3.2 Types of NMR Spectrometers

- There are two NMR spectrometers
    1. Continuous wave NMR spectrometry
    2. Pulsed or Fourier transformer [FT-NMR] spectroscopy.

Continuous- wave NMR spectrometers (or) spectroscopy(cw-NMR)

- A CW-NMR spectrometer consists of
    1. A magnet to separate the nuclear spin energy states.
    2. At least two RT channels.
    3. A sample proton containing coils for coupling the sample with RF fields.
    4. A detector to process the NMR signals.
    5. A sweep generator for sweeping either the magnetic or RF field through the resonance frequency of sample.
    6. A recorder to display the spectrum.

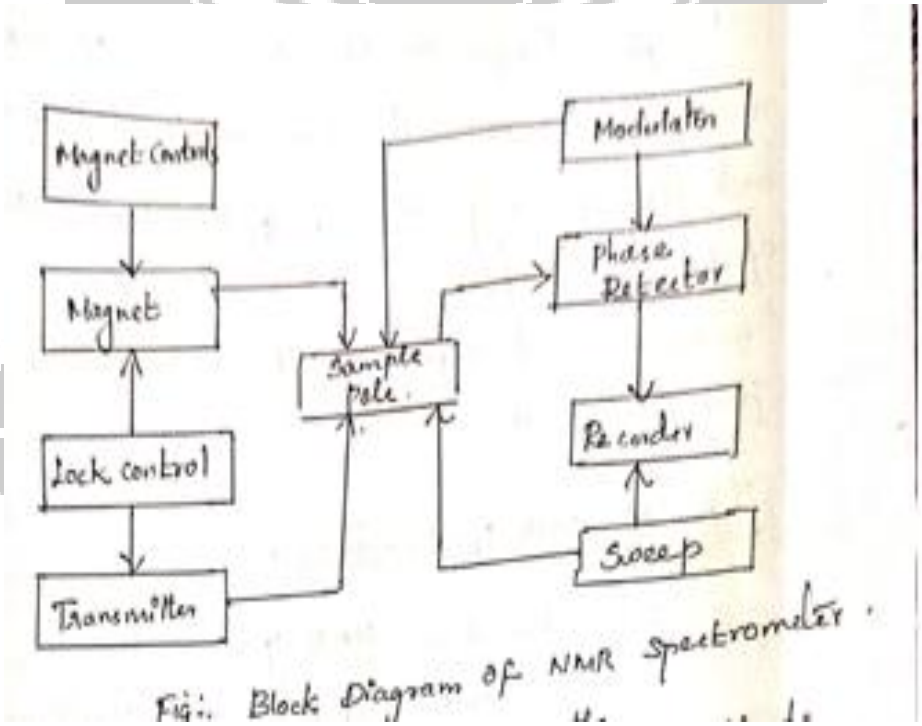


Fig:. Block Diagram of NMR Spectrometer.

The spectrum can be scanned by two methods,

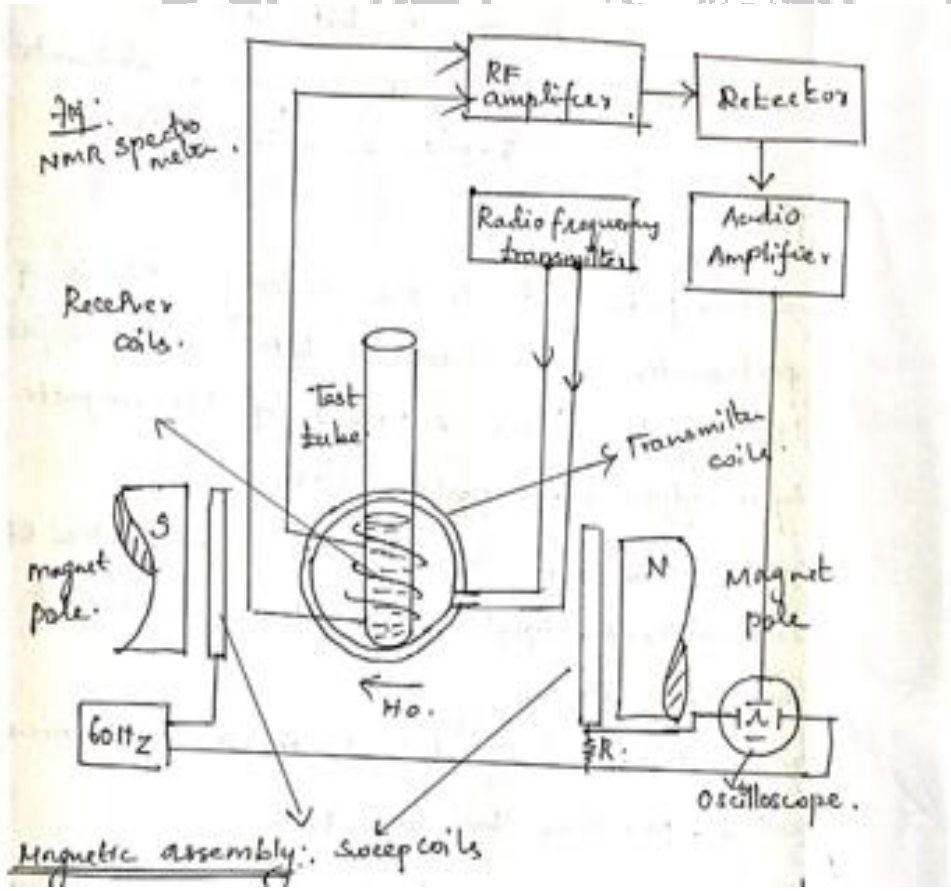
a) Frequency sweep method
b) Field sweep method

a) Frequency sweep method

In this frequency sweep method the magnetic field(H0) is held constant which keeps the nuclear spin energy level constant then the RF signal(H1) is swept [varied continuously] to determine the frequencies at which energy is absorbed.

b) Field sweep method

In this field sweep method, if the RF signal [H1] is held constant then the magnetic field is swept which varies the energy levels to determine the magnetic field strength that produce resonance at a fixed resonance frequency.



Magnetic assembly

- For high resolution work the magnetic field over the entire sample volume must be maintained uniform in space & time by,
    - ❖ The use of large poles pieces composed of a very homogenous alloy.
    - ❖ The polishing of poles faces to optical tolerances.
    - ❖ The use of narrow pole gap
    - ❖ Magnetic can be either permanent magnet or electromagnet.
    - ❖ Permanent magnets are simple & inexpensive to operate but requires elaborate power supplies & cooling systems.

Probe unit

- The probe unit is the sensing elements of the spectrometer. It is inserted between pole faces of the magnet in the X-Y plane the magnet air gap by adjustable probe holders.
- The probe unit houses the sample, the RF transmitters, output attenuator, receiver & phase sensitive detector.
- The sample is contained in a cylindrical, thin walled, percussion bore glass tube.
- The probe design can be of two types
    a) A single coil probe
    b) Crossed line

a) Single coil probe

A single coil probe has one coil that not only supplies the RF radiation to the sample but also serves as a part of the detector circuit, for the NMR absorption

signal. To detect resonance absorption & to separate the NMR signal from the imposed RF field a RF bridge is used.

b) Crossed coil

Crossed coil (nuclear induction) prober, have two coil, one for irradiating the sample & a second coil mounted orthogonally for signal.

Instrument stabilization

- Before the analysis or measurement the stabilization of the instrument is necessary the things that has to be considered for stabilization are
  - ❖ NMR spectrum should be recorded directly on precalibrated chart paper.
  - ❖ The ratio of RF frequency signal to field strength be very stable.
  - ❖ There is no problem with RF frequency but the magnet stability is a problem. So proper caring should be there in the selection of magnet.
  - ❖ A phase sensitive detector should be finely tunnel to sense only one component either the absorption or the dispersion mode.

Types of NMR spectrometer

1) Minimal type NMR spectrometer
2) Multipurpose NMR spectrometer
3) Wide line NMR spectrometer

1) Minimal type NMR spectrometer

- Among the Families of continuous wave NMR spectrometer, the minimal type has stressed reliability case of operation.
- It uses permanent magnet.

- So it is inexpressive, there is no need of power supply & cooling system.

2) Multipurpose NMR spectrometer

- The second family of NMR spectrometer is more diverse.
- These instruments are designed primarily for research & emphasis is on high performance versatility with cost being a secondary consideration.

3) Wide line NMR spectrometer

- It uses a frequency synthesis to generate the RF field & a permanent magnet or a compact light weight electromagnet slowly varying sun voltages are directly injected in the rectangular for the magnet power supply for the electromagnet.
- These signals cause a corresponding change in magnet energizing current, there by creating a scan of the magnetic field.

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Applications:

❖ NMR spectroscopy is used for the identification and structural identification of organic, metal and biochemical molecules.

❖ It helps in identifying the organic compound.

- It reveals the presence of particular functional groups such as hydroxyl groups in alcohols and phenols, aldehydes, carboxylic acids, amines, amides and the relative position of these groups.
- More than 200 isotopes have magnetic moments and these can studied by NMR.
- NMR technology has been applied increasingly to fields other than chemistry, such as biology, engineering, industrial quality control and medicine.
- One of the most important NMR applications is that of magnetic resonance imaging on MRI. In MRI, data from pulsed RF excitation of solid or semisolid objects are subjected to Fourier transformation & converted to three dimensional images of the interior of the objects.
- The primary advantage of MRI is that images of objects are formed noninvasively. This means that there is little or no potential of radiation injury or other damage to human or animal subjects as might be encounted with X-Rays CT scans or other similar methods.

MASS SPECTROMETERS:

- The mass spectrometer was first introduced by J.J.Thomas [1912], & Aston made some modification is that instrument [1919] to get the results of greater accuracy & precision.
- This mass spectrometer is an instrument which was designed for determining the relative mass of atoms & molecules.
- In mass spectrometer, the sample to be analyzed is first bombarded with an electron beam to produce ions.
- These ions are then sorted out by accelerating them through electric & magnetic fields according to their mass/charge ratio.
- Mass spectrometer separates the ions on the basis of their mass to charge ratio.

- But most of the ions are singly charged, so the ratio is equal to the mass of the ions.
- A record of the number of different kinds of ions is called the mass spectrometer.

- The advantage of mass spectrometry over other direct methods is that the impurities of masses different than the one being analyze do not interfere with the results & the other advantage is its greater sensitivity.