

TABLE OF CONTENTS

Chapter 1	SAMDRI®-PVT-3D Overview	
	SAMDRI®-PVT-3D	2
	SAMDRI®-PVT-3D Setup Overview	3
Chapter 2	Installing SAMDRI®-PVT-3D	
	Preparation of Installation Site	4
	Installation of the SAMDRI®-PVT-3D	4
Chapter 3	Operating SAMDRI®-PVT-3D	
	Operating SAMDRI®-PVT-3D	8
Chapter 4	Operational Notes	
	Safety Devices	12
	Installation	12
	Loading	12
	Chamber Cooling	13
	Solvent Exchange (PURGE)	13
	Critical Point Passage (HEAT)	13
	Pressure Reduction (BLEED)	15
	Typical Cycle	16
	Care of Dried Samples	16
Chapter 5	Theory of Critical Point Drying	
	The SAMDRI®-PVT-3D	17
	Preservation of Delicate Ultrastructure	17
	Sample drying at the Critical Point (SAMDRI®-ING Method)	17
	Transitional Fluid	18
	Sample Preparation before SAMDRI®-ING	19
	Water Exclusion	19
Chapter 6	Illustrations	21
Chapter 7	Maintenance and Support	
	Regular Maintenance Schedule	22
	Chamber Care	23
	LCO2 Filter Assembly	24
	LCO2 T-filter Element Installation	26
	FAQ	27
	Optional Accessories	30
	Warranty	30
	Repackaging for Shipment	31
Appendix		
	A. Chamber Lamp Replacement Installation	32
	B. Purge Line Filter Replacement Instruction	36
	C. Installation of the SAMDRI®-PVT-3D (with SOTER™ Condenser)	38
Illustration Index		50
User's Notes		51
Check Out Data Sheet		
Warranty		

SAMDRI®-PVT-3D Overview

SAMDRI®-PVT-3D

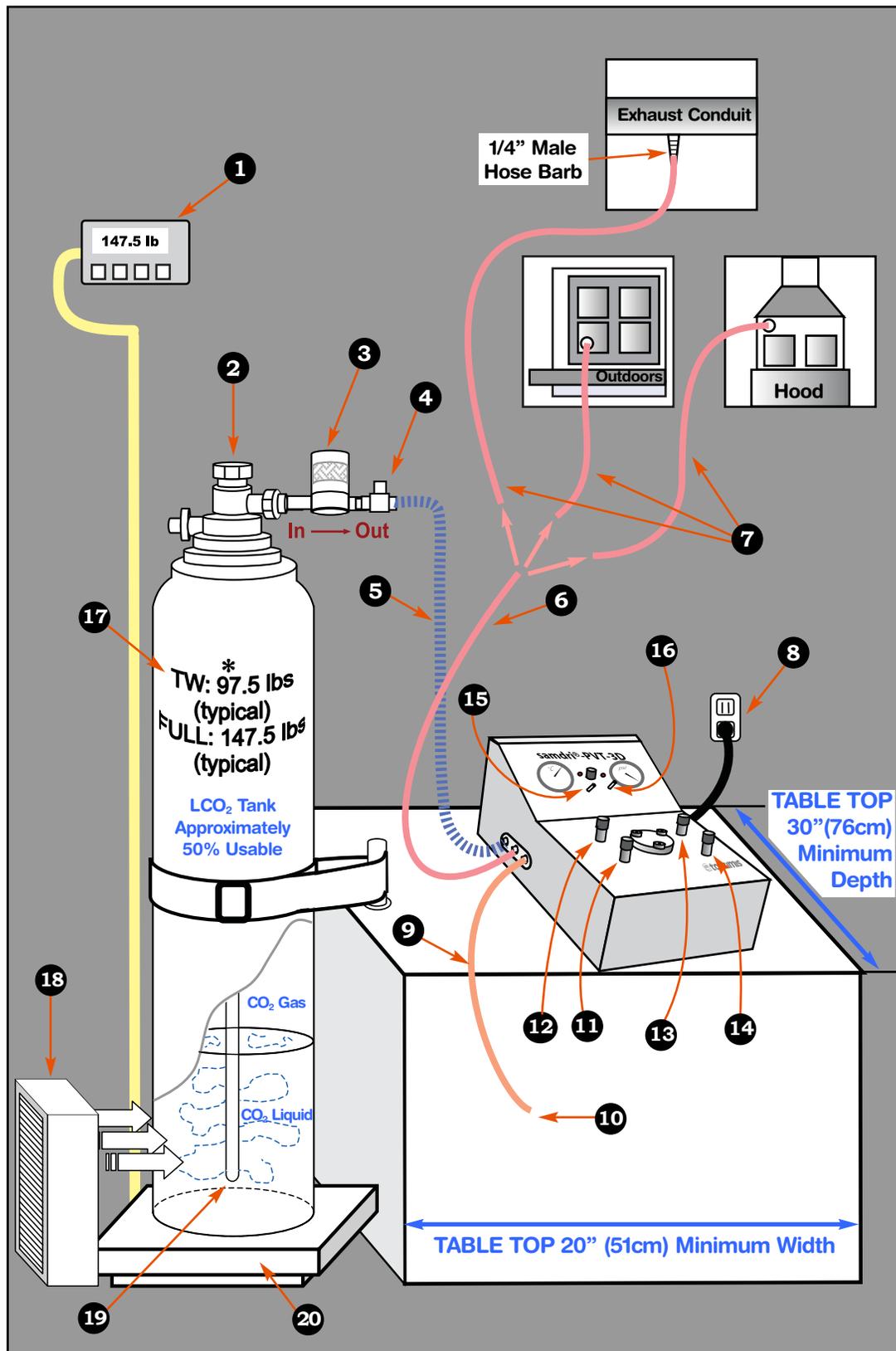
THE ADVANCED MANUAL CRITICAL POINT DRYER



tousimis® Catalog #8755B (SAMDRI®- PVT-3D)

SAMDRI®-PVT-3D Setup Overview

- 1 Remote LCD readout display of LCO₂ Tank Weight
- 2 Main LCO₂ Tank ON/OFF Valve
- 3 Oil/Water Filter Housing of LCO₂ Filter Assembly (#8784)
- 4 T-Filter w/ 0.5µm Element of LCO₂ Filter Assembly (#8784)
- 5 High Pressure Hose, 5ft (other lengths available upon request)
- 6 COOL / BLEED Exhaust Hose, White (1/4" ID), 6ft
- 7 COOL / BLEED CO₂ Exhaust Vented to either Hood, Exhaust Conduit, or Outdoors.
- 8 120V Properly Grounded Outlet (from either Wall Outlet or Transformer)
- 9 PURGE / VENT Exhaust Hose, Clear (1/4" ID), 6ft
- 10 PURGE/VENT Exhaust Hose routed to either an Erlenmeyer Flask, a Carboy (1 gal minimum) or Solvent Drain (if available)
- 11 FILL Meter Valve
- 12 COOL Meter Valve
- 13 BLEED Meter Valve
- 14 PURGE/VENT Meter Valve
- 15 POWER SWITCH
- 16 HEAT SWITCH
- 17 Tare Weight Varies (Consult your LCO₂ supplier. Usually stamped on tank)
- 18 Ceramic Heater or Heater Jacket maybe required in a cold tank placement position
- 19 Siphon or Dip Tube
- 20 LCO₂ Tank Scale (trc# 8770-54)



* At Room Temperature (20°C - 24°C), average LCO₂ tank pressure is 800psi (+/- 5%).

Installing SAMDRI®-PVT-3D

See Appendix C (p.38), to Install SAMDRI®-PVT-3D with optional SOTER™ Condenser (# 8777)



* BOC gas suppliers refer to 99.8% purity grade as “2.8” grade.

• Always order LCO₂ tanks with Siphon or dip tubes.

• Air Products gas suppliers refer to 99.8% purity grade as “CP” grade.

• Minimum LCO₂ purity required is 99.8%.

PREPARATION OF INSTALLATION SITE

Upon receipt of your SAMDRI®-PVT-3D, unpack the instrument carefully and check for any damage that may have occurred during shipment. Report (by Email trc@tousimis.com or Fax #301-881-5374) any irregularities immediately to tousimis®.

Insist on clean surfaces. Minimum table top space of approximately 20”(51cm) Width x 30”(76cm) Depth should be allotted for the SAMDRI®-PVT-3D with an additional 12”(31cm) x 12”(31cm) of floor space for the LCO₂ tank scale (See p.3).

- Use bone-dry (min. 99.8% Purity) LCO₂ with a syphon (dip-tube) tank* only.
- Do not use pressurized LCO₂ with Helium or any other high pressure substitute gas.
- Properly secure LCO₂ tank according to your facility’s safety protocol.

Tank pressure typically reads 800psi (±5%) at room temperature. The amount of LCO₂ in tank is best monitored with a LCO₂ tank scale (See accessories p.30).

Typical nominal LCO₂ tank weights for Net 50lb LCO₂ tanks:

- Full tank: 140 to 170 lbs / Tare of tank: 90 to 120 lbs.
- Most of the time, you may use 50% of a 50lb. net weight CO₂ tank.
- It is good practice to have spare LCO₂ tanks stored in reserve in case a tank runs out during mid process.

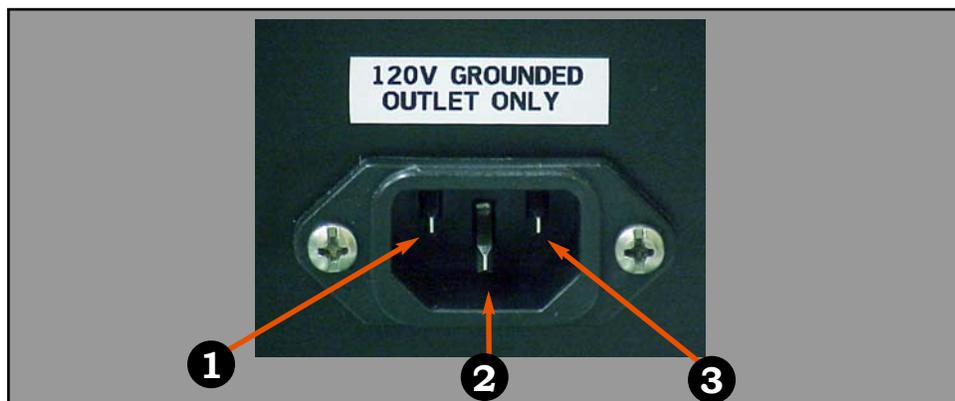
A properly grounded 120V/50-60Hz outlet or Transformer (Step-up/Step-Down) should be located within 4ft (1.2.m) of install site.

POWER Connection to SAMDRI®-PVT-3D

① AC (BLACK)
COLD

② GROUND
120AC/60HZ

③ AC (WHITE)
HOT





** LCO₂ tanks used in most facilities have tare weights ranging from 90 - 120lbs (40 - 55kg) and are delivered as "full" with net 50lbs (23kg) of compressed CO₂.

Generally, 50% of the net LCO₂ is useable.

Monitor the LCO₂ tank weight via a LCO₂ Tank Scale (#8770-54).

INSTALLATION OF THE SAMDRI® -PVT-3D

This SAMDRI®-PVT-3D is supplied with the basic accessories for operation with LCO₂ as a transitional fluid (for optional accessories, see p.30).

1. Install SAMDRI® within 4 feet (1.2m) of a properly grounded (120V/50-60Hz) electrical outlet.
2. Position a LCO₂ tank with dip tube within 5 feet (1.5m) of the SAMDRI®-PVT-3D. For a longer high pressure hose length, (see p.30), trc #8770-33. The LCO₂ tank should be mounted on a 400lb (182kg) capacity floor scale w/ remote LCD₂ display (trc #8770-54) to easily monitor LCO₂ supply**.
3. Pressure gauges or regulators should not be installed between the LCO₂ supply tank and the SAMDRI®-PVT-3D LCO₂ Connect Inlet. The stainless steel-sheathed high pressure hose accompanying the SAMDRI® is fitted with a CGA-320 female hex coupling which accepts a 3/4" plastic gasket (supplied) and joins the hose directly to the CGA-320 threaded male coupling at the tank top (See p.24). ALL LCO₂ tanks should be equipped with a rupture disc. All LCO₂ fittings and gasket surfaces should be kept clean and dry.

NEVER OVER-TIGHTEN FITTINGS...TUBING ATTACHED MAY BE DAMAGED.

Connecting High Pressure Hose to SAMDRI®-PVT-3D



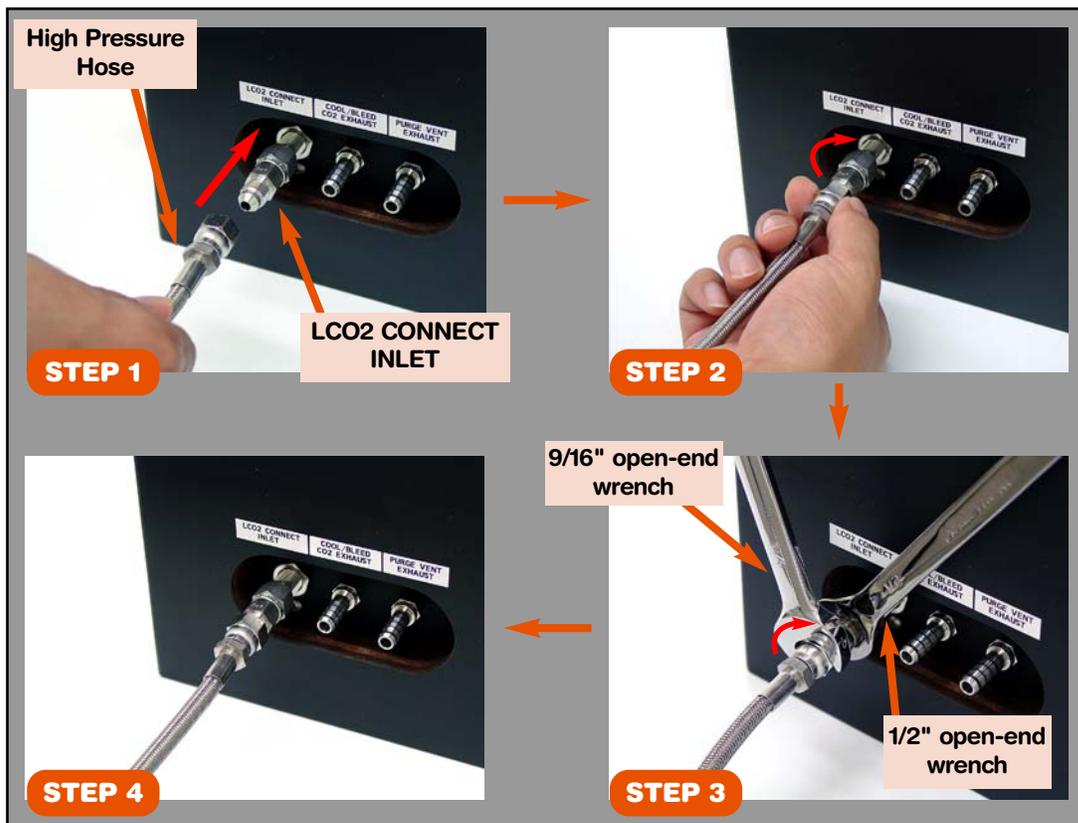
To attach LCO₂ high-pressure hose end fitting to the PVT-3D "LCO₂ Connect", use the supplied 9/16" and a 1/2" open-end wrenches.

First, slide 1/2" wrench over 1/2" fitting as shown in the photo. The 1/2" wrench will be used to hold the "LCO₂ Connect Assembly" steady as you gently tighten the high pressure hose end fitting snugly onto each of the flare connects with a 9/16" wrench.

DO NOT OVER-TIGHTEN

NO TEFLON® TAPE NECESSARY

4. Attach the HIGH PRESSURE HOSE onto the LCO₂ CONNECT INLET. Gently tighten the High Pressure Female Connector using the two open-end wrenches supplied (one on the hose and the other on the Inlet). Stop at the feel of first resistance; re-tighten if necessary, should you hear a leak.



5. The tousimis® LCO₂ Filter Assembly (catalog #8784) is factory installed directly onto the high pressure hose (See p.24-25). This helps prevent sample contamination from oil, water and particulates. Change the Oil/Water Filter Element (# 8784A) with every 500lbs (226.8kg) LCO₂ tanks changes. The secondary 0.5µm T-Filter Element (# 8783B) should be annually replaced.

Connecting Exhaust Hoses to SAMDRI®-PVT-3D

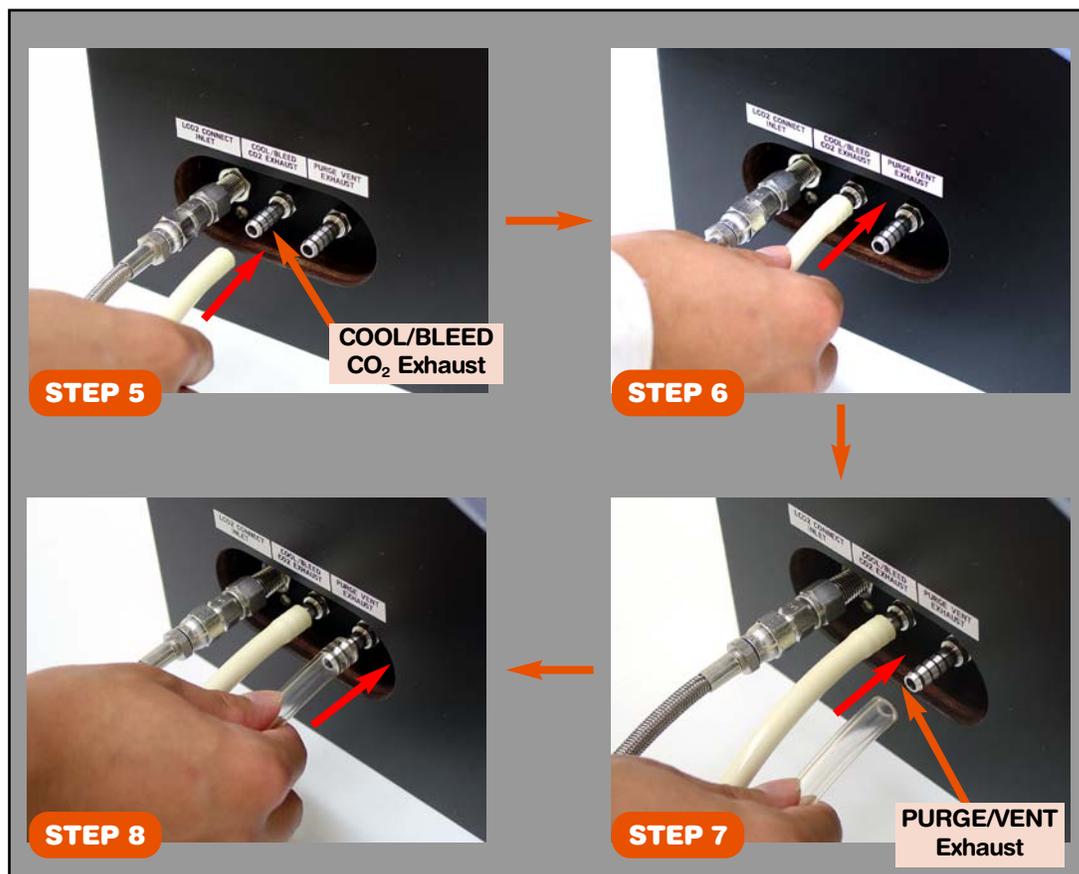
6. Attach the 6ft White COOL/BLEED Exhaust Tubing provided to the COOL/BLEED CO₂ Exhaust Outlet on the left-hand side of the SAMDRI®-PVT-3D, and the 6ft Clear Exhaust Tubing to the PURGE/VENT Exhaust outlet (See photos below). The tubing should be free of kinks which could block passage of rapidly exiting noisy CO₂ gas or solid flakes of CO₂.



NOTE:

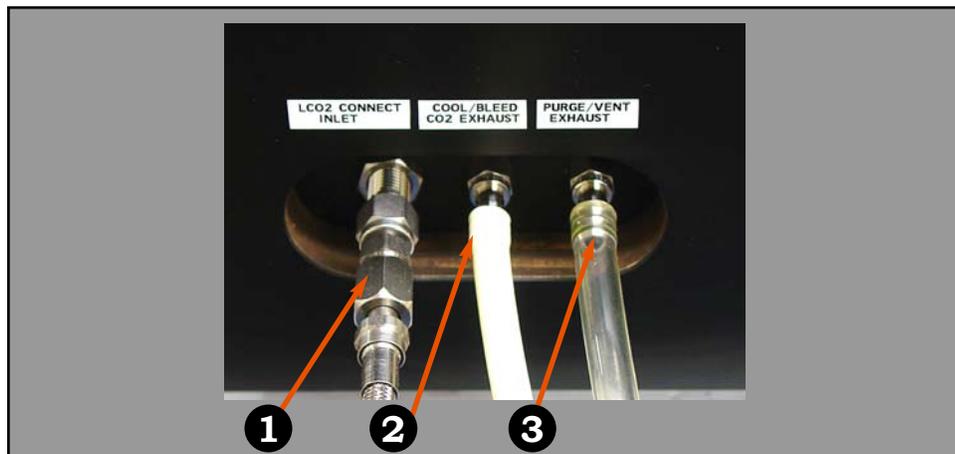
It is best to secure both the ends of the COOL/BLEED and PURGE/VENT Exhaust tubing so that the ends do not move during exhaust modes.

Be careful not to obstruct exhaust hose I.D. when securing.



Samdri®-PVT-3D Complete Inlet / Outlet Setting

- 1 LCO₂ Connect Inlet
- 2 COOL / BLEED CO₂ Exhaust
- 3 PURGE / VENT Exhaust



Samdri®-PVT-3D Chamber O-Ring



NOTE:

The chamber O-Ring should lie completely in its groove.

NEVER grease the O-Ring.



NOTE:

Do not expose chamber to HF or any other Acids.

Do not use Acetone.

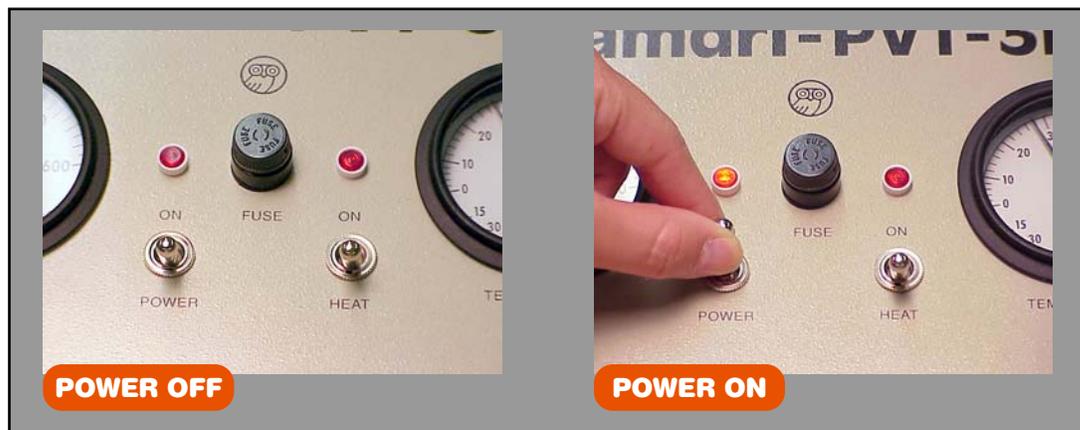
Please consult tousimis before using any non-recommended intermediate fluids or process any non-traditional CPD application material(s)

7. Check chamber, O-ring and windows; inspect unit to insure it is free of dust, oil and moisture. Chamber O-ring is NOT to be greased, but keep the chamber groove clean and dry.



Operating SAMDRI[®]-PVT-3D

Turning Power ON / OFF



NOTE:

Pressure measurement is no indication of the amount of LCO₂ left in the tank. The same PSI reading may be noted even after the liquid CO₂ drops below the level of the siphon tube.



NOTE:

* Use Ultrapure Alcohols only! (i.e. I.P.A., Methanol, Ethanol).

Use minimum 99.5% purity Alcohols for best results.

1. Turn power ON. Power light and chamber light will illuminate. Allow SAMDRI[®]-PVT-3D to warm up for 3 minutes prior to use. This allows time for the internal valve heaters to equilibrate.

2. Make certain all metering valves (FILL, COOL, BLEED, PURGE/VENT) are in the closed position (hand tight). Open the main LCO₂ tank valve. The Siphon (dip-tube) LCO₂ tank should have between 25 lbs - 50 lbs (13.6 kg -23 kg) net weight of LCO₂. (Subtract the tare weight of the tank from the tank weight to get the net weight of LCO₂).

3. Sample should be dehydrated completely in high purity (99.5+% minimum purity) alcohol. Pour enough high purity alcohol in the SAMDRI[®] chamber to completely cover your sample(s) prior to transfer. Now, transfer your sample into the process chamber. Make the transfer quickly in order to avoid the samples' exposure to air and moisture.

The chamber O-Ring should lie completely in its groove. NEVER grease the O-Ring. Place the chamber lid down over the chamber using the 3 knurl nuts; tighten each by hand with equal pressure. Never use channel locks or pliers for tightening the knurl nuts. Always hand tight!

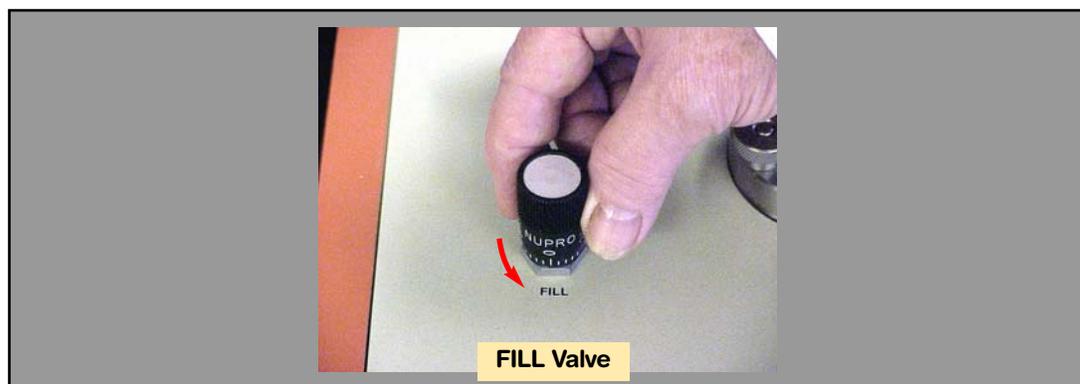
COOL Valve Adjust



COOL MODE

4. Open COOL valve to approximately the 0.50 position (See metering valve illustration, p. 21). The temperature of the chamber should drop to near 0°C from room temperature in approximately 1 minute. (The cool rate is a function of the amount of LCO₂ running through the COOL VALVE.) Usually a 0.50 setting on the COOL VALVE is satisfactory. It is normal to hear the COOL Exhaust making audible noise as it exhausts from white tubing. Close COOL VALVE when temperature reaches 0°C.

FILL Valve Adjust



FILL MODE

5. Open FILL metering valve slowly until the chamber is full. LCO₂ will enter the process chamber mixing with the high purity alcohol (A bubble (meniscus) will travel across the quartz viewing window indicating the chamber is full). Maintain the FILL valve open to a setting of about 0.50 turns (this setting could vary as per use of the metering valve).

PURGE-VENT Valve Adjust



PURGE MODE

6. Keep FILL valve open. To exchange all the ethanol with LCO_2 within the chamber; slowly open the PURGE / VENT metering valve (exhaust) you can visually see movement (from left to right) in the chamber. Make sure the “liquid” level in the chamber is always above the sample. The FILL rate adjust should always exceed the PURGE / VENT rate to insure that the sample remains covered in a liquid state. Place a carboy or an Erlenmeyer Flask at the end of the clear PURGE exhaust tube to collect waste alcohol. Continue in PURGE mode until all of the ethanol is exhausted. To be certain that all the ethanol has been purged, hold a clean piece of paper (glycine paper works best) about four inches from the exhaust tube and collect the solid white CO_2 that is being emitted. The solid CO_2 will sublime, and any visible “wet” marks on the paper indicates that there is still ethanol remaining.

If chamber temperature rises above 10°C during PURGE mode, open the COOL VALVE (this will not interfere with the PURGE mode) until the chamber is cooled to 0°C ; then, close the COOL valve and continue purging.

7. After ethanol is purged, close PURGE / VENT metering valve. Keep the FILL valve open to insure chamber is full with LCO_2 . Note that a meniscus will travel across the viewing window, indicating that the chamber is full. Close the FILL metering valve.

Turning HEAT ON / OFF





Never over tighten Meter Valves when closing to 0.00 position. Over Tightening may damage valves.

8. At this point, all the metering valves should be in the “CLOSE” (0.00) position (see page 21). Turn HEAT switch ON. HEAT light will go ON. As the temperature (°C) increase, so will the pressure. At approximately 1,350 psi ($\pm 5\%$ @ 20°C), the pressure relief valve will begin to vent the chamber pressure. The chamber’s pressure will be maintained above the critical pressure for CO₂ (1,072 psi). As the temperature passes the critical temperature for CO₂ (31°C), the heater will cut off (indicated by the heat light turning OFF) between 33°C - 39°C. The temperature may coast up to 44°C; this is normal. Do not turn off the HEAT switch. The SAMDRI®-PVT-3D will automatically maintain the chamber at a temperature higher than the critical temperature (31°C) of CO₂.

tousimis®
EQUILIBRIUM

9. At this point, the SAMDRI®-PVT-3D is above the critical pressure (1,072 psi) and temperature (31°C) for CO₂. It can maintain itself automatically at the tousimis® equilibrium state indefinitely. For best results, we recommend that you wait 4 minutes in equilibrium before proceeding to BLEED mode.

BLEED MODE

10. POWER and HEAT switches are ON. FILL, COOL, BLEED and PURGE / VENT metering valves are closed. Slowly open the BLEED metering valve to the BLEED meter valve adjust position noted on the Check-Out Data Sheet. The initial BLEED flow rate via the flow meter should be 8-10 SCFH (Fast decompression could result in CO₂ “condensation”). Chamber heater will cycle ON and OFF automatically to maintain temperature above the critical temperature during BLEED. Close BLEED metering valve at 400psi.

BLEED Valve Adjust



SAMDRIed samples could be hygroscopic; coating with conducting film immediately will best preserve surfaces of interest for your studies. However, even after coating with a conducting metal layer < 100Å thick, your sample should be kept dry and clean.



11. Open the PURGE / VENT metering valve to at least 0.50 setting to reduce chamber pressure to 0 psi. The pressure will reach 0 psi (atmospheric pressure) in 2-3 minutes, loosen the 3 Chamber Knurl Nuts evenly, by hand. Remove your sample and store in a dry environment (i.e. vacuum, desiccator, etc) for further processing.

12. You may begin your next process run immediately (if desired).

13. After last process run of the day, make certain the chamber is clean and O-ring is in place. Place lid on and tighten (by hand). Close the LCO₂ main tank valve and open all valves of the SAMDRI® to “drain” them from any residual line pressure, then close all the valves. Clean the SAMDRI® surfaces. Turn Power OFF.

Operational Notes

SAFETY DEVICES

The SAMDRI®-PVT-3D sample process chamber is a high pressure metal alloy vessel with viewing ports designed to withstand the pressures used in the critical point drying process. To ensure additional operator safety, the following features are incorporated:

- A mechanical Automatic Pressure Control System (APCS) which will maintain the chamber at a pressure between 1,350 psi \pm 5% (see Check-Out Data Sheet for your dryer's pressure relief valve specs).
- A rupture disc which will be activated (2,000 psi at 20°C) if the mechanical APCS should fail (see factory check-out data sheet for the setting(s) of your unit.)
- Quartz ports which will crack along the crystal line, rather than shatter, should they become unduly stressed (Immediately replace chamber lid if window becomes cracked).
- Metal-alloy (sintered stainless steel) filters placed internally along both the inlet and exhaust paths, preventing solid particles from fouling the gas lines/valves/fittings.
- An electronic heating system which is governed by two thermostats: one to maintain the chamber during critical point passage at temperatures 37°C \pm 2°C (see factory check-out data sheet for your unit) and a second safety thermostat to shut off all heating should a failure bring the chamber temperature near 49°C \pm 3°C.
- A bulb and a translucent diffuser plate below the sample chamber which illuminate chamber contents for easy viewing.

INSTALLATION

The SAMDRI®-PVT-3D is supplied with all necessary internal accessories for operation with pressurized LCO₂ as a transitional fluid. The user may also find it helpful to mount the LCO₂ cylinder on a tank scale (#8770-54) to monitor transitional fluid supply (by weight, not pressure!), since only approximately 50% of the original net LCO₂ may be used.

The SAMDRI®-PVT-3D connects to a standard 120V/50-60Hz, 15 Amp grounded outlet and can be used wherever space permits. Waste alcohol can be collected into a carboy or an Erlenmeyer Flask before the venting of the CO₂ commences. The work area should be clean and dry at all times.

LOADING

An adequate amount of intermediate fluid (Ultrapure Alcohol*, 99.9%+, enough to cover the sample and holder) should be poured carefully into the chamber before placing your samples into the chamber. This will minimize surface evaporation of the dehydrating intermediate fluid (ethanol) from the sample. This is done at room temperature to avoid moisture absorption from the atmospheric environment.



Methanol may also be used as an intermediate fluid

CHAMBER COOLING

The tousimis® cooling capability of the SAMDRI®-PVT-3D may be exercised at any time, for it is an entirely independent operation from FILL or PURGE/VENT. The chamber is brought down to a temperature of approximately 0°C or below (Consult Check-Out Data Sheet for your unit). The LCO₂ tank should have at least 50% net weight of LCO₂ for proper COOL operation.

Chamber cooling is necessary to ensure filling with the liquid phase of the transitional fluid (LCO₂). Cooling also insures expulsion of the gas phase ahead of the liquid phase in the pressure hose connecting the transitional fluid tank to the SAMDRI®-PVT-3D inlet circuit. CO₂ exiting from the COOL/BLEED Exhaust hose may appear in solid flakes as it encounters the lower pressure environment. A 6ft (1/4" I.D.) white static free exhaust tubing reduces the noisy rapid expansion of the LCO₂ exhaust. This is a standard accessory.

SOLVENT EXCHANGE (PURGE)

After cooling the chamber, transitional fluid (LCO₂) may now be allowed to simultaneously enter and leave the chamber. This process mode of incoming LCO₂ and outgoing alcohol and CO₂ is called PURGE. Characteristics visible to the operator during the PURGE mode are as follows:

- The intermediate fluid (alcohol) will appear as “oily currents” (schlieren patterns) within the chamber (especially forwards the onset of PURGE).
- The intermediate fluid will appear as a liquid or a slurry when exiting through the clear PURGE Exhaust hose. Once thoroughly exchanged, the chamber liquid will be perfectly clear; the exiting CO₂ will be solid white.

The Purge rate is manually adjustable with the PURGE / VENT valve, and should be set by the operator to allow for a steady flushing of transitional fluid without letting the liquid level drop away from the top chamber window.

A warming trend will be noted during the PURGE mode since the incoming fluid originates at room temperature. The operator should occasionally check the chamber temperature, and open the COOL valve as necessary, ensuring that the purging continues in the liquid state. It is best to keep the temperature between 0°-10°C during the PURGE mode.

The purge time required for complete solvent exchange is not standardized, but is dependent upon the size, density, and cellular characteristics of the sample. Exchange in a mono-layer of cells can be completed within several minutes, whereas large semipermeable structures may require 15 minutes. Generally, 24 samples of a size 4mm x 6mm x 2mm accommodated by a Sample Holder (tousimis® catalog #8763) will require about 10 minutes; these purge times are only suggested, however, and should be established in each case by process operator.

CRITICAL POINT PASSAGE (HEAT)

When all the intermediate fluid permeating the sample has been replaced by LCO₂, purging and cooling are terminated. Make sure the chamber is completely filled with LCO₂. Return to FILL again, if necessary. Heat is now applied to the chamber, elevating the transitional fluid to above its critical temperature and pressure. During the HEAT mode, the CO₂ may appear as oily currents due to the heat causing variations in LCO₂ densities and thus the



SUGGESTED TEST TO VERIFY COMPLETION OF PURGE:

Since more adiabatic cooling usually takes place near the exhaust orifice of the unit (evidenced by white flakes of CO₂, which will form at -79°C), and since intermediate fluids such as pure ethanol have even lower freezing points, these fluids will be flushed out as liquids. A soft, dry coated paper held momentarily in the exhausting stream will catch the white solid flakes of CO₂, and when shaken out, will show "wet" spots and indicate that there is remaining alcohol to be purged.



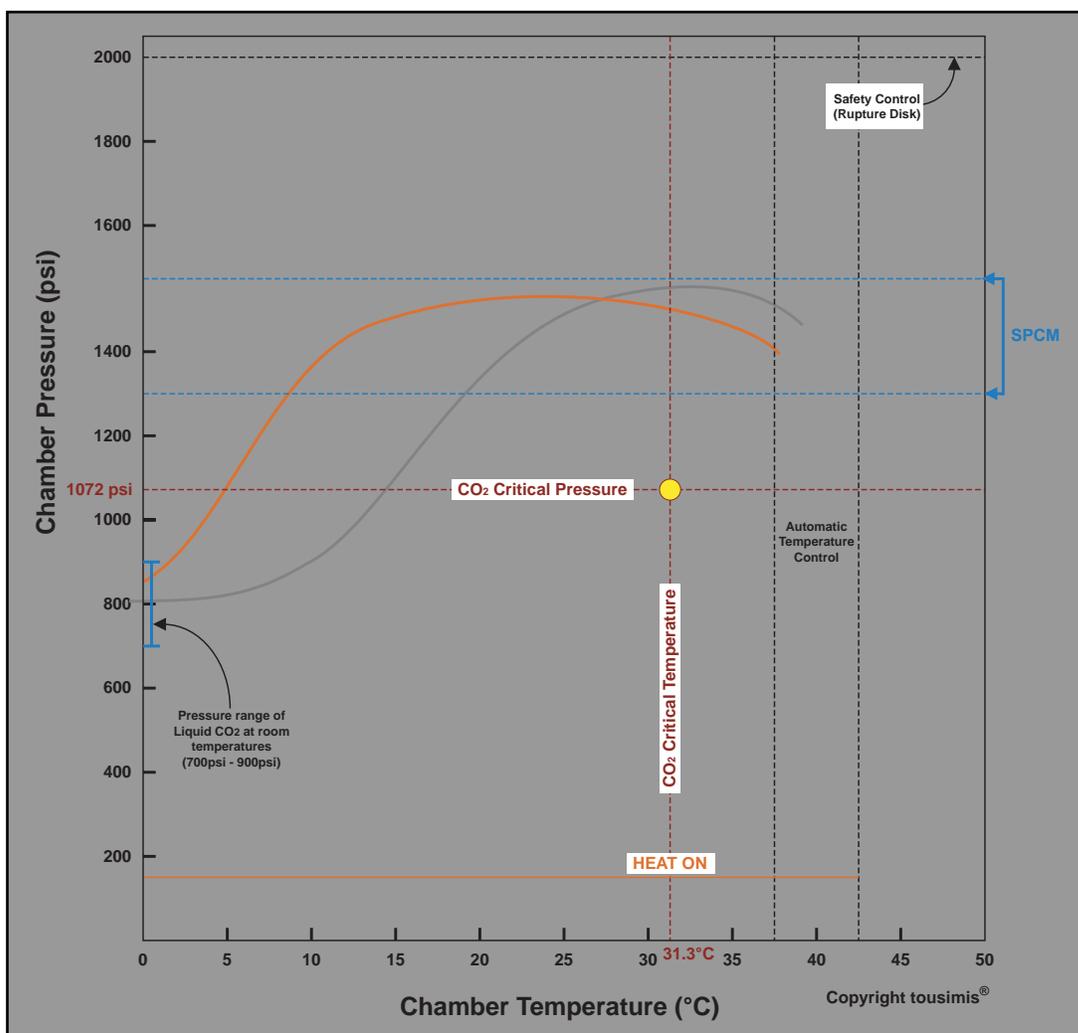
NEVER use a LCO₂ tank if it is kept outside in the winter. Bring it to room temperature for 2-3 days before use; otherwise, due to low LCO₂ tank temperature, the tank will not supply the SAMDRI[®] with LCO₂ at the proper pressure.

Schlieren pattern appearance.

The HEAT mode is thermostatically controlled to maintain a temperature at a safe margin above the critical values, yet low enough that thermal destruction (above 55°C) of any biological sample does not occur.

The chamber pressure increases relative to the increase in temperature. It is likely that the critical pressure will be reached before the critical temperature if the chamber was completely filled with LCO₂ when heat was applied. The APCS is factory-set for 1,350 (±5% @ 20°C) and will keep the chamber below 1,600 psi until the chamber temperature rises above the critical temperature.

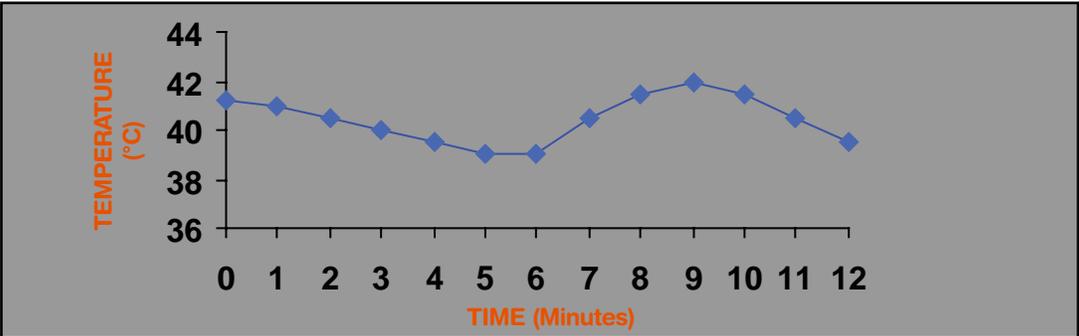
Typical Paths that Critical Point Passage May Follow During "Heat Mode"



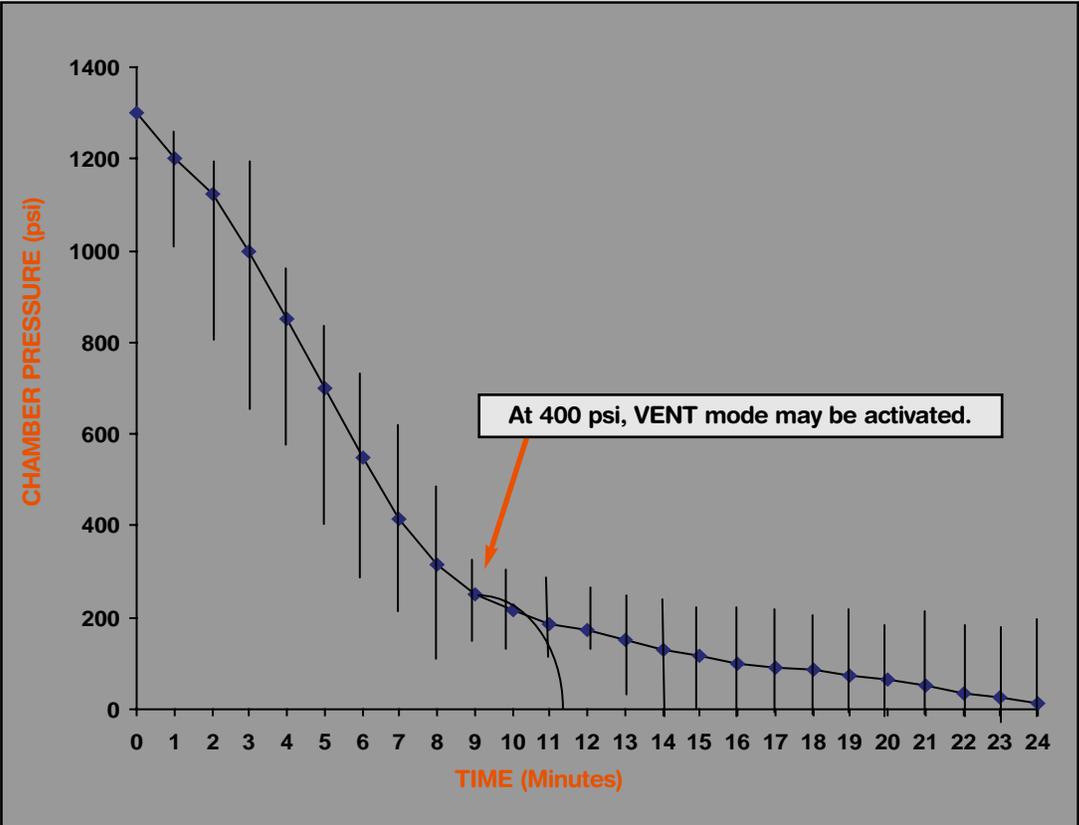
PRESSURE REDUCTION (BLEED Mode)

Once the critical point has been passed with an adequate margin of safety, the CO₂, which is now in a dense vapor state, is exhausted slowly from the process chamber.

Typical Chamber Temperature during Pressure Reduction (BLEED)



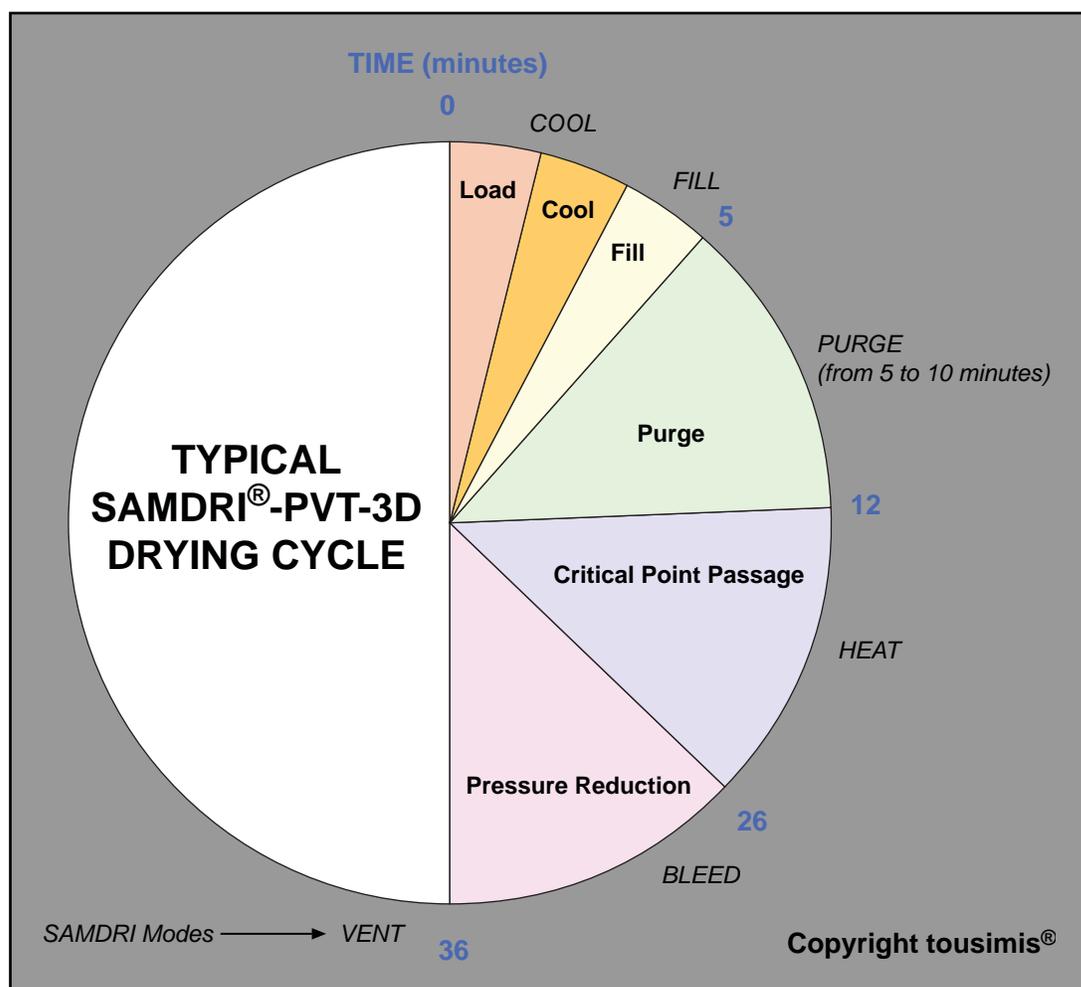
Typical Range of Paths that Pressure Reduction May Follow in the SAMDRI®-PVT-3D



TYPICAL PROCESS CYCLE

A typical process cycle, including 7 minutes of purge time, might run for about ½ hour, as shown in the following, and may use 1.5lbs (0.7kg) of CO₂.

Typical Drying Cycle



* Note that the SAMDRIed Biological samples could absorb certain glues! DO NOT USE glue to mount your sample since it will diffuse into the dried sample.

CARE OF DRIED SAMPLES

After drying, care should be taken to prevent damage to the sample surfaces. It is recommended that the sample be attached* to a tousimis® 10 mm diameter or Cambridge 1/2" stud and, as soon as possible, coated with the appropriate conducting layer. SAMDRI®-ed biological samples are receptive to a metal coating in a vacuum and do not require any preparatory carbon coating. If the sample cannot be further processed immediately after drying, it should be stored in an isolated and desiccated environment. After coating, samples should be maintained in a dry environment to prevent changes as a result of water absorption or possible cracking of the coating layer.

Theory of Critical Point Drying

THE SAMDRI®-PVT-3D

The tousimis® SAMDRI®-PVT-3D is a critical point drying apparatus which through a series of stages, the sample is processed by a technique which preserves and stabilizes the delicate 3-D morphology without freezing "rigid" chemical fixation (which alters the ultra-structure) embedding (paraffin, plastics, epoxies, etc.).

PRESERVATION OF DELICATE ULTRASTRUCTURE

Sample Drying (SAMDRI®) at the Critical Point: In order to preserve 3-D structure, one must avoid the sudden (air-drying) change of densities that occurs when the fluid "embedding" the sample and permeating its ultrastructure is converted to gas. Most liquids used to replace water in samples have a well-defined critical point -- a specific pressure and temperature at constant volume for each of them. At the critical point the densities of the gas and liquid phases of the transitional fluid are equal; therefore, a phase boundary does not exist. After the critical point transition from the liquid to vapor phase has taken place, the vapor phase can be removed (while above atmospheric pressure) and the dried sample preserved without structural damages due to surface tension forces.

SAMPLE DRYING AT THE CRITICAL POINT (SAMDRI®-ING) METHOD

SAMDRI®-ING involves immersion of the sample in an intermediate fluid [ethanol] and its substitution with LCO₂, under pressure. The LCO₂ is automatically converted to its gaseous phase at the critical point in the high pressure chamber and removed at temperatures higher than 31.1°C, thereby avoiding the distortion resulting from evaporation boundaries or thermal expansion.

Typical Paths that Critical Point Passage May Follow during "Heat Phase" (p. 14) shows the isotherm for CO₂, including the critical isotherm at 31.1°C. Superimposed is a path which an average critical drying cycle (SAMDRI®) can follow, divided here into four stages:

1st, the process chamber is COOLED and FILLED with LCO₂, and maintained at 0°C ± 10°C;

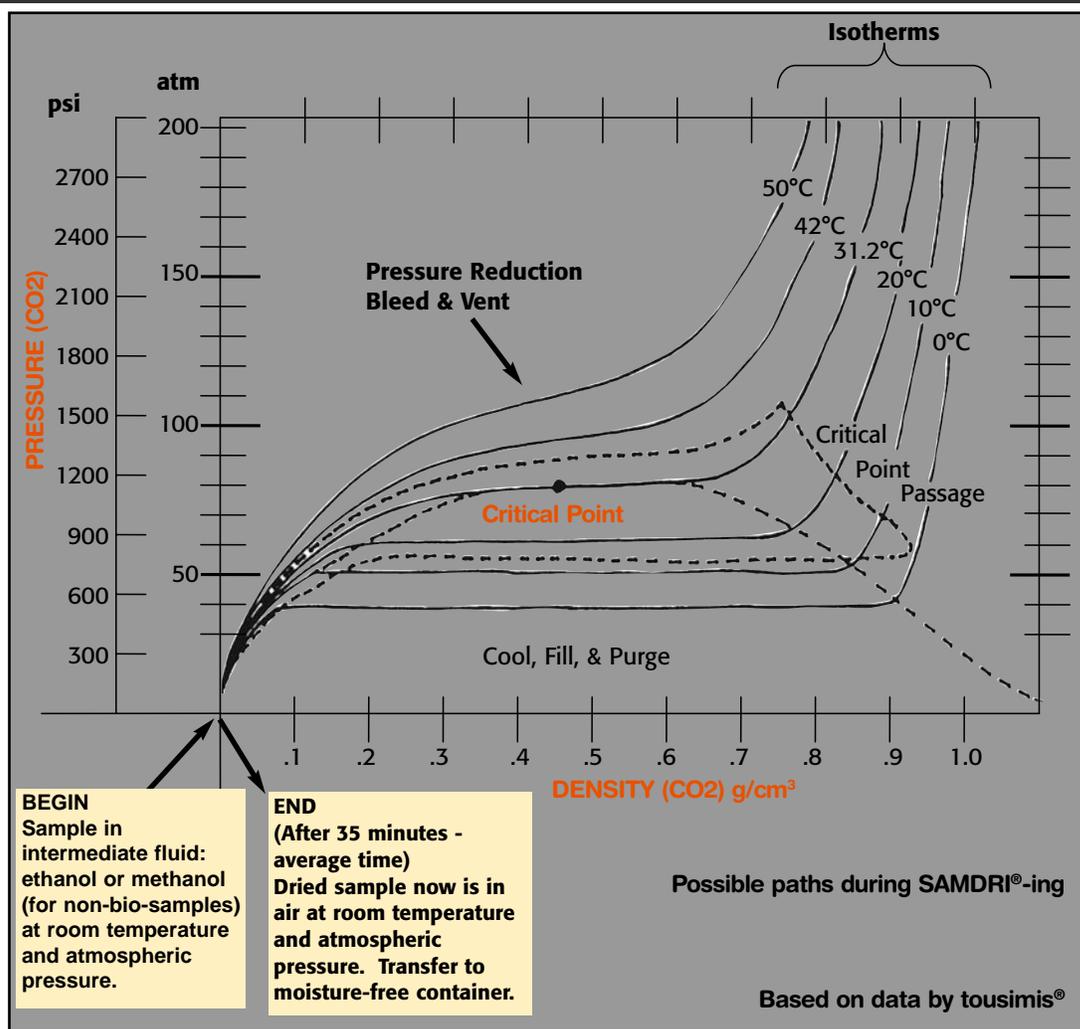
2nd, the sample in the alcohol under pressure is PURGED of the alcohol with LCO₂ until all traces of intermediate fluid [alcohol] are removed;

3rd, the chamber is heated automatically to a temperature above the critical temperature of CO₂. While the chamber volume is constant, there is an accompanying rise of pressure to above the critical pressure of CO₂, and the liquid phase is converted to the gaseous phase;

4th, the gaseous CO₂ is exhausted at a temperature above 31°C from the process

chamber, returning the sample to conditions of room temperature and pressure. The sample is now critically point dried and ready for coating (metal film coating under vacuum) and subsequent study in the SEM, TEM or electron probe X-ray microanalyzer.

Isothermal of Carbon Dioxide and an Average Drying Path of Sample in the SAMDRI®-PVT-3D



TRANSITIONAL FLUID

Liquid Carbon Dioxide (LCO₂) is the preferred transitional fluid in critical point drying, since it is easily accessible, more economical, less noxious, safe in a well-ventilated laboratory, and provides more consistent results than any other transitional fluid. Only research grade or filtered (see tousimis® combination filter, catalog #8784) "bone dry" LCO₂ should be used. Use of LCO₂ requires a "dip" or Siphon tube in the CO₂ cylinder tank, which provides liquid at the tank outlet for the first 25-30 lbs. Tanks should be kept at room temperature for at least 2-3 days prior to use.

LCO₂ is shipped to your laboratory by your local distributor in steel cylinders. Please note the tare weight and gross weight. You can only use about 50% of the net LCO₂ since the dip tube does not reach the bottom of the tank. Advise your supplier to never wash the empty steel cylinder with water or leave it "in the yard" to accumulate pollutants. ALWAYS KEEP SPARE FULL TANK IN RESERVE AT ROOM TEMPERATURE.

As you receive the LCO₂, it is under pressure at 20°C, 750 to 900 psi. [While CO₂ is a respiratory stimulant, humans cannot breathe air containing more than 10% carbon dioxide (at this level one could faint)]. Use the SAMDRI®-PVT-3D in a well-ventilated room, or exhaust all CO₂ into a working hood, exhaust conduit or the outdoors. Please consult your laboratory regulations for exhaust requirements. Usually one to two pounds of LCO₂ are used for each process run. The critical temperature and pressure of pure CO₂ are 31.3°C (88.84°F) and 72.9 atm (1072 psi), respectively.

SAMPLE PREPARATION BEFORE SAMDRI®-ING

It is not the intention of this manual to present an extensive review of sample preparation techniques. Certain samples present special problems in preparation and it is the responsibility of the investigator to determine the best procedures in each case. It is the general object of pre-critical point drying preparation steps to yield a sample in which the histological ultrastructure is well-stabilized and free of water.

Most biological samples preparation could include:

FIXATION - Fixation generally utilizes an aldehyde such as formaldehyde*, glutaraldehyde*, or acrolein** to provide rapid inter- and intra-cellular penetration and fixation, followed by osmium tetroxide for further fixation.

STAINING AND SPECIAL TECHNIQUES - A fixative/stain such as aqueous solutions of osmium tetroxide or silver is employed for SEM samples prior to dehydration and critical point drying.

DEHYDRATION - The fixed sample is taken through a series of increasing concentrations of a liquid such as ethanol, ending in a 100% ethanol of the highest possible purity (tousimis® CAT. #5086). Transfer to room temperature before placing it in alcohol in the SAMDRI®-PVT-3D chamber, and seal. Then you may initiate the process run.

WATER EXCLUSION

Water or oils remaining on the sample during critical point drying will not pass through the critical point with the transitional fluid (since their critical pressures and temperatures are different). Instead the water will evaporate from the sample surface and cause damage, or the oils will cover the surface and render sputtering or shadowcasting with an electrically conducting element impossible. Therefore, the sample must first be completely dehydrated, as mentioned above, and precautions must be taken to avoid subsequent rehydration before critical point drying (SAMDRI®-ING).



* High purity or ultra-pure fixatives are recommended. (see tousimis® CAT. #1008 and #1011)

** Acrolein is a very hazardous chemical. Avoid its use unless there is no other alternative, and then use as per special instructions in your laboratory and after careful reading of the MSDS.

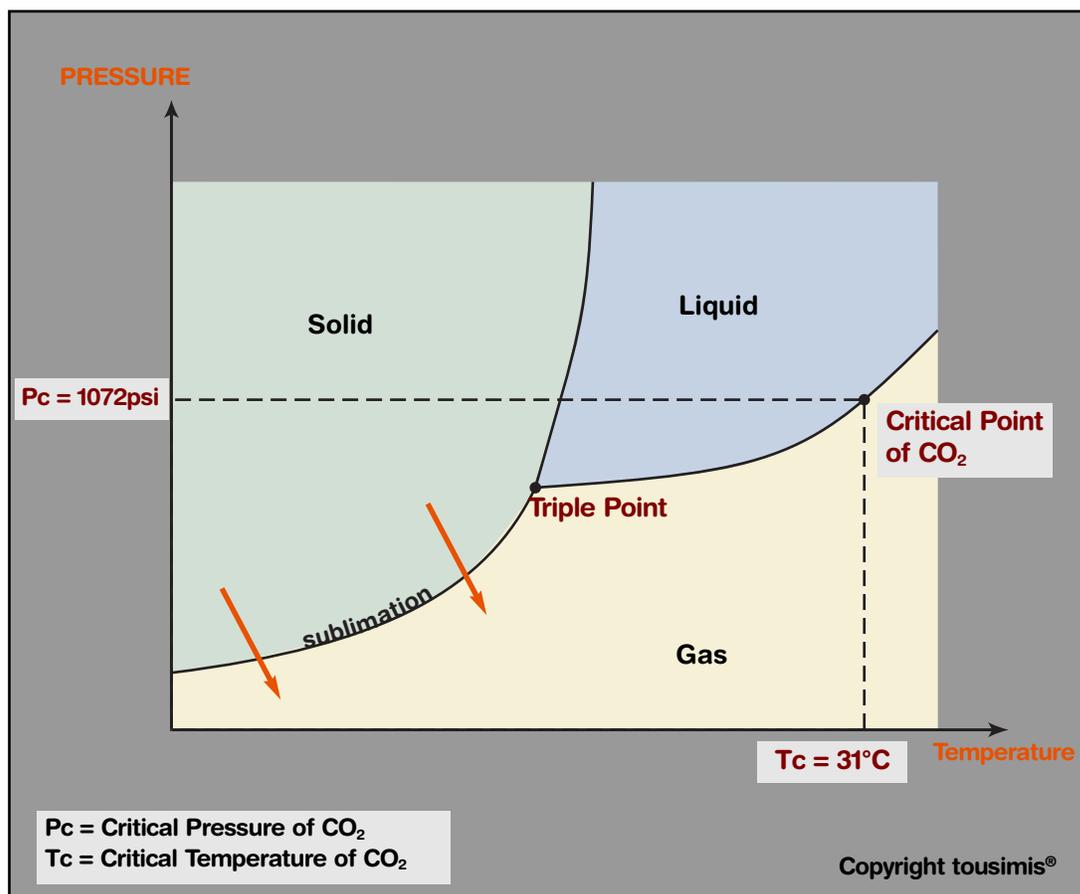


Avoid the use of powder-type CaSO_4 dehydrants in the ethanol. We suggest the use of clean silica gel-type dehydrants to avoid sample contamination from "powdering" CaSO_4 crystals.

The final dehydration step involves three conditions for the 100% dehydrating liquid:

1. The dehydrating liquid should fill the vessel containing the sample in order to reduce exposure to atmospheric moisture outside the SAMDRI®-PVT-3D.
2. The vessel should then be tightly stoppered with the lid (hand tighten).
3. The 100% fluid should be obtained from a newly-opened bottle of ultra-pure ethanol or that it contain a dehydrant to insure no water contamination. ALCOHOL IS HYGROSCOPIC.

General Form of Phase Boundaries Pressure vs. Temperature for CO_2



Illustrations

Metering Valve w/ Vernier Handle Setting Example



The numerical markings are arbitrary numerals for the sole purpose of meter valve adjustment position reference.

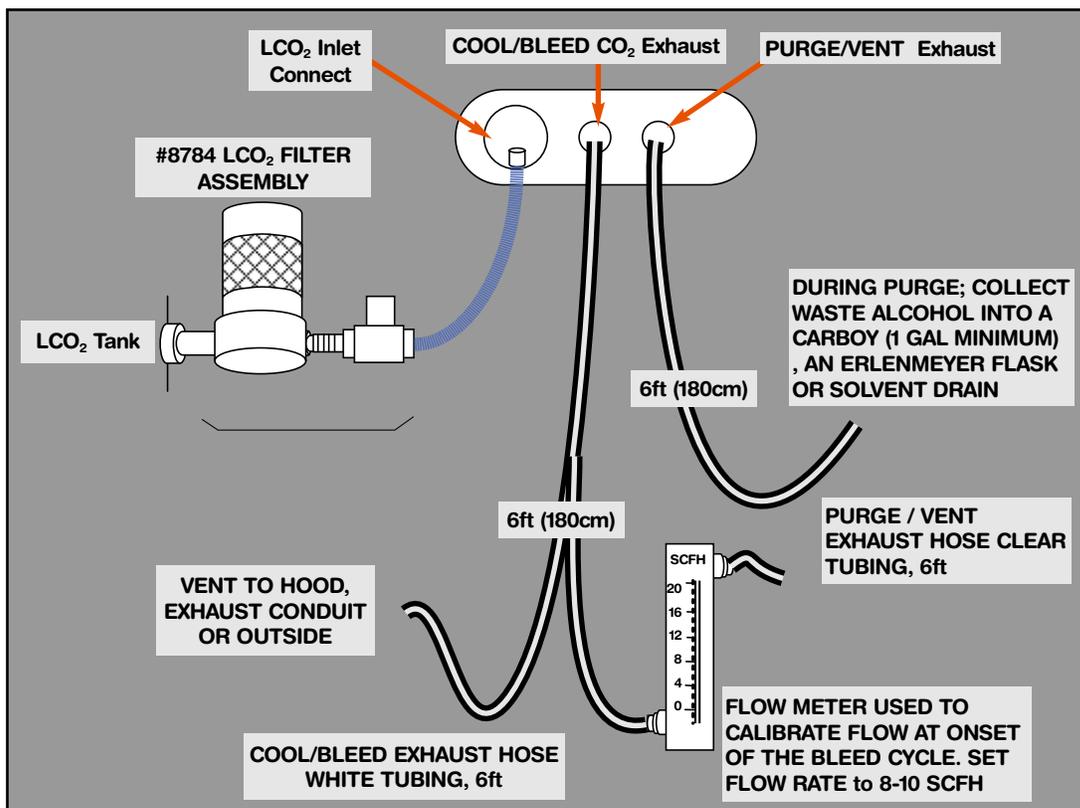
1 complete counter-clockwise 360 rotation from the 0.00 starting position is referred to as the '0.25 position'.



Venting Precautions



Only use Flow Meter to measure flow at onset of BLEED mode only.



Maintenance and Support

Regular Maintenance Schedule

Maintenance Activity	Recommended Frequency	Manual Reference	trc® Catalog Number
Keep Chamber Clean and Dry	Always	p. 23	N/A
Replace LCO ₂ Tank	After 50% net tank consumption	p. 28 (FAQ)	Contact local supplier
Replace LCO ₂ Oil/Water Filter in LCO ₂ Filter Assembly	500lb LCO ₂ Use	p. 24-25	# 8784A
Replace T-Filter Element in LCO ₂ Filter Assembly	1 Year*	p. 26	# 8770-83B
Chamber O-Ring	3 Months*	p. 7	# 8770-51
Internal Purge Line 0.5µm Filter	6 Months*	p. 36-37	# 8770-83
* May Vary Depending on Usage.			

Chamber Care



NOTE:

* Use Ultrapure Alcohols only! (i.e. I.P.A., Methanol, Ethanol).

Use minimum 99.5% purity Alcohols for best results.



NOTE:

Do not expose chamber to HF or any other Acids.

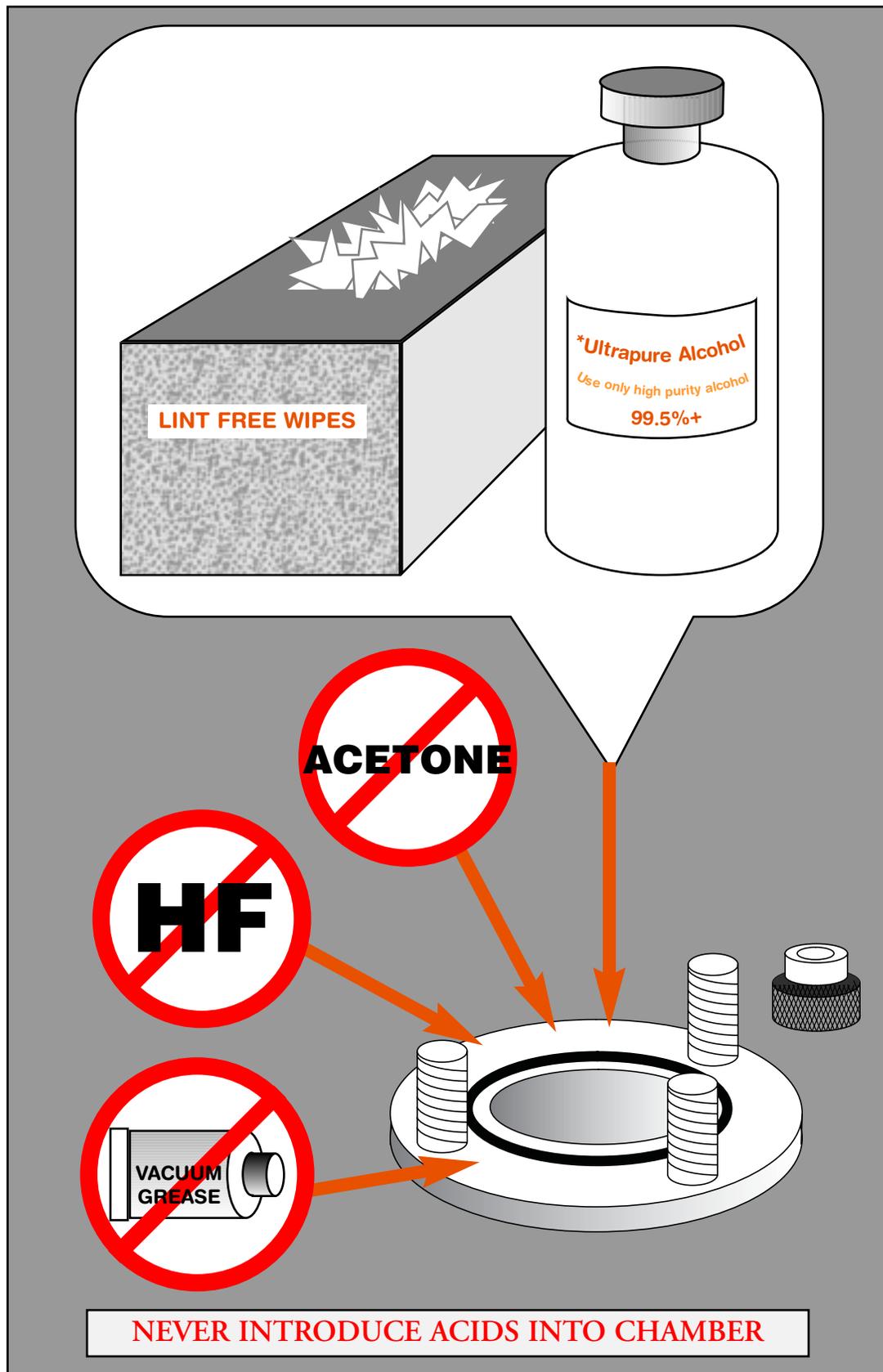
Do not use Acetone.

Please consult tousimis before using any non-recommended intermediate fluids or process any non-traditional CPD application material(s)



NOTE:

BE SURE TO KEEP CHAMBER O-RING GROVE AND FACE CLEAN AND DRY AT ALL TIMES.



LCO₂ Filter Assembly

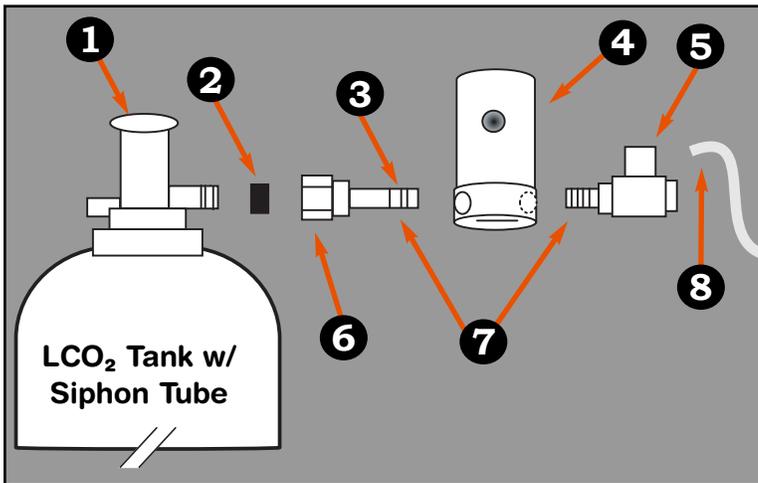
THE tousimis® LCO₂ FILTER ASSEMBLY (# 8784)

(4 + 5 Figure Shown Below)

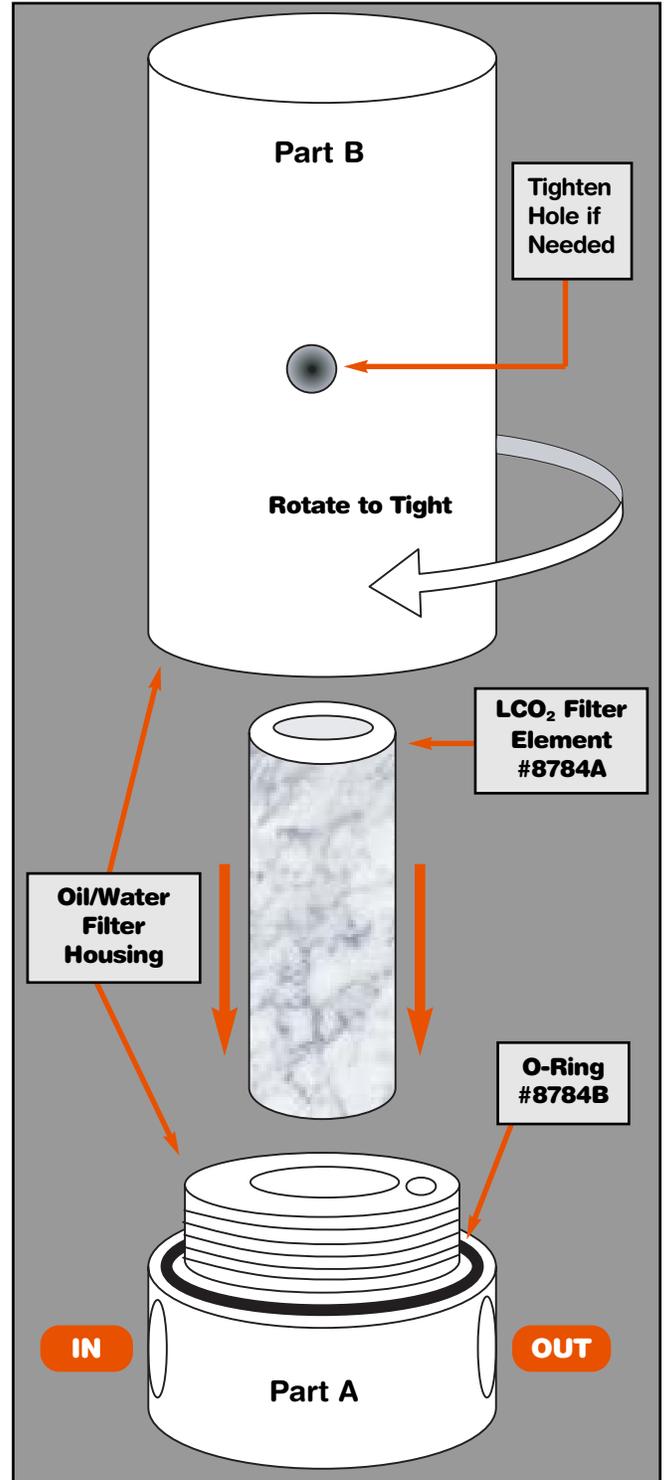
Installation Instructions

1. Uncouple CGA-320 S.S. nut from LCO₂ tank. Close main tank valve. Crack S.S. nut and bleed line pressure. Make certain there is no pressure within the high pressure hose.
2. Install filter element as shown in the diagram. (#8784A) Push element steadily into upper housing orifice until slight resistance is felt. Screw housing (Part B) onto upper housing (Part A). Be sure that teflon® O-Ring is in place.
3. The 0.5 µm T-Filter Element (# 8770-83B) is pre-installed into T-Filter.

* Change LCO₂ Filter Element (#8784A) with every 500 lbs of LCO₂ consumption, change T-Filter Element (#8770-83B) every 1 year to insure proper filtration.



- | | |
|---|---|
| 1 Main LCO ₂ Tank Valve | 5 T-Filter with T-Filter Element (0.5 µm, #8770-83B) |
| 2 Gasket (Teflon® or Nylon) | 6 CGA - 320 S.S. Coupler Nut |
| 3 CGA - 320 S.S. Nipple | 7 Teflon® tape |
| 4 Oil/Water Filter Housing w/ LCO ₂ Filter Element (#8784A) | 8 High Pressure Hose to Samdri® |



CAUTION: USE tousimis® LCO₂ FILTER ELEMENT (trc# 8784A) ONLY.

Operating pressure not to exceed 1000 psi

LCO₂ Filter Element Installation (#8784A)

STEP 1



After all of the pressure is safely bled from high pressure line, unscrew Oil/Water Filter Housing by hand.

STEP 2



Remove Oil/Water Filter Housing.

STEP 4



Place new LCO₂ Filter Element (#8784A) into lower Oil/Water Filter Housing. Let gravity hold element in place.

STEP 3



Position LCO₂ Filter Element (#8784A) into center of lower Oil/Water Filter Housing.

STEP 5



Carefully lower Oil/Water Filter Housing over replacement element (#8784A).

STEP 6



Hand tighten the housing body together firmly. O-Ring seats properly. Open main tank valve and check for leaks.

LCO₂ T-Filter Element Installation (#8770-83B)

STEP 1



After all of the pressure is safely bled from high pressure line, unscrew T-Filter cap using wrench.

STEP 2



Carefully remove T-filter element.

STEP 4



Top view of correctly installed T-Filter.

STEP 3



Install a new T-filter element (#8770-83B) into center of T-filter housing.

STEP 5



Hand tighten T-filter cap.

STEP 6



Use wrench, tighten the T-filter cap firmly. Open LCO₂ tank valve and check for leaks.



NOTE:
Do not over-tighten
T-Filter Cap.

FAQ

Should the LCO₂ tanks be secured?

YES. It is recommended that:

The LCO₂ Tanks be placed onto LCO₂ Tank Scales (trc #8770-54) and secured as per your facility safety regulations. Monitor the net LCO₂ used. When you approach 50% LCO₂ consumption, it is recommended that you replace the LCO₂ tank with a new one. Always keep several spare tanks of LCO₂ in reserve.

Do I need an In-line regulator between the tank and the SAMDRI®-PVT-3D?

NO. A regulator is not needed between the LCO₂ tank and the 3D. The 3D is designed to simply operate from LCO₂ direct tank pressure.

Is the chamber necessary to heat during BLEED mode?

YES. During the BLEED mode the HEAT switch should be kept ON. The HEAT mode will maintain chamber temperature automatically above 31°C and keep the process chamber dry.

How do I replace the #8784A filter elements?

To replace #8784A filter element, loosen and remove lower part B by turning counter clockwise. Remove old element and replace with new (See illustration p.24-25).

How do I secure the chamber lid?

Evenly finger-tighten the chamber lid with the 3 Knurled Nuts. Use even and steady finger pressure on all Knurl Nuts alternatively. Never use a non compatible tool or excessive force (i.e.: never use a wrench, etc).

Should I use “High-Pressure Head” tanks such as Helium or other high-pressure gases?

NO. Never use high-pressure tanks. Typical proper pressure range for LCO₂ tanks are between 750 – 900 psi. Higher pressures may damage SAMDRI®-PVT-3D and / or cause failures.

Can I use lower purity alcohol or acid in chamber?

NO. ONLY USE ULTRA-PURE ALCOHOL (ETHANOL, METHANOL, I.P.A., etc.) IN CHAMBER, NEVER ANY ACID! CONTACT TOUSIMIS PRIOR TO USING ANY OTHER INTERMEDIATE LIQUIDS.

Is a siphon or dip tube type LCO₂ tank necessary?

YES. It is essential that the LCO₂ tank has a siphon or dip tube on order to deliver liquid and not gaseous CO₂. The SAMDRI®-PVT-3D will not operate properly without liquid CO₂.

Why can I only use 50% of the net amount of LCO₂ and not 100% of the net LCO₂ within a new LCO₂ tank?

LCO₂ is lost during the process for 2 reasons:

- 1) As the liquid level drops in the LCO₂ tank, the gaseous head space created as the liquid level drops is taken up via gaseous CO₂ molecules.
- 2) Siphon (aka: dip-tubes) does not reach the absolute bottom of the LCO₂ tank.

What can I do to check that the BLEED rate is properly set should it appear unusually slow?

The flow rate should read 8 - 10 SCFH at the onset of the BLEED cycle. This setting should yield at average of approximately 100-150 psi/min reduction in pressure. This pressure reduction flow rate is the desired decompression rate between 1300 → 400 psi to avoid condensation.

You may periodically measure the BLEED rate (if desired) via the Flow Meter supplied by attaching the Flow Meter to the COOL/BLEED CO₂ Exhaust Outlet via a short (approximately 2') piece of Tygon tubing.

Flow Meter



What is the average FILL Time?

The FILL time should not exceed more than 2 minutes. Typically the chamber will FILL in less than 1 minute.

What is the average PURGE time?

The PURGE time will vary according to the volume of ethanol in the chamber. It takes approximately 7 minutes to purge 50% of the chamber volume (10-15ml Alcohol).

Is chamber pressure automatically regulated during Critical Point Passage?

YES. Chamber pressure will rise above 1072psi (Critical Pressure for CO₂) and automatically vent internally at 1350psi ±5% @ 20°C.

Is chamber temperature automatically regulated during Critical Point Passage?

YES. Temperature is maintained between 31°C-42°C automatically with HEAT switch ON.

Is there back-up pressure Safety?

YES. In addition to automatic internal pressure venting above the Critical Point. There is a “rupture disk” which automatically vents entire system at 2000psi.

What grade LCO₂ is best for the SAMDRI®-PVT-3D?

99.8% minimum purity.

May I replace or add parts without authorization from tousimis®?

NO. SAMDRI®-PVT-3D is a high pressure instrument with specific tolerances engineered into the design. Please contact tousimis® for authorization prior to attempting any desired changes.

What should I do if the initial COOL time is suddenly much slower than normal?

99% of the time the reason for this is lack of Liquid CO₂. The SAMDRI® is designed to COOL the process chamber via Liquid CO₂ and not gaseous CO₂.

Reasons that typically explain gaseous CO₂ delivery:

- 1) The Liquid CO₂ level has dropped below LCO₂ tank siphon (aka: dip tube) and only gaseous CO₂ is being delivered. [Note: The best way to monitor LCO₂ is via weight and not pressure. It is best to monitor tank weight via a "LCO₂ tank scale (trc#8770-54)"]
- 2) Gas supplier has delivered Gaseous rather than Liquid CO₂ by mistake. This can happen as the 2 tank types may look identical and the delivery/loading Gas Company staff accidentally delivered the wrong tank type to your site.

The simpler solution is to replace the existing supply tank with a new LCO₂ tank and initiate COOL mode. If the same problem persists; please contact customer service at either techsupport@tousimis.com or via tel# 301-881-2450.

OPTIONAL ACCESSORIES

Catalog #	Description
8777	SOTER™ CONDENSER
8760-01	Knurl Nut
8760-40	5 AMP Fuse
8770-10	Lamp, 120V/60Hz volt
8770-32	High Pressure Hose, 5 ft
8770-33	High Pressure Hose, 10 ft
8770-HPS	High Pressure Hose, Custom Length Up To 10m (33ft)
8770-45	Flow Meter - Use with any 1.25" dia. Samdri® Chamber (20 SCFH)
8770-50	Chamber Lid
8784	LCO2 Filter Housing for 8784A Filter Element
8784A	Replacement LCO2 Filter Element for 8784 Filter Housing
8784B	O-Ring for 8784 Filter Housing Seal
8784-05	Gasket for LCO2 Tank Connect
8770-83	Internal 0.5µm Brass Particulate Line Filter
8770-51	O-Ring for 1.25" Diameter Autosamdri® Chamber
8770-53	Power Cable, three prong, 120V/60Hz
8770-54	LCO2 Tank Scale, w/ Remote LCD Display (400lb capacity)
8770-55	LCO2 Tank Heater
8770-56	Step-Down Transformer, 220V → 110V
8770-57	Step-Up Transformer, 100V → 120V, 50/60hz
8761	Grid Holder, holds 12 grids.
8762	12 Sample Holder, each compartment measures 7.5 (dia.) x 6mm (ht.)
8763	24 Sample Holder, each compartment measures 4.5 (dia.) x 6mm (ht.)
8764	4 Sample Holder, each compartment measures 9 (dia.) x 16mm (ht.)
8766	Cover Slip Holder, for 9mm to 13mm diameter Glass Cover Slips
8766-01	Washer for Glass Slip Holder (for use with #8766)
8766-02	Stainless Steel Screens for Cover Slip Holder (for use with #8766)
8767	Cover Slip Holder, for 16mm to 22mm diameter Glass Cover Slips
8767-01	Washer for Glass Slip Holder (for use with #8767)
8767-02	Stainless Steel Screens for Cover Slip Holder (for use with #8767)

Contact tousimis @ 301-881-2450 or trc@tousimis.com for current pricing and availability, or visit our web site at www.tousimis.com.

WARRANTY

The SAMDRI®-PVT-3D is warranted to the original purchaser for two years from date of purchase against any defect in materials or workmanship. Should you have any questions, please feel free to contact us. If it is determined that the unit should be returned for repairs to our Service Department, we will send a written authorization for shipment. Parts and labor are free of charge; shipping charges are to be paid by customer (insure instrument at current list price).

DO NOT ship instrument via U.S. Mail. Use UPS, FedEx or other qualified shippers only.

Our mailing address: **tousimis research corporation**
2211 Lewis Avenue
Rockville, Maryland 20851
USA

Our shipping address: **tousimis research corporation**
Attention: Instrument Service Department
2211 Lewis Avenue
Rockville, Maryland 20851
USA

Telephone # 301-881-2450 Fax #301-881-5374 Email: techsupport@tousimis.com

REPACKAGING FOR SHIPMENT

After authorization for repairs is received (see warranty), this general rule may be followed in repackaging a tousimis® instrument for shipment:

- a) Attach identification tag to instrument. Tag should indicate owner name, the model and 4-digit serial number of the instrument, and the type of the service or repair desired.
- b) Secure chamber lid over a properly seated O-ring and tighten 8-knurl nuts evenly. Do not send the high-pressure hose or electrical power cord.
- c) Place instrument in original container, if available. If original container and packaging material is not available, new packaging may be purchased from tousimis

If original container is not used,

- d) Wrap the instrument in bubble plastic.
- e) Protect panel faces and instrument sides with foam or appropriate non-abrasive cushioning material. Use a minimum 6" of shock-absorbing packing material around all points of the instrument. Two double walled boxes are suggested for shipment; BOX-WITHIN-A-BOX AT 350LBS. TEST STRENGTH.
- f) Use heavy duty shipping tape or metal bands to seal container.
- g) Mark shipping container with "Delicate Instrument, Fragile", etc. and insure it.

Chamber Lamp Replacement Installation

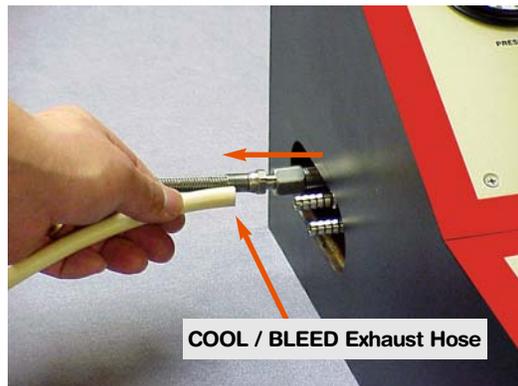
1. Turn the power switch "OFF" and unplug 120V power cord from the SAMDRI®-PVT-3D.



2. Disconnect 'PURGE / VENT' Exhaust Hose from the SAMDRI®-PVT-3D.

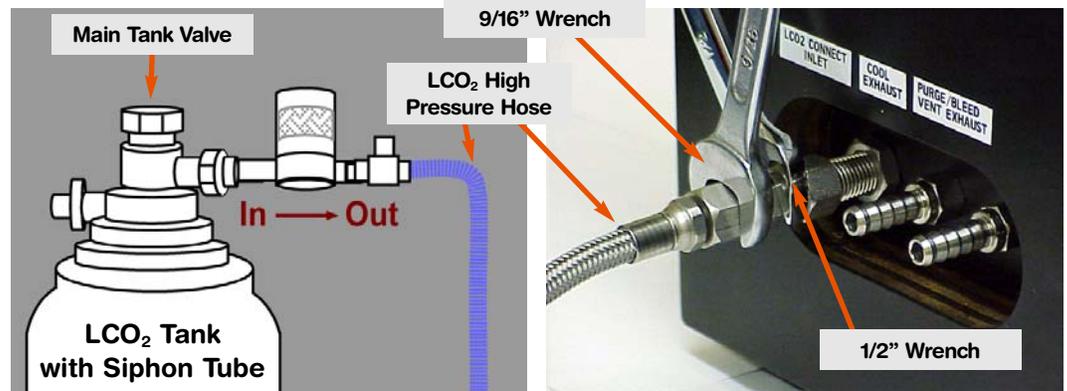


3. Disconnect 'COOL / BLEED' Exhaust Hose from the SAMDRI®-PVT-3D.



Chamber Lamp Installation

4. Close main LCO₂ tank valve, bleed LCO₂ High Pressure Hose. Disconnect 'LCO₂ High Pressure Hose' using supplied 9/16" Wrench and 1/2" Wrench from the SAMDRI®-PVT-3D Connect Inlet.



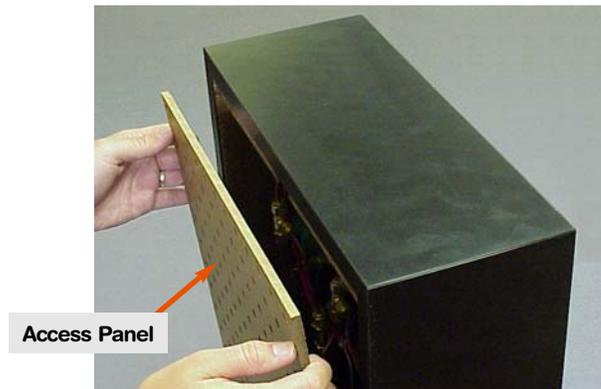
5. Carefully pick up the front end of SAMDRI®-PVT-3D and gently rest on its back end.



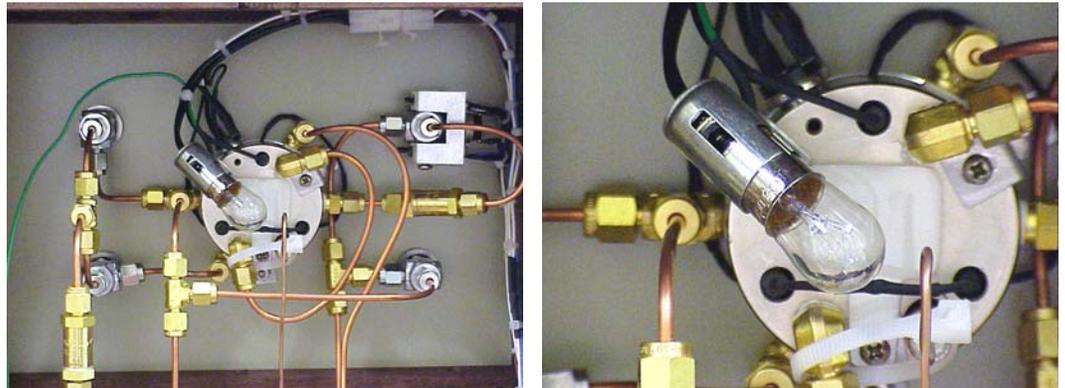
6. Unscrew the 4 black rubber legs that secure the access panel in place.



7. Remove and set aside the access panel. Note access panel orientation so that you may re-install panel in the same direction upon procedure completion.



8. Locate the Chamber Lamp as indicated below.

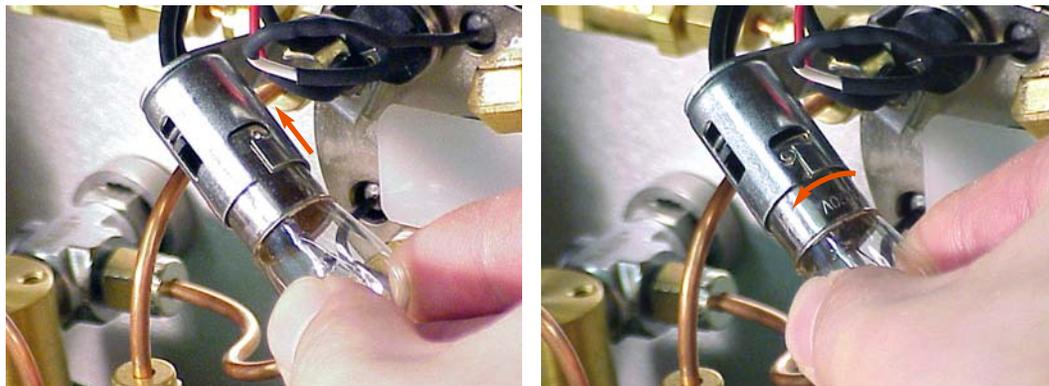


SAMDRI®-PVT-3D Chamber View

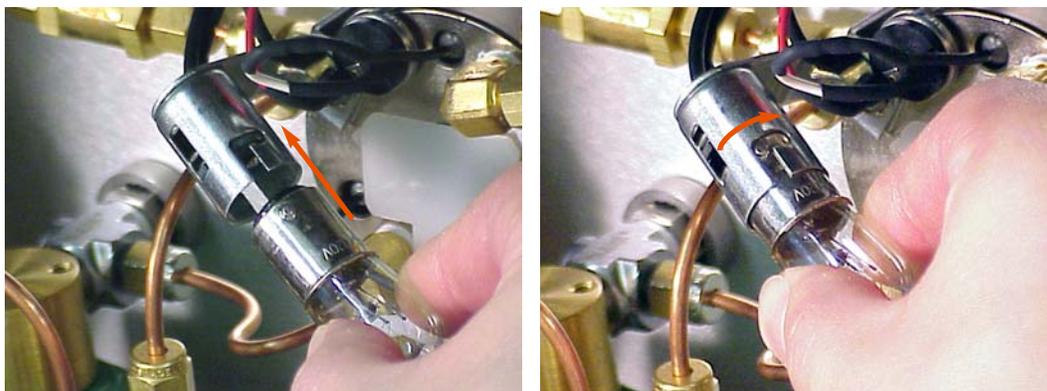


Chamber Lamp (Cat# 8770-10)

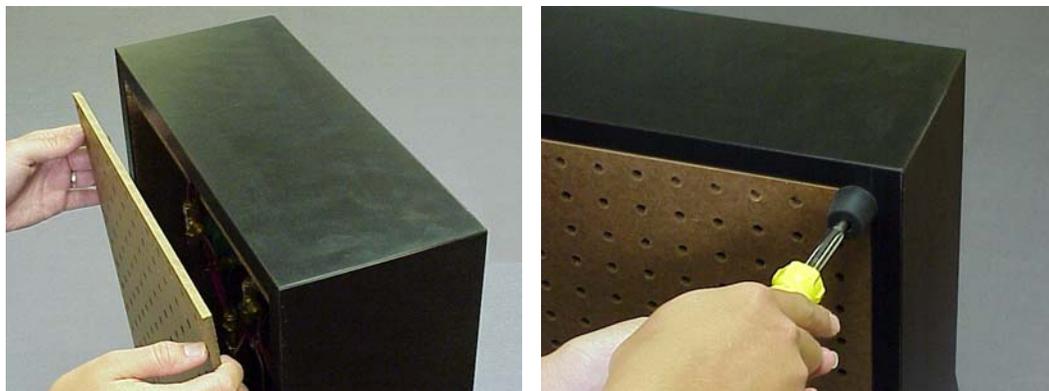
9. The Chamber Lamp is mounted into a "Bayonet" style lamp holder. Push Chamber Lamp towards the lamp housing and twist to the left to uninstall lamp (As shown below).



10. Install a new Chamber Lamp (Cat# 8770-10). Please note that you may need to support the Chamber Lamp housing with your free hand for support.

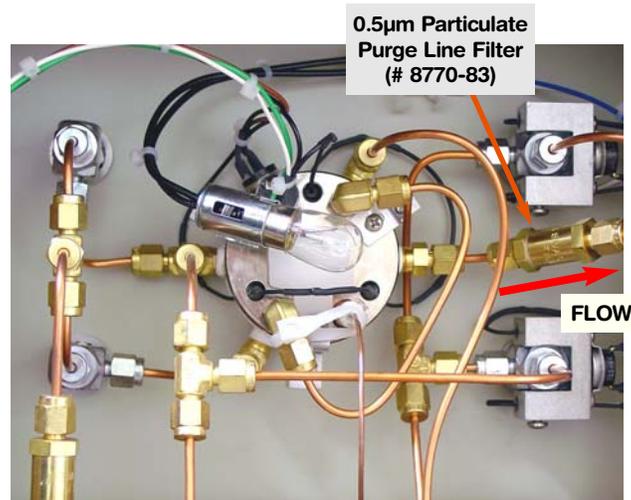


11. Re-install the access panel and re-connect power and all hose(s) prior to starting up system.

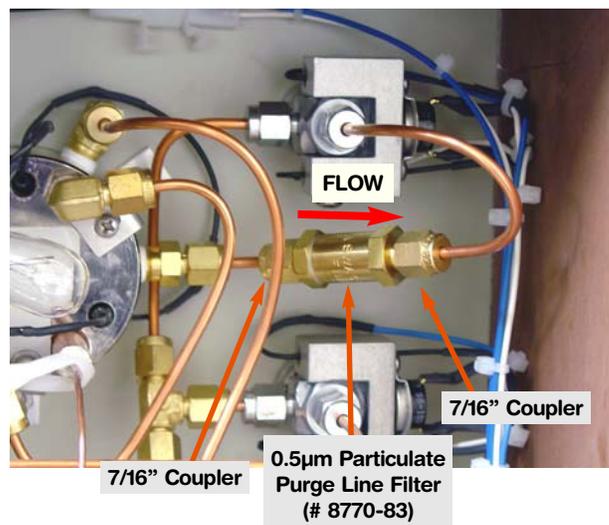


Purge Line Filter Replacement Instruction

1. Repeat Step 1-7 of previous section (Chamber Lamp Replacement Installation) in order to remove access panel.
2. Locate the Purge Line Filter (# 8770-83) as indicated below. Note the Purge flow direction.



SAMDRI®-PVT-3D Internal View



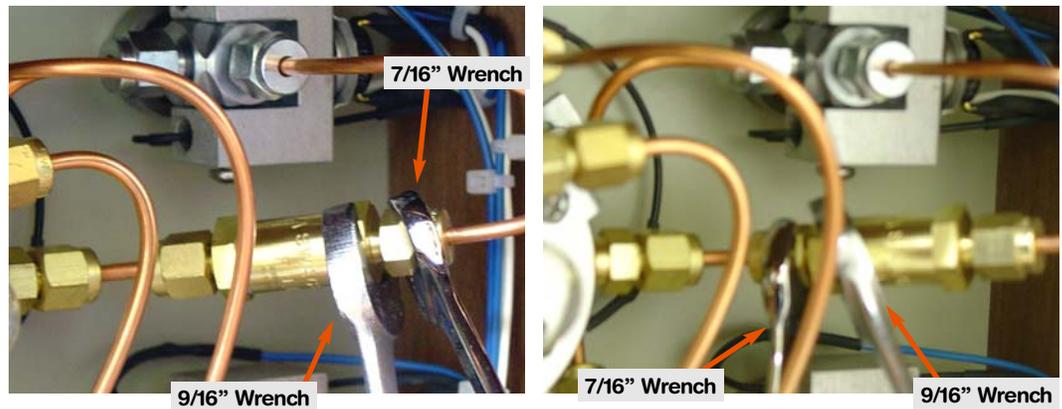
Purge Line Filter Replacement Instruction

Flow Direction



Purge Line Filter, 0.5 μm (#8770-83)

3. Disconnect Purge Line Filter by carefully holding Purge Line Filter steady with 9/16" Open-end wrench while disconnecting both 7/16" couplers. Carefully remove old Purge Line Filter and discard.

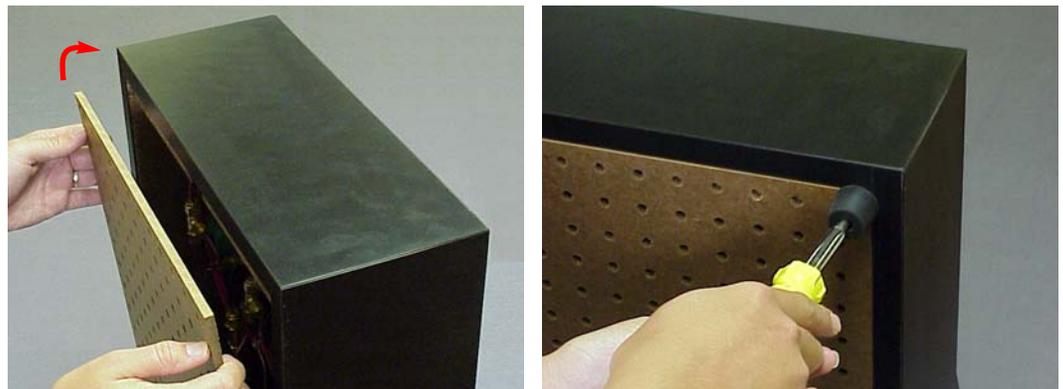


NOTE:

It is recommended that the "Purge Line Filter" be replaced every 6 months with regular use. Change more frequently with heavy use. Please contact techsupport@tousimis.com with any questions.

4. Install new Purge Line Filter paying attention to flow direction. Please note flow direction on the Purge Valve MUST be installed in-line with flow direction indicated in previous SAMDRI®-PVT-3D Chamber Plumbing picture.

5. Re-install access panel. Re-connect power and all connection hose(s) prior to starting up system.



Appendix C



Installing SAMDRI®-PVT-3D with SOTER™ Condenser

This section is for Installing SAMDRI®-PVT-3D with optional SOTER™ Condenser (# 8777). See Chapter 2 (p.4) for installing without SOTER™ Condenser.



* BOC gas suppliers refer to 99.8% purity grade as "2.8" grade. Air Products gas suppliers refer to 99.8% purity grade as "CP" grade. Always order LCO₂ tanks with Syphon (aka: dip) tubes. Minimum LCO₂ purity required is 99.8%.



** LCO₂ tanks used in most facilities have tare weights ranging from 90 - 120lbs (40 - 55kg) and are delivered as "full" with net 50lbs (23kg) of compressed LCO₂. GENERALLY, 50% IS USEABLE. You are advised to monitor the tank weight.

PREPARATION OF INSTALLATION SITE

Upon receipt of your SAMDRI®-PVT-3D, unpack the instrument carefully and check for any damage that may have occurred during shipment. Report (by Email trc@tousimis.com or Fax #301-881-5374) any irregularities immediately to tousimis®

Insist on clean surfaces. Minimum table top space of approximately 36"(92cm) Width x 30"(76cm) Depth should be allotted for the SAMDRI®-PVT-3D and optional SOTER™ Condenser with an additional 12"(31cm) x 12"(31cm) of floor space for the LCO₂ tank scale (See p.3).

Use bone-dry LCO₂ with a syphon (dip-tube) tank* only. Do not use pressurized LCO₂ with Helium or any other high pressure substitute gas. Secure LCO₂ tank according to your facility's safety protocol.

Tank pressure typically reads 800psi (±5%) at room temperature. The amount of LCO₂ in tank is best monitored with a LCO₂ tank scale (See accessories p.37).

Typical nominal LCO₂ tank weights for Net 50lb LCO₂ tanks:

- Full tank: 140 to 170 lbs / Tare of tank: 90 to 120 lbs.
- Most of the time, you may use 50% of a 50lb. net weight CO₂ tank.
- It is good practice to have spare LCO₂ tanks stored in reserve in case a tank runs out during mid process.

A properly grounded 120V/50-60Hz outlet should be located within 4ft (1.2m) of set-up site.

INSTALLATION OF THE SAMDRI® -PVT-3D

This SAMDRI®-PVT-3D is supplied with the basic accessories for operation with LCO₂ as a transitional fluid (for optional accessories, see p. 37).

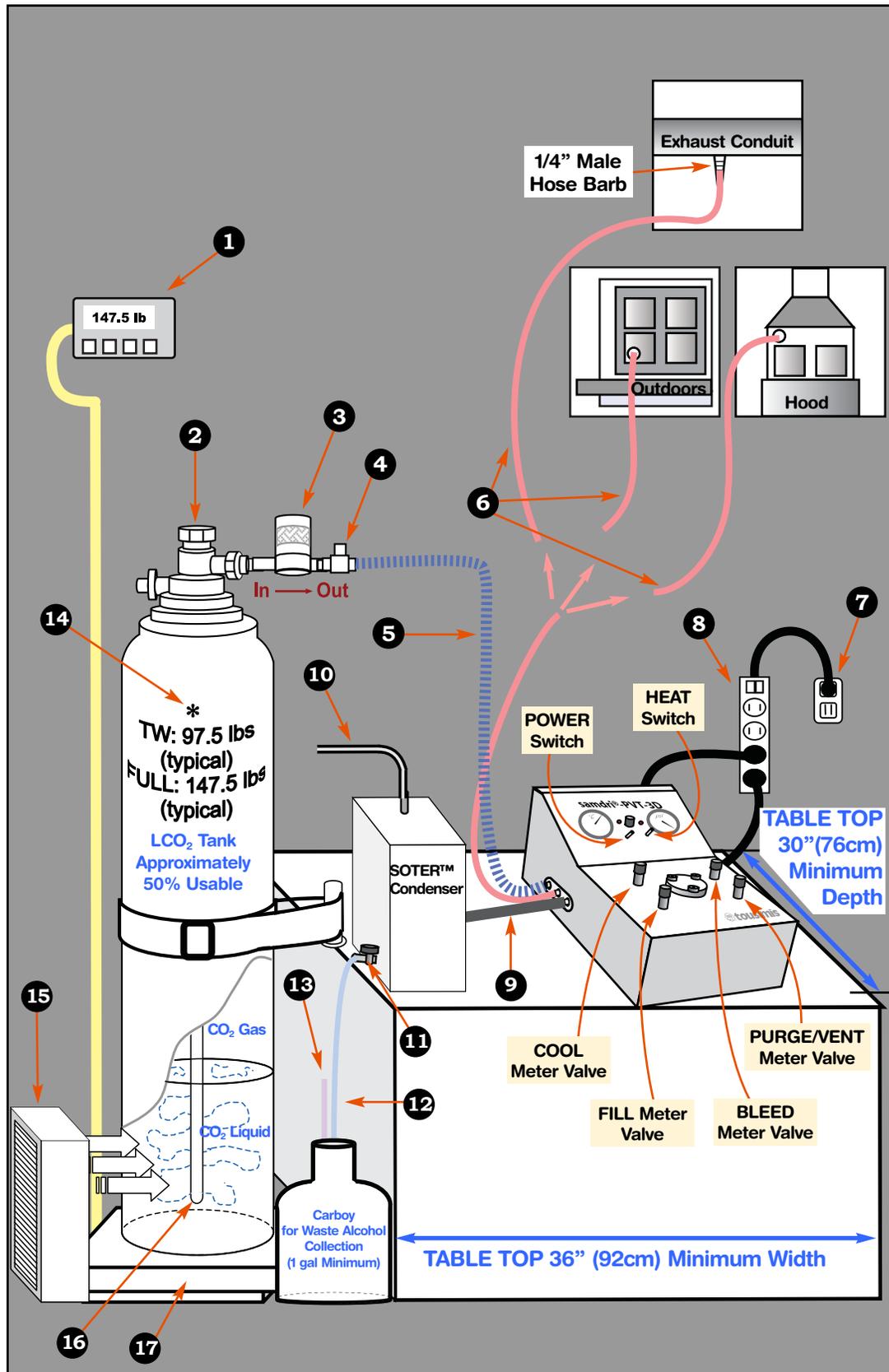
1. Position a LCO₂ tank with dip tube within 4 feet (1.2m) of the PVT-3D. For a longer high pressure hose length, see p.37, trc #8770-HPS. The LCO₂ tank should be mounted on a 400lb (182kg) capacity floor scale w/ remote LCD₂ display (trc #8770-54) to easily monitor LCO₂ supply**.

2. No pressure gauges or regulators need be installed between the supply LCO₂ tank and the SAMDRI®-PVT-3D LCO₂ connect inlet. The stainless steel-sheathed high pressure hose accompanying the unit is fitted with a CGA-320 hex coupling which accepts a 3/4" plastic gasket (supplied) and joins the hose directly to the CGA-320 threaded male nipple at the tank top (See p.31). ALL tanks are equipped with a rupture disc. All LCO₂ fittings and gasket surfaces should be kept clean and dry.

NEVER OVER-TIGHTEN FITTINGS...TUBING ATTACHED MAY BE DAMAGED.

SAMDRI®-PVT-3D Setup Overview

- 1 Remote LCD readout of LCO₂ Tank Weight
- 2 LCO₂ Cylinder ON/OFF Valve
- 3 External tousimis® Combination Filter (#8784)
- 4 T-Filter w/ 0.5µm Element
- 5 High Pressure Hose (5ft long, other lengths available upon request)
- 6 COOL/BLEED CO₂ Exhaust Tube to Hood, Exhaust Conduit or Outdoors. White (1/4" ID), 10ft
- 7 120V Properly Grounded Outlet (from either Wall or Transformer)
- 8 Power Strip for PVT-3D and SOTER™ Condenser
- 9 Exhaust Connect to SOTER™ Condenser
- 10 Condenser Vent Outlet (1/4" ID) to Hood, Exhaust Conduit or Outdoors., 10ft
- 11 Condenser Drain Valve. Always maintain "OPEN" position during process run, except when relocating SOTER™ Condenser or emptying Carboy
- 12 Condenser Clear Drain Tubing (1/4" ID), 3ft, routed to Carboy
- 13 Carboy Exhaust Tube to Hood or Outdoors, 15ft.
- 14 Tare Weight Varies (Consult your LCO₂ supplier or may be stamped on tank)
- 15 Ceramic Heater or Heater Jacket may be required in a cold tank placement position
- 16 Syphon(Dip) Tube
- 17 LCO₂ Tank Scale with Remote LCD Display (trc# 8770-54)

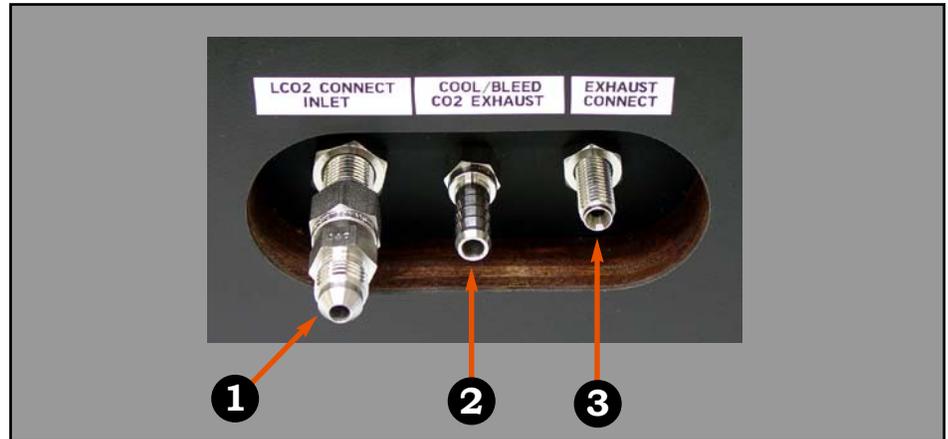


 * At Room Temperature (20°C - 24°C), average LCO₂ tank pressure is 800psi (+/- 5%).

3. The tousimis® LCO₂ Filter Assembly (tousimis® catalog #8784) is factory installed directly onto the high pressure hose (See p.31-32). This helps prevent sample contamination from oil, water and particulates. Change the #8784A filter element with every 500lbs (226.8kg) LCO₂ tanks changes. The secondary 0.5µm T-Filter element may be removed, ultra-sonicated and reinstalled every 1 year.

SAMDRI®-PVT-3D Inlet / Outlet Connect

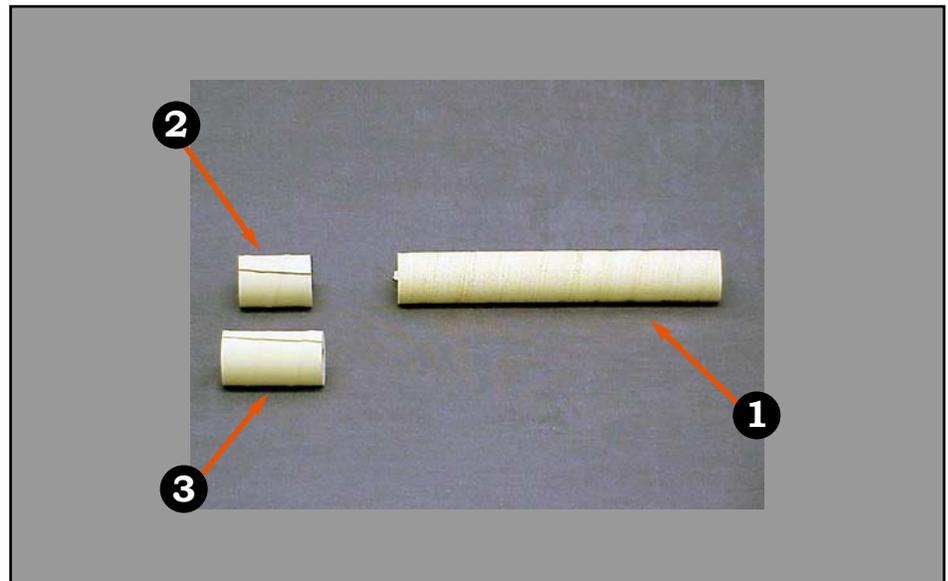
- 1 LCO₂ Connect Inlet
- 2 COOL/BLEED Exhaust Outlet
- 3 Exhaust Connect to SOTER™ Condenser (PURGE/VENT)



Exhaust Connect Line, Exhaust and Condenser Vent Insulates

4. Connect the SOTER™ Condenser to SAMDRI®-PVT-3D through 'Exhaust Connect', see picture below.

- 1 Exhaust Connect Line (7.0" Length)
- 2 Exhaust Insulate (1.75" Length)
- 3 Vent Insulate (2.25" Length)

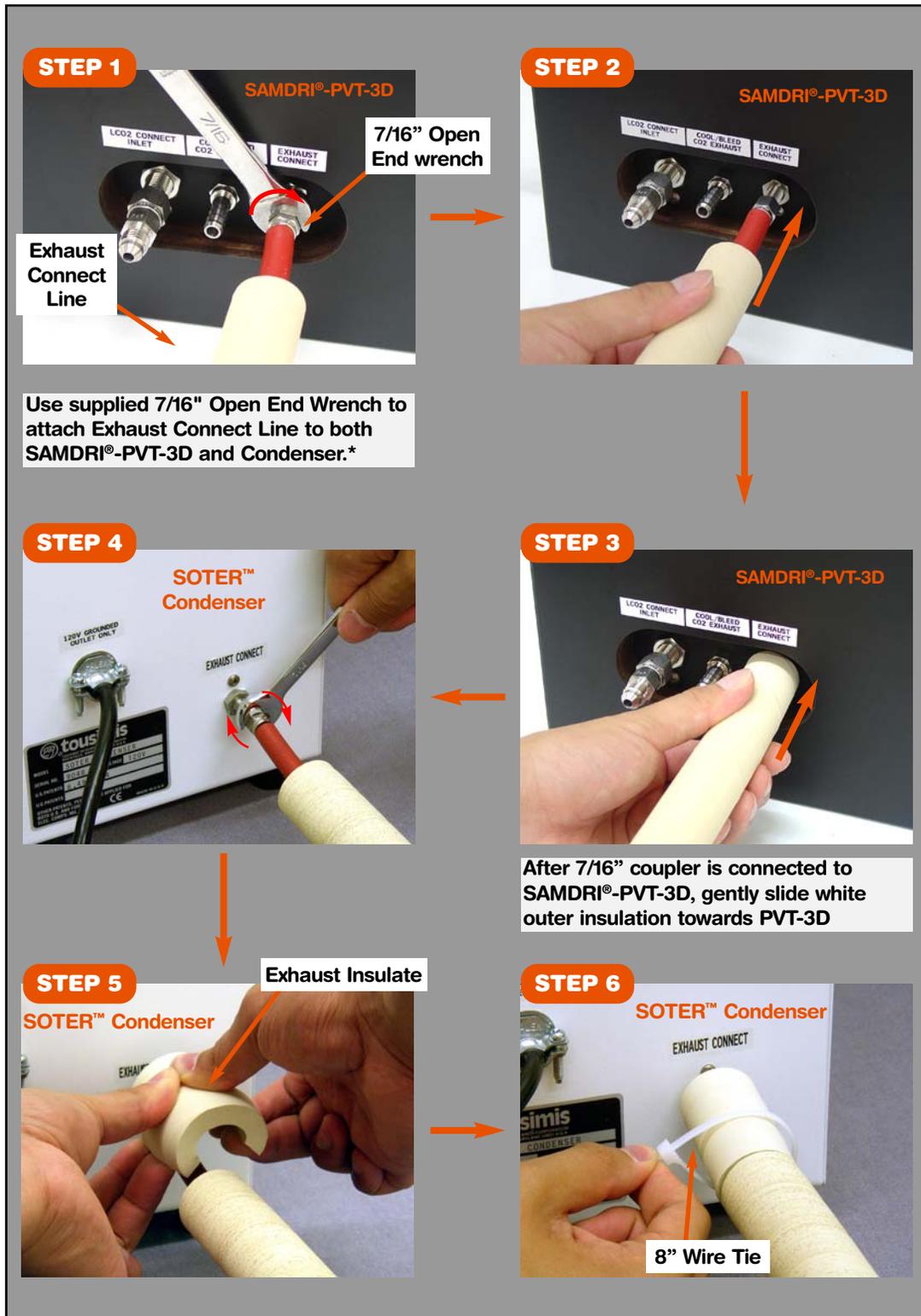


Connecting SOTER™ Condenser to SAMDRI®-PVT-3D



* NOTE:

Never over-tighten 7/16" fittings that connect "Exhaust Connect Line" to both SOTER™ Condenser and SAMDRI®-PVT-3D. Damage may result!



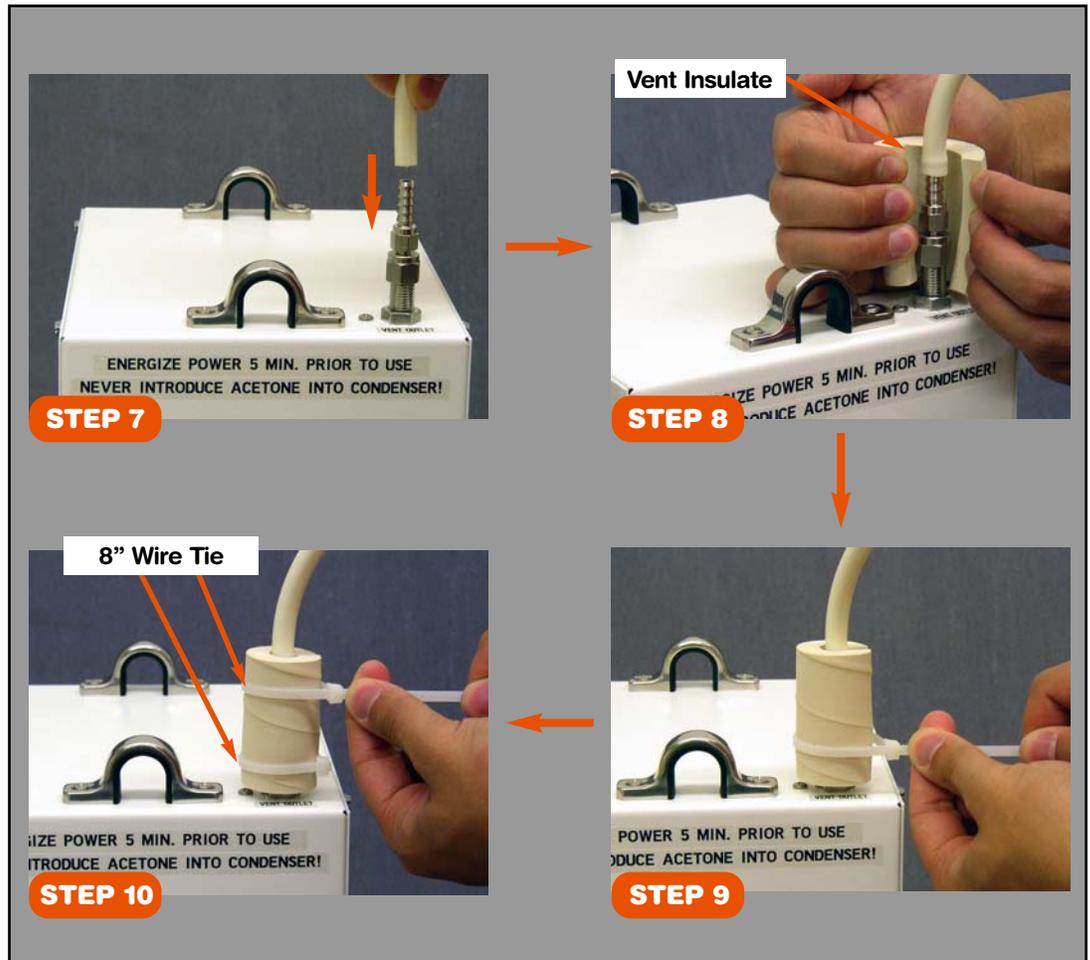
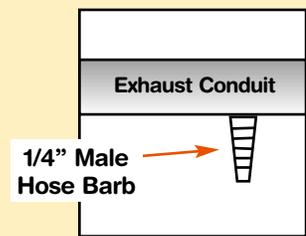
5. Connect the white 10' length of 'Condenser Exhaust Tubing' to the Condenser 'Vent Outlet' and cover the 'Vent Outlet' with the provided 'Vent Insulate' (See Step 8-9 on next page).

Connecting Condenser Exhaust



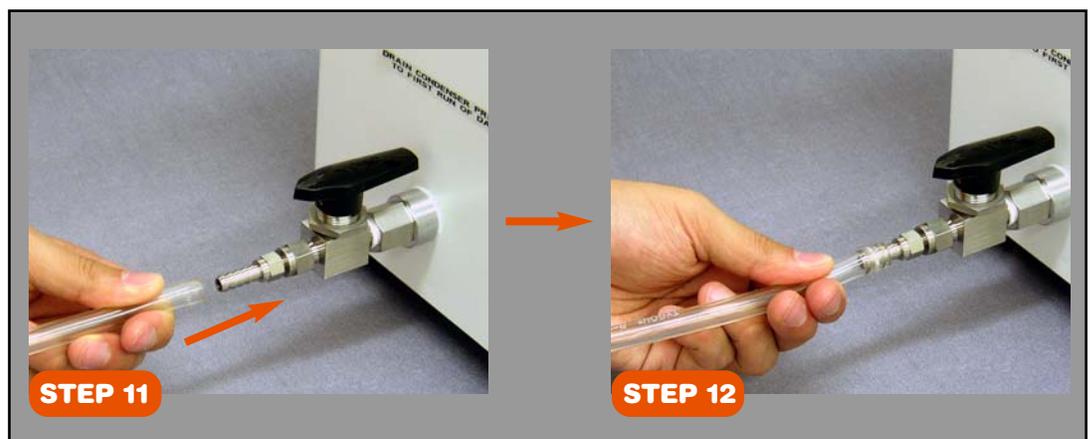
NOTE:

It is best to route Condenser Exhaust Tubing to facility Exhaust Conduit and attach via 1/4" male hose barb to conduit.



Connecting Condenser Drain

6. The 3' length of Clear Tygon 'Condenser Drain Tubing' should be placed onto the condenser's drain hose barb. The clear hose will allow you to visually see the alcohol draining into a carboy or Facility's Solvent Drain.



SOTER™ Condenser Exhaust Connect Overview

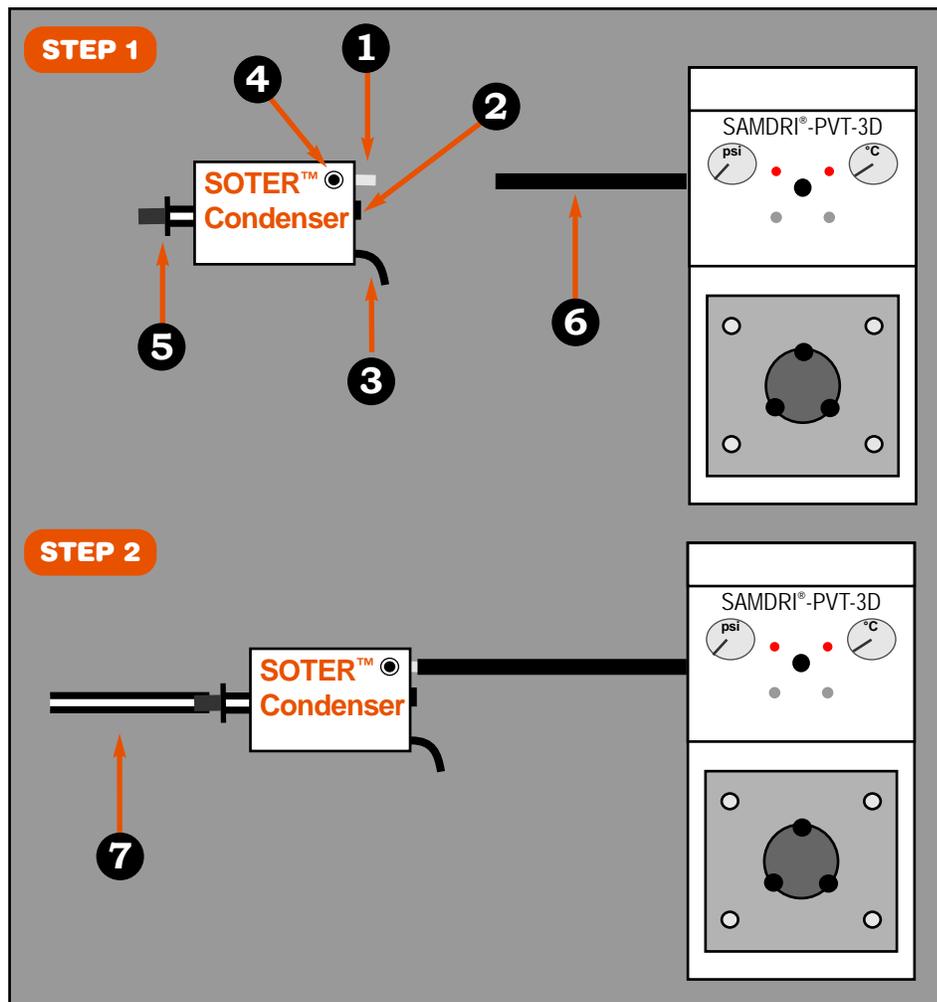
- 1 Exhaust Connect
- 2 Power ON / OFF
- 3 120V Power Cord
- 4 Vent Outlet (Attach 10' White Exhaust Tubing to Exhaust Conduit)
- 5 Alcohol Drain Valve (Leave in "Open" Position, except when relocating SOTER™ Condenser).
- 6 Exhaust Connect
- 7 Attach 3' clear Condenser drain tubing and lead to a Carboy (1 gal minimum) or Solvent Drain



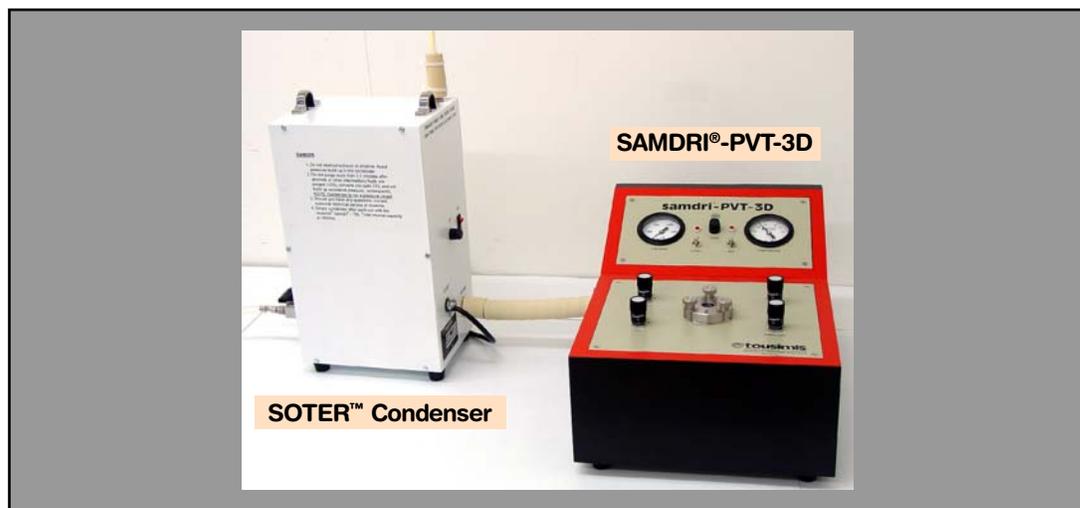
NOTE:

Do not over-tighten fittings on either end of Exhaust Connect. Check for leaks during the first run after connecting. Tighten as necessary with 7/16" wrench.

DO NOT OVER-TIGHTEN



SAMDRI®-PVT-3D with SOTER™ Condenser Overview

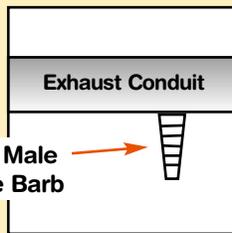


Connecting COOL/BLEED CO₂ Exhaust



NOTE:

It is best to route COOL / BLEED CO₂ EXHAUST to facility Exhaust Conduit via 1/4" male hose barb (if possible).

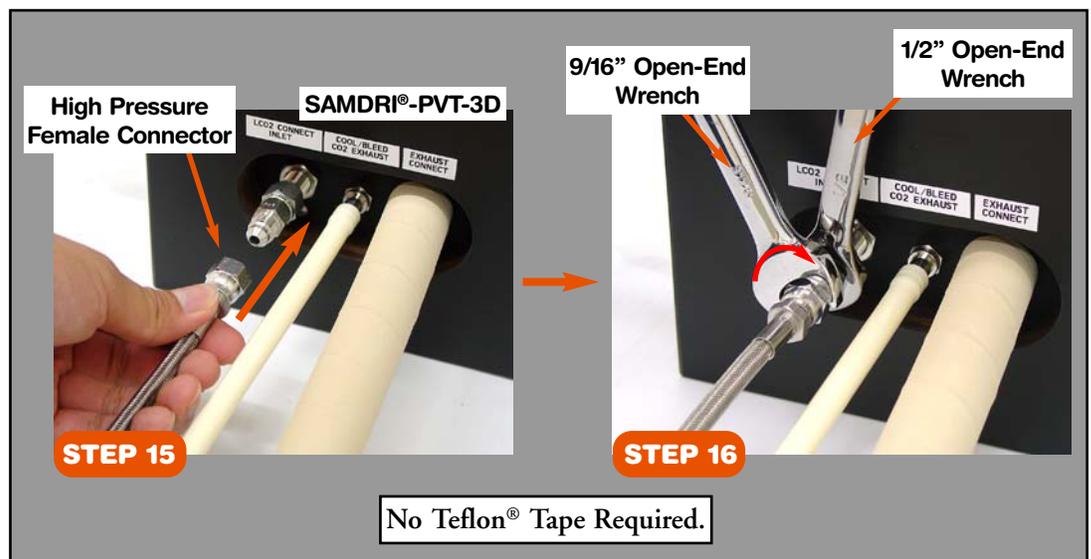


7. Attach the 10ft White exhaust tubing provided to the COOL/BLEED CO₂ Exhaust outlet on the left-hand side of the SAMDRI®-PVT-3D. Tubing should be free of kinks which could block passage of rapidly exiting noisy gas or solid flakes of CO₂.

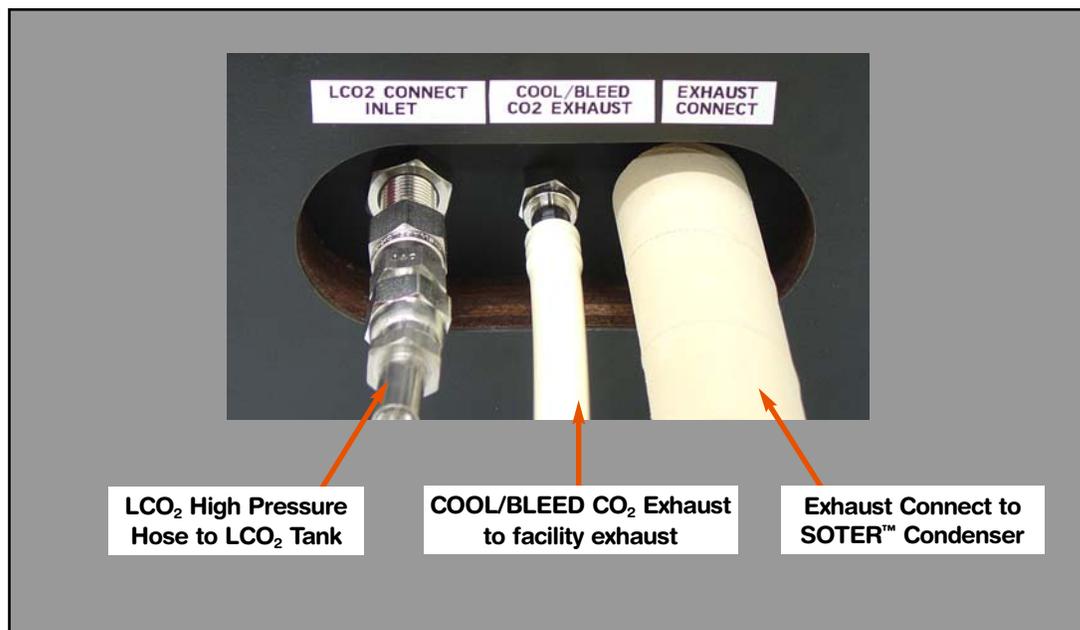


Connecting LCO₂ High Pressure Hose

8. Attach the HIGH PRESSURE HOSE to the SAMDRI®-PVT-3D LCO₂ CONNECT INLET. Gently tighten the High Pressure Female Connector using the two open end wrenches provided (the 9/16" onto the Hose and the 1/2" onto the Inlet). Stop at the feel of first resistance; re-tighten if necessary, should it leak.

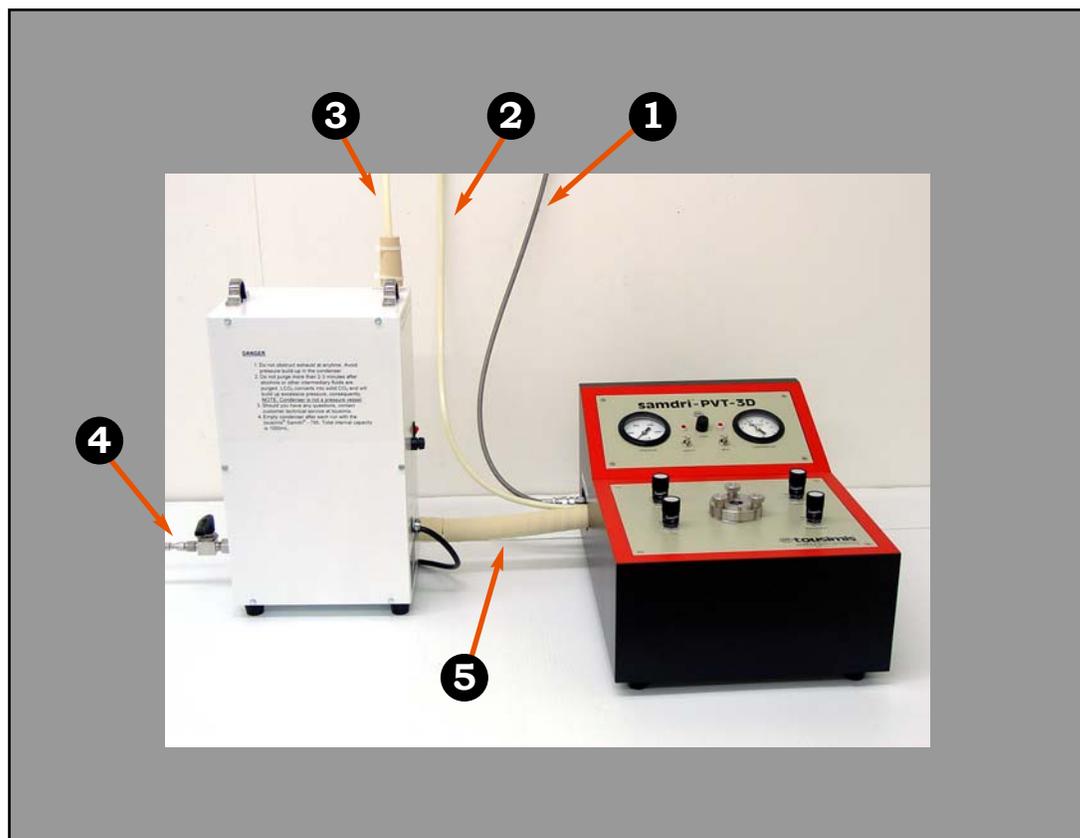


SAMDRI®-PVT-3D Inlet / Outlet Connect View



SAMDRI®-PVT-3D with Complete Connects

- 1 LCO₂ High Pressure Hose
- 2 COOL/BLEED CO₂ Exhaust Tubing
- 3 SOTER™ Condenser Vent Outlet
- 4 Condenser Drain Tubing to Carboy or Facility's Solvent Drain
- 5 Exhaust Connect



9. Connect Condenser Drain Tubing (clear) to a Carboy (1 gal minimum) or Facility's Solvent Drain. Condenser Drain Valve should always be maintained in the "OPEN" position during the process run, except when relocating Condenser or emptying Carboy. Connect Carboy Exhaust Tubing (white) to Fume Hood or Outdoors (See p.39 "Setup Overview").

Connecting Power Supply

10. Make certain facility 120V Power Supply is properly grounded and plug-in the Power Strip supplied. Next, plug in both the SOTER™ Condenser and SAMDRI®-PVT-3D Power Cords into the Power Strip.

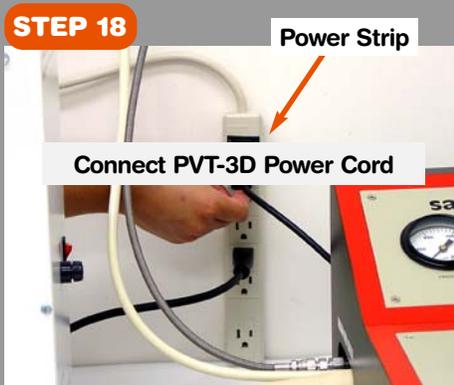


NOTE:

Please be certain that both SAMDRI®-PVT-3D and the SOTER™ Condenser power switches are in the 'OFF' position before plugging their power cords into the Power Strip.

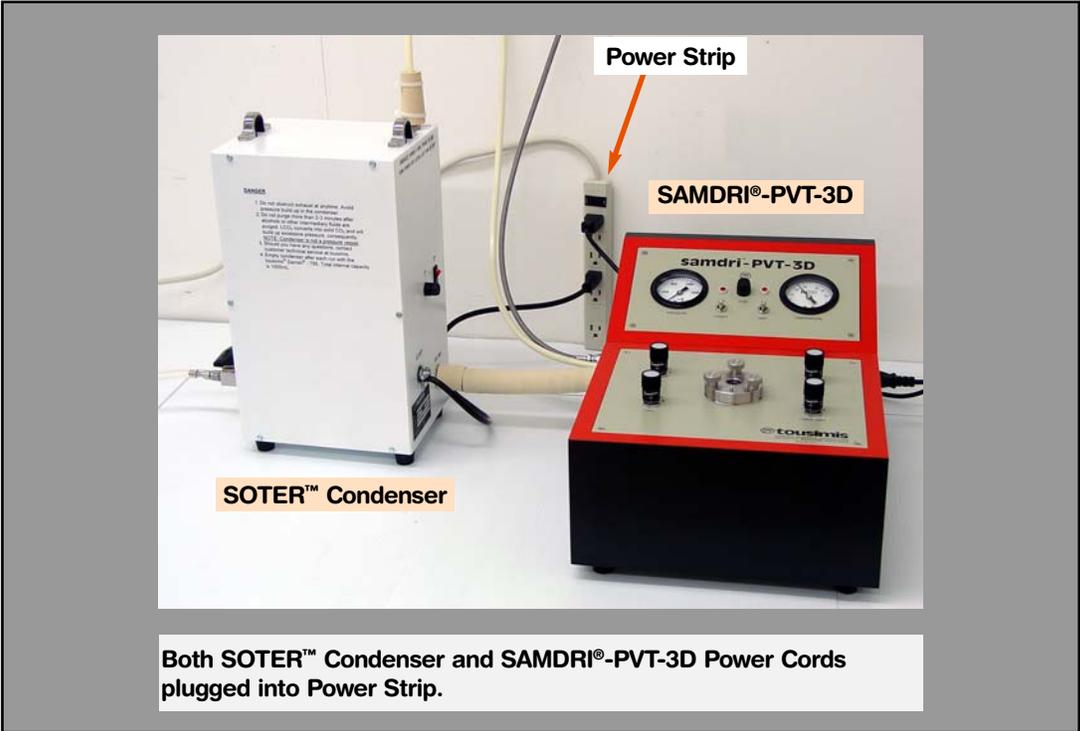


Connect SOTER™ Condenser Power Cord



Connect PVT-3D Power Cord

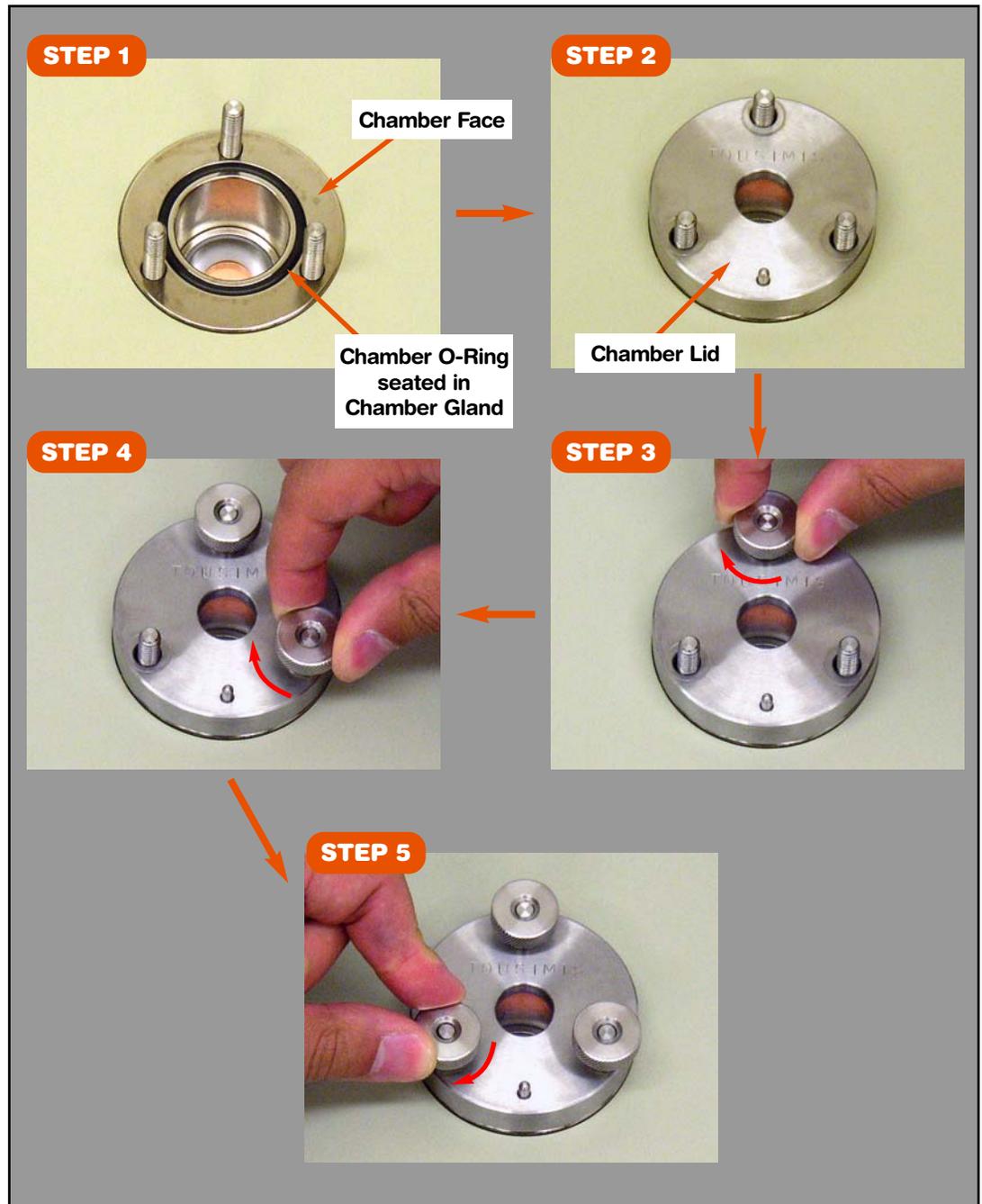
SAMDRI®-PVT-3D Set-Up Overview



Closing SAMDRI®-PVT-3D Process Chamber

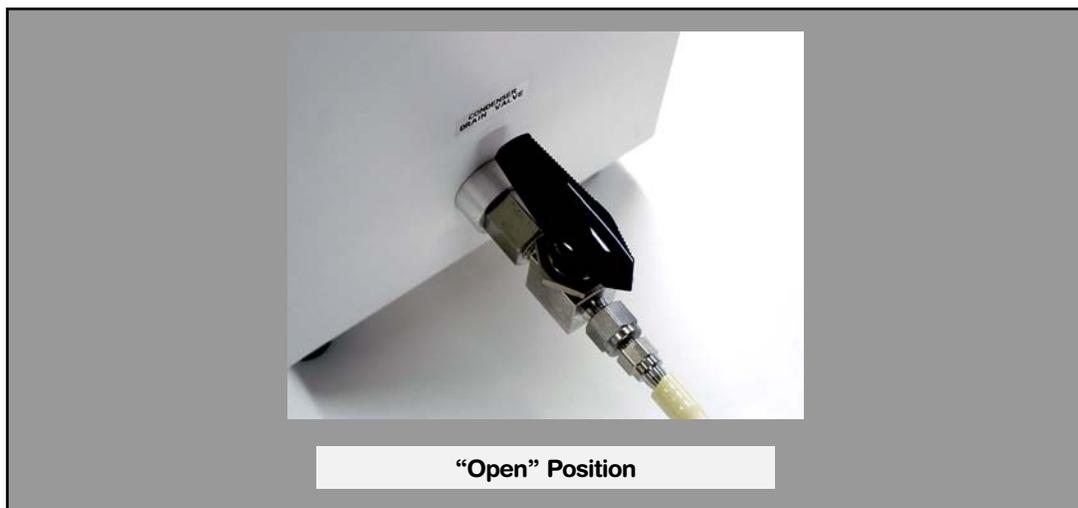
Chamber Sealing Procedure

1. Prior to placing chamber lid onto chamber, be sure that o-ring and surrounding chamber surface is clean and moisture free (NO Grease).
2. Carefully align chamber lid over chamber and carefully lower onto chamber face.
3. Carefully place the 3 knurl nuts onto each appropriate chamber post and turn in a clockwise direction until flush with chamber.



SOTER™ Condenser Drain Valve

Condenser Drain Valve. Always maintain “OPEN” position during process run, except when relocating SOTER™ Condenser or emptying Carboy.



SOTER™ Condenser Power Switch

Turn SOTER™ Condenser Power Switch “ON” 3-5 minutes prior to process run.

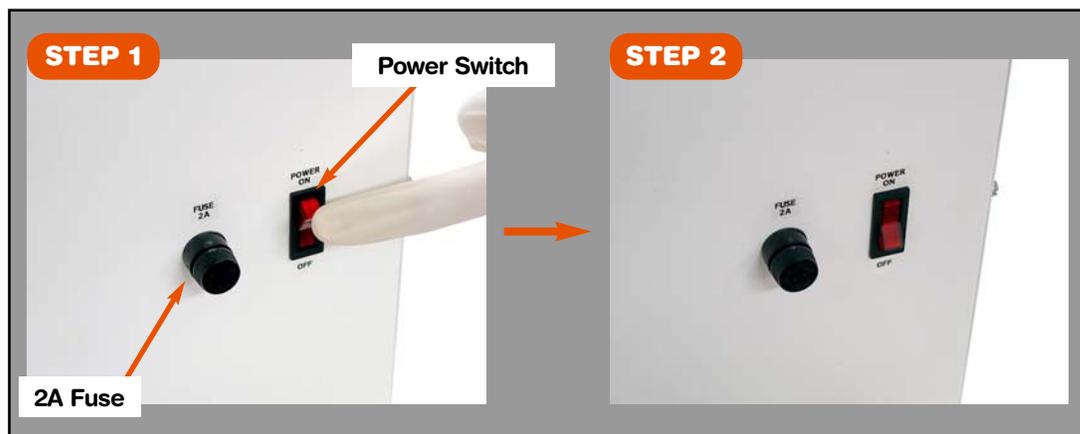


ILLUSTRATION INDEX

- Chamber Care, 23
- Chamber Lamp
 - Chamber Lamp, 34
 - Installation, 35
 - Removing, 32-34
- Flow Meter, 28
- General Form of Phase Boundaries Pressure vs. Temperature for CO₂, 20
- Isothermal of Carbon Dioxide and an Average Drying Path of Sample in the SAMDRI®-PVT-3D, 18
- LCO₂ Filter Assembly, 24-25
- LCO₂ T-Filter Element Installation, 26
- Purge Line Filter Replacement Instruction, 36-37
- SAMDRI®-PVT-3D, 2
- SAMDRI®-PVT-3D
 - Chamber O-Ring, 7
 - Dryer, 2
 - Exhaust Hose Connect, 6
 - High Pressure Hose Connect, 5
 - Inlet/Outlet Setting, 7
 - Metering Valve w/ Vernier Handle Setting Example, 21
 - Power Connection, 4
 - Setup Overview, 3, 39
 - Venting Precautions, 20
- SAMDRI®-PVT-3D Operation
 - BLEED Valve Adjust, 11
 - COOL Valve Adjust, 9
 - FILL Valve Adjust, 9
 - PURGE-VENT Valve Adjust, 10
 - Turning HEAT ON/OFF, 10
 - Turning Power ON/OFF, 8
 - Typical Drying Cycle, 16
- SAMDRI®-PVT-3D with SOTER™ Condenser
 - Chamber LCO₂ Supply Connect, 44
 - Condenser Drain, 42
 - Condenser Exhaust Connect, 42
 - Drain Valve, 49
 - Exhaust Connect Line, Exhaust & Condenser Vent Insulate, 40
 - High Pressure Hose Connect, 44
 - Inlet/Outlet Connect, 40, 45
 - Over View, 43, 45, 47
 - Power Switch, 49
 - Secure Chamber, 48
 - SOTER™ Condenser Connect, 41
 - Power Strip Connect, 46
- Typical Chamber Temperature during Pressure Reduction (BLEED), 15
- Typical Paths that Critical Point Passage May Follow during "Heat Phase", 14
- Typical Range of Paths that Pressure Reduction May Follow in the SAMDRI®-PVT-3D, 15



