

MJ CET

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/IV SEMESTER 1

ACADEMIC YEAR 2016-2017

VOLUME I : VOLUMETRIC ANALYSIS

Name: _____

Branch (section): _____

Roll No. 1604 - 16 - _____

MJCET

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CONTENTS

VOLUMETRIC ANALYSIS

TOPICS	PAGE NO.
1) SAFTEY INSTRUCTIONS	05
2) INTRODUCTION	06
3) DEMO EXPERIMENT	10
4) PERMANGANOMETRY	
Std FAS X KMnO_4	14
Std FAS X KMnO_4 X Fe^{2+}	17
Std O.A. X KMnO_4 X Fe^{2+}	20
5) DICHROMOMETRY	
Std FAS X $\text{K}_2\text{Cr}_2\text{O}_7$ X Fe^{2+}	24
Std $\text{K}_2\text{Cr}_2\text{O}_7$ & Estimation of Fe^{3+}	28
Std $\text{K}_2\text{Cr}_2\text{O}_7$ X FAS X COD	47
6) COMPLEXOMETRY	
Estimation of Hardness using EDTA	33
Std. MgSO_4 X EDTA X Total Hardness	38
7) ACIDMETRY	
Estimation of CO_3^{2-} & HCO_3^- Alkalinity	41
Std. Na_2CO_3 X HCl X CO_3^{2-} & HCO_3^-	44

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, use ventilating hoods. 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 in and inhale vapour is 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. Do not throw them into the wastepaper container or regular waste 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 amounts 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 and concentrate behind the lenses. If you are using contact lenses consult with your instructor. If possible try to wear a prescribed glasses under your safety goggles. In case of any accident that a chemical splashes near your eyes, immediately wash your eyes with lot of water and inform your instructor. Especially, when heating a test tube do not point its mouth to anyone or you.

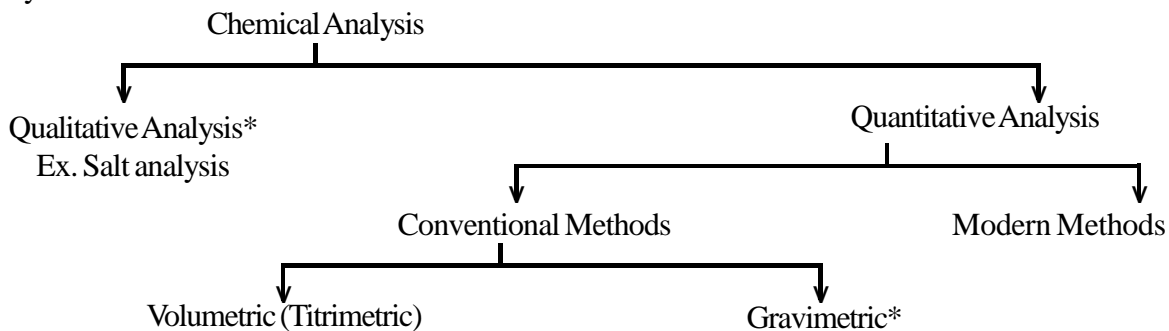
6) Fire: In case of fire or an accident, inform your instructor Immediately. Note the location of fire extinguishers as soon as you enter the laboratory so that you may use them if needed. Never perform an unauthorized experiment in the laboratory.

MUFFAKHAM JAH

COLLEGE OF ENGINEERING & TECHNOLOGY
(SULTAN-UL--ULOOM EDUCATION SOCIETY)

(VOL. 1 A MANUAL ON VOLUMETRIC ANALYSIS)

Note : This manual should be studied thoroughly for each experiment before going to the Laboratory.



(*Not in Syllabus)

INTRODUCTION : Volumetric analysis or Titrimetric analysis, just as gravimetric analysis gives a quantitative estimation of species. It involves a measurement of the volume of a solution of known concentration (standard solution) that is required to react completely with the species to be estimated. This method is applicable to fast reactions 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).

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), $\text{Fe SO}_4(\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$ (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

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:
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 :

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

Where N=Normality of the solution

V_{ml} = Volume of standard flask in ml

$$N_1 \times V_1 = N_2 \times V_2 \dots \dots \dots (2)$$

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:

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

* 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:

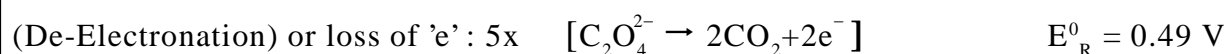
"Equivalent weights of species (substances) are derived from the balanced ionic (or molecular) equation of a particular reaction so that it involves one-electron transfer".

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

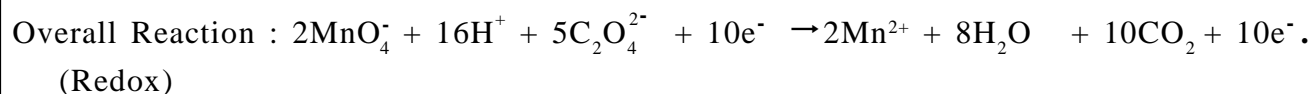
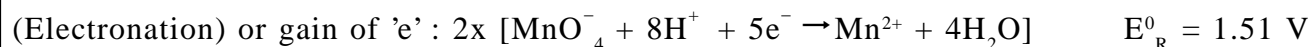
A redox reaction can be written as two parts or two half reactions, one indicating oxidation and the other indicating reduction. However, it should be clearly understood that the Redox process is a 'Simultaneous' process. **It takes place in the presence of both the oxidant (Oxidizing agent) and the Reductant (Reducing agent) at once 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 .

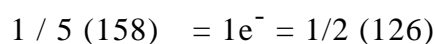
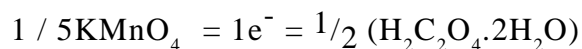
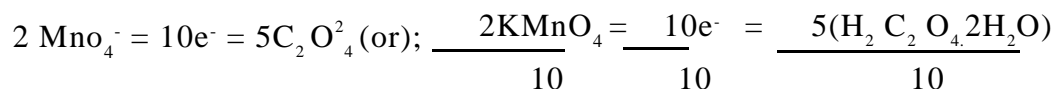
Half reaction : Oxidation :



Half reaction : Reduction :



In the above reaction it may be noted that the oxidant KMnO_4 gets reduced itself because of its High Reduction Potential ($E^0_{\text{R}} = 1.51\text{V}$) 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 ($E^0_{\text{R}} = 0.49\text{V}$). The equivalence established is as follows:



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 $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (Oxalic acid) and standardisation 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 our experimentation would now be briefly described.

If we wish to determine the normality of a solution (test solution) and thereby 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.

	Standard	link	Test
SYSTEM - I	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (Reductant)	KMnO_4 (Oxidant)	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (or) Fe^{2+} Amm. Sulphate (Reductant)
SYSTEM - II	$\text{K}_2\text{Cr}_2\text{O}_7$ (Oxidant)	Fe^{2+} Amm. Sulphate (Reductant)	$\text{K}_2\text{Cr}_2\text{O}_7$ (Oxidant)

A typical experiment involving a link titration, consists of Following steps :

- i) Prepare a **Standard Solution** of Oxalic acid.
- ii) Standardize KMnO_4 **Link solution** and then
- iii) Determine the normality of the given Ferrous Ammonium Sulphate solution (FAS) and also estimate the amount of Fe^{2+} present in the given **test solution**.

TITRATION DATA, TABULATION AND CALCULATIONS

Titration data should essentially be recorded as follows :

..... **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

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

Table.2. Weight of empty weighing bottle

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

i. Normality of prepared std. Oxalic acid solution :

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

(from tables of 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.)}	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.)

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

- Result :**
1. Wt. of oxalic acid =
 2. N std. oxalic acid =
 3. N KMnO_4 =

DEMO EXPERIMENT

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Ferrous Ammonium Sulphate Solution :

Table.1. Weight of bottle + Substance

						g.
--	--	--	--	--	--	----

Table.2. Weight of empty weighing bottle

						g.
--	--	--	--	--	--	----

$$\text{Wt. of F.A.S} = W_1 - W_2 =$$

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

(V_{ml} = Volume of Standard Flask = 100 ml)

Part B : Standardisation of KMnO_4 :

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

Sl. No.	$V_{\text{F.A.S}} (\text{Std.})$ ml	Burette Reading		V_{KMnO_4} ml
		Initial	Final	

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

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

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

Expt. No.

Date :

TABULATION AND CALCULATIONS

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

Table.1. Weight of bottle + Substance

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Table.2. Weight of empty weighing bottle

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

Wt. of F.A.S = $W_1 - W_2$.

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

(V_{ml} = 100 ml)

PART B : STANDARDISATION OF KMnO₄ :

Titration of Std. F.A.S. Vs. KMnO₄

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

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

'REDOX' TITRATION'
(PERMANGANOMETRY)

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

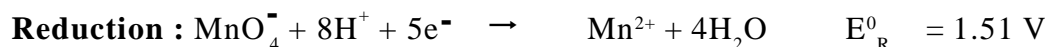
OBJECT : To standardize KMnO₄ solution using standard Ferrous Ammonium Sulphate solution.

Introduction : (Theory) : Potassium permanganate is a valuable and powerful oxidizing agent used in titrimetric analysis. In acid solution, Ferrous ion (Fe²⁺) is readily oxidized to Ferric ion (Fe³⁺) by KMnO₄; the redox reactions can be represented by the equation given below :

Ferrous ammonium sulphate FeSO₄(NH₄)₂SO₄·6H₂O is available in a high purity state and hence is used as primary standard instead of FeSO₄ for the standardization of KMnO₄.

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

(Partial Ionic equation.)



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

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

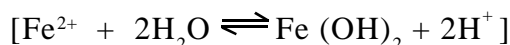
(Equivalent weight = Molecular Wt. / No : (or) No. of e⁻ transfer)

$$\text{Equivalent weights : KMnO}_4 / 5 = 158/5 = 31.6;$$

$$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O} / 1 = 392.1 / 1 = 392.1$$

PROCEDURE : A : PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE Fe SO₄ (NH₄)₂ SO₄ · 6H₂O SOLUTION : Weigh out in a clean weighing bottle accurately close to 3.9210 g Ferrous Ammonium sulphate FeSO₄ · (NH₄)₂ SO₄ · 6H₂O; transfer it to 100 ml std. standard Flask through a funnel ; dissolve it by adding distilled water including 10 ml dil. H₂ SO₄ (6N) (to prevent hydrolysis) and make the solution up to the mark. Shake it well to make it homogeneous.

HYDROLYSIS REACTIONS :



* 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 H_2SO_4 (6N) 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 dropwise with particular care to allow each drop to become decolorized before the next is added).
- iv) Repeat the process till at least two concordant values are obtained.

RESULTS :

1. Wt. of FAS = g
2. $N_{\text{std. FAS Solution}} = \underline{\hspace{2cm}}$
3. $N_{\text{KMnO}_4 \text{ Solution}} = \underline{\hspace{2cm}}$

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 ?

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

Signature of Faculty

'REDOX' TITRATION'
(PERMANGANOMETRY)

Experiment (2) : PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION, STANDARDISATION OF GIVEN POTTASIAM PERMANGANATE (KMnO_4) SOLUTION & ESTIMATION OF FERROUS IN GIVEN TEST SOLUTION.

OBJECT : To standardize KMnO_4 solution using Std. FAS solution.

INTRODUCTION : Refer Exp.1.

PART - A : PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE $\text{Fe SO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$ SOLUTION.

Refer Exp.1.

PART - B : STANDARDIZATION OF GIVEN KMnO_4 SOLUTION :

Refer Exp.1.

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 a 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.

* 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 POTASSIUM PERMANGANATE (KMnO₄) SOLUTION & ESTIMATION OF FERROUS IN GIVEN TEST SOLUTION.

Expt. No. **TABULATION AND CALCULATIONS** **Date :**

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

Table.1. Weight of bottle + Substance

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

Table.2. Weight of empty weighing bottle

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

$$\text{Wt of F.A.S} = W_1 - W_2 =$$

$$N_{(\text{std})\text{FAS}} = \frac{\text{wt}}{\text{Eq.wt}} \times \frac{1000}{V_{\text{ml}}} = \dots\dots\dots$$

(V_{ml} = 100 ml)

PART B. STANDARDISATION OF KMnO₄

Titration of Std. FAS Vs. KMnO₄

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

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

M J C E T

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	

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

$$\begin{aligned} \text{Wt. of Fe}^{2+} \text{ (in Test solution)} &= N_{\text{Fe}^{2+}} \times \text{Eq. Wt.} \\ &= \dots\dots\dots \text{gpl} \end{aligned}$$

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

Date : Expt. (3) Std. Oxalic Acid X K MnO_4 (Link) X Fe^{2+} (Test Soln.)

Expt. No.

TABULATION AND CALCULATIONS

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

Table.1. Weight of bottle + Substance

	.				
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Table.2. Weight of empty weighing bottle

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

$$\text{Wt. of Oxalic Acid} = W_1 - W_2$$

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

$$(V_{\text{ml}} = 250 \text{ ml})$$

PART B : STANDARDISATION OF KMnO_4 (LINK)

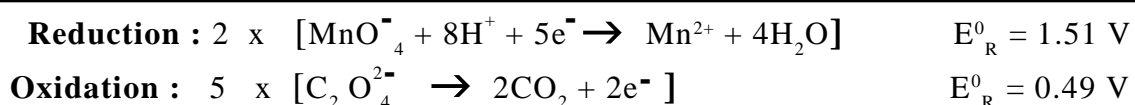
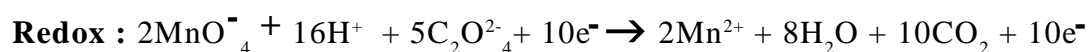
Titration of Std. $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ Vs. KMnO_4 (link)

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

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

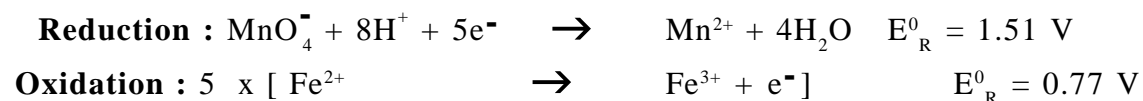
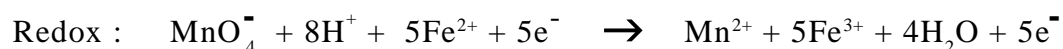
'REDOX LINK TITRATION'(PERMANGANOMETRY)**Experiment (3): Preparation of Standard Oxalic Acid, Standardisation of Potassium Permanganate (KMnO₄ / Link) Solution & Estimation of Fe²⁺ in the given Test Solution.****[NOTE : TYPICAL LINK TITRATION EXPERIMENT (AS A MODEL FOR WRITING IN THE RECORD)]**

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

PRINCIPLE:**1. Reaction between Oxalic and Permanganate****Overall ionic equation :**

Species: (KMnO₄) (H₂SO₄) (H₂C₂O₄) (MnSO₄)

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

2. Reaction between Ferrous and Permanganate**Overall ionic equation :**

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

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

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

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

Equivalent weights KMnO₄ / 5 = 158 / 5 = 31.6; H₂C₂O₄ . 2H₂O/2 = 126/2 = 63; Fe²⁺ = 55.8

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

Exact gm of Oxalic acid is weighed out accurately up to 4th decimal place in a clean weighing bottle. It is transferred to a 250 ml 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. The Normality of Standard Solution is calculated from the weighing data.

PART C: ESTIMATION OF Fe^{2+} IN THE GIVEN TEST SOLUTION.Titration : Fe^{2+} (Test Soln.) Vs. KMnO_4 (Link)

Sl. No.	$V_{\text{Fe}^{2+}}$ (test Sol.) ml	Burette Reading		V_{KMnO_4} ml
		Initial	Final	

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

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

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

Results:

- | | | | |
|----|--|---|-----------|
| 1. | Weight of Oxalic Acid | = | _____ g |
| 2. | $N_{\text{std. Oxalic acid}}$ | = | _____ |
| 3. | N_{KMnO_4} (Link) | = | _____ |
| 4. | $N_{\text{Fe}^{2+}}$ (Test) | = | _____ |
| 5. | Weight of Fe^{2+}
present in test solution | = | _____ gpl |

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

Signature of Faculty

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 solution (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 $\sim 70^\circ\text{C}$ (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. (last additions of KMnO_4 should be drop-wise, with particular care to allow each drop to become decolorized before the next is introduced).

The process is repeated till two concordant titre values are obtained. From the titration data, 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 a 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.

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.

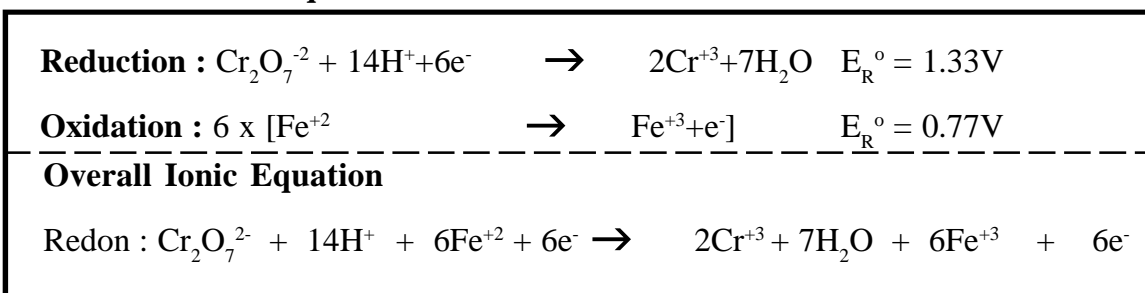
'REDOX' TITRATION'
(DICHROMATOMETRY)

Experiment (4) : Preparation of standard Ferrons Ammonium Sulphate, Standardisation of $K_2Cr_2O_4$ (Link) Solution & Estimation of Fe^{+2} in given test solution :

AIM : To estimate the amount of Fe^{+2} in test solution by preparing a standard FAS solution and using $K_2Cr_2O_7$.

Theory : Ferrons Ammonium Sulphate, Ferrons alum (Mohr's Salt) is a double salt of the combination $FeSO_4 (NH_4)_2SO_4 \cdot 6H_2O$ 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 $K_2Cr_2O_7$, the volume of oxidant ($K_2Cr_2O_4$) completely oxidizes ferrous iron Fe^{+2} to Ferric Iron (Fe^{+3}) is found out $K_2Cr_2O_7$ undergo 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 ($E_{Red}^0 = 0.76v$). In order to prevent the oxidation of diphenyl amine before the oxidation of Fe^{+2} , Phosphoric acid is added which reduces the reduction potential of iron couple from 0.77v to 0.44v.

Partial Ionic Equation :



Species : $(K_2Cr_2O_7)$ (H_2SO_4) $(FeSO_4)$ $Cr_2(SO_4)$ $Fe_2(SO_4)_3$

Colour Profile : (Orange) (X) (X) (Greenish) (X)

Equivalent Weight = Molecular Wt / No. of e^- transfer

(Eq. wts. of : $K_2Cr_2O_7 = 294.2/6 = 49.0$:

Eq. wts. of $FeSO_4 - (NH_4)_2SO_4 - 6H_2O = 392.1/1, : Fe^{+2} = 55.85/1$

X ----> Colourless

Experiment (4) : Preparation of standard Ferrons Ammonium Sulphate, Standardisation of $K_2Cr_2O_4$ (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 + Substance

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

Table.2. Weight of empty weighing bottle

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$$\text{Wt. of FAS} = W_1 - W_2 =$$

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

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

PART B. STANDARDISATION OF $K_2Cr_2O_7$

Titration of Std. FAS Vs. $K_2Cr_2O_7$ (link)

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

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

Procedure :**PART A : PREPARATION OF STD. F.A.S. SOLUTION :**

Exactly - $\square\square\square\square$ g of F.A.S is weighed out accurately upto 4th decimal place in a clean weighing bottle and it is transferred to a 100ml Std. volumetric flask through a funnel .Then dissolved in distilled water and the solution is made up to the mark, add 1/2 test tube dilute H_2SO_4 , it is shaken well to make homogenous. The normality of standard solution is calculated from the weighing data.

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

20ml of the prepared solution (standard FAS) 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, and then 3 to 5 drops of internal indicator diphenylamine and about 8ml (1/3 T. T) of Syrupy Phosphoric acid is added which lowers the reduction potential of Iron couple and facilitates the initial oxidation of Ferrons to Ferric. It is titrated with $\text{K}_2\text{Cr}_2\text{O}_7$ (taken in Burette) very slowly until the solution acquires a bluish greentint 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 bluish colour , which is stable.

The process is repeated till two concordant titre values are obtained. From the titation 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 pipetted 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 then 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 of 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?
7. Silky white precipitate is obtained due to _____.

PART C: ESTIMATION OF Fe^{2+} IN THE GIVEN TEST SOLUTION.Titration : Fe^{2+} (Test Soln.) Vs. $\text{K}_2\text{Cr}_2\text{O}_7$ (Link)

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

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

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

Results:

1. Weight of FAS = _____ g
2. $N_{\text{std. FAS}}$ = _____
3. $N_{\text{K}_2\text{Cr}_2\text{O}_7}$ (Link) = _____
4. $N_{\text{Fe}^{2+}}$ (Test) = _____
5. Weight of Fe^{2+} present in test solution = _____ gpl

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

Signature of Faculty

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

	.				
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Table.2. Weight of empty weighing bottle

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

$$\text{Wt. of K}_2\text{Cr}_2\text{O}_7 = W_1 - W_2 =$$

$$N_{(\text{std}) \text{K}_2\text{Cr}_2\text{O}_7} = \frac{\text{wt}}{\text{Eq. wt}} \times \frac{1000}{V_{\text{ml}}} = \dots\dots\dots$$

(V_{ml} = 250 ml)

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

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

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

$$\begin{aligned} \text{Wt. of Fe}^{2+} &= N_{\text{Fe}^{2+}} \times \text{Eq. wt. of Fe}^{2+} = \dots\dots\dots \times 55.8 \\ (\text{in solution mixture}) &= \dots\dots\dots \text{ gpl} \end{aligned}$$

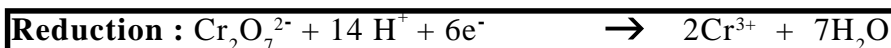
'REDOX' TITRATION'
(DICHROMATOMETRY)

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

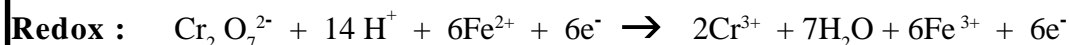
OBJECT : To estimate Iron Fe³⁺ from solution mixture using 0.1 N K₂Cr₂O₇ solution.

INTRODUCTION : (THEORY) : Ferrous ammonium sulphate, ferrous alum (Mohr's salt) is a double salt of the composition FeSO₄ · (NH₄)₂ SO₄ · 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 (Fe³⁺) and in the process gets reduced to Cr³⁺ which is green in colour even after equivalence point. In order to identify the equivalence point 3-5 drops of diphenyl-amine is added as an internal indicator (E⁰_{Red} = 0.76V). In order to prevent the oxidation of diphenyl amine 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: (K₂Cr₂O₇) (H₂SO₄) (FeSO₄) (Cr(SO₄)₃)

Color Profile: (Orange) (X) (X) (Green) X= Colorless

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

$$\text{(Eq. Wts.: K}_2\text{Cr}_2\text{O}_7/6 = 294.2/6 = 49.0 ;$$

$$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O} / 1 = 392.1; \text{Fe}^{2+} = 55.8)$$

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.	$\text{V}_{\text{Fe}^{2+} \& \text{Fe}^{3+}}$ (test Sol.) ml	Burette Reading		$\text{V}_{\text{K}_2\text{Cr}_2\text{O}_7}$ ml
		Initial	Final	

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

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

THERE FORE THE AMOUNT OF Fe^{3+} PRESENT = WT. OF TOTAL IRON -- WT. OF Fe^{2+}

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

M J C E T

Since HgCl_2 does not undergo any change (silky white ppt) it remains in the solution. After reduction of ferric iron to ferrous iron the 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 silky white 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 (A.R) $\text{K}_2\text{Cr}_2\text{O}_7$; 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 bluish violet colour appears near the end point.
- 5) Repeat the titration to obtain concordant values. Let the titre value be V 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 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 $\text{K}_2\text{Cr}_2\text{O}_7$.

- 1) To the above solution, add half test tube (10ml) of dil H_2SO_4 solution , 3-5 drops of DPA indicator followed by $\frac{1}{4}$ testube (5ml) of syrupy phosphoric acid.
- 2) Titrate this solution against Std $\text{K}_2\text{Cr}_2\text{O}_7$ solution until persistant bluish 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. Wt. of $\text{K}_2\text{Cr}_2\text{O}_7$ = _____ g
2. N $\text{K}_2\text{Cr}_2\text{O}_7$ solution = _____
3. Weight of Ferrous Iron = _____ g / lit.
4. Weight of Total Iron = _____ g / lit.
5. Wt. of Fe^{3+} in the test solution = _____ g / lit

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

Signature of Faculty

WATER ANALYSIS

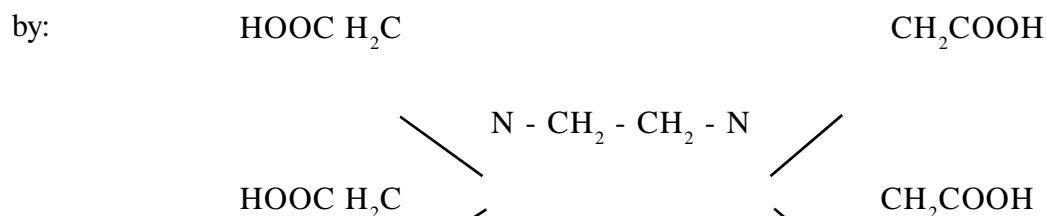
'COMPLEXOMETRIC' TITRATION

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

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 scales etc). Based on the degree of hardness, suitable treatment can be recommended for water supply or effluents.

EDTA = Ethylene Di ammine 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 Tetrabasic 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 makes 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.

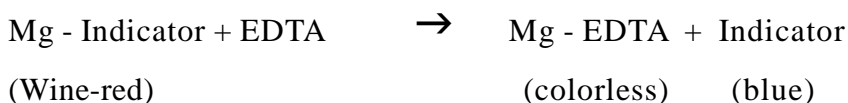
The order of stability is as follows:

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 the free Mg^{2+} ; and then with Mg^{2+} liberated from the Mg - Indicator complex (Wine-red) leaving the indicator above, which will be blue.



PREPARATION OF SOLUTIONS:

1. **Standard hard water :** weigh out accurately 1.0000 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 up to 1 litre with distilled water.
3. **Indicator :** 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 ($\text{Na}_2\text{H}_2\text{ ver. } 2\text{H}_2\text{O}$) dissolved in distilled water and made to 1 litre.

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

PROCEDURE : 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.

TOTAL HARDNESS (DETERMINATION) : Repeat the above process except that in step (i) sample hardwater is used instead of std. hard water.

Let the reading obtained now be V_2 ml. of EDTA.

PERMANENT HARDNESS (DETERMINATION) : Take a large quantity of sample hard water, say 100 ml. Boil it (to remove temporary hardness) to about one-fourth of this volume. Filter (through Whatman 42 filter paper) to remove insoluble CaCO_3 and MgCO_3 . Make up the volume to the original 100 ml by adding distilled water. Make the solution homogeneous. Now pipette out 20 ml of this solution into a conical flask. Then repeat the process of titration steps (ii) and (iii). Let the reading this time be V_3 ml of EDTA.

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]

Questions :

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 ?

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 water

$$1\text{ml EDTA} = ? \quad 1\text{ml EDTA} = \frac{20}{V_1} = \dots\dots\dots\text{mg CaCO}_3$$

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	

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

PART C : ESTIMATION OF PERMANENT HARDNESS

Titration of boiled sample water Vs EDTA

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

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

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

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	

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

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

RESULTS:

- Source: O/W, B/W = _____
- Depth (approximately) = _____
- Locality = _____
- Total Hardness = _____ ppm
- Permanent Hardness = _____ ppm
- Temporary Hardness = _____ ppm
- Total Hardness of municipal/Distilled water = _____ ppm

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

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Table.2. Weight of empty weighing bottle

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$$\text{Wt. of MgSO}_4 = W_1 - W_2 =$$

$$N_{(\text{std}) \text{MgSO}_4} = \frac{\text{wt}}{\text{eq. wt}} \times \frac{1000}{V_{\text{ml}}} = \dots\dots\dots$$

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

Part B : Standardisation of EDTA

Titration of Std. MgSO₄ Vs EDTA

Sl. No	V Std. MgSO ₄ Soln. ml	Burette Reading		V EDTA ml (V ₁)
		Initial	Final	

$$N_{\text{EDTA}} = \frac{N_{\text{MgSO}_4} \times V_{\text{MgSO}_4}}{V_{\text{EDTA}}} =$$

WATER ANALYSIS

'COMPLEXOMETRIC' TITRATION

EXPERIMENT (7): (ESTIMATION OF TOTAL HARDNESS OF WATER USING STD.**Mg SO₄ 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 make the 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 20 ml of the standard hardwater into a clean conical flask. Add about 5ml of (NH₄Cl+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 EBT - indicator to it. Titrate the contents of the conical flask against the EDTA solution taken in the burette until the wine red color 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	

$$N_{\text{Test Solution}} = \frac{N_{\text{EDTA}} \times V_{\text{EDTA}}}{V_{\text{Sample water}}} =$$

$$\text{Wt in terms of CaCO}_3 \text{ equivalents} = N \times \text{Eq. wt CaCO}_3 \\ = \quad \times 50 = \dots\dots\dots\text{gpl.}$$

$$\text{Total hardness of Test Solution} = \quad \times 1000 = \quad \text{mg/lit. (Or) ppm}$$

RESULTS:

1. Wt. of MgSO₄ = _____
2. Normality of MgSO₄ = _____
3. Normality of EDTA = _____
4. N 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:

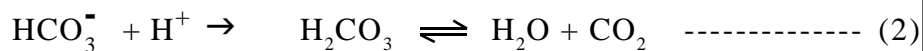
Alkalinity of natural waters may be attributed to the presence of salts of weak acids, such as bicarbonates and carbonates etc. Highly alkaline waters may lead to caustic embrittlement and deposition of precipitates and sludges in boilers. Bicarbonates of calcium and magnesium induce temporary hardness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

For water softening processes as well as boiler feed water analysis, it is essential to 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 .

* 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:**A. PREPARATION OF STANDARD N/50 Na₂CO₃ SOLUTION :**

Weigh out in a clean weighing bottle accurately to about 0.2622g of (A.R.) Na₂CO₃; transfer it to a clean 250ml. 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 .

$$N_{\text{Na}_2\text{CO}_3} = \frac{\text{Wt.}}{\text{Eq. Wt.}} \times \frac{1}{V} \times 1000 = \frac{\text{Wt.}}{53} \times \frac{1}{V} \times 1000$$

B. STANDARDIZATION 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 light pink colour; this is the end point. Repeat the process of titration till at least two concordant values are obtained).

$$N_{\text{HCl}} = \frac{N_{\text{Na}_2\text{CO}_3} \times V_{\text{Na}_2\text{CO}_3}}{V_{\text{HCl}}} = \frac{N_{\text{Na}_2\text{CO}_3} \times 20}{V_{\text{HCl}}}$$

D. 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. Follow the procedure as given in Part C for the estimation of CO₃²⁻ & HCO₃⁻ alkalinity in the given test solution.

Note - Instead of methyl orange, a mixture of bromocresol green and methyl red indicators can be used for better contrast at the end point. Color change is from Green to Reddish colour.

(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,

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 CO_3^{2-} = 2[P] = 2 x 10.4

ml = 20.8 ml and vol. of N/50 HCl equivalent to HCO_3^- = [M] - 2 [P] =

25.7 - 20.8 ml = 4.9 ml.

CALCULATION:

(i) CO_3^{2-} :

$$= \frac{N_{\text{HCl}} \times V_{\text{HCl}}}{100} = \frac{0.02 \times 20.8}{100} = 0.00416$$

$$\text{Wt. of} = \frac{N \times \text{eq.wt.}}{1000} \times 1000 \text{ g/l} = 0.00416 \times 30 \text{ g/l} = 0.1248 \text{ g/l}$$

$$\text{Wt. of CaCO}_3 \text{ eqt.} = 0.124 \times \frac{50}{30} \text{ g/l} = 0.208 \text{ g/l} = 208 \text{ mg/l} = 208 \text{ ppm}$$

(ii) HCO_3^- :

$$\frac{N_{\text{HCO}_3^-}}{V_{\text{HCO}_3^-}} = \frac{N_{\text{HCl}} \times V_{\text{HCl}}}{100} = \frac{0.02 \times 4.9}{100} = 0.00098$$

$$\text{Wt. of} = \frac{N_{\text{HCO}_3^-} \times \text{eq.wt.}}{1000} \times 1000 \text{ g/l} = 0.00098 \times 61 \text{ g/l} = 0.05978 \text{ g/l}$$

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

Expt. No. 9

Date :

PART - A: PREPARATION OF STANDARD Na_2CO_3

Table.1. Weight of bottle + Na_2CO_3

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Table.2. Weight of empty weighing bottle

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$$\text{Wt. of } \text{Na}_2\text{CO}_3 = W_1 - W_2 =$$

$$N \quad \text{wt} \quad 1000$$

$$(\text{std}) \text{Na}_2\text{CO}_3 = \frac{\quad}{\text{eq.wt}} \times \frac{\quad}{V_{\text{ml}}} = \dots\dots\dots$$

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

Part - B: Standardization of HCl Link solution

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

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

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

S.No	Vol. of test solution (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:(i) CO_3^{2-} :

$$N_{\text{CO}_3^{2-}} = \frac{N_{\text{HCl}} \times V_{\text{HCl}}}{V_{\text{CO}_3^{2-}}} =$$

$$\text{Wt. of } \text{CO}_3^{2-} = \frac{N_{\text{CO}_3^{2-}} \times \text{eq.wt.} \times 1000 \text{ g/l}}{1000} = \quad \text{g/l}$$

$$\text{Wt. of CaCO}_3 \text{ eqt.} = \text{Wt. of } \text{CO}_3^{2-} \times \frac{50}{30} \text{ g/l} = \quad \times 1000 = \quad \text{ppm}$$

(ii) HCO_3^- :

$$N_{\text{HCO}_3^-} = \frac{N_{\text{HCl}} \times V_{\text{HCl}}}{V_{\text{HCO}_3^-}} =$$

$$\text{Wt. of } \text{HCO}_3^- = \frac{N_{\text{HCO}_3^-} \times \text{eq.wt.}}{1000} \times 1000 \text{ g/l} = \quad \text{g/l}$$

$$\text{Wt. of CaCO}_3 \text{ eqt.} = \text{Wt. of } \text{HCO}_3^- \times \frac{50}{61} \text{ g/l} = \quad \times 1000 = \quad \text{ppm}$$

RESULTS:**I FOR TEST SOLUTION:-**

1. [P] = ml; [M]=..... ml ; [M-2(P)]=.....ml
2. $N_{CO_3^{2-}}$ =..... 3. Wt. of CO_3^{2-} in mixture =gpl
4. Carbonate Alkalinity = ppm
5. $N_{HCO_3^-}$ =..... 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

**PREPARATION OF STANDARD SOLUTION OF POTASSIUM DICHROMATE –
STANDARDIZATION OF MOHR SALT SOLUTION – DETERMINATION OF CHEMICAL**

Expt. No. 9

OXYGEN DEMAND

Date :

PART - A: PREPARATION OF STANDARD $K_2Cr_2O_7$

Table.1. Weight of bottle + $K_2Cr_2O_7$

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Table.2. Weight of empty weighing bottle

	.				
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$$\begin{aligned} \text{Wt. of } K_2Cr_2O_7 &= W_1 - W_2 = \\ N &= \frac{\text{wt}}{\text{eq. wt}} \times \frac{1000}{V_{ml}} = \dots\dots\dots \end{aligned}$$

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

PART - B: STANDARDIZATION OF FAS LINK SOLUTION

Titration of Std. FAS Vs. _____ (link)

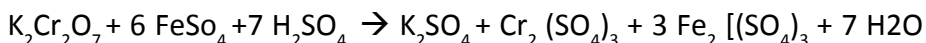
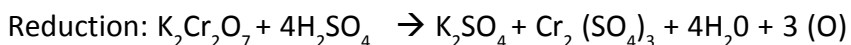
Sl. No.	V_{FAS} (Std.) ml	Burette Reading		$V_{K_2Cr_2O_7}$ ml
		Initial	Final	

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

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 in the water. It is the amount of oxygen equivalent used while oxidizing the 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 the given Fe^{2+} 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 (1/4th 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 a bluish green tint which shows that the end point is near. The Titration is continued now with dropwise addition of $K_2Cr_2O_7$ 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 $K_2Cr_2O_7$ 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 disulfuric acid and then slowly diluting to 500ml with distilled water , then pipette out 20 ml 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

$$= V_1 = \text{_____ ml}$$

Volume of mohr salt consumed in Blank Titration = $V_2 = \text{_____ ml}$

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 = _____

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

1 ml 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

= _____ gm

= _____ mg

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

= _____ ppm.

= _____

= _____ ppm

RESULTS:

1. Weight of $K_2Cr_2O_7$ = _____
2. N of $K_2Cr_2O_7$ = _____
3. N of FAS = _____
4. COD of given water sample = _____ ppm

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

Signature of Faculty

