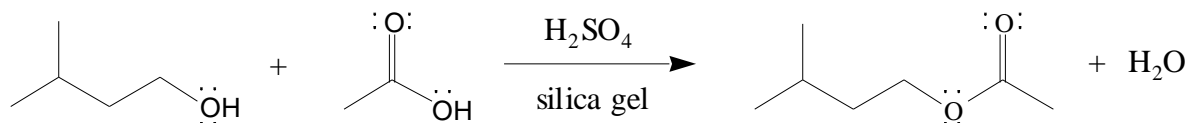


Esterification Reaction

Introduction

A carboxylic acid and an alcohol can be combined to make an ester using a catalytic amount of sulfuric acid. In this lab, we will use acetic acid and isopentyl alcohol to form isopentyl acetate, which is known as banana oil because it smells like bananas. It is found naturally in bananas, and is also used as an artificial flavoring in popsicles, candy, and baked goods.



This reaction is reversible, and the equilibrium constant is around 1. In order to favor the formation of the product, we will add silica gel, which will remove the water that is formed in the reaction.

We will run this reaction without a solvent, which is often referred to as running a reaction "neat." One of the reagents is liquid and the others dissolve in it. Because of this, we will do the necessary extractions in the conical vial rather than a separatory funnel to minimize loss of the product on the surface of the glassware.

Reagent	volume	density	mass	MW	mmoles	equivalents
isopentyl alcohol	1.50 ml					
glacial acetic acid	0.83 ml					
sulfuric acid (conc)	4 drops	x	x	x	x	catalytic
silica gel	x	x	100 mg	x	x	x
isopentyl acetate						

Boiling point of isopentyl alcohol: _____

Boiling point of isopentyl acetate: _____

Procedure

Before you come to lab:

- Review the esterification of carboxylic acids in your lecture notes. (If reaction this hasn't been covered in class, use the textbook or online sources to get an idea of what it involves.)
- Read through the lab procedure. Try to think of answers to the questions in italics.

- Review the following techniques:
 refluxing a reaction
 extraction and washing
 purifying a liquid by distillation
 measuring a boiling point
 characterizing a compound by IR spectroscopy
- Do the calculations in the table above, and locate the boiling points for isopentyl alcohol and isopentyl acetate.
- Write an introduction in your lab notebook, making sure to include the reaction and the reagent table.
- Do the on-line pre-lab questions.

During lab:

Set-up and run the reaction:

- Prepare a CaCl₂ drying tube. (*Why is this needed?*)
- Put the following in a conical vial: isopentyl alcohol, acetic acid, sulfuric acid, silica gel, and a spin vane.
- Set up a reflux condenser, and attach the conical vial and drying tube. Use a yellow clip to secure the drying tube to the reflux condenser, but not the condenser to the vial. (*Why not?*)
- Heat the reaction to reflux.
- Reflux the reaction for one hour. Check to make sure that the reflux condenser is cool to the touch. Turn up the heat if necessary to ensure that it continues to reflux. (*Why are these precautions necessary?*)

Isolate the product:

- Allow the reaction to cool.
- Transfer the liquid contents of the reaction to a clean conical vial (leaving the spin vane and silica gel behind).
- Washing #1: Add a layer of saturated aqueous NaCl, put a cap and septum on top, and shake to mix. Remove the water layer with a pipet. (*What does this accomplish?*)
- Washing #2: Add a layer of saturated aqueous NaHCO₃, cap and shake as before. Be careful to allow for evolving gas to escape. Remove the water layer. Repeat until no more bubbling occurs. (*What is happening? Where do the bubbles come from?*)
- Place the product layer in a 10 ml beaker and dry it over Na₂SO₄. Make sure to use a minimum of drying agent. (*Why?*)

Purify the product:

- Remove the crude product from the beaker to a clean conical vial. Use the markings on the vial to check the approximate amount of product you have obtained. If you have less than 0.3 ml, check with me about joining another group for this step.
- Set up a distillation apparatus with the conical vial and a spin vane, then a Hickman still, then a clean reflux condenser, then a thermometer, and finally some aluminum foil for insulation. Make sure to use a still that doesn't get narrow at the bottom.
- Slowly heat the crude product until it boils, monitoring the temperature. Record the boiling temperature of the liquid as it distills. (*What temperature should you expect?*) Don't continue heating when all of the liquid is gone, as this bakes the impurities onto the vial.
- Collect the distilled product in a tared container.

Characterize the product:

- Observe the appearance and odor of the product. (*What should it smell like?*)
- Obtain a mass and calculate the % yield that you obtained. The average student yield for this reaction is 34%.
- Compare the boiling point that you obtained during distillation to the boiling point of the authentic compound. (*Should you expect it to be exactly the same?*)
- Obtain an IR of the product. Compare it to the IR's of the two starting materials, looking for evidence of that the product was formed and the starting materials are not present. (*What should you see?*) Label your IR and turn it in with your lab report.
- Your conclusion should discuss the appearance and odor of your product; the mass and % yield, comparing it to the average student yield; the boiling point that you obtained, comparing it to the boiling point of the authentic compound; and the IR bands, including whether the expected bands were present, and whether any impurities were evident.

After lab:

- Finish writing up your procedure and observations.
- Print out the questions on the next page and fill them out. Turn in this sheet with the carbon copies of your lab and your IR (lab stapled to IR, questions separate).

Questions for Esterification Reaction

Name: _____

- 1) Draw the complete mechanism for this reaction, showing all steps individually! Identify the key nucleophile and electrophile in the reaction.
- 2) Use the mechanism above to explain why only four drops of sulfuric acid are needed.
- 3) How would the results of this reaction be different if no silica gel were added to this reaction?
- 4) If you were to use a thermometer to monitor the temperature of this reaction during reflux, what would you observe, and why?
- 5) What is the purpose of the bicarbonate washes? Write one of the two reactions which produces the bubbles.
- 6) Why is the boiling point measured during the distillation likely to be lower than the literature value, even if the product is pure?
- 7) How does IR give a good indication of the purity of the product in this reaction? What impurities would it reveal?