

Standard Operating Procedure (SOP) for Tousimis Supercritical Dryer (Model: Samdri-795)



Tousimis Samdri-795 supercritical dryer

User Name: _____		Run #: _____	
Date: _____			
Start Time: _____	Cool Time: _____ (60-90 sec)		
Stop Time: _____	Purge Time: _____ (~ 10 min)		
CO₂ weight (lbs)			
Start: _____	CO ₂ used (this run): _____ (~ 2.0 lbs)		
Stop: _____	Total CO ₂ (this tank): _____ (< 25 lbs)		
	Total CO ₂ (this filter): _____ (< 500 lbs)		
Heat Cycle		Bleed Cycle	
t (min)	T (°C)	P (psi)	
0			
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
Comments: _____			

Signature: _____ Form Last modified: March 19, 2003			

User log sheet

NOTE: This is the short SOP. The detailed SOP is found the Samdri-795 *User Manual* (S/N 8915). All users should read the user manual prior to operating the Samdri-795.

1. Turn on Samdri-795 by flipping the power switch on the right side panel. The vent light should illuminate. Leave the Samdri-795 on for *at least* five minutes before processing.
2. Fill out name, date, starting time, and run # on the log sheet.
3. Make sure the white COOL and clear PURGE-VENT BLEED hoses are leading to the 'dirty' fume hood. *Also make sure the PURGE-VENT BLEED hose exhausts into a beaker to catch any purged liquids.*
4. Set the purge timer to '2' (or 1.5, 1.75, etc. as necessary). This sets the purge time in five minute increments. A purge time of 10 minutes ('2' setting on purge timer) is usually sufficient.

NOTE: The user should not have to adjust any settings. The Samdri-795 has been adjusted to operate properly with minimal adjustments. Notify super-user if Samdri-795 does not operate properly.

5. Make sure the CO₂ tank is at room temperature. Open CO₂ tank valve. Measure weight (in lbs) and record total in the 'start' weight section of the log sheet. *Make sure that there is at least 25 lbs of CO₂ available (i.e. for a standard 50 lb tank, make sure that at most 25 lbs of CO₂ have been used.*
 - a. The tank should be on the scale and the digital read-out for the scale should be on. If it is not, remove the tank from the scale and press and hold 'on' and 'lb/kg' for several seconds.
 - b. The read-out should display '4400', then 'A_oF', and finally '0.0.' The scale is now zeroed and ready for use.
 - c. Place the CO₂ tank back on to the scale.
6. Open the Samdri-795 chamber. Fill the chamber with **ultra-pure** methanol (or ethanol; or IPA) and place sample in chamber making sure it is always covered with alcohol. The chamber should have sufficient alcohol in it to completely cover the sample. Close the chamber and tighten the three nuts uniformly by hand. *Never use a wrench to tighten the nuts.*
7. Press the COOL button. The Samdri-795 will cool the chamber to 0 °C (± 5 °C) and will automatically stop. Record the cool time on the log sheet. A typical cool time is 60-90 seconds.
8. Next, press the FILL button. The Samdri-795 will now fill with liquid CO₂. During fill, the chamber temperature is automatically maintained at $T < 10$ °C. After two minutes, the fill automatically stops.
9. The Samdri-795 now automatically advances to the PURGE mode (methanol or other ultra-pure alcohol is forced out of the chamber and is replaced completely by liquid CO₂). Monitor the chamber temperature (**check that the chamber temperature remains in the range -5 °C $< T < 10$ °C**). This ensures that the CO₂ remains in the liquid state. Note that the Samdri-795 will perform cooling automatically during the PURGE mode. During purge you will notice:
 - a. For the first few minutes the exhaust from the clear PURGE-VENT-BLEED hose will be clear liquid or slurry (i.e. alcohol). Make sure it is exhausted into a beaker or other proper container under the 'dirty' fume hood. The exhaust should be pure methanol (no contaminants!).
 - b. After all the alcohol has been purged from the chamber, only CO₂ will be exhausted. Because the CO₂ encounters a low-pressure environment (atmosphere) as it leaves the high-pressure chamber, it will cool and be exhausted as *clear white flakes*. This is normal. 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 CO₂ will not leave a wet spot on the cleanroom wipe, but alcohol will.

- c. The PURGE-VENT-BLEED hose may form condensation and/or ice due to the temperature drop as CO₂ exits the chamber. Wipe any condensation from the PURGE-VENT-BLEED hose with a cleanroom wipe to keep it dry.
 - d. During purge you will also notice ‘oily currents’ in the chamber. This is the intermediate fluid (alcohol).
10. Record the total purge time on the log sheet. The Samdri-795 will automatically advance to the HEAT cycle once the purge cycle is completed. Make sure the chamber is completely filled with liquid CO₂. If not, press the FILL button again to ensure complete filling of the chamber. During the heat cycle, record both temperature and pressure on the log sheet in increments of one minute. During the heat cycle you will notice:
 - a. *Schlieren* patterns: oily currents in the chamber caused by density variations of the CO₂ in the chamber.
 - b. Gradual increase in temperature and pressure of the chamber.
 - c. The critical point for CO₂ is at T = 31.3 °C and P = 1072 psi. The Samdri-795 has an automatic pressure valve that maintains the *maximum* pressure between 1200-1500 psi during heating.
11. Once critical point has been reached, continue HEAT cycle for another 2-4 minutes, recording the T and P values on the log sheet. Typical heat cycle time is 10 minutes. Then, proceed to the bleed cycle by pressing the BLEED button. Record T and P on the log sheet in 1 minute intervals. The chamber temperature will automatically be kept above the critical temperature T_C = 31.3 °C. The chamber pressure will gradually drop at a rate of around 100 psi/minute.
12. Once the chamber pressure is below 400 psi, press the VENT button. The chamber should reach atmospheric pressure in 3-5 minutes.
13. Once atmospheric pressure has been reached, open the chamber and remove your sample. Close the chamber immediately to prevent moisture from building up in the chamber.
14. Record the final CO₂ tank weight on the log sheet. Compute the CO₂ used in this run and enter in the log sheet. Compute the total CO₂ used for this tank (add the current CO₂ to the previous tank total). *This should be less than 25 lbs.* Compute the total CO₂ used for this filter (add the current CO₂ used to the previous filter total). *This should be less than 500 lbs.*
15. Enter any comments on the log sheet.
16. Close the CO₂ tank and leave the Samdri-795 in VENT mode for a few minutes to exhaust any remaining CO₂ in the system.
17. Turn off Samdri-795 and wipe the exhaust hoses. Roll up exhaust hoses and store.