

Standard Cryogenic Operation with an inline Cold Trap

X-Band EMX EPR system with ColdEdge, room SCH 162

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Table of Contents

Day 1, System startup after room-temp operation	2
Pumping down the vacuum insulation.....	2
Evacuation of a Sample Well, a Cold Trap, and a WaveGuide.....	2
Day 2: Cooldown	3
Sample change procedures.....	4
Insertion of the sample tube for an initial cooldown.....	4
Adjustment of sample tube depth to prevent vibrations.....	6
Troubleshooting	7
Warm up and clean up	8
Reverting to a Room-Temperature mode.....	9

NOTE 1: We have a Cold Trap permanently inserted in the supply line of the WaveGuide. It may be used warm (doing nothing) or cold (immersed in LN2 thus continuously cleaning the incoming helium). If you use it cold, the sample change protocol will include a step of pumping helium out of the Cold Trap through Four-Way Valve in "Both Sides" position. Warming up and evacuating of the system after the experiments are done naturally clean the Cold Trap.

NOTE 2: Sample requirements:

- 4mm tube
- use a sample gauge to test whether the tube is long enough to reach the bottom of the dewar
- mark the maximum depth on a tube
- 20mm to 40 mm sample height (no longer!).

Day 1, System startup after room-temp operation

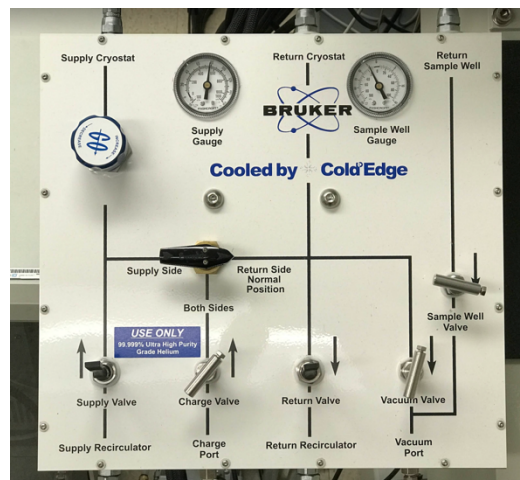
Current State: The sample well was open to air in room-temp operation

Pumping down the vacuum insulation

1. Note the positions of the gauge arms on the turbopump (to make sure they are not stuck - tap the gauge): **Black:** _____ Pa , **Red:** _____ Pa.
2. Check that both valves are closed: on a pump (towards minimum, down) and a WaveGuide (CW, down).
3. Turn on high-vacuum pump on.
4. Wait till pump display shows "at speed": _____ rpm
5. Record **Red Gauge** reading: RG= _____ Pa .
Note this value: this is the deepest vacuum the pump can achieve!
6. Open valve on pump all the way and 1/8 turn back.
7. Wait 15 sec - till pump display again shows "at speed".
8. Open valve on the WaveGuide Cryostat till you feel a stop and 1/4 turn back.
9. Leave Turbopump to run overnight.
10. Refill LN2 dewar for tomorrow

Evacuation of a Sample Well, a Cold Trap, and a WaveGuide

1. Go to a Research Grade He cylinder:
 - a. Open **Main valve**;
 - b. Open **Regulator valve** to 30 psi;
 - c. Open **Shutoff valve**.
2. Close sample well with a twist cap.
3. Turn on GAST pump (turn on a power strip).
4. Turn **Four-Way Valve** to **Both Sides**.
5. Open **Vacuum Valve** and **Sample Well Valve**.
6. Drop pressure to -27mm.
7. Close **Vacuum Valve** and **Sample Well Valve**.
8. Check vacuum: **Sample Well Gauge** must stay at -27.5 mm for one minute.
9. Purge Sample Well and WaveGuide:
 - a. Charge to 3 psi and close **Charge Valve**;
 - b. Open **Vacuum Valve** and **Sample Well Valve**;
 - c. Drop pressure to -27mm;
 - d. Close **Vacuum Valve** and **Sample Well Valve**;
 - e. Repeat charging and dropping pressure one more time.
10. Close main valve on a Research Helium cylinder. Record Regulator pressure: _____ psi
11. Open **Vacuum Valve** and **Sample Well Valve**
12. Check Turbopump pressures: black: _____, red: _____
13. Leave GAST pump to run overnight. (Pressure on pump: _____ mm)



Day 2: Cooldown

Current State: The WaveGuide was pumped overnight with a GAST pump. The turbopump was pumping the WaveGuide vacuum insulation. **Four-Way Valve - Both Sides**

We will cool down without opening the sample space to verify helium gas purity before we create a chance to contaminate helium by our changing samples. We should be able to reach 10K in 90 min.

It is recommended to immerse the Cold Trap in LN2. Alternatively, you may keep the Cold Trap warm. The penalty for having it warm is that if WaveGuide becomes blocked you will have to warm up overnight before you can restart the system with a Cold Trap in LN2 next day!

1. Check Turbopump: speed = _____ rpm, red gauge = _____ mBar.
2. On a Research Grade He cylinder:
 - a. Open the **main valve**
 - b. record **main gauge** reading: _____ psi.
3. Close **Vacuum Valve** and **Sample Well Valve**.
4. Check vacuum for one min.
5. Purge the Sample Well
 - a. Charge to 3 psi and close **Charge Valve**
 - b. Open **Vacuum Valve** and **Sample Well Valve**
 - c. Drop pressure to -27mm
 - d. Close **Vacuum Valve** and **Sample Well Valve**
 - e. Repeat charging and dropping pressure
6. Purge the WaveGuide
 - a. Set **Four-Way Valve** to **Supply Side**
 - b. Fully Open **Charge Valve** (gives 20-30 psi on a **Supply Gauge**)
 - c. Wait for **Sample Well Gauge** to reach 3 psi
NOTE: Takes 2-3 min.
 - d. Close **Charge Valve**
 - e. Open **Vacuum Valve** and **Sample Well Valve**
 - f. Drop pressure to -27mm
 - g. Slowly open the **Four-Way Valve** to **Both Sides**
 - h. Drop pressure to -27mm
 - i. Close **Vacuum Valve** and **Sample Well Valve**
7. Charge to positive 3 psi and close **Charge Valve**
8. Set **Four-Way Valve** to **Normal position**
9. Slowly lower the Trap in the LN2 dewar.
NOTE: This may be done before (1) to make it cold sooner.
10. Turn on the EPR instrument:
 - a. Open water (first "R", then "S");
 - b. Turn on the Haskris chiller;

- c. Turn on wall power to both EPR and Cryoplatfrom;
 - d. Turn on the Magnet Power Supply (Electricity ON, pause, Power ON);
 - e. Turn on EPR spectrometer
 - f. Turn on Temp Controller (on the back of the unit).
11. Turn on **Recirculator Compressor**
 12. Open **Return Valve**
 13. Open **Supply Valve**
 14. Record **Supply Gauge** reading
NOTE: If you suspect helium is still contaminated (incomplete cooldown in a previous run) - pause at this step for one hour to allow Cold Trap to work before a cold head is started.
 15. Turn on **F70** (start the cold head)
 16. Turn on Temp Controller Chart recorder on a computer to view a temperature graph.
 - a. In Firefox, open 192.168.1.12/utilities
 - b. open "Chart Recorder". It may not launch on first try. Keep trying.
 - c. Change file name for the recording and its folder
 - d. Click Sensor A (heat exchanger) and Sensor B (sample well).
 17. If **Supply Gauge** reading is smaller than 100 psi - add more helium by VERY SLOWLY opening **Charge Valve** till **Supply Gauge** reads 100 psi.
NOTE: It takes about 30 min to drop sample temperature below 20K, and another 30 min to stabilize.

Sample change procedures

NOTE 1: A crucial requirement for changing samples is to maintain a positive flow of helium out of the sample well at the moment when the samples are changed. This prevents atmospheric air from contaminating helium inside the system.

NOTE 2: It is critical for successful tuning that the sample tube presses against bottom of the dewar. However, after the sample tube fully cools to the low temperature, the tube contracts and, therefore, stops touching the bottom of the dewar. Additional adjustment is required to ensure that the cold tube is firmly engaged with the dewar bottom.

Insertion of the sample tube for an initial cooldown

Current State: The system is cold with sample well closed by a cap with or without a sample. If Cold Trap is immersed in LN₂, note additional instructions!

1. Close **Supply Valve**.
2. Immediately, start bleeding remaining pressure from Supply Side to the Return Side by SLOWLY and PARTIALLY turning the Four-Way Valve towards Both Sides while watching the Sample Well Gauge. It must read about 3-4 psi in the process.

IMPORTANT: DO NOT TURN Four-Way Valve ALL WAY to Both Sides at once! The excess pressure from the Cold Trap will blow up the Sample Well Gauge and plumbing!

NOTE: In case the Cold Trap is in LN2, this step is needed to avoid releasing contaminating air absorbed in a Cold Trap into the cold WaveGuide when pressure drops for a sample change. Instead, we bypass WaveGuide and send contaminants back into Recirculator Compressor to be recaptured later again.

3. When **Supply Gauge** drops to 0 psi, turn **Four-Way Valve** to **Normal Position**.
4. Close **Return Valve**.
5. Prepare a sample tube:
 - a. insert a WARM sample tube in the second cap;
 - b. put the cap on a depth gauge and push the tube through to reach the bottom of the gauge;
 - c. mark the height on a tube with a permanent marker;
6. Establishing an appropriate overpressure in a sample well
 - a. Open **Charge Valve** to see pressure increasing on a **Sample Well Gauge**.
 - b. As it passes 5 psi - unlock cap (hold hand to keep it); note the overpressure valve opening when you push on a cap to stop gas flow.
 - c. Open **Charge Valve** a little more to feel the He flow slightly lifting the cap (HOLD HAND ABOVE THE CAP!)
 - d. Twist-lock the cap
7. Freezing the sample tube:
 - a. freeze the sample volume in LN2: only immerse the part of the tube that contains sample, do not go deeper!
 - b. pull the tube up to till the frozen part approaches the cap.
NOTE: Do NOT pull frozen sample area further up into the cap! The O-ring in the cap will seize and the tube may break.
 - c. **Tighten up the collar on a cap.**
8. Quickly swap the caps and twist close.
9. Loosen the collar and carefully push the tube till you feel a hard stop. **BE SUPER CAREFUL AND SLOW!**
10. Tighten the collar on a cap.
11. Close **Charge Valve**.
12. Purge sample space:
 - a. Turn **Four-Way Valve** to **Both Sides**;
 - b. Charge to 3 psi and close **Charge Valve**;
 - c. Open **Vacuum Valve** and **Sample Well Valve**;
 - d. Drop pressure to -27mm;
 - e. Close **Vacuum Valve** and **Sample Well Valve**;
 - f. Repeat charging and dropping pressure two more times.
13. Test vacuum for one min.
14. Charge to positive 3 psi and close **Charge Valve**.

15. Turn **Four-Way Valve** to **Normal**.
16. Open **Return Valve**.
17. Open **Supply Valve**.
18. Wait for the Sensor B (sample) to reach the target temperature.

Adjustment of sample tube depth to prevent vibrations

Current state: *Sample tube is inserted and the system has cooled down to the setpoint. If you observe fluctuations of the resonance profile, the depth of the tube must be adjusted down to meet the dewar floor.*

1. Switch Xenon to **Tune** mode, click **Monitoring**, and make gauges big to see from the sample well position. Note how they fluctuate because of the vibrations.
2. Close **Supply Valve**.
3. Immediately, start bleeding remaining pressure from **Supply Side** to the **Return Side** by SLOWLY and PARTIALLY turning the **Four-Way Valve** towards **Both Sides** while watching the **Sample Well Gauge**. It must read about 3-4 psi in the process.

IMPORTANT: DO NOT TURN **Four-Way Valve** ALL WAY to **Both Sides** at once! The excess pressure from the Cold Trap will blow up the Sample Well Gauge and plumbing!

NOTE: In case the Cold Trap is in LN₂, this step is needed to avoid releasing contaminating air absorbed in a Cold Trap into the cold WaveGuide when pressure drops for a sample change. Instead, we bypass WaveGuide and send contaminants back into Recirculator Compressor to be recaptured later again.

4. When **Supply Gauge** drops to 0 psi, turn **Four-Way Valve** to **Normal Position**.
5. Close **Return Valve**.
6. Open **Charge Valve** to see pressure increasing on a **Sample Well Gauge**.
7. As it passes 5 psi, note the overpressure valve opening.
8. Adjust the sample tube down to stop vibrations:
 - a. Secure the sample tube from top with one hand (pressure will be pushing it up)
 - b. Loosen up the collar on the cap with another hand.
 - c. Push **gently** on the top of the tube and observe the gauges.
 - d. Once fluctuations reduce, **slowly** tighten the collar - while observing the gauges to see that fluctuations do not come back.
9. Close **Charge Valve**.
10. Purge sample space:
 - a. Turn **Four-Way Valve** to **Both Sides**;
 - b. Charge to 3 psi and close **Charge Valve**;
 - c. Open **Vacuum Valve** and **Sample Well Valve**;
 - d. Drop pressure to -27mm;

- e. Close **Vacuum Valve** and **Sample Well Valve**;
 - f. Repeat charging and dropping pressure two more times.
11. Test vacuum for one min.
 12. Charge to positive 3 psi and close **Charge Valve**.
 13. Turn **Four-Way Valve** to **Normal Position**.
 14. Open **Return Valve**.
 15. Open **Supply Valve**.
 16. Wait for the Sensor B (sample) to reach the target temperature.

Temperature Control

*NOTE 1: **Sensor B is a sample well** temperature. Sample A is a WaveGuide body sensor.*

NOTE 2: It is not necessary to use temperature control if your only goal is to record a spectrum near the lowest temperature (10K). You may just wait till it cools down and record and use the temperature controller as a temperature monitor. If you need to set specific Sensor B (sample well) temperature:

1. To start controlling temperature:
 - a. Push **SetPoint** button on the Temperature Controller face, enter temperature in K using number keyboard, and push **Enter**;
 - b. Push **Heater Range** button, use arrows to select power mode, push **Enter**.
NOTE: Use "Low" for 10-15K, "Med" for 15-60K, "High" above 60K
2. To stop temperature control :
 - a. Push **Heater Range** -> choose **Off** -> **Enter**
 - b. **Setpoint** -> dial **0 K** -> **Enter**

NOTE: It seems only necessary to tune once at the lowest temperature.

Troubleshooting

- If your sensor temperatures reduces to a target too slowly or start climbing up, this is a sign of a blockage in the heat exchanger inside the WaveGuide. To clear a small blockage:
 1. Turn off **F70**
 2. Wait for a sensor A to exceed 40K (about 10 min)
 3. Restart **F70**

If blockage is successfully cleared, the Sensor A will be below 10 K within 2 min.
- If blockage is still there, repeat the same steps but let sensor A warm up to 70K (oxygen melts at 54K, nitrogen - at 63K). This will take longer (maybe 30 min). Cooldown from 70K must be taking no longer than 5 min.

- If you still cannot quickly reach 10K after restarting F70, the Cold Trap may be saturated with air and does not provide clean helium to WaveGuide any more. In this case, proceed to "Warm up and clean up" section.

Warm up and clean up

The system is cold with the sample in a sample well and Cold Trap in LN2 dewar. This section is the end of work (either because you are done or your WaveGuide is blocked with air and does not cool down any more).

1. Stop temperature control (if used):
 - a. Push **Heater Range** -> choose **Off** -> **Enter**.
 - b. **Setpoint** -> dial **0 K** -> **Enter**.
2. Check that GAST pump is ON (or turn it ON)
3. Turn OFF the **F70**.
4. If Cold Trap is immersed in LN2: let Recirculator Compressor run for 30 min and clean helium while system is warming up.
5. Close **Supply Valve**.
6. If Cold Trap is immersed in LN2, bleed its contaminated helium to vacuum:

NOTE: This step allows to take all air accumulated in the Cold Trap out of the system.

 - a. Open the **Vacuum Valve** and **Sample Well Valve**;
 - b. Close **Return Valve**;
 - c. Carefully bleed pressure from a Cold Trap by SLOWLY and PARTIALLY turning the **Four-Way Valve** towards **Both Sides** while watching the **Sample Well Gauge**. It must read no more than 3-4 psi in the process!

IMPORTANT: DO NOT TURN **Four-Way Valve** ALL WAY TO **Both Sides** at once!
The excess pressure from the Cold Trap will blow up the Sample Well Gauge and plumbing!

 - d. When Supply Gauge drops to 0 psi, turn **Four-Way Valve** fully to **Both Sides**.
7. If Cold Trap is warm:
 - a. Wait till **Supply Gauge** goes to 0 (one min);
 - b. Turn **Four-Way Valve** fully to **Both Sides**;
 - c. Close **Return Valve**;
 - d. Open **Vacuum Valve** and **Sample Well Valve**.
8. Turn OFF the **Recirculating Compressor**.
9. Turn off EPR system:
 - a. Disconnect from spectrometer in Xenon;
 - b. Turn OFF the EPR spectrometer;
 - c. Turn OFF the Power Supply (Power Off, Electricity Off);
 - d. Flip DOWN both power switches on the wall (for EPR and to Cryoplatfom);

- e. Turn OFF the Haskris chiller;
 - f. Shut off water on the wall (both handles to horizontal, first "S", then "R");
 - g. Close vacuum valves on a Waveguide and a turbopump;
 - h. Stop the Turbopump.
10. Take sample out:
 - a. Close **Vacuum Valve** and **Sample Well Valve**;
 - b. Charge to 3 psi;
 - c. Close **Charge Valve**;
 - d. Take sample out;
 - e. Close Sample Well with a cap.
 11. Open **Vacuum Valve** and **Sample Well Valve**.
 12. Pump to -27.5 mm
 13. Close **Vacuum Valve** and **Sample Well Valve**.
 14. Test vacuum for one min.
 15. Open **Vacuum Valve** and **Sample Well Valve**.
 16. Close **Main** and **Shutoff** valves on Research Helium cylinder. **Main gauge:** _____ psi
 17. Take the Cold Trap out of LN2 dewar and rest it on a stand next to console.

Leave the system under vacuum with the GAST pump overnight to clean the Cold Trap and the WaveGuide body. Keep Temperature Controller ON to monitor how WaveGuide warms up.

Reverting to a Room-Temperature mode

The system is fully warmed up and was under vacuum (read "clean").

Four-Way Valve - **Both Sides**. Vacuum Valve and Sample Well Valve - **open**.

1. Record temperature controller readings: A = _____ K, B = _____ K
2. Turn off Temperature controller (on the back of the unit).
3. Open **main** and **shutoff** valves on Research Helium bottle; **regulator** valve should be around 30psi.
4. Close **Vacuum Valve** and **Sample Well Valve**.
5. Charge to 3 psi.
6. Close **Charge Valve**.
7. Turn **Four-Way Valve** to **Normal Position**.
8. Turn GAST pump **OFF**.
9. Research Grade Helium cylinder:
 - a. Record Helium pressure on the **main gauge:** _____ psi.
 - b. Close a **main valve** and a **shut-off valve**.
10. Unlock the sample well cap and release pressure.
11. Do NOT twist-lock the cap; it is only needed to protect the Sample Well from dust in a room-temperature operation.