

Guide to Operation of Nitrogen VT Experiment in Bruker EleXsys E500 EPR Spectrometer

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The current configuration of the nitrogen VT apparatus used with the Bruker EleXsys 500-A EPR spectrometer is designed to provide variable temperature EPR experiments in the range of 350°K/170°C to 100°K/-178°C. This is accomplished by two means. For experiments at or above room temperature house nitrogen is used and for experiments below room temperature liquid nitrogen is boiled into a gas. The temperature of the subsequent gas stream is then maintained via an in-line heating element. Setup of the apparatus is quite simple but close attention detail is suggested.

1. Connect the cryostat to your cavity. (See Figures 1 & 2.) Remove the top and bottom blue collet nuts and white plastic collets from your cavity. Carefully insert the quartz end of the cryostat assembly upwards through the bottom of your cavity. Screw the lower retaining nut of the dewar insert holder onto the collet stack of your cavity. (Do not cross thread or over tighten.)
2. Fit the sample holder assembly onto your cavity. Slide a large diameter plastic collet over the dewar insert and into the collet stack of your cavity. This will protect the quartz from breakage. Next, screw the upper VT collet adapter (part N) onto the collet stack of your cavity. Care should be taken to avoid cross threading the VT collet adapter as it is made of very soft material and can be easily damaged. (See figure 3.) Place a sample collet that is appropriately sized for your sample tube into the recession of the upper VT collet adapter. Screw a standard collet nut onto the upper VT collet adapter, again avoiding cross threading.

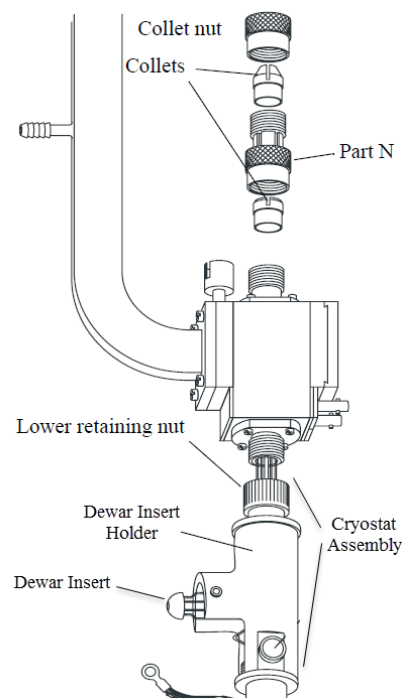


Figure 1. Cavity and Cryostat Assembly

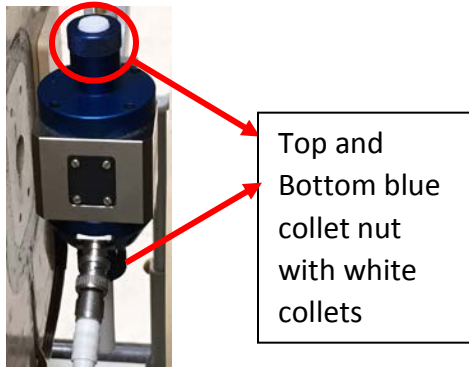


Figure 2. Blue collet nuts.



Figure 3. VT collet adapter.

3. Finally plug the thermocouple into the three-prong connector on the back of the temperature controller. (See Figure 4) When removing, be careful to pull on connector, not the cable!



Figure 1. Thermocouple to temperature controller.

4. The glass transfer can now be fitted to the cavity dewar insert. This can be tricky and care and patience is advised. The glass transfer line is delicate. The ball fitting of the cavity insert dewar should be lubricated with silicone vacuum grease prior to installation to insure a gas tight fit. (See Figures 5 and 6) Leaks along the gas stream path will result in temperature instability and icing which can affect temperature control and stability and inhibit your VT experiments. When applying silicone grease it is recommended that only a thin layer be applied and care taken not to get excess grease inside the ball fitting opening itself. This will restrict gas flow and affect the temperature stability during your experiments. If excess grease is observed inside the openings it can be removed with a cotton swab. Swabs can be found in

the equipment cabinets in the lab. Once the fittings are greased the transfer line should be connected to the cavity first. The transfer arm is held in place by spring tension from the bayonet collar. Position the bayonet collar so that the two holes are in the vertical or up and down position. Gently push the end of the transfer arm against the ball fitting on the rear of the cavity insert. (See Figures 5 and 6) Do not push too hard but apply gentle even pressure. Next rotate the black barrel connector 90° (CW) to engage the bayonet collar. Avoid turning the collar too far as it can cause the tension spring to slip past the bayonet posts and will make removal difficult. The Transfer arm should be held gently to the cavity dewar insert. If not repeat the installation steps again. Should the transfer arm become stuck onto the cavity insert do not apply excess force in its removal because both the cavity insert and the transfer arm can easily be broken. Use the transfer line support on the base plate to support the transfer arm parallel to the floor. (See Figure 5 and 7)

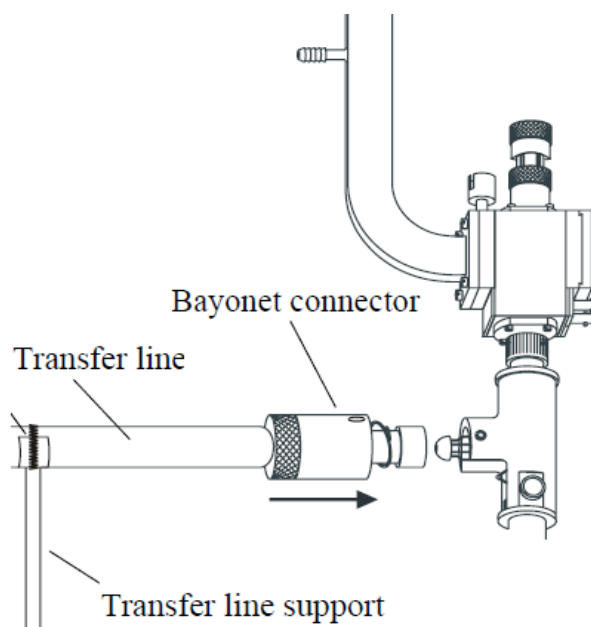


Figure 5. Transfer Arm and Cryostat



Figure 6. The ball fitting in the cavity insert dewar(left) and transfer arm(right)

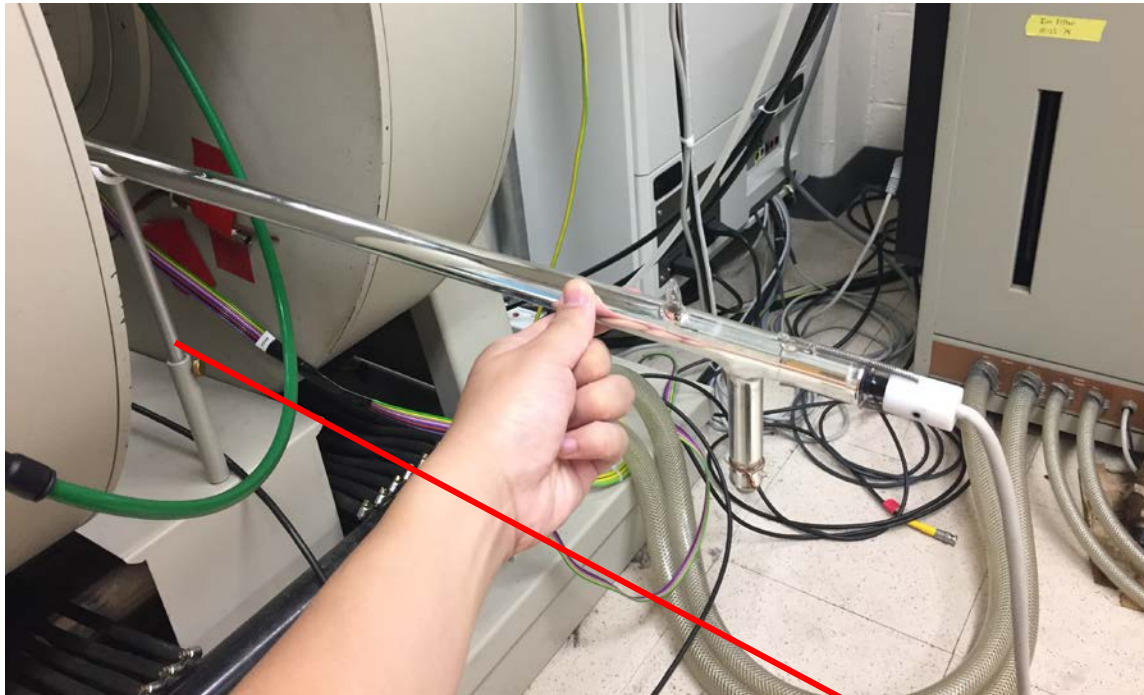


Figure 7. Transfer arm in position

Transfer line support

5. The next steps in the setup procedure are dependent on the temperature range needed for the given experiment. Elevated temperature experiments will use the house nitrogen gas supply and low temperature experiments will require liquid nitrogen.

Low Temperature setup

For low temperature experiments fill 25 liter supply dewar with liquid nitrogen and position it behind the magnet. Place the dewar insert heater into the top the dewar and use the big silver spring clip to fix the rubber stopper. (See Figure 8 and 9) This part may take a while to let the whole heater cool down. This insert contains the boil-off heater used to generate the cool gas stream needed for the low temperature experiments and liquid level sensors to warn the operator when the liquid level is low and when the dewar needs be refilled. If the liquid nitrogen level is low the N2 Fill indicator will light on the controller



Large rubber stopper

Figure 8. Dewar insert heater

front panel. When the dewar becomes empty the Empty indicator will light and the in-line heater and boil-off heaters will be disabled to avoid damage resulting from overheating. A thin application of silicone grease applied to the large rubber stopper (See Figure 8) on the dewar insert will help provide a good gas tight fit to the 25 liter supply dewar and help maintain a more stable gas flow. With the dewar insert in place connect the heater cable to the “LN2” connector that is labeled on the back of the temperature controller.

When the transfer arm has been attached the cavity insert gently slide the 25 liter supply dewar under the vertical end of the transfer arm and position the ball joints to fit. (See Figure 7 and 9) This can be difficult at times and will require patience to precisely align the ends. Avoid applying under pressure to the transfer arm and the cavity dewar insert. Make sure to obtain a good gas tight fit between the transfer arm and the supply dewar. Next attach the silver spring clips to the glass protrusions on either side of the transfer arm. (See Figure 9) Once this is in place adjust the support mount on the base plate inside the magnet cavity so it supports the transfer arm but does not push apart the ball joint fittings at the top of the supply dewar. The cable from the transfer arm can now be connected to the “Heater” connector labeled on the back of the temperature controller.

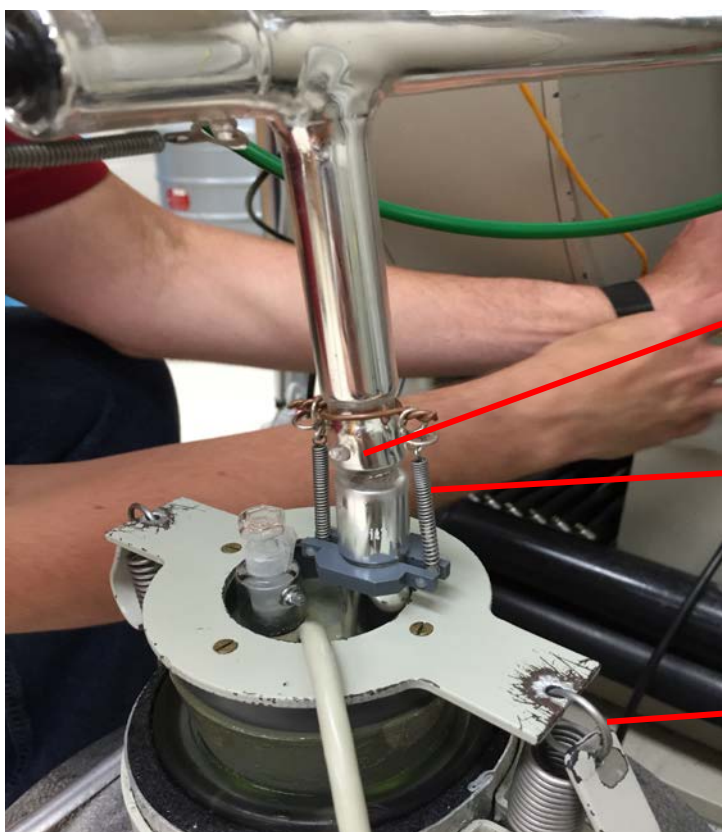


Figure 9. Connection between transfer arm and dewar insert heater

Tight fit part

Silver Spring

Big Silver Spring clip

Once the physical hardware has been assembled you can start the cool down process. Turn on the temperature controller positioned on top of the EPR magnet table. The power switch is located on the front right side of the controller. (See Figure 10) The desired operating temperature for your experiments will determine the required gas flow necessary to maintain adequate temperature stability. The gas flow is inversely proportional to cavity temperature. The higher the gas flow rate the lower the ultimate temperature obtainable. As the boil off rate increases so does liquid nitrogen consumption. Set your target temperature (setpoint) by clicking the orange up and down arrows below the LCD screen. The boil off heater, labeled “LN2” is located to the left of the power switch. The boil off rate is manually controlled via the up and down arrows on either side of the LN2 switch. Consult the accompanying graph to help determine the approximate setting of the LN2 heater for the desired experimental temperature. (on the right side of controller’s body) The panel meter above the LN2 switch gives a reading of the heater current being requested. Start the boil off heater and monitor the cavity temperature on the temperature display. Do not start the in-line heater labeled as “HEATER” at this time but allow the system to equilibrate and the transfer line and cavity to cool to operating temperature. Turn on the stream heater when the temperature is getting close to your target temperature. Wait for temperature equilibration.



Figure 10. Temperature controller

Elevated Temperature setup

You will still need the transfer arm to connect to the cavity insert. However, you don't need the dewar insert heater to connect to the vertical end of transfer arm. (See Figure 6) You will need to use house Nitrogen gas line to connect to the vertical end instead. (See Figure 11)

Before turning on the in-line heater you will need to set the temperature setpoint in the EuroTherm temperature controller. This is done before turning on the heater. Use the orange up and down arrow buttons below LCD screen on the EuroTherm controller to raise or lower the set point temperature to the desired level. (See Figure 9) Once the set point temperature has been adjusted and the displayed temperature is near the desired set point you may turn on the Heater switch labeled "HEATER". This will engage the automated temperature controller and will regulate the temperature of the gas stream. It may take 5-10 minutes for the system to equilibrate at the control temperature. Be patience.

Recommendation: Always test your sample in your VT range first by pre-cooling or pre-heating it. Make sure the sample tube and sample can hold your VT range. Then you can insert your sample to cavity. After you insert your sample, it may take a few minutes for the whole system to equilibrate again.



Figure 11. House Nitrogen gas line connected to vertical end of transfer arm

6. When you are done, turn off all the heaters in the temperature controller. Turn off the power of temperature controller. Then you can remove all the VT devices and put back the room temp devices. **Attention: the cavity part is either very hot or cold. Please be patience and watch out for it.** You can always use house Nitrogen gas line to connect to vertical end of transfer arm to flush the cavity. (See Figure 11)