SYNTHESIS OF AN ARTIFICIAL FLAVOR

Introduction
Esters occur naturally in plants and fruits. They are responsible for most of the fragrance given off from flowers and are what give fruits their characteristic aroma. These esters are commonly prepared synthetically in large quantities for commercial use as artificial fruit essences and other flavorings and as components of perfumes. Esters can be synthesized in the lab by combining an alcohol with a carboxylic acid, then heating it in the presence of an acid catalyst. In this experiment different alcohol/carboxylic acid combinations will be used to synthesize a range of esters. The acid catalyst will be concentrated sulfuric acid. The volume as well as the mass and percent yield of product will be determined. Observations of the physical properties will also be noted.

Experimental Procedure

NOTE: The organic liquids used are very flammable. Do not use an open flame to heat samples. Hot plates will be provided.

<table>
<thead>
<tr>
<th>Ester</th>
<th>Alcohol</th>
<th>Carboxylic acid</th>
<th>Theoretical yield of ester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isoamyl acetate</td>
<td>4.0ml isoamyl alcohol</td>
<td>3.0ml acetic acid</td>
<td>4.8g</td>
</tr>
<tr>
<td>Isoamyl butyrate</td>
<td>3.0ml isoamyl alcohol</td>
<td>2.0ml butyric acid</td>
<td>3.44g</td>
</tr>
<tr>
<td>methyl salicylate</td>
<td>5-6ml methanol</td>
<td>2.0g salicylic acid</td>
<td>2.20g</td>
</tr>
<tr>
<td>methyl butyrate</td>
<td>2.4ml methanol</td>
<td>3.0ml butyric acid</td>
<td>3.33g</td>
</tr>
</tbody>
</table>

Alcohol/Carboxylic acid combinations
1. Chill about 35ml water. Set aside for later use.
2. Add the measured amounts of alcohol and carboxylic acid together in a 50ml Erlenmeyer flask. Add 1ml concentrated sulfuric acid. Swirl the flask to thoroughly mix.
3. In your student hood located at your workstation, set up a hot water bath using a 250ml beaker of water and a hot plate. Place the Erlenmeyer flask from step 2 into the bath and heat the flask at a GENTLE boil for about 20 minutes. Do not let the ester in the flask boil away so adjust the temperature of the hot water bath accordingly.
4. Cool the reaction mixture in the flask.
5. Add 25ml of cold water prepared in step 1 to a beaker. Add the cooled reaction mixture. Stir thoroughly.
6. After stirring, let the mixture settle. Two layers will form. One layer is the ester and the other layer contains the water and water-soluble compounds. Determine which layer is the ester.
7. Pour the mixture into a container of small diameter. Pipet out the ester layer and place it in a clean dry preweighed beaker. (Do not discard either layer until the experiment is complete.)
8. Add 25ml saturated sodium bicarbonate solution to the separated ester layer. Swirl thoroughly and separate again as in step 7.
9. Measure the volume of the separated ester. Note the properties.

**Ester Densities**
- Isoamyl acetate – 0.867 g/ml
- Isoamyl butyrate – 0.866 g/ml
- Methyl butyrate – 0.898 g/ml
- Methyl salicylate – 1.174 g/ml
1. Volume of product (ml)

2. Mass of product (g)

3. Percent yield (%)

4. Appearance of product (color, odor, viscosity, etc.)

5. How did you identify the ester layer?

6. Your ester product is not water soluble, yet the starting alcohol and acids are water soluble. Draw molecular pictures indicating why this is possible.

7. What was the sodium bicarbonate used for? Write a chemical equation to show what happens when each of the starting materials is treated with sodium bicarbonate (a weak base).

8. Discuss reasons why the yield was not 100% (areas where loss could have occurred.)