

# *Users Manual*

## **ELECTRON SPIN RESONANCE SPECTROMETER** **Model: ESR-104**

Manufactured by

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## PREFACE

The subject of electron paramagnetic resonance has developed enormously over the twenty-eight years of its existence and is being applied to an ever widening range of problems of both fundamental and a practical nature. Inevitably a growing number of research workers require sufficient knowledge of principle and practice to enable them to tackle their particular problems. In this context, a need for an experiment in the field of electron paramagnetic resonance at the postgraduate level is obvious. Unfortunately, the equipment's in this field are very sophisticated and expensive, that have limited its use to only few research laboratories. However, with the introduction of this low cost, easy to operate spectrometer, we sincerely hope that the students would be able to appreciate the basic experimental technique at an M.Sc. level.

Our electron spin resonance spectrometer, based on Zavoisky's technique, has been specially designed for M.Sc. laboratories, keeping in view their requirements and limited resources.

Many students have complained of the difficulty of 'getting into the subject' that is very little in the text books of Solid State Physics and too much in the specialised text books on the 'Electron Paramagnetic Resonance'. To overcome, this difficulty, we have considerably increased the scope of this 'Operation Manual'. It is self contained booklet and has all the material on the electron paramagnetic resonance - background, basic theory, experimental technique in general and about the present set-up in particular, that a student at the postgraduate level is expected to know. In the end, some very useful references also given for the teachers and students, who might like to pursue the subject further.

Finally, and with much pleasure, we would like to express our gratitude to Dr. Kailash Chandra, Director, USIC and Dr. R. Mitra Professor, E. & CE., University Of Roorkee, Roorkee, for some very useful discussions and practical suggestions

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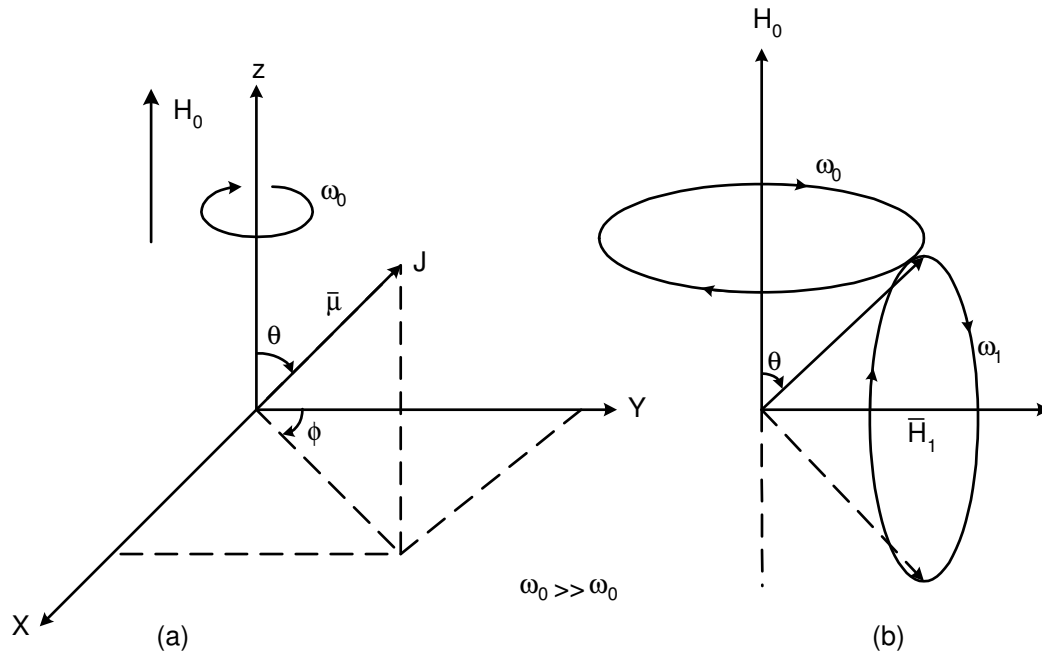


Fig. 1 : Precession of a magnetic moment  $\bar{\mu}$  when placed in a magnetic field  $\bar{H}_0$

- (a). The spin precesses with angular frequency  $\omega_0 = \gamma H_0$ ; the angle  $\theta$  is a constant of the motion.
- (b). In addition to  $\bar{H}_0$  a weak magnetic field  $\bar{H}_1$  is now also applied.  $\bar{H}_1$  is rotating about the z axis with angular frequency  $\omega_0$  and therefore  $\bar{\mu}$  precesses about  $\bar{H}_1$  with angular frequency  $\omega_1 = \gamma H_1$ ;  $\theta$  is not any more conserved.

# CHAPTER 1

## INTRODUCTION

Zavoisky in 1945 performed the earliest magnetic resonance experiments in a solid. He observed strong electron spin resonance absorption in several paramagnetic salts. Applications of electron magnetic spin resonance in solid state physics are of great importance. It is a very sensitive technique and has been applied in many fields. The chief of these are :

- (a) Paramagnetic ions in crystals,
- (b) Unpaired electron in semi-conductors and organic free radicals,
- (c) Colour centres, and radiation damage centres,
- (d) Ferro and anti-ferro magnetic materials.

## ELEMENTARY MAGNETIC RESONANCE

The fundamentals of elementary magnetic resonance may be understood in terms of simple classical concepts. Suppose a particle having a magnetic moment  $\vec{\mu}$  is placed in a uniform magnetic field of intensity  $\vec{H}_0$  (Fig.1a). Then the moment  $\vec{\mu}$  will precess around  $\vec{H}_0$  with an angular Larmor frequency

$$\omega_0 = g \left( \frac{e}{2mc} \right) H_0 \quad (1)$$

$g$  being the Lande' g-factor ( $g=1$  for pure orbital momentum and  $g=2$  for a free electron spin). For the case of anion in a crystal, the behaviour is modified by the environment and the g-factor may differ from the Lande' g-factor. This effective g- factor is known as the spectroscopic splitting factor.

We now introduce an additional weak magnetic field oriented in the xy plane and rotating about the z axis (in the same direction as the "Larmor precessing") with an angular frequency  $\omega_1$ . If the frequency  $\omega_1$  is different from  $\omega_0$  the angle between the field  $\vec{H}_1$  and the magnetic moment  $\vec{\mu}$  will continuously change so that their interaction will average out to zero. If, however,  $\omega_1=\omega_0$  the angle between  $\vec{\mu}$  and  $\vec{H}_1$  is maintained and net interaction is effective (Fig.1b). If we look at the system in a reference frame that is rotating about the z axis with the angular velocity  $\omega_0$  then the spin will appear to make an angle  $\psi=90-\theta$  with  $\vec{H}_1$ , and according to the previous argument will start to precess (in the rotating frame) about  $\vec{H}_1$ .

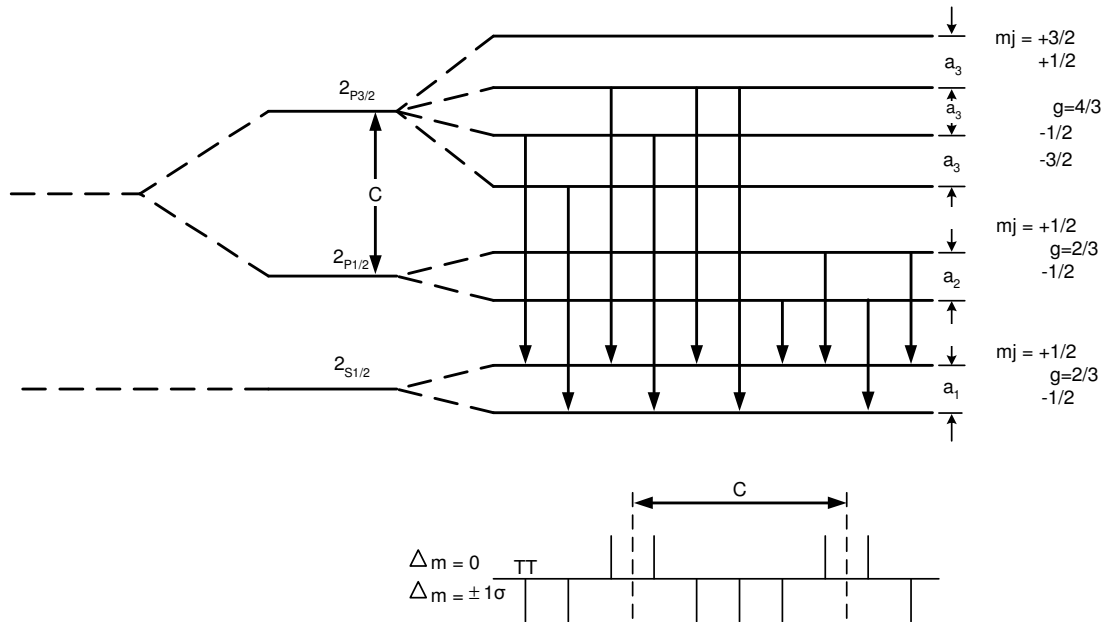


Fig. 2 : Energy levels of a single valence electron atom showing a P state and an S state. Due to the fine structure, the P state is split into a doublet with  $j=2/3$  and  $j = 1/2$ . Further, under the influence of an external magnetic field each of the three levels is split into sublevels as shown in the figure where account has been taken of the magnetic moment of the electron. The magnetic quantum number  $m_i$  for each sublevel is also shown as is the g factor for each level. The arrows indicate the allowed transitions between the initial and final states, and the structure of the line is shown in the lower part of the figure.

This corresponds to a "nutation" and a consequent change of the angle which implies a change in the potential energy of the particle in the magnetic field. The change in  $\theta$  is the classical analogy to a transition between sublevels with different  $m$ . We see that such transitions may take place only if the rotating field has an angular frequency  $\omega_1 = \omega_0$ .

Let us proceed to consider the quantum picture of elementary magnetic resonance. Suppose that the intrinsic angular momentum of the electron  $\bar{S}$  couples with the orbital angular momentum of electron  $\bar{L}$  to give a resultant  $\bar{J}$ . We know, that  $J+1$  magnetic sublevels labelled by the magnetic field  $\bar{H}_0$  by equal energy difference,

$$\Delta E = g\mu_0 H_0$$

between adjacent sublevels, where  $\mu_0$  is the Bohr magnetron and  $g$  is the Lande' factor or  $g$ -factor whose correct quantum mechanical value is

$$g = 1 + \frac{J(J+1) + S(S+1) - L(L+1)}{2J(J+1)}$$

Now, if the particle is subjected to a perturbation by an alternating magnetic field with a frequency  $\nu_1$  such that the quantum  $h\nu_1$  is exactly the same as the difference between the levels,  $\Delta E$  and if the direction of the alternating field is perpendicular to the direction of the static magnetic field, then there will be induced transitions between neighbouring sublevels according to the selection rules  $\Delta m = \pm 1$  for magnetic dipolar radiation.

Therefore, the condition for resonance is

$$\Delta E = g\mu_0 H_0 = h\nu_0 = h\nu_1 \quad (2)$$

Where  $\nu_1$  is the resonance frequency in cycles/sec. This requirement is identical with the classical condition  $\omega_1 = \omega_0$ .

In atomic spectroscopy, we do not observe the transitions between sublevels with different  $m$  (labelled a, a and selection rules  $\Delta L = \pm 1$ . Instead the splitting of a level is observed through small change in frequency of the radiation emitted in the transition between widely distant levels (Fig.2). It is clear that, if we could directly measure the frequency corresponding to a transition between the sublevels of the same state, a much more precise knowledge of the energy splitting would be obtained.

## ELECTRON SPIN RESONANCE IN SOLIDS

Let us proceed from the treatment of an isolated magnetic particle to a macroscopic body. The behaviour of a paramagnetic substance in a magnetic field will depend on the interaction of the particles with one another and with the diamagnetic particles. There are mainly two types of interactions.

(a) **Spin - Spin:** In which the spin interacts with a neighbouring spin but the total energy of the spin system remains constant.

(b) **Spin - Lattice:** In which the electron spin interact with entire solid or liquid, transforming energy from the spin system to the lattice which act as a thermal reservoir. As a matter of fact it is the spin-lattice interaction that makes possible the observation of energy absorption from the radio- frequency field when the resonance frequency is reached.

To understand this last statement, consider a paramagnetic substance in a magnetic field  $\overline{H_0}$  and say the equilibrium state has been reached. The population of individual energy levels will be determined by the Boltzmann distribution  $e^{-g\mu_0 H_0 m / kT}$  where  $m$  is the magnetic quantum number. It can be seen that the population of the lower energy levels are greater than those of the upper levels and, therefore when a periodic magnetic field with a resonance frequency is switched on; the number of induced radiation events will be more and as a result the substance will absorb energy from the radio-frequency field. Thus, two opposing processes take place in ESR. The radio frequency field tends to equalise the population of various levels and the spin lattice interaction tends to restore the Boltzmann distribution by conversion of the energy absorbed from the radio-frequency field into heat.

## RELAXATION AND SATURATION

In the preceding para, we see the mechanism through which the electron return from an excited state to the ground state or relax back to the ground state. This process in the field of magnetic resonance's (ESR, NMR, NQR ETC.) is know as relaxation and the time taken by the process is called the relaxation time. This complete process may be considered as two state process (provided the spin-spin interactions are much stronger than the spin-lattice interaction). First, the energy is absorbed from the radio frequency magnetic field and the equilibrium is established inside the 'spin system'. The time taken by this process is known as the spin-spin relaxation time and is a measure of the rate at which magnetic energy can be distributed within the spin system though total energy is conserved. Secondly, an exchange of energy occurs



between the spin system and the lattice. The time taken is known as the **spin lattice relaxation time** and is a measure of the rate of transfer of energy from the spin system to the lattice.

In optical spectroscopy of the relaxation time is usually very short ( $\sim 10^{-8}$  sec) so that the relaxation time does not impede the absorption rate. In radio frequency, on the other hand, typical relaxation times are in milliseconds or longer and the spin do not have time to relax if the energy is supplied at a faster rate. This situation is called the 'Saturation State'. In other words, no additional energy is absorbed, if the radio- frequency field power is increased beyond certain level.

## LINE WIDTH AND THE SPIN-SPIN AND SPIN-LATTICE INTERACTIONS

The effect of the spin-spin interaction is to slightly shift the exact position of energy level of any individual spin in the external field. This energy shift clearly depend on the relative orientation and distance of the spin and thus is different for each spin, resulting in apparent broadening of the energy level. Another way of thinking of the spin-spin interaction is that one electron spin produce a local magnetic field at the position of another spin. Thus, the width of absorption line due to spin-spin interaction may be estimated as  $\frac{1}{T'}$ , where  $T'$  is spin-spin relaxation time.

If the spin-lattice interactions are not weak the spin lattice relaxation time  $T$  will also be introduced. Let us consider the probability of a transition of an individual paramagnetic particle from one magnetic level to another under the influence of thermal motion. If the probability per second equal  $A$ ,  $T \sim \frac{1}{A}$  and the absorption line width would be of the order of  $\frac{1}{T}$ . In general case, however, the absorption line width may be estimated as  $\frac{1}{T} + \frac{1}{T'}$ .

Thus, we see that from the width of absorption line it is possible to measure the relaxation time. In fact most of the research in this field involve the study of relaxation phenomena which in turn provide information about internal interactions in solids and liquids.

The position and number of lines of paramagnetic resonance absorption also depend on the internal interactions.

## ELECTRON PARAMAGNETIC RESONANCE AND SPECTROSCOPY

Paramagnetic resonance is an integral part of spectroscopy, as it provides a means for determining the position of the energy levels of magnetic particles. It is interesting to consider the peculiar features of Paramagnetic resonance in comparison with optical spectroscopy.

1. Let us first note that the frequencies used in magnetic resonance experiments range from  $10^9$  to  $10^{11}$  cps. These frequencies situated below the limits of the infrared part of the spectrum, allow highly accurate investigation of energy level splitting so small that they are inaccessible or almost inaccessible by optical methods.
2. The probability of spontaneous transition in the radio-frequency region is very small, since this probability is proportional to  $\nu^3$ . Therefore, in paramagnetic resonance studies one is forced to deal only with induced absorption and emission.
3. While in the great majority of cases optical spectra arise from electric dipole transitions between energy levels, the lines of paramagnetic resonance absorption arise exclusively from magnetic dipole transitions. Consequently, the Einstein coefficients for induced absorption and emission will, in the case of paramagnetic resonance, be smaller by roughly four orders of magnitude.
4. As a result, the paramagnetic resonance effect is exceedingly small; if it can be observed at all is due to the high sensitivity of electronic methods of detection and the enormous number of photons coming into play (1mW corresponds to  $n \approx 10^{20}$  photons per sec at a frequency of  $10^{10}$  cps).
5. In the optical frequency region the line width is always very small in comparison with the fundamental frequency. In paramagnetic resonance the relation between these quantities becomes quite different, since the interactions causing a broadening of the lines can be of the same order of magnitude as the energy splitting which determines the resonance frequency. Because of this the width of paramagnetic resonance lines is often comparable to the fundamental frequency and can be measured with great accuracy. This opens up wide possibilities for investigation of different types of interactions in paramagnetic substances by means of analysis of the shape and width of a paramagnetic resonance line and of the character of its dependence upon various factors.
6. The most important factors determining the line width are magnetic dipole interactions, exchange forces, local electrical fields created by neighbouring magnetic particles, and

finally, thermal motion; the natural line widths of radio- frequency spectra are completely negligible.

7. In contrast with optical experiments, in radio-frequency spectroscopy it is customary to use radiation which is so monochromatic that the generated band of frequencies is incomparably narrower than the absorption line width.
8. Paramagnetic resonance spectra are not studied by varying the frequency of the incident radiation, but by varying the characteristic frequencies of the absorbing systems. This is achieved by varying the static magnetic field.

## CHAPTER 2

### GENERAL CONSIDERATIONS

For a free electron, by substituting the proper values of constants :  $g=2.00$ ,  $\mu_0=0.927 \times 10^{-20}$  erg / Gauss and  $h=6.625 \times 10^{-27}$  erg.sec. in equation (2), we get,

$$\frac{\nu_0}{H_0} = 2.8 \text{ MHz/Gauss}$$

That is ESR can be observed at radio frequencies in a magnetic field of few gauss or otherwise in the microwave region in a magnetic field of a few kilogauss. The latter alternate has many distinct advantages :

- (a) For each transition the absorbed energy is much larger, and thus the signal-to-noise ratio is much improved.
- (b) A high magnetic field is used, thus providing separation between levels that are intrinsically wide and would remain partially overlapped at low fields.

Because of these advantages, ESR in microwave region is preferred for research purpose, though, it is very sophisticated and expensive.

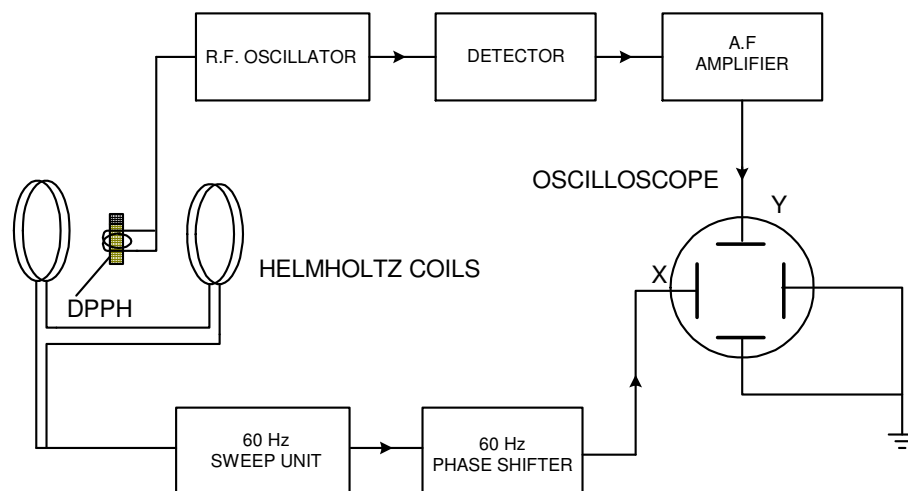
The former is preferable, where simplicity, easy operation and cost factor are the main considerations and no high resolution is required **ideal for postgraduate level experiments.**

### EXPERIMENTAL TECHNIQUE

In the radio frequency region, two types of methods are chiefly used:

1. The method of reaction on the generator
2. The method based on a determination of the change in a load factor of the oscillatory circuit due to paramagnetic loss.

The latter method has been used here. The sample under investigation is placed in an induction coil, which is the component of the tank circuit of the oscillator (generator). This is the Zavoisky's technique. It is based on the fact that under certain conditions such as absorption of power from generator, the watt load ( $\Delta w$ ) on the generator changes. This change of  $\Delta w$  is proportional to the change in base current  $\Delta I_b$  or collector current  $\Delta I_c$  of the generator. The proportionality, however, holds only the power dissipated by the sample due to paramagnetic absorption is small in comparison to the total losses in the circuit. This change in  $\Delta I_c$  is



**FIG. 3 Block diagram of the ESR set**



detected with the conventional circuits. To make the detection simple and more sensitive, the magnetic field and hence the Larmor frequency of the sample is modulated with a low frequency field 60Hz in the present set-up.

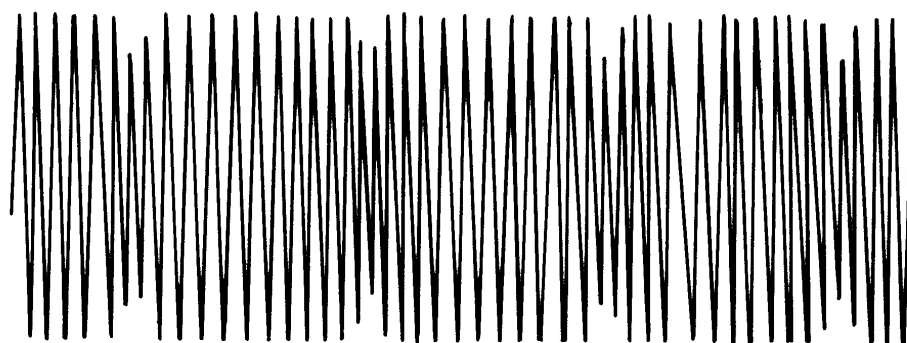
## DESCRIPTION OF THE ESR SPECTROMETER

A block diagram of the ESR Spectrometer is given in Fig. 3 and line-sketch on the next page.

**1. Basic Circuit:** The first stage of the ESR circuit consists of a critically adjusted (marginal) radio frequency oscillator having a frequency range of approximately 10–17MHz. A marginal oscillator is required here so that the slightest increase in its load decreases the amplitude of oscillation to an appreciable extent. The sample is kept inside the tank coil of this oscillator, which in turn, is placed in the 60Hz magnetic field, generated by the Helmholtz coils. At resonance, i.e. when the frequency of oscillation equal to the Larmor's frequency of the sample, the oscillator amplitude registers a dip due to the absorption of power by the sample. This obviously, occurs periodically - four times in each complete cycle of the Helmholtz coils supply voltage. The result is in amplitude modulated carrier (Fig. 4A) which is then detected using a diode detector and amplified by a chain of three low noise, high gain audio - frequency amplifiers of excellent stability. A sensitivity control is provided in the amplifier to suit the input requirement of any oscilloscope.

**2. Phase Shifter:** In order to make it possible to use an ordinary displaying type oscilloscope, instead of a measuring oscilloscope which preserve the phase between X and Y plates signals, a phase shifter is provided. This can compensate the phase difference which is introduced in the amplification stage of the ordinary oscilloscope.

The circuit diagram of the phase shifter is shown in Fig. 4B. The primary of the transformer is fed from the 110V, 60Hz mains and the secondary is centre tapped developing  $V_1$ -0- $V_1$  (say). The operation of the circuit may be explained with the help of the vector diagram shown in Fig. (4B). The vectors OA and BO represent the voltage developed in the secondary, in phase and magnitude. The current flowing in the circuit ADB leads the voltage vector BA due to the presence of capacitor C and is shown in the diagram as I. Voltage developed across resistance R, i.e.  $V_R$  is in phase with the current I, and the voltage across capacitor  $V_C$  is  $90^\circ$  (lag) out of phase with the current. The vector sum of  $V_C$  and  $V_R$  is equal to  $2V_1$ . These are also plotted in the diagram. It is clear from the diagram that as R is varied,  $V_R$  will change and the point D will trace a semicircle,



At A



At B

FIG. 4 (A)

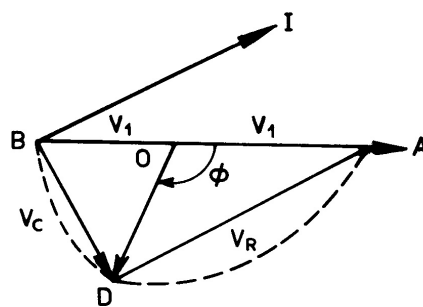
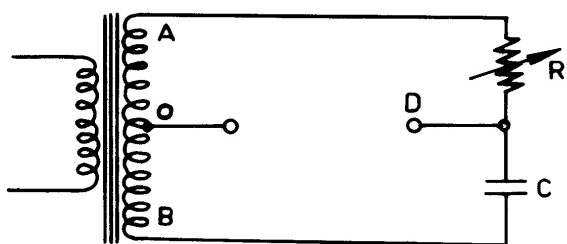


FIG. 4 (B)

shown dotted. The vector OD, or the voltage across points O and D, will, therefore, have a constant magnitude equal to  $V_1$  and its phase, variable from 0 to  $180^\circ$ . This is the voltage which is fed to the X-amplifier of the oscilloscope to correct for any phase change which might have taken place in the rest of the circuit.

**3. 60 Hz Sweep Unit :** For modulation with a low frequency magnetic field, a 60 Hz current flows through the helmholtz coils. As the resonance in this frequency range occurs at low magnetic fields, no static D.C. magnetic field is required.

#### **4. Power Supplies :**

a) D.C. Power Supply : The ESR circuit requires a highly stabilised almost ripple free voltage. It is obtained using an Integrated circuit regulator. The specification of the D.C. power supply are :

- (i) *D.C. Voltage* : 20 V
- (ii) *Load regulation* : 0.3%
- (iii) *Line regulation* : 0.01%
- (iv) *Ripple* : < 3 mV
- (v) *Long Term stability* : 0.1% per 1000 hrs.

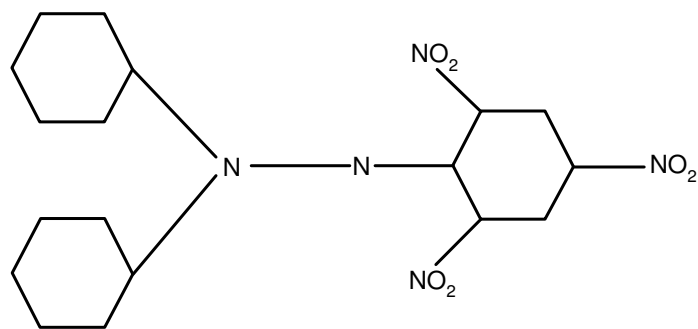
b) Helmholtz Coils Power Supply : The Helmholtz coils power supply consists of a step down transformer (110 to 35 V AC) separate winding on the mains transformer, a potentiometer (12 - 15 W) and a moving coil rectifier type meter. The output is taken from the two terminals provided on the panel.

**5. Helmholtz Coils :** There are two coils exactly alike and parallel to each other, so connected that current passes through them in the same direction. The two coils increase the uniformity of the field near the centre.

<i>Number of turns</i>	:	500 in each coil
<i>Diameter of the Windings</i>	:	15.4 cm
<i>Separation of the coil</i>	:	7.7 cm

In the centre of the coils, an attachment is provided to keep the sample in place and to minimise shocks and vibrations.





**Fig.: Chemical structure of DPPH (2,2-Diphenyl-1-picrylhydrazyl, (free radical, 95%))**

**6. Test Sample:** A test sample, Diphenyl Picryl Hydrazyl (DPPH) is placed in a plastic tube, which itself is in the induction coils. This increases the filling factor to the maximum. DPPH is a free radical and widely used as a standard for ESR measurements.

**7. R.F. Oscillator:** It is a transistorised radio frequency Oscillator suitable for the determination of resonance frequency. The power to it is provided from the ESR Spectrometer through a cable.

*Frequency range* : 9.5 MHz - 18.0 MHz

*Accuracy* : Better than 0.5%

**8. Controls & Terminals :** (Please refer to Fig. No. 4)

- (1) *Mains* : To switch 'ON' or 'OFF' the ESR Spectrometer.
- (2) *Sensitivity* : To adjust the amplitude of the output signal.
- (3) *Phase* : To adjust the phase between X and Y plates signals.
- (4) *Current* : To control current in Helmholtz coils.
- (5) *'H' Coils* : Terminals and switch for Helmholtz coils.
- (6) *Frequency* : To adjust the frequency of the Oscillator.
- (7) *X,Y,E* : For X, Y and Earth terminals of the Oscilloscope.

**9. Oscilloscope:** As the Oscilloscope is not supplied with the spectrometer, it is presumed that the worker is already familiar with the control knobs and functioning of the Oscilloscope in question.

Any Oscilloscope, normally available in the laboratory of the following specifications or better, will be quite suitable for the observation of ESR resonance :

*Screen diameter* : 12.5 cm

*Vertical amplifier sensitivity* : 50 mV/cm

## INSTALLATION

The instrument is thoroughly tested and checked before it is despatched from the factory. Please check for all the components from the list given in the delivery challan.

**It is strongly recommended to read the description of the ESR Spectrometer very carefully and get familiar with various knobs and terminals given in the preceding section.**

Now for installation proceed as follows :

- 1) Connect the Coaxial cable of the induction coil to the Oscillator through the socket.
- 2) Connect the Helmholtz coils to the terminal marked 'H' coil.
- 3) Connect the terminal marked X,Y, and E on the ESR Spectrometer to the X plate, Y plate input and earth of the Oscilloscope respectively and switch the Oscilloscope.
- 4) Connect the spectrometer with the AC mains 100 to 110 Volts, 60 Hz and switch - on the power. Ensure that the equipment is properly earthed.

## **OPERATION**

- (1) Switch on 'H COIL' power and adjust the current at 150 mA.
- (2) Set the front panel controls of ESR Spectrometer as follows

*Frequency* : Centred

*Sensitivity* : Fully clock-wise

*Phase* : Centred

- (3) Observe four peaks on the Screen of CRO. Now adjust the FREQUENCY and SENSITIVITY knobs of the Spectrometer and Sensitivity of the CRO to obtain the best results (i.e. sharp peaks and good signal to noise ratio).
- (4) Adjust the PHASE knob to coincide the two peaks with the other two as far as possible.

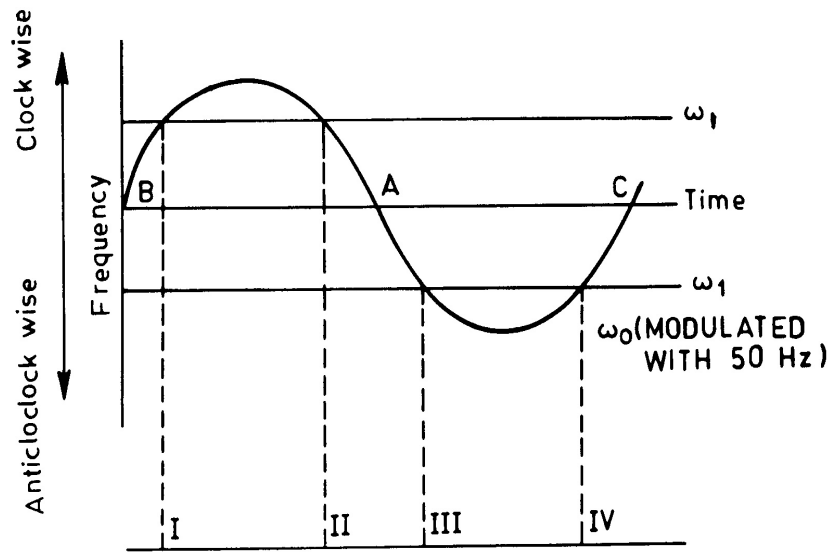


Fig. 5: The radio frequency field is linear polarised, which can be regarded as two circularly polarised fields of opposite direction (say clockwise and anti-clockwise). This is due to a change in direction (clockwise and anti-clockwise) of the magnetic field  $H_0$ . Resonance occurs when the two frequencies ( $\omega_1$  and  $\omega_0$ ) become equal in magnitude as well as in direction.

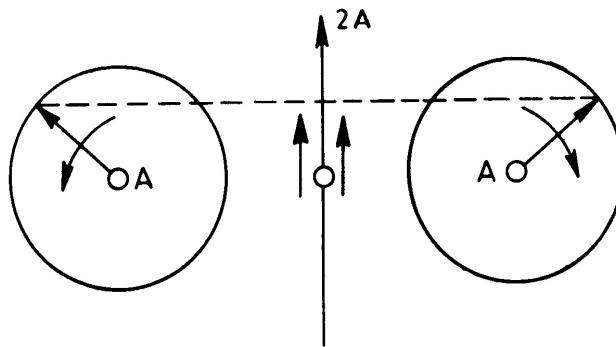


Fig. 6 : A linearly oscillating field of frequency  $\omega$  is equivalent to two fields rotating in opposite directions with the same frequency  $\omega$ .

## CHAPTER 3

### ORIGIN OF FOUR PEAKS

The observed peaks are in fact absorption dips, because the sample absorb power from the induction coil, reasons explained in Chapter 1. The reason for getting peaks is due to odd number of amplifying stages in the circuitry.

The spin precesses with Larmor's frequency ( $\omega_0 = \frac{eH_0}{2mc}$ ) and hence varies in magnitude and direction due to variation of magnetic field  $\overline{H_0}$  which is due to an alternating current in the Helmholtz coils. Now if the radio frequency field,  $\omega_1$  falls in the range of  $\omega_0$  the resonance occurs. The positions of the four peaks can be understood with Fig. (3).

If the X plate signal (60Hz) and Y plate signal (ESR output) are in phase the I and II peaks and III and IV peaks will coincide. The coincidence of peaks on the x-scale needs to be calibrated for magnetic field measurements. The coincidence ensures that the magnetic field is zero at the centre and has the peak values at the two ends. Complete merger of the peaks on y- scale may not occur due to many reasons such as 60Hz pick-ups, ripples in the power supply etc. Though, every effort has been made to minimise these factors but the large amplification ( $\cong 4000$ ) in the circuitry make them substantial. However, any non-coincidence on the y-scale is immaterial as neither any measurement of the y-scale is involved in the calculation of g- factor nor any measurement is made on it.

### DETERMINATION OF MAGNETIC FIELD

To calibrate the X-plate of CRO in terms of magnetic field proceed as follows :

1. Adjust the X amplifier (CRO) to obtain the maximum X deflection (say 'P' divisions).
2. Read current flowing in Helmholtz coils and calculate the magnetic field.

$$H = \frac{32 \pi n}{10 \sqrt{125} \cdot a} \cdot I$$

Where 'n' is the number of turns in each coil

'a' is the radius of the coils

'I' is the current (in amp) flowing through the coils.

This is the root mean square (rms) field. The peak to peak field will be  $2\sqrt{2}.H$  and represents 'P' division of the CRO X plate. The zero field is at the middle point.

3. Measure the positions of the two peaks. These should be at equal distances from the middle point (say "Q' division). The magnetic field at the resonance is thus

$$H_0 = \frac{2\sqrt{2}.H}{P}.Q \text{ gauss}$$

## DETERMINATION OF RESONANCE FREQUENCY

Keep the above set-up as it is. Power up the calibrated R.F. Oscillator by inserting its jack in "-9V socket" in ESR and bring it in the vicinity of the sample say at a distance of 1 feet. Tune the R.F. Oscillator to obtain beats (this will be seen on the CRO very easily) and read the frequency on the Oscillator dial. This will be the desired resonance frequency. If one observes the beats more carefully by varying the R.F. Oscillator frequency slowly, he will obtain high frequency beats twice with a zero beat in between. The frequency corresponding to the zero beat is the exact frequency. The inaccuracy involved in the frequency measurement will be exceedingly small even if the zero beat position could not be detected.

## CALCULATIONS

From equation (2)

$$h\nu_1 = g \mu_0 H$$

$$\text{or} \quad g = \frac{h \nu_1}{H_0 \mu_0}$$

Substitute the measured values of  $H_0$  and  $\nu_1$  universal constants  $h=6.625 \times 10^{-27}$  erg.sec and  $\mu_0=0.927 \times 10^{-20}$  erg/gauss to get the g factor.

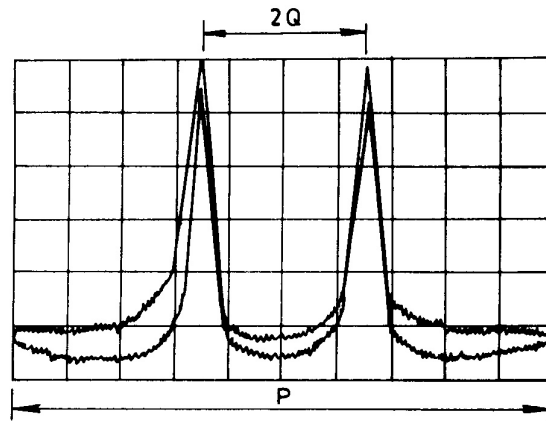


FIG. 7 LINE SKETCH OF THE RESONANCE PEAKS

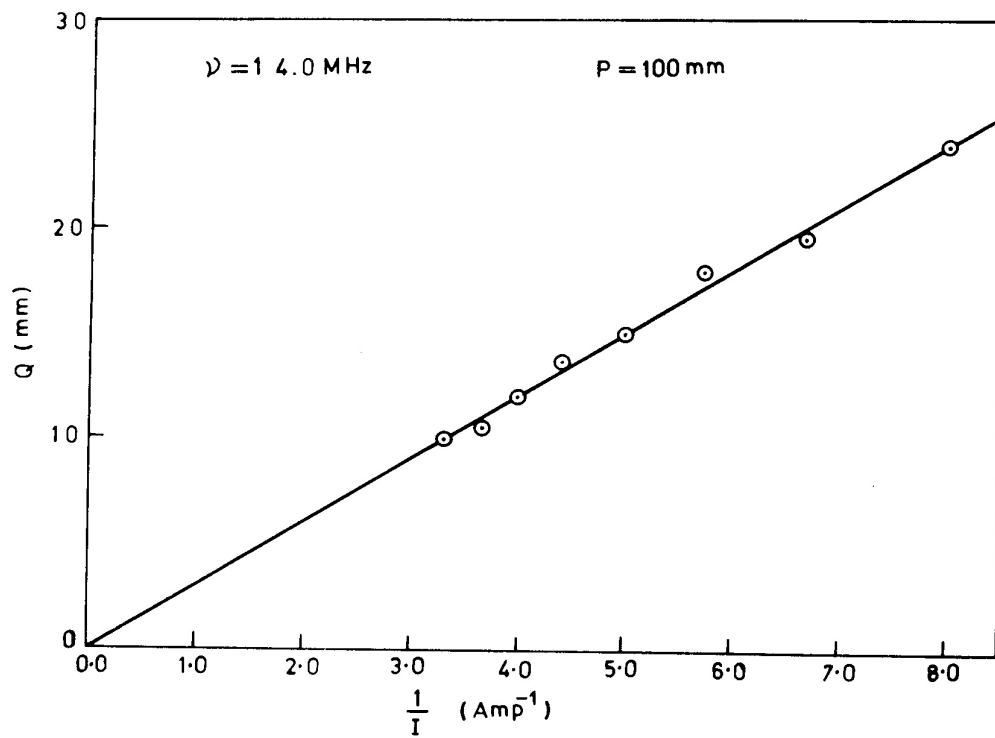


FIG. 8 GRAPH BETWEEN  $\frac{1}{I}$  VS  $Q$

## APPENDIX

### SAMPLE CALCULATIONS

Magnetic field at the centre of a Helmholtz coil is :

$$H_0 = \frac{32 \pi n}{10 \sqrt{125}} . I = KI \text{ gauss}$$

Where I is the current in amperes and other symbols have their usual meaning. Since the current measured is rms the magnetic field is also rms. The peak to peak magnetic field will be :

$$H_{PP} = 2\sqrt{2} H$$

The  $H_{PP}$  corresponds to the total X-deflection on the oscilloscope with zero field at the centre. Substituting the values  $a=7.7\text{cm}$ ;  $n=500$ , we obtain

$$H_{PP}=165.25 \text{ gauss/amp}$$

Let us take a typical observation (Fig. 7). The measured values are :

$$\nu_0=14.00 \text{ MHz}; I=200 \text{ mA}; P=100 \text{ mm and } 2Q=30 \text{ mm}$$

Since P corresponds to  $H_{PP}$ , the magnetic field per mm of the X-deflection will be  $H_{PP}/P$  and hence the magnetic field for either resonance is

$$H_0 = \frac{H_{PP}}{P} . Q = \frac{165.25 \times 0.2 \times 15}{100} = 4.96 \text{ gauss}$$

Substituting the values in the relation

$$h\nu_0=g\mu_0 H$$

$$g = \frac{h \nu_0}{\mu_0 H_0} = \frac{6.625 \times 10^{-27}}{0.927 \times 10^{-20}} \times \frac{14.00 \times 10^6}{4.96} = 2.04$$



## EXPERIMENTAL PROCEDURE

1. Increase the horizontal sensitivity of the Oscilloscope to the maximum **within the linear range**.
2. Obtain the best possible resonance peaks by varying the frequency, detection level and vertical sensitivity of the oscilloscope, keeping the current at 150 mA (say).
3. Keep the frequency fixed but vary the current flowing through the coils and measure the corresponding horizontal separation between the two peaks ( $2Q$ ) after adjusting the phase. Take five to six sets of observations.
4. Draw a graph in  $1/T$  Vs  $Q$  which should be a straight line. Calculate the g-factor using the  $QI$  value from the graph.
5. Repeat the experiment with different frequency.

The reason for drawing graph between  $1/T$  and  $Q$  with fixed  $I$  is that the measurements of  $I$  and  $Q$  are likely to contain some random errors.

## PRECAUTIONS

1. Experiment should be set up at a quite place free from mechanical and electrical disturbances.
2. Y - output from the ESR Spectrometer should be taken through shielded cable to minimise external pick-ups.
3. X and Y plates sensitivities of the Oscilloscope should be adjusted such that they should be in the linear range only.
4. Since the Helmholtz current is unstabilised, care should be taken that it should remain constant during the observation.
5. High currents (  $\sim 200$  mA ) should not be allowed to flow through the Helmholtz coils for an extended period. This will unnecessary heat the coils which may get damaged after some time.
6. If the peaks do not coincide on the x-scale, check the sinusoidal wave form of the mains voltage which may be distorted due to overloading of the main line due to other heavy gadgets working on the same line.
7. Do not use AC Stabiliser as it is likely to distort the sinusoidal wave form. If necessary use the variac.

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