

Standard operating procedure for operating JEOL EPR spectrometer:

1. Put on the Chiller placed at the back room. Turn the Front panel red knob clockwise to 'ON' position. – Then after some few seconds press 1 (Green label). Chiller comes on with all the settings done.
2. In the Spectrometer room, put on the main supply switch. Right corner – first switch from right. usually it is kept ON always.
3. Put on the Spectrometer (The unit kept at the bottom of the esr table) by pressing the Red switch to 'ON' position.
4. Wait for some time. Magnet power supply comes on.
5. Turn the PC on by putting on the first two supply switches. Here the last supply point is for the printer.
6. Give password as 'esr'. ESR software opens. Click 'unlock'.
7. Go to Q dip mode to insert/remove a sample tube in the cavity. When the Q dip is tuned properly using frequency, phase, and coupling close this window. Spectrometer is ready for making measurements.
8. Choose the right parameters for centre field, sweep width, modulation amplitude and also gain factor depending on the sample nature.
9. Choose 2 min. time to have a proper recording. For rough checking, 30 sec. option can be selected. When one analysis gets complete, wait till the cursor retraces its path to the left end and save it with appropriate file name for future reference.
10. For other software features/attachments, refer the manual, read, understand and then attempt.
11. Spectrometer is shut down while leaving in the evening. Click 'shut down'. Then click 'power off' option. Power supply to magnet goes off bringing the value to default setting. Then the spectrometer unit under the table can be turned off. PC is turned off subsequently.
12. Chiller is put off by pressing 'O'. Turn the red knob to 'off' position

ANALYTICAL METHOD

Instrumentation

- **ESR Instrument** : JEOL Japan Qband/X Band ESR Spectrometer
Model - JES FA-200E
- **Accessories used** : OD ESR tubes

Standard

Calibrate g-values with standard solid sample of diphenylpicrylhydrazyl (DPPH) taking peak position as $g = 2.0029$.

Instrument Parameters:

Instrument operating sensitivity - 7×10^9 spins/0.1 mT.

Measure ESR spectra at room temperature (298 K).

Typical ESR Q Band parameters keep on changing to enhance the signals like modulation width, the constant parameters are:

Amplitude at constant Frequency	:	9445 MHz
Microwave Power Output:	:	1 mW
Sweep Time:	:	2 min

Procedure:

Insert the sample tube containing the sample with 70%-80% infill in the thoroughly cleaned and dried quartz (Wilmad) 5 mm OD ESR tubes for ESR measurements.

Set the frequency to the resonance frequency of the cavity, which is determined at the time sample is inserted.

Determine the microwave output power necessary for measurement, it varies according to the sample to be measure, and adjustment of it is necessary.

Now amplify the signal and record the data.

Calculation and Report :

Calculate line width ($\Delta H_{1/2}$) and g-factor value (g) using system software and report the same for each sample.