Tousimis Model 810 Autosamdri

Standard Operating Procedure

Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)
Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)
(617) 627-3246 (From off-campus or from a cell phone)
Tufts Emergency Medical Services are at x66911.

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The purpose of critical point drying is to dry out a sample without introducing large forces on the structure due to phase boundaries (i.e. surface tension) in small gaps in the structure. This is critical for MEMS devices which often incorporate mechanical structures with small gaps and are sometimes released with a final wet etch. In that case, when drying the final solvent (usually methanol or isopropanol) out of the gaps, the structure may stick down if it does not have sufficient mechanical stiffness to overcome the capillary forces.

By using critical point drying, this problem can be reduced. For additional information, see:

Kim, Chang-Jin, John Y. Kim, and Balaji Sridharan. "Comparative evaluation of drying techniques for surface micromachining." *Sensors and Actuators A: Physical* 64.1 (1998): 17-26.

Mastrangelo, C. H. "Adhesion-related failure mechanisms in micromechanical devices." *Tribology Letters* 3.3 (1997): 223-238.

1.0 Material Requirements:

1.1 Personal Protective Equipment: safety goggles, nitrile gloves
1.2 1000 mL glass beaker to catch effluent (alcohol and CO_2)
1.3 Plastic carboy is used to catch and diffuse the CO_2 vented CO_2. The carboy will be in the fume hood.
1.4 PTFE dipper basket from Tousimis.

2.0 Safety:

2.1 Under normal operation, the process chamber will hold pressures as high as 1500 psi (100 atmospheres). Extreme care must be taken to ensure that the lid is properly fastened. Under no circumstances should the lid be opened while the chamber is under pressure.
2.2 CO_2 vapor and alcohol vapor are exhausted from the tool during the process. These vapors should be exhausted into the fume hood, as described below.
2.3 Some components will become cold due to expansion of liquid CO_2. These include the COOL hose and the surface of the process chamber.

If at any time you feel a situation is dangerous, turn off the CPD and close the CO_2 tank valve. Contact laboratory staff. Remember this is a very high pressure system.

Figure 1 - Front panel of Critical Point Dryer (CPD)

3.0 Setup (IMPORTANT):

3.1 As shown in figure 1, there are two needle valves that control fluid flow rate within the unit. The inlet adjust varies the rate at which CO_2 is introduced during purge, and the purge adjust varies the rate at which the mixture of CO_2 and alcohol is vented from the chamber during the purge step. *Do not adjust them as they have already been set for operation.* Setting these two rates appropriate is important to achieve a good result.

3.2 If you feel that they have been altered, there is an easy way for them to be readjusted:

- **3.2.1** Make sure the CPD is turned off, and turn both screws counterclockwise until they are closed (gently does it).
- **3.2.2** By inspecting the side of each screw, identify their zero position.
- **3.2.3** Open the INLET ADJUST screw exactly 5 full rotations.
- **3.2.4** Open the PURGE-VENT ADJUST screw 2 full rotations and continue until the number 15 is at the zero position.

4.0 Operation

4.1 Pressure vs. temperature profile for a typical run

The concept of critical point drying is that at high pressure and temperature (above the critical point of CO_2 – the CO_2 is called supercritical) there is no gas-liquid line … thus no phase change, and no surface tension. What is required is to start from standard lab conditions (20 ^oC, 15 psi), where the sample is bathed in alcohol, move to a state where CO_2 is liquid ("COOL" and "FILL"), purge the sample ("PURGE") to replace the alcohol with liquid CO_2, then move above the critical point into the supercritical phase ("HEAT"), and finally come back down to standard conditions ("BLEED" and "VENT") without ever crossing the liquid/gas line.

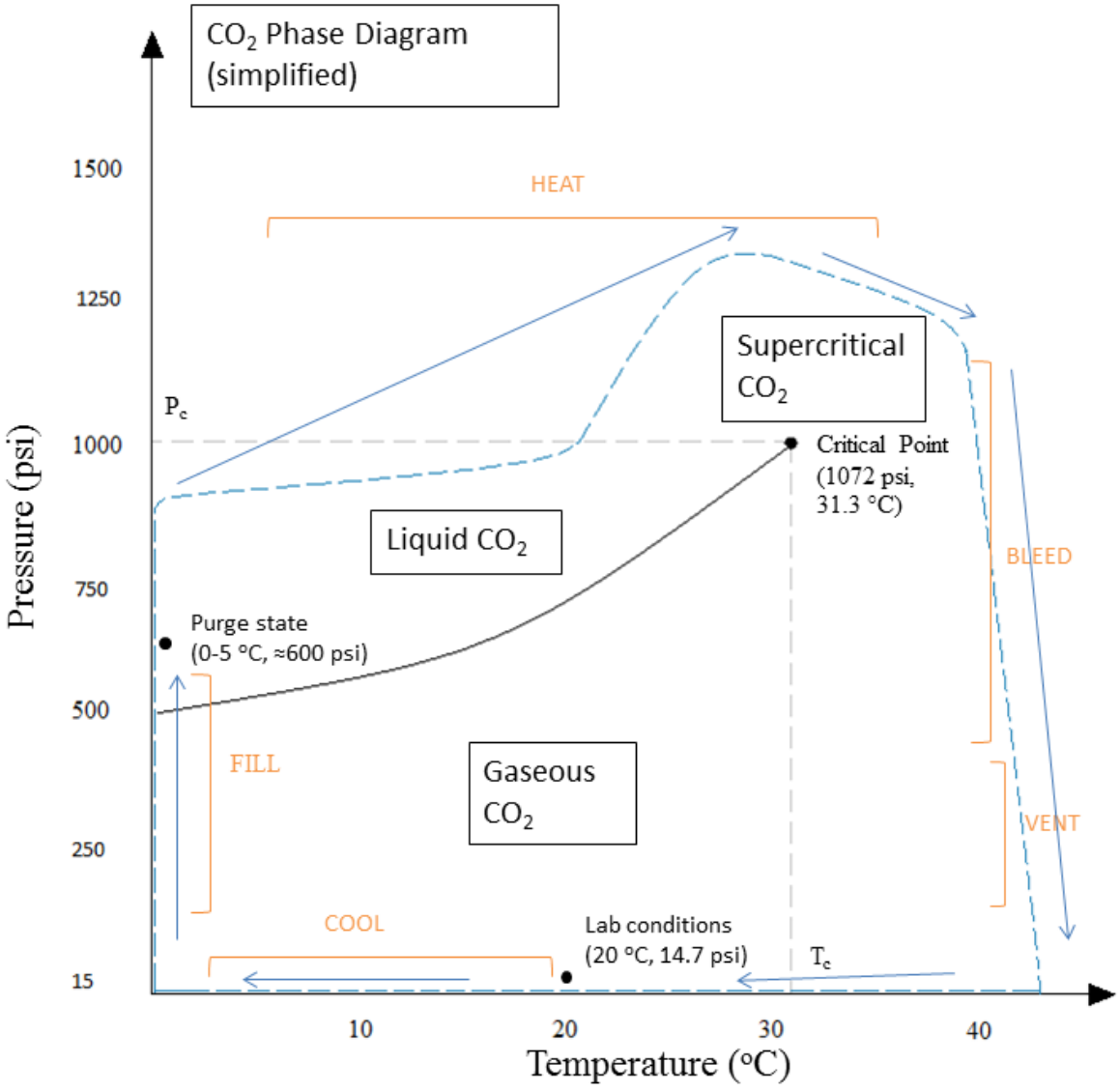

Figure 2 – Simplified phase diagram showing the concept of critical point drying as a path through the temperature-pressure plane.

4.2 Setting up

4.2.1 Make sure the white COOL and clear PURGE-VENT BLEED hoses are leading to the carboy, and the carboy is placed in the fume hood. The cap to the carboy should be left OPEN. See Figure 8.

4.2.2 Make sure the PURGE-VENT BLEED hose leads to a beaker 1000 mL glass beaker, also in the fume hood, to catch any purged liquids.

4.2.3 Turn on the autosamdri-810 by pushing the power switch. The POWER and PURGE lights should illuminate. See Figure 3 and 5.

4.2.4 Leave the unit turned on for at least five minutes before continuing with processing.

4.2.5 Push the button labeled Auto/Man so that "Auto" shows on the top – you are now in automatic mode. This SOP assumes you are operating in automatic mode. See Figure 7.

4.2.6 Open the CO_2 tank valve at the tank.

4.2.7 Measure weight of the tank (in lbs) and record total in the 'start' weight section of the log sheet.

4.2.8 **If the tank weight is 155 lbs or less, the tank needs to be changed.** This is for a standard 200-sized bone dry CO_2 siphon tank from airgas. When it arrives new from airgas it will weigh about 195 lbs (the empty tank is maybe about 130 lbs). Past experience says that if the weight gets below 155 lbs., the process will not run correctly – perhaps the dip tube is at or close to the surface of the liquid CO_2.

This is a bone dry CO_2, siphon tank. That means there is very little water vapor in there, and there is a dip tube that runs down to the bottom of the tank, so liquid CO_2, not gaseous CO_2, is extracted.

Since this is a liquid CO_2 tank – the pressure in the tank will always be around 800 psi; as the CO_2 is used up, more CO_2 will vaporize, maintaining the equilibrium vapor pressure of CO_2 in the tank at 800 psi (at 20 ^{o}C). Therefore, you cannot tell how much CO_2 is left in the tank based on the pressure in the tank. That is why we have the scale – it can tell you when the tank is going to run out.

4.3 Loading a sample

4.3.1 Remove the nuts from the chamber lid and remove the lid. Place the lid on a cleanroom wipe. See Figure 3.

4.3.2 Fill the chamber with ultra-pure methanol. The chamber should have sufficient alcohol in it to cover the sample. *Do not completely fill the chamber.*

4.3.3 Carefully transfer your wafer into the process chamber. In most cases you will want to have your sample held in a PTFE container – for MEMS processing we usually use a 5 chip 1 cm die holder from Tousimis.

4.3.4 Close the chamber and tighten the three nuts uniformly by hand. *Never use a wrench to tighten the nuts.* Refer to figure 3 to make sure lid and screws

have been properly fastened. *Make sure you don't have anything upside down!!!!*

4.3.5 Set the purge timer to 10 minutes (make sure switch is pressed to show that you are in the 0-15 minute range, and that the dial is set to 10). See Figure 6 and 7.

4.4 Critical Point Dryer Operation

4.4.1 Press ADV (advance). The autosamdri-810 will move into the cool step (see figure above), and cool the chamber to 0 °C (±5 °C) and will automatically stop. Record the cooling time (using a stopwatch). A typical cool time is 15-30 seconds.

4.4.2 Press ADV (advance). The autosamdri-810 will enter the FILL cycle. The process chamber will now fill with CO_2. During fill, the chamber temperature is automatically maintained at T < 10 °C. You may periodically hear the cooling process turn on and CO_2 will vent through the cool tube into the hood. This is normal. During this step, the pressure in the chamber should increase to approximately 800 psi. After two minutes, the autosamdri-810 will automatically advance to PURGE mode.

4.4.3 The autosamdri-810 will now enter PURGE mode. In this step, the alcohol is forced out of the chamber and is replaced completely by liquid CO_2. During this step, the FILL and PURGE lights should be on. Monitor the chamber temperature (check that the chamber temperature remains in the range –5 °C < T < 10 °C). The pressure within the chamber should be approximately 600 psi. This ensures that the CO_2 remains in the liquid state. (*Note the autosamdri-810 will perform cooling automatically during the PURGE mode)*. During purge you will notice:

4.4.3.1 For the first few minutes the exhaust from the clear PURGE-VENT-BLEED hose will be clear liquid or slurry. This is the alcohol being purged out. Make sure it is exhausting into a beaker in the fume hood.

4.4.3.2 After all the alcohol has been purged from the chamber, only CO_2 will be exhausted. Because the CO_2 encounters a low-pressure environment (atmosphere) as it leaves the high-pressure chamber, it will cool and be exhausted as clear white flakes. You can check to see if there is any alcohol in the exhaust by holding a cleanroom wipe at the end of the PURGE-VENT BLEED hose. Solid CO_2 will not leave a wet spot on the cleanroom wipe, but alcohol will.

4.4.3.3 The PURGE-VENT-BLEED hose may form condensation and/or ice due to the temperature drop as CO_2 exits the chamber. Wipe any condensation from the PURGE-VENT-BLEED hose with a cleanroom wipe to keep it dry.

4.4.3.4 During purge you will also notice 'oily currents' in the chamber. This is mixing between the alcohol and CO_2 or perhaps density variations in the liquid CO_2.

4.4.4 After 10 minutes (the time set by the purge timer), the autosamdri-810 will automatically advance to the HEAT cycle. The chamber should be

completely filled with liquid CO_2. If not, press the FILL button again to ensure complete filling of the chamber. During the HEAT cycle, record both temperature and pressure in increments of one minute. During the heat cycle you will notice:

4.4.4.1 Gradual increase in temperature and pressure of the chamber as the chamber moves up to temperatures and pressures above the critical point (see diagram above).

4.4.4.2 The critical point for CO_2 is at T = 31.3 °C and P = 1072 psi. The autosamdri-810 has an automatic pressure valve that maintains the maximum pressure between 1200-1500 psi during heating.

4.4.4.3 Schlieren patterns: oily currents in the chamber caused by density variations of the CO_2 in the chamber.

4.4.5 Next, the autosamdri-810 will automatically advance to the BLEED cycle (if 8-10 minutes have gone by above critical point and device hasn't advanced, press ADV). Record T and P in 2-minute intervals. *The chamber temperature will automatically be kept above the critical temperature T = 31.3 °C.* The chamber pressure will gradually drop at a rate of around 50 psi/minute.

4.4.6 Once the chamber pressure is below 400 psi, the autosamdri-810 will automatically go to the VENT cycle. If 5-7 minutes have gone by with a pressure reading < 400 psi, press ADV. The BLEED light should turn off and the PURGE light should turn on. The chamber should reach atmospheric pressure in 3-5 minutes.

4.4.7 Once atmospheric pressure has been reached, unscrew the nuts *by hand*, open the chamber and remove your sample. If the nuts feel too tight, then the pressure within the chamber might still be too high. Wait 5 minutes and try again. Close the chamber immediately to prevent moisture from building up in the chamber.

4.4.8 Record the final CO_2 tank weight. Compute the CO_2 used in this run and record it.

4.4.9 Close the CO_2 tank valve.

4.4.10 Leave the autosamdri-810 in VENT mode for a few minutes to exhaust any remaining CO_2 in the system.

4.4.11 Turn off autosamdri-810 and wipe the exhaust hoses. Roll up exhaust hoses and store. Clean the beaker.

<u>If at any time you feel a situation is dangerous, turn off the CPD and close the CO_2 tank. Contact laboratory staff. Remember this is a very high pressure system.</u>

Do not hesitate to call the safety office (x73246, Peter Nowak) the faculty supervisor/lab manager (x72210, Robert White), or Tufts Emergency Services (Police/Fire/Ambulance at x66911).

Additional Information

Figure 3: The CPD system.

1. Pressure gauge: displays the pressure present in the process chamber
2. Temperature gauge: displays the temperature present in the process chamber
3. Purge timer: sets purge time when running in automatic mode
4. Inlet adjust: sets flow rate of liquid CO_2 into the process chamber
5. Purge adjust: sets flow rate of fluid exiting process chamber (must be less than inlet flow rate)
6. Process chamber: chamber where devices will be dried
7. Controls: used to operate the device
8. Exhaust beaker: captures fluids exiting process chamber during PURGE cycle
9. Die holder handle: used to insert/remove die holder into/from the process chamber
10. Die holder container: holds devices that will be dried

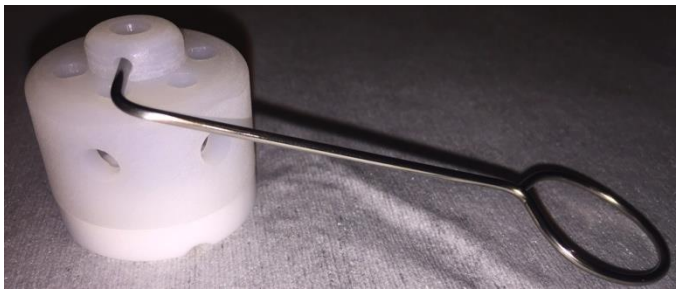

Figure 4: Die holder and handle.

Die holder & handle

Ensure that the process chamber is about 1/3 filled with methanol. Using the handle, insert the die holder into the chamber. Add methanol as needed to ensure the holder is completely submerged.

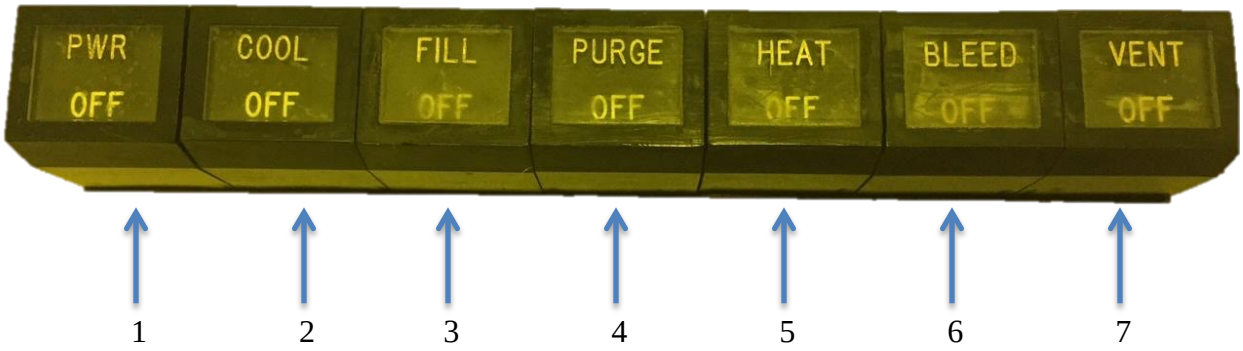

Figure 5: Power and cycle step buttons.

1. **PWR**: Turns on/off the device. After turning on, allow at least 5 minutes to pass before proceeding to the COOL phase.
2. **COOL**: Liquid CO_2 (LCO_2) from the CO_2 tank flows through an enclosure surrounding the process chamber and exits through the COOL hose. During this phase, the temperature of the process chamber decreases to 0 °C (±5 °C). A typical cool time is 15-30 seconds.
3. **FILL**: LCO_2 enters the process chamber via the inlet hose. If running in manual mode, allow at least 2 minutes to pass to ensure chamber is properly filled.
4. **PURGE**: Removes fluid from the process chamber. When running in automatic mode, the purge timer determines the run time of this phase. At 3 distinct locations on the purge timer knob, there are 2 numbers.

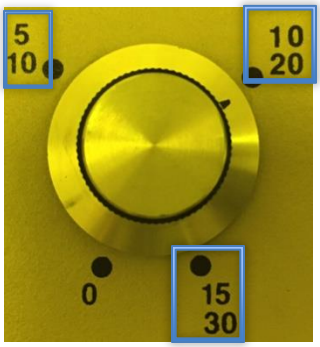

Figure 6: Purge timer.

i. If the TIME button reads 0-15, then the top numbers set the purge time. In other words, possible times are 0-5-10-15.
ii. If pushed, then it should read 0-30. In this case, the bottom numbers set the purge time (0-10-20-30).
iii. If running in manual mode, the purge timer is disabled.

NOTE: Regardless of the mode of operation (automatic vs. manual), the autosamdri-810 will automatically cool the temperature of the process chamber, if need be.

5. **HEAT**: Increases the temperature of the process chamber until it is above the critical temperature of CO_2. During this phase, the pressure of the chamber increases as well. A typical heat cycle takes ~10 minutes.
6. **BLEED**: The pressure of the process chamber decreases while maintaining a temperature above that of the critical temperature of CO_2. Fluid will be exiting the chamber through the BLEED/VENT hose. If running in manual mode, this button should be pushed until the pressure reading is below 400 psi.

NOTE: Regardless of the mode of operation, the autosamdri-810 will automatically maintain the temperature of the process chamber above the critical point of CO_2.

7. **VENT**: The pressure of the process chamber will decrease to atmospheric pressure. Fluid will be exiting the chamber through the BLEED/VENT hose. Even after reaching atmospheric pressure, allow the autosamdri-810 to remain in VENT mode for a few minutes to exhaust any remaining CO_2 in the system.

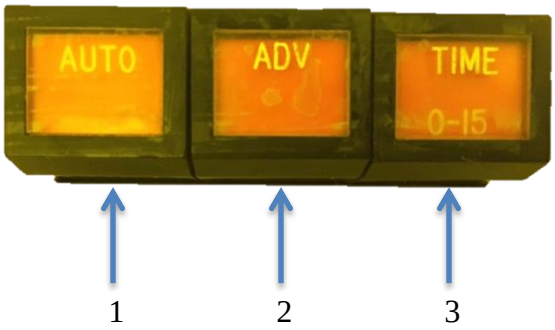

Figure 7: Auto, Advanced, and Time buttons.

1. **AUTO**: Indicates that the CPD is running in automatic mode. If pushed, the button should display MAN, indicating that the device is now running in manual mode.
2. **ADV**: Used in automatic mode to transition between phases. In a typical run, the advance button should only used when entering the COOL step and when entering the FILL cycle.
3. **TIME**: Indicates the set of numbers being read by purge timer. If pushed the display should change from ‘0-15’ to ‘0-30’.

Carboy

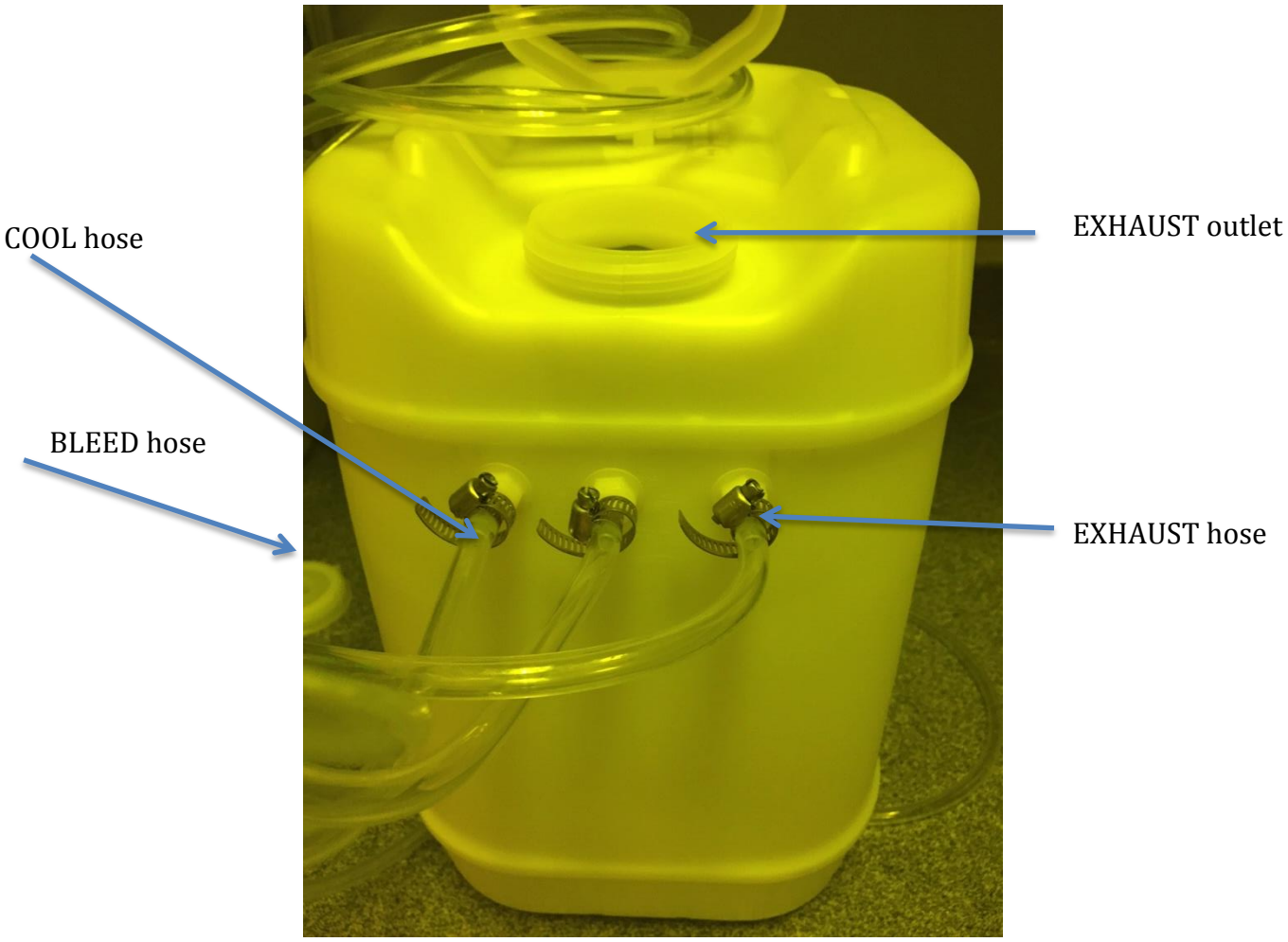

Figure 8: Carboy should be placed in the hood with the cap open.

Before starting operating the device, ensure that the carboy is securely placed in the fume hood and that the *lid is removed*.

- COOL hose: As the process chamber's temperature decreases, a mixture of gaseous and liquid CO_2 will enter the carboy.
- BLEED hose: During the BLEED cycle, gaseous CO_2 will enter the carboy.
- EXHAUST outlet/hose: Allow CO_2 to exit the carboy into the fume hood during operation.

NOTE: Failure to properly remove the lid will result in the carboy expanding due to rapid pressure buildup from vaporization of LCO_2.