

MUFFAKHAM JAH COLLEGE

OF

ENGINEERING AND TECHNOLOGY



(SULTAN-UL-ULOOM EDUCATION SOCIETY)

BANJARA HILLS, HYDERABAD - 500 034.

A MANUAL OF CHEMISTRY PRACTICALS

FOR B.E. I YEAR SEMESTER-I

ACADEMIC YEAR 2017-2018

VOLUME I : VOLUMETRIC ANALYSIS

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Branch (section): _____

Roll No. 1604 - 17 - _____

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Safety instructions to the students

1)Poisonous Chemicals: All of the chemicals have some degree of health hazard. *Never taste* any chemical in the laboratory unless specifically directed to do so. Avoid breathing toxic vapours. When working with volatile chemicals, strong acid and bases, using ventilating hood. If you are asked to taste the odour of a substance, do it by *wafting* a bit of the vapour towards your nose. Do not stick your nose inhale vapour directly from the test tube. Always wash your hand before leaving the laboratory.

2)Cut and Burns: Use glassware with care, Glassware is breakable and may cause cuts. When a piece of glass is heated, it gets hot very quickly. Since hot glass looks just like a cold one, handle it with a tong. Do not use any cracked or broken glass equipment. It may ruin an experiment and worse, it may cause serious injury. Place it in a waste glass container.

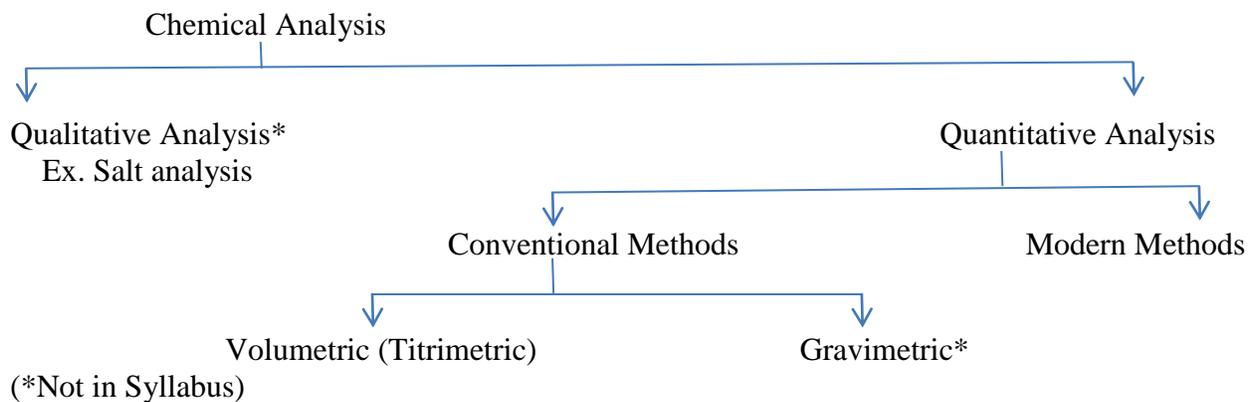
3)Eating and drinking: Any type of food is prohibited in the laboratory at all times and smoking is not allowed.

4)Clothing and Footwear: Everyone must wear a lab coat in the lab. Long hair should be securely tied back to avoid the risk of setting it on fire. If large amount of chemicals are spilled on your body, immediately remove the contaminated clothing and use the safety shower if available. Make sure to inform your instructor about the problem. Do not leave your coats and back packs on the bench. No Cell Phones, headphones and Walkman are allowed in the lab because they interfere with your ability to hear what is going on in the Lab.

5)Eye Protection: Because the eyes are particularly susceptible to permanent damage by corrosive chemical as well as flying objects, preferably wear safety goggles in the Laboratory. No sunglasses are allowed in the laboratory. Contact lenses have potential hazard because the chemical vapours dissolve in the liquids covering the eye concentrate behind the lenses. If you are using contact lenses consult with your instructor. If possible try to wear prescribed glasses under your safety goggles. In case of any accident that a chemical splashes near your eyes, immediately wash your eyes with a lot of water and inform your instructor. Especially, when heating a test tube do not point its mouth to anyone or you.

6)Fire: in case of fire or accident, inform your instructor immediately. Note the location of fire extinguishers as soon as you enter the laboratory so that you may use them if needed. Never perform an unauthorized experiment in the laboratory.

VOLUMETRIC ANALYSIS



INTRODUCTION:

Volumetric analysis or Titrimetry analysis, just as gravimetric analysis gives a quantitative estimation of species. It involves a measurement of the volume of a solution known as concentration (standard solution) that is required to react completely with the species to be estimated. This method is applicable to fast reaction in solutions. Its advantages include: simple apparatus, simple methods and lesser time with a high accuracy as compared to many other techniques.

The Chemical Analysis which includes both, Qualitative and Quantitative, can be done using instruments.

Since two reacting species would react in the same number of equivalents, the following equation is established:

$$N_1 \times V_1 = N_2 \times V_2$$

Where N and V refer to normality and volume respectively and the subscripts refer to species 1 and 2. Knowing the normality of one of the solutions (the standard solution) and the two reacting volumes, the normality N of the solution under test can be determined.

DEFINITIONS:

- i) **Titration:** The overall procedure of determining stoichiometric or equivalence point is called 'titration' or 'titrimetry'.
- ii) **Titrant:** The solution added in a titration.
- iii) **Titrand:** The solution to which the titrant is added.
- iv) **End point:** A point in the progress of the reaction which may be precisely located (almost coincident with stoichiometric or equivalence point).
- v) **Indicator:** A reagent used to indicate when the end point is reached. (In some cases, one of the reactants serves as its own indicator (self-indicator) as in the case of titrations involving KMnO_4)

*Education is a progressive discovery of our own ignorance

Primary and Secondary Standards:

In titrimetry certain chemicals are used frequently in definite concentrations as reference solutions. Such substances are referred to as Primary Standards or Secondary Standards.

A Primary standard is a compound of sufficient purity from which a standard solution can be prepared by direct weighing a quantity of it, followed by dilution to give a definite volume of solution. The solution produced is then a primary standard solution.

Requirements of a primary standard are: purity, easy availability, dryness and preserve in a pure state (a requirement not usually met by hydrated substances); should be unaltered in air during weighing (should not be hygroscopic, oxidized in air, or affected by CO_2), it should be of high relative molecular weight so as to minimize weighing errors; should be readily soluble.

Examples: $\text{K}_2\text{Cr}_2\text{O}_7$ (Potassium dichromate), Na_2CO_3 (Sodium Carbonate), $\text{Na}_2\text{B}_4\text{O}_7$ (Sodium Borate) $\text{H}_2\text{C}_2\text{O}_4$ (Oxalic Acid), $\text{FeSO}_4(\text{NH}_4)_2 \cdot \text{SO}_4 \cdot 6\text{H}_2\text{O}$ (Ferrous Ammonium sulphate)

A Secondary standard is a substance which is standardized and whose content of the active substance has been found by comparison against a primary standard. It follows that a secondary standard solution is a solution in which the concentration of dissolved solute has not been determined from the weight of the compound dissolved but by reaction (titration) of a volume of the solution against a measured volume of a primary standard solution.

Examples: KMnO_4 (Potassium Permanganate), $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ (Hypo)

Advantages of volumetric titration:

Apparatus used is simple

Easy to handle

Economical

Time saving

Good accuracy

*Education's purpose is to replace an empty mind with an open one

The set of experiments on Volumetric Analysis are designed with the object of giving a fair background to engineering students. The knowledge gained can thus be applied in solving typical scientific and industrial problems like general chemical analysis, ore analysis, water analysis and metallurgy etc.

The experiments are classified as follows:

- i) Acid-Base titrations: Estimation of carbonate and bicarbonate(alkalinity)
- ii) Oxidation and reduction (Redox) Titrations:
 - FAS Vs KMnO_4
 - Oxalic acid Vs KMnO_4 ; Fe^{2+} Vs. $\text{K}_2\text{Cr}_2\text{O}_7$
 - Also Iodometry: $\text{K}_2\text{Cr}_2\text{O}_7$ Vs. Hypo; Cu^{2+} Vs. Hypo.
- iii) Precipitation titration (NaCl Vs. Ag^+ ; Ag^+ Vs. CNS)
- iv) Complexometric titration(EDTA) method of estimation of hardness of water (total, permanent and temporary)

TITRATIONS:

(Redox Principle): Since 'Redox' titrations cover a basic principle, this type would be discussed briefly. Appropriate comments on other types would be included in the relevant experiments.

The principle of volumetric analysis, as already indicated earlier, could be expressed mathematically as follows:

$$N = \frac{\text{Wt.}}{\text{Eq.Wt.}} \times \frac{1}{V_{\text{ml}}} \times 1000 \text{-----(1)}$$

Where N=Normality of the solution

V_{ml} = Volume of standard flask in ml

$$N_1 \times V_1 = N_2 \times V_2 \text{-----(2)}$$

Where N and V refer to normality and volume respectively and the subscripts refer to the reacting solutions 1 and 2, taken in pipette & burette.

It is suggested that in practice, relation (2) should always be used with the species indicated clearly instead of using the numerical subscripts. For instance, it should be written as follows:



*It is the mark of an educated mind to be able to entertain a thought without accepting it

-Aristotle

Let us now briefly discuss the most important aspects of volumetric analysis or titrations, namely the concept of equivalent weight of a substance. This may be stated as follows:

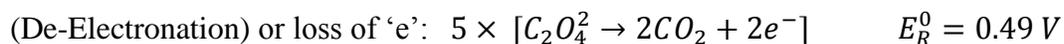
“Equivalent weights of species (substances) are derived from the balanced ionic (or molecular) equation of a particular reaction so that it involves one-electron transfer”.

The following example of the reaction of KMnO_4 Oxalic acid in acidic (H_2SO_4) medium illustrates this principle as applied to Redox reactions (and also to other electron exchange reactions).

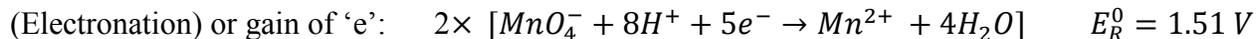
A redox reaction can be written as two parts or two half reactions, one indicating oxidation and the other indicating reduction. However, it should be clearly understood that the redox process is simultaneous process. **It takes place in the presence of both the Oxidant (oxidizing agent) and the Reductant (Reducing agent) at one and the same time.**

Example: The balanced reaction can preferably be written more elegantly in terms of only the reacting species which undergo Oxidation – Reduction.

Half reaction: Oxidation:



Half reaction: Reduction:



Overall Redox Reaction :



In the above reaction it may be noted that the oxidant KMnO_4 gets reduced itself because of its High Reduction Potential ($E_R^0 = 1.51 \text{ V}$) at the same time the reductant, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ gets oxidized itself because of Less Reduction Potential ($E_R^0 = 0.49 \text{ V}$). The equivalence established as follows:

$$2\text{MnO}_4^- = 10e^- = 5\text{C}_2\text{O}_4^{2-} \text{ (or); } \frac{2 \text{ KMnO}_4}{10} = \frac{10e^-}{10} = \frac{5(\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O})}{10}$$

$$1 / 5\text{KMnO}_4 = 1e^- = 1 / 2 (\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O})$$

$$1 / 5 (158) = 1e^- = 1 / 2 (126)$$

$$31.6\text{g} = 1e^- = 63 \text{ g.}$$

*Education is not filling a pail but the lighting of fire

Note 1: In a Redox reaction the Half reaction with high Reduction Potential will undergo reduction and the one with less Reduction Potential undergoes oxidation.

Note 2: It should be noted that in an experiment such as: “Preparation of a standard solution of $H_2C_2O_4 \cdot 2H_2O$ (Oxalic acid) and standardization of $KMnO_4$ ” (Redox Titration) the eq.wt. of Oxalic acid is $126/2=63.0$ g and that of $KMnO_4$ is $158/5=31.6$ g.

In other words the eq. wt. has to be defined as the weight of substance involving one electron transfer (Reduction or Oxidation).

LINK TITRATION (OR) DOUBLE TITRATION

Link Titration Principle: The Principle of link titration to be used ultimately in the experimentation would now be briefly described.

If it is required to determine the normality of a solution (test solution) and the amount of a species therein using a similar type of a standard solution, this can be done using a system of the following type, depending on the nature of the test solution.

System I	$\frac{\text{standard}}{H_2C_2O_4 \cdot 2H_2O}$	$\frac{\text{link}}{KMnO_4}$	$\frac{\text{Test}}{H_2C_2O_4 \cdot 2H_2O}$
	(Reductant)	(Oxidant)	(Fe^{2+} Amm. Sulphate)
	$H_2C_2O_4 \cdot 2H_2O$	$KMnO_4$	Fe^{2+}
	(Reductant)	(Oxidant)	(Reductant)
System II	$K_2Cr_2O_7$	Fe^{2+} Amm. Sulphate	$K_2Cr_2O_7$
	(Oxidant)	(Reductant)	(Oxidant)

A typical experiment involving a link titration, consists of following three steps for system I

- i) **Prepare a standard solution** of oxalic acid
- ii) **Standardize $KMnO_4$ Link solution** and then
- iii) **Estimate** the amount of Fe^{2+} present in the given **test solution**.

*Education is not preparation of life, education is life

TITRATION DATA, TABULATION AND CALCULATIONS

Titration data should essentially be recorded as follows:

The following example is for single titration: std $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ Vs. Fe^{2+} (test solution)

Table.1. Weight of bottle + Substance

12	.	7	6	3	4
----	---	---	---	---	---

Table.2. Weight of empty weighing bottle

11	.	0	7	8	2
----	---	---	---	---	---

i. Normality of prepared std. Oxalic acid solution :

Wt. of $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ = (12.7634 - 11.0782) g = 1.6852 g.

(from tables weight above)

N

$$(\text{std}) \text{ Oxalic acid} = \frac{\text{wt}}{\text{eq.wt}} \times \frac{1}{V} \times 1000 = \frac{1.6852}{63} \times \frac{1}{250} \times 1000 = 0.1070$$

Volume of std. flask = 250ml

Note: please do not take average value (for calculations consider the concordant value)

ii. Titration data : titration of Std. $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ Vs KMnO_4 (link)

Sl No.	V _{oxalic} (Std) (ml)	Burette Reading		V _{KMnO4} ml
		Initial	Final	
1.	20	0.0	19.9	19.9
2.	20	19.9	39.7	19.8
3.	20	0.0	19.8	19.8

$$i \quad N_{\text{KMnO}_4} \times V_{\text{KMnO}_4} = N_{\text{oxalic acid}} \times V_{\text{oxalic acid}}$$

(link) (link) (std.) (std.)

$$N_{\text{KMnO}_4} = \frac{N_{\text{Oxalic acid}} \times V_{\text{Oxalic acid}}}{V_{\text{KMnO}_4}} = \frac{0.1070 \times 20}{19.8} = 0.1080$$

V_{KMnO_4}

Result: 1. Wt. of oxalic acid = -----g.

2. N std. oxalic acid = -----

3. N_{KMnO_4} = -----

DEMO EXPERIMENT

TABULATION AND CALCULATIONS

Date _____

Part A : Preparation of std. ferrous Ammonium sulphate solution :

Table.1.weight of bottle + FAS

	•			
--	---	--	--	--

g.

Table.2.Weight of empty weighing bottle

	•			
--	---	--	--	--

Wt. of FAS = $W_1 - W_2 =$

$$N_{\text{std. F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = \dots\dots\dots$$

($V_{ml} = 100\text{ml}$)

Part B : Standardization of KMnO_4 :

Titration of Std. F.A.S. Vs. KMnO_4

Sl No.	$V_{\text{std FAS}}$ (ml)	Burette Reading		V_{KMnO_4} (ml)
		Initial	Final	

$$N_{\text{KMnO}_4} = \frac{N_{\text{FAS}} \times V_{\text{FAS}}}{V_{\text{KMnO}_4}}$$

Results:

1. Weight of FAS= _____ g

2. $N_{\text{std. FAS}}$ =_____3. N_{KMnO_4} =_____

Signature of Faculty

Experiment (1) : PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION & STANDARDISATION OF GIVEN POTASSIUM PERMANGANATE (KMnO₄) SOLUTION.

Date: _____

Expt. No. _____

TABULATION AND CALCULATIONS

PART A: PREPARATION OF STD. FERROUS AMMONIUM SULPHATE SOLUTION:

Table.1.weight of bottle + FAS

	.			
--	---	--	--	--

Table.2.Weight of empty weighing bottle

	.			
--	---	--	--	--

Wt. of FAS = $W_1 - W_2 =$

$$N_{\text{std F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = \dots\dots\dots$$

($V_{ml} = 100\text{ml}$)

Part B : Standardization of KMnO₄ :

Titration of Std. F.A.S. Vs. KMnO₄

Sl No.	VFAS (Std) (ml)	Burette Reading		V _{KMnO₄} (ml)
		Initial	Final	

$$N_{\text{KMnO}_4} = \frac{N_{\text{FAS}} \times V_{\text{FAS}}}{V_{\text{KMnO}_4}}$$

Experiment(1): PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION & STANDARDISATION OF GIVEN POTASSIUM PERMANGANATE (KMnO₄) SOLUTION.

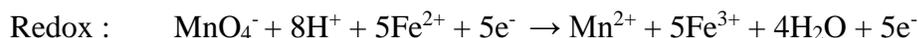
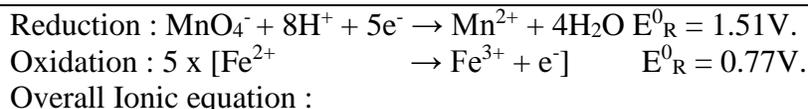
OBJECT: To standardize KMnO₄ solution using standard Ferrous Ammonium Sulphate solution.

Introduction: (Theory): Potassium permanganate is a valuable and powerful oxidizing agent used in titrimetric analysis. In acidic medium Ferrous ion (Fe²⁺) is readily oxidized to Ferric ion (Fe³⁺) by KMnO₄; the redox reactions can be represented by the equation given below. KMnO₄ is often contaminated with manganese-di-oxide and also undergoes decomposition it can be taken as secondary standard solution.

Ferrous ammonium sulphate FeSO₄·(NH₄)₂SO₄·6H₂O, (Mohr's salt- ore of iron) is available in a high purity state and hence is used as primary standard for the standardization of KMnO₄. KMnO₄ acts as self indicator. At the end point of titration, permanganate ions gives light pink colour.

APPLICATION: Titrimetric analysis, estimation of iron (most important quantitative method)

Redox reaction(Partial Ionic equation)

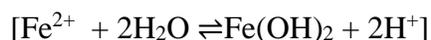


Species: (KMnO₄) (H₂SO₄) (FeSO₄) (MnSO₄)
 Color Profile: (Purple) (X) (X) (X) X=Colorless
 (Equivalent weight = Molecular Wt./ No. of e⁻ transfer)
 Equivalent weights : KMnO₄ / 5 = 158/5 = 31.6;
 FeSO₄·(NH₄)₂SO₄·6H₂O/1 = 392.1/1=392.1

PROCEDURE: PART A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE FeSO₄·(NH₄)₂SO₄·6H₂O SOLUTION:

Weigh out in a clean weighing bottle accurately close to 3.9210 g Ferrous Ammonium sulphate FeSO₄·(NH₄)₂SO₄·6H₂O; transfer it to 100 ml standard Flask through a funnel; dissolve it by adding distilled water including 100ml std. standard Flask through a funnel; dissolve it by adding distilled water including 10 ml dil. H₂SO₄ (6N) (to prevent hydrolysis) and make the solution upto the mark. Shake it well to make it homogeneous. Normality of Std FAS is calculated.

HYDROLYSIS REACTION :



*In your thirst for knowledge, be sure not to drown in all information.

B.STANDARDIZATION OF GIVEN KMnO_4 SOLUTION:

(Note: Before use, wash all glass apparatus with tap water, then rinse with distilled water, subsequently rinse pipette with standard F.A.S. solution and burette with KMnO_4 solutions)

- i) Pipette out 20 ml of the prepared std. Ferrous Amm. Sulphate solution into a clean conical flask.
- ii) Add 10 ml of dilute $\text{H}_2\text{SO}_4(6\text{N})$ solution (1/2 Test Tube)
- iii) Titrate with KMnO_4 (taken in the burette) to a faint pink colour which persists for at least a minute. This is the end point. (last addition of KMnO_4 should be drop wise with particular care to allow each drop to become decolorized before the next is added).
- iv) Repeat the process till at least two concordant vales are obtained.

The Normality of KMnO_4 is calculated .

RESULTS:

1. Wt. of FAS = _____g
2. N std FAS Solution= _____
3. N_{KMnO_4} = _____

QUESTIONS:

1. What is the colour of Mn^{2+} ion?
2. Why no indicator is added in this experiment?
3. Why is dilute H_2SO_4 added in the standard flask while making the standard solution?
4. Which substance gives the pink colour at the end point?
5. Can we use dilute HCl instead of dilute H_2SO_4 ?
6. Why is dilute H_2SO_4 added to Fe^{+2} solution in the conical flask, during titration with KMnO_4 ?
7. Why does KMnO_4 act as self indicator?

Marks	
Observations and calculations (20):	_____
Results (10):	_____
Discussion of results (5):	_____
Record (15):	_____
Total(50):	_____

Signature of Faculty

Experiment (2): Preparation of standard Ferrous ammonium sulphate solution & standardisation of given Potassium Permanganate (kmno₄) solution & Estimation of ferrous (iron) in given test solution.

OBJECT: To standardize KMnO₄ solution using Std. FAS solution.

INTRODUCTION : Principle- Refer Exp. 1.

PROCEDURE

PART-A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE FeSO₄(NH₄)₂SO₄.6H₂O SOLUTION.

Refer Exp. 1.

PART-B: STANDARDIZATION OF GIVEN KMnO₄ SOLUTION:

Refer Exp. 1.

PART-C: ESTIMATION OF Fe⁺² IN THE GIVEN TEST SOLUTION:

The given Fe²⁺ Amm. Sulphate test solution is made up to the mark with distilled water. The solution is thoroughly shaken to make it homogeneous. 20 ml. of the test Fe²⁺ solution is pipetted out into clean conical flask. Approximately (1/2 Test tube) 10 ml. of dilute H₂SO₄ (6N) is added to it. It is titrated with KMnO₄ (taken in a burette) to a Faint Pink colour which persists for at least a minute. This is the end point. The process is repeated till two concordant titre values are obtained. From the titration data the amount of Fe²⁺ in the given test solution is estimated.

From this titration ferrous iron is estimated from Ore of iron that is Mohrs salt(FAS)- ore analysis.

*Man's mind, once stretched by a new idea, never regains its original dimensions.

Experiment (2): Preparation of standard Ferrous ammonium sulphate solution, standardisation of given Pottasium Permanganate (kmno₄) solution & Estimation of ferrous iron in given test solution.

Date _____

Expt No. _____

PART A: PREPARATION OF STD. FERROUS AMMONIUM SULPHATE SOLUTION :

Table.1.weight of bottle + FAS

	.			
--	---	--	--	--

Table.2.Weight of empty weighing bottle

	.			
--	---	--	--	--

Wt. of FAS = $W_1 - W_2 =$

$$N_{\text{std. F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = \dots\dots\dots$$

($V_{ml} = 100\text{ml}$)

Part B : Standardization of KMnO₄ :

Titration of Std. F.A.S. Vs. KMnO₄

Sl No.	V _{std FAS} (ml)	Burette Reading		V _{KMnO₄} (ml)
		Initial	Final	

$$N_{\text{KMnO}_4} = \frac{N_{\text{FAS}} \times V_{\text{FAS}}}{V_{\text{KMnO}_4}}$$

PART C : ESTIMATION OF Fe²⁺ IN THE GIVEN TEST SOLUTIONTitration : Fe²⁺ (Test Soln) Vs KMnO₄ (Link)

Sl No.	V _{Fe²⁺} (test Sol.) (ml)	Burette Reading		V _{KMnO₄} (ml)
		Initial	Final	

$$N_{\text{Fe}^{2+}} (\text{Test}) = \frac{N_{\text{KMnO}_4} \times V_{\text{KMnO}_4}}{V_{\text{Fe}^{2+}}} = \dots\dots\dots$$

$$\text{Wt. of Fe}^{2+} = N_{\text{Fe}^{2+}} \times \text{Eq. Wt.}$$

.....gpl

Results:

1.Weight. of FAS= _____ g

2.N_{std.} FAS = _____3.N_{KMnO₄} (Link)= _____4.N_{Fe²⁺} (Test)= _____5.Weight of Fe²⁺

Present in test solution= _____ gpl

Marks

Observations and calculations (20): _____

Results (10): _____

Discussion of results (5): _____

Record (15): _____

Total(50): _____

Signature of Faculty

Expt (3) Std. Oxalic Acid X K MnO₄ (Link) X Fe²⁺ (Test Soln.)

Expt No. _____.

Date : _____

TABULATION AND CALCULATIONS

PART A: PREPARATION OF STD. OXALIC ACID (H₂C₂O₄. 2H₂O) SOLUTION:

Table.1.weight of bottle + Oxalic Acid

	.			
--	---	--	--	--

Table.2.Weight of empty weighing bottle

	.			
--	---	--	--	--

Wt. of Oxalic Acid = W₁-W₂ =

$$N_{\text{std. oxalic acid}} = \frac{wt}{Eq.wt} \times \frac{1000}{V_{ml}} = \dots\dots\dots$$

(V_{ml} = 250ml)**Part B : STANDARDIZATION OF KMnO₄ (link) :**Titration of Std. H₂C₂O₄.2H₂O Vs. KMnO₄ (link)

Sl No.	V _{oxalic acid} (Std) (ml)	Burette Reading		V _{KMnO₄} (ml)
		Initial	Final	

$$N_{\text{KMnO}_4} = \frac{N_{\text{Oxalic acid}} \times V_{\text{Oxalic acid}}}{V_{\text{KMnO}_4}}$$

'REDOX LINK' TITRATION
(PERMANGANOMETRY)

Experiment (3): Preparation of Standard Oxalic Acid, Standardisation of potassium permanganate (KMnO₄ / Link) Solution & Estimation of Fe²⁺ in the given Test Solution.

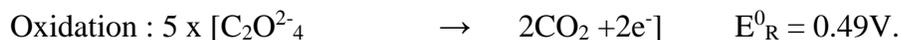
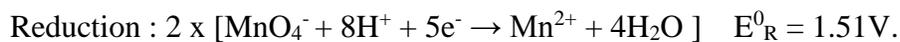
AIM: To estimate the amount of iron of Fe²⁺ in test solution by preparing a Standard Oxalic acid solution & using link KMnO₄ solution.

PRINCIPLE: Introduction (Theory)- KMnO₄ is a powerful oxidizing agent in acidic medium, but it needs to be standardized by using oxalic acid solution of high purity.

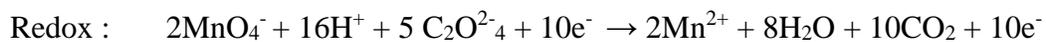
Oxalic acid is a mild reducing agent in acid medium. It is a covalent compound hence reaction with KMnO₄ is slow at room temperature. The reaction is initiated by heating oxalic acid and subsequently it is catalyzed by Mn²⁺ ions as one of the products. KMnO₄ acts as self indicator. At the end point of titration, permanganate ions give faint pink colour.

KMnO₄ a powerful oxidizing agent, oxidizes Oxalic acid to CO₂ in acidic medium in hot condition while itself undergoes reduction to MnSO₄. In estimation part it oxidizes Fe²⁺ to Fe³⁺ in acidic medium.

1. Reaction between Oxalic acid and Permanganate



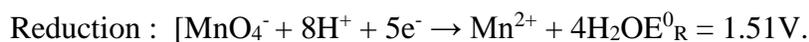
Overall Ionic equation :



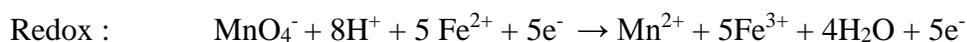
Species: (KMnO₄) (H₂C₂O₄) (MnSO₄)

Color Profile: (Purple) (X) (X) X=Colorless

2. Reaction between Ferrous and Permanganate



Overall Ionic equation :



Species: (KMnO₄) (H₂SO₄) (MnSO₄) (FeSO₄)

Color Profile: (Purple) (X) (X) (X) X=Colorless

(Equivalent weight = Molecular Wt./No. of e⁻ transfer)

Equivalent weights $\text{KMnO}_4/5 = 158/5 = 31.6$; $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}/2 = 126/2 = 63$; $\text{Fe}^{2+} = 55.8$

PROCEDURE

PART A: PREPARATION OF STD. OXALIC ACID ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) SOLUTION:

Exactly _____ gm of Oxalic acid is weighed out accurately in a clean weighing bottle. It is transferred to a 250ml standard Volumetric flask through a funnel then dissolved in distilled water and the solution is made upto the mark. It is shaken well to make it homogenous. The Normality of Standard Solution is calculated from the weighing data.

Part B: STANDARDISATION OF KMnO_4 (LINK SOLUTION)

(Note: Wash all glass apparatus with tap water then rinse with distilled water; also rinse pipette with Std. Oxalic acid solution and burette with KMnO_4 solutions).

20 ml. of the prepared solutions (standard Oxalic acid) is pipetted out into a clean conical flask. Approximately (1/2 Test tube) 10 ml. of dilute H_2SO_4 (6N) is added to it. The mixture is heated to -70°C (as observed by bubbling). While the solution is hot, it is titrated with KMnO_4 (taken in a burette) till a Faint Pink colour which persists for at least a minute. This is the end point. The process is repeated till two concordant titre values are obtained. From the titration data, the normality of KMnO_4 is calculated.

PART-C: ESTIMATION OF Fe^{+2} IN THE GIVEN TEST SOLUTION:

The given Fe^{2+} Amm. Sulphate test solution is made up to the mark with distilled water. The solution is thoroughly shaken to make it homogeneous. 20 ml. of the test Fe^{2+} solution is pipetted out into clean conical flask. Approximately (1/2 Test tube) 10 ml. of dilute H_2SO_4 (6N) is added to it. It is titrated with KMnO_4 (taken in a burette) to a Faint Pink colour which persists for at least a minute. This is the end point. The process is repeated till two concordant titre values are obtained. From the titration data the amount of Fe^{2+} in the given test solution is estimated.

Part C : ESTIMATION OF Fe²⁺ IN THE GIVEN TEST SOLUTION.Titration : Fe²⁺(Test Soln) Vs. KMnO₄ (link)

Sl No.	V _{Fe²⁺} (test Sol.)ml	Burette Reading		V _{KMnO₄} (ml)
		Initial	Final	

$$N_{\text{Fe}^{2+}} = \frac{N_{\text{KMnO}_4} \times V_{\text{KMnO}_4}}{V_{\text{Fe}^{2+}}}$$

$$\text{Wt. of Fe}^{2+} \text{ (in Test Solution)} = N_{\text{Fe}^{2+}} \times \text{Eq. Wt.}$$

RESULTS:

- 1.Weight. of Oxalic Acid= _____g
- 2.N_{std.} Oxalic Aid= _____
- 3.N_{KMnO₄} (Link)= _____
- 4.N_{Fe²⁺} (Test)= _____
- 5.Weight of Fe²⁺
Present in test solution= _____gpl

Marks

Observations and calculations (20):_____

Results (10):_____

Discussion of results (5):_____

Record (15):_____

Total(50):_____

Signature of Faculty

QUESTIONS:

1. What is the difference in nature of Mohr's salt and oxalic acid?
2. Why is heating required in this experiment?
3. While adding KMnO_4 to oxalic acid solution, the decolouration is slow initially, why?
4. At room temperature which reaction has higher activation energy? FAS Vs KMnO_4 (or) oxalic acid Vs KMnO_4 . Justify the answer.
5. Which is the oxidizing and reducing agent in this experiment.
6. Name the auto catalyst in this experiment.

'REDOX' TITRATION
(DICHROMATOMETRY)

Experiment (4): Preparation of Standard Ferrous Ammonium Sulphate, Standardisation of K₂Cr₂O₇ (Link) Solution & Estimation of Fe⁺² in the given Test Solution:

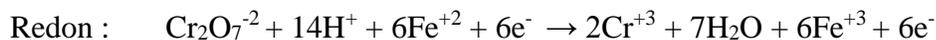
Aim: To estimate the amount iron of Fe⁺² in test solution by preparing a Standard FAS solution & using K₂Cr₂O₇.

Theory : FAS or Ferrous alum (Mohr's Salt) is a double salt of the combination FeSO₄(NH₄)₂SO₄.6H₂O this when dissolved in water breaks up into simple salts and water. Ferrous sulphate is a reducing substance. In the titration with a standard solution of K₂Cr₂O₇, the volume of oxidant (K₂Cr₂O₇) completely oxidizes ferrous iron Fe⁺² to Ferric Iron (Fe⁺³) is found out K₂Cr₂O₇ undergoes reduction because of high reduction potential. It oxidizes Fe⁺² (ous) to Fe⁺³ (ic) and in the process gets converted to Cr⁺³ which is green in colour even after equivalence point. In order to identify the equivalence point 3-5 drops of diphenylamine added as an internal indicator (E⁰_{Red} = 0.76v)]. In order to prevent the oxidation of diphenylamine before the oxidation of Fe⁺², Phosphoric acid is added which reduces the reduction potential of iron couple from 0.77v to 0.44v.] Diphenylamine is used as indicator. At the end point addition of excess dichromate oxidizes diphenylamine to diphenyl benzidine which is a deep violet colour (bluish violet)

Partial Ionic Equation



Overall Ionic equation :



Species:	(K ₂ Cr ₂ O ₇)	(H ₂ SO ₄)	(FeSO ₄)	Cr ₂ (SO ₄)	Fe ₂ (SO ₄) ₃
Color Profile:	(Orange)	(X)	(X)	(Greenish)	(X)

Equivalent weight = Molecular Wt./No. of e⁻ transfer)

Eq.wts. of : K₂Cr₂O₇ = 294.2/6 = 49.0:

Eq. wts. Of FeSO₄ – (NH₄)₂SO₄·6H₂O = 392.1/1, Fe⁺² = 55.85/1

In the presence of an acid mixture of H₂SO₄ and H₃PO₄, the reduction potential of Fe⁺³- Fe⁺² system is lowered to 0.61V due to complexion of Fe⁺² ion with PO₄⁻³ ion. Hence during titration, it is Fe⁺² ion that undergoes oxidation in preference to diphenylamine. The standard reduction potentials of Cr₂O₇⁻²-Cr⁺³ is 0.33V. Fe⁺³-Fe⁺² is 0.77v and diphenyl benzidine –diphenylane is 0.76v.

Experiment (4) : Preparation of standard Ferrous Ammonium Sulphate, Standardisation of $K_2Cr_2O_7$ (Link) Solution & Estimation of Fe^{+2} in given test solution:

Expt. No. _____

Date _____

TABULATION AND CALCULATIONS

Part A : Preparation of Std. FAS Solution :

Table.1.weight of bottle + FAS

--	--	--	--	--	--	--	--	--	--

Table.2.Weight of empty weighing bottle

--	--	--	--	--	--	--	--	--	--

Wt. of FAS = $W_1 - W_2 =$

$$N_{\text{std F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = \dots\dots\dots$$

($V_{ml} = 100ml$)

Part B : Standardization of $K_2Cr_2O_7$:

Titration of Std. F.A.S. Vs. $K_2Cr_2O_7$ (link)

Sl No.	V_{FAS} (ml)	Burette Reading		$V_{K_2Cr_2O_7}$ (ml)
		Initial	Final	

$$N_{K_2Cr_2O_7} = \frac{N_{FAS} \times V_{FAS}}{V_{K_2Cr_2O_7}}$$

Procedure:

PART A: PREPARATION OF STD. FAS SOLUTION:

Exactly _____g of FAS is weighed out in a clean weighing bottle. It is transferred to a 100ml standard Volumetric flask through a funnel, about half test tube dilute H_2SO_4 is added. The solution is made up to the mark with distilled water. It is shaken well to make homogenous. The normality of standard solution is calculated from the weighing data.

PART B: STANDARISATION OF $\text{K}_2\text{Cr}_2\text{O}_7$:

20 ml. of prepared solution of standard FAS is pipetted out into clean conical flask. Approximately 1/2 Test tube or 10 ml. of dilute H_2SO_4 (6N) is added to it and about 8ml or 1/3 tt of Syrupy Phosphoric acid is added followed by 3 to 5 drops of internal indicator diphenylamine. It is titrated with $\text{K}_2\text{Cr}_2\text{O}_7$ (taken in Burette) very slowly until the solution acquires a green colour which shows that the end point is near. The titration is continued now with dropwise addition of $\text{K}_2\text{Cr}_2\text{O}_7$ and good stirring until addition of a drop causes the formation of deep violet colour, which is the end point.

The titration is repeated till two concordant titre values are obtained. From the titration data normality of $\text{K}_2\text{Cr}_2\text{O}_7$ is calculated.

PART C: ESTIMATION OF Fe^{+2} (FERROUS) IRON IN GIVEN TEST SOLUTION:

The given Fe^{+2} test solution is made upto the mark with distilled water. The solution is thoroughly shaken to make it homogenous. 20ml of the test Fe^{+2} solution is pipette out in a clean conical flask. Follow the procedure mentioned in Part B

The process is repeated till two concordant titre values are obtained. From the data Normality of Fe^{+2} and the weight of Fe^{+2} in test solution is calculated.

Questions:

1. Why is $\text{K}_2\text{Cr}_2\text{O}_7$ not a self indicator?
2. Why is the colour of the contents of conical flask green before the end point?
3. Which substance gives the bluish violet colour at the end point?
4. What is the change in oxidation number chromium in this reaction?
5. What is the role of phosphoric acid in this experiment?
6. Which substance is used to reduce Ferric iron to Ferrous iron?

Part C : ESTIMATION OF Fe²⁺ IN THE GIVEN TEST SOLUTION.Titration : Fe²⁺(Test Soln) Vs. K₂Cr₂O₇ (Link)

Sl No.	V _{Fe²⁺} (test Sol.)ml	Burette Reading		V _{K₂Cr₂O₇} (ml)
		Initial	Final	

$$N_{\text{Fe}^{2+}}(\text{Test}) = \frac{N_{\text{K}_2\text{Cr}_2\text{O}_7} \times V_{\text{K}_2\text{Cr}_2\text{O}_7}}{V_{\text{Fe}^{2+}}} = \dots\dots\dots$$

$$\text{Wt. of Fe}^{2+} \text{ (in Test Solution)} = N_{\text{Fe}^{2+}} \times \text{Eq. Wt.}$$

Results:

- 1.Weight. of FAS = _____g
 2.N_{std.} FAS = _____
 3.N_{K₂Cr₂O₇} (Link) = _____
 4.N_{Fe²⁺} (Test) = _____
 5.Weight of Fe²⁺ Present in test solution = _____gpl

Marks	
Observations and calculations (20):	_____
Results (10):	_____
Discussion of results (5):	_____
Record (15):	_____
Total(50):	_____

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Experiment (5) : ESTIMATION OF Fe³⁺ (FERRIC) IRON FROM THE GIVEN SOLUTION MIXTURE USING STANDARD POTASSIUM DICHROMATE SOLUTION.

Expt No. _____

Date: _____

TABULATION AND CALCULATIONS

Part A : Preparation of Std. K₂Cr₂O₇ :

Table.1.weight of bottle + K₂Cr₂O₇

--	--	--	--	--

Table.2.Weight of empty weighing bottle

--	--	--	--	--

Wt. of K₂Cr₂O₇ = W₁-W₂ =

N

$$(\text{std}) \text{K}_2\text{Cr}_2\text{O}_7 = \frac{\text{wt}}{\text{eq.wt}} \times \frac{1000}{\text{Vml}} = \dots\dots\dots$$

(V_{ml} = 250ml)

Part B : ESTIMATION OF Fe²⁺ IN THE GIVEN TEST SOLUTION MIXTURE:

Sl No.	V _{Fe²⁺} (Test Sol) (ml)	Burette Reading		V K ₂ Cr ₂ O ₇ (ml)
		Initial	Final	

$$N_{\text{Fe}^{2+}} = \frac{N_{\text{K}_2\text{Cr}_2\text{O}_7} \times V_{\text{K}_2\text{Cr}_2\text{O}_7}}{V_{\text{Fe}^{2+}}} = \dots\dots\dots$$

$$\text{Wt. of Fe}^{2+} = N_{\text{Fe}^{2+}} \times \text{Eq. Wt. of Fe}^{2+} = \dots\dots\dots \times 55.8$$

$$= \dots\dots\dots \text{gpl}$$

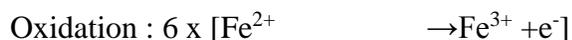
'REDOX' TITRATION
(DICHROMATOMETRY)

Experiment (5): ESTIMATION OF Fe³⁺ (FERRIC) IRON FROM THE GIVEN SOLUTION MIXTURE USING STANDARD POTASSIUM DICHROMATE SOLUTION.

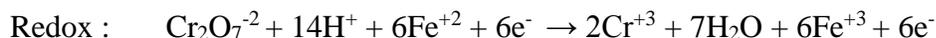
OBJECT: To estimate the Fe³⁺ from solution mixture using 0.1 N K₂Cr₂O₇ solution.

INTRODUCTION: (Theory) : Ferrous Ammonium Sulphate, Ferrous alum (Mohr's Salt) is a double salt of the combination FeSO₄ (NH₄)₂SO₄.6H₂O. This when dissolved in water breaks up into simple salts and water.

Ferrous sulphate is a reducing substance. In the titration with a standard solution of K₂Cr₂O₇ the reaction takes place in acidic medium provided by addition of dil. Sulphuric acid. Potassium dichromate is oxidizing substance it undergoes reduction because of high Reduction potential. It oxidizes ferrous iron (Fe²⁺) to Ferric Iron and in the process gets converted to Cr³⁺ which is green in colour even after equivalence point. In order to identify the equivalence point 3-5 drops of diphenylamine is added as an internal indicator (E⁰_{Red} = 0.76v)]. In order to prevent the oxidation of diphenylamine before the oxidation of Fe²⁺, Phosphoric acid is added which reduces the reduction potential of iron couple from 0.77v to 0.44v.



Overall Ionic equation :



Species:	(K ₂ Cr ₂ O ₇)	(H ₂ SO ₄)	(FeSO ₄)	(Cr ₂ (SO ₄) ₃)	
Color Profile:	(Orange)	(X)	(X)	(Greenish)	X=Colorless

Equivalent weight = Molecular Wt./No. of e⁻ transfer)

Eq.wts. of : K₂Cr₂O₇/6 = 294.2/6 = 49.0:

FeSO₄ (NH₄)₂SO₄.6H₂O/1 = 392.1/1, Fe²⁺ = 55.8)

PART C: ESTIMATION OF TOTAL IRON (FERROUS & FERRIC) IN THE GIVEN TEST SOLUTION MIXTURE:

Titration : Fe^{2+} (Test Soln) Vs. $\text{K}_2\text{Cr}_2\text{O}_7$ (Link)

Sl No.	$\text{V}_{\text{Fe}^{2+} \text{ \& } \text{Fe}^{3+}}$ (test Sol.)ml	Burette Reading		$\text{V}_{\text{K}_2\text{Cr}_2\text{O}_7}$ (ml)
		Initial	Final	

$$N_{\text{Fe}^{2+} \text{ \& } \text{Fe}^{3+}}(\text{Test}) = \frac{N_{\text{K}_2\text{Cr}_2\text{O}_7} \times V_{\text{K}_2\text{Cr}_2\text{O}_7}}{V_{\text{Total Iron}}} = \dots\dots\dots$$

$$\text{Wt. of Fe}^{2+} \text{ \& } \text{Fe}^{3+} \text{ (in Test Solution)} = N_{\text{Total Iron}} \times \text{Eq. Wt.} = \dots\dots\dots \times 55.8 \text{ gpl}$$

$$\text{THEREFORE THE AMOUNT OF Fe}^{3+} \text{ PRESENT} = \text{WT. OF TOTAL IRON} \text{ --- WT. OF Fe}^{2+} \\ = \dots\dots\dots \text{gpl.}$$

Since HgCl_2 does not undergo any change (silky white ppt) it remains in the solution. After reduction of ferric iron to ferrous iron to solution mixture contains Fe^{2+} originally present and Fe^{2+} obtained after reduction of Fe^{3+} . This solution containing total Fe^{2+} is titrated against std $\text{K}_2\text{Cr}_2\text{O}_7$ solution.

Precautions-

It should be noted that experiment is proceeded only when ppt is obtained. The solution is discarded in case, No ppt or grey/black ppt is obtained. If no ppt is obtained it is due to insufficient addition of SnCl_2 , if grey/black ppt is due to large excess of SnCl_2 . In either case, reject the solution and start with a fresh one.

PROCEDURE:

PART A: PREPARATION OF STD. $\text{K}_2\text{Cr}_2\text{O}_7$ SOLUTION

Weigh out in a clean weighing bottle, accurately close to 1.2250 g of $\text{K}_2\text{Cr}_2\text{O}_7$; dissolve it by adding distilled water in 250 ml standard volumetric flask and make up the solution up to the mark. Shake well to make it homogeneous.

PART B: ESTIMATION OF FERROUS IRON IN THE GIVEN TEST SOLUTION MIXTURE:

- 1) Make up the given (Fe^{+2} and Fe^{+3}) test solution mixture up to the mark by adding only distilled water.
- 2) Pipette out 20 ml. of this made up test solution into a conical flask.
- 3) Add half test tube (10ml) of dil H_2SO_4 solution, 3-5 drops of DPA indicator followed by $\frac{1}{4}$ test tube (5ml) of syrupy phosphoric acid.
- 4) Titrate this solution against Std $\text{K}_2\text{Cr}_2\text{O}_7$ solution until persistent deep violet colour appears near the end point.
- 5) Repeat the titration to obtain concordant values. Let the titre value be VI ml.
- 6) Calculate the amount of Ferrous iron present in the given test solution mixture.

PART C: ESTIMATION OF TOTAL IRON IN THE GIVEN TEST SOLUTION MIXTURE:

i) Reduction of Ferric iron to ferrous iron;

- 1) Pipette out 20ml of test solution mixture in a conical flask to perform reduction of ferric to ferrous iron.
- 2) Add 5ml of conc HCl and the heat the solution till it begins to boil.
- 3) To the hot solution stannous chloride(SnCl_2) is added dropwise from a burette until yellow colour of Fe^{+3} is discharged (becomes colourless). Now add 1 or 2 drops more SnCl_2 to ensure complete reduction. Avoid large excess.
- 4) The hot solution is cooled down to room temperature by holding the flask under tap. Dilute the solution by adding 40-50 ml of distilled water.
- 5) Add approximately one test tube (10ml) of saturated HgCl_2 (mercuric chloride) rapidly in one portion until a silky white ppt appears. The solution is allowed to stand for 5min. [if no ppt or black ppt appears, reject the solution and repeat the process]

(ii) Titration of total Fe⁺² against std K₂Cr₂O₇.

1) To the above solution, add half test tube (10ml) of dil H₂SO₄ solution, 3-5 drops of DPA indicator followed by ¼ test tube (5ml) of syrupy phosphoric acid.

2) Titrate this solution against Std K₂Cr₂O₇ solution until persistent deep violet colour appears near the end point.

3) Repeat the titration to obtain concordant values. Let the titre value be V₂ml.

4) Calculate the amount of total iron present in the solution.

5) Subtract the amount of ferrous iron (Part B) from amount of total iron (Part C) to estimate the Ferric iron (Fe⁺³) present in the given test solution mixture.

Results:

1. Weight. of K₂Cr₂O₇ = _____ g
2. K₂Cr₂O₇ solution = _____
3. Weight of Ferrous Iron = _____ g / lit.
4. Weight of Total Iron = _____ g / lit.
5. Weight of Fe³⁺
Present in test solution = _____ g / lit.

Marks	
Observations and calculations (20):	_____
Results (10):	_____
Discussion of results (5):	_____
Record (15):	_____
Total(50):	_____

Signature of Faculty

WATER ANALYSIS

‘COMPLEXOMETRIC’ TITRATION

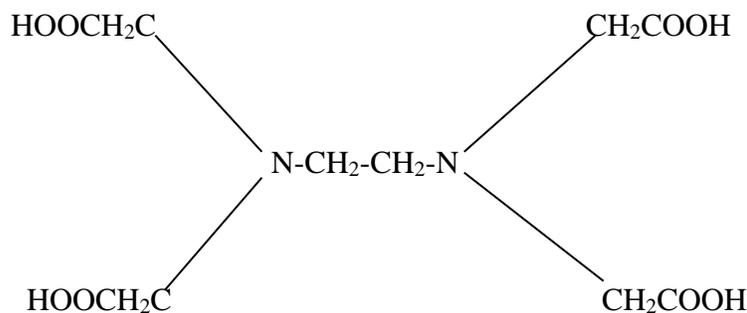
EXPERIMENT (6): ESTIMATION OF TEMPORARY & PERMANENT HARDNESS OF WATER USING EDTA SOLUTION.

Aim : To estimate the total hardness of water sample using EDTA solution.

INTRODUCTION: (THEORY): Hardness of water is a quantitative measure of the quality of water to judge its suitability for both drinking and industrial purposes. (Hard water causes health hazards, boiler sales etc). Based on the degree of hardness, suitable treatment can be recommended for water supply of effluents.

EDTA =Ethylene Di amine Tetra Acetic acid

EDTA has a very wide general application in analysis because of its powerful complexing action and commercial availability. In its unreacted form EDTA is a Tetra basic acid represented by:



(the disodium salt of EDTA available commercially is $\text{CH}_2\text{N}(\text{CH}_2\text{COOH})_2$
 $\text{CH}_2\text{N}(\text{CH}_2\text{COONa})_2 \cdot 2\text{H}_2\text{O}$ with formula wt=372.24. it is also written as Na_2H_2 versenate $2\text{H}_2\text{O}$
 i.e. disodium dihydrogen versenate)

TYPES OF HARDNESS IN WATER:

Temporary Hardness is due to the presence of soluble salts of Bicarbonates of Calcium and Magnesium. It can be removed by boiling.

Permanent Hardness is due to the presence of soluble salts of Chlorides and Sulphates of Calcium and Magnesium.

$$\text{Total Hardness} = \text{Temporary Hardness} + \text{Permanent Hardness}$$

The EDTA titration make use of Eriochrome Black 'T' as the indicator. The titration is sensitive to pH and therefore a buffer solution is used to maintain the best pH around 10. Beyond this pH 10, Mg is not sufficiently bound to the indicator (Eriochrome Black 'T') to give a sharp colour change at the end point.

The order of stability is as follows:

Ca –EDTA > Mg- EDTA > Mg – Indicator

(colourless)

(Wine – red)

When EDTA solution is added to hard water in the presence of Eriochrome Black 'T' indicator, it would first combine with free Ca^{2+} , then with free Mg^{+2} ions; and then with Mg^{2+} liberated from the Mg-Indicator complex (Wine-red) leaving the indicator aside, which will be blue.

Mg – Indicator + EDTA \rightarrow Mg- EDTA + Indicator

(wine-red)

(colourless) (blue)

PREPARATION OF SOLUTIONS:

1. **Standard hard water:** weigh out accurately 1.000 g. dried and pure (A.R.) CaCO_3 . Convert it to CaCl_2 by dissolving in minimum dilute (~6N) HCl. Make it up to 1 litre solution in distilled water. The strength is 1000 ppm.

2. **Buffer Solution:** 67.5 g. of NH_4Cl + 570 ml of conc. NH_4OH , made upto 1 litre with distilled water.

3. **Indicator:** or Dissolve 0.5 g. of Eriochrome black 'T' in 100 ml of alcohol or methanol.

4. **EDTA Solution:** 4.0 g. of disodium salt EDTA (Na_2H_2 ver. $2\text{H}_2\text{O}$) dissolved in distilled water and made to 1 litre.

PROCEDURE:

PART-A STANDARDIZATION OF EDTA:

i) Pipette out 20 ml of std. hard water into a conical flask.

ii) Add 5 ml of buffer solution and a few drops of Eriochrome Black 'T' indicator. The indicator, which is originally blue would assume a wine-red colour.

iii) Titrate with EDTA solution taken in the burette, till the wine-red colour changes to blue. This is the end point. Let this reading be V_1 ml of EDTA.

PART-B ESTIMATION OF TOTAL HARDNESS: Pipette out 20ml of sample water into a clean conical flask. Follow the procedure in PART-A. Let the reading obtained be V_2 ml. of EDTA.

PART-C ESTIMATION OF PERMANENT HARDNESS: Take about 100ml or 60ml of sample hard water in a beaker. Boil it to about one-fourth of its original volume to remove temporary hardness. Filter the boiled water through Whatman 42 filter paper into a clean beaker so that insoluble salts CaCO_3 and MgCO_3 are removed. Add distilled water in the filtered solution to make it to the original volume. Make the solution homogeneous. Now pipette out 20 ml of this boiled filtered water sample into a clean conical flask. Follow the procedure in PART-A of the experiment. Let the volume of EDTA be V_3 ml.

A simplified calculation would be as follows:

CALCULATIONS

$$\text{Total Hardness} = \frac{V_2}{V_1} \times 1000 = \text{ppm.}$$

$$\text{Permanent Hardness} = \frac{V_3}{V_1} \times 1000 = \text{ppm.}$$

$$\text{Temporary Hardness} = \frac{V_2 - V_3}{V_1} \times 1000 = \text{ppm.}$$

(difference)

(Round off the results to the nearest integer)

[Note: In this experiment, the standardization of EDTA could also be done by preparing a standard Hard water solution using $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, M.W .246.5 g]

Question:

1. To a 100ml water sample 58.5mg of NaCl is added. What would be the hardness of sample Water ?
2. How would you differentiate between distilled water and hard water sample ?

*He who has imagination without learning has wings but no feet.

EXPT. (6) ESTIMATION OF HARDNESS OF WATER – EDTA METHOD

Date:

Expt. No.

PART A: STANDARDISATION OF EDTA

Titration of Std. hard water Vs EDTA

Sl No.	V _{Std. hard water} (ml)	Burette Reading		V _{EDTA} ml (V ₁)
		Initial	Final	

V₁ ml of EDTA = 20ml Std. hard water1ml of EDTA = ? 1ml EDTA = $\frac{20}{V_1}$ = -----mg CaCO₃**PART B: ESTIMATION OF TOTAL HARDNESS OF SAMPLE WATER**

Titration of sample hard water Vs EDTA

Sl No.	V. Sample water ml	Burette Reading		V _{EDTA} ml (V ₂)
		Initial	Final	

Total hardness = $\frac{V_2}{V_1} \times 1000 = \dots\dots\dots = \dots\dots\dots$ ppm.

PART C: ESTIMATION OF PERMANENT HARDNESS

Titration of boiled hard water Vs EDTA

Sl No.	V _{boiled water} ml	Burette Reading		V _{EDTA} ml (V ₃)
		Initial	Final	

$$\text{Permanent hardness} = \frac{V_3}{V_1} \times 1000 = \dots\dots\dots = \dots\dots\dots \text{ppm.}$$

$$\text{Temporary hardness} = (\text{Total-permanent}) \text{ hardness} = \dots\dots\dots = \dots\dots\dots \text{ppm.}$$

PART D: ESTIMATION OF TOTAL HARDNESS OF MUNICIPAL/DISTILLED WATER

Sl No.	V _{Municipal water} ml	Burette Reading		V _{EDTA} ml (V ₄)
		Initial	Final	

$$\text{Hardness} = \frac{V_4}{V_1} \times 1000 = \dots\dots\dots = \dots\dots\dots \text{ppm.}$$

RESULTS:

- 1) Source: O/W, B/W = _____.
- 2) Depth (approximately) = _____.
- 3) Locality = _____.
- 4) Total Hardness = _____.
- 5) Permanent Hardness = _____.
- 6) Temporary Hardness = _____.
- 7) Total Hardness of Municipal/ Distilled water = _____.

Marks

Observations and calculations (20): _____

Results (10): _____

Discussion of results (5): _____

Record (15): _____

Total(50): _____

Signature of Faculty

**EXPERIMENT (7) : ESTIMATION OF TOTAL HARDNESS OF WATER USING
STD. MgSO₄ AND EDTA SOLUTION**

Expt. No. _____

Date: _____

TABULATION AND CALCULATIONS

Part A : Preparation of Std. MgSO₄

Table.1.weight of bottle + MgSO₄

	.				
--	---	--	--	--	--

Table.2.Weight of empty weighing bottle

	.				
--	---	--	--	--	--

Wt. of MgSO₄ = W₁-W₂ =

$$N \text{ std MgSO}_4 = \frac{wt}{eq.wt} \times \frac{1000}{Vml} = \dots\dots\dots$$

(V_{ml} = 100ml)

Part B : Standardisation of EDTA

Sl No.	V.Std MgSO ₄ Sol ml	Burette Reading		V _{EDTA} MI (V ₁)
		Initial	Final	

$$N_{EDTA} = \frac{N_{MgSO_4} \times V_{MgSO_4}}{V_{EDTA}} = \dots\dots\dots$$

WATER ANALYSIS
'COMPLEXOMETRIC' TITRATIONS

EXPERIMENT (7) : ESTIMATION OF TOTAL HARDNESS OF WATER USING

STD. MgSO₄ AND EDTA SOLUTION.

Aim : To estimate the total hardness of water sample using std MgSO₄ solution and EDTA solution.

Theory : (Refer to Experiment No. 6)

PROCEDURE :

PART-A: PREPARATION of STD. MgSO₄ SOLUTION.

Weigh accurately the given MgSO₄. 7H₂O (Epson salt) in a weighing bottle and let its weight be W₁ gms. Transfer the salt into a clean 100ml standard volumetric flask using funnel. Take the Weight of empty bottle and note down its weight as W₂ gms. Dissolve the substance in minimum Quantity of distilled water and the make solution upto the mark with distilled water. Shake the solution thoroughly to get uniform concentration. Calculate the normality of standard MgSO₄ solution.

PART-B : STANDARDISATION OF EDTA SOLUTION :

Pipette out 20ml of the standard hardwater into a clean conical flask. Add about 5ml of (NH₄ Cl+NH₃) buffer solution of pH = 10 and 3-4 drops of EBT indicator to it. The color of the solution changes to wine red. Titrate the solution against EDTA solution taken in the burette until the wine red colour of the solution changes to blue. Note down the burette reading and repeat the titration to get concordant titre values.

PART-C:ESTIMATION OF TOTAL HARDNESS OF WATER :

Pipette out 20 ml of given sample of water into a clean conical flask. Add about 5 ml of (NH₄Cl+NH₃) buffer solution of pH = 10 and 3 - 4 drops of EDTA solution taken in the burette

Until the wine red solution changes to blue. Note down the burette reading and repeat the titration to get concordant titre values

PART C: ESTIMATION OF TOTAL HARDNESS OF TEST SOLUTION

Titration of Test Solution Vs EDTA

Sl No.	V. Test Solution ml	Burette Reading		V _{EDTA} ml (V ₂)
		Initial	Final	

$$N_{\text{Test solution}} = \frac{N_{\text{EDTA}} \times V_{\text{EDTA}}}{V_{\text{test solution}}} =$$

$$\text{Wt in terms of CaCO}_3 \text{ equivalents} = N_{\text{test solution}} \times \text{Eq. wt CaCO}_3$$

$$= \text{_____} \times 50 = \text{_____ gpl.}$$

$$\text{Total hardness of Test Solution} = \text{_____} \times 1000 = \text{_____ mg/lit} = \text{_____ ppm.}$$

RESULTS:

- 1) Wt. of MgSO₄ = _____.
- 2) Normality of MgSO₄ = _____.
- 3) Normality of EDTA = _____.
- 4) Normality of Test Solution = _____.
- 5) Total Hardness in Test Solution = _____ ppm

Marks	
Observations and calculations (20):	_____
Results (10):	_____
Discussion of results (5):	_____
Record (15):	_____
Total(50):	_____

Signature of Faculty

WATER ANALYSIS

ACID-BASE TITRATION

Expt (8): ESTIMATION OF CARBONATE AND BICARBONATE ALKALINITY IN WATER

INTRODUCTION:

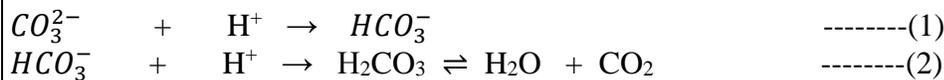
Alkalinity of natural waters may be attributed to the presence of salts of weak acids, such as bicarbonates and carbonates etc. Highly alkaline waters may lead to caustic embrittlement and deposition of precipitates and sludges in boilers. Bicarbonates of calcium and magnesium induce temporary hardness in water, which, if untreated, causes scale formation.

For water softening processes as well as boiler feed water analysis, it is essential have an idea about the nature and extent of alkalinity present.

The type and extent of alkalinity present in a water sample may be conveniently determined by titrating an aliquot of the sample with a standard acid to phenolphthalein end-point, [P], and then continuing the titration to methyl orange end-point, [M].

PRINCIPLE:

The reaction taking place may be represented by the following equations.



The volume of acid run down upto phenolphthalein end-point, [P] corresponds to the completion of equation (1) given above, while the volume of acid run down after [P], corresponds to the completion of equation (2). The total amount of acid used from the beginning of the experiment, i.e., [M] corresponds to the total alkalinity and represents the completion of reaction shown by equations (1) and (2).

Alkalinity is generally expressed as parts per million (ppm) in terms of $CaCO_3$.

Equivalent wt of $CaCO_3 = 100/2 = 50$, $CO_3^{2-} = 30$, $HCO_3^- = 61$

*Its not what you look at that matters, its what you see .

Reagents required:

- i) Standard HCl (N/50)
- ii) Phenolphthalein indicator
- iii) Methyl orange indicator

PROCEDURE:**PART A: PREPARATION OF STANDARD N/50 Na₂CO₃ SOLUTIONS:**

Weigh out in a clean weighing bottle accurately to about 0.2622g of (A.R.) Na₂CO₃; transfer it to a clean 250 ml. standard volumetric flask through a funnel; dissolve it in distilled water and make up the solution to the mark. Shake well to make it homogeneous – calculate the normality of the standard Na₂CO₃ solution.

$$N_{Na_2CO_3} = \frac{Wt.}{Eq.Wt.} \times \frac{1}{V} \times 1000 = \frac{Wt.}{53} \times \frac{1}{V} \times 1000$$

PART B: STANDARDISATION OF HCl (~N/50)

Pipette out 20ml of the prepared std. Na₂CO₃ solution into a clean conical flask. Add 2 drops of methyl orange indicator. The solution becomes yellow. Titrate with the given HCl (taken in the burette) to a initial light pink appears, this is the end point. Repeat the process of titration till at least two concordant values are obtained.

$$N_{HCl} = \frac{N_{Na_2CO_3} \times V_{Na_2CO_3}}{V_{HCl}} = \frac{N_{Na_2CO_3} \times 20}{V_{HCl}}$$

PART C: ESTIMATION OF CO₃²⁻ & HCO₃⁻ ALKALINITY IN TEST SOLUTION

Make up the given test solution upto the mark by adding distilled water. Make the solution homogeneous. Pipette 20 ml of test solution containing both CO₃²⁻ & HCO₃⁻ in a clean conical flask.

- i) Add 2 drops of phenolphthalein indicator, and titrate the sample with standard HCl. Until the pink colour just disappears-note the titre value as phenolphthalein end point [P].
- ii) Add 2 drops of methyl orange indicator to the same solution and continue the titration until colour changes from yellow to red or pink. Note the titre value as [M] end point

A Typical Titration Table: Estimation of Carbonate and Bi-Carbonate Alkalinity in water

S.No	Vol. of Water sample (ml)	Vol. of N/50 HCl run down (ml)			
		Phenolphthalein End point[P]	2 [P]	Methyl Orange end-point, [M]	M-2[P]
1	100	10.5	21.0	25.8	4.8
2	100	10.4	20.8	25.7	4.9
3	100	10.4	20.8	25.7	4.9

(Start the titration with '0' ml. mark on the burette)

Now, [P] = 10.4 ml; [M] = 25.7 ml, then 2 [P] should correspond to CO_3^{2-} and [M] - 2 [P] should correspond to HCO_3^- . Thus, vol. of N/50 HCl equivalent to $HCO_3^- = 2[P] = 2 \times 10.4 \text{ ml} = 20.8 \text{ ml}$ and vol. of N/50 HCl equivalent to $HCO_3^- = [M] - 2 [P] = 25.7 - 20.8 \text{ ml} = 4.9 \text{ ml}$.

CALCULATION: For estimation of carbonate and bicarbonate alkalinity in sample water

i) CO_3^{2-} :

$$NCO_3^{2-} = \frac{N_{HCl} \times V_{HCl}}{VCO_3^{2-}} = \frac{0.02 \times 20.8}{100} = 0.00416$$

$$\text{Wt. of } CO_3^{2-} = \frac{NCO_3^{2-} \times \text{eq. wt}}{g/l} = 0.00416 \times 30 \text{ g/l} = 0.1248 \text{ g/l}$$

$$\text{Wt. of } CaCO_3 \text{ eqt.} = 0.124 \times \frac{50}{30} \text{ g/l} = 0.208 \text{ g/l} = 208 \text{ mg/l} = 208 \text{ ppm.}$$

ii) HCO_3^-

$$NHCO_3^- = \frac{N_{HCl} \times V_{HCl}}{VHCO_3^-} = \frac{0.02 \times 4.9}{100} = 0.00098$$

$$\text{Wt. of } HCO_3^- = \frac{NHCO_3^- \times \text{eq. wt.}}{g/l} = 0.00098 \times 61 \text{ g/l} = 0.05978 \text{ g/l} = 59.7 \text{ mg/l} = 60 \text{ ppm}$$

Experiment (8) : ESTIMATION OF CARBONATE AND BICARBONATE ALKALINITY IN WATER

Expt No. ____

Date: _____

Part A : Preparation of Std. Na₂CO₃:

Table.1. weight of bottle + Na₂CO₃

	.			
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Table.2. Weight of empty weighing bottle

	.			
--	---	--	--	--

Wt. of Na₂CO₃ = W₁-W₂ =

N

$$\text{Std Na}_2\text{CO}_3 = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = \dots\dots\dots$$

(V_{ml} = 100ml)

Part B : STANDARDIZATION OF HCl LINK SOLUTION

Sl No.	V _{Na₂CO₃} (Std.) ml	Burette Reading		V _{HCl} ml
		Initial	Final	

$$N_{\text{HCl}} = \frac{N_{\text{Na}_2\text{CO}_3} \times V_{\text{Na}_2\text{CO}_3}}{V_{\text{HCl}}} = \dots\dots\dots$$

PART – C: ESTIMATION OF ALKALINITY IN TEST SOL.Table: Estimation of CO_3^{2-} & HCO_3^- Alkalinity in test sol.

S.No	Vol. of Water sample (ml)	Vol of N/50 HCl run down (ml)			
		Phenolphthalein End point[P]	2 [P]	Methyl Orange end-point, [M]	M-2[P]
1	20				
2	20				

CALCULATION:-**ESTIMATION OF CO_3^{2-} ALKALINITY**

$$N_{CO_3^{2-}} = \frac{N_{HCl} \times V_{HCl}}{V_{CO_3^{2-}}}$$

$$\text{Wt. of } CO_3^{2-} = N_{CO_3^{2-}} \times \text{eq.wt of } CO_3^{2-} = \text{_____} \times 30 = \text{_____ gpl}$$

$$\begin{aligned} \text{Wt.in terms of CaCO}_3 \text{ eq} &= \text{Wt. of } CO_3^{2-} \times \text{eq wt of CaCO}_3 / \text{eq wt of } CO_3^{2-} \\ &= \text{_____} \times 50 / 30 = \text{_____ gpl} \\ &= \text{_____ mg/L} = \text{_____ ppm} \end{aligned}$$

ESTIMATION OF HCO_3^- ALKALINITY

$$N_{HCO_3^-} = \frac{N_{HCl} \times V_{HCl}}{V_{HCO_3^-}}$$

$$\text{Wt. of } HCO_3^- = N_{HCO_3^-} \times \text{eq wt of } HCO_3^- = \text{_____} \times 61 = \text{_____ gpl}$$

$$\begin{aligned} \text{Wt.in terms of CaCO}_3 \text{ eq} &= \text{Wt. of } HCO_3^- \times \text{eq wt of CaCO}_3 / \text{eq wt of } HCO_3^- \\ &= \text{_____} \times 50 / 61 = \text{_____ gpl} \\ &= \text{_____ mg/L} = \text{_____ ppm} \end{aligned}$$

RESULTS:**FOR TEST SOLUTIONS:-**

- 1) [P] =ml; [M]=ml; [M-2(P)]=.....ml
- 2) NCO_3^{2-} =.....
- 3) Wt. of CO_3^{2-} in mixture =gpl.
- 4) Carbonate Alkalinity =ppm
- 5) NHCO_3^- =
- 6) Wt. of HCO_3^- in mixture =gpl.
- 7) Bicarbonate Alkalinity =ppm
- 8) Total Alkalinity in the given test soln =ppm

QUESTIONS:

- 1) Suggest a better alternative indicator to methyl orange in this experiment.
- 2) Total alkalinity of a sample water is 860 ppm while temporary hardness is 120 ppm. What is alkalinity due to HCO_3^- ?

Marks	
Observations and calculations (20):	_____
Results (10):	_____
Discussion of results (5):	_____
Record (15):	_____
Total(50):	_____

Signature of Faculty

**EXP.No.-9-PREPARATION OF STANDARD SOLUTION OF POTASSIUM
DICHROMATE-STANDARDIZATION OF MOHR SALT SOLUTION-
DETERMINATION OF CHEMICAL OXYGEN DEMAND**

Expt. No _____

Date:

PART A: PREPARATION OF STANDARD $K_2Cr_2O_7$

Table.1.weight of bottle + $K_2Cr_2O_7$

		•			
--	--	---	--	--	--

Table.2.Weight of empty weighing bottle

		•			
--	--	---	--	--	--

Wt. of $K_2Cr_2O_7 = W_1 - W_2 =$

N

$$\text{std } K_2Cr_2O_7 = \frac{wt}{eq.wt} \times \frac{1000}{Vml} = \dots\dots\dots$$

($V_{ml} = 100ml$)

Part B : Standardisation of FAS LINK SOLUTION

Titration of Std. $K_2Cr_2O_7$ vs FAS solution

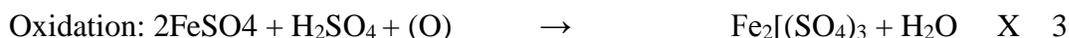
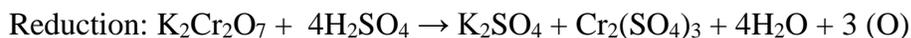
Sl No.	V_{FAS} (Std) ml	Burette Reading		$V_{K_2Cr_2O_7}$ ml
		Initial	Final	

$$N_{FAS} = \frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{FAS}} = \dots\dots\dots$$

EXP. (9)-PREPARATION OF STANDARD SOLUTION OF POTASSIUM DICHROMATE-STANDARDIZATION OF MOHR SALT SOLUTION – DETERMINATION OF CHEMICAL OXYGEN DEMAND.

OBJECT: to estimate the chemical oxygen demand (cod) of waste water.

Introduction: COD measures the total oxidisable impurities present in the sewage this include both biologically oxidisable and biologically inert which are only chemically oxidisable impurities present in the water, with a strong chemical oxidant $K_2Cr_2O_7$ in acidic medium. It is expressed in ppm.



PROCEDURE

PART A: PREPARATION OF STANDARD $K_2Cr_2O_7$

Weigh out in a clean weighing bottle, accurately close to 1.2250 g of (A.R) $K_2Cr_2O_7$; dissolve it by adding distilled water in 250 ml standard volumetric flask and make up the solution up to the mark. Shake well to make it homogeneous.

PART B: STANDARDIZATION OF FAS SOLUTION (LINK)

- i) Pipette out 20 ml. of given Ferrous Ammonium sulphate solution into a conical flask.
- ii) Add 10 ml of dilute H_2SO_4 (6N) and then 3 to 5 drops of internal indicator diphenylamine; (1% solution in concentrated H_2SO_4), followed by about 5 ml ($1/4^{\text{th}}$ Test tube) of syrupy phosphoric acid which lowers the reduction potential of Iron couple and facilitates the initial oxidation of Ferrous to Ferric Ions.
- iii) Now titrate very slowly with std. $K_2Cr_2O_7$. The solution initially acquires green colour due to formation of Cr^{+3} , its intensity increases the solution acquires bluish green tint which shows that the end pot is near. The Titration is continued now with dropwise addition of K_2CrO_7 and good stirring until addition of a drop causes the formation of an intense bluish violet colour at the end point. Bluish violet colour is due to oxidation of DPA by K_2CrO_7 which is stable. Repeat the titration to obtain at least two concordant values.

PART C: DETERMINATION OF COD:

Pipette out 20ml of test/waste water sample into a 250 ml conical flask with a round bottom flask (rbf), add 1 gm of HgSO_4 , followed by test tubes of silver sulphate or Sulphuric acid solution (prepared by dissolving about 5g of Ag_2SO_4 in 250 ml of concentrated sulphuric acid and then slowly diluting to 500ml with distilled water, then pipette out 20ml of $\text{K}_2\text{Cr}_2\text{O}_7$ solution into the flask fit the flask with the reflex condenser and reflux the mixture for about half an hour on cooling, rinse the inside of the condenser with distilled water into the flask. add 2-3 drops of ferroin indicator and titrate with standard Mohr's salt solution till the colour changes from bluish green to reddish brown.

Blank Titration: Repeat the above procedure by taking 20 ml of distilled water in place of sample waste water.

CALCULATIONS:

Volume of mohr salt consumed by excess of $\text{K}_2\text{Cr}_2\text{O}_7$ after oxidizing impurities in sample water = $V_1 = \underline{\hspace{2cm}}$ ml.

Volume of mohr salt consumed in Black Titration = $V_2 = \underline{\hspace{2cm}}$ ml

20 ml of $\text{K}_2\text{Cr}_2\text{O}_7$ solution + 20 ml of water sample = V_1 ml of N_1 N mohrs salt solution.

20 ml of $\text{K}_2\text{Cr}_2\text{O}_7$ alone = V_2 ml of N_1 N mohrs salt solution

Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution consumed by oxidisable impurities in terms of volume of mohrs salt solution = $(V_2 - V_1)$ ml = $\underline{\hspace{2cm}}$

1000 ml of 1N mohrs salt solution = 8gm of oxygen

1ml of 1N mohrs salt solution = $8/1000$ gm of oxygen

$(V_2 - V_1)$ ml N_1 N mohrs salt solution, $W_3 = 8(V_2 - V_1) \times N_1 / 1000$ g of oxygen

= wt of COD in 25 ml of waste water

= $\underline{\hspace{2cm}}$ gm

= $\underline{\hspace{2cm}}$ mg

COD of Sample = $W_3/20 \times 1000$

= $\underline{\hspace{2cm}}$ ppm.

= $\underline{\hspace{2cm}}$

RESULTS:

- 1) Weight of K_2CrO_7 = _____
- 2) Normality of K_2CrO_7 = _____
- 3) Normality of FAS = _____
- 4) COD of given water sample = _____

Marks

Observations and calculations (20): _____

Results (10): _____

Discussion of results (5): _____

Record (15): _____

Total(50): _____

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Table-I Elements and Atomic weight

S.No	Element	Atomic weight
1	H	1
2	C	12
3	N	14
4	O	16
5	Na	23
6	Mg	24
7	S	32
8	Cl	35.5
9	K	39
10	Ca	40
11	Cr	52
12	Mn	55
13	Fe	55.5~56
14	Cu	63.5

Table-II List of Chemicals with molecular formula and molecular weight

S.No	Name and Molecular formula of substance	Molecular Weight
1	Ferrous Ammonium Sulphate(FAS) Mohr's Salt. [FeSO ₄ (NH ₄) ₂ SO ₄ .6H ₂ O]	392.16
2	Potassium Permanganate [KMnO ₄]	158
3	Potassium Dichromate [K ₂ Cr ₂ O ₇]	294
4	Oxalic Acid [H ₂ C ₂ O ₄ .2H ₂ O]	126
5	Sodium thio sulphate [Na ₂ S ₂ O ₃ .5H ₂ O]	248.18
6	Copper sulphate [CuSO ₄ .5H ₂ O]	249.68
7	Sodium Carbonate [Na ₂ CO ₃]	106
8	Magnesium sulphate [MgSO ₄ .7H ₂ O]	246.5
9	Hydrochloric Acid [HCl]	36.5
10	Acetic Acid [CH ₃ COOH]	60

Viva Questions

I FAS Vs KMnO₄ Vs Fe⁺²

- 1) Write the chemical name and formula of FAS.
- 2) What is the reaction taking place between FAS and KMnO₄
- 3) Why dil.H₂SO₄ is added to prepare std. FAS solution in Volumetric flask.
- 4) Why dil.H₂SO₄ is added to FAS during the titration of FAS Vs KMnO₄ in conical flask.
- 5) Why indicator is not required for FAS Vs KMnO₄ titration.
- 6) Which substance gives pale pink colour at the end point.
- 7) What is the colour of Mn⁺² solution.
- 8) Name the oxidizing and reducing agents in this titration.
- 9) What is the change in oxidation number of Mn, when KMnO₄ changes to Mn⁺²
- 10) Name the primary and secondary standard solutions in this experiment.

II **Oxalic Acid Vs KMnO₄ Vs Fe⁺²**

- 1) What is the molecular formula of Oxalic acid? What type of compound is oxalic acid?
- 2) Which is the primary and secondary standard solutions. Why are they called so?
- 3) Name the Oxidizing and reducing agents in this experiment.
- 4) Why Oxalic acid is heated before titrating with KMnO₄?
- 5) What is the catalyst in this experiment?.
- 6) What are the factors affecting rate of reaction in this experiment?.
- 7) What is the difference between FAS Vs KMnO₄ and Oxalic acid Vs KMnO₄ titration.
- 8) At room temperature which reaction has high Activation energy? FAS Vs KMnO₄ (or) Oxalic acid Vs KMnO₄.
- 9) What is the difference between nature of Mohrs Salt and Oxalic Acid.
- 10) What is the indicator used in this titration? Why?
- 11) What is the change in oxidation number of Mn when KMnO₄ undergoes reduction.

III FAS Vs K₂Cr₂O₇ Vs Fe⁺²

- 1) What is meant by redox titration?
- 2) What is dichrometry and permanganometry?
- 3) What is the oxidation number of Cr in K₂Cr₂O₇.
- 4) Why the acid mixture (H₂SO₄ and H₃PO₄) are added during titration?
- 5) Name the indicator used in K₂Cr₂O₇ titration?
- 6) What is the colour change at the end point? Why is it so?
- 7) What is the difference between titration of KMnO₄ and K₂Cr₂O₇ with FAS?
- 8) Name the primary standard solution, link solution and test solution in this experiment?.
- 9) Can Potassium permanganate be used as primary standard solution?
- 10) Name the species giving green colour and blue colour in the titration?
- 11) Why K₂Cr₂O₇ do not act as self indicator, even though it is a coloured substance.

IV ESTIMATION OF HARDNESS BY EDTA METHOD

- 1) What is the difference between hard water and soft water?
- 2) What are the ions causing temporary and permanent hardness?
- 3) How is temporary hardness removed? What is the chemical change.
- 4) Write the structure and name of EDTA. Why it is used in estimation of hardness.
- 5) Name the reagents and indicator used?
- 6) Why Buffer solution is added to the solution?
- 7) What is the order of stability of complexes formed during titration?
- 8) What is the temporary hardness in ppm, if sample water contains 1.62 mg of $\text{Ca}(\text{HCO}_3)_2$ and 1.32 mg of Mg Cl_2 ?
- 9) Define ppm
- 10) What are the disadvantages of hard water?

V **ESTIMATION OF ALKALINITY IN WATER**

- 1) Alkalinity in water is due to presence of which ions?
- 2) What are the possible combinations of ions causing alkalinity in water?
- 3) What are the indicators used in this experiment?
- 4) Why alkalinity and hardness is measured in terms of CaCO_3 equivalents?
- 5) Temporary hardness is also known as _____ alkalinity.
- 6) Write the neutralization reactions for carbonate and bicarbonate ions.
- 7) What is the significance of [2P] end point?
- 8) Write the reactions involved in this acid-base titration?
- 9) If [P]=0, then alkalinity is due to _____ ions.
- 10) Alkalinity in water cannot be due to simultaneous presence of OH^- , CO_3^{2-} & HCO_3^- , why?