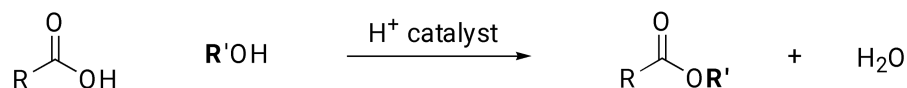


Synthesis of Fragrant Esters

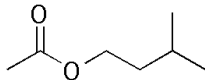
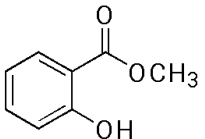
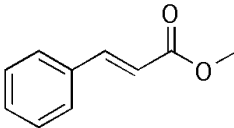
Background

Esters with low molar masses often have pleasant fruity or floral scents. Fragrant esters and other organic compounds can be extracted from natural sources, like fruits and flowers, with ethyl alcohol and concentrated to make what are known as essential oils. Esters can also be made in a laboratory; the flavor and fragrance industry makes many naturally occurring and synthetic esters that are added to perfume and food products.

In this lab you will synthesize three fragrant esters using the Fisher esterification reaction. In Fisher esterifications, a carboxylic acid and an alcohol are heated in the presence of an acid catalyst to form the ester.



You will synthesize, isolate and characterize isoamyl acetate, commonly known as banana oil. You will also synthesize two other esters, methyl salicylate and methyl cinnamate. These you will characterize by odor only and they do not require a mole table.

Alcohol	Acid	Ester	Structure	Odor
isopentyl alcohol	acetic acid	isopentyl acetate		bananas
methanol	salicylic acid	methyl salicylate		wintergreen
methanol	cinnamic acid	methyl cinnamate		strawberries

The odor of volatile compounds can vary with concentration. Also, different people may have different impressions of the same molecule. Don't worry about getting the "correct" scent in this lab. As always, write what you observe, not what you think you should observe.

Procedure

Synthesis of Isoamyl Acetate

Reaction Setup

Place 15 mL of isoamyl alcohol and 20 mL of glacial acetic acid in a 250 mL round bottom flask and add two boiling chips. Securely clamp the flask and **cautiously** add 1.5 mL of concentrated sulfuric acid. Attach a reflux condenser to the flask and begin heating on medium. Once the flask comes to a boil, reflux for 45 minutes then allow to cool to room temperature. Overheating may result in a dark by-product forming – reduce heat if you see color developing, which usually starts at the air-liquid interface.

Isolation and Purification

Pour 50 mL of water into the cooled reaction flask, then pour the contents of the flask into a separatory funnel. Remove the aqueous layer then wash the organic layer with two 25 mL portions of 5% bicarbonate solution. **Caution** – large amounts of carbon dioxide may be evolved – vent frequently! Next, wash with 25 mL of water. Separate the organic layer and dry it over magnesium sulfate.

Filter the organic layer into another round bottom flask and set up a simple distillation. When the stillhead reaches 137°C, begin collecting in a pre-weighed vial. Stop collecting when there is only a few drops left in the stillpot or when the temperature goes higher than 144°C.

Characterization

Record the boiling point range for the product as it distills. Calculate the percent yield. Characterize by IR and compare the fragrance of starting materials and products.

Synthesis of Methyl Salicylate and Methyl Cinnamate

Synthesis

For each ester, place 1.0 mL of the alcohol and 0.60 g of the acid in a test tube. **Cautiously** add 5 drops of concentrated sulfuric acid to the test tube. Heat gently in a 65-75°C water bath for 20 to 30 minutes. Cool the test tube in an ice bath then pour the contents into a 100 mL beaker containing 10 mL of deionized water.

Characterization

Characterize by scent, compare the odor of reactants and products. If the sharp scent of unreacted acid makes it difficult to identify the odor of the ester, add a few drops of 5% sodium bicarbonate to neutralize the acid smell.

Chemicals: isoamyl alcohol, acetic acid, concentrated sulfuric acid, 5% sodium bicarbonate, magnesium sulfate, methanol, salicylic acid, cinnamic acid.