

Nuclear Magnetic Resonance
1 – Proton NMR
Introduction and Instrumentation

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Introduction to Nuclear Magnetic Resonance

Nuclear Magnetic Resonance (NMR) is the most powerful tool available for organic structure determination.

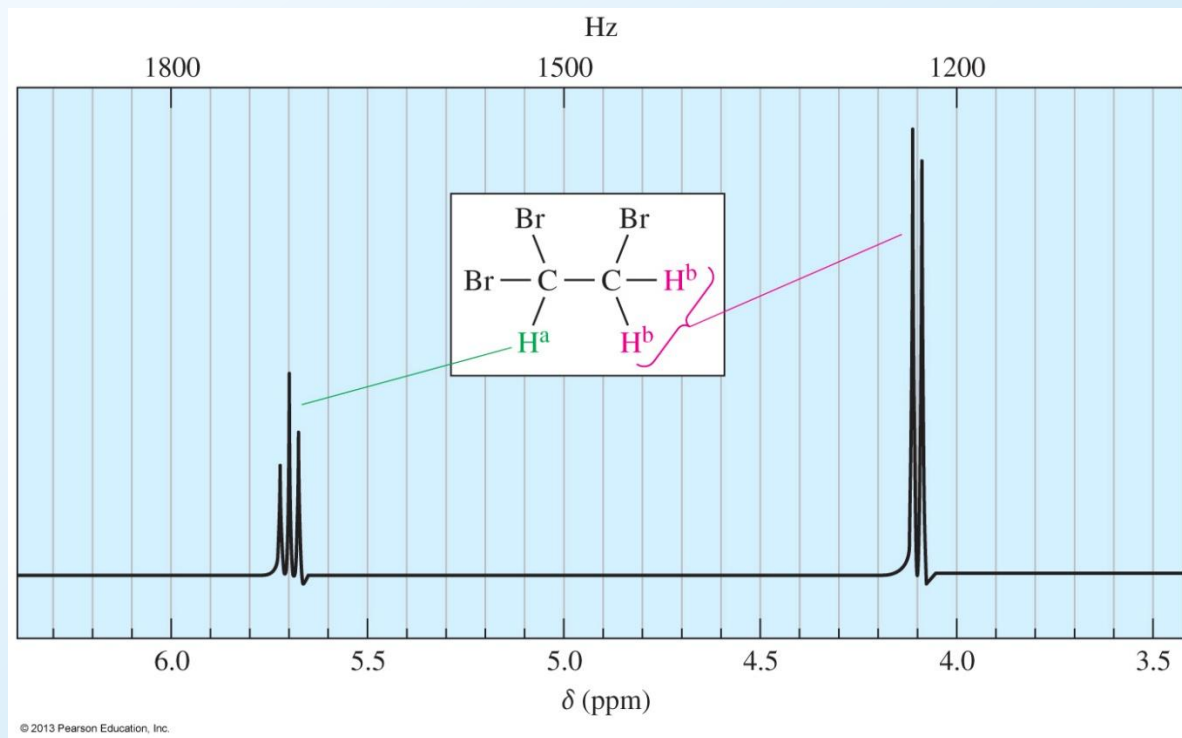
It is used to study a wide variety of nuclei: ^1H , ^{13}C , ^{15}N , ^{19}F , ^{31}P .

NMR can be used to determine the structure of compounds in gas, liquid or solid phases. The most common is solution of any of these phases.

We will focus on proton (hydrogen) – H-NMR, and carbon NMR, C13-NMR.

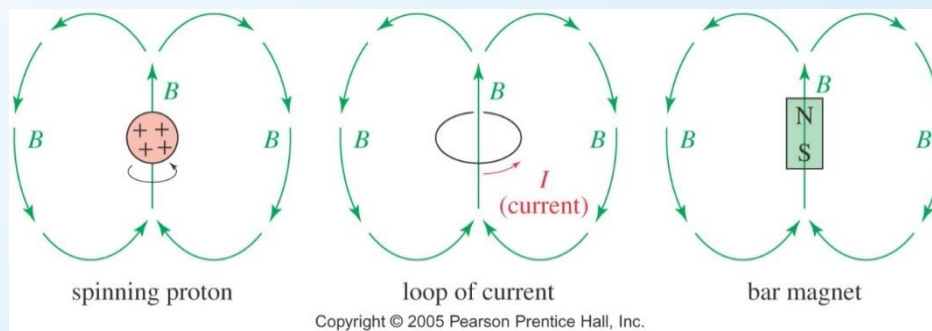
NMR Spectrum – A First Look

NMR graph consists of peaks. Each peak appears at a specific place and may be split, depending on the organic structure. Every peak in NMR is assigned to a hydrogen, meaning if there is a peak, it represents a proton. The x-axis is the chemical shift and y-axis as no measurement; however, the height of each peak is measured to determine the area under the peak.

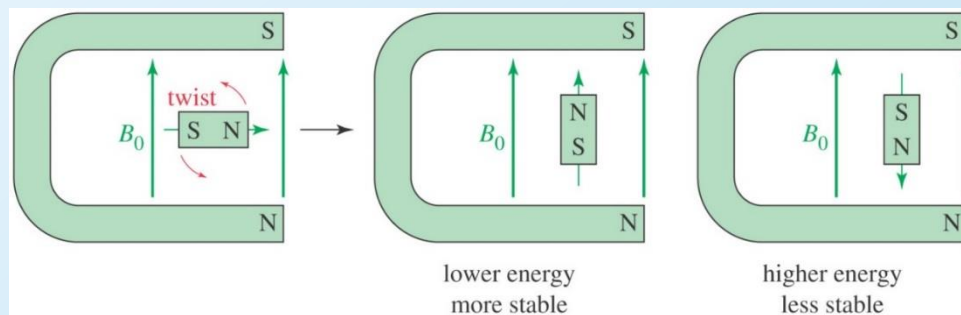


Theory – Nuclear Magnetic Resonance

Nuclear magnetic resonance (NMR) is a nuclear property. A nucleus with an odd atomic number or an odd mass number has a nuclear spin. Hydrogen is the most abundant element in organic compounds, and it has only one proton (odd number of proton). These protons have a magnetic spin (m_s number) and can generate a magnetic field.



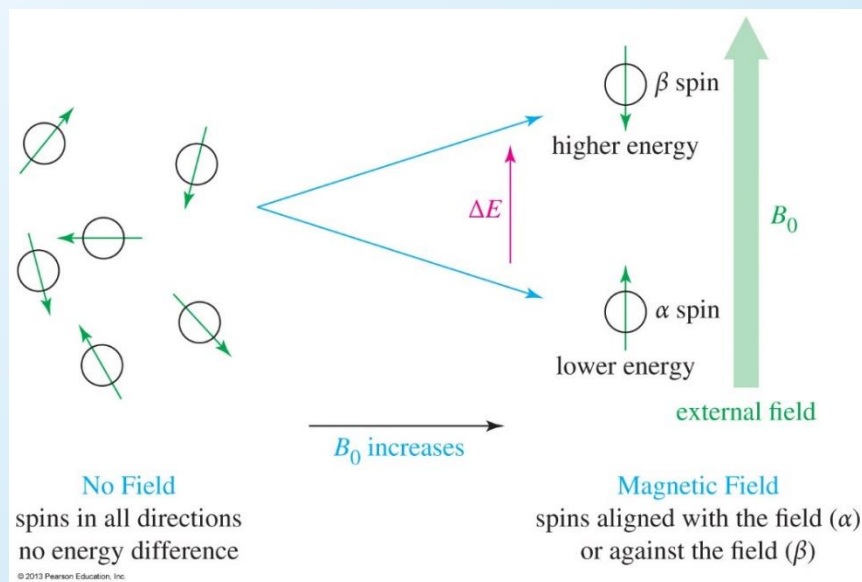
When placed in an external field, these spinning protons act like bar magnets.



Nuclei in Magnetic Field

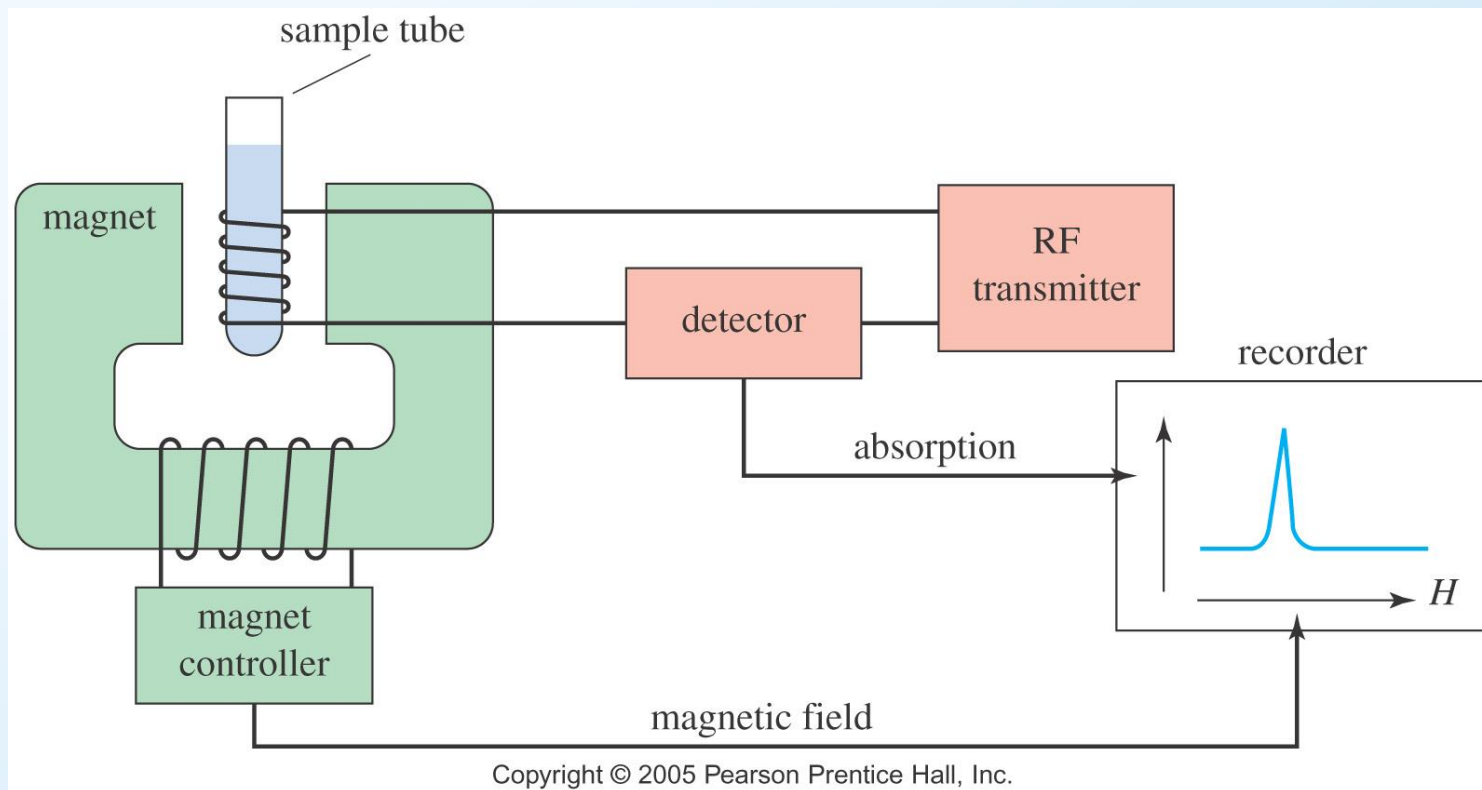
When the chaotic nuclei of an organic compound is brought near a magnetic field, the magnetic fields of spinning nuclei will align either *with* the external field, or *against* the field.

The right amount of energy can be absorbed and cause the spinning proton to flip from one orientation to another. The absorption of that energy is where the signal appears in NMR. Signals can appear at low or high energy. The energy in NMR is provided by the radiofrequency, which is a very low energy radiation.



The NMR Spectrometer

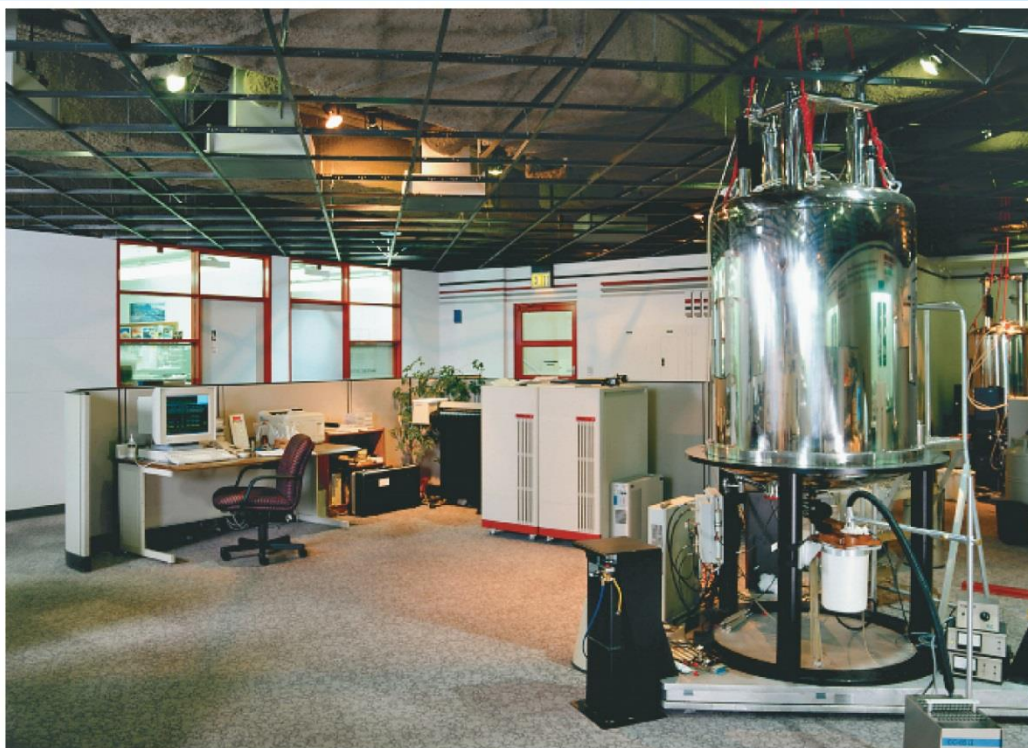
Below is a schematic showing sample tube in the magnet and the application of radiofrequency.



NMR at PNNL (Pacific Northwest National Lab)



A Typical 400 MHz NMR



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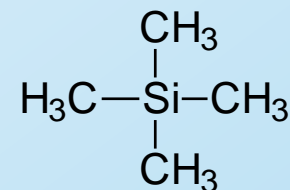
Sample Preparation

Solvent: Sample is dissolved in a solvent that is NMR inactive. Most solvents for organic compounds are hydrocarbons, e.g., chloroform (CHCl_3), dichloromethane (CH_2Cl_2), methanol (CH_3OH) and in some cases water (H_2O). These solvents are treated with deuterium (D), an NMR inactive isotope of H, so that the solvent solubility properties don't change drastically. CHCl_3 is CDCl_3 and H_2O is D_2O etc.

Samples are not destroyed during the analysis.

Sample Holder: An NMR tube (shown on the right) is used to make the sample.

Reference: Tetramethyl Silane (TMS), shown on the right, is added to the sample as a reference. Since silicon is less electronegative than carbon, TMS protons are highly shielded. The TMS signal is defined as zero. Organic protons absorb downfield (to the left) of the TMS signal.



Key Concepts

- Fundamentals of NMR