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Organic Chemistry Lab Report IX

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Fischer Esterification of Acetic Acid With Isopentyl Alcohol

Abstract

The main focus of this lab is to perform the esterification of acetic acid with isopentyl alcohol to get isopentyl acetate. Beads of silica gel, with anhydrous cobalt (II) chloride, are placed in a flask with isopentyl alcohol, glacial acetic acid, and concentrated sulfuric acid. While being connected to a water-jacketed reflux condenser, the flask is boiled in a hot sand bath. The top end of the reflux condenser contains spheres of anhydrous calcium chloride desiccant, this creates a dry atmosphere. This is left to run for 90 minutes, then transferred to a culture tube where the solution is washed with diethyl ether and saturated sodium bicarbonate. Liquid-liquid extraction occurs to remove the organic layer which is then dried with anhydrous sodium sulfate. The dried culture tube is placed in a hot sand bath to further evaporate the ether added for quantitative purposes and any excess water. The reaction yielded 768 mg of ester, which gave me a percent yield of 80%.

Procedure

The procedure adaptation is from Poulos, Z.J., Organic Chemistry I- "A Laboratory Manual for the Health Science Major," Fall 2019; Hayden-McNeil; Plymouth, MI, 2018. pp 141-150. The lab instructor made a few changes. The first being that instead of using a 5 mL reaction vial for our experiment, we were instructed to use a 10 mL round bottom flask. In

addition, instead of waiting a week to let the ether dry, we were instructed to speed up the drying period by placing out culture tubes into a hot sand bath to evaporate the ether. In addition, when adding the sulfuric acid, we did not use a glass Pasteur pipette but a plastic pipette.

Data and Calculations

Name	Theoretical yield (given by the textbook)	Tared culture tube	Final mass of culture tube	Actual yield (final mass of culture tube-tared culture tube)	Percent yield ($\frac{\text{actual yield}}{\text{theoretical yield}} \times 100$)
Isopentyl acetate	957 mg	11550 mg	12318 mg	12318 mg-11550 mg = 768 mg	$\frac{768 \text{ mg}}{957 \text{ mg}} \times 100 = 80.25$ $\approx 80 \%$

The theoretical yield is given by the textbook, but the calculations are:

(mmol of isopentyl acetate) x (MW of isopentyl acetate)

There values are also given in the textbook as:

(7.35 mmol) x (130.19 mg/mmol) = 957 mg

Discussion and Conclusion

An ester is a derivative of an alcohol and a carboxylic acid, a hydroxyl group is replaced by an alkyl group. For the experiment performed, the isopentyl is the alcohol and the carboxylic acid is acetic acid. The sulfuric acid is a catalyst meant to influence the reaction by increasing the rate of reaction without itself undergoing any chemical changes.

The process of esterification begins with protonation of the carboxylic acid. In the experiment, the sulfuric acid is used as a catalyst to protonate the oxygen of the alcohol which creates a positively charged alkoxonium ion. Without sulfuric acid, the esterification reaction will not work as stated in the textbook. The double bonded oxygen on the carboxylic acid will

remove the hydrogen from the alkoxonium ion to create a protonated carboxylic acid. Then the nucleophilic addition of alcohol to the protonated carboxylic acid takes place. A series of hydrogen transfers take place. A proton transfer and the loss of a water molecule through the collapse of the tetrahedral intermediate. The alcohol attaches to the main carbon which makes the carboxylic acid positively charged. The surrounding alcohol molecules takes the hydrogen off the connected alcohol. The OH group on the carboxylic acid takes a hydrogen from the protonated alkoxonium ion formed in the previous step. This creates a positive charge on the oxygen, which releases from the carbon atom. The end product is carboxylate ester and hydronium. In this experiment the carboxylic ester formed is isopentyl acetate.

From Le Chatelier's principle, removing water, a product, will favor formation of the ester. This is important because this principle means the reaction is reversible. In order to drive the reaction to favor the formation of the ester or forward, reagents must be added causing a limiting reagent and removal of product. The limiting reagent here is the isopentyl alcohol while acetic acid is added in excess. Product removal is due to drying agents such as the anhydrous cobalt (II) chloride covering the silica beads, while water molecules form, the anhydrous solution loses its blue color and becomes pink and clear.

The experiment began with the 10 mL round bottom beaker, a change made by the lab instructor, then I added 8 silica gel beads and 800 uL of isopentyl alcohol, 1.5 mL acetic acid, and 4 drops of sulfuric acid all at the same time. Since water would be a product in this reaction, the gel beads were added to absorb the water formed. The sulfuric acid works as a catalyst. A spin vane was added before the beaker was placed in the water reflux condenser. The reflux condenser had the top portion of the apparatus contain anhydrous calcium chloride, this was to

create a dry environment to limit loss of isopentyl alcohol and maximize absorption of water.

The hot plate was set to 7 and spin speed was set to 4, the beaker was placed in the middle of the hot plate into a hot sand bath and left for 90 minutes. Once the time had passed, the apparatus was taken apart and the miscible solution was placed in a culture tube. The solution was then made into immiscible organic and aqueous layers by the addition of 2 mL of saturated sodium bicarbonate. The top ethereal organic layer is where the ester is soluble and the bottom contains the aqueous layer. The organic layer is separated and placed in a new culture tube, then 2 mL of bicarbonate is added to the original culture tube and the liquid-liquid extraction happens once more, in total this process is done three times. To compensate for volume, 1 mL of diethyl ether was placed in the new culture tube. The organic layer was dried with anhydrous sodium sulfate twice by granules and through a pasteur pipette. Instead of leaving the layer to dry, I placed the culture tube inside a hot sand bath and massed the tube directly after to find I got 768 mg of isopentyl acetate.

The product was a yellow color and smelled like bananas. The recovered mass of the isopentyl acetate was 768 mg, when compared to the theoretical yield of 957 mg, given in the textbook, I had many human errors. My percent yield was calculated to be 80%, when compared with other students in my lab I found that my percent yield was somewhat higher than the rest. Despite this, human error in transferring the organic layer to a new culture tube led to loss of material. The continuous transfers from drying the organic layer led to loss of material. I did not time my experiment properly so the solution was overcooked or undercooked when heated in the sand baths at the beginning and end of experiment.

The infrared spectrum for acetic acid, there is a large peak at 3038 cm^{-1} and 2556 cm^{-1} this corresponds to the carboxyl group of the acid. This is because of hydrogen bonded dimer stretches which result in a broad range, not a series of sharp stretches. A carbonyl peak is seen at 1705 cm^{-1} . The infrared spectrum for isopentyl alcohol, there is a peak at 3323 cm^{-1} which shows the O-H stretch. Between the 2872 cm^{-1} and 2958 cm^{-1} stretch, there are three peaks that correspond to the C-H stretch of the alkyl group. There is a bending vibrations for both methyl and methylene at 1466 cm^{-1} and two peaks representing isopropyl groups at 1385 cm^{-1} and 1367 cm^{-1} . The infrared spectrum for isopentyl acetate, there are three peaks between 2958 cm^{-1} and 2873 cm^{-1} which correspond to the C-H stretching on the alkyl group. There are similar peaks between 1467 cm^{-1} and 1365 cm^{-1} which indicate isopropyl groups. A huge peak is seen on 1232 cm^{-1} which is associated with the single bond stretching of C-O on the carbonyl.