

Preparation of Banana Oil

Introduction

Many of the simple esters have pleasant fragrances which we find similar to that of fruits and flowers. These esters have been synthesized in laboratories and are sometimes used to duplicate natural fragrances and flavors in commercially prepared desserts and beverages. Interestingly, they are not used in expensive perfumes because the ester group is not stable to perspiration. It can decompose to produce acids which often have unpleasant odors.

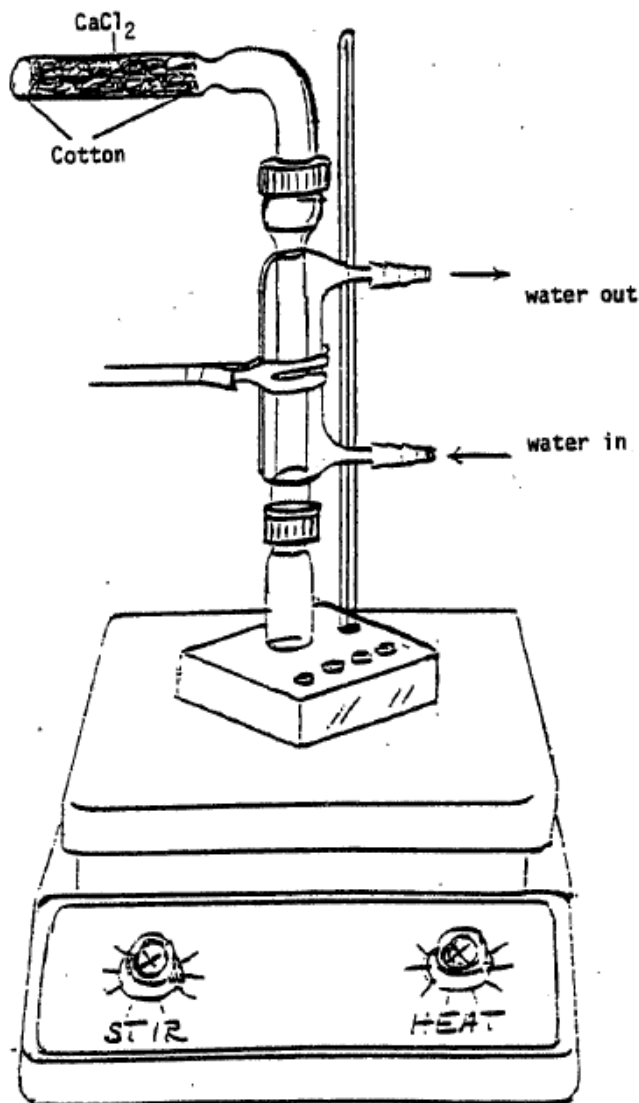
The ester prepared in this experiment has the fragrance of bananas. It has another interesting characteristic. It is identical to the pheromone of the honeybee. A pheromone is a chemical secreted by an organism that results in a specific response from other members of the same species. When a worker bee stings its victim, this pheromone is secreted along with the venom. The pheromone alerts other bees to join in the attack on the intruder.

In this experiment isopentyl acetate will be prepared from the reaction of acetic acid and isopentyl alcohol. The reaction does not yield significant amounts of the ester unless the reaction is forced toward the production of the ester. It can be forced by using an excessive amount of one of the starting materials. We will use an excess of acetic acid as it is less expensive than isopentyl alcohol. A small amount of sulfuric acid will be used as a catalyst. After the reaction is complete, the excess acetic acid and the sulfuric acid can be neutralized with sodium bicarbonate and subsequently separated from the water-insoluble ester.

Procedure

1. Using a graduated pipet, place approximately 0.7 mL of isopentyl alcohol (also called isoamyl alcohol) in a 5-mL conical vial.
2. Add approximately 1.4 mL of glacial acetic acid using a graduated pipet.
3. Using a disposable glass Pasteur pipet, add two or three drops of concentrated sulfuric acid.
4. Add a magnetic spin vane (magnetic stir bar).
5. Assemble a reflux apparatus using a water-cooled condenser. (Refer to figure.) Top the apparatus with a drying tube packed with calcium chloride held in place with a small amount of cotton.
6. Using a hot plate equipped with an aluminum block, bring the mixture to a boil. If desired, an aluminum collar can be wrapped around the vial to better control the temperature. Continue to heat the mixture for 60-75 minutes. STIR constantly using the magnetic spin vane.
7. Remove the apparatus from the heating source, continue running water through the condenser and allow the mixture to cool to room temperature.
8. When the mixture has cooled, take apart the apparatus and use clean forceps to remove the magnetic spin vane.
9. Using a calibrated Pasteur pipet slowly add 1.0 mL of 5% aqueous sodium bicarbonate solution. Stir with a microspatula until carbon dioxide evolution is no longer vigorous. (The sodium bicarbonate reacts with both the acetic acid and the sulfuric acid to produce carbon dioxide.)

10. Cap the vial (Teflon side in toward the product) and shake gently. Carbon dioxide will continue to be produced so it is important to open the cap carefully to vent the gas. Repeat the shaking and venting procedure until the evolution of the gas appears to be complete.
11. Using a filter-pipet, carefully remove the lower water layer and discard. The water-insoluble ester is in the top layer.
12. Repeat the process two more times with fresh portions of the sodium bicarbonate solution.
13. Transfer the ester to a dry 3-mL conical vial using a Pasteur pipet. Add two microspatulas full of granular anhydrous sodium sulfate. The sodium sulfate will absorb any remaining water. The sample should be left in contact with the sodium sulfate for at least 10-15 minutes.
14. Obtain infrared (IR) spectra of the isopentyl alcohol (the starting material) and the isopentyl acetate (the product). Locate the alcohol and carbonyl peaks.



Pre-lab Questions

1. Concentrated sulfuric acid is used in this reaction as a catalyst.
 - a. Briefly state the function of a catalyst in a chemical reaction.

 - b. How does the amount of catalyst used at the start of a reaction compare to the amount present at the end of a reaction?

2. In addition to the concentrated sulfuric acid, glacial acetic acid is also used in this experiment. Sulfuric acid is a strong acid, but acetic acid is a weak acid.
 - a. What does the term "glacial" indicate about the concentration of the acetic acid?

 - b. Compare the hazards these acids. (Is one of the acids more dangerous than the other?)

 - c. List two safety precautions that you should follow to reduce the possibility of injury from these acids.

3. Predict the major peaks in the infrared (IR) spectra of the starting alcohol (isopentyl alcohol) and the ester product (isopentyl acetate) in this experiment.

isopentyl alcohol

isopentyl acetate

Post-lab Questions

1. The procedure for this experiment used 1.4 mL of acetic acid, 0.7 mL of isopentyl alcohol, and 2-3 drops of concentrated sulfuric acid. What would be the impact on the reaction if the amounts of the reagents were altered in the following ways? Consider each case separately.
 - a. 3 mL of acetic acid

 - b. 3 mL of isopentyl alcohol

 - c. 10 drops of sulfuric acid

2. Suppose that the infrared (IR) spectrum of your ester contained a peak at around 3300 cm^{-1} . What substance(s) could be responsible for this peak?

3. What would be the likely cause of a vinegary smelling product in this reaction? Explain.

4. Name the ester that would be created by reacting the following:
 - a. 2-propanol and octanoic acid
 - b. cyclohexanol and benzoic acid

5. Draw a structure for the ester that would be created by reacting the following:
- 1-butanol and acetic acid
 - 2-methyl-2-propanol and formic acid
 - phenol and pentanoic acid
6. Name the alcohol and carboxylic acid used to create the following esters
- cyclopentyl decanoate
 - heptyl heptanoate
7. Write chemical equations, using structural formulas, for the reactions that form the following esters:
- ethyl benzoate
 - sec-butyl hexanoate
 - isobutyl acetate