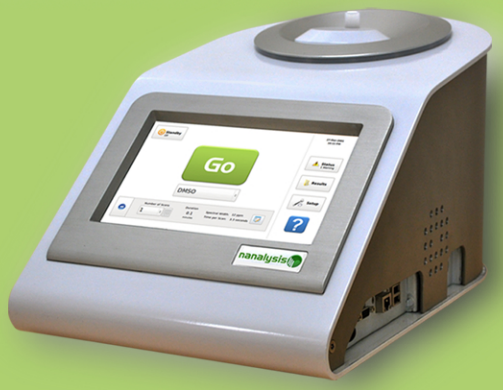
Introduction to NMR Spectrometers: An Instrumental Analysis

Incorporating instrumentation into undergraduate laboratories increases students' understanding of fundamental and practical characterization techniques.

The NMReady™ benchtop spectrometer offers a portable and affordable option with a modern, network accessible, easy-to-use interface that can be easily incorporated into teaching laboratories.



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Introduction to NMR Spectroscopy:

Nuclear Magnetic Resonance (NMR) Spectroscopy is one of the premier spectroscopic techniques avaliable for molecular structure identification. Information is obtained in form of a ***spectrum*** (plot of absoprtion frequency *vs.* intensity) when an analyte is placed in a magnetic field (B_o) & subjected to radio frequency (RF) irradiation. The spectrum of a sample is dependent on the nuclei present & the molecular structure of the analyte.

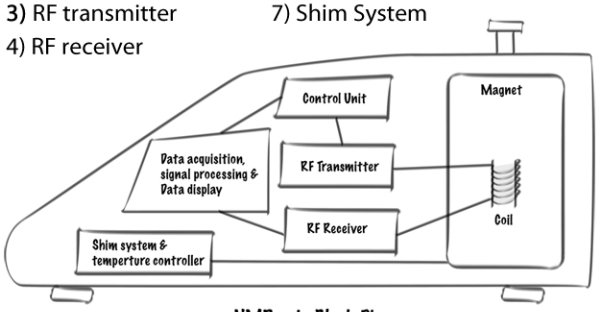
Although most commonly used for organic molecules containing ^1H & ^{13}C, any atomic nuclei with a ***nonzero spin*** can be exploited to determine chemical structure, relative configuration, etc without degrading the analyte. Modern NMR spectrometers are programmed with standard parameters, but a basic knowledge of the parameters & instrumentation is vital to obtaining optimal results.

The NMReady™ benchtop spectrometer provides an accessible medium for training both chemistry & chemical technician students about the fundamental parameters of NMR spectroscopy in a guided-discovery laboratory experiment.

Instrumentation:

NMR Spectrometers are made up of several components, the connectivity of which are illustrated below.

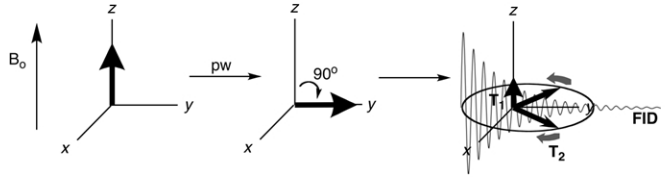
1) Magnet
2) Probe/coil
3) RF transmitter
4) RF receiver
5) Data acquisition/processing
6) Control Unit
7) Shim System



NMReady Block Diagram

Theory of Data Collection:

Nuclear spins tend to align in an external magnetic field B_o (*z* direction), a slight thermodyamic excess of parallel spins produce a net magnetization in the sample. When an RF pulse (pw) is applied, the nuclei are excited (rotated into the *xy* plane). The nuclei will ***precess*** & relax back to the equilibrium state through lattice (T_1) & spin-spin (T_2) relaxation processes giving rise to an oscillating, decaying sine wave called a free-induction decay (FID) (plot of emitted RF *vs.* time).



Procedure:

Allow the students to learn about & manipulate critical parameters with the NMReady One-touch software as they work through the procedure and related questions below.

1) Spectrometer Frequency - characteristic to nuclei & applied field of spectrometer. For ^1H NMR & the 1.41 T field of the NMReady this is 60 MHz.

$$v_0 = \left(\frac{\gamma}{2\pi}\right) B_o$$

where γ = magnetogyric ratio (property of nuclei that determines precession frequency. For ^1H $\gamma = 267.5 \times 10^6$ rad/s)
B_o = static magnetic field applied by magnet

2) Pulse Width (pw) - described by the angle (θ) the net magnetization is rotated through when RF pulse is applied. Power is applied for a time (tp in μs) to rotate to the desired angle, so pw can be discussed in terms of angle or time. The NMReady defaults to a 90° pulse.

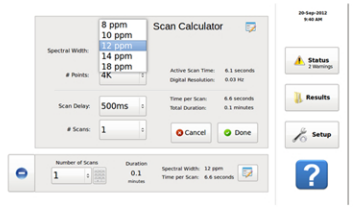
$$\theta = 360 \left(\frac{\gamma}{2\pi}\right) B_1 tp$$

where tp = time of pulse (μs)
B_1 = RF magnetic field applied by the coil

$$v_1 = \left(\frac{\gamma}{2\pi}\right) B_1$$

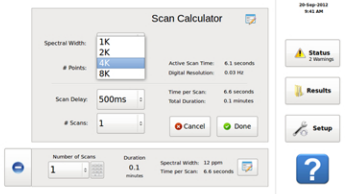
(i) What is the tp for a 90° pulse when B_1 = 290 uT? (ii) 180° ?

3) Spectral Width (sw) - (or ***sweep width***) frequency range analyzed by the spectrometer. It can be reported in ppm or Hz. ppm is independent of spectrometer frequency but Hz is not.



(iii) What is the typical range of chemical shifts for protons?
(iv) Given this information, what is a typical sw required for routine analysis of organic compounds?
(v) For a sw = 12 ppm, what is the range in Hz for a 60 MHz spectrometer versus a 400 MHz?
(vi) Changing only the sw determine what happens to the active scan time.

4) Number of Points (np) - controls the ***digital resolution (res)*** of the measured FID. ***Nyquist theorem*** states the analog signal must be sampled at a rate >sw to ensure each peak is properly reporduced in the spectrum.



Generally more points = higher resolution (res)

Typically the resolution must be greater than or equal to 1/2 of the peak line width (in Hz) at 50 % (LW_{50}).

$$res = \frac{2sw}{np}$$

(vii) For ^1H NMR spectra measured with SW = 12 ppm & a LW_{50} = 2.2 ppm on the NMReady what is the minimum np required?