Experiment IX – Fischer Esterification of Acetic Acid with Isopentyl Alcohol

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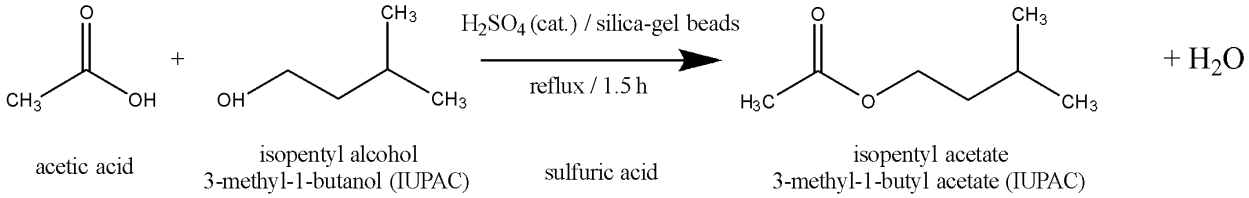
CHE231L7

Nucleophilic Acyl Substitution (S_NAc) Reaction - Fischer Esterification



acetic acid | isopentyl alcohol 3-methyl-1-butanol (IUPAC) | sulfuric acid | isopentyl acetate 3-methyl-1-butyl acetate (IUPAC)

Abstract :

The purpose of this experiment is to synthesize isopentyl acetate (3-methylbutyl acetate) by an esterification reaction between acetic acid and isopentyl alcohol (3-methylbutanol), using concentrated sulfuric acid as a catalyst. For Fischer esterification, we added isopentyl alcohol, glacial acetic acid, and a magnetic spin vane to a 5.0 mL reaction vial, and mixed well. After putting in silica gel beads, we added sulfuric acid to the swirling solution. Then we attached the flask to a reflux condenser and heated the mixture to reflux for 90 minutes. Then we quenched the reaction by adding saturated sodium bicarbonate solution; two immiscible layers were formed, and carbon dioxide gas was evolved. We removed the bottom aqueous layer and the isopentyl acetate ester product was dissolved in the top ethereal organic layer. We dried and obtained isopentyl acetate ester product (smell of banana observed) and measured the mass, 730mg, percent yield of 76%.

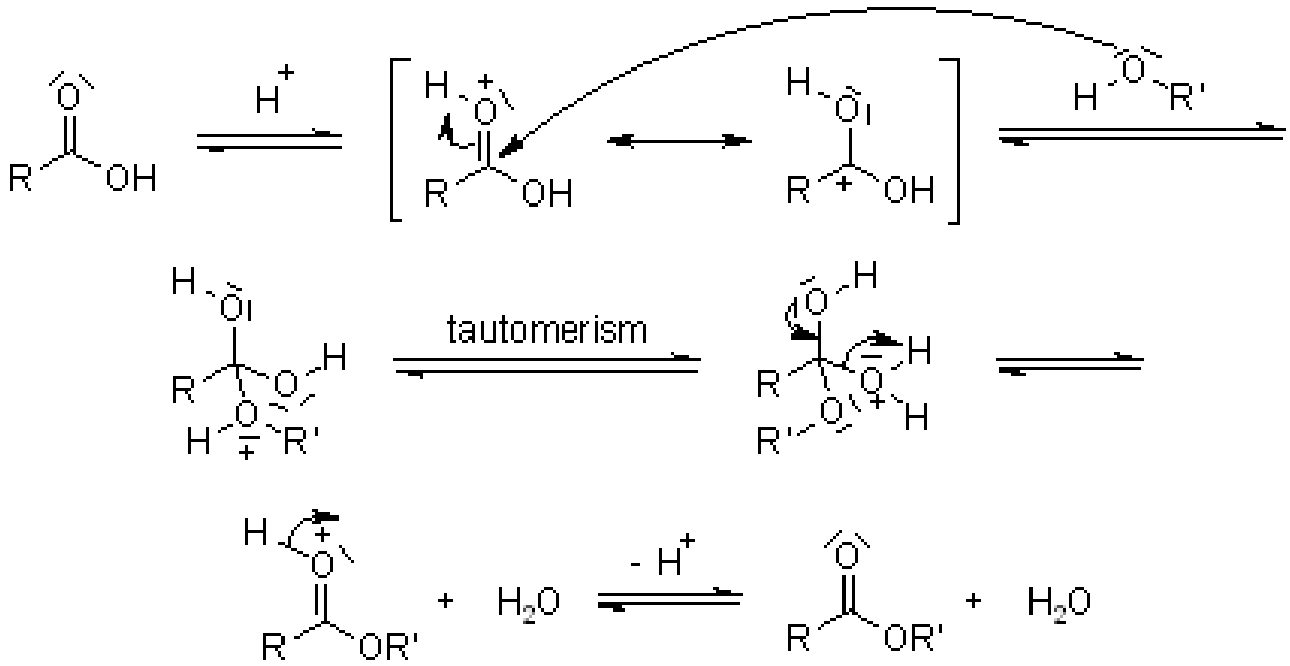
Procedure :

- Reference Procedure, title and source : Poulos, Zachary J. *A Laboratory Manual for the Health Science Major Organic Chemistry CHE231L*. Boston: Macmillan Learning, 2017. Print. Pg139-148
- List of changes : rather than putting the obtained isopentyl acetate ester product into the culture tube, we put in the product into the sand bath connected by the

tube and measured the mass when the liquid turned yellow and smelled like banana.

Reactions :

Reaction of the Esterification



Data and Calculations :

- Data table of measurement

  11/16, 2017

Original mass of cholesterol	Obtained mass of cholesterol dibromide	Measured melting point	Percent yield	Literature melting point provided by the instructor
102mg	111mg	108.5-112.6℃	74.4%	105-108.5℃

- Theoretical yield

  $102mg\ cholesterol \times \frac{1mmol}{386.66mg} = 0.264mmol$ cholesterol

Since mmol of cholesterol and cholesterol dibromide are equal due to the 1:1 ratio of cholesterol in to cholesterol dibromide out, 0.264 cholesterol dibromide will be obtained theoretically.

$0.264mmol \times \frac{606.5mg}{1mmol} = 149.2mg$

- Percent yield

$\frac{Actual\ yield}{Theoretical\ yield} \frac{111mg}{149.2mg} \times 100 = 74.4\%$

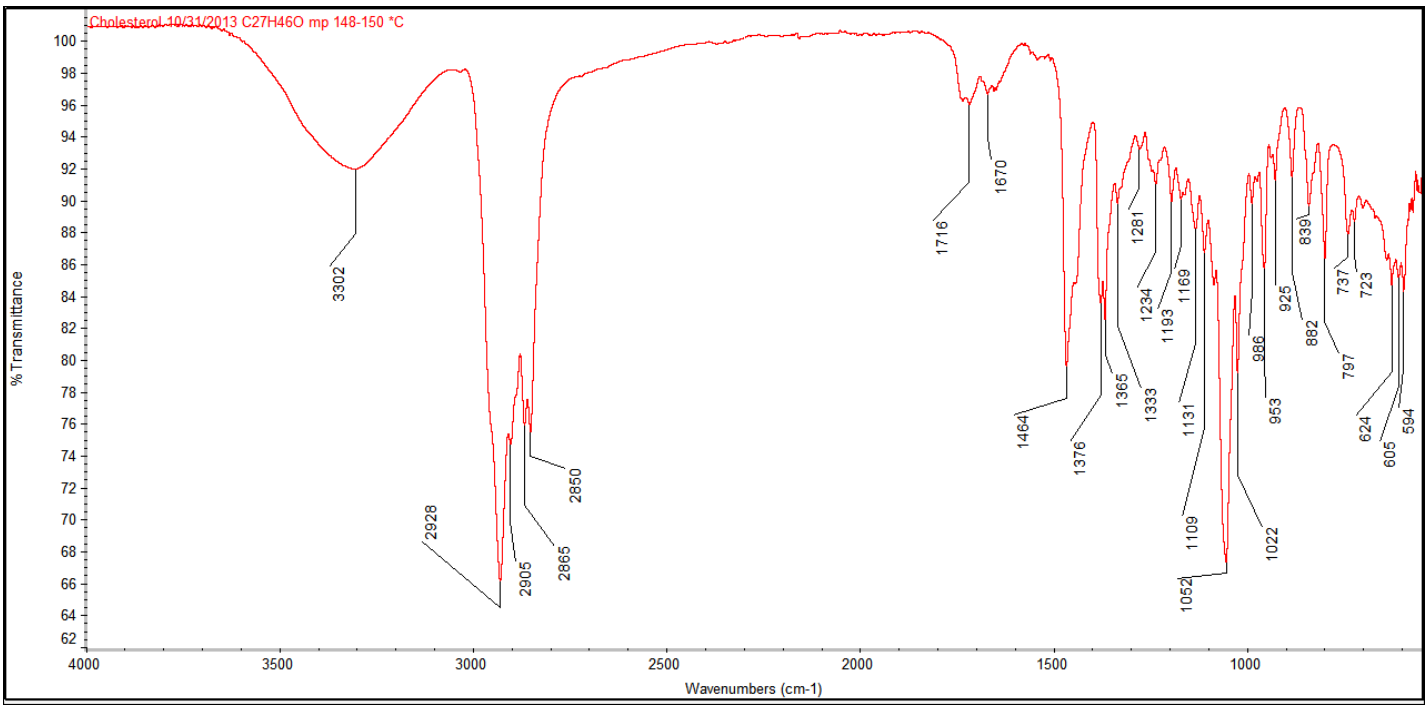
Discussion and Conclusion :

A halogen addition reaction is a simple organic reaction where a halogen molecule is added to the carbon-carbon double bond of an alkene functional group. The general chemical reaction of the halogen addition to an alkene is: C=C + X_2 → X−C−C−X (X represents the halogens bromine or chlorine.) and the product is a dihalide. This type of reaction is a halogenation and an electrophilic addition. The C=C double bond of alkenes makes the molecule much more reactive than an alkane and are capable of undergoing addition reactions, because π electrons can readily bond to other atoms. Addition of bromine to a double bond affords a product described as a vicinal dibromide because the halogen atoms are in the same vicinity. Bromine must donate a pair of electrons to the other end of the alkene as it is being attacked because there is no discrete carbocation intermediate. Instead of an intermediate cation, there is an intermediate bromonium ion that is then attacked by Br^-. So in our cholesterol dibromide, the connections of two bromines are opposite : 5α, 6β due to stereoselectivity of the bromonium ion (bromine is huge compared to a hydrogen, so if there's a carbocation next to a bromine atom its lone pairs of electrons are already getting close enough to interact with the positive charge because it is so big.)

To brominate cholesterol and convert it into cholesterol dibromide, I obtained 102mg of cholesterol. I added 2mL of MBTE to dissolve cholesterol. To this cholesterol in MBTE solution, I added red-brown solution of bromide, until the solution turned light yellow. If the solution turned yellow, there are no double bonds in the solution. When I swirled the solution gently, cholesterol dibromide started to crystallize. After cooling the solution in ice-water bath I vacuum filtrated the solution and white crystals of cholesterol dibromide came out. To get the solid cholesterol remaining in the glassware, I rinsed the Erlenmeyer flask with the wash solution (30% MBTE in glacial acetic acid). The actual yield of cholesterol dibromide was 111mg, and since the theoretical yield was 149mg, the percent yield was 74.4%. However, when I compared my result with other students and asked my instructor, my result seems fairly good. In the end of

the experiment, we measured the melting point of the obtained solid to measure its purification. The measured melting point was 108.5-112.6℃, and it matched the literature melting point which is 105-108.5℃ by no more than the range of 4℃. The difference in mass of theoretical yield and actual yield might be from the solid cholesterol sticking in the walls of glassware, or it might be from the imperfect vacuum filtration: there were still some solids left over in the mother liquid after the vacuum filtration.

IR discussion :



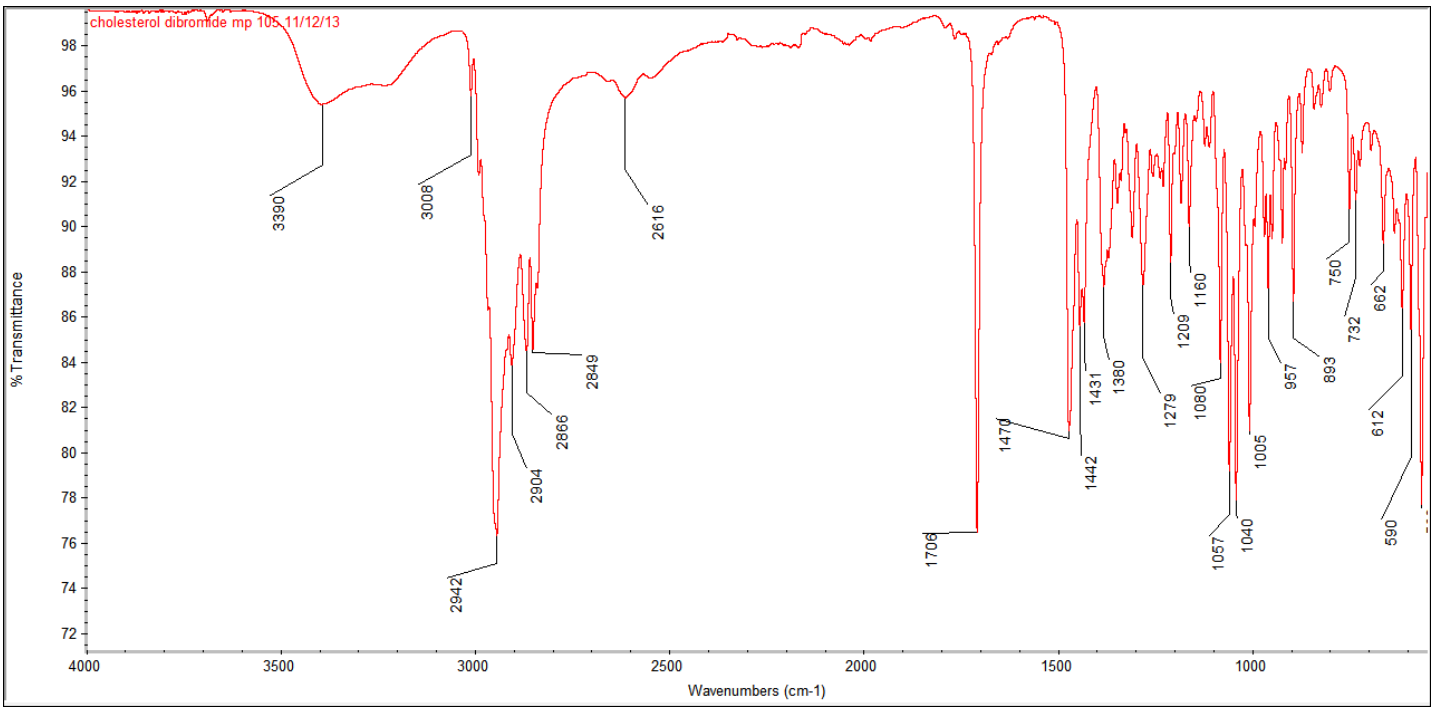
3302cm^{-1} O-H stretch (alcohol)

2928, 2905, 2885, 2850cm^{-1} C-H stretch

670cm^{-1} C=C stretch

1464, 1376, 1385cm^{-1} Ring C-C bend

1281cm^{-1} C-O stretch



3390cm^{-1} O-H stretch (alcohol)

3008, 2942, 2904, 2666cm^{-1} C-H stretch

1470, 1442, 1380cm^{-1} Ring C-C bend

1279cm^{-1} C-O stretch