Quick Loader and CryoMat Loader User Guide

Introduction

CryoMAT Loader is a Quick Loader with a Cryo option, The CryoMAT Loader is designed for dehydrated sample transfer, cryogenic cooling, and temperature control within a SEM or DualBeam microscope. It has a single stage transfer, a pre-set sample temperature control, and is integrated into the existing DB vacuum system.

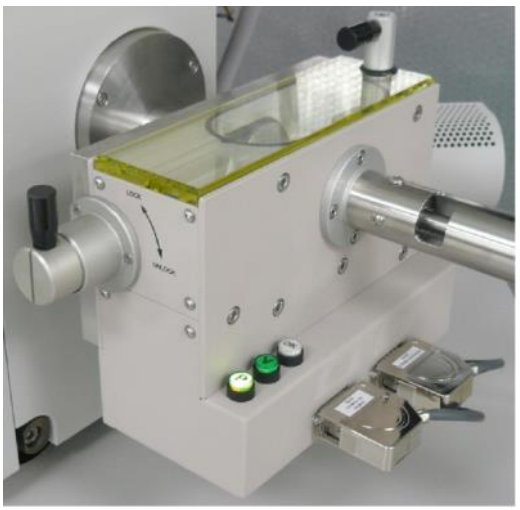
The CryoMAT combinations with QuickLoader is a simpler than a traditional cryo transfer system and can help stabilize non-wet materials (typically materials with < 2% water), operating temperature range +50^{0}C to -190^{0}C. The system does not require pre-freezing equipment or external vacuum system, and only requires a single transfer step using a QuickLoader. Temperature is microprocessor controlled and the stability is better than +/- 1 °C. The cooling design reduces liquid nitrogen consumption and in most cases requires only one filling per day.

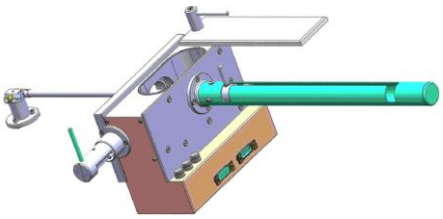
To be able to use CryoMat, the user must be familiar with the operation of QuickLoader.

QuickLoader: control buttons: P, V, OK

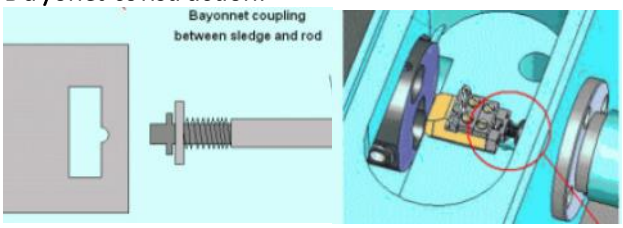
Control buttons are not shining when the system is recovering from vacuum status transition (e.g. immediately after the load/unload sample, during venting the chamber. The button is illuminated while in operation.

P (pump) button: press the P button to pump the loader chamber to required vacuum. When the P button is pressed, the stage moves to pre-defined loading position. If the system reaches appropriate vacuum level, the lever interlock is released and the gate valve can be opened.
V (vent) button: Press the V button to vent the loader chamber. The vent cycle continues till the P button is pressed or it is terminated by time-out.
OK button: Lights up when vacuum is reached. If the OK button light goes out: wait time has been exceeded; the appropriate vacuum for a transfer has been lost. Pressing the P button again will bring the system to vacuum OK status.





Bayonet construction:

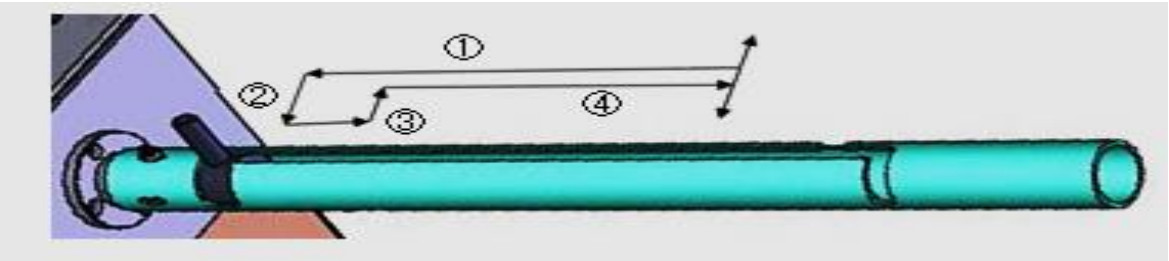


Loading Rod tracks: At the end of the track (farthest away from the loading chamber) is a slot for parking and attaching/releasing the sample carrier. At the other end of the track (closest to the chamber) is a large slot for coupling and decoupling the bayonet into or out of the sample shuttle when positioned in the Stage adapter in the microscope chamber.

Before loading/unloading:

Turns OFF the electron and ion beam accelerating voltage. Retract any retractable devices.

Sample Loading Sequence: 1-2-3-4 as indicated in the following drawing



1-Rod is into the chamber at hold position; 2-Rod rotates to the angle for separation; 3-pull to separate between Rod and the shutter; 4-Rod is retracted.

Step 1: Connecting Rod and shutter in the loading chamber

While holding the sample carrier on the loading table in the *loader chamber* move the rod out of the Parking position to the far left, and also to the back of the slot ,then forward to engage the bayonet into the sample carrier. Place the rod back into the Parking position after coupling to prevent the rod slowly creeping forwards. Close the loader chamber lid. After the lid is firmly closed the *P* button starts to shine.

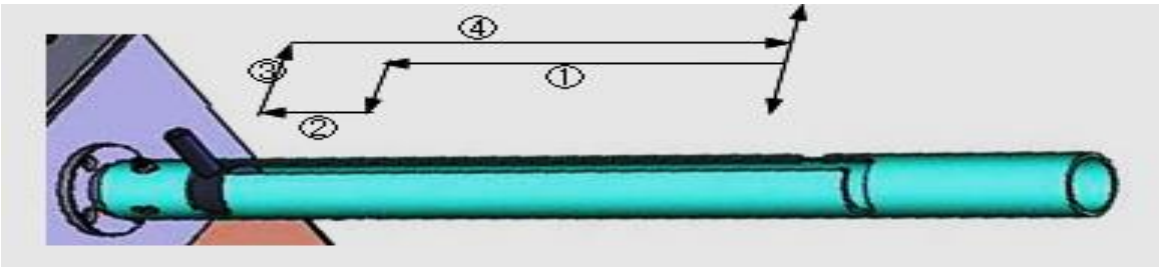
Step 2: Pump the Qloader chamber
Press the *P* button, the button stops to shine and the pumping cycle starts, the stage moves automatically to the loading position. The loading position is saved as pre-alignment procedure by a service engineer. If the loading position is wrong, it needs to re-align. When the vacuum in the loader chamber is correct the pump light starts to shine and the *OK* button lights up indicating operation can continue. The gate valve lever interlock is released.

Step 3: Open Gate Valve
Turn the Gate Valve knob lever from LOCK to UNLOCK position. Then pull the knob bar fully out from the first mark on the knob drum to the second mark. Turn anticlockwise of the knob bar to LOCK position.

Step 4: Insert the rod/sample
Activate CCD Quad and change to single quad mode so that successful loading can be confirmed visually. Move the loading rod from the Parking position into the chamber while still holding the rod bar. The Sample Carrier will engage with the Stage Adapter at the end of the rod travel. Turn the rod bar to the left (anticlockwise) to the base of the slot; pull back on the rod bar so it travels along the base of the slot, then turn to the right (clockwise) so that the rod bar is vertical and withdraw it back to the PARK slot at the far end of the rod guide.

While holding the sample carrier on the loading table in the loader chamber move the rod out of the Parking position to the far left, to the back of the slot and forward to engage the bayonet into the sample carrier. Place the rod back into the Parking position after coupling to prevent the rod slowly creeping forwards.

Sample unloading sequence: 1-2-3-4 as indicated in the drawing below:



1- Insert the rod; 2- Turn left (anti-clockwise angle) and push-in for connection; 3- Turn-Right (Clockwise) Connect the Rod to the shutter (lock it); 4-Pull-out to parking position.

Step 1: After the chamber lid is properly closed, the *P* button starts to light up. Press the *P* button, the button stop to shine and the pumping cycle starts, the stage moves automatically to the loading position at the same time. When

the vacuum in the quick loader chamber is correct the pump light starts to shine and the *OK* button lights up indicating operation can continue. The gate valve lever interlock is released at this moment.

Step 2: Turn the Gate Valve knob lever from LOCK to UNLOCK position. Then pull the knob bar fully out from the first mark on the knob drum to the second mark. Turn the knob bar (anticlockwise) to the LOCK position.

Step 3: Activate CCD Quad and change to single quad mode so that successful unloading can be confirmed visually. Move the rod as illustrated by the unload drawing. First, move the rod from the Parking position into the chamber while still holding the rod bar. When resistance is found turn the rod bar to the left (anticlockwise) to enter the bayonet. Push forward and turn the rod to the right (clockwise) and the bayonet will engage with the Sample Carrier on the Stage Adapter close to the end of the rod travel. Withdraw the rod back to the far end of the rod guide and place in the Parking position. The rod, bayonet and sample carrier are now out of the chamber and sit in the Loader chamber.

Note: Do not turn the rod at this movement, close the gate valve first. If not, the sample could drop inside the chamber.

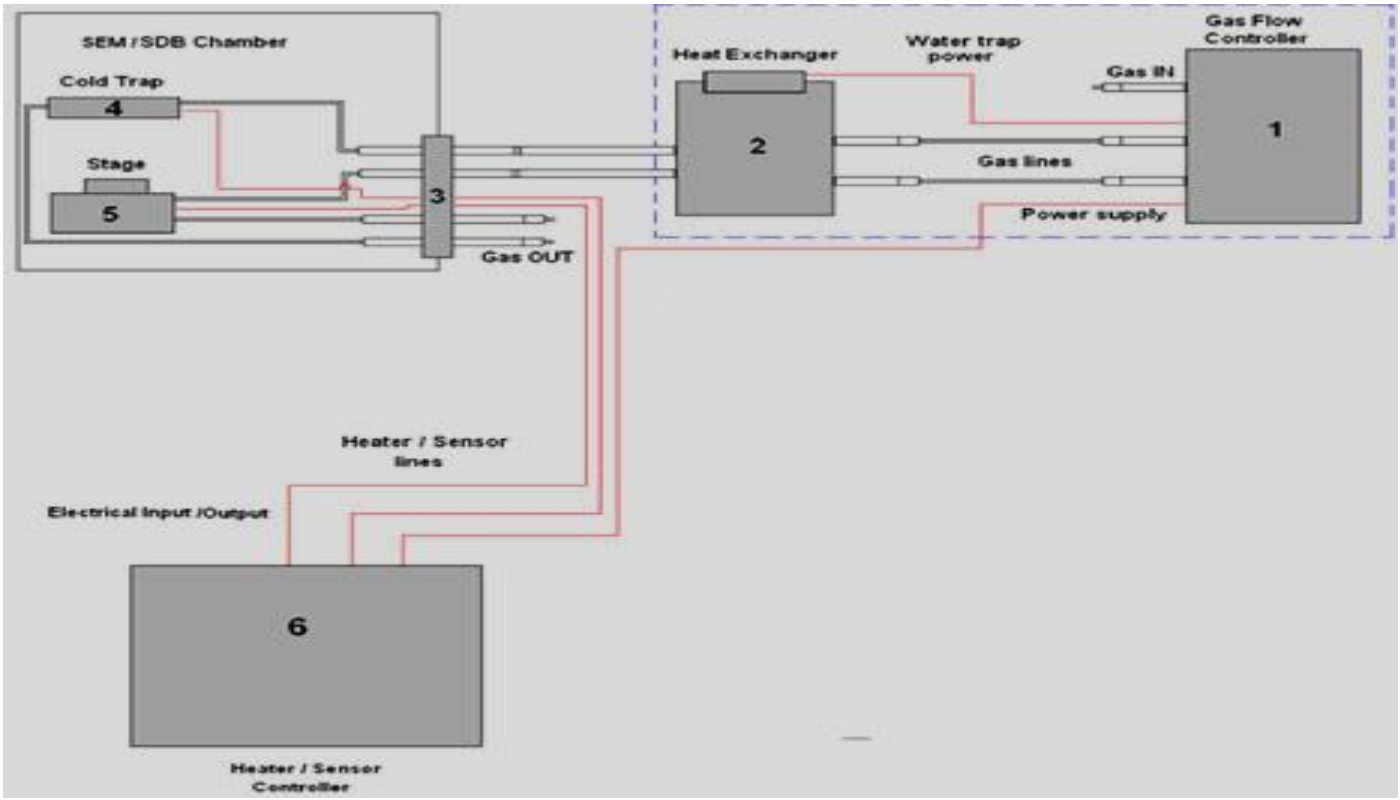
Step 3: Close the Gate Valve by turning the knob bar to the UNLOCK position and press the knob in to engage the valve over the opening. This can be seen through the lead glass lid, then turn the knob bar to the LOCK position to secure the valve.

Press *V* button once. The chamber will be vented and the lid can be opened. The sample carrier can be released by turning the rod bar to the far left and pulled back then returned to the parking position. Remove the sample carrier. Close the loader chamber lid. Press *P* button to evacuate the loader chamber.

Sample Size for QuickLoader: Stub diameter is up to 32mm; height is less than 9mm.

CryoMAT Loader

Overview of Components:

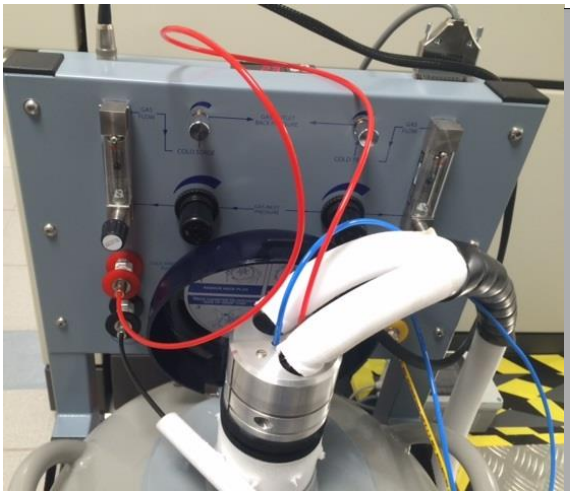


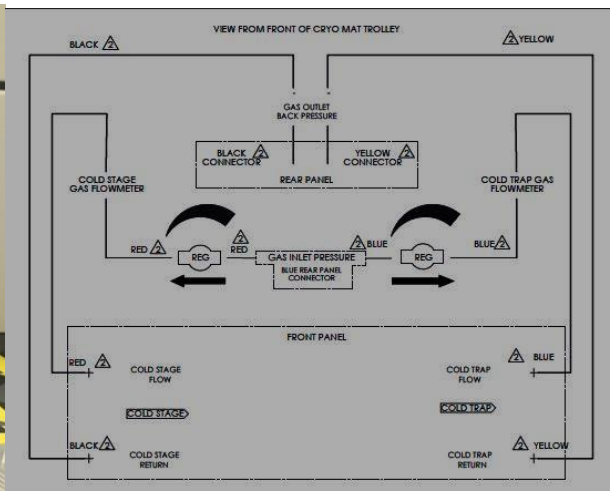
1-: N2 *Gas flow controller* on a trolley mount controls pressure and flow through cryo components.
2-: *Heat exchanger* cools down flowing N2 to cryogenic temperatures. A *water trap* captures water from the N2.
3-: *Gas and electrical interface flange* introduces the gas pipes and electrical cables through the chamber wall.
4-: *Cold trap* creates the coldest position in the chamber to trap condensing water vapour molecules.
5-: *Cryo stage* holds the sample at cryogenic temperature.
6-: *Cryo heater & sensor controller* allows temperature changes to the sample, gives temperature feedback to a visual display for the cold trap and cryo stage.

Gas Flow Controller (1)

Functions: The gas flow console panel is housed on trolley to control the N2 flowing through the LN2 heat exchanger for cooling the cold trap and the cryo-stage. Primary function of this unit is to regulate a separate gas flow to the cryo-stage and cold trap via the heat exchanger. The N2 pressure is regulated from the original supply to this unit at **1.5 bar (21.75 PSI) and 5 litres /minute** flow rate.

Trolley panel components:





• 2× pressure valves for regulation of the incoming gas pressure • 2× gas flow regulators 10 l/m scale • 2× back pressure end valves with non-closing action • 7× gas connections with inter-connective piping • Water trap heater temperature read out display with pre-set controls • Power supply to drive the temperature control, display and N2 water heater • Output socket to N2 water heater + cable • Temperature controller connection + cable • Power input cable and connection (at rear).

Note: When using the panel, fully open the regulators. Using the flow control achieve desired temperature this can minimise cryo-stage drift.

Heat Exchanger and LN2 Dewar (2)

The Dewar volume is 12 litres filled with LN_2. It accommodates four pipes with two in and two out. Gas flows from the gas flow controls to the two incoming pipes then further into the heat exchanger. At the end of the central core, the pipes connected into two copper coils. The two copper coils sit at the base of the Dewar creating cooling transferred by conduction from the LN2 to the N2 gas flowing in the pipes. The insulated pipes coming out of the central core then proceed to the gas flow and electrical interface flange situated on a chamber port. The cooled gas feeds the cold trap and cryo stage independently before returning to the interface flange. The return pipes with gas flowing back from the cryo stage and the cold trap also enter the insulation (neoprene) so that ice does not form at the port. These continue to the trolley entering gas connections through the two back pressure valves and then to atmosphere.

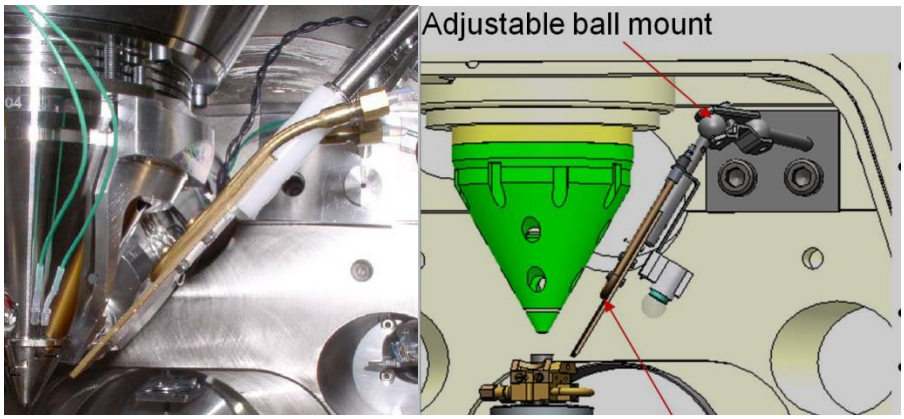
Heat Exchanger components • Stainless steel or aluminium vacuum Dewar • Water trap with heater and cable • Central pipe core with heat exchange coils at base • Insulated cold N2 pipes out of the central core • Non-insulated warm N2 pipes into the central core • Power supply (housed in trolley).

Water Trap

To prevent water entering the cooling system via the N2 gas, a water trap is designed into the top of the central core of the heat exchanger. The water trap chamber volume is heated to evaporate the unwanted water condensing into the trap. An independent power supply house in the trolley panel drives the heater.

Cold Trap (4)

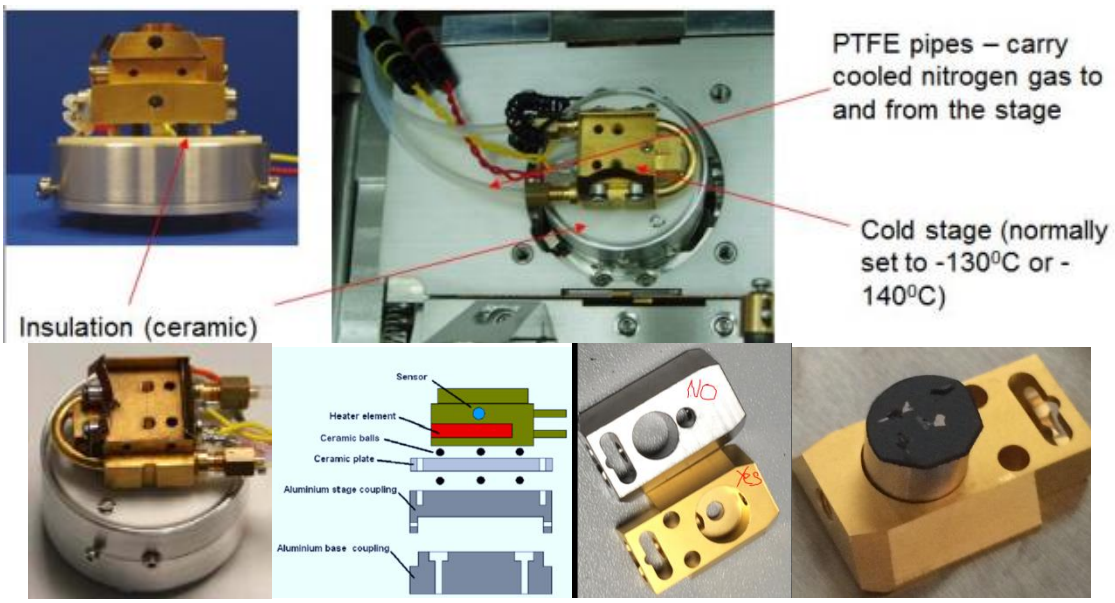
The cold trap protects the sample from water vapour condensing on its surface while at low temperature. This is the reason for keeping the cold trap 10-20°C cooler than your sample. It is suspended by a bracket from the back of the chamber to one side of the lens cone. The extension plate can be used (in addition) for even more effective anti-contamination of the sample. ***Cold Trap components*** • Cold trap plate • Mounting rod and insulator • Sensor element • Ground connector • Pivot bracket • Extension Plate.



Cryo Stage (5), Cryo Sample Carrier and Cryo Stubs

The cryo stage is a multi-construction allowing sample to be cooled down to cryogenic temperatures.
The cold part of the stage can be heated so temperature can be regulated with feedback via a thermal sensor. This allows the cold temperature of the sample to be carefully chosen. The sample can be tilted with the cryo stage to 52° tilt for milling.

The thermal insulation is constructed by a double ceramic minimized contact method to prevent temperature exchange from the FEI main stage to the sample and so that constant temperature can be maintained. The base plate is constructed in two parts so that the cryo stage can be removed quickly and conveniently without having to break connections or cut pipes. The base has three point contacts onto the FEI stage to eliminate vibration. In this way, the load position alignment remains valid when remounting the cryo stage. The X, Y and Z axes movements are not limited. Tilt is able to go to maximum but will only be restricted by the presence of the cold trap at approximately 56° tilt.



Cryo stage components • Mounting base plate • thermally insulated stage mount • Heater element to 100°C • Sensor element • Ground connector.

Note:

- The ceramic plate is very fragile and the 6 ceramics balls are easy to loss during assembling. Take care with handling the CryoStage.

- The cryo stage can be rotated maximum ± 20°! With the cryo stage installed do not use the Home Stage procedure.
- Do not take NavCam image with the cryostage mounted on the bulk stage.

The cryo sample carrier connects to the loading rod bayonet with the same mechanism as the ambient sample carrier. Release and re-connection to the rod use the same mechanism as the Quick Loader. The cryo sample carrier connects onto the cryo stage within the SEM/SDB with a "dovetail" device that is spring loaded on the cryo stage.

The sample stub hole is further along the carrier to a depth of 5.0 mm and of a diameter to accept with close fit a 10.0 mm diameter stub. The stub hole is terminated before penetration to the lower side of the sample carrier. A further 3.0 mm hole is drilled into the centre of the base of the 10.0 mm stub hole for pumping purposes. A small hex screw secures the stub through a threaded hole at the far end of the sample carrier.

Cryo Heater & Sensor Controller (6): Temperature Controller

Upper Red displays indicating real temperature of Cold Trap and Lower Green temperature is the set target temperature for Cryostage and the lower red displays CryoStage temperature.

Cold trap temperature is controlled purely by gas flow, usually the temperature set to -150^0 C to -190^{0}C, and it should be 10^0 to 30^0 lower than the cryostage. Typical temperatures for any type of samples are: cold stage -130^{0}C, cold trap: -150^{0}C.

3-Way temperature switch: Up position: high temperature preset (*SET 2 Warm*) for sample exchange or removing (up to +50° C, default +20° C). *Down* position: low temperature preset (*SET 1 Cold*) for sample cooling which using heating to offset the target cold stage temperature, for example if the target temperature is -130C, the N2 flow can lower that cold stage to -135C degree, the stage heating will raise the temperature by 5 degree.

At *Centre* position a temperature control is not in operation for stage heating or generating heating to achieve desired cold temperature.

Note: The best practice is to set the 3-way controller to the middle, use N2 flow control only to cold stage and cold trap temperatures.
Typical temperatures for any type of samples are: cold stage -130^{0}C, cold trap: -150^{0}C.

Operation guide: CryoMAT

1. Cold stage preparation:

- Turn on the dry N2 gas at its source and regulate the pressure to 1.5 Bar pressure to the Gas flow controller trolley.
- Turn on the control panel on trolley. The ON switch is on the back.
- Switch on the water heater at the back of the Gas flow controller trolley. Open the pressure regulators and flow meters fully. The back pressure regulators should always be open and are only used to create temporary extreme low temperatures not normally in use. They have built-in safe none-closing valves to prevent a closed circuit.
- Flow the N2 gas through the pipe lines to flush them while preparing the stage and samples. It requires flushing at least for a total of 45-90 minutes.
- Make sure that the heat exchanger rod is completely dry and void of trapped water.

- Vent the microscope chamber. Home the stage.
- Checking 4 -white PTFE (Teflon) tubing is not bent or twisted before and after mounting the CryoStage the Cryobase.
- Mount the Cryo-base/stage to the microscope stage, and make sure the pipe lines are pointing to the TLD/EDT detector direction, and rotate the stage relatively 90 degree to find a best guessing position that the shutter will be transfer smoothly to the CryoStage.
- Double check stage rotation between
- Close the chamber door without pumping.

2. Align loading/exchange stage position

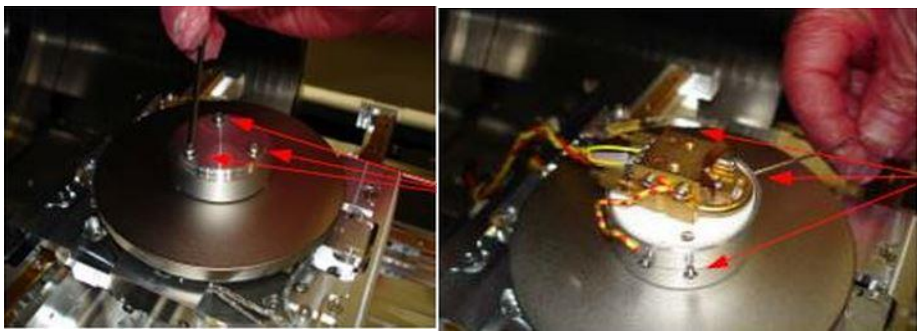
- Open the Qloader gate value when the chamber is vented and the door is closed.
- Press V to vent the loader chamber. After the chamber is vented, you can:
    - Press P button to pump the chamber again if needed (store sample under vacuum condition).
    - Press V button to release the mechanical interlock of the gate valve lever. Then you can open gate valve.
- Close the gate valve before microscope chamber pumping.
- Adjust stage x, y, and r positon to get the best position for the transfer.
- Transfer the sample through the gatevalve to the cold stage and also exchange it, to confirm loading exchange position is correct.
- After the stage position is aligned for the sample exchange, Make three marks on the CryoStage and the base to indicate orientations. When remounting is needed, use the marks as a reference.
- Export stage position: CryoMat.
- Pump down the chamber with gate valve is closed.
- Press “P” the stage will move to pre-saved loading/exchange position, this position is for non-cryo stage operation, so ignore this position.
- Open the gate valve.
- Import stage position “CryoMat”, double click the “CryoMat” in the stage navigation page before insert the transfer rod.
- Test the transfer/exchange, if necessary fine adjust the X,Y,R. Export the stage position, and resave it to “CryoMat”.
- Transfer the shutter from the stage to QLoader chamber.
- Close the Gate Valve.

Note: In case the sample carrier falls from the loading rod, vent the chamber with gate valve opened put the carrier back to a correct position and close the gate valve.

3. Mounting the Sample and operating at room temperature

Mount the sample with strong adhesive medium such as Carbon or Silver paint onto a stub. Do not use carbon adhesive pads because their adhesive can become ineffective at low temperature and may cause the sample to fall off the stub or drift. Sample size for CryoMat: less than 100mm in diameter, and 5 mm in thickness.

Load the sample through Loader.
After the sample has been transfer to CryoStage, rotate stage relatively 90 degree. This will bring the tubing towards to the back chamber/EDT/TLD detector direction. With the tubing at this direction, it will have less chance of bending when the stage is tilted to 52^0.
Prepare cross-sections as usual including deposition protections.
Once Cross-Sections are pre-prepared, the sample is ready to cool down for final cleaning.



4. Cooling Down the Sample

- With the Heat exchanger removed from the Dewar, fill the 12 litre Dewar with LN2.
- Allow 10 minutes to flush the system of any condensed water. Adjust the pressure regulators to show 5 L/M on the flow meters. Carefully immerse the heat exchanger rod into the 12 liter Dewar. Watch the temperature controller read-outs for the lowering of temperature.

Balancing the Flow Rate versus Temperature

- Wait for the boiling effect to subside before adjusting, this will be seen as bouncing balls in the flow meter tubes.
- The Cold trap will reach a lower temperature first because it is a shorter circuit, the cryo stage will follow. Therefore first regulate the flow meter to slow down and stop the cold trap at approximately -160°C. This will probably be between 4 and 5L/M.
- Immediately regulate the cryo stage gas flow so that it just overshoots -130°C. This will probably be between 3 and 4.5L/M.
- Switch the 3 Way Temperature switch to the middle (Not use) . Adjust flow rate to reach to -130C.
- The read-outs on the controller box will indicate the temperature stability at the cryo stage and cold trap. Do not switch on the beams till the sample is at the necessary low temperature. Once this is stable viewing or milling can begin on the sample. This is a procedure using the default temperature settings. Other temperatures can be used but the cold trap should always be at a lower temperature (at least 20°C but no greater than 40°C).

Sample Exchange / Removing

- When the sample needs to be warmed up to ambient temperature before being exchanged or removed, bring the stage to a non-tilt condition and switch off the HV.
- Switch the 3 Way Temperature switch up to SET 2 Warm preset temperature and wait for the system to reach the required temperature (usually after 20 minutes).
- During sample exchange keep the cryo stage at the SET 2 Warm temperature.

Finishing CryoMAT Loader Operation

- Switch the 3 Way Temperature switch up to SET 2 Warm preset temperature and wait for the system to reach the required temperature (usually after 20 minutes).
- Switch the 3 Way Temperature switch to centre position (Off).
- Remove the sample carrier from the SEM/SDB, vent the loader chamber and remove it from the loader with tweezers.

- Turn off the N2 gas supply to the 12 liter Dewar unit and wait for the supply pipes to the chamber interface to become flexible (approximately 15 minutes).
- Carefully remove the heat exchanger from the 12 liter Dewar and cap off the 12 liter Dewar to save LN2.
- Turn on the N2 gas supply to the 12 liter Dewar unit and warm the coils at the end of the core with a hair dryer of at least 1 000 Watt.
- When the temperatures on the controller box show ambient temperatures, stop heating the coils and turn off the N2 gas supply. Switch off the water trap heater on the trolley panel. This operation should only take approximately 15 minutes. Bring the stage to a non-tilt condition and switch off the HV.

Note:

- PTFE tubing is very vulnerable part. Bending of the tubing will cause the microscope chamber vacuum leading. The tuning must handle with care.
  - PTFE tuning must cut to a proper length. If the tubing is too long or too short, it will cause problems.