SOP Leica Critical Point Dryer

1 Syfte (Aim)

The following guide helps you through the usage of the CPD process.

2 Omfattning/avgränsning (Extent/Demarcation)

There are no notes here about different faults that can occur during use, refer to machine manual or your own notes or contact system responsible if there are unknown problems

3 Ansvar (Responibilites)

Maskinansvarig: Thomas Frisk

4 Genomförande (Implementation)

Run the system with machine responsible until he/she knows you are fit to use the system on your own. You will then be a 'licensed user' and be able to book the system in the lab booking system.

5 Bilagor (Attachments)

n/a

6 Ändringslogg (Changes)

Fastställd version	Dokumentdatum	Ändring	Namn
1	2019-06-20	Dokumentet upprättat	Thomas Frisk
1.2	2019-08-06	Dokumentet kompletterat.	Thomas Frisk
1.3	2019-08-06	Kompletterat med avsnitt om tömning av exchange bottle och kompletterat med illustrationer.	Thomas Frisk
1.4	2019-08-13	Added failure modes.	Thomas Frisk

Introduction

Samples sensitive to liquid forces are loaded, while wet, into the chamber, which is filled with liquid CO_2. A series of exchange cycles remove the original liquid and replace it with the liquid CO_2. Once the substitution is complete, the chamber is heated. This will raise the internal pressure, taking the CO_2 through its critical point. Once the critical point has been passed, the pressure is slowly released through the vent valve, and the samples can be removed.

Operation of the CPD System

Start-up of the system

1. Open the CO_2 valve in the service corridor behind the machine.
2. Turn on power with switch at back of machine (right hand side, black).

Loading procedure

1. Open the process chamber lid (unscrew it counter clockwise)
2. Remove sample holder beaker.
3. Make sure the magnetic stirrer is in place (small white plastic stirrer).
4. Pour some ethanol in the chamber, to approx. 1.5 cm height.
5. Transfer your sample to a sample holder (stored in the drawer at the front of the machine).
6. Put the sample holder “plug” in the bottom of the sample holder beaker.
7. Put the plastic spacer/distance (with hole) in a petri dish with concave side upwards.
8. Put the sample holder beaker on the spacer in the petri dish.
9. Pour some ethanol in the sample holder beaker.
10. Put the sample holder in the sample holder beaker.
11. Add “fillers” to 75-95% of the beaker volume.
12. Put the sample holder beaker into the process chamber.
13. Turn on the light in the chamber.
14. Add some more ethanol so that you can barely see the liquid meniscus in the window.
15. Close the lid (screw it on through clockwise rotation until it is reasonably tightened, mind you, it is made out of stainless steel, thus sensitive to overtightening!)

Liquid exchange procedure

1. Choose an appropriate program for your processing through pressing the button marked Programs
2. Modify the program according to your process needs.
3. Modify the Holder Filler ratio to appropriate configuration.
4. Return to main menu through press the button marked Main
5. Start the process through pressing the button marked Start
6. Initial cooling takes some time (5-8 minutes)

7. Some sputtering might be heard if there is gas or air in the CO_2 line.
8. Keep an eye on the pressure (Pc) as it should go up and down around the 55 bars
9. Keep an eye on the temperature (Tc) as it should stay below 15degC during the exchange cycles.
10. Some additional noise will be heard when the machine lets out the ethanol-CO_2-mixture.
11. There will be a smell of alcohol in the vicinity of the machine during the exchange cycles.
12. Keep an eye on the pressure changes during the final temperature ramp. The machine will have to let out some CO_2, but you don't want the pressure to go below 74.8 bars.
13. Make note in log book!

Unload procedure

1. When the machine indicates that it is done with the process (Yellow window indicating: Process Complete! Remove Samples Ready for next run), it is time to unload.
2. Open the process chamber lid (unscrew it counter clockwise)
3. Insert the holder lifter rod handle.
4. Lift the holder out from the process chamber.
5. Remove the fillers and sample holder.
6. Close the lid in order to keep the chamber clean.

In order to avoid contamination, don't leave the chamber open, exposing it to ambient air.

Shut down procedure

1. Close the CO2 valves in the service corridor
2. Shut off the power with the switch at the back of the machine.
3. Remove the separator bottle (press white bar under bottle to release) and empty it. Wipe dry the separator bottle holder of the machine.

Failure modes

- IPA (propanol) seems to yield residues. Use ethanol instead!
- Too few exchange cycles seems to yield residual solvent in chamber and on chip.
- Running the machine "from scratch" after bottle exchange gives poor performance.
- Beware of the humidity in the lab. It is uncontrolled, and especially bad late summer.
- Using the "plug" in the bottom of the sample holder beaker reduces the exchange greatly. Longer process time required.
- Beware of the pressure drop during final heating. Pressure should nog go below 74.8 bar.
- If chamber lid is not tightened correctly, gas will leak out. Stop process and re-tighten!
- If holders and fillers show signs of dirtiness, clean in ethanol and ultrasound bath!

Attachment

Process flow chart