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Use Our NMR service for all your NMR needs. Uses of NMR spectroscopy Nuclear Magnetic Resonance (NMR) spectroscopy is an analytical chemistry technique used in quality control and research for determining the content and purity of a sample as well as its molecular structure. For example, NMR can quantitatively analyze mixtures containing known compounds. For unknown compounds, NMR can either be used to match against spectral libraries or to infer the basic structure directly. Once the basic structure is known, NMR can be used to determine molecular conformation in solution as well as studying physical properties at the molecular level such as conformational exchange, phase changes, solubility, and diffusion. In order to achieve the desired results, a variety of NMR techniques are available. The basics of NMR are described here. You too can do NMR by using our NMR service. The basis of NMR The principle behind NMR is that many nuclei have spin and all nuclei are electrically charged. If an external magnetic field is applied, an energy transfer is possible between the base energy to a higher energy level (generally a single energy gap). The energy transfer takes place at a wavelength that corresponds to radio frequencies and when the spin returns to its base level, energy is emitted at the same frequency. The signal that matches this transfer is measured in many ways and processed in order to yield an NMR spectrum for the nucleus concerned. Fig. 1. The basis of NMR Fig.1, above, relates to spin-1/2 nuclei that include the most commonly used NMR nucleus, proton (1H or hydrogen-1) as well as many other nuclei such as 13C, 15N and 31P. Many nuclei such as deuterium (2H or hydrogen-2) have a higher spin and are therefore quadrupolar and although they yield NMR spectra, their energy diagram and some of their properties are different. Chemical shift The precise resonant frequency of the energy transition is dependent on the effective magnetic field at the nucleus. This field is affected by electron shielding which is in turn dependent on the chemical environment. As a result, information about the nucleus' chemical environment can be derived from its resonant frequency. In general, the more electronegative the nucleus is, the higher the resonant frequency. Other factors such as ring currents (anisotropy) and bond strain affect the frequency shift. It is customary to adopt tetramethylsilane (TMS) as the proton reference frequency. This is because the precise resonant frequency shift of each nucleus depends on the magnetic field used. The frequency is not easy to remember (for example, the frequency of benzene might be 400.132869 MHz) so it was decided to define chemical shift as follows to yield a more convenient number such as 7.17 ppm. $\delta = (\nu - \nu_0) / \nu_0$ The chemical shift, using this equation, is not dependent on the magnetic field and it is convenient to express it in ppm where ν_0 (for proton) TMS is set to ν_0 thereby giving it a chemical shift of zero. For other nuclei, ν_0 is defined as ν_0 / ν_{TMS} where ν (Greek letter Xi) is the frequency ratio of the nucleus (e. g., 25.145020% for 13C). In the case of the 1H NMR spectrum of ethyl benzene (fig. 2), the methyl (CH3) group is the most electron withdrawing (electronegative) and therefore resonates at the lowest chemical shift. The aromatic phenyl group is the most electron donating (electropositive) so has the highest chemical shift. The methylene (CH2) falls somewhere in the middle. However, if the chemical shift of the aromatics were due to electropositivity alone, then they would resonate between four and five ppm. The increased chemical shift is due to the delocalized ring current of the phenyl group. Fig. 2. 1H NMR spectrum of ethylbenzene This definition of chemical shift is sufficient for most purposes. However, complications arise when comparing chemical shifts under different conditions: solvent, temperature, etc. Chemical shifts are affected slightly by isotopic substitution, an effect that is known as an isotope shift. Spin-spin coupling The effective magnetic field is also affected by the orientation of neighboring nuclei. This effect is known as spin-spin coupling (fig. 3) which can cause splitting of the signal for each type of nucleus into two or more lines. Fig. 3. Spin-spin coupling The size of the splitting (coupling constant or J) is independent of the magnetic field and is therefore measured as an absolute frequency (usually Hertz). The number of splittings indicates the number of chemically bonded nuclei in the vicinity of the observed nucleus. Some common coupling patterns are shown below (fig. 4). (Click here for more examples of common homonuclear coupling-patterns and for their use in assigning 1H-NMR spectra as well as a description of heteronuclear coupling.) Fig. 4. Examples of coupling patterns showing coupling constants The above patterns are a first order approximation and are correct provided that all the coupled spins have widely separated chemical shifts. The different nuclei are labeled with the letters A and X (in a system of this type the letters come from widely separated parts of the alphabet). If the chemical shifts are similar then distortions in peak height occur as in the diagram below (the letters are also close together in the alphabet). For more than two spins, extra signals may appear. These effects are called second order coupling (fig. 5). Some examples are shown here and a detailed analysis of second order coupling is available in the literature. Fig. 5. An example of second order coupling Returning to the example of ethylbenzene (fig. 6), the methyl (CH3) group has a coupling pattern in the form of A3X2, which to a first order approximation looks like an AX2 multiplet. Likewise, the methylene (CH2) group has the form A2X3 that is equivalent to AX3. The first order approximation works because the groups are widely separated in the spectrum. The aromatic signals are close together and display second order effects. The ortho signal is a doublet AX while the meta and para signals are triplets. Fig. 6. Couplings in the ethylbenzene spectrum 1. NUCLEAR MAGNETIC RESONANCE By- Shivam Sharma , M.Pharm 1st Year P.S.I.T INSTRUMENTATION OF Presented to- Mr. ASHISH SRIVASTAVA Associate Professor 2. Introduction to NMR Spectroscopy Nuclear magnetic resonance spectroscopy is a powerful analytical technique used to characterize organic molecules by identifying carbon-hydrogen frameworks within molecules. Two common types of NMR spectroscopy are used to characterize organic structure: 1H NMR is used to determine the type and number of H atoms in a molecule; 13C NMR is used to determine the type of carbon atoms in the molecule. The source of energy in NMR is radio waves which have long wavelengths, and thus low energy and frequency. 3. Instrumentation The NMR spectrophotometer consists of following components:- 1. Sample holder 2. Permanent Magnet 3. Magnetic coil 4. Radio frequency generator 5. Radio frequency receiver 6. Read out system 4. 1. Sample holder The sample holder in NMR is normally tube- shaped and is therefore called the sample tube. The tube must be transparent to RF radiation, durable, and chemically inert. Glass or Pyrex tubes are commonly used. These are sturdy, practical, and cheap. They are usually about 6-8cm long and 0.3- 0.5cm in diameter, with a plastic cap to contain the sample. This type of tube is used for obtaining spectra of bulk samples and solutions. 5. 2. Permanent Magnet Permanent Magnet or electromagnet can be used in a NMR instrument. It should give stable and homogeneous magnetic field i.e. the strength and direction of magnetic field should not change point to point. Strength field should very high i.e. 20,000 Gauss (G). Because the chemical shifts are proportional to the field strength. The magnet size is 15 inches in diameter. 6. 3. Magnetic coil There is a relationship between the resonance frequency of nucleus and the strength of the magnetic field in which the sample is placed. Relationship is- $\nu = \text{Constant} \times H$ For the nucleus to resonate, the precessional frequency of the nucleus must equal to the applied RF radiation. If the ν is constant, the precessional frequency is fixed. 7. 4. Radio frequency generator In order to generate radio frequency radiation, radio frequency oscillator is used. To achieve the maximum interaction of the RF radiation with the sample, the coil of oscillator would be around the sample container. The oscillator irradiates the sample with a RF radiation. The oscillator coil is perpendicular to the applied magnetic field. 8. 5. Radio frequency receiver It is installed perpendicular to both magnetic field and the oscillator coil. It is tuned to the same frequency as transmitter. When precession frequency is match with RF radiation the nucleus induces (emf) in detector coil and this signal is amplified and sent to read out system. 9. 6. Read out system The read out system gives a spectrum as a plot of strength resonance signal on Y axis & strength of magnetic field on X axis. The strength of resonance signal is directly proportional to number of nuclei resonating at that particular field strength.

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