

- Add the solid (or add the more concentrated reagent) slowly, while swirling, to the volumetric flask. (Caution: Never dump it in!)
- Once the solid compound has dissolved or the more concentrated solution has been diluted, add water (dropwise if necessary) until the calibrated "mark" etched on the volumetric flask is reached (see Technique 16A for reading the meniscus). While securely holding the stopper, invert the flask slowly 10–15 times to ensure that the solution is homogeneous.

TECHNIQUE 6. MEASURING MASS



The mass measurement of a sample can be completed in two ways. In the traditional method, the mass of weighing paper or a clean, dry container (such as a beaker, watchglass, or weighing boat) is first measured and recorded. The sample is then placed on the weighing paper or in the container and this combined mass is measured. The mass of the weighing paper or container is then subtracted from the combined mass to record the mass of the sample.

On modern electronic balances, the mass of the weighing paper or container can be tared out—that is, the balance can be zeroed again after placing the weighing paper or container on the balance, in effect subtracting its mass immediately (and automatically). The sample is then placed on the weighing paper or in the container, and the balance reading is the mass of the sample.

For either method the resultant mass of the sample is the same and is called the tared mass of the sample.

Tared mass: mass of sample without regard to its container



Weighing paper.

The laboratory balance is perhaps the most used and abused piece of equipment in the chemistry laboratory. Therefore, because of its extensive use, you and others must follow several guidelines to maintain the longevity and accuracy of the balance:

- Handle with care; balances are expensive.
- If the balance is not leveled, see your laboratory instructor.
- Use weighing paper, a watchglass, a beaker, or some other container to measure the mass of chemicals; do not place chemicals directly on the balance pan.
- Do not drop anything on the balance pan.
- If the balance is not operating correctly, see your laboratory instructor. Do not attempt to fix it yourself.
- After completing a mass measurement, return the mass settings to the zero position.
- Clean the balance and balance area of any spilled chemicals.

Different balances, having varying degrees of sensitivity, are available for use in the laboratory. These are shown in Figures T.6a through T.6d.

Balance	Sensitivity (g)
Triple-beam (Figure T.6a)	±0.01
Top-loading (Figure T.6b)	±0.01 or ±0.001
Top-loading (Figure T.6c)	±0.0001
Analytical (Figure T.6d)	±0.00001



Figure T.6a Triple-beam balance, sensitivity of ±0.01 g.



Figure T.6b Electronic top-loading balance, sensitivity of ±0.01 g and/or ±0.001 g.



Figure T.6c Electronic analytical balance, sensitivity of ±0.0001 g.



Figure T.6d Electronic analytical balance, sensitivity of ±0.00001 g.

TECHNIQUE 7. HANDLING SMALL VOLUMES

The use of smaller quantities of chemicals for synthesis and testing in the laboratory offers many safety advantages and presents fewer chemical disposal problems. Many of the experimental procedures in this manual were designed with this in mind. Handling small volumes requires special apparatus and technique.

Small test tubes are the chemist's choice for handling small volumes. Common laboratory test tubes are generally of three sizes: the 75-mm (or 3-inch) test tube, the 150-mm (or 6-inch) test tube, and the 200-mm (or 8-inch) test tube (Figure T.7a). The approximate volumes of the three test tubes are as follows:

75-mm (3-inch) test tube	~3 mL
150-mm (6-inch) test tube	~25 mL
200-mm (8-inch) test tube	~75 mL

The 75-mm test tube is often recommended for "small volume" experiments.

A. Test Tubes for Small Volumes



Alternatively, a "well plate" can be used for a number/series of reaction vessels (Figure T.7b). The well plate is especially suited for experiments that require observations

B. Well Plates for Small Volumes

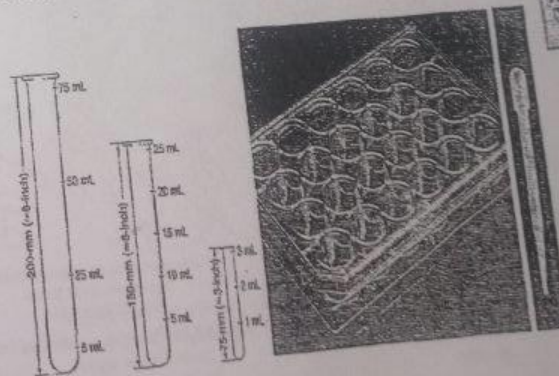


Figure T.7a The three common-sized test tubes for containing reagent solutions.
Figure T.7b A 24-well plate and Beral pipet are used for containing and transferring small quantities of reagent solutions.

Flush the precipitate from a beaker using a wash bottle containing the mixture's solvent (usually deionized water). Hold the beaker over the funnel or receiving vessel (Figure T.11c) at an angle such that the solvent will flow out and down the stirring rod.

D. Flushing a Precipitate from the Beaker

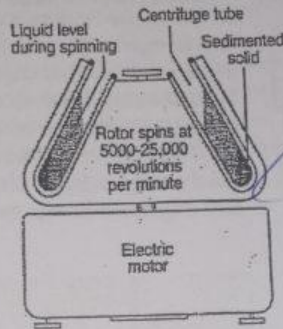


Set up the vacuum filtration apparatus as shown in Figure T.11f. Although a regular filter funnel can be used, the apex of the filter paper is easily ruptured when a vacuum is applied. A Büchner funnel (a disk of filter paper fits over the flat, perforated bottom of the funnel) set into a filter flask connected to a water aspirator is the apparatus normally used for vacuum filtration. Seal the disk of filter paper onto the bottom of the funnel by applying a light suction to the filter paper while adding a small amount of solvent.

E. Vacuum Filtration



Once the filter paper is sealed, turn the water faucet attached to the aspirator completely open to create a full suction. Transfer the mixture to the filter (Technique 10) and wash the precipitate with an appropriate liquid. To remove the suction, first disconnect the hose from the filter flask, and then turn off the water.



Handwritten notes in blue ink: $\frac{18}{30}$ and $\frac{40}{50}$. There is also some illegible red text next to the 18.

Figure T.11g A laboratory centrifuge forces the precipitate to the bottom of the centrifuge tube.

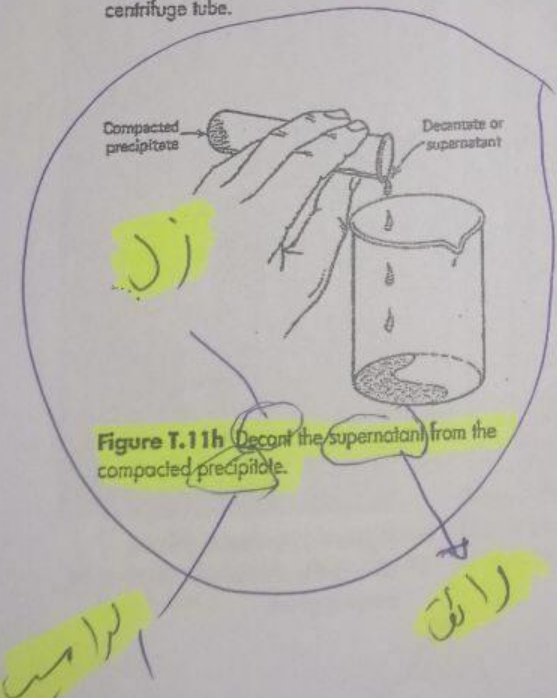


Figure T.11h Decant the supernatant from the compacted precipitate.

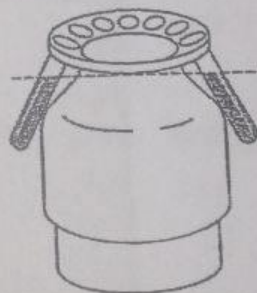


Figure T.11i Balance the centrifuge by placing tubes with equal volumes of liquid opposite each other inside the metal sleeves of the rotor.

تصنيف: كيمياء
 تاريخ: 11/11/2011
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 المحاضر المساعد: د. محمد عبد الحاميد



Figure T.15c Drying and/or firing a crucible and cover.

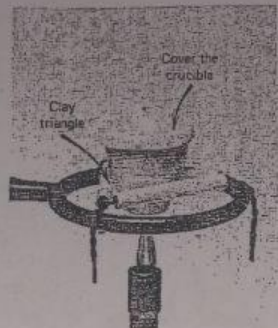


Figure T.15d Ignition of a solid sample in the absence of air.



Figure T.15e Ignition of a solid sample in the presence of air for complete combustion.

The careful measurement and recording of volumes of liquids are necessary to obtain quantitative data for a large number of chemical reactions that occur in solutions. Volumes must be read and recorded as accurately as possible to minimize errors in the data.

TECHNIQUE 16.
MEASURING
VOLUME

1. **Reading a Meniscus.** For measurements of liquids in graduated cylinders, pipets, burets, and volumetric flasks, the volume of a liquid is read at the *bottom of its meniscus*. Position the eye horizontally at the bottom of the meniscus (Figure T.16a) to read the level of the liquid. A clear or transparent liquid is read more easily, especially in a buret, by positioning a black mark (made on a white card) behind or just below the level portion of the liquid. The black background reflects off the bottom of the meniscus and better defines the level of the liquid (Figure T.16b). Substituting a finger for the black mark on the white card also helps in detecting the bottom of the meniscus but is not as effective.

A. Reading and Recording a Meniscus

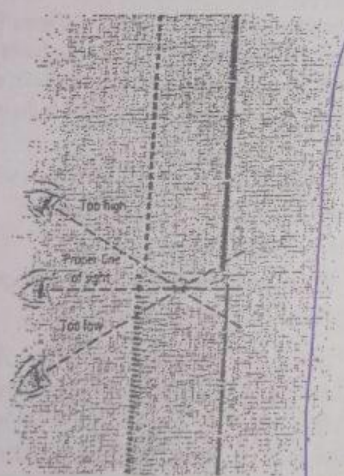


Figure T.16a Read the volume of a liquid with the eye horizontal to the bottom of the meniscus.

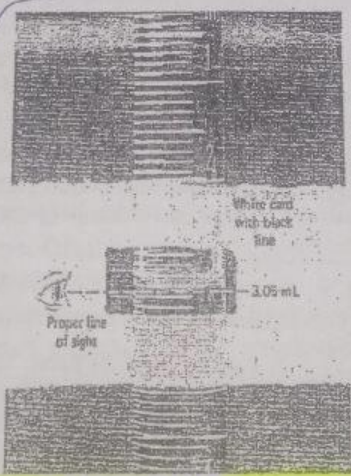


Figure T.16b Use a black line drawn on a white card to assist in pinpointing the location of the bottom of the meniscus.

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allow 10-15 seconds for the titrant to drain from the buret wall, read, and record the volume in the buret (Technique 16A).

To add less than a drop of titrant (commonly referred to as a "half-drop") to the receiving flask, suspend a drop from the buret tip, touch it to the side of the receiving flask, and wash the wall of the receiving flask (with deionized water).

5. **Cleanup.** After completing a series of titrations, drain the titrant from the buret, rinse the buret with several portions of deionized water, and drain each rinse through the tip. Discard the excess titrant and the rinses as advised in the experiment. Store the buret as advised by your laboratory instructor.



An educated nose is an important and very useful asset to the chemist. Use it with caution, however, because some vapors induce nausea and/or are toxic. *Never* hold your nose directly over a vessel. Fan some vapor toward your nose (Figure T.17a). Always consult your laboratory instructor before testing the odor of any chemical.

TECHNIQUE 17.

QUICK TESTS

A. Testing for Odor



To test the acidity/basicity of a solution with test paper, insert a *clean* stirring rod into the solution, withdraw it, and touch it to the test paper (Figure T.17b). For litmus paper acidic solutions turn blue litmus red; basic solutions turn red litmus blue. *Never* place the test paper directly into the solution.

B. Testing for Acidity/Basicity



Other paper-type indicators, such as pHydration paper (Figure T.17c), are also used to gauge the acidity/basicity of a solution.

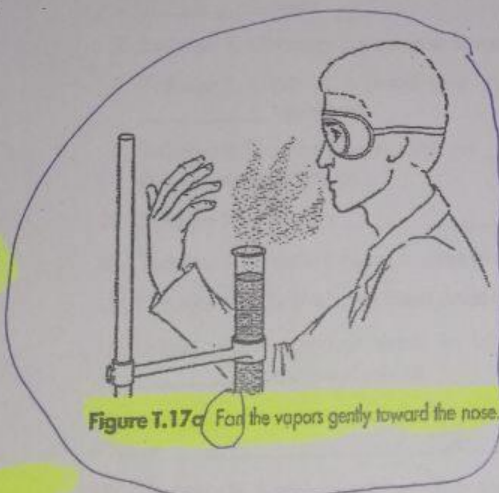


Figure T.17a Fan the vapors gently toward the nose.

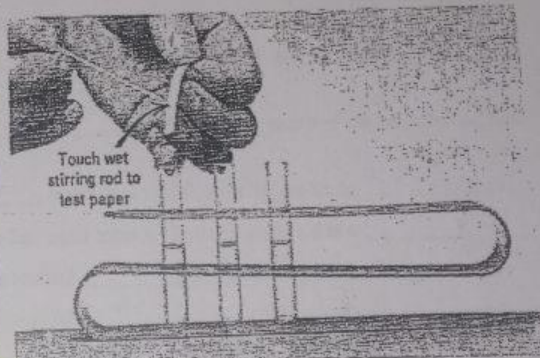


Figure T.17b Test for acidity/basicity.



Figure T.17c Test papers impregnated with a mixture of acid-base indicators can be used to measure the approximate pH of a solution.

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Fan

25

Liquids and solutions are often heated, for example, to promote the rate of a chemical reaction or to hasten a dissolution or precipitation, in a number of different vessels.

Caution: Flammable liquids should never be heated (directly or indirectly) with a flame. Always use a hot plate—refer to Techniques 13C and 13D where hot plates are used.

Hot liquids and solutions can be cooled by placing the glass vessel either under flowing tap water or in an ice bath.

TECHNIQUE 13. HEATING LIQUIDS AND SOLUTIONS



A cool flame is a nonluminous flame supplied with a reduced supply of fuel. In practice, the rule of thumb for creating a cool flame for heating a liquid in a test tube is as follows: if you can feel the heat of the flame with the hand that is holding the test tube clamp, the flame is too hot!

For heating a liquid in a test tube, the test tube should be less than one-third full of liquid. Hold the test tube with a test tube holder at an angle of about 45° with the flame. Move the test tube circularly and continuously in and out of the cool flame, heating from top to bottom, mostly near the top of the liquid (Figure T.13a). **Caution:** Never fix the position of the flame at the base of the test tube, and never point the test tube at anyone; the contents may be ejected violently if the test tube is not heated properly.

See Technique 13D for heating a solution in a test tube to a specified elevated temperature; the hot water bath in Technique 13D is a safer, but slower, procedure.

A. Test Tube Over a "Cool" Flame



An Erlenmeyer flask less than one-fourth full of liquid may be heated directly over a cool flame (see also Technique 13C). Hold the flask with a piece of tightly folded paper or flask tongs (not crucible tongs) and gently and continuously swirl the flask in and out of the flame (Figure T.13b). Set the flask on a wire gauze for cooling; do not place the hot flask directly on the laboratory bench.

B. Erlenmeyer Flask Over a "Cool" Flame

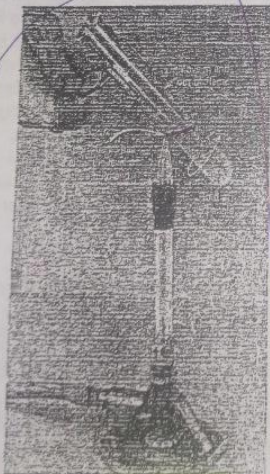


Figure T.13a Move the test tube circularly in and out of the cool flame, heating the liquid or solution from top to bottom.

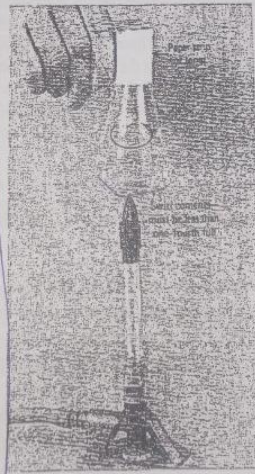


Figure T.13b Swirl the flask, less than one-fourth full, in and out of the cool flame.



Figure T.13c Place the flame directly beneath the tip of the stirring rod in the beaker. Boiling chips may also be placed in the beaker to avoid bumping.

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glass rod
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Bunsen burner
a mixture
carbon meth
burns with
and water
particles
a yellow
addition
volume of a substance, its density can be calculated.
In this experiment, the density of a water-insoluble solid and the
density of an unknown liquid are determined. The data for the mass
and the volume of water displaced (see Figure 1.6) are used to
calculate the density of the water-insoluble solid; the density of the
unknown liquid is calculated from mass and volume measurements
of the liquid.

Chemists conventionally express the units for density in the SI
units g/cm^3 for solids, g/mL for liquids, and g/L for gases at specified
temperatures and pressures.

Procedure Overview: A Bunsen flame is ignited, adjusted, and
analyzed. Various laboratory balances are operated and used. Mass
and volume data are collected and used to determine the density of a
solid and of a liquid.

Perform the experiment with a partner. At each circled superscript⁽¹⁻⁷⁾ in the procedure, stop, and record your observation on the
Report Sheet. Discuss your observations with your lab partner and
your instructor.

EXPERIMENTAL PROCEDURE



I. Lighting the Burner. Properly light a burner using the following
sequence of steps:

- Attach the tubing from the burner to the gas outlet on the lab
bench. Close the gas control valve on the burner (see Figure 1.1)
and fully open the gas valve at the outlet.
- Close the air holes at the base of the burner and slightly open
the gas control valve.
- Bring a lighted match or striker up the outside of the burner
barrel until the escaping gas at the top ignites.
- After the gas ignites, adjust the gas control valve until the flame
is pale blue and has two or more distinct cones.
- Slowly open the air control valve until you hear a slight
buzzing. This sound is characteristic of the hottest flame from
the burner. Too much air may blow the flame out. When the best
adjustment is reached, three distinct cones are visible (Figure
1.3).
- If the flame goes out, immediately close the gas valve at the
outlet and repeat the procedure for lighting the burner

A. Bunsen Burner

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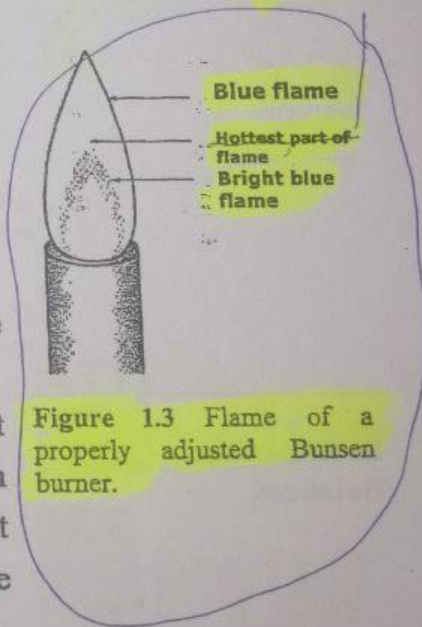


Figure 1.3 Flame of a
properly adjusted Bunsen
burner.

2. Observing Flame Temperatures Using a Wire Gauze

- Temperatures within the second (inner) cone of a nonluminous blue flame approach 1500°C.
- a. Using crucible tongs (or forceps), hold a wire gauze parallel to burner barrel about 1 cm above the burner top (Figure 1.4). Observe the relative heat zones of the flame. Sketch a diagram of your observations on the Report Sheet.
- b. Close the air control valve and repeat the observation with a luminous flame.

3. Observing Flame Temperatures Using the Melting Point of Metals.

- a. Adjust the burner to a nonluminous flame. Use crucible tongs to hold 2-cm strips of copper wire (melting point 1083°C), iron (melting point 1535°C), and aluminum (melting point 660°C) in the various regions of the flame.
- b. On the Report Sheet record the estimated temperature of the flame in the regions designated in Figure 1.5. Extinguish the burner when it is not being used by turning off the gas valve at the base on the lab bench.

wire gauze
burner



Figure 1.4 Hold the wire gauze parallel to burner barrel.

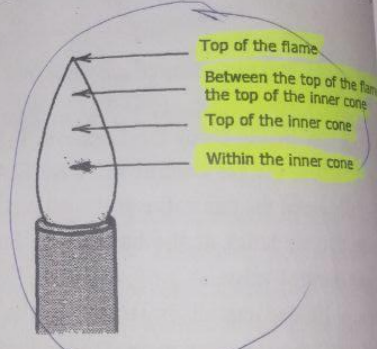


Figure 1.5 Regions of the flame temperature measurements.

B. Laboratory Balances

- 1. Practice Using the Balances. Refer to the Report Sheet for instructions suggested. Measure the mass of several objects. Use the analytical balance only after the instructor explains its operation and is sure to record the mass of the objects according to the sensitivity of the balance.® Refer to Technique 6.



your instructor which based on the densities of your unknowns. Verify your results.

Water-Insoluble Solid.

- Obtain an unknown solid and determine its assigned balance tare.
- Place the solid on the balance pan.
- Record the mass to the nearest 0.001 g.
- Half-fill a 10-mL graduated cylinder with water. Record the volume (Figure 1.6a).
- Place the solid in the cylinder and record a volume.
- Gently slide the known mass into the cylinder held at a 45° angle, removing any air bubbles.
- Record the new volume.
- The volume of the solid is the difference between the two levels. Repeat this procedure for Trial 2.

Figure 1.6 Apparatus for determining the volume of a solid.

Disposal: Check with your instructor regarding returning the solid to the stock container.

2. Liquid, Water-Soluble Solid

- a. Clean and dry the assigned beaker.

we will examine the solubility of a substance in three different solvents: water, cyclohexane, C_6H_{12} , and ethanol, C_2H_5OH . Qualitative solubility is recorded as being soluble (complete dissolution), slightly soluble (partial dissolution), or insoluble.

Density

Recall from Experiment 1 that density is the mass per unit volume. Substances with a large density have a large mass in a small volume. While we commonly say that lead is "heavy", we really mean that it has a high density—it doesn't require much volume in order to have a large mass of lead.

Boiling Point

When a liquid is gradually heated, there is a temperature at which bubbles form spontaneously and continue to form until the entire volume of liquid has been converted to a gas. This "constant" temperature is called the boiling point of the liquid. At the boiling point the temperature of the escaping vapor equals that of the liquid; this temperature depends on the prevailing atmospheric pressure. The boiling point values listed in Table 2.1 are measured at normal atmospheric pressure (called one atmosphere of pressure).

The boiling point of a substance is characteristic of the magnitude of the forces acting between molecules, called intermolecular forces. The stronger the magnitude of the intermolecular forces, the higher will be the boiling point. Since the magnitude of the intermolecular forces is different for each compound, each has a characteristic boiling point.

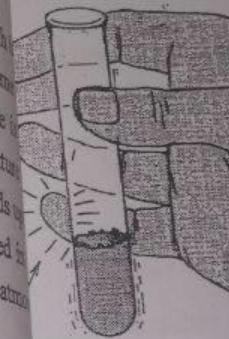


Figure 2.1. Shake the test tube with the pinky finger.

Table 2.1 Physical Properties of Some common Laboratory Chemicals Symbols used:

i = insoluble, sls = slightly soluble, s = soluble

Compound	Density (g/mL)	Boiling Point (°C)	Solubility		
			H ₂ O	C ₆ H ₁₂	C ₂ H ₅ OH
acetone	0.79	56	s	s	s
2-butanone	0.805	80	s	s	s
cyclohexane	0.79	80.74	i	-	s
cyclohexene	0.81	83	i	s	s
ethanol	0.79	79	s	s	-
ethylacetate	0.90	77	sls	s	s
heptane	0.684	98	i	s	s
n-hexane	0.66	68	i	s	s
1-hexene	0.67	63	i	s	s
isopropanol	0.79	83	s	s	s
methanol	0.805	65	s	s	s
n-propanol	1.00	97	s	s	s
water		100.0	-	i	s

EXPERIMENTAL PROCEDURE

Solubility

Solubility in Cyclohexane. Into a 75-mm test tube add 3 drops of your liquid unknown to 1 ml of cyclohexane (**Caution:** avoid breathing vapors. Agitate the contents (Figure 2.1). Does each drop dissolve? Partially dissolve? Record your observations.



Solubility in Water and Ethanol. Repeat the test with water and ethanol as solvents. Describe the solubility of your unknown as soluble, slightly soluble, or insoluble in each solvent.

hap



Tap-tap-tap w/pinky

Figure 2.1. Shake contents of the test tube with the "pinky" finger

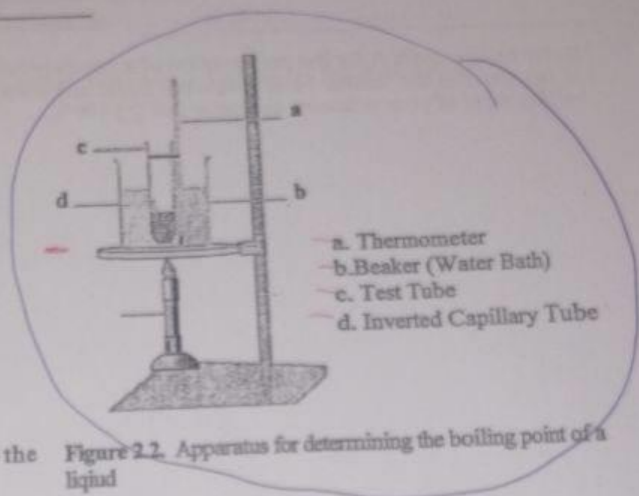


Figure 2.2. Apparatus for determining the boiling point of a liquid

- a. Thermometer
- b. Beaker (Water Bath)
- c. Test Tube
- d. Inverted Capillary Tube

B. Density

Complete two trials to determine the density of your unknown.

1. **Determine Mass and Volume of Unknown.** In a 150-mL beaker weigh (± 0.01 g) a 75-mm test tube. Pipet 2 mL of the liquid into the test tube and reweigh. (**Caution:** use a rubber pipet bulb in drawing the liquid into the pipet.) Calculate the density of the liquid.



C. Boiling Point

Check out a 110°C thermometer. Assemble the apparatus in Figure 2.2. Complete two measurements for the boiling point of your unknown.

1. **Assemble the Boiling Point Apparatus.** Place 10 drops of the unknown liquid (**Caution:** the liquid may be flammable-keep it away from an open Bunsen flame) into a 75-mm test tube. Position it beside the thermometer bulb using a rubber band. Invert (open end down) a 10-cm capillary tube in the liquid. Place the apparatus into the water bath.

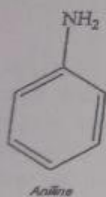
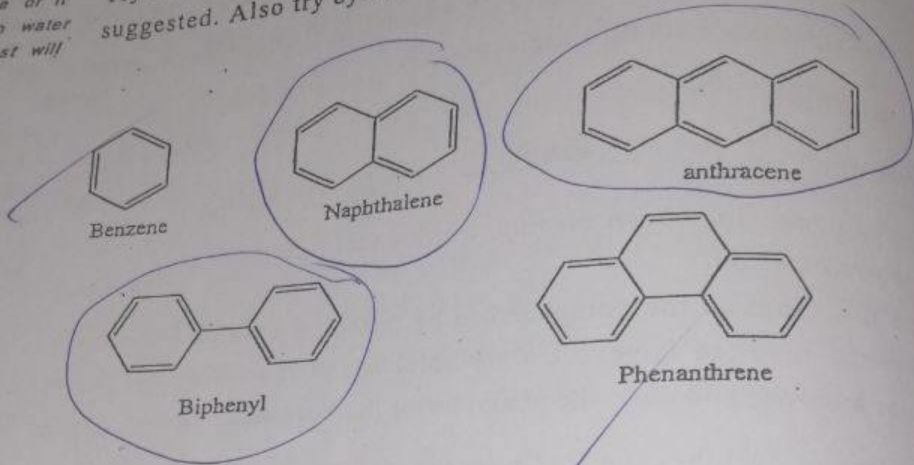


2. **Measure the Boiling Point.** Slowly heat the water in the water bath while stirring with the stirring rod. When a rapid and continuous stream of bubbles escapes the capillary tube, discontinue heating the

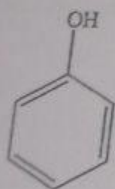


Caution:
Keep the Cover on
the aluminum
chloride as much
as possible or it
will pick up water
and the test will
fail

Incline the test tube and wet the walls with the solution.
Then while it is inclined, spill the aluminum chloride down
the wet wall, Note the color on the wall and in the solution.
Try the test with one or more solid aromatics such as those
suggested. Also try cyclohexane and cyclohexene.



Aniline



Phenol

Caution:
phenol is burns
and also an
anesthetic

F. Electrophilic Aromatic Substitution: **Bromination.**

Activated aromatic compounds such as phenol and aniline tribrominate so readily that they merely need to be shaken with an aqueous bromine solution. Since bromine is not too soluble in water, sodium bromide or potassium bromide is added to increase it. *Have care in using such solution; it will burn you. If you should get it on you, rinse the burn with sodium thiosulfate Solution and then rub glycerol in it gently.* Add 1 drop of the material to be tested to 1 ml of water. Add the bromine solution dropwise with shaking until a color persists. Note whether or not a precipitate is formed. Try this reaction with aniline, 90% phenol and chlorobenzene.

Expe
R
GRO

Name :

1. v

Experiment 1-6

MELTING POINT

Identify and Purity of Solid Organic Compounds

OBJECTIVES

1. Determining the melting point of a pure organic solid.
2. Determining the melting point of an impure organic solid (mixture).
3. Identifying an unknown from its melting point.

INTRODUCTION

The *melting point of a solid* is the temperature at which transition from solid to liquid occurs at atmospheric pressure; or the temperature at which solid and liquid phases are in equilibrium at a pressure of one atmosphere. The melting point is practically unaffected by changes in external pressure, making it a convenient physical constant for the identification of solids.

melting point: the temperature at which solid and liquid phases are in equilibrium at a pressure of one atmosphere

Many organic compounds are solids at room temperature as a result of strong intermolecular forces which hold the individual molecules together in a crystal lattice. The nature and strength of these intermolecular forces are responsible for the observed differences in melting point. In general, if the forces are strong, the melting point will be high, and if they are relatively weak, the melting point will be low.

Many organic compounds are solids at room temperature

A pure solid has a sharp melting point and will melt within a narrow range of $(0.5 - 1)^\circ\text{C}$. Soluble impurities affect the melting point of a solid in the following manner:

if the intermolecular forces are strong, the melting point will be high, and if they are relatively weak, the melting point will be low

a. Lower the melting point of the substance, with the upper limit considerably below the true melting point.

The presence of an impurity in the molten compound,

the intermolecular forces are three types:
Hydrogen,
Dipole-dipole and
London force

A pure solid has a sharp melting point

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ذوبت في
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Soluble Impurities
decreasing the melting
point of a solid

Insoluble Impurities
do not affect the melting
point or the melting point
range

reduces its vapor pressure thus lowering the melting point of the compound (Figure 1.1a). The greater the amount of impurity, the greater is the melting point depression (Figure 1.1b).

b. Broaden the melting point range. Depending on the amount of impurity, the melting process may extend over a range of (2 - 20)°C or more. Insoluble impurities (e.g., glass, sand ...etc.) do not affect the melting point or the melting point range.

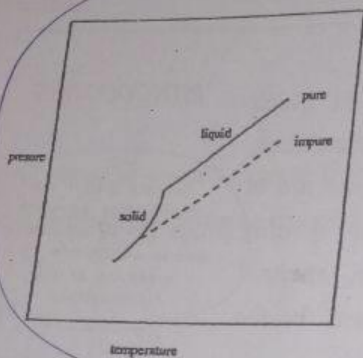


Figure 1.1a
Vapor pressure-temperature diagram.

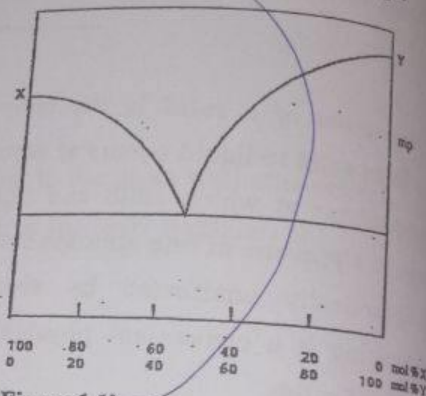
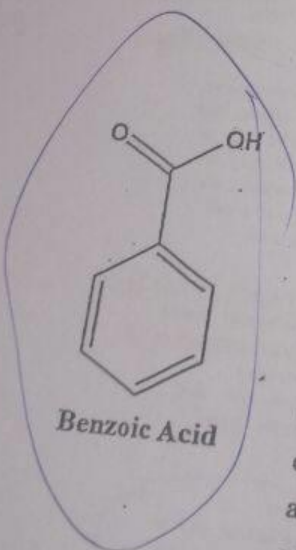


Figure 1.1b
Temperature-composition diagram.



Mixed melting points can be used in the following manner to determine whether two compounds are the same or different even though they have similar melting points. Assuming that a given organic compound (A) melts sharply at 120°C, and benzoic acid (compound B) also has a melting point of 120°C.

Is compound (A) benzoic acid or a different compound?
If compound (A) is benzoic acid, then a mixed melting point of (A) and (B) will melt sharply at 120°C, i.e. the same as each individual compound alone. If, on the other hand, compound (A) is not benzoic acid, then the melting point of a mixture of (A) and (B) will be lowered and the melting range will be broadened. Since they are different compounds each behaves as an impurity in the other.

Apparatus: A points is shown thermometer fitted long-necked flask high boiling and butylphthalate or The thermometer bottom of the flask fastened to the above the level close to and on Capillary melting and 1mm in diameter end of the capillary tube. The dry solid paper with a s then pushed in capillary tube bench top. This is densely packed 2-3 mm.

2. Procedure. small amount into a capillary thermometer heated rapidly point then stir per minute, at which the completely of that substance. The melting factors in a of material thickness

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2. Preparation of the Solution. To prepare the hot solution, the finely divided solid is placed in an Erlenmeyer flask and the selected solvent is added in small portions. The mixture is stirred and heated to boiling after each addition, *until the solid dissolves completely*. A slight excess of the solvent is usually added to compensate for any losses (through evaporation) during hot filtration.

Decolorizing charcoal may be added, if the solution is colored to remove the colored impurities

Decolorizing charcoal may be added at this stage if the solution is colored due to colored impurities. The flask should be removed from the heat source before adding charcoal to it, otherwise bumping will occur.

3. Hot Filtration (Gravity Filtration). Filtration of the hot solution is necessary to remove insoluble impurities. A fluted filter paper and a short-stem funnel (Figure 3.4) allow rapid filtration and avoid premature crystallization inside the stem and on the filter paper.

Filtration of the hot solution is necessary to remove insoluble impurities

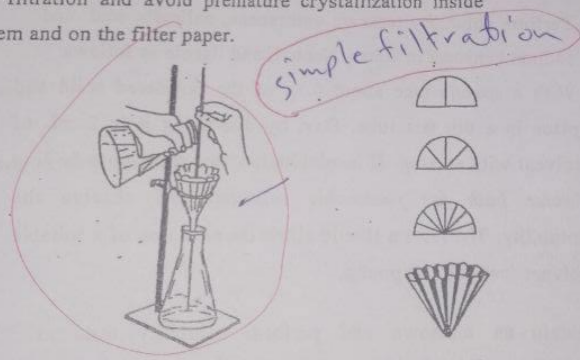


Figure 3.4 : Rapid filtration of a hot solution using a fluted filter paper.

4. Cooling. To induce crystallization, the clear, hot filtrate is allowed to cool down to room temperature, undisturbed, until a large amount of crystals has formed. (Scratching of the inner surface of the glass can help in crystal formation). *The mixture finally chilled in ice to complete crystallization.*

Scratching of the inner surface of the glass can help in crystal formation

Acids

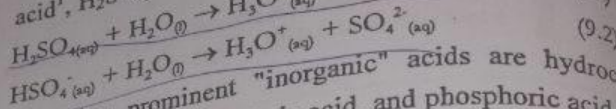
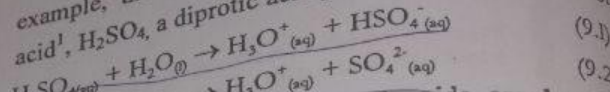
Litmus: a common laboratory acid-base indicator

Since H_3O^+ is a H^+ attached to a water molecule, H_3O^+ acids are also defined as producing H^+ in water.

Diprotic: a substance having two protons (H^+) that can ionize in an aqueous system

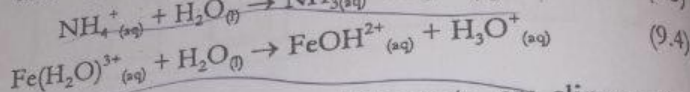
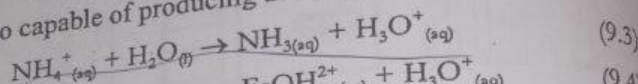
Hydrated: ions having bonded water molecules are hydrated.

Acidic solutions have a sour or tart taste, cause a prickle on the skin, and turn blue litmus red. Nearly all of the foods we consume are acidic ... think of lemon juice as being a taste but milk not quite so. All acids are substances that produce hydronium ion, H_3O^+ , in aqueous solution. For example, the most versatile of all chemicals worldwide is sulfuric acid¹, H_2SO_4 , a diprotic acid producing H_3O^+ in two steps:

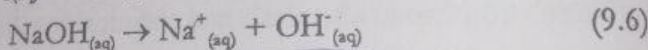
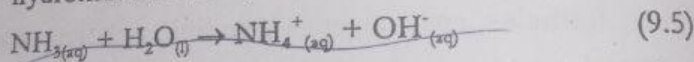


Other prominent "inorganic" acids are hydrochloric acid (called muriatic acid), nitric acid, and phosphoric acid (Figure 9.1). Some common "organic" acids are acetic acid found in vinegar, citric acid found in citrus fruits, and ascorbic acid, the vitamin C (Figure 9.2).

Many cations, such as ammonium and (hydrated) ferric iron, are also capable of producing acidic solutions as well.

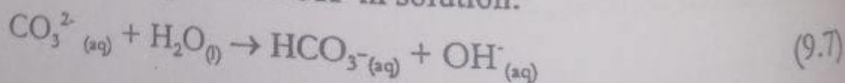


Basic solutions have a bitter taste, are slippery to the touch, and turn red litmus blue. Our palates are unaccustomed to the taste of bases ... think of antacids, soaps, detergents, and household cleaning products. All bases are substances that produce hydroxide ion, OH^- , in aqueous solution. For example, the most common base is ammonia (a naturally produced chemical), the most common laboratory base is sodium hydroxide. In water;



Sodium hydroxide, found in oven and drain cleaners, is commonly called lye or caustic soda; calcium hydroxide is called slaked lime; potassium hydroxide is called caustic potash; magnesium hydroxide, commonly called milk of magnesia, is an antacid and purgative (Figure 9.3).

A large number of anions, such as carbonate and phosphate, are also capable of producing basic solutions as well. For example, sodium carbonate, known as soda ash and washing soda (often used in detergents), produces OH^- in solution.



Bases



Figure 9.1 Phosphoric acid that delivers the tart taste in many soft drinks.

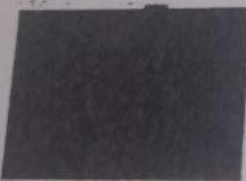


Figure 9.2 Common household acids.

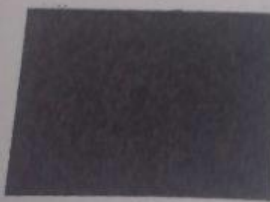


Figure 9.3 Common household bases.

The acidity of most aqueous solutions is measured in terms of the low concentrations of hydronium ion, H_3O^+ . A convenient mathematical expression² used to describe the concentrations of hydronium ion. pH is defined as the negative logarithm of the molar concentration of hydronium ion:

$$pH = -\log[H_3O^+]$$

At 25°C, pure (and neutral) water has a hydronium ion concentration (and a hydroxide ion concentration) of $1.0 \times 10^{-7} M$. The pH of a solution is $pH = 7.0$. Solutions with hydronium ion concentrations greater than $1.0 \times 10^{-7} M$ have lower pH's ($pH = 2.0$). Solutions with hydroxide ion concentrations greater than $1.0 \times 10^{-7} M$ have higher pH's ($pH = 12.0$). Some familiar solutions are shown in Figure 9.4.

or dish about 1 g of
ial 1 on the Repon
e-mL beaker and
with a stirring rod
settle. Leave the

the solution (80.
boil!) for 15 min
pt warm, proceed
he heating.
precipitate to
ature.

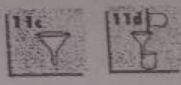
about 30 ml



Withdraw and Save Supernatant. Once the precipitate has settled and the supernatant has cleared in Part A.2b, use a dropping pipet to withdraw enough supernatant to half-fill two 75-mm test tubes, labeled "1" and "2."

Filter the BaSO₄ Precipitate. While the remaining solution from Part A.4 is still warm, quantitatively transfer the precipitate to the filter. Transfer any precipitate on the wall of the beaker to the filter with the aid of a **rubber policeman**; wash any remaining precipitate onto the filter with three or four 5-mL volumes of warm water (from Part A.2c).

Dry and Measure the Amount of BaSO₄ Precipitate. Remove the filter paper and precipitate from the filter funnel. Air-dry the precipitate on the filter paper until the next laboratory period or dry in a 110°C constant temperature drying oven for 30-40 minutes or overnight⁴. Determine the combined mass (±0.001 g) of the precipitate and filter paper. Record.

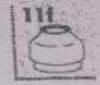


Rubber policeman: a spatula-like rubber tip attached to a stirring rod



- 1. Clarify the Supernatant.** Centrifuge the two collected supernatant samples from Part A.4.
- 2. Test for Excess SO₄²⁻.** Add 2 drops of the test reagent 0.5 M BaCl₂ to the supernatant liquid in test tube 1. If a precipitate forms, the SO₄²⁻ is in excess and Ba²⁺ is the limiting reactant in the original salt mixture.
- 3. Test for Excess Ba²⁺.** Add 2 drops of the test reagent 0.5 M Na₂SO₄ to the supernatant liquid in test tube 2. If a precipitate forms, the Ba²⁺ is in excess and SO₄²⁻ is the limiting reactant in the original salt mixture. An obvious formation of precipitate should appear in only one of the tests.

B. Determination of the Limiting Reactant



Disposal: Dispose of the barium phosphate, including the filter paper, in the "Waste Solids" container. Dispose of the waste solutions in the "Waste Liquids" container.



CLEANUP: Rinse each beaker with small portions of warm water and discard in the "Waste Liquids" container. Rinse twice with tap water and twice with deionized water and discard in the sink.



¹ This is called **digesting** the precipitate. This heating procedure causes the formation of larger BaSO₄ particles for the purpose of a better separation during the filtering procedure.



A vacuum filtering apparatus (Technique 11 E) can also be used; the filtering procedure will be more rapid, but because of the small crystal size, more precipitate will pass through the filter paper.

³ Whatman No. 42 and Fisherbrand Q2 filter papers are both fine-porosity filter papers; a fine-porosity filter paper is required to filter the finely divided BaSO₄ precipitate.

⁴ The drying time can be reduced by placing the precipitate and filter paper into a microwave oven.

Handwritten notes in Arabic and English: "مهم Digest", "to make large particles", "30 دقيقة", "المسحوق".

Handwritten mark resembling the number '13'.

مؤسسة التميز
تعدون - جامعة لعمدة تجميل
الميزانية التشغيلية

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Experiment 96

REACTIONS OF THE FUNCTIONAL GROUPS I ALKANES, ALKENES, AND AROMATICS

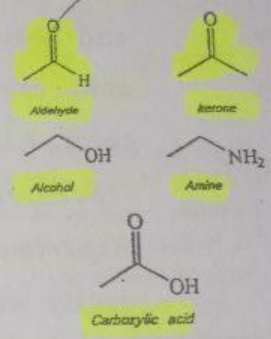
OBJECTIVES

1. Distinguish between the functional group by chemical Reaction.
2. Understanding the Aromatic compound Reaction

Organic compounds are divided into functional groups for the obvious reason that they have different chemical behavior. Each functional group responds or fails to respond to certain classes of chemical reactions. They vary from the reactive aldehydes to the relatively unreactive alkanes. In Experiments 4, 7, and 8, we shall examine the characteristic chemical behavior of the main organic functional groups.

INTRODUCTION

Some of function group that will be studied



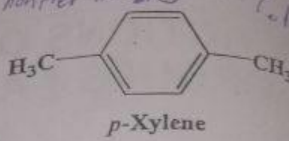
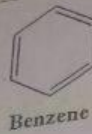
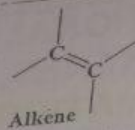
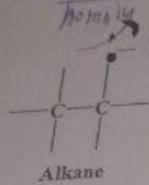
In this experiment, the characteristic reactions of alkanes, alkenes, and aromatic hydrocarbons are examined. alkanes are saturated hydrocarbons in which each carbon has a maximum number of bonded atoms (four). All bonds are single bonds. Alkenes have one or more double bonds. Aromatic compounds include a variety of resonance stabilized substances, of which benzene is the most common. Because benzene as recently come under suspicion as causing leukemia, it will not be used here. It is added along with xylene and toluene to high-octane gasolines.

Alkanes are saturated hydrocarbons in which each carbon has a maximum number of bonded atoms (four)

Alkenes are Unsaturated hydrocarbons in which have one or more double bonds

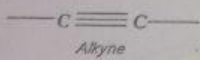
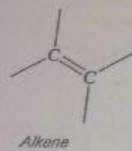
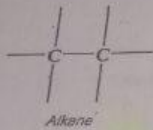
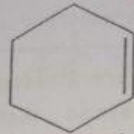
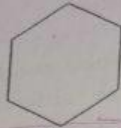
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Physical properties



The following compounds will be used in most of the tests described in the procedures.

Alkynes are inactive



Aluminum Chloride
Anhydrous: $AlCl_3$

Chloroform $CHCl_3$

The alkanes will betray themselves by their inactivity in almost any test we shall try. The alkenes, alkynes, and some aromatics dissolve in concentrated sulfuric acid (Experimental Procedure Part A), often with color. Alkenes and alkynes add bromine (Experimental Procedure Part B) readily and decolorize permanganate (Experimental Procedure Part C). Aromatics substitute readily with strong Lewis acid-type reagents, such as test D (in Experimental Procedure) using aluminum chloride and chloroform. Alkynes are rather uncommon and the best tests provide explosive compounds so they will not be tested here. The tests will be performed both on compounds that give positive reactions and on those that do not. Note and record any physical observations such as heat evolution, color change, formation of a precipitate, and formation or disappearance of layers.

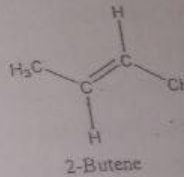
Alkenes + Alkynes are active

$C \equiv C$
Key reminder

H.C. + O₂ → CO₂
Energy heat

solubility → alkene-dissolve
likes water - polar

A. Sulfuric Acid (H_2SO_4)



With care, put about in a 10 X 75-mm hydrocarbon to be tested tube with your left top. Gently strike downward motion practice, using w mixing without s Observe careful dissolving. A y has taken place her the hydroc meniscus Look more drops o does not dis fails test C i Try this test

B. Bromine

Alkane:

Experiment 6 MOLAR MASS OF A VOLATILE LIQUID

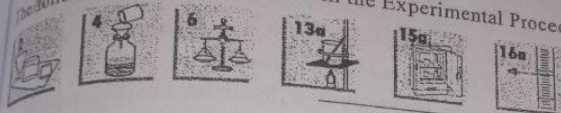
The mercury barometer accurately measures atmospheric pressure in mmHg (or torr).

To determine the molar mass (molecular weight) of a volatile liquid

OBJECTIVES

The following techniques are used in the Experimental Procedure

TECHNIQUES



The measurement of the molar mass of a compound is one of the first determinations a chemist makes after the synthesis of a new compound. In fact, in working with chemicals in academia, research, and industry, chemists must always know the properties of the chemicals with which they are working; one of the most fundamental properties is the molar mass of their compounds.

INTRODUCTION

In this experiment the molar mass of a low boiling-point liquid is determined by the Dumas Method (John Dumas, 1800-1884). The procedure involves vaporizing the liquid into a fixed-volume vessel at a measured temperature and barometric pressure. From the data and the use of the ideal gas law equation (assuming ideal gas behavior), the moles of vaporized liquid, n_{vapor} , is calculated:

$$n_{\text{vapor}} = \frac{PV}{RT} = \frac{\text{pressure (atm)} \times \text{volume (L)}}{R (\text{L} \cdot \text{atm/mol} \cdot \text{K}) \times \text{temperature (K)}} \quad (3.1)$$

In this equation, R is the universal gas constant, P is the barometric pressure, V is the volume of the vessel into which the liquid is vaporized, and T is the temperature of the vapor. $R = 0.08206 \text{ L} \cdot \text{atm/mol} \cdot \text{K}$

The mass of the vapor, m_{vapor} , is determined from the difference between the "empty" vessel and the vapor-filled vessel.

$$m_{\text{vapor}} = m_{\text{flask + vapor}} - m_{\text{flask}} \quad (3.2)$$

The molar mass of the compound, M_{compound} , is then calculated from the data.

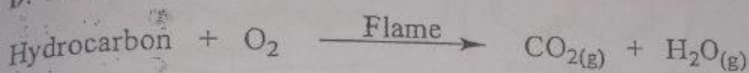
$$M_{\text{compound}} = \frac{m_{\text{vapor}}}{n_{\text{vapor}}} \quad (3.3)$$

If too much permanganate is used; the test may be masked by the intense color of the excess.

Place 1ml of ethanol in each of three test tubes. Add 5 to 10 drops of cyclohexane to the first, cyclohexene to the second, and Chlorobenzene to the third. Add a dilute solution of potassium permanganate drop wise and observe for an immediate reaction.

Caution:
Be careful with adding the $KMnO_4$. If too much is used; the test may be masked by the intense color of the excess.

D. Combustion

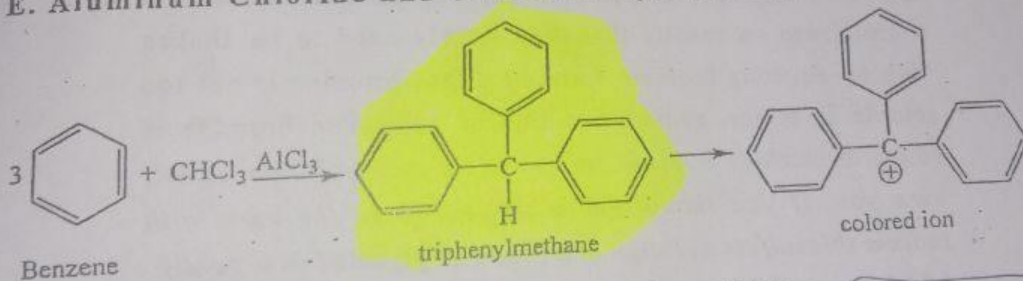


Saturated compounds burn cleanly, while unsaturated ones tend to produce soot.

Place a few drops of the compound to be tested on a watch glass or the inverted cover of a crucible. Ignite it with a match or a burner and note the character of the burning.

Try the test on cyclohexane, cyclohexene, and toluene.

E. Aluminum Chloride and Chloroform. ($AlCl_3 \setminus CHCl_3$)



This is a complex reaction, involving several reactions ending in a colored product somewhat as illustrated above. Aromatic compounds produce colors whereas alkenes do not. Benzenes will give orange-to-red colors, naphthalenes will be blue, biphenyls and phenanthrenes will be purple, and anthracenes will be green. You may wish to do the following test in a hood. Add a small spatula of solid aromatic compound to 1 ml of dry chloroform in a 10 X 75-mm test tube. Get a little anhydrous aluminum chloride on a spatula.

Reaction of aromatic compound with Aluminum Chloride and Chloroform will give colored ion like:

Benzene	orange-red
Naphthalene	blue,
biphenyl	purple
phenanthrene	purple
Anthracene	green.

General Chemistry Laboratory

Experiment 1 :

◆ Tools ...

 Funnel	 Erlenmeyer flask قارورة	 Beaker	 graduated cylinder مدرجة
 Pipette	 Test Tube	 Burette	 Test Tube Rack رف
 Wire Gauze شاش	 Dropper	 Wash Bottle	 Bunsen Burner موتد
 Iron Ring	 Stand	 Clamp معلق	 Pipette Filler
 Crucible Tongs	 Clay Triangle	 Crucible & Lid	 Glass Rod قضيب زجاجي
 Test Tube Brush	 Goggles	 Spatula	

MANDATORY SIGNS

CHEMICALS MANUFACTURING



HAIR MUST BE CONTAINED	PROTECTIVE CLOTHING MUST BE WORN	FACE MASK MUST BE WORN	USE ANTI CORROSIVE GLOVES	USE ANTI CORROSIVE BOOTS
SAFETY GOGGLES MUST BE WORN	POISON GAS USE BREATHING APPARATUS	EXPLOSIVE GAS KEEP DOOR LOCKED	FLAMMABLE GAS NO OPEN FLAMES	CHEMICAL HAZARD AUTHORIZED PERSONNEL ONLY

Flammable	Poison	Radioactive مشعة
Non-ionizing Radiation	Biohazard	High Voltage
Use Gas Mask	Risk of Explosion انفجار	
SELECT CARCINOGEN مادة مسرطنة	Corrosive substance أحماض (مذيبة)	

