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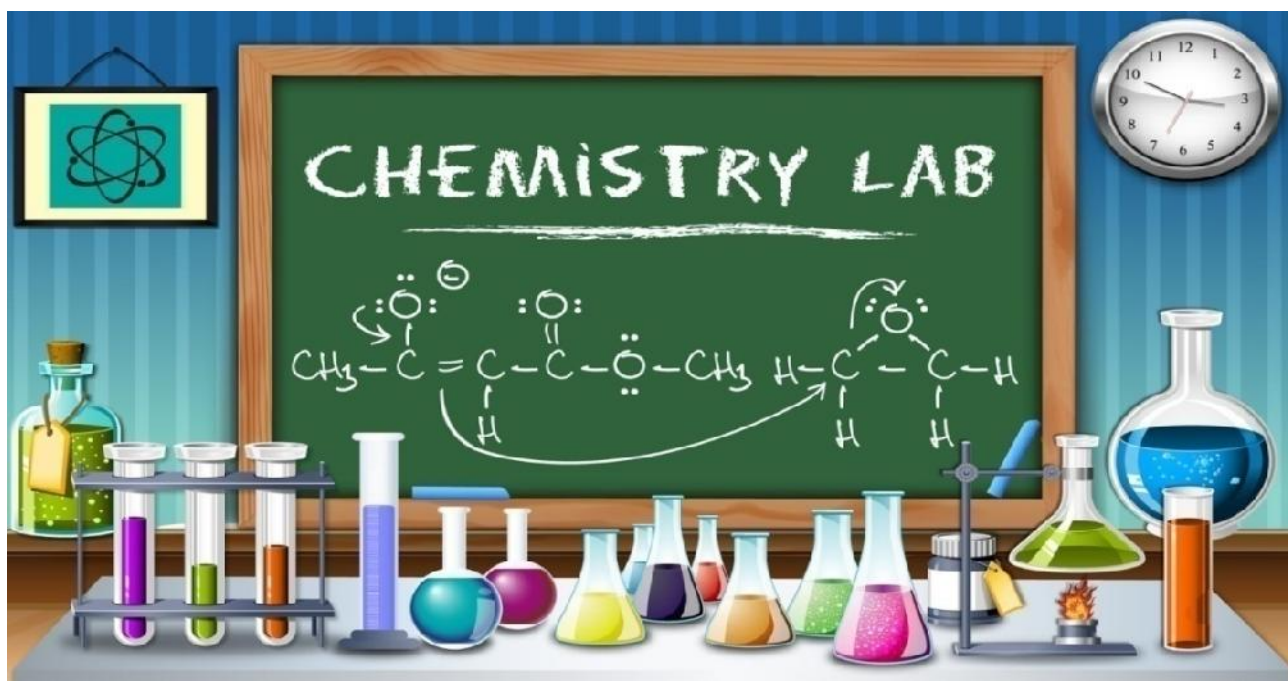
LAB MANUAL

Regulation   : 2013

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GE6262-CHEMISTRY LABORATORY - II

ANNA UNIVERSITY - CHENNAI

GE6262 - CHEMISTRY LABORATORY II

LIST OF EXPERIMENTS

(Any 5 Experiments)

1. Determination of alkalinity in water sample

2. Determination of total, temporary & permanent hardness of water by EDTA method

3. Estimation of copper content of the given solution by EDTA method

4. Estimation of iron content of the given solution using potentiometer

5. Conductometric precipitation titration using $BaCl_2$ and Na_2SO_4

6. Corrosion experiment – weight loss method

7. Determination of Calcium Oxalate (CaO) in Cement

INDEX

Ex. No.	Date	Name of Experiment	Signature of the Staff	Remarks
1.		**Determination of Alkalinity in Water Sample**		
2.		**Determination of Total, Permanent, Temporary Hardness of Water Sample by EDTA Method**		
3.		**Estimation of Copper Content of the Given solution by EDTA Method using Fast Sulphone Black-F Indicator**		
4.		**Estimation of Ferrous ion by Potentiometric Titration**		
5.		**Conductometric Precipitation Titration using $BaCl_2$ and Na_2SO_4**		
6.		**Determination of Rate of Corrosion by Weight Loss Method**		
7.		**Determination of Calcium Oxalate (CaO) in Cement**		

EX. NO:1
**DATE:**

DETERMINATION OF ALKALINITY IN WATER SAMPLE

AIM

To determine the typeand amount of alkalinity present in the given water sample. A standard solution of sodium hydroxide of strength …….N and a link solution of hydrochloric acid are provided.

PRINCIPLE

Alkalinity is caused by the presence of hydroxide, carbonate and bicarbonate. There are five alkalinity conditions possible in a given sample of water, hydroxide only, carbonate only, bicarbonate only, combination of carbonate and hydroxide or carbonate and bicarbonate. The various alkalinities can be determined by titrating with a standard acid using phenolphthalein and methyl orange indicators successively.

1. Phenolphthalein end point

When alkaline water is titrated with acid using phenolphthalein indicator, hydroxide alkalinity is completely neutralized and carbonate alkalinity is partially neutralized.

$$OH^- + H^+ \longrightarrow H_2O$$

$$CO_3^{2-} + H^+ \longrightarrow HCO_3^-$$
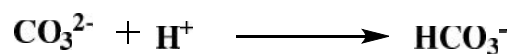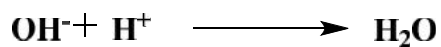
2. Methyl orange end point

After thephenolphthalein end point, methyl orange indicator is added and titrated with acid. Bicarbonate neutralization occurs.

$$HCO_3^- + H^+ \longrightarrow CO_2 + H_2O$$
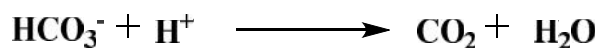
From the two titre values the different alkalinities are calculated.

When,

$P = M,$ hydroxide alkalinity

$$2P = M, \text{ carbonate alkalinity}$$

$$P = 0, \quad \text{bicarbonate alkalinity}$$

$$P < \frac{1}{2}M, \text{ carbonate and bicarbonate alkalinity}$$

$$CO_3{}^{2-} = 2P, HCO_3{}^{-} = M - 2P$$

PROCEDURE

TITRATION – I
Standardisation of HCl

The burette is washed well with water and rinsed with the given hydrochloric acid solution. It is then filled with the same upto zero mark. 20 ml of the standard sodium hydroxide solution is pipetted out in a clean conical flask. 2 – 3 drops of phenolphthalein indicator is added, the colour turn to pink colour. It is then titrated against the hydrochloric acid taken in the burette. The end point is disappearance of pink colour. The titration is repeated to get concordant values. From the titre values, the normality of HCl is calculated.

TITRATION – II
With Phenolphthalein Indicator

20 ml of the water sample is pipetted out in a clean conical flask. A drop of phenolphthalein indicator is added. Pink colour is observed. This solution is titrated against the standard HCl, already taken in the burette, until pink colour is disappeared. The end point is noted. This titre value correspondstophenolphthalein end point (P).

TITRATION – III
With Methyl orange Indicator

Few drops of methyl orange indicator is added to the same solution after the phenolphthalein end point. The titration is continued until the solution becomes red orange. The total titre value is noted. This titre value corresponds to methyl orange end point (M). The titration is repeated for concordant values.

From the titre values the amount of each alkalinity present in given water sample is calculated.

TABLE – I
Titre values and different alkalinities

S. No	Result of titration of [P] and [M]	Hardness causing ions		
		OH^-	CO_3^{2-}	HCO_3^-
1.	[P] = 0	0	0	[M]
2.	[P] = [M]	[P] or [M]	0	0
3.	$P = \dfrac{1}{2}M$	0	2[P] or [M]	0
4.	$P\ 0\dfrac{1}{2}M$	2[P] – [M]	2[M] – [P]	0
5.	$P\ M\dfrac{1}{2}M$	0	2[P]	[M] – 2[P]

TITRATION – I

HCl Vs Std. NaOH

Sl. No	Volume of NaOH ml	Burette Readings		Concordant Volume of HCl ml	Indicator
		Initial ml	Final ml		
1.	20	0			Phenolphthalein
2.	20	0			

Volume of **NaOH** V_1 = **20 ml**

Normality of **NaOH** N_1 = ………………N

Volume of **HCl** V_2 = ………………ml

Normality of **NaOH** N_2 =?

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times ....N}{...............}$$

TITRATION II & III

Water Sample Vs. Std. HCl

Sl. No	Volume of water sample ml	Burette Readings		Concordant Volume of HCl	
		Phenolphthalein end point [P] Ml	Methyl Orange end point [M] ml	P ml	M ml
1.	20	0			
2.	20	0			

CALCULATIONS:

If the data satisfies the condition $P > \frac{1}{2}M$

     i.) Volume of `HCl` required for [`OH` ⁻] alkalinity **= 2 * P – M**

$$= 2 \text{ x } .... - ....$$

$$= ....\textbf{ml}$$

     ii.) Volume of `HCl` required for [`CO`$_3^{2-}$] alkalinity = **2 * M – 2 * P**

$$= 2 \text{ x } .... - 2 \text{ x } ....$$

1. Calculation of OH⁻ alkalinity

     Volume of **HCl** V_1 = ...**ml**

     Strength of **HCl** N_1 = ...**N**

     Volume of water sample V_2 = **20 ml**

Strength of water sample $N_2 = $?

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{\text{..........ml} \times \text{.....................N}}{20} = \text{...........N}$$

Amount of OH^- content present in 1 litre of water sample, in terms of $CaCO_3$ equivalent   =

Strength of water sample x eqv.Wt.of$CaCO_3$

ie.,OH^- alkalinity interms of $CaCO_3$ equivalent = **..................N x 50 x 1000ppm**

Alkalinity due to OH^-ions = ...ppm

$$P > \frac{1}{2}M, \quad \text{carbonate and hydroxide alkalinity}$$

$$CO_3^{2-} = 2(M–P), \quad OH^- = 2P – M$$

Alkalinity values are expressed in terms of milligrams per litre as calcium carbonate.

2. Calculation of CO_3^{2-} alkalinity

Volume of HCl $V_1 = $ ....ml

Strength of HCl $N_1 = $ ....N

Volume of water sample $V_2 = 20$ ml

Strength of water sample $N_2 = $?

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{\dots\dots ml \times \dots\dots\dots N}{20} = \dots\dots\dots\dots N$$
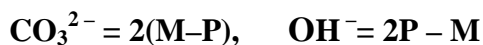
Amount of CO_3^{2-} content present in 1 litre of water sample, in terms of $CaCO_3$ equivalent =

Strength of water sample x eqv. Wt.of $CaCO_3$

ie.,CO_3^{2-} alkalinity in terms of $CaCO_3$ equivalent = **………………..N x 50 x 1000ppm**

Alkalinity due to CO_3^{2-} ions = ………………..ppm

RESULT

Water sample contains the following alkalinity

i.) Hydroxide alkalinity (OH^-)   = ………………….**ppm**

ii.) Carbonate alkalinity (CO_3^{2-})   = ………………….**ppm**

iii.) Total alkalinity ($OH^- + CO_3^{2-}$)   = ………………….**ppm**

EX. NO: 2
DATE:

DETERMINATION OF TOTAL, PERMANENT, TEMPORARY HARDNESS OF WATER SAMPLE BY EDTA METHOD
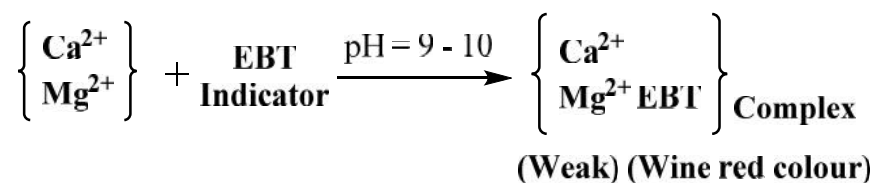
AIM

To determine the total, permanent, temporary hardness in the given water sample of hard water by EDTA method. A standard hardwater of ……………N and an EDTA solution are provided.
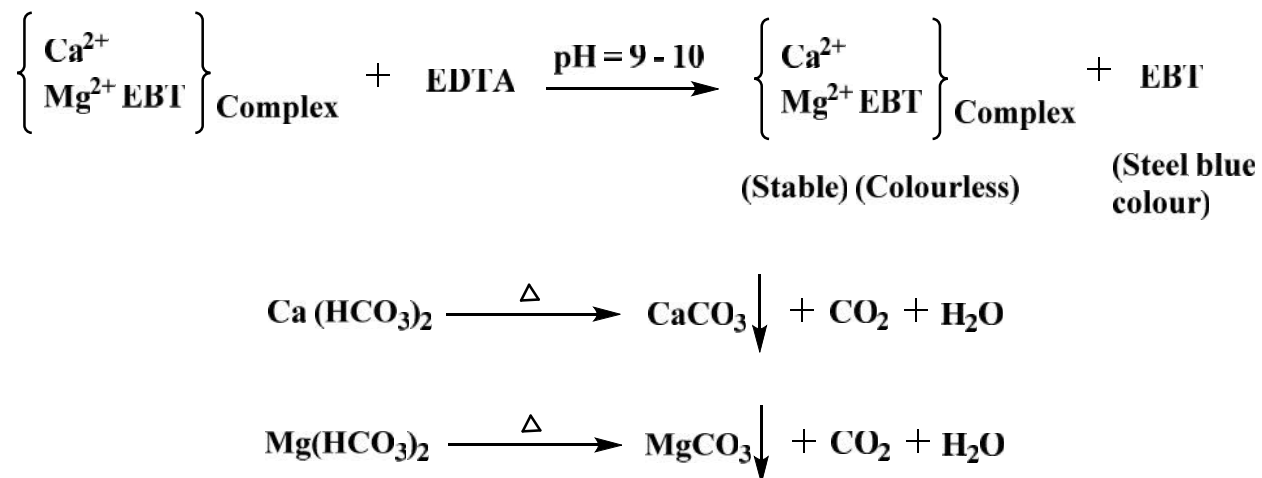
PRINCIPLE

Disodium salt of Ethylene Diamine Tetra Acetic acid (EDTA) is a well-known complexing agent. Its structure is shown in the figure 1.

Disodium salt of EDTA is used to estimate the various hardness of the given hard water containing Ca^{2+} and Mg^{2+} ions. When EDTA is added to hard water, it reacts with calcium and magnesium ions present in hard water to form stable EDTA metal complexes. From the volume of EDTA consumed the hardness can be calculated. Eriochrome Black – T is used as an indicator. The indicator forms a weak complex with the metal ions present in the hard water and gives wine red colour.

$$\begin{Bmatrix} Ca^{2+} \\ Mg^{2+} \end{Bmatrix} + \begin{matrix} EBT \\ Indicator \end{matrix} \xrightarrow{pH = 9 - 10} \begin{Bmatrix} Ca^{2+} \\ Mg^{2+} \, EBT \end{Bmatrix} Complex$$
(Weak) (Wine red colour)

When EDTA is added into the hard water, the metal ions from a stable metal complex with EDTA by leaving the indicator. When all the metal ions are taken by EDTA from the indicator metal ion complex, the wine red colour changes into steel blue, which denotes the end point. The metal EDTA complex is stable at pH 8-10. This pH range can be maintained by adding ammoniacal buffer ($NH_4Cl + NH_4OH$).

$$\left.\begin{array}{l} Ca^{2+} \\ Mg^{2+} \, EBT \end{array}\right\}_{Complex} + \; EBT \; \xrightarrow{pH = 9 - 10} \; \left.\begin{array}{l} Ca^{2+} \\ Mg^{2+} \, EBT \end{array}\right\}_{Complex} + \; EBT$$

(Stable) (Colourless)   (Steel blue colour)

$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3 \downarrow + CO_2 + H_2O$$

$$Mg(HCO_3)_2 \xrightarrow{\Delta} MgCO_3 \downarrow + CO_2 + H_2O$$

The filtrate is collected in the conical flask, EBT indicator and buffer solutions are added. It is then titrated against the EDTA.

Temporary hardness = Total hardness – Permanent hardness

MATERIALS REQUIRED

1. EDTA solution, 2. Standard hard water, 3.EBT indicator, 4.Buffer solution, 5.Burette, pipeete, conical flask, 250 ml beaker, 100 ml std. flask, 6. Sample hard water

PROCEDURE

Step I

TITRATION – I
Standardisation of EDTA

The burette is washed well with the distilled water and rinsed with a little amount of the given EDTA solution. It is then filled with the same EDTA solution upto the zero level without air bubbles. Initial reading of the burette is noted. 20 ml of standard hard water solution is pipette out into a clean conical flask. 5 ml of ammonia buffer solution and 2 drops of EriochromeBlack – T indicator are added. The solution turns wine red in colour and it is then titrated against EDTA taken in the burette. The change of wine red colour to steel blue colour is the end point. The final reading in the burette is noted. The difference in the burette reading gives the volume of the EDTA solution. The titration is repeated to get concordant values.

Step II

TITRATION – II
Determination of Total hardness of Hardwater Sample

20 ml of the given hard water sample is pipetted out into a clean conical flask. 5 ml of ammonia buffer solution and 2 drops of Eriochrome Black – T indicator are added. The solution turns wine red in colour. This solution is titrated against the EDTA taken in the burette. The change in colour from wine red to steel blue colour is the end point. The titration is repeated to get concordant values.

Step III

TITRATION – II

Determination of Total hardness of Hardwater Sample

100 ml of the sample of hard water is taken in a clean 250 ml beaker and boiled for 10-15 minutes. It is then cooled and filtered. The filtrate is collected in a 100 ml standard flask and made upto the mark using distilled water. 20 ml of this made up solution is pipette out into a clean conical flask. 5 ml of ammonia buffer solution and 2 drops of Eriochrome Black – T indicator are added. The solution turns wine red in colour. This solution is titrated against the EDTA taken in the burette. The change in colour from wine red to steel blue colour is the end point. The titration is repeated to get concordant values.
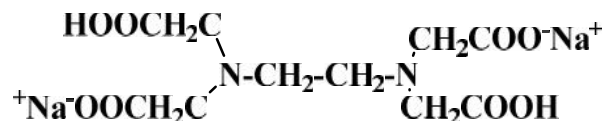
Step IV

Determination of temporary Hardness

Temporary hardness of the water sample is calculated by subtracting permanent hardness from total hardness.

Temporary hardness = Total hardness – permanent hardness

Structure of Disodium salt of EDTA

$$HOOCH_2C \diagdown$$
$$N\text{-}CH_2\text{-}CH_2\text{-}N \quad CH_2COO^-Na^+$$
$$^+Na^-OOCH_2C \diagup \qquad CH_2COOH$$

Fig. 1

Step I: STANDARDISATION OF EDTA

TITRATION – I

Standard Hard water vs. EDTA

Sl. No	Volume of water sample ml	Burette Readings		Concordant volume of EDTA (V_1) ml	Indicator
		Initial ml	Final ml		
1.	20	0			EBT
2.	20	0			

CALCULATION:

Volume of standard hard water $V_1 = 20$ **ml**

Strength of standard hard water $N_1 =$ ……………**N**

Volume of EDTA $V_2 =$ …………..**ml**

Strength of EDTA $N_2 = ?$

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times \text{....................}N}{\text{...............}}$$

Strength of EDTA = ……………….**N**

Step II: DETERMINATION OF TOTAL HARDNESS OF HARDWATER SAMPLE

<div align="center">

TITRATION – II
Hardwater Sample Vs. Std. EDTA

</div>

Sl. No	Volume of sample hard water ml	Burette Readings		Concordant volume of EDTA (V_2) ml	Indicator
		Initial ml	Final ml		
1.	20	0			EBT
2.	20	0			

Calculation of the total hardness of hard water

Volume of EDTA V_1 = …………. ml

Strength of EDTA N_1 = ……………N

Volume of the hard water sample V_2 = 20 ml

Strength of the hard water sample N_2 =?

According to the law of volumetric analysis

$$\boxed{\begin{array}{c} \mathbf{V_1N_1 = V_2N_2} \\[2mm] \mathbf{N_2 = \dfrac{V1 \times N1}{V2}} \end{array}}$$

$$N_2 = \frac{\text{……………} \times \text{…………………N}}{20}$$

Strength of hard water sample = ……………….N

Total hardness of hard water sample = ……………….N x 50x 1000

$$= \text{…………….ppm}$$

Step III: DETERMINATION OF PERMANENT HARDNESS

<u>**TITRATION – III**</u>
Boiled Hardwater Sample Vs. Std. EDTA

Sl. No	Volume of boiled hard water ml	Burette Readings		Concordant volume of EDTA (V_3) ml	Indicator
		Initial ml	Final ml		
1.	20	0			EBT
2.	20	0			

Calculation of the permanent hardness of the hard water

Volume of **EDTA** $V_1 = $ …………. **ml**

Strength of **EDTA** $N_1 = $ ……………**N**

Volume of the boiled hard water sample $V_2 = $ **20 ml**

Strength of the boiled hard water sample $N_2 = $**?**

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$= \frac{\text{…………} \times \text{………………}N}{20}$$

N_2 $= $ ………………**.N**

Permanent hardness of hard water sample $= $ ………………**.**N x 50x 1000

$= $ …………….ppm

Step IV: Calculation of the temporary hardness of the hard water

Temporary hardness = Total hardness – Permanent hardness

$$= \text{………………...} - \text{………………}$$

$$= \text{………………..}\textbf{ppm}$$

RESULT

1. Amount of total hardness of the given sample of water = ………………**ppm**
2. Amount of permanent hardness of the given sample of water = ……………….**ppm**
3. Amount of temporary hardness of the given sample of water = ……………..**ppm**
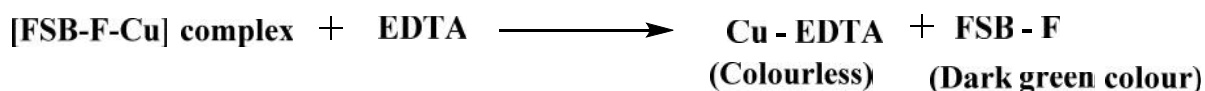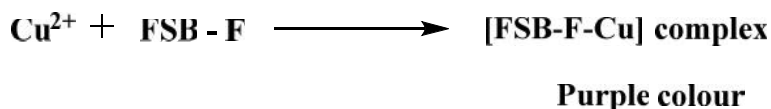
EX. NO: 3
DATE:

ESTIMATION OF COPPER CONTENT OF THE GIVEN SOLUTION BY EDTA METHOD USING FAST SULPHONE BLACK-F INDICATOR

AIM

To estimate the amount of copper present in 100 ml of the given solution being supplied withstandard solution of $ZnSO_4$ of strength …………..N and a link solution of EDTA.

PRINCIPLE

The Cu^{2+} ions are determined by the usual complexometric method using EDTA in presence ofFastsulphone black – F indicator.

FSB-F

$$Cu^{2+} + FSB\text{-}F \longrightarrow [FSB\text{-}F\text{-}Cu] \text{ complex}$$
$$\text{Purple colour}$$

$$[FSB\text{-}F\text{-}Cu] \text{ complex} + EDTA \longrightarrow \underset{\text{(Colourless)}}{Cu\text{-}EDTA} + \underset{\text{(Dark green colour)}}{FSB\text{-}F}$$

MATERIALS REQUIRED

1. Std. $ZnSO_4$ solution, 2. EDTA solution, 3. Fast sulphone black-F indicator, 4. Copper solution, 5. Ammonia buffer solution, 6. Burette, pipette and conical flask.

PROCEDURE

Step I

TITRATION – I
Standardisation of EDTA

The burette is washed well with distilled water and rinsed with the small amount of given EDTAsolution. It is then filled with the same EDTA solution upto the zero mark without any air bubbles.

The pipette is washed with distilled water and rinsed with the small amount of standard Zn^{2+} solution. 20 ml of this solution is pipetted out into a clean conical flask. 10 ml of ammonia

buffersolution and 3-4 drops of EBT indicator are added.The solution turns wine red in colour. It is then titrated against EDTA solution taken in theburette. The change of wine red colour to steel blue colour is the end point. The final reading is noted. The titration is repeated to get concordant values. From the volume of EDTA consumed, strength of EDTA solution is calculated.

Step II

TITRATION – II
Estimation of Copper

The given copper solution is made upto 100 ml in a standard flask. 20 ml of this solution is pipetted out into a clean conical flask. 10 ml of ammonia solution and 3-4 drops of Fast Sulphone Black-F (FSB-F) indicator areadded. The solution is titrated against standard EDTA solution taken in the burette. The change of purple colour into dark green colour is the end point. The titration is repeated to get concordant value.

Step I: STANDARDISATION OF EDTA

TITRATION – I
Standard Zn^{2+} solution Vs. EDTA

Sl. No	Volume of Std Zn^{2+} solution (V_1) ml	Burette Readings		Concordant volume of EDTA (V_2) ml	Indicator
		Initial ml	Final ml		
1.	20	0			EBT
2.	20	0			

Calculation of strength of EDTA solution

Volume of stdZn^{2+} solution $V_1 = 20$ ml

Strength of stdZn^{2+} solution $N_1 = $ ……………N

Volume of **EDTA** solution $V_2 = $ …………..ml

Strength of **EDTA**Solution $N_2 = ?$

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times ....\mathbf{N}}{.................}$$

∴ Strength of EDTAsolution (N_2) = ……………….**N**

Step II: ESTIMATION OF COPPER

TITRATION – II
Copper solution Vs Std. EDTA

Sl. No	Volume of copper solution (V_1) ml	Burette Readings		Concordant volume of EDTA (V_2) ml	Indicator
		Initial ml	Final ml		
1.	20	0			Fast Sulphone Black-F
2.	20	0			

Calculation of strength of copper solution

Volume of **copper** solution V_1 = **20 ml**

Strength of **copper**solution N_1= ……………**N**

Volume of **EDTA** solution V_2 = …………..**ml**

Strength of **EDTA** Solution N_2 =?

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \text{ x}.....................\textbf{N}}{...............}$$

Strength of **copper** solution = ………………**N**

Calculation of amount of copper present in the solution

Amount of Cu present in 1 litre of the given solution   =

                                                    Strength of Cu solution x Atomic mass of Cu

                                                    = …....................N x 63.54

                                                    = …....................g/L

Amount of Cu^{2+} present in 100 ml of the given solution   =..............N x 63.54 x 100/1000 gms

                                                    = ………………gms

RESULT

1. The amount of copper present in the whole of the given solution   = …....................**gms**

2. The amount of copper present in 100 ml the given solution   = …....................**gms**

EX. NO: 4
DATE:

ESTIMATION OF FERROUS ION BY POTENTIOMETRIC TITRATION

AIM

To estimate the amount of ferrous ion (Fe^{2+}) present in 100 ml of the given solution potentiometrically. A standard solution of potassium dichromate of strength …............N is provided.

PROCEDURE

Potentiometric titrations depend on emf between reference electrode and an indicator electrode. When a solution of ferrous ion is titrated with a solution of potassim dichromate, the following redox reaction takes place.

$$6\,Fe^{2+} + Cr_2O_7^{2-} + 14\,H^+ \longrightarrow 6\,Fe^{3+} + 2\,Cr^{3+} + 7\,H_2O$$
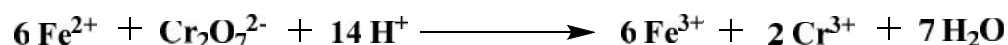
During the titration Fe^{2+} is converted into Fe^{3+}, whose concentration increases. At the endpoint, there will be a sharp change due to sudden removal of all Fe^{2+} ions.

The cell is set up by connecting this redox electrode with a calomel electrode as shown below.

$$Hg / Hg_2Cl_{2(S)}, KCl // Fe^{2+}, Fe^{3+} / Pt$$

A graph between emf measured against the volume of $K_2Cr_2O_7$ added is drawn and the endpoint is noted from the graph.

MATERIALS REQUIRED

1. Potentiometer, 2. Pt electrode, 3.Saturated Calomel electrode, 4.Standard $K_2Cr_2O_7$ solution, 5.Given ferrous ion solution, 6.Burette and pipette, 7.Dil. H_2SO_4, 8. Distilled water

PROCEDURE

The given ferrous ion solution is transferred into 100 ml standard flask and made upto the markusing distilled water. 20 ml of this made up solution is pipetted out into a clean 100 ml beaker. About 10 ml of dil H_2SO_4 and 20 ml of distilled water are added in it. A platinum

electrode is dipped into the solution. This electrode is then coupled with a saturated calomel electrode and the cell isintroduced into potentiometric circuit.

TITRATION – I

The burette is washed well with water and rinsed with the given $K_2Cr_2O_7$ solution. It is then filled with the same upto zero mark and titrated against the ferrous ion solution taken in the conical flask.

The addition is carried out by adding std. $K_2Cr_2O_7$ solution in portions of 1 ml and the emfofthe cell is measured after each addition. The addition of $K_2Cr_2O_7$ is continued even after the endpoint and the range at which end point lies is found out by plotting volume of $K_2Cr_2O_7$ added againstemf (graph-1)

TITRATION – II

Another titration is carried out by adding std. $K_2Cr_2O_7$ solution in portions of 0.1 ml near theend point and the emf of the cell is measured after each addition. The addition of $K_2Cr_2O_7$ iscontinued even after the end point for further 1 ml. The accurate end point is determined by plotting$\sqcup E$ / $\sqcup V$Vs Volume of $K_2Cr_2O_7$ added (graph-2). From the end point, the strength of ferrous ionsolution and hence its amount is calculated.
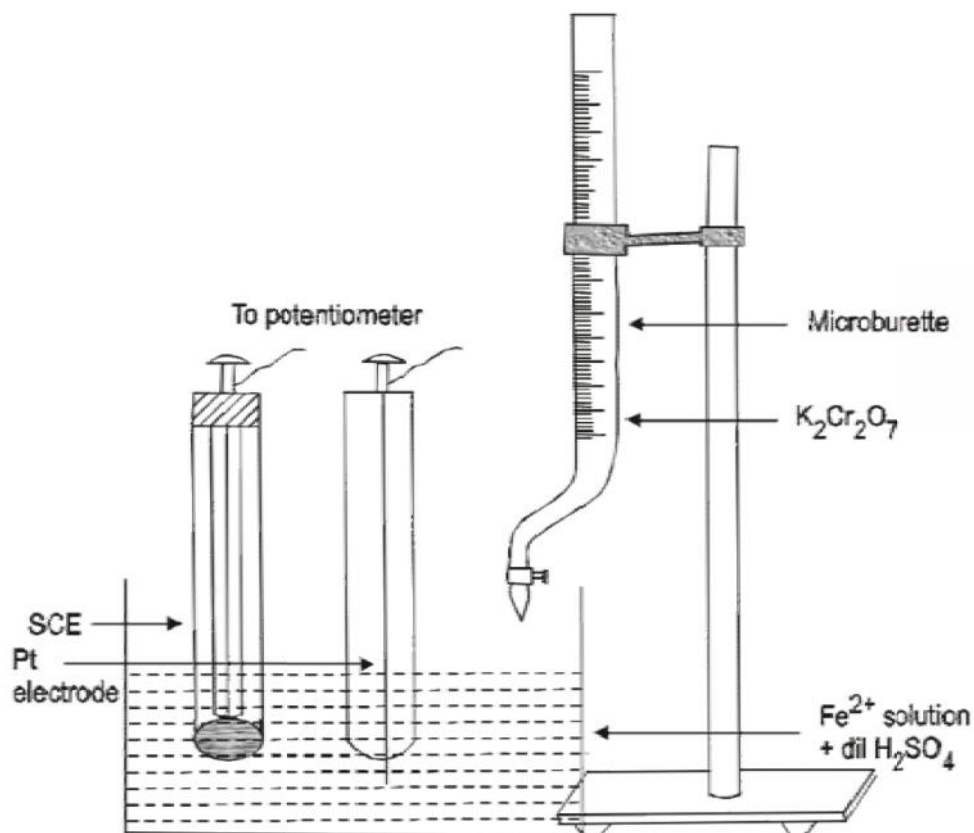
Graph 1: Volume ofK$_2$Cr$_2$O$_7$ Vsemf   **Graph 2: Volume of K$_2$Cr$_2$O$_7$ Vs ∪E / ∪V**
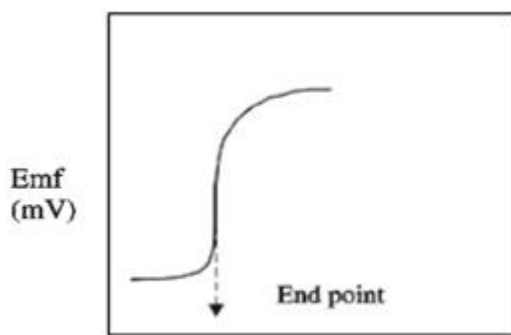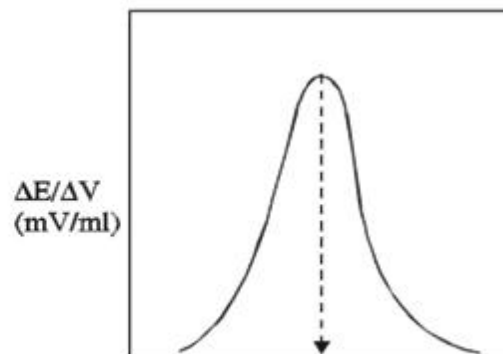
Step I

TABLE – I

TITRATION – I
K$_2$Cr$_2$O$_7$ Vs Iron Solution

Volume of Ferrous Ion Solution = 20 ml

S. No	Volume of K$_2$Cr$_2$O$_7$ ml	Emf (Volts)

TABLE – II

TITRATION – II
K₂Cr₂O₇ Vs Iron Solution

Volume of Ferrous Ion Solution = 20 ml

S. No	Volume of K₂Cr₂O₇ ml	Emf (Volts)	U𝐄 Volts	U𝐕 Ml	U𝐄 / U𝐕 volts / ml

Step – II

Calculation of strength of ferrous ion solution

Volume of **ferrous ion** solution	V_1 = 20 ml
Strength of **ferrous ion** solution	N_1= ?
Volume of $K_2Cr_2O_7$	V_2= …...........ml
Strength of $K_2Cr_2O_7$	N_2 = …...............N

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_1 = \frac{V2 \times N2}{V1}$$

$$N_2 = \frac{20 \times ....N}{...............}$$

Strength of **ferrous ion** solution = ……………….N

Calculation of amount of ferrous ion

The amount of ferrous ion present in 100 ml of the given solution = …...........N x 55.85 x $\frac{100}{1000}$gms

= ……………………………gms.

RESULT

The amount of ferrous ion present in 100 ml of the given solution = …....................**gms**

EX. NO: 5
DATE:

# CONDUCTOMETRIC PRECIPITATION TITRATION USING $BaCl_2$ - Na_2SO_4

AIM

To determine the amount of $BaCl_2$ present in one litre of the given solution byconductometrictitration using standard Na_2SO_4 of …....................N.

PRINCIPLE

Solution of electrolytes conducts electricity due to the presence of ions. Since specificconductance of a solution is proportional to the concentration of ions in it, conductance of the solutionis measured during titration.

In the precipitation titration, the ions are converted into insoluble precipitate, which will not contribute to the conductance.

When Na_2SO_4 is added slowly from the burete to the solution of $BaCl_2$, $BaSO_4$getsprecipitated while the chloride ions remain unchanged.

$$BaCl_2 \; + \; Na_2SO_4 \longrightarrow BaSO_4 \; + \; 2\,NaCl$$
$$\textbf{Unionised}$$
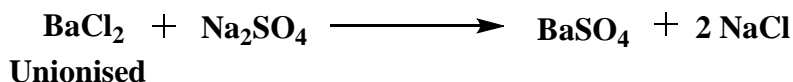
The Ba^{2+} ions in the solution are replaced by free Na^+ ions. Since the mobility of Na^+ ions are less than that of Ba^{2+} ions, the conductance of the solution decreases.

After the end point, when all the Ba^{2+} ions are replaced, further addition of Na_2SO_4 increasesthe conductance. This is due to the increase of Na^+ and SO_4^{2-} ions in the solution.

MATERIALS REQUIRED

1. Conductivity bridge, 2.Conductivity cell, 3.100 ml beaker, 4.Sandard Na_2SO_4 solution, 5.Given $BaCl_2$ solution, 6.Burette, pipette, glass rod etc., 7.Distilled water.

PROCEDURE

TITRATION

The burette is washed well with water and rinsed with the given Na_2SO_4 solution. It is thenfilled with the same Na_2SO_4 solution upto the zero level. 20 ml of the given $BaCl_2$ solution

ispipetted out into a clean 100 ml beaker. The conductivity cell is placed in it and then diluted to 50 ml by adding conductivity water. The two terminals of the cell are connected with a conductivity bridge.

Now 1 ml of Na_2SO_4 from the burette is added to the solution, taken in the beaker, stirred, andthen conductivity is measured. This is continued upto the end point. After the end point, againNa_2SO_4 is gradually added and few more readings are noted.

Thus, the conductivity is continuously measured for each addition of Na_2SO_4 and is tabulated.Now the graph is plotted between the volume of Na_2SO_4 and conductivity. The end point is theintersection of the two curves. From the volume of Na_2SO_4, the strength and hence the amount of $BaCl_2$present in 1 litre is calculated.

TABLE – I
$BaCl_2$ VsNa_2SO_4

S. No	Volume of Na_2SO_4 added ml	Conductance mho	Remarks

CALCULATIONS:

Calculation of Strength of $BaCl_2$

Volume of $BaCl_2$ solution	**$V_1 = 20$ ml**	
Strength of $BaCl_2$ solution	**$N_1 = ?$**	
Volume of Na_2SO_4 solution	**$V_2 = $ …............ml**	
Strength of Na_2SO_4 solution	**$N_2 = $ …...............N**	

According to the law of volumetric analysis

$$\boxed{\begin{array}{c} \mathbf{V_1N_1 = V_2N_2} \\[2mm] \mathbf{N_1 = \dfrac{V2 \times N2}{V1}} \end{array}}$$

$$N_2 = \frac{\text{…...............X..................N}}{20}$$

Strength of $BaCl_2$ solution = ………………N

Calculation of amount of $BaCl_2$

Amount of **$BaCl_2$** present in 1 litre of the given solution =

Strength of $BaCl_2$ x Eq. wt. of $BaCl_2$

= …...................N x 122.14

= …....................gms/L

RESULT

The amount of $BaCl_2$ present in 1 litre of the given solution = …..................**gms**

EX. NO: 6
DATE:

DETERMINATION OF RATE OF CORROSION BY WEIGHT LOSS METHOD

AIM

To determine the rate of corrosion of mild steel specimen in hydrochloric acid of different concentrations at constant temperature.

PRINCIPLE

Generally metal surfaces are covered with impurities like rust and scales. These impurities if present at the time of coating will produce porous and discontinuous coatings. In order to get a uniform, smooth, and adherent coating, these substances must be removed by acid cleaning (or) pickling. For which dil. HCl is used as pickling solution.

Acid cleaning is applicable to remove the scale by oxidation of metal

$$Fe \longrightarrow Fe^{2+} + 2e^-$$

During this process the metal ions, along with rust, get dissolved in the pickling solution. Such dissolution is called corrosion. As the time increases the rate of corrosion also increases.

MATERIALS REQUIRED

1. Mild steel specimens, 2. HCl of different concentrations, 3.100 ml beakers, 4. 100 ml measuring jars, 5. 100 ml standard flask, 6.Weighing balance.

PROCEDURE

Mild steel specimens of known dimensions are taken, washed with distilled water and air dried. Initial weights of the specimens are noted as W_1 gms.

Similarly hydrochloric acids of different concentrations (say 3%, 6%, 9%, 12%, and 15%) are prepared (called pickling solutions).

Now the steel specimens are dipped in 100 ml of various HCl solutions for 15-30 minutes. After that they are taken out and washed with distilled water thoroughly and air dried. Again the specimens are weighed and the final weights are noted as W_2 gms.

From the weight loss the rate of corrosion can be calculated using the following relation.

$$\text{Rate of corrosion} = \frac{534 \, X \, W1 - W2 \, gms}{DXAXT}$$

Where,

W_1 = Initial weights of the steel specimen (in gms)

W_2 = Final weights of the steel specimen (in gms)

D = Density of the specimen (gm/cm^3)

A = Area of the specimen (inch^2)

T = Time of exposure (in hrs)

TABLE –I

Observation

S. No.	Initial Weight of steel specimen (W₁) gms	Final weight of steel specimen (W₂) gms	Percentage of HCl	Weight loss (W₁ – W₂) gms

Calculation:

Rate of corrosion =

$$\frac{534 \, X \; W1 - W2 \; \text{gms}}{DXAXT}$$

$$= \frac{534 \, X \dots\dots\dots\dots\dots\dots\dots\dots}{\dots\dots\dots X \dots\dots\dots X \dots\dots}$$

Density of the specimen = **7.874 gm/ cm³**

RESULT

The rate of corrosion is found to be …………………………

EX. NO: 7
DATE:

DETERMINATION OF CALCIUM OXIDE (CaO) IN CEMENT
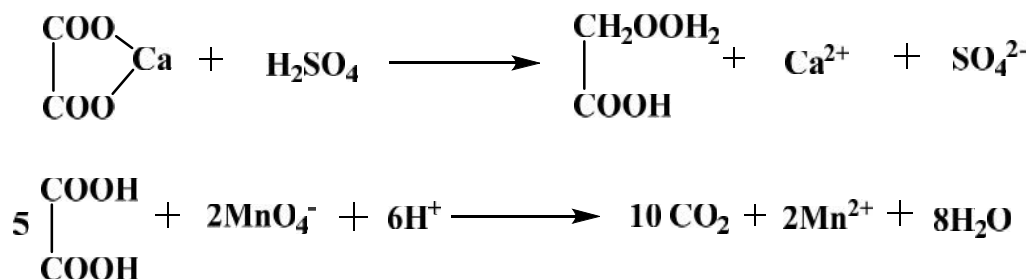
AIM

To determine the amount of CaO present in ……… gms of cement. You are provided with ……… N solution of $KMnO_4$.

PRINCIPLE

Cement is a complex substance containing silicates of calcium as the main constituent.The analysis of cement involves the estimation of calcium. Calcium in cement extract is precipitated as calcium oxalate by the addition of ammonium oxalate in alkaline medium.

$$Ca^{2+} \; + \; \begin{matrix} COO^- \\ | \\ COO^- \end{matrix} \longrightarrow \begin{matrix} COO \\ | \quad Ca \downarrow \\ COO \end{matrix}$$
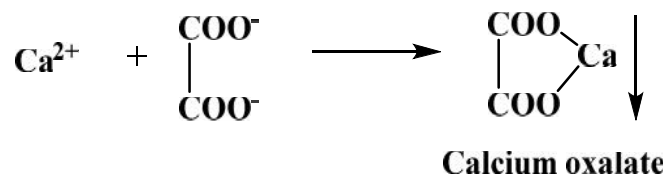
Calcium oxalate

The calcium oxalate precipitate is dissolved in dil. H_2SO_4.

$$\begin{matrix} COO \\ | \quad Ca \\ COO \end{matrix} + \; H_2SO_4 \longrightarrow \begin{matrix} CH_2OOH_2 \\ | \\ COOH \end{matrix} + \; Ca^{2+} \; + \; SO_4^{2-}$$

$$5 \begin{matrix} COOH \\ | \\ COOH \end{matrix} + \; 2MnO_4^- \; + \; 6H^+ \longrightarrow 10\,CO_2 + 2Mn^{2+} + 8H_2O$$

The liberated oxalic acid is estimated by using std. $KMnO_4$ solution.

MATERIALS REQUIRED

1.   Cement sample, 2. Con. HCl, 3. Ammonium oxalate solution, 4. Ammonium hydroxide solution, 5.Whatmann filter papper No. 40, 6. 0.1 % ammonium oxalate solution, 7. Dil. H_2SO_4, 8.Std. $KMnO_4$ solution, 9. China dish, 100 ml std flask, 500 ml beaker, glass rod, conical flask.

PROCEDURE

Preparation of cement extract

About 1 gram of the cementis exactly weighed in a china dish, 5 ml of con. HCl is added and strongly heated to convert calcium ions into chlorides. The calcium ions are extracted with hot water and filtered. The residue is washed with water and the filtrate is made up to 100 ml in standard flask using distilled water.   (or)

The given cement extract solution is transferred into 100 ml standard flask and made upto the mark using distilled water.

Preparation of cement solution

20 ml of the made up solution (cement extract) is pipetted out into a clean 500 ml beaker, heated and 20 ml of ammonium oxalate solution is slowly added with constant stirring. Ammonium hydroxide solution is added drop wise until the red colour of solution changes to yellow. After vigorous stirring, the solution is allowed to stand for ½ an hour. It is then filtered through whatman filter paper No. 40. The precipitate is washed several times with 0.1 % ammonium oxalate solution and again by distilled water.

A hole is made in the filter paper with a glass rod and transferred the whole precipitate into 100 ml std flask made upto the zero mark with 20 ml of dil. sulphuric acid.

TITRATION

Determination of calcium oxide

The burette is washed and ringed with given standard $KMnO_4$ solution. It is then filled with the same $KMnO_4$ solution up to zero mark. 20 ml other made-up solution is pipetted out into a clean conical flask. 5 ml of dil. H_2SO_4 is added and heated up to 60°C and titrated against std. $KMnO_4$ solution taken in the burette. Appearance of pale pink colour is the end point. The titration is repeated for concordant value. From the titre value, normality and hence amount of CaO present in whole of the given solution is calculated.

Calculaions:

Weighing of cement for preparation of cement extract

Weight of the empty weighing bottle (or) paper = **A** = …. **gms**

Weight of the empty weighing bottle (or) paper + cement = **B** = ……. **gms**

Weight of the cement = **B – A** = …… (-) ……

x = ….. **gms**

TITRATION

Standard KMnO₄vs Cement solution

S. No	Volume of cement solution (V_A) ml	Burette Readings		Concordant volume of KMnO₄ (V₂) ml	Indicator
		Initial ml	Final ml		
1.	20	0			
2.	20	0			Self
3.	20	0			

Calculation of strength of CaO in cement extract

Volume of cement extract solution taken for precipitation = **20 ml**

Volume of prepared cement solution taken for titration (**V_A**) = **20 ml**

ie., 100 ml of prepared cement solution = **20 ml** of cement extract

20 ml of prepared cement solution $= \dfrac{20 \times 20}{100}$

= 4 ml of cementextract (**V₁ ml**)

Volume of **cement** extract, **V₁ = 4 ml**

Normality of **cement** extract, **N₁= ?**

Volume of **KMnO₄,** **V₂** = …………**ml**

Normality of **KMnO₄,** **N₂** = ………….**N**

According to volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_1 = \frac{V2 \times N2}{V1}$$

Normality of CaO in cement extract $N_1 = \dfrac{\ldots\ldots\ldots ml \times \ldots\ldots\ldots N}{4}$

Calculation of amount of CaO in cement extract

Amount of **CaO** present in whole (1000 ml) of the given cement sample

= **Normality ion x Eq. wt. of CaO**

$$= \frac{\ldots\ldots\ldots ml \times \ldots\ldots\ldots N}{4} \times 28 \text{ gms}$$

Amount of **CaO** present in 100 ml of the given cement solution

$$= \frac{\ldots\ldots\ldots ml \times \ldots\ldots\ldots N}{4} \times \frac{100}{1000} \text{ gms}$$

(or)

Amount of **CaO** present in ……. (xgms) of the cement $= \dfrac{\ldots\ldots\ldots ml \times \ldots\ldots\ldots N}{4} \times \dfrac{100}{1000} \text{ gms}$

$$= \ldots\ldots\ldots \text{ gms}$$

RESULT

1. The amount of CaO present in the whole of the given cement extract = …… **gms**

2. The amount of CaO present in …… (x) gms of the cement = ……….**gms**