LABORATORY SAFETY AND HAZARD ASSESSMENT FORM

Course: CHE 214L Organic Chemistry Laboratory

Experiment No. 5: Preparation of Synthetic Fruit Flavors

Personnel Involved: undergraduates, laboratory staff, and instructors

Substances Involved*	Quantity Used in Experiment	Hazards and Toxicities	Precautions and First Aid

**identify all chemical reagents / substances that you will use in the experiment.*

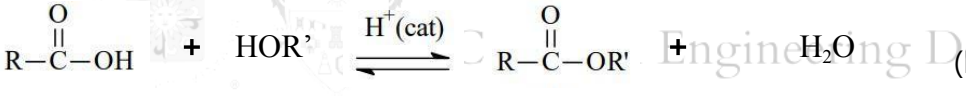
Prepared by: _____ Instructor: _____

Experiment 5

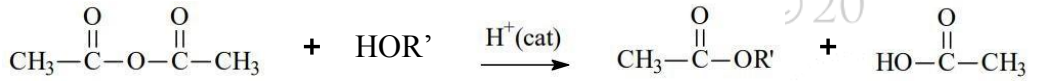
PREPARATION OF SYNTHETIC FRUIT FLAVORS

BACKGROUND

Esters are naturally-occurring compounds that contribute to flavors and aromas of fruits and flowers. They also make up the bulk of animal fats and vegetable oils. An ester can be synthesized via Fischer esterification, which involves the reaction of a carboxylic acid and an alcohol under heat in the presence of a strong acid catalyst as shown in EQ 1 where R' and R" are hydrocarbon chains. Fischer esterification, however, is reversible and only slightly favors the products. In this experiment, an ester will be prepared from acetic acid using acetic anhydride. This modified esterification process (EQ 2) is also acid catalyzed like Fischer esterification, but is very fast and irreversible. The resulting mixture will then be purified by extraction.



carboxylic acid + alcohol ⇌ ester + water side product (EQ 1)



acetic anhydride + alcohol → an acetate ester + acetic acid side product (EQ 2)

Extraction is a separation process applied to mixtures with components that can be chemically converted to ionic form. The components converted to ionic form are dissolved in water (the aqueous layer), while the non-ionic and organic components report into an organic solvent (the organic layer). The two immiscible layers – the aqueous and organic layers – are then made to separate inside a separatory funnel where the lower layer can be manually collected, separate from the upper layer, through the stopcock.

Table 1. Contents of each separated layer during extraction

		Solvent	
		$NaHCO_3$	NaCl
Contents	Organic Layer	CH_3CO-OR (ester) some H_2O	CH_3CO-OR trace H_2O
	Aqueous Layer	$NaHCO_3$ (and $CO_{2(g)}$) R-OH (unreacted alcohol) CH_3COO-Na^+ SO_4^{-2} H_2O	NaCl H_2O

In this experiment, the synthesized ester will be purified by extraction using $NaHCO_3$ twice and NaCl once. In the process, unreacted alcohol, sulfuric acid, acetic anhydride, and most of the water, which all report to the aqueous layer, are separated from the ester in the organic layer as shown in Table 1.

OBJECTIVES

- to synthesize a synthetic fruit flavor (ester) via the modified Fischer esterification reaction,
- to purify ester through the process of extraction, and
- to determine the percentage yield of ester.

MATERIALS AND EQUIPMENT

isoamyl, *n*-propyl, and octyl alcohol
acetic anhydride
concentrated H_2SO_4
saturated $NaHCO_3$ solution
saturated NaCl solution
anhydrous Na_2SO_4
ice cubes*
test tubes

Bunsen burner or heating plate
thermometer
distilling flask
condenser
rubber tubing
wire gauze
iron stand
tripod

dropper
250-ml beaker
spatula
*to be brought by students

grease
boiling chips
separatory funnel

PROCEDURES

Refer to Table 2 for specifications to be followed for the assigned synthetic flavoring.

Table 2. Preparation of synthetic flavorings

Synthetic Flavoring	Alcohol	Volume of Anhydride	Reflux Time
Banana	isoamyl alcohol	2.6 ml	30 mins
Pear	*n*-propanol	4.0 ml	15 mins
Orange	octyl alcohol	2.0 ml	15 mins

1. Measure 2.5 mL of the assigned alcohol in a dry test tube and label it as test tube A.
2. Measure the specified volume of acetic anhydride in a separate dry test tube and label it as test tube B.
3. Put both test tubes A and B in an ice-water bath for 5 minutes.

Concentrated sulfuric acid (H_2SO_4) is corrosive and oxidizing.
Use the fume hood when working with this acid.
Reaction is exothermic. Wear goggles.

4. Carefully add 5 drops of concentrated H_2SO_4 in test tube B. Slowly swirl the test tube to induce proper mixing.
5. While keeping both test tubes in an ice bath, slowly add, drop-wise, the contents of test tube B to test tube A. Swirl the test tube.
6. Using a 250-mL beaker, prepare a water bath at 80°C.
7. Transfer the resulting mixture in a reflux set-up as shown in Figure 1. Connect one end of the condenser to a water source and the other end to a drain. Add

3-5 pieces of boiling chips to the mixture in the flask and heat the flask in a water bath at 80°C for the specified period of time according to the assigned flavoring.

8. Remove the mixture from the hot bath, cool for a minute and pour the contents in a 250 mL small beaker containing a cube of ice.
9. Transfer the mixture in a separatory funnel (as shown in Figure 2) containing 15 mL of saturated $NaHCO_3$ solution. Secure the funnel with the cap.

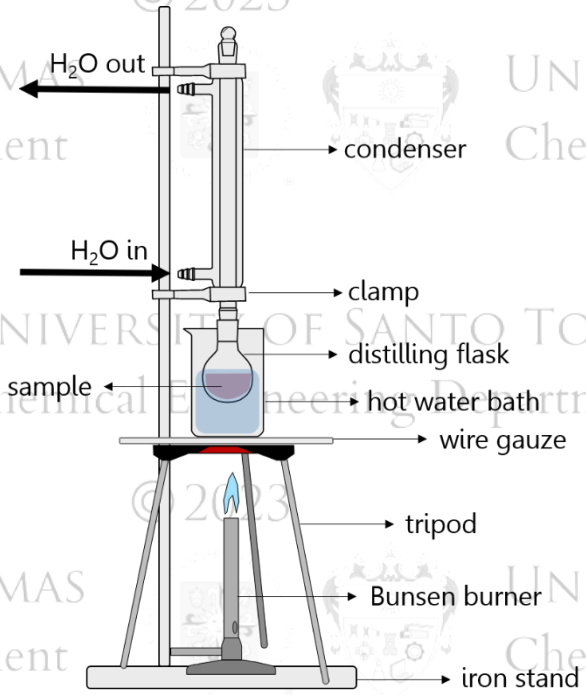


Figure 1. The reflux set-up

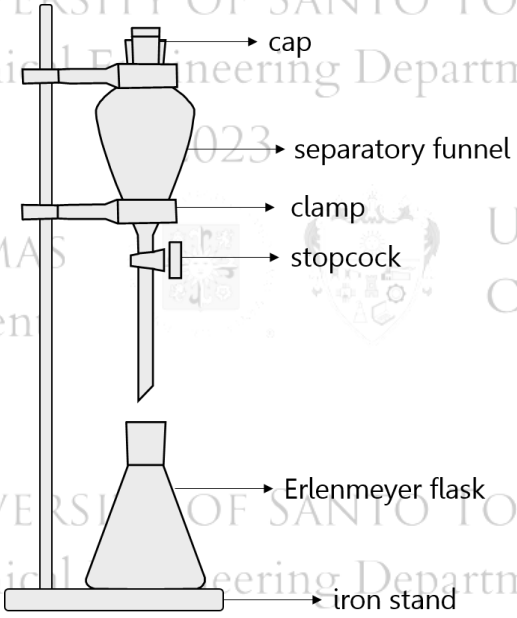


Figure 2. Separatory funnel

10. Swirl the separatory funnel gently to avoid forming emulsions. To decrease gas-pressure build up, invert the separatory funnel, secure the cap with one hand, and release the gas through the stopcock (shown in Figure 3).

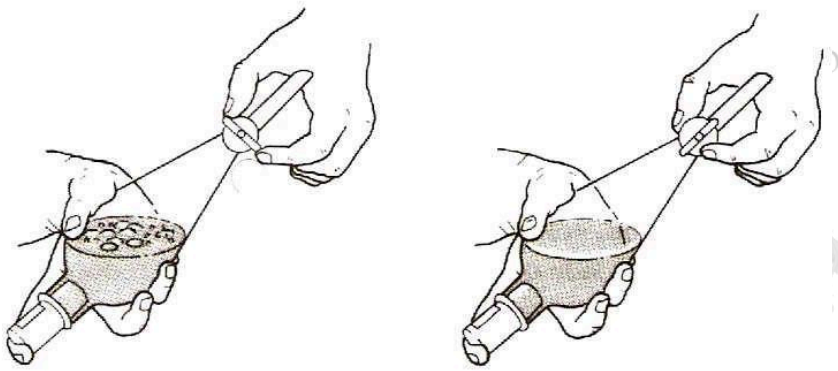


Figure 3. Releasing the pressure building up inside the funnel

11. After mixing, allow the two layers to separate. Remove the cap and discard the (aqueous) lower layer.
12. Repeat the extraction with another 15 mL $NaHCO_3$.
13. Then, extract with 15 mL NaCl solution and discard the (aqueous) lower layer.
14. Put 0.5 grams of Na_2SO_4 inside a dry clean test tube and then transfer the (organic) upper layer into the test tube. Mix the solution gently.
15. Allow the solution to stand for several minutes until the Na_2SO_4 solids settle down. Carefully decant the liquid from the solution and weight the final liquid product.
16. Keep the liquid product sealed and keep it inside your cabinet for use in another experiment.

TREATMENT OF RESULTS

1. Considering EQ 2 and the actual volumes of alcohol and anhydride used in the experiment, determine (a) the limiting reactant, (b) the excess reactant, and the (c) theoretical yield of ester in grams.

2. From the theoretical yield that you have determined and the weight of the ester that you obtained from the experiment, compute for the percentage yield of ester using EQ 3.

$$\frac{actual\ yield}{theoretical\ yield} \times 100\% = \%\ yield \qquad \text{(EQ 3)}$$

GUIDE QUESTIONS

1. What is the purpose of refluxing the mixture?
2. Why was the mixture extracted using saturated NaCl solution instead of distilled water?
3. Why was the product mixed with anhydrous Na_2SO_4 after extraction?
4. What other mixtures can be separated by extraction to manufacture commercially-available products?

REFERENCES

1. Garcia, C. (2014). *Laboratory Experiments in Organic Chemistry*.
2. Paar, L., Elbert, J., & Manfredi, K. (2008). *Organic Chemistry Laboratory Experiments*.
3. Roberts, J., Stewart, R., & Caserio, M. (1971). *Organic chemistry: Methane to macromolecules*. New York, New York: W.A. Benjamin, Inc.
4. UWI Jamaica. (2009). Separation of an Unknown Mixture by Acid/Base Extraction. Retrieved from http://wwwchem.uwimona.edu.jm/lab_manuals/c1901exp8.html
5. UWI Mona. (2009). Separation of mixtures.
6. Winona State University. Synthesis of esters using acetic anhydride. Retrieved from http://course1.winona.edu/tnalli/spring05/209labs/expt5.pdf

Data Sheet

Experiment No. 5

Preparation of Synthetic Fruit Flavors

Name: _____ Analysis Score: _____

Section: _____ Group No.: _____ Date(s) Performed: _____

Check one:

☐ Banana ☐ Pear ☐ Orange

Have each set-up approved by the laboratory instructor before proceeding.

Reflux Set-up ☐ Instructor's Signature

Extraction Set-up ☐ Instructor's Signature

A. Weight of the Product

Weight of empty test tube	☐ g
Weight of test tube with product	☐ g
Weight of product	☐ g

B. Physical Properties of the Product

State (*i.e.* solid, liquid, or gaseous)	
Color	
Odor (Do not sniff directly!)	
Other observations	