

Operating Instructions – Critical Point Dryer

Manufacturer: Tousimis

Model: Autosamdri-931 w

Safety:

- **CO₂ at high pressure (800 psi/55 atm). Pay attention to loose connections or strange sounds.**
- **Inform the Core Facility staff for any safety issue or misuse of the instrument.**
- **Contact Information: Koby Gvili (E-mail: yaacov.gvili@gtit.edu.cn)**
Ricardo CHEN (Phone Number:131-125-38546)

1. Equipment and chemicals

- 1) Ethanol: >99.5% purity.
- 2) Beakers: 100 ml and 250 ml
- 3) Tweezers

2. Turn on the machine for preheating

- 1) Switch on the machine by pushing the **POWER** button up for 5 seconds. The lights above the **POWER** button and inside the chamber should turn on.



- 2) Make sure the 4 valves (**COOL, FILL, BLEED, PURGE/VENT**) are in their closed position (turned all the way clockwise).



Control board. The chamber is in the center.



- 3) Open the valve of the CO₂ tank (anti-clockwise).



CO₂ tank valve

3. Transfer the samples to the chamber

- 1) Manually, remove the three screws of the chamber.
- 2) Take the **Sample Container** from the small box stored in the drawer. Remove the lid of the **Sample Container** and put it into a 100 ml beaker.
- 3) Pour ethanol (Purity: > 99.5%) over the **Sample Container** until it is covered with ethanol.
- 4) Use tweezers to transfer the sample into the **Sample Container**. Remove the **Sample Container** from the beaker and screw the lid to it.
- 5) Transfer the **Sample Container** into the dryer chamber. Attach the chamber lid and tight up the three screws.



Sample Container

4. Cooling Mode

- 1) Open a window in the lab, place the CO₂ exhaust pipe out of the window. Prepare a 250ml beaker, and put the PURGE/VENT exhaust pipe into the beaker for collection of ethanol waste.



Exhaust pipes

- 2) Slowly open the **COOL** valve to decrease the chamber temperature to 0°C. When the temperature has decreased to around 0°C, close the **COOL** valve. A sound will be heard from the CO₂ exhaust pipe due to gas discharge.

5. Bleeding Mode

- 1) Slowly open the **FILL** valve. The liquid CO₂ will flow into the chamber and mix with the ethanol. When bubbles will appear around the chamber lid, the sample chamber is full with liquid CO₂.
- 2) Keep the position of the **FILL** valve at a level of 0.5 until the pressure increases up to 800 psi.

6. Permutation Mode

- 1) Keep the **FILL** valve at 0.5 level.
- 2) Slowly open the **PURGE/VENT** valve. Make sure that the level of the **PURGE/VENT** valve is lower than the level of the **FILL** valve to guarantee that liquid CO₂ is always covering the sample inside the chamber. The ethanol will be exhausted through the pipe and into the beaker.
- 3) When most of the ethanol has been removed, put a dry tissue paper at the end of the **PURGE/VENT** exhaust pipe to check if any additional ethanol is still coming out. Close the **PURGE/VENT** valve and then close **FILL** valve.

Note:

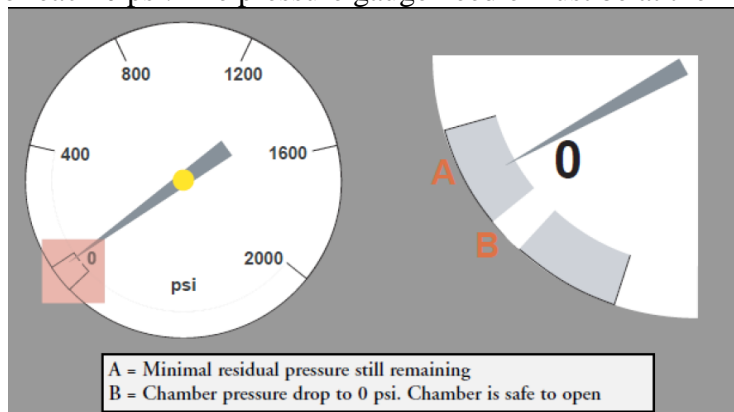
1. If the temperature has risen to above 10°C during this process, open the **COOL** valve until the temperature drops to 0°C and then close it.

7. Heating Mode

- 1) Make sure all 4 valves are closed and nothing coming out from the both 2 pipes.
- 2) Switch on the **HEAT** button (the light will be on). At this stage, the temperature and pressure will rise slowly. With time, the pressure will reach the range of 1,200-1,400 psi. The temperature will reach the range of 33-39°C. During this step, no action is required. The dryer will adjust to the critical point conditions.
- 3) When the light turns off (**HEAT** button is still on), maintain the critical point conditions for 5 minutes.

8. Bleeding Mode

- 1) Make sure the **HEAT** button is on and all 4 valves are closed.
- 2) Slowly and gradually open the **BLEED** valve. When the pressure decreases to below 400 psi, the **BLEED** valve can be opened to its maximum level.
- 3) Wait for the pressure to reach **0** psi. The pressure gauge needle must be at the **B** position for safety.



9. Take out sample and turn off the machine

- 1) When the pressure reaches 0 psi, remove the 3 screws of the chamber lid, take out the **sample container**. Prepare a tube or a box for your sample.
- 2) Close the lid of the chamber, put the **Sample Container** back to the box.
- 3) Close the valve of the CO₂ bottle.
- 4) Slightly open the 4 valves on the control board to exhaust the CO₂ leftovers inside the chamber.
- 5) Put the CO₂ exhaust pipe back into the room, close the room window, pour the ethanol to an organic waste container.
- 6) Switch off the instrument.