Applications of a Benchtop NMR Spectrometer in an Undergraduate Chemistry Laboratory

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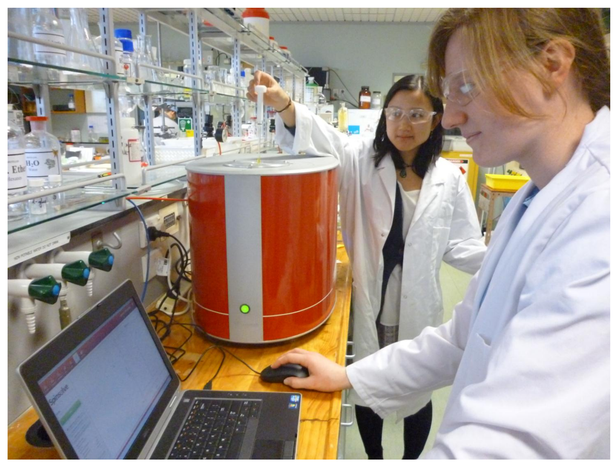
The Magritek Spinsolve benchtop NMR spectrometer.

Introduction

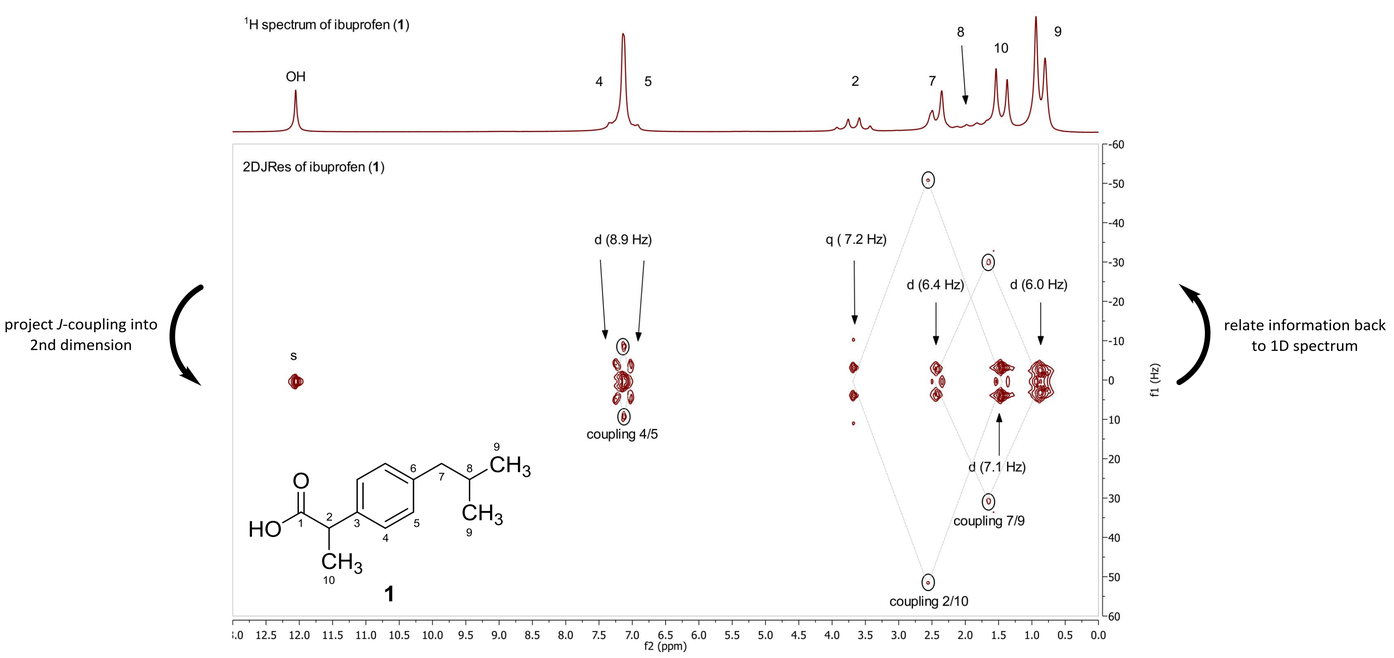
Teaching NMR spectroscopy in undergraduate chemistry programmes is fast becoming a more routine practice – particularly with the advent of benchtop NMR spectrometers. Unlike traditional superconducting spectrometers, this new class of NMR spectrometer is sufficiently robust, safe, and stable to be deployed directly in a typical chemistry laboratory and simple enough to be easily operated by students. We have been using the Spinsolve benchtop NMR spectrometer, manufactured by the New Zealand company Magritek, over the last six months and are exploring avenues in which to integrate the use of this instrument and its range of 1D- and 2D-NMR experiments into our undergraduate chemistry teaching programmes.



The Magritek Spinsolve benchtop NMR spectrometer in action.

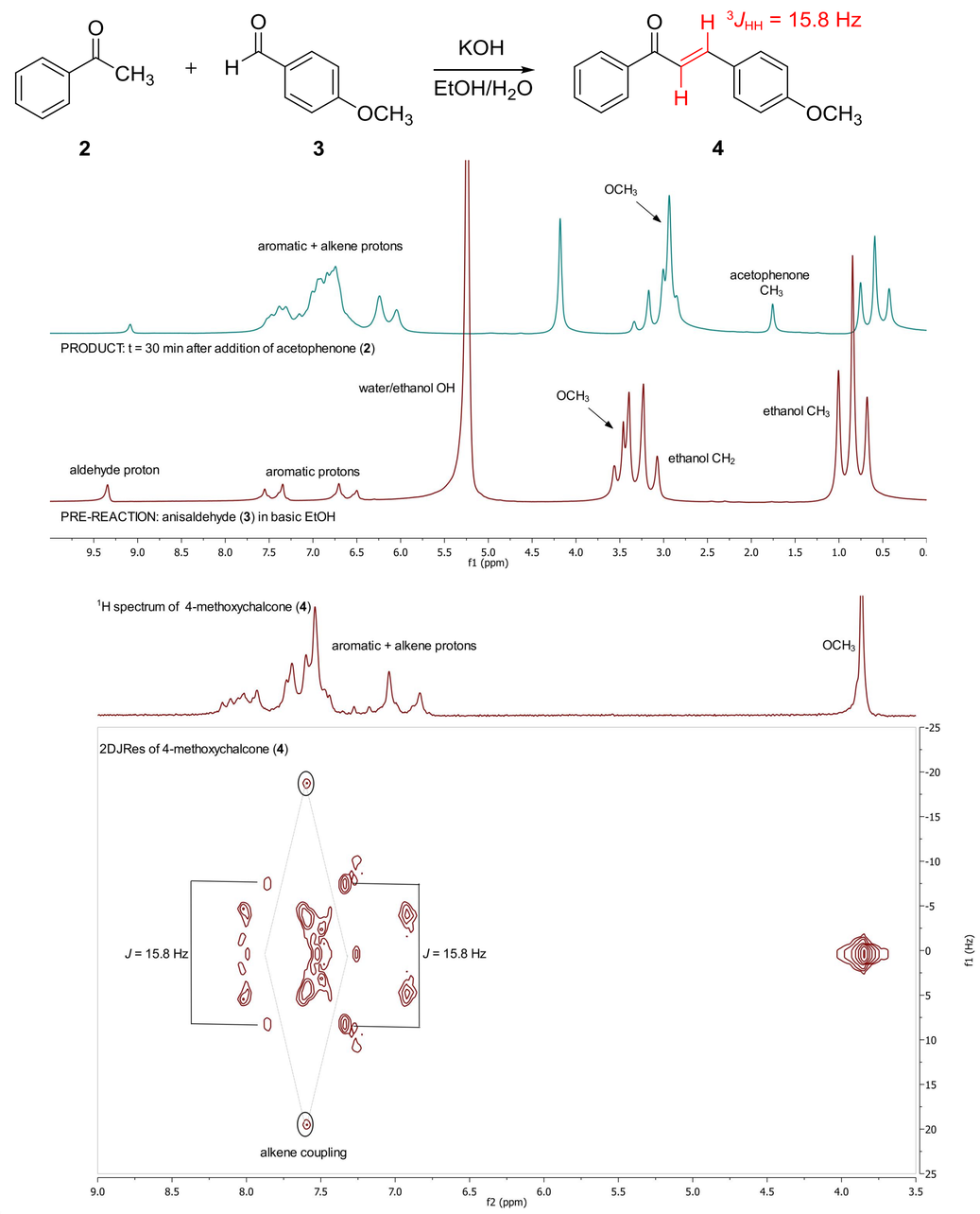
First Year: 2D-NMR as a Teaching Tool

Interpretation of ^{1}H NMR spectra is often the most difficult aspect for students to learn, especially when factors such as *J*-coupling, peak overlap and second-order effects dominate the spectrum. By introducing the 2D *J*-resolved experiment at an earlier stage of teaching, students are able to observe these effects independently by placing *J*-coupling on to the second dimension. As demonstrated with ibuprofen (**1**), the precise chemical shifts and multiplicities of all protons (with the exception of H-8) can be assigned. The presence of equidistant satellites are indicative of through-bond coupling.



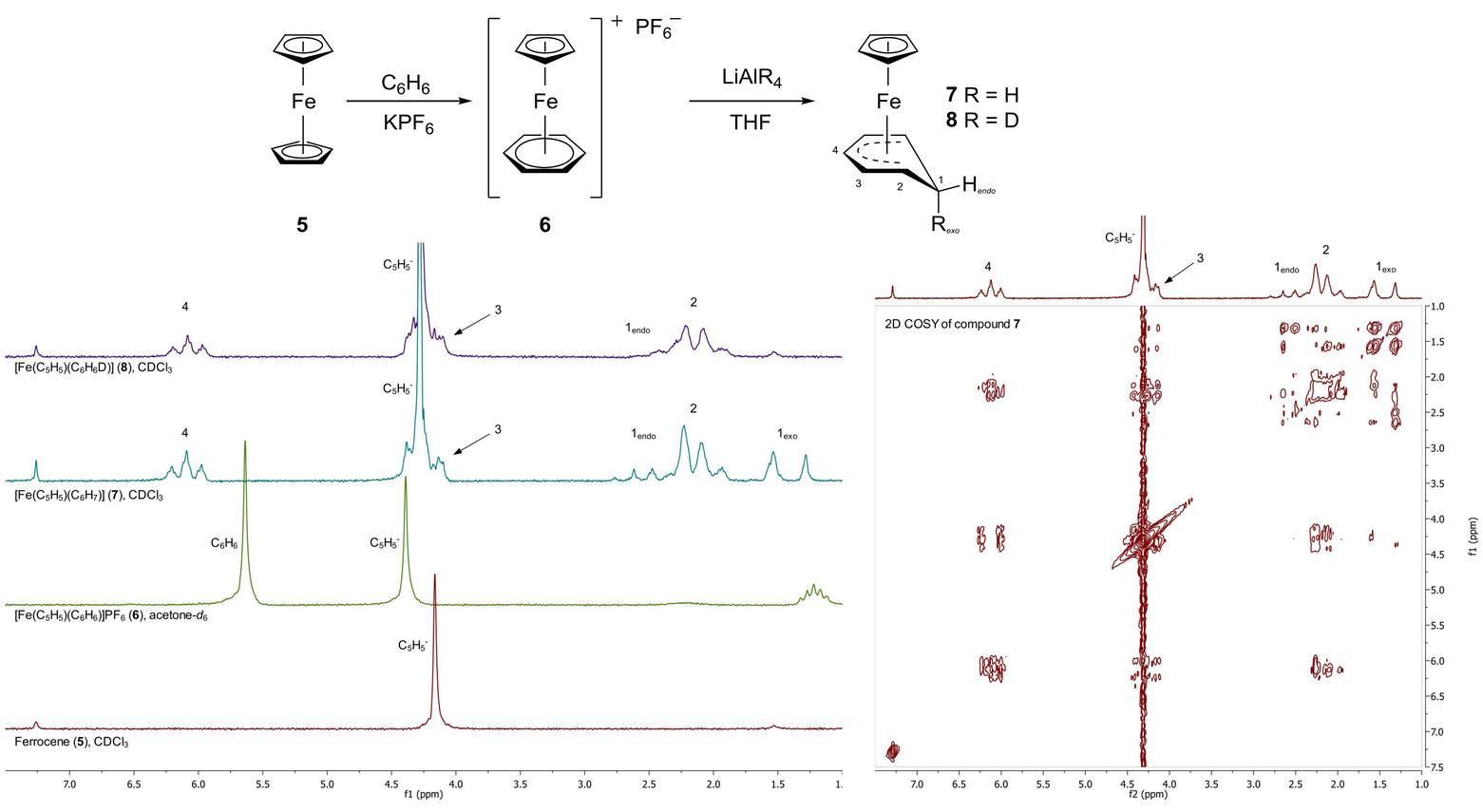
Second Year: Reaction Monitoring

The ability to monitor chemical reactions in real-time provides valuable information about synthetic processes. For example, acetophenone (**2**) and anisaldehyde (**3**) are reacted in ethanolic KOH to produce 4-methoxychalcone (**4**). By performing the reaction in the NMR tube, students can determine when the reaction is sufficiently complete before proceeding with work-up and purification. Furthermore, the geometry about the alkene can be determined by analyzing the 2D *J*-resolved spectrum of the purified compound.



Third Year: Advanced Experiments and Interpretation

NMR spectroscopy is an important tool in the understanding of chemical principles. Starting with the π-complex ferrocene (**5**), one cyclopentadienyl anion is substituted for a benzene molecule to form cationic species **6**. Nucleophilic attack on the benzene ring in compound **6** using $LiAlH_4$ affords the neutral η^5-C_6H_7 complex **7**. The analogous reaction with $LiAlD_4$ shows that the attack occurs on the *exo* face of the benzene ring to form complex **8**. Analysis of ^{1}H and COSY spectra acquired on the Spinsolve allows detailed structural information to be at hand within minutes of sample preparation.



Future Directions

We have demonstrated how the incorporation of a benchtop NMR spectrometer can enhance both the teaching and learning experience in the laboratory. The ease of use and accessibility of the Spinsolve means we can approach existing experiments in a different manner, introduce other experiments which employ NMR methods (*e.g.* kinetic studies), or utilize other NMR-active nuclei such as ^{13}C and ^{19}F.

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