1. From Oil of Wintergreen to Salicylic Acid

Introduction

What happens during a chemical reaction? Reactant molecules are converted to product molecules as chemical bonds are formed or broken. Because the properties of a molecule depend in part on the bonds within the molecule, the properties of the reactants differ from those of the products. These differences are often large.

This experiment deals with a chemical reaction in which there is a significant and easily noticed difference between the properties of a reactant and those of a product: The reactant is a liquid and the product is a solid.

Some of the substances in this experiment are called organic compounds. *Organic chemistry* deals with compounds in which carbon is the principal element.

Purpose

You will examine the chemical reaction in which methyl salicylate (oil of wintergreen), a liquid, is converted to salicylic acid, a solid. The actual yield of this product will be compared with the theoretical yield.

Some interesting facts

Methyl salicylate, well known for its characteristic wintergreen odor, is used as a flavoring and as an ingredient in various liniments. Salicylic acid, which is used in the preparation of aspirin, shares aspirin's ability to relieve pain.

Concept of the experiment

When methyl salicylate ($HOC_6H_4COOCH_3$) is heated in water, salicylic acid (HOC_6H_4COOH) and methyl alcohol (CH_3OH) are formed slowly. This reaction is shown in Figure 1.1. You can obtain a faster reaction if you replace the water with an aqueous solution of sodium hydroxide. When the reaction is complete, the sodium hydroxide can be removed chemically by the addition of sulfuric acid. Although the conversion of methyl salicylate to salicylic acid becomes slightly more complicated under these conditions, the overall results are virtually identical to those shown in Figure 1.1.

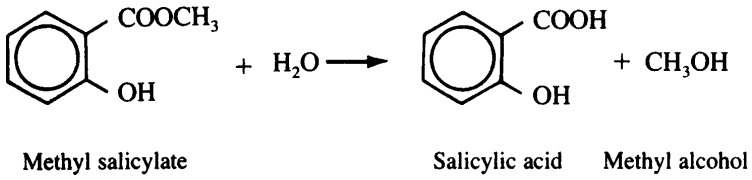

FIGURE 1.1
The reaction of methyl salicylate (oil of wintergreen) with water to give salicylic acid and methyl alcohol.

Salicylic acid is not very soluble in an aqueous solution of sulfuric acid. As a result, salicylic acid precipitates when you add sulfuric acid. You will use suction filtration to separate the precipitated solid from the solution. This filtration technique is described in the Introduction to this manual.

The product that you have recovered may be crude and impure. You will use a technique called *recrystallization* to remove possible impurities. In this technique, a crude solid product from a reaction is dissolved in a liquid, and crystallization is induced. In the simplest use of the technique, purification will occur if the impurities are more soluble in the liquid than the desired compound is. The impurities will remain in solution after the purified product has crystallized.

After recrystallization, the crystals must be dried. There are two possible techniques. The one you use will depend on the time available.

You will also have an opportunity to calculate the theoretical yield and the percentage yield of salicylic acid (Ebbing/Gammon, Section 3.8) on the basis of the mass of the limiting reactant. You will probably obtain a percentage yield greater than 80%.

Procedure

Getting started

1. Make sure that you have read and understood the description of suction filtration in the Introduction to this manual.
2. Observe the following safety precaution:

   CAUTION: The solutions of sodium hydroxide and sulfuric acid that you will encounter must be handled carefully. These solutions can cause chemical burns in addition to ruining your clothing. If you spill one of these solutions on you, wash the contaminated area thoroughly and report the incident to your laboratory instructor. You may require further treatment.

3. Add 60 mL of distilled water to a 250-mL beaker from a graduated cylinder. Mark the location of the upper surface of the water on the outer wall of the beaker, using a marking pencil. You will use this beaker for the reaction. First, however, discard the water and dry the beaker.
4. Ask your laboratory instructor which drying technique you should use for the crystals of salicylic acid.

Doing the reaction

1. Obtain 4.0 mL of methyl salicylate in a clean, dry 10-mL graduated cylinder. Measure and record the mass, using a platform balance, triple beam balance, or an electronic top-loading balance. Your laboratory instructor will provide you with instructions for using these balances.
2. Add as much of the methyl salicylate from Step 1 as you can to the dry, marked 250-mL beaker.

3. Measure and record the mass of the graduated cylinder. Calculate the mass of the salicylate that you poured into the beaker.
4. Add 40 mL of 6 *M* NaOH from a clean graduated cylinder. A white precipitate will form immediately. This precipitate is not the desired product. The ionic compound in this precipitate is $NaOC_6H_4COOCH_3$.
5. Stir the mixture thoroughly with a glass stirring rod. Do not remove the stirring rod.
6. Set up a ring stand with an iron ring, and place a piece of wire gauze on the ring. Adjust the height of the ring so that the wire gauze will be in the hottest part of the flame from a laboratory burner. Do not light the burner until you have made this adjustment.

   CAUTION: Avoid burning your fingers. Do not touch the iron ring or the wire gauze at any time while the contents of the beaker are being heated.

7. Place the beaker on the wire gauze and heat the mixture to a *gentle* boil. Stir the solution occasionally. The precipitate will dissolve.
8. Continue to boil the solution for 15 min after the precipitate has dissolved. Use a stream of distilled water from a plastic wash bottle to rinse any solids from the inner walls of the beaker into the solution. However, do not let the total volume exceed 60 mL, as indicated by your mark.
9. Take this opportunity to assemble the glassware for suction filtration.
10. After the period of heating is completed, remove the flame and cool the beaker in ice until it is only warm when you touch it.
11. Add 50 mL of distilled water to the beaker, and then *cautiously* add 50 mL of 8 *M* H_2SO_4 with stirring. A white precipitate of the crude product should form during the addition.
12. Cool the beaker again in ice until it is cold. At the same time, cool 50 mL of distilled water in an Erlenmeyer flask. You will use the cold water to wash the precipitate.
13. Filter the cold mixture by suction filtration, and wash the crude product with the cold water.
14. The suction should be continued for several minutes to partially dry the precipitate.
15. Wash and dry the 250-mL beaker during this time.

Recrystallizing the product

1. Tilt the Büchner funnel over the 250-mL beaker. Use a metal spatula to carefully separate the precipitate from the filter paper and to transfer the precipitate to the beaker.
2. Add 100 mL of distilled water to the beaker from a graduated cylinder.
3. Place the beaker on the wire gauze. Heat the mixture until a gentle boil occurs. Stir with a stirring rod. Several minutes of boiling are usually required before all of the precipitate dissolves.
4. Remove the flame and allow the solution to cool for 5 min without disturbing it. You should see crystals of salicylic acid begin to appear. Record a description of their appearance.

5. Cool the beaker in ice until it is cold. At the same time, cool 50 mL of distilled water in an Erlenmeyer flask.
6. Filter the cold mixture by suction filtration. Wash the crystals with the cold water.
7. Continue the suction for 15 min.
8. Wash and dry the 250-mL beaker during this time.

Drying the crystals

1. Measure and record the mass of the dry 250-mL beaker, using a platform balance, triple beam balance, or an electronic top-loading balance.
2. Transfer the crystals to the beaker, using the techniques that you used earlier.
3. If you have only a limited amount of time, follow Steps 4 through 7 and Steps 9 and 10. However, if it is possible to wait until the next laboratory period, follow Steps 8 through 10.
4. Cover the beaker with a clean watch glass. Place the beaker on the wire gauze.
5. Follow the directions in this step carefully. Brush the flame back and forth across the underside of the wire gauze for about 30 s. Do not allow the flame to linger in any one place. You should see a very thin layer of water condense on the watch glass.
6. Allow the beaker to cool briefly, and remove the watch glass. Measure and record the mass of the beaker and its contents.
7. Replace the beaker and watch glass on the wire gauze. Repeat Steps 4, 5, and 6. Repeat until the mass does not change significantly; but, in no circumstance, should the change in mass exceed 0.2 g.
8. Write your name on the beaker with a marking pencil. Cover it with a clean watch glass and set it aside so that the crystals can dry in the air.
9. When the crystals are dry, remove the watch glass. Measure and record the mass of the beaker and its contents.

CAUTION: Do not ingest your sample under any circumstances.

10. Show your sample to your laboratory instructor. Obtain instructions for its disposal.

CAUTION: Before you leave the laboratory, make sure that your gas outlet and those of your neighbors are closed.

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Date:	.....	Student name:	.....
Course:	.....	Team members:	.....
Section:	.....		.....
Instructor:	.....		.....

Prelaboratory assignment

1. a. Draw sketches of methyl salicylate and salicylic acid, showing every bond.

2. What is the limiting reactant in this experiment? Why? The density of methyl salicylate is 1.18 g/mL at 20°C.

3. What safety precautions must be observed during this experiment?

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Section: .....

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Results

Mass of methyl salicylate and graduated cylinder (g): .....

Mass of graduated cylinder (g): .....

Mass of methyl salicylate (g): .....

Description of crystals of salicylic acid:

Mass of salicylic acid and beaker (g): .....

Mass of beaker (g): .....

Mass of salicylic acid (g): .....

Questions

1. a. What is the actual yield of salicylic acid?

   b. Calculate the theoretical yield. Remember that you deduced the limiting reactant in the Prelaboratory Assignment.

c. Calculate the percentage yield.

2. What covalent bonds in methyl salicylate and water were broken during the reaction? What bonds were formed?