Electron Spin Resonance Spectroscopy

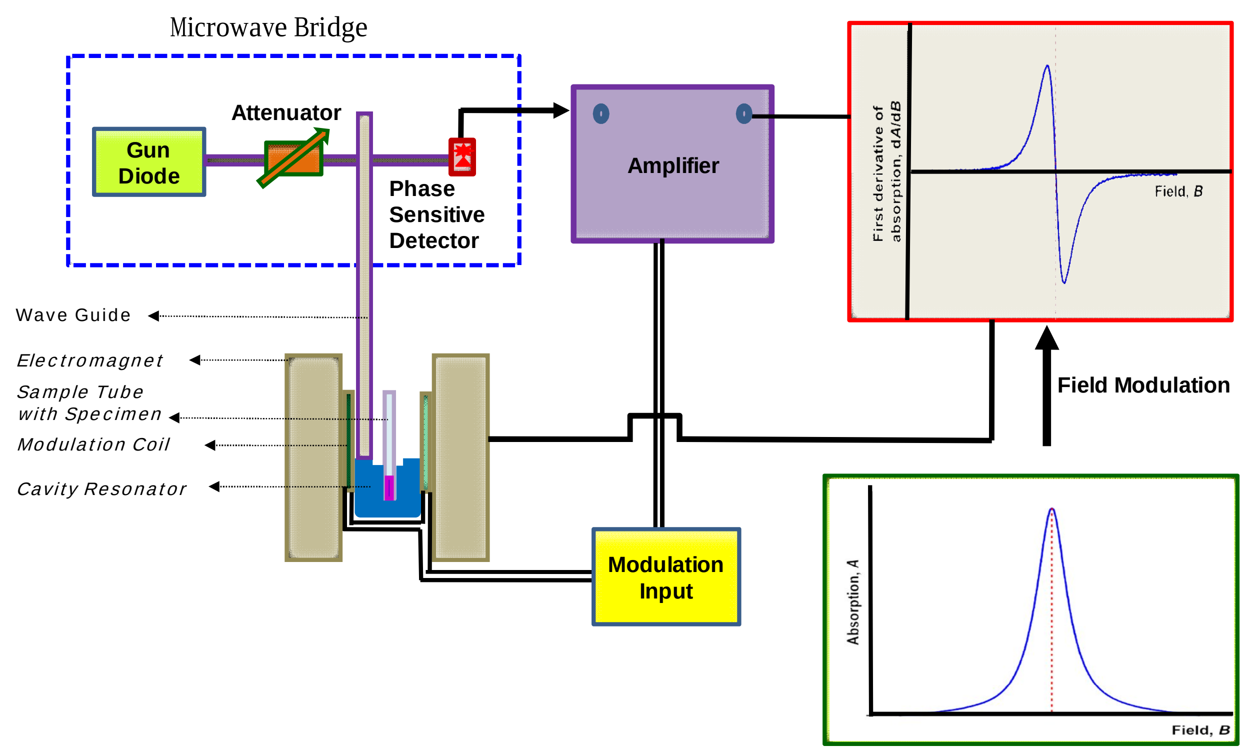
Electron Spin Resonance (ESR) spectroscopy, also referred to as Electron Paramagnetic Resonance (EPR) spectroscopy, is a versatile, nondestructive analytical technique based on the absorption of microwave radiation in presence of an applied field by paramagnetic species.

When any species that contains unpaired electron(s) is subjected to a static magnetic field, the interaction between the magnetic moment of the electron and the applied magnetic field splits the spin energy levels (Zeeman splitting), and transitions between these spin levels are induced by applying suitable microwave radiation perpendicular to the magnetic field. Further interactions will depend on the presence of other factors such as the nuclear spins present in both the origin and other neigbours. The resulting absorption of the microwave radiation is modulated to record the first derivative of the absorption. Conventionally, EPR spectra are recorded as first derivative rather than absorption to improve the resolution. In fact, second derivative operation is also possible when needed to get better resolution of complex splitting patterns.

Description of the Instrument:

The block diagram of JES200 ESR spectrometer available at SAIF, IITM is shown below. This is a continuous wave (CW) ESR spectrometer that employs field modulation and phase sensitive detection. The Microwave Bridge consists of the Gundiode (as source of radiation) and the Detector. Both X-band (8.75 - 9.65 GHz) and Q-band (35.5GHz) frequency units are available. The sample is placed in a resonant cavity which admits microwaves through an iris. The cavity is located in the middle of an electromagnet and helps to amplify the weak signals from the sample. Other components such as an attenuator, field modulator and amplifier are also included to enhance the performance of the instrument. X- Band Electron Nuclear DOuble Resonance (ENDOR) facility is also available to study the nature of hyperfine interactions of very low magnitude, noting that this may be used only after complete understanding of the electronic structure of the molecule.

The ESR parameters that are monitored include the measurement of the g-factor at the centre of the spectrum and the hyperfine splitting due to interaction with nuclei having spin $I \neq 0$. Zero-field splitting (or fine structure) characteristic of transition metal complexes and other species with two or more unpaired electrons ($S \geq 1$) can be observed in solid samples.



Block Diagram of JES FA200 CW ESR Spectrometer

Applications:

1. To elucidate the electronic structure of free radicals and paramagnetic transition metal complexes

2. To study the reaction mechanism of free radicals, photochemical and polymerization reactions.

3. Study the magnetic properties of the materials and its order of orientations

4. To explore the dimensionality of exchange coupling of the magnetically coupled materials.

5. To study conducting properties of the organic conducting polymers.

6. To investigate *the* active sites on the surface of the catalyst.

7. To study the oxidation state and conducting properties of the super conducting materials.

8. To determine the presence of oil under the earth.

9. To investigate the active sites in the metal enzymes

10. To study the structure and activities of hemoglobin and other biological samples.

11. To analyze the hole and electron centers in the semiconductor materials

12. To study the ground state energy levels of the metal cluster complexes

13. To determine the unknown concentration of the metal ion in paramagnetic compounds.

14. To determine the presence of trace amounts of metal ions in the polluted samples.

Further Reading:

1. Wertz, J. E.; Bolton, J. R., Electron Spin Resonance: McGraw-Hill: New York, 1972.

2. Pilbrow, J. R. Transition Ion Electron Paramagnetic Resonance; Clarendon Press: Oxford, U.K., 1990.

3. Mabbs, F. E.; Collison, D.; Electron Paramagnetic Resonance of d Transition Metal Compounds. Studies in Inorganic Chemistry 16. Elsevier Science Publishers; Amsterdam, The Netherlands. 1992.

4. Lund, Anders, Shiotani, Masaru, Shimada, Shigetaka, Principles and Applications of ESR Spectroscopy, Springer, 2011.