

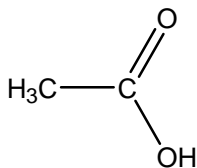
ESTERS

An Introduction to Organic Chemistry Reactions

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In contrast to inorganic reactions, which usually involve metals combining with non-metals or polyatomic ions, organic reactions occur as additions or substitutions into a compound or as a condensation-type reaction where removal of certain atoms or groups cause two molecules to combine. One type of reaction, that will be observed in this experiment, is a dehydration between an organic acid and an alcohol to produce an ester.

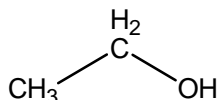
An organic acid has a structure:



This compound, which contains two carbon atoms with a  group (called a carboxyl

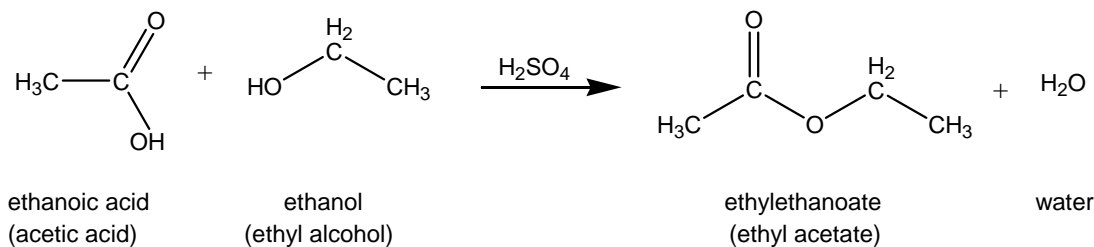
group) on one end of the molecule is ethanoic acid, commonly called acetic acid or vinegar (a 5% solution of acetic acid).

An organic alcohol has a structure:



This compound, which contains two carbon atoms with a $-\text{C}-\text{OH}$ structure on one end of the molecule is ethanol, commonly called ethyl alcohol.

When an organic acid is mixed with an alcohol in the presence of a strong, dehydrating acid, such as sulfuric acid, H_2SO_4 , the molecules are joined together to form an ester with the removal of a water molecule:



The ester in the reaction, above, is called ethylethanoate, or commonly, ethyl acetate.

To obtain the pure ester, one would have to distill it from the mixture of products obtained in the reaction.

Esters occur in many plants and are, in part, responsible for the distinctive odors of those plants and the flavors of their fruits.

Esters are commonly used in the preparation of artificial flavors or fragrances. Flavors and fragrances may utilize a single ester or a mixture of esters and other substances to duplicate the odor and flavor of a particular fruit. Some esters that smell like common materials that can be prepared in the laboratory are listed in Table 1.

Table 1. Common esters used for flavors and fragrances

Ester	Smells like	Prepared from	
		Alcohol	Acid
isoamyl acetate	Banana	Isoamyl alcohol	Acetic acid
ethyl butyrate	Pineapple	Ethanol	Butanoic acid
amyl acetate	Pineapple	Amyl alcohol	Acetic acid
benzyl acetate	Peaches	Benzyl alcohol	Acetic acid
n-propyl acetate	Pears	n-propyl alcohol	Acetic acid
benzyl butyrate	Flowers	Benzyl alcohol	Butanoic acid
methyl butyrate	Apples	Methanol	Butanoic acid
isobutyl propionate	Rum	Isobutyl alcohol	Propionic acid
octyl acetate	Oranges	Octanol	Acetic acid
methyl anthranilate	Grapes	methanol	2-aminobenzoic acid
isobutyl formate	Raspberry	Isobutyl alcohol	Formic acid
ethyl valerate	Walnut	Ethanol	Valeric acid

Esters can also be found in products such as scratch and sniff spots which are often used in magazines, direct mail advertisements, stickers, or children's books. In these products, the esters are microencapsulated. An individual usually uses a fingernail to mechanically break the small microcapsules to release the contents.

Microencapsulation is a process where substances such as inks or dyes, adhesives, cosmetics, pharmaceuticals, or fragrances are contained in microscopic capsules, 20 to 150 microns in diameter, which can be broken mechanically, electrically, or chemically to release the contents. The microcapsules are composed of different materials depending on the substance packaged and the method by which it is to be released. Gelatin is widely used as an encapsulating agent. The advantage of microencapsulation is that the capsules remain stable and inert until broken.

In this experiment, you will prepare a microscale quantity of a selected ester from the list of compounds in Table 1 and isolate it.. Since different groups, in the class, will prepare different esters, you will be able to compare the odors of the different esters.

PREPARING ESTERS

PROCEDURE

MATERIALS NEEDED:

Scratch and sniff sticker or similar
Organic acids and alcohols (see Table 1)
sulfuric acid, H_2SO_4 , (concentrated)
sodium carbonate, Na_2CO_3 10% solution
magnesium sulfate (anhydrous), MgSO_4
screw cap vial with a Teflon liner
copper wire to make a handle for the vial
graduated cylinder, 10 mL
test tubes (12 x 75 mm and either 15 x 150 mm or 18 x 150 mm)
400 mL beaker for water bath
Stirring rod
Beral pipet
Pasteur pipet
Glass wool
hot plate

SAFETY PRECAUTIONS:

Wear safety goggles or glasses

Sulfuric acid is corrosive causing severe burns. In the event of contact, rinse well with water for at least 10 minutes, then seek qualified medical assistance.

Phosphoric acid is corrosive causing severe burns. In the event of contact, rinse well with water for at least 10 minutes, then seek qualified medical assistance.

Concentrated acids react with water to produce heat. Always dilute acids by adding the acid to water slowly and with stirring.

Organic acids may be irritants to the skin and eyes. Some have strong odors. Use any compounds with strong odors under a fume hood.

Look up individual properties and hazards of each acid and take the recommended precautions.

Organic alcohols are flammable. Avoid flames.

Never heat any of the materials in this experiment directly, use a water bath.

If passing esters around for individuals to smell, use small quantities with a piece of glass wool in the container to prevent splashing or spilling.

DISPOSAL:

All organic materials should be disposed in the proper organic waste containers.

Dispose of concentrated acids by first diluting them by pouring the acid into a large beaker of water in a hood, and then neutralizing the solution with small amounts of sodium carbonate

(washing soda) or sodium bicarbonate (baking soda). The neutralized solution can be poured down the drain.

EXPERIMENTAL PROCEDURE:

Examining a Scratch and Sniff Sticker

Note the odor of a Smelly Patch/Sticker.

Scratch the Smelly Sticker once or twice with your fingernail. Does the intensity of the odor increase?

Bend the Smelly Sticker back and forth several times. Does the intensity of the odor increase?

Preparation of esters

This will be a group experiment. Each group will prepare one ester. The products will be placed in small vials and passed around to different groups.

Set up a water bath by placing 300 mL of distiller water in a 400-mL beaker on a hot plate. Heat the water to boiling as you prepare your reaction mixture.

Obtain a reaction vial. (The vials with a green colored cap contains a Teflon liner.)

Using Table 1 ("Prepared from" column) as a guide, mix 1.2 mL of alcohol with 2.5 mL of organic acid in the reaction vial. If the organic acid is solid, use 0.5 g of the compound. **NOTE:** not all the alcohols and acids listed in Table 1 will be available.

Add 5 drops sulfuric acid to the reaction vial.

Seal the vial tightly. Mix the reaction mixture by inverting the reaction vial several times.

Wrap the copper wire around the vial leaving several inches to serve as a handle.

Place the sealed vial in the boiling water bath. Bend the copper wire handle over the edge of the beaker to keep the vial upright.

Watch the vial to see if any bubbles are coming out of the vial cap. If this occurs, the vial is not tightly sealed. Removed the vial from the water bath, allow it to cool, then tighten the vial cap. Place the vial back in the boiling water bath.

Heat the vial for 20 to 25 minutes.

At the end of the heating period, remove the vial from the water bath and allow it to cool for about 2 minutes. Then place the vial in an ice water bath for 3 minutes to cool it completely.

Add 10 mL of ice cold water to a 15 x 150 mm or 18 x 150 mm test tube.

Wearing chemically resistant gloves, carefully open the reaction vial. **CAUTION:** The reaction mixture may squirt out of the vial.

Pour the reaction mixture into the ice water in the test tube. Gently stir the mixture to dissolve any unreacted materials.

Rinse out the reaction vial with distilled water and allow it to dry.

Use a Pasteur pipet to remove the organic layer (this is the upper layer) in the test tube and add it to a small 12 x 75 mm test tube.

Wash the crude ester twice with 2 mL of 10% sodium carbonate solution. **CAUTION:** The sodium carbonate will react vigorously with any sulfuric acid remaining in the crude ester forming carbon dioxide, CO_2 . Add the sodium carbonate solution dropwise to prevent the mixture from bubbling out of the test tube.

After each wash, use a pipet to remove as much of the lower aqueous layer from the test tube as possible.

Dry the ester with a small amount of anhydrous magnesium sulfate.

Pour the dry ester into the dry reaction vial. Place a small piece of glass wool into the vial. This will prevent spilling or splashing if it is passed around to a group of people.

Label the vial with the name of the ester and the names of the individuals in your group.

These samples of esters should remain stable for several months.

RESULTS:

Ester Name	Smells like
The ester our group prepared is:	