Mimicking a GHz NMR

- Applying auxiliary agents for structure verification

For most NMR applications, higher external magnetic field strengths are preferred for better dispersion and sensitivity, but are usually outside the budgets of educational labs. The good news is the Fourier 80 can do the job with a little help from auxiliary agents and Bruker's step-by-step protocols designed for students.

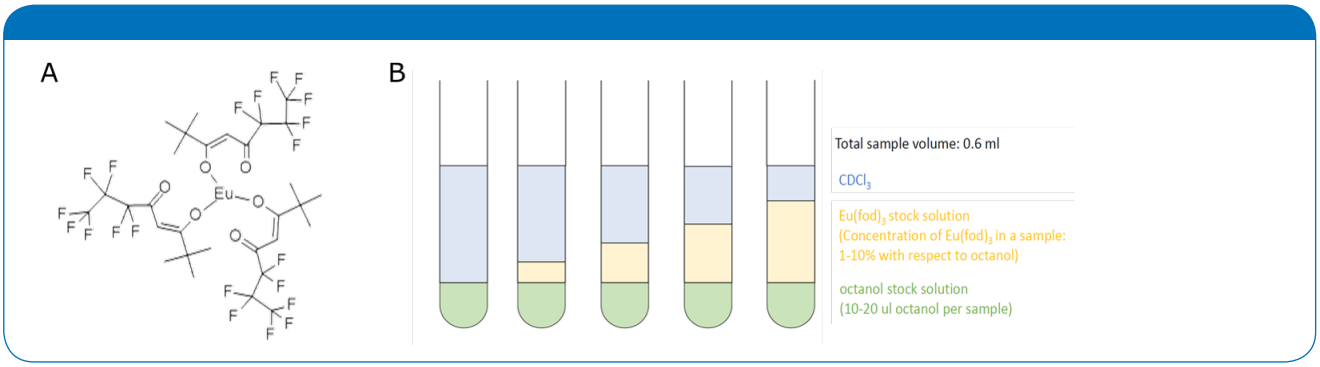
Performing this experiment, students will learn in detail about chemical agents that can be used to manipulate the behavior of NMR-active nuclei and how and when to make use of those effects.

Key Learnings

- Applying auxiliary agents
- Understanding dispersion
- Setting up a homonuclear 2D COSY experiment

What are auxiliary agents used for?

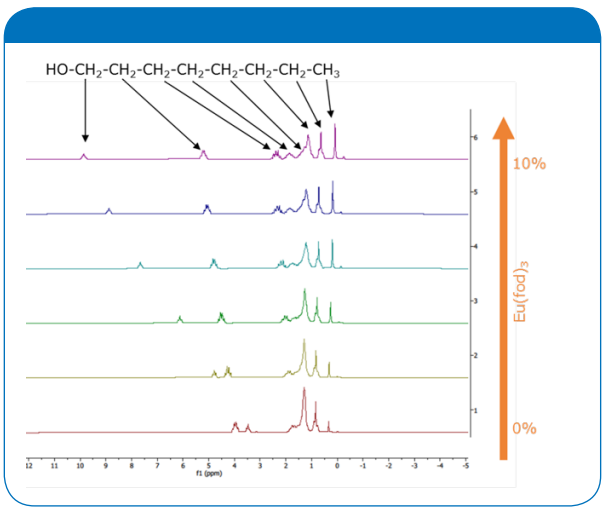
When using NMR, it is sometimes useful to manipulate the spins of a sample. Most common are paramagnetic auxiliary (also known as 'doping') agents that have an accelerating effect on relaxation times, which allow the NMR spectroscopist to overcome long delays between experiments. Another example of how an auxiliary agent can be applied is shown in this protocol using an europium complex.



(A) Structure of the auxiliary agent $Eu(fod)_3$, an europium complex with three fluorinated di-keton ligands. (B) Samples prepared contain of octanol in $CDCl_3$ with increasing concentration of $Eu(fod)_3$ from zero to ten percent.

How to mimic a GHz spectrometer?

In NMR spectra, paramagnetic complexes cover a large chemical shift region (e.g. 1000 ppm in ^1H spectra), because their unpaired electrons have a strong influence on the chemical shift. Therefore, assigning those NMR spectra can be very challenging and not very straightforward. However, we can make use of this effect for molecules featuring unpaired electrons to enhance the chemical shift differences of protons within one molecule. When the molecule binds to such a paramagnetic complex, protons are shifted downfield (to the left of the spectrum). The proximity of the proton to the binding site determines the extent of the effect. In such a way it is possible to enhance dispersion - and this is exactly the effect we will use to mimic a GHz spectrometer.



While the $Eu(fod)_3$ complex itself only shows one singlet (tert-Butyl group), the alkalic signals of the octanol are shifted more downfield the closer the protons are to the metal center. The effect scales with the $Eu(fod)_3$ concentration.

Where to find more information

This, as well as many other experiments, are included in the Fourier EduLab. Next to a detailed step by step protocol for students, we also deliver an instructor's guide featuring additional information. **Contact us for details!**