

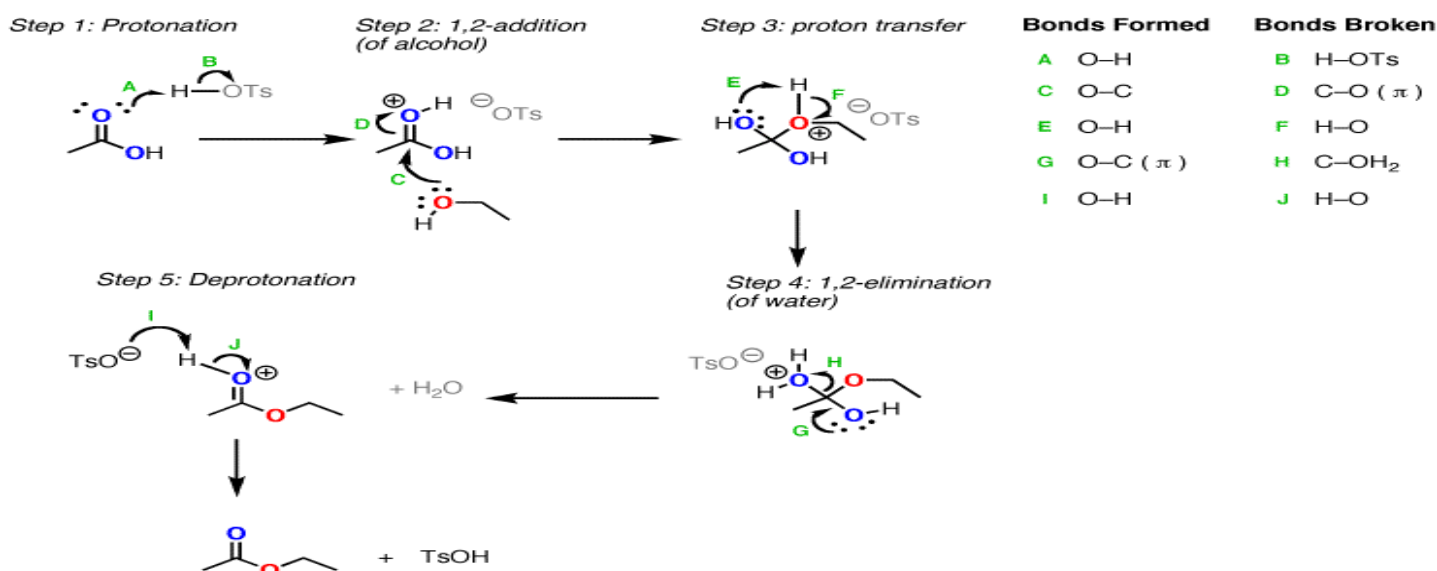
Abstract

Carboxyl group of organic compound that are not modified, they are not reactive enough to undergo an attack referred to as nucleophilic by nucleophiles therefore they must be activated by a mineral acid, in this experiment sulphuric acid is used for activation. The reaction, Fisher Esterification is conducted, its involves the reaction between an alcohol and a carboxylic acid in the presence of a mineral acid. The Aim of this experiment is to prepare isoamyl butyrate by reacting isopentanol and butyric acid using the methods of reflux, extraction, drying, and distillation. The theoretical yield is 3.142g. The actual yield is 0.222 g. The yield was low due to some of the errors that included unclean glassware which might have produced some side reaction, or the water may not have circulated faster during distillation hence affecting the process.

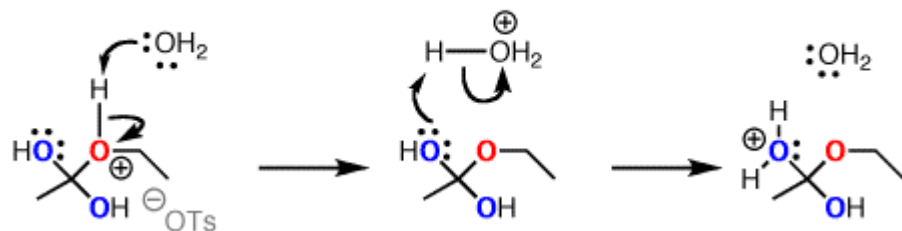
Introduction

The Fisher Esterification, mechanism, uses, and pitfalls

Fischer esterification is an acid catalyzed nucleophilic acyl substitution reaction of a carboxylic acid with an alcohol. The reactivity of Carboxylic acids is not strong enough so their reactivity is increased by the presence of a strong acid like H₂SO₄. The sulphuric acid protonates the carbonyl group oxygen giving it a positive charge and making it a better leaving group, increasing the reactivity of the carboxylic acid as below



Alternative proton transfer mechanism



The overall effect of the reaction is replacement an -OH group with an -OR . When large volume of isopentanol is present as a solvent the formation of isoamyl butyrate is favored.

There are two ways of making an alkene: one is by the process of dehydration and the other is by dehydrohalogenation. In this experiment the dehydration is used as the first method of elimination.

Dehydration is done by treatment of alcohol by a strong acid i.e. sulphuric acid to form the alkene which undergo the loss of the -OH group and a H to form the water molecule.

Conversion of -OH group to -OH_2^+ is the initial stage of the mechanism of the dehydration producing a leaving group

The next mechanism can be either E1 OR E2. About E1 mechanism. The alkyl cation is made by elimination of water which in return eliminates the proton making a double bond. About E2 mechanism, a bimolecular molecule is produced in which water acts as a base abstracting a hydrogen making an alkene.

The Purpose of this experiment is to prepare isoamyl butyrate by reacting isopentanol and butyric acid using the methods of reflux, extraction, drying, and distillation.

Materials and Methods

Procedure: adapted from Pavia, D. L.; Lampman, G. M.; Kriz, G. S.; Engel, R. G. Introduction to Laboratory Techniques, 4th Ed. Thomson Brooks/Cole: Mason, OH,2006; pp 106-108.

Isopentol (0.1 moles) and butyric acid (0.25 mole) was combined in a 100ml round bottomed flask. 4 mL of concentrated sulfuric acid was added to the mixture and swirled gently. Three precaution was observed during performing this experiment: measuring Butyric acid in the fume chamber due to bad odor, Handling sulphuric acid with gloves due to acid burns and finally the precaution of not adding the mixture to the acid but adding

the acid to the mixture this avoided acid from splashing. Three black boiling chips were added to the mixture. The Reflux condenser was attached and refluxed the mixture for three hours.

When the reaction completed, the mixture was allowed to cool. The vessel was vented with a syringe needle to cool the ice bath and to also to avoid the sudden pressure release when the vessel was opened up.

The product mixture was transferred to 20 ml falcon tube. The organic layer was then washed by 10% sodium bicarbonate solution. The organic layer was transferred to the Erlenmeyer flask and dried over sodium sulfate. The ester was allowed to stand until the liquid was clear.

The crude ester was decanted into 50 ml round bottomed flask, 4 boiling stones was added and then there was a simple distillation set up as fig 1

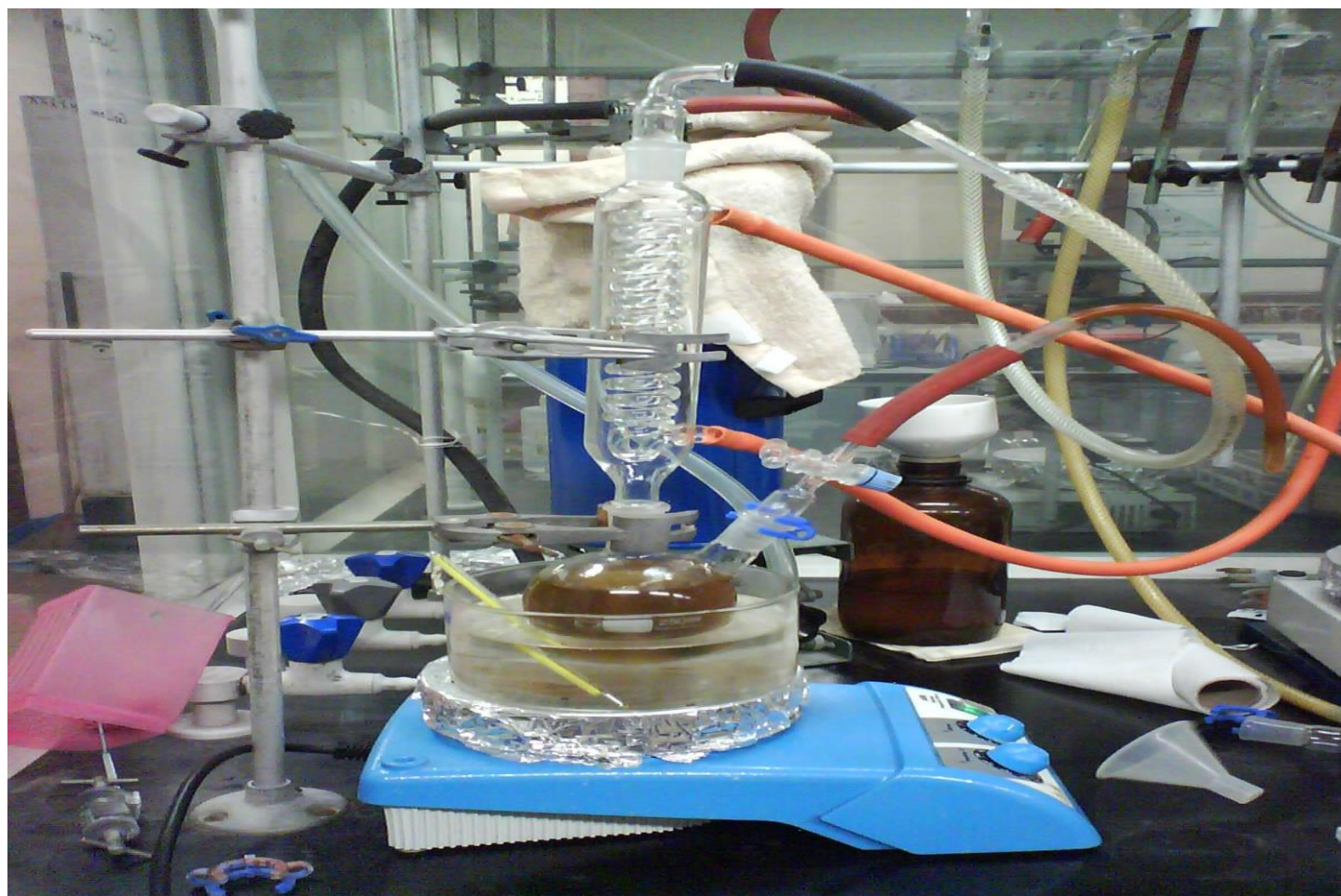


Fig 1.

The yield and the boiling point were recorded and IR spectrum was obtained.

Results

0.222 grams of dried product was collected at a boiling point of 186 °C

Mass of 100mL round bottom flask	54.206 g
Mass of 100mL round bottom flask with alcohol	55.880 g
Mass of alcohol (round bottom flask with alcohol- round bottom flask)	55.880g-54.206g=1.674
Mass of 50mL round bottom flask	22.100 g
Mass of 50mL round bottom flask with distillate	22.800 g
Mass of distillate (round bottom flask with distillate - round bottom flask)	22.800 g-22.100 g=0.700 g

1) Finding of limiting reagent

$$1.674 \text{ g isopentanolol}) \left(\frac{1 \text{ mole isopentanolol alcohol}}{88.2 \text{ g isopentanolol alcohol}} \right) = 0.0199 \text{ moles of isopentanol alcohol}$$

$$(1.5 \text{ mL butyric acid}) \left(\frac{1.06 \text{ g}}{1 \text{ mL}} \right) \left(\frac{1 \text{ mole butyric acid}}{88.1 \text{ g}} \right) = 2.646 \times 10^{-2} \text{ mole butyric acid}$$

$$\left(\frac{2.646 \times 10^{-2} \text{ mole excess butyric acid}}{1.999 \times 10^{-2} \text{ mole limiting isopentanol alcohol}} \right) = 1.32$$

2) Theoretical yield

$$(\text{moles of limiting}) \left(\frac{\text{excess}}{\text{limiting}} \right) \left(\frac{158.241 \text{ g isoamyl butyrate}}{1 \text{ mol isoamyl butyrate}} \right) (MW) = \text{theoretical yield}$$

$$(1.99 \times 10^{-2} \text{ mole isopentanol alcohol}) \left(\frac{1 \text{ mole isoamyl butyrate}}{1 \text{ mole isopentanol alcohol}} \right) \left(\frac{158.241 \text{ g isoamyl butyrate}}{1 \text{ mol isoamylbutyrate}} \right) = 3.142 \text{ g isoamyl butylate}$$

3) Percent yield

Formula: (amount prepared/amount possible) X 100% = percent yield

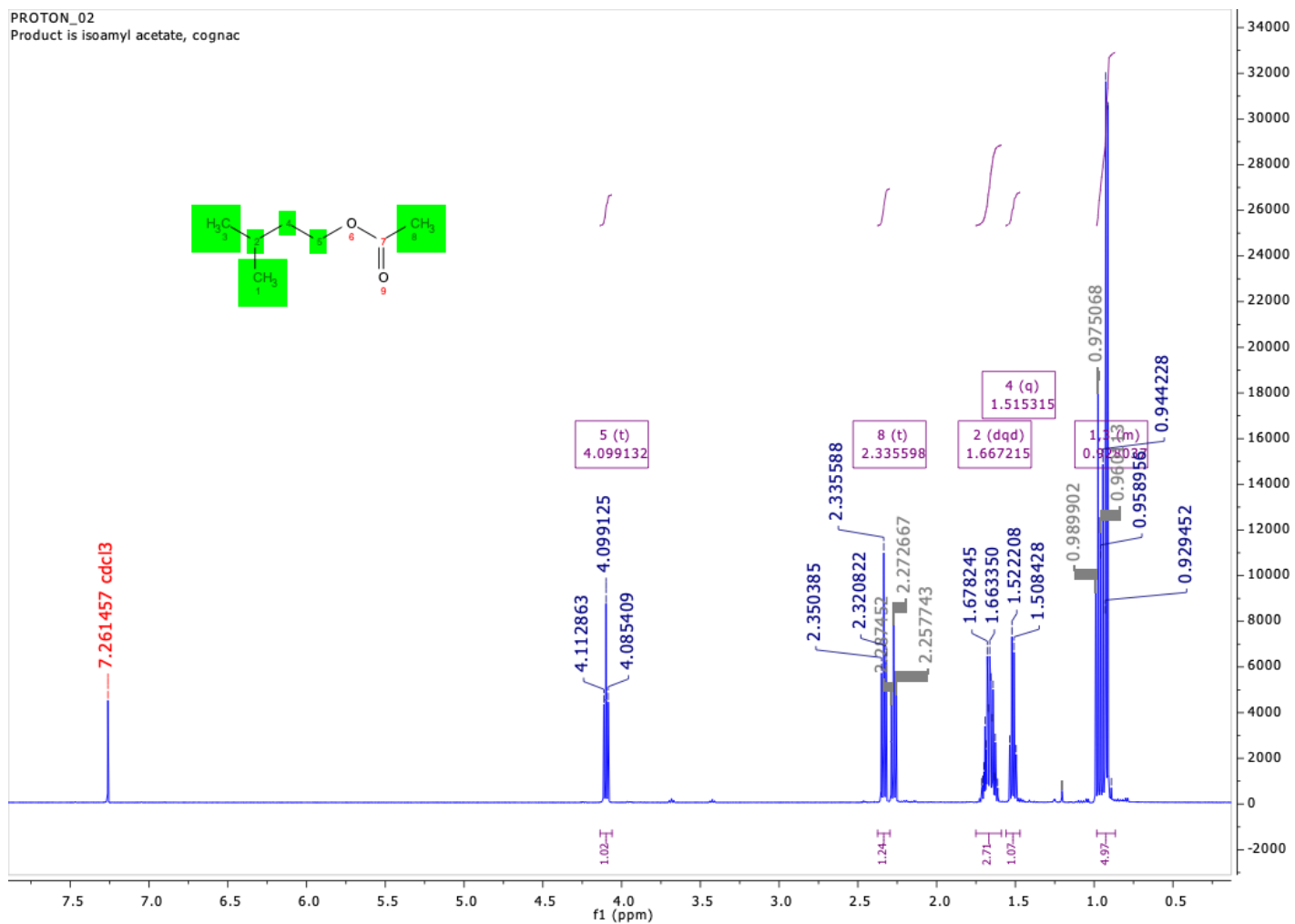
$$\left(0.700 \text{ g} \frac{1}{3.142} \text{ g isoamylbutyrate} \right) \times 100\% = 22.27\% \text{ percent yield isoamylbutyrate}$$

Conclusion

The ester isoamyl butyrate was synthesized by Fisher Esterification process. During this experiment 0.7 g of ester isoamyl butyrate was formed by direct esterification of butyric acid with isopentanol alcohol. The catalyst used in this reaction was sulphuric acid. Excess of isoamyl butyrate was used to move the reaction to the right to enable esterification occur. When the reaction was isolated, the excess butyric acid and isopentanol was gotten rid by sodium bicarbonate and the isoamyl butylate was purified more by anhydrous sulphate and also distillation.

The Percentage yield of the isoamyl butylate was 22.27 then the theoretical yield was 3.142g. For this experiment, the butyric acid was in excess and the isopentanol was the limiting reagent. This means the process of reaction was dependent on isopentanol alcohol available. The experiment was a success.

The spectra of isoamyl butylate is shown below. The C=O stretch of isoamyl butylate (3200) is at a higher wavelength



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