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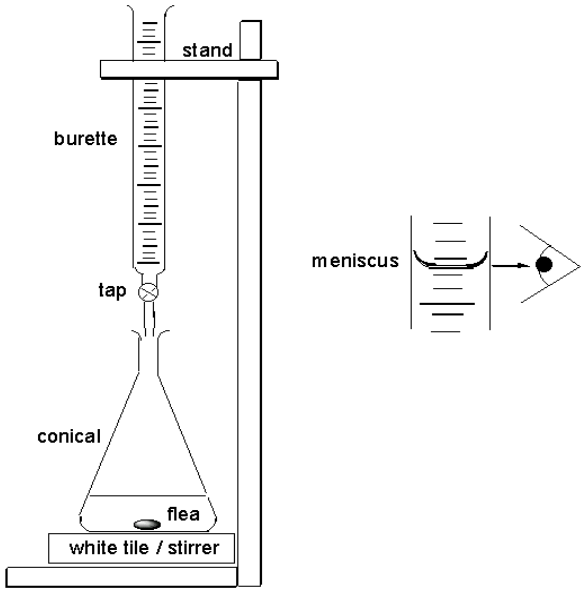
CHEMISTRY DIVISION

Chemistry Sessional

LIST OF EXPERIMENTS

Sl	Name of The Experiment
01	Standardization of Sodium Hydroxide (NaOH) Solution with Standard Oxalic Acid dihydrate ($C_2H_2O_4.2H_2O$) Solution. (STRONG BASE WEAK ACID NEUTRALIZATION)
02	Standardization of Hydrochloric Acid (HCl) Solution with Standard Sodium Hydroxide (NaOH) Solution. (STRONG ACID STRONG BASE NEUTRALIZATION)
03	Standardization of Hydrochloric Acid (HCl) Solution with Standard Sodium Carbonate (Na_2CO_3) Solution. (STRONG ACID WEAK BASE NEUTRALIZATION)
04	Determination of Calcium (Ca) Content in a Calcium Chloride dihydrate ($CaCl_2.2H_2O$) Solution with Standard Di-Sodium Ethylene Diammine TetraAceticAcid (Na_2EDTA) Solution. (COMPLEXOMETRIC TITRATION)
05	Standardization of Sodium Thiosulphate Pentahydrate ($Na_2S_2O_3.5H_2O$) Solution with Standard Potassium Dichromate ($K_2Cr_2O_7$) Solution. (IODOMETRIC TITRATION)
06	Estimation of Copper (Cu) Content in a Copper Sulphate Pentahydrate ($CuSO_4.5H_2O$) (Blue Vitriol) Solutions by Iodometric Method with Standard Sodium Thiosulphate Pentahydrate ($Na_2S_2O_3.5H_2O$) Solution. (IODOMETRIC TITRATION)
07	Standardization of Potassium Permanganate ($KMnO_4$) Solution with Standard Oxalic Acid dihydrate ($C_2H_2O_4.2H_2O$) Solution. (OXIDATION REDUCTION TITRATION)
08	Determination of Ferrous (Fe) Content in a Ammonium Ferrous Sulphate (Mohr`s Salt) [$FeSO_4.(NH_4)_2SO_4.6H_2O$] Solution with Standard Potassium Permanganate ($KMnO_4$) Solution. (REDOX TITRATION)
09	Determination of Zinc (Zn) Content in a Zinc Sulphate Heptahydrate ($ZnSO_4.7H_2O$) Solution with Standard Di-Sodium EDTA (Na_2-EDTA) Solution by using Eriochrome black T indicator. (COMPLEXOMETRIC TITRATION)
10	Determination of Ferrous (Fe) (Iron) Content in a Supplied Solution with Standard Potassium Permanganate ($KMnO_4$) Solution.
11	Determination of the Hardness of Water With Standard Di-Sodium EDTA (Na_2-EDTA) Solution by Using Eriochrome black T indicator. (*ANALYSIS* HARDNESS OF H_2O)

MILITARY INSTITUTE OF SCIENCE AND TECHNOLOGY



Exp No : **Course Code : CHEM -** **DEPT:**

Name : ……

Roll No : ……

Level / Term & Group : ……

Date of Exp : ……

Signature of Teacher : ……

Exp No:01

Name of the Exp: Standardization of 0.1 (N) (Approx) Sodium Hydroxide (NaOH) Solution with Standard Oxalic Acid dihydrate($C_2H_2O_4.2H_2O$) Solution. *(NORMALITY)*

1. Theory:

The titration of a weak acid with a strong base involves the direct transfer of protons from the weak acid to the hydoxide ion. The reaction of the weak acid, oxalic acid, with a strong base, NaOH, can be seen below. In the reaction the acid and base react in a one to one ratio.

$$C_2H_2O_4\,(aq) + 2OH^-(aq) \rightarrow C_2O_4^{2-}(aq) + H_2O\,(l)$$

$$H_2C_2O_4(aq) + 2\,NaOH(aq) \rightarrow Na_2C_2O_4(aq) + 2\,H_2O(l)$$

. In this reaction a buret is used to administer one solution to another. The solution administered from the buret is called the titrant. The solution that the titrant is added to is called the analyte. In a titration of a Weak Acid with a Strong Base the titrant is a strong base and the analyte is a weak acid. In order to fully understand this type of titration the reaction, titration curve, and type of titration problems will be introduced

2. Apparatus: Volumetric flask (100ml),Conical flask(250ml),Burette, Pipette(10ml), Funnel, Wash Bottle, Iron Stand etc.

3. Chemicals:

- **Sodium Hydroxide (NaOH)**
- **Oxalic Acid di-hydrate ($C_2H_2O_4.2H_2O$)**
- **Phenolphthalein ($C_{20}H_{14}O_4$)**

4. Phase: The expt. can be done two phases.

a. Preparation of Standard 0.1(N) $C_2H_2O_4.2H_2O$ soln.
b. Standardization of NaOH With Standard $C_2H_2O_4.2H_2O$ soln.

5. Procedure:

a. Preparation of Standard (approx) 0.1 (N) $C_2H_2O_4.2H_2O$ Soln: Wt out **0.63** gm of $C_2H_2O_4.2H_2O$ into a 100ml Volumetric flask, then make up to the mark by adding distilled water (Stirring until the sample dissolves completely).

Strength of $C_2H_2O_4.2H_2O$ soln = (Wt taken in gm / Wt to be taken in gm) X N/10

= (N)

= (N)

b. <u>Standardization of NaOH Soln With Standard $C_2H_2O_4.2H_2O$ Soln</u>:

(1) Take 10 ml Oxalic acid ($C_2H_2O_4.2H_2O$) Solution into a Conical flask.

(2) Dilute up to 100 ml distilled water.

(3) 2/3 drops of phenolphthalein indicator, colorless solution appears.

(4) Titrate with Sodium Hydroxide (NaOH) solution (**which to be determined**).

(5) Pink color appears at the end point.

<u>Table for Vol of Sodium Hydroxide (NaOH)</u>

Number of Observation	**Vol of $C_2H_2O_4.2H_2O$ soln,V_1(ml)**	**<u>Vol of NaOH soln</u>**				**Remarks**
		IBR(ml)	**FBR(ml)**	**Diff(ml)**	**MeanVol, $V_{2\,(ml)}$**	
1						Minimum one observation must be checked and signed
2						
3						
4						

6. <u>Experimental Data:</u>

V_1(Vol of $C_2H_2O_4.2H_2O$ soln) = ml

S_1(Strg of $C_2H_2O_4.2H_2O$ soln) = (N)

V_2 (Vol of NaOH soln) = ml

S_2 (Strg of NaOH soln) = ?

7. <u>Calculation</u>: $S_2 = V_1S_1/V_2$

= ---------- (N) Soln

= (N) Soln

8. <u>Result</u>: The Strength of Supplied Sodium Hydroxide **(NaOH)** Sample Solution is ----

---------- (N)

9. <u>% of error</u> :

[(Std result ~ Obs result) / Std result] X 100

=

= %

10. <u>Discussion / Viva voce</u>:

KEY POINT (EXP:01)

1. **Molarity**

2. Normality

3. Titration

4. Indicator

5. Equivalent weight

ABSTRACT / PRECIS

Exp No:02

Name of the Exp : **Standardization of Hydrochloric Acid (HCl) Solutions with Standard Sodium Hydroxide (NaOH) Solution.** ***(NORMALITY)***

1. **Theory:**

Strong acid-Strong base titration is performed using a phenolphthalein indicator. Phenolphtalein is chosen because it changes color in a pH range between 8.3 - 10. It will appear pink in basic solutions and clear in acidic solutions. In the case of a strong acid-strong base titration, this pH transition would take place within a fraction of a drop of actual neutralization, since the strength of the base is high.
The addition of reactants is done from a burette. The reactant of unknown concentration is deposited into an Erlenmeyer flask and is called the analyte. The other reactant of known concentration remains in a burette to be delivered during the reaction. It is known as the titrant. The indicator—phenolphthalein, in this case—has been added to the analyte in the Erlenmeyer flask.
Neutralization is the reaction between an acid and a base, producing a salt and neutralized base. For example, hydrochloric acid and sodium hydroxide form sodium chloride and water:

$$\text{Acid} + \text{Base} \rightarrow \text{Salt} + H_2O$$

$$HCl\ (aq) + NaOH\ (aq) \rightarrow H_2O\ (l) + NaCl\ (aq)$$

Neutralization is the basis of titration. A pH indicator shows the equivalence point—the point at which the equivalent number of moles of a base have been added to an acid. It is often wrongly assumed that neutralization should result in a solution with pH 7.0; this is only the case in a strong acid and strong base titration.

2. **Apparatus**: Volumetric flask(100ml),Conical flask(250ml),Burette, Pipette(10ml), Funnel, Wash Bottle, Iron Stand etc.

3. **Chemicals:**
   - **Hydrochloric Acid (HCl),**
   - **Sodium Hydroxide (NaOH)**
   - **Phenolphthalein ($C_{20}H_{14}O_4$)**

4. **Phase**: The exp can be done two phases.

   a. **Preparation of standard 0.1(N) NaOH soln.**
   b. **Standardization of supplied HCl solution with standard NaOH soln.**

5. **Procedure**

a. **Preparation of 0.1 (N) NaOH Soln**: Wt out **0.40** gm of NaOH into a 100 ml volumetric flask, then make up to the mark by adding distilled water.

Strength of NaOH = **(**Wt taken in gm / Wt to be taken in gm) X N/10

= (N)

= (N)

b. <u>Standardization of HCl solution with standard NaOH soln .</u>

(1) Take 10 ml of Sodium hydroxide (NaOH) Solution into a Conical flask.

(2) Dilute up to 100 ml distilled water.

(3) 2-3 drops of phenolphthalein indicator, pink color solution appears.

(4) Titrate with supplied HCl solution (**which to be determined**).

(5) Colorless solution appears at the end point.

<u>Table for Vol of Approx 0.1(N) HCl</u>

Number of Observation	**Vol of NaOH soln,V_1(ml)**	**<u>Vol of HCl soln</u>**				**Remarks**
		IBR(ml)	**FBR(ml)**	**Diff(ml)**	**MeanVol, $V_{2\,(ml)}$**	
1						Minimum one observation must be checked and signed
2						
3						
4						

6. **<u>Experimental Data</u>**:

V_1(Vol of NaOH Soln) = ml

S_1(Strg of NaOH Soln) = (N)

V_2 (Vol of HCl soln) = ml

S_2 (Str of HCl Soln, which to be determined) = ?

7. **Calculation:** $S_2 = V_1S_1/V_2$

= ---------- (N) Soln

= (N) Soln

8. **Result:** The Strength of Supplied **HCl** Soln is ----------(N)

9. % of error :

[(Std result ~ Obs result) / Std result] X 100

=

= %

10. Discussion / Viva voce :

ABSTRACT / PRECIS

KEY POINT (EXP:02)

1. **pH Scale**

2. **Different kinds of Indicator for diff.pH**

3. **Principle of Acid-Base Titration**

4. **Mechanism of Phenolphthalein**

Exp No:03

Name of the Exp: **Standardization of Hydrochloric Acid (HCl) solutions with Standard Sodium Carbonate (Na_2CO_3) Solution.** ***(MOLARITY)***

1. **Theory:**

Find the concentration of dilute hydrochloric acid by titration. This process is known as standardizing the hydrochloric acid. The reaction between sodium carbonate and hydrochloric acid takes place in two stages:

$$Na_2CO_3\,(aq) + HCl\,(aq) \rightarrow NaHCO_3\,(aq) + NaCl\,(aq) \quad (1)$$
$$NaHCO_3\,(aq) + HCl\,(aq) \rightarrow NaCl\,(aq) + H_2CO_3\,(CO_2(g) + H_2O\,(l)) \quad (2)$$

2. **Apparatus**: Volumetric flask(100ml),Conical flask(250ml),Burette, Pipette(10ml), Funnel, Wash Bottle, Iron Stand etc.

3. **Chemicals:**
   - **Hydrochloric Acid (HCl)**
   - **Sodium Carbonate (Na_2CO_3),**
   - **Phenolphthalein ($C_{20}H_{14}O_4$).**

4. **Phase**: The exp can be done two phases.

   a. Preparation of standard 0.1(M) Na_2CO_3 soln.
   b. Standardization of Supplied HCl solutions with standard Na_2CO_3 soln.

5. Procedure

a. **Preparation of 0.1 (M) Na_2CO_3 Soln**: Wt out **1.06** gm of Na_2CO_3 into a 100ml volumetric flask, then make up to the mark by adding distilled water (Stirring until the sample dissolves completely).

Strength of Na_2CO_3 = (Wt taken in gm / Wt to be taken in gm) X M/10

= (M)

= (M)

b. Standardization of HCl solution with standard Na_2CO_3 soln.

—

(1) Take 10 ml of Sodium Carbonate (Na_2CO_3) Solution into a Conical flask.

(2) Dilute up to 100 ml distilled water.

(3) 2/3 drops of phenolphthalein indicator, pink color solution appears.

(4) Titrate with supplied HCl solution (**which to be determined**).

(5) Color changes from pink to colorless at the end point.

Table for Vol of HCl

Number of Observation	**Vol of Na_2CO_3 soln,V_1(ml)**	**Vol of HCl soln**				**Remarks**
		IBR(ml)	**FBR(ml)**	**Diff(ml)**	**Mean $V_{2\ (ml)}$**	
1						Minimum one observation must be checked and signed
2						
3						
4						

6. **Experimental Data**:

V_1 (Vol of Na_2CO_3 Soln) = ml

S_1 (Strg of Na_2CO_3 Soln) = (M)

V_2 (Vol of HCl soln) = ml

S_2 (Strg of HCl Soln, which to be determined) = ?

7. **Calculation:**

V_1 X S_1 (**Strg of Na_2CO_3**) / V_2 X S_2 (**Strg of HCl**) = 1 mol / 2 mol

$$S_2 = 2 V_1S_1 / V_2$$

= ---------- (M) Soln

= (M) Soln

10

8. **Result:** The Strength of Supplied **HCl** Soln is ---------- (M)

Over all Reaction

Na_2CO_3 (aq) + 2 HCl (aq) → 2 NaCl (aq) + H_2CO_3 (CO_2(g) + H_2O (l))

9. % of error:

[(Std result ~ Obs result) / Std result] X 100

=

= %

10. Discussion / Viva voce :

ABSTRACT / PRECIS

KEY POINT (EXP:03)

1. **Primary Standard Substance**

2. Secondary Standard Substance

3. Characteristics Primary Standard & Secondary Standard Substance

4. Mechanism of Phenolphthalein

Exp No:04

Name of the Exp: Determination of Calcium (Ca) Content in a Calcium Chloride dihydrate ($CaCl_2$) Solutions with Standard Di-Sodium EthyleneDiamine Tetra AceticAcid (Na_2 - EDTA) Solution. (*Complexometric Titration*). *(MOLARITY).*

1. Theory:

Complexometric titration (sometimes chelatometry) is a form of volumetric analysis in which the formation of a colored complex is used to indicate the end point of a titration. Complexometric titrations are particularly useful for the determination of a mixture of different metal ions in solution. An indicator capable of producing an unambiguous color change is usually used to detect the end-point of the titration.

A covalent chemical bond between two atoms that is produced when one atom shares a pair of ele ctrons with another atom lackingsuch a pair. called coordinate covalent bond.

EDTA, ethylenediaminetetraacetic acid, has four carboxyl groups and two amine groups that can act as electron pair donors, or Lewis bases. The ability of EDTA to potentially donate its six lone pairs of electrons for the formation of coordinate covalent bonds to metal cations makes EDTA a **hexadentate** ligand. However, in practice EDTA is usually only partially ionized, and thus forms fewer than six coordinate covalent bonds with metal cations.

Disodium EDTA is commonly used to standardize aqueous solutions of transition metal cations.

2. Apparatus: Volumetric flask(100ml), Conical flask(250ml),Burette, Pipette(10ml), Funnel, pH meter, wash bottle, Iron stand etc.

3.Chemicals:

- **Calcium Chloride Dihydrate ($CaCl_2.2H_2O$),**
- **Di-Sodium Ethylene Diammine TetraAceticAcid (Na_2-EDTA)($C_{10}H_{14}N_2Na_2O_8.2H_2O$) *Ligand*,**
- **Sodium Hydroxide (NaOH)**
- **Eriochrome blue ($C_{20}H_{13}N_2NaO_5S$) (Calcon).**

4. Phase: The exp can be done two phases.

a. **Prep. of Standard 0.1(M) Di Sodium EDTA (Na_2-EDTA) Solution.**

b. Calcium Chloride Dihydrate ($CaCl_2$,$2H_2O$) as Supplied solution.

5. **Procedure:**

a. Prep. of Standard 0.1(M) Di Sodium EDTA (Na_2-EDTA) solution : Wt out **3.72** gm of Di Sodium EDTA (**Na_2-EDTA**) and transfer into a 100ml Volumetric flask. Then dissolve and make up to the mark with distilled water

Strength of Na_2-EDTA = **(**Wt taken in gm / Wt to be taken in gm) X M/10

= (M)

= (M)

b. Prep of standard 1.25 (M) Sodium hydroxide (NaOH) solution : Wt out **3.0** gm of Sodium hydroxide (**NaOH**) and transfer into a **100 ml Beake**r. Then add **60** ml distilled water and well shake.

6. Content of Calcium in Calcium Chloride dihydrate ($CaCl_2.2H_2O$):

(a) **Preparation of Calcium Chloride dihydrate ($CaCl_2.2H_2O$):** The solution is **Supplied as sample** or **1.47 gm** of Calcium Chloride dihydrate transfer into a 100 ml Volumetric flask. Then dissolve and make up to the mark with distilled water.

"Complexometric titrations involve reaction between metal ions and complexing agent (ligand) to give to soluble complex. (coordinate complex)." "Complexometric titrations involve reaction between metal ions and complexing agent (ligand) to give to soluble complex. (coordinate complex)."

This Titration occurs in Basic media

Complexometric titration are useful for determining a large number of metals.

(b) **Titration Method:**

(1) 10 ml Calcium Chloride Dihydrate ($CaCl_2.2H_2O$) Solution in Conical flask.

(2) Dilute up to 80 ml distilled water.

(3) **10** ml **1.25 (M)** NaOH solution.

(4) 2/3 drop Eriochrome blue (Calcon) Indicator.

(5) Titrate with 0.1(M) Di Sodium EDTA (Na_2-EDTA) solutions.

(6) Light Blue color appears at the end point.

Table for Vol of 0.1 (M) Di-Sodium EDTA (Na_2-EDTA)

Number of Observation	Vol of Calcium Chloride Dihydrate($CaCl_2.H_2O$)	Vol of Di Sodium EDTA (Na_2-EDTA)				Rmks
		IBR (ml)	FBR(ml)	Diff(ml)	Mean	
1					X	Minimum one observation must be checked and signed
2						
3						

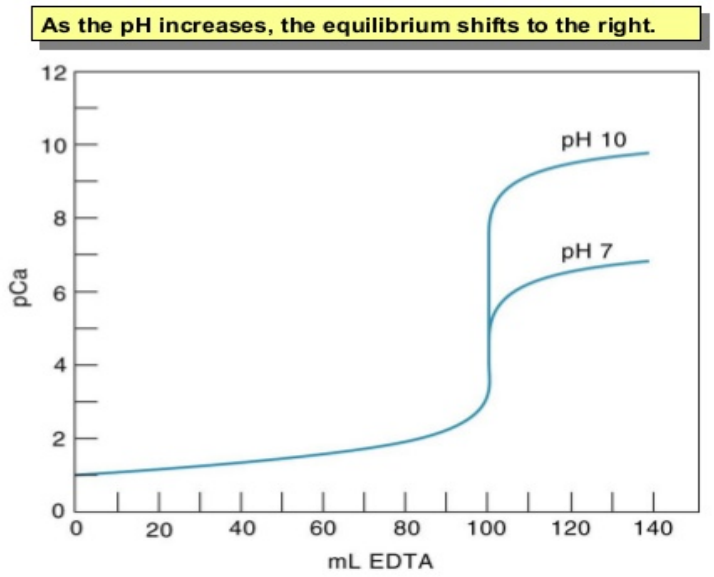
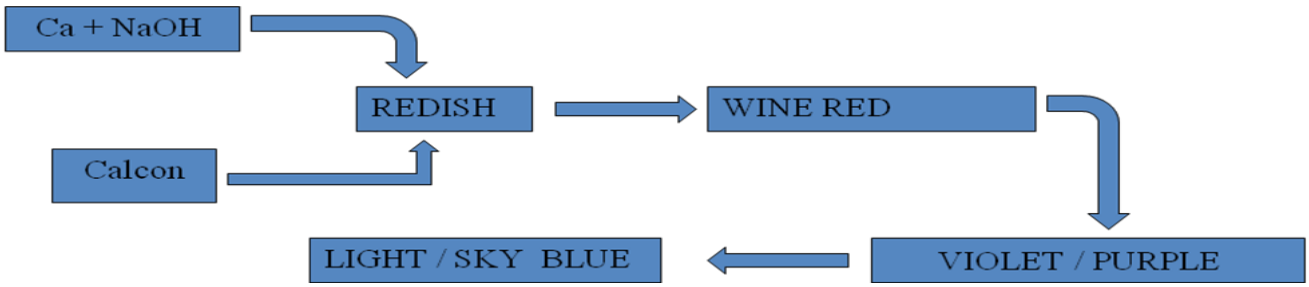


Fig. Titration Curve for 0.1 Calcium VS 0.1 Na_2EDTA

FLOW SHEET



Determination of Calcium (Ca) Content

1000 ml of 1 (M) Na_2-EDTA can consume 40.08 gms of Ca^{2+}

1 ml 1 ,, ,, 40.08 / 1000 gms of Ca^{2+}

X ml **0.1 (M)** ,, ,, **(40.08 x X x 0.1) / 1000** gms of Ca^{2+}

= **P** gms of Ca^{2+}

10 ml Calcium Chloride Dihydrate ($CaCl_2.H_2O$) solution contain **p** gms of Ca^{2+}

1 ml ,, ,, ,, **p**/10 gms of Ca^{2+}

100 ml ,, ,, ,, **p** X 100 /10 gms of Ca^{2+}

= gms of Ca^{2+}

7. Result :

The Amount of **Calcium(Ca)** in 100 ml Supplied Calcium Chloride Dihydrate

($CaCl_2.H_2O$) Solution is ..... gms

8. % of error :

[(Std result ~ Obs result) / Std result] X 100

=

= %

9. Discussion / Viva voice :

ABSTRACT / PRECIS

KEY POINT (EXP: 04)

1. **Co-ordinate bond**

2. **Formation of Co-ordinate bond**

3. **Ligand / Chelate**

4. **Structure of Na_2-EDTA**

5. **Zwitterions**

6. **Complexones**

Exp No:05

Nameof the Exp: **Standardization of Sodium Thiosulphate Penta hydrate ($Na_2S_2O_3.5H_2O$) solution with standard Potassium Dichromate ($K_2Cr_2O_7$) solution.**

1. **Theory:**

In this experiment the strength of Sodium Thiosulphate solution is determined with the help of a standard Potassium Dichromate solution. This is an oxidation and reduction reaction, as well as "Iodometric reaction". Titration involving with iodine or dealing with iodine liberated in chemical reaction is called Iodimetric and Iodometric titration respectively. This reaction is iodometric because iodine is got from KI. The reactions of this experiment are:

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

$$(6\ I^- + 14\ H^+ + Cr_2O_7^{2-} + 6e^- = 2\ Cr^{3+} + 3\ I_2 + 7\ H_2O)$$

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$$

$$(S_2O_3^{2-} + e^- = S_4O_6^{2-} + I^-)$$

Here $K_2Cr_2O_7$ is an oxidizing agent and I- is a reducing agent. Again in the second reaction I2 is an oxidizing agent and S2O3 -- is a reducing agent. In the 2nd Step of the reaction a specific indicator is used that is "Starch"- which has a significant characteristic on iodine. Starch+I2=Starch-Iodine (blue colour) Starch-Iodine+6S2O3 --=Starch +6I- + 3S4 O6 — If to a solution containing a little iodine, some starch solution is added and Na2S2O3 is run in from the burette, the blue color of the starch-iodine complex will disappear from the solution as soon as all the iodine has been reduced to iodide ion.

2. Apparatus: Volumetric flask(100ml),Conical flask(100ml),Burette, Pipette(10ml), Funnel, Wash Bottle, Iron Stand etc.

3. **Chemicals:**

- **Potassium dichromate ($K_2Cr_2O_7$)**
- **Sodium Thiosulphate Pentahydrate ($Na_2S_2O_3.5H_2O$)**
- **Potassium Iodide (KI)**
- **Sulfuric Acid (H_2SO_4)**
- **Sodium bi-carbonate ($NaHCO_3$)**
- **Starch ($(C_6(H_2O)_5)_n$**

4. Phase: The expt. can be done two phases

a. **Prep of standard 0.1(N) Potassium dichromate ($K_2Cr_2O_7$) solution.**

b. Standardization of Sodium Thiosulphate ($Na_2S_2O_3.5H_2O$) solution with standard ($K_2Cr_2O_7$) solution

.

5. **Procedure:**

a. <u>Prep of standard 0.1(N) Potassium dichromatre ($K_2Cr_2O_7$) solution</u> : Wt out **0.49** gm of $K_2Cr_2O_7$ and transfer into a 100ml Volumetric flask. Then dissolve and make up to the mark with distilled water.

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Strength of $K_2Cr_2O_7$ = **(**Wt taken in gm / Wt to be taken in gm) X N/10

= (N)

= (N)

b. <u>Standardization of approx 0.1(N) Sodium Thiosulphate ($Na_2S_2O_3.5H_2O$) with standard ($K_2Cr_2O_7$) solution.</u>

(1) Take 10ml 12% potassium Iodide (KI) solution into a conical flask.

(2) Add about 80 ml distilled water and about 2 gm sodium-bi-carbonate ($NaHCO_3$).

(3) Add 10 ml of 3N sulphuric acid (H_2SO_4).

(4) Add 10 ml of standard potassium dichromate ($K_2Cr_2O_7$) solution, shake, cover with watch glass and keep in dark place for 10minutes.

(5) Reddish colour appears, rinse the watch glass sharply and dilute up to 100ml with distilled water (if need).

(6) Titrate the liberated iodine with sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution.

(7) Light yellow color appears.

(8) Add starch indicator solution and dark colour appears.

(9) Continue titration with sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution.

(10) Light green or light blue colour appears at the end point.

<u>FLOW SHEET</u> <u>(STANDARDIZATION OF SODIUM THIOSULPHATE)</u>

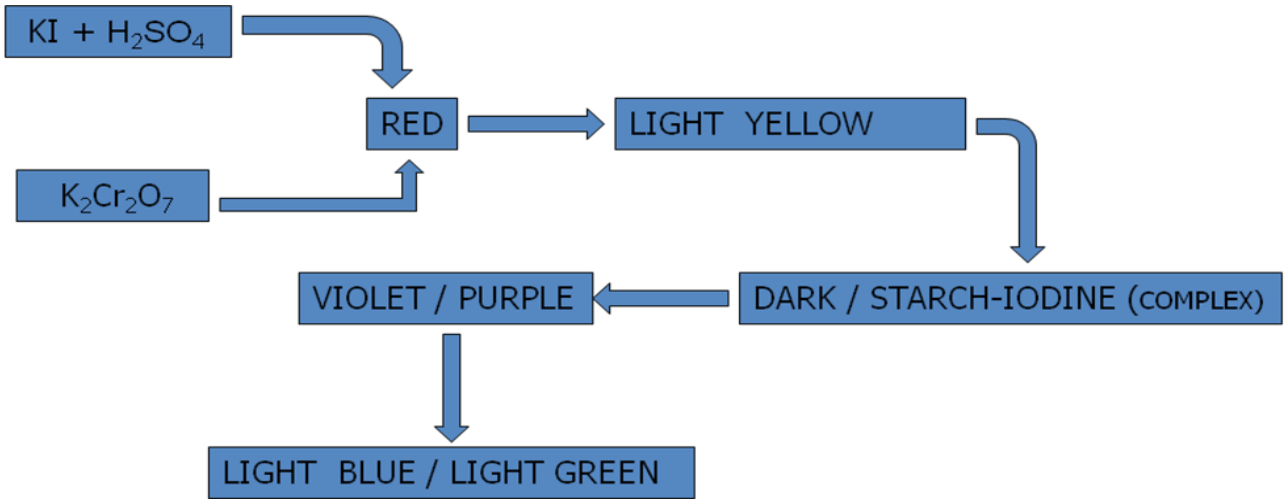


Table for vol of $Na_2S_2O_3.5H_2O$

Number of Observation	Vol of $K_2Cr_2O_7$ soln, V_1(ml)	Vol of $Na_2S_2O_3.5H_2O$				Remarks
		IBR(ml)	FBR(ml)	Diff (ml)	Mean	
1						Minimum one observation must be checked and signed
2						
3						
4						

6. **Experimental Data**:

V_1(Vol of $K_2Cr_2O_7$ Soln) = ml

S_1(Str of $K_2Cr_2O_7$ Soln) = (N)

V_2 (Vol of $Na_2S_2O_3.5H_2O$ soln) = ml

S_2 (Str of $Na_2S_2O_3.5H_2O$ Soln, which to be determined) = ?

7. **Calculation:** $S_2 = V_1S_1/V_2$

= ---------- (N) Soln

= (N) Soln

8. **Result:** The Strength of Solution **$Na_2S_2O_3.5H_2O$** Soln is ---------- (N)

9. % of error :

[(Std result ~ Obs result) / Std result] X 100

=

= %

10. Discussion / Viva voce :

KEY POINT (EXP:05)

1. **Oxidation Number**

2. **Equivalent weight of $K_2Cr_2O_7$**

3. **Iodometric Titration**

4. **Reaction With Balance**

5. **Half Oxidation and Reduction Titration**

6. **Why keep dark for Iodometric Titration**

7. **Why use $NaHCO_3$ for Iodometric Titration**

8. **Flow sheet of this Iodometric Titration**

Exp No:06

Name of the Exp: **Estimation of Copper (Cu) Content in a Copper Sulphate penta hydrate ($CuSO_4.5H_2O$) Solution by Iodometric Method with Standard Sodium Thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) Solution.**

1. **Theory :**

In this experiment, the amount of copper in a copper salt solution is determined. The reaction that occurs here is oxidation and reduction reaction as well as iodometric reaction. Titration involving with iodine or dealing with iodine liberated in chemical reaction is called Iodimetric and Iodometric titration respectively. This reaction is iodometric because iodine is got from KI. The reaction takes place in two steps:

1. $Cu^{2+} + KI + H^+ = I_2 + \ldots\ldots$
2. $Na_2S_2O_3 + I_2 = Na_2S_4O_6 + \ldots\ldots$

In the 2nd Step of the reaction a specific indicator is used that is "Starch"- which has a significant characteristic on iodine. Here, for the first part of the experiment ,the basis of the volumetric measurement is that one equivalent weight of an oxidizing agent will completely react with one equivalent weight of a reducing agent. To perform the second part of the experiment, we can follow the process discussed below:-

1000 ml (N) $Na_2S_2O_3$ solution ≡ 1000 ml (N) iodine solution ≡ 1 equivalent weight of iodine ≡ 1 atomic weight of iodine

Since one atom of iodine is set free for each atom of copper 1000 ml (N) $Na_2S_2O_3$ solution should be equivalent to one gram molecular weight that is 63.54 gram of copper. Therefore,

1 ml of (N) Na2S2O3 ≡ 0.06354 gram of copper

So, the weight of copper in the supplied solution may then be easily calculated using the above relation.

2.**Apparatus:** Volumetric flask(100ml),Conical flask(100ml),Burette, Pipette(10ml), Funnel, Wash Bottle, Iron Stand etc.

3. **Chemicals:**

- **Copper Sulphate Pentahydrate ($CuSO_4.5H_2O$)**

- **Potassium dichromate ($K_2Cr_2O_7$),**
- **Sodium Thiosulphate Pentahydrate ($Na_2S_2O_3.5H_2O$)**
- **Potassium Iodide (KI),**
- **Sodium bi-carbonate ($NaHCO_3$)**
- **Sulfuric Acid (H_2SO_4)**
- **Ammonium Thiocyanate (NH_4SCN)**
- **Starch $(C_6(H_2O)_5)_n$**

4. **Phase**: The expt. can be done two phases

Phase: 01

a. **Prep of standard 0.1(N) Potassium dichromatre ($K_2Cr_2O_7$) solution.**

b. Standardization of approx 0.1(N) Sodium Thiosulphate ($Na_2S_2O_3$) with standard ($K_2Cr_2O_7$) solution.

Phase: 02

Estimation of Copper Content in a Copper Sulphate ($CuSO_4.5H_2O$) Solution by Iodometric method.

5. **Procedure:**

a. Prep of standard 0.1(N) Potassium dichromatre ($K_2Cr_2O_7$) solution a.Wt out **0.49** gm of $K_2Cr_2O_7$ and it transfer into a 100ml Volumetric flask. Then dissolve and make up to the mark.

Strength of $K_2Cr_2O_7$ = **(**Wt taken in gm / Wt to be taken in gm) X N/10

= (N)

= (N)

b. Standardization of approx 0.1(N) Sodium Thiosulphate ($Na_2S_2O_3.5H_2O$) with standard($K_2Cr_2O_7$) solution.

(1) Take 10 ml 12% potassium iodide (KI) solution into a conical flask.

(2) Add about 80ml distilled water and about 2 gm sodium-bi-carbonate ($NaHCO_3$)

(3) 10ml (3N) sulphuric acid (H_2SO_4).

(4) 10 ml potassium dichromate ($K_2Cr_2O_7$) solution shakes, cover with watch glass and keep in dark place for 10minutes.

(5) Reddish colour appears, rinse the watch glass sharply and dilute up to 100ml with distilled water (if need).

(6) Titrate with sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution, light yellow colour appears.

(7) Add starch indicator solution, dark colour appears.

(8) Continue titrate with sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution.

(9) Light green or light blue colour appears at the end point.

Table for vol of $Na_2S_2O_3.5H_2O$

Number of Observation	Vol of $K_2Cr_2O_7$ soln,V_1(ml)	Vol of $Na_2S_2O_3.5H_2O$				Remarks
		IBR(ml)	FBR(ml)	Diff (ml)	Mean	
1						Minimum one observation must be checked and signed
2						
3						
4						

6. **Experimental Data**:

V_1(Vol of $K_2Cr_2O_7$ Soln) = ml

S_1(Str of $K_2Cr_2O_7$ Soln) = (N)

V_2 (Vol of $Na_2S_2O_3.5H_2O$ soln) = ml

S_2 (Str of $Na_2S_2O_3.5H_2O$ Soln, which to be determined) = ?

7. **Calculation:** $S_2 = V_1S_1/V_2$

= ---------- (N) Soln

= (N) Soln

8. The Strength of $Na_2S_2O_3.5H_2O$ Soln is ----------**Y** (N)

9. **Content of Copper(Cu) in $CuSO_4.5H_2O$ Solution :**

(c) **Preparation of 0.1 (N) $CuSO_4.5H_2O$:** Wt out 2.497 gm of $CuSO_4.5H_2O$ in 100 ml Volumetric flax, then add make up to the mark by distilled water ***Or Supplied Solution.***

(d) **Titration (Iodometric Method):**

(1) 10 ml Copper Sulphate (**$CuSO_4.5H_2O$**) solution in Conical flax.

(2) Dilute up to 80 ml distilled water.

(3) Add 2 gm of sodium-bi-carbonate ($NaHCO_3$)

(4) 10ml (3N) sulphuric acid (H_2SO_4)

(5) Take 10ml 12% potassium iodide (KI) solution into a conical flask and cover with (watch glass and keep in dark place for 10minutes.

(6) Reddish colour appears, rinse the watch glass sharply and dilute up to 100 ml with distilled water.

(7) Titrate with 0.1 (N) sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solutions, light yellow colour appears.

(8) Add starch indicator solution, dark colour appears.

(9) 5 ml 10 % Ammonium Thiocyanate (NH_4CNS) Solution.

(9) Continue titrate with sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution.

(10) White or Off-White colour appears at the end point.

Table for vol of $Na_2S_2O_3.5H_2O$

Number of	**Vol of**	**Vol of $Na_2S_2O_3.5H_2O$**	**Remarks**

Observation	$CuSO_4.5H_2O$ soln	IBR(ml)	FBR(ml)	Diff (ml)	Mean	
1					**X**	Minimum one observation must be checked and signed
2						
3						
4						

As it often happens in the case of multistage procedures, equation that describes whole process is only an oversimplification of the real procedure. What is important is that it preserves the stoichiometry of the process, so it can be used for the calculation of titration results:

$$\mathbf{2Cu^{2+} + 2S_2O_3^{2-} + 2I^- \rightarrow 2CuI(s) + S_4O_6^{2-}}$$

10. <u>Calculation for the Estimation of Copper(Cu) content in supplied $CuSO_4.5H_2O$ solution :</u>

1000 ml 1 (N) $Na_2S_2O_3$ Solution can consume 63.54 gm of Cu^{2+}

1 ml 1 (N) ,, ,, 63.54/1000 gm of Cu^{2+}

X ml **Y** (N) ,, ,, **(63.54 x X x Y) / 1000** gm of Cu^{2+}

= p gm of Cu^{2+}.

*[Here **X** and **Y** are the Vol and Strength of $Na_2S_2O_3$]*

10 ml $CuSO_4$ Contains **p gm** of Cu^{2+}

1 ml ,, ,, **p** / 10 **gm** of Cu^{2+}

100 ml ,, ,, **p** X 100 / 10 gm of Cu^{2+}

= gm of Cu^{2+}

12. **<u>Result:</u>** The Amount of **Copper (Cu)** in 100 ml Supplied **$CuSO_4$** Solution is

..... gm

13. % of <u>error</u>:

[(Std result ~ Obs result) / Std result] X 100

=

= %

14. Discussion / Viva voce :

KEY POINT (EXP:06)

1. **Iodometric Titration**

2. **Iodimetric Titration**

3. **Difference between Iodometric & Iodimetric Titration**

4. **Reaction With Balance**

5. **Half Oxidation and Reduction Titration**

6. **Why use Starch Middle of the reaction**

7. **Why use $NaHCO_3$ for Iodometric Titration**

8. **Why use NH_4SCN for Iodometric Titration**

9. Flow sheet of this Iodometric Titration

Exp No:07

Name of the Exp: Standardization of Potassium Permanganate ($KMnO_4$) Soln with standard Oxalic Acid ($C_2H_2O_4.2H_2O$) Solution. ***REDOX TITRATION.***

1 . Theory:

In this experiment, potassium permanganate solution is standardized with the help of standard Oxalic Acid solution. The reaction occurs in temperature .The reaction that occurs here is oxidation and reduction reaction. An oxidation-reduction reaction (red ox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers.

Oxidation is the half reaction in which there is loss of electrons by a species (or increase of oxidation number of an atom).

Reduction is the half reaction in which there is a gain of electrons by a species (or decrease of oxidation number of an atom).
Reaction involved in this reaction is:

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

$$2\,KMnO_4 + 5\,H_2C_2O_4 + 8\,H_2SO_4 \rightarrow K_2SO_4 + 2\,MnSO_4 + 5\,H_2SO_4 + 10\,CO_2 + 8\,H_2O$$

2. Apparatus: Volumetric flask(100ml),Conical flask(250ml),Burette, Pipette(10ml),Funnel, Wash Bottle, Iron Stand etc.

3. **Chemicals:**

- **Oxalic Acid Dihydrate ($C_2H_2O_4.2H_2O$),**
- **Potassium permanganate ($KMnO_4$),**
- **Sulfuric Acid (H_2SO_4).**

4. Phase: The expt. can be done two phases

a. Preparation of standard 0.1(N) $C_2H_2O_4.2H_2O$ (oxalic acid) Soln.
b. Standardization of approx 0.1(N) Potassium Permanganate ($KMnO_4$)

5. Procedure :

a. **<u>Preparation of Standard 0.1(N) $C_2H_2O_4.2H_2O$ Soln</u>:** Wt out **0.63** gm Wt out **$C_2H_2O_4.2H_2O$** into a 100ml Volumetric flask then add make up to the mark by adding distilled water.

Strength of $C_2H_2O_4$ = (Wt taken in gm / Wt to be taken in gm) X 0.1(N)

= **(N)**

= **(N)**

b. Standardization of approx 0.1 (N) Potassium Permanganate ($KMnO_4$) With standard oxalic acid ($C_2H_2O_4.2H_2O$):

(1) 10 ml Oxalic acid di-hydrate ($C_2H_2O_4.2H_2O$) solution.

(2) Dilute up to 100 ml distilled water.

(3) 10 ml (3N) sulphuric acid (H_2SO_4) solution.

(4) Heat the solution upto $(60\sim70)^0$C temperature (use thermometer).

(5) Slowly titrate the solution with Potassium permanganate ($KMnO_4$) solution.

(6) Color disappears and at the end point pink colour appears.

<u>Table for Vol of $KMnO_4$</u>

Number of Observation	**Vol of $C_2H_2O_4$ soln**	**Vol of $KMnO_4$ soln**				**Remarks**
		IBR(ml)	**FBR(ml)**	**Diff(ml)**	**MeanVol, $V_{2\,(ml)}$**	
1						Minimum one observation must be checked and signed
2						
3						
4						

6. **<u>Experimental Data</u>**:

V_1(Vol of $C_2H_2O_{4\ Soln}$) = ml

S_1(Str Vol of $C_2H_2O_{4\ Soln}$) = (N)

V_2(Vol of $KMnO_{4\ Soln}$) = ml

S_2(Str of $KMnO_{4\ Soln}$) = ?

7. **Calculation**: $S_2 = V_1S_1/V_2$

= ---------- (N) Soln

= (N)
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8. **Result**: The Strength of **$KMnO_4$** Soln is ----------(N)

9. % of error :

[(Std result ~ Obs result) / Std result] X 100

=

= %

10. Discussion / Viva voce :uj

KEY POINT (EXP:07)

1. **Electronic concept of Oxidation and Reduction**

2. **Oxidizing agent and Reducing agent**

3. **Auto catalyst**

4. **Why heat is needed for the reaction(Influence of Tempr)**

5. **Half Oxidation and Reduction Titration**

6. **Why use H_2SO_4 for this titration(why not HCl or HNO_3)**

7. **Activation Energy.**

8. **Arrhenius Equation .**

Exp No :08

Name of the Exp: Determination of Ferrous (Iron) (Fe) Content in a Solution with Standard Potassium Permanganate($KMnO_4$) Solution. REDOX *Titration*.

1. Theory:

Ammonium iron(II) sulfate, or **Mohr's Salt (named after German scientist Karl Friedrich Mohr)** , is the inorganic compound with the formula $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$. Containing two different cations, Fe^{2+} and NH_4^+, it is classified as a double salt of ferrous sulfateand ammonium sulfate. It is a common laboratory reagent. Like the other ferrous sulfate salts, ferrous ammonium sulfate dissolves in water to give the aquo complex $[Fe(H_2O)_6]^{2+}$, which has octahedral molecular geometry.

This compound is a member of a group of double sulfates called Schönites or Tutton's salts. which form monoclinic crystals and have formula $M_2N(SO_4)_2.6H_2O$. This group can mix metals and crystallise on other schönite crystals.

The amount of ferrous ion (Fe^{2+}) in the solution can be determined by a redox titration with standard potassium permanganate ($KMnO_4$).

The ionic equations of ferrous sulphate with acidified potassium permanganate are given below-

$$[\ Fe^{2+}\ \text{(Oxidation)} \rightarrow\ Fe^{3+} + e^-\ ]\ X\ 5$$

$$MnO^{4-} + 8H^+ + 5\ e^-\ \text{(Reduction)} \rightarrow Mn^{2+} + 4\ H_2O$$

2. Apparatus: Volumetric flask(100ml), Conical flask(250ml),Burette, Pipette(10ml), Funnel, Thermometer, Tripod stand, Buner, wash bottle, Iron stand etc.

3. Chemicals:

- **Mohr`s Salt [$FeSO_4.(NH_4)_2SO_4.6H_2O$]**
- **Oxalic Acid Dihydrate ($C_2H_2O_4$, $2H_2O$),**
- **Potassium permanganate ($KMnO_4$),**
- **Sulphuric Acid (H_2SO_4)**

4. Phase: The exp can be two phases.

a. Preparation of standard oxalic acid solution.

b. Standardization of Potassium permanganate solution with standard oxalic acid solution.

5. Procedure :

a. **Preparation of 0.1(N) oxalic acid solution**:Wt out of 0.63gm ($C_2H_2O_4$) and transfer it into a 100ml volumetric flask then dissolve and make up to the mark with distilled water.

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Str. of $C_2H_2O_4$. $2H_2O$ solution = (Wt taken in gm / Wt to be taken) X 0.1(N)

= (N)

= (N)

b. Standardization of Potassium permanganate ($KMnO_4$) solution:

(1) Take10 ml oxalic acid solution in Conical flax

(2) Dilute up to 100 ml distilled water.

(3) 10 ml (3N) sulphuric acid (H_2SO_4) solution.

(4) Heat the solution up to 60^0~70^0 C temperation (use up thermometer)

(5) Slowly titrate the solution with potassium permanganate ($KMnO_4$) solution.

(6) Colour disappears and at the end point pink colour appears.

Table for vol of $KMnO_4$

Number of Observation	Vol of $C_2H_2O_2.H_2O$	Vol of $KMnO_4$				Remarks
		IBR(ml)	FBR(ml)	Diff (ml)	Mean	
1						
2						
3						

6. Experimental Data:

V_1 (Vol of $C_2H_2O_4$) = ml

S_1 (Str of $C_2H_2O_4$) = (N)

V_2 (Vol of $KMnO_4$) = ml

S_2 (Str of $KMnO_4$) = (N)

7. **Calculation**: $S_2 = V_1S_1/V_2$

= ---------- (N) Soln

=

Strength of 0.1 (N) Potassium permanganate ($KMnO_4$) is .....Y.....(N)

8. Content of Ferrous (Iron) in Mohr`s Salts[$FeSO_4.(NH_4)_2SO_4.6H_2O$] :

(e) **Preparation of 0.1 (N) Mohr Salts [$FeSO_4.(NH_4)_2SO_4.6H_2O$] : The solution is supplied as sample.**

(f) **Titration Method:**

(1) 10 ml Mohr`s Salt [$FeSO_4.(NH_4)_2SO_4.6H_2O$] solution in Conical flax.

(2) Dilute up to 100 ml distilled water.

(3) 10 ml (3N) sulphuric acid (H_2SO_4)

(4) Slowly Titrate with "**Y**" (N) Potassium permanganate ($KMnO_4$**)** solutions.

(5) Color disappears and at the end point pink colour appears.

Table for vol of "Y"(N) Potassium permanganate ($KMnO_4$)

Number of Observation	Vol of Mohr Salt [$FeSO_4.(NH_4)_2SO_4.6H_2O$]	Vol of Potassium permanganate ($KMnO_4$)				Remarks
		IBR(ml)	FBR(ml)	Diff(ml)	Mean	
1					X	

2						
3						

Determination of Ferrous (Iron)Content

1000 ml of 1 (N) $KMnO_4$ Can Consume 55.84 gms of Fe^{2+}

1 ml 1 ,, ,, 55.84 / 1000 gms of Fe^{2+}
X ml **Y** ,, ,, **(55.84 x X x Y) / 1000** gms of Fe^{2+}
= **p** gms of Fe^{2+}

*[Here **X** and **Y** are the Vol and Strength of $KMnO_4$]*

10 ml Mohr`s Salt $[FeSO_4.(NH_4)_2SO_4.6H_2O]$ solution contain **p** gms of Fe^{2+}

1 ml ,, ,, ,, **p**/10 gms of Fe^{2+}

100 ml ,, ,, ,, **p** X 100 /10 gms of Fe^{2+}

= gms of Fe^{2+}

9. Result :

The Amount of **Ferrous (Iron)** in 100 ml Supplied Mohr Salt **$[FeSO_4.(NH_4)_2SO_4.6H_2O]$**

Solution is ..... gms

10. % of error :

[(Std result ~ Obs result) / Std result] X 100

=

= %

10. Discussion / Viva voice :

KEY POINT (EXP:08)

1. **Molecular formulae and Analysis of Mohr's Salt**

2. **Oxidizing agent and Reducing agent**

3. **Example (Three) of Oxidizing agent and Reducing agent**

3. **Self Indicator.**

4. **Advantage and disadvantage of $KMnO_4$**

5. **Half Oxidation and Reduction Titration**