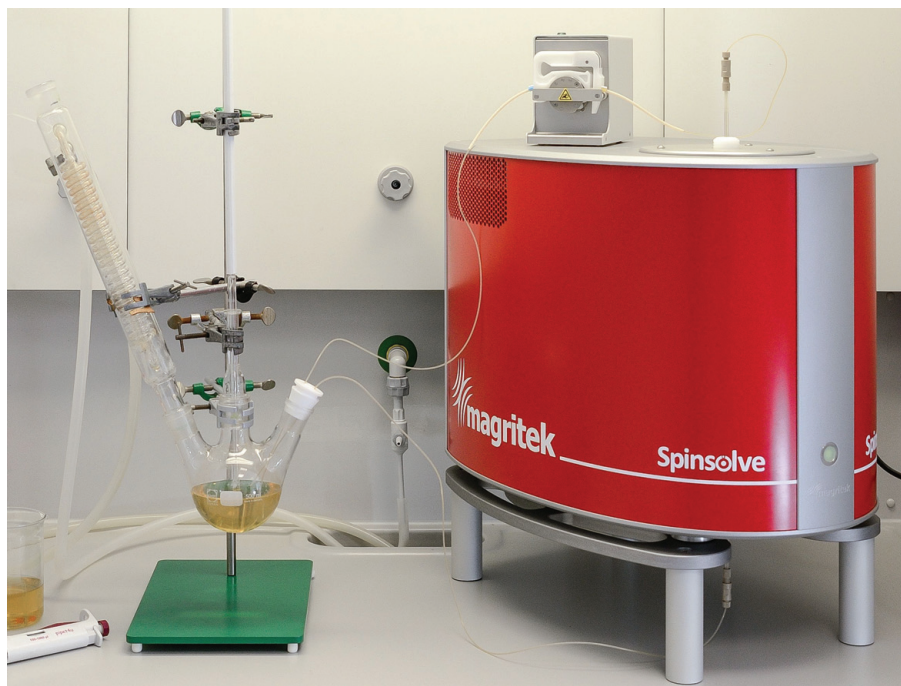
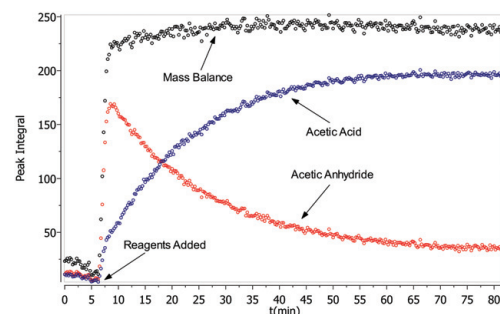


On-line Benchtop NMR Reaction Monitoring



Reaction monitoring with the Spinsolve® Benchtop NMR spectrometer and the glass flow cell.



Example: Hydrolysis of acetic anhydride with mass balance. (see Application Note 9)

Why use NMR for Reaction Monitoring

NMR spectra are chemically specific enabling the measurements to be sensitive to molecular structure and reaction kinetics. The measurements are quantitative with only minimal calibration. The signal has a linear response to concentration and it is generally not sensitive to the matrix. The measurement is of the complete sample and is non destructive.

The advantages of the Spinsolve® benchtop NMR spectrometer

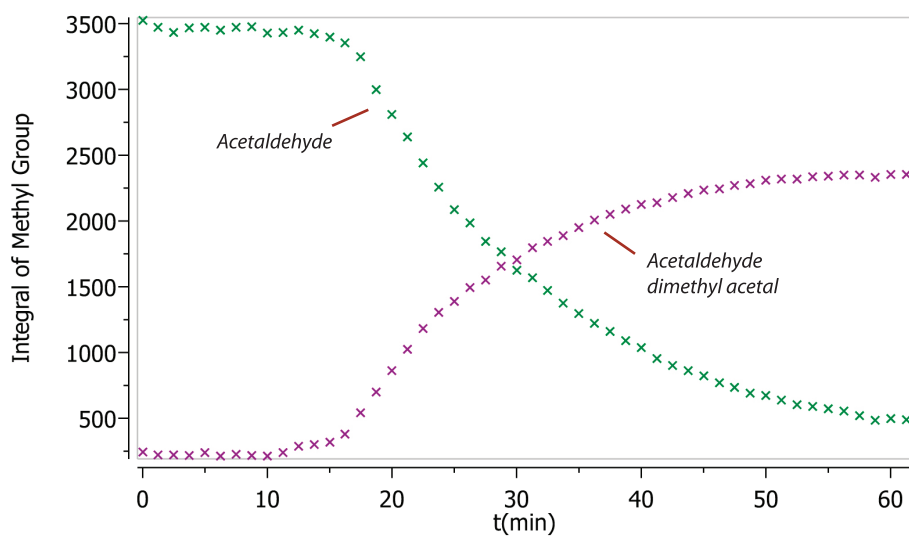
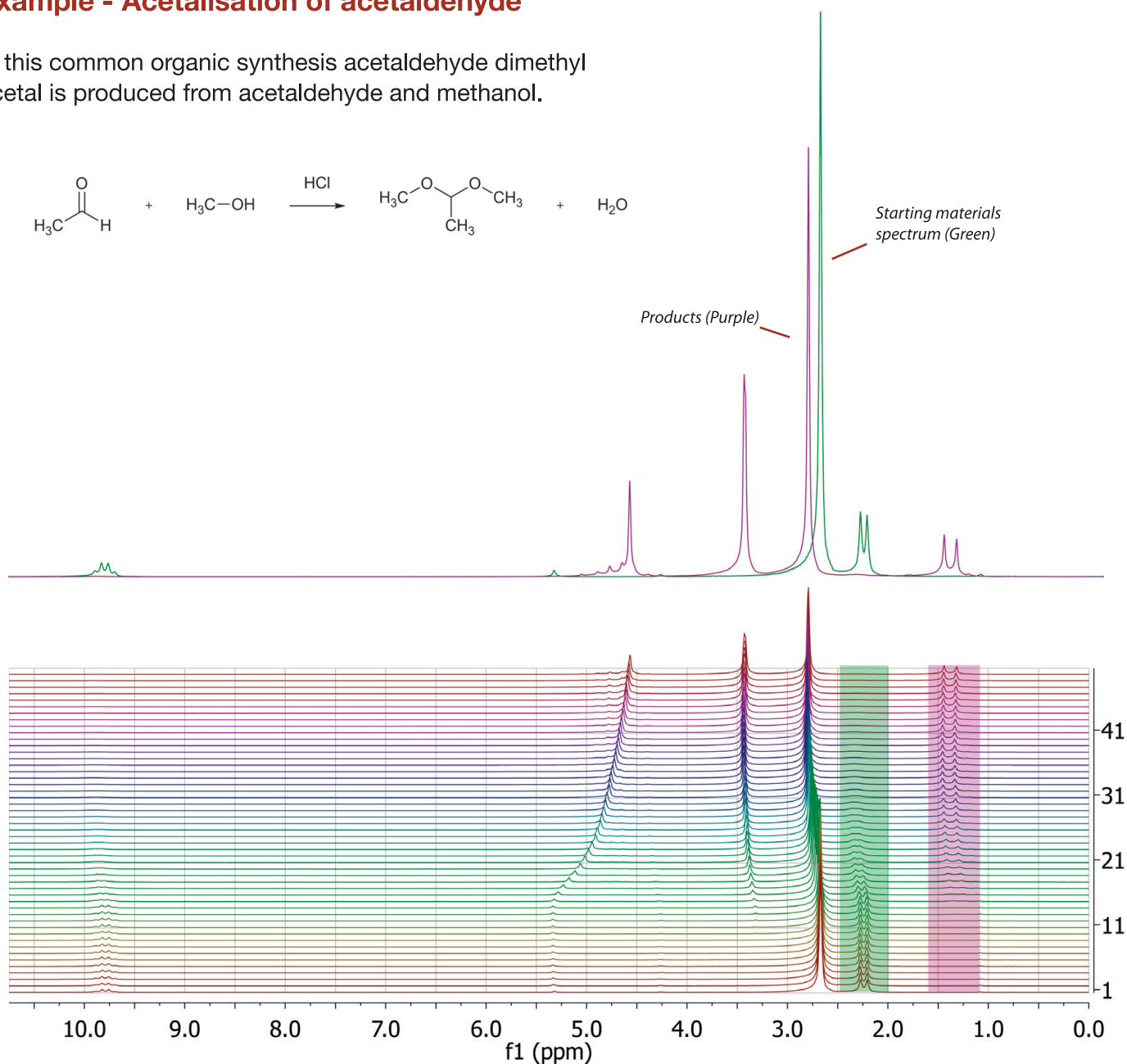
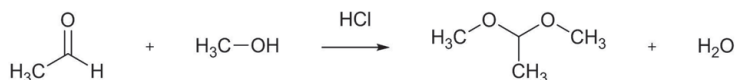
- Large sample volume with high sensitivity for fast measurements and high signal-to-noise
- High spectral resolution with narrow base width for accurate component integrals
- High stability for long measurement times with no user intervention
- Easy to use - does not require a dedicated technician for operation

Applications

- Determine reaction end points
- Determine reaction kinetics
- Identify intermediates and by-products.
- Maximize yields

Example - Acetalisation of acetaldehyde

In this common organic synthesis acetaldehyde dimethyl acetal is produced from acetaldehyde and methanol.



By monitoring the methyl group of the acetaldehyde at 2.25 ppm and also the methyl group of the product acetaldehyde dimethyl acetal at 1.4 ppm we can easily and accurately monitor the progress of the reaction as it occurs.

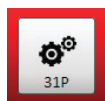
Software

It is easy to set up a reaction monitoring experiment with Spinsolve® software scripting.

In the example shown here we monitor an oxidation of triphenylphosphine to triphenylphosphine sulfide observing the signal from the phosphorus nucleus. The experiment runs for just over two hours with a spectrum taken every 20 minutes. The results are plotted in Mnova.

Step 1

Choose an existing reaction monitoring script template or modify one to suit.



Step 2

Press the green start button to commence monitoring. Start your reaction.

Here we take a ^{31}P spectrum every 20 minutes with 8 repetitions. Then open the data in Mnova.

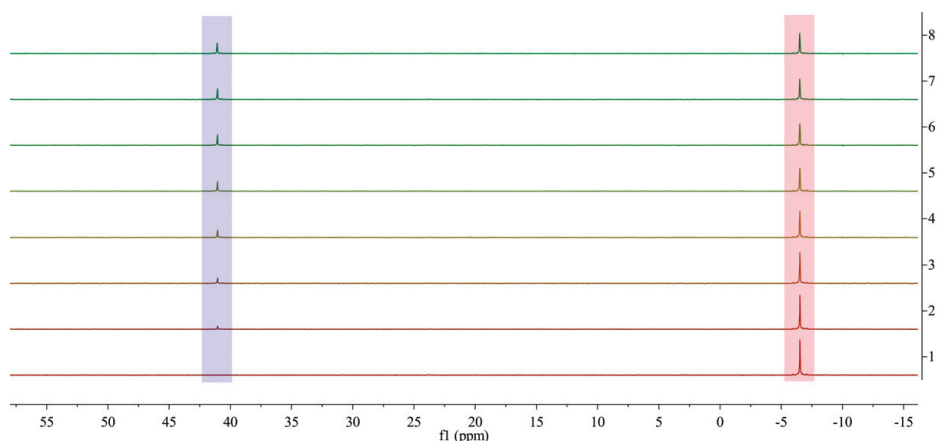
Step 3

Follow your progress with the sequence bar.

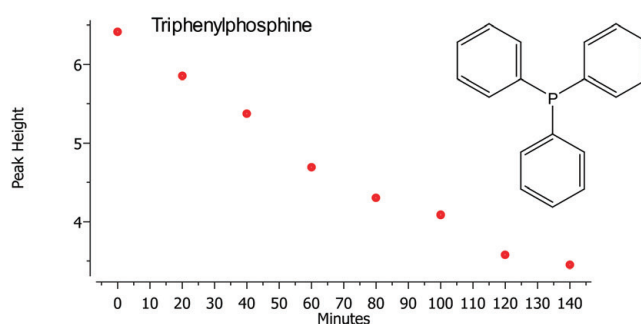
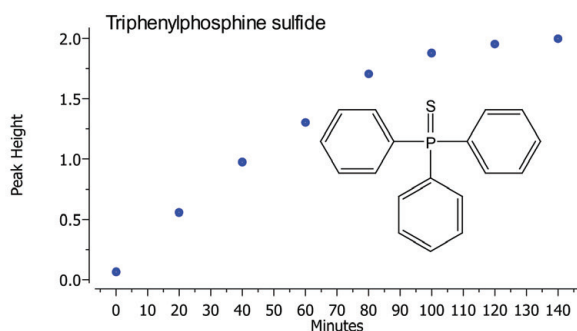
Track the reaction results with Mnova Listener or with a Spinsolve® software script driving Mnova.

Step 4

At the end of the reaction the script will automatically plot your data in Mnova.



The script can also include processing instructions for Mnova to generate the component quantities over time.

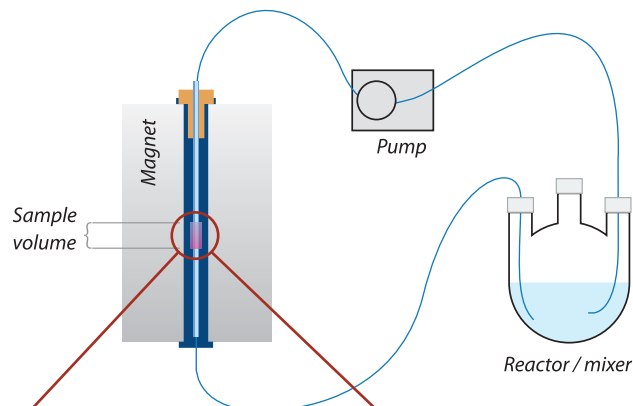


Hardware Options

Magritek offers two hardware kit options for reaction monitoring – indicated in the figure below.

For more details on each of these hardware options please see the specific datasheets.

The Spinsolve software provides complete control of pump operation enabling either stop flow or continuous flow operation with either of these hardware configurations.



PTFE Tubing (Kit RM1)

Single piece of PTFE tubing with a glass tube guide in the spectrometer bore to center the tubing at the sampling zone.

- Simplest configuration
- Easy to set up and run

Glass Flow Cell (Kit RM2)

Use of a glass flow cell with an expanded ID in the sample zone gives the highest performance.

- Large sample volume for maximum sensitivity
- Higher flow rate possible without signal drop from T_1 and T_2 effects.

Published Articles by Users

Several Spinsolve users have published results:

A self optimizing synthetic organic reactor system using real-time in-line NMR spectroscopy, V. Sans, L. Porwol, V. Dragone and L. Cronin, Chem. Sci., 6, (2015), 1258-1264. (School of Chemistry, The University of Glasgow, UK).

Simultaneous ^{19}F - ^1H medium resolution NMR spectroscopy for online reaction monitoring, N. Zientek, et al. J Mag Res, 249 (2014), 53–62. (BAM Federal Institute for Research and Testing, Berlin, Germany).

Online monitoring of fermentation processes via non-invasive low-field NMR. D.Kreyenschulte, E. Paciok, L. Regestein, B. Blümich and J. Büchs, Biotechnol. Bioeng., 112, Issue 9, (2015), 1810-1821.