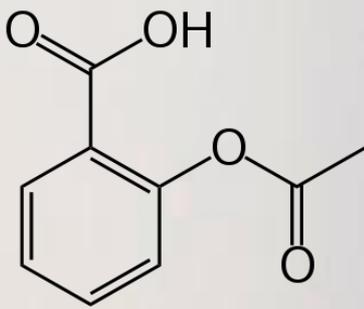


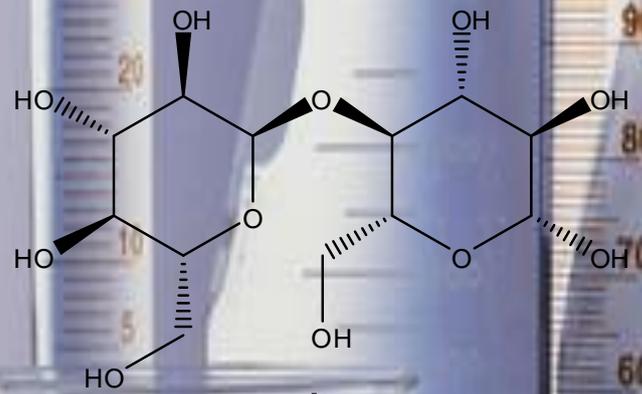


Mutah University
Faculty of Science
Department of Chemistry

**Laboratory Experiments For
General & Organic Chemistry
(Chem. 0303003)
*For Medical student
(2023/2024)***



Aspirin



maltose

كيمياء عامة عضوية عملي (ك003) لطلبة الطب



Mutah University
Faculty of Science
Department of Chemistry
General and Organic Chemistry Laboratory for
Students of Medicine (0303003)
First Semester 2023/2024

Course Contents

<i>Exp.#</i>	<i>Experiment Name</i>	<i>Week</i>	<i>Date</i>
1	Basic Laboratory Operations	3	22/10 - 26/10
2	Identification of Compounds: Chemical Properties	4	29/10 – 2/11
3	Limiting Reactant	5	5/11 – 9/11
4	Acids and Bases	6	12/11 – 16/11
5	Antacid Analysis	7	19/11 – 16/11
6	Molar Mass of a Volatile Liquid	8	26/11 – 30/11
7	Preparation of Aspirin	9	3/12 – 7/12
8	Functional groups (I): Reactions of Alkanes, Alkenes and Aromatics.	10	10/12 – 14/12
9	Functional groups (II): Alcohols, Aldehydes, and Ketones	11	17/12 – 21/12
	Final Exam	12	Thur. 28/12/2023
	-----	13	31/12 – 04/1
	University Final Exams for Online Courses	14	06/1 – 11/1
	<i>University Final Exams</i>	15	13/1(Sat.)–25/1(Thurs.)

Evaluation:

Final Exam: 25% of the whole general and Organic Chemistry Course (108).

The End

Contents

NO.	Subject	Page No.
1	Laboratory Safety in (English)	3
2	Laboratory Techniques	7
3	Laboratory Safety in (Arabic)	33
4	Special Laboratory Equipment	35
5	Syllabus	37
6	Experiments	
	Basic Laboratory Operations	39
	Identification Of A compound: Chemical Properties	51
	Limiting Reactant	63
	Acids and Bases	74
	Antacid Analysis	85
	Molar Mass of a Volatile Liquid	93
	Preparation of Aspirin	101
	Functional groups (I):	108
	Reactions of Alkanes, Alkenes and Aromatics.	
	Functional groups (II):	117
	Alcohols, Aldehydes, and Ketones.	
7	Appendix G: Solubility rules	127



Laboratory Safety and Guidelines

Wearing proper laboratory attire protects against chemical burns and irritations.

The chemistry laboratory is one of the safest environments on campus or in an industrial setting. Every chemist, trained to be aware of the potential dangers of chemicals, is additionally careful in handling, storing, and disposing of chemicals. Laboratory safety should be a constant concern to everyone in the laboratory.

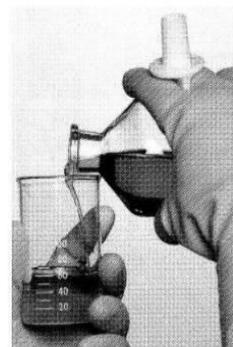
Be sure that you and your partners practice laboratory safety and follow basic laboratory rules. It is your responsibility, *not* the instructor's, to **play it safe**. A little extra effort on your part will assure others that the chemistry laboratory continues to be safe. Accidents do and will occur, but most often they are caused by carelessness, thoughtlessness, or neglect.

On the inside front cover of this book, there is space to list the location of important safety equipment and other valuable reference information that are useful in the laboratory. You will be asked to complete this at your earliest laboratory meeting.

This section of the manual has guidelines for making laboratory work a safe and meaningful venture. Depending on the specific laboratory setting or experiment, other guidelines for a safe laboratory may be enforced. Study the following guidelines carefully before answering the questions on the Report Sheet of Dry Lab I.

1. Approved safety goggles or eye shields *must be worn* at all times to guard against the laboratory accidents of others as well as your own. Contact lenses should be replaced with prescription glasses. In rare cases where contact lenses must be worn, eye protection (safety goggles) is absolutely necessary. A person wearing prescription glasses must also wear safety goggles or an eye shield. Discuss any interpretations of this with your laboratory instructor.
2. Shoes *must* be worn. Wear only shoes that shed liquids. Sandals, canvas shoes, and high-heeled shoes are *not* permitted.
3. Secure long hair and remove (or secure) neckties and scarves.
4. Wear nonsynthetic (cotton) clothing that is not torn or frayed. Shirts and blouses should not be frilled or flared, and the sleeves should be close-fit. In case of fire, synthetic (e.g., nylon, spandex) clothes may be difficult to remove quickly because they tend to stick to the skin.
5. Clothing should cover the skin from "neck to (below the) knee" and *at least* to the wrist. Discuss any interpretations of this with your laboratory instructor. See the opening photo.
6. Gloves are often worn to protect the hand when transferring corrosive liquids. Always consult with your laboratory instructor.

A. Self-Protection

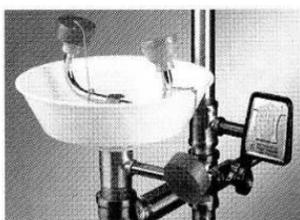


Laboratory gloves protect the skin from chemicals.

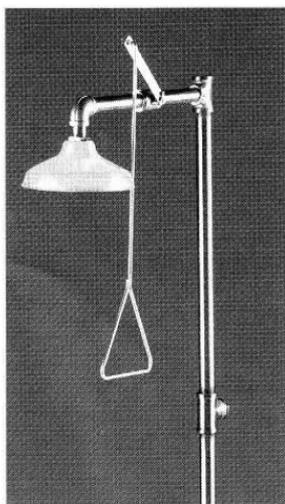


7. To protect outer clothing, the wearing of laboratory aprons or coats (with snap fasteners only) is encouraged.
8. *Never* taste, smell, or touch a chemical or solution unless specifically directed to do so (see B.4 below). Individual allergic or sensitivity responses to chemicals cannot be anticipated. Poisonous substances are not always labeled.
9. For additional information on handling chemicals in the laboratory, refer to Technique 3, page 14.

B. In Case of an Accident



An eye wash can quickly remove chemicals from the eyes.



A safety shower can quickly remove chemicals from the body.



1. *Do not panic.* The most important first action after an accident is the care of the individual. **Alert your laboratory instructor immediately!** If a person is injured, provide or seek aid *immediately*; clothing and books can be replaced and experiments can be performed again later. Second, take the appropriate action regarding the accident: clean up the chemical (see B.8 below), use the fire extinguisher (see B.6 below), and so on.
2. Even if the accident or injury is regarded as minor, *notify your instructor* at once. A written report of an(y) accident may be required. Check with your laboratory instructor.
3. Wash your hands often during the laboratory. Always wash your hands before leaving the laboratory and your arms and face immediately thereafter in the washroom. Toxic or otherwise dangerous chemicals may be inadvertently transferred to the skin and from the skin to the mouth.
4. Whenever your skin (hands, arms, face, etc.) comes into contact with chemicals, quickly flush the affected area for several minutes with tap water followed by thorough washing with soap and water. Use the eyewash fountain to flush chemicals from the eyes and face. *Get help immediately.* Do *not* rub the affected area, especially the face or eyes, with your hands before washing.
5. Chemical spills over a large part of the body require immediate action. Using the safety shower, flood the affected area for at least 5 minutes. Remove all contaminated clothing if necessary. Use a mild detergent and water only (no salves, creams, lotions, etc.). Get medical attention.
6. In case of fire, discharge a fire extinguisher at the base of the flames and move it from one side to the other. Small flames can be smothered with a watchglass (do *not* use a towel, it may catch on fire). Do *not* discharge a fire extinguisher when a person's clothing is on fire—use the safety shower.
7. For abrasions or cuts, flush the affected area with water. Any further treatment should be given only after consulting with the laboratory instructor.
For burns, the affected area should be rubbed with ice, submerged in an ice/water bath, and/or placed under running water for several minutes to withdraw heat from the burned area.
8. Treat chemical spills in the laboratory as follows:
 - Alert your neighbors and the laboratory instructor.
 - Clean up the spill as directed by the laboratory instructor.
 - If the substance is volatile, flammable, or toxic, warn everyone of the accident.
9. For additional information on the disposal of chemicals in the laboratory, refer to Technique 4, page 15.

C. Laboratory Rules

In addition to the guidelines for self-protection (Part A), the following rules must be followed.

1. *Smoking, drinking, eating, and chewing* (including gum and tobacco) are not permitted at any time because chemicals may inadvertently enter the mouth or lungs. Your hands may be contaminated with an “unsafe” chemical. Do not place any ob-

jects, including pens or pencils, in your mouth during or after the laboratory period. These objects may have picked up a contaminant from the laboratory bench.

2. Do *not* work in the laboratory alone. The laboratory instructor must be present.
3. Inquisitiveness and creativeness in the laboratory are encouraged. However, variations or alterations of the Experimental Procedure are forbidden without prior approval of the laboratory instructor. If your chemical intuition suggests further experimentation, consult with your laboratory instructor.
4. Maintain an orderly, clean laboratory desk and drawer. Immediately clean up all chemical spills, paper scraps, and glassware. Discard wastes as directed by your laboratory instructor.
5. Keep drawers or cabinets closed and the aisles free of any obstructions. Do *not* place book bags, athletic equipment, or other items on the floor near any lab bench.
6. At the end of the laboratory period, completely clear the lab bench of equipment, clean it with a damp sponge or paper towel (and properly discard), and clean the sinks of all debris. Also clean all glassware used in the experiment (see Technique 2, page 13).
7. Be aware of your neighbors' activities; you may be a victim of their mistakes. Advise them of improper techniques or unsafe practices. If necessary, tell the instructor.
8. For all other rules, **listen to your instructor!**



1. Maintain a wholesome, professional attitude. Horseplay and other careless acts are prohibited. No personal audio or other "entertainment" equipment is allowed in the laboratory.
2. Do *not* entertain guests in the laboratory. Your total concentration on the experiment is required for a safe, meaningful laboratory experience. You may socialize with others in the lab, but do not have a party! You are expected to maintain a learning environment.
3. Scientists learn much by discussion with one another. Likewise, you may profit by discussion with your laboratory instructor or classmates, but *not* by copying from them.
4. *Prepare* for each experiment by completing the Prelaboratory Assignment and by studying the Objectives, Techniques, Introduction, and Experimental Procedure *before lab*. Advanced preparation will save you time, reduce the chances of personal injury and damage to equipment, and provide a more meaningful learning experience (and a better lab grade).

D. Working in the Laboratory



Laboratory facilities must be designed for safety.

5. Are any calculations required before you begin to collect the data for the experiment? Complete those before the laboratory period begins.
6. As a guide to studying the Experimental Procedure, always try to understand the reason for each step and why you are doing it. **Think**, whistle if you like, while working in the laboratory.
7. Note beforehand the need for any extra equipment in the experiment and obtain it all at once from the stockroom.
8. Review the Laboratory Questions at the conclusion of the Report Sheet before *and* as you perform the experiment. These questions are intended to enhance your understanding of the chemical principles on which the experiment is based.

NOTES ON LABORATORY SAFETY AND GUIDELINES



The Application of proper laboratory techniques improves data reliability.

Laboratory Techniques

Scientific data that are used to analyze the characteristics of a chemical or physical change must be collected with care and patience. The data must be accurate; that is, it must be reproducible to within an “acceptable” margin of error. Reproducible data implies that the data collected from an observed chemical or physical change can be again collected at a later date by the same scientist or another scientist in another laboratory.

A scientist who has good laboratory skills and techniques generally collects good, reproducible data (called **quantitative data**). For that reason, careful attention as to the method (or methods) and procedures by which the data are collected is extremely important. This section of the laboratory manual describes a number of techniques that you will need to develop for collecting quantitative data in the chemistry laboratory. You do not need to know the details for all of the techniques at this time (that will come with each successive experiment that you encounter), but you should be aware of their importance, features, and location in the laboratory manual. Become *very* familiar with this section of the laboratory manual! Consult with your laboratory instructor about the completion of the Laboratory Assignment at the end of this section.

In the Experimental Procedure of each experiment, icons are placed in the margin at a position where the corresponding laboratory technique is to be applied for the collection of “better” data. The following index of icons identifies the laboratory techniques and page numbers on which they appear:



Technique 1. Inserting Glass Tubing through a Rubber Stopper *p. 13*



Technique 2. Cleaning Glassware *p. 13*



Technique 3. Handling Chemicals *p. 14*



Technique 4. Disposing of Chemicals *p. 15*



Technique 5. Preparing Solutions *p. 15*



Technique 6. Measuring Mass *p. 16*



Technique 7. Handling Small Volumes *p. 17*

A. Test Tubes for Small Volumes *p. 17*



B. Well Plates for Small Volumes *p. 17*

Technique 8. Collecting Gases p. 18



A. Water-Soluble Gases, Air Displacement p. 18



B. Water-Insoluble Gases, Water Displacement p. 19



Technique 9. Transferring Solids p. 19



Technique 10. Transferring Liquids and Solutions p. 20



Technique 11. Separating a Liquid or Solution from a Solid p. 21

A. Decanting a Liquid or Solution from a Solid p. 21



B. Preparing Filter Paper for a Filter Funnel p. 21



C. Gravity Filtration p. 21



D. Flushing a Precipitate from the Beaker p. 23



E. Vacuum Filtration p. 23



F. Centrifugation p. 24



Technique 12. Venting Gases p. 24



Technique 13. Heating Liquids and Solutions p. 25

A. Test Tubes over a "Cool" Flame p. 25



B. Erlenmeyer Flask over a "Cool" Flame p. 25



C. Beaker (or Flask) p. 26



D. A Hot Water Bath or Hot Plate p. 26



Technique 14. Evaporating Liquids p. 26

A. Nonflammable Liquids p. 27



B. Flammable Liquids p. 27



Technique 15. Heating Solids p. 27

A. Heating in a Drying Oven p. 27



B. Cooling in a Desiccator p. 27



C. Using a Crucible p. 28

1. Drying and/or Firing the Crucible
2. Igniting Contents in the Absence of Air
3. Igniting Contents for Combustion



Technique 16. Measuring Volume p. 29

A. Reading and Recording a Meniscus p. 29

1. Reading a Meniscus
2. Recording a Volume



B. Pipetting a Liquid p. 30

1. Preparation of the Pipet
2. Filling of the Pipet
3. Delivery of the Liquid
4. Cleanup



C. Titrating a Liquid (Solution) p. 30

1. Preparation of the Buret
2. Preparation of the Titrant
3. Operation of the Buret
4. Addition of Titrant to Receiving Flask
5. Cleanup



Technique 17. Quick Tests p. 33

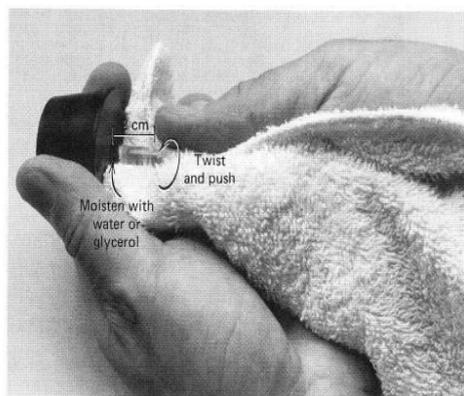
A. Testing for Odor p. 33



B. Testing for Acidity/Basicity p. 33

Caution: Perhaps more accidents occur in the general chemistry laboratory as a result of neglect in this simple operation than all other accidents combined. Please review and practice this technique correctly when working with glass tubing. Serious injury can occur to the hand if this technique is performed incorrectly.

Moisten the glass tubing and the hole in the rubber stopper with glycerol or water (**note:** glycerol works best). Place your hand on the tubing 2–3 cm (1 in.) from the stopper. Protect your hand with a cloth towel (Figure T.1). Simultaneously *twist* and *push* the tubing slowly and carefully through the hole. Wash off any excess glycerol on the glass or stopper with water and dry.



TECHNIQUE 1. INSERTING GLASS TUBING THROUGH A RUBBER STOPPER

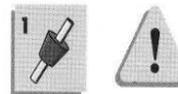


Figure T.1 Inserting glass tubing through a rubber stopper.

It's like eating from a dirty plate! A chemist is very concerned about contaminants causing errors in experimental data. Cleanliness is extremely important in minimizing errors in the precision and accuracy of data.

Clean all glassware with a soap or detergent solution using *tap* water. Use a laboratory sponge or a test tube, pipet, or buret brush as appropriate. Once the glassware is thoroughly cleaned, first rinse several times with tap water and then once or twice with small amounts of deionized water. Roll each rinse around the entire inner surface of the glass wall for a complete rinse. Discard each rinse through the delivery point of the vessel (i.e., buret tip, pipet tip, beaker spout). Deionized water should never be used for washing glassware; it is too expensive.

Invert the clean glassware on a paper towel or rubber mat to dry (Figure T.2a); do *not* wipe or blow-dry because of possible contamination. Do *not* dry heavy glassware (graduated cylinders, volumetric flasks, or bottles), or for that matter any glassware, over a direct flame.

The glassware is clean if, following the final rinse, no water droplets adhere to the clean part of the glassware (Figure T.2b).

TECHNIQUE 2. CLEANING GLASSWARE



A laboratory detergent.



Figure T.2a Invert clean glassware on a paper towel or rubber mat to air-dry.

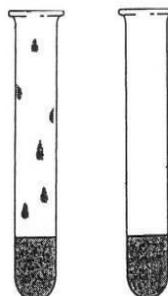


Figure T.2b Water droplets (left) do *not* adhere to the wall of clean glassware (right).

TECHNIQUE 3. HANDLING CHEMICALS

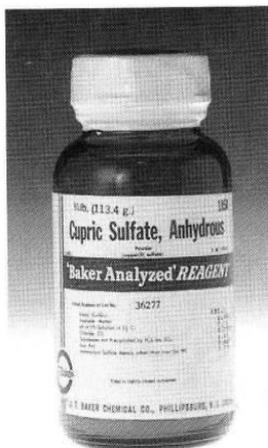


Figure T.3a Chemicals are labeled with systematic names.

Handling chemicals is as safe as handling your dog; if it is done carefully, no injuries will occur. Most techniques for handling chemicals are also regarded as safety guidelines.

- Read the label on a reagent bottle at least *twice* before removing any chemicals (Figure T.3a). The wrong chemical may lead to serious accidents or “unexplainable” results in your experiments (see Dry Labs 2 for an understanding of the rules of chemical nomenclature). Techniques 9 and 10 illustrate the correct procedures for transferring solids and liquid reagents.
- Avoid using excessive amounts of reagents. *Never* dispense more than the experiment calls for. *Do not return excess chemicals to the reagent bottle!*
- *Never* touch, taste, or smell chemicals unless specifically directed to do so. Skin, nasal, and/or eye irritations may result. If inadvertent contact with a chemical does occur, wash the affected area immediately with copious amounts of water and inform your laboratory instructor (see Laboratory Safety B.4, 5).

Chemicals are often labeled according to National Fire Protection Association (NFPA) standards which describe the four possible hazards of a chemical and a numerical rating from 0 to 4. The four hazards are health hazard (blue), fire hazard (red), reactivity (yellow), and specific hazard (white). A photograph of a label is shown in Figure T.3b.

If you wish to know more about the properties and hazards of the chemicals with which you will be working in the laboratory, safety information about the reagents is available in a bound collection of the Material Safety Data Sheets (MSDS). The MSDS collection is also accessible at various sites on the World Wide Web (see Laboratory Data, Part C).



In this manual, the international caution sign (shown at left) is used to identify a potential danger in the handling of a solid chemical or reagent solution or hazardous equipment.



Figure T.3b Hazardous materials classification system.

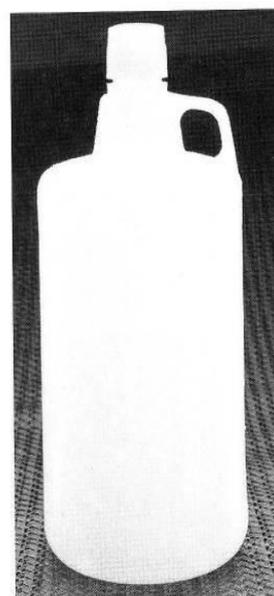


Figure T.4 Waste disposal containers are available in the laboratory.

Disposal of waste chemicals has become an increasingly important concern in recent years. Although most of the chemicals in this manual are considered "safe," any carelessness or abuse in their handling can be dangerous.

You must properly dispose of each test chemical. Assume that *nothing* (besides soap and water!) is to be discarded in the sink. Discard waste chemicals as directed in the Experimental Procedure of each experiment or by the laboratory instructor. There are no "general" waste disposal containers; each is labeled for a specific waste (Figure T.4). Instructions for the disposal of the various test chemicals are listed in the Experimental Procedure of each experiment.

Know the proper procedure for disposal before carelessly discarding your test solution. Double-check all labels before discarding. Improper waste disposal is foolish and potentially disastrous to you, your friends, your apparatus, and the laboratory!

A disposal icon is used throughout the manual to bring attention to the proper disposal of chemicals.

The final disposal of chemicals is the responsibility of the stockroom personnel. Information for the proper disposal of chemicals is available from the MSDS collection or at various sites on the World Wide Web (see Laboratory Data, Part C).

TECHNIQUE 4. DISPOSING OF CHEMICALS



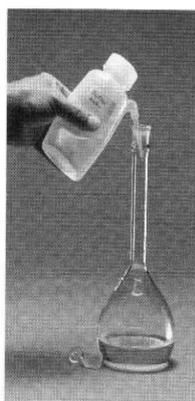
The preparation of an aqueous solution is often required for an experimental procedure. The preparation begins with either a solid reagent or a solution more concentrated than the one needed for the experiment. At either starting point, the minimum amount (the number of *moles*) of compound required for the experiment is calculated: from a solid, the mass and the molar mass of the compound are needed to calculate the number of moles of compound required for the preparation of the solution; from a more concentrated solution, the concentration and volume (or mass) of the concentrated solution must be known in order to calculate the number of moles of compound needed for the preparation of the aqueous solution. In both cases, the calculated (and then also measured) moles of compound are diluted to final volume. Knowledge of moles and mole calculations is absolutely necessary.

In the laboratory preparation, *never* insert a pipet, spatula, or dropping pipet into the reagent used for the solution preparation. Always transfer the calculated amount from the reagent bottle as described in Techniques 9 and 10.

Solutions are commonly prepared in volumetric flasks (Figure T.5) according to the following procedure:

- Place water (or the less concentrated solution) into the volumetric flask until it is one-third to one-half full.

TECHNIQUE 5. PREPARING SOLUTIONS



(a)



(b)



(c)



(d)

Figure T.5 Place water (or the less concentrated solution) into the flask before slowly adding the solid or more concentrated solution. Dilute the solution to the "mark" with water; stopper and invert the flask 10–15 times.

- 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



Tared mass: mass of sample without regard to its container



Weighing paper.

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, watch-glass, 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.

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

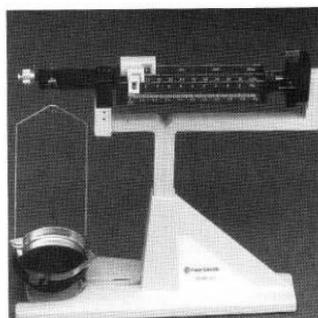


Figure T.6a Triple-beam balance, sensitivity of ± 0.01 g.



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

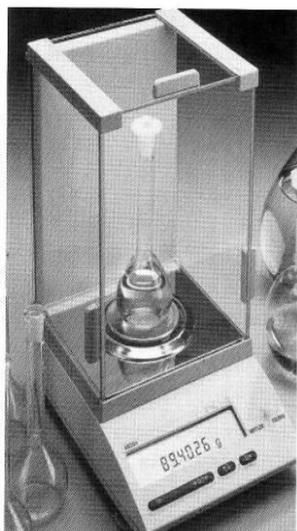


Figure T.6c Electronic analytical balance, sensitivity of ± 0.0001 g.



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

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.

TECHNIQUE 7. HANDLING SMALL VOLUMES

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

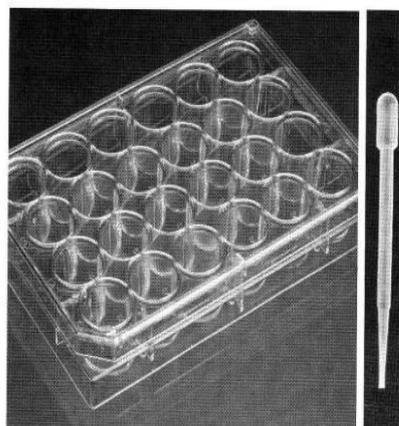
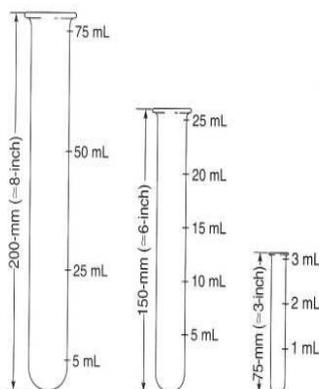


Figure T.7a The three common-sized test tubes for containing reagent solutions.

Figure T.7b A 24-well plate and Beral pipet are used for containing and transferring small quantities of reagent solutions.

from repeated or comparative reactions. The well plate most often recommended is the 24-well plate in which each well has an approximate volume of 3.5 mL (compared to a 3 mL for a small test tube).

For either technique the Beral pipet, a plastic, disposable pipet, or a dropping pipet is often used to transfer small volumes of solutions to and from the test tubes or well plate. The Beral pipet has a capacity of about 2 mL, and some have the volume graduation marks on the stem.

TECHNIQUE 8. COLLECTING GASES

The solubility and density of a gas determine the apparatus used for its collection. Water-soluble gases should not be collected over water, but rather by air displacement.

A. Water-Soluble Gases, Air Displacement



Water-soluble gases *more* dense than air are collected by air displacement (Figure T.8a). The more dense gas pushes the less dense air up and out of the gas bottle. Water-soluble gases *less* dense than air are also collected by air displacement (Figure

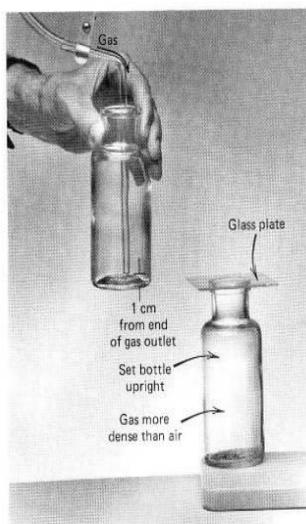


Figure T.8a Collection of water-soluble gases more dense than air.

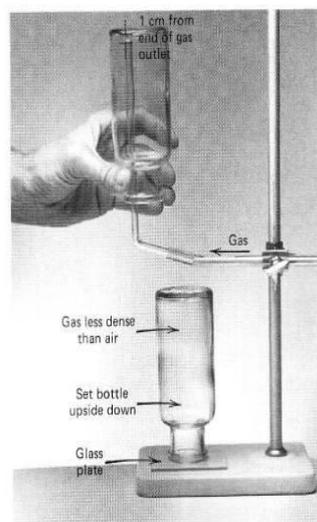


Figure T.8b Collection of water-soluble gases less dense than air.

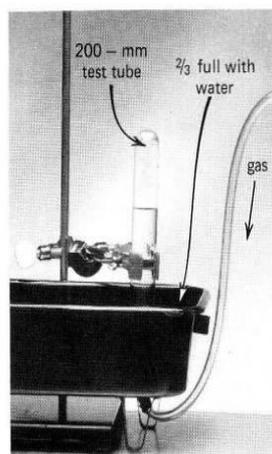
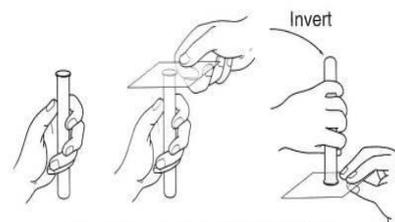


Figure T.8c Collection of water-insoluble gas by the displacement of water.



Slide glass plate over top of test tube.
Do not allow an air bubble
to enter the test tube.

Figure T.8d Inverting a water-filled test tube.

T.8b) except that, in this case, the less dense gas pushes the more dense air down and out of the gas bottle. Note that the gas outlet tube should extend to within 1 cm of the bottom (or top) of the gas bottle.

Gases that are relatively insoluble in water are collected by water displacement. The gas pushes the water down and out of the water-filled gas-collecting vessel (Figure T.8c). The gas-collecting vessel (generally a flask or test tube) is first filled with water, covered with a glass plate or plastic wrap (no air bubbles must enter the vessel, Figure T.8d), and then inverted into a deep pan or tray half-filled with water. The glass plate or plastic wrap is removed, and the tubing from the gas generator is inserted into the mouth of the gas-collecting vessel.

B. Water-Insoluble Gases, Water Displacement



Before transferring a solid chemical for use in the preparation of a solution or for study of its chemical properties, read the label on the bottle *twice* to be sure it is the correct chemical. For example, is the chemical iron(II) acetate or iron(III) acetate? Is it the anhydrous, trihydrate, or pentahydrate form of copper(II) sulfate?

If the reagent bottle has a hollow glass stopper or if it has a screw cap, place the stopper (or cap) top side down on the bench (Figure T.9a). To dispense solid from the bottle, hold the label against your hand, tilt, and roll the solid reagent back and forth.

TECHNIQUE 9. TRANSFERRING SOLIDS



- For *larger quantities* of solid reagent, dispense the solid into a beaker (Figure T.9b) until the estimated amount has been transferred. Try not to dispense any more reagent than is necessary for the experiment. Do not return any excess reagent to the reagent bottle—share the excess with another chemist.
- For *smaller quantities* of solid reagent, first dispense the solid into the inverted hollow glass stopper or screw cap. And then transfer the estimated amount of

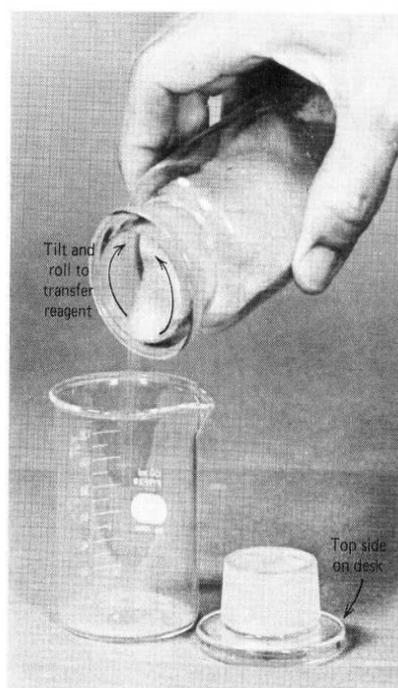


Figure T.9a Transferring a solid chemical from a glass ground reagent bottle. Place the glass stopper top side down.



Figure T.9b Tilt and roll the reagent bottle back and forth until the desired amount of solid chemical has been dispensed.

reagent needed for the experiment from the stopper/screw cap to an appropriate vessel. Return the excess reagent in the glass stopper or screw cap to the reagent bottle—in effect, the solid reagent has never left the reagent bottle.

For either situation, *never* use a spatula or any other object to break up or transfer the reagent to the appropriate container unless your laboratory instructor specifically instructs you to do so.

When you have finished dispensing the solid chemical, *recap* the reagent bottle.

TECHNIQUE 10.
TRANSFERRING
LIQUIDS AND
SOLUTIONS



When a liquid or solution is to be transferred from a reagent bottle, remove the glass stopper and hold it between the fingers of the hand used to grasp the reagent bottle (Figures T.10a, b). Never lay the glass stopper on the laboratory bench; impurities may be picked up and thus contaminate the liquid when the stopper is returned.

To transfer a liquid from one vessel to another, hold a stirring rod against the lip of the vessel containing the liquid and pour the liquid down the stirring rod, which, in

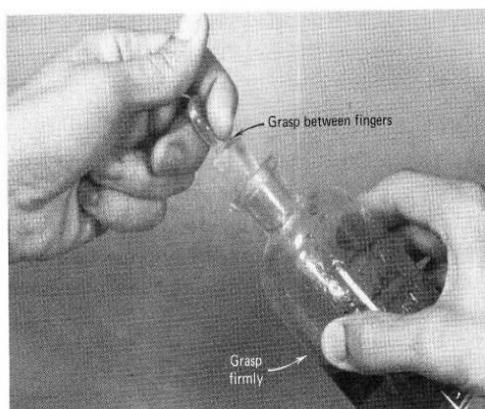


Figure T.10a Remove the glass stopper and hold it between the fingers of the hand that grasps the reagent bottle.

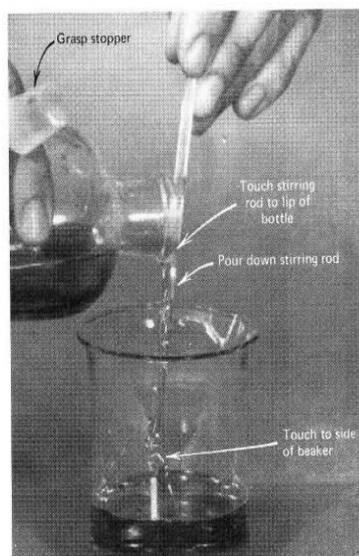


Figure T.10b Transfer the liquid from the reagent bottle with the aid of the stirring rod.

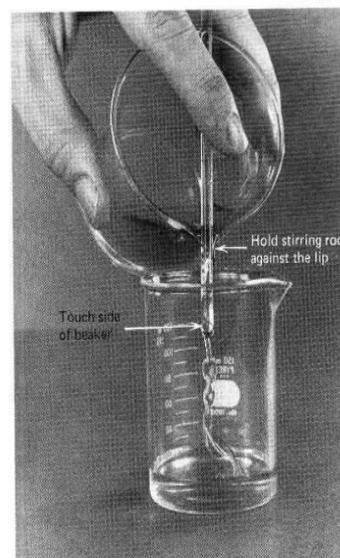


Figure T.10c The stirring rod should touch the lip of the transfer vessel and the inner wall of the receiving vessel.

turn, should touch the inner wall of the receiving vessel (Figures T.10b, c). Return the glass stopper to the reagent bottle.

Do *not* transfer more liquid than is needed for the experiment; do *not* return any excess or unused liquid to the original reagent bottle.

A liquid can be decanted (poured off the top) from a solid if the solid clearly separates from the liquid in a reasonably short period of time. Allow the solid to settle to the bottom of the vessel (Figure T.11a). Transfer the liquid (called the **supernatant**) with the aid of a clean stirring rod (Figure T.11b). Do this slowly so as not to disturb the solid. Review Technique 10 for the transfer of a liquid from one vessel to another.

TECHNIQUE 11. SEPARATING A LIQUID OR SOLUTION FROM A SOLID

A. Decanting a Liquid or Solution from a Solid

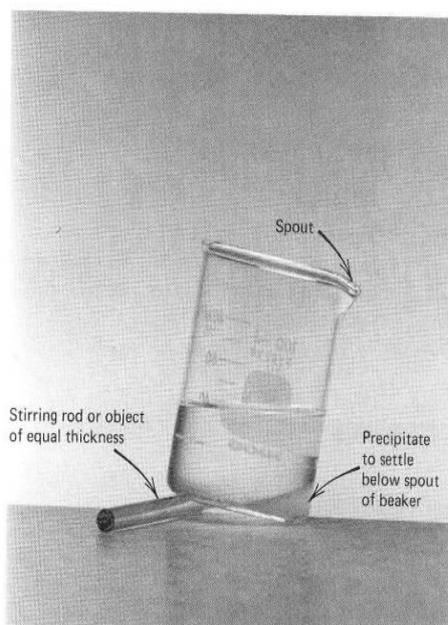


Figure T.11a Tilt the beaker to allow the precipitate to settle at the side. Use a stirring rod or a similar object to tilt the beaker.

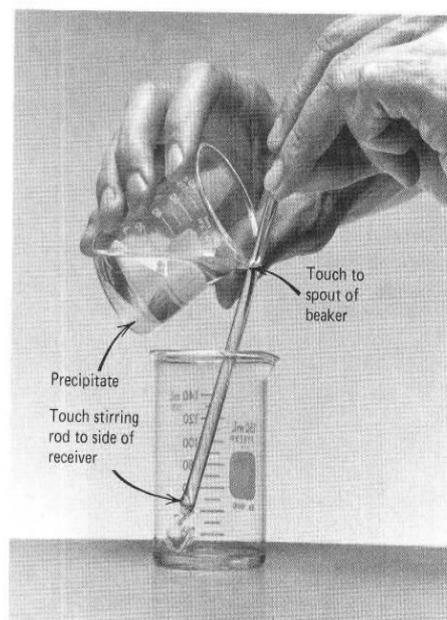


Figure T.11b Transfer the supernatant to a receiving vessel with the aid of a stirring rod.

If a solid is to be separated from the liquid using a filtering process, then the filter paper must be properly prepared. For a gravity filtration procedure, first fold the filter paper in half (Figure T.11c), again fold the filter paper to within about 10° of a 90° fold, tear off the corner of the outer fold unequally, and open. The tear enables a close seal to be made across the paper's folded portion when placed in a funnel.

Place the folded filter paper snugly into the funnel. Moisten the filter paper with the solvent of the liquid/solid mixture being filtered (most likely this will be deionized water) and press the filter paper against the top wall of the funnel to form a seal. Support the funnel with a clamp or in a funnel rack.

B. Preparing Filter Paper for a Filter Funnel



Transfer the liquid as described in Technique 10 (Figure T.11d). The tip of the funnel should touch the wall of the receiving beaker to reduce any splashing of the **filtrate**. Fill the bowl of the funnel until it is less than two-thirds full with the mixture. Always keep the funnel stem full with the filtrate; the weight of the filtrate in the funnel stem creates a slight suction on the filter in the funnel, and this hastens the filtration process.

C. Gravity Filtration



Filtrate: the solution that passes through the filter in a filtration procedure

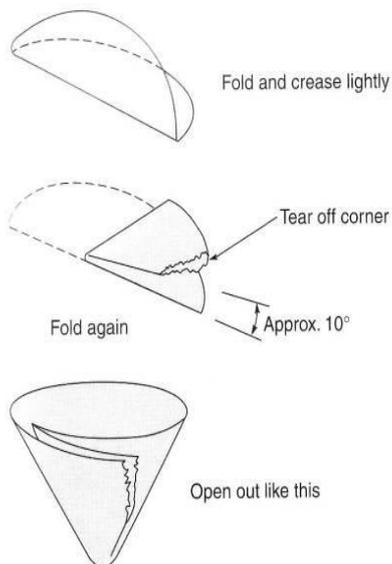


Figure T.11c The sequence of folding filter paper for a filter funnel in a gravity filtration procedure.

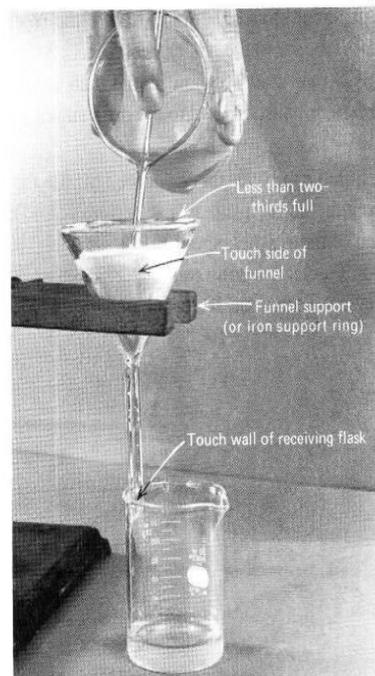


Figure T.11d The tip of the funnel should touch the wall of the receiving flask, and the bowl of the funnel should be one-half to two-thirds full.

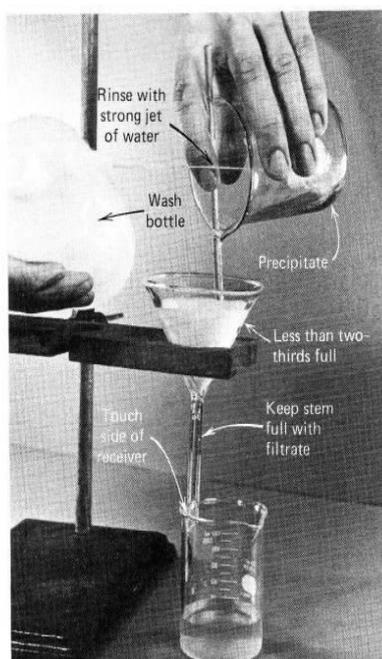


Figure T.11e Flushing the precipitate from a beaker with the aid of a "wash" bottle.

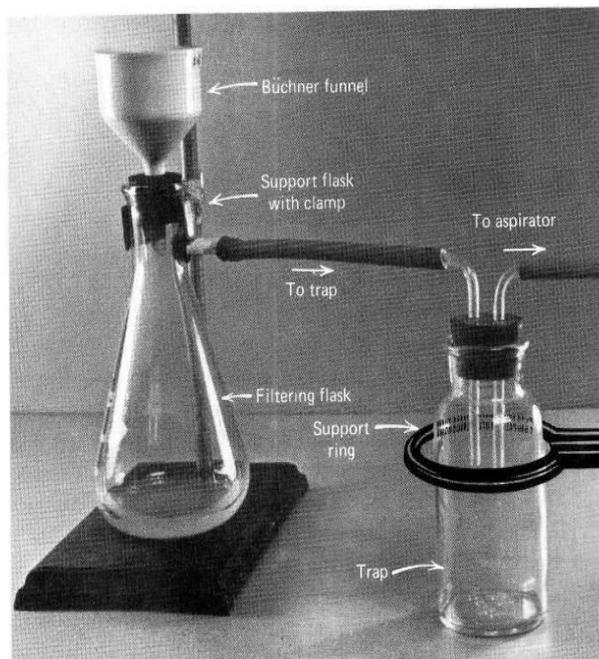


Figure T.11f The aspirator should be fully open during the vacuum filtering operation.

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.11e) 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.

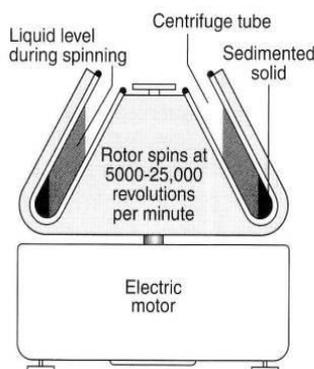
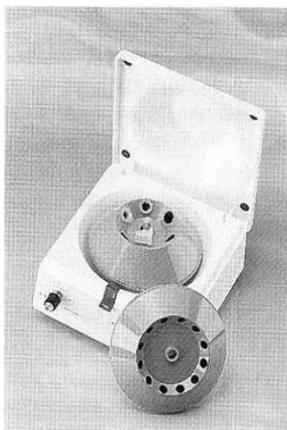


Figure T.11g A laboratory centrifuge forces the precipitate to the bottom of the centrifuge tube.

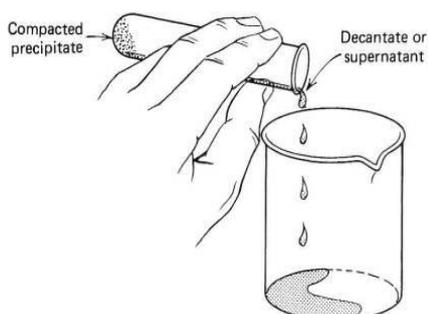


Figure T.11h Decant the supernatant from the compacted precipitate.

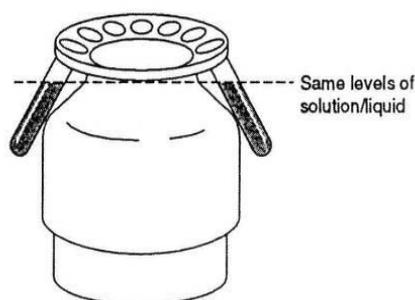


Figure T.11i Balance the centrifuge by placing tubes with equal volumes of liquid opposite each other inside the metal sleeves of the rotor.

F. Centrifugation



Supernatant: the clear liquid covering a precipitate

A centrifuge (Figure T.11g) spins at velocities of 5000 to 25,000 revolutions per minute! A solid/liquid mixture in a small test tube or centrifuge tube is placed into a sleeve of the rotor of the centrifuge. By centrifugal force the solid is forced to the bottom of the test tube or centrifuge tube and compacted. The clear liquid, called the **supernatant**, is then easily decanted without any loss of solid (Figure T.11h). This quick separation of liquid from solid requires 20–40 seconds.

Observe these precautions in operating a centrifuge:

- Never fill the centrifuge tubes to a height more than 1 cm from the top.
- Label the centrifuge tubes to avoid confusion of samples.
- *Always* operate the centrifuge with an *even* number of centrifuge tubes containing equal volumes of liquid placed opposite one another in the centrifuge. This *balances* the centrifuge and eliminates excessive vibration and wear. If only one tube needs to be centrifuged, balance the centrifuge with a tube containing the same volume of solvent (Figure T.11i).
- *Never* attempt to manually stop a centrifuge. When the centrifuge is turned off, let the rotor come to rest on its own.

TECHNIQUE 12. VENTING GASES



Removing “undesirable” gases from a chemical reaction should be accomplished in a fume hood (Figure T.12a). Locate the fume hood in your laboratory.

On occasion the space in the fume hoods is not adequate for an entire class to perform the experiment in a timely manner. With the *approval of your laboratory instructor*, an improvised hood (Figure T.12b) can be assembled. For the operation of an improvised hood, a water aspirator draws the gaseous product from above the reaction vessel—the gas dissolves in the water. To operate the “hood,” completely open the faucet that is connected to the aspirator in order to provide the best suction for the removal of the gases. But, as a reminder, *never* substitute an improvised hood for a fume hood if space is available in the fume hood.



Figure T.12a A modern laboratory fume hood.

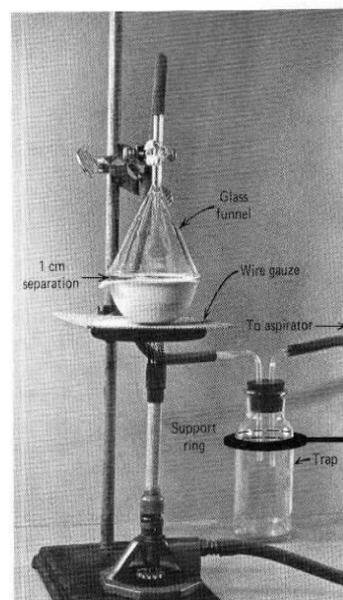


Figure T.12b Position a funnel, connected to a water aspirator, over the escaping gases.

Liquids and solutions are often heated, for example, to promote the rate of a chemical reaction to or 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

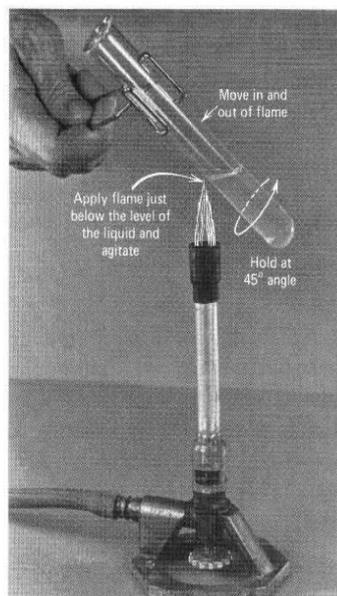


Figure T.13a Move the test tube circularly in and out of the cool flame, heating the liquid or solution from top to bottom.

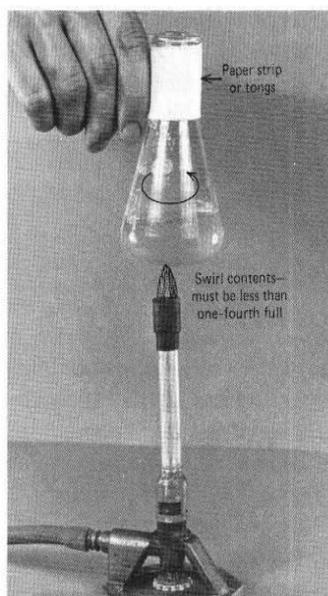


Figure T.13b Swirl the flask, less than one-fourth full, in and out of the cool flame.

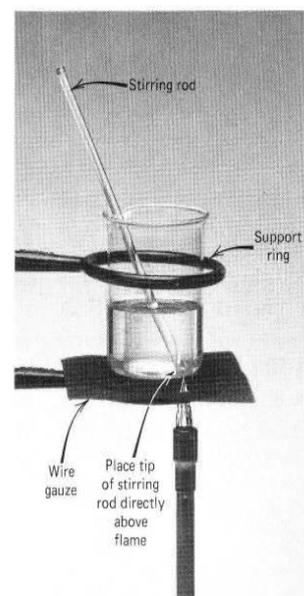


Figure T.13c Place the flame directly beneath the tip of the stirring rod in the beaker. Boiling chips may also be placed in the beaker to avoid “bumping.”

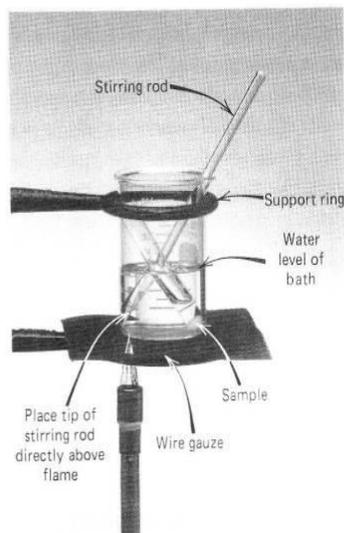


Figure T.13d A hot water bath may be used to maintain solution in test tubes at a constant, elevated temperature for an extended time period.

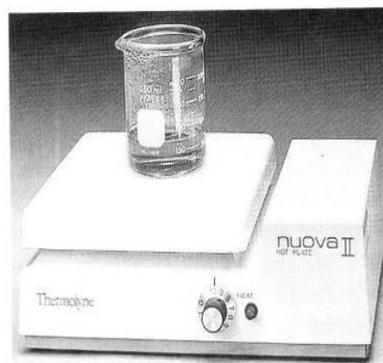


Figure T.13e A hot plate may be used to maintain solutions in a beaker or flask at a constant, elevated temperature for an extended time period.

C. Beaker (or Flask)



Nonflammable liquids in beakers or flasks that are more than one-fourth full can be heated directly using either a direct flame or a hot plate, provided a safe apparatus is assembled. If a flame is to be used, support the beaker (or flask) on a wire gauze that is centered over an iron ring; for a hot plate, merely center the beaker (or flask) on the surface.



Boiling chips.

Place a support (iron) ring around the top of the beaker (or flask) to prevent it from being accidentally knocked off. To avoid the problem of bumping (the sudden formation of bubbles from the superheated liquid), place a glass stirring rod and/or several **boiling chips** in the beaker. For heating with a flame, position the flame directly beneath the tip of the stirring rod (Figure T.13c). For either method of heating, heat slowly—occasional agitation with a stirring rod allows for more uniform heating of the liquid and minimizes bumping.

*Boiling chips (also called **boiling stones**): small, porous ceramic pieces—when heated, the air contained within the porous structure is released, gently agitating the liquid and minimizing bumping. Boiling chips also provide nucleation sites on which bubbles can form.*

D. A Hot Water Bath or Hot Plate



Small quantities of liquids in test tubes that need to be maintained at a constant, elevated temperature over a period of time can be placed in a hot water bath (Figure T.13d). The heat source may be a hot plate or direct flame, depending on the chemicals being used. The setup is the same as that for heating a liquid in a beaker (Technique 13C).

If the liquid is in a beaker or Erlenmeyer flask instead of a test tube, place the beaker or flask on a hot plate and *slowly* heat the liquid (Figure T.13e).

TECHNIQUE 14. EVAPORATING LIQUIDS

To remove a liquid from a vessel by evaporation, the flammability of the liquid must be considered. This is a safety precaution.

Use a fume hood or an improvised hood (Technique 12) as recommended to remove irritating or toxic vapors.

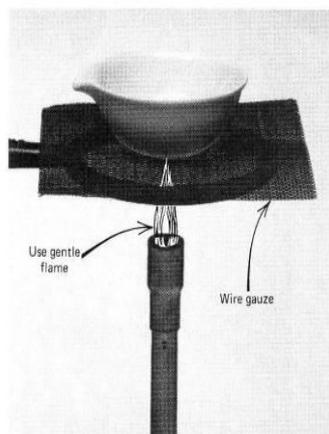


Figure T.14a Evaporation of a nonflammable liquid over a low, direct flame. A hot plate may be substituted for the flame.

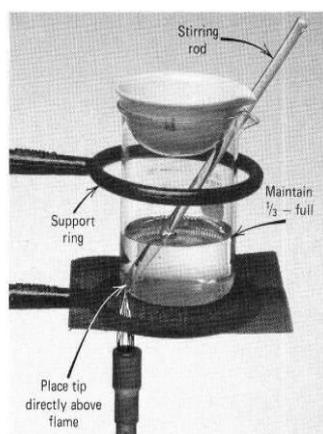


Figure T.14b Evaporation of a nonflammable liquid over a steam bath. A hot plate may be substituted for the flame.

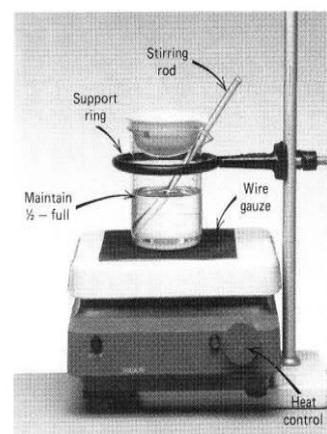


Figure T.14c Evaporation of a flammable liquid over a steam bath using a hot plate for the heat source.

A nonflammable liquid can be evaporated either with a direct flame (Figure T.14a) or over a steam bath (Figure T.14b). If a direct flame is used, place the liquid in an evaporating dish centered on a wire gauze and iron ring. Use a gentle, “cool” flame to slowly evaporate the liquid.

If a steam bath is used, place the liquid in an evaporating dish on top of a beaker set up according to Figure T.14b. Gentle boiling of the water in the beaker is more efficient than rapid boiling for evaporating the liquid. Avoid breathing any vapors.

No open flames should be near flammable liquids. Substitute a hot plate for the open flame in Figure T.14b (Figure T.14c). The use of a fume hood or an improvised hood (Technique 12) is suggested if large amounts are evaporated in a laboratory with inadequate ventilation. Consult your laboratory instructor.

Solids are heated to dry them or to test their thermal stability. A drying oven is often used for low temperature heating, and porcelain crucibles are used for high temperature heating. Beakers and test tubes can be used for moderately high temperature heating.

When solid chemicals are left exposed to the atmosphere they often absorb moisture. If an exact mass of a solid chemical is required for a solution preparation or for a reaction, the absorbed water must be removed before the mass measurement is made on the balance. The chemical is often placed in an open container (usually a Petri dish or beaker) in a drying oven (Figure T.15a) set at a temperature well above room temperature (most often at $\sim 110^{\circ}\text{C}$) for several hours to remove the adsorbed water. The container is then removed from the drying oven and placed in a desiccator (Technique 15B) for cooling to room temperature. **Caution:** *Hot glass and cold glass look the same—the container from the drying oven is hot and should be handled accordingly.* See your laboratory instructor.

When crucibles, crucible lids, and solid chemicals are cooled in the laboratory, moisture tends to condense on the outer surface, adding to the total mass. To minimize this mass error, and for quantitative work, substances and mixtures that may tend to be hygroscopic are placed into a desiccator (Figure T.15b) until they have reached ambient temperature.

A. Nonflammable Liquids



B. Flammable Liquids



TECHNIQUE 15. HEATING SOLIDS

A. Heating in a Drying Oven



B. Cooling in a Desiccator





Figure T.15a A modern laboratory drying oven.

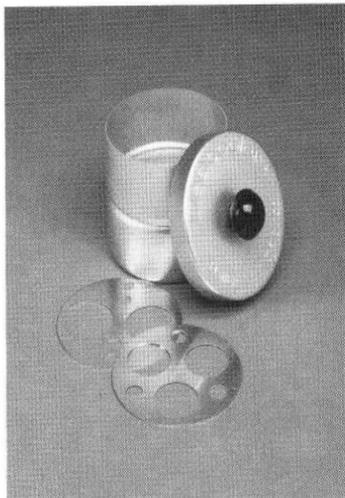


Figure T.15b A simple laboratory desiccator (left) or a glass desiccator (right) contains a desiccant (usually anhydrous CaCl_2) to provide a dry atmosphere.

A desiccator is a laboratory apparatus that provides a dry atmosphere. A desiccant, typically anhydrous calcium chloride, absorbs the water vapor from within the enclosure of the desiccator. The anhydrous calcium chloride forms $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$; the hydrated water molecules can be easily removed with heat, and the calcium chloride can be recycled for subsequent use in the desiccator.

C. Using a Crucible



For high temperature combustion or decomposition of a chemical, porcelain crucibles are commonly used. To avoid contamination of the solid sample, thoroughly clean the crucible (so it is void of volatile impurities) prior to use. Often-used crucibles tend to form stress fractures or fissures. Check the crucible for flaws; if any are found return the crucible to the stockroom and check out and examine a second crucible.



- Drying and/or Firing the Crucible.** Support the crucible and cover on a clay triangle (Figure T.15c) and heat in a hot flame until the bottom of the crucible glows a dull red. Rotate the crucible with crucible tongs to ensure complete “firing” of the crucible, i.e., the combustion and volatilization of any impurities in the crucible. Allow the crucible and cover to cool to room temperature while on the clay triangle or after several minutes in a desiccator (Technique 15B). If the crucible still contains detectable impurities, add 1–2 mL of 6 M HNO_3 (**caution: avoid skin contact, flush immediately with water**) and evaporate *slowly* to dryness in the fume hood.
- Igniting Contents in the Absence of Air.** To heat a solid sample to a high temperature but *not* allow it to react with the oxygen of the air, set the crucible upright in the clay triangle with the cover in place (Figure T.15d). Use the crucible tongs to adjust the cover.
- Igniting Contents for Combustion.** To heat a solid sample to a high temperature and allow it to react with the oxygen of the air, slightly tilt the crucible on the clay triangle and adjust the cover so that about two-thirds of the crucible remains covered (Figure T.15e). Use the crucible tongs to adjust the cover.

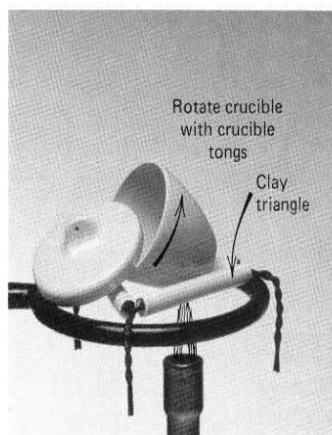


Figure T.15c Drying and/or firing a crucible and cover.

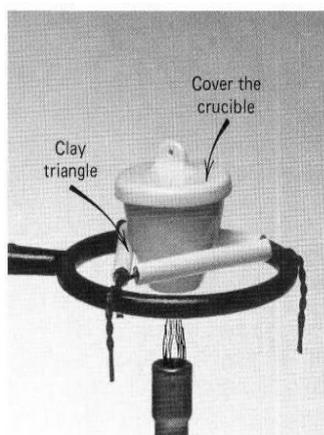


Figure T.15d Ignition of a solid sample in the absence of air.

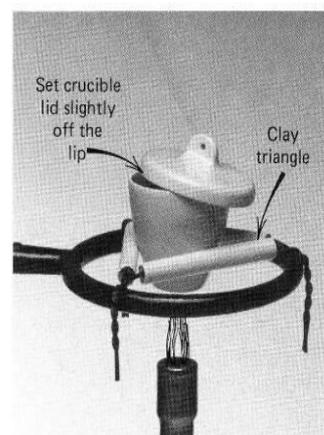


Figure T.15e Ignition of a solid sample in the presence of air for complete combustion.

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

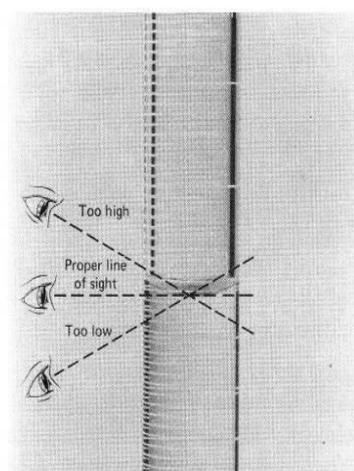


Figure T.16a Read the volume of a liquid with the eye horizontal to the bottom of the meniscus.

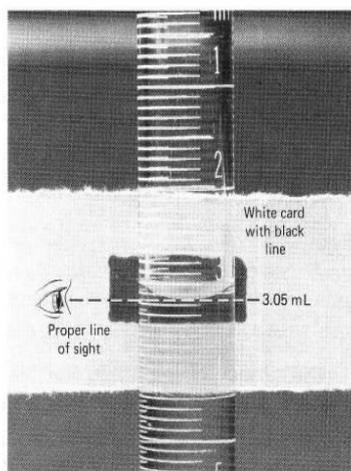


Figure T.16b Use a black line drawn on a white card to assist in pinpointing the location of the bottom of the meniscus.

Volumetric glassware: glassware that has a calibration mark(s) that indicates a calibrated volume, as determined by the manufacturer

2. **Recording a Volume.** Record the volume of a liquid in **volumetric glassware** using all certain digits (from the labeled calibration marks on the glassware) *plus* one uncertain digit (the last digit which is the best estimate between the calibration marks). In Figure T.16b, the volume of solution in the buret is between the calibration marks of 3.0 and 3.1; the “3” and the “0” are certain, the “5” is the estimate between 3.0 and 3.1. The reading is 3.05 mL. Be aware that all volume readings do *not* end in “5!”

This guideline for reading volumes also applies to reading and recording temperatures on a thermometer.

B. Pipetting a Liquid



Dust/lint-free tissue.

The most common type of pipet in the laboratory is labeled TD at 20°C. A pipet labeled TD at 20°C (to deliver at 20°C) means that the volume of the pipet is calibrated according to the volume it delivers from gravity flow only. The liquid retained in the pipet tip is considered in the calibration of the pipet and therefore is *not* to be blown out.

A clean pipet in conjunction with the proper technique for dispensing a liquid from a pipet are important in any quantitative determination.

1. **Preparation of the Pipet.** See Technique 2 for cleaning glassware. A clean pipet should have no water droplets adhering to its inner wall. Inspect the pipet to ensure it is free of chips or cracks. Transfer the liquid that you intend to pipet from the reagent bottle into a clean, dry beaker; do *not* insert the pipet tip directly into the reagent bottle. Dry the outside of the pipet tip with a clean, dust-free towel or tissue (e.g., Kimwipe). Using the suction from a collapsed rubber (pipet) bulb, draw several 2- to 3-mL portions into the pipet as a rinse. Roll each rinse around in the pipet to make certain that the liquid washes the entire surface of the inner wall. Deliver each rinse through the pipet tip into a waste beaker. Discard the rinse as directed in the experiment.
2. **Filling of the Pipet.** Place the pipet tip well below the surface of the liquid in the beaker. Using the collapsed pipet bulb (or a pipet pump—*never* use your mouth!), draw the liquid into the pipet until the level is 2–3 cm above the “mark” on the pipet (Figure T.16c). Remove the bulb and quickly cover the top of the pipet with your index finger (*not* your thumb!). Remove the tip from the liquid and wipe off the pipet tip with a clean, dust-free towel or tissue. Holding the pipet in a vertical position over a waste beaker, control the delivery of the excess liquid until the level is “at the mark” in the pipet. Practice (Figure T.16d)! Read the meniscus correctly. Remove any drops suspended from the pipet tip by touching it to the wall of the waste beaker.
3. **Delivery of the Liquid.** Deliver the liquid to the receiving vessel (Figure T.16e) by releasing the index finger from the top of the pipet. The pipet tip should touch the wall of the receiving vessel to avoid splashing. Do *not* blow or shake out the last bit of liquid that remains in the tip; this liquid has been included in the calibration of the pipet . . . remember this is a TD at 20°C pipet!
4. **Cleanup.** Once it is no longer needed in the experiment, rinse the pipet with several portions of deionized water and drain each rinse through the tip.



C. Titrating a Liquid (Solution)



Titratant: the solution in the buret

A clean buret in conjunction with the proper technique for measuring and dispensing a liquid from a buret is important in any quantitative analysis determination.

1. **Preparation of the Buret.** See Technique 2 for cleaning glassware. If a buret brush is needed, be careful to avoid scratching the buret wall with the wire handle. Once the buret is judged to be “clean,” close the stopcock. Rinse the buret with several 3- to 5-mL portions of water and then **titrant**. Tilt and roll the barrel of the buret so that the rinse comes into contact with the entire inner wall. Drain each rinse through the buret tip into the waste beaker. Dispose of the rinse as advised in the experiment. Support the buret with a buret clamp (Figure T.16f).



Figure T.16c Draw the liquid into the pipet with the aid of a rubber pipet bulb (*not* the mouth!).

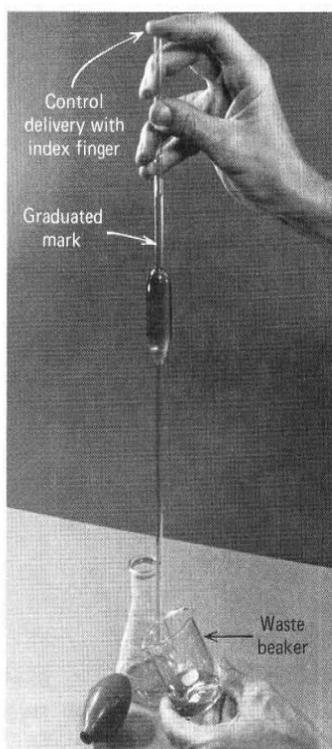


Figure T.16d Control the delivery of the liquid from the pipet with the forefinger (*not* the thumb!).

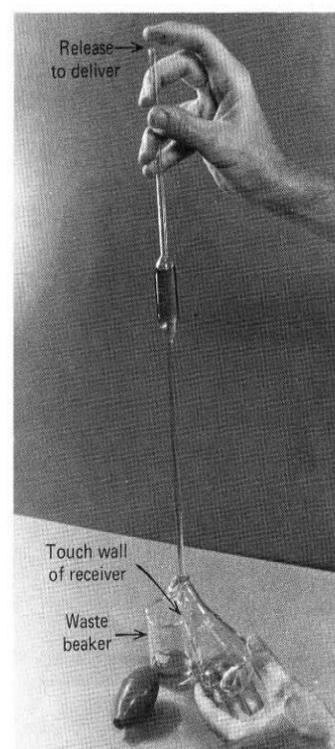


Figure T.16e Deliver the liquid from the pipet with the tip touching the wall of the receiving flask.

2. **Preparation of the Titrant.** Close the stopcock. With the aid of a *clean* funnel, fill the buret with the titrant to just above the zero mark. Open the stopcock briefly to release any air bubbles in the tip *and* allow the meniscus of the titrant to go below the uppermost graduation on the buret. Allow 10–15 seconds for the titrant to drain from the wall; **record the volume** (Technique 16A) of titrant in the buret. Note that the graduations on a buret *increase* in value from the top down (Figure T.16g).
3. **Operation of the Buret.** During the addition of the titrant from the buret, operate the stopcock with your left hand (if right-handed) and swirl the Erlenmeyer flask with your right hand (Figure T.16h). This prevents the stopcock from sliding out of its barrel and allows you to maintain a normal, constant swirling motion of the reaction mixture in the receiving flask as the titrant is added. The opposite procedure, of course, is applicable if you are left-handed (Figure T.16i). Fill the buret after each analysis. Use an Erlenmeyer flask as a receiving flask, rather than a beaker, unless the solution is to be stirred with magnetic stirrer.
4. **Addition of Titrant to Receiving Flask.** Have a white background (a piece of white paper) beneath the receiving flask, generally an Erlenmeyer flask, so that you can better see the endpoint of a titration (the point at which the indicator turns color). If the endpoint is a change from colorless to white, a black background is preferred. Add the titrant to the Erlenmeyer flask as described above; periodically stop its addition and wash the wall of the flask with the solvent (generally deionized water) from a wash bottle (Figure T.16j). Near the endpoint (slower color fade of the indicator, Figure T.16k), slow the rate of titrant addition until a drop (or less) makes the color change of the indicator persist for 30 seconds. **Stop,**

Record the volume: read the volume in the buret using all certain digits (from the labeled calibration marks on the buret) plus one uncertain digit (the last digit which is the best estimate between the calibration marks)

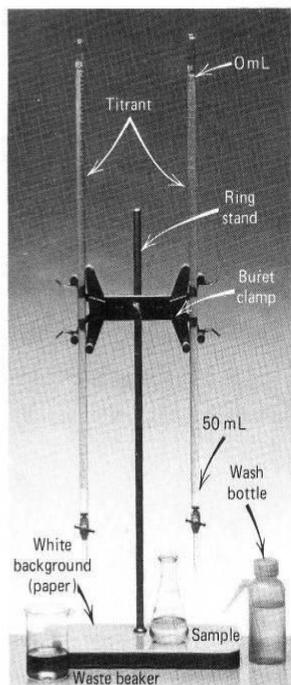


Figure T.16f Setup for a titration analysis.

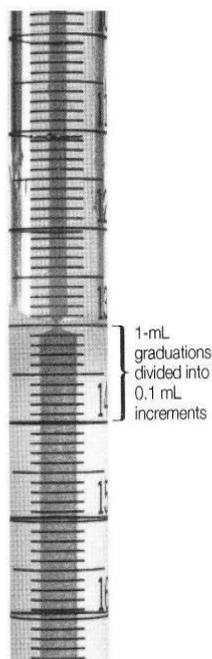


Figure T.16g A 50-mL buret is marked from top to bottom, 0 to 50 mL, with 1-mL graduations divided into 0.1-mL increments.

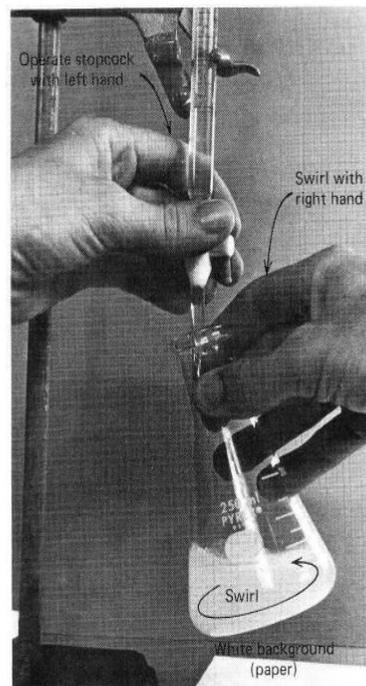


Figure T.16h Titration technique for right-handers.

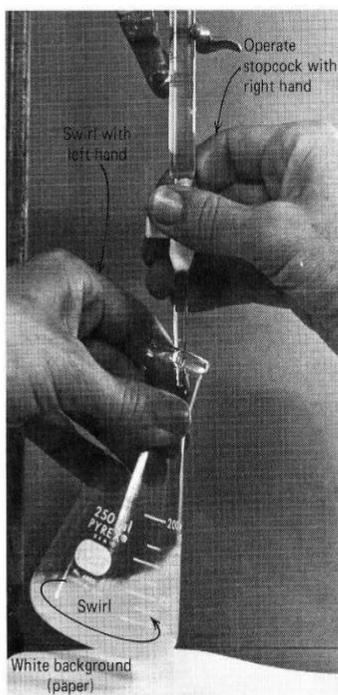


Figure T.16i Titration technique for left-handers.

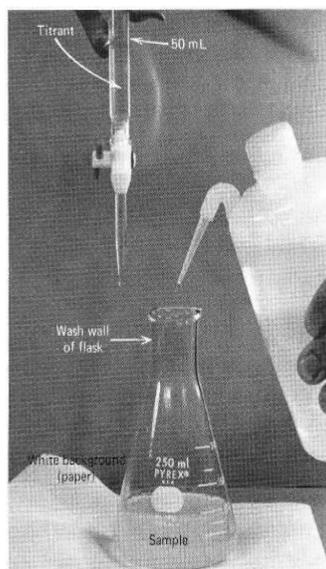


Figure T.16j Place a white background beneath the receiving flask and wash the wall of the receiving flask periodically during the titration.

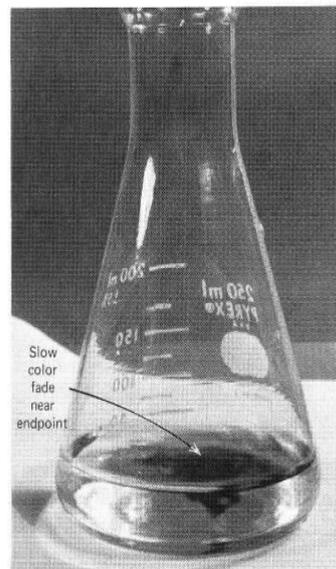


Figure T.16k A slow color fade of the indicator occurs near the endpoint in the titration.

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.

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

B. Testing for Acidity/Basicity

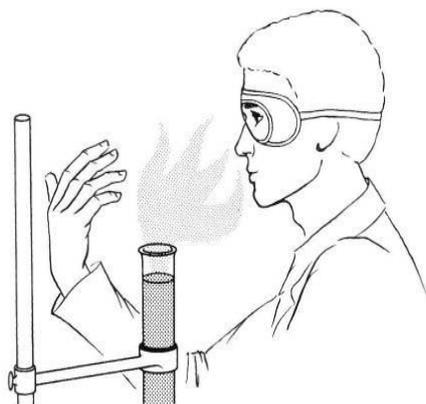


Figure T.17a Fan the vapors gently toward the nose.

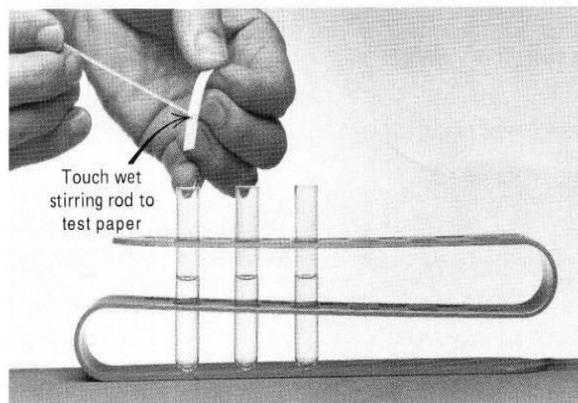


Figure T.17b Test for acidity/basicity.



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

Disclaimer: The material contained in the Laboratory Safety and Laboratory Techniques sections of this manual has been compiled from sources believed to be reliable and to represent the best opinions of safety in the laboratory. This manual is intended to provide basic guidelines for safe practices in the undergraduate chemistry laboratory. It cannot be assumed that all necessary warning and precautionary measures are contained in this manual, or that other or additional information or measures may not be required.

Further discussions of these and other laboratory techniques can be found on the World Wide Web. Refer to Laboratory Data, Part C.

NOTES ON LABORATORY TECHNIQUES

Laboratory Techniques

Date _____ Lab Sec. _____ Name _____ Desk No. _____

Complete with the correct word(s), phrase, or value(s).

Ask your instructor to identify the questions you are to complete.

1. Technique 1. To insert glass tubing through a rubber stopper, first moisten the glass with _____ or _____.
2. Technique 2. Glassware should first be washed with _____ water and a detergent solution followed by final rinses with _____ water.
3. Technique 2. Glassware is clean when _____.
4. Technique 4. Information on the properties and disposal of chemicals can be found in the stockroom or online from the _____ collection.
5. Technique 5. Most solutions used for quantitative work are prepared in flasks called _____ flasks.
6. Technique 6. The mass of a sample measured on a balance without regard to its container is called its _____ mass.
7. Technique 6. After completing a mass measurement, the mass settings are to be set to _____.
8. Technique 7. A well of a 24-well plate has the same approximate volume that is slightly larger than a _____ mm test tube.
9. Technique 8. To collect a water-soluble gas _____ dense than air, the mouth of the gas-collecting flask should be pointed downward.
10. Technique 9. Do not use a _____ to transfer a solid from its reagent bottle.
11. Technique 10. Transfer liquids or solutions from the reagent bottle to a beaker with the aid of a _____.
12. Technique 11c. The bowl of the funnel should be less than _____ full when gravity filtering a mixture.
13. Technique 11f. A centrifuge should be balanced with _____ numbers of test tubes containing _____ volumes of solution.
14. Technique 12. If a noxious or nauseating gas is evolved from a reaction mixture, it is good advice to perform the reaction _____ or _____.
15. Technique 13a. A nonluminous flame with a reduced supply of fuel is called a _____. Such a flame is most critical when heating liquids and solutions contained in a _____.
16. Technique 13c. A nonflammable liquid in a flask or beaker that is *greater than* one-fourth full, can be heated using the laboratory setup shown in Figure _____.
17. Technique 13d. Solutions in test tubes can be maintained at a constant "higher" temperature with the use of a _____.
18. Technique 15a, b. Solids are commonly heated to dryness in a drying oven and then cooled (ideally) in a _____.
19. Technique 16a. The volume of a liquid should be read at the _____ of the meniscus.
20. Technique 16b. The volume of a liquid in a pipet should be controlled with the _____ finger.
21. Technique 16c. A buret should be rinsed with several 3- to 5-mL portions of _____ before being filled.

22. Technique 16c. During the titration procedure, the stopcock of the buret should be controlled with the _____ hand for those chemists who are left handed.
23. Technique 16c. The color change of the indicator at the endpoint should persist for _____ seconds.
24. Technique 17. The acidity or basicity of a solution is easily and quickly tested with _____ or _____.
25. All techniques. Technique advisories appear in the margin of Experiment Procedure for each experiment by the presence of an appropriate _____.

True or False

Ask your instructor to identify the questions you are to complete.

- _____ 1. Dry all clean glassware with a clean towel.
- _____ 2. While cleaning glassware, discard all washes and rinses from the delivery point of the glass vessel.
- _____ 3. To avoid waste in the use of chemicals, always return the unused portion directly to the original reagent bottle.
- _____ 4. Never touch, taste, or smell a chemical unless specifically told to do so.
- _____ 5. Most all chemicals used in experiments can be discarded into the sink.
- _____ 6. The mass of a dry solid can be measured directly on the balance pan.
- _____ 7. If a 3-inch test tube has a volume of 3 mL, then an 8-inch test tube must have a volume of 8 mL.
- _____ 8. To transfer a solution, a stirring rod touches the delivery point of the reagent vessel and the wall of the receiving vessel.
- _____ 9. Only 1 mL of a liquid mixture should be present in a centrifuge tube when placed in the centrifuge.
- _____ 10. A test tube should be less than one-third full when heating with a direct flame.
- _____ 11. If the heat of a flame is felt with the hand holding the test tube clamp (holding the test tube), the flame is a cool flame.
- _____ 12. Blow out the solution remaining in the pipet tip after the solution has drained from the pipet.
- _____ 13. A buret must *always* be filled to the top (the zero mark) before every titration procedure.
- _____ 14. The volume of solution in a buret should be read and recorded 10–15 seconds after completing the titration.
- _____ 15. It is possible to add a half-drop of solution from a buret.
- _____ 16. To test the acidity of a solution with litmus paper, place the litmus paper directly in the solution.
- _____ 17. The odor of a chemical should not be tested unless specifically instructed to do so. The vapors of the chemical should be fanned toward the nose.
- _____ 18. A “blue 3” on a reagent bottle indicates the reagent poses an extreme danger.

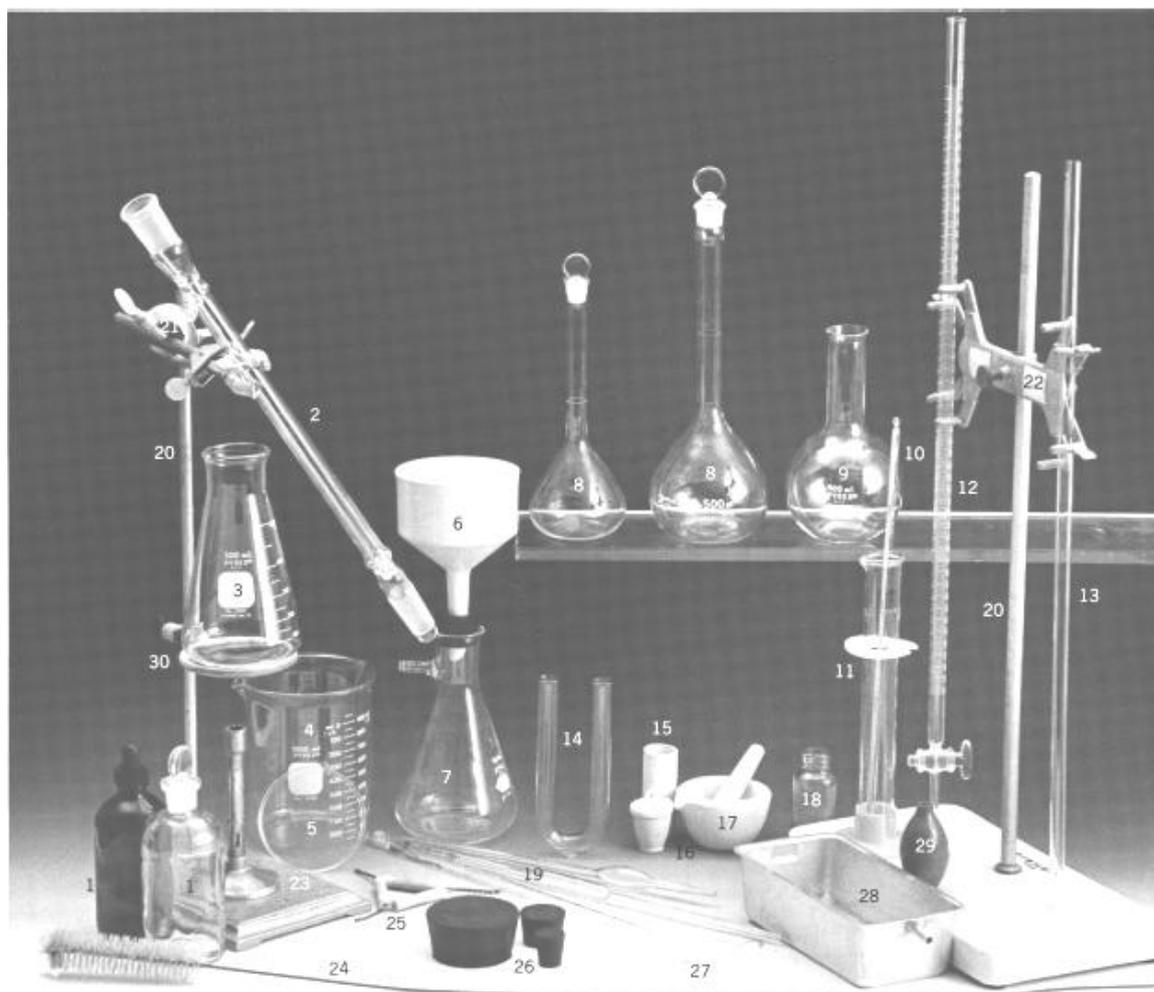
Summarize the “Disclaimer” in your own words.

متطلبات السلامة العامة في مختبرات الكيمياء

ارتداء مريول المختبر	-2	الامتناع عن الأكل والشرب داخل المختبر
يجب على الطالب إحضار بشكير وكبريت واسفنجة	-4	كل من يتسبب في كسر أي أداة يتحمل ثمنها
إحضار مفتاح الخزانة في كل مختبر	-6	استعمال النظارات الواقية للعينين إجباري
عدم دخول المختبر قبل الوقت الرسمي المحدد في البرنامج إلا بإذن رسمي		
عدم عمل تجارب خارجة عن المنهاج وكل من يخالف ذلك يتحمل مسؤولية		
يجب على كل طالب أن يقرأ التجربة في البيت قبل دخوله المختبر		
يجب على كل طالب أن يتعامل مع الأثاث والخزائن والأجهزة المخبرية بليونة		
يجب على كل طالب أن يعرف كيفية استعمال طفايات الحريق		
عند سكب الأحماض والقواعد في المغسلة يجب أن يتبع ذلك سكب كمية كافية من الماء		
عدم وضع فراشي التنظيف في الحوامض والقواعد لأن ذلك يتلفها		
عند إشعال الغاز يجب إشعال عود الثقاب أولاً ثم فتح حنفية الغاز		
يجب التعامل مع الموازين والأجهزة بليونة وكل من يلتبس عليه شيء يراجع المشرف على المختبر		
التفاعلات التي ينتج عنها غازات سامة يجب إجرائها في خزانة الأبخرة		
إذا حصل وسكبت محاليل كيميائية بطريق الخطأ على ملابسك أو جلدك فاغسلها جيداً بالماء		
عدم إلقاء أعواد الثقاب والنفايات الصلبة في المغاسل	-19	يجب عدم الإسراف في استعمال المواد
إذا حصل معك حادث مهما كان بسيطاً فيجب عليك إخبار المشرف على المختبر		
لا تسخن المواد المتطايرة أو القابلة للاشتعال على اللهب مباشرة لأن ذلك يسبب حريقاً في المختبر		
لا تترك أي مادة كيميائية أو محلول كيميائي في وعاء دون أن تكتب على الوعاء اسم المادة أو المحلول		
يجب على كل طالب أن يحافظ على مكان عمله نظيفاً		
إذا حصل واشتعلت النار في ملابسك فلا تركض نم على الأرض وتدرج حتى يتم التخلص من الحريق		
ليس من الضروري أن يحصل لك الضرر من أغلاطك بل ربما من أغلاط جارك فلذلك يجب عليك الحذر وإذا رأيت جارك يقوم بعملاً خاطئاً فعليك إخبار المشرف على المختبر فوراً		
لا تعمل في المختبر لوحدك بل يجب أن يكون هناك مشرف على المختبر		
لا تضبع وقتك في المزاح واللعب وحافظ على جو من المسؤولية والجد والنظام		
عند إذابة المحاليل لبعضها البعض لا تضعهم دفعة واحدة بل على دفعات وبالتدريج مع التحريك المستمر وعادة تضاف المحاليل المركزة إلى الأقل تركيز ويضاف الحامض إلى الماء وليس العكس		
لا تجعل فوهة أنبوب الاختبار باتجاه وجهك أو وجه زميلك أثناء العمل		
عند نهاية المختبر ضع جميع أدواتك الزجاجية نظيفة في الخزانة وأغلق الخزانة وحافظ على مفاتيحها ونأكد من حنفية الغاز والماء مغلقة واغسل يديك جيداً قبل مغادرتك المختبر		

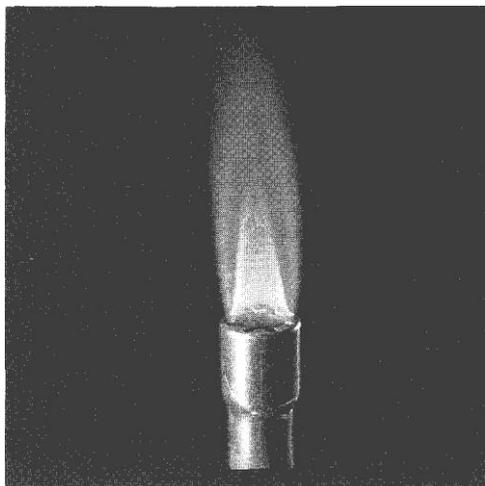
Special Laboratory Equipment

Number	Item	Number	Item
1	reagent bottles	16	porcelain crucible and cover
2	condenser	17	mortar and pestle
3	500-mL Erlenmeyer flask	18	glass bottle
4	1000-mL beaker	19	pipets
5	Petri dish	20	ring and buret stands
6	Büchner funnel	21	clamp
7	Büchner (filter) flask	22	double buret clamp
8	volumetric flasks	23	Bunsen burner
9	500-mL Florence flask	24	buret brush
10	10°C–110°C thermometer	25	clay pipe-stem triangle
11	100-mL graduated cylinder	26	rubber stoppers
12	50-mL buret	27	wire loop for flame test
13	glass tubing	28	pneumatic trough
14	U-tube	29	rubber pipet bulb
15	porous ceramic cup	30	iron support ring



Experiment 1

Basic Laboratory Operations



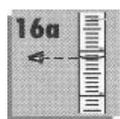
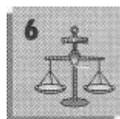
A properly adjusted Bunsen flame burns with a blue, nonluminous flame

- To light and properly adjust the flame of a Bunsen burner
- To develop the skill for properly operating a balance
- To develop the technique of using a pipet
- To determine the density of an unknown substance

OBJECTIVES

The following techniques are used in the Experimental Procedure

TECHNIQUES



You will use a number of techniques repeatedly throughout your laboratory experience principal techniques are fully described under Laboratory Techniques in this manual. Become familiar with each of these techniques before you are required to use them in an experiment.

INTRODUCTION

In this experiment you will learn several common techniques that are used repeatedly throughout this laboratory manual-you will learn to light and adjust a Bunsen burner, to use a laboratory balance, and to use a pipet. With the skills developed in using a balance and pipet, you will determine the density of a metal and a liquid.

Laboratory burners come in many shapes and sizes, but all accomplish one main purpose: a combustible gas-air mixture yields a hot, efficient flame. Because Robert Bunsen (1811-1899) was the first to design and perfect this burner, his name is given to most burners of this type used in the general chemistry laboratory (Figure1.1).

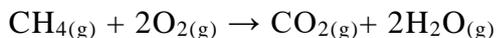
Bunsen Burner

Hydrocarbon: a molecule consisting of only the elements carbon and hydrogen

Nonluminous: nonglowing or nonilluminating

Incandescence: glowing with intense heat

The combustible gas used to supply the fuel for the Bunsen burner in most laboratories is natural gas. Natural gas is a mixture of gaseous **hydrocarbons**, but primarily the hydrocarbon methane, CH₄. If sufficient oxygen is supplied, methane burns with a blue, **nonluminous** flame, producing carbon dioxide and water as combustion products.



with an insufficient supply of oxygen, small carbon particles are produced which, when heated to **incandescence**, produce a yellow, luminous flame. The combustion products may, in addition to carbon dioxide and water, include carbon monoxide.

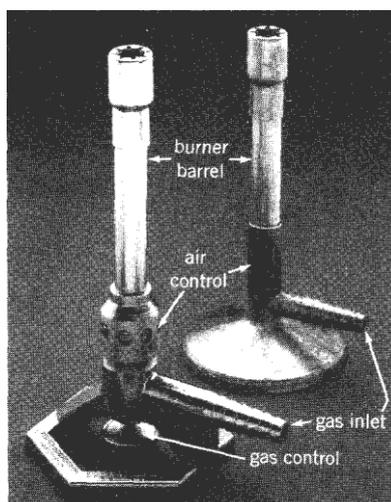


Figure 1.1 Bunsen-type burners.

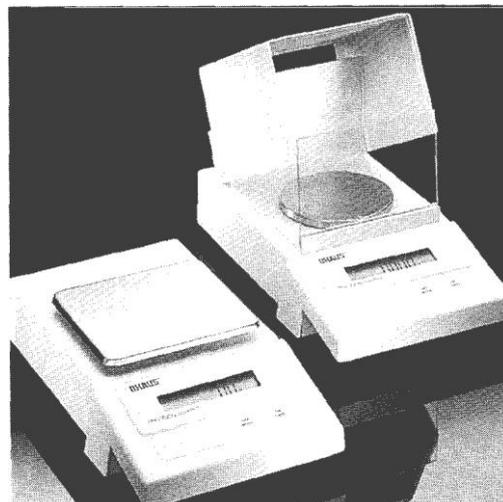


Figure 1.2 Two balances with different sensitivities.

Laboratory Balances

The laboratory balance is perhaps the most common and most often used piece of laboratory apparatus. Balances, not scales, are of different makes, models, and sensitivities (Figure 1.?). The selection of the appropriate balance for a mass measurement depends upon the degree of precision required for the analysis. The triple-beam and top-loading balances are the most common. Read Technique 6 under Laboratory Techniques for instructions on the proper operation of the laboratory balance.

Density

Intensive property: property independent of sample size

density = mass/ volume

Each pure substance exhibits its own set of **intensive properties**. One such property is density, the mass of a substance per unit volume. In the English system the density of water at 4°C is 8.34 lb/gal or 6.22 lb/ft³, whereas in SI the **density** of water at 4°C is 1.00 g/cm³ or 1.00 g/mL. By measuring the mass and

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 as 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

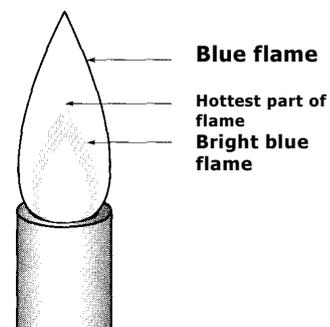


Figure 1.3 Flame of a properly adjusted Bunsen burner.

2. Observing Flame Temperatures Using a Wire Gauze.

Temperatures within the second (inner) cone of a nonluminous blue flame approach 1500°C .

- Using crucible tongs (or forceps), hold a wire gauze parallel to the 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.
- Close the air control valve and repeat the observation with a luminous flame.

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

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

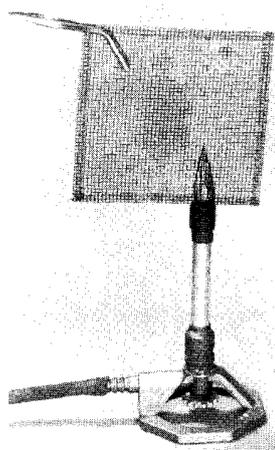


Figure 1.4 Hold the wire gauze parallel to burner barrel.

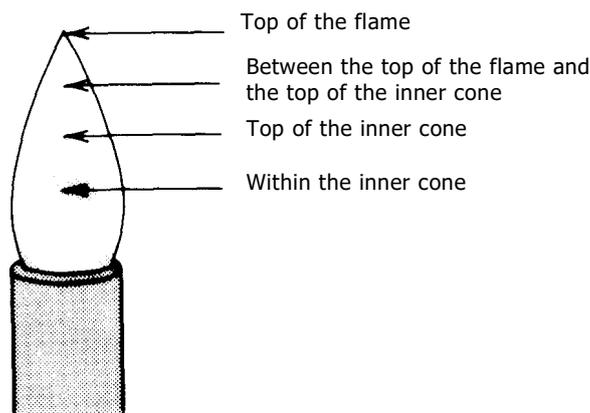


Figure 1.5 Regions of the flame temperature measurements.

B. Laboratory Balances



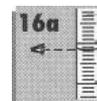
- 1. Practice Using the Balances.** Refer to the Report Sheet. As suggested. Measure the mass of several objects. Use the top-loading balance only after the instructor explains its operation. Be sure to record the mass of the objects according to the sensitivity of the balance.[®] Refer to Technique 6.

Ask your instructor which balance you are to use to determine the densities of your unknowns. Write the balance number on the Report **C. Density**

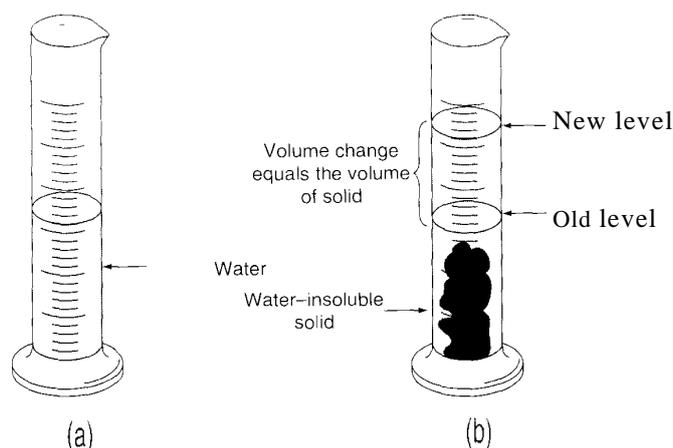


1. Water-Insoluble Solid.

- Obtain an unknown solid and record its number.® Using the assigned balance tare the mass of a piece of weighing paper, place the solid on the weighing paper, and measure its mass. Record the mass to the sensitivity allowed by the balance.
- Half-fill a 10-mL graduated cylinder with water and record its volume (Figure 1.6a). Refer to Technique 16A for reading and recording a volume.
- Gently slide the known mass of solid into the graduated cylinder held at a 45° angle. Roll the solid around in the cylinder, removing any air bubbles that are trapped or that adhere to the solid. Record the new water level (Figure 1.6b).



The volume of the solid is the difference between the two water levels. Repeat this procedure with a second "dry" solid sample for Trial 2.



3

Figure 1.6 Apparatus for measuring the density of a water-insoluble solid.

Disposal: Check with your instructor for the procedure of properly returning the solid sample.



2. Liquid, Water.

- Clean and dry your smallest laboratory beaker. Using your assigned balance, tare its mass. Pipet 5 mL of water into the beaker.



- b. Measure the mass of the water. Calculate the density of water from the available data. Repeat the density determination for Trial ?.
- c. Collect and record the density value of water at room temperature from five additional laboratory measurements from classmates. Calculate the average density of water at room temperature.

3. Liquid, Unknown.



Flammable: capable of igniting in air (generally initiated with a flame or spark)

- a. Dry, the beaker and pipet. Ask the instructor for a liquid unknown and record its number[Ⓢ] (**Caution:** The unknown liquid may be **flammable**. Do not inhale the fumes of the liquid: extinguish all flames.)
- b. Rinse the pipet with two 1-ml- quantities of the unknown liquid and discard. Repeat the measurements of Part C.1. substituting the unknown liquid for the water. Repeat this experiment for Trial 2. Calculate the average density of the liquid.



Disposal: Check with your instructor. Dispose of the unknown liquid and the rinses in the "Waste Liquids" container.

Experiment 1 *Prelaboratory Assignment*

Basic Laboratory Operations

Date _____ Lab Sec. _____ Name _____ Desk No. _____

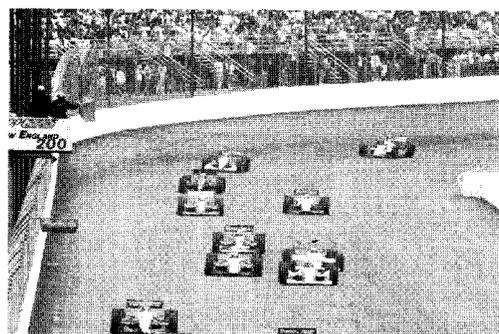
1. a. What is the dominant color of a nonluminous flame from a Bunsen burner? Explain...

b. Is the temperature of a luminous flame greater or less than that of a non luminous flame? Explain...

c. Why does a luminous flame appear yellow?

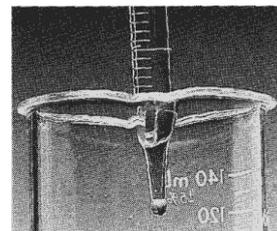
2. Experimental Procedure, Part C. I. hat is the meaning of the phrase, "tare the mass of a piece of weighing paper?"

3. A fire in the "pits" at the Indianapolis 500 Motor Speedway is especially dangerous because the flame from the fuel used in the race cars is nearly colorless and non luminous, unlike that of a gasoline fire. How might the fuel used in the Indianapolis 500 race cars differ from that of gasoline?



4. Refer to Technique 16B.

a. Remove the drop suspended from a pipet tip by ...



b. The finger used to control the delivery of liquid from a pipet is the ...

c. A pipet is filled with the aid of a ...

d. Most pipets are calibrated as "TD 20°C". Define "TD" and what is its meaning regarding the volume of liquid a pipet delivers?

5. Experimental Procedure, Part C. 1. The density of diamond is 3.51 g/cm^3 and the density of lead is 11.3 g/cm^3 . If equal masses of diamond and lead are transferred to equal volumes of water in separate graduated cylinders, which graduated cylinder would have the greatest volume change? Explain.

6. Experimental Procedure, Part C.3. The mass of a beaker is 5.333 g. After 5.00 mL of hexane, C_6H_{14} is pipetted into the beaker, the combined mass of the beaker and the hexane sample is 8.613 g. From the data, what is the measured density of hexane?

Experiment 1 *Report Sheet*

Basic Laboratory Operations

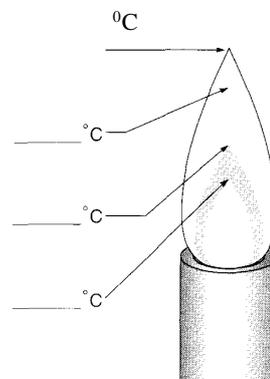
Date _____ Lab Sec. _____ Name _____ Desk No. _____

A. Bunsen Burner

1. [ⓐ] Instructor's Approval of a well-adjusted Bunsen flame.
 - a. At right, draw a sketch of the heat zones for a nonluminous flame as directed with the wire gauze positioned parallel to the burner barrel. Label the "cool" and "hot" zones.

 - b. What happens to the "cool" and "hot" zones of the flame when the air control valve is closed? Explain

2. [ⓑ] Based upon the melting points of the metals, estimate (on the diagram) the temperature of the following regions of the flame (see Figure 1.5).
 - a. Top of the flame
 - b. Between the top of the flame and the inner cone
 - c. Top of the inner cone
 - d. Within the inner cone



3. Does the diagram agree with your sketch from the wire gauze test? Explain.

B. Laboratory Balances

③ Determine the mass of the following objects on the respective balance. Express your results with the Correct sensitivity. Compare the masses recorded for the same object on the different balances.

Object	Triple-Beam Balance	Top-Loading Balance
Test tube	_____	_____
Beaker	_____	_____
Erlenmeyer flask	_____	_____

C. Density

④ What is your balance number?

1. ④ Solid Unknown Number	Trial 1	Trial 2
a. Tared mass of solid (g)	_____	_____
b. Volume of water (cm ³)	_____	_____
c. Volume of water and solid (cm ³)	_____	_____
d. Volume of solid (cm ³)	_____	_____
e. Density of solid (g/cm ³)	_____	_____
f. Average density of solid (g/cm ³)	_____	

2. Liquid

Water

	Trial 1	Trial 2
a. Tared mass of liquid (g)	_____	_____
b. Volume of liquid (mL)	_____	_____
c. Density of liquid (g/mL)	_____	_____
d. Average density of liquid (g/mL)	_____	

Laboratory Questions

Circle the questions that have been assigned.

1. Part A.1b. Suppose the air holes at the base of the Bunsen burner are not closed. What would most likely happen the striker (or lighted match) is used in Part A.1 c? Explain.

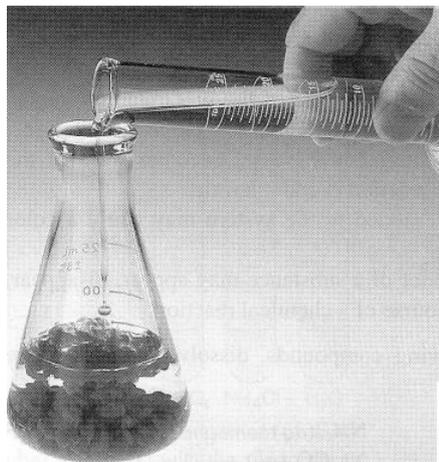
2. Part A. 1e. If the air control valve is opened too wide, what happens to the flame'? Explain.



3. Part C.1 c. An air bubble adheres to the surface of the solid when it is submerged in the water. Explain how this phenomenon affects the reported density of the solid.

4. Part C.2. Suppose that after delivery several drops of the water cling to the inner wall of the pipet (because the pipet wall is dirty). How does this technique error affect the reported density of water? See drawing at right.

5. Part C.3. The unknown liquid is known to be volatile. If some of the liquid evaporates between the time that the liquid is delivered to the beaker and the time that its mass is measured, will the reported density of the liquid be too high, too low. or unaffected') Explain.



A potassium chromate solution added to a silver nitrate solution results in the formation of insoluble silver chromate

Experiment 2

Identification of a Compound: Chemical Properties

- To identify a compound on the basis of its chemical properties
- To design a systematic procedure for determining the presence of a particular compound in aqueous solution

The following techniques are used in the Experimental Procedure



TECHNIQUES

Chemical property: characteristic of a substance that is dependent on its chemical environment

Chemists, and scientists in general, develop and design experiments in an attempt to understand, explain, and predict various chemical phenomena. Carefully controlled (laboratory) conditions are needed to minimize the many parameters that affect the observations. Chemists organize and categorize their data, and then systematically analyze the data to reach some conclusion; often, the conclusion may be to carefully plan more experiments!

It is presumptuous to believe that a chemist must know the result of an experiment before it is ever attempted; most often an experiment is designed to determine the presence or absence of a **substance** or to determine or measure a parameter. A goal of the environmental or synthesis research chemist is, for example, to separate the substances of a reaction mixture (either one generated in the laboratory or one found in nature) and then identify each substance through a systematic, or sometimes a **trial-and-error**, study of their chemical and physical properties. As you will experience later, Experiments 37-39 are designed to identify a specific ion (by taking advantage of its unique chemical properties) in a mixture of ions through a systematic sequence of analyses.

INTRODUCTION

Substance: a pure element or compound having a unique set of chemical and physical properties

Trial-and-error study: a method that is often used to seek a pattern in the accumulated data

On occasion an experiment reveals an observation and data that are uncharacteristic of any other set of properties for a known substance, in which case either a new compound has been synthesized/discovered or experimental errors and/or interpretations have infiltrated the study.

In this experiment, you will observe chemical reactions that are characteristic of various compounds under controlled conditions. After collecting and organizing your data, you will be given an unknown compound; a compound that you have previously investigated. The interpretations of the collected data will assist you in identifying your compound.

What observations will you be looking for? Chemical changes are generally accompanied by one or more of the following:

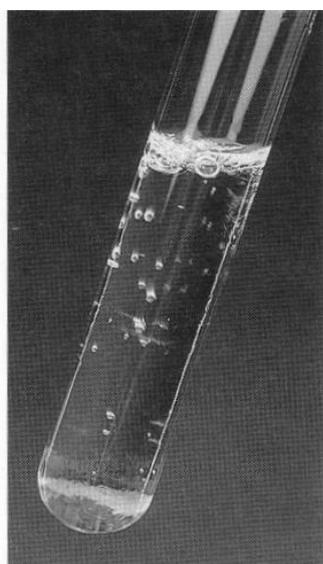


Figure 3.1 A reaction mixture of $\text{NaHCO}_3(\text{aq})$ and $\text{HCl}(\text{aq})$ produces CO_2 gas.

- A gas is evolved. This evolution may be quite rapid or it may be a "fizzing" sound (Figure 3.1).
- A precipitate appears (or disappears). The nature of the precipitate is important; it may be crystalline, it may have color, it may merely cloud the solution. Heat may be evolved or absorbed. The reaction vessel becomes warm if the reaction is exothermic or cools if the reaction is endothermic.
- A color *change* occurs. A substance added to the system may cause a color change.
- A *change in odor* is detected. The odor of a substance may appear, disappear, or become more intense during the course of a chemical reaction.

The chemical properties of the following compounds, dissolved in water, are investigated in Part A (1, 2, 3, 4) of this experiment:

Sodium chloride	$\text{NaCl}_{(\text{aq})}$
Sodium carbonate	$\text{Na}_2\text{CO}_{3(\text{aq})}$
Sodium sulfate	$\text{Na}_2\text{SO}_{4(\text{aq})}$
Barium chloride	$\text{BaCl}_{2(\text{aq})}$
Zinc sulfate	$\text{ZnSO}_{4(\text{aq})}$
Ammonium chloride	$\text{NH}_4\text{Cl}_{(\text{aq})}$
Water	$\text{H}_2\text{O}_{(\text{l})}$

Reagent: a solid chemical or a solution having a known concentration of solute

The following test reagents are used to identify and characterize these compounds:

Silver nitrate	$\text{AgNO}_{3(\text{aq})}$
Barium nitrate	$\text{Ba}(\text{NO}_3)_{2(\text{aq})}$
Sodium hydroxide	$\text{NaOH}_{(\text{aq})}$
Sulfuric acid	$\text{H}_2\text{SO}_{4(\text{aq})}$

In Part A5 of this experiment the chemical properties of five compounds in aqueous solutions, labeled 1 through 7, are investigated with four reagents, labeled A, B, C, and D. Chemical tests will be performed with these eleven solutions. An unknown will then be issued and matched with one of the solutions, labeled 1 through 7.

Procedure Overview: In Part A(1, 2 3 4) a series of tests for the chemical properties of known compounds in aqueous solutions are conducted. A similar series of tests are conducted on an unknown set of compounds in Part A5. In each case, an unknown compound is identified on the basis of the chemical properties observed.

You should discuss and interpret your observations on the known chemical tests with a partner, but each of you should analyze your own unknown compound. At each circled superscript⁽¹⁻⁷⁾ in the procedure, *stop*, and record your observation on the Report Sheet.

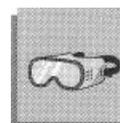
To organize your work, you will conduct a test on each known compound in the seven aqueous solutions and the unknown compound with a single test reagent. It is therefore extremely important that you are organized and that you describe your observations as completely and clearly as possible. The Report Sheet provides a "reaction matrix" for you to describe your observations; because the space is limited, you may want to devise a code, e.g.,

- p-precipitate + color
- c-cloudy + color
- nr-no reaction
- g-gas, no odor
- go-gas, odor

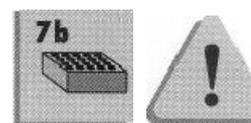
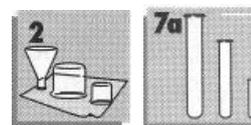
1. Observations with Silver Nitrate Test Reagent.

- Use a permanent marker to label seven small, clean test tubes (Figure 3.2a) or set up a clean 24-well plate (Figure 3.2b). Ask your instructor which setup you should use. Place 5-10 drops of each of the seven "known" solutions into the labeled test tubes .
- Use a dropper pipet (or a dropper bottle) for the delivery of the silver nitrate solution. (**Caution:** AgNO_3 forms black stains on the skin. The stain, caused by silver metal, causes no harm.) If after adding several drops you observe a chemical change, add 5-10 drops to see if there are additional changes. Record your observation in the matrix on the report sheet.[Ⓞ]

EXPERIMENTAL PROCEDURE



A. Chemical Properties of Known Compounds



Appendix G

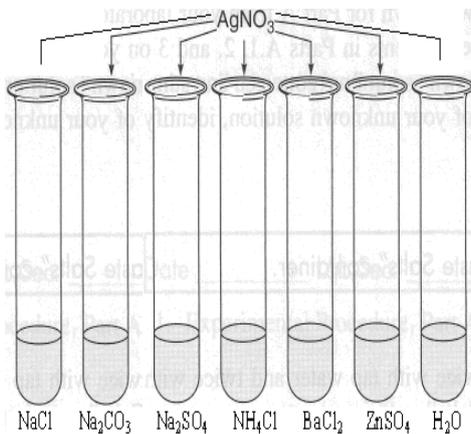


Figure 3.2a Arrangement of test tubes for testing with the silver nitrate

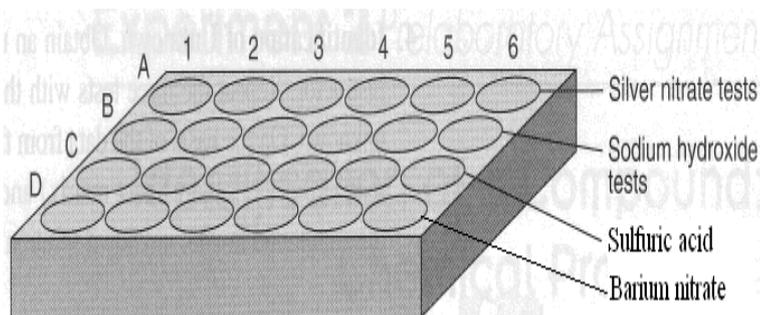


Figure 3.2b Arrangement of test solutions in the 24-well plate for testing salts

2. Observations with Sodium Hydroxide Test Reagent.

- Use a permanent marker to label five additional small, clean test tubes (Figure 3.3). Place 5-10 drops of each of the seven "known" solutions into this second set of labeled test tubes.
- To each of these solutions slowly add 5-10 drops of the sodium hydroxide solution; make observations as you add the solution. Check to see if a gas evolves in any of the tests. Check for odor. What is the nature of any precipitates that form? Observe *closely*[ⓐ]



Appendix G

3. Observations with Sulfuric Acid Test Reagent.

- Use a permanent marker to label seven additional small, clean test tubes (Figure 3.4). Place 5-10 drops of each of the seven "known" solutions into this four set of labeled test tubes (Figure 3.2b).
- Add the sulfuric test reagent to the solutions and record your observations. Check to see if any gas is evolved. Check for odor. Observe *closely*.[ⓑ]

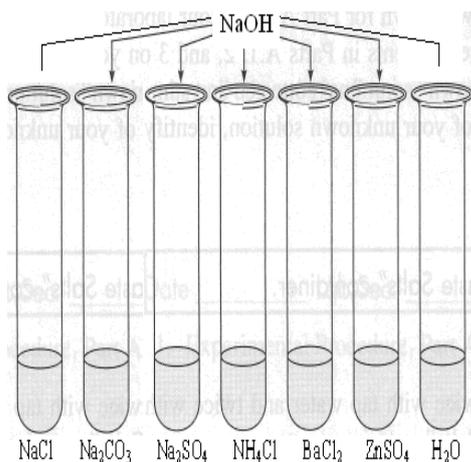


Figure 3.3 Arrangement of test tubes for testing with the sodium hydroxide reagent

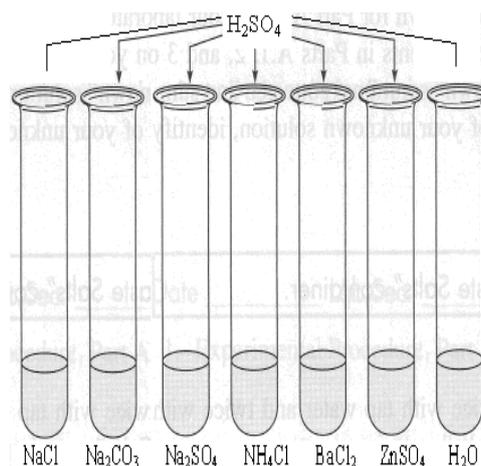


Figure 3.4 Arrangement of test tubes for testing with the sulfuric acid reagent

4. Observations with Barium Nitrate Test Reagent.

- Use a permanent marker to label seven small, clean test tubes (Figure 3.5 or set up a clean 24-well plate (Figure 3.2b). Ask your instructor which setup you should use. Place 5-10 drops of each of the seven "known" solutions into the labeled test tubes .
- Use a dropper pipet (or a dropper bottle) for the delivery of the barium nitrate solution. *If* after adding several drops you observe a chemical change, add 5-10 drops to see if there are additional changes. Record your observation in the matrix on the report sheet.[Ⓢ]

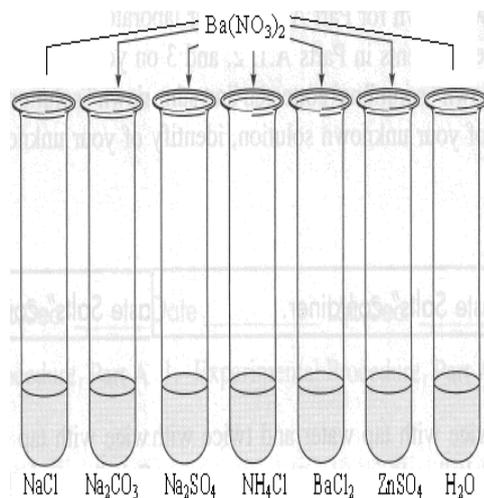


Figure 3.5 Arrangement of test tubes for testing with the barium nitrate reagent

- 5. Identification of Unknown.** Obtain an unknown for Part A from your laboratory instructor. Repeat the four tests with the reagents in Parts A.1, 2, 3, and 4 on your unknown. On the basis of the data from the "known" solutions (collected and summarized in the Report Sheet matrix) and that of your unknown solution, identify the compound in your unknown solution.[Ⓢ]

Disposal : Discard the test solutions in the "Waste Salts" container



CLEANUP: Rinse the test tubes or well plate twice with tap water and twice with deionized water. Discard each rinse in the "Waste Salts" container.



Experiment 2 Prelaboratory Assignment

Identification of a Compound: Chemical Properties

Date _____ Lab Sec. _____ Name _____ Desk No. _____

1. Experimental Procedure, Part A.

a. What is the criterion for clean glassware?



b. What is the size and volume of a "small, clean test tube?"



2. Experimental Procedure, Part A.2. Describe the technique for testing the odor of a chemical.



3. a. Depending upon the tip of a dropper pipet, there are approximately 20 drops per milliliter of water. What is the approximate volume (in ml-) of one drop of an aqueous solution?

b. A micropipet delivers 153 drops of alcohol for each milliliter. Calculate the volume (in ml-) of alcohol in each drop.

4. Write a balanced equation for the following observed reactions:

a. Aqueous solutions of sodium hydroxide, NaOH, and sulfuric acid, H₂SO₄, are mixed. The neutralization products are water and one other compound with the evolution of heat.

b. Aqueous solutions of copper(II) nitrate, Cu(NO₃)₂, and sodium carbonate, Na₂CO₃, are mixed. A

blue precipitate of copper(II) carbonate forms in addition to one other compound.

5. Experimental Procedure, Part A. The substances, NaCl, Na₂CO₃, Na₂SO₄, and NH₄Cl used for test solutions, are all soluble ionic compounds. For each substance indicate the ions present in its respective test solution.

NaCl: _____

Na₂CO₃: _____

Na₂SO₄: _____

NH₄Cl: _____

6. Three colorless solutions in test tubes, with no labels, are in a test tube rack on the laboratory bench. Lying beside the test tubes are three labels: potassium iodide, KI, silver nitrate, AgNO₃, and sodium sulfide, Na₂S. You are to place the labels on the test tubes using only the three solutions present. Here are your tests:

A portion of test tube #1 added to a portion of test tube #3 produces a yellow, silver iodide precipitate. A portion of test tube #1 added to a portion of test tube #2 produces a black, silver sulfide precipitate.

- a. Your conclusions are:

Test Tube 1 _____

Test Tube 2 _____

Test Tube 3 _____

- b. Write the balanced equation for the formation of silver iodide, AgI.

- c. Write the balanced equation for the formation of sulfide, Ag₂S.

Identification of a Compound: Chemical Properties

Date _____ Lab Sec. _____ Name _____ Desk No. _____

A. Chemical Properties of Known Compounds

Test	NaCl	Na ₂ CO ₃	Na ₂ SO ₄	NH ₄ Cl	BaCl ₂	ZnSO ₄	H ₂ O	Unknown
AgNO ₃								
NaOH								
H ₂ SO ₄								
Ba(NO ₃) ₂								

Sample No. of unknown for part A.5 _____

Compound in unknown solution is _____

Laboratory Questions

Circle the questions that have been assigned.

1. What *chemical* test will distinguish calcium chloride, CaCl_2 , from calcium carbonate, CaCO_3 ? Explain.

2. What test reagent will distinguish a soluble Cl^- salt from a soluble SO_4^{2-} salt? Explain.

3. Predict what would be observed (and why) from an aqueous mixture for each of the following:

- potassium carbonate and hydrochloric acid
- zinc chloride and silver nitrate
- magnesium chloride and sodium hydroxide
- ammonium nitrate and sodium hydroxide

4. For a solution of the cations Ag^+ , Ba^{2+} , Mg^{2+} , or Cu^{2+} , the following experimental observations were collected:

	$\text{NH}_3(\text{aq})$	$\text{HCl}(\text{aq})$	$\text{H}_2\text{SO}_4(\text{aq})$
Ag^+	No change	White ppt ^a	No change
Ba^{2+}	No change	No change	White ppt
Mg^{2+}	White ppt	No change	No change
Cu^{2+}	Blue ppt/deep blue soln with excess	No change	No change

^aExample: when an aqueous solution of hydrochloric acid is added to a solution containing Ag^+ , a white precipitate (ppt) forms.

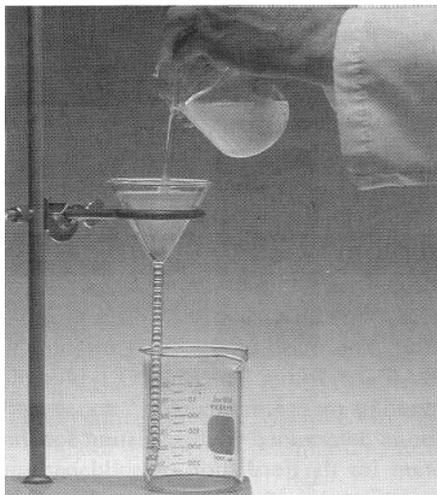
- Identify a reagent that distinguishes the chemical properties of Ag^+ and Mg^{2+} . Explain.
- Identify a reagent that distinguishes the chemical properties of HCl and H_2SO_4 . Explain.
- Identify a reagent that distinguishes the chemical properties of Ba^{2+} and Cu^{2+} . Explain.
- *d. Identify a reagent that distinguishes the chemical properties of Cu^{2+} and Mg^{2+} . Explain.

5. Three colorless solutions in test tubes, with no labels, are in a test tube rack on the laboratory bench. Lying beside the tests tubes are three labels: 0.10 M Na_2CO_3 , 0.10 M HCl , and 0.10 M KOH . You are to place the labels on the test tubes using only the three solutions present. Here are your tests:
- A few drops of the solution from test tube #1 added to a similar volume of the solution in test tube #2 produces no visible reaction but the solution becomes warm.
 - A few drops of the solution from test tube #1 added to a similar volume of the solution in test tube #3 produces carbon dioxide gas.

Identify the labels for test tube #1, #2, and #3.

Experiment 3

Limiting Reactant



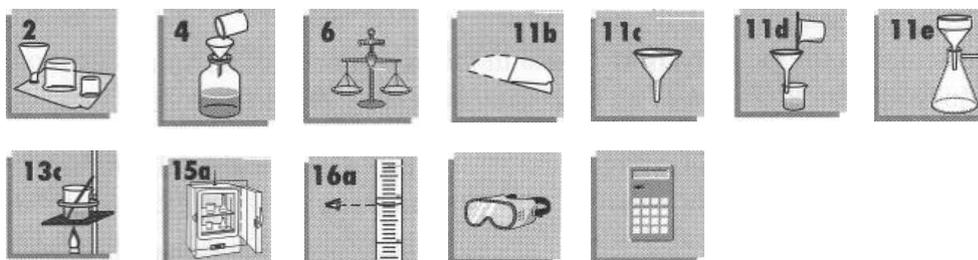
Gravity filtration is used to filter finely divided precipitates.

- To determine the limiting reactant in a mixture of two soluble salts
- To determine the percent composition of each substance in a salt mixture

OBJECTIVES

The following techniques are used in the Experimental Procedure

TECHNIQUES



Percent composition: the mass ratio of the components of a mixture or compound to the total mass of the sample.

Two factors affect the yield of products in a chemical reaction: (1) the amounts of starting materials (reactants) and (2) the **percent yield** of the reaction. Many experimental conditions, for example, temperature and pressure, can be adjusted to increase the yield of a desired product in a chemical reaction, but because chemicals react according to fixed mole ratios (**stoichiometrically**), only a limited amount of product can form from given amounts of starting materials. The reactant determining the amount of product generated in a chemical reaction is called the **limiting reactant** in the chemical system.

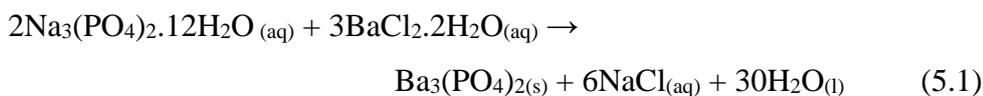
INTRODUCTION

Percent yield:

$$\left(\frac{\text{actual yield}}{\text{theoretical yield}} \right) \times 100$$

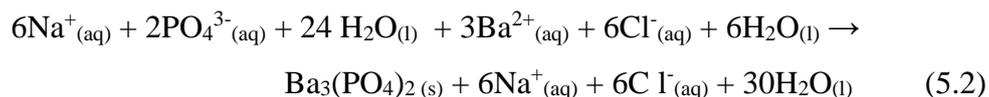
Stoichiometrically: by a study of a chemical reaction using a balanced equation.

To better understand the concept of the limiting reactant, let us look at the reaction under investigation in this experiment, the reaction of sodium phosphate dodecahydrate, $\text{Na}_3(\text{PO}_4)_2 \cdot 12\text{H}_2\text{O}$, and barium chloride dihydrate, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, in an aqueous system. The molecular form of the equation for the reaction in aqueous solution is:



As the two reactant salts and sodium chloride are soluble in water but

barium sulfate is insoluble, the ionic equation for the reaction is



Spectator ions: cations or anions that do not participate in any observable or detectable chemical reaction

Presenting only the ions that show evidence of a chemical reaction occurring, i.e., the formation of a precipitate (and removing **spectator ions** from the equation), we have the **net ionic equation** for the observed reaction:



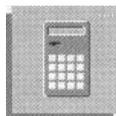
Net ionic equation: an equation that includes only those ions that participate in the observed chemical reaction.

From the balanced net ionic equation, 2 mol of phosphate ion (from the 2 mol of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, molar mass = 380.12 g/mol, or 760.24 g) reacts with 3 mol of barium ion (from 3 mol of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, molar mass = 244.27 g/mol, or 732.81 g) if the reaction proceeds to completion. The equation also predicts the formation of 1 mol of $\text{Ba}_3(\text{PO}_4)_2$ (molar mass = 601.93 g/mol), or 601.93 g.

In Part A of this experiment the solid salts $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ and $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ form a heterogeneous mixture of unknown composition. The mass of the solid mixture is measured and then added to water-insoluble $\text{Ba}_3(\text{PO}_4)_2$ forms. The $\text{Ba}_3(\text{PO}_4)_2$ precipitate is collected, via gravity filtration and dried, and its mass is measured.

The percent composition of the salt **mixture is determined by first testing for the limiting reactant**. In Part B, the limiting reactant for the formation of solid barium phosphate is determined from two precipitation tests of the solution: (1) the solution is tested for an excess of barium ion with a phosphate reagent-observed formation of a precipitate indicates the presence of an excess of barium ion (and a limited amount of phosphate ion) in the salt mixture; (2) the solution is also tested for an excess of phosphate ion with a barium reagent-observed formation of a precipitate indicates the presence of an excess of phosphate ion (and a limited amount of barium ion) in the salt mixture.

Calculations



The calculations for the analysis of the data in this experiment require some attention. The question, after collection of all of the data, becomes, "How do I proceed to determine the percent composition of a salt mixture containing the salts $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ and $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ from the data of a precipitation reaction?" Consider the following scenario: A 0.942 g sample of the salt mixture is added to water and 0.188 g of $\text{Ba}_3(\text{PO}_4)_2$ precipitate forms. Tests reveal that $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ is the limiting reactant. What is the

percent composition of the salt mixture?

Using the experimental fact that $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ is the limiting reactant, the stoichiometry of the reaction indicates that 1 mol $\text{Ba}_3(\text{PO}_4)_2$ precipitate requires 3 mol Ba^{2+} (and therefore 3 mol $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) to form (Equation 8.2). As 0.188 g $\text{Ba}_3(\text{PO}_4)_2$ forms, then

$$0.188 \text{ g Ba}_3(\text{PO}_4)_2 \times \frac{1 \text{ mol Ba}_3(\text{PO}_4)_2}{601.93 \text{ g}} \times \frac{3 \text{ mol Ba}^{2+}}{1 \text{ mol Ba}_3(\text{PO}_4)_2} = 9.37 \times 10^{-4} \text{ mol Ba}^{2+}$$

reacts, and

$$9.37 \times 10^{-4} \text{ mol Ba}^{2+} \times \frac{1 \text{ mol BaCl}_2 \cdot 2\text{H}_2\text{O}}{1 \text{ mol Ba}^{2+}} \times \frac{244.27 \text{ g BaCl}_2 \cdot 2\text{H}_2\text{O}}{1 \text{ mol BaCl}_2 \cdot 2\text{H}_2\text{O}} = 0.229 \text{ g}$$

$\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ is a part of the original salt mixture.

The mass of the $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ in the salt mixture must be the difference between the total mass of the original salt sample and the mass of the $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, or $(0.942 \text{ g} - 0.229 \text{ g}) = 0.713 \text{ g}$

The percent $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in the salt mixture is

$$\frac{0.229 \text{ g}}{0.942 \text{ g}} \times 100 = 24.3 \%$$

The percent $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ in the salt mixture is

$$\frac{0.713 \text{ g}}{0.942 \text{ g}} \times 100 = 75.7 \%$$

Procedure Overview: In Part A a measured mass of a solid $\text{Na}_2\text{SO}_4/\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ salt mixture of unknown composition is added to water. The precipitate that forms is digested, filtered, and dried, and its mass measured. Observations from tests on the **supernatant** solution in Part B determine which salt in the mixture is the limiting reactant. An analysis of the data provides the determination of the percent composition of the salt mixture.

Two trials are recommended for this experiment. To hasten the analyses, measure the mass of duplicate unknown solid salt mixtures and simultaneously follow the procedure for each. Label the beakers accordingly for Trial 1 and Trial 2 to avoid the intermixing of samples and solutions.

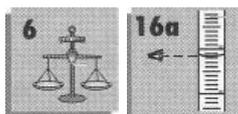
Obtain about 2-3 g of an unknown $\text{Na}_2\text{SO}_4/\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ salt mixture.

EXPERIMENTAL PROCEDURE

Supernatant: the clear solution that exists after the precipitate has settled



A. Precipitation of BaSO_4 from the Salt Mixture



Cool flame: a nonluminous flame with limited amount of gas being burned.

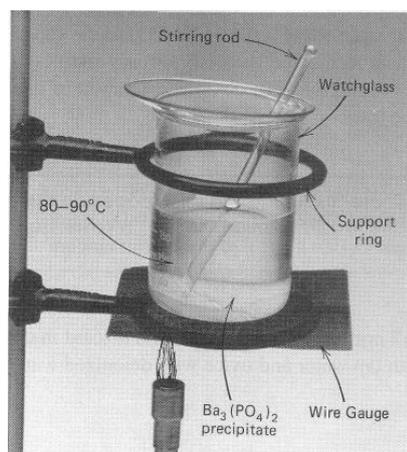


Figure 5.1 Warming and digesting the precipitate

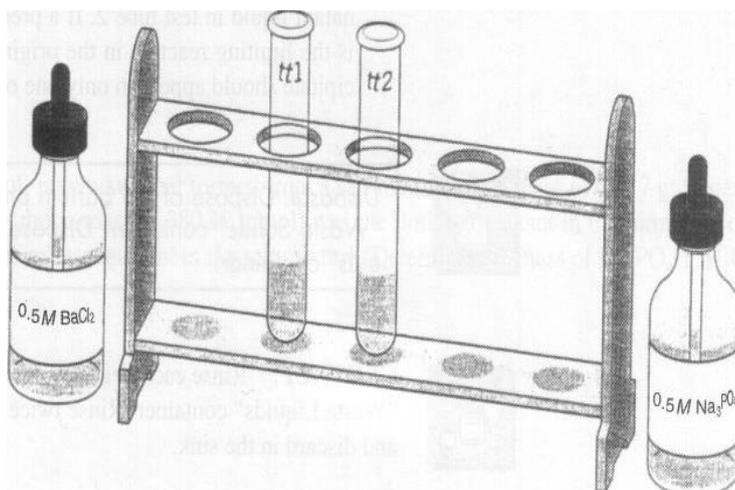


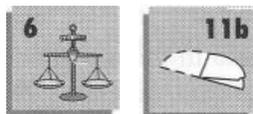
Figure 5.2 Testing for the excess (and the limiting) reactant..

1. Prepare the Salt Mixture.

- Measure (± 0.001 g) on a weighing paper or dish about 1 g of the salt mixture. Record this tared mass for Trial 1 on the Report Sheet. Repeat for Trial 2.
- Transfer the salt mixture to a labeled 250-mL beaker and add ~150 mL of deionized water. Stir the mixture with a stirring rod for 2-3 minutes and then allow the precipitate to settle. Leave the stirring rod in the beaker.

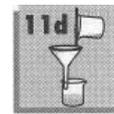
2. Digest the Precipitate.

- Cover the beaker with a watch glass and warm the solution ($80-90^\circ\text{C}$) over a steam bath or with a **cool flame** (*don't boil!*) for 15 minutes¹ (Figure 8.1). While the precipitate is being kept warm, proceed to Part A.3. Periodically check on the progress of the heating.
- After 15 minutes, remove the heat and allow the precipitate to settle; the solution does not need to cool to room temperature.
- While the precipitate is settling, heat ($70-80^\circ\text{C}$) about 30 mL of deionized water for use as wash water in Part A.5.



- Set Up a Gravity (or Vacuum²) Filtering Apparatus.** Place your initials (in pencil) on a piece of Whatman No. 42 or Fisherbrand Q2 filter paper³, fold, and tear off its corner. Determine its mass (± 0.001 g). Seal the filter paper into the filter funnel with a small amount of deionized water. Discard the deionized water from the receiving flask. Have your instructor inspect your apparatus before continuing (see opening photo). Return to Part A.2b.

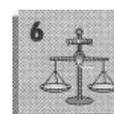
4. Withdraw and Save Supernatant. Once the precipitate has settled and the supernatant has cleared in Part A.2b, use a dropping pipet to withdraw enough supernatant to half-fill two 75-mm test tubes, labeled "1" and "2."



5. 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).

6. 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.

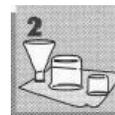
B. Determination of the Limiting Reactant



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.

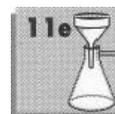


¹ This is called digesting the precipitate. This heating procedure causes the formation of larger BaSO₄ particles for the purpose of a better separation during the filtering procedure.

² 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.

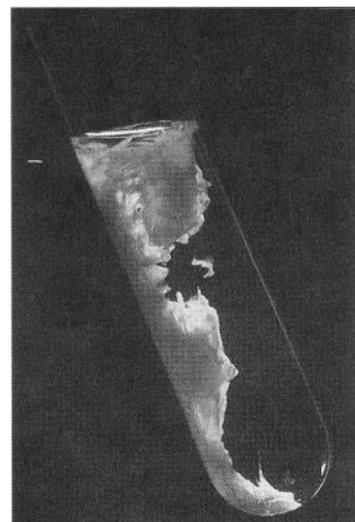
³ Whatman No. 42 and Fisherbrand Q2 filter papers are both fine-porosity filter papers; a fine-porosity filter paper is required to filter the finely divided BaSO₄ precipitate.

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



4. A 1.276-g mixture of the solid salts Na_2SO_4 and $\text{Pb}(\text{NO}_3)_2$ forms an aqueous solution with the precipitation of PbSO_4 . The precipitate was filtered and dried, and its mass was determined to be 0.717 g. The limiting reactant was determined to be $\text{Pb}(\text{NO}_3)_2$.

a. Write the molecular form of the equation for the reaction.



b. Write the net ionic equation for the reaction.

c. How many moles and grams of $\text{Pb}(\text{NO}_3)_2$ are in the reaction mixture?

d. What is the percent by mass of each salt in the mixture?

Experiment 3 *Report Sheet*

Limiting Reactant

Date _____ Lab Sec. _____ Name _____ Desk No. _____

A. Precipitation of BaSO₄ from the Salt Mixture

Unknown number _____

1. Tared mass of salt mixture (g) _____
2. Mass of filter paper (g) _____
3. Mass of filter paper and BaSO₄ (g) _____
4. Mass of BaSO₄ (g) _____

B. Determination of Limiting Reactant

1. Limiting reactant in salt mixture (write complete formula) _____
2. Excess reactant in salt mixture (write complete formula) _____

Data Analysis

1. Moles of BaSO₄ precipitated (mol) _____
2. Moles of limiting reactant in salt mixture (mol)
 - formula of limiting hydrate _____
3. Mass of limiting reactant in salt mixture (g)
 - formula of limiting hydrate _____
4. Mass of excess reactant in salt mixture (g)
 - formula of excess hydrate _____
5. Percent limiting reactant in salt mixture (%)
 - formula of limiting hydrate _____
6. Percent excess reactant in salt mixture (%)
 - formula of excess hydrate _____

*** Show all calculations:**

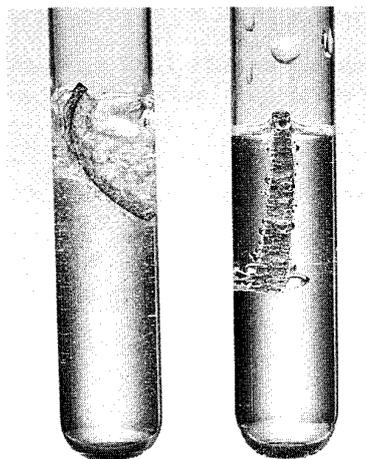
Laboratory Questions

Circle the questions that have been assigned.

1. Part A.2. If the step for digesting the precipitate were omitted, what would be the probable consequence of reporting the "percent limiting reactant" in the salt mixture? Explain.
2. Part A.3. A couple of drops of water were accidentally placed on the properly folded filter paper before its mass was measured. However, in Part A.6, the BaSO_4 precipitate and the filter paper were dry. How does this sloppy technique affect the reported mass of the limiting reactant in the original salt mixture? Explain.
3. Part A.5. Because of the porosity of the filter paper and the finely divided precipitate, some of the BaSO_4 precipitate passes through the filter paper. Will the reported percent of the limiting reactant in the original salt mixture be reported too high or too low? Explain.
4. Part A.5. Excessive quantities of wash water are added to the BaSO_4 precipitate. How does this affect the mass of BaSO_4 precipitate reported in Part A.6?
5. Part A.6. The BaSO_4 precipitate is not completely dry when its "dried" mass is determined. Will the reported mass of the limiting reactant in the original salt mixture be reported too high or too low? Explain.

Experiment 4

Acids And Bases



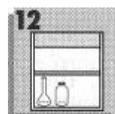
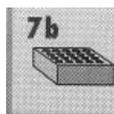
Hydrogen gas is produced at different rates from the reaction of zinc metal with hydrochloric acid (left) and phosphoric acid (right).

OBJECTIVES

- To become familiar with the chemical properties of acids and bases.
- To develop the concept of pH and approximate the pH of common acids, bases.

The following techniques are used in the Experimental Procedure

TECHNIQUES



Many of the chemical compounds that you will encounter in the laboratory (and in lecture) can be classified as an acid, a base, or a salt. The nomenclature and the formulas of these compounds were derived systematically in Dry Lab 2. Using the correct formulas in a chemical equation allows chemists to express chemical reactions and chemical systems that are simply understood without writing extensive paragraphs. *Balanced* equations lead to an understanding of stoichiometry.

INTRODUCTION

However, names, formulas, and equations have little meaning unless there is some tangible relationship to chemicals and chemical reactions. To a chemist, sulfur is not simply an element with the symbol S that reacts with oxygen to form sulfur dioxide, but rather a yellow solid that can be held in the hand and burns in air with a blue flame, producing a choking irritant called sulfur dioxide.

Stoichiometry: a study of a chemical reaction using a balanced equation

In this experiment, you will observe some of the chemical properties and reactions of acids, bases, and salts. What does it mean when a substance is identified as being acidic? basic? soluble? insoluble?

Acids

Acidic solutions have a sour or tart taste, cause a pricking sensation

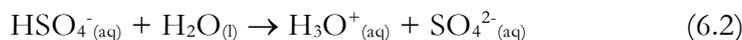
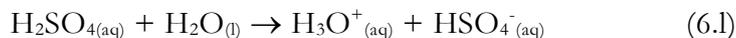
Litmus: a common laboratory acid-base indicator

Since H_3O^+ is a H^+ attached to a water molecule, $H_2O.H^+$, 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.

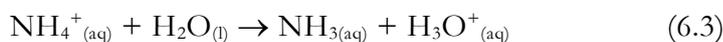
on the skin, and turn blue litmus red. Nearly all of the foods and drinks we consume are acidic ... think of lemon juice as being quite acidic to taste but milk not quite so. All acids are substances that produce hydronium ion, H_3O^+ , in aqueous solutions. For example, the most versatile of all chemicals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



Other prominent "inorganic" acids are hydrochloric acid (also called muriatic acid), nitric acid, and phosphoric acid (Figure 6.1).

Some common "organic" acids are acetic acid found in vinegar, citric acid found in citrus fruits, and ascorbic acid, the vitamin C acid (Figure 6.2).

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



Bases



Figure 6.1 Phosphoric acid that delivers the tart taste in many soft drinks.

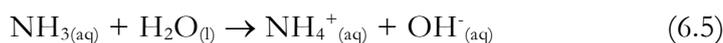


Figure 6.2 Common household acids.



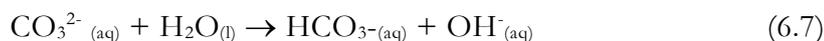
Figure 6.3 Common household bases.

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 ammonia. All bases are substances that produce hydroxide ion, OH^- , in aqueous solutions. For example, the most common base is ammonia (the #2 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, called milk of magnesia, is an antacid and purgative (Figure 6.3).

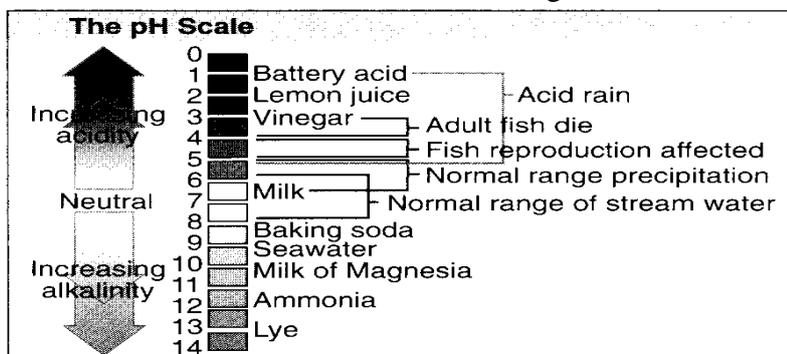
A large number of anions, such as carbonate and phosphate ions, also are capable of producing basic solutions as well. For example, sodium carbonate, known as soda ash and washing soda (often added to detergents), produces OH^- in solution.



The acidity of most aqueous solutions is frequently the result of low concentrations of hydronium ion, H_3O^+ . "pH" is a convenient mathematical expression² used to express low concentrations of hydronium ion. pH is defined as the negative logarithm of the molar concentration of hydronium ion.

$$\text{pH} = -\log[\text{H}_3\text{O}^+] \quad (6.12)$$

At 25°C, pure (and neutral) water has a hydronium concentration (and a hydroxide concentration) of 1×10^{-7} mol/L: $[\text{H}_3\text{O}^+] = 1.0 \times 10^{-7}$ pH = 7.0. Solutions having higher concentrations of hydronium ion, (e.g., 1.0×10^{-2} mol/L) have lower pH's (pH = 2.0). Solutions having lower hydronium concentrations or higher hydroxide concentrations (e.g., $[\text{H}_3\text{O}^+] = 1 \times 10^{-10}$ mol/L) have higher pH's (pH = 10.0). The pH ranges of some familiar solutions are shown in Figure 6.5.



The pH of solutions is often measured with an acid-base indicator. Litmus being red in an acidic solution and blue in a basic solution is one of the more common laboratory indicators. Different indicators change colors over different pH ranges. Mixtures of acid-base indicators can also be used to approximate the pH of a solution. Various pH test papers (Figure 6.6) and the universal indicator are examples of mixed indicators.

pH

[]: Brackets placed around an ion indicate the molar concentration of that ion.

$$\text{pH} = -\log[1 \times 10^{-7}] = 7.0.$$



Figure 6.6 The pH of a solution can be estimated with pH paper, a strip of paper that is impregnated with a mixture of acid-base indicators. A gradation of colors on the pH paper approximates the pH of the solution.

EXPERIMENTAL PROCEDURE

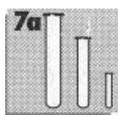


Procedure Overview: The chemical properties of a range of acids, bases, and salts are observed. Chemical (ionic and net ionic) equations are used to account for the observations. The pH of selected acids, bases, and salts are estimated with pH test paper or universal indicator.

Perform the experiment with a partner. At each circled superscript D_n in the procedure, *stop*, and record your observation on the Report Sheet. Discuss your observation with your partner and write an equation expressing your observation. Caution: *Dilute and*

concentrated (*conc*) acids and bases cause severe skin burns and irritation to mucous membranes. Be very careful in handling these chemicals. Clean up all spills immediately with excess water, followed by a covering of baking soda, NaHCO_3 . Refer to the Laboratory Safety section at the beginning of this manual.

A. Acids and Acidic solution



1. Action of Acids on Metals. a. Place a small (~1 cm) polished (with steel wool or sandpaper) strip of Mg, Zn, and Cu into separate small test tubes. To each test tube add just enough 6 M HCl to submerge the metal and observe for several minutes. Record your observations on the Report Sheet

b. Repeat the test of the three metals with 6 M H_3PO_4 and then again with 6 M CH_3COOH

2. Effect of Acid Concentration on Reaction Rate. Set up six small clean test tubes containing about 1/2 mL of the acid solutions shown in Figure 6.7. Add a small (~1 cm) polished strip of magnesium to each solution and observe. Explain your observations. Account for any similarities and differences between HCl and CH_3COOH . What effect do differences in the acids *and* the concentrations of the acids have on the reaction rate?

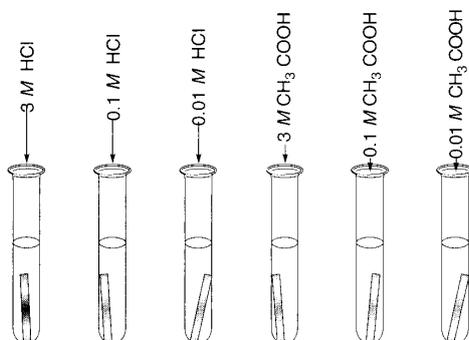


Figure 6.7 A setup for testing the effect of different acids and acid strengths on their reactivity with a meta

1. Reaction of Aqueous Sodium Hydroxide with Acid. Place about 1 mL of 1 M NaOH in a small test tube. Test the solution with litmus. Add and count drops of 6 M HCl-after each drop, agitate the mixture and test the solution with litmus until the litmus changes color. Record your data.
2. Dissolution of Sodium Hydroxide. Place a small "BB-sized" sample of NaOH in a clean small test tube. Hold the test tube in your hand containing the NaOH to detect a heat change and add drops of water to the sample. Test the solution with litmus paper and record your observations.
3. Dissolution of Sodium Carbonate. Place a small "BB-sized" sample of anhydrous Na_2CO_3 in a clean small test tube. Hold the test tube in your hand containing the Na_2CO_3 and add drops of water to the sample. Test the solution with litmus paper and record your observations.

B. Basis and Basic Solutions



Anhydrous salt: a salt having no hydrated water molecules in its solid structure



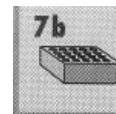
Disposal: Discard the Base solutions in the "Waste Bases" container.

CLEANUP: Rinse all of the used test tubes twice with tap water and twice with deionized water.

Boil 10-20 mL of deionized water for about ~5 minutes to expel any dissolved gases, specifically carbon dioxide. Set the water aside to cool.

1. **pH of Water.** Clean a 24-well plate (small test tubes may also be used) with soap and water and *thoroughly* rinse with deionized water. Half-fill wells A1-A3 with boiled (but cooled) deionized water, unboiled deionized water, and tap water, respectively. Add 1-2 drops of universal indicator to each. Account for any differences in the pH.
2. **Estimate the pH of Acids and Bases.** Place 1 mL of each solution listed on the Report Sheet into wells B1-B4 and add 1-2 drops of universal indicator to each. Estimate the pH of each solution. Write an equation showing the origin of the free H_3O^+ or OH^- in each solution.
3. **Estimate the pH of "Common" Solutions.** By the same method as in Part C.2, use wells C1-C4 to determine the pH of vinegar, lemon juice, household ammonia, detergent solution, or substitutes designated by your instructor.

C. pH Measurements



1 Also called oil of vitriol, sulfuric acid production (#1 produced chemical worldwide) exceeds the #2 produced chemical (ammonia) by a margin of ~3:1 in the United States annually. For that reason sulfuric acid is often called the "old horse of chemistry."

² Sgren P. L. Sgrensen, a Danish biochemist and brewmaster is credited for instituting this expression.

Experiment 4 Report Sheet

Acids And Bases

Name _____

A. Acids and Acidic Solutions

1. **Action of Acids on Metals.** In the following table, state whether or not a reaction is observed. Indicate the relative reaction rate (i.e., fast, moderate, slow) of the metals with the corresponding acid.

	Mg	Zn	Cu
6 M HCl			
6 M H ₃ PO ₄			
6 M CH ₃ COOH			

2. **Effect of Acid Concentration on Reaction Rate.** Explain how changes in HCl concentration affect the reaction rate with Mg.

Explain how changes in CH₃COOH concentration affect the reaction rate with Mg.

Discuss the similarities and differences of HCl and CH₃COOH in the observed reactions with magnesium.

What effect do concentration changes have on the reaction rate?

B. Bases and Basic Solutions

1. **Reaction of Aqueous Sodium Hydroxide with Acid.** On the basis of your observations of the litmus test, describe the progress of the reaction of NaOH with HCl.
2. **Dissolution of Sodium Hydroxide.** Describe your observations.
3. **Dissolution of Sodium Carbonate.** Describe your observations. Write an equation that accounts for your observations.

C. pH Measurements

Water	Approximate pH	Briefly account for the pH if not equal to 7
Boiled, deionized		
Unboiled, deionized		
Tap		

Solution	Approximate pH	Balanced equation showing acid ity/basicity
0.010 M HCl		
0.010 M CH ₃ COOH		
0.01 M NaOH		
0.010 M NH ₃		
Vinegar		

Laboratory Questions

Circle the questions that have been assigned.

1. Part A.1. The observation for the reaction of 6 M HCl was obviously different from that of 6 M CH₃COOH. What were the contrasting observations? How do the two acids differ? Explain.
2. Part A.2. As the molar concentration of an acid decreases, the reaction rate with an active metal, such as magnesium, is expected to ----- . Explain.
3. Part B.1. 6 M H₂SO₄ is substituted for 6 M HCl. Will more or less drops of 6 M H₂SO₄ be required for the litmus to change color? Explain.
4. Part B.3. Sodium carbonate dissolved in water produces a basic solution. Water-soluble potassium phosphate, K₃PO₄, also produces a basic solution. Write an equation that justifies its basicity.
5. Part B.3. The setting of mortar is a time-consuming process that involves a chemical reaction of quicklime, CaO, with the carbon dioxide and water of the atmosphere, forming CaCO₃ and Ca(OH)₂ respectively. Write the two balanced equations that represent the setting of mortar.
6. Part C.1. The unboiled, deionized water has a measured pH less than seven. Explain.



Experiment 5

ANTACID ANALYSIS

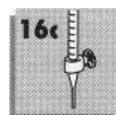
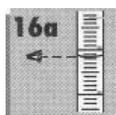
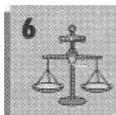
All antacids, as weak bases, reduce the acidity of the stomach.

- To determine the neutralizing effectiveness per gram of a commercial **Antacid**.

OBJECTIVES

The following techniques are used in the Experimental Procedure

TECHNIQUES



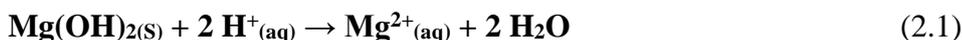
Various commercial antacids claim to be the "most effective" for relieving acid indigestion. All antacids, regardless of their claims or effectiveness have one purpose: to neutralize the *excess* hydrogen ion in the stomach to relieve acid indigestion.

INTRODUCTION

The acidity (and basicity) of an aqueous solution is often expressed in terms of its **pH**.¹ Acidic solutions have a pH less than seven with the lower values being the more acidic; basic solutions have a pH greater than seven with the higher values being more basic. Further experimental studies regarding the understanding of pH are presented in later experiments.

The negative logarithm of the molar concentration of the hydrogen ion is pH
 $pH = -\log[H^+]$

The pH of the stomach ranges from 1.0 to 2.0; acid indigestion and heartburn generally occur at a lower pH. Milk of magnesia, an aqueous suspension of magnesium hydroxide, $Mg(OH)_2$, and sodium bicarbonate, $NaHCO_3$, commonly called baking soda, are simple antacids (and thus, bases) that neutralize hydrogen ion, H^+ :



The release of carbon dioxide gas from the reaction of sodium bicarbonate with hydrogen ion (Equation 2.2) causes one to "belch".

The more common, "faster relief" commercial antacids that buffer excess acid in the stomach are those containing calcium carbonate, $CaCO_3$, and/or

A buffer system resists large changes in the acidity of a solution

sodium bicarbonate. A $\text{HCO}_3^- / \text{CO}_3^{2-}$ **buffer system**² is established in the stomach with these antacids.



RoloidsTM, containing dihydroxyaluminum sodium carbonate, $\text{NaAl}(\text{OH})_2\text{CO}_3$, is a combination antacid that reacts with hydrogen ion, releasing bicarbonate ion, HCO_3^- .



In this experiment, the total effectiveness of several antacids is determined using a strong acid-strong base titration. To avoid the possibility of a buffer system from being established, an excess of hydrochloric acid, HCl, is added to the dissolved antacid solution, shifting the reaction in Equation 2.4 to the right. The solution is heated to drive off the excess carbon dioxide. The excess HCl is titrated with a standardized sodium hydroxide, NaOH, solution³. This-technique is referred to as a "**back**" titration.

The total number of equivalents⁴ of the antacid in the commercial sample plus the number of equivalents of NaOH used in the titration equals the number of equivalents of HCl added.

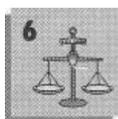
$$\text{equivalents}(\text{antacid}) + \text{equivalents}(\text{NaOH}) = \text{equivalents}(\text{HCl}) \quad (2.6)$$

A rearrangement of the equation provides the equivalents of antacid in the sample.

$$\text{equivalents}(\text{antacid}) = \text{equivalents}(\text{HCl}) - \text{equivalents}(\text{NaOH}) \quad (2.7)$$

The equivalents of antacid per gram of antacid provides the data required for a comparison of the antacid effectiveness of commercial antacids.

EXPERIMENTAL PROCEDURE



A. Dissolving the Antacid

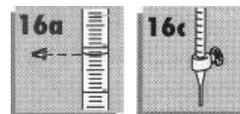
1. Measuring the Mass of Antacid for Analysis. If your antacid is a tablet, pulverize and/ or grind the antacid tablet with a mortar and pestle. Weigh (± 0.001 g) approximately 0.7 g of the pulverized commercial antacid tablet (or 0.7 g of a liquid antacid) in a previously weighed 250-mL Erlenmeyer flask.

2. Preparation of the Antacid for Analysis. Pipet 50.0 mL of 0.1 N HCl into the flask and swirl.⁵ Record the actual normality of the HCl on the Report Sheet. Heat the solution to a gentle boil and maintain the heat for 1 minute to remove dissolved CO_2 .

Add 4-8 drops of bromophenol blue indicator.⁶ If the solution is blue, pipet an additional 25.0 mL of 0.1 N HCl into the solution and boil again.

B. Analyzing the Antacid Sample

- 1. Prepare the Buret for Titration.** Prepare a *clean* buret. Rinse the clean buret with two 3 to 5 mL portions of a standardized NaOH solution. Record the actual normality of the NaOH on the Report Sheet. Fill the buret with the NaOH solution; be sure no air bubbles are in the buret tip. Wait for 30 seconds, then read and record its initial volume (± 0.01 mL).
- 2. Titrate the Sample.** Titrate the antacid sample solution with the NaOH solution to a blue endpoint. When a single drop of NaOH changes the sample solution from yellow to blue, STOP. Wait for 30 seconds and then read the final volume of NaOH solution in the buret.
- 3. Repeat the Titration of the Same Sample.** Refill the buret and repeat the experiment.
- 4. Analyze Another Antacid.** Perform the experiment, in duplicate, for another antacid. Record all data on the Report Sheet.



Disposal: Dispose of the test solutions as directed by your instructor.



¹ The negative logarithm of the molar concentration of the hydrogen ion is $\text{pH} : -\log[\text{H}^+] = \text{pH}$.

² A buffer system resists large changes in the acidity of a solution. To analyze for the amount of antacid in this experiment we want to remove this buffering property in order to determine the total effectiveness of the antacid. To do this, we "swamp" the system with an excess of strong acid, HCl, and then titrate with a strong base, NaOH, to determine the amount of strong acid that was not neutralized by the antacid (base).

³ A standardized NaOH solution is one in which the concentration of NaOH has been very carefully determined (for this experiment) by the stockroom personnel.

⁴ An equivalent (abbreviated "eq.") of any substance is merely an expression of its amount present in a system. For a soluble substance, its number of equivalents present in solution is found by multiplying its volume (in liters) by its normality, N (equivalents/liter, abbreviated eq/L)

⁵ If the sample is a tablet, swirl to dissolve. Some of the inert ingredients of the antacid tablet may not dissolve.

⁶ Bromophenol blue is yellow in an acidic solution and blue in a basic solution.

Experiment 5 Prelaboratory Assignment

ANTIACID ANALYSIS

Date _____ Lab Sec. _____ Name _____ Desk No. _____

1. Write the balanced equation for the reaction of one mole of the active ingredient in RoloidsTM with an *excess* of hydrogen ion. (Hint: consider Equations 2.4 and 2.5)

2. a. Write a balanced equation representing the antacid effect of sodium citrate, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$. Citric acid is a weak triprotic acid, $\text{H}_3\text{C}_6\text{H}_5\text{O}_7$.

b. Is sodium citrate more or less effective per mole than $\text{Mg}(\text{OH})_2$? per gram of $\text{Mg}(\text{OH})_2$? Show calculations.

3. a. In your text find the color range of the pH change for bromophenol blue.
What is its color in an acidic solution?
What is its color in a basic solution?

b. Describe the color change that occurs at the endpoint in this experiment.

4. Why do some antacids cause gas to accumulate in the stomach? What is the gas?

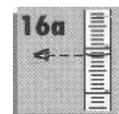
5. A 50-mL volume of 0.104 N HCl is added to a sample of an unknown base. The HCl *not neutralized*

(the excess HCl) by the base is titrated to a bromophenol blue endpoint with 10.7 mL of 0.0841 N NaOH. How many equivalents of unknown base (antacid) are present in the original sample?

6. How much time should be allowed for the titrant to drain from the buret wall before a reading is made ?



7- What criterion is followed in reading and recording the volume of titrant of a buret ?



Experiment 5 Report Sheet

ANTIACID ANALYSIS

Date _____ Lab Sec. _____ Name _____ Desk No. _____

Commercial Antacid

	Trial 1	Trial 2
1. Mass of flask + antacid (g)
2. Mass of flask (g)
3. Mass of antacid (g)
4. Volume of HCl added (mL)
5. Normality of HCl (eq/L)	
6. Normality of NaOH (eq/L)	
7. Buret reading, final (mL)
8. Buret reading, initial (mL)
9. Volume of NaOH (mL)

Calculations

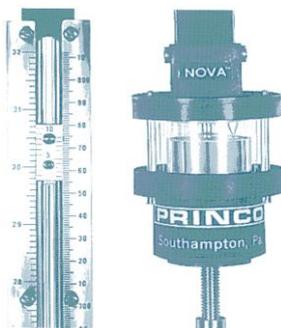
1. Eq. of HCl added, total
2. Eq. of NaOH added
3. Eq. of antacid
4. Eq. per gram of antacid
5. Ave. eq. of antacid/gram (eq/g)	

Laboratory Questions

1. If the CO_2 is not removed by boiling after the 0.1 N HCl is added, how does this affect the amount of NaOH required to reach the bromophenol blue endpoint? Explain. (Hint: CO_2 produces a low concentration of carbonic acid, H_2CO_3 , when dissolved in water.)

2. If the results from Trial 1 and 2 differ by a substantial amount ($\geq 5\%$), what would you do before presenting your data to the laboratory instructor?

3. If the antacid selected for analysis is known to contain *only* milk of magnesia, how could the Experimental Procedure be modified to expedite the analysis?



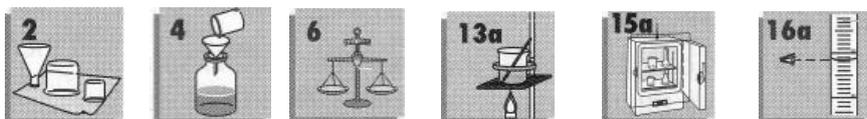
The mercury barometer accurately measures atmospheric pressure in mmHg (or torr).

Experiment 6

FORMULA MASS OF A VOLATILE LIQUID

- To determine the formula mass (molecular weight) of a volatile liquid **OBJECTIVES**

The following techniques are used in the Experimental Procedure **TECHNIQUES**



The measurement of the formula 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 formula mass of their compounds.

INTRODUCTION

In this experiment the formula 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.

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

The formula mass of the compound, FM_{compound} , is then calculated from the data.

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

Gases and liquids that have relatively large intermolecular forces and large molecular volumes do *not* calculate according to the ideal gas law equation; in fact, some compounds that we normally consider as liquids deviate significantly from ideal gas behavior at temperatures at or slightly above their boiling points. At these conditions, the intermolecular force and molecular volume corrections can be considered in the determination of the formula mass of the compound by using a modification of the ideal gas law equation; this is called the van der Waal's equation.

$$\left(P + \frac{n^2a}{V^2}\right)(V - nb) = nRT \quad (3.4)$$

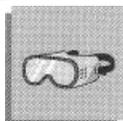
In this equation, P, V, T, R, and n have the same meanings as in Equation 3.1. *a* is an experimental value that is representative of the intermolecular forces of the vapor and *b* is an experimental value that is representative of the volume of the molecules.

For a more accurate determination of the moles of vapor, n_{vapor} in the flask, van der Waal's equation can be used instead of the ideal gas law equation. Some values of *a* and *b* for a number of low boiling-point liquids are listed in Table 3.1.

Table 3.1 : Van der Wool's Constants for Some Low Boiling-Point Compounds

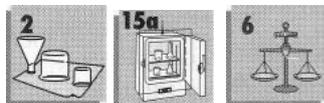
Name	a $\left(\frac{\text{L}^2 \cdot \text{atm}}{\text{mol}^2}\right)$	b (L/mol)
methanol	9.523	0.06702
ethanol	12.02	0.08407
acetone	13.91	0.0994
propanol	14.92	0.1019
hexane	24.39	0.1735
cyclohexane	22.81	0.1424
n-pentane	19.01	0.1460

EXPERIMENTAL PROCEDURE



Procedure Overview : You are to complete three trials in determining the formula mass of your unknown low boiling-point liquid. Initially obtain 15 to 20 mL of liquid from your instructor. The same apparatus is used for each trial.

A. Preparing the Sample



1. Prepare the Flask for the Sample. Fit a *clean*, 125-mL Erlenmeyer flask with a one-hole stopper. Dry the flask either in a drying oven or by allowing it to air-dry. Do *not* wipe dry or heat over a direct flame. Weigh (± 0.001 g) the *dry* flask and the one-hole rubber stopper.

2. Place the Sample in the Flask. Add about 6 mL of the unknown liquid to the flask and insert the stopper. You do *not* need to weigh the liquid and flask. Cover the stopper with a small piece of aluminum foil (Figure 3.1). Secure the aluminum foil with a rubber band and, with a pin, pierce the aluminum foil several times at the point where the foil covers the hole in the rubber stopper.¹



3. Prepare a Boiling Water Bath. Fill a 600-mL beaker half y with water, support it on a wire gauze and near the top, with a support ring. Bring the water to a boil.

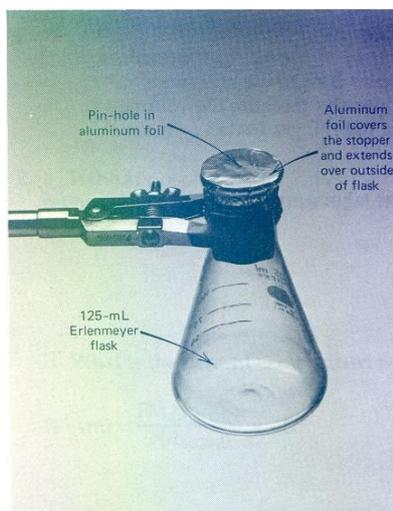


Figure 3.1. Preparation of a flask for the placement of the volatile liquid

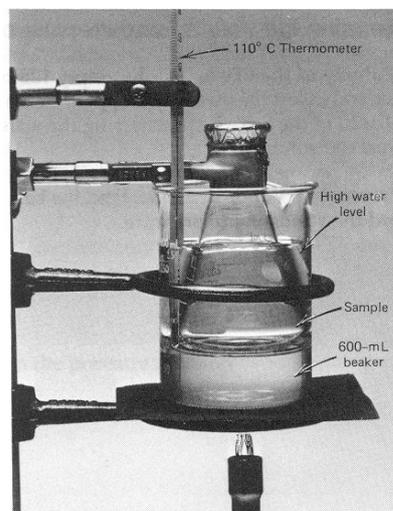


Figure 3.2. Apparatus for determining the formula mass of the volatile liquid

B. Collecting the Data

1. Place the Flask/Sample in the Bath. Remove the heat from the boiling water bath. Lower the flask/sample in the bath and secure it with a utility clamp. Be certain that neither the flask or the clamp touches the beaker wall. Adjust the water level *high* on the neck of the flask (Figure 3.2).

2. Heat the Sample to the Temperature of Boiling Water. Resume heating until the water boils. (**Caution:** *most unknowns are flammable. Use a moderate flame for heating.*) When the liquid in the flask or the vapors from the holes in the aluminum foil are no longer visible, continue heating for another 5 minutes. Record the temperature ($\pm 0.01^\circ\text{C}$) of the boiling water.



3. Measure Mass of Flask/Sample. Remove the flask and allow it to cool to room temperature. Sometimes the remaining vapor in the flask condenses. That's O.K.



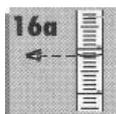
Dry the outside of the flask and weigh (± 0.001 g, use the same balance!) it, the rubber stopper, and the vapor. (Don't forget to remove the aluminum foil before weighing!)

4. Do It Again and Again. Repeat the experiment for Trials 2 and 3.



Disposal : Dispose of the leftover unknown liquid in the "Waste Liquids" container.

C. Determine the Volume and Pressure of the Vapor ;,



1. Volume of the Flask. Fill the empty 125-mL Erlenmeyer flask to the brim with water and insert the one-hole stopper. Remove the stopper and measure the volume (± 1 mL) of the flask by transferring the water to a 50 or 100-mL graduated cylinder. Record the volume.

2. Pressure of Vapor in Flask. Find the barometer in the laboratory. Read and record the atmospheric pressure.

¹ The aluminum foil should cover the stopper and the outside of the flask. Do not insert the foil inside the flask.

Experiment 6 *Prelaboratory Assignment*

FORMULA MASS OF A VOLATILE LIQUID

Date _____ Lab Sec. _____ Name _____ Desk No. _____

1. A mass of 0.777 grams of an unknown vapor occupies 314 mL at 98.7°C and 740 torr. Assume ideal gas behavior.

a. How many moles of vapor are present?

b. What is the formula mass of the vapor?

2. a. If the atmospheric pressure is mistakenly recorded as 760 torr in Question 1, what is the reported formula mass of the vapor?

b. What is the percent error caused by the error in the pressure reading?

$$\% \text{ error} = \frac{\text{FM}_{\text{difference}}}{\text{FM}_{\text{actual}}} \times 100$$

3. Explain how the mass of the vaporized unknown is determined in this experiment.

4. a. How many trials are to be completed in this experiment?

b. What volume of unknown liquid should be obtained from your instructor?

5. The formula mass of a compound is measured to be 43.7, 42.9, 43.3, and 43.0 in four trials.
- What is the average formula mass of the compound?
 - Calculate the standard deviation (see **Appendix B**) for the determination of the formula mass.
6. Rearrange van der Waal's equation, placing all n terms on the same side of the equation. (Hint: the result is a cubic equation!)

Experiment 6 *Report Sheet*

FORMULA MASS OF A VOLATILE LIQUID

Date _____ Lab Sec. _____ Name _____ Desk No. _____

Unknown Number	Trial 1	Trial 2
1. Mass of dry flask and stopper (g)
2. Temperature of boiling water ($^{\circ}\text{C}$, K)
3. Mass of dry flask, stopper, and vapor (g)
4. Volume of 125-mL flask (L)	
5. Atmospheric pressure (atm)	

Calculations

1. Moles of vapor, n_{vapor} (mol)
2. Mass of vapor, m_{vapor} (g)
3. Formula mass of compound (g/mol)
4. Average formula mass	
5. Standard deviation of formula mass	

*Calculation of Trial 1. Show work here.

Laboratory Questions

Circle the questions that have been assigned.

1. If the outside of the flask is *not* dried after vaporizing the liquid, will the formula mass of the compound be too high or too low? Explain.
2. a. Suppose you assumed that the atmospheric pressure was one atmosphere instead of the pressure that you recorded. Would this have caused you to report a formula mass that was too high or too low? Explain.

b. What would have been the percent error in your formula mass had this occurred?
3. If the volume of the vapor had been assumed to be 125 mL, instead of your measured volume, what would have been the percent error in your reported formula mass of the compound? Explain. Show your work.
4. If all of the unknown liquid does *not* vaporize in the 125-mL flask, will the reported formula mass be too high or too low? Explain.
5. Suppose the thermometer is miscalibrated and reads 0.3°C higher than actual. Does this affect the reported formula mass of the compound? If so, will the reported formula mass be too high or too low? Explain.

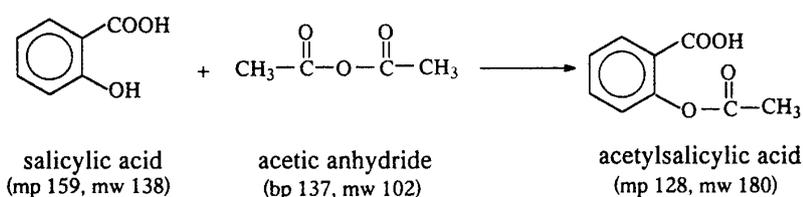
Experiment 7

Preparation Of Aspirin

Aspirin (acetylsalicylic acid) is a widely used drug in modern society. It is an analgesic (pain killer), a powerful antipyretic (fever-reducing), and an anti-inflammatory (swelling-reducing) substance.

Salicylic acid (which is a constituent of certain plants) is itself an analgesic and was originally administered as sodium salicylate. Since salicylic acid has an irritating effect on the stomach lining, chemists sought a modification that would retain its properties while decreasing the adverse side effects. Conversion to the ester satisfied this requirement and acetylsalicylic acid (aspirin) proved to be as effective as sodium salicylate without the irritation typical of phenolic compounds. Aspirin gets, however, hydrolyzed to salicylic acid in the alkaline medium of the intestines.

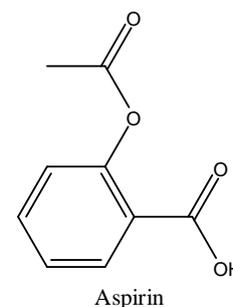
Aspirin is obtained from salicylic acid by acetylation with acetic anhydride in the presence of sulfuric acid as a catalyst. Because of its low solubility in water (0.25g/100mL), it is isolated from the reaction mixture by precipitation with water.



The name aspirin is derived from the spirea willow tree, the bark of which was used in the Middle Ages as a medicine. In the 1600s, extracts of this bark were shown to have fever-reducing properties. In 1826, the active principle, salicylic acid, was identified and by 1874 it was in large-scale production.

Salicylic acid caused severe gastric irritation and so the sodium salt (sodium salicylate) was tried with little more success. Salol, introduced in 1886, caused less gastric irritation but because it was hydrolyzed to salicylic acid and phenol in the small intestine, phenol poisoning was a danger

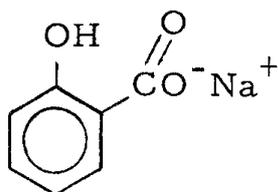
INTRODUCTION



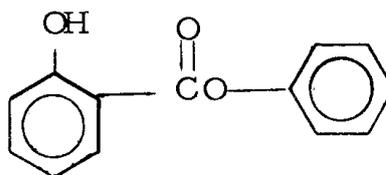
Aspirin is analgesic (pain killer), a powerful antipyretic (fever-reducing) and an anti-inflammatory (swelling-reducing) substance.

The name aspirin is derived from the spirea willow tree

Salicylic acid causes severe gastric irritation



Sodium salicylate



Salol

*Felix Hofmann
discovered the
medicinal properties
of aspirin.*

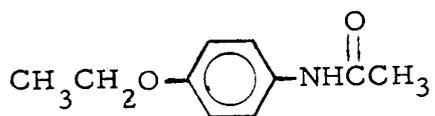
*aspirin
is an organic acid (-
COOH is an organic
acid group)*

Toward the end of the nineteenth century, Felix Hofmann, who worked for the Bayer Company, discovered the medicinal properties of aspirin by experimenting on his father, a severe arthritic. Today, with so many brands of aspirin on the market, a consumer can really be in a quandary as to which to purchase. Consumers Union's The Medicine Show, 1970, states that "the only significant difference among brands of aspirin, plain or buffered, is price."

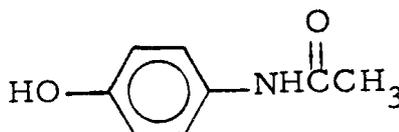
Aspirin is frequently combined with other ingredients as a combination of pain relievers. Since aspirin is an acid (-COOH is an organic acid group), there are some people who suffer from mild gastric

irritation, especially when taking aspirin on an "empty" stomach. Thus, several buffered aspirins containing antacids are on the market.

Two pain relievers sold individually or in combination with aspirin, comparable to aspirin in effect, are *phenacetin* and *acetaminophen*.

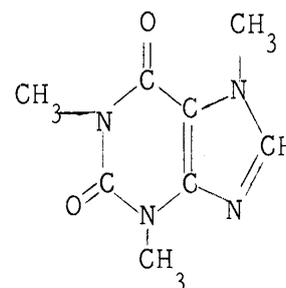


Phenacetin



Acetaminophen

These are both used by individuals who experience unpleasant reactions to aspirin. Phenacetin has been tentatively implicated in kidney damage and has been removed from several combination pain relievers. More recently, it has been found that large overdoses of acetaminophen can cause serious liver damage. APC tablets, thought by many to be a more powerful combination than plain aspirin, contain aspirin, phenacetin, and caffeine.

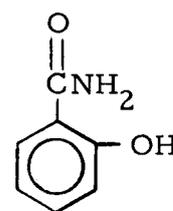


Caffeine

The AMA Drug Evaluations (1971) sees no sound reason for combining aspirin and phenacetin since both have essentially the same effect. The amount of caffeine in leading formulations is less than the amount in a cup of coffee and there is no reliable evidence that caffeine aids in the effect of aspirin.

A final substance used in some combination pain relievers is salicylamide. It is much less effective than aspirin and too weak and unreliable to be useful alone.

In this experiment, aspirin will be synthesized from salicylic acid and acetic anhydride.



Salicylamide

Experimental

Materials Needed

Glassware: 100 ml Erlenmeyer flask, Buchner funnel, filter flask, graduated cylinder (10 ml), 2 test tubes.

Chemicals: 5.0g Salicylic acid, 5 ml acetic anhydride, 4.0 ml of glacial acetic acid, 1mL concentrated sulfuric acid, ice.

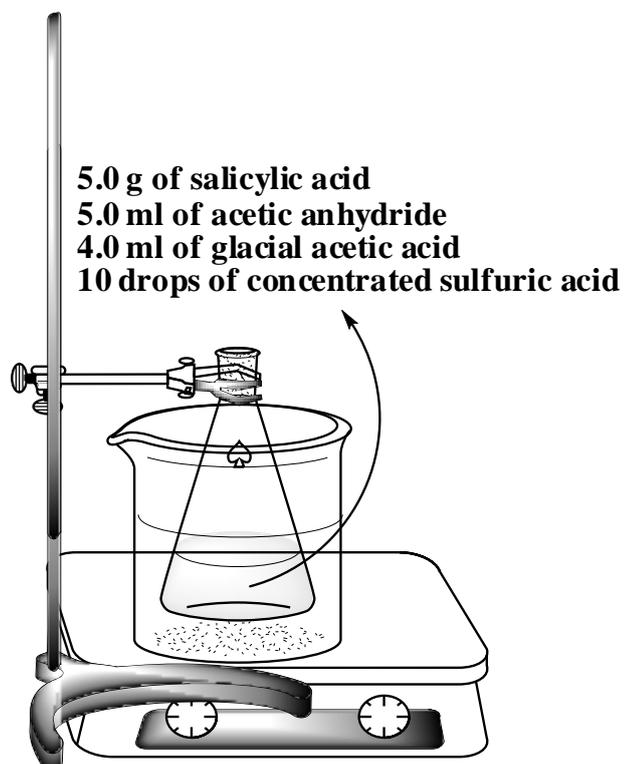
Experimental Procedure

Fill a 400-ml or a 600-ml beaker half full of water and heat to boiling. Place 5.0 g of salicylic acid in a 125-ml Erlenmeyer flask. Add to this in the following order: 5.0 ml of acetic anhydride, 4.0 ml of glacial acetic acid, and 10 drops of concentrated sulfuric acid. Place the flask in the boiling water and rotate gently until the salicylic acid goes into solution. Remove the flask and *pour 40 ml of hot water into the flask*. Heat to boiling with a burner and allow to boil for 3 to 5 min, or long enough to wash down the sides of the flask.

Place the Erlenmeyer flask on an asbestos square and allow it to crystallize undisturbed for 20 min. Then cool the flask in an ice bath for at least 15 min to promote further crystallization.

Suction-filter the crystallized aspirin. Allow to dry in your desk until the next lab period. Weigh the product and determine the melting point. Calculate the percent yield.

**The aspirin is reasonably pure as is but could be recrystallized from water.*



Experiment 7 *Prelaboratory Assignment*

Preparation Of Aspirin

Name : Section :

1. Which organic reaction represent the preparation of Aspirin (*just the name of the reaction*)
2. In this experiment (preparation of aspirin), where can you find an application (*example*) of LeChâtelier's Principle?
3. Write an equation showing the preparation of each of the following from salicylic acid.
 - (a) Sodium salicylate.
 - (b) Salol.
 - (c) Salicylamide.
 - (d) Methyl salicylate, oil of wintergreen.
4. Using condensation reactions, write equations showing the preparation of the following.
 - (a) Phenacetin.
 - (b) Acetaminophen.

Experiment 7 *Report Sheet*

Preparation Of Aspirin

Name : Section :

Preparation of Aspirin (*Reaction Equation*) :

1. Volume of acetic anhydride (ml) _____
2. Density of acetic anhydride (g/ml) _____
3. Mass of acetic anhydride (g) _____
4. Mass of Salicylic acid (g) _____
5. Mole of acetic anhydride _____
6. Mole of Salicylic acid (mol) _____
7. **Limiting Reactant is** _____
8. Moles of Aspirin (mol) (*Theoretical*) _____
9. **Mass of Aspirin (Theoretical yield) (g)** _____
10. Mass of filter paper (g) _____
11. Mass of filter paper and Aspirin (g) _____
12. **Mass of Aspirin (g) (*Actual Yield*)** _____
13. **Percentage yield of Aspirin (%)** _____
14. **Melting point of Aspirin:** _____

* Show Calculations :

Laboratory Questions :

1. When aspirin is left standing under moist conditions, it takes on a vinegary odor. Explain. Write an equation.
2. Write an equation showing the hydrolysis of aspirin in the alkaline environment of the small intestine.
3. What was the purpose of the 40 ml of boiling water in this experiment?
4. Examine the labels of several brands of pain relievers (including combination pain relievers) in a drug store. List the ingredients of each brand. Make a cost analysis of aspirin tablets.ere?

Experiment 8

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

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

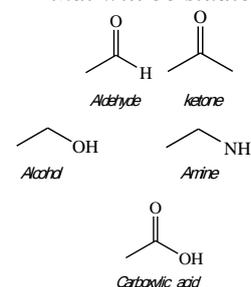
OBJECTIVES

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.

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 has recently come under suspicion as causing leukemia, it will not be used here. It is added along with xylene and toluene to high-octane gasoline.

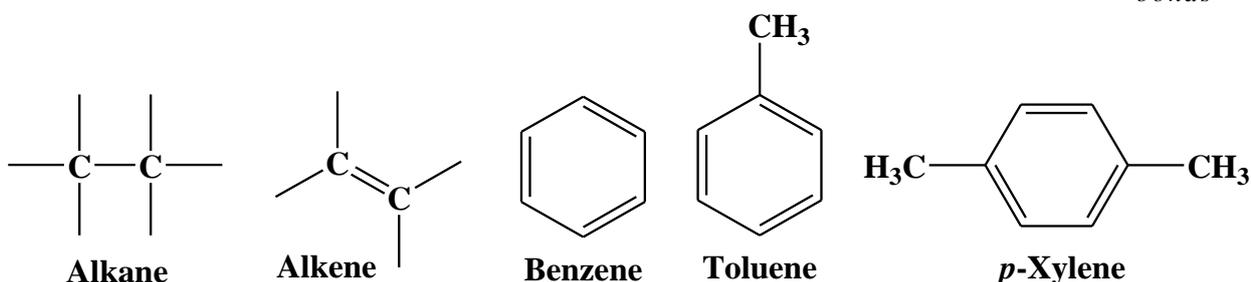
INTRODUCTION

Some of the function groups that will be studied

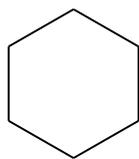


Alkanes are saturated hydrocarbons in which each carbon has a maximum number of bonded atoms (four)

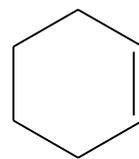
Alkenes are Unsaturated hydrocarbons which have one or more double bonds



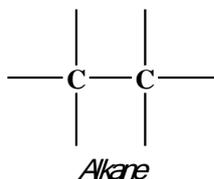
The following compounds will be used in most of the tests described in the procedures.



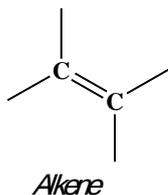
Cyclohexane



Cyclohexene



Alkane



Alkene



Alkyne

Aluminum Chloride

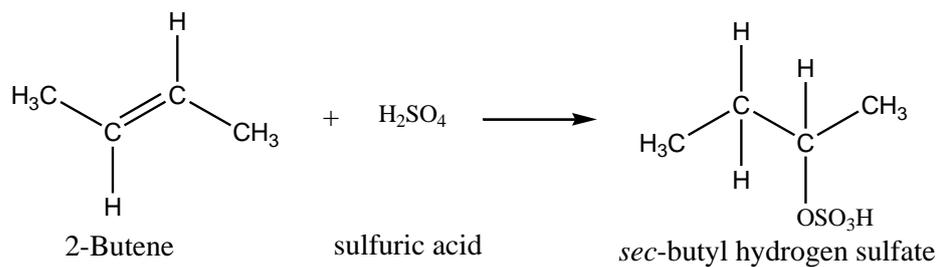
Anhydrous: AlCl₃

Chloroform CHCl₃

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.***

Experimental Procedure

A. Sulfuric Acid (H₂SO₄)



With care, put about 1 ml of concentrated sulfuric acid in a 10 X 75-mm test tube. Add several drops of hydrocarbon to be tested and agitate carefully. Hold the tube with your left thumb and index figure at the very top. Gently strike the bottom of the tube with a downward motion with your right index finger. A little practice, using water, will teach you the technique of mixing without splashing.

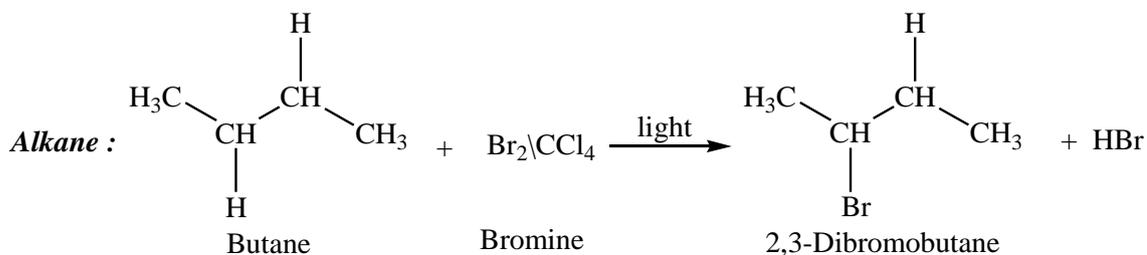
Observe carefully whether or not the hydrocarbon is dissolving. A yellow-to-brown color indicates solution has taken place. The biggest problem is to note whether the hydrocarbon is dissolving or just hiding in the meniscus. Look very carefully, and perhaps add a few more drops of hydrocarbon to be sure. If the material does not dissolve, it is probably an alkane. If it also fails test C it is an alkane.

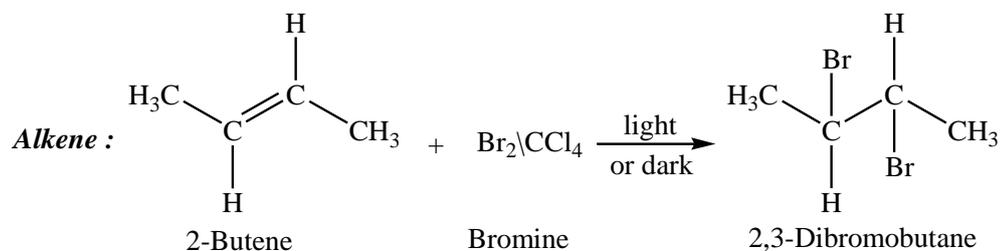
Try this test on cyclohexane, cyclohexene, and toluene.

Caution:
Be careful with handling the concentrated H₂SO₄ because it's skin irritation and burning

Look very carefully The biggest problem is to note whether the hydrocarbon is dissolving or just hiding in the meniscus

B. Bromine in Carbon Tetrachloride (Br₂/CCl₄)





Alkenes decolorize bromine rapidly and completely with a dilute solution of bromine in carbon tetrachloride, even in the dark

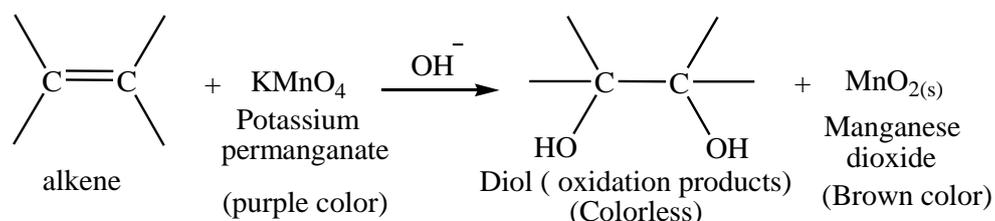
Alkenes decolorize bromine rapidly and completely with a dilute solution of bromine in carbon tetrachloride, even in the dark. Alkanes react very slowly by substitution only in the presence of ultraviolet light.

Put a little (½ mL) of hydrocarbon in a 10 X 75-mm test tube and add the dilute bromine in carbon tetrachloride solution drop by drop. If you add too much solution for the amount of alkenes you have, the extra bromine will color the solution and mask the test. So observe carefully after each drop to determine whether or not the bromine is really adding.

Do the test on cyclohexene and chlorobenzene and chlorobenzene. (If you wish, you may take the cyclohexane and chlorobenzene tubes out in the sunlight, shake them, and observe) HBr is produced in one tube and may be detected by carefully holding a strip of moist blue litmus down in the test tube.

Caution: the extra bromine will color the solution and mask the test so you will not observe the change

C. Potassium Permanganate (KMnO₄⁻), The Baeyer Test



Potassium permanganate will react with double bonds forming diols, followed by more extensive oxidation. It will take place in either acidic or basic solution. In base, the purple color disappears, forming manganese dioxide. If concentrated enough, it will precipitate as a brown flocculent precipitate.

*Potassium Permanganate
KMnO₄*

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

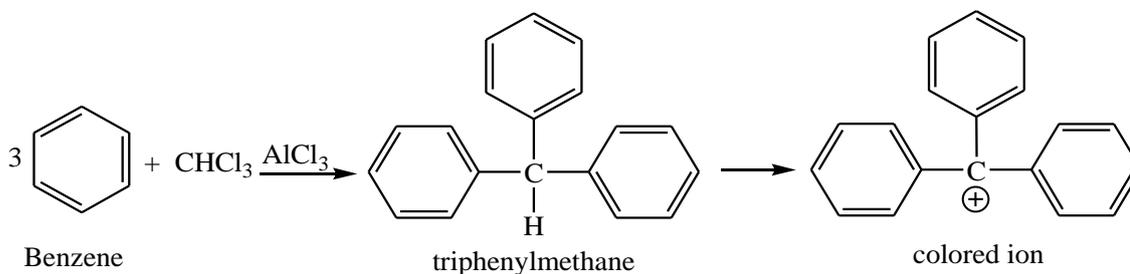


Saturated compounds burn cleanly, while unsaturated ones tend to produce soot.

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. ($AlCl_3/CHCl_3$)



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.

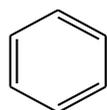
You may wish to do the following test in a hood. Add a small spatula of solid aromatic compound to 1 ml of dry chloroform in a 10 X 75-mm test tube. Get a little anhydrous aluminum chloride on a spatula.

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.

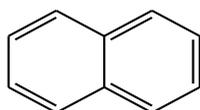
*Caution:
Keep the Cover on
the aluminum
chloride as much
as possible or it
will pick up water
and the test will
fail*

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.*

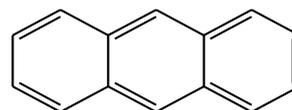
Try the test with one or more solid aromatics such as those suggested. Also try cyclohexane and cyclohexene.



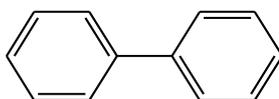
Benzene



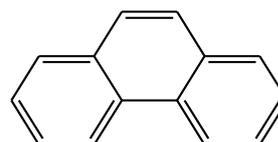
Naphthalene



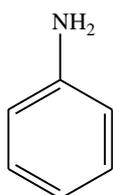
anthracene



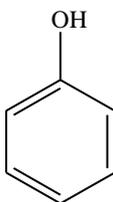
Biphenyl



Phenanthrene



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.

Experiment 8 Prelaboratory Assignment

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

Name : Section :

1. write the chemical structure for the following Organic Compound:

- a) n-Pentane
- b) 2-Pentene
- c) Anthracene
- d) *p*-Chlorotoluene

2. Write an example for each following Organic Functional Group:

- a) Ketone
- b) Aldehyde
- c) Amine
- d) Alcohol
- e) Ester
- f) Carboxylic acid

3. Write the resonance structure for Benzene.

Experiment 8 *Report Sheet*

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

Name : Section :

Test A, B, C, and D

Test	reagent	Observations and Results			
		Alkane	Alkene	Aromatic	
		Cyclohexane	Cyclohexene	Toluene	Chlorobenzene
A.	H ₂ SO ₄				X
B.	Br ₂ /CCl ₄			X	
C.	KMnO ₄			X	
D.	Combustion				X

Test	Positive Test Reaction Equation
A.	
B.	
C.	
D.	

E. Result, AlCl₃/CHCl₃ test with three Aromatic compound.

(Equation and Observation)

1.

2.

3.

F. Results, Bromine water ($\text{Br}_2/\text{H}_2\text{O}$) test with:

(Equation and Observation)

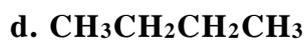
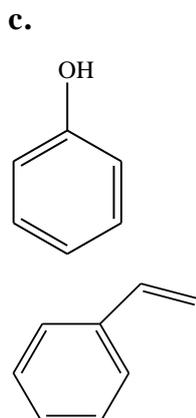
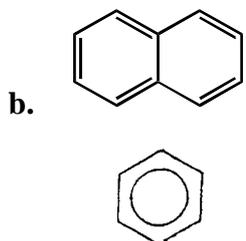
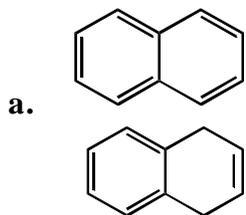
1. Chlorobenzene

2. Phenol

3. Aniline

Laboratory Questions :

1. Give the chemical test that you can distinguish between the following pairs of the compounds.



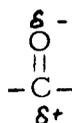
Experiment 9

REACTIONS OF THE FUNCTIONAL GROUPS II: ALDEHYDES, KETONES, AND ALCOHOLS

Aldehydes and ketones are both characterized by the carbonyl group, - **INTRODUCTION**
C=O. Aldehydes have at least one hydrogen bonded to the carbonyl
carbon whereas in ketones, both bonds are to alkyl. or aryl groups.



Aldehydes



Carbonyl group



Ketones

Aldehydes have at least one hydrogen bonded to the carbonyl carbon whereas in ketones, both bonds are to alkyl. or aryl groups.

Two types of chemical behavior will be tested here. First, in both aldehydes and ketones, the carbonyl group reacts well with many reagents to form derivatives. Secondly, aldehydes are readily oxidized by numerous mild oxidizing agents that do not react with ketones. Several special tests that work for methyl ketones will also be tried.

Structurally, alcohols have a hydroxy group bonded (usually) to a saturated carbon.

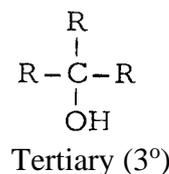
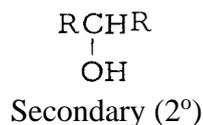
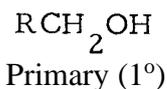


The reactions of alcohols involve the polar carbon-oxygen and oxygen-hydrogen bonds. Alcohols are classified as primary, secondary, or tertiary, and their rates of reaction frequently vary depending on the structure.

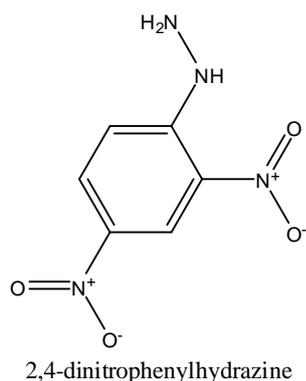
Type of Alcohol

Primary (1°)

Secondary (2°) Tertiary (3°)



Experimental Procedure

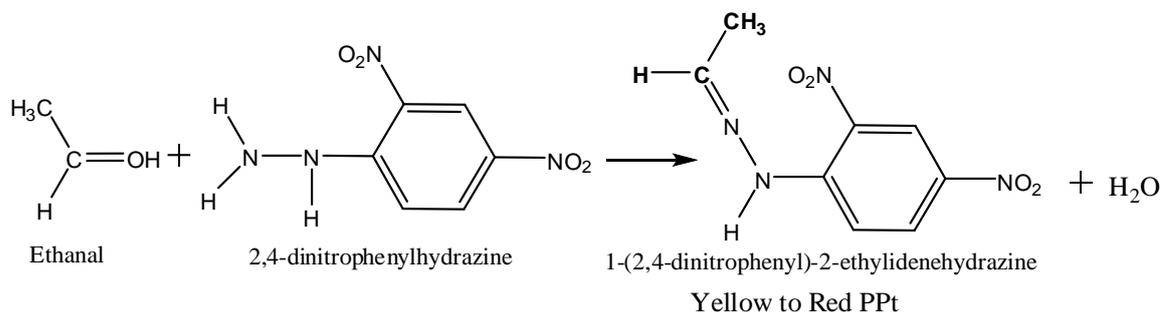


A. 2, 4-Dinitrophenylhydrazine

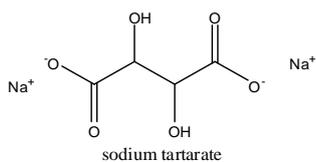
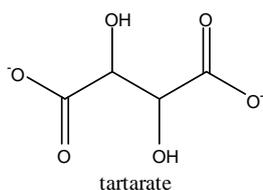
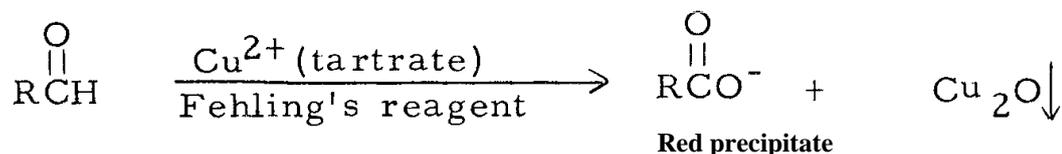
This orange-red solution of 2,4-dinitrophenylhydrazine in sulfuric acid produces an immediate precipitate with most aldehydes and ketones, colored yellow to deep red. Recrystallization and determination of the melting point is a valuable clue to their identity.

Put 1 ml of the 2,4-DNP reagent (careful, it stains!) in a 10 X 75-mm test tube. Add 2 to 3 drops of the compound to be tested and shake carefully.

Try the test with acetone, benzaldehyde, cinnamaldehyde, acetophenone, and cyclohexanol.



B. Fehling's Test

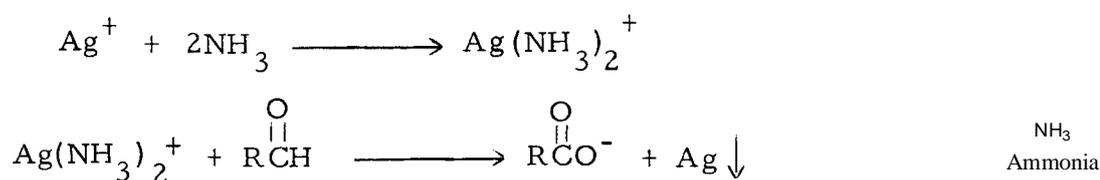


Fehling's solution is an alkaline solution of sodium tartrate, in which copper(II) ion is soluble because of complex formation. (Copper(II) ion is ordinarily insoluble in base.) This solution is a very mild oxidizing agent, sufficiently strong to oxidize aldehydes but not much else. In the reaction, the copper(II) ion is reduced to copper(I) and precipitates as a red solid, the copper(I) oxide. Enough reagent should be made at one time to handle all of your tests. The reagent reacts well at about 60°C. You should, however, place one tube of the reagent without any test compound in the bath to make sure that any positive tests you may get are not simply the decomposition of the reagent. When the reaction is positive, the deep blue solution of the reagent will turn to a murky olive-green suspension and the red precipitate will gradually collect on the bottom of the tube.

Take 5 ml of Fehling's A and 5 ml of Fehling's B and mix. Place 2 ml of this solution in each of three test tubes. Add 2 to 3 drops of the compounds to be tested to each tube and place the tubes in the warm-water bath. Also, place one tube in the bath that has only the reagent in it for a blank. Examine for any change in color or for a red precipitate on the bottom of the tube. Test on aqueous acetaldehyde and acetone

*When the reaction is **positive**, the deep blue solution of the reagent will turn to a murky olive-green suspension and the red precipitate will gradually collect on the bottom of the tube.*

C. Tollens' Test



This test is very similar to Fehling's test above in that Tollens' reagent is an ammonia solution of silver ion that is a very mild oxidizing agent. It produces silver metal, either as a fine black precipitate or as a mirror on the tube!

This test is very similar to Fehling's test

This is how mirrors were made in the past; now they are made by evaporating aluminum metal onto glass. After we test for aldehydes, we shall make a mirror.

1. Test for Aldehydes

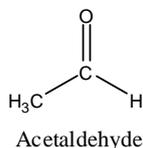
Put 5 ml of 0.1M silver nitrate solution into a test tube. Add dilute ammonia dropwise until the brown precipitate that forms at first almost redissolves. Shake the tube between each drop so that you do not add too much ammonia. Place 1 ml of the solution in a 10 X 75-mm test tube and add 2 drops of the compound to be tested. You may warm it gently, but be careful as you may just decompose the reagent. You may test this by warming a sample of the reagent alone. With an aldehydes, you

Silver nitrate
AgNO₃

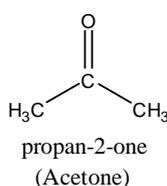
The black precipitate
that formed is
 $Ag_{(s)}$

will observe a fine black precipitate, or if you are lucky and the tube is clean, you may silver the inside of the tube, giving a mirror that you can see from the outside. Try the test on the aqueous acetaldehyde and acetone.

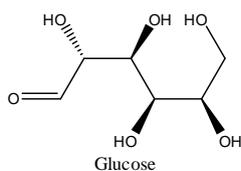
2. Preparation of a Silver Mirror



Thoroughly clean a 90-mm watch glass and a 1 x 3-in. microscope slide by washing them with soap, rinsing with water and then acetone, and allowing to dry. Place 50 ml of water in a 400-ml beaker supported on a tripod or iron ring. Set the watch glass over the open end of the beaker and lay the microscope slide across the watch glass. Have a burner ready to heat the water.

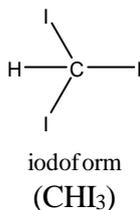


Pour 25 ml of 0.1 M $AgNO_3$ into a small beaker. Add concentrated ammonia dropwise until the dark precipitate that forms after the first drop or so just disappears. Add a spatula (0.2 to 0.3g) of glucose (grape sugar, blood sugar - the main energy source for animals; human blood contains 0.08% to 0.1% glucose). Stir or shake to dissolve the glucose and pour the solution into the watch glass. Bring the water in the 400-ml beaker to a boil. Remove the flame as soon as the first traces of mirror appear. Allow the plating to proceed for 3 to 5 min. With tongs, carefully remove the slide (handle on edges) and rinse with water and then with a little acetone. Place a piece of dark electrical or masking tape on the side with the better silver surface and rub the silver off the other side. Observe the mirror formed on the watch glass. The silver can be removed from the watch glass with a few milliliters of 6 N HNO_3 .



Note:

The silver can be removed from the Glassware with a few milliliters of 6N HNO_3 .

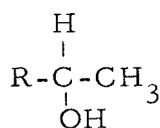


D. Iodoform Test

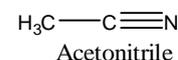
The iodoform test is positive for aldehydes and ketones, which have a methyl group directly connected to the carbon-oxygen double bond. It also is positive for primary and secondary methyl carbinols.



A methyl carbonyl



A methyl carbinol

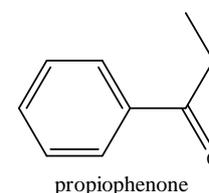
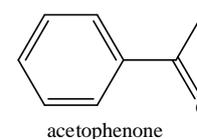
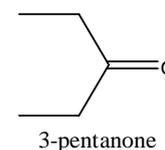
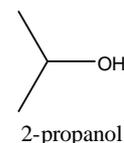
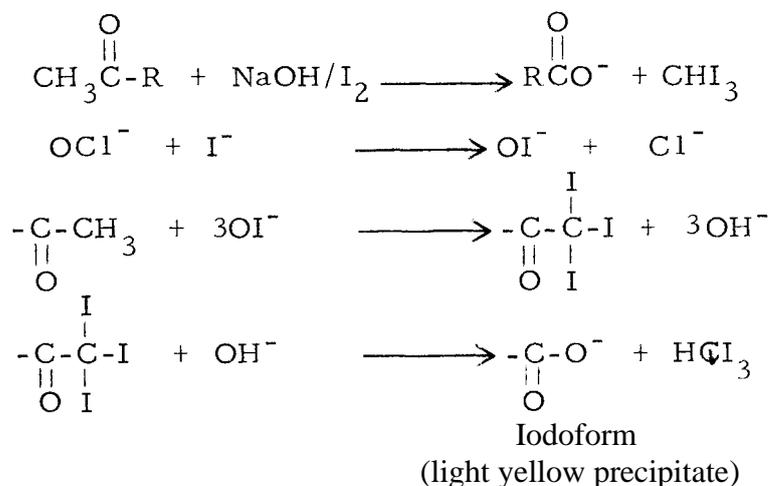


Put 2 to 3 ml of 10% potassium iodide solution (water /acetonitrile) in a 13 X 100-mm test tube Warm briefly on a steam bath or a boiling-water bath. Add 2 drops (no more) of the compound to be tested. Shake, then add 1 to 1.5 ml of Clorox Agitate the solution. Look for the yellow precipitate, iodoform. Iodoform has a characteristic odor. You may have to filter the solution and isolate the iodoform to be sure the test is positive.

potassium iodide
KI

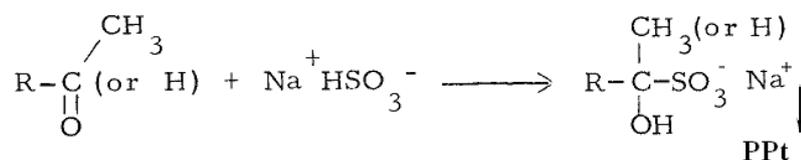
Clorox
(sodium hypochlorite)
NaOCl

Secondary methyl carbinols are oxidized to methyl ketones and will therefore also give the test. *Try the test with acetone, 2-propanol, 3-pentanone, acetophenone, and propiophenone.*



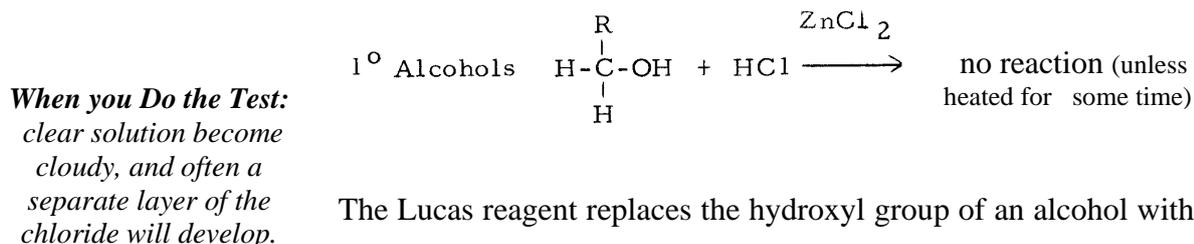
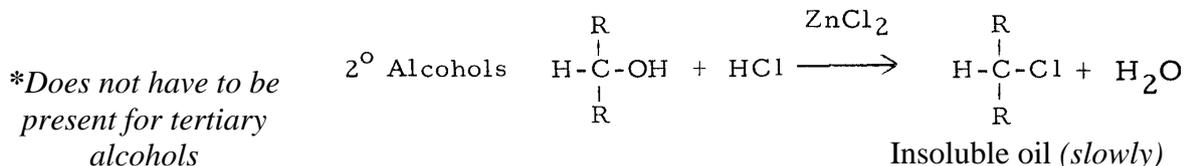
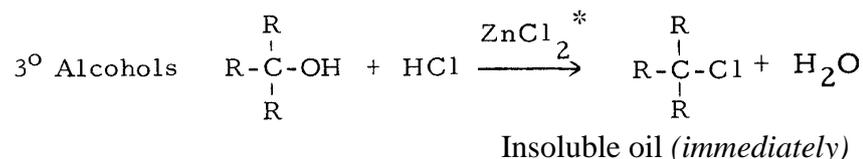
E. Sodium Bisulfite

Sodium bisulfite adds to aldehydes and methyl ketones to give crystalline addition products.



Shake 1 to 2 ml of a saturated solution of sodium bisulfite, NaHSO₃, (not meta-bisulfite) with a few drops of the compound to be tested. Aldehydes and methyl ketones give a precipitate of the addition product. Try the test with acetone, acetophenone, benzaldehyde, and cyclohexanol.

F. Lucas Test



Tertiary alcohol give the test immediately at room temperature.

Secondary alcohols take 5 to 10 min at room temperature or a minute or two at 60 °C.

Primary alcohols will react eventually but take much longer.

**Phenols
&
Carboxylic acids
do not react**

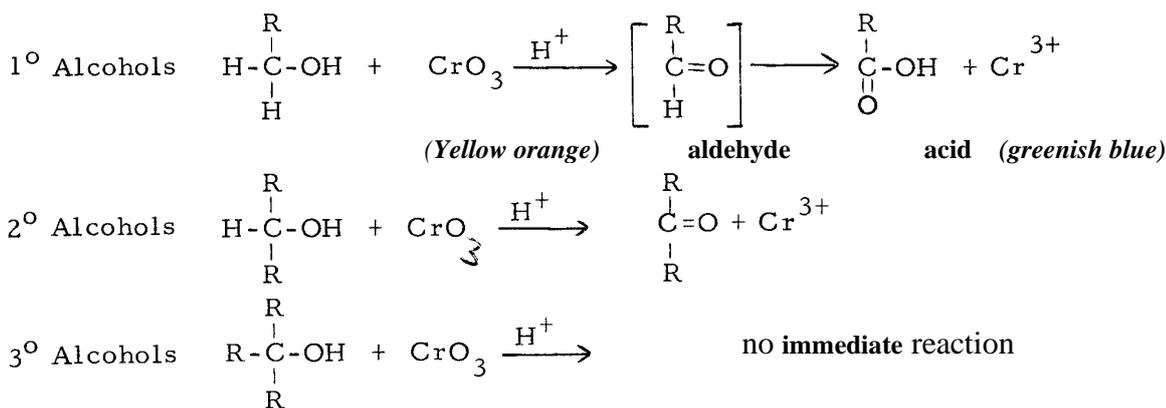
Caution :
Be careful with Handling phenol solutions. They are almost invariable strong vesicants (blisterers) or irritants.

The Lucas reagent replaces the hydroxyl group of an alcohol with a chloride. The alcohol is usually soluble in the reagent, and the chloride is not. One therefore sees a clear solution become cloudy, and often a separate layer of the chloride will develop. Tertiary alcohol give the test immediately at room temperature. Secondary alcohols take 5 to 10 min at room temperature or a minute or two at 60 °C. Primary alcohols will react eventually but take much longer. Phenols and acids do not react. Since the reagent sometimes becomes weaker with age, you cannot rely on time alone. Always do the test with different kinds of alcohols simultaneously so that you can see the contrast. Put 1 to 2 ml of the prepared Lucas reagent (anhydrous zinc chloride dissolved in concentrated hydrochloric acid) in a 10 X 75-mm test tube. Add 5 to 10 drops of the alcohol and shake briefly. Wait several minutes and then put the test tubes with no reaction into a beaker of water of 60 °C. The tertiary alcohols should react at room temperature and the secondary alcohols at 60°C; the primary alcohols won't react.

Try the test with 1-butanol, 2-butanol, and t-butyl alcohol. If desired, you may try a few drops of a 90% solution of phenol and a few drops of acetic acid in separate test tubes to perceive the non reaction.

For contrast, repeat the tests with concentrated hydrochloric acid. Only the tertiary alcohol will react.

G. Chromic Anhydride - Oxidation of Alcohols



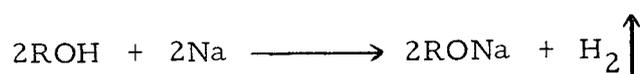
Primary alcohols can be oxidized to aldehydes and then acids. Secondary alcohols are readily oxidized to ketones. Tertiary alcohols are not readily oxidized.

Given just a little time, however, the tertiary alcohol will undergo changes such as unsaturation that will allow extensive oxidation. This hesitation is the basis of this test. Primary and secondary alcohols will react with this reagent within 2 sec. Tertiary alcohols hesitate for a few seconds, then react.

Add 1 to 2 ml of acetone to each 10 X 75-mm test tube. Add 1 to 2 drops of the compound to be tested and then add an equal amount of the chromic acid reagent.

The yellow color should change immediately to the greenish blue of the chromic ion. If the solution remains yellow for 2 sec, the test is negative. Be sure to do a blank on the acetone Test 1-butanol, 2-butanol, and t-butyl alcohol.

H. Reaction with Sodium Metal



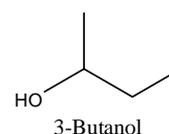
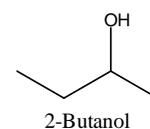
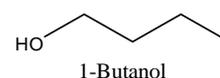
Alcohols can be distinguished from many other kinds of compounds - such as hydrocarbons, ethers, and esters - by their reactivity with sodium metal. Great care must be used in handling metallic sodium and in interpreting the results of this test. The test detects the active hydrogen on the hydroxyl. Therefore, any other active hydrogen will interfere, particularly water. A moist ether would appear positive.

Place 1 ml of the compound in a 10 X 75-mm test tube. Insert a small freshly cut sliver of metallic sodium and look for the steady but possibly slow evolution of hydrogen gas. Occasionally the sodium will adsorb enough water to give a little hydrogen. Look for the steady and continual evolution that indicates reaction with the compound itself. Primary alcohols react most rapidly, secondaries next, and tertiaries slowly if at all. The sodium should eventually completely dissolve.

Try the test on 1-butanol, 2-butanol, and t-butyl alcohol.

Primary and secondary alcohols will react with this reagent within 2 sec.

Tertiary alcohols hesitate for a few seconds, then react.



Primary alcohols react most rapidly, secondaries next, and tertiaries slowly if at all

Experiment 9 *Prelaboratory Assignment*

REACTIONS OF THE FUNCTIONAL GROUPSII: ALDEHYDES, KETONES, AND ALCOHOLS

Name : Section :

4. Which functional group will be discussed in these experiments?
5. Write the chemical structure for the following organic compounds:
 - e) 3-Pentanol
 - f) Pentanal
 - g) 3-Pentanone
6. How could you prepare 3-Pentanone from 3-Pentanol?
7. How could you prepare Pentanal from n-Pentanol?
8. Name the following organic compounds:
 - $\text{CH}_3(\text{CH}_2)_5\text{CHO}$
 - $\text{CH}_3(\text{CH}_2)_5\text{OH}$
 - $(\text{CH}_3\text{CH}_2)_2\text{CO}$
 - $\text{C}_6\text{H}_6\text{OH}$
 - $(\text{CH}_3\text{CH}_2)_2\text{COHCH}_3$

Experiment 9 *Report Sheet*

REACTIONS OF THE FUNCTIONAL GROUPSII: ALDEHYDES, KETONES, AND ALCOHOLS

Name : Section :

Results; Observations						
Test	Reagent	Acetone	Acetophenone	Cinnamaldehyde	Cyclohexanol	Benzaldehyde
A.	2, 4-Dinitro Phenyl hydrazine					

Results; Observations			
Test	Reagent	Acetone	Acetaldehyde
B.	Fehling's		

Results; Observations			
Test	Reagent	Acetone	Acetaldehyde
C.	Tollens'		

* Results of silver mirror preparation

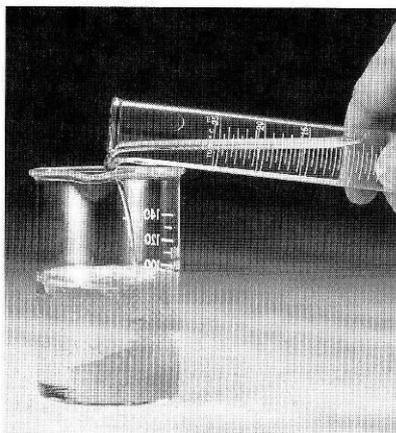
Results; Observations						
Test	Reagent	Acetone	Acetophenone	3-Pentanone	2-Propanol	Benzaldehyde
D.	Iodoform					

Results; Observations						
Test	Reagent	Acetone	Acetophenone	Cyclohexanol	2-Propanol	Benzaldehyde
E.	Sodium Bisulfite					

Results; Observations				
Test	Reagent	1 -Butanol	2-Butanol	t-Butyl alcohol
F.	Lucas			
G.	Chromic Anhydride			
H.	Sodium Meta			

Laboratory Questions :

1. Write a chemical equation for all positive test in this experiment.



Appendix **G**

Water Solubility of Inorganic Salts

Many salts, such as cadmium sulfide, have very low solubilities.

-
1. All salts of the chloride ion, Cl^- , bromide ion, Br^- , and iodide ion, I^- , are soluble **Water-Soluble Salts** *except* those of Ag^+ , Hg_2^{2+} , Pb^{2+} , Cu^+ , and Tl^+ . BiI_3 and SnI_4 are insoluble. PbCl_2 is three to five times more soluble in hot water than in cold water.
2. All salts of the acetate ion, CH_3CO_2^- , nitrate ion, NO_3^- , chlorate ion, ClO_3^- , perchlorate ion, ClO_4^- , and permanganate ion, MnO_4^- , are soluble.
3. All common salts of the Group 1A cations and ammonium ion, NH_4^+ , are soluble.
4. All common salts of the sulfate ion, SO_4^{2-} , are soluble *except* those of Ba^{2+} , Sr^{2+} , Pb^{2+} , and Hg^{2+} .
5. All Group 1A and 2A salts of the bicarbonate ion, HCO_3^- , are soluble.
6. *Most* salts of the fluorosilicate ion, SiF_6^{2-} , thiocyanate ion, SCN^- , and thiosulfate ion, $\text{S}_2\text{O}_3^{2-}$, are soluble. *Exceptions* are the Ba^{2+} and Group 1A fluorosilicates, the Ag^+ , Hg_2^{2+} , and Pb^{2+} thiocyanates, and the Ag^+ and Pb^{2+} thiosulfates.

-
1. All common salts of the fluoride ion, F^- , are insoluble *except* those of Ag^+ , NH_4^+ , and Group 1A cations. **Water-Insoluble Salts**
2. In general, all common salts of the carbonate ion, CO_3^{2-} , phosphate ion, PO_4^{3-} , borate ion, BO_3^{3-} , arsenate ion, AsO_4^{3-} , arsenite ion, AsO_3^{3-} , cyanide ion, CN^- , ferricyanide ion, $[\text{Fe}(\text{CN})_6]^{3-}$, ferrocyanide ion, $[\text{Fe}(\text{CN})_6]^{4-}$, oxalate ion, $\text{C}_2\text{O}_4^{2-}$, and the sulfite ion, SO_3^{2-} , are insoluble, *except* those of NH_4^+ and the Group 1A cations.
3. All common salts of the oxide ion, O^{2-} , and the hydroxide ion, OH^- , are insoluble *except* those of the Group 1A cations, Ba^{2+} , Sr^{2+} , and NH_4^+ . $\text{Ca}(\text{OH})_2$ is slightly soluble. Soluble oxides produce the corresponding hydroxides in water.
4. All common salts of the sulfide ion, S^{2-} , are insoluble *except* those of NH_4^+ and the cations that are isoelectronic with a noble gas (e.g., the Group 1A cations, the Group 2A cations, Al^{3+} , etc.).
5. Most common salts of the chromate ion, CrO_4^{2-} , are insoluble *except* those of NH_4^+ , Ca^{2+} , Cu^{2+} , Mg^{2+} , and the Group 1A cations.
6. All common salts of the silicate ion, SiO_3^{2-} , are insoluble *except* those of the Group 1A cations.

Table G.1 Summary of the Solubility of Salts

Anion	Soluble Salts with These Cations	“Insoluble” Salts with These Cations
acetate, CH_3CO_2^-	most cations	none
arsenate, AsO_4^{3-}	NH_4^+ , Group 1A (except Li^+)	most cations
arsenite, AsO_3^{3-}	NH_4^+ , Group 1A (except Li^+)	most cations
borate, BO_3^{3-}	NH_4^+ , Group 1A (except Li^+)	most cations
bromide, Br^-	most cations	Ag^+ , Hg_2^{2+} , Pb^{2+} , Cu^+ , Tl^+
carbonate, CO_3^{2-}	NH_4^+ , Group 1A (except Li^+)	most cations
chlorate, ClO_3^-	most cations	none
chloride, Cl^-	most cations	Ag^+ , Hg_2^{2+} , Pb^{2+} , Cu^+ , Tl^+
chromate, CrO_4^{2-}	NH_4^+ , Ca^{2+} , Cu^{2+} , Mg^{2+} , Group 1A	most cations
cyanide, CN^-	NH_4^+ , Group 1A (except Li^+)	most cations
ferricyanide, $[\text{Fe}(\text{CN})_6]^{3-}$	NH_4^+ , Group 1A (except Li^+)	most cations
ferrocyanide, $[\text{Fe}(\text{CN})_6]^{4-}$	NH_4^+ , Group 1A (except Li^+)	most cations
fluoride, F^-	Ag^+ , NH_4^+ , Group 1A	most cations
fluorosilicate, SiF_6^{2-}	most cations	Ba^{2+} , Group 1A
hydroxide, OH^-	NH_4^+ , Sr^{2+} , Ba^{2+} , Group 1A	most cations
iodide, I^-	most cations	Ag^+ , Hg_2^{2+} , Pb^{2+} , Cu^+ , Tl^+ , Br^{3+} , Sn^{4+}
nitrate, NO_3^-	most cations	none
nitrite, NO_2^-	most cations	none
oxalate, $\text{C}_2\text{O}_4^{2-}$	NH_4^+ , Group 1A (except Li^+)	most cations
oxide, O^{2-}	NH_4^+ , Sr^{2+} , Ba^{2+} , Group 1A	most cations
perchlorate, ClO_4^-	most cations	none
permanganate, MnO_4^-	most cations	none
phosphate, PO_4^{3-}	NH_4^+ , Group 1A (except Li^+)	most cations
silicate, SiO_3^{2-}	Group 1A	most cations
sulfate, SO_4^{2-}	most cations	Sr^{2+} , Ba^{2+} , Pb^{2+} , Hg^{2+}
sulfide, S^{2-}	NH_4^+ , Groups 1A and 2A	most cations
sulfite, SO_3^{2-}	NH_4^+ , Group 1A (except Li^+)	most cations
thiocyanate, SCN^-	most cations	Ag^+ , Hg_2^{2+} , Pb^{2+}
thiosulfate, $\text{S}_2\text{O}_3^{2-}$	most cations	Ag^+ , Pb^{2+}
Cations	Soluble Salts with These Anions	“Insoluble” Salts with These Anions
ammonium, NH_4^+	most anions	no common anions
Group 1A	most anions	no common anions