Preparation of Methyl Benzoate

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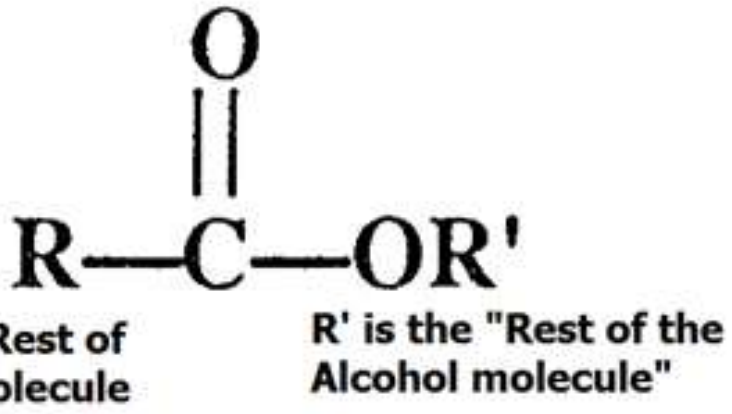
Esters **are chemical compounds derived from an acid (organic or inorganic) in which at least one –OH (hydroxyl) group is replaced by an –O–alkyl (alkoxy) group.Usually, esters are derived from a carboxylic acid and an alcohol.**



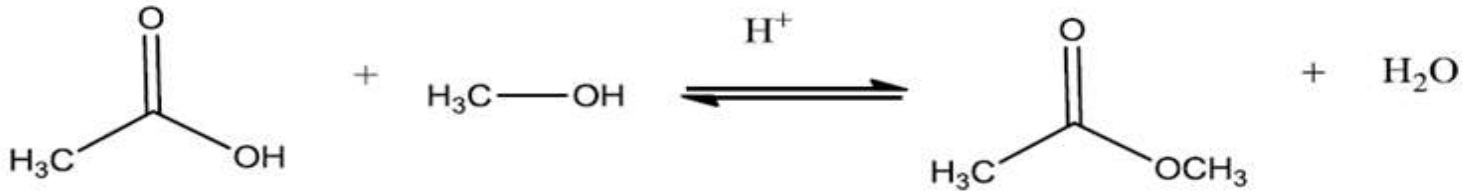
Outline

The ester group is an important functional group that can be synthesized in a number of different ways. The low-molecular-weight esters have very pleasant odors and indeed are the major components of the flavor and odor aspects of a number of fruits.

Although the natural flavor may contain nearly a hundred different compounds, single esters approximate the natural odors and are often used in the food industry for artificial flavors and fragrances.

Esters can be prepared by the reaction of a carboxylic acid with an alcohol in the presence of a catalyst such as concentrated sulfuric acid, hydrogen chloride

p-toluenesulfonic acid, or the acid form of an ion exchange resin:

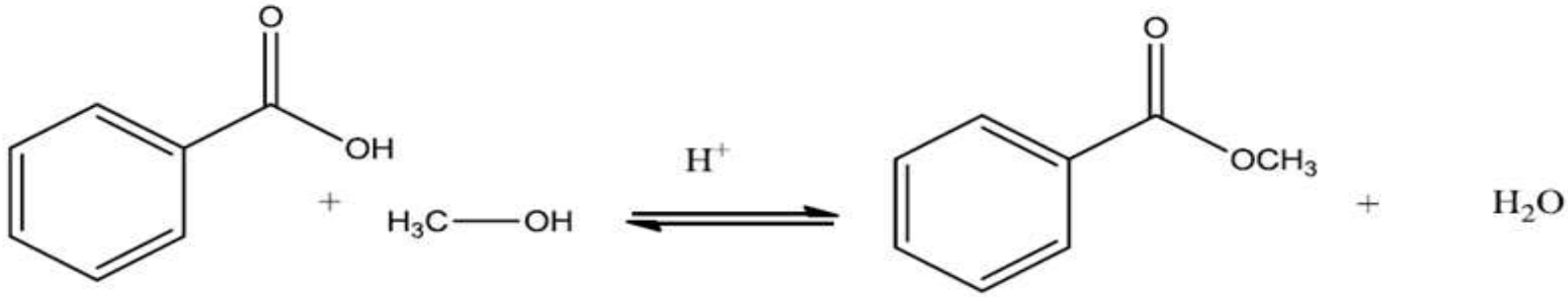


This Fischer esterification reaction reaches equilibrium after a few hours of refluxing. The position of the equilibrium can be shifted by adding

- **more of the acid or of the alcohol,**
- **depending on cost or availability.**

The mechanism of the reaction involves initial protonation of the carboxyl group, attack by the nucleophilic hydroxyl, a proton transfer, and loss of water followed by loss of the catalyzing proton to give the ester.

In this experiment you will prepare methyl benzoate by reacting benzoic acid with methanol using sulfuric acid as a catalyst. Since this is a reversible reaction, it will reach an equilibrium that is described by the equilibrium constant, Keq .



For this experiment, you will isolate the product, methyl benzoate, and any unreacted benzoic acid. Using this data, you will calculate the equilibrium constant for the reaction .

Chemicals Required:

- 0.1 mol benzoic acid
- 40 mL methanol
- 3.0 mL of concentrated sulfuric acid
- 50mL methylene chloride
- 20 mL 5% sodium carbonate

Procedure : (((A. Reflux)))

1. Add 0.61 g of benzoic acid to a round bottom flask.

2. Add 2 mL of methanol

3. Add 0.15 mL of concentrated sulfuric acid

4. Mix all reactants and add boiling stones

5. Attach a water cooled reflux condenser

6. Reflux for one hour

(((B. Separation)))

1. Cool the mixture to room temperature

2. Decant the mixture into a separatory funnel

3. Rinse the round bottom flask with 10 mL of methylene chloride into the funnel

4. Add 40 mL of methylene chloride and 40 mL of water to the funnel

5. Extract the methyl benzoate into the methylene chloride layer by shaking

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6. Separate the organic and aqueous phases

(((C. Washing))) (do not discard any materials during this step)

1. **Wash the organic layer with 20 mL of R. I. water**
2. **Wash the organic layer with 20 mL of 5% sodium carbonate**
3. **Repeat step 2**
4. **Wash the organic layer with water**
5. **Drain the organic layer into a dry flask and dry with magnesium sulfate**

6. The aqueous layer from the sodium carbonate wash should be acidified with concentrated HCl. When this aqueous material is made strongly acidic with hydrochloric acid, unreacted benzoic acid may be observed. The unreacted benzoic acid should precipitate. Remove the solvent by vacuum filtration.

- **Collect and dry the solid benzoic acid and let it air-dry until the next lab period. You will need this weight for the Keq calculations.**

(((D. Distillations)))

1. Transfer the dried product (minus the solids) and methylene chloride to a round bottom flask large enough to accommodate the total volume
2. Attach a three way adapter and short condenser and thermometer
3. Distill off the solvent, methylene chloride.
4. Transfer the residue from step 3 into a preweighed container. If needed, transfer the residue from step 3 to a small round bottom flask as possible, reattach the adapter and condenser and thermometer and distill the product into a preweighed container

(((E. Analysis)))

1. Determine the actual yield of the product and the unreacted (recovered) benzoic acid.

2. Determine the refractive index of the product

3. Obtain an IR spectrum of the product (reference spectrum are at the end of this document)

4. Calculate Keq

5. Calculate theoretical yield and determine % yield .

CALCULATIONS

Calculation of the % Yield based upon amount of benzoic acid that is consumed:

$$\%\ \text{yield} = \frac{(\text{moles methyl benzoate obtained})(100)}{(\text{Initial moles benzoic acid})}$$