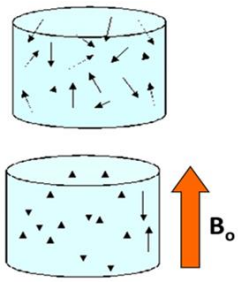
Nuclear Magnetic Resonance Spectroscopy Laboratory (NMRL)

Nuclear Magnetic Resonance (NMR) is a spectroscopic technique which provides information about the structural and chemical properties of molecules. Using NMR, information about bonding structure and molecular formula can also be obtained.

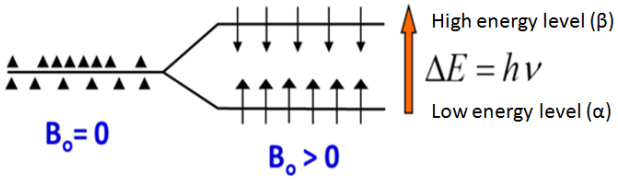
There are two NMR spectrometers in NMR facility at the METU Central Laboratory which are used for this purpose. These are High Resolution Digital 300 MHz (7 Tesla) NMR Spectrometer and High Power Solid State 300 MHz NMR Spectrometer of Bruker Biospin.

BASIC PRINCIPLES

Nuclear magnetic Resonance (NMR) is a physical phenomenon which depends on the magnetic properties of the nucleus of the atom. Briefly, it is the absorption of electromagnetic radiation in radiofrequency region by an atomic nucleus when it is placed in a strong magnetic field. Magnetic moment of atomic nuclei can be measured by using NMR. NMR measurements are useful to analyze the molecular structure of the chemicals since the value of magnetic moment depends on the chemical environment of the nuclei.



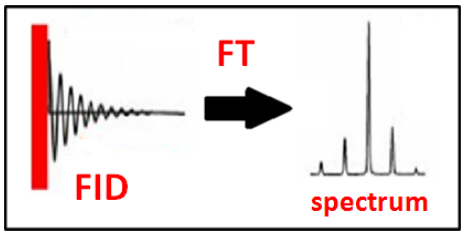
When a nucleus is placed in an external magnetic field B_0, magnetic moments align parallel and anti parallel to the applied field. There are two energy levels where the magnetic moment of nuclei may fall into.



There is a slight excess of nuclear moments aligned parallel to the applied field which are in lower energy level (α). By irradiating with the proper frequency ν, lower energy state (α) is flipped to higher energy state (β) with the absorption of energy. As a result of this, resonance condition is observed for nucleus. This resonance is used for nuclear magnetic resonance spectroscopy and nuclear magnetic resonance imaging.

The sensitivity of NMR depends on the ratio of the number of protons distributed between these two energy levels according to Boltzman distribution. When more protons are resonanced, this increases the power of NMR signal that much.

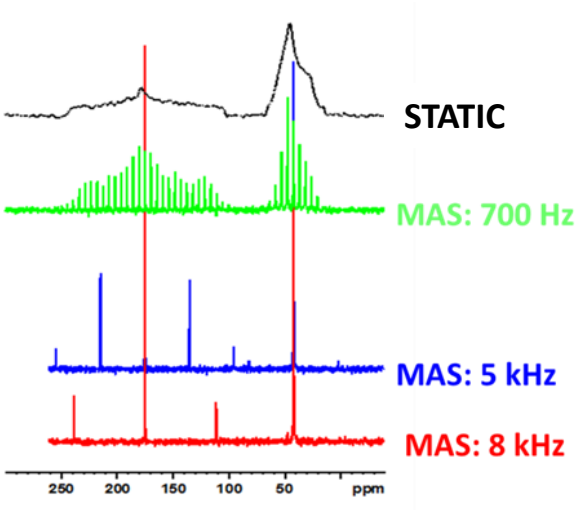
Once excited to the higher energy state by a radiofrequency (RF) pulse, the spins will return to their initial equilibrium condition by means of relaxation mechanisms. When this happens, at first a time domain emission signal is recorded and then a frequency domain NMR spectrum is obtained by Fourier Transformation (FT).



Different from Liquid NMR spectrometer, Solid State NMR spectrometer is used in NMR laboratory to analyze samples which are insoluble or those wanted to be analyzed in solid form due to their structures. In Solid State NMR, the nuclei of solid samples are very close to each other and there are interactions among them. This decreases the resolution of the spectrum and as a result of this, peaks are getting broader.

In order to eliminate these problems, there are some techniques used in Solid State NMR. These are; MAS (magic angle spinning) and CP (cross polarization) methods. MAS is shortly a method where spinning is applied to magnetic field at a certain “magic angle” to obtain a beter solid NMR spectrum. Whereas in CP, NMR signal of 13C nucleus is inreased by using cross polarization method.

In addition to this, peaks can be obtained in better resolution by spinning the samples at higher frequencies.



LIQUID NMR

High Resolution Digital 300 MHz NMR Spectrometer - Bruker Biospin

Probe: 5 mm BBO 1H, 13C, 31P, 19F

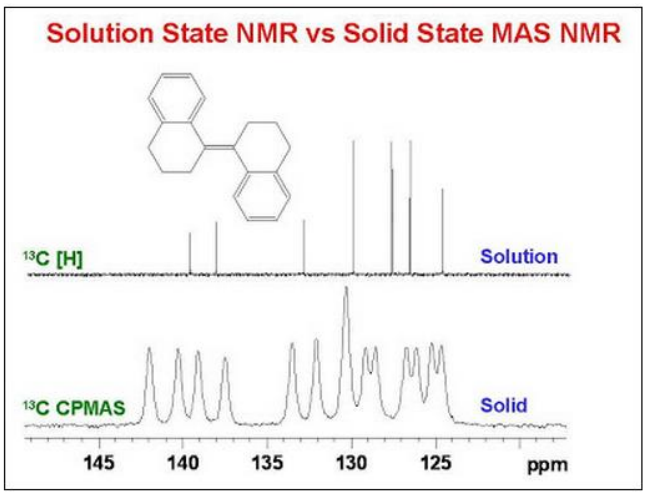
Experiments: 1D: 1H, 13C, 31P, 19F nuclei, dept-45, dept-90, dept-135.

Sample Requirements: Pure samples must be supplied with indication of solvent. For 13C, minimum 40 - 50 mg of sample, for 1H, minimum 10-20 mg of sample is required.

Deuterium lock solvents available are: Chloroform-D1, Dimethyl sulfoxide-D6, Deuterated water, Acetone-D6, Benzene-D6, Toluene, Pyridine, Dimethyl formamide, dichloromethane-D2.

MATERIALS

High Resolution Liquid NMR spectrometer is used to analyze different samples either in solid, powder or liquid form from petroleum, chemical, food, drug and pharmaceutical industries after solving them in proper deuterated solvents.



SOLID NMR

High Power Solid State 300 MHz NMR Spectrometer - Bruker Superconducting FT-NMR Spectrometer Avance TM 300 MHz WB

Probe: 2.5 mm MAS, 4 mm MAS , 7 mm MAS

Experiments: NMR spectrum of 13C, 29Si, 27Al and some other nuclei, MAS (Magic Angle Spining) and CP (Cross Polarization) techniques.

Sample Requirements: Dry and pure powder solid samples at least 250 mg for 2.5 mm probe, 500 mg for 4 mm probe, 1000 mg for 7 mm probe. Samples containing metals are not accepted for NMR analysis due to their unwanted effects on NMR magnets.

MATERIALS

In solid NMR the samples which are insoluble in certain solvents or the samples whose chemical structure may be destroyed due to solving process can be analyzed.

Apart from the liquid NMR analysis, the solid state NMR allows one to analyse organic crystalline compounds, catalysts, zeolites, amorphous compounds, liquid crystals, polymers and biopolymers.

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