Molecular Science - Spectrometry

Nuclear Magnetic Resonance

This is a modern high-field nuclear magnetic resonance spectrometer which observes protons at 300 MHz and carbon-13 at about 75 MHz. And to do this, we need to generate a large magnetic field of about 7 Tesla.

So this is produced by a super-conducting magnet which essentially a very efficient cryostat is containing a solenoid immersed in a bath of liquid helium. And careful design of this cryostat including the use of an outer jacket of liquid nitrogen, means that the liquid helium, which is very expensive, only has to be topped up every four months or so.

The main body of the spectrometer contains the hardware for generating the radio frequency radiation and for receiving and detecting the NMR signals. Finally, there's the computer which controls all the functions of the spectrometer. And of course it also analyses the data and displays the results.

Careful sample preparation is an important part of any NMR experiment. We need to dissolve the sample in a suitable solvent and between 1 and 10 milligrams is adequate for a proton NMR work. However, for carbon-13 spectra, we need to use as concentrated a solution as possible because of the low sensitivity of the carbon-13 nucleus. Deuteron-chloroform is often the solvent of choice. The reference compound for either proton or carbon-13 NMR is tetramethylsilane, that's TMS for short. And a few drops are added to the solution. A minimum depth is required to give adequate resolution. A tight fitting cap is required since both solvent and TMS are volatile. The sample is now ready for the NMR spectrometer.

The first thing to do is to insert the sample into a spinner turbine, and adjust to a pre-determined optimum position. It's this turbine, powered by compressed air that allows the sample to be spun once it's in the NMR probe. This particular instrument has an auto-sampler attachment and it's particularly useful for running a batch of samples overnight. We can also use it to load our sample. The sample, once delivered to the top of the cryostat, is gently lowered on a bed of compressed air into the body of the magnet, and so into the NMR probe.

It's now just a matter of setting the parameters for the experiment in the computer. We're going to run a broad band decoupled carbon-13 NMR spectrum, but we'll need to accumulate quite a few spectra and add them together in order to get a reasonable spectrum without too much noise. Overall it will take about 10 to 20 minutes. The final result is a familiar carbon-13 stick spectrum, and there are four distinct carbon-13 resonances. And this is consistent with the chemical structure of our sample - it's ethyl acetate, or more correctly ethyl ethanoate. And this is clearly the carbon of the carboxylate group.

So this instrument can carry out many different experiments, all of which are designed to help in unravelling the structures of often complex molecules.