2.3: What sample considerations are important?

What nucleus should I detect?

Just as you might make a choice between measuring a UV or an IR spectrum, in NMR we often have a choice in the nucleus we can use for the measurement. A wide range of nuclei can be measured, with the spin ½ nuclei ^{1}H, ^{31}P, ^{13}C, ^{15}N, ^{19}F, ^{29}Si, and ^{31}P among the most common. However, most quantitative NMR experiments make use of ^{1}H, because of the inherent sensitivity of this nucleus and its high relative abundance (nearly 100%). In addition, as we will see in the next section, the relaxation properties of nuclei are also important to consider in quantitative NMR experiments, and compared with many other nuclei like ^{13}C, ^{1}H nuclei have more favorable T_1 relaxation times. The choice of the observe nucleus can depend on whether one seeks universal detection (for organic compounds ^{1}H and ^{13}C fall into this category) or selective detection. For example fluoride ions can be easily detected in fluorinated water at the sub-ppm level, in large part because of the selectivity of the measurement – one expects to find very few other sources of fluorine in water. Similarly phosphorous containing compounds like ATP, ADP, and inorganic phosphate can be detected and even quantified in live cells, tissue or organisms.

How concentrated is my sample?

In the Beer-Lambert law you are probably familiar with from UV-visible spectroscopy, absorbance is directly related to the concentration of the analyte. Similarly, in NMR the signal we detect scales linearly with concentration. Since NMR is not a very sensitive method, you would ideally like to work with reasonably concentrated samples, for protons this means analyte concentrations typically in the millimolar to molar range, depending on the instrument you will be using. Other nuclei are less sensitive than protons. The sensitivity issue has two components, the inherent sensitivity, which depends on the magnetogyric ratio (γ), and the relative abundance of the nucleus (for example ^{19}F is 100% abundant, but ^{13}C is only 1.1% of all carbon atoms)

What other practical issues do I need to consider?

The sensitivity of an NMR experiment can also be affected by the homogeneity of the magnetic field that the sample feels. It is normal to adjust the field homogeneity through a process known as shimming. NMR samples should be free of particulate matter, because particles can make it difficult to achieve good line shape by shimming. You will also have better luck with shimming if you have a sample volume sufficient to meet or exceed the minimum volume recommended by your instrument manufacturer.