Instructions for Sample Drying at the Critical Point

Tousimis Critical Point Dryer, “Samdri-780A”

1. Make sure all valves (INLET, COOL, BLEED, and PURGE-VENT) are closed. (hand tight)

2. Place 5-10 ml of 100% ethanol (use newly opened bottle to ensure dryness, especially in summer) in the Samdri high-pressure chamber.

3. Transfer the sample holder into the chamber. Do this quickly, and make sure sample surfaces are not exposed to air.

4. Replace lid, “pip” side up, and evenly hand-tighten 3 retainer nuts to secure it.

5. Turn POWER/LAMP switch to ON. Note light comes on in chamber.

6. Open the CO2 cylinder valve, several turns. (NOTE: You may hear a hissing sound as CO2 escapes from around the O-ring seal of the cylindrical filter unit, but it should stop quickly as the O-ring saturates with CO2. This is normal for this type of O-ring. IF IT CONTINUES TO HISS, NOTIFY STAFF.)

7. Open the COOL valve a few turns. Close the valve when temperature approaches 0°C…should take about 60-90 seconds. Keep the temperature in the 0 -- 10°C range throughout the run. Reason for doing this is to help ensure that liquid CO2 remains liquid as it enters the high pressure chamber.

8. Open the INLET valve slowly. Watch the LCO2 enter the chamber and mix with the ethanol. Check the pressure gauge…it should read about 850-900 psi. When chamber is full and the pressure stabilized, open the INLET valve all the way.

9. Open the PURGE-VENT valve (to allow outflow of LCO2 just a few turns, until the outflow / purge rate is steady and less than the rate of LCO2 inflow.

10. Allow purge to run for 5 minutes for most samples…small pieces or single cells. For larger samples, it’s good to purge for 5 minutes, then close the purge valve and allow the samples to soak in the LCO2 for 3 minutes, then purge again for 3 more minutes. If you do the extended purge, though, be sure to note it on the log sheet. (IMPORTANT!! During purging the temperature will rise quickly. Open and close the COOL valve as needed to keep the temperature between 0°C and 10°C.)

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11. When purging is finished, close the valves in the following order:
   a. The COOL valve, if open.
   b. The PURGE-VENT valve.
   c. The INLET valve.
   d. The CO2 cylinder valve.

12. Turn the HEAT switch ON. The HEAT light will go on. After the temperature passes 31\(^\circ\)C, usually somewhere between 33 – 39\(^\circ\)C, the HEAT light should go off. Do not turn HEAT switch off.

13. Listen for the pressure maintenance valve to open, with a quiet hissing sound. This valve opens to maintain the system above the “critical point” of CO2. (approx. 1,200 psi and 31\(^\circ\)C) Once this valve opens, set the timer for 4 minutes.

14. After 4 minutes at the critical point, open the BLEED valve very slowly and carefully…it’s hard to control. Adjust to maintain a pressure decrease rate of about 100 psi / minute. Allow about 10-12 minutes to reach approximately 250 psi. The chamber heater will cycle on / off to maintain temperature above 32\(^\circ\)C.

15. When chamber pressure reaches 250 psi, open the PURGE-VENT valve all the way and decrease the pressure to 0 psi.

16. Loosen the 3 knurled chamber cover retainer nuts evenly, by hand. Never use tools.

17. Remove samples and store in a dry environment.

18. Turn the Heat and Power switches to OFF

19. Make sure the chamber is clean and the O-ring is in place and in good condition. Replace lid and attach retainer nuts, but do not tighten them as this will compress the O-ring and prevent a good seal next time it’s used.