Experiment 6

Synthesis of Wintergreen Oil

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

Methyl salicylate, commonly known as wintergreen oil, will be synthesized from salicylic acid and methanol, isolated, and purified to calculate a percent yield.

Background

Synthetic chemistry involves the practice in lab of performing a chemical reaction and then isolating, purifying, and characterizing the products. Stoichiometry allows the calculation of the amount of reactants needed and the expected amount of product made. Isolation and purification of products typically involves separation techniques which utilize physical and chemical changes. Characterization can be any of a number of tests whose results can be used to identify the product, including measuring the substance's melting or boiling point, calculating density or molar mass, spectroscopic methods, or other qualitative tests. Synthetic chemistry is widely and frequently practiced due to its ability to transform reactants into a variety of products.

For a reaction, the reactant that will produce the least amount of product is called the limiting reactant. Any reactants that make more product than the limiting are known as reactants in excess. The amount of product calculated to be made by the limiting reactant is known as the theoretical yield. In the laboratory setting, it can be very difficult to collect all of the product made, as some can be lost due to transfer between glassware or spills. As a result, the amount of product physically collected when the reaction is performed is known as the actual yield and is usually lower than the theoretical yield. An actual yield that equals the theoretical yield indicates that all of the product was successfully collected. An actual yield greater than the theoretical yield indicates that the product may contain extra impurities. For any synthetic experiment, a percent yield should always be reported as a way to expression how much of the calculated product was successfully collected. The formula for calculating percent yield is shown in Eqn. 1.

$$\text{percent yield} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100 \qquad \text{Eqn. 1}$$

Salicylic acid ($C_7H_6O_3$) is a precursor to the commercial drug aspirin, known scientifically as acetylsalicylic acid. It is commonly used for pain relief. When reacted with methanol (CH_3OH), salicylic acid produces wintergreen oil, a naturally-occurring extract that is commonly used in cosmetics for its pleasant aroma as well as an analgesic. The balanced reaction between salicylic acid and methanol is shown in Eqn. 2.

$$C_7H_6O_3(l) + CH_3OH(l) \rightarrow C_8H_8O_3(s) + H_2O(l) \qquad \text{Eqn. 2}$$

The reaction also requires an acid catalyst to be present. Catalysts are compounds that speed up the reaction but are not consumed by it, meaning that they are neither reactants nor products. They are present both at the beginning and end of the reaction. Because of this, catalysts are never limiting reactants and their exact amount does not matter.

Example Exercise: Determining Limiting Reactant and Percent Yield

A 0.1150 g sample of salicylic acid is reacted with 5.0 mL of methanol and 3 drops of concentrated sulfuric acid to produce methyl salicylate. After the reaction, 0.0890g of product was collected. Find the limiting reactant, theoretical yield of product, and percent yield for the reaction. Methanol has a density of 0.792 g/mL.

Step 1: Calculate the amount of product made by each reactant.

$$0.1150 \text{ g } C_7H_6O_3 \times \frac{1 \text{ mol } C_7H_6O_3}{138.12 \text{ g } C_7H_6O_3} \times \frac{1 \text{ mol } C_8H_8O_3}{1 \text{ mol } C_7H_6O_3} \times \frac{152.15 \text{ g } C_8H_8O_3}{1 \text{ mol } C_8H_8O_3}$$

$$= 0.1267 \text{ g } C_8H_8O_3$$

$$5.0 \text{ mL } CH_3OH \times \frac{0.792 \text{ g } CH_3OH}{1 \text{ mL}} \times \frac{1 \text{ mol } CH_3OH}{32.04 \text{ g } CH_3OH} \times \frac{1 \text{ mol } C_8H_8O_3}{1 \text{ mol } CH_3OH} \times \frac{152.15 \text{ g } C_8H_8O_3}{1 \text{ mol } C_8H_8O_3} = 19 \text{ g } C_8H_8O_3$$

Step 2: Find the limiting reactant as the one that makes the least amount of product

$C_7H_6O_3$ is the limiting reactant with a theoretical yield of 0.1267 g $C_8H_8O_3$

Step 3: Use Eqn. 1 to find the percent yield

$$\frac{0.0890 \text{ g } C_8H_8O_3}{0.1267 \text{ g } C_8H_8O_3} \times 100 = 70.2 \text{ \% yield}$$

Procedure

Part I: Synthesis of Wintergreen Oil

1. Fill a 400 mL beaker approximately halfway with tap water. Place it on a hot plate.

2. Collect a glass thermometer and split stopper. Slide the thermometer through the hole. Clamp the thermometer in the water approximately 1 cm from the bottom, but not touching the bottom or the sides.

3. Heat the water bath in Step 1 to approximately 60°C.

4. Weigh a clean, dry large test tube. Record this weight in your data sheet.

5. Add approximately 0.25 g of salicylic acid to the test tube in Step 4. Record the exact weight in your data sheet.

6. Add approximately 4.0 mL of methanol and 3 drops of concentrated $H_2SO_4(aq)$ to the test tube in Step 5. Record the exact amounts in your data sheet.

H_2SO_4 is extremely corrosive!

7. Note the odor of the starting reactant mixture. Remember to waft the air above the container toward your nose; never directly sniff or smell a chemical!

8. Place the test tube in Step 6 in the hot water bath. Heat for 30 minutes, keeping the bath's temperature between 59-64°C.

9. Remove the test tube from the water bath and allow to cool to room temperature.

10. To the cooled test tube, add approximately 4.0 mL of methylene chloride (dichloromethane) and 4.0 mL deionized water.

11. Mix the two liquids with a clean glass stirring rod, but do **not** spill or leak any solution out of the test tube. Remove the glass rod and allow the solution to settle.

12. Two immiscible liquid layers, one of methylene chloride and product, the other of deionized water and excess reactants, should be visible. The denser liquid between methylene chloride and deionized water will be on the bottom.

Note: you will need to research the densities of methylene chloride and water.

13. Using your transfer pipet (dropper), very carefully remove the top layer from the test tube without disturbing the lower layer. Put the top layer into a beaker.

14. To the remaining layer in the test tube, add approximately 3.0 mL of 10% sodium bicarbonate ($NaHCO_3(aq)$) solution. Mix the two liquids with a clean glass stirring rod. Remove the rod and allow the liquids to settle.

15. Two immiscible liquid layers, one of methylene chloride and product, the other of sodium bicarbonate, water, and other impurities, should be visible. The denser liquid is on the bottom.

16. Using your transfer pipet, very carefully remove the top layer from the test tube without disturbing the lower layer. Add this top layer to the same beaker as in Step 13.

17. Repeat Steps 14-16 two more times, for a total of three additions of $NaHCO_3$(aq) solution.

18. To the remaining layer in the test tube, add approximately 3.0 mL of deionized water. Mix the two liquids with a clean glass stirring rod. Remove the rod and allow the liquids to settle.

19. Two immiscible liquid layers, one of methylene chloride and product, the other of water and other impurities, should be visible. The denser liquid is on the bottom.

20. Using your transfer pipet, very carefully remove the top layer from the test tube without disturbing the lower layer. Add this top layer to the same beaker as in Step 13.

21. Dispose of the aqueous layers collected in the beaker in the appropriate waste container.

22. Add a boiling chip to the remaining liquid in the test tube.

23. In a fume hood, fill a 250 mL beaker approximately half full with water and place it on a hot plate. Put the test tube carefully into the water bath, avoiding spilling or tipping, and gently heat until the liquid inside the test tube begins to boil.

24. Continue to heat the solution until the liquid inside the test tube ceases boiling. There should only be an oily residue remaining. This is your product. Note the odor of your product by wafting.

25. At your counter, let the test tube and product cool to room temperature. Remove the boiling chip, being careful to not lose your product. Weigh accurately. Record the weight on your data sheet.

26. Determine the theoretical yield of product, limiting reactant, and percent yield for your reaction.

Experiment 6—Data Sheet

Name: ______________________________________

1. Mass test tube (g) ______________

2. Mass test tube and salicylic acid (g) ______________

3. Volume methanol (mL) ______________

4. Mass test tube and product (g) ______________

5. Mass salicylic acid (g) ______________
show calculation:

6. Limiting Reactant ______________
show calculation:

7. Theoretical Yield of Product (g) ______________

8. Actual Yield of Product (g) ______________
show calculation:

9. Percent Yield of Product (%) ______________
show calculation:

10. Odor of Product:

Experiment 6—Post-Lab Assignment

1. Give three experimental reasons why your percent yield may or may not be 100%.

2. Solid iron rusts in the presence of oxygen gas to produce iron(III) oxide.

   a. Write the balanced chemical equation for this reaction.

   b. A 5.15 g sample of iron reacts with 3.65 g oxygen gas. Find the limiting reactant and theoretical yield of iron(III) oxide produced.

   Limiting Reactant ________________ Theoretical Yield ________________

   c. A student performed the reaction described above and collected 4.5675 g of iron(III) oxide product. Find the percent yield for this reaction.

3. Most of gasoline is octane, C_8H_{18}, which combusts in a car engine to produce energy.

a. Write the balanced chemical reaction for the combustion of octane.

b. A student uses 10.0 gallons of gas to commute every week. Calculate how many grams of gasoline the student uses per year. (1 year = 52 weeks, 1 gallon = 3.785 L, density of octane = 0.703 g/mL)

c. Calculate the grams of CO_2 produced by the student's commute each year.

d. List three assumptions made in your above calculations that would make your answer inaccurate.

Experiment 6—Pre-Lab Assignment

Name: ____

For all calculations, show all work and draw a box around the final answers.

1. What is the purpose of adding sodium bicarbonate ($NaHCO_3$) *after* the reaction?

2. A student performed the synthesis of wintergreen oil and recorded the following data.

Mass salicylic acid (g)	0.450 g
Volume methanol (mL)	8.0 mL
Volume 9.0 M H_2SO_4 (drops)	3 drops
Mass Test Tube (g)	28.6555 g
Mass Test Tube and product (g)	29.0561 g

Find the limiting reactant, theoretical yield of methyl salicylate, and percent yield for the reaction.