Users' operation manual for the Janis cryostat on the Maj K endstation at VLS-PGM

Sections

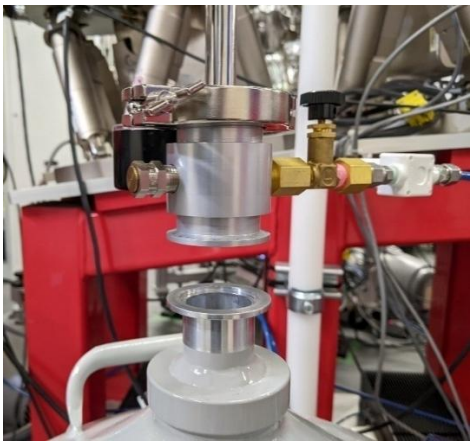
1. Filling the 20L dewar with LN2

All Users that are operating the system must have read the CLS 11.14.52.3 Cryogenic Safety document and completed the CLS Cryogenic Safety Training.

Filling and moving the dewar is considered Use of large amounts of LN2 (>10L) and requires additional PPE, such as face shield, safety glasses, cryogenic gloves, cryogenic apron, long pants and closed shoes.

Refer to the table in the CLS 11.14.52.3 Cryogenic Safety document for detailed PPE requirements.

The cryogenic transfer line needs to be removed from the top of the dewar prior to filling. See Section 6: *Removing the transfer line from the dewar*	  *Dewar shown with transfer line removed; transfer line seen stowed in plastic storage tube*

Remove the lower clamp and O-ring from the dewar assembly, store both parts safely. Insert the upper assembly into the white plastic storage tube.	 *Assembly disconnected from dewar*
Roll the dewar to the filling station located at the loading dock. Filling is started with the green start button at the station, marked 5-minute run. The dewar will fill quickly. Fill only up to the yellow “Max Fill” line marked on the outside of the dewar. This is to avoid liquid splashing when the dewar is moved and to reduce stress on the dewar. Regularly stop the fill by pressing the red Stop button and monitor the level by dipping the black plastic dipstick in the dewar. The dewar is full when you see that frost forms on the tip of the dipstick once withdrawn.	 Insert non-pressurized fill hose into the neck of the dewar as shown. 

Wearing a cryogenic apron, roll the dewar back to the Beamline area. Use caution to not splash any contents out of the uncapped dewar during transport. Re-attach with the O-ring and clamp.	

2. Setup

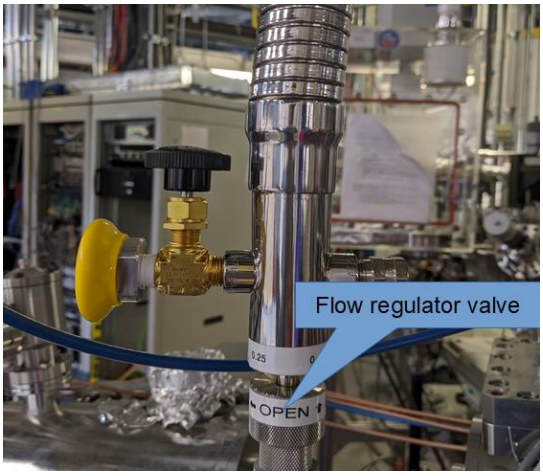
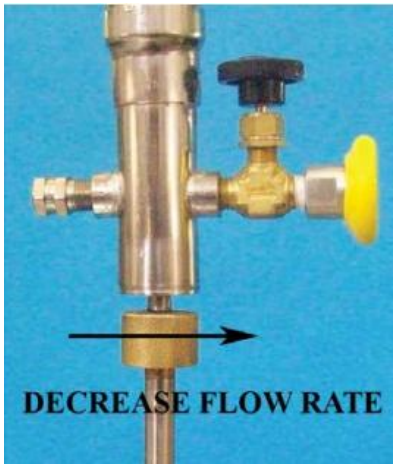
Refer to the table in the CLS 11.14.52.3 Cryogenic Safety document for detailed PPE requirements.

PPE which must be worn when setting up, operating, or taking down the cryostat will include:

- Safety glasses
- Face shield
- Cryogenic gloves
- Long pants and closed shoes

Safety glasses should be worn at all times due to the flow of cryogen.

Additional PPE may be required; refer to the CLS 11.14.52.3 Cryogenic Safety document.

Loosely close the flow regulator valve on the transfer line finger tight.	 *Dewar side of transfer line with flow regulator valve indicated* 

Close the dewar pressurization valve by rotating it clockwise.	
Remove the metal dust cap from the dewar assembly and store it in the bag attached to the dewar.	 *Uncapped dewar assembly*
A stepladder is needed beside the dewar to complete the next step. Wearing all PPE noted above, including face and eye protection, lift the dewar side of the transfer line out of its plastic storage tube. This tube must only be handled with cryo-gloves, as it will become extremely cold. Be aware that there is a small vent hole on the side of this tube that cold gas may exit from during the next step.	 *Dewar side of transfer line in white storage tube*

Insert the tube of the transfer line into the liquid nitrogen dewar, until it stops at the white collar.	 *Transfer line seated and collar threaded onto dewar assembly*
Loosely thread the O-ring collar onto the dewar tube.	

Open the dewar pressurization valve three turns.	
After a few minutes, the dewar pressure gauge will read approximately 4psi. *If any parameters of the cryostat system during operation aren't performing as expected, and/or as indicated in this manual, contact ASAP the Beamline Staff.*	

3. Starting operation

Before starting operation, please review the appendix, *Understanding the Temperature Controller.*

a. Adjusting the temperature setpoint

The endstation gate valve must be closed and both MCP and SDD detector HVs switched off whenever the cryostat is out of a steady equilibrium state, including whenever the setpoint temperature is adjusted.

If the setpoint is to be increased from a lower value, the SDD cooling should also be switched off (if applicable) and the sample moved to Load/Unload position.

See Section 5: *Shutting off the cryostat.*

Any temperature from slightly below ambient (~273K) down to approximately 90K can be selected in this step.

I. From the control panel of temperature controller, press the **SETPOINT** button.

II. Input the first temperature setpoint (in Kelvin) you need for your experiment using the number keys, then press **ENTER**.

You can change the setpoint again later once the system is running by repeating step a.

b. Activating the temperature controller

III. Press **Heater Range**
IV. Press the blue up or down arrows to change the range to **High**
V. Press **ENTER.**

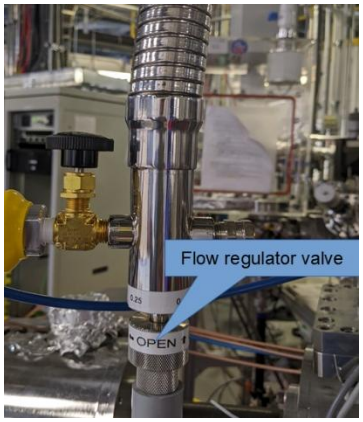
A red LED will light on the controller indicating Output 1.

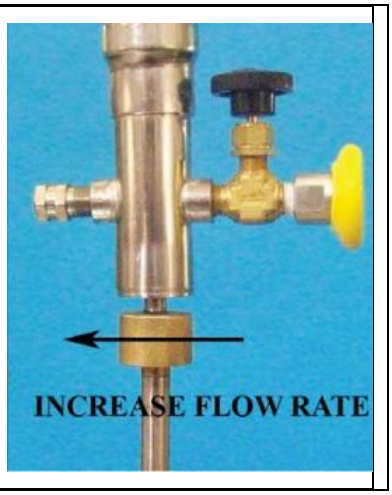
A **Heater %** number will appear on the screen of the temperature controller, next to the **heater range High.**

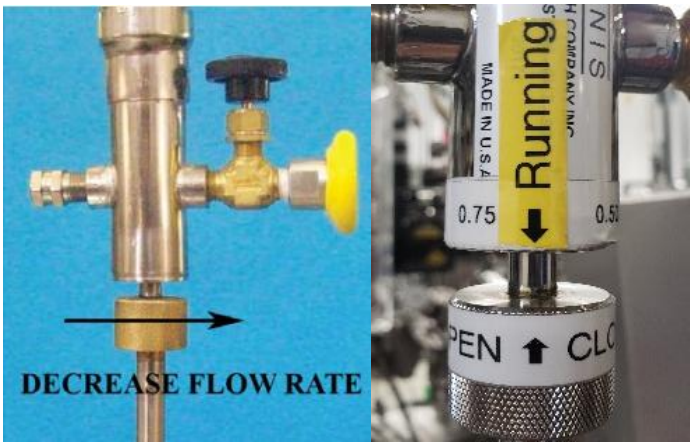
The front panel of the temperature controller, activated on range High, reading A, B, and setpoint 1 temperatures of ~100K, with 0% power flowing to the heater.

c. Starting cooling flow

Turn the flow regulator valve 2 full turns in the open direction.





After about 7 minutes, the temperatures indicated by the temperature controller should start to drop at roughly 0.3K per second.	
Once the **A** temperature is steadily dropping towards the setpoint • smoothly close the flow regulator valve one full turn, then • close the valve an additional half turn, until the position marked with the yellow “Running” arrow That is, the valve should be about one-half turn from fully closed.	 *The regulator valve and default “Running” setpoint.*

You may choose to adjust the setpoint temperature to achieve the desired final Sample holder (**B**) temperature.

After the **B** temperature readings stabilize at the desired value and the sample plate has equilibrated *(see Section 4: Changing samples)*, you may now begin XAS measurements of the sample as usual.

The temperatures can be changed by following Section a: *Adjusting the temperature setpoint.*

The flow regulator valve position marked “Running” is usually acceptable for all temperature setpoints.

While running, monitor the sensor temperatures and the **Heater %**.

Heater % might read 100% for a short period while reaching the set temperature and it should then stabilize to between 20% and 80%. It is normal for the cooling power to fluctuate moderately over second to minute scales.

If you observe the **A** temperature is above the target setpoint for a long period, and Heater % is near zero and/or unstable, the dewar fill level may be low, or the cooling

power through the transfer line may have decreased. If this situation persists, the flow regulator valve can be opened in steps of a tenth of a rotation.

If the **A** temperature is below the setpoint, or the **Heater %** is near 100% for a long period, the temperature controller is overwhelmed by the cooling, and the flow regulator valve can be slowly closed in small increments.

In either case, if you observe any unexpected variations in the cryostat during sociable hours, contact the Beamline Staff.

Heat flows slowly between the Sensor A position and the sample (Sensor B). If you wish to raise the sample temperature from a low value to a significantly higher value, it is often helpful to first input a setpoint temperature about 30K above the target sample temperature.

Once Sensor A reaches this raised setpoint, change the setpoint back to the actual target sample temperature you wish to reach.

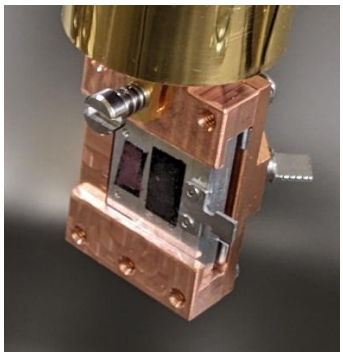
4. Changing samples

⚠ Caution

Sample plates which have been removed from the cold cryostat will remain extremely cold after transfer to the load-lock.

Burns may occur if the load-lock is vented and these samples are handled; use tongs to avoid contact with skin.

Sample plates may be loaded and unloaded according to the normal procedures for transfer from the load-lock.



A sample plate loaded for analysis.

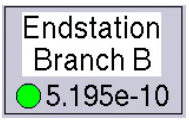
When a warm sample plate is introduced into the cold cryostat, the temperature indicated by **B** (sample holder) will rise, typically on the order of 15K.

As the sample plate cools, the **B** value will slowly drop back towards its initial value.

It is useful to record the **B** temperature before and after a sample change, to have a rough indication of when the plate temperature has stabilized.

It can take 30 minutes for the sample plate to fully equilibrate with the holder in this way, depending on setpoint.

5. Shutting off the cryostat

The goal of a controlled shutdown is to avoid contamination from gases condensed on the sample or thermal shock to the sample. • Close the endstation gate valve and switch off and ramp down all detector HVs as well as the SDD cooler. • Move the sample to the Load/Unload position using the preset positions screen. • From the temperature controller, progressively enter temperature setpoints in 50K steps back to ambient temperature (273K), waiting a few minutes between each step. • Between each step, monitor the chamber pressure for large spikes; pause at a temperature setpoint if the chamber pressure spikes.	
• Once the cryostat temperature (**A**) is back to ambient temperature (273K), fully close finger-tight the **flow regulator valve**.	
• Press the red **All Off** button on the temperature controller. ○ Ensure the temperature controller reads **0% Off** • Ensure the setpoint of the temperature controller is set back to 273K.	
• Close the dewar pressurization valve, and continue to Section 6: *Removing the transfer line from the dewar*	

6. Removing the transfer line from the dewar

Follow these steps after the cryostat is shut down. The transfer line is to be removed when refilling the dewar, or when the session is complete and the system will not be operated for some time.

In the next step any remaining dewar pressure will be vented.

Be aware that cold gas will be released around the collar, and the tube itself will remain extremely cold.

Wearing all required PPE (including face shield, safety glasses, and cryo gloves), unscrew the O-ring collar.

Remove the transfer line from the dewar and insert it back into storage.

Losen O-ring collar

Transfer line moved back to storage

After waiting 10 minutes for the dewar to settle, the metal cap can be loosely inserted back onto the dewar assembly to prevent debris falling into it.

7. Appendix: Understanding the temperature controller

The temperature controller adjusts the temperature of **Sensor A** to reach the configured setpoint. This sensor is located right beside the internal heater located inside the cryostat.

Sensor B is located on the sample holder itself and is a better indication of the true temperature of the sample.

It takes time for heat or cooling to flow to the sample holder (**B**) from **A**, and the sample holder (**B**) will also stabilize a little bit hotter than **A**.

The display of the temperature controller, showing:

- **Sensor A:** control temperature [K]
- **Sensor B:** sample holder temp [K]
- **1:** Target Setpoint temp [K]
- **Heater %.**

You must wait for the **B** temperature to reach the desired value, and for the sample plate to reach equilibrium, as discussed in Section 4: *Changing Samples,* before starting your measurements.