

Experiment 26G

SYNTHESIS OF ESTERS

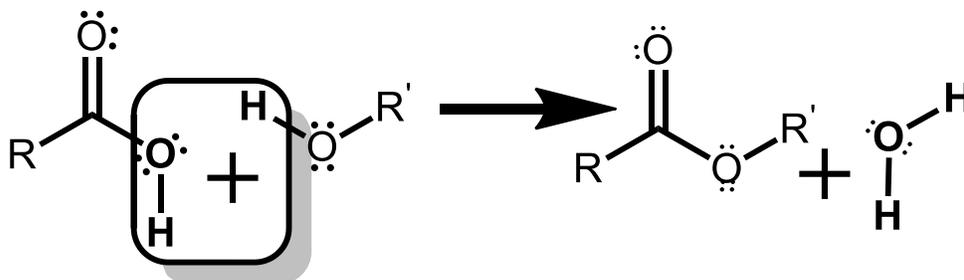
PROBLEM: Synthesize an ester that smells like bananas using the combinatorial chemistry technique.

LEARNING OBJECTIVE: By the end of this experiment, the midshipman should be able to demonstrate the following proficiencies:

1. Synthesize an ester.
2. Use combinatorial chemistry to find a compound with desired properties.

BACKGROUND:

An ester is synthesized by the reaction of a carboxylic acid with an alcohol via a condensation reaction.



where R, R' represent a hydrocarbon group. In this lab, R can be either, $-\text{CH}_3$, $-\text{C}_6\text{H}_5$, $-\text{C}_6\text{H}_5\text{O}$ or $-\text{C}_6\text{H}_6\text{N}$, and R' can be either, $-\text{CH}_3$, $-\text{CH}_2\text{CH}_3$, $-\text{CH}_2\text{CH}_2\text{CH}_3$ or $-\text{CH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2$.

Many pleasant smells in nature are esters. Different fruits, for example, will have a characteristic smell of a particular ester that plant makes. Rather than randomly choosing a particular carboxylic acid to react with a particular alcohol, and hoping for the one which smells like bananas, we will use a combinatorial chemistry approach. This is a technique for producing large numbers of diverse, but related compounds. Thus, we make a small amount of many related compounds and then tested at the same time. For example, if all combinations of four different carboxylic acids and four different alcohols are used, sixteen esters may be synthesized and checked for the sought after banana aroma.

MATERIALS: 400 mL beaker, 50 mL beaker, test tube rack, hot plate, thermometer, glass stirring rod, small weighing dishes, spatulas, 13 x 100 mm test tubes, disposable plastic pipettes, 18 M H₂SO₄, glacial acetic acid, benzoic acid, salicylic acid, anthranilic acid, methanol, ethanol, 1-propanol, 3-methyl-1-butanol, waste container.

PROCEDURE:

Part A. Preparation and Smelling of One Assigned Ester

Caution: Glacial acetic acid and sulfuric acid are corrosive, and should be handled carefully!

1. Assemble a hot water bath by half filling a large beaker with tap water and heating it on a hot plate. Maintain a temperature of 80–85 °C
2. In a test tube measure and add either 0.2 g (if solid) or 10 drops (if liquid) of the assigned carboxylic acid, 20 drops of the assigned alcohol, and 5 drops of sulfuric acid, unless anthranilic acid is assigned. Note: If you are assigned anthranilic acid, use 60 drops of alcohol and 15 drops of sulfuric acid. Add the sulfuric acid very slowly because the reaction is exothermic.
3. Swirl the mixture well. Place in hot water bath for 30 minutes. If one of the reactants is a solid, continue to mix with a stirring rod until the solution is homogeneous (no solid remains).
4. Turn off the hot plate. Remove the test tube from the hot water bath, and pour the ester into a small beaker containing about 2 mL of distilled water.
5. Cautiously smell the ester.

Part B. Smelling of All Prepared Esters

1. After all sixteen esters have been synthesized by the class, cautiously smell and record observations for each ester.
2. List the reactant pairs that make the ester that smells like bananas.

Clean up:

1. Check that the hot plate is turned off.
2. Dispose of your ester in the waste beaker.
3. Dispose of your test tube in the container for broken glass.

Name _____

Section _____

Date _____

DATA SECTION
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Part A. Aromas of Esters Synthesized From Reactants

Alcohol / Carboxylic Acid	benzoic acid	salicylic acid	acetic acid	anthranilic acid
	methanol			
ethanol				
1-propanol				
3-methyl-1-butanol				

Part B. List of Reactant Pairs That Make the Ester That Smells Like Bananas

Carboxylic Acid and Alcohol: _____ and _____

Name _____

Section _____

Date _____

PRE-LAB EXERCISES
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1. Draw the condensed structural formula for the carboxylic acids used in this experiment.

benzoic acid	salicylic acid	acetic acid	anthranilic acid
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2. Draw the condensed structural formula for the alcohols used in this experiment.

methanol	ethanol	1-propanol	3-methyl-1-butanol
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3. Using condensed structural formulas, write a balanced chemical equation for the reaction of benzoic acid and ethanol.
4. What is the procedure for safely detecting the odor of chemicals?