

Laboratory 24: Properties of Carboxylic Acids and Esters

Introduction

In this laboratory we will explore the chemical and physical properties of carboxylic acids and esters.

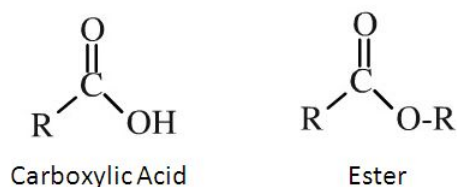
Discussion

Structure of Carboxylic Acids and Esters

Carboxylic Acids and Esters are organic compounds containing a carbonyl carbon ($R-COO^-$) (Figure 1 below) connected with an oxygen atom. The carbonyl carbon is a polar group with the carbon having a slight excess of positive charge and the oxygen atom having a slight excess of negative charge. The addition of a second oxygen atom increases the strength of the dipole. Both molecules are capable of dipole-dipole interactions. One major difference between the molecules is the hydrogen atom attached to the oxygen atom in carboxylic acids which allows for the formation of hydrogen bonds between molecules.

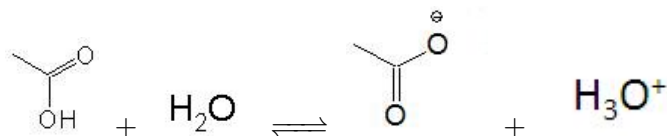
Carboxylic acids are often responsible for many flavors such as the tart taste of vinegar which is commonly called acetic acid but using IUPAC rules should be called ethanoic acid. The sour taste of fruits such as lemons is due to the presence of citric acid.

Unlike carboxylic acids which often have tart tastes or unpleasant odors, many esters have pleasant flavors and fragrances. The odor and flavor of oranges is due to octyl propanoate, that of pears due to pentyl propanoate, and raspberries from 2-methylpropyl methanoate.

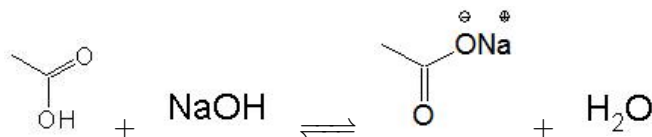


Chemical Properties

Carboxylic acids are considered weak acids because the carboxyl group ionizes slightly in water.

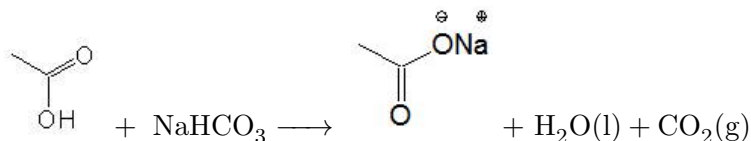


Carboxylic acids can react with bases (typically NaOH) to form carboxylic acid salts. Large carboxylic acids are insoluble in water, however, conversion to a salt greatly increases the solubility in water. Many carboxylic acids found in food products or medications are found in their soluble salt form rather than as the acid itself.

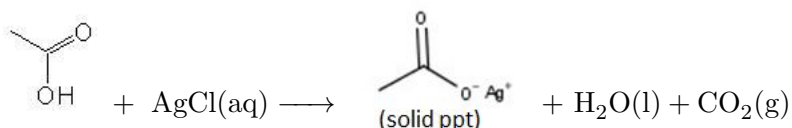


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A simple diagnostic test to differentiate between Carboxylic Acids and Esters is by reaction with sodium bicarbonate (a weak base) to produce a carboxylic acid salt, and carbonic acid (which decomposes to give carbon dioxide gas and water). Carboxylic Acids will give a positive result while Esters will give a negative result.



Another simple diagnostic test for the Carboxylic Acid functional groups by reaction with silver nitrate to form Carboxylic Acid Salt and an acid. Unlike normal carboxylic acid salts, the silver salt, is generally insoluble, though the addition of nitric acid will cause the precipitate to dissolve.



Esters are most often produced by the dehydration reaction between a carboxylic acid and an alcohol. The reaction generally takes place in the presence of an acid catalyst and produces an ester and water. A saponification reaction involves an ester hydrolysis in the presence of a strong base and results in the formation of a carboxylic acid salt and an alcohol. In general the ester is usually insoluble in water while the corresponding carboxylic acid salt and alcohol (if small) are not.

- Hydrolysis: Ester + H₂O $\xrightarrow{[\text{H}^+ \text{ or Enzyme}]}$ Carboxylic Acid + Alcohol.
- Saponification: Ester + Strong Base $\xrightarrow{[\text{H}_2\text{O}]}$ Carboxylic Acid Salt + Alcohol

Procedure

A. Odors of Carboxylic Acids and Esters

1. Carefully detect the odor of all the compounds by carefully wafting.

B. Carboxylic Acids - Solubility and Acid Character

1. Test the solubility of each of the listed substance with water by adding 10 drops of the substance to be tested to a test tube. For the solids use the amount that fits on the tip of the spatula. Add 2 mL of water to each test tube.
2. Mix each of the test tubes vigorously for 15 seconds. Wait 30 seconds. Sketch a picture of your results. In the sketch, label each liquid or solid in the test tube. Note which pairs are soluble/insoluble or miscible/immiscible and which are not.
3. For any solutions that are insoluble note the relative density of the compounds with respect to water.
4. Test the pH of each solution using the pH paper supplied. Compare the color on the paper to the color chart on the container and report the pH.
5. For any insoluble carboxylic acids, place them in a hot water bath for 3 minutes. Record your results. Note which pairs are soluble/insoluble or miscible/immiscible and which are not. Allow the tubes to cool (10-15 minutes) and record your results.
6. Add drops of 10% NaOH to each test tube until a drop of the solution turns red litmus paper blue (This should be approximately 10 drops). Record your observations. Note which pairs are soluble/insoluble or miscible/immiscible and which are not.

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7. Add 10 drops of 10% HCl to each solution until they are neutralized (blue litmus paper turns red). Record your observations. Note which pairs are soluble/insoluble or miscible/immiscible and which are not.
8. Dispose of the solutions in the waste bottle labeled "E24 Waste".

C. Reaction with Sodium Bicarbonate

1. Mix 10 drops or 0.5 mL (or 0.1 gram) of the compound to be tested with 1 mL of methanol.
2. Carefully (drop wise) 1 mL of saturated sodium bicarbonate solution to the above mixture.
3. Record your observations.
4. Dispose of the solutions in the sink.

D. Reaction with Silver Nitrate

1. Mix 10 drop or 0.5 mL (or 0.1 gram) of the compound to be tested with 2 mL of 2% ethanolic silver nitrate solution.
2. If no reaction is observed after 5 minutes at room temperature, heat the solution to boiling.
3. Record your observations.
4. If a precipitate forms, add 5 drops of 6 M HNO₃. Record your observations.
5. Dispose of the solutions in the waste bottle labeled "E24 Waste".

E. Formation of Esters

1. Make only your assigned ester.
2. Place 2 mL of the alcohol in a test tube.
3. Add 3 mL of the carboxylic acid (or 0.5 grams if solid) to the test tube.
4. Carefully (holding the test tube pointed away from you) add 10 drops of concentrated sulfuric acid to each test tube.
5. Heat each test tube in boiling water for 3 minutes.
6. Carefully detect the odor of each of the esters produced.
7. Dispose of the contents of the test tubes in the waste bottle labeled "E24 Waste".
8. Make sure to draw the structure of **ALL** reactants and products, not just the reaction you were assigned.
9. Make sure to name each product produced.

F. Hydrolysis of Esters

1. Place 3 mL of water in a test tube. Add 5 drops of methyl salicylate. Record the appearance and odor of the ester.
2. Add 1 mL (20 drops) of 10% NaOH solution. Two layers should form in the test tube. Place the test tube in a boiling water bath for 30 minutes or until the top layer disappears.
3. Record any changes in the odor of the ester.
4. Remove the test tube and cool it to room temperature in a cold water bath.
5. After the solution is cool, add about 1 mL (20 drops) of 10% HCl dropwise until one drop turns blue litmus paper red. Record your observations.
6. Dispose of the contents of the test tubes in the waste bottle labeled "E24 Waste".

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Name: _____

Class: _____

Date: _____

Results

A. Structures and Odor of Common Carboxylic Acids and Esters

Compound	Structure	Odor
2-methylpropyl methanoate (isobutyl formate)		
ethyl butanoate (ethyl butyrate)		
3-methylbutyl ethanoate (isoamyl acetate)		
octyl ethanoate (octyl acetate)		
methyl 2-hydroxybenzoate (methyl salicylate)		
ethanoic acid (acetic acid)		

Table 1: Results - Structures and Odors

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B. Solubility of Carboxylic Acids and Esters

Test	Acetic Acid (6 M)	Benzoic Acid
Structure		
Solubility in cold water		
pH		
Solubility in warm water		
Solubility after cooling		
Addition of NaOH		
Addition of HCl		

Table 2: Results - Carboxylic Acids

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C. and D. Sodium Bicarbonate Test and Silver Nitrate

Compound	NaHCO ₃ Test	Silver Nitrate
Acetic Acid		
Benzoic Acid		
Ester Assigned: _____		

Table 3: Results - Sodium Bicarbonate and Silver Nitrate

1. Write the complete (including states) balanced chemical reaction that occurs when acetic acid reacts with sodium bicarbonate

2. Write the complete (including states) balanced chemical reaction that occurs when acetic acid reacts with silver nitrate.

3. Did any test results not match expectations? Explain.

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E. Preparation of Esters

Reactants (Draw Line or Lewis Structure)	Odor	Structure and IUPAC Name of Ester
methanol + salicylic acid (o-hydroxybenzoic acid)		
1-pentanol + acetic acid		
1-octanol + acetic acid		
1-Propanol + acetic acid		
phenylmethanol + acetic acid		

Table 4: Results - Preparation of Esters

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F. Hydrolysis of Esters

Reactants	Observations
Structure of methyl salicylate	
Appearance and Odor	
Observations after adding NaOH and heating	
Chemical equation for saponification of methyl salicylate	
Observations on addition of HCl	
Chemical equation for above observations	

Table 5: Results - Hydrolysis of Esters

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Name: _____

Class: _____

Date: _____

Prelab Questions

1. Draw the Line or Lewis structure of all molecules in Part A, B and E of the Results section, and show it to your instructor before beginning the laboratory.
2. Draw an example showing the hydrogen bonding which occurs between two acetic acid molecules.
3. Why are large carboxylic acids generally solids?
4. What is the formula of sodium bicarbonate? What is the visible evidence of a positive result for the sodium bicarbonate test?
5. What is the formula of silver nitrate? What is the visible evidence of a positive result for the silver nitrate test?

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Another chance meeting. Are you stalking me? Oh good, I only thought that in my head and did not say it out loud. Shall we continue our pleasant conversation? What is your favorite food?