

## Attachment 1: Instrustions

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### Setup

- T-station 75: automatic pumping system for vacuum in Cryostat
- OptistatDN: cryogen free optical Cryostat
- Mercury iTC: control unit for Cryostat

### After 2 month of not using the cryogen free optical Cryostat

#### Vacuum in Cryostat

1. Take vacuum hose, which is connected to T-station, and join it to the Cryostat vacuum valve (1) for pumping the **Outer Vacuum Chamber (OVC)**. Valve vent of OVC should be closed!
2. T-station: Start the T-station ( $\langle \rangle$ ) and go to Turbo Set-Point (by  $\triangleright$ , TSP LED is on). TSP should be set to default "OFF". If not: Press the Enter ( $\leftrightarrow$ ) and then Next ( $\triangleright$ ) select "OFF", then press Enter to confirm it ( $\leftrightarrow$ ).
3. T-station: Press Next ( $\triangleright$ ) and get to Turbo screen (Turbo LED is going to glow). Press the Enter ( $\leftrightarrow$ ) and you can watch percentage of Turbo speed. Move to Gauge menu by pressing once Next ( $\triangleright$ ).
4. When the pressure reach 1 mbar you can start to slowly opening vent valve (1).
5. **GOAL:** OVC should be evacuated to less than  $10^{-4}$  mbar. During the sorb process Cryostat OVC must be pumped continuously.

#### Heating process - Activating the sorb

1. Plug cable (CWA0123) between Cryostat and control unit cable.
2. Control Unit: Set the Heater properties. Tap the Heater. Tap Resistance and set it to 150  $\Omega$ . Tap Voltage and change it to 12V. Tap Home to return to the home screen.
3. Control Unit: Set the Desorb process. Tap Control. Tap Sweep. Tap Load and select file DN-Desorb. Tap Close. Tap Heater auto. Tap Fixed.
4. Control that if heater is 100% then the Voltage is 12 V. After 600 min at 308 K the temperature is reduced by switching off the heater (Safety setting - Heater is set to 50K). After Cryostat is cooled back to room temperature, wait at least 2 hours before disconnecting the pump and closing the valve!

## Before measurement

### Preparing the System

1. Use normal cable for direct connection between control unit and Cryostat (Plugged into "SENSOR" connector).
2. control unit: Tap Heater. Tap Resistance Set and control that the resistance is  $20.22 \Omega$ . If not then change it.
3. Tap Voltage and control that it is 40 V.
4. Insert plug into sample space or control that the sample space is closed. (Without sample!)

### Pumping OVC and sample space

1. Take vacuum hose connected to T-station and join it to Cryostat vacuum valve (1) for pumping the **Outer Vacuum Chamber (OVC)**. Valve vent (1) of OVC should be closed.
2. T-station: Start the T-station ( $\langle \rangle$ ) and go to Turbo Set-Point (by  $\triangleright$ , TSP LED is on). TSP should be default "OFF". If not: Press the Enter ( $\leftrightarrow$ ) and then Next ( $\triangleright$ ) select "OFF", then press Enter to confirm it ( $\leftrightarrow$ ).
3. T-station: Pump the OVC to **at least**  $10^{-5}$  mbar. Press Next ( $\triangleright$ ) and get to Turbo screen (Turbo LED is lit). T-station: Press the Enter ( $\leftrightarrow$ ) and you can watch percentage of Turbo speed. Move to Gauge menu by pressing Next ( $\triangleright$ ).
4. When the pressure reach 1 mbar you can start to slowly opening vent valve (1).
5. After OVC is evacuated, close the vent valve (1) and switch off T-station. Then wait about 10 min until Turbo speed get to 0%. Change vacuum hose to sample space vacuum valve (4). Sample space vacuum valve vent is closed!
6. T-station: Go to Turbo Set-Point (by  $\triangleright$ , TSP LED is on). TSP should be set to pressure set-point lower than " $5.0^0$ ". If not: Press the Enter ( $\leftrightarrow$ ) and then Next ( $\triangleright$ ) select display with number (set point), then press Enter to confirm it ( $\leftrightarrow$ ). To change set-point press and hold Enter ( $\leftrightarrow$ ) to start number entry mode. Value for each digit is changed by Next ( $\triangleright$ ) and confirmed by Enter ( $\leftrightarrow$ ).
7. Prepare bladder full of helium gas (at least  $\phi$  50 cm, don't worry...) and closed it with stopper. (Stopper is on the bladder before helium is pump into it :) Pump the OVC to **around** 2.5 – 3 mbar. T-station: Press Next ( $\triangleright$ ) and get to Turbo screen (Should be zero all the time). Press the Enter ( $\leftrightarrow$ ). Move to Gauge menu by pressing Next ( $\triangleright$ ). (If vacuum pressure stops before it reach  $10^1$  mbar then the hose is not connected properly to the Cryostat or it has to be cleaned of dust.) **Now be fast, because the sample space is not made for low pressure condition.**
8. Close sample space vacuum valve vent (4) and change vacuum hose for the helium bladder with reduction. Now you can open the stopper. You should hear gas getting into the sample space.

### **Sample preparation for measurement**

1. Sample in plastic cuvette (1 cm, UV or VIS, Kartell, made in Italy) put into sample holder on the rod. The plug must be covered by a rubber.
2. Open sample space vacuum valve vent (4) to 1/2 turn. (Stopper on helium bladder is open!)
3. Unseal plug on sample space. And quickly change it for the sample rod. And seal the rod. (During changing, helium should be blowing from Cryostat.)

### **During measurement**

#### **Cooling the sample to base**

1. Connect funnel to the Nitrogen port (6, between valves 1 and 4). Use a filter in funnel. Fit 20 cm polyethylene tube on the second Nitrogen port (7).
2. Fill the reservoir through Nitrogen port (6) until liquid comes out of other vent. Usually, 3 dewar bottles.
3. Control unit: Tap Control and set "set point" to 77K or below.
4. Open the exhaust needle valve (5) by 2 or 3 turns. After 77 K let exhaust needle valve (5) open to 1 turn. (If it is closed too much, temperature will rise.)
5. For one measurement you don't have to refill the reservoir.

#### **Changing temperature (Control Unit Menu)**

1. Close the valve fully and open it by 1/4 or 1/2 turn.
2. Control unit: Tap Control. Set the temperature. And then change to Auto.
3. After the temperature is reached, optimize the flow of liquid nitrogen so that the heater voltage is less than 12V

### **After measurement**

1. Fast: Control unit: Tap Control Set temperature to room temperature. Close the needle valve (5) and allow to warm it to room temperature.
2. Slow: Let the needle valve open. It will slowly (12hours) warm itself after all nitrogen is used.
3. Next day, the sample should be removed of the cryostat

### **Other information**

1. If the heater is not working. Go to its settings on control unit and tap calibration. Then it should be OK.
2. Cryostat don't have to be cooled down directly to 77K.
3. Cleaning of windows in cryostat: optic cloth with methanol and then put it on wire rot. Clean the window by soft rotating of the rot and then do the same without methanol.
- 4.

## Literature

- Optistat DN2, Operator's Handbook
- T-Station 75. Instruction Manual

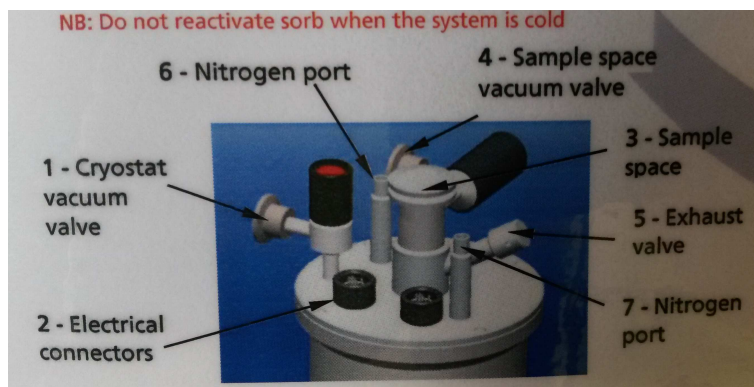


Figure 1: Cryostat valves and caps.