User's Manual

NUCLEAR MAGNETIC RESONANCE SPECTROMETER
Model: NMR-01
(Rev : 01/06/2016)

Manufactured by:
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CE

ISO 9001:2015
Certified Company

CONTENTS

COPYRIGHT AND WARRANTY

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COPYRIGHT NOTICE

LIMITED WARRANTY

SES Instruments Pvt. Ltd warrants this product to be free from defects in materials and workmanship for a period of one year from the date of shipment to the customer. SES Instruments Pvt. Ltd will repair or replace, at its option, any part of the product which is deemed to be defective in material or workmanship. This warranty does not cover damage to the product caused by abuse or improper use. Determination of whether a product failure is the result of manufacturing defect or improper use by the customer shall be made solely by SES Instruments Pvt. Ltd. Responsibility for the return of equipment for warranty repair belongs to the customer. Equipment must be properly packed to prevent damage and shipped postage or freight prepaid. (Damage caused by improper packaging of the equipment for return shipment will not be covered by the warranty). Shipping costs for returning the equipment, after repair, will be paid by SES Instruments Pvt. Ltd.

EQUIPMENT RETURN

Should this product have to be returned to SES Instruments Pvt. Ltd, for whatever reason, notify SES Instruments Pvt. Ltd BEFORE returning the product. Upon notification, the return authorization and shipping instructions will be promptly issued.

Note: No equipment will be accepted for return without an authorization.

When returning equipment for repair, the units must be packed properly. Carriers will not accept responsibility for damage by improper packing. To be certain the unit will not be damaged in shipment, observe the following rules:

1. The carton must be strong enough for the item shipped.
2. Make certain there is at least two inches of packing material between any point on the apparatus and the inside walls of the carton.
3. Make certain that the packing material can not displace in the box, or get compressed, thus letting the instrument come in contact with the edge of the box.

SAFETY INFORMATION

This Section addresses safety considerations and describes symbols that may appear on the Instrument or in the manual.

A **Warning** Statement identifies conditions or practices that could result in injury or death. A **Caution** statement identifies conditions or practices that could result in damage to the Instrument or equipment to which it is connected.

 **Warning**

> **To avoid electric shock, personal injury, or death, carefully read the information in Table-1, "Safety Information," before attempting to install, use, or service the Instrument.**

GENERAL SAFETY SUMMARY

This equipment is Class 1 equipment tested in accordance with the European Standard publication EN 61010-1.

This manual contains information and warnings that must be observed to keep the Instrument in a safe condition and ensure safe operation.

To use the Instrument correctly and safely, read and follow the precautions in Table 1 and follow all safety instructions or warnings given throughout this manual that relate to specific measurement functions. In addition, follow all generally accepted safety practices and procedures required when working with and around electricity.

SYMBOLS

Table 2 lists safety and electrical symbols that appear on the Instrument or in this manual.

Table 2. Safety and Electrical Symbols

Symbols	Description	Symbols	Description
	Risk of danger. Important information. See Manual.		Earth ground
	Hazardous voltage. Voltage >30Vdc or ac peak might be present.		Potentially hazardous voltage
	Static awareness. Static discharge can damage parts.		Do not dispose of this product as unsorted municipal waste. Contact SES or a qualified recycle for disposal.

Table 1. Safety Information

⚠ ⚠ **Warning**
To avoid possible electric shock, personal injury, or death, read the following before using the Instrument:
• **Use the Instrument only as specified in this manual, or the protection provided by the Instrument might be impaired.**
• **Do not use the Instrument in wet environments**
• **Inspect the Instrument in wet environments.**
• **Inspect the Instrument before using it. Do not use the Instrument if it appears damaged.**
• **Inspect the connecting lead before use. Do not use them if insulation is damaged or metal is exposed. Check the connecting leads for continuity. Replace damaged connecting leads before using the Instrument.**
• **Whenever it is likely that safety protection has been impaired, make the Instrument inoperative and secure it against any unintended operation.**
• **Have the Instrument serviced only by qualified service personnel.**
• **Always use the power cord and connector appropriate for the voltage and outlet of he country or location in which you are working.**
• **Never remove the cover or open the case of the Instrument before without first removing it from the main power source.**
• **Never operate the Instrument with the cover removed or the case open.**
• **Use only the replacement fuses specified by the manual.**
• **Do not operate the Instrument around explosive gas, vapor or dust.**
• **When servicing the Instrument, use only specified replacement parts.**
• **The equipment can remain Switched on continuously for five hours**
• **The equipment must remain Switched off for at least fifteen minutes before being switched on again.**
• **The equipment is only for the intended use**
• **Use the equipment only as specified in this manual.**

Unpacking and Inspecting the Instrument

Every care is taken in the choice of packing material to ensure that your Instrument will reach you in perfect condition. If the Instrument has been subject to excessive handling in transit, there may be visible external damage to the shipping container and packing material for the carrier's inspection.

Carefully unpack the Instrument from its shipping container and inspect the contents for damaged or missing items. If the Instrument appears damaged or something is missing, contacts the carrier and SES immediately. Save the container and packing material in case you have to return the Instrument.

Storing and Shipping the Instrument

To prepare the Instrument for storage or shipping, if possible, use the original shipping container along with thermo coal corners, as it provides shock isolation for normal handling operations. If the original shipping container is not available, use any good cardboard box which is at least 2-3 inches bigger than the instrument on all sides, with cushioning material (thermo coal or Styrofoam etc) that fills the space between the Instrument and the side of this box.

To store the Instrument, place the box under cover in a location that complies with the storage environment specification described in the "Environment Sections" below.

Environment

Temperature

Operating …………………………………. 0°C to 50°C

Storage …………………………………… 40°C to 70°C

Warm Up …………………………………15 min to full uncertainty specification

Relatively Humidity (non-condensing)

Operating ………………………………….Uncontrolled (<10°C)
<90 % (10°C to 30°C)
<75 % (30°C to 40°C)
<45 % (40°C to 50°C)

Storage………………………………….. -10°C to 60°C <95 %

Power Considerations

The Instrument operates on varying power distribution standards found throughout the world and must be set up to operate on the line voltage that will power it. The Instrument is packed ready for use with a line voltage determined at the time of ordering.

Replacing the Fuses

The Instrument uses one fuse to protect the line-power input and two fuses to protect current-measurement inputs.

Line-Power Fuse

The Instrument has a line-power fuse in series with the power supply. Table 3 indicates the proper fuse for each of the four line-voltage selections. The line-power fuse is accessed through the real panel.

1. Unplug the power cord.
2. Rotate the fuse holder cap to the right until the fuse POPS out.
3. Remove the fuse and replace it with a fuse of an appropriate rating for the selected line-power voltage. See Table 2.

Warning

To avoid electric shock or fire, do not use makeshift fuses or short-circuit the fuse holder.

Table 2. Line Voltage to Fuse Rating

Line Voltage Selection	Fuse Rating
220/ 240 V	1A, 250V (Slow blow)
100/ 120 V	2A, 250V (Slow blow)

Connecting to Line Power

Warning

To avoid shock hazard, connect the factory supplies three conductor line power cord to a properly grounded power outlet. Do not use a two-conductor adapter or extension cord, as this will break the protective ground connection. If a two conductor power cord must be used, a protective grounding wire must be connected between the ground terminal and earth ground before connecting the power cord or operating the Instrument.

1. Verify that the Line voltage is set to the correct setting.

2. Verify that the correct fuse for the line voltage is installed.

3. Connect the power cord to a properly grounded three-prong outlet. See Figure 1 for line-power cord types available from SES. Refer to Table 3 for description of the line-power cords.

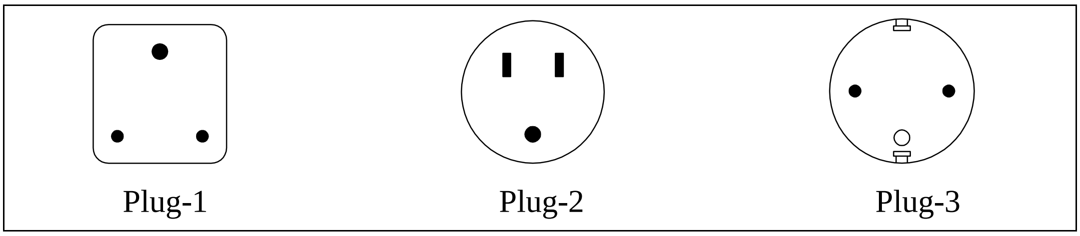


Figure 1. Line-Power Cord Types Available from SES

Table 3. Line-Power Cord Types Available from SES

Type	Voltage/Current	SES Model Number
India	240 V/ 5 A	Plug-1
North America	120 V/15 A	Plug-2
Universal Euro	220 V/16 A	Plug-3

Turning Power On

The On-Off switch on the front panel when points towards “ON” signs, indicates that the equipment has been switched on.

Cleaning the Instrument

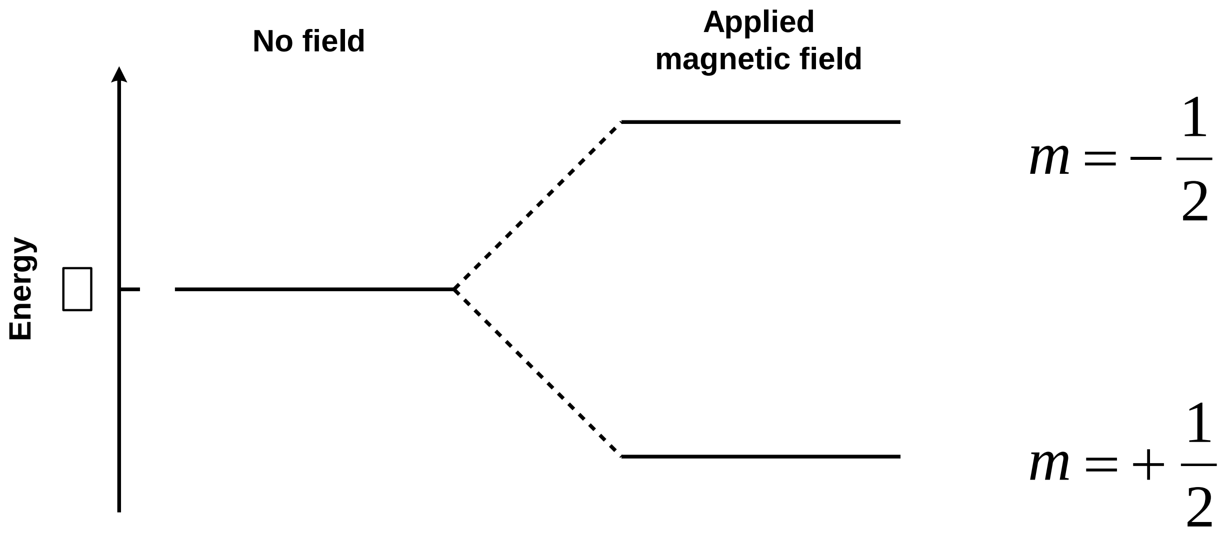
⚠ ⚠ **Warning**

To avoid electric shock or damage to the Instrument, never get water inside the Instrument.

⚠ **Caution**

To avoid damaging the Instrument’s housing, do not apply solvents to the Instrument.

If the Instrument requires cleaning, wipe it down with a cloth that is lightly dampened with water or a mild detergent. Do not use aromatic hydrocarbons, alcohol, chlorinated solvents, or methanol-based fluids when wiping down the Instrument.



Energy levels for a nucleus with spin quantum number 1/2

Fig. 1

NUCLEAR MAGNETIC RESONANCE

(I) THEORY

Nuclear Magnetic Resonance (NMR) is a powerful analytical tool. It was discovered by Bloch and Purcell in 1945. NMR spectroscopy is nowadays an important standard method in chemistry and biology. In medical applications it is known as magnetic resonance imaging (MRI) and supplements other scanning techniques.

It is based on the magnetic properties of the atomic nucleus. A nucleus which possesses a nuclear spin I has a magnetic moment μ given by

$$\vec{\mu} = g\,\mu_n\,\vec{I}\;. \qquad \text{.... (1)}$$

Here μ_n is nuclear magnetron and g is the nuclear *g*-factor. The nuclear spin can have 2I + 1 possible orientation with respect to a magnetic field. In the absence of magnetic field, these orientations are of equal energy. If a magnetic field *B* is applied or is present at the site of the nucleus, then the energy levels split. This is analogous to the Zeeman Effect. Each level is characterized by a magnetic quantum m and has energy given by

$$\begin{aligned} E_m &= -\,\vec{\mu}\,.\vec{B} \\ &= -\,g\,\mu_n\,\vec{I}\,.\vec{B} \\ &= -\,g\,\mu_n\,B\,m, \end{aligned} \qquad \text{.... (2)}$$

with m taking values - *I*, -(*I* – 1), -(*I* – 2), ...(*I* – 1), *I* . A nucleus with spin ½ will have two possible orientations, Fig.1.

Consider the specimen having these nuclei. When they are in a magnetic field, the initial populations of the energy levels are determined by thermodynamics, as described by the Boltzmann distribution. This is very important, and it means that the lower energy level will contain slightly more nuclei than the higher level. The actual differential between the levels depends on μ, *B* and the temperature *T*. It is possible to excite these nuclei into the higher level with electromagnetic radiation. The frequency of radiation needed is determined by the difference in energy between the energy levels. This energy difference is given by

$$\Delta E \;=\; g\,\mu_n\,B\,, \qquad \text{.... (3)}$$

as $\Delta m = \pm 1$.

In order to understand the absorption of radiation by a nucleus (of spin ½) in a magnetic field, imagine that it is in the lower energy level. Its magnetic moment is aligned with the field and is not opposed to it. Remember that the nucleus also has an angular momentum (the spin) and a torque $\vec{\tau}$, which is perpendicular to the angular momentum $\vec{I}$, acts on it,

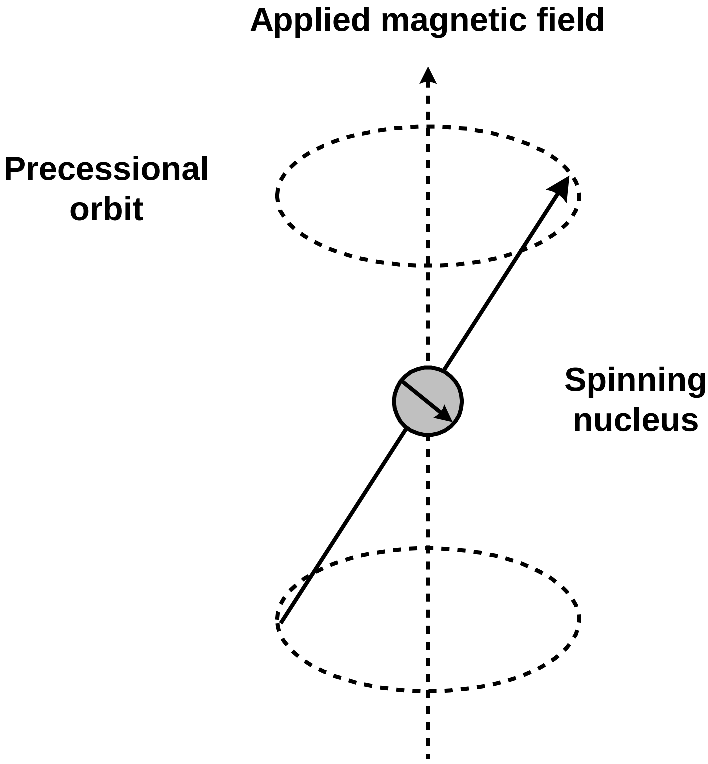

Applied magnetic field
Precessional
orbit
Spinning
nucleus

Fig. 2

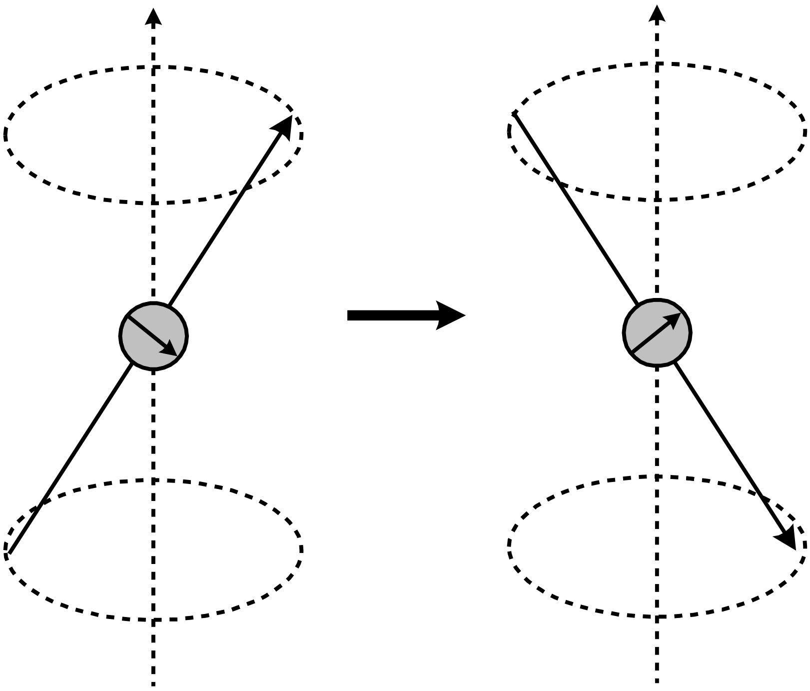


Fig. 3

$$\vec{\tau} = \vec{\mu} \times \vec{B}$$

$$= g\, \mu_n\, \vec{I} \times \vec{B} \qquad .... (4)$$

In the presence of a magnetic field, the axis of rotation will *precess* around the magnetic field, Fig 2. The frequency of precession is called the Larmor *frequency*. It is given by

$$\vec{\nu} = \frac{g\, \mu_n}{h} \vec{B}. \qquad .... (5)$$

It is identical to the transition frequency $\Delta E/h$. A resonance absorption of electromagnetic power occurs when photons (of the radio-frequency field) bombarding the specimen have the proper energy to excite transitions between these levels, i.e., satisfy the condition of resonance. The strength of the absorption depends upon the difference in population of the levels involved.

When the energy is absorbed by the nucleus, then the angle of precession will change. For a nucleus of spin ½, the absorption of radiation "flips" the magnetic moment so that it now opposes the field, Fig 3.

It is important to realize that only a small proportion of "target" nuclei are in the lower state (and can absorb radiation).

There is now the possibility that by exciting these nuclei, the populations of the higher and lower energy levels will become equal. When this occurs, then there will be no further absorption of radiation. The spin system gets saturated. In order to have a sustained absorption, there must be relaxation processes which return nuclei to the lower energy state.

There are two major relaxation processes: (1) Spin-lattice relaxation, (2) Spin-spin relaxation. The later one is not very effective. Consider the former. The atoms containing these nuclei are in rotational and vibration motion. The magnetic fields generated by these motions interact with nuclear precession and cause the nuclei to lose energy and return to the lower state. The energy that a nucleus loses increases the amount of vibration and rotation within the lattice resulting in a little increase in the temperature of the sample.

(II) EXPERIMENTAL TECHNIQUES

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.

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 loads (Δw) on the generator changes. This change of Δw is proportional to the change in base current ΔI_b or collector current ΔI_c of the generator. The proportionality, however, holds only the power dissipated by the sample due to absorption is small in comparison to the total losses in the circuit. This change in ΔI_c is 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 50Hz in the present set-up.

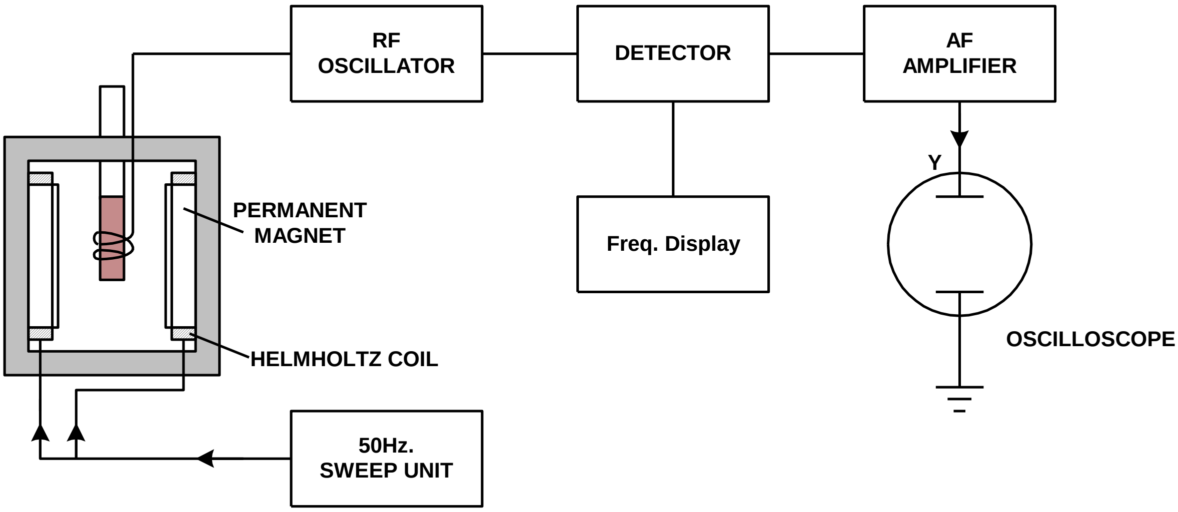


Fig. 4 : Block Diagram of NMR Spectrometer

(III) DESCRIPTION OF THE NMR SPECTROMETER

It consists of three units. A block diagram of the NMR spectrometer is given in Fig. 4 and panel diagrams in Fig. 5.

(i) Unit 1: This unit contains a critically adjusted (marginal) radio frequency oscillator in the desired frequency range for ^1H and ^{13}F resonance. A marginal oscillator is required here so that slightest increase in its load decreases the amplitude of oscillations to an appreciable extent. The sample is kept inside the tank coil of unit 2. 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 two times in each complete cycle of modulating filed (Helmohltz coil supply voltage). The result is in amplitude modulated carrier (Fig. 6A) which is then detected and amplified by high gain low noise audio frequency amplifier.

(ii) Unit 2: This unit consists of two permanent magnets of high magnetic field and placed in the structure (shown in Fig-5 unit 2). The magnets are very accurately aligned to produces the desired very uniform magnetic field. The magnetic field in the centre is 5.02 KG. For modulation with a low frequency magnetic field a pair of coils (Helmohltz coils) is mounted over the permanent magnets. Inside the magnetic field the inductance of the tank coil of the R.F. Oscillator is mounted accurately in the uniform field and such that sample change is very conveniently done.

(iii) Unit 3: This unit consists of three sections:

a) A frequency counter to display the frequency of Oscillations on a 4 digit display
b) 50 HZ Sweep Unit for modulation with a low frequency magnetic field, 50 Hz current flows through the Helmholtz coils.
c) Phase shifter – In order to make it possible to use an ordinary display type oscilloscope in X-Y mode a phase shifter is provided.

The circuit diagram of the phase shifter is shown in Fig. 7B. The primary of the transformer is fed from the 220V, 50Hz (or 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° (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, shown dotted. The vector OD, or the voltage across points 0 and D, will, therefore, have a constant magnitude equal to V_1 and its phase, variable from 0 to 180°. 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.

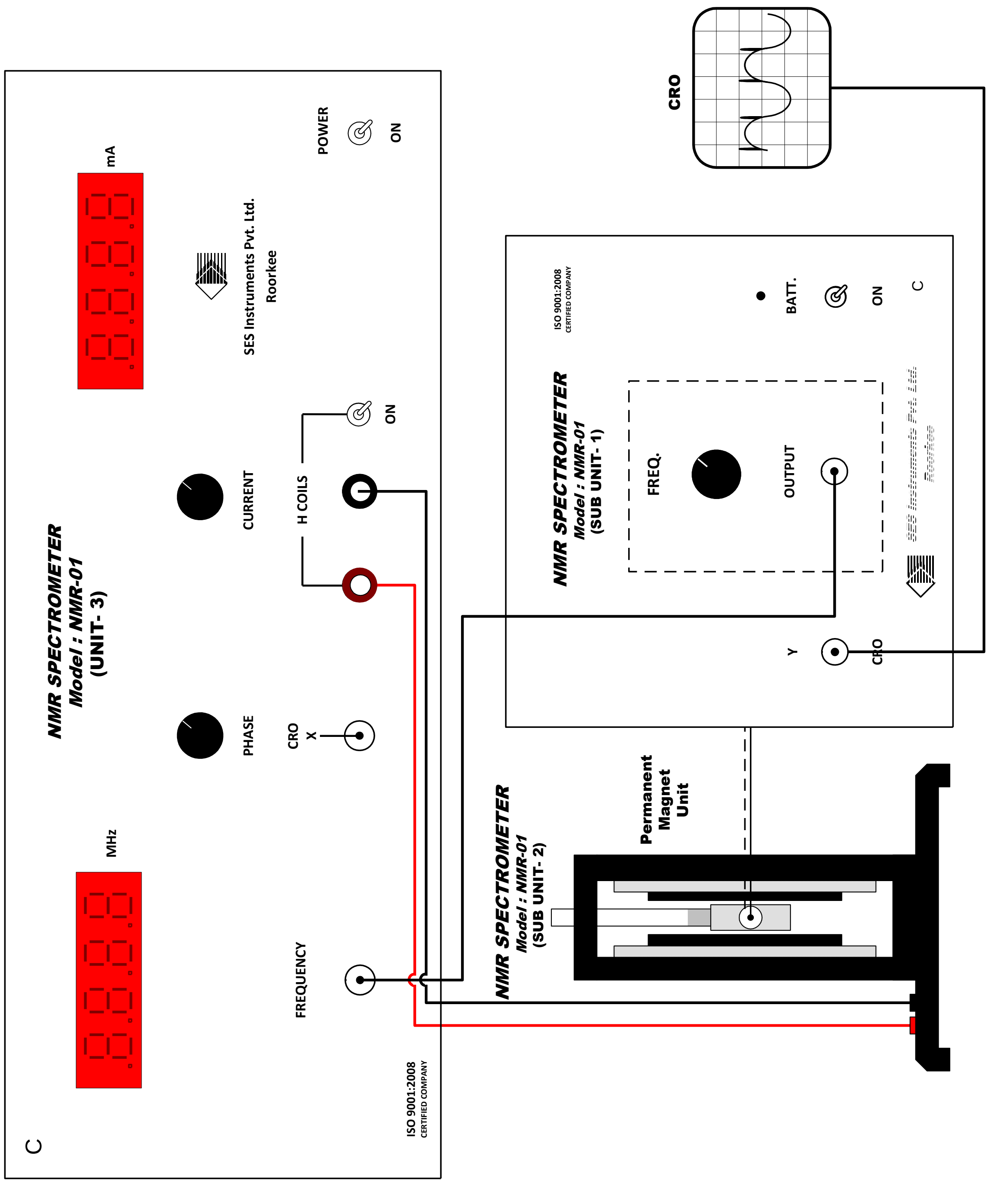


Fig.5: Panel Diagram of Nuclear Magnetic Resonance Spectrometer, NMR-01

(IV) TEST SAMPLES AND THEIR PREPARATION

(a) $FeCl_3$ (b) $CuSO_4$ (c) Glycerine (d) Tap Water

(e) Polytetraflouroethylene (PTFE)

Four sample tubes are also provided. PTFE sample can be used directly. While for Glycerine fill about half in the sample tube. For $FeCl_3$ and $CuSO_4$ take the sample tube and fill ordinary water upto half and add small quantity of $FeCl_3$, $CuSO_4$ in two separate tubes. For Water add normal tap water in the empty sample tube.

(V) CONTROLS AND TERMINALS

(i) Unit 1: ON-OFF Switch: To switch on the battery placed inside the unit

FREQUENCY: To adjust the frequency of the oscillator.

BNC - CRO Y : For Y input terminal of CRO

BNC – Frequency: To connect to FREQ BNC of Unit 2

(ii) Unit 2: Hole on the top: To insert sample tube in the tank coil.

Red & Black terminals: To connect to similar terminals on Unit 3.

(iii) Unit 3: FREQUENCY BNC: To connect Frequency ‘OUT PUT’ of unit 1.

CRO ‘X’ BNC: To connect to CRO ‘X’ input terminal.

H COILS: To connect to ‘RED’, ‘BLACK’ connector of Unit 2.

POWER ‘ON’: To switch ‘ON’ power of this unit.

PACKING LIST

1) Unit 1
2) Unit 2
3) Unit 3
4) Sample tube: 4 nos.
5) Samples : PTFE, Glycerine, $FeCl_3$, $CuSO_4$
6) Sample tube stand.
7) BNC Cables – 3 nos.
8) H Coils leads.

INSTALLATION

Proceed as follows:

1) Connect Unit – 1 ‘Y’ to CRO input ‘Y’ with a BNC cable.
2) Connect Unit – 1 ‘FREQ. OUTPUT’ to Unit – 3 ‘FREQUENCY’ with BNC cable.
3) Connect Unit – 3 ‘H COILS’ to connectors of Unit - 2.
4) Turn fully anti clockwise the knobs on Unit -1 and Unit – 3.
5) Ensure that ‘ON-OFF” switch of BATT (Unit – 1) ‘H COILS” (Unit – 3) Power (Unit – 3) are in OFF Position.
6) Connect mains cord of Unit – 3 to AC mains.
7) Ensure that Unit – 3 is properly earthed.

OPERATING INSTRUCTIONS

(a) Switch ‘ON’ power switch of Unit – 3.
(b) Switch ‘ON’ switch of ‘H-COILS’ and adjust current to 80 mA.
(c) Insert the sample tube containing preferably Cupric Sulphate from the top of Unit – 2.
(d) Switch ‘ON’ Battery switch of Unit – 1.
(e) Adjust the ‘Y input’ amplitude knob to 50 mV/div. for proton and 20 mV/div for fluorine.
(f) Rotate frequency knob (Unit – 1) slowly until the peaks are seen.
(g) Further slowly rotate this knob till the distance between peaks in 10 ms.
(h) Note the value of frequency displayed on Unit – 3.
(i) Please note that the magnetic field at sample coil is 5.02 KG or 0.502 Tesla.
(j) Sample Test Results are given in Appendix-3.

CALCULATIONS

From Eq. 5

$$g = \frac{h}{\mu_n}, \frac{\nu}{B_o}$$

With h = 6.625×10^{-34} Js

$\mu_n = 5.051 \times 10^{-27}$ J/T

On substituting that values h, μ_n, ν (obtained at h step above) and B_o given at (i) step above one can calculate the value of g.

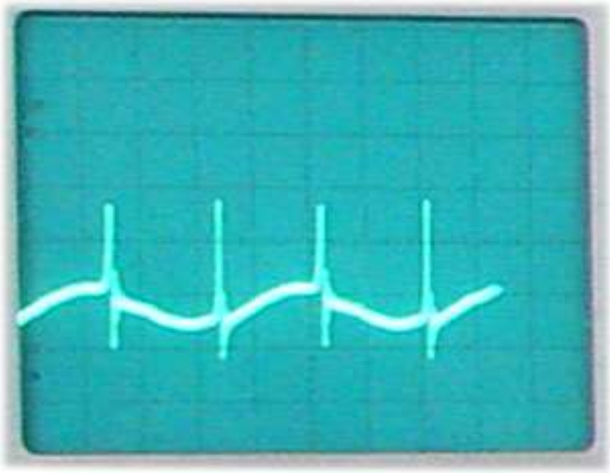


Fig. 6a: Photograph of resonance

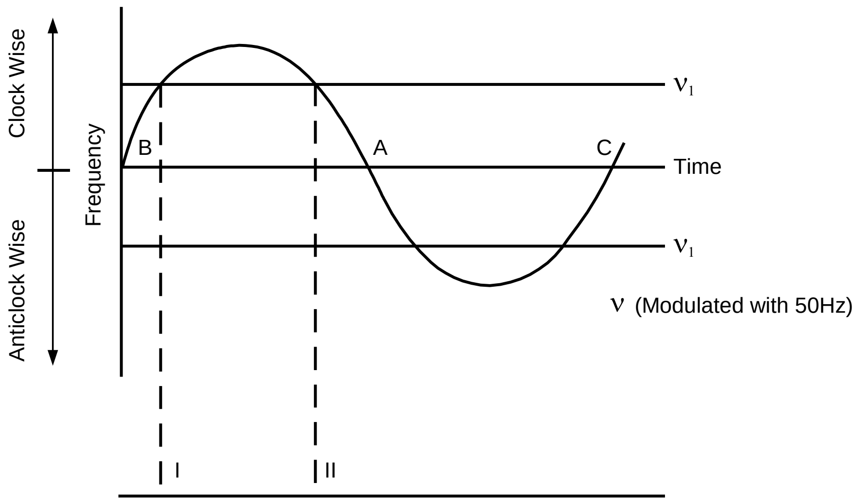


Fig. 6b: The radio frequency is linearly polarised, which can be regarded as two circularly polarised fields of opposite direction (say clockwise and anti clockwise). Further magnetic field B_0 does not changes direction. Thus resonance occurs when the two frequencies (ν_1 and ν_0) becomes equal in magnitude as well as direction i.e. two times in one full cycle of B+B'(modulating small field due to Helmohltz current).

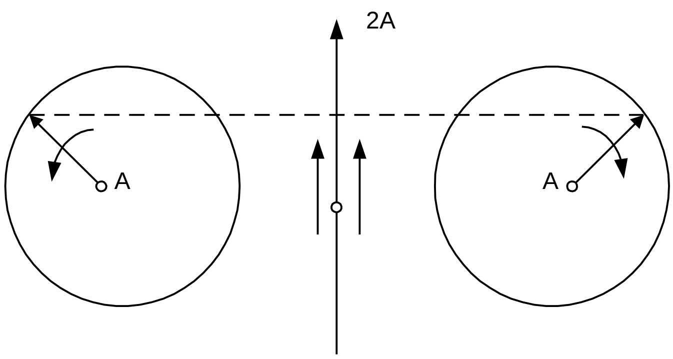


Fig. 6c : A linearly field of frequency is equivalent to two fields rotating in opposite direction with the same frequency ν

APPENDIX - 1

ORIGIN OF TWO PEAKS

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

The spin precesses with Larmor's frequency $(\vec{\nu} = \frac{g\mu_n}{h}\vec{B})$ and hence varies in magnitude due to small variation of magnetic field $\overline{B}$ which is due to an alternating current in the Helmholtz coils. Now if the radio frequency field, ω_1 falls in the range of υ the resonance occurs. The positions of the two peaks can be understood with Fig 6.

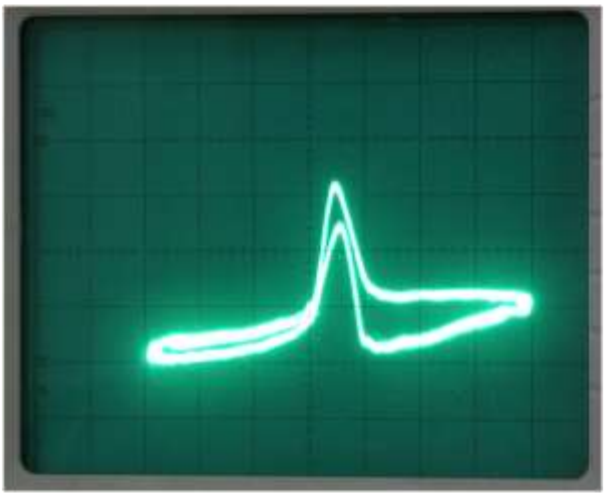


Fig. 7

APPENDIX - 2

Advantage of Phase Shifter

In case a measuring (calibrated) oscilloscope is not available, the frequency of resonance can be determined in the following way:

1. With a BNC cable connects CRO ‘X’ of Unit-3 to X input of CRO and bring CRO in X-Y mode.
2. With the phase shifter overlap the two peaks, Fig 7.
3. Using frequency control of Unit-1, bring the two over lapped peaks in the centre of display.
4. Read the frequency of Unit-3 and this will be the resonance frequency υ.

APPENDIX - 3

SAMPLE TEST RESULTS OF NMR SPECTROMETER, NMR-01

Object : To determine the g-factor by the NMR Spectrometer

Sample = Cupric Sulphate ($CuSO_4.5H_2O$) & Teflon

Observation:

(a) Frequency displayed on Unit -3 when the distance between peaks 10ms for Cupric Sulphate (CuSO4.5H2O): 20.82 MHz

(b) Frequency displayed on Unit-3 when the distance between peaks 10ms for Teflon:19.59 MHz

Calculation for Cupric Sulphate ($CuSO_4.5H_2O$):

We know from Eq. 5 (Refer Manual page no. 8)

$$g = \frac{h}{\mu_n} x \frac{\nu}{B_0}$$

Where,

h = Planck's Constant = 6.625×10^{-34} JS

$\mu_n = 5.051 \times 10^{-27}$ J/T

$B_0 = 0.483$ T

Experimental value of **g = 5.654**

Standard value of g = 5.5857

Calculation for Teflon:

We know from Eq. 5 (Refer Manual page no. 8)

$$g = \frac{h}{\mu_n} x \frac{\nu}{B_0}$$

Where,

h = Planck's Constant = 6.625×10^{-27} erg.sec

$\mu_n = 5.051 \times 10^{-27}$ J/T

$B_0 = 0.483$ T

Experimental value of **g = 5.32**

Standard value of g = 5.6

SOURCE OF ERRORS

The main source of error in the result is the determination of magnetic field at the resonance peaks due to following factor:

1. In the estimation of physical dimensions of the Helmholtz coils.
2. Due to distortions in the AC main's waveforms.