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702

Using Microscale Techniques

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PURPOSE OF THE EXPERIMENT

Use glassware commonly found in microscale procedures. Weigh and transfer small amounts of materials. Use air or nitrogen to speed evaporation of volatile solvents. Calculate the percent recovery of benzoic acid dissolved in acetone. Calibrate a micropipet. Calculate the density of cyclohexane. Measure the refractive index and boiling point of cyclohexane. Identify an unknown using physical properties.

BACKGROUND REQUIRED

You should know how to weigh chemicals using a milligram balance.

BACKGROUND INFORMATION

The use of microscale techniques in the general organic laboratory began in response to concerns about laboratory safety and the expense of chemical purchase and disposal. Conducting experiments using solvent volumes of one milliliter instead of one hundred milliliters can significantly reduce the exposure of students and laboratory instructors to large volumes of hazardous organic chemicals.

Microscale experiments also introduce students to a variety of manipulations of small amounts of compounds, each requiring careful technique. A small spillage from one hundred milliliters may not greatly affect the yield in a macroscale experiment. Any observable loss from a one-milliliter sample could result in a significant yield reduction in a microscale experiment.

Reviewing Reaction Vials and Apparatus

Two major styles of microscale glassware are available for use in the organic laboratory. Reactions are conducted in either conical vials or round-bottom flasks. Capacities of these containers are five milliliters or less.

One style typically uses conical vials. The glassware pieces have ground glass fittings that are secured by threaded caps, as shown in Figure 1(a) on the next page.

The other glassware style commonly uses round-bottom flasks. The glassware pieces are connected by special elastomeric connectors. These connectors also contain a detachable rod that can be connected to a support stand, as shown in Figure 1(b) on the next page.

Many microscale reactions can be conducted in test tubes, minimizing the need to purchase special glassware. Reactions can also be conducted in standard laboratory glassware using ten-milliliter round-bottom

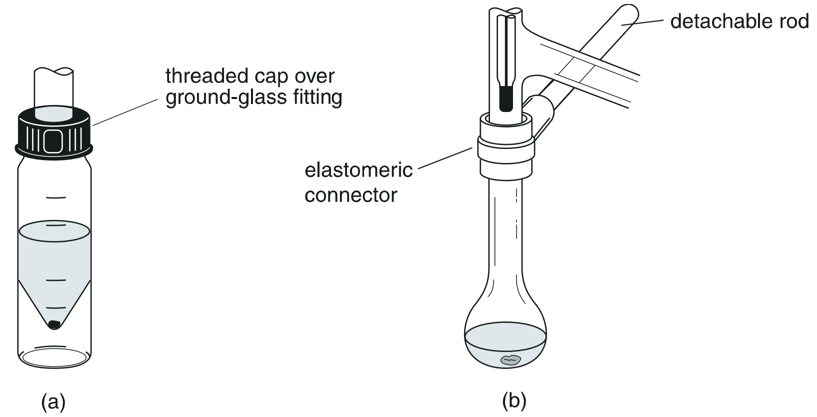


Figure 1 Two styles of microscale glassware: (a) concial vial with threaded cap; (b) round-bottom flask with elastomeric connector

flasks. Reactions using small standard glassware are considered **semi-microscale.**

Heating Microscale Glassware

Sand baths are used to heat microscale reaction mixtures because sand is a poor conductor of heat. As a result, the sand-bath temperature varies depending on the depth below the surface of the sand. A reaction vial set deeply into the sand will be heated to a higher temperature than one set on the sand's surface.

Sand baths are usually constructed in either of two ways. One method is to use a crystallizing dish filled with sand and placed on an electric heater-stirrer, as shown in Figure 7 later in this module. The heater-stirrer can both heat the sand and power a magnetic stirring bar or vane in the reaction container. Alternatively, an electric heating well can be filled with sand, as shown in Figure 8 later in this module. The heat controller for the heating well also heats the sand. If magnetic stirring is required, the heating well can be placed on top of a magnetic stirrer.

Weighing and Transferring Solids and Liquids

Balances capable of weighing to the nearest milligram, 0.001 g, are necessary to measure the masses of the small quantities of chemicals used in microscale experiments. Top-loading electronic balances typically contain a tare function. **Tare** is the mass of an empty container. The tare function resets the balance to 0.000 g with the container on the balance.

For weighing solids, a stainless-steel microspatula is used to transfer a solid directly into a container. At times, glazed weighing paper is used instead of a container. Glazed paper is necessary because filter paper and other coarse papers trap small quantities of solids in their fibers.

For weighing liquids, a Pasteur pipet or a Pasteur filter pipet is used to deliver a liquid directly from one container to another. Many volatile liquids do not adhere well to glass. These liquids are less likely to drip from the bottom of a pipet if it is packed at the tip with a small cotton plug. The cotton plug slows the liquid flow, making leakage less likely. A Pasteur pipet can be modified into a filter pipet, as shown in Figure 2.

The mass of a liquid can often be measured more accurately than its volume. However, transferring known volumes of liquids usually is faster than weighing them. Two common devices for dispensing small liquid volumes are the micropipet and the bottle-top dispenser, which are shown in Figure 3.

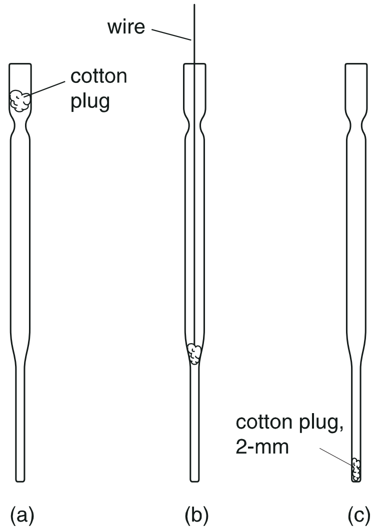
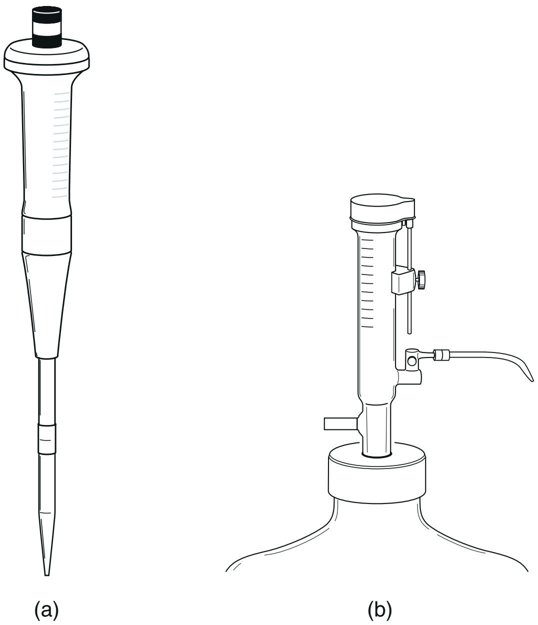


Figure 2 Constructing a Pasteur filter pipet: (a) cotton placed in pipet; (b) wire used to push cotton to pipet tip; (c) finished filter pipet

Figure 3 Two devices used to dispense small volumes of liquid: (a) micropipet; (b) bottle-top dispenser



Using Air or Nitrogen to Speed Evaporation

When a solid is dissolved in a solvent, the solid can be recovered by evaporating the solvent. Placing the vial containing the dissolved solid in a heated sand bath and passing a *gentle* stream of nitrogen or air into the vial significantly increases the evaporation rate.

Calculating Percent Recovery

If the initial mass of material is known, and if the mass of material recovered after drying is known, the percent recovery can be calculated. The mass of the recovered material is divided by the initial mass of the material and multiplied by one hundred percent, as shown by Equation 1.

$$\text{percent recovery, } \% = \left(\frac{\text{mass of dried recovered compound}}{\text{initial mass of compound}}\right)(100\%) \qquad \text{(Eq. 1)}$$

Using and Calibrating Micropipets

Micropipets are available that deliver either fixed volumes or variable volumes. A fixed-volume micropipet dispenses a predetermined volume—for example, 100 microliters (μL)—each time the pipet is used. The volume of liquid delivered by a variable-volume micropipet can be changed. Typically, these micropipets are adjustable over a limited range of volumes. For example, one micropipet is adjustable from 1–10 μL, another is adjustable from 20–200 μL, and a third is adjustable from 100–1000 μL. Pipets calibrated to deliver other volume ranges are also available.

It is important to know the calibrated range of a micropipet. The adjustment mechanism on a micropipet may allow delivery volumes to be set that are outside the calibrated range of accuracy for the micropipet. For example, if a micropipet is made to deliver from 100–1000 μL, and it is set at 75 μL, the micropipet cannot be trusted to accurately deliver 75 μL.

Micropipets have disposable plastic tips. The tips are changed when the liquid being dispensed is changed. Some models have built-in ejectors for the tips.

Setting a volume on a micropipet does not guarantee the micropipet delivers that volume. Periodically, a micropipet must be calibrated to check its accuracy. Water is used for calibration because of its availability and its slow rate of evaporation.

The density of water varies slightly with temperature. Table 1 shows the mass of one milliliter (1000 μL) of water at several temperatures.

Table 1 Mass of 1.00000 mL of water at various temperatures

°C	*mass (g)*	°C	*mass (g)*
4	1.00000	22	0.99780
5	0.99999	23	0.99757
10	0.99973	24	0.99733
15	0.99913	25	0.99708
20	0.99823	26	0.99682
21	0.99802	30	0.99568

The calibration is performed using Equation 2.

$$\left(\frac{\text{measured mass, g}}{\text{mass from Table 1, g}}\right)(1000\ \mu\text{L}) = \text{volume dispensed} \qquad \text{(Eq. 2)}$$

For example, if use of a micropipet set to dispense 1000 μL results in a water mass of 0.992 g at 20 °C, the calibrated volume would be calculated using Equation 2.

$$\left(\frac{0.992\ \text{g}}{0.998\ \text{g}}\right)(1000\ \mu\text{L}) = 994\ \mu\text{L}$$

This calculation means that a setting of 1000 μL would deliver only 994 μL.

Calculating Density

Density is one of several physical properties used to characterize organic compounds. Densities of solids and liquids are typically listed in handbooks of chemical compounds. Density is usually reported in grams per milliliter (g/mL).

Densities can be used to convert volume measurements to mass measurements and vice versa. For example, suppose a procedure calls for 50 milligrams (mg) of cyclohexane. The density of cyclohexane can be used to convert 50 mg to the equivalent volume in μL. The density of cyclohexane is 0.779 g/mL. The equation for calculating density is shown in Equation 3.

$$\text{density, g / mL} = \frac{\text{mass, g}}{\text{volume, mL}} \qquad \text{(Eq. 3)}$$

If both grams and milliliters are divided by one thousand, their ratio remains the same. Thus, Equation 3 can be restated as Equation 4.

$$\text{density, mg / }\mu\text{L} = \frac{\text{mass, mg}}{\text{volume, }\mu\text{L}} \qquad \text{(Eq. 4)}$$

The data can be substituted into this form of the equation.

$$0.779\ \text{mg / }\mu\text{L} = \frac{50\ \text{mg}}{\text{volume, }\mu\text{L}}$$

$$\text{volume, } \mu L = \frac{50 \text{ mg}}{0.779 \text{ mg}/\mu L} = 64 \ \mu L$$

A micropipet can be used to dispense 64 µL of cyclohexane, replacing the need to weigh 50 mg of the compound.

An experimentally determined density can be used to help characterize an unknown compound. Three commonly used devices for density determination are shown in Figure 4. For each device, the mass of the empty device is measured. Then, the device is filled with a known volume of liquid and weighed again. If both the volume of the device and the mass of the liquid are known, the density can be calculated using Equation 4.

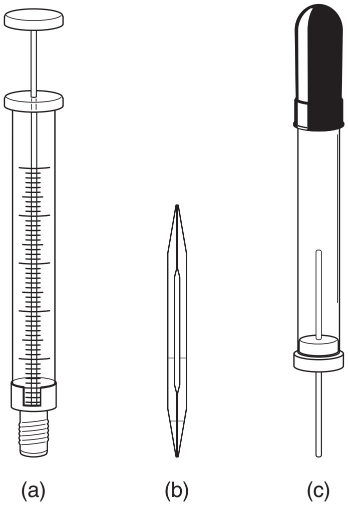


Figure 4 Common devices for density determination: (a) microsyringe; (b) pycnometer; (c) microcapillary pipet

A microsyringe is the most versatile of the three devices because the syringe can measure a range of volumes. Also, the liquid can be recovered after the density measurement is completed.

The pycnometer is a more accurate device for density measurements. The pycnometer fills by capillary action to a precisely known volume. When the end of the pycnometer is touched to the surface of the liquid to be measured, the pycnometer fills automatically.

Microcapillary pipets are also made to hold a precisely known volume of liquid. The pipet is filled either by touching its end to the surface of the liquid and allowing capillary action to draw the liquid into the pipet, or by using a bulb to draw the liquid into the pipet.

Measuring Refractive Index

Refractive index is another physical property that is useful in characterizing liquid organic compounds. When light moves from air into another medium, such as an organic liquid, the light bends because its velocity changes. **Refractive index** is a measure of this change in velocity of light. Figure 5 on the next page shows the refraction of the light as it moves from air into another medium.

Refractive index, n, is measured by comparing the angle of incidence of light in air, θ, to the angle of refraction of the light in the new medium, ϕ, as shown in Figure 5 and Equation 5.

$$n = \frac{\text{velocity}_{\text{air}}}{\text{velocity}_{\text{medium}}} = \frac{\sin\theta}{\sin\phi} \qquad \text{(Eq. 5)}$$

Because different wavelengths of light are refracted to different degrees, refractive index is usually measured using the sodium *D*-line, which has a wavelength of 589 nm, as a reference. A **refractometer** measures the refractive index. The Abbé refractometer shown in Figure 6 on the next page is the most common type. It uses white light, but it is designed to compensate and give values typical of the sodium *D*-line.

Temperature also affects the refraction; therefore, the temperature must be recorded when a measurement is made. Most reported refractive indices are measured at 20 °C, so the refractive index reported for cyclohexane, for example, will be listed as n_D^{20} 1.4266, where *D* indicates the sodium *D*-line and 20 indicates 20 °C.

Two options exist for obtaining a refractive index of a compound at 20 °C. A refractometer can be connected to a constant temperature bath at 20 °C, and the refractive index can be measured directly. Alternatively, the refractive index can be measured at ambient temperature, and a temperature correction can be applied to the reading. The refractive

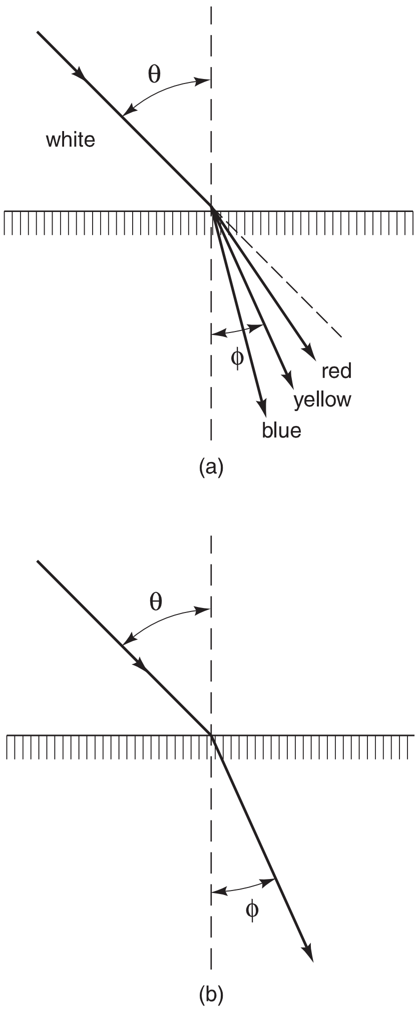


Figure 5 Refraction of: (a) white light; (b) yellow light of sodium at 589 nm

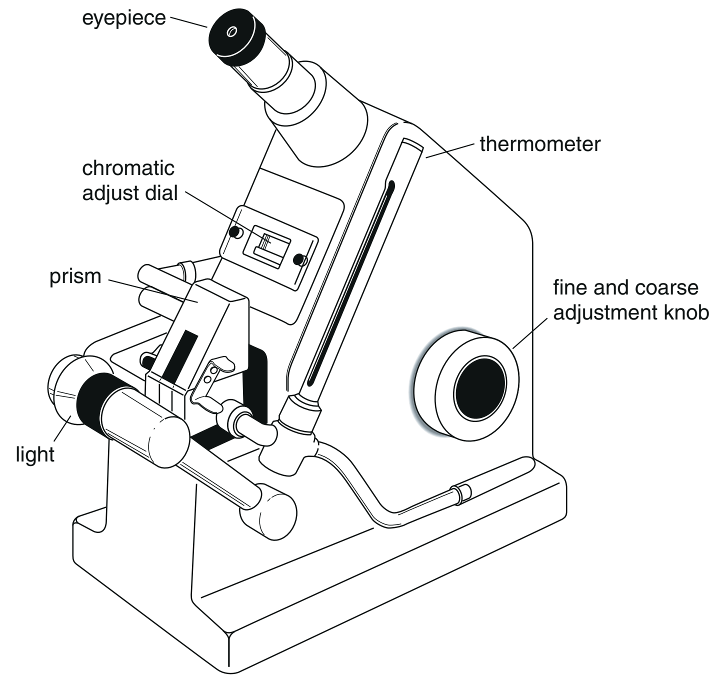


Figure 6 Abbé refractometer

index at 20 °C is calculated by using Equation 6, where T is the ambient temperature in degrees Celsius and n_D^T is the refractive index measured at ambient temperature.

$$n_D^{20} = n_D^T + 0.00045(T - 20\ °\text{C}) \qquad \text{(Eq. 6)}$$

For example, if the refractive index for cyclohexane measured at 24 °C is 1.4248, the corrected measurement is 1.4266.

$$n_D^{20} = 1.4248 + 0.00045(24\ °\text{C} - 20\ °\text{C}) = 1.4266$$

Refractive index is useful for measuring purity. The more pure a compound is, the closer its refractive index will be to the literature value. For an unknown compound, the refractive index, like density, is one additional physical property used to characterize the compound.

Measuring Boiling Points

Boiling points are also useful physical properties for indicating the purity of an organic compound. **Boiling point** is the temperature at which the vapor pressure of a liquid equals atmospheric pressure or some other applied pressure. A boiling point is commonly measured during a **distillation,** in which a liquid is heated to form vapor, and then the vapor is condensed and collected in another container. The boiling temperature is measured as distillation vapor covers the bulb of a thermometer suspended above the boiling liquid. Typically, the most accurate boiling point measurement is the relatively constant temperature achieved during a distillation. Figures 7 and 8, on the next page, show two common microscale distillation setups.

A **capillary bell**, a tiny glass tube open at one end, is used to measure the boiling point of a very small volume of liquid. A bell is placed *open side down* into a melting point capillary tube containing the liquid to be measured. Figure 9(a) on the next page shows this setup.

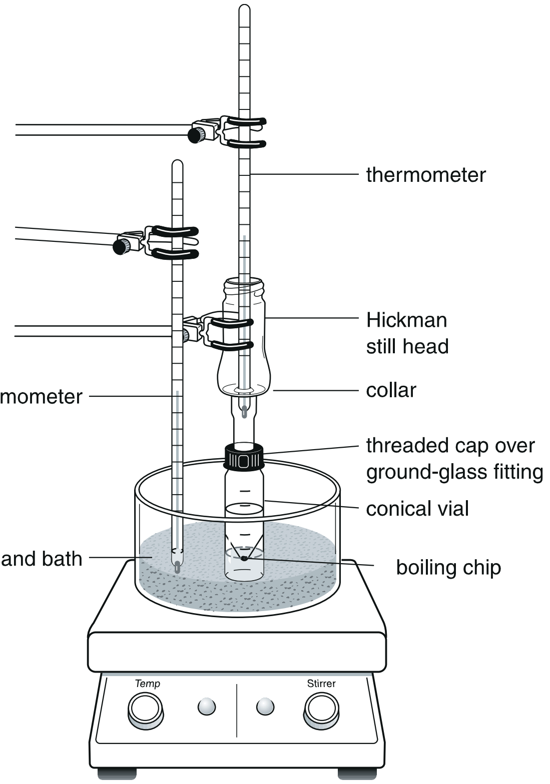


Figure 7 Simple distillation apparatus using a conical vial with ground glass fittings

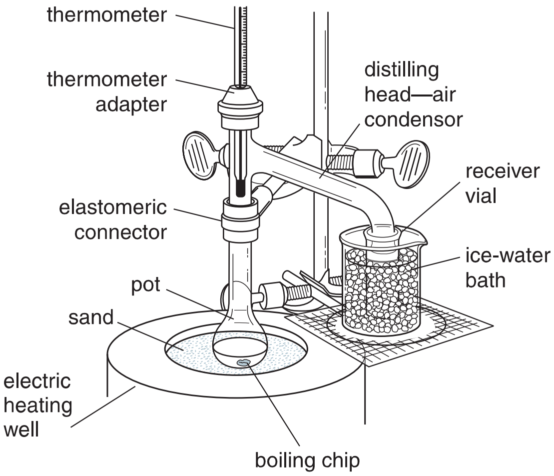


Figure 8 Simple distillation apparatus using a round-bottom flask with elastomeric connectors

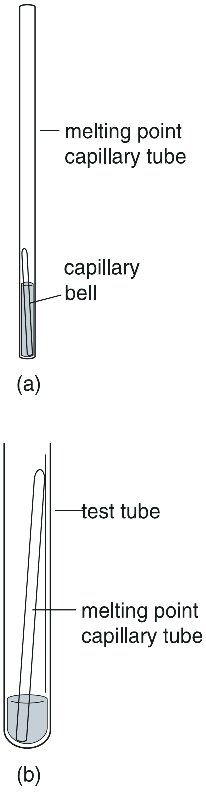


Figure 9 Microscale boiling point setup: (a) capillary bell in a melting point capillary tube; (b) melting point capillary tube in a test tube

The tube is heated in a capillary melting point apparatus to a temperature higher than the boiling point of the liquid. Figure 10 on the next page shows typical melting point apparatus.

The applied heat from the melting point apparatus drives the air out the bell. The air is replaced by vapor from the liquid. As long as the vapor temperature is higher than the boiling point of the liquid, the pressure of the vapor in the bell keeps the liquid out of the bell. When the vapor

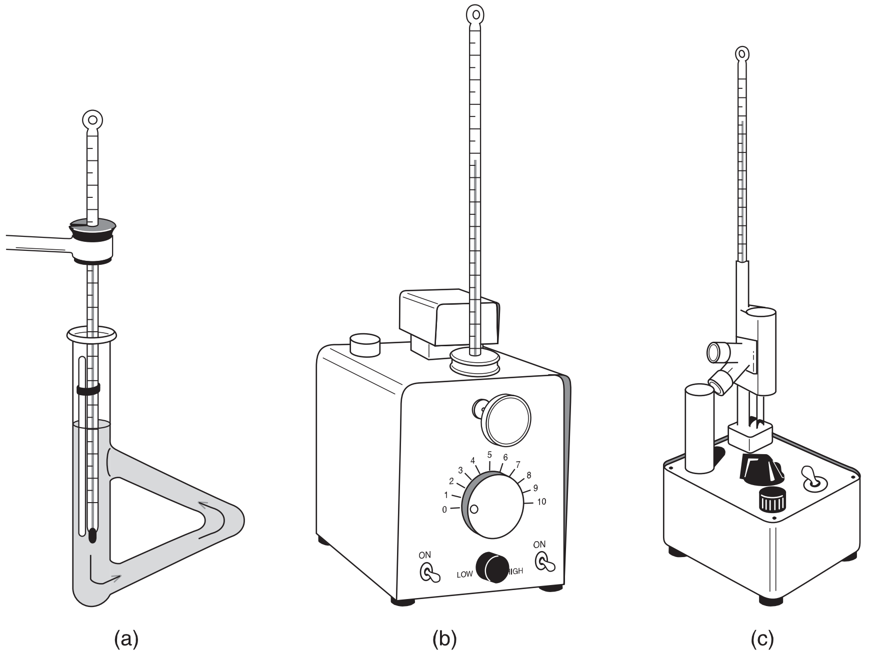


Figure 10 Different types of melting point apparatus: (a) Thiele tube; (b) Thomas-Hoover apparatus; (c) Mel-Temp apparatus

temperature cools to the boiling point of the liquid, atmospheric pressure forces the liquid into the bell. The observed temperature at which the liquid enters the bell is the boiling point.

A modification of the capillary bell method is used when larger liquid volumes are available. The liquid is placed into a small test tube. Then a melting point capillary tube is placed *open side down* into the test tube, as shown in Figure 9(b). The capillary tube functions as the bell. The test tube is heated using either a Thiele tube or a beaker containing mineral oil. The boiling point is measured in the same way as with the capillary bell.

In this experiment, you will use microscale equipment and techniques to weigh benzoic acid, dissolve it in acetone, and evaporate the acetone to recover the benzoic acid. You will use water to calibrate a micropipet. Using cyclohexane, you will use microscale techniques to calculate density, and to measure refractive index and boiling point. Finally, you will use physical properties to identify an unknown compound.

Using Microscale Techniques

Equipment

2 beakers, 25-mL
3 capillary tubes, 1-mm dia. closed-end, melting point type
5 capillary tubes, 1-mm dia. open-end*
2 conical vials, 5.0-mL[†]
cotton
glass scorer
rubber band[‡]
microburner
1000-µL micropipet, with tips
microspatula
500–1000 µL microsyringe, without needle
5 Pasteur pipets, with bulb
5-mm polyethylene tubing
metric ruler
2 × 100-mm test tube[‡]
weighing paper
20-cm copper wire

*for constructing capillary bells

[†] or Erlenmeyer flasks, 10-mL

[‡] for capillary test-tube method

Reagents and Properties

substance	*quantity*	*molar mass (g/mol)*	*mp (°C)*	*bp (°C)*	*density (g/mL)*	n_D^{20}
acetone	1 g	58		56		
benzoic acid	0.100 g	122	122			
1-butanol		74		118	0.810	1.3992
2-butanol		74		100	0.808	1.3954
cyclohexane	1.5 mL	84	6.5	81	0.779	1.4266
1-propanol		62		97	0.780	1.3850
2-propanol		62		82	0.785	1.3776

PROCEDURE

Caution: Wear departmentally approved safety goggles at all times while in the chemistry laboratory.

Always use caution in the laboratory. Many chemicals are potentially harmful. Prevent contact with your eyes, skin, and clothing. Avoid ingesting any of the reagents.

1. Weighing and Transferring Small Amounts of Materials

Caution: Benzoic acid is an irritant. Acetone is flammable and irritating. If possible, use acetone under a ***fume hood***.

Prepare a Pasteur filter pipet by placing a small piece of cotton in the top of a Pasteur pipet, as illustrated in Figure 2 earlier in this module. Use a copper wire to push the cotton to the bottom of the pipet to form a plug 2 mm high. Do not make the plug so tight that liquid is difficult to draw into the pipet.

Zero a top-loading balance by pressing the tare function. Place a 5.0-mL conical vial (or a 10-mL Erlenmeyer flask) on the balance and record the mass of the vial. Again press the tare function to reset the balance at 0.000 g. Using a microspatula, carefully transfer approximately 0.100 g of benzoic acid into the vial. Record the mass to the nearest milligram, 0.001 g.

Again press the tare function to reset the balance at 0.000 g. Use a Pasteur filter pipet to add approximately 1 g of acetone to the vial. Gently swirl the vial to dissolve the benzoic acid.

NOTE 1: The first resistance point on a micropipet is the position for filling the micropipet with liquid for the set volume. If you push the plunger all the way down before you fill the pipet, too much liquid will be drawn into the pipet. If you release the plunger too rapidly when filling the pipet, air will be drawn into the tip, and the volume will not be accurate.

2. Using Air or Nitrogen to Speed Evaporation

Place the vial containing the benzoic acid–acetone solution in a sand bath at a drying station under the fume hood. Use a *gentle* stream of air or nitrogen, one you *barely* feel against your hand, to evaporate the acetone. Focus the stream at the surface of the solution. When evaporation is complete, cool the vial to room temperature.

Zero a top-loading balance, weigh the vial containing the benzoic acid, and record the mass. Subtract the mass of the empty vial from the total mass. Place the benzoic acid into the container labeled "Recovered Benzoic Acid", provided by your laboratory instructor.

3. Using and Calibrating a Micropipet

Fill a 25-mL beaker with distilled or deionized water. Place a tip on a micropipet. To fill the micropipet, press the plunger to the first resistance point, and put the pipet tip into the water. [NOTE 1] Keep the pipet vertical. Slowly release the plunger to allow the pipet to fill. Be careful to keep the tip submerged in the water until the pipet is filled.

To transfer the water, hold the micropipet over another beaker and press the plunger *all the way* down, pushing the water out of the pipet tip. Practice filling and emptying the micropipet.

Zero a top-loading balance containing a 5.0-mL conical vial (or a 10-mL Erlenmeyer flask) by pressing the tare function. Use a micropipet set at 1.000 mL (1000 μL) to dispense water into the vial. Record the mass to the nearest milligram. [NOTE 2]

Do not empty the vial. Zero the balance containing the vial and its contents. Again dispense 1.000 mL of water into the vial, and record the mass to the nearest milligram. Zero the balance a third time, and dispense a third 1.000 mL of water into the vial. Record the mass.

If any one of the three masses varies by more than 0.020 g from the other masses, continue weighing 1.000-mL volumes until your results are consistent. Average the last three masses. Compare your result to the 0.997 g expected for 1.000 mL of water at 25 °C. Refer to Table 1 earlier in this module for the expected water mass if your temperature is outside the 24–26 °C range. Notify your laboratory instructor if the average mass is not consistently within the expected range.

NOTE 2: When weighing the water, your results may vary from the 0.997 g expected for 1.000 mL of water at 25 °C, especially if the pipet is not correctly calibrated. Strive for the consistent measurements that reflect consistent dispensing technique.

4. Calculating the Density of Cyclohexane

Caution: Cyclohexane is flammable and irritating. If possible, use cyclohexane under a ***fume hood.***

Zero a top-loading balance by pressing the tare function. Place a clean, dry microsyringe without attached needle on the balance pan. Record the mass of the syringe to the nearest milligram.

Carefully fill the syringe to the 500-μL mark with cyclohexane. Zero the balance and weigh the filled syringe to the nearest milligram. Record the mass. Subtract the tare of the empty syringe from the total mass of the filled syringe. [NOTE 3]

Place the cyclohexane from the microsyringe into the appropriate container labeled "Recovered Cyclohexane", provided by your laboratory instructor.

NOTE 3: If your syringe retains a small amount of liquid in the tip, reverse the procedure, weighing the full syringe first, then weighing the "empty" syringe containing the small amount of liquid in its tip.

5. Measuring the Refractive Index of Cyclohexane

Caution: Cyclohexane, acetone, and ethanol are flammable and irritating. If possible, use these compounds under a ***fume hood.***

Place a plastic tip extension onto a Pasteur filter pipet, using either a 5-mm piece of polyethylene tubing or a micropipet tip. Open the prisms on the front of the Abbé refractometer. [NOTE 4] Using the extended pipet, place 2–4 drops of cyclohexane on the bottom prism, and close the prisms.

Turn on the light. Look into the eyepiece and move the light source to maximize brightness in the eyepiece. Rotate the fine and coarse adjustment knob on the right side of the refractometer until a light–dark interface is seen.

If the interface is not in focus, or if a colored band is visible, rotate the chromatic adjust dial below the eyepiece until a sharp light–dark interface is obtained. Even if the interface is in focus, rotate the chromatic adjust dial so you will see the effect when it is not in focus. Refocus the interface.

Using the fine adjustment knob, move the interface to the exact middle of the cross hair, as shown in Figure 11.

Depress the button on the left side of the refractometer and keep it depressed while you read the refractive index through the eyepiece. Read and record the measurement to four decimal places. Record the refractometer temperature.

NOTE 4: This procedure is based on the Abbé design for refractometers. Refer to Figure 6. If the refractometer used in your laboratory differs significantly from this design, your laboratory instructor will provide any necessary changes in the procedure.

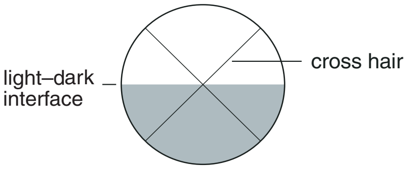


Figure 11 Light–dark interface located at the cross hair of a refractometer

Turn off the light. Open the prisms and clean both prisms with lens paper moistened with ethanol or acetone. Close the prisms.

6. Measuring the Boiling Point of Cyclohexane

[NOTE 5]

NOTE 5: Use boiling point method *A* or *B,* as designated by your laboratory instructor.

Caution: Cyclohexane is flammable and irritating. If possible, use cyclohexane under a ***fume hood***.

Caution: Hot glass looks just like cold glass. Be careful to avoid burns when heating glass.

A. Using the Capillary Bell–Capillary Tube Method

Prepare several capillary bells from 1-mm diameter open-end capillary tubes. Refer to Figure 12.

Rotate the center of a tube in the top of the flame cone from a microburner. As the tube softens, *remove it from the flame,* and quickly pull it straight out in line with the tube until the thin section length is approximately 10 cm. Allow the tube to cool.

Use a glass scorer to score the glass, and break the thin section of the tubing from the capillary tube. Score and break the thin section into pieces approximately 1 cm in length. Touch one end of each piece to the flame to seal the glass, leaving the other end open.

Use a microsyringe to add cyclohexane to a 1-mm diameter melting point capillary tube to a depth of 6–7 mm. If the liquid adheres to the sides of the tube, tap the tube with your finger to cause the liquid to settle to the bottom. Do not overfill. Make certain the liquid depth in the tube is approximately two-thirds the length of the capillary bell.

If tapping does not cause the liquid to settle to the bottom of the melting point tube, place the melting point tube inside a centrifuge tube. Place the centrifuge tube into a tube holder in the centrifuge. Use the centrifuge to force the liquid to the bottom.

Add a capillary bell *open side down* to the melting point capillary tube. Tap the tube, centrifuge the tube, or use a wire to move the bell to the bottom of the tube, as shown in Figure 9(a) earlier in this module.

Place the capillary tube into the melting point apparatus or attach it to the thermometer of a Thiele tube. Make certain the temperature

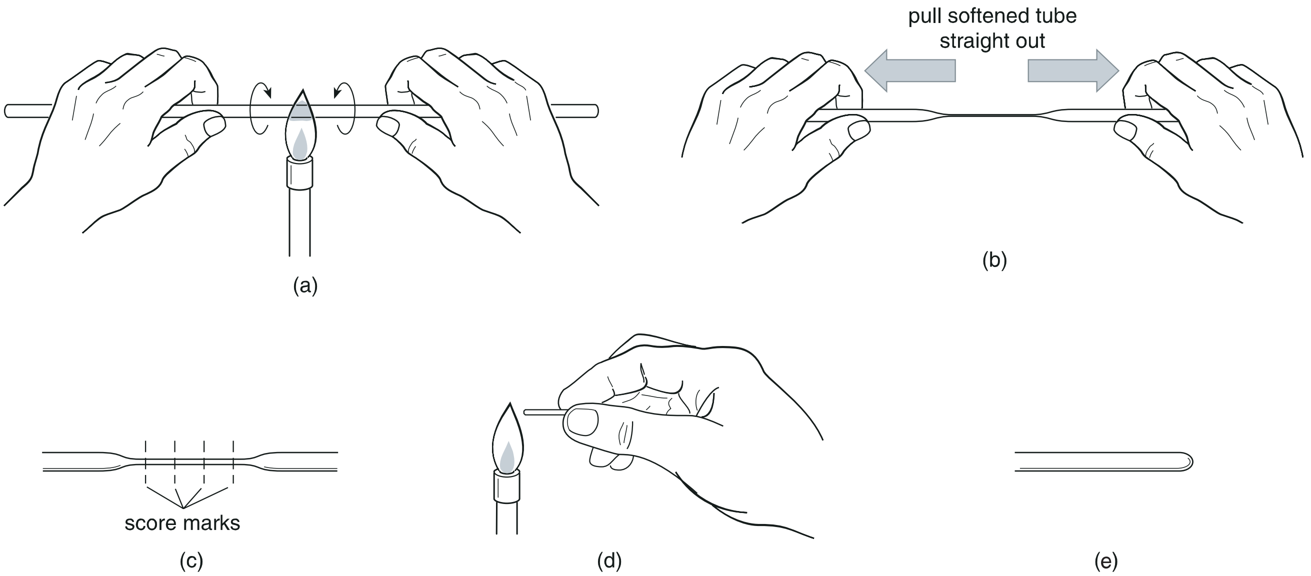


Figure 12 Constructing capillary bells using capillary tubes

of the melting point apparatus is a least 10 °C lower than the expected boiling point.

Slowly heat the capillary tube in the melting point apparatus until bubbles begin to emerge from the bell. Continue heating until bubbles emerge so rapidly that individual bubbles are hard to distinguish. [NOTE 6] Continue heating until the temperature rises 5–10 °C more. Stop heating the tube and allow the system to cool.

NOTE 6: It is important to continue heating the capillary tube in the melting point apparatus until an abundance of bubbles emerges from the capillary bell. Removing the heat before you reach the liquid's boiling point will cause the liquid to immediately fill the bell because the vapor pressure inside the bell is below atmospheric pressure. As a result, you will record a boiling point that is too low.

When the bubbles stop and the liquid *just begins to enter* the bell, observe the temperature. Record this temperature as the boiling point of the liquid. Using new capillary bells each time, repeat the procedure until you obtain consistent results. Compare your results with those of two or more classmates.

B. Using the Capillary Tube–Test Tube Method

Place 1 mL of cyclohexane into a 12 × 100-mm test tube. Place a melting point capillary tube *open side down* into the test tube, as shown in Figure 9(b) earlier in this module.

Use a rubber band to attach the test tube to a thermometer. Insert the thermometer–test tube assembly into a Thiele tube. Keep the rubber band well above the level of the oil in the Thiele tube.

Make certain the temperature of the oil in the Thiele tube is 10 °C or more lower than the expected boiling point.

Slowly heat the Thiele tube until bubbles begin to emerge from the capillary tube. Continue heating until bubbles emerge so rapidly that individual bubbles are hard to distinguish. [NOTE 6] Continue heating until the temperature rises 5–10 °C more. Stop heating the tube and allow the system to cool.

When the bubbles stop and the liquid *just begins to enter* the capillary tube, observe the temperature. Record this temperature as the boiling point of the liquid. Using new capillary tubes each time, repeat the procedure until you obtain consistent results. Compare your results with those of two or more classmates.

7. Using Physical Properties to Identify an Unknown

Caution: 1-Propanol, 2-propanol, 1-butanol, and 2-butanol are flammable and irritating. If possible, use these compounds under a ***fume hood***.

Obtain an unknown liquid compound from your laboratory instructor. Record the identification number. Determine the boiling point, the density, and the refractive index of your unknown. Compare the data for your unknown with the data for the compounds listed in Table 2. Identify your unknown.

Table 2 Physical properties of compounds to be compared with unknowns

compound	*bp (°C)*	*density (mg/μL)*	*refractive index at 20 °C*
1-propanol	97	0.780	1.3850
2-propanol	82	0.785	1.3776
1-butanol	118	0.810	1.3992
2-butanol	100	0.808	1.3954

8. Cleaning Up

Use the labeled collection containers provided by your laboratory instructor.

Your Pasteur filter pipets are clean if they were used only to deliver cyclohexane. Store the filter pipets for future use. If a filter pipet is broken, place it in the container labeled “Broken Glass”. Clean all other glassware with soap or detergent.

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.

Post-Laboratory Questions

1. What was your percent recovery of benzoic acid? Show your calculations.
2. List the procedural errors that could result in a percent recovery of benzoic acid that is (a) too high or (b) too low.
3. (a) What was the average mass of water you obtained in calibrating the micropipet? List the three masses you averaged.
   (b) Using Equation 2, calculate the water volume dispensed by your micropipet.
4. (a) What was your experimentally determined density of cyclohexane? Show your calculation.
   (b) How would you expect the laboratory temperature to affect the result? Briefly explain.
5. (a) What is the refractive index of cyclohexane you measured with the refractometer?
   (b) How did the refractive index change when you corrected for temperature? Show this calculation.
   (c) How closely does the refractive index you obtained for cyclohexane agree with the literature value?
6. What boiling point did you determine for cyclohexane? Were your results reproducible? How did your boiling point compare to boiling points obtained by your classmates?
7. List the physical data for your unknown compound. What compound from Table 2 matches the data for your unknown? Explain your choice.

NAME SECTION DATE

TECH 702/Using Microscale Techniques

Pre-Laboratory Assignment

1. When compared to macroscale experiments, what safety advantage does the use of microscale experiments in organic chemistry offer students and laboratory instructors?

2. Briefly explain why sand baths are commonly used in microscale organic laboratories.

3. What is meant by “tare”?

4. Why should solids be weighed on glazed paper instead of on filter paper?

5. What is the purpose of the cotton plug in a Pasteur filter pipet?

6. What error in volume occurs if a micropipet plunger is pressed all the way down before filling the pipet?

7. How many microliters are occupied by 150 mg of cyclohexane at 25 °C? Show your calculation.

8. How can a refractive index measurement be used to indicate the purity of an organic compound?

9. When using a refractometer, is it possible to measure the refractive index if no light–dark interface is found, but a color band is seen? Briefly explain.

10. What causes a liquid to enter a capillary bell during a boiling point measurement?

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