Steam Distillation of Cinnamon

Introduction

Essential oils have been used since ancient times as perfumes, flavorings and even medicines. So-called because they seem to contain the odor and flavor "essence" of a plant, essential oils are volatile organic liquids obtained from flowers, leaves, twigs, fruits, nuts or seeds.

Concepts

- Essential oil
- Solvent extraction
- Steam distillation
- Aldehyde functional group

Background

There are three main methods for obtaining essential oils from plants: steam distillation, extraction with a solvent, and expression or cold pressing. *Steam distillation* involves heating plant components with water or steam and collecting the liquid distillate. The latter consists of an immiscible mixture of water and the essential oil.

When a mixture of two immiscible liquids, such as water and oil, is heated, *each liquid exerts its vapor pressure independently of the other liquid*. The total vapor pressure (P_{total}) at a given temperature is therefore equal to the *sum* of the vapor pressures of the two immiscible liquids. Equation 1 describes the total vapor pressure for a mixture of an essential oil and water.

$$P_{total} = P_{H_2O} + P_{Oil} \qquad \textit{Equation 1}$$

A mixture of two immiscible liquids will boil when their combined vapor pressure is equal to 760 mm Hg. The boiling point of a mixture of water and essential oil will thus be *less than* the boiling point of either component separately. This is an advantage in the distillation of natural products that have high boiling points and would decompose at high temperatures.

The amounts of water and essential oil that will co-distill during steam distillation can be determined using Equation 1 and the ideal gas law. The *mole ratio* of oil and water in the distillate is equal to their relative vapor pressures (P) at the boiling point (Equation 2, n = number of moles).

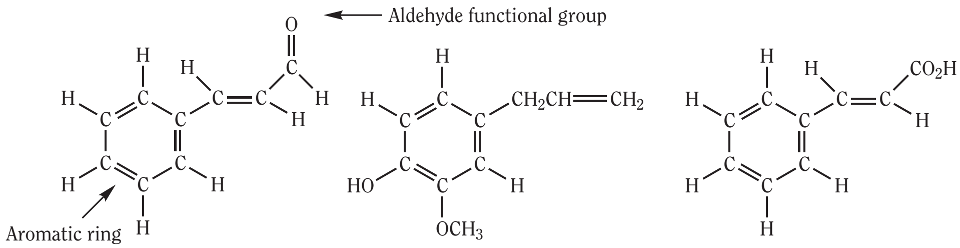
$$\frac{n(\text{oil})}{n(\text{H}_2\text{O})} = \frac{P_{oil}}{P_{H_2O}} \qquad \textit{Equation 2}$$

Multiplying each term in Equation 2 by the molar mass (*MM*, g/mole) of the substance gives the mass of water in grams that will co-distill with an essential oil (Equation 3).

$$\frac{n(\text{oil}) \times MM(\text{oil})}{n(\text{H}_2\text{O}) \times MM\ (\text{H}_2\text{O})} = \frac{\text{mass of oil (g)}}{\text{mass of water (g)}} = \frac{P_{oil} \times MM\ (\text{oil})}{P_{H_2O} \times MM\ (\text{water})} \qquad \textit{Equation 3}$$

Cinnamon is obtained from the inner bark of *Cinnamomum zeylanicum*, a small evergreen that is native to Sri Lanka and India. Oil of cinnamon is obtained from cinnamon bark by steam distillation. The major componant of oil of cinnamon is cinnamaldehyde (70–80%). *Cinnamaldehyde* is classified as an aromatic aldehyde—it contains an *aldehyde* functional group and has a benzene (aromatic) ring (see Figure 1). The compound has natural antimicrobial properties but is highly irritating to the skin. Minor components of oil of cinnamon include eugenol (10–20%) and cinnamic acid (5–10%).

Publication No. 10962
061616



Cinnamaldehyde *Eugenol* *Cinnamic Acid*

Figure 1. Components of Oil of Cinnamon.

The purpose of this activity is to isolate oil of cinnamon by steam distillation and study its chemical properties. The mixture of oil and water in the distillate will be extracted with hexane to dissolve the oil and the solvent will then be evaporated. The presence of cinnamaldehyde can be identified using a qualitative test.

Materials *(for each student group)*

Cinnamaldehyde, 0.5 mL
Cinnamon sticks, fresh, 10 g
Hexane, 15 mL
Schiff reagent, 2 mL
Sodium chloride, 10 g
Water
Custom distillation head (condenser and adapters)
Hot plate
Thermometer and thermometer adapter
Beral pipets, 3
Beaker, 400-mL
Clamps, 2
Erlenmeyer flasks, 50-mL, 2
Erlenmeyer flask, 125-mL
Heating mantle and variable transformer
Round-bottom flask, 250-mL
Round-bottom flask, 100-mL
Rubber tubing, 2 pieces
Separatory funnel, 125-mL
Stirring rod
Stopcock grease (optional)
Support (ring) stands, 2
Test tube, large
Test tubes, small, 2
Weighing dish, large

Safety Precautions

Oil of cinnamon and cinnamaldehyde are severe skin irritants. Hexane is a flammable liquid and a dangerous fire risk—do not use around flames. Avoid contact of all chemicals with eyes and skin. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Safety Data Sheets for additional safety, handling, and disposal information. Wash hands thoroughly with soap and water before leaving the laboratory.

Procedure

1. Obtain about 10 g of fresh cinnamon sticks in a large weighing dish and break the sticks into small pieces.
2. Add the broken cinnamon pieces to a 250-mL round-bottom flask and fill the flask about one-half full with water. This is the distilling flask.
3. Place the distilling flask into a heating mantle and clamp the flask to a support (ring) stand.
4. Place the one-piece custom distillation head (or a three-way adapter, followed by a condenser and a second adapter) into the distilling flask (see Figure 2).
5. Place the thermometer holder into the adapter in the distillation head. Carefully insert the thermometer so that the thermometer bulb rests just below the side arm that leads to the condenser.
6. Place a 100-mL round-bottom flask into the side-arm adapter attached to the distillation head. Gently clamp the receiving flask to a support stand. Cool the flask in an ice bath.

7. Attach one piece of rubber tubing from the inlet nozzle on the condenser to the faucet, and place the second piece of rubber tubing from the outlet nozzle on the condenser into the sink to drain.
8. Turn on the faucet to allow water to flow through the condenser. Make sure the rubber tubing connections are tight and check for leaks.
9. Plug the heating mantle into the variable transformer and turn on the power to begin heating the flask.
10. When the water in the flask boils, the vapor will rise in the distillation apparatus and condense in the condenser. The liquid will collect in the receiving flask.
11. Continue distilling the mixture for 45 minutes, until about 30 mL of distillate has been collected. Record the boiling point of the distillate and its appearance in the data table.
12. Turn off the heating mantle. Allow the apparatus to cool and then remove the receiving flask.
13. Pour the distillate into a large test tube and add solid sodium chloride until the solution is saturated. Pour the liquid (*not* the solid) into a separatory funnel.
14. Add about 7 mL of hexane to the separatory funnel and shake. Run off the lower aqueous layer back into the large test tube, and collect the upper hexane layer in a 125-mL Erlenmeyer flask.

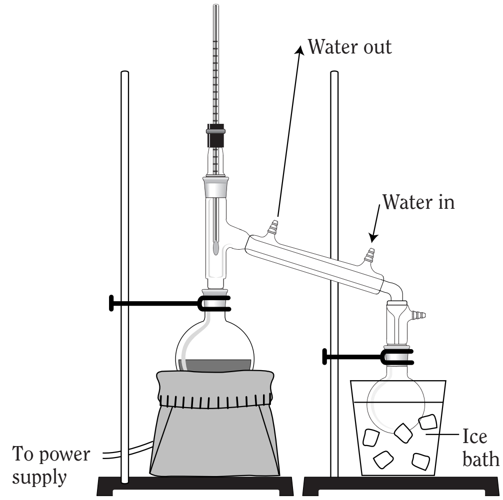


Figure 2. Steam Distillation Apparatus.

15. Return the aqueous layer back into the separatory funnel, and repeat step 14. Combine the hexane extracts in the same Erlenmeyer flask.
16. Working in the hood, place a stirring rod in the hexane solution and heat the solution on a hot plate at a medium setting. Allow all of the hexane to evaporate.
17. *(Optional)* Allow the hexane to evaporate at room temperature overnight.
18. Cool the flask to room temperature. Observe the appearance of oil of cinnamon and carefully waft the vapor to your nose to detect the odor. In the data table, record the appearance and odor of the essential oil.
19. Obtain two small test tubes and place about 1 mL of Schiff reagent into each test tube.
20. Add one drop of pure cinnamaldehyde to the first test tube and one drop of the essential oil to the second test tube. Record observations.

Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines and specific procedures, and review all federal, state and local regulations that may apply, before proceeding. The reaction product may be disposed of down the drain with cold running water according to Flinn Suggested Disposal Method #26b.

Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
Evidence, models, and explanation
Content Standards: Grades 9–12
Content Standard B: Physical Science, structure and properties of matter
Content Standard G: History and Nature of Science, nature of scientific knowledge, historical perspectives

Tips

- This lab is best suited for an advanced or honors chemistry class or as a demonstration. There are two good stopping points if the lab cannot be finished in the scheduled class time. After extracting the oil from the distillate, save the hexane extract before evaporating the solvent. Testing the oil of cinnamon will only take a few minutes. This step can easily be completed on a second day.
- Demonstrate the proper use of a separatory funnel. Always invert the stoppered separatory funnel and vent the vapor *before* shaking the funnel, and then again after every shake. Failure to vent the funnel may result in pressure building

up and solvent spraying out of the separatory funnel when the stopper is removed.

- For best results, always use fresh cinnamon sticks for steam distillation. Cinnamaldehyde and other components of cinnamon oil are volatile liquids and may evaporate from opened packages of cinnamon.

- Schiff's reagent is used as a qualitative test for aldehydes. It consists of an indicator dye, fuchsin hydrochloride, in a satu rated solution of sulfur dioxide. Sulfur dioxide decolorizes the dye. When an aldehyde is added to Schiff's reagent, it reacts with the SO_2 and thus restores the deep reddish purple color of the dye.

- Essential oils are considered secondary metabolites. Many leaf oils and root oils are natural pesticides, protecting plants against insects and parasites. Essential oils from flowers may help plants attract insects for pollination. Many essential oils also have natural antifungal and antibacterial ability.

Sample Data Table *(Student data will vary.)*

Boiling Point and Appearance of Distillate	*Boiling Point = 99–100 °C. The distillate looked oily and separated into two layers in the receiving flask.*
Oil of Cinnamon—Appearance and Odor	*The oil was a pale yellow liquid with a strong cinnamon odor.*
Results of Schiff Test for Aldehydes	
Cinnamaldehyde	*The sample gave a deep magenta (purple) solution after two minutes.*
Oil of Cinnamon	*Same as above—deep purple product.*

Answers to Questions *(Student answers will vary.)*

1. *The distillate looked like oily water. It was two layers. When the liquid in the condenser collected in the receiving flask, the oil seemed to be the bottom layer. Oil of cinnamon is thus more dense than water.* ***Note to teachers:*** *Most essential oils are less dense than water. Oil of cinnamon is an exception.*
2. *Cinnamon oil has a stronger smell than cinnamon sticks. That is because there may be many more components of the odor of fresh natural cinnamon in addition to the essential oil. The other components modify the odor.*
3. *Cinnamaldehyde is a known aldehyde. It served as a positive control for the test with Schiff's reagent. A positive test was marked by the formation of a dark magenta (reddish purple) solution. Oil of cinnamon gave a positive test.*
4. *Vapor pressure of water = 760 – 5 mm Hg = 755 mm Hg.*

Cinnamaldehyde, C_9H_8O*, molar mass 131 g/mole*

$$\frac{\text{mass of cinnamaldehyde (g)}}{\text{mass of water (g)}} = \frac{P_{oil} \times MM\text{ (oil)}}{P_{H_2O} \times MM\text{ (water)}}$$

$$\frac{x\text{ grams cinnamaldehyde}}{30\text{ g water}} = \frac{(5\text{ mm Hg})(131\text{ g/mole})}{(755\text{ mm Hg})(18\text{ g/mole})}$$

$x = 1.5$ *g cinnamaldehyde per 30 g of water*

5. *The combined vapor pressure of a mixture of two immiscible liquids is equal to the sum of their individual vapor pressures. Because a liquid will boil when the total vapor pressure is equal to atmospheric pressure, the boiling point of the mixture will be less than the boiling point of either component.*
6. *Essential oils are responsible for the strong odors or fragrance of plants. Odors help plants attract insects for pollination. Also, some odors may actually repel insects that may be harmful.* ***Note to teachers:*** *A good example is citronellol, which is the essential oil from lemongrass. Citronella candles are used to keep mosquitoes away.*

Materials for *Steam Distillation of Cinnamon* are available from Flinn Scientific, Inc.

Catalog No.	Description
C0400	Cinnamaldehyde, 100-mL
H0002	Hexanes, Reagent, 500 mL
S0180	Schiff Reagent, 100 mL
S0134	Sodium Chloride, 500 g

Consult your *Flinn Scientific Catalog/Reference Manual* for current prices.

Steam Distillation of Cinnamon Worksheet

Data Table

Boiling Point and Appearance of Distillate	
Oil of Cinnamon—Appearance and Odor	
Results of Schiff Test for Aldehydes	
Cinnamaldehyde	
Oil of Cinnamon	

Questions *(Use a separate sheet of paper to answer the following questions.)*

1. Describe the appearance of the distillate. Is cinnamaldehyde (oil of cinnamon) less dense or more dense than water?

2. Compare the odor of cinnamon oil versus cinnamon sticks. What factors may account for the difference in smell?

3. Schiff's reagent is used as a qualitative test for aldehydes. What compound was used as a positive control or reference compound for this test? Describe the appearance of a positive test result. Did oil of cinnamon give a positive test?

4. The vapor pressure of cinnamaldehyde at 99–100°C is about 5 mm Hg. Assuming that the barometric pressure was 760 mm Hg, use Equations 1 and 3 in the *Background* section to calculate the mass of cinnamon oil that would co-distill with 30 g of water during steam distillation.

5. Why is the boiling point of a mixture of an essential oil and water always less than 100 °C?

6. Essential oils and other natural products are considered secondary metabolites—they are not responsible for the primary structure and function of a cell. What are some possible biochemical functions for an essential oil?