Esterification: Synthesis of Isopentyl Acetate (Banana Oil)

STUDENT HANDOUT

Equipment

Microscale kits, stirring hot plates, gas chromatograph

Supplies

Safety goggles, isopentyl alcohol, glacial acetic acid, concentrated sulfuric acid, aluminum heating block and collar, 5% aqueous sodium bicarbonate, Pasteur pipets, distilled water, acetone, numbered plastic vial, microtubes.

Purpose

To synthesize isopentyl acetate.

Procedure

1. Assemble the reflux apparatus according to written and verbal directions. The teacher will review this process before the van visit. See Figure 1.

2. Take a 3.0mL conical vial with a magnetic spin vane, and using a micropipettor preset to 1.0mL by the teacher add 1.0mL of isopentyl alcohol.

3. Using a preset micropipettor add 0.550mL of glacial acetic acid.

4. Add four drops of concentrated sulfuric acid using a dropper bottle.

5. Replace 3mL vial on apparatus. **Do not overtighten.** Lower apparatus onto hotplate. Turn on the magnetic stirrer.

6. Make sure water is running through the condenser. Heat the mixture to a boil in a heating block on the hot plate (be sure to stir the mixture with a magnetic stirrer.)

7. After the reflux band is visible, continue heating under reflux for 20 minutes. Maintain the temperature between 160°C and 180°C.
Figure 1. Synthesis apparatus
8. Remove the apparatus from the hot plate and allow the mixture to cool to room temperature. Air cool for a couple of minutes and then use a damp paper towel wrapped around the 3mL vial to speed cooling. CAUTION: Do not place the vial on cool or cold surfaces until it has cooled sufficiently to avoid cracking the vial.

9. While waiting for the vial to cool, prepare a 5mL conical vial with 2mL of 5% aqueous sodium bicarbonate solution (NaHCO₃). NOTE: The micropipette can only deliver 1mL, so use it twice. You will be transferring your refluxed solution to this vial for extraction later.

10. Use a Pasteur pipet to transfer your solution to the 5mL conical vial prepared in step 9. Wait for the initial fizzing to subside, then cap the vial.

11. For this extraction, shake the capped vial and vent additional gas (shake and vent three times), then allow to stand until separate layers are formed.

12. Using a Pasteur pipet, remove the lower, aqueous layer and discard it into a waste collection beaker or sink.

13. Extract again by adding a fresh 2.0mL portion of 5% NaHCO₃ solution to the 5.0mL conical vial.

14. Again, shake and vent three times, then allow to stand until separate layers are formed.

15. Using your Pasteur pipet, again remove the lower aqueous layer and discard it.

16. Pour product into plastic numbered vial. Record the vial number and give vial to instructor.

--------------------------------------------------------------------------------------------------------------------------END OF DAY 1--------------------------------------------------------------------------------------------------------------------------

17. Pour product from numbered vial into 5mL glass vial.

18. Add a small amount of anhydrous MgSO₄ to your product in the 5mL vial to dry the organic layer. (This amount varies depending on how much ester you have made - please see instructor.)

STOP!! IF YOU HAVE LESS THAN 40 MINUTES LEFT, DO THE FOLLOWING:

a) Pre-weigh a colored microtube and record its mass.

b) Carefully pour sample into the microtube leaving the drying agent behind.
c) Weigh the sample microtube and contents and determine the mass of the ester by subtracting.

d) Clean the 5mL conical vial by rinsing with distilled water and finally rinsing with acetone.

**GAS CHROMATOGRAPHIC ANALYSIS OF PRODUCT:**

24. Following your teachers instructions, inject one μL of your sample into a gas chromatograph. Identify and label the peaks and their retention times on your print-out. Compare your results with those of the known standards. Complete the data table using your printout.
Figure 2. Distillation Apparatus
DATA COLLECTION AND CALCULATIONS

Calculate the percentage yield of the ester using the following steps and data from your experiment.

1. Calculate the number of moles of isopentyl alcohol
   (MW = 88.2 g/mole, density = 0.813 g/mL).
   \[ \text{_______ mole} \]

2. Calculate the number of moles of acetic acid
   (MW = 60.1 g/mole, density = 1.06 g/mL).
   \[ \text{_______ mole} \]

3. What is the limiting reactant?
   \[ \text{__________________________} \]

4. Calculate the theoretical yield.
   \[ \text{Yield = ______ g} \]

5. Calculate the percent yield
   (Remember: % yield = actual/theoretical x 100)
   Mass of your product and microtube
   \[ \text{________ g} \]
   Mass of your microtube only
   \[ \text{_______ g} \]
   Mass of your product
   \[ \text{_______ g} \]

   \[ \% \text{yield} = ______ \]
6. **From your computer printout, complete the following tables.**

Gas Chromatograph Code  A  B  Circle the appropriate code.

Be sure to use retention times for standards from the same gas chromatograph as your sample is run on.

<table>
<thead>
<tr>
<th>Known Standards</th>
<th>Student Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Compound</strong></td>
<td><strong>Peak</strong></td>
</tr>
<tr>
<td><strong>Retention Times</strong></td>
<td><strong>Retention Time</strong></td>
</tr>
<tr>
<td>Water</td>
<td>Peak 1</td>
</tr>
<tr>
<td>Acetic Acid</td>
<td>Peak 2</td>
</tr>
<tr>
<td>Isopentyl Alcohol</td>
<td>Peak 3</td>
</tr>
<tr>
<td>Isopentyl Acetate</td>
<td>Peak 4</td>
</tr>
</tbody>
</table>
7. By comparing retention times between your sample and the known standards, identify your peaks.

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>% OF SAMPLE (from printout)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak 1</td>
<td></td>
</tr>
<tr>
<td>Peak 2</td>
<td></td>
</tr>
<tr>
<td>Peak 3</td>
<td></td>
</tr>
<tr>
<td>Peak 4</td>
<td></td>
</tr>
</tbody>
</table>

8. Why shouldn’t you have found any acid in your final sample?

9. Why did you find water in your final sample?

10. How could you have reduced the amount of water in your final sample?

11. Why did you find isopentyl alcohol in your final sample?
12. How could you have reduced the amount of alcohol in your final sample?

13. By following the best possible techniques, which peaks should be minimized, which should be maximized?

14. How did your group compare to others in your class/school?

15. What suggestions would you have to improve your results?