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Preparation of Esters

Student Laboratory Kit

Catalog No. AP4362 Publication No. 4362

Introduction:

The purpose of this kit is to form and determine several types of esters.

Background:

Many esters have the characteristic property of a pleasant odor. As such, they are used as artificial flavors and perfumes. The flavors from products such as peppermint gum or banana-flavored ice cream most likely originate from organic compounds known as esters. Not all esters have odors, but most volatile esters do. An example of an odorless ester would be vegetable oil.

Esters are formed by the process of dehydration synthesis using an alcohol with an acid. The name of the ester is formed from the alcohol and acid used in their making. For example, when making the ester methyl salicylate (oil of wintergreen), the alcohol and acid used would be methyl alcohol and salicylic acid, respectively. This ester has a pleasant "peppermint" odor. Another example would be the ester butyl formate which is produced when reacting butyl alcohol with formic acid.

When an organic acid (RCOOH) reacts with an alcohol (R*OH), water is produced and an ester is formed. This process is an example of dehydration synthesis. Unfortunately, the reverse reaction also occurs so keeping a large excess of alcohol and minimizing the amount of water helps increase the yield of esters.

$$RCOOH + R^*OH \rightleftharpoons H_2O + RCOOR^*$$

for example...

$$\underset{\text{salicylic acid}}{C_6H_5OCOOH} + \underset{\text{methyl alcohol}}{CH_3OH} \longrightarrow \underset{\text{water}}{H_2O} + \underset{\text{methyl salicylate}}{C_6H_5OCOOCH_3}$$

Some artificial flavors are formed by the mixing of esters in an attempt to duplicate the flavor or odor of a natural substance. For example, the odor of raspberries originates from a mixture of isobutyl formate and isobutyl acetate. In this experiment three different esters having the characteristic odors of banana, peppermint, and fruit essences will be made. The product formed will be quite crude; however, the three different odors will be detected, and the basic method of producing esters will be performed.

Chemical Concepts:

- Esters
- Dehydration synthesis

Materials (for each lab group):

Equipment:

Styrofoam® cup (6 oz)
Beral pipets (thin-stem/long), 5
Beral pipets (extra large bulb/modified), 3
Cork stoppers (#00), 3
Bunsen burner
Thermometer (–10 to 110 °C)
Spatula

Chemicals:

Sulfuric acid, 18 M, H_2SO_4, 7 drops
Acetic acid (glacial), CH_3CO_2H, 20 drops
Salicylic acid, 2–HOC_6H_4COOH, .25 g
Isopentyl alcohol, $(CH_3)_2 CHCH_2CH_2OH$, 10 drops
Methyl alcohol, CH_3OH, 20 drops
Ethyl alcohol, C_2H_5OH, 10 drops
Hot water (70–80 °C), 50 mL

Safety Precautions:

Sulfuric acid is severely corrosive to eyes, skin, and other tissue. Acetic acid is corrosive to skin and tissue, is a moderate fire risk, and is moderately toxic by ingestion and inhalation. Salicylic acid is moderately toxic by ingestion. Isopentyl alcohol is slightly toxic by ingestion and inhalation, and a moderate fire risk. Methyl alcohol is a dangerous fire risk and is toxic by ingestion. Ethyl alcohol is a dangerous fire risk; addition of denaturant makes the product poisonous—it cannot be made non-poisonous. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron.

Procedure:

1. **Preparation of Three Esters:**

   Label the three modified extra large bulb pipets I, E, and M.

   Place into the pipet labeled "I" (see Figure 1): 10 drops of isopentyl alcohol, 10 drops of glacial acetic acid, and cautiously add 1 drop of concentrated sulfuric acid.

   Now stopper the pipet with a small cork and set the unit aside.

   Into the "E" labeled pipet, place 10 drops of ethyl alcohol, 10 drops of glacial acetic acid, and cautiously add 1 drop of concentrated sulfuric acid. Stopper this pipet and set aside.

   To the "M" labeled pipet, use a plastic funnel to fill the bulb about ¼ full (0.25 g) salicylic acid; then add 20 drops methyl alcohol to dissolve the salicylic acid. Now cautiously add 5 drops of concentrated sulfuric acid and stopper this unit.

2. **Examination of the Three Esters:**

   With the use of a Styrofoam cup, place all 3 units into a hot water bath (see Figure 2). The water used should be between 70 and 80 degrees Celsius. Careful, this is hot. After 10 minutes, remove the stopper and cautiously smell the odor of the esters. Pipet I contains isopentyl acetate (banana), and pipet E contains ethyl acetate (fruity). Before smelling the odor of pipet M, methyl salicylate, (peppermint) commonly known as "oil of wintergreen," add 5 drops of water to the ester.

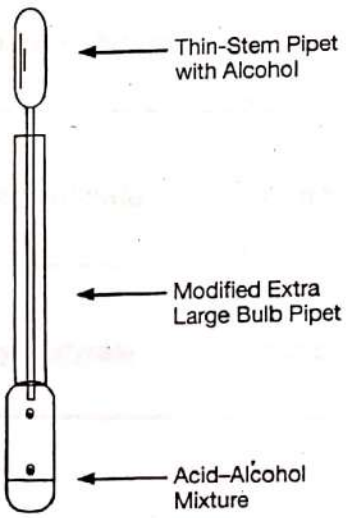

Figure 1

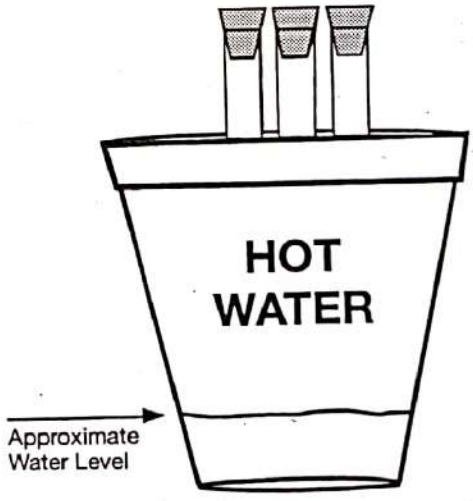

Figure 2

3. **Disposal:**

   See instructor for disposal procedures.

Name: _____ Date: _____ Pd: _____

Preparation of Esters Lab Experiment - Organic Chemistry

Data Sheet and Questions

Pre-Lab:

You will need to READ the "Preparation of Esters" handout. You will write numbered steps out for the procedure in your own words on a separate sheet of paper (attach it!) Failure to do so will result in not being allowed to complete the lab experiment and you will receive a ZERO.

Important: *You should start off with heating some water while you prepare your three esters so it is hot when you are ready to use it!*

Lab Observations: Record your scent observations in the table below.

Pipet Letter	Alcohol Used	Acid Used	Ester Created	Scent Observed
I				
E				
M				

Concept Questions:

1. Using the example from the "Background" section on the lab write-up, write a chemical reaction for each ester prepared above. You should have a "**word equation**" with the names of the reactants and products and a "**chemical equation**" with either the chemical formulas or drawn structures.

I:

E:

M:

2. Fill in the alcohol and acid that would be needed to create the esters in the table.

	Ester	Odor	Alcohol	Acid
1	methyl acetate	sweet		
2	isopentyl isovalerate	apple		
3	ethyl acetate	fruity		
4	octyl acetate	orange		
5	ethyl butyrate	peach		
6	isopentyl acetate	banana		
7	methyl butyrate	pineapple		
8	amyl butyrate	apricot		

*****Don't forget to attach your pre-lab write-up!*****