

Balzers Critical Point Dryer (CPD) 020

Operation Manual

It is recommended that the operator become thoroughly familiar with the CPD 020 before running the real samples.

LOAD:

1. Be sure all three valves (5, 7, 8 on the Figure) on the CPD 020 are closed.
2. Make sure there is no pressure (9) in the chamber. Unscrew the chamber lid (1). *The pressure gauge is glycerol dampened – a bubble is normally present.*
3. Fill the chamber with enough transferring liquid (ethanol, amyl acetate etc.), so that the baskets containing the specimens are just covered.
4. Rapidly transfer the baskets which contain the dehydrated sample to the CPD chamber. Screw down the lid. **HAND TIGHT ONLY!**
5. Turn on the MAIN switch (2).
6. Open the valve on the CO₂ cylinder all the way on.

COOLING

7. Set the chamber cooling temperature to +15°C (3).
8. Depress the TEMP button (4). The CPD will begin automatically cooling the specimen chamber to the selected temperature. When the temperature is reached, the CPD will automatically maintain it within $\pm 3^{\circ}\text{C}$, and exchange of the transferring liquid with the drying gas can begin.

EXCHANGE

9. ***Slowly*** open the gas inlet valve (5). Observe via the sight glass the filling of the specimen chamber with liquid. Notice the Schlieren line patterns that occur. This indicates the mixing of the fluids and signals the beginning of exchange.
10. When the specimen chamber is nearly full, close the gas inlet valve (5) and press the STIRRER button (6). This will ensure the mixing of the drying agent with the transferring liquid. Let the liquids mix for about 2 minutes.
11. Open the GAS OUT VALVE (7) one or more turns. Carefully open the METERING VALVE (8) and ***slowly*** drain the liquid from the specimen chamber while observing the level through the sight glass.
12. When the level of the remaining liquid is just above the specimen baskets (or specimen inside the baskets), close the METERING and GAS valves.
13. Repeat steps 9-12 several times (could up to 20 times), until all traces of the transferring liquid are gone. It is extremely important that the transferring liquid be completely replaced by the drying agent. Otherwise, the samples will not dry properly.

DRYING

14. When the transferring liquid has been ***completely*** washed out, fill the chamber ***halfway*** or to the point where liquid just covers the specimens inside the baskets, whichever is greater. To

prevent damage to the burst of cellular membrane of your samples, do **NOT** fill chamber completely.

15. Make sure that all valves are closed (including the CO₂ tank). Shut off the magnetic stirrer.
16. Set the TEMPERATURE selector (3) to +40°C. The CPD will automatically begin warming the specimen chamber. Notice also that the pressure within the chamber begins to rise. **If the pressure begins to exceed 120 bar, reduce the pressure to 80-85 bar with the gas out and metering valves. If pressure approaches 150 bar, the burst membrane will rupture, and your samples will be ruined.**
17. As the critical temperature and critical pressure are approached, the drying agent will begin to go from a liquid to a gaseous state (critical point).
18. To be assured that complete drying has taken place, wait until the critical temperature and pressure are exceeded.

Critical value for CO₂: 40°C/80-85 bar

19. Begin pressure reduction by opening the GAS OUT valve (7) and **slowly** opening the METERING valve (8). The METERING valve will allow you to precisely regulate the rate of pressure reduction. Pressure reduction should take about 15 minutes. If the gas within the chamber begins to recondense, you are reducing the pressure too quickly. **Exercise special care during venting to prevent condensation of the drying gas.**
20. Only when the pressure in the chamber is 0 bar can the cover be opened and the samples removed.
21. Slowly open the gas inlet valve (5) to release the remaining CO₂ in the tube. Close all valves and shut off MAINS.

At this point, the samples will be very hygroscopic. Care should be taken to prevent them from absorbing water from the atmosphere.

