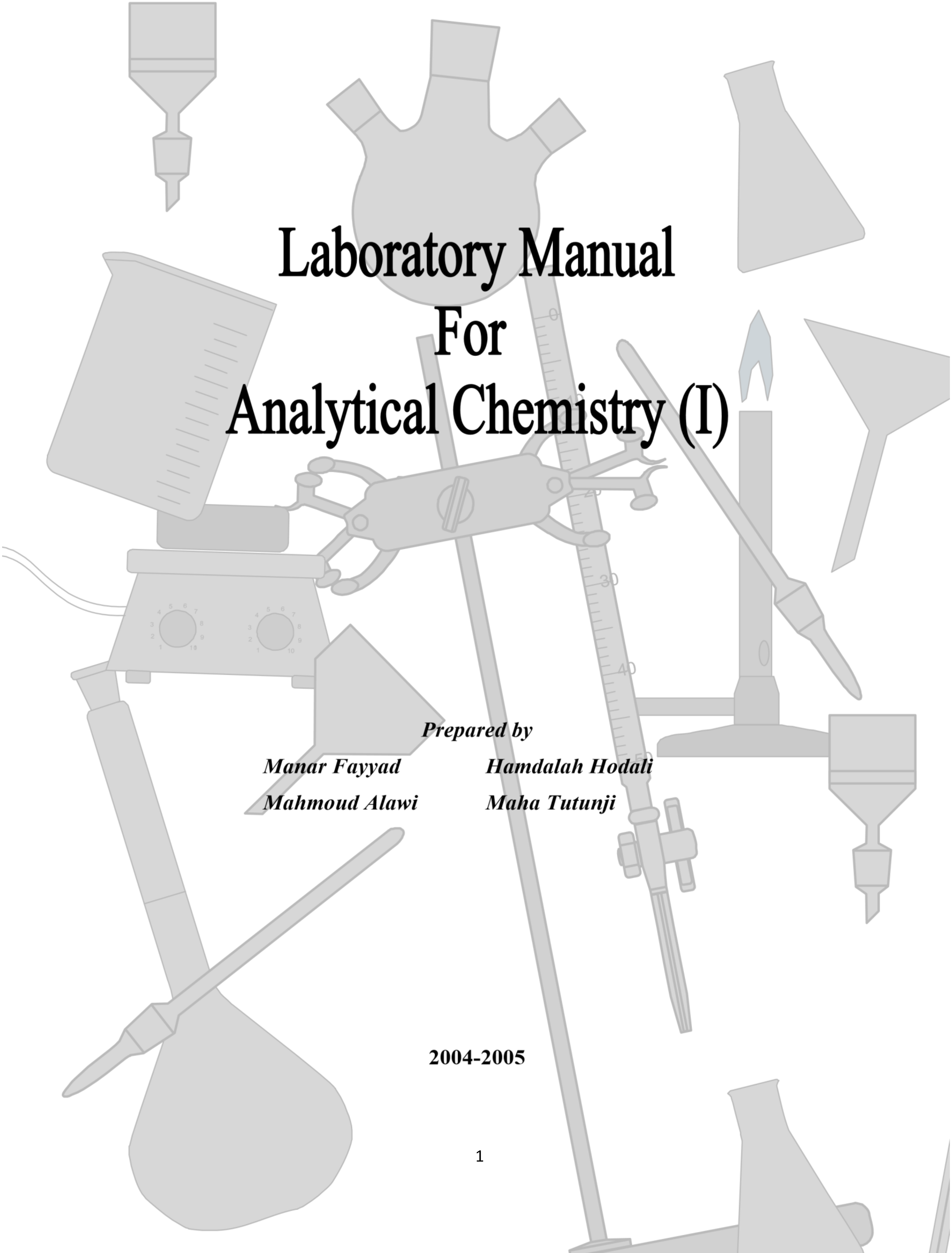


Laboratory Manual
For
Analytical Chemistry (I)

Prepared by

Manar Fayyad ***Hamdalah Hodali***
Mahmoud Alawi ***Maha Tutunji***

2004-2005

CONTENTS

EXPERIMENT (1)
SAFETY AND EQUIPMENTS

Working in a laboratory should be a safe experience. This will happen, however, only if certain precautions are followed without exception. The practice of safety requires (1) the desire on the part of the individual to protect himself or herself as well as those around him or her and (2) the need to rigidly follow a well-defined set of laboratory rules. The safety rules to be followed by all midshipmen in the Chemistry Department laboratories will be discussed by your instructor during the first laboratory class period of the semester. These rules are posted in the laboratory spaces and are outlined on the following pages of this manual. While it is the obligation of the instructor to explain these rules, it is the responsibility of everyone in the laboratory to follow the safety regulations. They will be rigidly and impartially enforced, with non-compliance resulting in the dismissal of the guilty party from the laboratory.

A. Safety Equipment

The location and use of the safety equipment in laboratory will be discussed by your instructor the first day that the class meets in the lab space. All students should become familiar with the proper use of the:

Safety shower, Eye-wash fountain, Fire extinguisher, and neutralizing solutions for spills. The location of the equipment is important, as well as when to use each piece of equipment.

Safety goggles for eye protection are to be worn by all students while in the laboratory. If a midshipman fails to bring the goggles to class, he or she will not be allowed to inter the laboratory. Laboratory coats are to be worn by all midshipmen while in the lab.

B. Laboratory Do's and Don'ts.

The following is a list of do's and don'ts which must be adhered to by all students.

1. The laboratory stools belong to the lab islands. They are designed to be used by the students in the lab only.
2. Bring only the necessary materials into the laboratory: lab manual, pencil/pen, calculator, graph paper, ruler, coats and goggles.
3. Do not touch chemicals with your hands. Spatulas and forceps have been provided for handling solid materials.

4. No wet chemicals should ever be placed on the islands or the pull-out writing surfaces. In addition, no stock solutions bottles are to go to individual lab stations. Midshipmen should take an appropriate container to the stock solution to obtain the needed amount.
5. Do not eat or drink in the laboratory. Do not taste any chemical. Do not smell any chemicals directly. Use your hand to waft the odor to your nose.
6. Do not pipit solutions by mouth. Rubber pipit bulbs are provided at each lab station.
7. Do not put flammable liquids near an open flame.
8. When heating a test tube, make certain that the open end of the tube is directed away from your face of other students. If overheating or superheating causes the contents to bump out, no one in the laboratory will be splashed.
9. When finished with your Bunsen burner for a given portion of an experiment, turn it off. Be careful not to place lit burners under the shelf above the lab station.
10. Do not sit on the lab benches. We give you nice stools – use them and save your uniform from damage.[1]
11. Do not engage in games or horseplay in the laboratory. Failure to follow this rule will result in immediate dismissal from the lab and subsequent conduct action.
12. Do not pour any chemical into a sink without authorization from the instructor.
13. All broken glassware should be reported to your instructor immediately. Broken glass should be disposed of in the specially marked receptacles.
14. Do all reactions, particularly those involving malodorous, noxious or dangerous chemicals, in a fume hood.
15. If a chemical gets on your skin, immediately wash the affected area with large quantities of water. The instructor should be notified, no matter how insignificant the incident might seem.
16. When pouring one liquid into another, do so slowly and cautiously. To dilute an acid, pour the acid into the water; never pour water into an acid.
17. No student shall be permitted to work alone in the lab. You may not do unauthorized experiments or variations of any experiment.
18. Exercise good housekeeping practices in the laboratory. Be sure that the lab benches remain free of clutter during the experiment. In the event of a spill, clean the area immediately. Be sure to use a wet sponge to wipe off the work station at the conclusion of the lab.
19. Know what you are to do before entering the lab. Read the experiment carefully before coming to the lab. Be cautious and think about what you are doing. Use common sense.
20. **Wash your hands before leaving the lab**.

C. Standard Operating Procedure of the Chemistry Department for Classroom or Laboratory:

Evacuation due to Fire, Fire Drill or Bomb Threat:

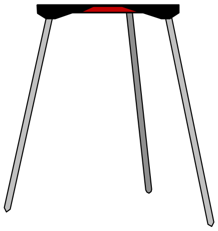
In the event of a fire, fire drill or bomb threat during a class period, a quite distinguishable alarm will sound. In response to the alarm, the midshipmen will take the following actions.

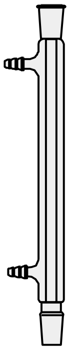
1. All students will exit the class in an orderly and safe fashion. All students will form-up, by section, in a pre-designated area. This area will be specified by your instructor on the first day of class, each semester.
2. Section leaders will take a muster of the section as soon as possible after evacuation of the building. The section leaders should promptly report the results of the muster to the instructor.
3. No midshipmen will be allowed to leave the muster area until directed to do so by the instructor.
4. Fire department personnel will advise the instructors when it is safe to return to the building to resume the normal schedule of classes.

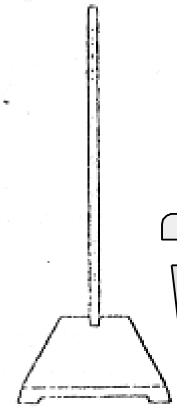
D. Standard Operating Procedure for Student Injuries in Laboratory

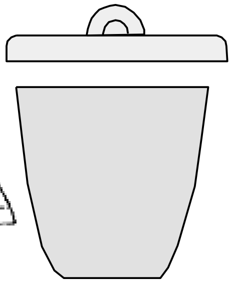
1. Students should report *all* injuries, no matter how small, to the instructor.
2. The instructor will use conservative judgment about the severity of the injury. For anything more serious than a paper cut the student will be sent, with escort, to the Medical Clinic.
3. If the injury is a chemical burn, the burned area is to be flushed with water for fifteen minutes before departure to Medical treatments in the university medical center.
4. The escort accompanying an injured student to Medical should deliver the Prep Room phone number to medical personnel at the reception desk, along with the MSDS, if appropriate. The escort should then report back to the instructor with any pertinent information.
5. The instructor will enter time and date of injury, a detailed description of the injury, and remedial steps taken in the injury log in Prep Room office.

LABORATORY APPARATUS


Tripod

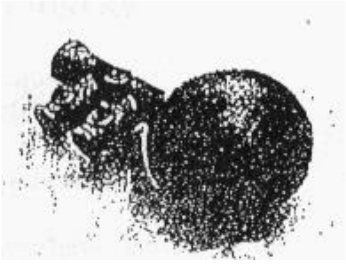

Condenser

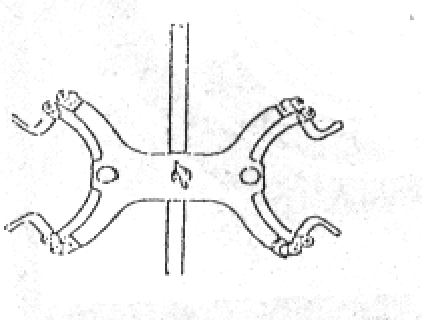

Stand


Crucible


Volumetric pipette


Reagent bottle

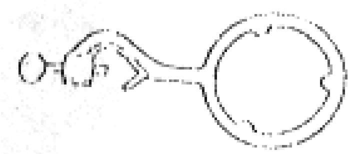

Pipette filler

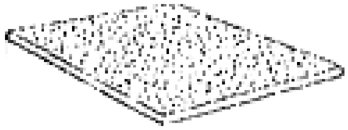

Burette clamp

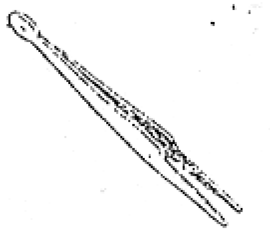

Test tube brush

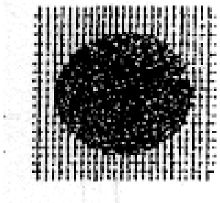

Metal spatula


Test tube holder

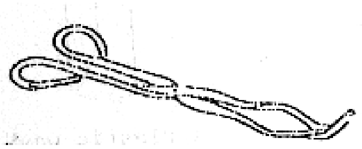

Ring clamp


Ceramic square

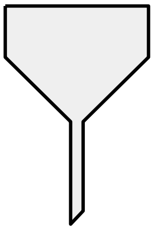

Forceps


Wire gauze

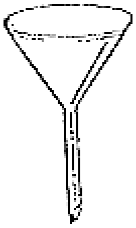

Watch glass


Crucible tongs

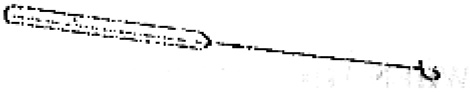
Beaker



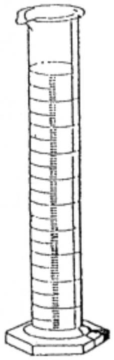
Buchner Funnel



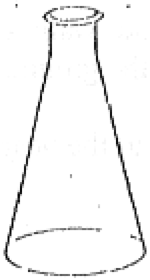
Funnel



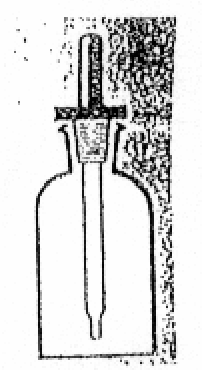
Glass rod with platinum wire (for flame testing)



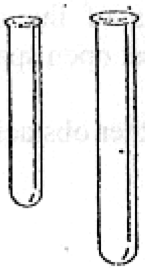
Graduated cylinder



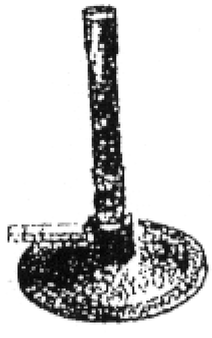
Erlenmeyer flask



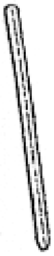
Dropping bottle



Test tubes



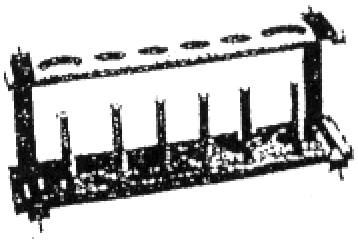
Bunsen burner



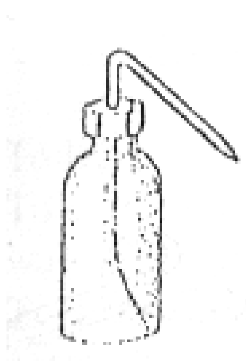
Stirring rod

Dropper

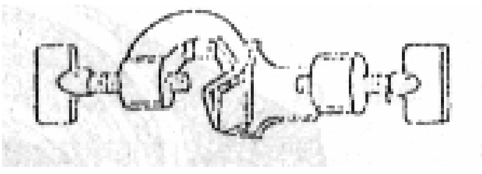
Safety glasses (Goggles)



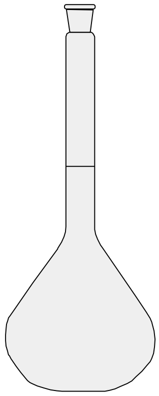
Test tube rack



Plastic wash bottle



Clamp holder



Volumetric flask

Burette

(C): CHEMISTRY LABORATORY SAFETY AGR

EEMENT

Before working in the chemistry laboratory, read carefully the safety precautions and techniques for handling chemicals. Give this agreement, signed and dated, to your laboratory instructor on the first day of lab.

When you are in the laboratory, THINK about what you are doing at all times.

1. If any fume hood in your laboratory is on, you must wear your approved chemical splash goggles.
2. Do not attempt any unauthorized experiments.
3. Know the location and operation of safety equipment.
4. Bring only necessary materials into lab. Book bags, jackets, etc., are to be left in the hallway.
5. Never work alone in the laboratory.
6. Never eat or drink in the laboratory.
7. Use the fume hood when necessary.
8. Keep your work area uncluttered.
9. Use only equipment that is in good condition.
10. Dispose of waste and excess materials according to your laboratory instructor’s directions.
11. Don’t sit or lean on laboratory work surfaces.
12. Handle chemicals with caution.
    (a) Read labels carefully.
    (b) Use only the amount required.
    (c) Leave reagent containers in their proper places.
    (d) Clean up all spills immediately.
    (e) Label all chemical containers.
13. Thoroughly wash your hands any time you leave the laboratory.
14. Immediately report all accidents and physical/chemical injuries, no matter how minor, to your laboratory instructor. Be ready to take immediate action as needed to assist any injured classmate.
15. Don’t leave the laboratory without your instructor’s approval.

I have carefully read all the safety precautions on the above pages , and recognize that it is my responsibility to observe them throughout my chemistry lab course.

I have read and understood the 'Safety Rules in the Laboratory' and the 'Fire regulations'. I agree to follow these rules.

Name: ______________________________ **ID No.**___________

Signature: _____________________

Course: _______________________

Section: _______________________

Date: _________________________

EXPERIMENT (2)
VOLUMETRIC GLASSWARE AND BALANCES

I. INTRODUCTION

Two basic types of equipment in an analytical laboratory which are volume measuring devices and balances. Volumetric glassware used in analysis should be clean. Glassware that looks clean may or may not be clean. Surfaces, that have no visible dirts, are sometimes contaminated with invisible film of greasy materials. Glassware into which a brush can be inserted, such as beaker or erlenmeyer flasks, are best cleaned with soap or synthetic detergent[1]. Pipets, burettes, and volumetric flasks, on the other hand, are ordinarily cleaned with cleaning solution[2], whose strong oxidizing properties ensure clean glass surfaces in most cases. After cleaning, apparatus should be rinsed several times with tap water, then with small portions of distilled water, and finally allowed to drain.

Table 1. Tolerances for volumetric glasses (mL)

Capacity less and including	Volumetric flasks	Transfer pipets	Burettes
2		0.006	
5		0.01	0.01
10		0.02	0.02
25	0.03	0.03	0.03
50	0.05	0.05	0.05
100	0.08	0.08	0.10
200	0.10	0.10	
500	0.15		
1000	0.30		

Calibration of volumetric glassware used in volumetric analysis is sometimes necessary. Table (1) shows some of the tolerance values established for volumetric glassware by the National Bureau of Standards (NBS). Very often low-priced glassware does not meet NBS tolerances. For this reason, and also because the history of a particular item involving possible damage may be obscure, it is often advisable for the student to calibrate his

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[1] For example alkonox

[2] Cleaning solution can be prepared by dissolving 20 to 25 g of technical-grade sodium dichromate in 5 mL of water in a 600 mL beaker. Then add slowly and carefully about 450 mL of technical-grade sulfuric acid with occasional stirring. Cool the solution and store it in a glassstoppered bottle.

volumetric glassware. One of the methods employed in calibration is based on finding the volume of water obtained from a measuring device; from weight of water, and its density (Table 2). This type of calibration involves the use of an analytical balance.

Table 2. Density of water at various temperatures.

°C	g/mL	°C	g/mL
10	0.9984	21	0.9970
11	0.9983	22	0.9968
12	0.9982	23	0.9966
13	0.9981	24	0.9964
14	0.9980	25	0.9962
15	0.9979	26	0.9959
16	0.9978	27	0.9957
17	0.9977	28	0.9954
18	0.9975	29	0.9952
19	0.9974	30	0.9943
20	0.9972		0.9943

In order to use the analytical balance safely and accurately the following rules should be strictly followed:

• RULES FOR THE CARE AND USE OF BALANCES

1. The balance should be kept clean. Remove dust and spills (there should not be any) from the pan and floor of the balance with a brush before you begin weighing. The brush will be found in the balance case.

2. Never weigh an object heavier than the capacity of your balance. Make a rough weighing on the triple beam if you are in doubt about whether the object exceeds the capacity of the single beam balance.

3. The object to be weighed should be at room temperature. It is wise not to handle the object with your fingers since fingerprints contribute weight. Glass objects should not be rubbed with a dry cloth prior to weighing since this can put a static charge on the object which may affect the accuracy of the reading.

4. Chemicals are never placed directly on the balance pan. For quantitative weighing, the following procedure <u>must</u> be carried out; a technique called “weighing by difference.”

    a) The substance to be weighed is placed in a clean, dry weighing bottle. Fingerprints are wiped off and the weighing bottle here forth manipulated by means of a strip of paper.
    b) The stoppered weighing bottle is weighed and the mass recorded to four decimal places.
    c) With the strip of paper, the weighing bottle is removed from the balance, the stopper removed with clean forceps on a clean surface. The material is transferred from the weighing bottle to the receiving vessel by tipping the bottle and gently rotating it back and forth until the material begins to fall into the receiver. <u>If any spills occur outside the receiver, you must</u> <u>begin the process again</u> with a clean receiver and a new weight of the weighing bottle and contents.
    d) Transfer material, re-stopper and re-weigh the weighing bottle until the amount of material transferred (difference in weights) is approximately equal to the desired quantity.

    The use of weighing paper is not an acceptable practice for quantitative determinations since the sample can pick up atmospheric moisture off the weighing paper.

5. At the completion of a weighing make sure that:

    a) the weight is recorded in your notebook.
    b) the balance is clean.
    c) no objects are left on the pan.

This experiment is designed to provide practice in the use of the analytical balance and calibration of volumetric glassware.

II. EXPERIMENTAL PROCEDURE:

A. Cleaning.

Clean the burette using a mild detergent and brush. Thoroughly rinse and test the burette for dirt by allowing water to drain from the burette. If the burette is clean, an even film of water will develop on the surface, if the burette is still dirty, water droplets will form as the liquid is allowed to drain from the burette. Thus, it is necessary to clean to burette with dichromate cleaning solution. However, care must be taken when using this cleaning solution since it can cause burns.

B. Calibration of a burette.

After the burette has been cleaned and rinsed, fill it with distilled water so that the meniscus is above the zero mark. Then slowly open the stopcocks and allow the solution to fill the tip. If air bubbles appear in the tip, these must be removed by quickly turning the stopcock open and closed until the bubbles leave through the tip. By opening the stopcock, adjust the level of solution in the burette so that the meniscus is above the zero mark, Fig (1). (Be sure there are no air bubbles in the burette tip). Record the temperature of the water.

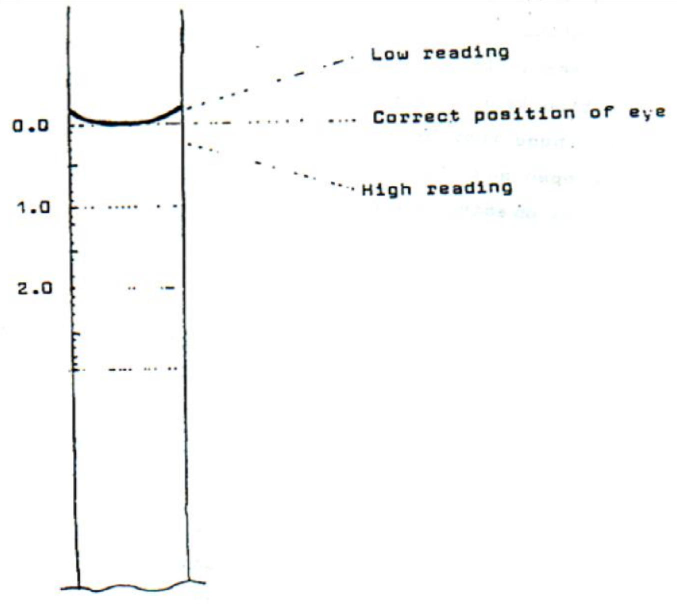

Fig. (1) Section of a buret

Weigh[3] a glass-stoppered weighing bottle (or a stoppered 50 mL Erlenmeyer flask) up to four decimal points. Now withdraw water more slowly until the meniscus is at, or slightly below, the zero mark on the burette. After drainage is complete (at least 30 sec.) read the burette to two decimal points. Record this initial reading. Remove the water droplet at the burette tip by touching it to a piece of glass. (e.g.: inside wall of a beaker) slowly allow a little less than 5 mL of water to drain into the weighing bottle, wait 30 seconds for drainage from the wall of the burette, and adjust the meniscus to drain about 5.00 mL.

Touch the tip of the burette with the inside wall of the weighing bottle. Quickly stopper the bottle. Read the burette after allowing time for drainage and record the “final” reading. Then weigh the stoppered bottle to four decimal points record this weight. Repeat the procedure three more times. All volume readings should be reported to two decimal points. The weighing bottle should be emptied, the neck dried with a clean towel, and reweighed. Tabulate your results in the following tables.

When your experimental work is complete, rinse all your volumetric glassware with large quantities of water, store the burette filled with distilled water and capped. Be sure all equipment is returned to its appropriate place.

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[3] See rules for the care and use of balances.

EXPERIMENT (2)

VOLUMETRIC GLASSWARE AND BALANCES

Name: **Group NO.:**

III DATA

	Trial 1	Trial2	Trial 3	Trial4
Initial burette reading, mL				
Final burette reading, mL				
Apparent volume, mL				
Initial weight of flask, g				
Final weight of flask, g				
Weight of water, g				
Temperature, °C				
Actual volume, mL				
Correction, mL				

Instructor's Signature

IV. CALCULATION

Note: use significant figures and decimal places in the calculations

(Trial No ………..)

1- Apparent Volume (mL) = Final burette reading – Initial burette reading

2- Weight of water(g) =

3- Actual Volume (mL) =

4- Correction (mL) =

ERRORS

- **According to the following rules:**

z = x + y or z = x - y ⇒ $\Delta z = \sqrt{\Delta X^2 + \Delta y^2}$

z = x * y or $z = \frac{x}{y}$ ⇒ $\Delta z = z * \sqrt{\left(\frac{\Delta X}{x}\right)^2 + \left(\frac{\Delta y}{y}\right)^2}$

Calculate the following errors:

1- Initial (ΔR_i) and final (ΔR_f) burette reading errors =

2- Apparent volume error (ΔA) =

3- Initial (ΔW_i) and final (ΔW_f) weight of the flask errors= Error in the balance =

4- Weight of water error (ΔW) =

5- Temperature error (ΔT) =

6- Actual volume error(ΔAct) =

QUESTIONS

Q1) A 25 mL pipette was found to deliver 24.876 g of water when calibrated against stainless steel weight at 25 °C. Use the data in Table 2 to calculate the volume delivered by the pipette at this temperature, repeat the calculation at 19 °C.

Q2) Suggest some sources of error in this experiment.

EXPERIMENT (3)
SAMPLING AND STATISTICAL HANDLING OF DATA

INTRODUCTION

An important part of any analytical process is to get a representative sample. If the sample is homogenous, any portion of it can be taken. If the sample is not homogenous, a statistical sampling is required. This experiment illustrates the importance of sampling, practice with the burette and the statistical handling of data.

The experiment consists of two parts; first titration of a homogenous sample and second titration of a heterogeneous sample.

For each of these titrations, a blank sample must be analyzed. Constant errors affecting physical measurements can be evaluated with a blank determination. A blank sample is done through performing all steps of the analysis in the absence of sample. The result is then applied as a correction to the actual measurements.

II. EXPERIMENTML PROCEDURE

a. Homogenous sample

1. Titration of potassium hydrogen phthalate (KHP)

   Wight accurately about 0.5g of pure KHP into a 250 mL Erlenmeyer flask. add 25 mL distilled water and three drops of Phenolphthalein. Titrate with 0.10 M NaOH till the indicator color becomes pink. Repeat 5 times.

b. Heterogeneous sample

1. Mixture of KHP and sugar.

   Wight accurately about 0.5gof the KHP and sugar mixture into 250 mL Erlenmeyer flask, add 25 mL distilled water and three drops of phenolphthalein. Titrate against 0.10 M NaOH. Repeat 5 times.

EXPERIMENT (3)

SAMPLING AND STATISTICAL HANDLING OF DATA

Name: **Group NO.:**

a) **Homogenous sample**

	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Mass of KHP(g)					
Volume of NaOH (mL)					
Molarity of NaOH (M)					
Average molarity of NaOH					
Standard deviation (S_a)					

b) **Heterogeneous sample**

	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Mass of mixture(g)					
Volume of NaOH (mL)					
Molarity of NaOH (M)					
Average molarity of NaOH					
Standard deviation (S_t)					

c) Calculation the sampling error (Ss)

a) Homogeneous sample:

1- Molarity of NaOH for trial 1 =

2- Average molarity of NaOH (Ave.x) =

3- The standard deviation of Homogeneous sample (S_a):

Standard deviation (S) = $\sqrt{\frac{\sum(x_i - Ave.x)^2}{n-1}}$

Sa =

b) Heterogeneous sample (HS):

1- Molarity of NaOH for trial 1

2- Average molarity of NaOH =

3- The standard deviation of Heterogeneous sample (S_t):

Sampling error (S_s):

Remember that: $S_t^2 = S_a^2 + S_s^2$

EXPERIMENT (4) AND (5)
GRAVIMETRIC ANALYSIS

I. INTRODUCTION

Gravimetric analysis is one of the most accurate and precise methods of macroquantitative analysis. In gravimetric analysis the measurement signal is mass. The analyte is selectively converted to an insoluble form, which is then separated, washed, dried or ignited and then weighed accurately. From the weight of the precipitate and knowledge of its chemical composition, the concentration of the analyte in the sample can be calculated.

Gravimetric analysis normally involves processes of:

1. Preparation of the analyte solution.
2. Quantitative precipitation: it is necessary to ensure complete precipitation.
3. Digestion: the precipitate must be digested for purposes of coagulation or increase in the particle size.
4. Filtration: quantitative transfer of the precipitate using proper techniques as will be illustrated later.
5. Washing: this is an important step for removal of other coprecipitated species. Proper choice of washing solvent is necessary for this purpose.
6. Drying or ignition: the precipitate must be stable at the drying temperature. Drying time should ensure complete removal of the solvent.
7. Weighing: the washed and dried precipitate is then weighed to "constant weight" on an analytical balance.

Equipment used in Gravimetric Analysis:

Desiccator

A desiccator is a vessel, usually of glass but occasionally of metal, which is used to equilibrate objects with a controlled atmosphere. Since the desiccator usually stands in the open, the temperature of this atmosphere generally approaches room temperature. It is normally the humidity of this atmosphere which is of interest. Objects such as weighing bottles or crucibles, and chemical substances, tend to pick up moisture from the air.

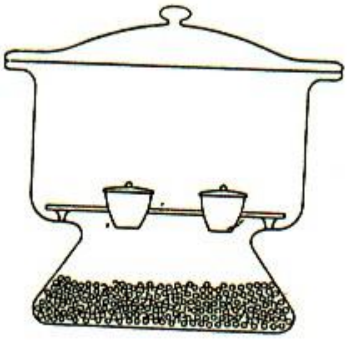

Fig. (1) .Desiccator

The desiccator provides an opportunity for such materials to come to equilibrium with an atmosphere of low and controlled moisture can tent so that errors due to the weighing of water along with the objects can be avoided. A common type of desiccator is shown in Fig. (1)

Table 1. EFFICIENCY OF CHEMICAL DESICCANT

Material	Residual water (mg/Liter)
$Mg(ClO_4)_2.0.12H_2O$	0.2
$Mg(ClO_4)_2.1.4H_2O$	1.5
BaO(96.2%)	2.8
Al_2O_3 (anhydrous)	2.9
P_2O_5	3.5
Molecular Sieve 5A (Linde)	3.9
$Mg(ClO_4)_2$ (88%) + $KMnO_4$ (0.86%)	4.4
ClO_4^- (anhydrous)	13
$CaCl_2.0.18H_2O$	67
$CaCl_2.0.02H_2O$ (Drierite)	67
Silica gel	70
NaOH (91%, Aacarite)	93
$CaCl_2$ (anhydrous)	137
$CaSO_4.0.21H_2O$ (anhydrocel)	207
$NaOH.0.03H_2O$	513
$Sa(ClO_4)_2$ (anhydrous)	599

The nature of the drying agent placed in the bottom of the desiccator determines the equilibrium partial pressure of water vapor in the desiccator space.

After reagents or objects such as crucibles have been dried in the oven, or perhaps at even higher temperatures, they are usually cooled to room temperature in the desiccator prior to weighing. When a hot object cools in the desiccator, a partial vacuum is created, and care must be taken in opening the vessel since a sudden rush of air blows material out of' a crucible or disturb the desiccant itself. For this reason, and also because glass is a very poor conductor of heat, it is usually best to allow a very hot object to cool well toward room temperature before it is placed in the desiccator. After a hot object has been placed in the desiccator, it is well to cover the vessel in such a way as to leave a small opening at one side. This allows air displaced by the warm object to reenter as the object cools, and hence minimizes the tendency to form a vacuum. The desiccator is completely closed during the final stages of cooling.

Funnels and Filter Paper

In gravimetric procedures the desired constituent is often separated in the form of a precipitate. This precipitate must be collected, washed free of undesirable contaminants from the mother liquor, dried, and weighed, either as such or after conversion into another form.

Filtration is the common way of collecting precipitates, and washing is often accomplished during the same operation. Filtration is carried out with either funnels or filter paper or filtering crucibles. The important factors in choosing between the two are the temperature to which the precipitate must be heated to convert it into the desired weighing form and the ease with which the precipitate may be handled. Fig. (2)

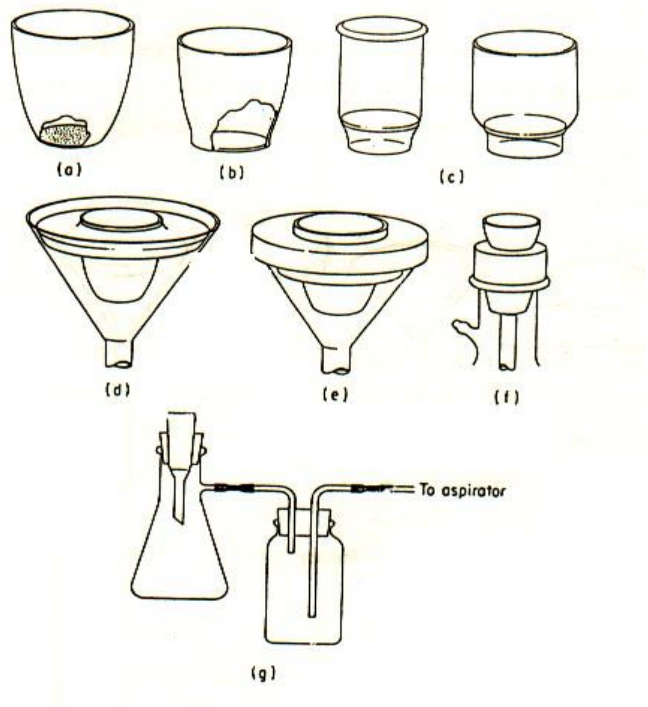

Fig. (2). Filtering crucibles: (a) Gooch, (b) porous porcelain, (c) fritted glass, high and low forms, (d) Sargent holder, (e) Bailey holder, (f) Water holder, and (g) Filtration with suction, water holder.

Various types of filter papers are available for quantitative work, only paper of the so-called “ashless” quality should be used. (A typical figure for the ash from one circular paper 11 cm in diameter 0.13 mg.). The nature of the precipitate to be collected dictates the choice of paper. “Fast” papers are used for gelatinous flocculent precipitates such as hydrous iron (III) oxide and for coarsely crystalline precipitates such as magnesium ammonium phosphate.

Filter paper circles are available in various diameters. The size to be used depends upon the quantity of precipitate, not the volume of solution to be filtered. Larger paper than necessary should be avoided: the paper and the funnel should match with regard to size. It is especially important that the paper not extend above the edge of the glass funnel but come within 1 or 2 cm of this edge. The precipitate should occupy about one-third of the paper cone and never more than one-half.

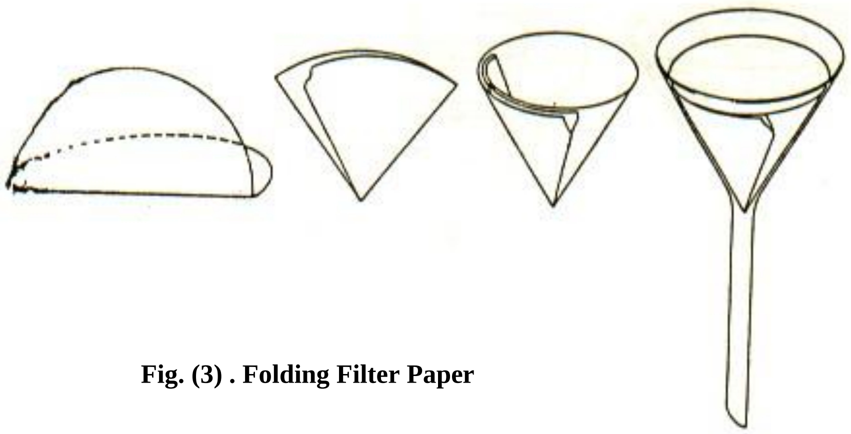

Fig. (3) . Folding Filter Paper

Filter paper is normally folded so as to provide a space between the paper and the funnel, except at the top of the paper, which should fit snugly to the glass.

Technique of Washing and Filtering a Precipitate

Usually a precipitate is washed, either with water or a specified wash solution, before it is dried and weighed. The washing is generally carried out in conjunction with the filtration step Fig. (4+5), where in the precipitate separated from its mother liquor in compact form.

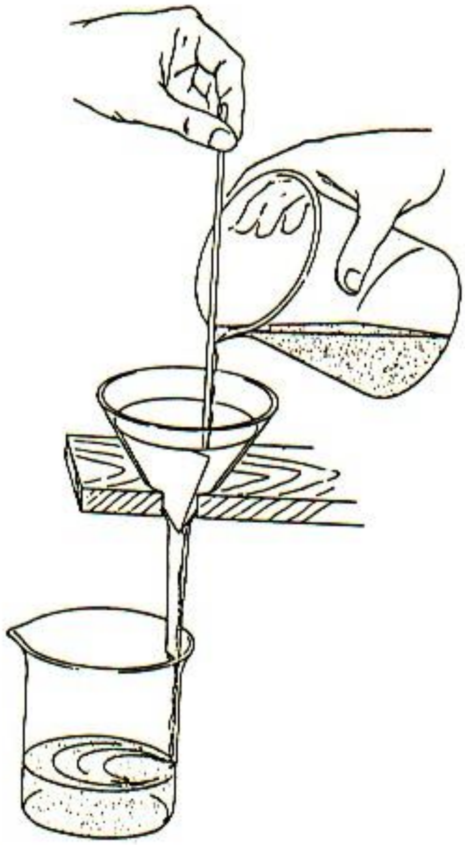

Fig. (4). Technique of filtration with filter paper

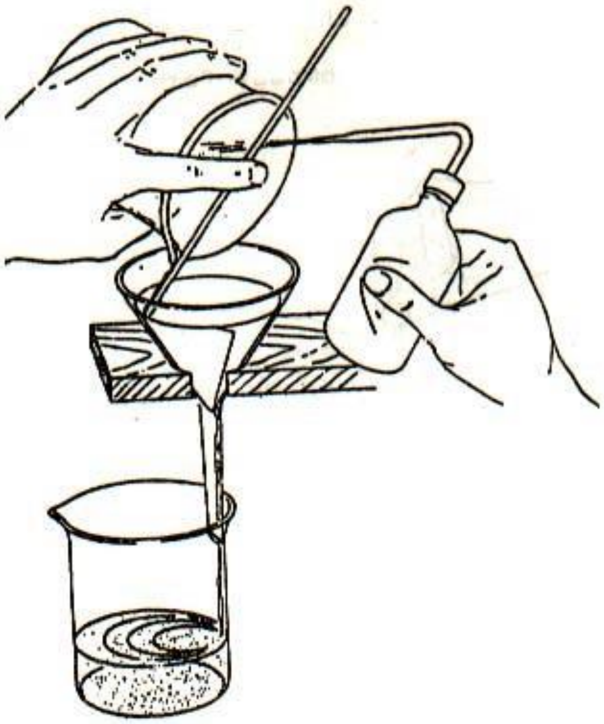

Fig. (5). Use of wash bottle in transferring precipitate

Ignition of Precipitate with Filter Paper

After the filter paper has drained as much as possible in the funnel, the top of the paper is folded over to encase the precipitate. The bundle of paper is transferred from the funnel to the prepared crucible (see the discussion of crucibles below). It is better to handle the paper where it is three layers thick rather than by the other side. The next steps in the ignition of the material in the crucible are generally as follows:

1. Drying the Paper and Precipitate. This may be done in an oven at temperatures of 100 to 125 °C if the schedule permits setting aside the experiment at this stage. If the ignition is to be followed through immediately, the drying may be accomplished with a burner. Place the covered crucible in a slanted position in a clay or silica triangle, and place a small flame beneath the crucible at about the middle of the underside.

   Too strong heating must be avoided; and the flame should not touch the crucible.

2. Charring the Paper. (See Fig. (6)) After the precipitate and paper are Entirely dry, the crucible cover is set ajar to permit access of air, and the heating is increased to char the paper. Increase the size of the flame slightly and move it to burn off with a flame. If the paper bursts into flame, cover the crucible immediately to extinguish it.

3. Burning Off the Carbon from the Paper. After the paper has completely charred and the danger of its catching fire is past, the size of the flame is increased until the bottom or the crucible becomes red.

4. Final Stage of Ignition. To conclude the ignition, Plate the crucible upright, removing the cover to admit air freely, and heat at the temperature recommended for the particular precipitate.

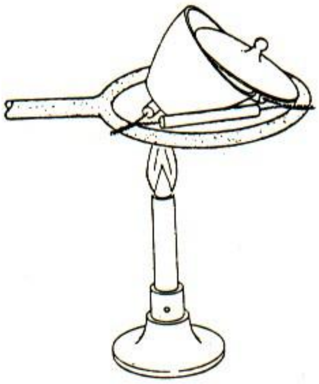

Fig. (6). Ignition of precipitate

EXPERIMENT (4)
GRAVIMETRIC DETERMINATION OF CHLORIDE

I. INTRODUCTION

The chloride content of a soluble sample is determined by precipitating silver chloride by using silver nitrate as a precipitating agent:

$$Ag^{+}_{(aq)} + Cl^{-}_{(aq)} \longrightarrow AgCl_{(s)}$$

This colloidal precipitate is digested, filtered, washed, dried and then, weighed. The chloride content is calculated from the weight of AgCl using the gravimetric factor:

$$\text{mass of Cl} = \frac{\text{mass of AgCl } x \text{ Molar mass of Cl}}{\text{Molar mass of AgCl}}$$

II. EXPERIMENTAL PROCEDURE

Pipet 10.0 mL of the unknown chloride solution into a 600 mL beaker. Dilute with 100-150 mL of distilled water. Add about 1 mL of conc. nitric acid. Cover the beaker with a clean watch class.

Slowly and with string add about 20 mL of the 0.1 M $AgNO_3$ solution. Heat the suspension nearly to boiling with frequent stirring[4] to coagulate the silver chloride. Let the precipitate settle, and test for complete precipitation by carefully adding few drops of silver nitrate to the clear supernatant liquid. If more precipitate or cloudiness appears, add a few more milliliters of silver nitrate solution, stir well, heat, let the precipitate settle, and test again. Continue in this way until precipitation is complete. Let the covered beaker stand in the desk, protected from light, for 30 min. before filtration.

Preparation of the Gooch crucible:

Clean the crucible from surface contamination using soap and water, rinse, then place it in a crucible holder in a suction flask, if chemical cleaning is required, draw a little concentrated HNO_3 slowly through the porous filtering

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[4] The stirring helps prevent bumping of the solution during heating and the danger of loss of precipitate.

disc. With gentle suction, draw several small portions of distilled water through the filter. Mark the crucible with your name, and put it the oven at the drying temperature of the precipitate (120-130 ^{0}C) for half an hour. With clean crucible tongs, transfer the hot crucible to a desiccator, cool for 10 minutes, and weigh. Repeat drying until constant weights within ± 0.3 mg are obtained.

Filtration and washing of the precipitate

Decant the solution through the weighed crucible, pouring the solution down by stirring rod, and using gentle suction.

Prepare the wash solution by mixing 5 mL of concentrated HNO_3 with 200 mL water. To the precipitate in the beaker add about 25 mL of the wash solution, stir well, let the precipitate settle, and decant the solution through the filter crucible.

Repeat the washing by decantation two times, and finally bring the precipitate into the filter; use small portions of the wash solution for transfer. Remove with a rubber policeman any solid particles adhering to the beaker Continue washing the precipitate in the crucible with the wash solution until the last portions of washing give a negative test for silver ions. Silver ion is tested for by adding a drop of hydrochloric acid.

Drying and Weighing of the Precipitate:

Place the crucible containing the precipitate in the oven for (25-30) minutes at 120 -130 ^{0}C. Cool the crucible in the desiccator, and weigh accurately. Repeat for 15 minutes periods to obtain constant weights of ± 0.3 mg.

EXPERIMENT (4)
GRAVIMETRIC DETERMINATION OF CHLORIDE

Name: **Group NO.:**

III DATA

Volume of unknown solution used, mL	
Mass of empty crucible, g	
Mass of crucible + precipitate, g	
Mass of AgCl precipitate, g	

IV CALCULATION

1- Mass of Cl in the volume used, g =

2- Mass of Cl in 1L (g/L) =

V QUESTIONS

Q1) What is the effect of the following phenomena on the chloride determination:

a) Insufficient amount of $AgNO_3$ added.

b) Incomplete transfer of the precipitate.

c) Coprecipitation

d) Washing too little

e) Washing too much.

f) Incomplete drying of precipitate.

g) Photodecomposition of silver chloride.

Q2) What is digestion of a precipitate? Why is it necessary?

Q3) Dilute HNO_3 is used as a wash solvent. Explain?

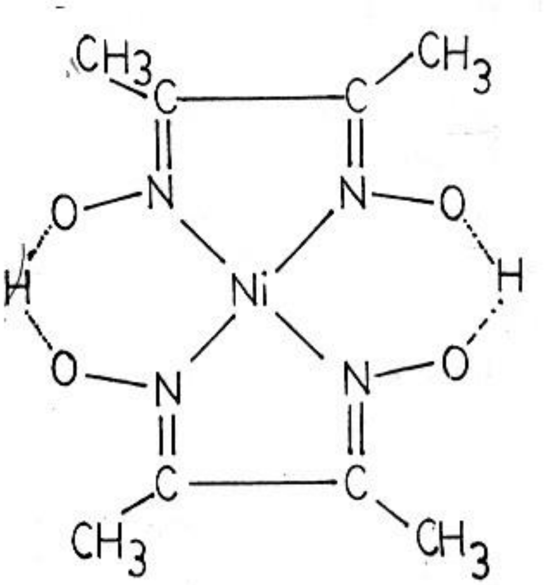
EXPERIMENT (5)
GRAVIMETRIC DETERMINATION OF Ni AS Ni(DMG)$_2$

I. INTRODUCTION

In the gravimetric determination of a substance the precipitating agent should be as specific as possible for that substance. Dimethylglyoxime (DMG) has a specificity to precipitate only palladium from acid medium and only nickel from weakly basic solutions, In ammoniacal solution, nickel is precipitated quantitatively by DMG as a bright strawberry complex, Ni(DMG)$_2$.

$$2HDMG + Ni^{2+} \rightleftharpoons Ni(DMG)_{2\,(s)} + 2H^+$$

The specificity of DMG arises from the unusual nature of the nickel chelate. Two DMG ligands are coordinated to each nickel ion, with the nickel and all four coordinating nitrogen atoms in the same plane:



Ni(DMG)$_2$ is bulky when first precipitated; the precipitate also tends to creep up the walls of the vessel containing it. For these reasons, only small quantities of nickel can be handled.

This method is used for gravimetric determination of nickel in steel.

II. EXPERIMENTML PROCEDURE:

Pipet 10 mL from the unknown nickel salt solution into a 400 mL beaker. Add about 5 mL of dilute HCl then dilute to about 200 mL with distilled water. Heat to 70-80 ^{0}C (use a thermometer).

Reduce the flame and add 30 mL of DMG. With good stirring, add dilute ammonia dropwise, until precipitation takes place and then add a slight excess. Heat very gently on a small flame and test the solution for complete precipitation when the red precipitate has settled out.

Allow the precipitate to stand for 20-30 minutes at room temperature. Clean up a gooch crucible and heat it to constant weight at 110-120 ^{0}C. Filter the cold solution through the crucible, wash the precipitate with warm water and then with 30% alcohol, to dissolve any excess DMG from the precipitate.

Finally, put the crucible in an oven at 110-120 ^{o}C for 30 minutes. Allow to cool in a desiccator and weigh. Repeat drying until constant weight is achieved.

EXPERIMENT (5)

GRAVIMETRIC DETERMINATION OF NICKEL AS BIS(DIMETHYLGLOXIMATO)-NICKEL(II) NI(DMG)$_2$

Name:	**Group NO.:**

III DATA

Volume of unknown solution used, mL	
Mass of empty crucible, g	
Mass of crucible + Ni(DMG)$_2$, g	
Mass of Ni(DMG)$_2$ precipitate, g	

IV CALCULATION

1- Mass of Ni in the volume used, g =

2- Mass of Ni in 1L (g/L) =

V QUESTIONS

Q1) Why precipitation is carried out in an ammonia solution?

Q2) Why a large excess of DMG should not be added?

Q3) Why ethanol is used for washing?

EXPERIMENT (6)

NEUTRALISATION TITRATION IN AQUEOUS MEDIUM

I. INTRODUCTION.

Neutralization titrations involve acid-base reactions. End point detection is based upon the abrupt change in pH at the equivalence point. In laboratory practice it is customary to prepare and standardize one solution of an acid and one of a base. These two solutions can then be used to analyze unknown samples of acids and bases. In this experiment a solution of about 0.1 M HCl is going to be standardized against a primary standard solution of sodium carbonate.

The standardized HCl solution will then be used for standardization of about 0.1 M NaOH solution. The standardized NaOH will be used for determination of phosphoric acid in commercial acid and for determination of the equivalent weight of an unknown organic acid.

II. EXPERIMENTAL PROCEDURE

a) Standardization of Sodium hydroxide

Weigh accurately about 1.50g of primary standard KHP in a weighing bottle. Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Adjust the volume to the mark and mix well, calculate the molarity of the KHP solution.

Pipet 10 mL of the KHP solution into an Erlenmeyer flask, add 3 drops of Phenolphthalein indicator and 20 mL distilled water then titrate with the NaOH solution until the solution changes from colorless to pink. Repeat three times and calculate the exact molarity of the NaOH solution.

b) Determination of Phosphoric acid in commercial acid

Phosphoric acid H_3PO_4 is a tribasic acid having three replicable hydrogen atoms. Neutralization of this acid leads to the production of the dihydrogen phosphate, the monohydrogen phosphate and the tribasic phosphate. The pH at the equivalence point at each stage is 4.6, 9.7 and 12.6 respectively. Thus, in the titration of this acid an appropriate indicator with a transition pH range which matches the pH of the stage at the equivalence point should be selected. No satisfactory indicator is known for the third stage.

1- Titrate 10.0 mL from the provided phosphoric acid with the 0.1M NaOH solution using 3-5 drops of methyl orange or bromocresol green as indicator. Repeat 2 times. Calculate the concentration of H_3PO_4 in the unknown phosphoric acid solution in grams per liter.

2- Titrate another 10.0 mL from the provided phosphoric acid with the 0.1 M NaOH using 3-5 drops of phenolphthalein indicator. Repeat two times. Calculate the concentration of H_3PO_4 in the unknown phosphoric acid solution in grams per liter.

c) Determination of the equivalent weight of an unknown organic acid. **(theoretical)**

Equivalent weight of a weak acid is useful for identification purposes. It can be determined by titration with a strong base using an indicator that changes color from pH 8 to pH 10. It is necessary to carry out preliminary solubility tests.

1- Weigh accurately about 0.3 g sample of the unknown acid. Transfer the sample into 200 mL beaker and dissolve it in about 50 mL of distilled water. In some cases, heating may be required to dissolve the sample. Also, sometimes it is necessary to dissolve the sample in an alcohol water mixture. Transfer the solution quantitatively into a 100 mL volumetric flask and dilute to the mark. Mix well.

2- Titrate 10 mL of the above solution with the provided 0.1M NaOH solution using 3-4 drops of phenolphthalein indicator. Repeat three times. Calculate the equivalent weight of the unknown acid.

EXPERIMENT (6)

NEUTRALISATION TITRATION IN AQUEOUS MEDIUM

Name:	**Group NO.:**

III DATA

d) a) Standardization of Sodium hydroxide

Weight of KHP (g)………………………….

	Trial 1	Trial2	Trial 3
Volume of KHP mL			
Volume of NAOH, mL			

Average vol. of NaOH……………… Molar Conc. NaOH………………….

b) Determination of Phosphoric acid in commercial acid

	Volume of H_3PO_4	Volume of NaOH	Indicator
Trial 1			BCG
Trial 2			BCG
Trial 3			Phph
Trial 4			Phph

Molar conc. H_3PO_4……….. Average vol. NaOH ……….	BCG
Molar conc. H_3PO_4……….. Average vol. NaOH ……….	Phph

III. CALCULATIONS

a) .a Standardization of Sodium hydroxide

-The balanced equation for the reaction is:

-Molarity of KHP(mol/L) =

-Molarity of NaOH (mol/L) =

b) (Determination of Phosphoric acid in commercial acid)

.b1 (Using BCG indicator)

-The balanced equation for the reaction is:

-Molar Conc. of H_3PO_4 (mol/L) =

-Conc. of H_3PO_4 (g/L) =

b2 (Using Phph. indicator)

The balanced equation for the reaction is:

-Molar Conc. of H_3PO_4 (mol/L) =

-Conc. of H_3PO_4 (g/L) =

EXPERIMENT (7)
THE NEUTRALIZING CAPACITY OF ANTACID TABLETS

Introduction:

Hydrochloric acid (HCl) is one of the substances found in gastric juices secreted by the lining of the stomach. HCl is needed by the enzyme pepsin to catalyze the digestion of proteins in the food we eat. Heartburn is a symptom that results when the stomach produces too much acid (hyperacidity).

Antacids are bases used to neutralize the acid that causes heartburn. Despite the many commercial brand, almost all antacids act on excess stomach acid by neutralizing it with weak bases. The most common of these bases are hydroxides, carbonates, or bicarbonates. The following table contains a list of the active ingredients found in several common commercial antacids, and the reactions by which these antacids neutralize the HCl in stomach acid.

Compound	Chemical Formula	Chemical Reaction
Aluminum hydroxide	$Al(OH)_3$	$Al(OH)_{3(s)} + 3HCl_{(aq)} \rightarrow AlCl_{3(aq)} + 3H_2O_{(l)}$
Calcium carbonate	$CaCO_3$	$CaCO_{3(s)} + 2HCl_{(aq)} \rightarrow CaCl_{2(aq)} + H_2O_{(l)} + CO_{2(g)}$
Magnesium carbonate	$MgCO_3$	$MgCO_{3(s)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + H_2O_{(l)} + CO_{2(g)}$
Magnesium hydroxide	$Mg(OH)_2$	$Mg(OH)_{2(s)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + 2H_2O_{(l)}$
Sodium bicarbonate	$NaHCO_3$	$NaHCO_{3(aq)} + HCl_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)} + CO_{2(g)}$

In this experiment, several brands of antacids will be analyzed to determine the number of moles of acid neutralized per tablet and the cost analysis of each tablet. The analytical procedure used is known as back titration. In this procedure, a known amount of HCl, which is in excess, will be reacted with a weighed portion of a ground antacid tablet. The HCl remaining after the antacid neutralization reaction occurs will be determined by titration with a standardized NaOH solution to a phenolphthalein OR bromothymol blue endpoint. The number of moles of HCl neutralized by the antacid (HCl neutralized) is the difference between the moles of HCl initially present in the excess (HCl added) and the moles of HCl titrated by the NaOH (HCl titrated)

EXPERIMENTAL PROCEDURE

a) Standardization of the sodium hydroxide solution

Weigh accurately about 1.50g of primary standard KHP in a weighing bottle. Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Adjust the volume to the mark and mix well, calculate the molarity of the KHP solution.

Pipet 10 mL of the KHP solution into an Erlenmeyer flask, add 3 drops of Phenolphthalein indicator and 20 mL distilled water then titrate with the NaOH solution until the solution changes from colorless to pink. Repeat three times and calculate the exact molarity of the NaOH solution.

b) Standardization of the HCl solution

Pipet 5.0 mL of about 0.2 M HCl solution into an Erlenmeyer flask. Add 25 mL of distilled water and 3 drops of phenolphthalein indicator. Titrate with the standard NaOH solution until the solution changes from colorless to pink. Repeat three times end calculate the exact molarity of the sodium hydroxide solution.

c) Determination of antacid capacity

1. Clean, rinse, and fill a burette with 0.100 M NaOH solution. Record the initial NaOH volume reading from the burette.
2. Weigh a sample of about (0.150-0.200) grams of crushed antacid tablet in a weighing bottle.
3. Transfer the sample to a clean 250 mL Erlenmeyer flask.
4. Add exactly 25.0 ml of 0.200 M HCl
5. Swirl to help dissolve the antacid. Since the antacid tablet may contain inert ingredients, much of the tablet may not dissolve and the liquid will be cloudy. The active antacid ingredient will dissolve and react with the acid very quickly while the liquid will remain cloudy.
6. Boil the solution to get rid of CO_2
7. Leave to cool
8. Add 3 - 4 drops of the ph.ph indicator to the flask, and titrate the acidic solution to the endpoint. Record the final NaOH volume.
9. Repeat this analysis on two other samples of antacid tablets.

EXPERIMENT (7)
THE NEUTRALIZING CAPACITY OF ANTACID TABLETS

Name:	**Group NO.:**

III DATA

a) Standardization of NaOH solution			
Weight of KHP (g)…………………………			
	Trial 1	Trial2	Trial 3
Volume of KHP, mL			
Volume of NaOH mL			
Average vol. of NaOH……………… Molar Conc. NaOH………………….			
b) Standardization of the HCl solution			
	Trial 1	Trial2	Trial 3
Volume of HCl, mL			
Volume of NaOH, mL			
Average vol. of NaOH……………… Molar Conc. HCl………………….			

c) Determination of Neutralizing capacity of Antacid		
	Trial 1	Trial 2
Mass of Antacid sample (g)		
Volume of HCl (mL)		
Moles of HCl (used to dissolved Antacid)		
Volume of NaOH added		
Capacity of Antacid (mole/g)		
Average Capacity of Antacid (mole/g)………………		

Calculations:

a) <u>Standardization of NaOH against KHP:</u>

- The balanced equation for the reaction is:

- Mass of KHP in g =

- Mole of KHP =

- Molarity of KHP (mol/L) =

- Avg. Molarity of NaOH (mol/L) =

b) <u>Standardization of HCl against NaOH:</u>

- The balanced equation for the reaction is:

- Avg. Molarity of HCl (mol/L) =

c) Determination of Capacity of Antacid:

- Assume one mole of Antacids reacts with one mole of HCl.

➢ Calculation for Trial 1:

1. Total moles of HCl added =
2. Moles of NaOH needed to neutralize excess HCl =
3. Moles of excess HCl =
4. Moles of acid needed to neutralize Antacid=
   (total moles of acid – moles of excess acid)
5. Capacity of Antacid for Trial 1=
   (Moles of acid needed to neutralize Antacid / mass of Antacid)

➢ Calculation for Trial 2:

Repeat the above calculation to find the Capacity of Antacid for Trial 2.

Capacity of Antacid for Trial 2 =

➢ Avg. Capacity of Antacid:

Avg. Capacity of Antacid =

Questions:

1- Why strong bases like NaOH cannot be used to neutralize the acid in the stomach?

2- Why was the back titration technique used in this experiment instead of direct titration?

3- A student is given 0.543g sample of Antacid to find its capacity and it’s found to be 0.00813 mol/g. The sample was dissolved and treated by 40.00 ml of 0.151 M of HCl, then the solution was titrated with 10.20 ml NaOH. Calculate the concentration of NaOH used.

EXPERIMENT (8)
PRECIPITATION TITRATIONS (ARGENTIMETRY)

I. INTRODUCTION

The general example of precipitation titrations is the argentimetric methods where silver nitrate, is used as the titrant. Argentimetric methods are used for determination of halides, cyanide, isocyante and to a less extent for phosphate, arsenate, sulphide and other ions. Silver may be determined by using some of these methods in reverse.

The general methods of argentimetry are:

- Mohr's method potassium chromate is used as indicator in a neutral solution (pH 6.5-9). A colored sparingly soluble salt will be formed at the equivalence point.

- Fajan's method: Adsorption indicators are used e.g., fluorescein and rhodamine. The role of these indicators depends upon their adsorption by the precipitate at the equivalence point which is accompanied also by a color change.

- Volhard's method: It is an indirect method, where a known excess of silver nitrate is added to the halide solution and the residual silver nitrate is back titrated with a standard thiocyanate solution using ferric ion as indicator.

II. EXPERIMENTAL PROCEDURE

(A) ~~Standardization of silver nitrate by Mohr's method.~~

1. ~~Dry sodium chloride at 105 °C 1-2 hours and weigh accurately about 0.60g. Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Complete to the mark. Shake well.~~
2. ~~Pipet 10 mL of the standard chloride solution into a 250 mL conical flask and titrate with the silver nitrate solution using 1 mL of the chromate indicator until a faint but distinct change in color occur (faint reddish-brown). Repeat the titration using 1 mL of the mixed indicator (chromate and dichromate) and titrate to the first permanent color change.~~
3. ~~Calculate the molarity and weight per liter of silver nitrate.~~

~~Use the calculated concentration only in part D.1~~

(B) Standardization of silver nitrate by Fajan's method

Dry sodium chloride at 105 °C 1-2 hours and weigh accurately about 0.60g.

Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Complete to the mark. Shake well.

Pipet 10 mL of the standard chloride solution into a conical flask, add 5 drops of the dichlorofluorescein indicator and titrate using the above standardized silver nitrate solution until the precipitate suddenly assumes a pronounced pink color. Repeat the titration and calculate the molarity and weight per liter of $AgNO_3$. Use the calculated concentration in parts C and D.2.

(C) Standardization of potassium thiocyanate by Volhard's method

Pipet 10 mL of the standard silver nitrate solution into a conical flask. Add about 2-3 mL of the 6 M HNO_3 and 1 mL of the ferric indicator solution. Titrate with thiocyanate solution. Shake vigorously during the titration until the red brown color of $FeSCN^{2+}$ is permanent for one minute.
Calculate the molarity and weight per liter of the thiocyanate solution.

(D) Determination of a mixture of halides (NaCl + KCl)

1. According to Mohr's method

Pipet 10 mL of the unknown mixture solution into a conical flask and add 1 mL chromate indicator (or the mixed indicator) and titrate with the standard silver nitrate. Find the volume of silver nitrate which reacts with the mixture. Duplicate the titration.

If W_1 = 0.0650 g is the weight of the mixture in 10.0 mL which contains x g of NaCl and y g of KCl then

$$W_1 = x + y = 0.0650 \text{ g}$$

$$\frac{x}{58.5} + \frac{y}{74.5} = M.V$$

Where M is the molarity of $AgNO_3$ and V is the volume of $AgNO_3$ required for the titration.

By solving the above two equations determine the weight of each of NaCl and KC1 per liter.

2. <u>According to Volhard's method</u>

Pipet 10.0 mL of the unknown mixture into a conical flask. Add an excess volume of 0.1 M $AgNO_3$ (15.0 mL exactly), about 2-3 m1 of 6 M HNO_3 and 1 mL of the ferric indicator solution, and 1 mL of nitrobenzene. Titrate with the standardized potassium thiocyanate solution according to Volhard's method. Duplicate the titration. Determine the weight of NaCl and KCl per liter. Compare the two results obtained from steps (1) and (2)

EXPERIMENT (8)
PRECIPITATION TITRATIONS (ARGENTIMETRY)

Name:	**Group No.:**

III DATA

Standardization of silver nitrate			
Weight of NaCl……………… g			
~~a) ***Mohr's Method***~~	~~Trial 1~~	~~Trial 2~~	~~Trial 3~~
~~Volume of NaCl~~			
~~Volume of $AgNO_3$~~			
~~Average vol. of $AgNO_3$…………mL Molar conc. $AgNO_3$…………….~~			
b) ***Fajan's Method***	Trial 1	Trial 2	Trial 3
Volume of NaCl			
Volume of $AgNO_3$			
Average vol. of $AgNO_3$…………mL Molar conc. $AgNO_3$…………….			
Standardization of potassium thiocyanate			
c) ***Volhard's Method***	Trial 1	Trial 2	Trial 3
Volume of $AgNO_3$			
Volume of KSCN			
Average vol. of KSCN…………….mL Molar conc. KSCN…………….			
Determination of a mixture of halides (NaCl + KCl)			
~~a) ***Mohr's Method***~~	~~Trial 1~~	~~Trial 2~~	~~Trial 3~~
~~Volume of unknown~~			
~~Volume of $AgNO_3$~~			
~~Average vol. of $AgNO_3$…………mL~~			
b) ***Volhard's Method***		Volume of $AgNO_3$……………..	
	Trial 1	Trial 2	Trial 3
Volume of unknown			
Volume of KSCN			
Average vol. of KSCN…………….mL			

Instructor's Signature

IV CALCULATIONS

• **~~Part A. (Mohr's method)~~**

~~-Molarity of NaCl (mol/L) =~~

~~-Molar Conc. of $AgNO_3$ (mol/L) =~~

~~-Weight of $AgNO_3$ (g/L) =~~

• **Part B. (Fajan's method)**

-Molar Conc. of $AgNO_3$ (mol/L) =

-Weight of $AgNO_3$ (g/L) =

• **Part C. (Volhard's method)** (Use the concentration of $AgNO_3$ from part B)

-Molar Conc. of KSCN (mol/L) =
-Weight of KSCN (g/L) =

- **Part D. (Mixture of halides NaCl and KCl)**

-Weight of mixture in one liter = 6.50 g
-Weight of mixture in 10 mL (W_1) = 0.0650 g

~~o **Part D.1 (Mohr's Method)** (Use the concentration of $AgNO_3$ from part A)~~

~~-Moles of the unknown =~~

~~-Weight of NaCl (x) and KCl (y) =~~

~~-Concentration of NaCl and KCl (g/L)=~~

o **Part D.2 (Volhard's Methods)** (Use the concentration of $AgNO_3$ from part B)

-Moles of $AgNO_3$ added =

-Moles of KSCN =

-Moles of Unknown =

-Weight of NaCl (x) and KCl (y) =

-Concentration of NaCl and KCl (g/L)=

V QUESTIONS

Q1) Why precipitation titrations by the Mohr's method require a neutral solution?

Q2) What is the effect of using acidic solution in the Fajan's method?

Q3) What is the effect of using $K_2Cr_2O_7$ instead of K_2CrO_4 as an indicator on the titration result?

Q4) What is the effect of using basic solution on the results in precipitation titration?

Q5) Why nitrobenzene is added in case of titration of Cl^- by Volhard's method?

EXPERIMENT (9)
REDOX TITRATION (DICHROMATE TITRATION)

I. INTRODUCTION

Potassium dichromate is an oxidizing agent which has advantages over potassium permanganate. It can be obtained in a state of high purity, its solution can stand for a long time without changing its titer, there is no stable oxidation states between +6 and +3, chloride ion in dilute solution is not oxidized by dichromate, and its solution can be boiled without decomposition. In spite of the above advantages of $K_2Cr_2O_7$ it is not as strong an oxidizing agent as $KMnO_4$.

The reaction of dichromate with reducing agents proceeds as follows:

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7H_2O$$

Indicators appropriate for these titrations are redox indicators. A redox indicator is a substance whose oxidized form is of different color from its reduced form. The change in color takes place at a definite potential. Thus, it is necessary that the redox indicator for a particular titration has a potential that coincides with that of the system.
Diphenylamine sulfonic acid and diphenyl bezidine sulfonic acid are the best indicators used with dichromate solution.

II. EXPERIMENTAL PROCEDURE

(a) Determination of chromium in a soluble chromic salt:

Chromic salts are oxidized to dichromate by boiling with an excess of a persulphate solution in the presence of a little $AgNO_3$. The dichromate resulted is determined by adding an excess of a standard ferrus solution and titration of the excess of Fe^{2+} with standard $K_2Cr_2O_7$.

1. Weigh out accurately about 0.5 g of the provided chromic salt, and dissolve it in about 30 mL of distilled water. Add about 5 mL of 0.1M $AgNO_3$ solution, followed by 10 mL of 10% solution of ammonium persulfate. Boil

the liquid gently for 20 minutes. Cool and dilute to 100 mL in a volumetric flask.

2. Pipet 10 mL of the above solution into a 250 mL conical flask, add 10 mL of the standard ferrous ammonium sulfate solution, 40 mL of 1M H_2SO_4, 2-4 drops of diphenylamine indicator and 3-5 mL of syrupy phosphoric acid. Titrate the excess ferrous ion with the standard $K_2Cr_2O_7$. Duplicate the titration. Calculate the percentage of chromium in the sample.

(b) Determination of Fe^{2+} and Fe^{3+} in a mixture

1- Determination of Fe^{2+} in the mixture

Pipet 10 mL of the provided mixture and add about 10 mL of 1M H_2SO_4 into a conical flask. Add 2-4 drops of diphenylamine indicator and about 5 mL of phosphoric acid (H_3PO_4). Titrate with the standard $K_2Cr_2O_7$ solution until the color changes to violet-blue. Repeat the titration and calculate the amount of iron (II) in a liter of mixture.

$$Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \longrightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

2- Determination of Fe^{3+} in the mixture

2.1. Preparation of the Johnes reductor (Zinc amalgam)

Into 250 mL conical flask, cover about 10 g of pure zinc (20-30 mesh) with 2 percent-mercuric chloride solution (about 10 mL). The mixture is stirred vigorously for about 5 minutes, and the solution is decanted off. Wash the zinc amalgam with distilled water 3-4 times by decantation.

2.2. Reduction procedure:

Add 10 mL of the unknown mixture and about 40 mL of 1M H_2SO_4 on the amalgamated zinc granules. Insert a funnel in the neck of the flask and boil the solution until the yellow color has disappeared entirely, this confirms that all Fe^{3+} has been reduced to Fe^{2+}.

To check the complete reduction of Fe^{3+}, one drop of the solution is added to a drop of NH_4CNS on a porcelain tile.
No pink color should appear.

When the solution has become colorless cool it thoroughly under the tap, wash the drops of liquid from the funnel into the flask. Insert a piece of glass wool (cotton wool as an alternative) into the funnel and filter the solution into a large conical flask (500 mL).

Wash the original flask, funnel and the glass wool several times with distilled water. Add about 5 mL of syrupy H_3PO_4 and 2-4 drops of diphenylamine indicator; titrate with the standard $H_2Cr_2O_7$ as in procedure (b1) above.

EXPERIMENT (9)
REDOX TITRATIONS (DICHROMATE TITRATIONS)

Name:	**Group NO.:**

III DATA

(b) Determination of Fe^{2+} and Fe^{3+} in a mixture		
Molarity of $K_2Cr_2O_7$..........................		
b.1) Determination of Fe^{2+} in the mixture		
	Trial 1	Trial2
Volume of unknown		
Volume of $K_2Cr_2O_7$		
Average vol. of $K_2Cr_2O_7$...................................		
b.2) Determination of Fe^{3+} in the mixture		
Volume of unknown		
Volume of $K_2Cr_2O_7$		
Weight of Fe^{2+} ………………. g / L	Weight of Fe^{3+} ………………. g / L	

Instructor's Signature

IV. CALCULATIONS

b. Determination of Fe^{2+} and Fe^{3+} in a mixture

b.1 Determination of Fe^{2+} in the mixture

- Molar conc. of Fe^{2+} (mol/L) =

- Weight of Fe^{2+} (g/L) =

b.2 Determination of Fe^{3+} in the mixture

- Moles of Fe^{2+} in the mixture =

- Moles of Fe^{2+} and Fe^{3+} in the mixture =

- Moles of Fe^{3+} in the mixture =

- Molar conc. of Fe^{3+} (mol/L) =

Weight of Fe^{3+} (g/L) =

V. QUESTIONS

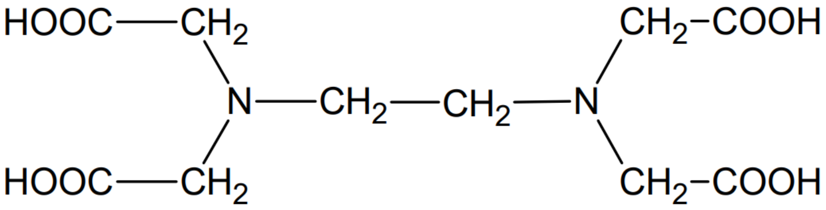
Q1) What is the role of H_3PO_4 in determination of Fe^{2+} by titration with $K_2Cr_2O_7$?

Q2) Can you determine iron ores using $K_2Cr_2O_7$?

EXPERIMENT (10)
COMPLEXOMETRIC TITRATIONS (TITRATIONS WITH EDTA)

I. INTRODUCTION

Most complexometric titrations involve the titrant ethylendiamine tetraacetic acid (EDTA):



EDTA forms stable, water soluble 1:1 chelates with polyvalent metal ions. It can be obtained in high purity as the free acid H_4Y and as the disodium salt Na_2H_2Y. The salt can be dissolved directly in water; the acid requires sodium hydroxide to be added for dissolution. A titrant solution prepared in either way is stable indefinitely. The usefulness of EDTA as a titrant is due to the presence of four or six atoms which are available for coordination to a metal cation in such a way that stable five membered rings are produced.

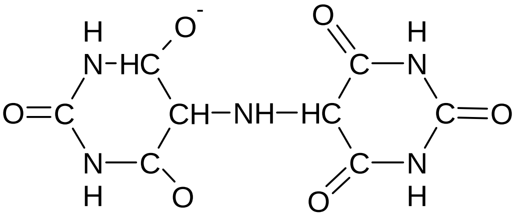
EDTA is a tetraprotic acid. The values of the four acid dissociation constants are 1.0×10^{-2}, 2.1×10^{-3}, 6.9×10^{-7} and 7.4×10^{-11}. From these values it is evident that two of the protons are strongly acidic and two are weakly acidic. From the values of these constants it can also be concluded that in a solution of about pH 5 (closely attained in a solution of the disodium salt of EDTA) the predominant species is H_2Y^{2-}. If EDTA is formally represented by H_4Y at that pH, the formation of a 1:1 complex (which is one of the most interesting features of EDTA) between EDTA and a metal ion, M^{n+} , can be represented as:

$$M^{n+} + H_2Y^{2-} \rightleftharpoons MY^{(n-4)} + 2H^+$$

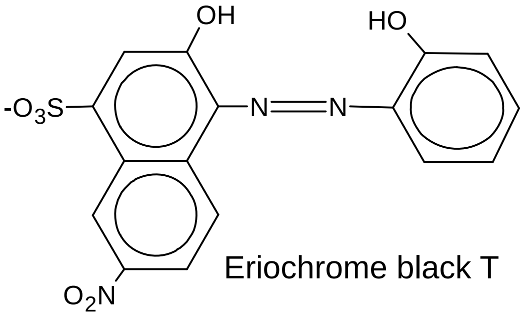
as we can see, two hydrogen ions are freed. This reaction is reversible, and consequently the more the complex is extensively dissociated the higher the acidity of the solution. In other words, the proton competes with metal ion for complexation with the EDTA.

This is an important fact because it requires the performance of EDTA titration above a certain limiting pH value; otherwise; the complex dissociates to a great extent and the titration fails. The limiting pH value depends on the stability of the complex formed during the titration. The lower the stability of the relevant metal-EDTA complex is, the higher the pH value must be, in some cases, it is advisable to do the reaction in a buffer medium.

Indicators appropriate for these titrations are organic dyes that form colored chelates with metal ions which must be weaker than the chelate formed by the metal with EDTA. Most of these indicators possess acid-base indicator properties, thus a strict control of the pH is necessary. The most widely used indicators are Murexide and Eriochrome black T:

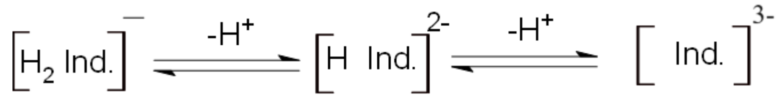


Murexide



Eriochrome black T

The acidic proton of the sulfonic acid group dissociates in the pH-range of interest 7-12. The formula of the Eriochrome black T indicator can be written as H_2Ind^-. The color of the indicator changes as follows:



$$[H_2\ Ind.]^{-} \underset{}{\overset{-H^+}{\rightleftharpoons}} [H\ Ind.]^{2-} \overset{-H^+}{\rightleftharpoons} [Ind.]^{3-}$$

pH = 6.3 pH = 11.5

wine red deep blue orang

II. EXPERIMENTAL PROCEDURE

A. Standardization of about 0.01M EDTA solution with a standard zinc sulfate solution:

1. Weigh accurately about 0.60-0.70 g of A.R. Zinc sulfate ($ZnSO_4.7H_2O$). Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Complete the volume to the mark using distilled water.
2. Pipet 10 mL of $ZnSO_4$ solution into a titration flask and add 5 mL of ammonia-ammonium chloride buffer solution followed by 5-6 drops Eriochrome black T indicator.
3. Titrate with about 0.01 M EDTA solution until the color changes from wine red through purple to pure blue. Repeat the titration three times and calculate the exact molarity of the EDTA solution.

B. Determination of water hardness

1. Pipet 50 mL of tap water into a conical flask. Add 2 mL of ammonium chloride buffer solution and 5-6 drops of Eriochrome black-T indicator.
2. Titrate with the standard EDTA solution until the color changes from wine red to pure blue. This titration determines the sum of calcium and magnesium (V_1), repeat one more time.
3. Pipet another 50 mL of the tap water into a titration flask. Add 1 mL of 5.0 M NaOH to precipitate the Mg^{2+} ions as $Mg(OH)_2$. Then add 5-6 drops murexide indicator.
4. Titrate until the color changes from pink to purple. This titration determines the calcium only (V_2). The volume corresponding to magnesium is ($V_1 – V_2$). Repeat the titration one more time.

Calculate the calcium and magnesium content in ppm.
Calculate the total water hardness as mg $CaCO_3$/L.

Determination of the concentration of calcium and magnesium in an unknown solution:

1. Pipet 10 mL of your unknown solution into a conical flask. Add about 25 mL distilled water, 2 mL of ammonia-ammonium chloride buffer and 5-6 drops of the Eriochrome black-T indicator. Titrate with the standard EDTA solution until the color changes from wine red to pure blue. Repeat one more time.

2. Pipet another 10 mL of your unknown solution into a conical flask. Add about 25 mL distilled water, 1 mL of 5 M NaOH and 5-6 drops murexide indicator. Titrate with the standard EDTA solution until the color changes from pink to purple. Repeat the titration one more time.
   Calculate the concentration of calcium and magnesium in your unknown solution in ppm.

EXPERIMENT (10)

COMPLEXOMETRIC TITRATIONS (TITRATIONS WITH EDTA)

Name: **Group NO.:**

III DATA

Standardization of about 0.01M EDTA

Weight of $ZnSO_4.7H_2O$………………………g

	Trial 1	Trial 2	Trial 3
Volume of $ZnSO_4.7H_2O$			
Volume of EDTA			

Average vol. of EDTA……………..mL Molar conc. EDTA……………..mol/L

Determination of water hardness

	Volume of H_2O	Volume of EDTA	Note
Trial 1 (V1)			------------
Trial 2 (V1)			
Trial 1 (V2)			In present of NaOH
Trial 2 (V2)			

Average V1 =.. Average V2 =...

Conc. of $CaCO_3$……………………………mg/L

Determination of the concentration of Ca and Mg in an unknown solution

	Volume of unknown	Volume of EDTA	Note
Trial 1 (V1)			------------
Trial 2 (V1)			
Trial 1 (V2)			In present of NaOH
Trial 2 (V2)			

Average V1 =.. Average V2 =...

ppm Ca……………….……………… ppm Mg……………………………

Instructor's Signature

IV. CALCULATIONS

- Part II.A. (Standardization of about 0.01M EDTA solution)

  -Molar conc. of Zn^{2+} (mol / L)

  -Molar Conc. of EDTA (mol / L)

- Part II.B. (Determination of water hardness)

  -Conc. of Ca (ppm)

  -Conc. of Mg (ppm)

  -Total water hardness, as mg $CaCO_3$ /L

- Part II.C. (Determination of the conc. of Ca and Mg in an unknown solution)

  -Conc. of Ca (ppm)

  -Conc. of Mg (ppm)

EXPERIMENT (11)
DETERMINATION OF UNKNOWN BASES BY STANDARDIZED HCl

I. EXPERIMENTAL PROCEDURE

a) Standardization of hydrochloric acid against sodium carbonate

1. Weight out four 0.2 g sodium carbonate sample by difference from the weighing bottle (to avoid absorption of moisture) into 250 mL flasks and dissolve each of them in 50-100 mL of distilled water.

2. Add 2-3 drops of phenolphthalein indicator to each of the titration flask, and then titrate with the HCl solution until the solution changes from red to colorless. Calculate the molarity of the HCl solution, repeat one more time.

$$Na_2CO_3 + HC1 \longrightarrow NaHCO_3 + NaC1$$

b) Determination of the composition of the unknown

1. Weight out 1.00-1.50 g samples into 100 mL Volumetric flasks and complete to the mark with distilled water. Pipette 10mL of the solution into 250mL flask (**four times**). Titrate the first two samples with HCl using bromocresol green indicator and the second two samples using phenolphthalein indicator. Use the data in Table (1) to determine the composition of the unknown, and then calculate the percentage of each component in the unknown.

Table (1): Volume relationships in the analysis of mixtures containing hydroxide, carbonate, and hydrogen carbonate ions.

Constituents in sample	Relation between V_{Phph} and V_{BCG}
NaOH	$V_{Phph} = V_{BCG}$
Na_2CO_3	$V_{Phph} = 1/2\ V_{BCG}$
$NaHCO_3$	$V_{Phph} = 0; \quad V_{BCG} > 0$
NaOH , Na_2CO_3	$V_{Phph} > 1/2\ V_{BCG}$
Na_2CO_3, $NaHCO_3$	$V_{Phph} < 1/2\ V_{BCG}$

EXPERIMENT (11)

DETERMINATION OF UNKNOWN BASES BY STANDARDIZED HCI

Name: **Group NO.:**

II DATA

a) Standardization of hydrochloric acid against sodium carbonate			
	Trial 1	Trial2	Trial 3
Weight of Na_2CO_3, g			
Volume of HCl, mL			
Molar Conc. HCl, mol/L			
Average molar conc. of HCl ………………..			

b) Determination of the composition of the unknown						
	Phph			BCG		
	Trial 1	Trial2	Average	Trial 1	Trial2	Average
Volume, mL						
Wt. of unknown						
The first component of the unknown is ……………… and its Molarity ……………M						
The second component of the unknown is …………… and its Molarity ……………M						

Instructor's Signature

III. CALCULATIONS

- Molar conc. of HCl (mol / L)

- Percentage of the first component

- Percentage of the second component

- IV. QUESTIONS

Q1) If a basic unknown solution was titrated with Phenolphthalein and need 20ml and when it titrated with bromocresol green needs 40ml, the unknown will consist of which type of basic material?

Q2) where you can find the mix of these basic materials in the environments?

1A	2A											3A	4A	5A	6A	7A	8A
1 **H** 1.008																	2 **He** 4.003
3 **Li** 6.941	4 **Be** 9.012											5 **B** 10.81	6 **C** 12.01	7 **N** 14.01	8 **O** 16.00	9 **F** 19.00	10 **Ne** 20.18
11 **Na** 22.99	12 **Mg** 24.31											13 **Al** 26.98	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.07	17 **Cl** 35.45	18 **Ar** 39.95
19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.88	23 **V** 50.94	24 **Cr** 52.00	25 **Mn** 54.94	26 **Fe** 55.85	27 **Co** 58.93	28 **Ni** 58.69	29 **Cu** 63.55	30 **Zn** 65.38	31 **Ga** 69.72	32 **Ge** 72.59	33 **As** 74.92	34 **Se** 78.96	35 **Br** 79.90	36 **Kr** 83.80
37 **Rb** 85.47	38 **Sr** 87.62	39 **Y** 88.91	40 **Zr** 91.22	41 **Nb** 92.91	42 **Mo** 95.94	43 **Tc** (98)	44 **Ru** 101.1	45 **Rh** 102.9	46 **Pd** 106.4	47 **Ag** 107.9	48 **Cd** 112.4	49 **In** 114.8	50 **Sn** 118.7	51 **Sb** 121.8	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
55 **Cs** 132.9	56 **Ba** 137.3	57 **La*** 138.9	72 **Hf** 178.5	73 **Ta** 180.9	74 **W** 183.9	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.1	79 **Au** 197.0	80 **Hg** 200.6	81 **Tl** 204.4	82 **Pb** 207.2	83 **Bi** 209.0	84 **Po** (209)	85 **At** (210)	86 **Rn** (222)
87 **Fr** (223)	88 **Ra** 226	89 **Ac**† (227)															

*Lanthanides	58 **Ce** 140.1	59 **Pr** 140.9	60 **Nd** 144.2	61 **Pm** (145)	62 **Sm** 150.4	63 **Eu** 152.0	64 **Gd** 157.3	65 **Tb** 158.9	66 **Dy** 162.5	67 **Ho** 164.9	68 **Er** 167.3	69 **Tm** 168.9	70 **Yb** 173.0	71 **Lu** 175.0
†Actinides	90 **Th** 232.0	91 **Pa** (231)	92 **U** 238.0	93 **Np** (237)	94 **Pu** (244)	95 **Am** (243)	96 **Cm** (247)	97 **Bk** (247)	98 **Cf** (251)	99 **Es** (252)	100 **Fm** (257)	101 **Md** (258)	102 **No** (259)	103 **Lr** (260)