

Name (Last, First):	Ratzlaff, Natalie	PID:	A17091327
---------------------	-------------------	------	-----------

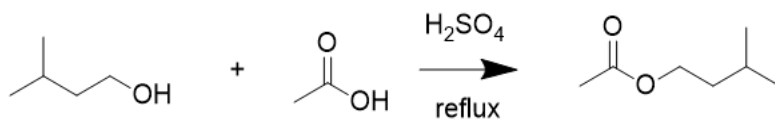
## Experiment 6: Fischer Esterification

### Introduction

The objective of this experiment was to perform a Fischer-Speier esterification reaction to convert an unknown alcohol into an ester with acetic acid. The resulting ester was then compared with 4 known ester samples using Gas Chromatography to determine the identity of the original unknown alcohol.

Esterification is important in many different applications, so running a Fischer-Speier esterification is very useful. An example of esterification at an industrial scale can be seen in the perfume industry, as esters typically have very strong, pleasant smells.

### Scientific Principles



**Scheme 1.** Balanced equation for the Fischer Esterification of 3-Methyl-1-Butanol and Acetic Acid.

1. Briefly explain how reflux is used to control the reaction temperature. Briefly explain how the reaction setup minimizes solvent loss.

Attaching a reflux tube to the top of the beaker allows evaporated solvent to condense in the reflux tube as it moves further from the hot plate, rather than leaving the entire system. The condensed solvent is then able to stay in the system, so long as the condensation does not go all the way up the reflux tube (because then vapors would be leaving the system due to the temperature being too high).

2. The enthalpy of reaction ( $\Delta H_R$ ) for the Fischer esterification reaction is usually  $\sim 0$ , indicating that neither products nor reactants are strongly favored ( $K_{eq} \sim 1$ ). Explain how the reaction is driven to completion (e.g. all alcohol is transformed into ester) in this experiment. Based on your understanding of equilibrium, propose one different strategy to maximize the formation of the ester product.

In an acidic environment, the alcohol is protonated, and this triggers a reaction between the protonated alcohol and the carboxylic acid, producing the intended ester and water as products. If water is removed (i.e. evaporated off at high temperatures), then the reaction will be pushed continuously to the ester (products) side. Alternately, continuously adding more reactants (and keeping one of them in excess for good measure) would have the same effect of pushing the equilibrium to the product side.

3. How was the excess acetic acid removed in this experiment? If instead the alcohol was used in excess, explain whether the *same* method could work to remove the excess alcohol.

The ester product is nonpolar due to its lack of O-H bonds relative to acetic acid and alcohols in this reaction. To filter out excess acetic acid, the product was filtered through silica, which removes polar compounds. Since both the acetic acid and alcohol are polar, this method would also work to remove excess alcohol.

4. If one would attempt to use column chromatography on silica gel to separate the product ester and excess reagent after a Fischer esterification, what characteristic should the mobile phase have? Briefly explain the experimental analysis needed to find the proper conditions for such a separation.

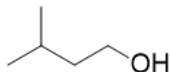
The mobile phase should be nonpolar, such that the nonpolar ester product is more miscible in the mobile phase, and the excess polar compounds are more miscible in the stationary silica phase.

5. GC analysis was performed on a mixture of the product ester with a 1:1:1:1 sample of the four possible esters. Briefly explain why this analysis is required, after one has already obtained the GC traces of the product ester and the 1:1:1:1 sample of the four possible esters separately.

Although it is possible to approximate the identity of the sample with retention time alone, retention time varies slightly from run to run, and machine to machine. So, it is possible to get a retention time that appears to match a certain standard, even if the compound is different. However, by mixing the product ester with all 4 standards, there will be 4 peaks, with one being stronger than the other, so long as the product ester actually is a match for one of the unknowns. If the product ester is not actually an ester or does not match one of the standards for another reason, there will be 5 peaks.

## Result: Identification of the Unknown Alcohol

Unknown ID# 377 has been identified as 3-Methyl-1-Butanol



3-Methyl-1-Butanol

## Discussion

The objectives of this experiment were met, as an ester product was able to be formed from an unknown alcohol, and this ester product was identified through GC comparison with the 4 standards, as well as IR spectroscopy.

The mixed GC analysis yielded 4 relevant peaks (there was a small acetone peak, which is not relevant to the experiment), 3 of which had the same 11 area%, and 1 of which had an area% of 53, and a retention time of 0.814, the lowest retention time of all the peaks. Because Isopentyl acetate is the smallest of the possible ester products, it is the one expected to have the lowest of the 4 retention times, so the unknown product ester was determined to be Isopentyl acetate. Additionally, because the peak for the product ester overlapped with one of the standard peaks, it is likely that the ester was actually formed.

Additionally, the IR spectra of the ester product contained a broad cluster of peaks around 2950 cm<sup>-1</sup>, with an intensity of 75%T, as well as a sharp peak at 1740 cm<sup>-1</sup>, with an intensity of 37%T. The 2950 cm<sup>-1</sup> peaks are indicative of the different C-H bonds in the ester product, and the 1740 cm<sup>-1</sup> peak is indicative of the C=O double bond in the ester product. There are no peaks above 3000 cm<sup>-1</sup> that would indicate the presence of O-H bonds, indicating that the ester product is pure.

The IR spectra of the alcohol contained a broad peak at 3324 cm<sup>-1</sup> with an intensity of 84%T, which is indicative of an O-H bond, as well as a collection of peaks around 2955 cm<sup>-1</sup> with a maximum 68%T, and these peaks are indicative of the different C-H bonds in the alcohol. Unlike the ester IR, there is no peak at a lower wavelength that would indicate the presence of a C=O bond, which makes sense for an alcohol.

## Conclusion

The product ester was identified as Isopentyl acetate based off GC comparison with the 4 standards. Based off of the presence of a lower wavelength peak indicative of a C=O bond, and the absence of a >3000 cm<sup>-1</sup> broad peak indicative of an O-H bond in the IR spectra for the ester product, it can be concluded that the ester product was a pure ester, not a mix of ester product and excess alcohol. Because the ester product was identified as Isopentyl acetate, the initial unknown alcohol must have been 3-Methyl-1-butanol.