General laboratory Rules

1- Regular and prompt attendance of all scheduled laboratory
2- You must be prepared for laboratory . this includes goggles . calculators , pens . notebook and proper attire.
3- Students reports must be written and prepared individually .
4- Be prepared for the experiment.
5- Goggles are to be worn at all times in the laboratory . if you need to remove your goggles . step out of the room and into the hallway.
6- Wear appropriate clothing . cloth covered shoes , loos clothing, shorts or cut-off that expose open skin to boiling water or chemical reagents.
7- Eating and drinking are not permitted in the laboratory.
8- If chemicals are spilled onto your body , or into your eyes, immediately wash the areas with large amounts of water .
9- Keep clean bench-top . Clutter usually results in broken glass.
10- Inform the instructor of any accidents or problems encountered.

Formal report instructions general chemistry laboratory

The formal report should include the following :

Cover page

The cover page is to have on it the following :your Name ;your partner(s) Name(s) in parentheses ; title of experiment; course title and number; and the date . the body of the report should start on a separate page. Each section should be clearly labeled as follows:

1. **The purpose of experiment**

The purpose statement gives the underlying reason for doing the experiment .

2. **Introduction (the theory)**

   In the introduction you should write in both words and equations a description of the chemistry that you are expecting to carry out.

3. **procedure (Method)**
4. **Data**

   This section contains observations and data organized neatly in a table.

5. **Calculations and Graphs**

   Show calculations that lead from data to results. Calculations should be readable.

6. **Results**

   This section summarizes any conclusions you can make based upon your data and calculations.

7. **Discussion**

   The discussion section is a detailed evaluation of the procedure and results . Discuss possible errors that would lead to your results being incorrect or inconsistent. Include in this section the answers to any questions that were asked in the lab manual.

8. **References**

   A complete list of all references which provides the title of the reference, author or editor, publisher , year of publication, and page(s) used. For web site references give the complete URL.

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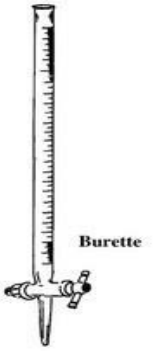
General chemistry Equipments

Picture	Name	Description
	Beaker	Used to hold and heat liquids. Multipurpose and essential in the lab .
	Conical Flask Erlenmeyer Flask	The Erlenmeyer Flask is used to heat and store liquids.
	Cylinder	Use to measure the volume of liquids
	Burette	The burette is used in titrations to measure precisely how much liquid is used.

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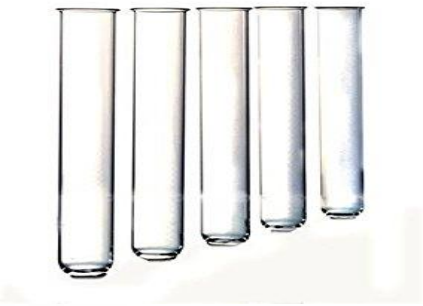
	Stand	Stand is used to hold items using clamps or rings
	Clamp	Used to catch items
	Ring Stand	Ring stands are used to hold items being heated .
	Test Tube	Used in chemical reactions
	Test Tube rack	The test tube rack is used to hold test tubes while reactions happen in them or while they are not needed .

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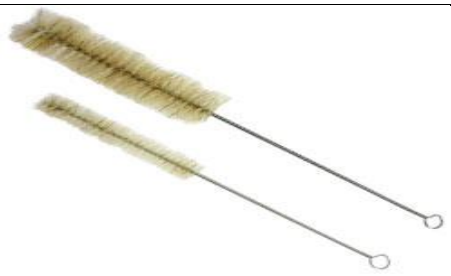
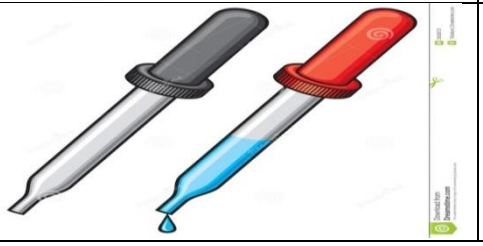
	Test tube holder	The holder is used to hold test tubes when they are hot and untouchable.
	Test tube brush	The test tube brush is used to easily clean the inside of a test tube.
	Pipette	The pipette is used for moving small amounts of liquid from place to place
	Volumetric Flask	The volumetric flask is used to measure one specific volume.
	Wash bottle	Used to wash the glassware
	Dropper	Use to add drops of liquid

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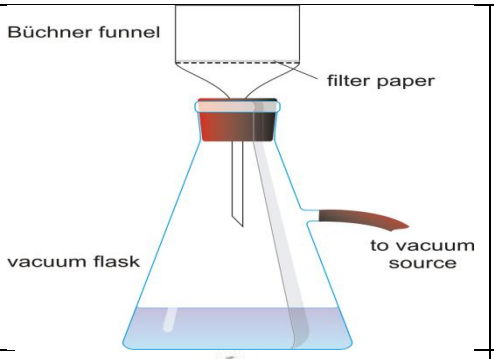	Buchner funnel And Filtering flask	Used to rapidly filtration
	Triangle	The triangle is used to hold crucibles when they are being heated. they usually sit on a ring stand
	Funnel	The funnel is a piece of equipment that is used in the lab but is not confined to the lab. the funnel can be used to target liquids in to any container so they will not be lost or spilled
	Watch Glass	The watch glass is used to hold solids when being weighed or transported. they should never be heated.
	Evaporating Dish	The Evaporating Dish is used to heat and evaporate liquids.

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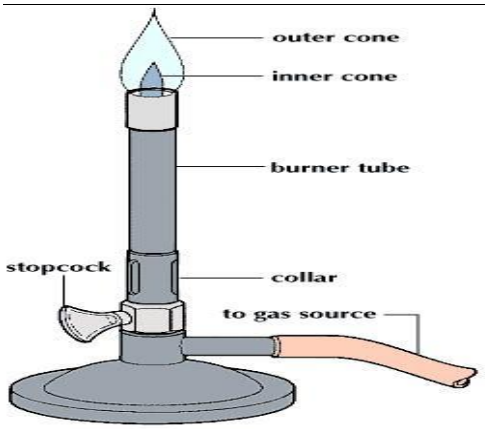	Bunsen burner	Bunsen burners are used for heating and exposing items to flame. They have many more uses than a hot plate but do not replace a hot plate.
	Electronic balance	An electronic balance ,directly measures the mass of an object placed on the weighing pan.
	High sensitivity Balance	Use to measure the mass of an object in high accuracy.
	Hot plate	Use to heat objects.
	Crucibles	Crucibles are used to heat small quantities to very high temperatures.

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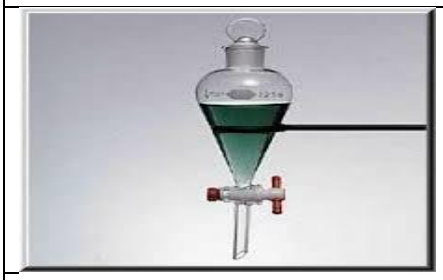
	Tripod Stand	Use to hold objects on it to heating them with Bunsen burners.
	Thermometer	The thermometer is used to take temperature of solids ,liquids, and gases. they are usually in ^{0}C , but can also be in ^{o}F
	Mortar and pestle	the mortar and pestle are used to crush solids into powders for experiments, usually to better dissolve the solids.
	Separation funnels	Use to separate two unmixable liquids.
	Spatula	The spatula, is used for moving small amounts of solid from place to place.

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Lab.2

Type of Analytical Methods

1-Qualitative Analysis

2- Quantitative Analysis

Qualitative analysis:

to express the composition of substances and to describe the qualitative and qualitative change which occur during chemical reactions in a precise . short , and straightforward way we use chemical symbols and formulae .

qualitative analysis may be carried out on various scales > in macro analysis the quantity of the substance employed is (0.1-1) gram and the volume of solution taken for the analysis is about 20ml , in what is usually termed semi-micro analysis , the quantity used for analysis is reduced by a factor of (0.1-0.05) gram and the volume of solution to about 1ml , for micro analysis the factor is of the order of 0.01 or less.

General description of analytical groups

For the purpose of systematic qualitative analysis, cations are classified into five groups on the basis of their behavior against some reagent called GROUP REAGENTS. Classification is based on whether a cation reacts with these reagents with the formation of the precipitate or not.

Group I

Cations of this group form precipitates with dilute hydrochloric acid. Ions of this group are lead Pb^{2+}, mercury(I) Hg_2^{2+} and silver Ag^{+}.

Group II

The cations of this group are divided into IIA and IIB groups. IIA contains mercury(II) Hg^{2+}, copper Cu^{2+}, bismuth Bi^{3+}, cadmium Cd^{2+} and lead Pb^{2+}. The presence of lead cations in groups I and IIA is caused by partial solubility of $PbCl_2$ in diluted hydrochloric acid and for that reason lead

ions, if present in the sample, are not completely precipitated with Group I and are carried over into Group IIA. The IIB group contains arsenic As^{3+}, antimony Sb^{3+} and tin Sn^{2+}. We will not test for the Pb^{2+} and Group IIB cations in the qualitative analysis of Group II cations.

Group III

The cations of this group do not react either with diluted hydrochloric acid, or with hydrogen sulfide in diluted mineral acid medium. However they form precipitates with ammonium sulfide in neutral or ammoniac medium. Cations of this group are: cobalt Co^{2+}, nickel Ni^{2+}, iron(II) Fe^{2+}, iron(III) Fe^{3+}, chromium(III) Cr^{3+}, manganese(II) Mn^{2+}, aluminum Al^{3+} and zinc Zn^{2+}.

Group IV

The cations of this group do not react with the reagents of Groups I, II and III. They form precipitates with ammonium carbonate in the presence of ammonium chloride and ammonia. Cations of this group are: calcium Ca^{2+}, strontium Sr^{2+} and barium Ba^{2+}.

Group V

Common cation, which do not react with reagents of the previous groups, form the last group of cations . They are: magnesium Mg^{2+}, sodium Na^{+}, potassium K^{+} and ammonium NH^{4+}.

Exercise 1: Separation of I Group cations (Ag^{+}, Pb^{2+}, Hg_2^{2+})

Group I cations (Ag^{+}, Hg_2^{2+}, Pb^{2+}) form insoluble chlorides. Upon the addition of hydrochloric acid Ag^{+}, Pb^{2+}, Hg_2^{2+} ions will precipitate as AgCl, $PbCl_2$ and Hg_2Cl_2

(reaction 1)…… ??
(reaction 2) ……??
(reaction 3)……??

The solubility of $PbCl_2$ increases approximately threefold as the temperature of the solution increases from 20°C to 100°C. Thus, $PbCl_2$ will dissolve in hot water while AgCl and Hg_2Cl_2 remains insoluble. The

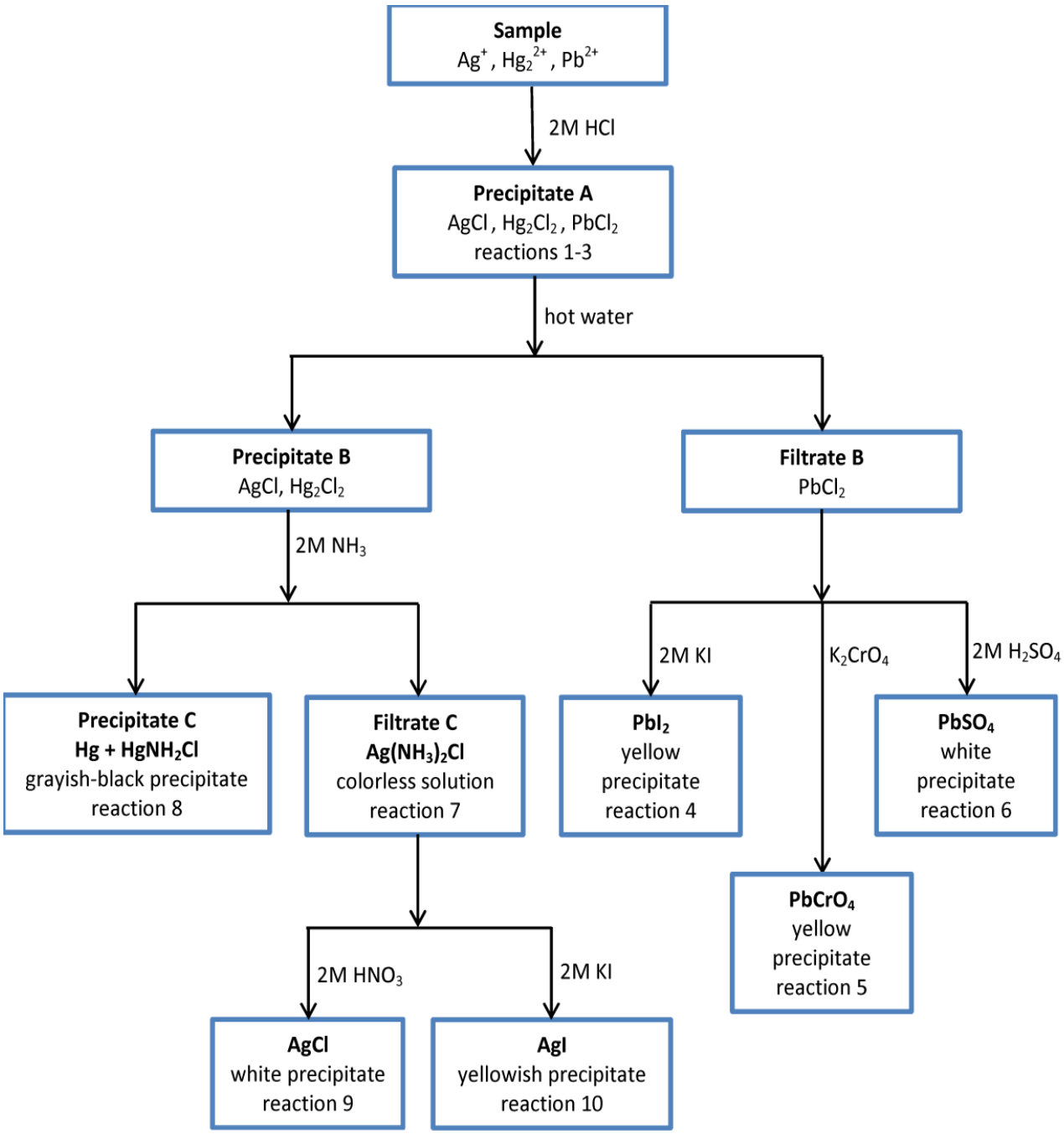
presence of Pb^{2+} ions in obtained solution can be proved with KI, K_2CrO_4 and H_2SO_4 solutions. Their addition yield a golden yellow precipitates of PbI2 and $PbCrO_4$ (reactions 4 and 5), and white precipitate of $PbSO_4$ (reaction 6).

The precipitate still may contain Hg_2Cl_2 and AgCl . Of those two compounds, only the silver chloride is soluble in aqueous ammonia due to the formation a colorless solution of $Ag(NH_3)_2Cl$ (reaction 7), whereas mercury(I) chloride turns into Hg metal and $HgNH_2Cl$ visible as grayish-black precipitate which is insoluble in ammonia solution (reaction 8)

The formation of white precipitate of AgCl in reaction of $Ag(NH_3)_2Cl$ with diluted HNO_3 proves the presence of silver (reaction 9). The additional confirmation of silver ions is yellowish precipitate of silver iodide AgI obtained in the reaction with potassium iodide KI (reaction 10).

2. The identification of the sample composition

To identify cations present in the given sample you can follow the *Scheme 1* presented below with the use of flow chart.



Scheme 1. Analysis of the Group I.

Procedure

1). Take 5 ml of a sample destined for identification and add 10 ml of 2M HCl . You should obtain white precipitate (Precipitate A).

2). Separate the precipitate by the filtration and wash it twice with cold distilled water and discard the washing.

3). Put clean test tube under the funnel with the precipitate. Then wash the precipitate with 10 ml of hot distilled water and collect the supernatant (Filtrate B). It is very important to remove all $PbCl_2$ from the precipitate to follow further analysis of remaining AgCl and Hg_2Cl_2 which may be present in the Precipitate B.

4). Now you can confirm the presence of Pb^{2+} ions. In this purpose divide the solution of Filtrate B into three part and add KI, K_2CrO_4 and H_2SO_4 to each test tube respectively. Addition of those chemicals successively will yield a golden yellow precipitates of PbI_2 (reaction 4) and $PbCrO_4$ (reaction 5), and white precipitate of $PbSO_4$ (reaction 6).

5). After confirming the presence of Pb^{2+} ions in the solution wash the Precipitate B with additional portion of hot water until the washing give no precipitate with K_2CrO_4 solution.

White Precipitate B may still contain Hg_2Cl_2 and AgCl. Of those two compounds, only the silver chloride is soluble in aqueous ammonia due to the formation a colorless solution of $Ag(NH_3)_2Cl$ (Filtrate C, reaction **7)**. Mercury(I) chloride reacts with ammonia solution to form grayish-black precipitate of Hg metal and HgNH2Cl which are insoluble in ammonia solution and remain on the filter (Precipitate C, reaction 8).

6). Pour ammonia solution onto the filter with Precipitate B and collect colorless solution in clean test tube (Filtrate C). Observe also the filter, because the precipitate may dissolve completely or turn black. If the

color will change into black it means that Hg_2^{2+} is present in the initial sample. This is the final confirmation of the Hg_2^{2+} presence.

The lack of precipitate indicate the absence of Hg_2Cl_2 and the lack of Hg_2^{2+} cations in the initial sample. Therefore, the last step is to follow the test for Ag^+ cations which is given below.

7). Divide the Filtrate C into two parts. To the first one add dilute 2M nitric acid HNO_3 (reaction 9). The formation of white precipitate of AgCl prove the presence of silver. To the second test tube add the solution of potassium iodide KI. Yellowish precipitate of silver iodide AgI is the additional confirmation of the presence of silver ions in the sample (reaction 10).

As you perform the experiment, collect discard all in the appropriate waste containers. DO NOT POUR ANY OF THE SOLUTIONS DOWN THE DRAIN.

Results:

Prepare your results for Group I on the report sheet provided. Be sure to include your all positive cations present in your initial sample. Use correct formulas for reagents and products.

Volumetric analysis

Some terms used in volumetric analysis

Quantitative Analysis : the Quantitative analysis it is including volumetric and Gravimetric analysis.

Volumetric method: A volumetric method is one in which the analysis is completed by measuring the volume of a solution of Known concentration needed to react completely with the substance being determined.

Titration: is a process for determining the amount of a substance by measurement of the quantity of a reagent (titrant) required to react completely with that substance.

There are a required for titrations :

The reaction should be equilibrium which is mean the reaction between analyst and standard solution have a chemical equilibrium between equivalent .

$CH_3COOH + NaOH \longleftrightarrow CH_3COONa + H_2O$

A standard solution : is a reagent of exactly known composition used in a titration .

A primary standard : is a highly purified chemical compound .

NaOH is not standard solution because it is absorb CO_2 from air to compound Na_2CO_3 therefore the concentration of NaOH is not stable and we should titrated with primary standard solution like potassium phthalate ($C_8H_5O_4K$) .

Standardization : is a process whereby the concentration of a standard solution is determined by titrating with a primary standard

Requirements of primary standard :

1- It is must be of the highest purity .
2- It should be stable and not attacked by atmosphere.

3- It should not be hygroscopic.
4- it should be available and not too expensive.
5- It should have high equivalent to minimize weighing errors.

The equivalent point : is the point where the amount of standard solution added is chemically equivalent to substance with which it reacts.

The End point of a titration : is the point at which physical changes associated with the equivalent point can be observed .

Indicator : is a chemical compound that exhibits a change in colour as a result of result of concentration changes occurring near the equivalent point .

Volumetric calculations :

The concentration of solution can be expressed in several ways:

1- **Normality :** is the number of equivalent weight contained in one liter of solution .
2- **Molarity :** is the number of gram molecular weights or the number of moles of solute in one liter of solution.
3- **Formality :** is the number of gram formula weights per liter of solution .

Volumetric methods can be divided in to four types :

1- Acid –Base titrations
2- Oxidation –reductions titrations
3- Complex metric titrations
4- Precipitations titrations

Lab. 3 **Acid – Base Titration**

preparation and standardization of an acid

a) Preparation of 0.1N HCL from concentrated HCL:-

$$N = \frac{\text{specific gravity} \times \%W/W \times 1000}{\text{Eq.wt of Hcl}}$$

Sp.g = 1.18

Eq.wt of HCl = 36.5

%w/w of HCl is obtained from the reagent bottle

N of concentration HCl =12

To calculation the volume of concentrated HCl that should be taken to prepare 250ml of 0.1N HCl

N_1V_1 of conc. = N_2V_2 of diluted

$12 \times V_1 = 0.1 \times 250$

$V_1 = 2.2$ ml

Should be taken from concentrated HCl and should be diluted with distilled water in 250 ml volumetric flask to obtaine 0.1N HCl .

b) Standardization of prepared of 0.1N HCl solution :-
   1- Fill the burette with the prepare HCl solution .
   2- Transfer 10ml of exactly 0.1N Na_2CO_3 (primary standard) into a conical flask by using a bulb pipette (0.1N Na_2CO_3) solution is prepared by weighing exactly 5.3 gm of Na_2CO_3 and diluted to 1000 ml with distilled water in a volumetric flask .
   3- Add 2 drop of methyl orange as an indicator , Yellow color is obtained
   4- Titrate with HCl solution drop by drop from the burette into the conical flask until a faint orange color is obtained.

5- The exact normality of HCl can be calculated from the following equation.

$Na_2CO_3 + HCl \longleftrightarrow 2NaCl + H_2O + CO_2$

C) Determination of the normality of 0.1 N NaOH solution :-

1) Preparation of an 0.1 N NaOH dissolve and dilute to one liter with distilled water .
2) Standardization of 0.1 N NaOH solution :
   a. Transfer 10 ml of standard HCl solution to a conical flask .
   b. Add 1-2 drop of ph-ph as an indicator .
   c. Fill the burette with the prepared NaOH solution .
   d. Add NaOH drop by drop into the conical flask until the color of the solution is faint pink .
   e. The exact normality of NaOH is obtained from :-

$NaOH + HCl \longrightarrow NaCl + H_2O$

$N_1 V_1 (NaOH) = N_2 V_2 (HCl)$

Lab -4 Determination of the percentage of acetic acid in a vinegar sample

Vinegar is essentially a solution of acetic acid CH_3COOH in water .

Acetic acid is an example of a carboxylic acid .

Acetic acid reacts with sodium hydroxide , a base , according to the reaction :

$$CH_3COOH + NaOH \longrightarrow H_2O + CH_3COONa$$

This is an example of an acid – base neutralization reaction in which an acid and base react to produce water plus a salt.

Procedure:

i. Preparation of unknown acetic acid solution :

Transfer 10 ml of unknown into 100 ml volumetric flask . complete the volume with distilled water stopper the flask and shake well .

ii. Determination of the % W/v of acetic acid in vinegar .
   a. Transfer with a bulb pipette 10 ml of the prepared solution to a conical flask . Add 2 drop of ph- ph as an indicator .
   b. Fill the burette with 0.1 N NaOH solution .
   c. Titrate the prepared acetic acid solution (diluted unknown) with standard NaOH until the color of the indicator becomes faint pink .
   d. The normality of NaOH must be determined by titration with standard HCl (mentioned before) .

   NV (HCl) = NV (NaOH)

iii. Calculation :

$N_1 V_1$ (HAC) = $N_2 V_2$ (NaOH)

$$N_2 V_2 \text{ (NaOH)} = \frac{\text{wt (HAC)}}{\text{Eq. wt.}} \times 1000$$

$$\text{Wt (HAC)} = \frac{\text{(NV) NaOH} \times \text{eq.wt. HAC}}{1000}$$

= gm of acetic acid (HAC) in 10 ml of the diluted unknown

$$\%w/v \text{ of acetic acid} = \frac{(NV)\ NaOH \times \text{eq. wt. HAC}}{1000}$$

$$= \frac{N \times 10 \times 60 \times 10}{1000}$$

= gm /100ml

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Oxidation – Reduction Reaction

Chemical reactions in which a transfer of electrons occurs are known as oxidation – reduction , or redox reaction . Oxidation involves the loss of electrons by a substance , and reduction the gain of electrons .

In any oxidation – reduction reaction , the number of electrons lost by the one substance is equal to the number gained by other .

Oxidaizing agents readily gain electrons lost by reducing agents . permanganate ion function as an oxidizing agent in acid being reduced to Mn^{+2}

$$MnO^-_4 + 8\,H^+ + 5e^- \longleftrightarrow Mn^{+2} + 4H_2O$$

While Fe^{+2} is a reducing agent being oxidized tom Fe^{+3}

$$Fe^{+2} \longleftrightarrow Fe^{+3} + e^-$$

The equivalent weight in redox-reaction is equal to the weight which , directly or indirectly , consumes produces 1 mole of electrons.

$$\text{Eq. wt.} = \frac{\text{M.wt.}}{\text{No. of electrons gained or lost}}$$

Lab-5 Preparation and standardization of (0.1N) $KMnO_4$

1- Preparation of 0.1N $KMnO_4$ solution:
   a. Dissolve 0.8 gm of solid $KMnO_4$ in 50 -100 ml of distilled water .
   b. Heat the solution on a hotplate for 1`0 min. at 70- 80 C^0.
   c. Cool the solution to room temperature , then add 2.5 ml of concentrated sulfuric acid .
   d. Complete the volume up to 250 ml by distilled water . Stopper the volumetric flask and mix. Well store the solution in the dark.

2- Standardization of $KMnO_4$ solution against sodium oxalate :
   a. Fill the burette with the $KMnO_4$ solution .
   b. Transfer 10 ml of 0.1 N sodium oxalate solution to a conical flask . Add 5 ml of concentrated H_2SO_4.
   c. Heat to 65^0C . Titrate against $KMnO_4$. The end point is the first permanent pink color .

$$2\ MnO^-_4 + 2\ C_2O_4^{2-} + 16\ H^+ \longleftarrow 2\ Mn^{+2} + 10\ CO_2 + 8H_2O$$

Calculation :

$N1V1\ (KMnO_4) = N2V2\ (Na_2\ C_2O_4)$

Lab- 6- Determination of Ferrous Iron in Mohr's salt

$$FeSO_4.(NH_4)_2SO_4 .6H_2O$$

Procedure :-

1- Dissolve the sample in distilled water . Transfer to 50 ml volumetric flask , complete the volume up to the mark and mix. Well.
2- Transfer 20 ml of the sample solution to a conical flask , add 5 ml of conc. H_2SO_4 .
3- Titrate against standard $KMnO_4$ solution . the end point is faint pink .

$$MnO^-_4 + 5Fe^{2+} + 8H^+ \longleftrightarrow Mn^{2+} + 5Fe^{3+} + 4\,H_2O$$

Calculate

$$N1V1_{\,KMnO4} = \frac{\text{wt. of Mohr's salt}}{\text{Eq, wt.}} \times 1000$$

wt. of Mohr's salt = gm in 20ml of solution

$$\text{wt. of ferrous iron} = \text{wt. of Mohr's salt} \times \frac{55.85}{392.14} \times 2.5$$

= gm Fe^{2+} in the sample

Note : M. wt of Mohr's salt = 392.14
Eq.wt of Fe^{+2} = atomic wt . = 55.85

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Lab- 7 **precipitation Titration**

Determination of chloride by Mohr Method

Precipitation titrations are based upon reactions that yield ionic compounds of limited solubility . The most important precipitating reagent is silver nitrate are sometimes termed argentometric methods. Potassiumchromate can serve as an end point indicator for the argentometric determination of chloride , bromide and cyanide ions by reacting with silver ions to form a brick –red silver chromate precipitate in the equivalence point region

The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a silver nitrate standard solution . After all the chloride has been precipitated as white silver chloride ,the first excess of titrant results in the formation of a silver chromate. Precipitate. Which signals the end point.

The chloride ion is titrated with standard silver nitrate, AgCl is formed as a precipitate .the end point is the appearance of brick-red color of silver chromate Ag_2CrO_4.

$$NaCl + AgNO_3 \longrightarrow AgCl + NaNO_3$$

$$AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2\,KNO_3$$

Procedure

1- Transfer 10 ml of unknown solution containing KCl or NaCl into a conical Flask .
2- Add 1 ml of K_2CrO_4 as an indicator.
3- Titrate with standard 0.1N silver nitrate solution , slowly , until a faint reddish color appears.

Calculation :-

$$N\ V\ (AgNO_3) = \frac{\text{wt. of } Cl^-\ \times 1000}{\text{Eq. wt}}$$

Weight of chloride in liter of solution .

Standardization of $AgNO_3$ solution :-

1- Transfer 10 ml of standard 0.1 N NaCl solution in to a conical flask .
2- Add 10 ml of K_2CrO_4 indicator and titrate slowly silver nitrate solute until a faint red color is formed .

N V ($AgNO_3$) = N V (NaCl)

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Lab – **8** **Complexometric Titration**

Determination of Total Hardness in Tab Water

Water hardness is an expression for the sum of calcium and magnesium cation concentration in a water sample . These cations form insoluble salts with soap , decreasing soap = s cleaning effectiveness . They also form hard water deposits in hot water heaters . The standard way to express water hardness is in ppm $CaCO_3$ which has the formula weight of 100.1 g/mole .

An excellent way to determine water hardness is perform a complexometric titration using a standard ethylene diamine tetra acetic acid (EDTA) solution . due to steric hindrances , EDTA will complex with calcium and magnesium in a one –to-one molar ratio . The end point in this experiment will be determined using a calmagite indicator . The indicator imparts a red color to the solution while there are calcium and magnesium ions that have not complexed with EDTA . Once the end point has been reached and there is no more uncomplexed Ca or Mg , the indicator will give a blue color . No hint of red color will be left.

This lab will be graded primarily on the accuracy of your individual results . Due to the fact that you will be using the EDTA as a primary standard , it is important that you be extremely careful in your weighing procedure . Any mistakes will carry through the entire experiment and greatly affect the accuracy of your results . Carful titrations will give you high precision and accuracy.

1- Determination of total hardness in tap water :
   a. Transfer 50 ml tap water into a conical flask .
   b. Add 10 ml of buffer 10 .
   c. Add 0.2 gm of Eriochrome black – T as an indicator .
   d. Titrate aginst 0.01 F EDTA solution until the color of the solution changes from red to blue .

   Not : $CaCO_3$ represents the total hardness of H_2O

$$V \quad M_{(EDTA)} = \frac{\text{wt of } CaCO_3}{\text{M. wt}} \times 1000$$

   M.wt of $CaCO_3$ = 100

$$Ppm_{CaCO3} = \frac{\text{wt of } CaCO_3}{50} \times 10^6$$

2- Determination of calcium –Hardness :

a. Transfer with a pipette 50 ml tap water into a conical flask .
b. Add 2 ml of buffer 12 .
c. Add 0.2 gm of murexide indicator and titrate against 0.01 F EDTA solution until the color changes from pink to violet .

$$V\ M_{(EDTA)} = \frac{\text{wt of } Ca^{+2}}{\text{At. Wt}} \times 1000$$

Atomic wt. of $Ca^{+2} = 40$

$$Ppm\ Ca^{+2} = \frac{\text{wt. of } Ca^{+2}}{50} \times 10^6$$

3- Determination of magnesium – Hardness :

$$V_{EDTA,total} - V_{EDTA,\ Ca\ 2+} = V_{EDTA,\ Mg\ 2+}$$

$$V_{EDTA,\ Mg\ 2+} \times M_{EDTA} = \frac{\text{wt. of } Mg^{+2}}{\text{At. wt}} \times 1000$$

Atomic wt. of $Mg^{+2} = 24$

$$Ppm_{Mg\ +2} = \frac{\text{wt. of } Mg^{+2}}{50} \times 10^6$$

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Lab -9 **Gravimetric Determination of Nickel**

Dimethyglyoxime is a chemical compound described by formula $CH_3(NOH)C(NOH)CH_3$. this colourless solid is the dioximederivative of the diketone diacetyl (also known as 2,3- butanedione) . DMG H_2 is used in the analysis of pallsdium or nickel . Its coordination complexes are of theoretical interest as models for enzymes and as catalysts . Many related ligand can be prepared from other diketones .

Procedure

1- Transfer the sample solution to a volumetric flask . complete the volume up to the mark with distilled water .
2- Transfer 20 ml of the diluted nickel solution into 400- 500 ml beaker . add to it a mixture of 50 ml of distilled water plus 2 ml concentrated HCl . heat to 80^0C on a water bath .
3- Add 20 ml of di methyl glyoxime , then introduce sufficient NH_4OH (1:1) with good stirring until red precipitate appear . The odor of NH_3 indicates that the medium is alkaline .
4- Leave the precipitate of N – dimethyl glyoximate for half an hour to complete the precipitate.
5- Clean , dry and weigh sintered glass crucible filter by decontation using Buchner suction pump.
6- Wash the precipitate with hot distilled water several times .
7- Dry the precipitate with crucible in the oven at 110 – 120 ^{0}C for 20 min.
8- Weigh the crucible plus the precipitate.