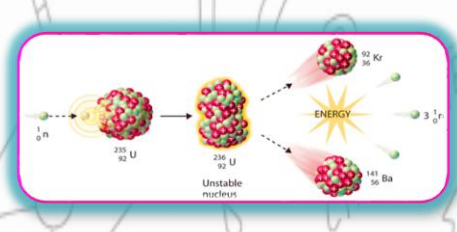
RAMAIAH
College of Arts, Science & Commerce - Autonomous

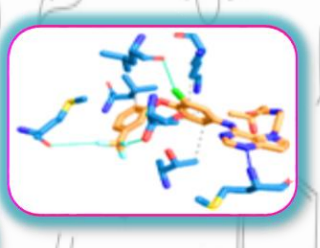
Department of Chemistry & Biochemistry

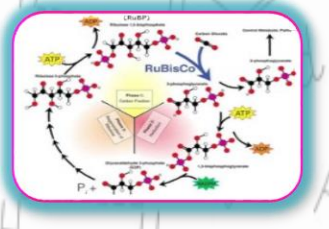
LAB MANUAL

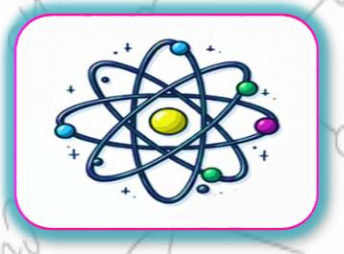
I Semester BSc - Chemistry

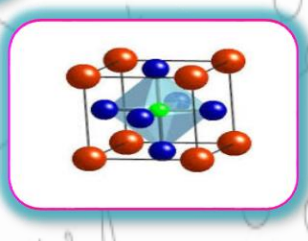
CHEMISTRY – I PRACTICAL











Dr. Amreen Khanum

Department of Chemistry/Biochemistry

I Semester B. Sc., Chemistry

Practical Manual

Name: …… ……

Register Number: …… ……..

Section: ……

SAFETY MEASURES

1. Students must wear apron in the lab.

2. Read label carefully before transferring a chemical from a container.

3. While pipetting, if any solution is swallowed, wash the mouth repeatedly with water.

4. Handle with care the chemicals, glassware and electrical instruments.

5. The electronic balance should be handled carefully under the directions of a technical assistant.

6. Do not perform unauthorized experiment in the laboratory.

7. Despite the best precautions sometimes an accident may occur. Seek immediate help and rescue from the laboratory staff.

8. Keep the work bench neat and tidy.

BASIC CONCEPTS IN VOLUMETRIC ANALYSIS

Chemical analysis of the substances is carried out in two ways

1. **Qualitative analysis:** Identification of the constituents, e.g. elements or functional groups, present in a substance.
2. **Quantitative analysis:** Determines the quantity of a component present in substance. It is carried out in two ways
   a. Gravimetric analysis
   b. Volumetric analysis

   Gravimetric analysis involves the estimation of the amount of a given compound from the results of weighing. Volumetric analysis is based on measuring volume of the solution of a substance.
3. **Types of Titrations:**
   a. **Acid – Base titrations**: A titration in which an acid of known concentration is added to a solution of base of unknown concentration, or the converse.
   b. **Complexometric titrations:** Titrations in which complex is formed between the analyte and the titrant.
   c. **Redox titrations**: Redox titrations are based on an oxidation-reduction reaction between an oxidizing agent and a reducing agent.
   c. **Precipitation Titrations:** Titrations based on the formation of a precipitate between the titrant and the analyte are termed as precipitation titrations.

4. **Terms involved in volumetric analysis**
   a. **Titration:** The process of finding out the volume of one of the solution required to react completely with the definite volume of other solution
   b. **Titrant:** The solution of known strength.
   c. **Titrand or Analyte:** The solution which contains a substance whose strength is to be estimated.
   d. **Indicator:** The substance which indicates the end point of titration is called indicator. The indicator indicates the completion of reaction by change in colour at the end point. Eg: Phenolphthalein for acid base titration.

e. **Strength:** The amount of substance dissolved in one litre of a solution is expressed in terms of strength of a solution. Strength of solution can be expressed in any of the following ways.

i. **Normality**: It is the number of gram equivalents of solute present in one liter of solution. It is denoted by N.

ii. **Molarity:** The number of moles of a given substance per litre of solution. It is denoted by M.

iii. **Molality**: The number of moles of solute per kilogram of solvent. It is denoted by m.

f. **Standard solution:** It is a chemical term which describes a solution of known concentration. The concentration of the solution is normally expressed in units of moles per liter. Standard solutions are normally used in titrations to determine the unknown concentration of a substance in sample solution.

g. **Primary standard**: Any substance stable, pure, readily soluble in water, with high equivalent weight and the composition of its solution should not change on long standing or storage.
Examples: Oxalic acid, Magnesium sulphate, Potassium dichromate.

h. **Secondary Standard**: The composition of these solutions changes on long standing or storage with duration of time. These are standardized by Primary standards.
Examples: Ferrous sulphate, EDTA, Potassium permanganate

i. **Equivalent Weight:** Equivalent Weight of a substance may be defined as the number of parts by weight of it which can combine or displace 1.008 parts of hydrogen or 8 parts of oxygen or equivalent weight of any substance.

Equivalent weight of acid = Mol. Wt of acid / no. of replaceable H^+ ions

a. Molecular weight of sulfuric acid(H_2SO_4) = 98.07 g/mol
Number of replaceable H^+ ions or basicity = 2
Equivalent weight = 98.07 / 2 = 49.035 g/ equivalent of H^+

b. Molecular weight of Hydrochloric acid (HCl) = 36.5 g/mol

Number of replaceable H^+ ions = 1

Equivalent weight = 36.5 /1 = 36.5 g/ equivalent of H^+

Equivalent weight of base = Mol. Wt of base / no. of replaceable OH^- ions

a. Molecular weight of ammonium hydroxide (NH_4OH) = 35.00 g/mol

Number of replaceable OH^- ions or acidity = 1

Equivalent weight = 35.00 /1 = 35.00 g/equivalents of OH^-

b. Molecular weight of sodium hydroxide (NaOH) = 40.00 g/mol

Number of replaceable OH^- ions or acidity = 1

Equivalent weight = 40.00 /1 = 40.00 g/equivalents of OH^-

Equivalent weight of an oxidizing agent

Oxidation process involves loss of electrons. An oxidizing agent is one which accepts electrons; it oxidizes the other substance by stripping off electrons from it.

Equivalent weight of oxidizing agent = Mol. Wt / number of electrons gained

a. **Potassium permanganate** ($KMnO_4$)**:**

i). Acidic **medium:**

Equivalent weight of $KMnO_4$ = Mol. Wt / 5

$MnO_4^- + 8\ H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$ (gained 5 electrons from reductant)

Equivalent weight of $KMnO_4$ = 158.04 / 5 = 31.608

ii). **Alkaline medium**

Equivalent weight of $KMnO_4$ = Mol. Wt / 1 = 158.04/1

$MnO_4^- + e^- \rightarrow MnO_4^{2-}$

Permanganate ion — Manganate ion

iii). **Neutral Medium**

Equivalent weight of $KMnO_4$ = Mol.Wt / 3 = 158.04/3 = 52.68

$MnO_4^- + 2\ H_2O + 6e^- \rightarrow MnO_2\downarrow + 4\ OH^-$

Manganese dioxide ppt

e

b. Potassium dichromate($K_2Cr_2O_7$):

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

Equivalent weight of $K_2Cr_2O_7$ = Mol.Wt / 6 = 294.185/6 = 49.03

Equivalent Weight of reducing agent:

Reduction process involves gain of electrons. A reducing agent is one which loses the electrons; it reduces the other substance by donating electrons to it.

Equivalent weight of a reducing agent = Mol. Wt / No. of electrons lost

a. Equivalent Wt. of Mohr's Salt ($Fe\ SO_4(NH_4)_2\ SO_4.6\ H_2O$) = Mol.Wt / 1 = 392.13/1

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

b. Equivalent Wt. of Oxalic acid ($H_2C_2O_4.2\ H_2O$) = Mol.Wt / 2 = 126/2

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-$$

c. Equivalent Wt. of Sodiumthiosulphate($Na_2S_2O_3.5H_2O$)= Mol.Wt/1=248.18/1

$$S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\ e^-$$

CONTENTS

OBSERVATIONS AND CALCULATIONS:

1. Calibration of pipette

Mass of empty beaker = m_1g

Mass of empty beaker + water = m_2g

Mass of water = (m_2 -m_1) g

Volume of water = $\frac{(m2-m1)g}{\text{Density of Water}}$

2. Calibration of volumetric flask:

Mass of empty volumetric flask = m_1g

Mass of flask + water = m_2 g

Mass of water = (m_2 -m_1)g

Volume of water = $\frac{(m2-m1)g}{\text{Density of Water}}$

3.Calibration of burette:

Burette readings	Mass of water Transferred(g)	Volume of water $=\frac{(m2-m1)g}{\text{Density of Water}}$
0	m_1	------
5	m_2	$v_1=\frac{(m2-m1)g}{\text{Density of Water}}$
10	m_3	$v_2=\frac{(m2-m1)g}{\text{Density of Water}}$
15	m_4	$v_3=\frac{(m2-m1)g}{\text{Density of Water}}$

EXPERIMENT NO: 1

CALIBRATION OF VOLUMETRIC GLASS WARES

Aim: To calibrate glassware: i) **Pipette** ii) **Burette** iii) **Volumetric flask**

Principle: The object of calibration is to detect inaccuracies in the graduations of glassware like the pipette, burette and the volumetric flask. They can be calibrated by measuring the real volume of solution delivered or contained by weighing mass of the water. Weighing is done with very good accuracy. Under a constant temperature and atmospheric pressure, the density of distilled water is constant. Knowing the density of water, we can calculate volume of the given mass of water at a given temperature. Thus, we can determine exact capacity of the glassware.

Mass of the water can be converted to the volume using equation: $V=\boldsymbol{m/d}$

Where; m is the mass of water and d its density

Procedure:

1. **Calibration of pipette:** A clean beaker is accurately weighed. Distilled water is drawn into the pipette which is to be calibrated and the lower meniscus is exactly adjusted to the mark. The water is now discharged into the weighed beaker. The beaker is again weighed accurately. The volume of water can be calculated at the given temperature.

2. **Calibration of volumetric flask:** A volumetric flask to be calibrated is accurately weighed. The mass of the flask m1g is recorded. Distilled water is filled into the flask and the lower meniscus is exactly adjusted to the mark. The flask is again weighed accurately. Let the mass found out be m2g. The volume of water at the given temperature is calculated.

3. **Calibration of burette:** The burette is calibrated by transferring water between any two markings on the burette and then finding out its mass. The calibration of the burette is done for every 5 ml. The initial reading on the burette is noted down and the first 5ml (ie 0-5ml) is transferred into a previously weighed beaker (m_1g) .The beaker is with the transferred water is weighed again (m_2g). The calibrated volume is calculated.

Similarly the calibration is done for every 5ml (i e. between 0-5, 5-10, 10-15, markings) and the calibrated volume in each case is calculated. The observations are recorded in a tabular column.

Result: 1. Correct Volume of Pipette=…….

2. Correct Volume of Volumetric Flask=……

3. Correct Volume of Burette=…….

OBSERVATIONS:

REACTION:

$$2KMnO_4+5Na_2C_2O_4+8H_2SO_4 \rightarrow K_2SO_4+2MnSO_4+5Na_2SO_4+8H_2O+10CO_2$$

PART–I: Preparation of standard sodium oxalate solution

1. Mass of empty weighing bottle +sodium oxalate crystals(m_1)= g
2. Mass of empty weighing bottle (m_2)= g
3. Mass of sodium oxalate (m_1-m_2) = g

Normality of sodium oxalate = $\frac{(m1-m2)\times 10}{67}$ =.....N

PART-II: ESTIMATION OF $KMnO_4$

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Volume of $KMnO_4$ run down in cm^3(V_2)			

(V_2N_2) $KMnO_4$ = (V_1N_1) $Na_2C_2O_4$

Normality of KMnO4 (N_2) = (V_1N_1) $Na_2C_2O_4$)/V_2

Mass of $KMnO_4$ crystals present in 100 $cm^3$of its solution = $\frac{N2\times 31.6}{10}$ ..... g

EXPERIMENT NO: 2

AIM: ESTIMATION OF POTASSIUM PERMANGANATE USING STANDARD SODIUM OXALATE SOLUTION

PROCEDURE:

Part I: Preparation of standard solution of sodium oxalate

0.67 g of sodium oxalate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in a 100 cm3 standard flask. It is mixed well for uniform concentration. The normality of sodium oxalate is calculated.

Part II: Estimation of Potassium permanganate using std. sodium oxalate solution

10 cm3 of the made up sodium oxalate solution is pipetted out into a clean conical flask. One test tube of dilute sulphuric acid is added to it. The contents of the conical flask are heated nearly to boiling. The hot solution is titrated against potassium permanganate solution taken in the burette slowly with drop wise addition until a permanent pale pink colour is obtained. The titration is repeated for concordant values.

RESULT: Mass of potassium permanganate present in 100 cm^3= …… g.

OBSERVATIONS:

REACTION:

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

$$2K_3Fe(CN)_6 + 3FeSO_4 \rightarrow Fe_3[Fe(CN)_6]_2 + 3K_2SO_4$$

Part I: Preparation of standard potassium dichromate solution

1. Mass of the empty weighing bottle +potassium dichromate crystals(m_1)=……g
2. Mass of the empty weighing bottle with the remaining(m_2)……=.....g
3. Mass of potassium dichromate crystals transferred (m_1.m_2)..... =.....g

Normality of $K_2Cr_2O_7$ (N_1) $= \frac{(m1-m2)\times 10}{49}$

= …… N

Part II: Estimation of Ferrous ammonium sulphate

Burette: $K_2Cr_2O_7$ solution
Conical flask: 10$cm^3$of FAS soln. + 1test tube of dil.H_2SO_4+1t.t of water
Indicator: Diphenyl amine
Endpoint: Green to Violet

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Volume run down in cm^3			

$(V_2N_2)FAS = (V1N1)\ K_2Cr_2O_7$

Normality of FAS (N_2) = $\frac{BR\times N1}{10}$

Mass of Ferrous ammonium sulphate crystals in 100 cm^3 of its solution

$= \frac{N2\times Gr,Eq\ Wt}{10}$

$= \frac{N2\times 392}{10} =$ …… g

EXPERIMENT NO: 3

AIM: ESTIMATION OF FERROUS AMMONIUM SULPHATE USING STANDARD POTASSIUM DICHROMATE SOLUTION AND DIPHENYLAMINE AS AN INTERNAL INDICATOR

PROCEDURE:

Part I: Preparation of standard solution of Potassium dichromate

0.49 g of potassium dichromate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 100 cm^3standard flask. It is mixed well for uniform concentration. The normality of $K_2Cr_2O_7$ is calculated.

Part II: Estimation of Ferrous ammonium sulphate using standard potassium dichromate solution.

Pipette out 10 cm3 of the given FAS solution into a clean conical flask. One test tube of dil.sulphuric acid, ½ test tube of phosphoric acid, one test tube of distilled water and 2-3 drops of diphenyl amine indicator are added. The resulting mixture is titrated against the prepared standard potassium dichromate solution till the appearance of deep violet colour. The titration is repeated for concordant values.

RESULT: Mass of Ferrous ammonium sulphate crystals present in 100cm^3 of its solution is.....g

OBSERVATIONS and CALCULATION:

$K_2Cr_2O_7+7H_2SO_4+6KI \rightarrow 4K_2SO_4+Cr_2(SO_4)_3+3I_2+7H_2O$

$2Na_2S_2O_3 + I_2 \rightarrow 2NaI+Na_2S_4O_6$ (Colourless)

Part I: Preparation of standard potassium dichromate solution

Mass of the empty weighing bottle + potassium dichromate crystals (m_1) = ……g

Mass of the empty weighing bottle with the remaining (m_2) = ...g

Mass of potassium dichromate crystals transferred (m_1-m_2) = ...g

Normality of $K_2Cr_2O_7$ (N_1) = $\frac{(m1-m2)\times10}{49}$ = ...N

Part II: Estimation of Sodium thiosulphate

Burette: $Na_2S_2O_3$ solution

Conical flask: 10 $cm^3$of $K_2Cr_2O_7$+ ¼ test tube of Conc.HCl + 1t.t of KI

Indicator: Freshly prepared starch

End point: Disappearance of blue colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Volume of $Na_2S_2O_3$ run down in cm^3 (V_2)			

(V_2N_2) hypo= (V_1N_1) $K_2Cr_2O_7$

Normality of Sodium thiosulphate (N_2) = $\frac{V1\times N1}{V2}$

Mass of hypo present in 100 cm^3 of the solution = $\frac{N2\times248}{10}$ = ...g

EXPERIMENT NO: 04

AIM: ESTIMATION OF SODIUM THIOSULPHATE USING STANDARD POTASSIUM DICHROMATE SOLUTION

PRINCIPLE: Potassium dichromate liberates iodine from KI quantitatively. Iodine liberated in the reaction is the measure of the concentration of potassium dichromate used. It is titrated against hypo solution using starch an internal indicator near the end point.

PROCEDURE:

Part I: Preparation of standard solution of potassium dichromate:

0.49 g of potassium dichromate crystals is accurately weighed and dissolved in distilled water and the solution is made up to the mark in 100 cm3 standard flask. It is mixed well for uniform concentration. The normality of $K_2Cr_2O_7$ is calculated.

Part II: Estimation of Sodium thiosulphate using standard potassium dichromate

Pipette out 10 cm^3 of the prepared $K_2Cr_2O_7$ solution into a clean conical flask. Add 1/2 test tube of KI followed by 1/4 test tube of Conc.HCl. Also, add a test tube of distilled water. The reaction mixture is shaken and kept aside for one minute. The liberated iodine solution is titrated against the made up sodium thiosulphate solution taken in the burette till a pale yellow colour is obtained, 2-3 drops of the freshly prepared starch indicator is added, the solution turns blue. The blue solution is titrated against the same hypo solution till green colour is obtained or blue colour disappears. The titration is repeated for concordant values.

RESULT: Mass of Sodium thiosulphate crystals present in 100 cm^3 =.....g

OBSERVATIONS and **Reactions**:

$Na_2H_2Y \rightarrow 2Na^+ + H_2Y^{2-}$

$Zn^{2+} + H_2Y^{2-} \rightarrow ZnY^{2-} + 2H^+$

Na_2H_2Y = Disodium salt of EDTA – $C_{10}H_{14}N_2Na_2.2H_2O$

PART-I;a) Preparation of standard Zinc sulphate solution

1. Mass of weighing bottle with $ZnSO_4.7H_2$ crystals (W_1) =……g
2. Mass of Empty weighing bottle after transfer (W_2) =……g
3. Mass of $ZnSO_4.7H_2O$ crystals transferred (W_1-W_2) =……g

Molarity of $ZnSO_4$ solution (M_1) = $\frac{(W_1-W_2) \times 10}{287.5}$ = =……M

b) Standarization of EDTA solution:

Burette: EDTA solution

Conical flask: 10 cm^3 of $ZnSO_4$ solution + 2 cm^3 of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(V_2 cm^3)			

Concordant Value = …… cm^3

$(V_2M_2)_{EDTA} = (V_1M_1)_{ZnSO4}$

Molarity of EDTA solution (M_2) = 10 x M_1/V_2 = ……M

PART-II: Estimation of Zinc

Burette: Standardised EDTA solution

Conical flask: 10 cm^3 of $ZnSO_4$ solution (given) + 2 cm^3 of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm^3)			

Concordant Value = …… cm^3

$(V_3M_3)_{Zn2+} = (V_2M_2)_{EDTA}$

Molarity of Zn^{2+} solution (M_3) = V_2 x M_2/V_3 = ……M

Mass of Zn^{2+} ions present in 100 cm^3 of the = Molar mass x Molarity (M_3)/10 =

$$\frac{(65.37 \times \text{Molarity})}{10}$$

= ……g

EXPERIMENT NO: 5

AIM: TO ESTIMATE THE AMOUNT OF ZINC PRESENT IN 100 Cm^3 OF THE GIVEN SOLUTION USING 0.01M EDTA SOLUTION AND ZINC SULPHATE CRYSTALS

PRINCIPLE: Zinc ions in solution form coloured complex with the indicator as well as colourles complex with EDTA. Zinc-indicator complex is less stable than Zn-EDTA complex. At the end point all the free Zinc ions are complexed with EDTA. Now EDTA removes the metal forms the less stable metal-indicator complex releasing the free indicator which has a blue colour at pH-10.

PROCEDURE:

PART I: PREPARATION OF STANDARD ZINC SULPHATE SOLUTION AND

STANDARDIZATION OF EDTA SOLUTION

About 0.3 g of pure zinc sulphate crystals are weighed accurately dissolved in about one test tube of dil.sulphuric acid and transferred into 100 cm^3 standard flask and made up to the mark using distilled water and shaken well for uniform concentration.

10 cm^3 of the made up solution is pipetted out into a clean conical flask, 2 cm^3 of ammonia-ammonium chloride buffer of pH-10 are added and a pinch of Erio-chrome black-T indicator is added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

PART-II: ESTIMATION OF ZINC

The given Zinc sulphate solution in the standard flask is made up to the mark using distilled water and shaken well. 10 cm^3 of the made up solution is pipetted out into a clean conical flask, 2cm^3 of ammonia-ammonium chloride buffer of pH-10 are added and a pinch of Eriochrome black-T indicator is added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

RESULT: Mass of Zn^{2+} present in 100 cm^3 of the given solution is = ...g

OBSERVATIONS:

Reactions: $Na_2H_2Y \rightarrow 2Na^+ + H_2Y^{2-}$

$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+$ (M = Ca, Mg, Zn)

Na_2H_2Y = Disodium salt of EDTA – $C_{10}H_{14}N_2Na_2.2H_2O$

PART-I:a) Preparation of standard Zinc sulphate solution

1. Mass of weighing bottle with $ZnSO_4.7H_2O$ crystals (W_1) =…….g
2. Mass of Empty weighing bottle after transfer (W_2) =……...g
3. Mass of $ZnSO_4.7H_2O$ crystals transferred (W_1-W_2) =…….g

Molarity of $ZnSO_4$ solution (M_1) = $\frac{(W_1-W_2)x10}{287.5}$ = =……M

b) Standarization of EDTA solution:

Burette: EDTA solution

Conical flask: 10 cm^3 of $ZnSO_4$ solution + 2 cm^3 of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm^3)			

Concordant value = …… cm^3

$(V_2M_2)_{EDTA} = (V_1M_1)_{ZnSO4}$

Molarity of EDTA solution (M_2) = 10x M_1/V_2 = 10 x……...

= ……M

PART-II: **Determination of Total hardness of water**

Burette: Standardised EDTA solution

Conical flask: 25 cm^3 of the given sample of water + 2 cm^3 of pH-10 buffer solution

Indicator: 3-4 crystals of Eriochrome black-T;

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm3)			

Concordant Value = …… cm^3

$(V_3M_3)_{Ca2+} = (V_2M_2)_{EDTA}$

Molarity of Ca^{2+}solution (M^3) = V_2 x M_2/V_3 = ……. x……../10

= ……..M

Hence Mass of $CaCO_3$ present in 100 cm^3 = M_3x 100/10 =……g

Amount of $CaCO_3$ in 10^6 cm^3= ……x 10^6/100 =……ppm

EXPERIMENT NO: 6
DETERMINATION OF TOTAL HARDNESS OF OF WATER

AIM: TO ESTIMATE THE TOTAL HARDNESS OF THE GIVEN SAMPLE OF WATER USING 0.01M EDTASOLUTION AND ZINC SULPHATE CRYSTALS

PRINCIPLE: Hardness of water is due to the presence of calcium and magnesium ions. These ions form characteristic coloured complexes with the indicator and colourless complex with the metal ions. The EDTA –metal ion complex is more stable than the metal-indicator complex.

The metal-ion indicator complex is wine red in colour while the free indicator is blue between the pH 7-11. During titration of a sample of hard water against EDTA, the colour of the solution changes from wine red to deep blue. Total hardness of water is calculated from the titre value and expressed in parts per millions of CaCO3.

PROCEDURE:

PART I : PREPARATION OF STANDARD ZINC SULPHATE AND STANDARDIZATION OF EDTA SOLUTION

About 0.3 g of pure zinc sulphate crystals are weighed accurately dissolved in about one test tube of dil.sulphuric acid and transferred into 100 cm^3 standard flask and made up to the mark using distilled water and shaken well for uniform concentration.

10 cm^3 of the made up solution is pipetted out into a clean conical flask, 2 cm^3 of ammonia-ammonium chloride buffer of pH-10 are added and about 2 crystals of Erio-chrome black-T indicator are added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

PART II: **DETERMINATION OF TOTAL HARDNESS OF WATER**

25 cm^3 of the given sample of hard water is pipette out into a clean conical flask, 2 cm^3ofammonia-ammonium chloride buffer of pH-10 are added and about 2 crystals of Erio-chrome black-T indicator are added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

RESULT: The total hardness of the given sample of water is ……ppm of $CaCO_3$

OBSERVATIONS:

Reactions: $Fe_2O_3 + 6HCl \rightarrow FeCl_3 + 3H_2O$

$2Fe^{3+} + Sn^{2+} \rightarrow 2Fe^{2+} + Sn^{4+}$

$SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + Hg_2Cl_2$

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

PART-I: **a) Preparation of standard ferrous ammonium sulphate solution**

1. Mass of weighing bottle with Ferrous Ammonium Sulphate crystals (W_1)=g
2. Mass of Empty weighing bottle after transfer (W_2) =……g
3. Mass of Ferrous Ammonium Sulphate crystals transferred (W_1-W_2) =……g

Normality of FAS = $\frac{(W_1-W_2)x10}{392}$ = =……N

b)Standarization of Potassium dichromate solution:

Burette: Potassium dichromate solution;

Conical flask: 10 cm^3 of FAS solution + 4 cm^3 of $H_3PO_4 + H_2SO_4$ mixture

Indicator: Diphenylamine indicator,

End point: Green to violet.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $K_2Cr_2O_7$(cm^3)			

Concordant Value = …… cm^3

$V_2N_2 = V_1N_1$, ,

($K_2Cr_2O_7$) (FAS) Normality of $K_2Cr_2O_7$ solution (N_2) = $\frac{10\times N1}{V2}$ = ...N

PART-II: **Estimation of Iron**:

Burette: Potassium dichromate solution;

Conical flask: 10 cm^3 of reduced haematite ore solution + 4 cm^3 of $H_3PO_4 + H_2SO_4$ mixture

Indicator: Diphenyl amine

End point: Green to violet.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $K_2Cr_2O_7$(cm^3)			

Concordant Value = …… cm^3

Normality of Ore solution containing iron (N_3) = $V_2x\ N_2/V_3$ = ……x…… /10 =

= _____ N

Mass of Iron present in 100 cm^3 of the given Haematite ore solution = $\frac{N3\times Gr.Eq.Wt}{10}$

$=\frac{N3X\ 55.45}{10}$

= ……g

Percentage of Iron in the given sample of Haematite ore = $\frac{Mass\ of\ Iron\ \times 100}{1.2}$=.....

EXPERIMENT NO.7 (ANALYSIS HAEMATITE ORE)

AIM: DETERMINATION OF THE PERCENTAGE OF IRON IN THE GIVEN SAMPLE OF HAEMATITE ORE(1.2 g of the ore is dissolved in in 100 cm^3 of the given solution) USING POTASSIUM DICHROMATE SOLUTION AND FERROUS AMMONIUM SULPHATE CRYSTALS

PRINCIPLE: Haematite ore mainly contains iron having the composition Fe_2O_3 dissolved in 1:1 HCl and filtered. Ferric ions in the solution are reduced to ferrous state by stannous chloride and con. HCl, the resulting ferrous ions is estimated using potassium dichromate solution in acid medium and diphenyl amine as internal indicator.

PROCEDURE:

PART-I: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION AND STANDARDIZATION OF POTASSIUM DICHROMATE SOLUTION

About of 2.5 g of ferrous ammonium sulphate crystals are weighed in a weighing bottle and transferred into 100 cm^3 beaker, 0.5 cm^3 of conc. sulphuric acid is added, dissolved in water and transferred into 100 cm^3 standard flask and made up to the mark using distilled water.

10cm^3 of the made up solution is pipetted out into a clean conical flask, one test tube of acid mixture (Half test tube of phosphoric acid and half test tube of con. sulphuric acid mixture) are added into it, 2 drops of diphenyl amine indicator is added and titrated against the given potassium dichromate solution taken in the burette till the green solution changes into violet. The titration is repeated for concordant values.

PART-II: ESTIMATION OF PERCENTAGE OF IRON IN THE HAEMATITE ORE

The given haematite ore solution in the standard flask is made up to the mark using distilled water and shaken well. 10cm^3 of the made up solution is pipetted out into a clean conical flask, to this solution 2cm^3 of Conc. HCl is added. The resulting solution is boiled; stannous chloride solution is added in drops till the solution becomes colourless (to ensure the complete reduction 2 drops are added in excess), 2 test tubes of water are added and cooled to the room temperature. Half test tube of saturated mercuric chloride is added at a stretch, silky white precipitate is obtained (if it turns grey, the solution is rejected and the reduction is repeated). To this solution, 1 test tube of acid mixture is added (Half test tube of phosphoric acid and half test tube of conc. sulphuric acid mixture) are added into it, 2 drops of diphenyl amine indicator is added and titrated against the standardized potassium dichromate solution till the colour of the solution changes green to violet. The titration is repeated for concordant readings.

Result:
1. Amount/Mass of iron present in 100 cm^3 of the given solution = ……g
2. The percentage of iron in the given sample of Haematite ore =.

REACTIONS: **1.** $CO_3^{2-} + H_3O^+ \rightarrow HCO^- + H_2O$

2. $HCO_3^- + H_3O^+ \rightarrow H_2CO_3 + H_2O$

Burette: 0.1N HCl solution
Conical flask: W g of sodium carbonate and sodium bicarbonate mixture
Indicator: a) Phenolphthalein b) Methyl orange
Endpoint: a) Appearance of permanent pale pink b)Yellow to pale pink

a)Phenolphthalein end point:

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Volume of HCl run down in cm^3			= x

b)Methyl orange end point:

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Volume of HCl run down in cm^3			= y

Percentage of sodium carbonate=y x 0.1 x 106 x100/ w

Percentage of sodium bicarbonate= (x-y) x0.1x 84x100/w

EXPERIMENT NO.8

AIM: **ESTIMATION OF CARBONATE AND BI-CARBONATE IN A GIVEN MIXTURE**

Principle: Carbonic acid is a diprotic acid, which furnishes two protons per molecule. The estimation of carbonate and bi-carbonate in a mixture of the two is based on equilibria of di protic acid and its salt. The first pK_a is 6.34 and the second is 10.36, Phenolphthalein is a suitable indicator for the first end point and methyl orange for the second. Mixtures of carbonates and bicarbonates are analyzed using the double indicator method.

PROCEDURE:A known mass (w g) of a mixture of sodium carbonate and sodium bicarbonate is transferred into a clean conical flask followed by one test tube of distilled water and two drops of phenolphthalein indicator and the solution is titrated against standard 0.1N HCl until a permanent pale pink color is got. Let the average value got is x cc, At this stage two drops of methyl orange is added and the titration is continued till the orange color changes to permanent pale pink. Let the average titre value be y cc.

RESULT: The percentage of carbonate and bicarbonate in the given salt is…….

OBSERVATIONS

REACTIONS:

$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$

$2K_3Fe(CN)_6 + 3FeSO_4 \rightarrow Fe_3[Fe(CN)_6]_2 + 3K_2SO_4$

Part I: Estimation of ferrous iron

Burette: 0.1N $K_2Cr_2O_7$ solution

Conical flask: 25 cm^3 of Ferrous-ferric mixture+2test tubes of dil.H_2SO_4 +1t.t of water,

Indicator: Di-phenyl amine .

End point: Appearance of violet colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. run down in c.c			

$(V_2N_2)_{\text{Ferrous iron}} = (V_1N_1)_{\text{pot.dich.}}$; Normality of Fe^{2+} iron(N_2) = ...x 0.1/25 =

Amount of ferrous iron present in 250 cm^3 of its solution=N_2 x g.eq.wt/4

= .....x 27.925/4 =.....g

Part I: Estimation of ferric iron

Burette: 0.1N $K_2Cr_2O_7$ solution

Conical flask: 25 cm^3 of Ferrous-ferric mixture+2test tubes of dil.H_2SO_4 +1t.t of water,

Indicator: Di-phenyl amine,

End point: Appearance of violet colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. run down in c.c			

$(V_2N_2)_{\text{Ferric iron}} = (V_1N_1)_{\text{pot.dich.}}$; Normality of Fe^{3+} iron(N_2) = ...x 0.1/25 =

Amount of total ferric iron present in 250 cm^3 of its solution=N_2 x g.eq.wt/4

= .....x 55.85/4 =.....g

EXPERIMENT NO. 09

AIM:ESTIMATION OF FERROUS AND FERRIC IRON IN A GIVEN USING STANDARD POTASSIUM DICHROMATE SOLUTION

PROCEDURE:1. ESTIMATION OF FERROUS IRON

25 cc of the given ferrous ferric mixture is pipetted out into a clean conical flask. Two test tubes of dil.sulphuric acid, ½ test tube of phosphoric acid, one test tube of distilled water and 2-3 drops of diphenyl amine indicator are added. The resulting mixture is titrated versus the standard potassium dichromate solution till the appearance of deep violet colour. The titration is repeated for concordant values.

2.ESTIMATION OF TOTAL IRON:

25cc of the given ferrous ferric mixture is pipetted out into a clean conical flask followed by ½ a test tube of 2N con.HCl. The solution is heated, to the hot solution stannous chloride solution is added drop wise from a burette until it becomes just colourless, 2-3 drops are added in excess and the conical flask is cooled externally by running cold water from a tap. To the cold solution 5 ml of mercuric chloride is added at a stretch when a silky white precipitate is got. This solution is titrated against standard potassium dichromate solution taken in a burette using diphenyl amine as indicator till violet colour is obtained as the end point. The experiment is repeated for concordant values.

RESULT: Amount of ferrous iron present in the given solution is .....g and theamount of ferric iron present in the given solution is .....g.

$CaOCl_2+2CH_3COOH+2KI \rightarrow CaCl_2+ 3CH_3COOK +3I_2+H_2O$

$2Na_2S_2O_3+I_2 \rightarrow 2NaI+Na_2S_4O_6$ (Colourless)

Burette: 0.1N $Na_2S_2O_3$
Conical flask: 10cm^3 of bleaching powder soln.+2 cm^3 of glacial acetic acid + 1/2 test tube of distilled water
Indicator: Freshly prepared starch
End point: Disappearance of blue colour

Trial no.	1	2	3
Final burette reading			
Initial burette reading			
Vol. run down in cm^3			

$(VxN)_{\text{bleaching powder}} = (VxN)_{\text{hypo}}$;

Normality of bleaching powder $= \frac{0.1x\ldots.}{10}$

$= \ldots\ldots\ldots\ldots\ldots$ N

Mass or amount of chlorine in100cm^3=35.5xN/10 $= \frac{35.5\times\ldots\ldots}{10} =$ g

The mass of bleaching powder dissolved is 'm' g

Hence percentage of available chlorine in the bleaching powder =w x100/m

$\frac{=\ldots\ldots\ldots x100}{m} = \ldots\ldots\ldots\ldots\ldots$ (z)

EXPERIMENTNO.10

AIM: DETERMINATION OF PERCENTAGE OF AVAILABLE CHLORINE IN THE GIVEN SAMPLE OF BLEACHING POWDER

PRINCIPLE: Available chlorine in a sample of bleaching powder is estimated volumetrically.

A known mass of bleaching powder as a suspension of water is treated with excess of potassium iodide solution. The solution is then acidified with a strong solution of acetic acid. The liberated iodine is titrated against standard sodium thiosulphate solution using starch as indicator.

PROCEDURE: About 2g of bleaching powder is weighed and dissolved in Con. hydrochloric acid and the solution is made up to 100 ml using distilled water in a standard flask. 10 cm^3 of the made up solution is pipetted out into a clean conical flask followed by one test tube of distilled water, 2cm^3 of 10% KI solution and 2cm^3 of glacial acetic acid. The liberated iodine is titrated against standard 0.1N sodium thiosulphate solution taken in the burette till a pale yellow colour is obtained. 2-3 drops of freshly prepared starch indicator is added. The resulting blue solution obtained is titrated against the same hypo solution till the blue colour just discharges. The titration is repeated for concordant values.

RESULT: The percentage of the available chlorine in the given sample of bleaching powder is……..

Periodic Table of the Elements

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1 H Hydrogen 1.01																	2 He Helium 4.00
3 Li Lithium 6.94	4 Be Beryllium 9.01											5 B Boron 10.81	6 C Carbon 12.01	7 N Nitrogen 14.01	8 O Oxygen 16.00	9 F Fluorine 19.00	10 Ne Neon 20.18
11 Na Sodium 22.99	12 Mg Magnesium 24.31											13 Al Aluminum 26.98	14 Si Silicon 28.09	15 P Phosphorus 30.97	16 S Sulfur 32.06	17 Cl Chlorine 35.45	18 Ar Argon 39.95
19 K Potassium 39.10	20 Ca Calcium 40.08	21 Sc Scandium 44.96	22 Ti Titanium 47.88	23 V Vanadium 50.94	24 Cr Chromium 51.99	25 Mn Manganese 54.94	26 Fe Iron 55.85	27 Co Cobalt 58.93	28 Ni Nickel 58.69	29 Cu Copper 63.55	30 Zn Zinc 65.38	31 Ga Gallium 69.72	32 Ge Germanium 72.63	33 As Arsenic 74.92	34 Se Selenium 78.97	35 Br Bromine 79.90	36 Kr Krypton 83.80
37 Rb Rubidium 85.47	38 Sr Strontium 87.62	39 Y Yttrium 88.91	40 Zr Zirconium 91.22	41 Nb Niobium 92.91	42 Mo Molybdenum 95.95	43 Tc Technetium 98.91	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.91	46 Pd Palladium 106.42	47 Ag Silver 107.87	48 Cd Cadmium 112.41	49 In Indium 114.82	50 Sn Tin 118.71	51 Sb Antimony 121.76	52 Te Tellurium 127.6	53 I Iodine 126.90	54 Xe Xenon 131.29
55 Cs Cesium 132.91	56 Ba Barium 137.33	57-71 Lanthanides	72 Hf Hafnium 178.49	73 Ta Tantalum 180.95	74 W Tungsten 183.85	75 Re Rhenium 186.21	76 Os Osmium 190.23	77 Ir Iridium 192.22	78 Pt Platinum 195.08	79 Au Gold 196.97	80 Hg Mercury 200.59	81 Tl Thallium 204.38	82 Pb Lead 207.20	83 Bi Bismuth 208.98	84 Po Polonium [208.98]	85 At Astatine 209.98	86 Rn Radon 222.02
87 Fr Francium 223.02	88 Ra Radium 226.03	89-103 Actinides	104 Rf Rutherfordium [261]	105 Db Dubnium [262]	106 Sg Seaborgium [266]	107 Bh Bohrium [264]	108 Hs Hassium [269]	109 Mt Meitnerium [278]	110 Ds Darmstadtium [281]	111 Rg Roentgenium [280]	112 Cn Copernicium [285]	113 Nh Nihonium [286]	114 Fl Flerovium [289]	115 Mc Moscovium [289]	116 Lv Livermorium [293]	117 Ts Tennessine [294]	118 Og Oganesson [294]

57 La Lanthanum 138.91	58 Ce Cerium 140.12	59 Pr Praseodymium 140.91	60 Nd Neodymium 144.24	61 Pm Promethium 144.91	62 Sm Samarium 150.36	63 Eu Europium 151.96	64 Gd Gadolinium 157.25	65 Tb Terbium 158.93	66 Dy Dysprosium 162.50	67 Ho Holmium 164.93	68 Er Erbium 167.26	69 Tm Thulium 168.93	70 Yb Ytterbium 173.06	71 Lu Lutetium 174.97
89 Ac Actinium 227.03	90 Th Thorium 232.04	91 Pa Protactinium 231.04	92 U Uranium 238.03	93 Np Neptunium 237.05	94 Pu Plutonium 244.06	95 Am Americium 243.06	96 Cm Curium 247.07	97 Bk Berkelium 247.07	98 Cf Californium 251.08	99 Es Einsteinium [254]	100 Fm Fermium 257.10	101 Md Mendelevium 258.10	102 No Nobelium 259.10	103 Lr Lawrencium [262]

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Alkali Metal | Alkaline Earth | Transition Metal | Basic Metal | Metalloid | Nonmetal | Halogen | Noble Gas | Lanthanide | Actinide