

Operating procedure for Critical Point Dryer (Revised: 12/12/02).

Equipment Check

1. Ensure that the temperature reading is at 15°C (<20°C).
2. Unscrew door of critical point dryer chamber.
3. Clean the door, boat and rim with wipe using ethanol. Acetone may be used but the lifetime of the O-ring will be reduced.
4. Screw the door back in the critical point dryer.
5. Ensure all valves are closed.
6. Open valve of the CO₂ cylinder located at the grey area.
7. Open valve and check that liquid CO₂ is flowing. Just flow a little bit as this is just to check liquid CO₂ flow.
8. Open the bottom valve to let CO₂ out.
9. Close the bottom valve after all has liquid CO₂ drained out.

Sample loading and cycling

1. Make sure wipes are placed near the door and at the bottom valve. Make sure more are within easy reach.
2. Unscrew door of critical point dryer.
3. Immerse boat with sample in a large beaker containing the solvent e.g. ethanol or methanol. Acetone may be used for drying but it reduces the life time of O-ring.
4. Transfer sample to the boat while immersed in the solvent. If sample is small use a ladle.
5. Load boat into the critical point dryer chamber.
6. Screw the door back in the critical point dryer as far as possible. Ethanol would drain as soon as the boat is loaded.
7. Flow in liquid CO₂ as soon as possible. Open the bottom valve slowly to let ethanol to drain. Use a wipe to soak in the ethanol. Open the top valve slowly to let trapped air out. Ensure that ethanol is completely removed and that the liquid CO₂ meniscus is above the boat and hence covering the sample.
8. Flush the chamber for about 7-10 mins – bottom valve open and liquid CO₂ is flowing in. Ensure that ethanol is completely removed and that the liquid CO₂ meniscus is above the boat and hence covering the sample.
9. After flushing, close the bottom valve. Then close the top liquid CO₂ valve.
10. Start cycle as below:

Step	Set temperature (°C)	Observed Temperature(°C)	Expected Pressure (psi)	Duration between steps (mins)
1	30	~25 - 27	~1000	~ 20
2	35	~29 - 31	~1100	~ 10
3	40	~34 - 36	~1200	~ 10

11. After cycling is done, release pressure slowly until a pressure of 500 psi is reached. May speed up after that. Too fast a release burst cells!!
12. Unscrew door of critical point dryer.
13. Unload boat and remove sample.
14. Clean boat, rim and door of dryer.
15. Screw the door back in.
16. Close the valve of the liquid CO₂ cylinder at the grey area.
17. Vent the liquid CO₂ line by opening top valve.
18. Set temperature controller back to 15°C. It usually takes about 30-40 mins to get back to 15°C.