Standard Operating Procedure (SOP) for the attoDRY1000 System

Instrument Location: Central Facility, Quantum Cryogenic Lab (QCL), IIT Bombay

System: attoDRY1000XL/1T/1T/1T Cryostat System with Superconducting Magnet (SU system)

Safety Precautions

- High voltage is present; risk of electric shock. Follow all electrical safety guidelines.
- Never open the dewar or the outer vacuum space when the system is cold, as warming up may cause high internal pressures.
- Every vacuum space must have an operational overpressure valve. Ensure no valves are blocked, and do not modify them.
- If the system is opened without proper venting, it can lead to ice formation and dangerous pressures during warm-up.
- The system contains fragile components. Avoid any mechanical or thermal work on tubes or valves, and never use blocked components.

System Overview

The **attoDRY 1000** is a cryogen-free cryostat capable of reaching temperatures as low as 4 K and magnetic fields up to 9 T. It utilizes pulse tube technology for cooling without the need for cryogenic liquids, minimizing vibrations and allowing for easy sample exchange.

Main Components

- **Pulse Tube Cold Head:** Drives the cooling via gas compression and displacement.
- **Sample Vacuum Space:** Provides isolation and thermal coupling for the sample.
- **Outer Vacuum Space:** Encloses the sample vacuum space, providing an additional insulation layer.
- **Compressor Unit:** Provides high-pressure helium to the pulse tube.
- **Overpressure Valves:** Ensures pressure safety in both the inner and outer vacuum chambers.

1 Preparation of the attoDRY 1000 for Cool Down

Adjustment Procedure of the x-y Plate

1. Remove all the side walls from the cryostat rack in order to take off the top cover panel.
2. Remove the ISO K o-ring from the entry port and carefully slide the vacuum tube into the crystat.
3. Slightly loosen the screws of the x-y tilt plate (Figure 1).

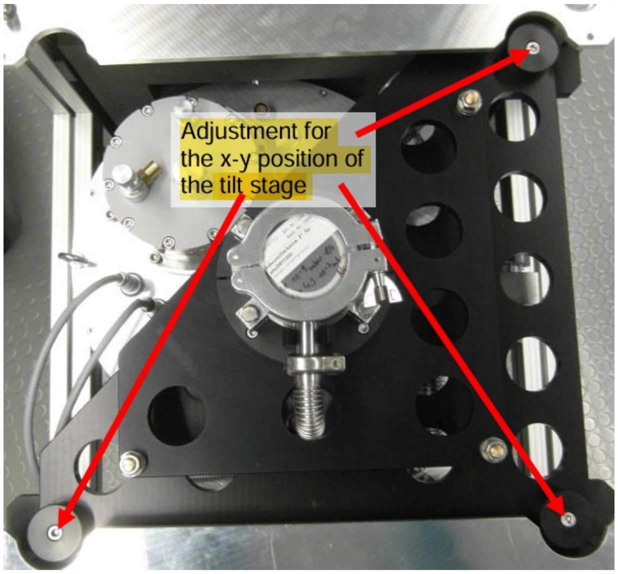

Figure 1: Top view of the attoDRY1100 with the adjustment possibility of the x-y tilt plate.

4. Tilt the vacuum tube (Figure 2) to the left and right and to the front and back to check if the vacuum tube is centered in the attoDRY1000.

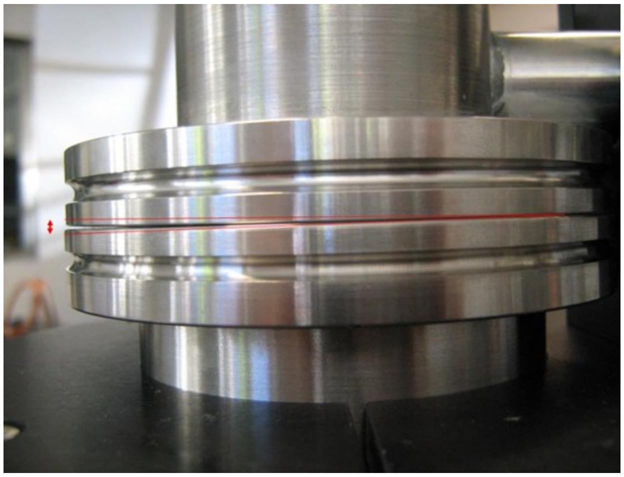

Figure 2: Slightly tilt the vacuum tube to see if it's centred correctly

5. If the vacuum tube is not centerd accurately, move the whole x-y tilt plate to adjust the center position of the vacuum tube.
6. Thighten the screws of the x-y tilt plate.
7. Remove the vacuum tube and put the ISO-K63 O-ring on top of the attoDRY1000 flange.
8. Reinsert the vauum tube, connect heater and temperature sensor cables and thighten the ISO-K63 flange with the clamps.
9. Close the KF50 flange of the vacuum tube with the dummy flange.
10. Mount all side walls onto the cryostat rack.
11. Do not forget to re-connect the flexible hose for the Helium exchange gas, compare (Figure 3).

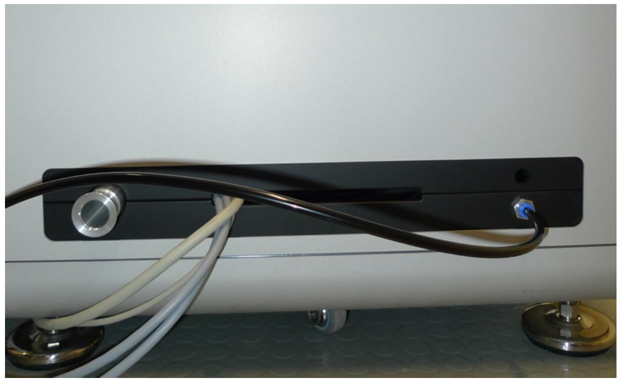

Figure 3: Preparing the attoDRY for inserting a microscope stick.

12. Remove the microscope tube.
13. Insert the 'insertion guide' as shown in (Figure 4). This part is needed to protect the internal membrane bellows of the outer vacuum space from being damage during the insertion of the vacuum tube.

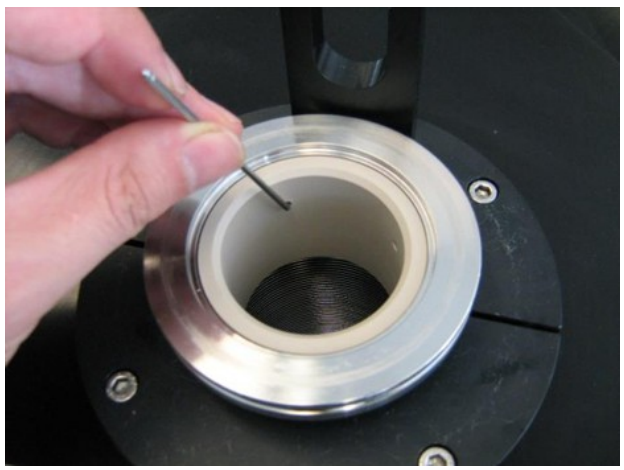

Figure 4: Insertion guide

Evacuation of the Sample Space and Outer Vacuum Space

Please connect the KF25 valve of the isolation vacuum to a turbo pump (Figure 8 : refer manual). Open the valve to the isolation vacuum and turn on the pump to evacuate the cryostat. Due to the large surface areas, especially of the super insulation foil, inside the cryostat it is recommended to pump the vacuum over night down to a pressure in the range of 10^{-4}*mbar*.

Evacuating the Outer Vacuum Space

Connect a hose to the KF25 on the back of the attoDry1000 to a turbo pump (Figure below Figure 9 : refer manual). After opening the pumping valve of the outer vacuum space one can turn on the turbo pump.

2 Cooling Down the attoDRY Pulse-Tube Cryostat

Turn on the compressor in the following order.
1. Turn on the cooling water (flow 7 l/min), depending on its temperature
2. Main circuit breaker on
3. Power switch on
4. Compressor on (green button)

3 Inserting the Microscope

Connect the black hose on the back of the attoDRY1000 to a He gas Cylinder.
1. (See section IV of the manual : *Microscope Stick and Vacuum Tube*)
2. Make sure that all valves (outer vacuum space valve, cryostat isolation vacuum valve) on the attoDRY pulse tube refrigerator are closed.
3. Prepare a connection to the He gas cylinder (Figure 5).

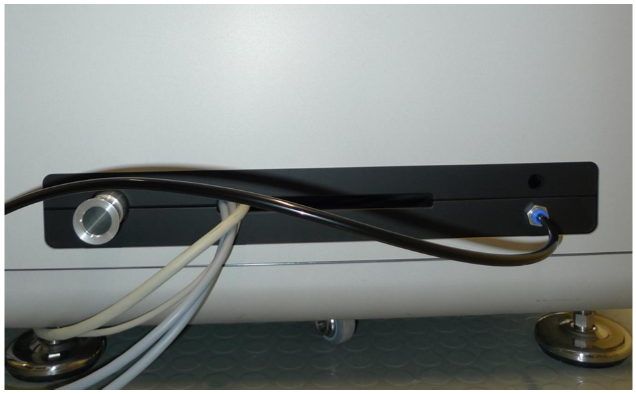

Figure 5: Preparing the attoDRY for inserting a microscope stick.

4. Flush the tube that comes from the He gas cylinder with Helium gas.
5. Connect the gas cylinder to the valve of the outer vacuum space.
6. Open the venting valve (Figure 6) of the sample tube and open the valve on the gas cylinder.

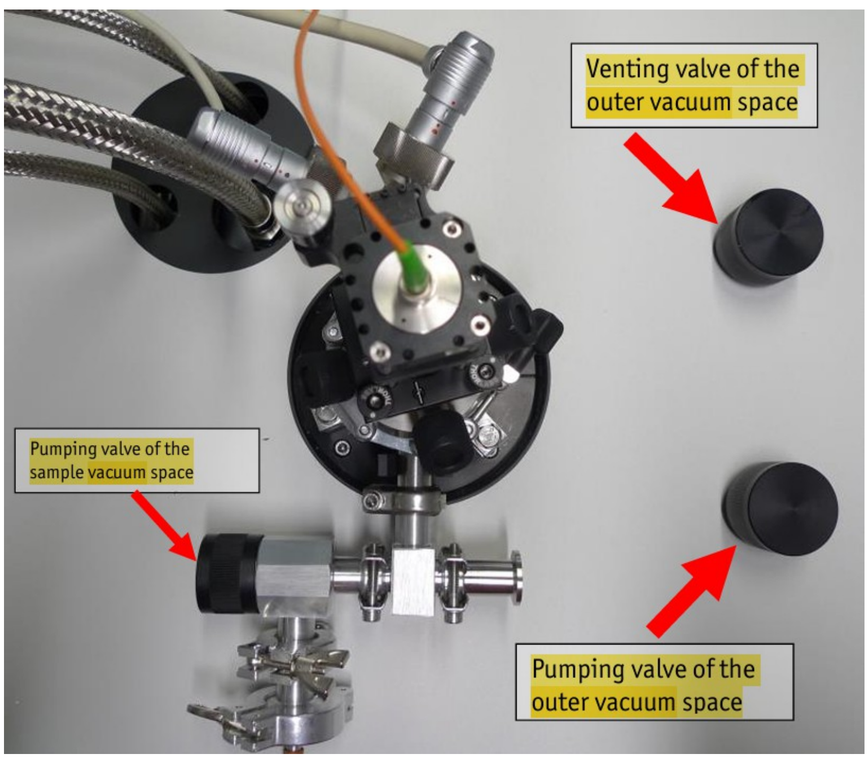

Figure 6: top view of the attoDry1000 with the venting and pumping valve of the outer vacuum space.

7. Let gas flow into the sample tube until the overpressure valve opens.

4 Taking out the Microscope

Connect the black hose on the back of the attoDRY1000 to a He gas cylinder.

12. (See section IV : *Microscope Stick and Vacuum Tube*).
13. Make sure that all valves (sample tube valve, isolation vacuum valve) on the attoDRY pulse tube refrig- erator are closed.
14. Prepare a connection to the He gas cylinder (Figure 7).

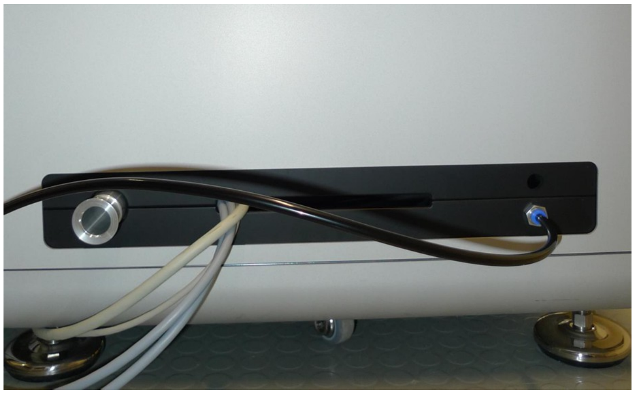

Figure 7: Preparing the attoDRY for inserting a microscope stick.

15. Flush the tube that comes from the He gas cylinder with Helium gas.
16. Cconnect the gas cylinder to the sample tube valve.
17. Open the venting valve (Figure 8) of the sample tube and open the valve on the gas cylinder.

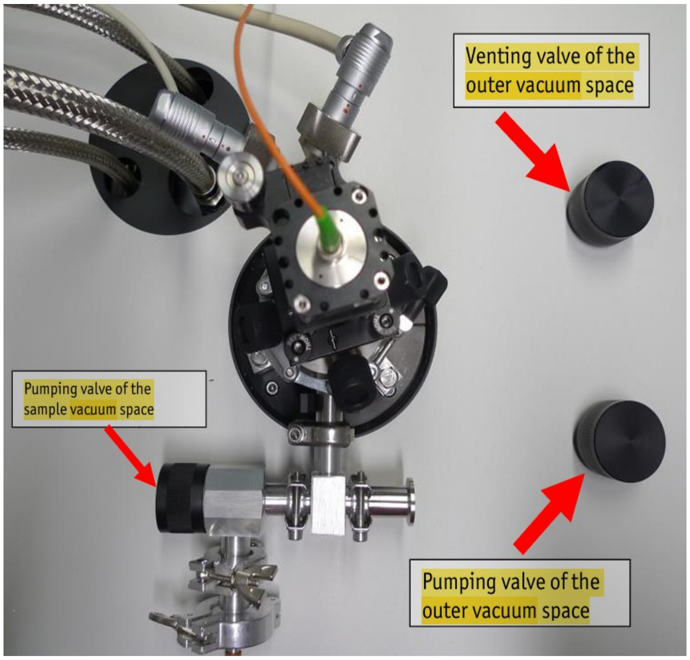

Figure 8: top view of the attoDry1000 with the venting and pumping valve of the outer vacuum space.

18. Let gas flow into the sample tube until the overpressure valve opens.
19. Now one can open the top cover flange (ISO K 63) of the sample tube while helium gas is flowing all the time.
20. Take out the microscope stick and put a blind flange on top of the sample tube, tight the clamps on the flange again.
21. Close all the valves carefully.

5 Microscope Stick

The top of the microscope stick contains all electrical connectors and feedthroughs, the vacuum window, and the fiber feed-through. The design of the microscope stick offers the possibility to have free optical access to the sample either by a fiber or by free beam.

1. Carefully remove cryostat from the chamber and placed it on cryostat stand.
2. Place the sample on the PCB in the microscope stick.

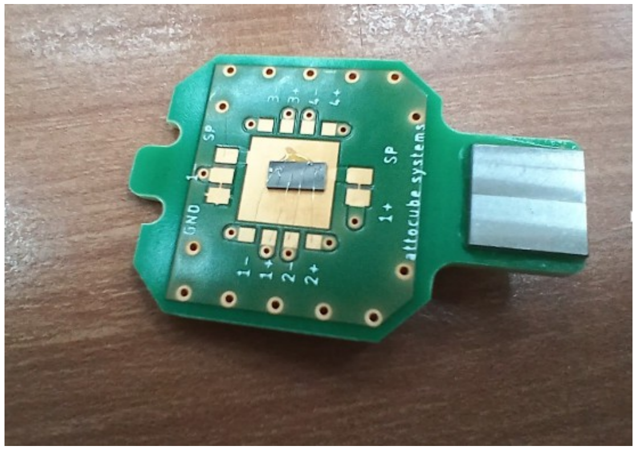

Figure 9: Sample mounted on PCB

3. Then carefully mount the PCB onto the piezo stack, ensuring it aligns precisely for accurate operation and signal stability.
4. Secure all screws and connections to prevent movement or drift during experiments.
5. Wait till temperature go down to 4K. Then the stick has to be mounted into the vacuum tube.

Figure 10: Microscope stick with attached microscope module

6 Vacuum Tube

The diameter can vary in case of customized solutions. The main flange is a DN50KF and the side flange is a DN25KF. The overall length of the tube is customized to the individual setup. The tube is made of non-magnetic stainless steel. The microscope stick is to be inserted into this vacuum tube.

NOTE:

1. The walls of the tube are fragile. Be careful not to damage or dent the walls.
2. Be sure that no contaminations (dust, oil etc…) gets inside the tube.

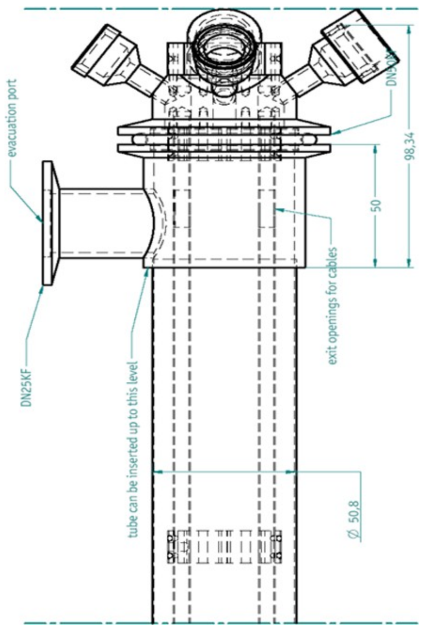

Figure 11: Schematic drawing of the vacuum tube and zoom-in on the side view of the vacuum tube with the microscope stick inserted.

7 Vacuum Valve (Evacuation of chamber)

1. As temperature increases above 70 K, we need to evacuate the chamber. A vacuum pump is needed to evacuate the microscope stick.
2. A pressure of 10^{-4} to 10^{-5} mbar is suggested to remove any water vapor and other residual gases from the vacuum tube.
3. A small turbo pump together with a rotating pump serves this purpose well, but special care needs to be taken to avoid vaporized oil flowing back into the stick. For this reason, a dry turbo- and backing pump would be the best solution.
4. To ensure a good thermal contact, in order to reach the base temperature, we suggest introducing 20 mbar He gas into the microscope stick.
5. To avoid water vapor one can clean the He gas with a cold trap before inserting into the stick.

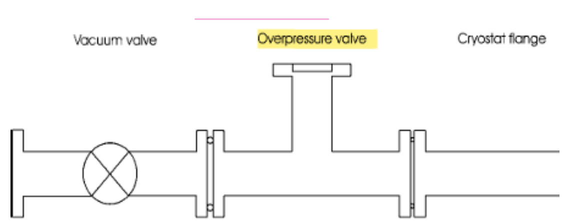

Figure 12: Schematic drawing of vacuum valves

8 Magnetic Field and Temperature Control

- For magnetic fields above 4 T, ensure appropriate He pressures in both the inner and outer volumes.
- Avoid applying fields if the sample temperature is outside the defined safe range (4–4.5 K) to prevent magnet quenching.
- Ensure pressures in the inner and outer vacuum chambers align with the field and temperature param- eters.

Operation of the Piezo Stack

The piezo stack should be controlled via a stable power source to avoid abrupt movements or vibrations. Regularly inspect the setup to prevent misalignment and wear, ensuring consistency during temperature changes or adjustments.

Operation of the Magnets

1. Magnetic Field Control: The cryostat system allows magnetic fields up few Tesla, depending on the temperature. Monitor the He pressures in both inner and outer chambers to prevent magnet quenching.
2. Handling Quenches: In the event of a magnet quench (such as due to water chiller failure), halt experiments immediately. Ensure the compressor is functioning and that there is no dust accumulation on the chiller to prevent temperature spikes that could impact superconducting magnets.

Note : If base temperature goes above 15 K then magnet will quenched. As a result of which, the compressor turned off and the cryo temperature started to increase. Above 15K, the magnetic coils lose their superconducting nature, and behave as open circuit for such a high current. So, the sudden rise in temperature acted as a power cut to the coils.

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Cryostats Overview

	attoCMC	attoDRY800(xs)	attoDRY1000	attoDRY2100	attoDRY2200
Base temperature	2.3 K		4 K	1.8 K	1.8 K
Temperature range		3.8 .. 320 K	4 .. 70 K	1.8 .. 300 K	1.8 .. 300 K
Variable temperature		●	○	●●	●●
Low vibration		●	●	●	●●
Automated temperature control	●	●		●	●
Superconducting (vector) magnet			●	●	●
Toploading (sample in exchange gas)			●	●	●
Sample in vacuum	●	●			
Optical access to sample		●●	●	●	●
attoAFM I			●	○	●●
attoAFM III			○		●●
attoAFM/CFM			○		●●
CFM base kit			●●	●●	●●
attoCFM I & IV			●●	●●	●●
cryoRaman		on request		●●	●●
atto3DR				●	●
19-inch rack compatibility	●●	● (xs/IGLU)			
Req. infrastructure	single phase	three phase, water/air colled	three phase, water cooled	three phase, water cooled	three phase, water cooled
IGLU compressor	Incl.	optional			

○ yes, but with limited performance
● yes, performs very well
●● yes, performs excellent

attocube
WITTENSTEIN group

Technical Specifications

General Specifications

technology	low vibration, pulse-tube based closed-cycle cryostat, designed for confocal microscopy
sample environment	He exchange gas, 4-5 different pressure ranges depending on desired sample temperature, requires manual control
sample space	49.7 mm diameter probe bore fitting all attocube inserts
~~sample exchange~~	~~top loading system for quick access~~
vibration & acoustic noise damping system	proprietary low vibration design

Performance Data

temperature control	manual, requires optional temperature controller
temperature range	4 .. 80 K (guaranteed), 4 .. 300 K (expected); optional temp. controller required
base temperature	< 4 K
~~magnetic field control~~	~~manual control via magnet power supply, via~~ remote control
max. magnetic field	100 % (e.g. 9 T) @ 4 .. 10 K sample temperature, 67% (e.g. 6 T out of 9 T) @ 10 .. 300 K sample temp
cool down time of sample	approx. 2 h (depending on insert)
~~cool down time of system (system incl. 9 T magnet)~~	~~approx. 10 .. 15 h (unattended)~~
cool down time of system (system without magnet)	approx. 5 .. 10 h (unattended)
temperature stability	< ± 10 mK expected (4 .. 50 K), < ± 25 mK guaranteed (4 .. 50 K)

Compressor

power consumption	max. 9.0 kW, 7.2 kW steady state
cooling of compressor	water cooling (requires local infrastructure)

Size and Dimensions

cryostat (width x depth x height)	1120 x 640 x 1050 mm³
~~required min. ceiling height~~	~~approx. 2.60 m (depending on magnet)~~
optional electronics rack (width x depth x height)	640 x 640 x 1050 mm³

Options and Upgrades

superconducting magnet	solenoids: 9 T, 12 T, vector magnets: e.g.: 9/3 T, 9/1/1 T, ...
~~bipolar magnet power supply~~	~~included (with optional magnet)~~
~~temperature controller~~	~~2 channel (magnet + sample temperature)~~
pumping kit	turbomolecular pump with suitable backing

Compatibility

confocal microscopes	attoCFM I, attoCFM IV
atomic force microscopes	attoAFM I, AFM upgrade options (MFM, KPFM, PFM, conductive-tip AFM)


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