

Standard Cryogenic Operation with an inline Cold Trap

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

8/30/2024

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NOTE: 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

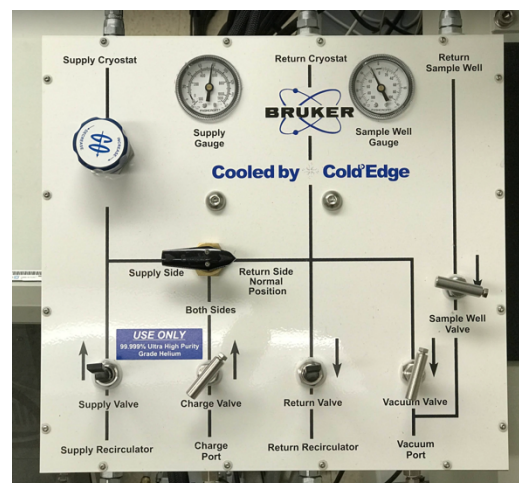
Current State: The sample well was open to air in room-temp operation

Pumping down the vacuum insulation

1. Note the positions of the gauge arms on the turbopump (tap the gauge bottom!):
Black: _____ Pa (expected: "air") **Red:** _____ Pa (expected: 10^{-2})
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 (expected: 27k)
5. Record **Red Gauge** reading: RG= _____ Pa .
Note this value: this is the deepest vacuum the pump can achieve!
6. Open valve on pump CCW all the way and then 1/8 turn back.
7. Wait 15 sec - till pump display again shows "at speed".
8. Open valve on the WaveGuide Cryostat: do ten 1/2 turns of the valve CCW.
9. Leave Turbopump to run overnight.

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

1. Check that **Charge Valve** is closed
2. Go to a Research Grade He cylinder:
 - a. Open **Main valve**;
 - b. Note the gas pressure: _____ psi
 - c. Open **Shutoff valve**.
 - d. Note pressure on the **Regulator valve** gauge: _____ psi;
3. Close sample well with a twist cap. Make sure the O-ring is in place!
4. Close **Vacuum Valve** and **Sample Well Valve**
5. Turn on GAST pump (turn on a power strip).
6. GAST pump reached _____ mm when pumping on itself. Must be -27 mm
7. Turn **Four-Way Valve** to **Both Sides**.
8. Open **Vacuum Valve** and **Sample Well Valve**.
9. Drop pressure to negative 26-27 mm Hg.
10. Close **Vacuum Valve** and **Sample Well Valve**.
11. Check vacuum: **Sample Well Gauge** must stay at -27.5 mm for one minute.
12. 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 items c-d
13. Close main valve on a Research Helium cylinder.



14. Record Regulator pressure: _____ psi
NOTE: if this pressure is different than 30-40 psi, proceed with the protocol but contact NMR staff to ask to adjust at the earliest convenience.
15. Check vacuum: **Sample Well Gauge** must be at -27.5 mm
16. Open **Vacuum Valve** and **Sample Well Valve**
17. Check Turbopump pressures: black: _____, red: _____
18. 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 60-90 min.

1. Refill LN2 dewar for the cold trap to the top of the cylindrical portion (not the neck!)
2. Check Turbopump: speed = _____ rpm, red gauge = _____ mBar.
3. Check vacuum in the sample well:
 GAST gauge = _____ mm, Sample Well Gauge = _____ mm
4. On a Research Grade He cylinder:
 - a. Open the **main valve**
 - b. record **main gauge** reading: _____ psi.
5. Close **Vacuum Valve** and **Sample Well Valve**.
6. Check vacuum for one min.
7. 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
8. 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**
9. Charge to positive 3 psi and close **Charge Valve**
10. Set **Four-Way Valve** to **Normal position**

11. Slowly lower the Trap in the LN2 dewar.
Wear proper PPE: cryogenic gloves, safety goggles and closed-toe shoes!
12. **Turn on N2 purge gas:**
 - a. turn the yellow handle to horizontal position.
 - b. adjust the ball on the flow gauge to be at to 10 liters per minute.
13. Turn on the EPR instrument:
 - a. Open water (first "R", then "S"); *Check for water leaks!*
 - b. Turn on the Haskris chiller; *Check for water leaks again!*
 - c. Turn on wall power to **both EPR and Cryoplatfom**;
 - 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).
Sensor A is a heat exchanger and Sensor B is a sample well.
14. Test tuning of the empty EPR cavity: must tune around 9.47 GHz.
15. Check that **Return Valve** and **Supply Valve** are closed (horizontal)
16. Turn on **Recirculator Compressor**
17. Open **Return Valve**
18. Open **Supply Valve**
19. Record **Supply Gauge** reading: _____ psi
NOTE: If you suspect helium is still contaminated (incomplete cooldown in a previous run), you should pause at this step for one hour to allow Cold Trap to work before the F70 is started.
20. Record the pressure reading on F70 gauge: _____ psi (inner scale) - must be 195psi
21. Turn on **F70** (start the cold head)
22. Turn on Temp Controller Chart recorder on a computer to view a temperature graph.
 - a. In Firefox, open 192.168.1.12
 - b. Click **Utilities**
 - c. open "Chart Recorder". It may not launch on first try. Close the Java window (click (x)) and relaunch from the web page button. Keep trying.
 - d. Change file name for the recording and its folder
 - e. Check boxes for **Input A** (heat exchanger) and **Input B** (sample well).
 - f. Click **Start** to begin recording
23. If **Supply Gauge** reading is smaller than 100 psi - add more helium by **VERY SLOWLY** opening **Charge Valve** till **Supply Gauge** reads 100 psi.
24. Reduce **N2 purge gas** flow to 2 liters per minute
25. Wait for 60 min minimum to cool down below 20K
26. Test tuning of the empty EPR cavity: must tune around 9.47 GHz

Sample change procedures

NOTE: 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.

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. Once the gauge arm passes 5 psi -
 - i. if a previous sample is there: loosen the collar and pull tube 2 inches up (to remove it from the dewar in the EPR cavity); tighten the collar back
NOTE: if the O-ring is too hard and tube does NOT move - warm up the cap with a hair drier.
 - ii. unlock cap (hold hand to keep it) to see if gas lifts the cap; note the overpressure valve opening when you push on a cap to stop gas flow.
 - c. If pressure is not enough, reset all:
 - i. lock the cap,
 - ii. close the **Charge Valve**,
 - iii. open **Vacuum Valve** to drop pressure;

- iv. close Vacuum Valve.
 - d. Open **Charge Valve** a more this time and repeat the test **b-c**
 - e. 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 **b-e** one more time.
- 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.
- 19. Turn on Xenon, tune and record a quick spectrum. If the system cannot tune automatically, tune manually. If you will see sharp periodic spikes on the spectrum: the tube vibrates and needs adjustment - see next section.

NOTE: Your sample is cooled down slower than the sensor, therefore, stable sensor reading does not mean that the sample is also at this temperature yet. You should give the sample time to fully cool down at least 10 min after sample temperature dropped to within 5K from the target. Keep this time the same for all your measurements for reproducibility.

Adjustment of sample tube depth to prevent vibrations

Current state: Sample tube is inserted and the system has cooled down to the setpoint.

Observation: you observe fluctuations of the resonance profile such that the system cannot tune or you see significant spikes on the EPR spectrum.

Solution: the depth of the tube must be adjusted down to meet the dewar floor.

1. Switch Xenon to **Tune** mode, select **Operate**, 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. 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:
 - 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. CAUTION: Push **gently** on the top of the tube to positively meet the bottom of the dewar. PUSHING TOO HARD WILL CRUSH THE DEWAR!
 - d. **Slowly** tighten the collar.
9. At this point, fluctuations are either gone or become smaller. If they are still present, check the tube adjustment: place your finger on top of the tube and provide **light and constant pressure downwards** while observing the gauges. If fluctuations stopped, the tube is properly down but is too heavy and you will have to provide this pressure while measuring (see the NOTE below).
10. Close **Charge Valve**.
11. Vacuum test:
 - a. Turn **Four-Way Valve** to **Both Sides**;
 - b. Open **Vacuum Valve** and **Sample Well Valve**;
 - c. Drop pressure to -27mm;
 - d. Close **Vacuum Valve** and **Sample Well Valve**;
 - e. 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.

NOTE: The tube with a large and heavy upper portion, even when adjusted properly down, **will vibrate** due to operation of the WaveGuide and **will produce fluctuations that prevent tuning and recording smooth spectra**. In this case, it is sufficient to keep your finger **lightly pressing down onto the tube cap** to dampen these fluctuations while system is tuning or recording.

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 temperature reduces towards a target too slowly or starts climbing up, this is a sign of a blockage in the heat exchanger inside the WaveGuide. To clear a small blockage:
 1. Adjust the sample tube up by 1 cm
 - close Supply Valve
 - remove helium pressure from the system: SLOWLY and PARTIALLY turning the Four-Way Valve towards Both Sides while watching the Sample Well Gauge
 - When Supply Gauge drops to 0 psi, turn Four-Way Valve to Normal Position.
 - Close Return Valve.

- Open **Charge Valve** to see pressure increasing on a **Sample Well Gauge**.
 - Once the gauge arm passes 5 psi, loosen the collar and move the tube up 1 cm. NOTE: if the O-ring is too hard and tube does NOT move - warm up the cap with a hair drier.
 - close the **Charge Valve**
 - Turn **Four-Way Valve** to **Normal**.
 - Open **Return Valve**.
 - Open **Supply Valve**.
2. Turn off **F70**
 3. Wait for a sensor A to exceed 40K (about 10 min)
 4. 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.
 - Once you cooled down successfully, adjust the tube down to the bottom of the dewar following ***Adjustment of sample tube depth to prevent vibrations***

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. Take sample out:

IMPORTANT! Warming up WITH the sample may lead to tube breaking due to thermal expansion!

 - a. Prepare a "plugged cap": a cap without a sample and with a plug in the top.
 - b. Close **Supply Valve**
 - c. Immediately, start bleeding remaining pressure from **Supply Side** to the **Return Side** by by SLOWLY and PARTIALLY turning the **Four-Way Valve** towards **Both Sides**
 - d. When **Supply Gauge** drops to 0 psi, turn **Four-Way Valve** to **Normal Position**.
 - e. Close **Return Valve**.
 - f. Open **Charge Valve** to see pressure increasing on a **Sample Well Gauge**;
 - g. As it passes 5 psi, note the overpressure valve opening.
 - h. Take sample out.
 - i. Close Sample Well with the plugged cap.
 - j. Close **Charge Valve**.
2. Vacuum test:

- a. Turn **Four-Way Valve** to **Both Sides**;
 - b. Open **Vacuum Valve** and **Sample Well Valve**;
 - c. Drop pressure to -27mm;
 - d. Close **Vacuum Valve** and **Sample Well Valve**;
 - a. Test vacuum for one min
3. Open **Return Valve**.
4. Open **Supply Valve**.
5. Stop temperature control (if used):
 - a. Push **Heater Range** -> choose **Off** -> **Enter**.
 - b. **Setpoint** -> dial **0 K** -> **Enter**.
6. Turn OFF the **F70**.
7. If Cold Trap is immersed in LN2: let the Recirculator Compressor run for 30 min and clean helium while system is warming up.
8. Close **Supply Valve**.
9. 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!
 - d. When Supply Gauge drops to 0 psi, turn **Four-Way Valve** fully to **Both Sides**.
10. 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**.
11. Turn OFF the **Recirculating Compressor**.
12. **Shut OFF N2 purge gas**: turn the yellow handle to a vertical position.
13. 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 CW;
 - h. Stop the Turbopump.
14. Test vacuum
 - a. Verify that vacuum on **Sample Well Gauge** is about -27 mm
 - b. Close **Vacuum Valve** and **Sample Well Valve**.
 - c. Test vacuum for one min.
 - d. Open **Vacuum Valve** and **Sample Well Valve**.

15. Close **Main** and **Shutoff** valves on Research Helium cylinder. **Main gauge:** _____ psi
16. 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 the WaveGuide warms up.

Day 3: Reverting to a Room-Temperature mode

The system is fully warmed up and was under vacuum overnight (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.
12. **Check if N2 purge gas line is closed** (yellow handle in a vertical position)