Practical Quantitative Analysis

(Volumetric and Gravimetric Analysis)

Practical skills – Quantitative Analysis

Principles of Volumetric Analysis

Volumetric analysis is a method to determine the amount of substances in a given sample. A procedure called **titration** is used in volumetric analysis. In titration, a solution of known concentration, called a **standard solution**, is added to a measured volume of an unknown solution until the reaction is completed.

The main apparatus used in volumetric analysis are:

1. **Volumetric flask** - to make up a solution to a certain volume accurately, e.g. 250 cm^3
2. **Burette** - to deliver variable volumes of solution accurately
3. **Pipette** - to deliver a fixed volume of solution accurately, e.g. 10 cm^3 or 25 cm^3
4. **Conical flask** - it has a narrow neck to prevent solution from spurting when being shaken

When carrying out a titration, a known volume of solution is placed in the conical flask using the pipette. Then the standard solution is run from the burette until the two solutions have just reacted completely. This is the *end point* of the titration.

Standard Solutions and Standardization

Before estimations involving acids or alkalis can be carried out on given substances or mixtures, it is necessary to obtain acids or alkalis in which their concentrations are accurately known. Solutions with **accurately known concentration** are called *standard solutions*.

The common acids and alkalis cannot be employed directly for making these standard solutions (Standard solution is a solution in which its concentration is accurately known.) because they are variable in composition for the reasons given below:

Hydrochloric acid	-	this is volatile in high concentration
Sulphuric acid	-	this is hygroscopic
Sodium hydroxide and Potassium hydroxide	-	they are deliquescent and react with carbon dioxide in the air
Calcium hydroxide	-	this is insufficiently soluble and also reacts with carbon dioxide in the air
Ammonia	-	this is volatile, and is a solution of variable concentration

To prepare a standard solution, we need to obtain a pure compound. The required mass of this compound is weighed out accurately, dissolved in some deionized water, and the solution made up to a definite volume in a volumetric flask.

Most compounds are not suitable for preparing standard solutions because they are hard to obtain in a pure form. For example, sulphuric acid is hygroscopic while sodium hydroxide is deliquescent. Usually anhydrous sodium carbonate is used to prepare standard solution because its pure form can be obtained without difficulty.

The concentrations of other solutions can be found out by using a standard solution. This is called *standardization*. For example, we can find out the exact concentration of sulphuric acid by titrating it with a standard solution of sodium carbonate.

Characteristics of a good standardizing agent

1. It should be obtainable in high degree of purity.

2. It should be stable and unaffected by the atmosphere. It should not be deliquescent or efflorescent, so that it may weighed easily and accurately.

3. It should be fairly cheap.

For standardization of acids, the materials commonly used as the **primary standards** are:

1. Pure sodium carbonate prepared by heating sodium hydrogencarbonate.
2. Pure borax, $Na_2B_4O_7$. 10 H_2O , sodium metaborate.
3. Pure calcium carbonate.

Alkaline solutions may be standardized by using solid crystalline organic acids such as :

1. oxalic acid, $H_2C_2O_4$. 2 H_2O , or
2. succinic acid, $HOOCCH_2CH_2COOH$, which can be obtained in a high state of purity.

Simple Theory of Acid-Base Indicators

An acid-base indicator is a fairly weak organic acid:

$$HIn\ (aq) \rightleftharpoons H^+\ (aq) + In^-\ (aq)$$

which on ionization undergoes a rearrangement of electrons so that the two forms, HIn and In^-, absorb light of *different wavelength*. Indicators are chosen so that some of this absorbed light is in the visible region and HIn and In^- have different colours.
There are some examples of indicators and their colour change:

Indicator	pH range	Colour in	
		Acid	Alkaline
Methyl orange	3 - 4	red	yellow
Phenolphthalein	9 - 10	colourless	pink
Litmus	6.5 - 7.5	red	blue

Redox Titration

Potassium permanganate is a powerful oxidizing agent and is used for the estimation of many reducing agents, especially compounds of iron, oxalic acid and its salts.

In acidic solution, one molecule of potassium permanganate reacts with, for example, five iron(II) ions:

$$2\ KMnO_4 + 8\ H_2SO_4 + 10\ FeSO_4 \longrightarrow K_2SO_4 + 5\ Fe_2(SO_4)_3 + 8\ H_2O$$

In alkaline solution, potassium permanganate, by a different reaction, yields manganese(IV) oxide as a brown precipitate.

Consideration of these facts make it clear at once that for quantitative work, potassium permanganate must be used in conditions which exclude entirely one of these reactions. In practice, potassium permanganate is almost always used to titrate solutions which are sufficiently acidic to exclude altogether the formation of manganese(IV) oxide.

Of the three mineral acids available, only sulphuric acid is suitable for use with potassium permanganate. Hydrochloric acid is not suitable because it reacts with potassium permanganate according to the following equation:

$$2\ KMnO_4 + 16\ HCl \longrightarrow 2\ KCl + 2\ MnCl_2 + 8\ H_2O + 5\ Cl_2$$

While for nitric acid, it itself is an oxidizing agent and may therefore interfere with the oxidizing action of the permanganate.

In this titration, the solution must be *sufficiently acidic* to prevent the formation of any precipitate of manganese(IV) oxide. As the titration proceeds, manganese(II) ions accumulate, but at the concentration used as in ordinary titration, it gives a colourless solution. As soon as the permanganate is in excess, the solution becomes pink and therefore the permanganate ion itself acts as the indicator. The end point being the first permanent pink colour.

A potassium permanganate solution decompose slowly and therefore should be protected from light and standardized again at intervals because organic matter in the atmosphere or in water may reduce the permanganate solution.

It may be standardized by a pure iron(II) salt or a pure oxalate. Usually iron(II) ammonium sulphate, $FeSO_4 . (NH_4)_2SO_4 . 6 H_2O$, is used because it can be obtained in a high grade of purity, no efflorescence and no atmospheric oxidation.

Titration - General Technique

1. Cleaning laboratory glasswares

   Cleanliness is an essential prerequisite. Volumetric glassware should always be **clean** prior to use. The most obvious indication of dirty glassware is the appearance of globules of liquid adhering to the glass when liquid has been drained from the apparatus. The residual liquid forms a uniform file, not globules, when the glassware is clean.

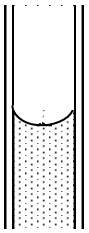
   To rinse laboratory glassware, it is much more effective to use several small amount of rinsing solution (deionized water or, in the case of a pipette or burette, the solution next to be introduced), rather than one large quantity.

2. Reading (or adjusting) the position of a meniscus

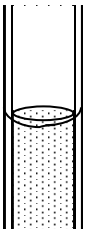
   Except with very deeply colored solutions, the bottom of the meniscus is the most easily located part of the liquid surface and is generally taken as the reference point.

   A piece of white paper may be placed behind the burette to sharpen the bottom of the meniscus and to aid in the estimation of the correct reading.

   In order to prevent parallax errors, hold the stem of liquid vertically and bring the eye so that it is horizontal to the meniscus, i.e. so that if a calibration mark completely encircles the glass at the position of the meniscus, it appears as a straight line.



Correct . The meniscus viewed at eye level.



Incorrect . The meniscus viewed at an angle.

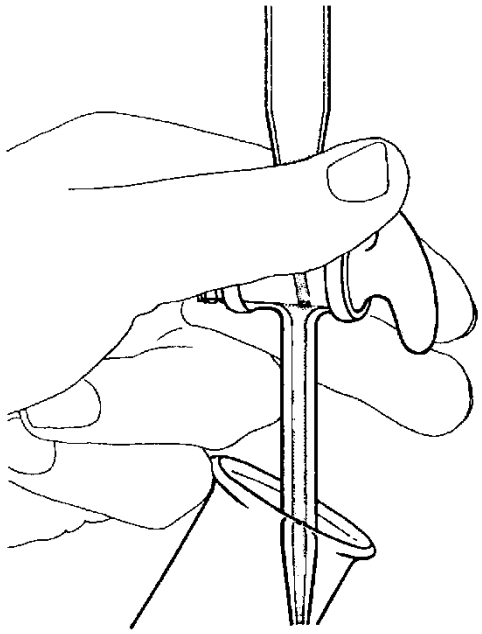
3. Using a burette

If you use a funnel to fill a burette, tilt it to one side so that the liquid runs down the side of the burette, otherwise you may get an air blockage and spill the liquid.

Allowing the solution to run smoothly down the side of the burette also avoids air bubbles being produced which may adhere to the wall of the burette.

Clamp the burette vertically in a stand. Open the stopcock briefly to fill the part below it with the solution. You must, of course, expel *all* of the air from the tip of the burette before you start, or a remaining air bubble may become dislodged during the actual titration.

With practice, the majority of people find the most convenient procedure for delivering liquids from a burette is to place the fingers of the left hand round the back of the burette and the thumb in front, and to hold the stopcock tap between the thumb and fore and middle fingers. In this way there is no tendency to pull out the tap, a very delicate touch can be developed and the right hand is left free to swirl the titration flask.



After adjusting the initial level of solutions, wait until there is no change in meniscus level through drainage before taking the initial reading or re-adjusting exactly to the zero mark.

Remove any droplet adhering to the tip of the burette by touching the tip against, for example, the inside of a beaker.

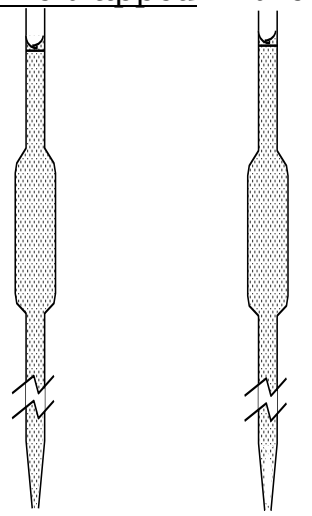
4. Using a volumetric flask

Before making up to the mark, loosen the stopper and allow any liquid caught there to run down. If you wish avoid getting the ground glass wet, you can introduce the solution by using a funnel and swirling the flask as you fill it up rather than shaking it.

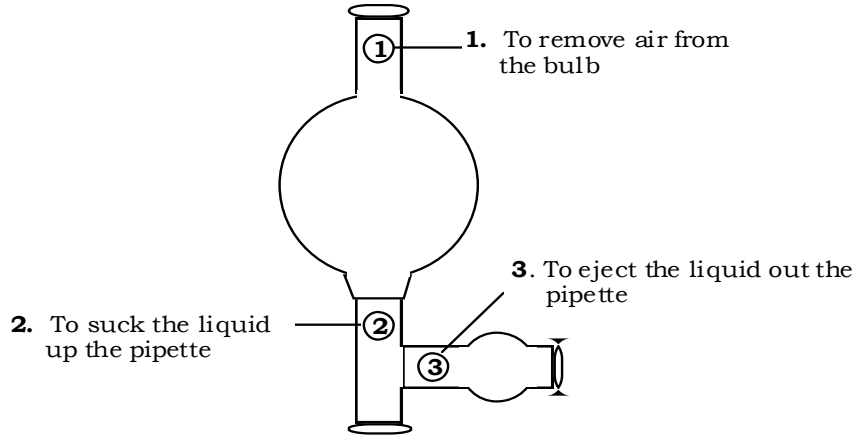
The shape of a volumetric flask prohibits good mixing when the flask is filled to the mark. To make sure that the final solution is homogeneous it is necessary to hold the stopper and keep on inverting and shaking the flask gently a number of times. For the same reason too, it is as well to make sure that all solids are dissolved when the flask is only half full.

5. Using a pipette

When liquid is being drawn into the pipette, the tip of the pipette must be kept below the liquid level in the container. If the tip becomes exposed, air will be drawn into the pipette and bubbles will become trapped in the pipette stem.



A pipette filler should always be used for the sake of safety. The pipette filler comprises a bulb and three pinch valves controlling influx and efflux of pipette content. The operation of a pipette filler is summarized below:



Press the valve 1 and the bulb of the pipette filler simltaneously until the bulb is flattened. This is to remove all the air out of the bulb. Fit the pipette filler to the end of the pipette.

Draw up the required solution to above the graduation mark by pressing the valve 2. Lift the pipette out of the liquid and wipe the outside of the lower stem free from any adhering droplets which may later run down or drop into the delivery vessel.

Adjust the meniscus to the mark by pressing on the valve 3; bring the tip of the pipette just into contact with the surface of the solution again so as to remove any drop adhering to the tip.

Carefully, so as to avoid spurting and loss of liquid, bring the pipette tip over the delivery vessel and holding the pipette vertically, allow the liquid to run out freely by remove the pipette filler. Allow 15 seconds for drainage, taken from the time that the stream breaks into droplets, keeping the tip clear of the liquid surface in the flask.

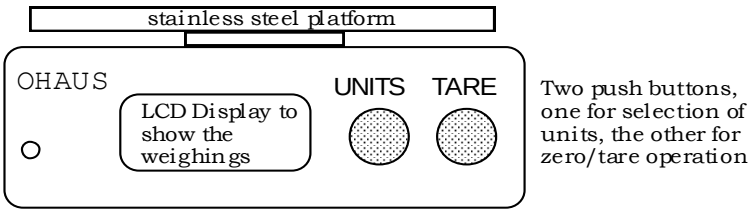
Finally touch the side of the container with the tip of the pipette to remove any partial drop which has formed. Do not blow or shake out the residue or more than the marked volume will be delivered. Similarly, you will not obtain the required volume if you do not allow the specified drainage time; nor, if you do not allow for drainage, and about the same time in each case, will your delivered volumes be very consistent.

The contents of a pipette should always be delivered in the same way, otherwise the volume discharged will not be consistent.

6. Weighing

You are less likely to loss (or contaminated 被染污) material using a weighing bottle rather than a watch glass.

The following diagram shows the front panel of an electric balance common used in the laboratory:



The Front Panel of an Electric Balance

Procedure for accurate weighing

A. Direct Weighing

1. Determine the mass of a weighing bottle accurately*
2. Remove the weighing bottle
3. Add the approximate amount of material to the bottle
4. Replace the weighing bottle on the balance
5. Obtain the accurate mass of the container plus sample
6. Carefully transfer the entire contents of the weighing bottle into the receiver (e.g. volumetric flask).

* Note: ***Taring*** - some user prefer to use taring, i.e. to set the balance to zero when weighing the empty weighing bottle. Then when sample is added, its mass is read directly.
But if you wanted to weigh the weighing bottle directly, you would need to set the balance to zero *before* placing the container on the balance pan.

B. Weighing by Difference

1. Add the approximate amount of material to the bottle
2. Obtain the accurate mass of the container plus sample
3. Carefully discharge the sample from the weighing bottle into the apparatus (e.g. volumetric flask)
4. Obtain the accurate mass of the container less the sample transferred

7. Recording your results

   To save yourself uncertainty and perhaps having to repeat work, record your results immediately you obtain them and in the proper place, not on an insubstantial scrap of paper.

   1.6 g means somewhere between 1.55 g and 1.65 g . On the other hand, 1.6000 g means somewhere between 1.59995 g and 1.60005 g . For the convenience of other people and of yourself, if you mean 1.6000 g , then write 1.6000 g.

8. End points

   Don't ask the teacher "Is this the end point ?"

   The end point of a colorimetric indicator is a perceptible change and you yourself, if you watched your solution on addition of the last drop of titrant, are the most qualified person to judge if any change has taken place.

   If you are uncertain, you can always record the burette reading temporarily and note the effect of adding further titrant. In any case, some end points are fugitive and although the correct colour change may have taken place, it may have reversed by the time you consult a second person.

Errors in Titrations

1. Non-coincidence of equivalent point and indicator end point.
2. Impurities present in the solutions.
3. Difficult to locate the end point, especially for weak acid and weak base.
4. Errors in measuring volumes, weighing, etc.

Experimental No. 1

Standardization of HCl using standard solution of sodium carbonate then determination of concentration of sodium hydroxide and carbonate sodium in mixture (Neutralization Reaction)

Part one:

Standardization of HCl using standard solution of sodium carbonate

1- Aim: Calculate $\mathbf{N}_{HCl}$

2- Theory:

$HCl + Na_2CO_3 \rightarrow NaCl + NaHCO_3$ pH = 8.7

$HCl + NaHCO_3 \rightarrow NaCl + H_2CO_3$ pH = 3.7

3- Chemical :

HCl (0.1N), Na_2CO_3 (0.1N), ph ph and MO as indicators

4- Apparatus:

Conical flask & burette & Pipette & Beaker

5- Procedures:

a- Transfer 10 mL Na_2CO_3 solution into conical flask

b- Add 2-3 drops of ph ph (pink color)

c- Titrate using HCl till (colourless)

d- Repeat the above steps with MO (color changes from yellow to red)

6- Calculation:

$(N \times V)_{HCl} = (N \times V)_{Na2CO3}$

Note: in case ph ph $V_{HCl} = 2V$ of burette

In case MO V_{HCl} = V of burette

Results

In case of ph.ph.

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

In case of MO

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Part two:

• Determination of concentration of sodium hydroxide and carbonate sodium in mixture

1- Aim: Calculate N_{NaOH} & N_{Na2CO3}

2- Theory:

$(NaOH + Na_2CO_3)$ **+** $HCl \rightarrow 2NaCl + NaHCO_3 + H2O$

$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$

In case ph ph

$V1_{HCl}$ = all hydroxide + 1/2 carbonate

In case MO

$V2_{HCl}$ = all hydroxide + all carbonate

3- Chemical:

Mixture solution (unknown), HCl (0.1N), indicators

4- Procedures:

a) In case of ph.ph.

1- Transfer 10 ml mixture into conical flask

2- Add 2-3 drops of ph.ph. (pink)

3- Titrate with HCl till colorless

b) In case of MO.

1- Transfer 10 ml mixture into conical flask

2- Add 2-3 drops of MO. (yellow)

3- Titrate with HCl till red

5- Calculations:

V1 = all hydroxide + 1/2 carbonate

V2 = all hydroxide + all carbonate

(V_2-V_1) = 1/2 carbonate

$2(V_2-V_1)$ = all carbonate

$2V_1 - V_2$ = all hydroxide

(N(unknown) x 10)NaOH = [N x $2(V_2-V_1)$]HCl

(N(unknown) x 10)Na_2CO_3 = [N x $(2V_1 - V_2)$]HCl

Calculate the strength for both carbonate and hydroxide

Results

a) **Incase of ph.ph.**

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

a) Incase of MO.

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Experimental No. 2

Determination of ammonia using standard solution of HCl (back titration)

1- Aim: calculate N_{NH3}

2- Theory:

$NH_4Cl + (NaOH)excess \leftrightarrow NaCl + (NaOH)_{unreacted\ remained} + NH_4OH$

Then boil the total solution to librate NH4OH as ammonia gas then titrate against HCl

Note: NH_3 is volatile gas so determination of ammonia will be using indirect titration **(back titration)**

Back titration is suitable in these cases:

1) Volatile compounds

2) When the sample is solid or insoluble

3) When a large excess of reagent be needed

4) When we need boiling

3- Procedure:

1) Transfer 10 ml ammonia solution in to C.F.

2) Add 30 ml NaOH

3) Boiling for about 15 min.

4) Cool then add 2-3 drops of MO

5) Titrate against HCl till red

4- Calculate:

V_{HCl} = unreacted NaOH

Reacted NaOH = 30 - V_{HCl}(unreacted NaOH)

$(N \times V)_{NaOH} = (N \times V)_{NH4Cl}$

$0.1 \times (30 - VHCl) = N \times 10$

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Experimental No. 3

Determination of concentration of H_3PO_4 and HCl in mixture using 0.1 N NaOH

1- Aim: Calculate N_{H3PO4} & N_{HCl}

2- Theory:

$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + NaCl$ (1/3 of complete reaction)

$NaH_2PO_4 + NaOH \rightarrow Na_2HPO_4 + NaCl$ (2/3 of complete reaction)

$Na_2HPO_4 + NaOH \rightarrow Na_3PO_4 + NaCl$ (3/3 or all reaction)

$HCl + NaOH \rightarrow NaCl + H_2O$ (one step for all reaction)

In case MO

$V1_{NaOH}$ = 1/3 H_3PO_4 + all HCl

In case ph ph

$V2_{NaOH}$ = 2/3 H_3PO_4 + all HCl

3- Chemical:

Mixture solution (unknown), NaOH (0.1N), indicators

4- Procedures:

a) In case of MO.

1- Transfer 10 ml mixture (phosphoric and hydrochloric) into conical flask

2- Add 2-3 drops of MO. (red)

3- Titrate with NaOH till yellow

b) In case of ph.ph.

1- Transfer 10 ml mixture into conical flask

2- Add 2-3 drops of ph.ph. (colorless)

3- Titrate with NaOH till pink

5- Calculations:

V_1 = 1/3 phosphoric + all hydrochloric

V_2 = 2/3 phosphoric + all hydrochloric

(V_2-V_1) = 1/3 phosphoric

3(V_2-V_1) = all phosphoric

$2V_1 - V_2$ = all hydrochloric

(N(unknown) x 10)H_3PO_4 = [0.1 x 3(V_2-V_1)]NaOH

(N(unknown) x 10)HCl = [0.1 x ($2V_1 - V_2$)]NaOH

Calculate the strength for both phosphoric and hydrochloric

Results

a) **Incase of MO.**

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

a) Incase of ph.ph.

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Experimental No. 4

Standardization of potassium permanganate using oxalic acid (Redox Reaction)

Define: Oxidation, Reduction, Oxidizing agent, Reducing agent???

1- Aim: Calculate N_{KMnO4}

2- Theory: $KMnO_4$ is a 2nd standard substance

$\underline{KMnO_4} + H_2C_2O_4 + H_2SO_4 = K_2SO_4 + \underline{MnSO_4} + CO_2 + H_2O$

The optimum conditions for $KMnO_4$ titration:

1- Acidic medium using 20 ml 2N H_2SO_4, Why should you avoid HNO_3 and HCl???

2- Heating to 70 ^oC (not boiling, why??)

3- Procedures

1) Transfer 10 ml 0.1 N oxalic or oxalate soln. in to C.F.

2) Add 20 ml 2N H_2SO_4

3) Heat to 70 ^oC

4) Titrate with $KMnO_4$ gradually with stirring till pink color appears.

5) Calculate (N x V)$KMnO_4$ = (N x V)oxalic

Results

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Experimental No. 5

Iodometry and Iodimetry using sodium sulphate

Introduction:

Q1. What is the difference between [self-indicator ($KMnO_4$), specific indicator (I_2), external indicator (pot-ferricynaide), internal indicator (diphenyl amin)]??

Q2. What is the difference between Iodimetry and Iodometry?

Q3. How do you prepare Iodine solution?

Q4. Why is I_2 secondary standard?

Q5. How do you prepare starch as indicator and when use it?

Standardization of $Na_2S_2O_3$ using $K_2Cr_2O_7$

1- Aim: Calculate $N_{Na2S2O3}$

2- Theory: $Na_2S_2O_3$ (2^{nd} standard) and $K_2Cr_2O_7$(1st standard)

a- Iodometry:

$K_2Cr_2O_7 + KI + HCl \leftrightarrow KCl + CrCl_3 + I_2 + H_2O$

b- Iodimetry:

$I_2 + Na_2S_2O_3 = NaI + Na_2S_4O_6$

NOTE: $S_2O_3^{2-} = I_2 = KI = K_2Cr_2O_7$

3- **Materials:**

$Na_2S_2O_3$ (unknown soln.), $K_2Cr_2O_7$(0.1N),

KI (5%), HCl (1N), starch (freshly prepared)

4- Procedure:

1- Transfer 10 ml $K_2Cr_2O_7$ soln. into C.F.

2- Add 3 ml HCl

3- Add 20 ml KI soln.

4- Titrate against $Na_2S_2O_3$ in presence of starch till faint green

5- **Calculate:**

(N x V)$_{K2Cr2O7}$ = (N x V)$_{Na2S2O3}$ = (N x V)I_2

Results

	Burette reading		Difference
Exp.	**Initial**	**Final**	
1.			
2.			
3.			

Experimental No. 6

Silver nitrate titrations by Mohr method (precipitation titration)

1- Aim: Calculate N_{Cl^-} or N_{Br^-}

2- Theory:

NaCl (unknown soln.) + $AgNO_3$ (titrant) ↔AgCl (white ppt.) + $NaNO_3$

At the end point: $AgNO_3$ (titrant) + K_2CrO_4 = KNO_3 + Ag_2CrO_4 (red ppt.)

3- Chemicals:

$AgNO_3$ (0.1N), NaCl (unknown), K_2CrO_4 (Indicator)

4- Procedure:

1- Transfer 10 ml of NaCl solution in C.F.

2- Add 3 drops of chromate indicator pH (6.5-9)

3- Titrate against 0.1N $AgNO_3$ (white ppt. is formed)

4- At end point the excess of $AgNO_3$ will react with Ind. To form red ppt.

5- Calculations

$(N \times V)_{NaCl} = (N \times V)_{AgNO3}$

Results

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Experimental No. 7

Application with EBT and Murexide indicators by analysis of binary mixture of calcium plus magnesium (Complexiметry)

Introduction

Q1. Define: Complex, Ligand, Metalochromic indicator (EBT and Murexide)

Q2. What are the types of ligands?

Q3.What the conditions required to form the complex?

1- Aim: calculate M_{Ca2+} and M_{Mg2+} in binary mixture

2- Theory:

This experiment gives accurate results for the total of Ca^{2+} + Mg^{2+} ions but not for calcium ions alone because when magnesium ions are precipitated at pH 12 some of Ca^{2+} are coprecipitated along with Mg^{2+} as hydroxide.

To overcome this problem:

1- Adding just less than the required amount of EDTA before adding NaOH so that virtually all of Ca^{2+} are complexed with EDTA before $Mg(OH)_2$ precipitates

2- Excess of EDTA may be added to mixture before addition of NaOH and then the remained EDTA may be determined by back titration with standard calcium ions solution using murexide but incomplete precipitation of Mg ions in presence of EDTA.

3- Masking Mg ions in the mixture by tartrate ions then titrate Ca ions with EDTA using murexide (desirable method)

Preparation of Ca+Mg mixtures and recommended procedures:

Prepare two stock solutions A and B

Solution A: 0.008 M Ca ions + 0.004 M Mg ions (0.8 g $CaCO_3$ + 1.0 g $MgSO_4.7H_2O$ per liter).

Solution B: 0.004 M Ca ions + 0.008 M Mg ions (0.4 g $CaCO_3$ + 2.0 g $MgSO_4.7H_2O$ per liter).

3-Procedures:

• (Major method for all mixture ions Ca^{2+} + Mg^{2+})

1- Transfer 10 ml mix. into C.F. then add 5 ml ammonical buffer pH 10 then EBT

2- Titrate against 0.01M EDTA to first change color from red to pure blue

3- EDTA volume(X ml) equivalent to all mixture (Ca^{2+} + Mg^{2+})

• (A method to determine Ca^{2+} ion alone)

1- Transfer 10 ml mixture in to c.f. then add 2 ml (1M) NaOH solution to precipitate Mg as hydroxide at pH 12

2- Add a speck of murexide and titrate with EDTA to first change in color from red to purple

3- The volume EDTA (Y ml) equivalent to Ca ions

But volume EDTA equivalent to Mg ions = (X - Y)

Results

	Burette reading		Difference
Exp.	**Initial**	**Final**	
1.			
2.			
3.			

Exp.	Burette reading		Difference
	Initial	Final	
1.			
2.			
3.			

Experimental No. 8

Determination of crystalline water in barium chloride (Gravimetry)

Introduction

Q1. **Define**: Solubility product, Precipitant, Adsorption, Occlusion, Peptization

Q2. What is the difference between Coprecipitation and Post precipitation?

Q3.What is the characteristics of the precipitate?

Q4. What is the difference between precipitate and weighed form? give examples??

Q5. What are the conditions of precipitation?

Part A:

1- Aim: Number of moles of water

2- Procedures:

1- Weigh out about 1.5 g of fresh recrystallized **$\underline{BaCl_2.xH_2O}$** in the weighing bottle (W1)

2- Dry the sample in an electric oven at 130 ^oC for about two hours.

3- At the end of 2 hours transfer the bottle to a desiccator by means of crucible tongs.

4- Weigh the bottle (W2)

5- Replace the bottle with the substance in the drying oven and keep it there for 1/2 hours, cool and weight it again.

If the second weight gives the same results, it is assumed that all the water of crystallization has been removed

3- Calculations:

Weight of bottle empty =

Weight of bottle + salt =

Weight of hydrated salt =

(Before drying)

Weight of bottle + salt =

(After drying)

Weight of water =

$$BaCl_2.xH_2O \rightarrow xH2O$$

$$Mol.wt + 18x \rightarrow 18x$$

Wt of hydrated sample → Wt of water

Experimental No. 9

Determination of barium ion as $BaSO_4$

Theory

Barium could be precipitated as a sparingly soluble precipitate $BaSO_4$ using dil sulphoric acid solution:

$$\text{Ba salt} + H_2SO_4 \rightarrow BaSO_4\downarrow \text{ (white ppt)}$$

Procedure

1- Weight exactly 0.5 g of barium salt and then dissolve it in 150 mL dist water.
2- Add 5 mL HCl (1:1) to the dil barium soln.
3- Heat the solution till 70 ^{o}C.
4- After heating, add H_2SO_4 drop by drop with continuous stirring.
5- Put the ppt on a heater after precipitation for 5 min.
6- Allow the ppt to stand for 1/2 h, and test for complete precipitation using H_2SO_4.
7- Filter the ppt and wash it with hot water.
8- Dry the ppt in oven at 120 ^{o}C for 30 min.
9- Wight the ppt and filter paper.

Calculations

Wight of Barium salt = …..g

Wight of dry empty filter paper =……g

Wight of ppt and filter paper after drying = …..g

Wight of ppt ($BaSO_4$) only =……g

$$BaSO_4\downarrow \rightarrow Ba^{2+}$$

$$MWt \rightarrow AWt$$

$$\ldots\ldots \rightarrow \ldots\ldots$$

$$\text{Wight of ppt} \rightarrow x\ g\ Ba^{2+}$$

$$\ldots\ldots \rightarrow \ldots\ldots$$

So, weight of Ba =

Calculate also,

The practical yield

The percent of Ba in the original salt

The percent of Ba in the formed ppt

Experimental No. 10

Determination of lead as lead chromate ($PbCrO_4$)

Theory

Lead may be precipitated as a slightly soluble precipitate $PbCrO_4$ using Potassium chromate solution:

$$Pb\ salt + K_2CrO_4 \rightarrow PbCrO_4\downarrow \text{ (yellow ppt)}$$

Procedure

1- Weight exactly 0.3 g of lead salt and then dissolve it in 150 mL dist water.

2- Add 5 mL CH_3COOH (1:1) to the dil lead soln.

3- Heat the solution till 70 ^oC.

4- After heating, add K_2CrO_4 drop by drop with continuous stirring.

5- Put the ppt on a heater after precipitation for 5 min.

6- Allow the ppt to stand for 1/2 h, and test for complete precipitation using K_2CrO_4.

7- Filter the yellow ppt and wash it with hot water.

8- Dry the ppt in oven at 120 ^oC for 30 min.

9- Wight the ppt and filter paper after drying.

Calculations

Wight of lead salt = …..g

Wight of dry empty filter paper =……g

Wight of yellow ppt and filter paper after drying = …..g

Wight of ppt ($PbCrO_4$) only =……g

$PbCrO_4\downarrow \rightarrow Pb^{2+}$

MWt → AWt

……. → …….

Wight of ppt → x g Pb^{2+}

……. → …….

So, weight of Pb =

Calculate also,

The practical yield

The percent of Pb in the original salt

The percent of Pb in the formed ppt

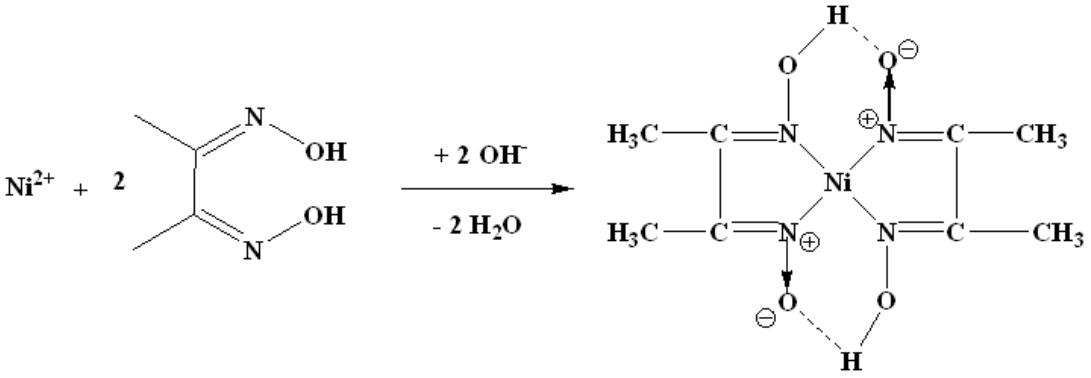
Experimental No. 11

Determination of nickel as nickel – dimethylglyoxime (DMG - organic precipitant)

Theory

The nickel is precipitated as nickel dimethyl glyoxime by adding alcoholic solution of dimethyl glyoxime $C_4H_6(NOH)_2$ and then adding a slight excess of aqueous ammonia solution.

A slow increase in the concentration of ammonia in the solution causes a slight increase in the pH gradually and results in the precipitation of the complex. The result is the formation of a denser precipitate. Once the filtrate has been collected and dried, the nickel content of the solution is calculated stoichiometrically from the weight of the precipitate.



Procedure

1- Weight exactly 0.3 g of nickel salt and then dissolve it in 150 mL dist water.

2- Add 5 mL HCl (1:1) to the dil lead soln.

3- Heat the solution till 70 ^{o}C.

4- After heating, add 30 mL of DMG solution.

5- Add NH4OH drop by drop with continuous stirring.

6- Put the red ppt on a heater after precipitation for 5 min.

7- Allow the ppt to stand for 1/2 h, and test for complete precipitation using NH_4OH or DMG.

8- Filter the yellow ppt and wash it with hot water.

9- Dry the ppt in oven at 120 ^oC for 30 min.

10- Wight the ppt and filter paper after drying.

Calculations

Wight of nickel salt = …..g

Wight of dry empty filter paper =……g

Wight of red ppt and filter paper after drying = …..g

Wight of ppt (Ni-DMG) only =……g

$$\text{Ni-DMG}\downarrow \rightarrow \text{Ni}^{2+}$$

$$\text{MWt} \rightarrow \text{AWt}$$

$$\ldots\ldots \rightarrow \ldots\ldots$$

$$\text{Wight of ppt} \rightarrow \text{x g Ni}^{2+}$$

$$\ldots\ldots \rightarrow \ldots\ldots$$

So, weight of Ni =

Calculate also,

The practical yield

The percent of Ni in the original salt

The percent of Ni in the formed ppt