

Bruker EPR spectrometer Operating Instructions

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If you have any questions, please let me know. WK

1. Switch ON
 - a. Water
 - b. Console
 - c. Computer program: Bruker EMX
 - d. Power Supply
2. TUNE
 - a. Open the microwave bridge control dialog box (MW button)
 - b. Switch the Tune
 - c. Set the attenuator to 25 dB
 - d. Clean the sample tube and place the sample tube into the cavity
 - e. Tune the bridge and cavity by clicking Auto Tune
 - f. When the tuning process is done, the program would switch to Operate automatically. Check the bars for AFC and Diode are both in the center of green zones. If not, click the Fine Tune, re-auto tune the bridge and cavity, or tune it manually (procedures see below).
3. OPERATE/OBTAIN SPECTRUM
 - a. Click on Create New Spectrum button
 - b. Open the Standard Parameter window
 - c. Set X: field sweep; Y: no Y-sweep; Power: 20dB (= 2mW)
Time constant and conversion Time can stay unchanged.
Change Receiver Gain, Modulation Phase, and Number of X-scans if needed.
 - d. Click OK to close the window
 - e. Click on Run button to acquire a spectrum
Red Stop button: stop immediately. Green button: stop after complete a scan.
 - f. To center the field, click on Interaction Change Button and right click on the center of the spectrum. Open the Standard Parameter window and unclick g=2. Acquire the spectrum again.
4. TURNING OFF SPECTROMETER
 - a. MW to Stand By
 - b. Power Supply
 - c. Computer program: Bruker EMX
 - d. Console
 - e. Water

**** TRY NOT TO SWITCH BETWEEN STAND-BY and TUNE MANY TIMES****
**** IF YOU ARE ONLY SWITCHING SAMPLES OR TEMPERATELY NOT USING THE INSTRUMENT (<2 HR), JUST LEAVE IT IN TUNE MODE****

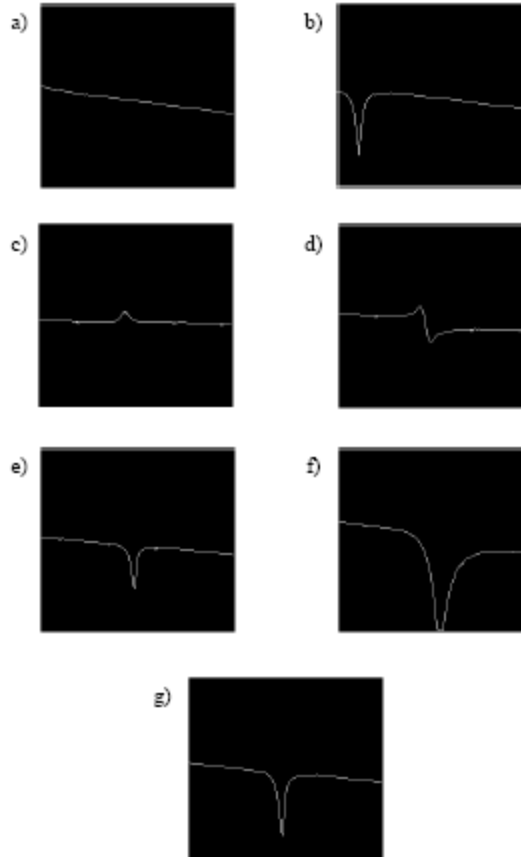
Tuning a Microwave Bridge Manually (5.1)

1. Follow the tuning procedures above 2: a-d.
2. Observe the mode pattern on the display monitor (Gunn Diode Microwave Sources). Have the dip to be the same as Figure 5-2 (g)

Figure 5-2

Mode tuning patterns for a Gunn diode microwave source.

- a) Off resonance.
- b) Slightly off resonance
- c) On resonance, phase 180° off.
- d) On resonance, phase 90° off.
- e) On resonance, correct phase, undercoupled.
- f) On resonance, correct phase, overcoupled.
- g) On resonance, correct phase, critically coupled.



3. Tune the signal phase. Adjust the Frequency and Signal Phase until the dip is in the center and the depth of the dip is maximized and symmetric.
4. Fine-tune the MW source frequency. Click on the Operate button. Adjust the Frequency until the needle of the AFC meter is centered.
5. Adjust the bias level. Set MW to 50dB, and then adjust the bias slider bar until the diode meter needle is centered.
6. Match the cavity. Increase the MW power by 10dB. Click the $\uparrow\downarrow$ iris buttons until the diode needle is centered. Then decrease MW power and repeat the procedure until reach the attenuator to 10dB. When reach 10dB MW attenuation, adjust the Signal Phase slide bar until it reaches a local maximum in the diode current. Verify that you have achieved critical coupling by changing the MW attenuation from 10dB to 50dB with virtually no change in the diode current. For the higher power, set the attenuation to 0dB and then adjust the iris.

Power Saturation (5.4)

1. Follow the procedures on page 1, but change the Y experiment setting to MW power sweep.
2. Set the starting power to 29 dB with the step value of -2 dB. Set the Resolution in Y to 12 (for O₂ and N₂), or 13 (for 20 mM NiEDDA).
3. Click on Run to acquire 2D data set. Transfer the data to WIN-EPR by clicking on the button.
4. In WIN-EPR program, click on 2-D processing under the WINEPR system. You can change the display to Stacked Plot or Scan Plot.
5. For pick peaking purpose, set the display to Scan Plot. Then click on Peak Picking under 2-D processing.
6. Define region: select the region that you want to pick peaks
7. Parameter: change PC value, Peaks Sign: Both, and click on Process
8. Report. Record the peak intensity onto a spread sheet, substrate these two values to obtain p-p amp than enter the values into the PSE.exe

| Power (dB) | Power (mW) | Int + | Int - | p-p Amp |
|------------|------------|-------|-------|---------|
| 29 | 0.250 | | | |
| 27 | 0.398 | | | |
| 25 | 0.632 | | | |
| 23 | 1.001 | | | |
| 21 | 1.586 | | | |
| 19 | 2.514 | | | |
| 17 | 3.985 | | | |
| 15 | 6.315 | | | |
| 13 | 10.01 | | | |
| 11 | 15.86 | | | |
| 9 | 25.14 | | | |
| 7 | 39.85 | | | |
| 5 | 63.31 | | | |

Converting .par files to txt. Files (in order to open these files in EPR programs)

1. Open WINEPR.EXE and load your spectrum or transfer the file directly from Bruker EMX.
2. Click on 1D processing under the tab of WinEPR system. 2D files, such as power saturation data can not be converted using this routine.
3. Under parameters, click on list data file. Save the data in another file name (may need play around the file names).
4. Open Origin, import your new .txt file via ASCII. Delete the first two columns and just leave the last column with the Y coordinate. Saving this file via Export ASCII and this is the format that all EPR programs can read.