

Benchtop NMR for Educational Experiments

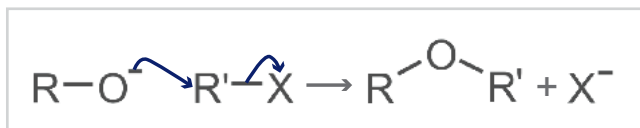
Williamson Ether Synthesis Reaction

This experiment is designed to teach the practical aspects and principles of the Williamson ether synthesis reaction, in this case via an S_N2 reaction:

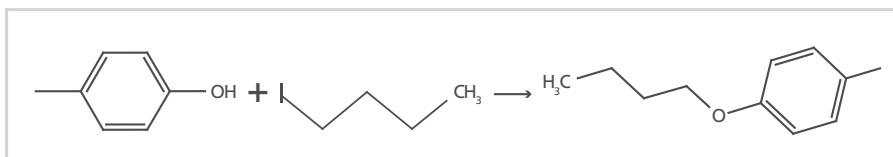
Refluxing, isolation of organic compounds, purification by solvent evaporation.

Total experiment time: 2 hours

The Williamson ether synthesis reaction involves the reaction of an alkoxide ion with a primary alkyl halide via an S_N1 or S_N2 reaction forming an ether group, and is of the general equation:



An example is the reaction between p-cresol and 1-iodobutane to form p-butoxytoluene.



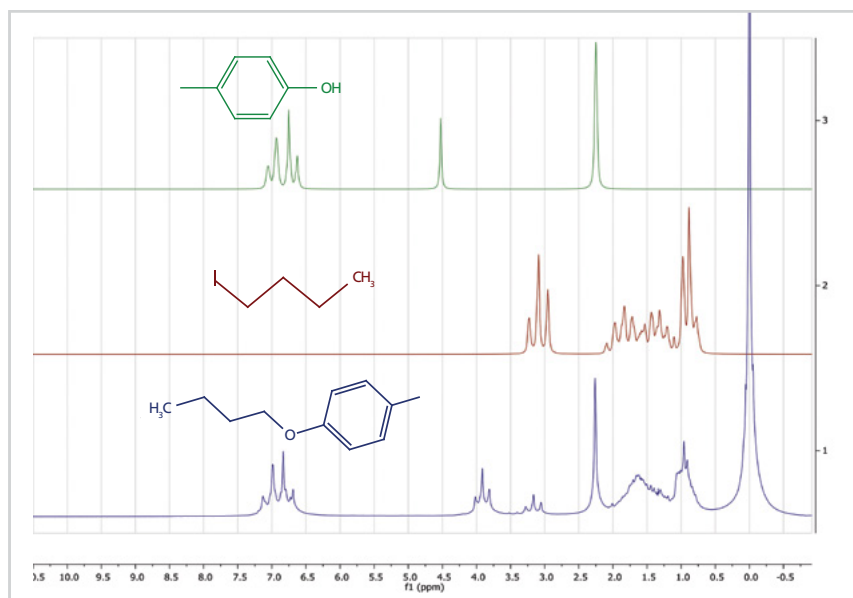
Pulsar™, a 60MHz benchtop NMR spectrometer can be used to measure the spectra of the starting materials and reaction product confirming that the reaction has completed and no starting materials remain. High quality spectra can be collected within 2 minutes. The spectra of the starting materials p-cresol and 1-iodobutane, along with the final product p-butoxytoluene are shown in Figure 1 (see over page).

It is clear from these spectra that p-cresol is not present in the spectrum of the product. However, the peak at about 3.2ppm and the overlap of the peaks in the region 1.2-2.0ppm suggests there is still some 1-iodobutane present in the spectrum of p-butoxytoluene. The p-butoxytoluene has tetramethylsilane (TMS) added as a chemical shift reference material showing a peak at 0ppm chemical shift.

The student can sign the peaks in the spectrum and generate peak integrals in order to verify the identity of the reaction product.

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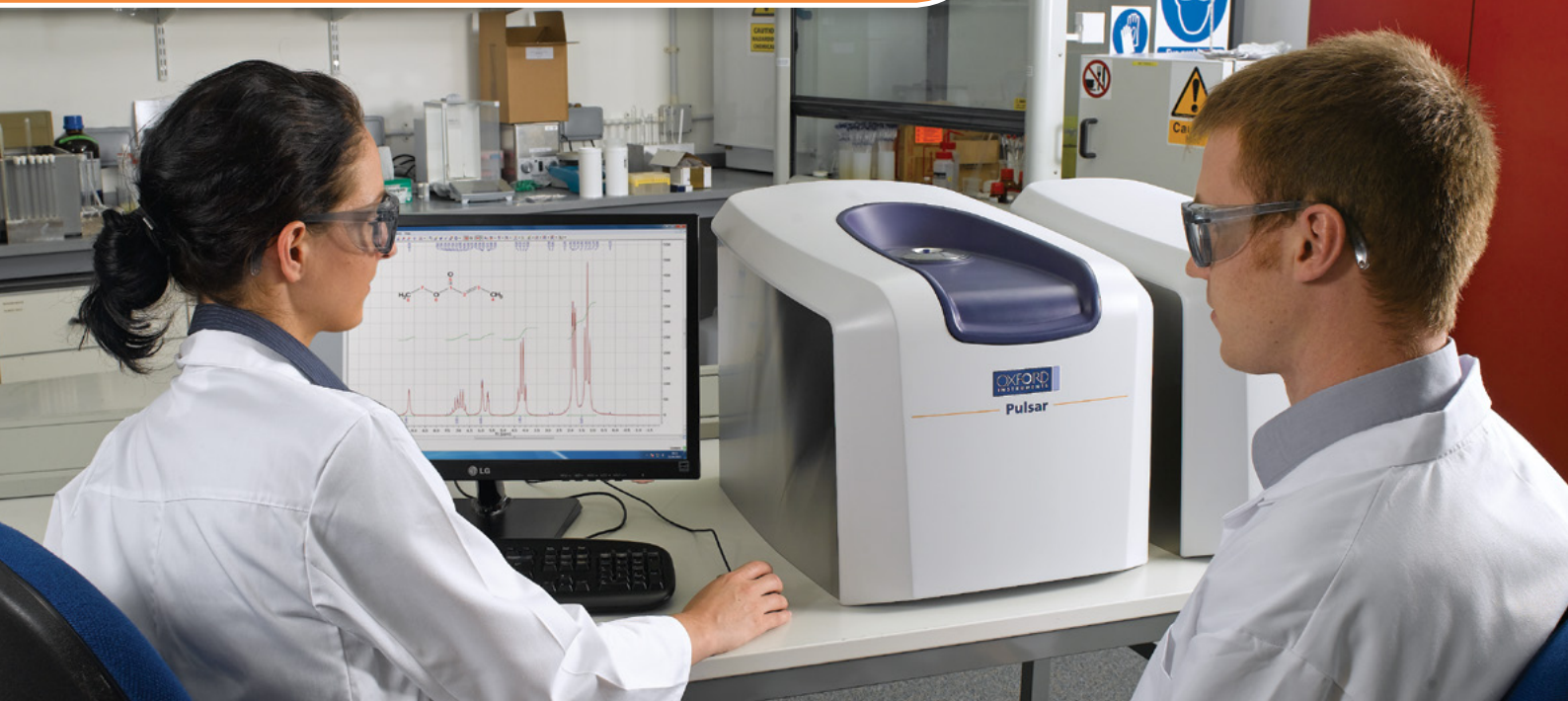


Pulsar is a cryogen-free benchtop NMR spectrometer that can easily be sited in the undergraduate chemistry laboratory allowing hands-on NMR for students.

A simple to use software interface and standard sampling using 5mm NMR tubes enables a high throughput of samples in a busy laboratory.

Figure 1. From top to bottom: *p*-cresol, 1-iodobutane and *p*-butoxytoluene spectra

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