

Samples are carried out in solution and so a solvent is required, however, since we are measuring using hydrogen and carbon isotopes, this solvent cannot contain carbon or hydrogen or else it would produce a signal of its own. Instead, a solvent using the deuterium isotope of hydrogen is used, known as a deuterated solvent, e.g. CDCl_3 . This does not produce a signal as there are an even number of particles in the nucleus, so all particles with opposite spins are paired up. In a carbon-13 spectrum, the signal produced by the carbon in the solvent is easily removed, and afterwards the solvent can easily be evaporated off to recover the original sample.

In carbon-13 NMR spectroscopy, the carbon isotope is used to identify the different environments that carbons in a sample are in. The number of different peaks produced will correspond to the different environments, and the chemical shift value of a peak will indicate what kind of environment this is, however, different environments can have overlapping ranges of chemical shift values, so it is not always obvious what environment a peak is indicating.

Environments are only the same when everything around them is exactly identical, not just the atoms bonded to the carbon: this will only happen in molecules which are symmetrical. A central atom bonded to four methyl groups will produce only one peak, as each carbon is an equivalent environment. A chain of five carbons with an OH group at one end will produce 5 different peaks, as each carbon is a different distance away from the OH group, and therefore each carbon will have a unique environment. By counting the number of environments in a molecule, it is fairly straightforward to be able to match it up with a spectrum by counting the number of peaks produced. A molecule with 3 different environments can only correspond with a spectrum with 3 different peaks. The chemical shift value of the peaks can then be used to identify the nature of the carbon environment, for example, if the peak has a chemical shift value roughly between 190-220 δ/ppm , then the carbon in question is joined to an oxygen atom with a double bond, $\text{C}=\text{O}$.

NMR spectroscopy using protons (^1H nuclei) investigates hydrogen environments, as opposed to carbon environments. The key aspects are the same, however, proton NMR spectroscopy also gives additional information that carbon-13 does not. The heights of the peak are proportional to the number of protons in the environment, so the peak heights can be used to determine the ratio of hydrogens in each environment. The splitting patterns of the peaks can also be used to determine the number of hydrogens adjacent to the environment – this arises because of something called “spin-spin coupling”. As you know, protons can spin either up or down, and normally particles with opposite spins would pair up. Protons adjacent to each other can align their spin states in different ways – there are many different combinations, e.g. you could have two up, or two up and one down, or one up and one down. These different magnetic fields generated will cause what would otherwise be a single peak to be split into several, depending on the number possible combinations. This is where the $n+1$ rule comes in: for n protons on an adjacent carbon, the number of peaks in a splitting pattern is $n+1$. In other words, if there are n protons on a carbon adjacent to a particular environment, there will be $n+1$ peaks generated by that environment. For example, if a triplet peak (three peaks) is generated, there is a carbon with two protons on it adjacent to the environment generating the triplet.

The last key feature of proton NMR spectroscopy is the spectra of samples containing OH and NH groups. These groups are very difficult to see on a spectrum, as the range of chemical shift values is incredibly wide and overlaps with many other environments, and there is usually no splitting pattern. Peaks generated by the protons in these groups would make it very difficult to read a spectrum without mistakes, so these peaks are filtered out using D_2O , deuterium oxide. This is a water molecule with the deuterium isotope instead of hydrogen. As you already know, deuterium will not produce a peak, as it has an even number of particles in the nucleus. The deuterium will