Standard Operating Procedure for

Critical Point Dryer

Do not attempt to use this instrument until you have been ***trained by FMIC staff***

**Do not** operate the CPD unless there is an **EM center staff person available** (ie. in the FMIC) for the entire run time. Protective Equipment: lab coat, safety glasses, gloves

1. Place fully dehydrated samples into CPD sample holders in a Petri dish filled with 100% Ethanol
2. Turn on power, **wait 3 minutes** for CPD to warm up before use.
3. Turn Fill, Cool, Purge, and Bleed valves to Close (hand tight – do not use tools to tighten).
4. Open LCO2 (liquid carbon dioxide) Tank Valve.
5. Place 5-10 ml of 100% ethanol (using a plastic pipet – do not pour from bottle with desiccant) in the Sadri high pressure chamber.
6. Transfer sample holder into the chamber. Avoid exposing sample surfaces to air.
7. Dry any alcohol that may be in the O-ring groove. Examine rubber O-ring for defects, then place O-ring in groove..
8. Put lid on and evenly hand tighten the knurled knobs - using equal pressure on all three knobs.
9. Open **Cool Valve** and reduce the temperature of the chamber to near 0°C (the temp should drop to near zero in about 1 minute, if longer the LCO2 tank maybe empty).
10. Close the **Cool Valve**.
11. Open **Fill Valve** to fill the chamber with LCO2. A bubble (meniscus) will travel across the window indicating the chamber is full. Set **Fill Valve** to 0.75 turns – do not close.
12. Slowly open the **Purge**/ **Vent Valve**.
    1. **Warn people working in the hood that you will be venting the CPD**.
    2. Check the beaker in the hood next to the CPD to see if all the ethanol is exhausted.
    3. If the temperature of the chamber goes above **10°C** during purging of the alcohol, open the cool valve, this will not interfere with the purging mode, until the chamber is cooled to about 0°C, then close it.
13. Close the **Purge**/ **Vent Valve**. Open the **“Fill”** valve as needed
14. Watch as the “bubble” under the lid-window disappears, then set the **Fill Valve** to 0.25 turns
15. Wait 10 minutes.
16. Repeat the **Cool**, **Purge**, Fill and **Wait** (10 min.) steps until only CO2 is exhausted (ie. filter paper is not wet after purge).
    1. NOTE: Usually 2 - 3 changes of CO2 of 10 minutes each are needed to completely purge all the ethanol.
17. Wipe dry any moisture that may have condensed on the lid.
18. Fill chamber with CO2 then Close the **Fill** valve.
19. Flip heat switch to “On”
20. Heat light will go on. Close the **LCO2 tank valve**
21. Wait for temperature to reach 31°C – heat light may go on and off, do not turn off the heat switch
22. Set a timer for **4** minutes and wait as the liquid converts to gas.
23. After the 4 minute wait, use either “Bleed” or “Purge” valve to adjust pressure decrease rate to about 100 psi per minute.
    1. Note: Fast decompression could result in artifacts and CO2 “condensation”.
24. Allow at least 9 minutes for pressure to reach 350 psi.
25. Adjust **Purge**/ **Vent Valve** so that pressure reaches 0 psi in 2 - 3 minutes,
26. Loosen the three knurled knobs evenly by hand.
27. Remove sample and transfer it to a dry environment (vacuum dessicator) or sputter coat.
28. Remove O-ring, make sure the chamber is clean and O-ring is in good condition.
29. Check to be sure the valve is closed on the LCO2 tank.
30. Open all valves of the Samdri to “drain” them from any and all residual line.
31. Turn off heat and power switches.

Note: Samdri samples are extremely hygroscopic, place in vacuum dessicator at least 30 minutes to overnight to allow outgassing prior to sputter coating.