

M.Sc. Chemistry

(Core Course- X)

Application of Spectroscopy

Unit- III Magnetic Resonance Spectroscopy

Introduction: NMR Spectroscopy is abbreviated as Nuclear Magnetic Resonance spectroscopy. Nuclear magnetic resonance (NMR) spectroscopy is the study of molecules by recording the interaction of radiofrequency (Rf) electromagnetic radiations with the nuclei of molecules placed in a strong magnetic field.

Zeeman first observed the strange behaviour of certain nuclei when subjected to a strong magnetic field at the end of the nineteenth century, but the practical use of the so-called “Zeeman effect” was only made in the 1950s when NMR spectrometers became commercially available.

It is a research technique that exploits the magnetic properties of certain atomic nuclei. The NMR spectroscopy determines the physical and chemical properties of atoms or molecules. It relies on the phenomenon of nuclear magnetic resonance and provides detailed information about the structure, dynamics, reaction state, and chemical environment of molecules.

Basis of NMR Spectroscopy:

Nuclear Magnetic Resonance (NMR) was first detected experimentally at the end of 1945, nearly concurrently with the work groups Felix Bloch, Stanford University and Edward Purcell, Harvard University. The first NMR spectrum was first published in the same issue of the Physical Review in January 1946. Bloch and Purcell were jointly awarded the 1952 Nobel Prize in Physics for their research of Nuclear Magnetic Resonance Spectroscopy.

Nuclear magnetic resonance (NMR) spectroscopy is a crucial analytical tool for organic chemists. The research in the organic lab has been significantly improved with the aid of the NMR. Not only can it provide information on the structure of the molecule, it can also determine the content and purity of the sample. Proton (^1H) NMR is one of the most widely used NMR methods by organic chemists. The protons present in the molecule will behave differently depending on the surrounding chemical environment, making it possible to elucidate their structure.

NMR Spectroscopy Principle:

Many nuclei have spin, and all nuclei are electrically charged, according to the NMR principle. An energy transfer from the base energy to a higher energy level is achievable when an external magnetic field is supplied.

- All nuclei are electrically charged and many have spin.
- Transfer of energy is possible from base energy to higher energy levels when an external magnetic field is applied.
- The transfer of energy occurs at a wavelength that coincides with the radio frequency.
- Also, energy is emitted at the same frequency when the spin comes back to its base level.
- Therefore, by measuring the signal which matches this transfer the processing of the NMR spectrum for the concerned nucleus is yield.

Chemical Shift in NMR Spectroscopy:

In nuclear magnetic resonance (NMR) spectroscopy, the chemical shift is the resonant frequency of an atomic nucleus relative to a standard in a magnetic field. Often the position and number of chemical shifts are diagnostic of the structure of a molecule. Chemical shifts are also used to describe signals in other forms of spectroscopy such as photoemission spectroscopy.

Some atomic nuclei possess a magnetic moment (nuclear spin), which gives rise to different energy levels and resonance frequencies in a magnetic field. The total magnetic field experienced by a nucleus includes local magnetic fields induced by currents of electrons in the molecular orbitals (electrons have a magnetic moment themselves). The electron distribution of the same type of nucleus (e.g. ^1H , ^{13}C , ^{15}N) usually varies according to the local geometry (binding partners, bond lengths, angles between bonds, and so on), and with it the local magnetic field at each nucleus. This is reflected in the spin energy levels (and resonance frequencies). The variations of nuclear magnetic resonance frequencies of the same kind of nucleus, due to variations in the electron distribution, is called the chemical shift. The size of the chemical shift is given with respect to a reference frequency or reference sample, usually a molecule with a barely distorted electron distribution.

NMR Spectroscopy Applications:

Scientists utilise a myriad of techniques to study the molecular properties of matter, and when it comes to useable data Nuclear Magnetic Resonance (NMR) Spectroscopy is one of the most valuable. The advanced analytical technique allows researchers to study the unique chemical, physical and biological characteristics of a molecules, with the method used across a wide range of fields and professions. Few applications of NMR Spectroscopy are discussed below:

- **Chemistry laboratories:** Chemists rely on NMR Spectroscopy as a tool to chart the complex molecular structures of matter. The technique sees samples placed

in a specialised NMR spectrometer, where it's exposed to a strong magnetic field. The gravitational pull generated by the field excites the nuclei of some atoms and creates resonant frequencies. These resonant frequencies are then tracked, measured and used to generate useful data.

- **Food quality control and research:** Before hitting the consumer market, foods undergo strict quality control and research studies. NMR Spectroscopy is widely used across the industry to map protein structures, profile amino acids, identify carotenoids and quantify metabolites.
- **MRI scans:** Most people are familiar with Magnetic Resonance Imaging (MRI) scans which use powerful magnetic fields and radio waves to reveal detailed images of the internal organs. Data is generated based on rates of absorption and energy emission recorded in the radiofrequency (RF) range. The medical imaging technique is founded in NMR Spectroscopy and uses the same basic principles.
- **Identifying human disorders:** When used alongside metabolomics data, NMR Spectroscopy is an invaluable tool for identifying human disorders. The cellular metabolism contains important biomarkers used to diagnose a wide range of conditions, including tuberculosis, pneumonia and malaria. NMR Spectroscopy allows researchers to identify these tell-tale biomarkers and treat patients accordingly. The technique can also be used to investigate Parkinson's disease, as well as cardiovascular diseases and a range of neuropsychiatric disorders such as bipolar, schizophrenia, major depression and autism-spectrum disorders.
- **Cancer diagnosis:** Over the past few decades, NMR Spectroscopy has become a key technique for cancer diagnosis. The ability to analyse abnormal behaviour in the cellular metabolism allows scientists to detect the metabolite-based biomarkers associated with cancers.
- **Environmental monitoring:** It's not just the medical field where NMR Spectroscopy is considered a valuable tool, with the technique also used for environmental monitoring. Spectrometers are used to detect and characterise contaminants in air, soil and water samples as well as monitor the metabolic responses of organisms exposed to these contaminants.