



Lab Manual

[Marvell Nanolab](#)[Member login](#)[Lab Manual Index](#)[Mercury Web](#)[Berkeley Microlab](#)

Tousimis 815 Critical-Point Dryer (small samples)

(cpd2)

1.0 **Title**

Tousimis Model 815 **Autosamdri** CO₂ critical-point drying system

2.0 **Purpose**

This document has specific information about the capabilities, configuration, and proper operation of the Model 815 critical-point drying system

3.0 **Scope**

3.1 The Autosamdri-815 (cpd2) is a fully automatic CO₂ critical point drying apparatus. Its front panel consists of a pressure gauge, a temperature gauge, a purge timer knob, and six process push buttons (COOL, FILL, PURGE, HEAT, BLEED, and VENT) with green LEDs. Only COOL and FILL buttons need to be pushed for a complete process. The unit can also operate under semi-automatic mode, but it is not recommended for the inexperienced users. The sample holders for cpd2 are 1-inch in diameter.

3.2 On the flat panel of the unit, there are two handle screws that control the FILL and PURGE/VENT flow. They are factory set. DO NOT ADJUST THE VALVES.

3.3 If you feel that the PURGE flow rates have been altered; there is an easy to adjust via a **Dry Run** (that is without any alcohol or sample. This way you can visualize the flow of the LCO₂ through the chamber via the chamber window:

3.3.1 Make sure that both FILL and PURGE meter valves are free to turn by making sure that both 1/32" hex head screws are backed off and loose so you may freely turn the valves.

3.3.2 Turn CPD on.

3.3.3 Set Purge Timer to pos# 2 (10 min.) to allow for enough time to comfortably make the following adjustments.

3.3.4 Press COOL.

3.3.5 Press FILL after COOL shut off so that you may visualize the incoming LCO₂ from the left hand side of the chamber. The LCO₂ will look like a steady stream entering the chamber. As the chamber level fills with LCO₂; the stream will disappear.

3.3.6 After the 2 minute FILL mode; the process will advance into the PURGE mode. This is where the most critical adjustment is necessary.

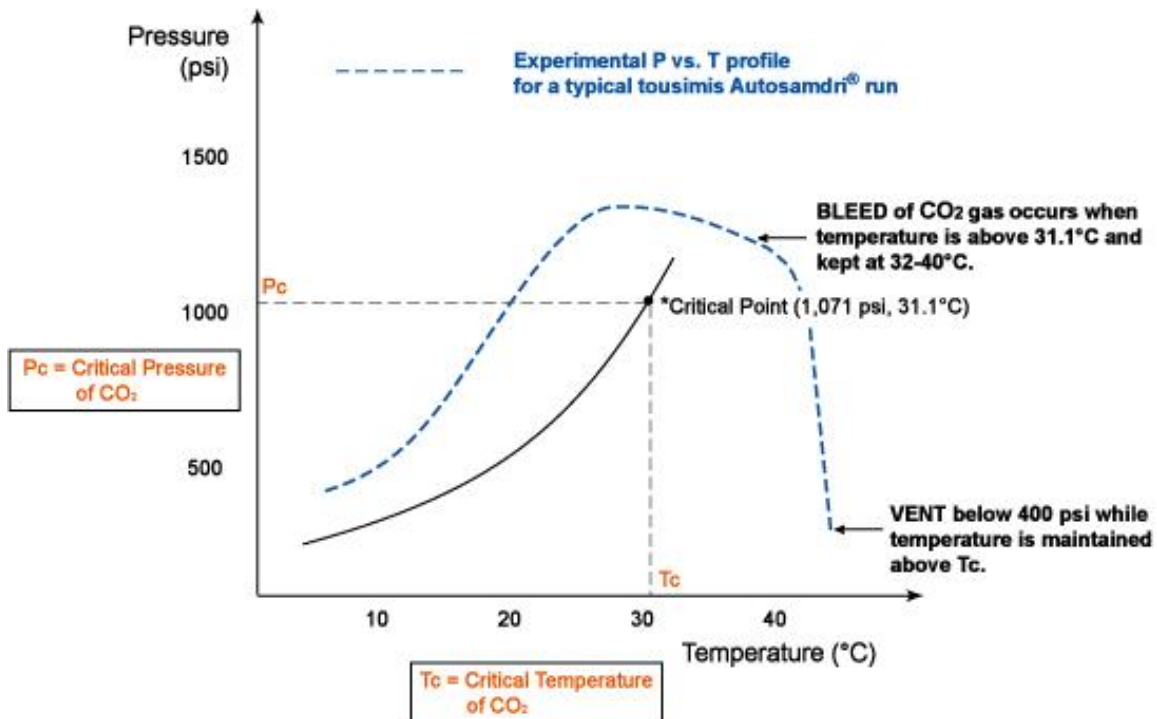
[It is essential that the incoming FILL rate exceeds the outgoing PURGE rate so that the LCO₂ level does not drip below the level of the devices being dried. You also; want to be sure that you have steady flow so that exchange of alcohol for LCO₂ occurs. Therefore; the PURGE meter valve is adjusted one way another until you see movement from left to right within the chamber (indicating flow) until the 1st hint of small bubbles are evident. Stop at this point. This is the point where you have good flow; yet the LCO₂ level is not dropping.]

3.3.7 Let the 815 process complete its course automatically

4.0 Applicable Documents

[Revision History](#)

Figure III. Pressure (P) vs. Temperature (T) Graph For AUTOSAMDRI®



* At critical point, the liquid CO₂ transforms into gaseous CO₂ and above critical point isotherm, it is always a gas. Below this point, it can exist as a liquid, gas or both.

© 2002 tousimis

[Verified Temperature Pressure Graph of Microlab CPD Process](#)

5.0 Definitions, Process Terminology, Nomenclature

- 5.1 CPD: critical-point drying (dryer)
- 5.2 LCO₂: liquid carbon dioxide

6.0 Safety

- 6.1 The CPD system normally operates under very high chamber pressures. DO NOT DEVIATE FROM THE CORRECT PROCEDURE FOR LOADING THE SAMPLE AND SECURING THE CHAMBER LID – personal injury is quite possible otherwise.
- 6.2 An uncontrolled release of CO₂ can cause personal injury due to frostbite. Again, DO NOT DEVIATE from the correct procedures. Make very sure that persons near the system are always wearing protective eye ware.

7.0 Statistical/Process Data

8.0 Available Processes, Process Notes

- 8.1 CPD uses copious amounts of LCO₂. Requests for LCO₂ cylinder changes are the primary CPD problem report and result in decreased tool availability for all lab members. Review the following Process Notes and attempt to minimize your LCO₂ usage.
- 8.2 Extra long purge time does NOT increase the effectiveness of your critical point drying process; it just wastes LCO₂.
- 8.3 Minimize the amount of alcohol you add to the chamber. You only need enough alcohol to just cover your parts.
- 8.4 Maximize the volume of the CPD chamber filled with Teflon inserts. The more Teflon in the chamber, the less alcohol in the chamber, and the less LCO₂ needed.

Most of the LCO₂ is consumed during the PURGE process. Each number of the PURGE dial indicator corresponds to 5 minutes of flowing LCO₂; i.e., setting 1 = 5 min, setting 2 = 10 min, setting 4 = 20 min, etc. The goal of the purge process is to replace essentially all the methanol/IPA in the chamber with LCO₂. Since methanol and IPA are especially soluble in LCO₂, there is NOT a linear relationship between purge time and alcohol removal. Most alcohol is removed from the chamber after only one LCO₂ volume exchange. Once the alcohol has been removed, the LCO₂ can be heated to supercritical conditions and a phase change from liquid to vapor can take place without crossing a liquid/vapor equilibrium line and generating the surface tension forces that result in adhesion of released MEMS parts. (See Section 4, [Pressure vs. Temperature Graph for CPD Process](#)).

Process staff has verified that there was no remaining alcohol and therefore a completely effective CPD process was performed with the following samples and volumes of alcohol.

Sample Holder	Teflon [®]	Steel	None
Amt Methanol	10 ml	17 ml	25 ml
Purge Setting	1	1	2
Purge Time	6.5 min	6.9 min	11.17 min
Cool CO ₂ Usage	0.5 lb	0.5 lb	0.5 lb
Chamber CO ₂ Usage	1.5 lb	1.0 lb	3.0 lb

A purge setting of 1 is sufficient for most needs and there should not be any need for a purge setting greater than 2.

CPD is often one of the last steps of a lengthy MEMS process. Users are wary to risk their parts to an ineffective CPD process. If you would like to verify that a lower purge time is suitable for your process – run a dummy process one time. Add a non-critical sample and just enough alcohol to cover the sample. Use the recommended minimum purge time. Verify that the chamber is absolutely dry (no residual alcohol) at the end of the process. If the chamber is dry, you have performed a fully effective CPD process.

If you ever observe residual alcohol in the chamber using the minimum recommended times, report it as a CPD problem on the wand.

Important Note: Any residual HF remaining on your samples from previous steps can easily damage the **High Pressure Sensor** on the cpd tool. Therefore, extra care must be taken to thoroughly rinse off your part(s) before entering the cpd chamber.

9.0 Operation

9.1 Preparation

- 9.1.1 Before starting to use cpd2, the user should check the liquid CO₂ cylinder weight. If the weight is 25 lbs (or more) below its full weight, report a fault on the WAND. This will give

the staff adequate time to change the tank. Typically, each run consumes approximately 1 lb and a full tank provides 30 lbs of CO₂ according to the vendor. Therefore, the cylinder has to be changed before 30 lb has been used up.

- 9.1.2 Clean samples carefully. Photoresist residue may re-deposit on the dried samples or the chamber walls.
- 9.1.3 Make sure your samples are sufficiently rinsed after a wet process that involves HF and/or other acid treatments, before entering the cpd chamber.
- 9.1.4 The user should also decide on the purge time beforehand and set the purge timer knob accordingly. A setting of 2 indicates 10 minutes, a setting of 3 indicates 15 minutes, and so on to a maximum of 30 minutes. See Section 8 for Process Notes regarding appropriate Purge times.

9.2 Operation

- 9.2.1 Check for reservations on the WAND for cpd or cpd2. The two systems share one CO₂ tank; therefore they cannot be used simultaneously. Likewise, if either of the systems is enabled already, the **ON** indicator above the system (on the front of the hood) will be illuminated.
- 9.2.2 Enable cpd2 on the WAND. Note the **ON** indicator at the front of the hood lights up.
- 9.2.3 Ensure that the three-way manual valve on the front of the hood points toward **CPD2**. If not, rotate the handle 180° until it does.
- 9.2.4 Verify that the cylinder valves of both liquid CO₂ cylinders are open. Check the weight of the cylinders. Both liquid CO₂ cylinders are located behind the tools on the other side of the 432B wall. One is labeled **CHAMBER CO2** and the other, **COOL CO2**. Report a fault on the WAND if the weight is 20 lbs or more below its full weight.
- 9.2.5 Record the weight of both CO₂ cylinders (see 9.2.18 below).
- 9.2.6 Clean the chamber and lid using methanol/IPA and dry with N₂ gun. Repeat several times if necessary. Use Techni-cloth cleanroom wipes.
- 9.2.7 Let unit warm up for 3-5 minutes. This will stabilize all plumbing components, the metering valves, and the Static Pressure Control module.
- 9.2.8 Press the COOL button. The LED on the button will go on (VENT LED will go off). Now place only enough Methanol or IPA into the chamber to cover your samples.
- 9.2.9 Place your sample(s) in the chamber. There are three steel sample holders available, and one Teflon sample holder for 10mm samples. The sample holders are stored in a drawer below the cpd systems. Alternatively, some users have custom sample holders, and in fact the system can be used without any sample holder at all, by placing the sample on the bottom of the chamber.

Be careful to keep the surface of the sample wet while transferring it into the chamber. Practice and experience are very helpful here.
- 9.2.10 Place the lid on the chamber. The side with the **tousimis** stamp should face up. Make sure that the O-ring is in place. Finger-tighten three knurled nuts evenly. DO NOT USE A WRENCH OR OTHER TOOLS. The unit will go on cooling by itself until slightly below 0°C. If it takes more than 2 minutes to reach 0°C, the liquid CO₂ cylinder may be depleted.
- 9.2.11 Once the cooling reaches ~0°C, press the FILL button. The unit will start filling the chamber with liquid CO₂. The user can see a visible liquid flow through the fill port. If not, the liquid CO₂ cylinder may be depleted. Now the unit is in the AUTOMATIC mode. It is not necessary to push any more buttons till the process has completed.

- 9.2.12 After 2 minutes, the unit switches to PURGE Mode automatically. The PURGE LED will be on. The Methanol/IPA is exchanged with liquid CO₂. The PURGE time depends on the setting of the timer knob the user set before the process starts. **See Section 8 for important notes on PURGE time settings.** There should be no liquid coming out the chamber at the end of PURGE time. The unit will switch to the HEAT mode. The HEAT LED will be on.
- 9.2.13 When the pressure reaches 1250 psi and the temperature is above 31°C, the Tousimis Equilibrium cycle starts. At this point the HEAT LED will be blinking for 4 minutes.
- 9.2.14 At the end of the Tousimis Equilibrium, the unit switches to BLEED cycle and the BLEED LED will be on. The Bleed flow should be around 7 SCFH for pressures between 1,200 and 400 psi. The temperature should be above 31°C.
- 9.2.15 There may be a hissing or cracking sound in the BLEED cycle. It is normal because gaseous CO₂ or dry ice may be coming out the unit.
- 9.2.16 When the pressure drops below 360 psi, the unit switches to VENT mode. VENT LED will be on. Do not try to loosen the nuts until the chamber is completely vented. It will damage the threads on the nuts and the chamber. After 3 to 4 minutes, the chamber will come to the atmospheric pressure. NOW the user should be able to loosen the knurled nuts with fingers. It is time to open the chamber and remove your samples.
- 9.2.17 Clean and blow-dry the chamber and lid with methanol/IPA if you see any residue. Close the lid to keep the chamber clean and moisture free.
- 9.2.18 Leave the cylinder valves of both liquid CO₂ cylinders open. Report the initial and final cylinder weights on the WAND comment when disabling cpd2. If either of the final cylinder weights is below 20 lb of the full weight, report fault on the WAND also.

10.0 Troubleshooting Guidelines

11.0 Qualification Procedures

- 11.1 Potential new users should arrange *ahead of time* to have an existing user review cpd2 operation. Casual questioning (e.g. **Hey, can you review this with me for a moment?**) is not to be considered an official review period.
- 11.2 The first question that should be asked is whether Section 2.7, **Tousimis Critical Point Dryer** of the WAND has been thoroughly reviewed? If not, insist that it be reviewed before the meeting.
- 11.3 The **meeting** between the current and new user should be scheduled for enough time to explain equipment operation, load & unload sequence, etc.
- Equipment time can be recharged to new user's account if necessary.
- 11.4 Step 11.3 should be done at least twice by two different current users.
- 11.5 Superusers will first ask: **Who trained you?** of the new user. **Training** implies adequately following Steps 11.1-4 above. The users identified as being the ones who did the training will be responsible for the information conveyed by the new user during the qualification.