

Critical Point Dryer

Standard Operating Procedure

4D LABS Confidential

Revision: 1.0 — Last Updated: Jul.27/2011, Revised by Nathanael Sieb

Overview

This document will provide a detailed operation procedure of the Tousimis 815B Critical Point Dryer. Formal Training is required for all users prior to using the system.

Revision History

#	Revised by:	Date	Modification
1	Nathanael Sieb	07/27/11	Initial release
2			
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5			

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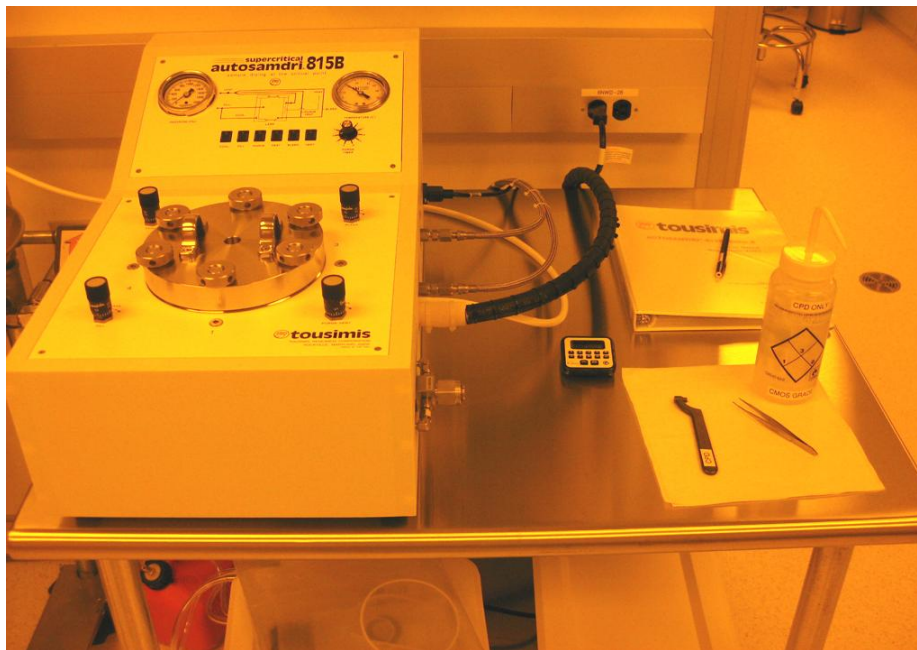
General Information

The Critical Point Dryer (CPD) is used to avoid stiction and surface tension problems when drying fragile samples. Before using the CPD, all samples must be dehydrated in isopropanol (IPA) and COMPLETELY free of acid. Any residual acid will damage the CPD and any residual water will damage the sample. When preparing the sample, rinse it thoroughly and exchange the water with IPA *at least* 3 times for 5 minutes each.

The basic operation of the CPD is as follows:

1. Immerse sample in IPA in the chamber.
2. Cool the chamber with liquid CO₂.
3. Fill chamber with liquid CO₂.
4. Continually replace IPA/CO₂ mixture with pure liquid CO₂.
5. Heat the chamber past the critical point of CO₂ (31 °C, 1072 psi).
6. Vent the gaseous CO₂.
7. Remove the sample.

The rest of this SOP will describe how to operate the tool.



Operation

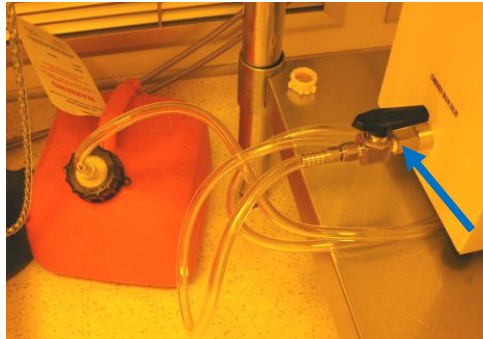
1. Sign in on the tool log book.
2. Turn the condenser power on (located under the table) and wait 5 minutes.



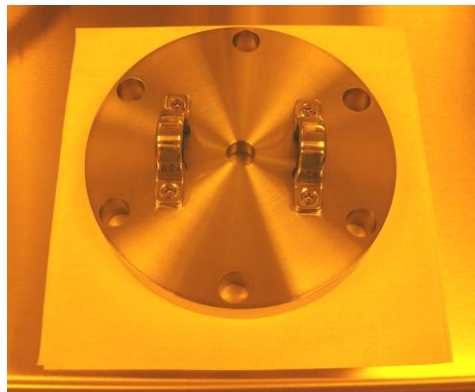
3. Turn the main power on for the Critical Point Dryer (CPD). The Vent light will turn on.



4. Make sure that the drain valve is open (parallel to tubing).



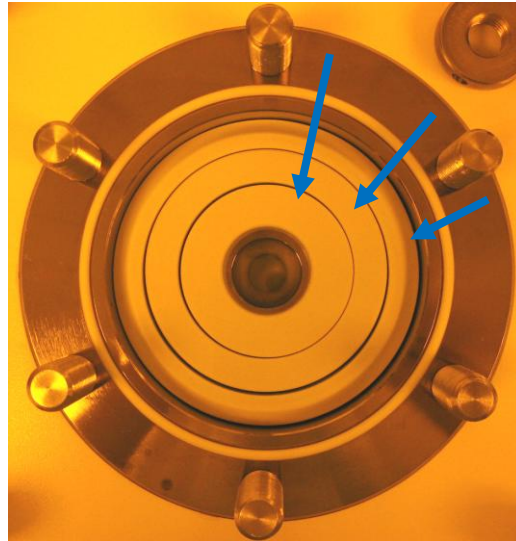
5. Remove the lug nuts and place the chamber lid on the clean wipes.



6. Place wet sample into an appropriate sample holder.



- Place inserts into chamber as needed to reduce total chamber volume.

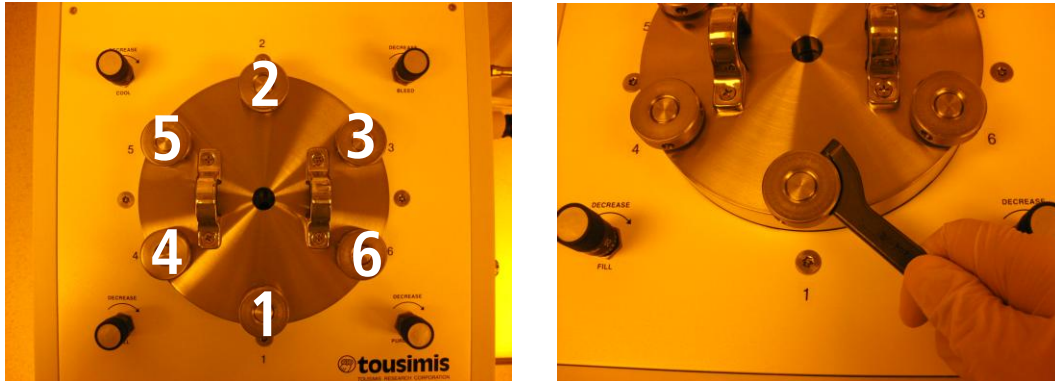


- Press Vent once to close the purge valve. The Vent light will begin to flash.



- Add enough isopropanol (IPA) into the chamber to completely cover the sample in the next step.
- Place sample holder into the IPA-filled chamber.
- Put the chamber lid back on.
- Reattach the lug nuts.

13. Partially tighten the lug nuts in a 'star pattern' using the special wrench. Continue repeating the star pattern until the lug nuts are completely tight.



14. Press Cool. The user will hear the liquid CO₂ (LCO₂) entering the chamber walls and the gas exiting into the exhaust. The system will take ~2 minutes to cool to 0 °C.



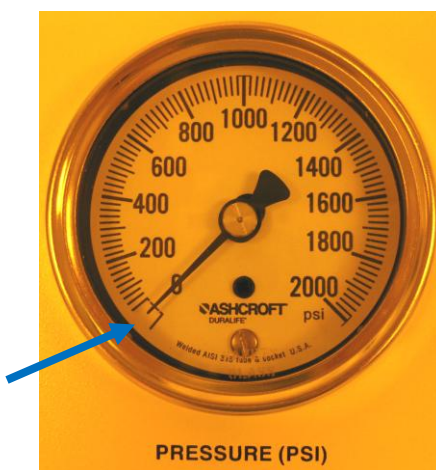
15. Ensure that the purge timer is set to the desired value (time = setting * 5 minutes). Setting 2 (10 minutes) is a typical value.



16. Press Fill. The rest of the sequence will take place automatically as follows:

- a. During the Fill cycle, LCO₂ enters the chamber and mixes with the IPA. The squealing noise is normal as the pressure equilibrates to ~800 psi. The fill cycle lasts for 8 minutes and then the system moves to purge.

- b. During the purge cycle, pure LCO₂ enters the chamber while the LCO₂/IPA mix exits the chamber to the drain carboy. The purge cycle lasts the duration of the time set earlier in step 14 (e.g., 10 minutes).
 - c. During the post-purge fill, the fill and purge lights are lit. This step lasts 4 minutes while the chamber is filled with LCO₂.
 - d. The heat cycle takes ~20 minutes while the chamber is heated to ~38 °C and the LCO₂ pressure increases to ~1360 psi. The pressure will be reached before the temperature, and excess pressure will bleed off via the pressure relief valve.
 - e. When the system reaches the defined critical point ($P > 1200$ psi, $T > 36^{\circ}\text{C}$), the heat light flashes and the CPD waits for 4 minutes before proceeding to the bleed cycle.
 - f. The bleed cycle slowly vents the system until the pressure reaches ~400 psi (~20 minutes).
 - g. At 400 psi, the system enters the vent mode and complete vents the chamber.
17. When the system has reached 0 psi, the chamber may be opened. Remove the lug nuts in a 'star pattern'.



18. Place the chamber lid on the clean wipes.
19. Remove the sample from the chamber and clean the chamber out with IPA.
20. Replace the chamber lid and place the lug nuts loosely on the system.
21. Turn off the main system and condenser power.
22. Sign out on the tool log book.



References and Files

Critical Point Dryer Manual and training notes.

Contact Information

Questions or comments in regard to this document should be directed towards Nathanael Sieb (sieb@4dlabs.ca) in 4D LABS at Simon Fraser University, Burnaby, BC, Canada.