Synthesis of banana flavor essay sample

Abstract

The purpose of this experiment was to synthesize isopentyl acetate via Fischer esterification reaction between acetic anhydride and isoamyl alcohol, using concentrated sulfuric acid as a catalyst. This reaction is characterized by the combining of an alcohol and an acid (with an acid catalyst) to yield and ester plus water. In order to accomplish this reaction, the reactants were refluxed for 30 minutes at 80 degrees Celsius , to yield a mixture, the mixture was then mixed with sodium hydrogen carbonate and dried with anhydrous sodium sulfate. The product obtained from this reaction was then weighed and yielded 3. 5753g, the total percent yield of isopentyl acetate obtained was 60 %. The advantages of using this particular esterification process, is that is fairly simple to set up and recreate, as long as the proper acidic conditions are present. Keywords: Esterification, Reflux, Extraction, Esters, Isopentyl acetate

Introduction

Esters have the general formula of R’COOR, in which R’ can be H, an alkyl or an aromatic hydrocarbon group and R is an alkyl or an aromatic hydrocarbon group (Bettelheim et al., 2007). The volatile compounds in natural fruits and flowers are usually complex mixtures of compounds, where esters frequently predominate. Many artificial flavorings contain esters or mixtures of esters. Esters are a group of naturally occurring compound that generally possess distinctive odor. Many of the processed foods that you buy today come with an ingredient label that lists “ artificial flavors” as one of the key ingredients. Artificial flavors are simply chemical mixtures that mimic a natural flavor in some way. Most organic fragrances and fruit flavours belong to this class of

compounds. Anything that we smell has to contain some sort of volatile chemical — a chemical that evaporates and enters a person’s nose. In the case of taste, a chemical has to activate the taste buds. Taste is a fairly crude sense — there are only four values that your tongue can sense such as sweet, salty, sour, and bitter — while the nose can sense thousands of different odors.

Carboxylic acid esters of low molecular weight are colourless, volatile liquids with pleasant odours, slightly soluble in water. Many esters are responsible for the fragrance and flavour of flowers and fruits; for example, isopentyl acetate is present in bananas, methyl salicylate in wintergreen, and ethyl butyrate in pineapples. These and other volatile esters with characteristic odors are used in synthetic flavours, perfumes, and cosmetics. Certain volatile esters are also used as solvents for lacquers, paints, and varnishes; for this purpose, large quantities of ethyl acetate and butyl acetate are commercially produced. Waxes secreted by animals and plants are esters formed from long-chain carboxylic acids and long-chain alcohols. Fats and oils are esters of long-chain carboxylic acids and glycerol. Therefore most artificial flavors have both taste and smell components. Any natural flavor is normally quite complex, with dozens or hundreds of chemicals interacting to create the taste/smell. But it turns out that many flavors — particularly fruit flavors – have just one or a few dominant chemical components that carry the bulk of the taste/smell signal. Many of these chemicals are called esters. The primary objective of this experiment is to prepare the assigned ester via Fischer reaction and calculate its percent yield. Fischer reaction for the

synthesis of esters is an acid catalyzed reaction of an alcohol and a carboxylic acid.

Scheme 1. 0 Example of a Fischer Esterification

An example of this reaction is seen in Scheme 1. 0, the acid and alcohol reactants are in equilibrium with ester product. It involves heating of a mixture of an alcohol and a carboxylic acid in the presence of strong mineral acid catalyst like H2SO4 to yield an ester and water as the end product. Isopentyl acetate is known as banana oil because of its characteristic odor. This ester has also been shown to be one of the active substances in the alarm pheromone of the honeybee. The pheromone causes other bees to become aggressive and attack the intruder. In the experiment, the Fischer esterification will make the acid and alcohol reactants to be in equilibrium with the assigned ester, isopentyl acetate, to prepare an artificial Banana flavoring. Then isoamyl alcohol will be refluxed with acetic anhydride and with concentrated Sulfuric acid as the catalyst. The product is isolated using a combination of technique such as acid-base extraction, drying, and in special case of distillation. Results and Discussion

Isopentyl acetate

Acetic anhydride

Isoamyl alcohol

Scheme 1. 1 Fischer Esterification of Banana Flavor

The scheme above summarize the whole experiment, its shows the reaction between isoamyl alcohol and acetic anhydride together with the catalyst

which is sulfuric acid. Before the experiment was performed, the Limiting reagent was first computed between isoamyl alcohol and acetic anhydride, to obtain the theoretical yield. The computation would not be possible without the different molecular weight, volumes and densities given in Table 1. 0: Table 1. 0 Molecular Weight, Volumes and Densities of the Reagents and Products.

Isoamyl alcohol| |

MW = 88 g/mol| |

d = 0. 8092 g/mL| |

V = 5. 00 mL| |

Acetic anhydride| |

MW = 102 g/mol| |

d = 1. 089 g/mL| |

V = 5. 20 mL| |

| Isopentyl acetate|

| MW = 130 g/mol|

| d = 0. 876 g/mL|

| V = ? mL|

5ml C5H12O x 0. 8092g/ml = 4. 046 C5H12O (1mol/88g C5H12O) = 0. 046mol C5H12O 5. 2ml C4H6O3 x 1. 089g/ml = 5. 616g C4H6O3 (1MOL/102g C4H6O3) = 0. 055mol C4H6O3

Scheme 1. 2 Limiting reagent

0. 046mol C5H12O x (1mol C7H14O2/ 1mol C5H12O) x (130g C7H14O2 / 1mol C7H14O2) = 5. 98g C7H14O2

Based on the computation above, C5H12 is the limiting reagent for it will be the first to be consumed in the reaction. Aside from the computing the limiting reagent the theoretical yield was also taken into consideration.

Scheme 1. 3 Theoretical yield

This scheme above that the ester obtained from the reaction is most likely to be 5. 98g. Table 1. 1 Summary of Results

Volume of Isoamyl alcohol:| 5. 00 mL|

Volume of Acetic Anhydride:| 5. 20 mL|

Limiting Reagent:| 0. 046mol C5H12O|

Excess Reagent:| 0. 055mol C4H6O3|

Theoretical Yield:| 5. 98g C7H14O2|

Actual Yield:| 3. 5753g C7H14O2|

Percent Yield:| 60 %|

Description of Banana flavour:| Colorless, Banana Odor|

In the experiment, the isoamyl alcohol and acetic anhydride underwent Fischer esterification to prepare an artificial fruit extract namely, Isopentyl acetate or also known as Banana Flavoring. The mechanism used here was Fischer Esterification, it is the process of forming an ester by refluxing a carboxylic acid and an alcohol in the presence of an acid catalyst. The reaction mixture was refluxed for 30 minutes at 80 degrees Celsius, this was done in order to thermally accelerate the reaction by conducting it at an elevated temperature, without losing any of the compounds in the set-up. The addition of concentrated sulfuric acid is important in Fischer reaction because, it acts as an acid and dehydrating agent which accomplishes the task by injecting a proton into the carboxylic acid structure through the

reaction H2SO4+R-COOH→HSO4-+R-C+(OH)2. In this part, Le Chatelier's Principle kicks in, and the equilibrium point is slammed to the right as the forwards reaction tries to oppose the lack of water that the sulfuric acid has caused.

Le Chatelier's principle suggests that the amount of ester produced in an equilibrium reaction might be increased either by using an excess of one of the reactants or by removing one of the products. This means that lots of ester gets made. Le Chatelier's principle states that " if external stress is applied to a system at equilibrium, the system adjusts in such a way that the stress is partially offset as it tries to reestablish the equilibrium" (Chang and Overby, 2011). In addition, the H2SO4 also serves as an acid catalyst which somehow speeds up the reaction. The swirling of the separatory funnel, which contains NaHCO3 together with the resulting mixture, was done gently because too hard will cause the mixture to emulsify. NaHCO3 was preferred to use than NaOH because it gives less moles of H2O shown in Scheme 1. 4 and Scheme 1. 5. 2NaOH(aq) + H2SO4 → Na2SO4(aq) + 2H2O

2 NaHCO3 + H2SO4 → Na2SO4 + 2H2CO3

H2CO3 → H2O + CO2(gas)

Scheme 1. 4 Reaction of NaHCO3 with Na2SO4

Scheme 1. 5 Reaction of NaOH with Na2SO

After swirling, the mixture created two layers as the funnel is swirled gently, this was discarded to prevent the reaction from undergoing hydrolysis which would push the reaction to the reactants. The purpose of extracting the saturated NaCl (lower layer) from the mixture is to remove the bulks of water

remaining in the mixture. Also, the purpose of extracting the NaCl with sodium bicarbonate is to remove the excess acids from the mixture. Lastly the purpose of filtering the mixture with anhydrous sulphate is to dry the mixture from the water, because anhydrous sulphate are capable of binding in water .

The pimary goal of this experiment is to prepare the assigned ester via Fischer Reaction and calculate its percent yield. In the experiment, Lewis or Brønsted acid-catalyzed esterification of carboxylic acids with alcohols (isoamyl alcohol) to give esters is a typical reaction in which the products and reactants are inequilibrium. The equilibrium may be influenced by either removing one product from there action mixture (in the experiment, removal of the water by liquid-liquid distillation or absorption by molecular sieves) or by employing an excess of one reactant (Acetic Anhydride). At the start of Fischer reaction, the protonation from sulfuric acid (acid catalyst) to the carbonyl group of acetic anhydride, making it electrophilic, it is a state where in atoms, molecules, and ions behave as electron acceptors. The oxygen atom from isoamyl alcohol attacks the electrophilic carbon of the carbonyl group.

A proton transfer from the oxygen to second molecule of isoamyl alcohol gives an activated complex. Protonation of one of the hydroxyl groups in the activated complex gives a new oxonium ion (removing of OH). Some sources of experimental errors really depends on the discarding of certain compounds, because if these are not discarded well it may affect the actual yield, which will further affect the percent yield in the experiment. Through several extractions with the use of sodium bicarbonate and saturated sodium

chloride will remove the neutral water molecules from the oxonium ion. After the water loss and subsequent deprotonation, the reaction yielded a purified, colorless product namely , Isopentyl acetate. As a result, the esterification reaction between acetic anhydride and isoamyl alcohol, using concentrated sulfuric acid as a catalyst, was successful and yielded isopentyl acetate. The percent yield of this reaction was 60%.

Experimental methodology

Two dry test tubes labeled A and B, containing 5. 0 mL isoamyl Figure 1. 0 alcohol and 5. 2 mL acetic anhydride respectively, were placed in an ice-bath. Ten drops of concentrated sulfuric acid was drop- wisely added into test tube B. While both test tubes are in the ice- bath, the contents of test tube B were added drop-wise into test tube A, these steps were shown in Figure 1. 0.

Figure 1. 1The test tube was swirled constantly. The resulting mixture was transferred into a reflux set-up, shown in Figure 1. 1. Three to five boiling chips were added and heated in a beaker containing 125mL of water at 80°C for 30 minutes. After 30 minutes, the hot bath was removed, cooled for a minute and the contents were poured in a small beaker containing a cube of ice (Figure 1. 2).

Figure 1. 2

ice cube

NaHCO3

30 mL

NaHCO3

Figure 1. 3

The resulting mixture was transferred in a separatory funnel, shown in Figure 1. 2, containing 30mL saturated NaHCO3 solution. The bubbles were waited until it disappeared, then the funnel was secured with the cap. The funnel was gently swirled to avoid forming emulsion. The two layers were allowed to separate. The cap was removed and the lower layer was discarded, retaining the organic layer. The extraction was repeated with another 30mL of NaHCO3. After the lower layer was discarded again, the organic layer were obtained and was transferred into a clean and dry beaker.

30mL NaHCO3.

The final product was filtered using a funnel with a cotton containing a half spatula of anhydrous sodium sulfate, shown in Figure 1. 4, and the final product was then weighed and the % yield was calculated. Figure 1. 4

References

Bettelheim, F. A., Brown, W. H., Campbell, M. K., & Farrell, S. O. Introduction to General, Organic and Biochemistry; Brooks/Cole: Singapore, 2007; p 465-467.

Chang, R. & Overby, J. General Chemistry: The Essential Concepts; McGraw-Hill Companies Inc. : New York, 2011; p 386-387.

Hart, H., Hart, D., Craine, L., & Hadad, C. Organic Chemistry: Short Course; Houghton Mifflin Company: USA, 2007; p 300-318.

Klein, D. Organic Chemistry; John Wiley & Sons, Inc. : USA, 2012; p. 997-1000.

Lehman, J. A. Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course; Pearson Education Inc. : New Jersey, 2009; p 70-75.

Ouellette, R. J. Organic Chemistry: A Brief Introduction; Macmillan Publishing Company: New York, 1994; p 427-435.

http://images. ustbio1. multiply. multiplycontent. com/attachment/0/ThwvpQooCpIAADlac0E1/Banana. pdf? key= ustbio1: journal: 10&nmid= 468734014 (retrieved on December 16, 2012). http://users. stlcc. edu/mhauser/Syn%20Isopentyl%20Acetate. pdf(retrieved on December 16, 2012). http://www. wisegeek. com/what-is-the-function-of-sulfuric-acid-in-esterification. htm (retrieved on December 16, 2012).