

OPERATING INSTRUCTIONS FOR THE ELECTRON PARAMAGNETIC RESONANCE (EPR) SPECTROMETER (MODEL BRUKER EMX-PLUS)

Location – BRWN 5106

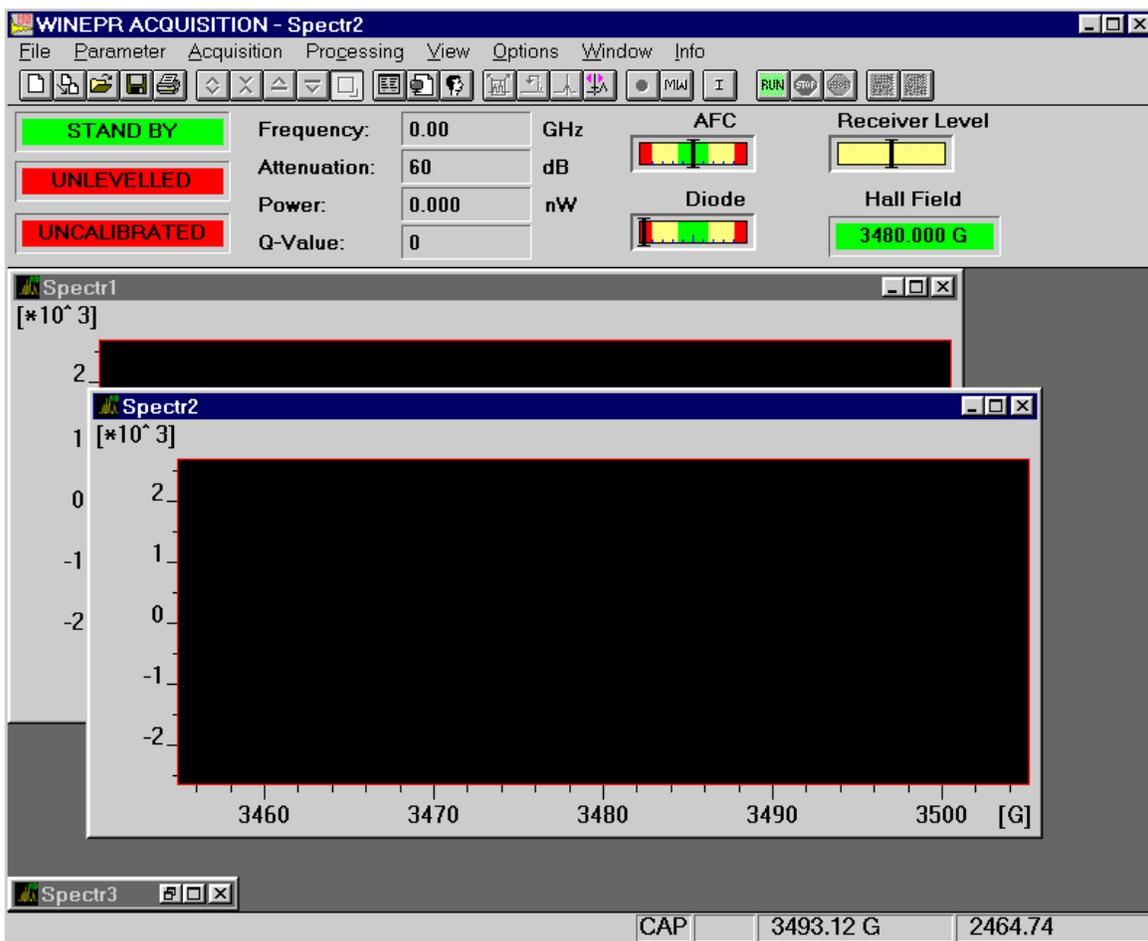
Revised on – 2/2022

A. INTRODUCTION

The Bruker EMX-Plus EPR is a general-purpose X band spectrometer that can be outfitted with a wide variety of sample cavities (resonators). Among the cavities available in addition to the standard cavity are: 1) an ER 4119 HS that is a high sensitivity cavity suitable for general use, 2) an ER 4116 DM useful in investigating forbidden EPR transition, 3) and ER 4123 DL for performing power saturation experiments on high dielectric samples. Many of these cavities will accommodate dewar inserts for temperature control.

Due to the different needs of each research group and the variety of sampling techniques, each user is responsible for providing his/her sample preparation materials and notifying Research Instrumentation Center (RIC) staff when instrument reconfiguration is needed.

The instrument is completely software driven using the WinEPR acquisition package provided by Bruker. There are no physical buttons or knobs. Fortunately, the user interface follows familiar Windows conventions. The figure below is a screen shot of the WinEPR Acquisition main window. This interface window can be divided into the five main areas described below.



Menu Bar – Like all Windows programs, there is a Menu Bar at the top of the window used to access all other program features. All of the features listed in the other areas of the interface are directly accessible via the menu bar.

Button Bar – This bar is located just below the Menu and can be toggled visible/invisible using the View option in the Menu bar. It is highly recommended to leave it visible since all spectrometer functions needed for data acquisitions are most easily accessed from the tool pallet. The table below shows each icon and lists its “tip strip”. The specific functions of many are discussed in the relevant sections below.

 New Spectrum  Duplicate Spectrum  Open Spectrum  Save Spectrum  Print Spectrum	 Experimental Parameters  Experimental Options  Comment Parameters	 Run / Stop Script  Run Acquisition  Stop Acquisition  Stop Acquisition at End Scan
 Times 2  Divide by 2  Expand  Contract	 Change Center Field and Sweep Width  Change Center Field  Change Static Field  Interactive Receiver Level	 Send Spectrum to WinEPR  Send Spectrum to Simfonia
 Reset Display  Rectangular Zooming  Cursor Moving	 Microwave Fine Tune  Microwave Bridge Control  Interactive Spectrometer Control	

Instrument Monitor – This area is located just below the Button Bar and shows the status of the microwave bridge and signal channel. The displays can be toggled visible/invisible by using the View option in the Menu bar.

Data Area – This area fills the center of the screen and is populated by various windows. The windows can be any combination of spectra, parameters, and instrument control windows.

Status Line – This is located at the bottom of the window and displays current instrument status and messages.

B. OPERATION: STARTUP

Follow the procedure below to insure proper instrument operation. Although spectra can be obtained immediately after startup, waiting at least an hour for the magnet and electronic components to thermally stabilize before taking spectra is recommended.

EMX STARTUP

1. Nitrogen Purge – Open the gas supply valve on the liquid nitrogen dewar. This will purge the EPR cavity, waveguide, and microwave bridge of water vapor which absorbs microwave energy and oxygen which gives a signal since it is paramagnetic
2. Cooling water – Open the two chilled water valves on the wall to the right of the computer desk.
3. Heat Exchanger – Use the rotary switch labeled “Heat Exchanger” located on the control box above the water filter. Be careful to not mistakenly push the Main power button.
4. Magnet power supply – The magnet power supply not only controls current in the electro magnet but powers the console and microwave bridge as well. It is powered on in a two-step process as detailed below.
 - a. Press the ELECT. ON (Electronics) button in far left, upper corner. Wait for the five red LED-warning lights on far right to extinguish before continuing. If the LEDs do not extinguish, confirm that the cooling water is on and repeat this step.
 - b. Press POWER ON, which is located to the immediate right of the ELECT. ON switch.
5. Console Power – Press the round button located in the lower, center of the cabinet. This also provides power for the Microwave Bridge located on the top, left corner of the shelf above the electro magnet. The console houses:
 - 1) Signal Channel – A phase sensitive detector measuring the EPR signal by comparison with a signal of known frequency.
 - 2) Magnetic Field Controller – Controls both the magnitude and sweep rate of the magnetic field during a scan.
 - 3) Modulation Amplifier – This unit provides the modulated reference signal for detection in the signal channel.

**NOTE – The console houses ~\$150,000 of electronics.
DO NOT set samples or your coffee on it!!!!**

COMPUTER LOGON

The computer is usually left on and can be awakened by pressing any key on the keyboard. If the screen does not respond within 30 seconds of pressing a key, press the power button on the front of the case. At the Logon prompt enter your “User Name” and “Password”. The “Domain” entry should be set to BoilerAD.

Once logged on, WinEPR shortcuts are visible in the upper right-hand corner of the desktop. “WinEPR Acquisition” is the application used to run the spectrometer and obtain data. “WinEPR Processing” and “WinEPR SimFonia” are for analysis and simulation, respectively. This training document focuses only on the acquisition software and basic instrument operation.

Launch the WinEPR Acquisition by double clicking its icon. It takes about 90 seconds for the software to fully launch. Once the window opens, click the Interactive Spectrometer Control  button and click the “Calibrated” check box in the upper right of the window that opens.

C. OPERATION: LOADING SAMPLES

Inserting / Changing Samples

(Room Temperature)

1. If the Microwave Bridge Controller window is not open, click on the Microwave Bridge  button on the tool bar.
2. Select the STANDBY or TUNE mode. See the image below.
3. To avoid contaminating the cavity, clean the outside of the sample tube with a Chem Wipe.
4. Center the sample tube in the collet (loosen the collet ring if necessary) and gently slide it down until either the sample is centered in the cavity or the tube is resting on the pedestal. Tighten the collet ring just enough to prevent the sample tube from moving.

(Helium Variable temperature)

1. If the Microwave Bridge Controller window is not open, click on the Microwave Bridge  button on the tool bar.
2. Select the STANDBY or TUNE mode. See the image below.
3. Position the sample tube in the top-hat using the cavity gauge drawing to center the sample in the cavity. Tighten the top-hat ring so that the sample tube is snug.

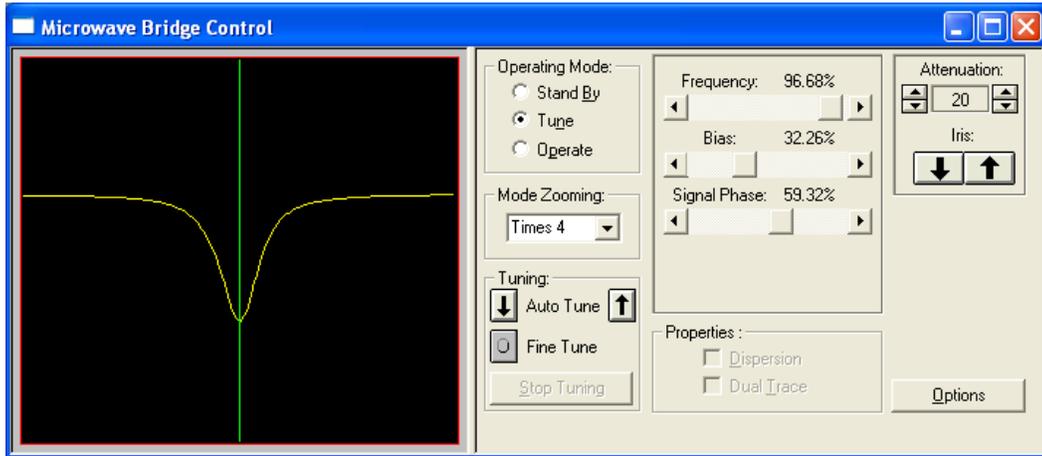
Notes – Do not over loosen or remove the upper portion of the top-hat.

- The sample should be located in the center of the cavity for the best response.
- Samples must be frozen prior to inserting them in the cavity!

4. To avoid contaminating the cavity, clean the outside of the sample tube with a Chem Wipe. Be sure to remove any frost.
5. In as smooth and rapid a motion as possible remove the top-hat currently on the VT cavity and replace it with the new sample by carefully sliding the sample tube straight down into the cavity. Once the top-hat contacts the vent tube, apply gentle but firm pressure until the top-hat snaps into place.
6. Tune the cavity and set parameters to the instrument as usual.
7. Wait for the sample to thermally equilibrate (~10 minutes).

Tuning the Microwave Cavity and Bridge

1. If the Microwave Bridge Controller window is not open, click on the Microwave Bridge  button on the tool bar.
2. Select the TUNE mode. See the image below.



3. Use the pairs of up/down arrow keys set the attenuation to 25dB. The leftmost pair changes in units of 10dB and the rightmost pair changes in 1dB units.
4. Use the right/left arrow buttons of the Frequency slider to center the tuning dip as shown above. The system takes long fractions of a second to respond so do not click too fast. Use the right/left arrow buttons on the Signal Phase slider to adjust phase until the tuning dip is symmetrical as shown above. The system takes long fractions of a second to respond so do not click too fast.
5. Either click the Up or Down arrow to start the cavity auto-tune process. The autotune routine will then adjust the frequency, phase, bias, and iris coupling for optimal performance. Wait for this process to complete as indicated by the three green indicators in the left of the Instrument Monitor window.

Note – If auto-tune fails, follow the procedure in Appendix C.

6. Click the Microwave Bridge  button to close the window.

D. OPERATION: TAKING SPECTRA

Setting Parameters

Instrument parameters may be loaded from disk by opening an existing spectrum, or set manually as described below. Whichever method is chosen it is important to verify that the cavity calibration file is loaded and is being used.

1. Setting parameters manually
 - a. Click the New Spectrum  button to ensure that a previously open spectrum is not accidentally overwritten.

- b. Click the Experimental Parameters  button and enter the desired parameters.

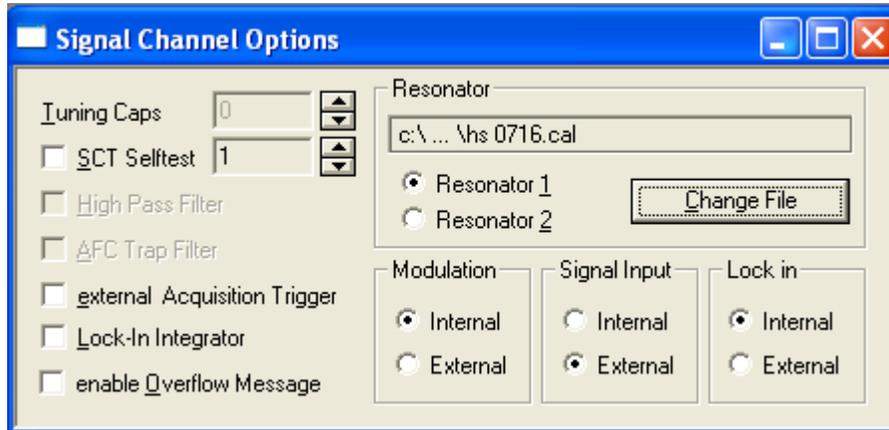
Notes

- a) If looking for radicals centered around $g = 2$, check the box just above the “Center Field” numeric to automatically set the “Center Field” parameter.
- b) Microwave power – The range is 1 to 200 mW. Usual values are 1 to 20 mW.
- c) Modulation Amplitude – The range is 0.1 to ~20 (dependent on cavity) gauss. 5 to 10 gauss is typical. The amplitude should be no higher than the width of the narrowest line (in gauss) in the spectrum. See page 2-19 in the Bruker WinEPR acquisition manual for a discussion of over modulation.
- d) Receiver Gain – Typical range 100 to 5000.
- e) Conversion Time – This is the time allotted for the A/D process and directly influences resolution in the Y-axis. This value multiplied by Resolution in X (# of points collected across spectrum) yields the sweep time for the spectrum.
- f) Time constant – This value should be less than one tenth the time needed to scan the narrowest line in the spectrum. See page 2-20 in the Bruker WinEPR acquisition manual for a discussion of using an excessively long time constant.
- g) If a single scan does not yield spectra with reasonable signal-to-noise, either of the two options below can help.
 - a. Repetitive scan
 - b. Increased time constant with either increased conversion time and/or X resolution to slow the scan through the signal

2. Loading/Verified cavity calibration

- a. Click the Interactive Spectrometer Control  button.

- b. Verify that the “Calibrated” check box in the upper right is checked.
- c. Click the “SCT Options” button at the lower right to open the Signal Channel Options dialog shown below.



- d. Verify that the loaded calibration file in use matches the name of the cavity currently installed. See the table below for the calibration file names. If necessary, click the “Change File” button and select the proper file.

Resonator Name	Calibration File
ER 4102 ST	ST 0203.cal
ER 4103 TM	TM 9304.cal
ER 4119 HS	HS 0716.cal
ER 4116 DM	DM 0708.cal
ER 4123 D	D 0247.cal

Acquiring Data

After setting the scan parameters, click the Run  button to initiate data collection. Acquisition can be terminated immediately using the Stop Acquisition  button or at the end of the current scan via the  button. This latter operation is useful during averaging operations if the desired spectral quality has been achieved.

E. INSTRUMENT OPERATION: SHUTDOWN

1. Set Microwave Bridge controller to STANDBY
 - a. If the Microwave Bridge Controller window is not open, click on the Microwave Bridge  button on the tool bar.

- b. Select the STANDBY mode.
2. Exit the WinEPR software.
3. “Sign Out ...” - Right click on your name at the top of the Windows “Start” menu.
4. Remove the sample from the cavity and replace the cap.
5. Power down in this order.
 - a. Turn off the console by pressing the lit button in the center of the unit.
 - b. Turn off the magnet power supply (PWR first and then ELECT).
6. Turn off the Heat Exchanger – Be careful not to mistakenly push the Main power button.
7. Close the chilled water valves.
8. Close the nitrogen gas valve.
9. Sign the logbook.

F. CONTACTS

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Appendix A – VARIABLE TEMPERATURE OPERATION (COLDEDGE)

Using high purity helium as a coolant, sample temperature can be varied from 150 K to ~5 K. This is done by flowing helium gas at the desired temperature through a dewar assembly installed within the cavity that surrounds the sample. The cold gas is generated by passing 99.999% helium at room temperature through a heat exchanger connected to a Sumitomo closed-loop compressor system. This eliminates the need for using liquid helium and reduces the cost of operation by a factor of 10.

Due to the extreme low temperatures, great care must be taken when inserting samples not to thermally stress the sample/cavity, contaminate the cavity with room temperature air, or crush the heater and thermocouple that sit just below the sample. To reduce the thermal shock, which usually results in broken sample tubes, all samples **MUST** be frozen in liquid nitrogen before placing them in the cavity. Prefreezing samples also greatly reduces the time needed for temperature equilibration. Sample tubes **MUST** be rigorously cleaned to avoid cavity contamination. Wash your sample tubes between uses with an appropriate solvent and wipe them off with a ChemWipe before inserting them in the cavity. System maintenance due to contamination by a careless user may be billed to the PI at a cost of \$60/hr with a 4-hour minimum!

PROCEDURES – The procedures below are intended to be used as needed and are not listed sequentially.

STARTUP, COOL-DOWN, SHUTDOWN

Due to the complexity of the ColdEdge VT system, center staff will perform these operations. Users will need to coordinate startup and shutdown times with center staff.

SETTING TEMPERATURE

The Oxford Instruments Cryostat and LakeShore controller combination uses a heater to warm the flowing cold helium gas to the desired temperature. To set the temperature press the “Setpoint” button and enter the desired temperature and hit “Enter”. If you find that you have entered an incorrect value or menu that you didn’t want, simply press the cancel button.

CHANGING/INSERTING SAMPLES

1. Place the Microwave Bridge Controller in STANDBY mode.
2. With one hand, remove the sample/cap from the cavity. With the other hand, insert the new sample.

Notes – This must be done as quickly as possible to prevent room temperature air from entering the cavity.

– Samples must be frozen prior to inserting them in the cavity!

3. Tune the cavity and set parameters to the instrument as usual.
4. Wait for the sample to thermally equilibrate (~10 minutes) and fine-tune the cavity.

Appendix B – MANUAL TUNING THE MICROWAVE CAVITY

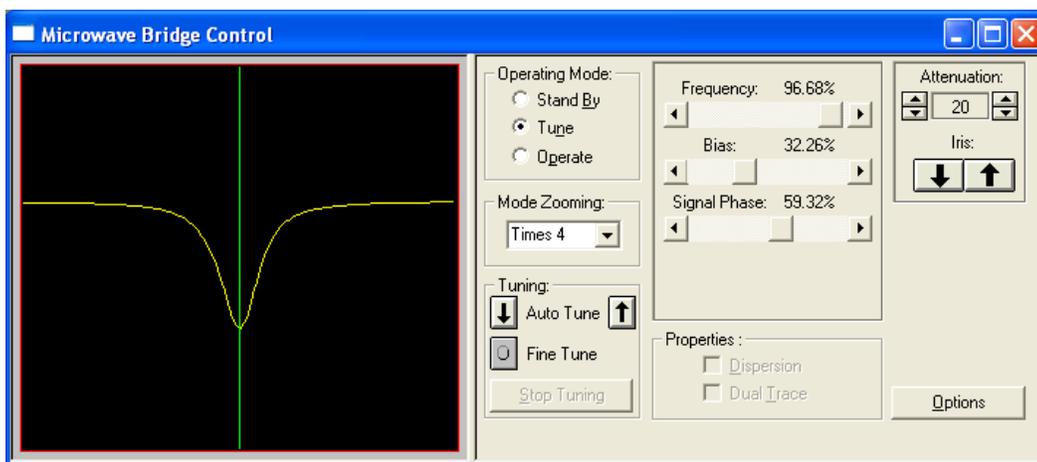
In case the automatic tuning operation (in Part V. of the training outline) fails to properly tune the cavity and bridge, a message indicating this failure will be shown in the mode indicator in the center of the microwave bridge controller display.

Some samples may be somewhat "lossy," i.e., the sample or solvent changes the conditions in the cavity to decrease the cavity absorption dip. If this effect is only minimal, it may still be possible to manually tune the cavity and bridge by the following procedure.

If the sample is very "lossy," it may be impossible to observe a sufficient cavity dip for lock-on by the control system. If the control system cannot be stabilized as indicated by inability to center the LOCK OFFSET meter or the DIODE CURRENT meter, then a different (aqueous) cavity will need to be installed. Contact RIC staff to make this change.

Manually Tuning Procedure (Summarized from section 5.1 of the WinEPR acquisition manual)

1. If the Microwave Bridge Controller window is not open, click on the Microwave Bridge  button on the tool bar.
2. Select the TUNE mode. See the image below.



3. Use the pairs of up/down arrow keys set the attenuation to 25dB. The leftmost pair changes in units of 10dB and the rightmost pair changes in 1dB units.
4. Use the right/left arrow buttons of the Frequency slider to center the tuning dip as shown above. The system takes long fractions of a second to respond so do not click too fast.
5. The dip should cover come about 2/3 of the way to the baseline. If the dip is too small or large, decrease or increase, respectively the attenuation in 1dB steps.

6. Use the right/left arrow buttons of the Signal Phase slider to adjust phase until the tuning dip is symmetrical as shown above and is as deep as possible. If the dip points up, the phase is 180 degrees off. If it has positive and negative lobes, it is ~90 degrees off. The system takes long fractions of a second to respond so do not click too fast.
7. Select the Operate mode and then fine-tune the Frequency to center the AFC indicator in the Instrument Monitor window. Readjust as needed if the AFC drifts during subsequent steps.



8. Set attenuation to 50dB and use the right/left arrow buttons of the Bias slider to center the Diode Current meter (200 μ A) in the Instrument Monitor Window
9. The steps below adjusting the iris (critical coupling) are iterative in nature.
 - a. Lower the attenuation by 10dB.
 - b. Use the Up/Down keys of the Iris, bring the diode current back to center.
 - c. Repeat the steps above until reaching 10dB.

Note – If the AFC lock drifts, center it by adjusting the frequency.
10. While at 10dB of attenuation, adjust phase to achieve maximum Diode current.
11. Cycle through 10, 20, 30, 40, 50 dB to verify that the Diode current remains constant. If not, repeat the tuning process.
12. Click the Microwave Bridge  button to close the window.