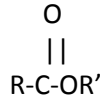
Synthesis of Esters

Introduction

Esters are a class of compounds widely distributed in nature. They have the general formula

(R and R' represent generic carbon groups.)



The simple esters tend to have pleasant odors. In many cases, although not exclusively so, the characteristic flavors and fragrances of flowers and fruits are due to compounds with the ester functional group. An exception is the case of essential oils. The organoleptic qualities (odors and flavors) of fruits and flowers may often be due to a single ester, but more often the flavor or aroma is due to a complex mixture in which a single ester predominates. Some common flavor principles are listed below.

Ester	Odor
isoamyl acetate	banana
ethyl acetate	fingernail polish remover
methyl salicylate	wintergreen
ethyl butyrate	strawberry
benzyl butyrate	cherry
ethyl propionate	rum
benzyl acetate	peach
methyl butyrate	apple
octyl acetate	orange
n-propyl acetate	pear
ethyl phenylacetate	honey

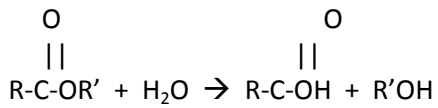
Food and beverage manufacturers are thoroughly familiar with these esters and often use them as additives to spruce up the flavor or odor of a dessert or beverage. Many times odors do not have a natural basis, as is the case with the "juicy fruit" principle, isopentenyl acetate. An instant pudding that has the flavor of rum may never have seen its alcoholic namesake—this flavor can be duplicated by the proper admixture, along with other minor components, of ethyl formate and isobutyl propionate. The natural flavor and odor are not exactly duplicated, but most people can be fooled. Often only a trained person with a high degree of gustatory perception, a professional taster, can tell the difference.

A single compound is rarely used in good-quality imitation flavoring agents. A formula for imitation pineapple flavor that might fool an expert includes 10 esters and carboxylic acids that can easily be synthesized in the laboratory, and 7 essential oils that are isolated from natural sources.

Flavor is a combination of taste, sensation and odor transmitted by receptors in the mouth (taste buds) and nose (olfactory receptors). There are four different tastes (sweet, sour, salty, and bitter). The perception of flavor, however, is not so simple. The human actually possesses 9000 taste buds and odor plays a big role in the perception of taste.

Although the "fruity" tastes and odor of esters are pleasant, they are seldom used in perfumes or scents that are applied to the body. The reason for this is that the ester group is not as stable to perspiration as the ingredients of the more expensive essential oils. The latter are usually hydrocarbons (terpenes), ketones and ethers extracted from natural sources. Esters are only used for the cheapest toilet waters, since on contact with

sweat, they hydrolyze, giving organic acids. These acids, unlike their precursor esters, generally do not have a pleasant odor.



Butyric acid, for instance, has a strong odor like that of rancid butter (of which it is an ingredient) and is a component of what we normally call body odor. Ethyl butyrate and methyl butyrate, however, are esters that smell like strawberry and apple, respectively.

In this experiment you will note the odor of six carboxylic acids and six alcohols. You will prepare up to eight esters using various combinations of these carboxylic acids and alcohols. From the odor of the esters and the list above you will identify the ester and then the carboxylic acid and alcohol from which it was made.

Materials

The lab will be set up with samples of 6 carboxylic acids and 6 alcohols.

You will also need:

- concentrated H_2SO_4
- Small test tubes
- test tube rack
- test tube holder
- glass stirring rod
- pieces of filter paper (or strips of paper towel)
- 150 mL beaker (to be use as a hot water bath)
- boiling chips

Waste & Safety

Dispose all of the ester products in the organic waste container.

Caution! Concentrated sulfuric acid will burn the eyes and skin. Wear goggles at all times. Use gloves. Wash your hands. Spills should be cleaned immediately; ask your instructor for assistance. Wash the counters at the end of lab.

WORK IN THE HOOD! Wear gloves. A few of the reagents give unpleasant odors that can be overwhelming. Keep your reagents capped and work only in the hood. Do not bring the reagents out to the benchtops! When asked to smell the reagents, carefully waft odors towards your nose; do not inhale!

Procedure

1. Start a hot water bath in a 150 mL beaker.
2. Each lab table will work together on the 8 trials. Each pair of lab partners can prepare 4 unique esters.
3. Put one drop of carboxylic acid and one drop of alcohol on opposite sides of a piece of filter paper. Waft the vapor toward your nose and describe the odor of the acid and alcohol. Open the cap of the container and waft the vapors toward your nose. Record the odor is the appropriate space in the following data table. The solid carboxylic acids used in this lab do not have a strong odor, so if you are assigned a solid, do not do this part.
4. Record the scent of your starting carboxylic acid and alcohol in your data table.
5. Add the number of drops/quantity (assigned to you) of each reagent to a small test tube. Swirl gently to mix the contents. Make sure to label your test tube with the trial letter you are assigned (A, B, C, etc.)
6. Add a drop of concentrated sulfuric acid to the test tube. (Sulfuric acid acts as a catalyst.) Add a boiling chip.

7. Using the test tube holder, place the test tube in a boiling water bath for 2-3 minutes. Watch the contents carefully to avoid boiling over. If the reaction mixture begins to boil too quickly, remove it from the water bath for a few seconds and slowly return it. Each test tube must be in the water bath for two minutes.
8. Use a stirring rod to transfer a drop of the reaction mixture to a clean piece of filter paper (or strip of paper towel). Waft the vapors toward your nose and record the odor of the new compound. If the mixture solidifies, waft the vapors from the solid material on the end of the stirring rod.
9. Identify the ester by the odor of the ester produced. Use the identification of the ester to identify the carboxylic acid and alcohol used in the reaction to produce the ester.

Data

TRIAL	Acid	Quantity	Acid Name	Alcohol	Quantity	Alcohol Name	Ester Name	Odor
A	#1	10 drops		#2	20 drops			
B	#1	10 drops		#3	20 drops			
C	#3	10 drops		#2	10 drops			
D	#4	20 drops		#2	15 drops			
E	#5	0.1 gram		#4	15 drops			
F	#6	0.4 gram		#2	20 drops			
G	#1	10 drops		#6	20 drops			
H	#3	10 drops		#1	20 drops			

Pre-lab Questions

1. Concentrated sulfuric acid is used as a catalyst in this experiment.
    a. Briefly state the function of a catalyst in a chemical reaction.
    b. How does the amount of catalyst used at the start of a reaction compare to the amount present at the end of a reaction?
2. Concentrated sulfuric acid can cause serious chemical burns. List two safety precautions that you can follow to reduce the possibility of injury from concentrated sulfuric acid.

Post-lab Questions

1. Write equations for each of the prepared esters (A-H).
2. If esters have a pleasant smell, why are they not commonly used for fragrances and perfumes? As part of your explanation, provide a reaction using the strawberry ester, ethyl butyrate.