

Benchtop NMR for Educational Experiments

Esterification Reaction

This experiment is designed to teach the practical aspects and principles of the Fischer esterification reaction:

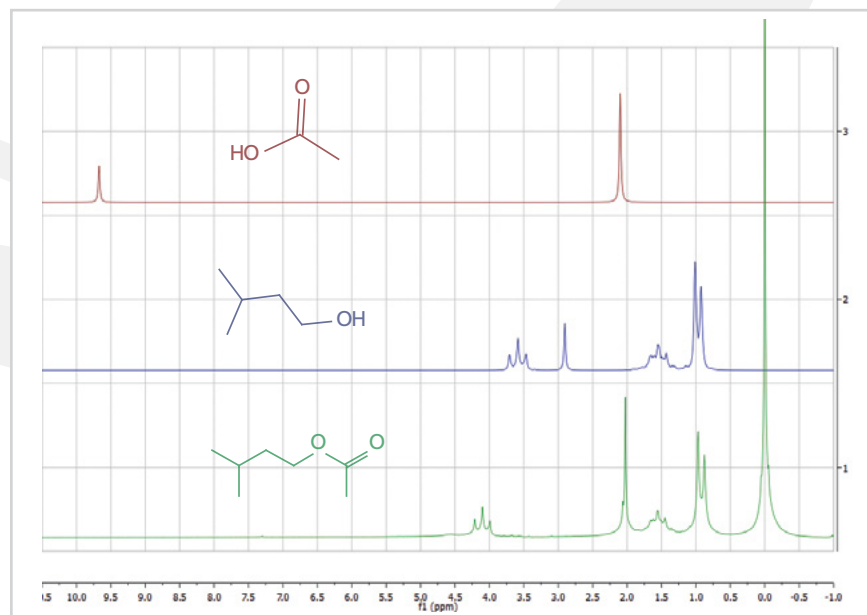
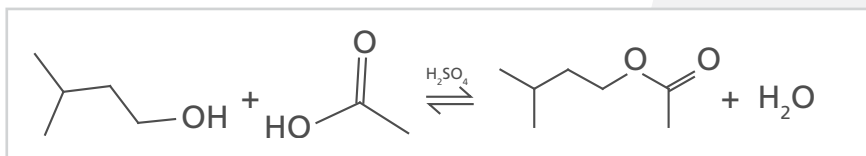
Refluxing, isolation of organic compounds, purification by distillation.

Total experiment time: 2 hours

The esterification reaction involves the reaction of a carboxylic acid with an alcohol.

The reaction is a reversible reaction taking place under acidic conditions with the application of heat.

An example is the reaction of 3-methyl-1-butanol with glacial acetic acid to form isoamyl acetate. The reaction is performed using sulphuric acid as a general acid catalyst using an excess of glacial acetic acid:



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. The spectra of the starting materials glacial acetic acid and 3-methyl-1-butanol, along with the final product isoamyl acetate are shown in Figure 1.

Figure 1. From top to bottom: Glacial acetic acid, 3-methyl-1-butanol and isoamyl acetate spectra

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It is clear from these spectra that neither glacial acetic acid nor 3-methyl-1-butanol is present in the spectrum of isoamyl acetate. The isoamyl acetate has tetramethylsilane (TMS) added as a chemical shift reference material showing a peak at 0ppm chemical shift.

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

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.

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