5.3: Instrumentation

Objectives

After completing this section, you should be able to:

- have basic understanding of how the NMR works
- understand the basic components of the NMR spectrometer

NMR spectroscopy works by varying the machine's emitted frequency over a small range while the sample is inside a constant magnetic field. Most of the magnets used in NMR instruments are superconducting to create the magnetic field range from 6 to 24 T. The diagram below is of an NMR spectrometer. In a basic experiment, an organic sample is dissolved in a suitable solvent, which is often deuterated chloroform or another deuterated solvent, and placed in a thin glass tube. A reasonable concentration for your sample is 5 to 25 mg of desired compound dissolved in 0.6 to 1.0 mL of a suitable solvent, which should be free of particulate matter for solution NMR experiments. The sample is inserted through the NMR spinner and pneumatically lowered into the NMR probe, which is between two magnetic poles. The coils around the NMR tube in the diagram below is where the sample must be. NMR experiments require a uniform magnetic field over the whole of the NMR sample volume that sits within the detection coil. Deviation from this ideal introduces various line shape distortions, compromising both sensitivity and resolution.The strong magnetic field causes the ^{1}H nuclei (or other NMR active nuclei in other experiments) in a molecule to align in one of the two possible orientations. The sample is then subjected to a frequency from the radio wave source. A detector then interprets the results and sends it to the main console. Using a data analysis program, the free induction decay (FID) data is transformed into the spectrum typically shown in papers and books.

Diagram of NMR spectrometer

NMR Instrument

An NMR can be divided into three main components: the workstation computer where one operates the NMR instrument, the NMR spectrometer console, and the NMR magnet, which is shown in the picture below.

400 MHz NMR Spectrometer

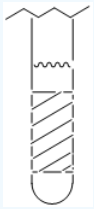
The NMR is layered with the superconducting magnet just outside the probe towards the center of the spectrometer (diagram below). The magnet only works if a coil is kept very cool, so it is immersed in the first layer inside the NMR, which is liquid helium (4.2 K). To reduce the boil off rate of the liquid helium, the next layer is filled with liquid nitrogen at 77 K. The liquid nitrogen reservoir space is mostly above the magnet. This way, it can act as a less expensive refrigerant to block infrared radiation

from reaching the liquid helium jacket. Aluminum and stainless steel are also used to contain the liquids and block infrared irradiation as well. All the layers are working to keep the magnet coil very cold.

Diagram of the main layers inside an NMR instrument

? Exercise 5.3.1

Consider a sample in an NMR tube.The crosshatched region in the tube is the area over which signal is recorded.Why is it important that B_{APPL} be homogeneous over this entire region?



Answer

NMR experiments require a uniform magnetic field over the whole of the NMR sample volume that sits within the detection coil, which is represented by the crosshatched region. Deviation from this ideal introduces various line shape distortions, compromising both sensitivity and resolution.
