Undergraduate education with benchtop NMR

Spinsolve is the perfect way to provide NMR as part of an undergraduate teaching program. The high sensitivity and advanced capability means it is fast and informative, critically important for a busy class. Spinsolve provides convenient, high performance NMR at a fraction of the cost of traditional NMR systems.
In this second-year chemistry experiment, students synthesise \( p \)-nitroaniline through the hydrolysis of \( p \)-nitroacetanilide.

Spinsolve NMR spectra were acquired within a few minutes of sample preparation. Comparison measurements were also made on a standard 300 MHz instrument.

Figure 1 shows the \(^{1}H\) NMR spectra of 200 mM solutions of starting material (red) and final product (blue) in DMSO-\(d_6\). The disappearance of the methyl peak (CH\(_3\)) and the conversion of the NH peak from 10.5 ppm into a NH\(_2\) at 6.6 ppm are evidence for a successful reaction. The results show that there is no loss of chemical information by using a lower NMR frequency.

- Spinsolve is fast: students obtain informative spectra within a minute of sample preparation.
- Convenient: it’s in the lab, on the bench, next to the students.
- Students can work with concentrations of tens to hundreds of mmol due to the superb sensitivity.
- Spinsolve 43 MHz spectra provide similar chemical information as high field.

Save time by taking NMR measurements in the teaching lab

The cost and ease of access to high-field NMR spectrometers is often cited as a main limitation to the practical use of NMR in undergraduate teaching programs. Due to the superb spectral resolution of the Magritek Spinsolve, most small molecules synthesised in chemistry teaching laboratories can be analysed right there at low field without loss of chemical information.

• Resolution: Spinsolve spectra provide similar chemical information as at 300 MHz
• Sensitivity: Students can work with concentrations of hundreds of mmol
• Convenient: It’s in the lab, on the bench, next to the students.
• Fast: Students obtain informative spectra within a minute of sample preparation.
Reduce cost

As well as being less expensive than high field NMR, Spinsolve reduces costs because expensive deuterated solvents are not necessary.

In this second-year chemistry experiment, students synthesise \( p \)-nitroacetanilide through the nitration of acetanilide.

Figure 2 shows the NMR spectra of starting material (red) and final (blue) product, in deuterated and non-deuterated DMSO.

Although there is a large peak for the non-deuterated solvent, all peak assignments can still be made for the molecule.

- Reduce cost - deuterated solvents are not necessary.
- Low cost budget NMR tubes can be used.
- Samples can be analysed in native solutions.
- Solubility issues are resolved – any solvent can be used.
- Enables in-line monitoring of reactions.

![Figure 2](image-url)
Easy to run

Setting up different NMR experiments can be a challenge for someone new to NMR. The simple Spinsolve user interface makes this easy.

For example, many inorganic complexes are paramagnetic ions. These extend the chemical shift range of proton spectra to hundreds of ppm. The Paramagnetic protocol in the Spinsolve software is particularly tailored for scanning such samples and is easy to run.

This example from a third-year chemistry experiment demonstrates the reaction of coordinated ligands using the conversion of paramagnetic $[\text{Co(phen)}_3]^2^+$ to diamagnetic $[\text{Co(phen)}_3]^3^+$ complexes.

Figure 3 shows how the chemical shift range differs dramatically between the paramagnetic and diamagnetic cobalt complexes.

*Spectra courtesy of Paul S Donnelly, University of Melbourne.*

- Simple to use interface permits students to run different experiments.
- Paramagnetic samples can be analysed easily.
- Advanced 2D NMR experiments are simple to run.
- Students can also measure $^{19}\text{F}$ spectra with the click of a button.
Advanced NMR

When first being exposed to NMR, many students find it difficult to understand and distinguish between chemical shift and $j$-coupling. Two-dimensional NMR experiments project additional information into a second dimension, which tremendously facilitates the interpretation of NMR spectra.

As an example, we consider the $^1$H NMR spectrum of ibuprofen dissolved in CDCl$_3$. The COSY experiment is used to assign which proton resonances are coupled to each other. Cross-peaks in the 2D spectrum indicate through-bond coupling. The COSY spectrum for ibuprofen is shown in Figure 4.

From the cross-peaks two spin systems are observed: CH-$2$/CH$_3$-$10$ and CH$_2$-$7$/CH$_3$-$9$.

- The COSY experiment identifies the proton signals from magnetically coupled chemical groups.
- 2D COSY is one of the most commonly used 2D NMR experiments.
- Takes 10 minutes to run, ideal for a teaching laboratory.

Figure 4

Advanced NMR understanding with two-dimensional experiments

When first being exposed to NMR, many students find it difficult to understand and distinguish between chemical shift and $j$-coupling. Two-dimensional NMR experiments project additional information into a second dimension, which tremendously facilitates the interpretation of NMR spectra.

2D NMR experiments can now be easily incorporated into laboratory teaching experiments. This enables students to use 2D experiments in order to better interpret 1D NMR spectra.

- The 2D homonuclear $j$-decoupled experiment separates the $j$-multiplets from chemical shift.
- Students experience couplings "hands-on".
- The 2D homonuclear $j$-decoupled spectrum of ibuprofen immediately clarifies the peak multiplicities.

Student example

As an example, we consider the $^1$H NMR spectrum of ibuprofen dissolved in CDCl$_3$. The COSY experiment is used to assign which proton resonances are coupled to each other. Cross-peaks in the 2D spectrum indicate through-bond coupling. The COSY spectrum for ibuprofen is shown below.

From the cross-peaks two spin systems are observed: CH-$2$/CH$_3$-$10$ and CH$_2$-$7$/CH$_3$-$9$.

The 2D homonuclear $j$-resolved experiment can be used to gain information about chemical shift and $j$-coupling, especially where spectral overlap makes it difficult to resolve. The $j$-resolved spectrum is shown below, along with the peak assignment and the 1D spectrum in blue. The experiment clearly confirms the doublets at 1.02, 1.62 and 2.60 ppm, quartet at 3.84 ppm, and multiplets at 2.00 and 7.30 ppm.
Fluorine NMR now available for Magritek Spinsolve

Why use Fluorine NMR?
• The 19F nucleus is one of the most important nuclei for NMR spectroscopy
• Due to its 100% natural abundance and high frequency its NMR sensitivity is very high
• Organofluorine compounds are often used in the pharmaceutical industry
• Fluorine is often used as a molecular tag

How does it work?
• Coming in version 1.1 of the Spinsolve software, we will offer a 1D Fluorine protocol
• No hardware changes are required, old systems can be upgraded easily
• Proton and Fluorine experiments can be run on the same sample, without further adjustments

Example spectra

Spinsolve software is beautiful and intuitive which means it can be easily operated by anyone in the chemistry lab with minimal training. Most experiments can be run with a single click of a button. Switching between experiments also involves a single click. This simple, easy to use operation reduces time spent learning how to operate the system and increases throughput.

The screenshot above shows the new 19F capability built into the latest version of the Spinsolve software.
"Now the students are able to acquire their own NMR spectra as well as carry out the analysis of the compounds they have made. This makes their undergraduate experiment more applicable to both research and industry settings and increases their enthusiasm for chemistry."

- Professor Frances Separovic, Head of Chemistry at the University of Melbourne
Why the Spinsolve will suit your education needs:

Reduce cost
• Low cost to purchase compared to high field.
• Non-deuterated solvents can be used.
• Low power consumption.
• Budget NMR tubes can be used.

Save time
• Nearby - fits easily on laboratory bench.
• Standard 5 mm NMR tubes enables rapid sample exchange.
• Fast - students get on and off quickly.
• Easy to use - simple and intuitive software.
• Safe - no stray magnetic field.

Informative
• Enables leading NMR education.
• Obtain high-resolution NMR data in as little as 10 seconds.
• Now available with 2D, multi-pulse experiments (2DJRes, COSY) and $^{19}$F Fluorine.
• Students and teaching staff gain hands-on experience with NMR.

Contact us now for a quote, to request a demo or to measure your samples

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