MUFFAKHAM JAH COLLEGE
OF
ENGINEERING AND TECHNOLOGY

(SULTAN-UL-ULOOM EDUCATION SOCIETY)

BANJARA HILLS, HYDERABAD - 500 034.

LABORATORY MANUAL IN CHEMISTRY

FOR B.E. IST YEAR; SEMESTER- I AND II

VOLUMETRIC ANALYSIS & INSTRUMENTAL ANALYSIS

Name: ____

Branch (section): ____________________________________

Roll No. 1604 - ___________________________________

DEPARTMENT OF CHEMISTRY
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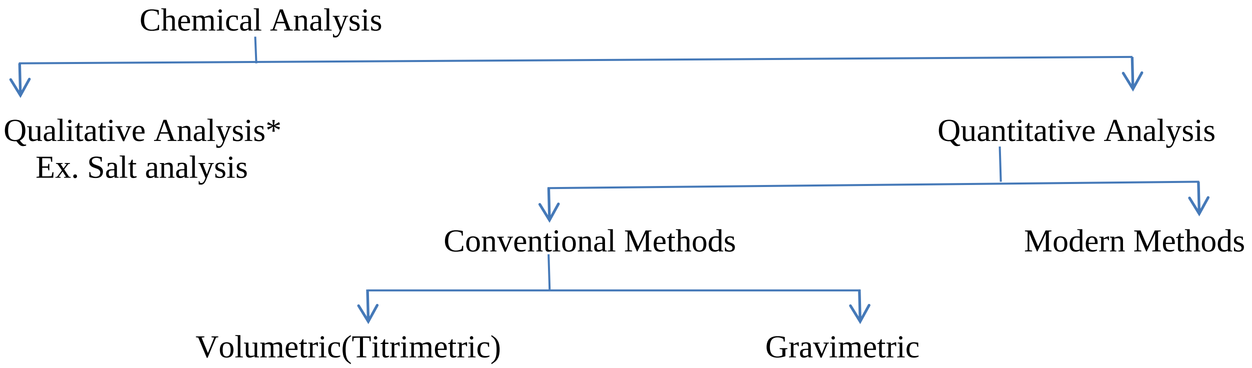
Revised- 2022

Chemistry Laboratory Safety Instructions

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 hands 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. Hair should be covered or securely tied back to avoid the risk of falling/getting color by touch of chemicals or 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 bag packs on the bench. No Cell Phones and headphones 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 glassesunder 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.

CONTENTS

VOLUMETRIC ANALYSIS



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 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:

$$\mathbf{N_1 \times V_1 = N_2 \times V_2}$$

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: $FeSO_4(NH_4)_2\ SO_4.\ 6H_2O$(Ferrous Ammonium sulphate)$K_2Cr_2O_7$(Potassium dichromate), $H_2C_2O_4$(Oxalic Acid),Na_2CO_3(Sodium Carbonate),$Na_2B_4O_7$(Sodium Borate)

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), NaOH (Sodium hydroxide), $Na_2S_2O_3.5H_2O$(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**:
FAS Vs $KMnO_4$, Fe^{2+}Vs. $K_2Cr_2O_7$ and Oxalic acid Vs $KMnO_4$.
Also Iodometry: $K_2Cr_2O_7$Vs. Hypo; Cu^{2+} Vs. Hypo.
iii) Precipitation titration **:**Estimation of chloride in water sample (NaCl Vs. Ag^+)
(Ag^+ Vs CNS)
iv) **Complexometric titration** (EDTA) method of estimation of hardness of water(total, permanent and temporary hardness)

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:

$$\mathbf{N = \frac{Wt.}{Eq.Wt.} X \frac{1}{V_{ml}} X1000} \text{---------}(1)$$

Where N=Normality of the solution

V_{ml}= Volume of standard flask in ml

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

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, Oxalic acid Vs KMnO, it should be written as follows:

$$\mathbf{N_{KMnO4} \times V_{KMnO4} = N_{Oxalic\ acid} \times V_{Oxalic\ acid}}$$

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

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 Oxalic acid Vs $KMnO_4$ 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:

(De-Electronation) or loss of 'e': $5 \times \ [C_2O_4^{-2} \rightarrow 2CO_2 + 2e^-]E_R^0 = 0.49\ V$

Half reaction: Reduction:

(Electronation) or gain of 'e': $2\times \ [MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} \ + 4H_2O]E_R^0 = 1.51\ V$

Overall Redox Reaction:

$2MnO^-_4 + 16H^+ + 5C_2O_4^{2-} + 10e^- \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2 + 10e^-$

In the above reaction it may be noted that the oxidant $KMnO_4$ gets reduced itself because of its High Reduction Potential ($E_R^0 = 1.51\ V$) at the same time,oxalic acid($H_2C_2O_4.2H_2O$)

gets oxidized itself because of Less Reduction Potential ($E_R^0 = 0.49\ V$). The equivalence established as follows:

$2MnO^-_4 = 10e^- = 5C_2O_4^{2-}$ (or); $\frac{2\ KMnO_4}{10} = \frac{10_{e^.}}{10} = \frac{5(H_2C_2O_4 2H_2O)}{10}$

$1 / 5KMnO_4 = 1e^- = 1 / 2\ (H_2C_2O_4.2H_2O)$

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

31.6g = $1e^-$ = 63 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 $H_2C_2O_4.2H_2O$ (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 our 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 $\frac{standard\quad link\quad Test}{FAS\quad KMnO_4\quad Fe+2}$	(Reductant)	(Oxidant)	(Reductant)
	FAS	$KMnO_4$	Fe^{2+}
	(Reductant)	(Oxidant)	(Reductant)
System II (FAS Vs $K_2Cr_2O_7$ Vs Fe^{+2})	FAS	$K_2Cr_2O_7$	Fe^{2+}

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

i)**Preparation of a standard solution** of FAS

ii)**Standardisation of** $KMnO_4$ **Link solution** and then

iii) **Estimation of** 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 Ferrous ammonium sulphate Vs. Potassium permanganate

Table.1.Weight of bottle +Substance

12	.	7	6	3	4

Table.2.Weight of empty weighing bottle

8	.	6	3	4	0

i. Normality of prepared std.FAS solution :

Wt. of FAS= (12.7634 -8.6340) g = 4.125g.

N(std) FAS= $\frac{wt}{eq.wt}$x $\frac{1}{V}$x 1000= $\frac{4.125}{392}$ x$\frac{1}{100}$ x 1000 =0.1052

Where Volume of std. flask = 100 ml

ii. **Standardisation of KMnO4** : Titration of Std. $H_2C_2O_4 . 2H_2O$ Vs $KMnO_4$ (link)

Sl No.	Voxalic (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

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

N_{KMnO4}x V_{KMnO4} = N_{FAS} x V_{FAS}

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

$$N_{KMnO4} = \frac{N_{FAS}\ X\ V\ FAS}{V_{KMnO4}} = \frac{0.1052\ X\ 20}{19.8} = 0.0106$$

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

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

3. $NKMnO_4$ = ------------------

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. $[FeSO_4(NH_4)_2SO_4.6H_2O$	392.16
2	Potassium Permanganate $[KMnO_4]$	158
3	Potassium Dichromate $[K_2Cr_2O_7]$	294
4	Oxalic Acid $[H_2 C_2 O_4.2H_2O]$	126
5	Sodium thio sulphate $[Na_2S_2O_3.5H_2O]$	248.18
6	Copper Sulphate $[CuSO_4.5H_2O]$	249.68
7	Sodium Carbonate $[Na_2CO_3]$	106
8	Magnesium Sulphate $[MgSO_4.7H_2O]$	246.5
9	Hydrochloric Acid [HCl]	36.5
10	Acetic Acid $[CH_3COOH]$	60

Standard Electrode Potentials in Aqueous Solution at 25°C

Cathode (Reduction) Half-Reaction	**Standard Potential E° (volts)**
$Li^+(aq) + e^- \rightarrow Li(s)$	-3.04
$K^+(aq) + e^- \rightarrow K(s)$	-2.92
$Na^+(aq) + e^- \rightarrow Na(s)$	-2.71
$Mg^{2+}(aq) + 2e^- \rightarrow Mg(s)$	-2.38
$Al^{3+}(aq) + 3e^- \rightarrow Al(s)$	-1.66
$2H_2O(l) + 2e^- \rightarrow H_2(g) + 2OH^-(aq)$	-0.83
$Zn^{2+}(aq) + 2e^- \rightarrow Zn(s)$	-0.76
$Cr^{3+}(aq) + 3e^- \rightarrow Cr(s)$	-0.74
$Fe^{2+}(aq) + 2e^- \rightarrow Fe(s)$	-0.41
$Cd^{2+}(aq) + 2e^- \rightarrow Cd(s)$	-0.40
$Ni^{2+}(aq) + 2e^- \rightarrow Ni(s)$	-0.23
$Sn^{2+}(aq) + 2e^- \rightarrow Sn(s)$	-0.14
$Pb^{2+}(aq) + 2e^- \rightarrow Pb(s)$	-0.13
$Fe^{3+}(aq) + 3e^- \rightarrow Fe(s)$	-0.04
$2H^+(aq) + 2e^- \rightarrow H_2(g)$	**0.00**
$Cu^{2+}(aq) + e^- \rightarrow Cu^+(aq)$	0.16
$ClO_4^-(aq) + H_2O(l) + 2e^- \rightarrow ClO_3^-(aq) + 2OH^-(aq)$	0.17
$Cu^{2+}(aq) + 2e^- \rightarrow Cu(s)$	0.34
$Cu^+(aq) + e^- \rightarrow Cu(s)$	0.52
$I_2(s) + 2e^- \rightarrow 2I^-(aq)$	0.54
$Fe^{3+}(aq) + e^- \rightarrow Fe^{2+}(aq)$	0.77
$Hg_2^{2+}(aq) + 2e^- \rightarrow 2Hg(l)$	0.80
$Ag^+(aq) + e^- \rightarrow Ag(s)$	0.80
$Hg^{2+}(aq) + 2e^- \rightarrow Hg(l)$	0.85
$Br_2(l) + 2e^- \rightarrow 2Br^-(aq)$	1.07
$O_2(g) + 4H^+(aq) + 4e^- \rightarrow 2H_2O(l)$	1.23
$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$	1.33
$Cl_2(g) + 2e^- \rightarrow 2Cl^-(aq)$	1.36
$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$	1.49
$H_2O_2(aq) + 2H^+(aq) + 2e^- \rightarrow 2H_2O(l)$	1.78
$S_2O_8^{2-}(aq) + 2e^- \rightarrow 2SO_4^{2-}(aq)$	2.01
$F_2(g) + 2e^- \rightarrow 2F^-(aq)$	2.87

Experiment (0): PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION& STANDARDISATION OF GIVEN POTASSIUM PERMANGANATE ($KMnO_4$) SOLUTION.

OBJECT: To standardize $KMnO_4$ 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 (Fe^{2+}) is readily oxidized to Ferric ion ($Fe^{3+)}$by $KMnO_4$; the redox reactions can be represented by the equation given below.$KMnO_4$ is often contaminated with manganese-di-oxide and also undergoes decomposition it can be taken as secondary standard solution. Ferrous ammonium sulphate $FeSO_4(NH_4)_2SO_4.6H_2O$ is available in a high purity state and hence is used as primary standard for the standardization of $KMnO_4$. $KMnO_4$ acts as self-indicator. At the end point of titration, light pink color is obtained due to excess drop of potassium permanganate added .

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

PRINCIPLE-Redox reaction (Partial Ionic equation)

Reduction: $MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$ $E^0_R = 1.51V$.

Oxidation: $5 \times [Fe^{2+} \rightarrow Fe^{3+} + e^-]$ $E^0_R = 0.77V$.

Overall Ionic equation
Redox : $MnO_4^- + 8H^+ + 5Fe^{2+} + 5e^- \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O + 5e^-$

Species:	($KMnO_4$)	(H_2SO_4)	($FeSO_4$)	($MnSO_4$)	
Color Profile:	(Purple)	(X)	(X)	(X)	X=Colorless

(Equivalent weight = Molecular Wt. / No. of e^- transfer)
Equivalent weights:$KMnO_4$/ 5 = 158/5 = 31.6;
$FeSO_4$. $(NH_4)_2SO_4$ $6H_2O$/1 = 392.1/1=392.1
Fe^{+2} = 55.8, approximately 56

PROCEDURE: PART A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE $FeSO_4(NH_4)_2SO_4.6H_2O$ SOLUTION:

Exactly 3.9210g of Ferrous Ammonium sulphate $FeSO_4$. $(NH_4)_2SO_4$.6H2O is weighed out in a clean weighing bottle; transfered to 100 ml standard Flask through a funnel; dissolved by adding distilled water a funnel **including 10 ml dil. H_2SO_4** (6N) (to prevent hydrolysis) and make the solution up to the mark. Shake it well to make it homogeneous.

HYDROLYSIS REACTION:

$$[Fe^{2+} + 2H_2O \rightleftharpoons Fe(OH)_2 + 2H^+]$$

PART 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) 20 ml of the prepared std. Ferrous Amm. Sulphate solution is pipetted out into a clean conical flask.
ii) 10 ml of dilute H_2SO_4(6N) solution (1/2 Test Tube is added to provide acidic medium.
iii) This solution is titrated with $KMnO_4$ (taken in the burette) to a faint pink color 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) The process is repeated till, atleast two concordant values are obtained.

RESULTS:

1.Wt. of FAS= _____________g
2.N std FAS= _____________
3.N_{KMnO4} = _____________

QUESTIONS:

1.What is the color 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 color 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

DEMO EXPERIMENT

Experiment (0): PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION & STANDARDISATION OF GIVEN POTASSIUM PERMANGANATE ($KMnO_4$) 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 =

$N_{\text{std F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{Vml} =$ ………………

(V_{ml} = 100ml), Eq Wt FAS=392.1/1

Part B: Standardization of $KMnO_4$:

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

Sl No.	V_{FAS} (Std) (ml)	Burette Reading		V_{KMnO_4} (ml)
		Initial	Final	

$$N_{KMnO4} = \frac{N_{FAS} \times V_{FAS}}{V\ KMnO4}$$

Experiment(1): PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION & STANDARDISATION OF GIVEN POTASSIUM PERMANGANATE ($KMnO_4$) SOLUTION & ESTIMATION OF FERROUS (IRON) IN GIVEN TEST SOLUTION.

AIM: To estimate Fe+2 from the given test solution and to standardize $KMnO_4$ solution using Std. FAS solution

INTRODUCTION and PRINCIPLE : Refer Exp. 0, Demo Exp, Pg no.13

PROCEDURE

PART-A: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE $FeSO_4 (NH_4)_2SO_4.6H_2O$ 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, dissolved in ½ test tube dilute H_2SO_4. The solution is made up to the mark with distilled water. It is shaken well to make homogenous. The Normality of standard FAS solution is calculated from the weighing data.

PART-B: STANDARDIZATION OF GIVEN $KMnO_4$ (Potassium permanganate) SOLUTION:

1) 20 ml of the prepared std. Ferrous Amm. Sulphate solution is pipetted out into a clean conical flask.
2) 10 ml of dilute H_2SO_4(6N) solution (1/2 Test Tube is added to provide acidic medium).
3) This solution is titrated with $KMnO_4$ (taken in the burette) to a faint pink color 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).
4) The process is repeated till, atleast two concordant values are obtained.
5) 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 pippetted 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.

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

Experiment (1): PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION, STANDARDISATION OF GIVEN POTASSIUM PERMANGANATE ($KMnO_4$) SOLUTION & ESTIMATION OF FERROUS IRON IN GIVEN TEST 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 =

$N_{\text{std. F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{Vml} = \ldots\ldots\ldots\ldots\ldots\ldots$

(V_{ml} = 100ml),Eq Wt FAS=392.1/1

Part B: Standardization of $KMnO_4$:

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

Sl No.	V_{stdFAS} (ml)	Burette Reading		V_{KMnO_4} (ml)
		Initial	Final	

$$N_{KMnO4} = \frac{N_{FAS} \times V_{FAS}}{V\ KMnO4}$$

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

Titration: Fe^{2+} (Test Soln) Vs $KMnO_4$ (Link)

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

$$N_{Fe^{2+}} \text{ (Test)} = \frac{N_{KMnO4} \times V_{KMnO4}}{V_{Fe^{2+}}} = \ldots\ldots\ldots\ldots$$

Wt. of Fe^{2+}= $N_{Fe^{2+}}$ X Eq. Wt.= ……………………..gpl

Results:

1.Weight. of FAS= ______________g
2.$N_{std.}$ FAS = ______________
3.N_{KMnO4} (Link)= ______________
4.$N_{Fe^{2+}}$ (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 (2): Preparation of Standard Ferrous Ammonium Sulphate, Standardization of $K_2Cr_2O_7$ (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 $K_2Cr_2O_7$.

THEORY:FAS or Ferrous alum (Mohr's Salt) is a double salt of the combination $FeSO_4$ $(NH_4)_2SO_4.6H_2O$, this when dissolved in water breaks up into simple salts and water. Ferrous sulphate is a reducing agent. $K_2Cr_2O_7$ is oxidizing agent in acidic medium. 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 $K_2Cr_2O_7$ 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 color even after equivalence point. In order to identify the equivalence point 3-5 drops of diphenylamine added as an internal indicator (E^0_{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 diphenylbenzidine which is a deep violet color (bluish violet).

Partial Ionic Equation

Reduction: $Cr_2O_7^{-2} + 14H^+ + 6e^- \rightarrow 2Cr^{+3} + 7H_2O E^0_R = 1.33V$.

Oxidation: $6 \times [Fe^{+2} \rightarrow Fe^{+3} + e^-]$ $E^0_R = 0.77V$.

Overall Ionic equation:

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

Species: ($K_2Cr_2O_7$) (H_2SO4) ($FeSO_4$) $Cr_2(SO_4)$ $Fe_2(SO_4)_3$

↓ ↓

Color Profile: (Orange) (X) (X) (Greenish) (X)

Equivalent weight = Molecular Wt./No. of e^- transferred)

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

Eq. wts. Of $FeSO_4 - (NH_4)_2SO_4\text{-}6H_2O$ = 392.1/1, Fe+2 = 55.85/1, approximately 56

In the presence of an acid mixture of H_2SO_4 and H_3PO_4, the reduction potential of Fe^{+3}- Fe^{+2} system is lowered to 0.61V due to complexation of Fe^{+2} ion with PO_4^{-3} ion. Hence during titration, it is Fe^{+2} ion that undergoes oxidation in preference to diphenylamine.The standard reduction potentials of $Cr_2O_7^{-2}$-Cr^{+3} is 0.33V. Fe^{+3}-Fe^{+2} is 0.77v and diphenylbenzidine –diphenylene is 0.76v.

Experiment (2):Preparation of standard Ferrous Ammonium Sulphate, Standardization 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 =

$N_{\text{std F.A.S}} = \frac{wt}{eq.wt} \times \frac{1000}{Vml}$ = ………………

(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.	$V_{\text{std FAS}}$ (ml)	Burette Reading		$V_{K_2Cr_2O_7}$ (ml)
		Initial	Final	

$$N_{K_2Cr_2O_7} = \frac{N_{FAS} \times V_{FAS}}{V_{K2Cr2O7}}$$

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, dissolved in ½ test tube dilute H_2SO_4. The solution is made up to the mark with distilled water. It is shaken well to make homogenous. The normality of standard FAS solution is calculated from the weighing data.

PART B: STANDARISATION OF $K_2Cr_2O_7$:

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 provide acidic medium), about 1/4 test tube of syrupy Phosphoric acid is added followed by 3 to 5 drops of internal indicator diphenylamine are added.It is titrated with $K_2Cr_2O_7$ (taken in Burette) very slowly until the solution acquires a green color (due to chromium ions). The titration is continued with drop wise addition of $K_2Cr_2O_7$ and good stirring until addition of a drop causes the formation of deep violet color, which is the end point.

The process is repeated till two concordant titre values are obtained. From the titration data Normality of $K_2Cr_2O_7$ is calculated.

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

The given Fe^{+2} test solution is made up to 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.

Approximately 1/2 Test tube or 10 ml. of dilute H_2SO_4(6N) is added(to provide acidic medium), about 1/4 test tube of syrupy Phosphoric acid is added followed by 3 to 5 drops of internal indicator diphenylamine are added.It is titrated with $K_2Cr_2O_7$ (taken in Burette) very slowly until the solution acquires a green color (due to chromium ions). The titration is continued with drop wise addition of $K_2Cr_2O_7$ and good stirring until addition of a drop causes the formation of deep violet color, which is the end point.

The process is repeated till two concordant title 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 $K_2Cr_2O_7$ not a self-indicator?
2.Why is the color of the contents of conical flask green before the end point?
3.Which substance gives the bluish violet color 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?

Part C: ESTIMATION OF Fe^{+2} IN THE GIVEN TEST SOLUTION.

Titration: Fe^{2+}(Test Soln) Vs. $K_2Cr_2O_7$ (Link)

Sl No.	$V_{Fe^{2+}}$ (test Sol.)ml	Burette Reading		$V_{K_2Cr_2O_7}$ (ml)
		Initial	Final	

$$N_{Fe^{2+}}(\text{Test}) = \frac{N_{K2Cr2O7} \times V_{K2Cr2O7}}{V_{Fe^{2+}}} = \ldots\ldots\ldots$$

Wt. of Fe^{2+} = $N_{Fe^{2+}}$ X Eq. Wt.= ------------------------
(in Test Solution)

Results:

1.Weight. of FAS= ______________g
2.$N_{std.}$ FAS= ______________
3.$N_{K2Cr2O7}$ (Link)= ______________
4.$N_{Fe^{2+}}$ (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

WATER ANALYSIS
COMPLEXOMETRIC TITRATION

EXPERIMENT (3): ESTIMATION OF TOTAL HARDNESS OF WATER USING STANDARD.$MgSO_4$ AND EDTA SOLUTION.

Aim: To estimate the total hardness of water sample using STD $MgSO_4$ solution and 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 and effluents.

Hardness of water is due to the presence of salts of calcium, magnesium and other metal ions. Hardwater does not give lather easily with soap.Units of Hardness is PPM or mg/l expressed in terms of calcium carbonate equivalents.

TYPES OF HARDNESS IN WATER:

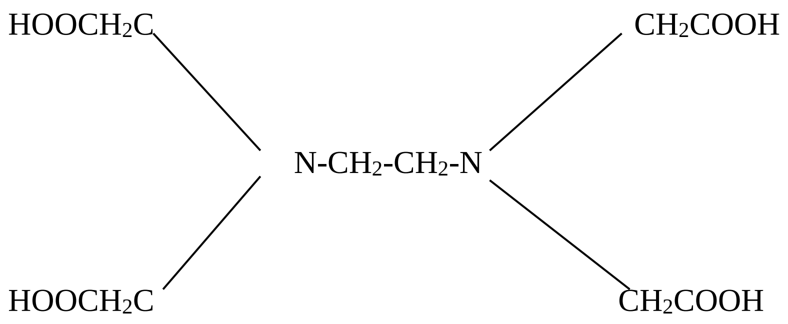
Temporary Hardness is due to the presence of soluble salts of Bicarbonates of Calcium and Magnesium. It can be removed by boiling.On boiling, bicarbonates are converted into insoluble carbonates.

Permanent Hardness is due to the presence of soluble salts of Chlorides and Sulphates of Calcium and Magnesium.It can be removed by only chemical treatment and ion exchange method.

Total Hardness = Temporary Hardness + Permanent Hardness

EDTA =Ethylene Di amine Tetra Acetic acid

EDTA is a strong complexing agent and is easily commercially availabe. In its unreacted form EDTA is a Tetra basic acid (H_4Y) represented by:



(The disodium salt of EDTA, (Na_2H_2Y) available commercially is $CH_2.N(CH_2.COOH)_2$ $CH_2.N(CH_2COONa)_2.2H_2O$ with formula wt=372.24. it is also written as Na_2H_2versenate

$2H_2O$ i.e. disodium dihydrogen versenate).It reacts quantitatively with metal ions to form soluble, stable,1:1 complex. EDTA being insoluble in water, it is generally used as sodium salt that readily dissolves in water.EDTA forms stable colorless complexes with calcium and Magnesium metal ions.

The reaction with metal ions is written as: $M^{+2} + H^2Y^{-2} \rightarrow M^{+2}Y^{-2} + 2H^+$ (Y= versene)

PRINCIPLE:

Hardness of water is determined by titrating a known volume of water sample with EDTA,using Eriochrome black T (EBT) as indicator, in the presence of ammonium hydroxide-ammonium chloride buffer solution (P^H 10). When a few drops of EBT indicator are added, it forms wine red coloured complex with metal ions.

M^{+2} + EBT → M-EBT complex

(wine red)

When the solution is titrated, EDTA reacts with free metal ions present in the solution.Near the endpoint, when free metal ions are exhausted in the solution , further addition of EDTA dissociates the M-EBT complex, consumes metal ions and releases free indicator, which is blue in colour.Therefore colour change is from wine red to blue.

M –EBT complex + EDTA→ M- EDTA complex + EBT indicator (Blue)

(Wine-red) (Colorless)

The indicator shows colour change at P^H range 9-11.therefore the P^H of solution is maintained around 9 using NH4OH + NH_4Cl buffer.

The order of stability of complex formed is as follows:

[Ca –EDTA] > [Mg- EDTA] > [Mg – Indicator]

PREPARATION OF SOLUTIONS:

Buffer Solution: 67.5 g. of NH_4Cl + 570 ml of conc. NH_4OH, made up to 1 liter with distilled water.

Indicator: Dissolve 0.5 g. of Eriochrome black ‘T’ in 100 ml of alcohol or methanol.

PROCEDURE:

PART-A: PREPARATION of STD. $MgSO_4$ SOLUTION.

Weigh accurately the given $MgSO_4$. $7H_2O$ (Epsom salt) in a weighing bottle . Transfer the salt into a clean 100ml standard volumetric flask using funnel.. 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 $MgSo_4$solution.

PART-B: STANDARDISATION OF EDTA SOLUTION:

Pipette out 20ml of the standard hardwater into a clean conical flask. Add about 5ml (1/4 test tube) of (NH_4 CI+NH_3) buffer solution of pH = 10 and 3-4 drops of EBT indicator to it. The color of the solution becomes wine red. Titrate this solution against EDTA solution taken in the burette until the wine red colour of the solution changes to blue. Note down the burette reading (V_1) and repeat the titration to get concordant titre values. Calculate the Normality of EDTA

PART-C: ESTIMATION OF TOTAL HARDNESS IN TEST SOLUTION:

Pipette out 20 ml of given Test solution into a clean conical flask. Add about 5ml of (NH_4CI+NH_3) buffer solution of pH = 10 and 3 - 4 drops of EBT indicator to it. The color of the solution becomes wine red. Titrate this solution against EDTA solution taken in the burette until the wine red solution changes to blue. Note down the burette reading (V_2) and repeat the titration to get concordant titre values.

Calculate the Total hardness in the given test solution in PPM and report the results.

EXPERIMENT (3): ESTIMATION OF TOTAL HARDNESS OF WATER USING STD. $MgSO_4$ AND EDTA SOLUTION

Expt. No.____________ Date: ________

TABULATION AND CALCULATIONS

Part A: Preparation of Std. $MgSO_4$

Table.1.weight of bottle + $MgSO_4$

	•				

Table.2.Weight of empty weighing bottle

	•				

Wt. of $MgSO_4 = W_1 - W_2 =$

Nstd $MgSO_4 = \frac{wt}{eq.wt} \times \frac{1000}{Vml} =$ ………………

(V_{ml} = 100ml), Eq,Wt $MgSO_4 . 7H_2O$=246/2= 123

Part B: Standardization of EDTA

Sl No.	$V_{.StdMgSO4}$ Sol ml	Burette Reading		V_{EDTA} Ml (V_1)
		Initial	Final	

$N_{EDTA} = \frac{N_{MgSO4} \times V_{MgSO4}}{V_{EDTA}} =$ …………..

PART C: ESTIMATION OF TOTAL HARDNESS IN TEST SOLUTION

Titration of Test Solution Vs EDTA

Sl No.	$V_{\text{. Test Solution}}$ ml	Burette Reading		V_{EDTA} ml (V_2)
		Initial	Final	

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

Wt in terms of $CaCO_3$ equivalents = $N_{\text{test solution}}$ x Eq. wt $CaCO_3$

= ___________ x50 = ______________gpl.

Total hardness of Test Solution = _________x 1000 =_______________ mg/lit = _________ppm.

RESULTS:

1)Wt. of $MgSO_4$= ___________. 2) Normality of $MgSO_4$= ___________. 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

COMPLEXOMETRIC TITRATION

EXPERIMENT-4: ESTIMATION OF TOTAL AND PERMANENT HARDNESS IN SAMPLE WATER USING STD. $MgSO_4$ AND EDTA SOLUTION

Aim- To estimate the Total ,Permanent and temporary hardness in sample water.

****Introduction,Principle and Procedure part A, part B from Experiment-3**

PART-C: ESTIMATION OF TOTAL HARDNESS IN SAMPLE WATER-

Pipette out 20 ml of sample water into a clean conical flask. Add about 5ml of (NH_4CI+NH_3) 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 (V_2) and repeat the titration to get concordant titre values. Calculate the Total hardness in Sample water ,(students bring the sample water,water used for washing from their home)

PART D: ESTIMATION OF PERMANENT HARDNESS IN SAMPLE WATER-

Take a large quantity of sample hard water, say 100 ml in a beaker.

Boil it to about one-fourth of its volume (to remove temporary hardness).

Filter (through Whatman 42 filter paper) to remove insoluble carbonates of calcium and magnesium.Collect the filtrate in a clean beaker.

Make up the volume of filtrate to the original 100 ml by adding remaining distilled water.(Distilled water is assumed to have zero hardness) Make the solution homogeneous.

Now pipette out 20 ml of this solution into a conical flask. Then repeat the procedure of titration steps as in part C .Let the reading this time be V_3 ml of EDTA.This gives permanent hardness in sample water.

Calculate the Temporary hardness by removing permanent hardness from total hardness in PPM.

NOTE-Estimation of hardness can be done following the above procedure for sample water of any source.

EXPERIMENT (4): ESTIMATION OF TOTAL AND PERMANANT HARDNESS IN SAMPLE WATER

Expt. No.____________ Date: ________

TABULATION AND CALCULATIONS

Part A: Preparation of Std. $MgSO_4$

Table.1.weight of bottle + $MgSO_4$

	.				

Table.2.Weight of empty weighing bottle

	.				

Wt. of $MgSO_4$ = W_1-W_2 =

Nstd $MgSO_4$ = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ = ………………

(V_{ml} = 100ml), Eq,Wt $MgSO_4$. $7H_2O$=246/2= 123

Part B: Standardization of EDTA

Sl No.	$V_{.StdMgSO4}$ Sol ml	Burette Reading		V_{EDTA} ml (V_1)
		Initial	Final	

N_{EDTA}=$\frac{N_{MgSO4} \text{ x } V_{MgSO4}}{V_{EDTA}}$ = …………..

PART C: ESTIMATION OF TOTAL HARDNESS IN SAMPLE WATER.

Titration of Sample water Vs EDTA

Sl No.	V. Sample water (ml)	Burette Reading		V_{EDTA} ml (V_2)
		Initial	Final	

$$N_{\text{sample water}} = \frac{N_{EDTA} X\ V_{EDTA}}{V_{\text{Sample water}}} =$$

Wt in terms of $CaCO_3$ equivalents = $N_{\text{Sample water}}$ x Eq. wt $CaCO_3$

= __________ x50 = _____________gpl.

Total hardness in sample water = _________x 1000 =______________ mg/lit = _________ppm.

PART D: ESTIMATION OF PERMANANT HARDNESS IN SAMPLE WATER –

Titration of boiled/filtered hard water Vs EDTA

Sl No.	$V_{\text{boiled/filtered water}}$ ml	Burette Reading		V_{EDTA} ml (V_3)
		Initial	Final	

$$N_{B/F \text{ sample water}} = \frac{N_{EDTA} X\ V_{EDTA}}{\text{VB/F Sample water}} =$$

Wt in terms of $CaCO_3$ equivalents = N $_{\text{B?F Sample water}}$ x Eq. wt $CaCO_3$

= __________ x50 = _____________gpl.

Permanant hardness in sample water = _________x 1000 =______________ mg/lit = _________ppm.

Temporary hardness = (Total-permanent) hardness = ……………. . = ……………. ppm.

RESULTS: Sample Water

1)Wt. of $MgSO_4$= __________.

2) Normality of $MgSO_4$= __________.

3) Normality of EDTA = __________.

4) Normality of Sample water= __________.

5) Total Hardness in Sample Water= ________ppm

1) Source: O/W, B/W= __________.

2) Depth (approximately) = __________.

3) Locality= __________.

4) Total Hardness = __________.

5) Permanent Hardness = __________.

6) Temporary Hardness = __________.

Marks

Observations and calculations (20):______

Results (10):_____________

Discussion of results (5):_____________

Record (15):______________

Total (50):______________

Signature of Faculty

WATER ANALYSIS

ACID-BASE TITRATION

Expt (5): 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.

$$CO_3^{2-} + H^+ \rightarrow HCO_3^- \text{--------(1)}$$
$$HCO_3^- + H^+ \rightarrow H_2CO_3 \rightleftharpoons H_2O + CO_2 \quad \text{--------(2)}$$

The volume of acid run down up to phenolphthalein end-point, [P] corresponds to the completion of equation (1) , half neutralization of carbonates. (Then 2[P]=vol of acid for complete neutralization of carbonates.

The amount of total acid consumed 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).i.e, neutralization of bicarbonates initially present in the test solution mixture and also the bicarbonates from half neutralization of carbonates. (Then [M-2P]=vol. of acid for neutralization of bicarbonates alone.

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:

PART A: PREPARATION OF STANDARD N/50 Na_2CO_3 SOLUTIONS:

Weigh out in a clean weighing bottle accurately to about 0.2622g of Na_2CO_3; 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 Na_2CO_3 solution.

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

Pipette out 20ml of the prepared std. Na_2CO_3 solution into a clean conical flask. Add 2 drops of methyl orange indicator. The solution becomes yellow. Titrate with the given HCl solution (taken in the burette) until reddish pink appears, this is the end point. Repeat the process of titration till at least two concordant values are obtained.

PART C: ESTIMATION OF CO_3^{2-} & HCO_3^- ALKALINITY IN TEST SOLUTION

Make up the given test solution(mixture CO_3^{2-}&HCO_3^-) up to the mark by adding distilled water. Make the solution homogeneous. Pipette 20 ml of test solution in a clean conical flask.

i) Add 2 drops of phenolphthalein indicator,the solution turns light pink then titrate the with standard HCl until the pink color just disappears (colourless)-note the titre value as phenolphthalein end point [P].

Add 2 drops of methyl orange indicator to the same solution, the solution turns yellow. Continue the titration from [P]end point until color changes from yellow to reddish pink. Note the titre value as [M] end point.

ii) Repeat the titration by pipetting 20ml test solution by following procedure of step (i), but in step (ii), instead of methyl orange another indicator ,ie, mixture of BCG (bromo cresol green) and methyl red can be used for better contrast at the end point.

AN EXAMPLE FOR CALCULATIONS: 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-} = 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:$\boldsymbol{CO_3^{2-}}$ **Alkalanity-**

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

$$\text{Wt. of } CO_3^{2-} = \frac{NCO_3^{2-}\ x\ eq.wt}{1000} \text{ x } 1000\text{g/l} = 0.00416 \text{ x } 30 \text{ g/1} = 0.1248 \text{ g/l}$$

Wt. of $CaCO_3$eqt. = 0.124 x $\frac{50}{30}$ g/1 = 0.208 g/1 = 208 mg/1 =208 ppm.

i) $\boldsymbol{HCO_3^{-}}$ **Alkalanity**

$$NHCO_3^{-} = \frac{N_{HCl} \text{ X } V_{HCl}}{VHCO_3^{2-}} = \frac{0.02 \text{ x } 4.9}{100} = 0.00098$$

Wt. of HCO_3^{-} = $N_{HCO^{-}_{3}}{}^{x\ eq.wt.}$ x 1000 g/l = 0.00098 x 61 g/l = 0.05978 g/l

Wt. of $CaCO_3$eqt. = 0.05978 x $\frac{50}{61}$ g/1 = 0.049g/1 =

Bi-carbonate alkalinity= 49mg/1 =49 ppm.

Experiment (5) : ESTIMATION OF CARBONATE AND BICARBONATE ALKALINITY IN WATER

Expt No. Date:________

Part A : Preparation of Std. Na_2CO_3:

Table.1.weight of bottle + Na_2CO_3

	•			

Table.2.Weight of empty weighing bottle

	•			

Wt. of $Na_2CO_3 = W_1 - W_2 =$

$$N_{\text{Std } Na_2CO_3} = \frac{wt}{eq.wt} \times \frac{1000}{Vml} = \ldots\ldots\ldots\ldots\ldots\ldots$$

(V_{ml} = 250ml)

Part B : STANDARDIZATION OF HCl LINK SOLUTION

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

$$N_{HCl} = \frac{N_{Na2CO3} \times V_{Na2CO3}}{V_{HCl}} = \ldots\ldots\ldots\ldots$$

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				(MeOH- Ind)
2	20				(BCG-Ind)

CALCULATION:-*<u>ESTIMATION OF CO_3^{2-} ALKALINITY</u>*

$$NCO_3^{2-} = \frac{N_{HCl} \text{ X } V_{HCl}}{\text{V } CO_3^{2-}} =$$ (VHCl=[2P])

Wt. of CO_3^{2-} = N CO_3^{2-} x eq.wtof CO_3^{-2} = ___________x 30 = ____________gpl

Wt.in terms of CaCO3^{-2}eq = Wt. of CO_3^{2-} x eqwt of $CaCO_3$/eqwt of CO_3^{-2}

= _____________ x 50 / 30 = _________gpl

= _____________mg/L = ______________ ppm

<u>ESTIMATION OF HCO_3^- ALKALINITY</u>

$$HCO_3^- = \frac{N_{HCl} \text{ X } V_{HCl}}{\text{V } HCO_3^-} =$$ (V_{HC}l=M-2P)

Wt. of HCO_3^- = N HCO_3^- x eqwt of HCO_3^- =_________ x 61 = ____________gpl

Wt.in terms of $CaCO_3$eq = Wt. of HCO_3^- x eq wt of $CaCO_3$/eqwt of HCO_3^-

= _____________ x 50 / 61 = ____________gpl

= _____________mg/L = ______________ ppm

RESULTS:

FOR TEST SOLUTIONS:-

1)[P] = ……..ml; [M]= ………ml; [M-2(P)]=……..ml

2)$N_{CO3}{}^{2-}$ =……….

3)Wt. of $N_{CO3}{}^{2-}$ in mixture = ……gpl.

4)Carbonate Alkalinity = ………ppm

5)N_{HCO3-}= ………..

6)Wt. of N_{HCO3-} 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 Bicarbonate ions-------------?

Marks:

Observations and calculations (20):______

Results(10):_____________

Discussion of results (5):___________

Record (15):______________

Total(50):______________

Signature of Faculty

Viva Questions

EXPERIMENT No. 1: FAS X $KMnO_4$X Fe^{+2}

1) Write the chemical name and formula of FAS.
2) What is ionic equation for the overall reaction between FAS and $KMnO_4$.
3) Why dil.H_2SO_4 is added to prepare std. FAS solution in Volumetric flask.
4) Why dil.H_2SO_4 is added to FAS during the titration of FAS Vs $KMnO_4$ in conical flask.
5) Why indicator is not required for FAS Vs $KMnO_4$ 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 $KMnO_4$ changes to Mn^{+2}
10) Name the primary and secondary standard solutions in this experiment.

EXPERIMENT No. 2: FAS X $K_2Cr_2O_7$ X Fe^{+2}

1) What is meant by redox titration?
2) What is dichrometry and permanganometry?
3) What is the oxidation number of Cr in $K_2Cr_2O_7$.
4) Why the acids (H_2SO_4 and H_3PO_4) are added to FAS solution during titration?
5) Name the indicator used in $K_2Cr_2O_7$ titration?
6) What is the colour change at the end point.
7) What is the difference between titration of $KMnO_4$and $K_2Cr_2O_7$ with FAS?
8) Name the primary standard solution and link solution and test solution in this experiment?.
9) Can $K_2Cr_2O_7$ used as primary standard solution? Why? Why Not?
10) Name the species gives green colour and blue colour in the titration?
11) Why $K_2Cr_2O_7$ do not act as self indicator?

EXPERIMENT No. 3: ESTIMATION OF HARDNESS BY EDTA METHOD

1) What is the difference between hard water and soft water?
2) What are the salts causing temporary and permanent hardness?
3) How is temporary hardness removed? What is the chemical change.
4) Write the structure and name of EDTA.
5) What is the type of titration, 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 contains0.555g of $CaCl_2$?
9) Why hardness is expressed in terms of CaCO3 equivalents.
10) What are the units of hardness?

EXPERIMENT No. 5: ESTIMATION OF ALKALANITY 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 is measured in terms of $CaCO_3$ equivalents?

5) Temporary hardness is also known as ___________alkalinity.

6) What is the application of alkalinity measurement?

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) [M] endpoint indicates neutralization of which ions.

INSTRUMENTAL CHEMICAL ANALYSIS

Introduction:

Quantitative chemical analysis can also be done with the help of various analytical instruments like Colorimeter, Conductometer, P^H meter, Potentiometer and Spectrophotometer.

Advantages of Instrumental analysis:

- Low concentration of sample required.
- Highly sensitive and selective
- Reliable measurements.
- Fast determination.
- Indicators are not required.
- Very small amount of salt can be estimated.

Method	Instrument	Phenomenon underlying the method	Quantity measured
Optical methods			
1. Colorimetry	1.colorimeter	Interaction of matter with Electromagnetic radiation	Absorbance (OD) Or Transmittance
2. SpectroPhoto-metry	2.Spectrophoto-meter		
Electrochemical methods		Changes during chemical reactions	
1.Conductometry.	1.Conductometer	Conductivity of the solution	Conductance
2.pH metry	2.pH meter	H^+ ion concentration of the solution	pH
3.Potentiometry	3.Potentiometer	Electrode potential difference of the cell	Emf

CONDUCTOMETRY

The electrical conduction in electrolytes is due to flow/movement of the ions followed by their electrode reaction at the anode and cathode respectively. This amounts to indirect transfer of alter electrons through the electrolyte (conduction in liquids) as free electrons cannot exist in solution .During the measurement of conductivity of the solution, the concentration of electrolyte (C) and the applied e.m.f. (E) must be constant. This is possible by using alternate current (A/C) of high frequency and coating the platinum electrodes with platinum black (to prevent the back e.m.f.).

CONDUCTIVITY CELL:

Conductivity cell is a special type of cell used for measuring the conductance of an electrolytic solution. The cell consists of two electrodes. Each electrode is a platinum disc or plate coated with finely divided platinum black. The electrode is connected to a platinum wire which is fused to a glass tube. The glass tubes are firmly fixed in the cell so that the distance between the electrodes would not change during the experiment. The cell constant is fixed for the conductivity cell. The cell is open at one end. An aqueous solution of the electrolyte whose conductance has to be measured is placed in a beaker. The conductivity cell is kept in the solution and is connected to the conductivity meter.

CELL CONSTANT:

The cell constant (x) of a conductivity cell is the ratio of the distance between the two electrodes to the area of cross-section of each electrode.

$$X = \text{Cell Constant} = \frac{\text{Distance between the electrodes}}{\text{Cross-sectional area of electrode}} = \frac{l}{a}$$

Units of cell constant = cm^{-1}; m^{-1}

*There are no foolish questions, & no man becomes a fool until he has stopped asking questions.

SPECIFIC CONDUCTIVITY (K):

Specific conductivity is the conductivity by the ions present in one cubic centimeter of the solution. If the cell constant of a conductivity cell is 1.0, the measured conductivity of the electrolyte will also be its specific conductivity. The specific conductivity of an electrolyte is expressed in ohm^{-1} cm^{-1}, mho cm^{-1}, S.cm^{-1}, S.m^{-1}.

$$\text{Cell Constant} = \frac{\text{Specific conductance of KCl}}{\text{Measuredconductance of KCl}}$$

CONDUCTOMETRIC ACID – BASE TITRATIONS

THEORY:

The electrical conductance of an electrolytic solution depends on :

i) Number of ions present in solution i.e., ionic concentration.
ii) The ionic mobilities (size of ions)
iii) Temperature of the solution
iv) Nature of electrolyte
v) Dilution of solution

The ionic mobilities of various ions in aqueous solution at 25°C are generally in the range. 4.8×10^{-4} cm sec^{-1} under potential of 1 Volt cm^{-1}. But the ionic mobilities of H^+ and OH^- are abnormally high ($H^+ = 36.8\times10^{-4}$, $OH^-=20.5 \times 10^{-4}$ cm2 sec^{-1} Volt^{-1}). Thus the conductance of an electrolyte is quite sensitive to the concentration and ionic mobilities in acid-base titrations of H^+ and OH^- ions.

In the conductometric acid-base titrations the course of the titration as base is added to the base, is followed from the gradual change in the conductivity of the titrand.

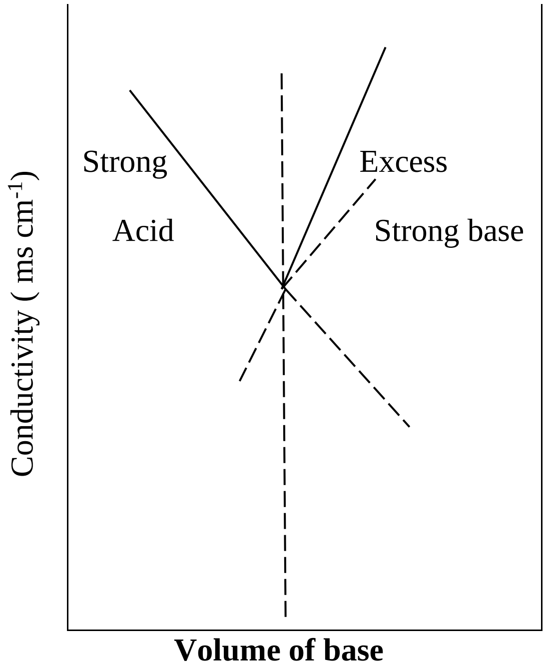
> *The important thing is not to stop questioning
>
> - Einstein.

1. Titration of Strong Acid (HCl) with Strong Base (NaOH)

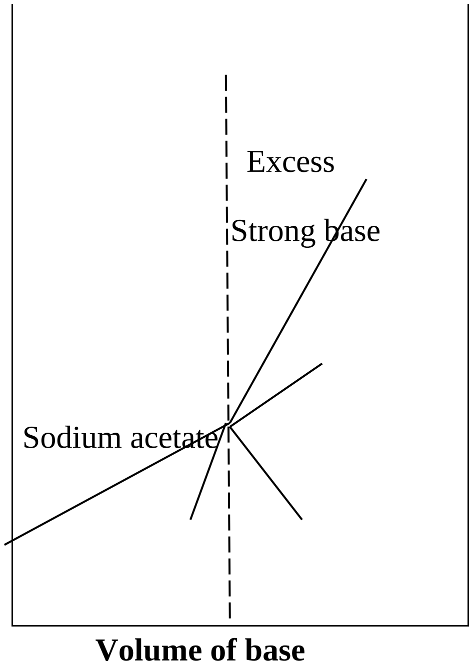
In a solution HCl is completely ionized. The mobility of H^+ ions is high, hence the conductance of HCl solution will also be high. As NaOH is added to HCl, the fast moving H^+ ions are removed by OH^- ions of the base as water.

$H^+ + Cl^- + Na^+ + OH^- \rightarrow Na^+ + Cl^- + H_2O$ (Undissociated)

In solution water is little ionized and Na^+ ions added are slow moving compared to H^+ ions of the acid. Hence, as the titration proceeds, the conductance of the titrand (HCl) decreases gradually till theequivalence point is reached. At equivalence point the conductance of HCl will be minimum. Addition of the base beyond the equivalence point results in a gradual increase in conductance mainly due to the fast moving OH^- ions. Model graph for titration of (A) Strong acid Vs Strong base and (B) Weak acid Vs Strong base is given below.



GRAPH- (A)



GRAPH- (B)

The plot of the conductance of the solution against the volume of the titrant (base) added gives two straight lines. The point of intersection of the interpolated lines is the equivalence point of the titration.

*No problem can withstand the assault of sustained thinking

2. Titration of Weak Acid (CH_2COOH) with Strong Base (NaOH)

The conductance of an aqueous solution of acetic acid will be low, because of the low concentration of H^+ ions, as acetic acid is feebly ionized. As NaOH is added, **highly ionized sodium acetate** is formed and effectively the net result of the titration is the replacement of unionized CH_3COOH molecules by the highly ionized sodium acetate. Therefore there will be a gradual increase in the conductance.

$CH_3COOH + Na^+ + OH^- \rightleftharpoons CH_3COO^- + Na^+ + H_2O$

After the equivalence point, any further addition of NaOH will produce a more rapid increase in conductance due to highly mobile OH^- ions. The point of intersection of the lines, in the plot of the conductance against the volume of the alkali added, is the end point.

3. Titration of a mixture of strong and weak acids

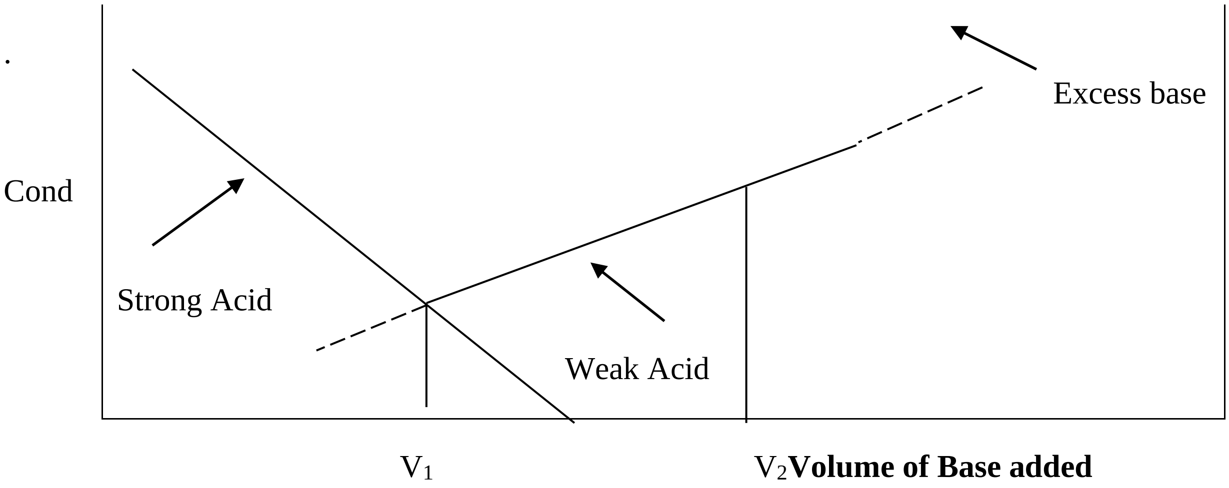
(HCl + CH_3COOH) against strong base (NaOH)

In the mixture of acids, the H^+ ion concentration is almost exclusively from HCl. When the base is added HCl will react first, as indicated by a gradual decrease in conductance. When all the HCl is used up, CH_3COOH starts reacting, leading to a gradual increase in conductance. Beyond the equivalence point with the addition of excess NaOH conductance will shoot up. The Plot of the conductance Vs the volume of alkali added will show two independent end points on the graph, first one is equivalence point for the end point of HCl Vs NaOH and the second one for the end point of (HCl + CH_3COOH) Vs NaOH.

V_1= Volume of NaOH required for neutralization of HCl.

(V_2-V_1) = Vol. of NaOH required for neutralization of CH_3COOH

*Reading without reflecting is like eating without digesting.



Procedure:

Preparation of Std. Oxalic Acid Solution ($C_2H_2O_4 .2H_2O$):

Weigh about 0.63 g. of oxalic acid, in a weighing bottle. Transfer it into a clean 100 ml std. flask through funnel. Dissolve it in distilled water and make a homogeneous solution upto the mark. From the wt. of Oxalic acid calculate the normality of std solution.

A.Standardisation of NaOH (link) solution:

Pipette out 20 ml of the oxalic acid solution into a clean conical flask. Add 2-3 drops of phenolphthalein indicator. Titrate the contents of the flask till a light pink colour appears. Repeat the titration to get two concordant titre values.

Requirements: Conductivity meter, conductivity cell, burette, pipette, beakers.

Chemicals: HCl : ~0.05N ($N/_{20}$)

CH_3COOH : ~0.05N ($N/_{20}$)

NaOH : ~0.5N ($N/_2$)

How to use conductivity Meter:

1. Connect the instrument to A/C mains and Switch it on.
2. Take out the conductivity cell from the water beaker. Rinse it with distilled water and wipe it with tissue paper.
3. Dip the conductivity cell in the respective (test) solution.
4. Press the cell switches to **‘1’.**
5. Select the conductivity range by pressing either 200mho (for strong acid) or 20mho switch (for weak & mixture of acids).
6. For calibration-Adjust the temperature control knob to 25°C.
7. Calibrate the conductivity meter by pushing the MEAS/CAL switch to CAL position **(release the button)** and adjust CAL control knob to get 100.0 displayed on the read out (for conductivity range 200 ms range) and 10.00 is displayed (for conductivity range 20 ms)
8. For measurement **-** adjust the temperature control knob to the room temperature (30°C)
9. Read the conductivity of the solution by pressing CAL/MEAS switch to MEAS position **(pressed).**
10. At the end of the experiment follow the winding up instructions.

Note: Do not take out the conductivity cell from the solution when switch is in MEAS position (pressed).

B. Estimation of amount of HCl/CH_3COOH present in the given test solution using conductometry.

Procedure:

Make up the given test solution (HCl/CH_3COOH or HCl + CH_3COOH) upto the mark. Shake it thoroughly to make the solutions homogenous. 40 ml of the test solution is placed in a 100 ml beaker. The conductivity cell is dipped in this solution. The initial conductance is measured before titration. Now, NaOH is added from the burette in intervals of 0.5 ml. After the addition of each instalment of NaOH, the solution is mixed gently with a stirrer. Conductance is measured. Conductance is plotted against the volume of the alkali added. From the graph the neutralization point is obtained.

The above experiment is also done using an acid mixture (containing HCl and CH_3COOH)

From the graph equivalence points for each acid is obtained.

<u>Application of conductometric measurements of aqueous and non-aqueous solutions:</u>

Used in cooling towers, reverse osmosis, steam boilers, acid, salt and alkali concentrations,

Industries which use conductance:-

Chemical, Power generation, hospitals, textiles, agriculture, food processing, brewing, petroleum etc.

<u>Questions:</u>

1. In the titration of HCl vs NaOH even at the neutralization point some conductivity is observed. Why?
2. What are the advantages of instrumental Chemical analysis over conventional volumetric analysis?
3. The initial conductance in a solution of HCl is due to which ions?
4. Name the factors which affect the conductivity of a solution?
5. How can you calculate molecular and equivalent conductivities of a solution from observed conductivities?
6. Why the concentration of base is kept 10 times higher than the concentration of acids?
7. Is it possible to determine the volume of strong and weak acids in a mixture?

Expt. (6) Conductometric Titration of Strong Acid Vs Strong Base

Date:

Expt No:

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution ($C_2H_2O_4 .2H_2O$) :

Table.1.weight of bottle + Oxalic Acid

	.				

Table.2.Weight of empty weighing bottle

	.				

Wt. of Oxalic Acid = W_1-W_2 =

$N_{(std)}$ Oxalic Acid = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ =

(V_{ml} = 100ml)

Eq Wt of Oxalic acid=126/2=63

Part B : Standardization NaOH

Titration of Std. Oxalic AcidVs. NaOH (link)

Sl No.	$V_{oxalic\ acid}$ (ml)	Burette Reading		V_{NaOH} (ml)
		Initial	Final	

$$N_{NaOH} = \frac{N_{oxalic} \text{ x } V_{oxalic}}{V\ NaOH}$$

Part – C Estimation of HCl in the test solution:

Table: Conductometric titration of HCl X NaOH

S.No.	V_{NaOH} (ml)	Conductivity ($mscm^{-1}$)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V\ HCl}$$

=

V_{NaOH} = Inflection point from the graph

$Wt_{HCl} = N_{HCl} \times 36.5 =$

Results:

1. Weight of Oxalic Acid = ____________g
2. N_{std}. Oxalic Acid = ____________
3. N_{NaOH} (Link) = ____________
4. VNaOH end point (from graph) = ____________ (ml)
5. N_{HCl} (Test) = ____________
6. Weight of HCl in test solution = ____________ gpl

Experiment (7): Conductometric Titration of Weak Acid Vs Strong Base

Part – C Estimation of CH_3COOH in the test solution:

Table: Conductometric titration of CH_3COOH X NaOH

S.No.	V_{NaOH} (ml)	Conductivity ($mscm^{-1}$)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

$$N_{CH3COOH} = \frac{N_{NaOH} \times V_{NaOH}}{V_{CH3COOH}}$$

V_{NaOH} = Inflection point from the graph

$Wt_{CH3\ COOH} = N_{CH3\ COOH} \times 60$ = ___________gm/lit

Marks

Observations and calculations (20):______

Results (10):_____________

Discussion of results (5):______________

Record (15):______________

Total(50):______________

Results:

1. Weight of Oxalic Acid = ___________g
2. N_{std}. Oxalic Acid = ____________
3. N_{NaOH} (Link) = ____________
4. V_{NaOH} @ end point (from graph) = ____________ (ml)
5. $N_{CH3\ COOH}$ (Test) = ____________
6. Weight of CH_3 COOH test solution = ____________ gpl

Signature of Faculty

*Inaction saps the vigor of the mind.

Expt (8) Conductometric Titration of Mixture of Acids Vs Strong Base

Date:

Expt No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.				

Table.2.Weight of empty weighing bottle

	.				

Wt. of Oxalic Acid = W_1-W_2 =

$N_{(std)}$ Oxalic Acid = $\frac{wt}{eq.wt} \times \frac{1000}{Vml}$ =

(V_{ml} = 100ml)

Part B : Standardization NaOH

Titration of Std. Oxalic. Vs. NaOH (link)

Sl No.	V_{oxalic} (Std) (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

Marks

Observations and calculations (20):______

Results (10):_____________

Discussion of results (5):______________

Record (15):______________

Total(50):______________

$$N_{NaOH} = \frac{N_{oxalic} \times V_{oxalic}}{V\ NaOH}$$

Part – C -Estimation of HCl and CH_3COOH in the test solution:

Table: Conductometric titration of HCl + CH_3COOH X NaOH

S.No.	V_{NaOH} (ml)	Conductivity ($mscm^{-1}$)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		

Note: Vol of HCl and CH_3COOH =40ml

$$\mathbf{N_{HCl}} = \frac{N_{NaOH} \text{x } V_{NaOH}}{V_{HCl}}$$

=

V_{NaOH} = Inflection point from the graph (V_1) =

Wt_{HCl} = N_{HCl} x 36.5

$$\mathbf{N_{CH3\ COOH}} = \frac{N_{NaOH} \text{x } V_{NaOH}}{V_{CH3\ COOH}}$$

V_{NaOH} = Inflection point from the graph (V_2- V_1)

$Wt_{CH3\ COOH}$ = $N_{CH3\ COOH}$ X 60=____________gm/lit

Results:

1. Weight of Oxalic Acid= ____________g
2. $N_{std.Oxalic\ Acid}$= ____________
3. N_{NaOH} (Link) = ____________
4. V_{NaOH} at end point (from graph) for HCl= ____________ (ml)
5. N_{HCl} (Test) = ____________
6. **Weight of HCl in test solution** = ____________ gpl
7. V_{NaOH} at end point (from graph) for CH_3COOH = ____________
8. $N_{CH3COOH}$ (Test) = ____________
9. **Weight of CH_3COOH in test solution**. = ____________gpl

POTENTIOMETRY

THEORY:

In Potentiometry, cell potential measurement are taken for the location of the end point in titrimetric methods of analysis like acid-base, redox titrations etc

The equipment used consists of a potentiometer (a potential measuring device) a reference electrode and an indicator electrode.

The half-cell potential of the reference electrode is a known constant and this electrode is completely insensitive to the composition of the solution under study. Usually, saturated calomel electrode is used as a reference electrode in potentiometric titrations.

Along with the reference electrode, an indicator or working electrode is also employed which responds to the changes in the concentration of the solution under study.

A cell is constructed by combining the reference electrode an indicator electrode and the test solution in the cell is titrated against the standard solution. During the course of the titration, the changing values of cell potential are noted down and later plotted as a graph from which end point of the titration is noted and concentration of the test solution is calculated.

Examples of Potentiometry Redox titrations:

1) Fe^{2+} x $KMnO_4$
2) Fe^{2+} x $K_2Cr_2O_7$

Examples of Potentiometry Acid – Base titrations:

i) Strong Acid (HCl) x Strong Base (NaOH)
ii) Weak Acid (CH3COOH) x Strong Base (NaOH)
iii) Mixture of Acids (HCl + CH3COOH) x Strong base (NaOH)

*Begin Challenging your own assumptions your assumptions are your windows on the world.

Scrub them off every once in a while, or the light won’t come in.

Exp-(9)-Estimation of Fe^{2+}Vs $KMnO_4$

Aim: To titrate Potentiometrically Fe^{2+} against standard $KMnO_4$

Theory: The reference electrode used here is saturated calomel electrode (SCE). It consists of mercury metal covered with a paste of $Hg + Hg_2 Cl_2 \downarrow$ in contact with saturated KCl solution and Pt-Wire for electrical contact. The reduction potential of this electrode is 0.242V. This saturated calomel electrode functions as ANODE.

The **Indicator electrode** is a platinum electrode which responds rapidly to oxidation- reduction couples and senses the potential which depends upon the concentration ratio of the reactants & products of redox reactions. Here, the Pt electrode is in contact with a Ferrous-Ferric couple. This electrode functions as CATHODE.

Cell Representation:

(-) Pt, $Hg_{(1)}$ | , $Hg_2 Cl_2(s)$ | $KCl_{(sat)}$ || Fe^{3+}, Fe^{2+} | Pt (+)

Cell Reaction:

Anode:- $2 Hg + 2CI^- \rightarrow Hg_2Cl_2 + 2e^-$

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

Cell E.m.f.:

$$E_{cell} = E^o (Fe^{3+} / Fe^{2+}) + \frac{2.303RT}{F} \log \frac{[Fe^{3+}]}{[Fe^{2+}]} - E_{SCE}$$

The cell potential is measured during the course of reaction and graphs are plotted. From the graphs end point of the titration is located and concentration is calculated.

Requirements: Saturated calomel electrode, platinum plate electrode, potentiometer, beakers (100ml) burette, pipette, stirrer and salt bridge.

Chemicals:

i) Ferrous ammonium sulphate (~N/80) Test solution.
ii) Potassium permanganate (~N/40) solution

PROCEDURE:

PART-A: PREPARATION OF STANDARD F.A.S. SOLUTION:

The given Mohr salt (F.A.S.) is weighed accurately in a clean weighing bottle and transferred into a clean 100 ml standard flask through a funnel. It is then dissolved in ½ test tube (~10 ml) of dil $H2SO_4$ and the solution is made up to the mark with distilled water. The solution is shaken thoroughly to make it homogeneous. From the weight of FAS the Normality of Standard solution is calculated.

PART-B: Standardization of $KMnO_4$ solution:

20ml of the prepared standard FAS solution is pippeted out into a clean conical flask. Approximately (1/2 Test Tube) 10 ml of dilute H_2SO_4(6N) is added to provide acidic medium. It is then titrated with $KMnO_4$ taken in burette very slowly until the solution acquires pale pink colour which persists for at least a minute as end point. The titre value is noted.

The process is repeated till 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:

1. Make the given test solution of Fe^{+2}, up to the mark by adding distilled water. Make it homogeneous.

2. Connect the potentiometer to A/C mains, Switch it on.

3. Pipette out 20 ml of test solution in a clean 100 ml beaker, place the platinum electrode in the solution, which creates a Fe^{+2}/Fe^{+3} couple. Connect the electrode to pH terminal.

4. In another 100 ml beaker a Saturated calomel electrode (SCE) is placed in KCl(Sat.) solution. This is connected to R.E. terminal.

5. The two solutions are connected by means of salt bridge to form the Galvanic cell
   $^{(-)}PtHg_{(1)}, \mid Hg_2\,Cl_2(s) \mid KCl_{(sat)} \parallel Fe^{+3}, Fe^{+2} \mid Pt^{(+)}$

6. Switch on the instrument. Keep the mv current RED switch in pressed position (mv mode). The display reads EMF of cell in mill volts. Adjust the initial EMF to a present value with right side knob.
7. Add $KMnO_4$ from burette in 1 ml portions to the ferrous solution, stir it and note the EMF. (Table-1).
8. Continue the titration till a sudden inflection in EMF occurs. Then take about 5 to 6 readings after inflection in 1 ml INTERVALS.
9. From the titrations approximate volume of $KMnO_4$ required is found out.

*Invest a few moments in. It will pay good interest.

10. The titration is repeated with addition of $KMnO_4$ in 0.1 ml lots in the vicinity of end point (In 2 ml range). (Table-2).

11. Draw a graph of E_{cell}Vs volume of $KMnO_4$ added; the inflexion point gives an approximate equivalence. From table-1

12. Differential graph is drawn by plotting $\frac{\Delta E}{\Delta V}$ (Y-axis) Vs V_{KMnO4} (X-axis) to get a sharp peak, which corresponds to the precise equivalence point of titration. From table-2

TABULATION

Table : 1.

S.No.	V_{KMnO4}	Ecell
1		
2		
3		

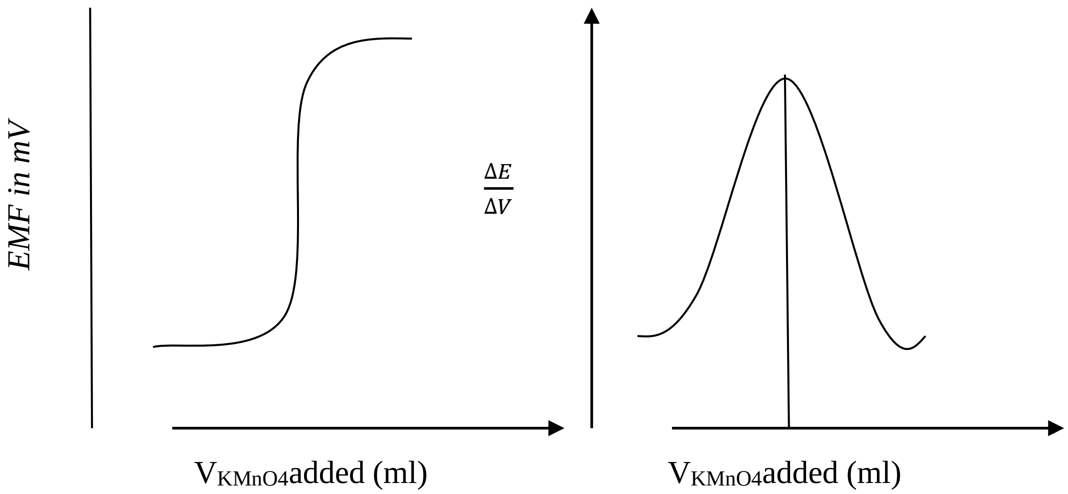
[Draw 17 lines in full page]

Table : 2.

S.No.	V_{KMnO4} (ml)	Ecell (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				

[Draw 25 lines in full page]

GRAPHS



(A) (B)

*Genius is nothing but a great aptitude for patience..

Exp. No. (9) POTENTIOMETRY REDOX TITRATION

Date:

Exp. 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 =

$N_{(std)}$ FAS = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ =

Part B : Standardization of $KMnO_4$ solution.

Titration of Std. FAS. Vs. $KMnO_4$ (link)

Sl No.	V_{Fe}^{2+} (ml)	Burette Reading		V_{KMnO4} ml
		Initial	Final	

$$N_{KMnO4} = \frac{N_{FAS} \text{ x } V_{FAS}}{V_{KMnO4}}$$

PART-C: Estimation of Fe^{+2} in the given test solution:

Table : 1.

S.No.	Vol of $KMnO_4$ added (ml)	E_{cell} (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

Table : 2.

S.No.	V_{KMnO4} (ml)	E_{cell} (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{Fe^{2+}} = \frac{N_{\mathbf{KMnO4}} \times V_{\mathbf{KMnO4}}}{V_{Fe^{2+}}}$$

$V_{\mathbf{KMnO_4}}$ = Peak from differential graph –B)

Wt of Fe^{2+} = NFe^{2+} x 56 =

= ------------------------------gpl

Results:

1. Weight of FAS = ____________g
2. N_{std}. FAS = _____________
3. N_{KMnO4} (Link) = _____________
4. V_{KMnO4} @ end point (from graph) = _____________ (ml)
5. NFe^{2+} (Test) = ______________
6. Weight of Fe^{2+} in test solution = _____________ gpl

Questions:

1. Which property or parameter is measured in Potentiometry?
2. Which electrode acts as anode and which one as cathode in this experiment?
3. What are the reactions taking place at anode and cathode?
4. What is salt bridge? What is its role?
5. Why emf of cell increases on addition of $KMnO_4$ to Fe^{2+} solution?

Marks

Observations and calculations (20):_______________

Results & Graphs (10):______________

Discussion of results (5):________________________

Record (15):________________________

Total (50):________________________

Signature of Faculty

*The world we have created is a product of our thinking it cannot be changed without changing our thinking.

-Einstein

Exp. No. (10) POTENTIOMETRY ACID-BASE TITRATION

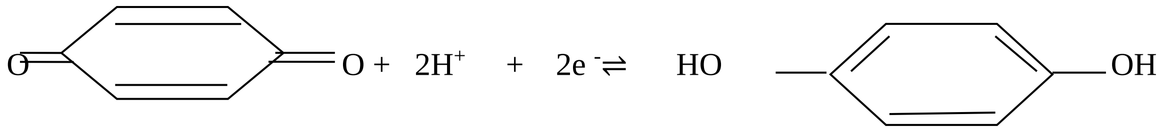
ESTIMATION OF HCl Vs NaOH (Strong Acid Vs Strong Base)

Aim: To Titrate potentiometrically HCl against NaOH.

Theory: Reference electrode used here is saturated calomel electrode (SCE) with reduction potential 0.242V. This electrode functions as ANODE.

The indicator electrode in Quinhydrone electrode (QE). Quinhydrone is an equimolar

mixture (1:1) of Quinone (Q) and Hydroquinone (QH_2). When a pinch of quinhydrone is added to an acid solution, in contact with Pt electrode the following equilibrium is set up.



Quinone

Hydroquinone

This electrode is reversible with respect to H_3O^+ ions in the solution. The reduction potential of this system is given by

$$E_{Q/QH_2} = E^{o}_{Q/QH_2} - 0.0591P^{H}$$

Where E^{o} Q/QH_2 (SRP of Quinhydrone electrode) is 0.6996V at 25°C. The potential of Quinhydrone electrode depends on P^H of the solution. This Quinhydrone electrode functions as CATHODE.

The electrochemical cell is constructed by combining the saturated calomel electrode (SCE) with the quinhydrone electrode (QE).

*Few minds wear out; most rust out.

Cell Representation:-

$$(-)\ Pt \mid Hg_{(1)}, Hg_2Cl_2(s) \mid KCl_{(sat)} \parallel Q, QH_2 \mid Pt^{(+)}$$

Cell Reaction:-

Anode:- $2Hg + 2Cl^- \rightarrow Hg_2Cl_2 + 2e^-$

Cathode:- $Q + 2H^+ + 2e^- \rightarrow QH_2$

<u>Cell E.m.f:-</u>

$E_{cell} = E_{QE} - E_{SCE}$

$= \left[E^{o}_{QE-}0.0591pH\right] - 0.242$

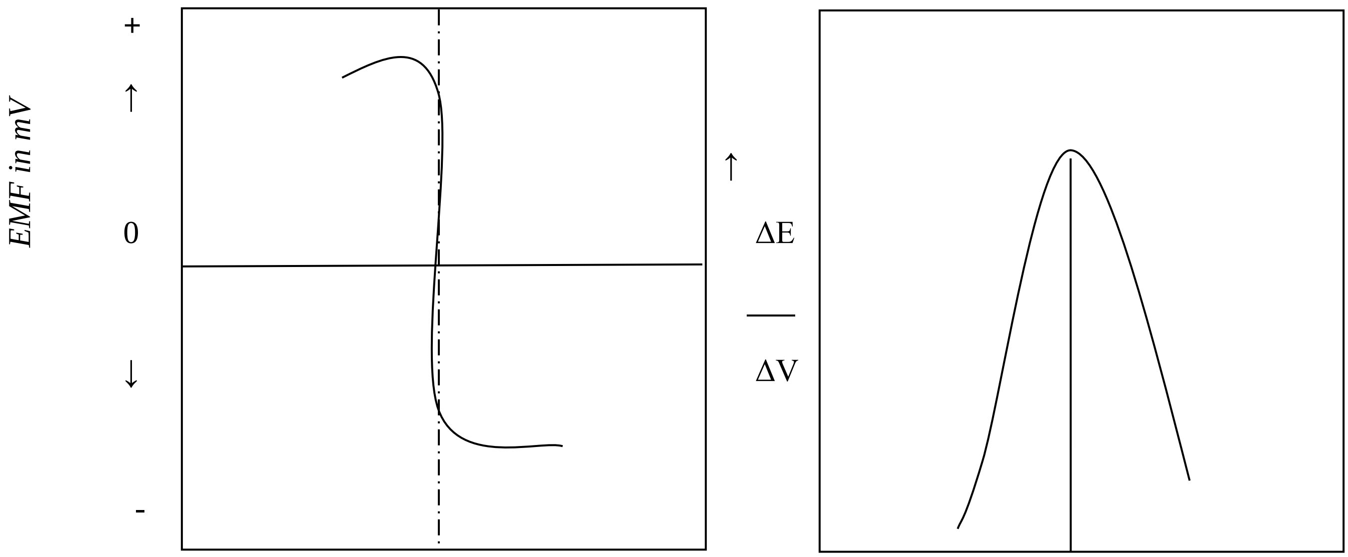
$= E_{cell} = 0.6996 - 0.0591pH - 0.242$

$= E_{cell} = 0.458 - 0.0591pH$

E_{cell} is a function of pH.

During the titration, as base is added to the acid, the H^+ ion concentration in the half cell containing Quinhydrone will decrease. Correspondingly, there will be a decrease in the E_{QE} and E_{cell} values also.(But P^H increases, as P^H=-log $[H^+]$) Ecell values are noted down, graph is plotted, end point is located and concentration is calculated.

Potentiometric Acid-Base Titrations: Graphs



V_{NaoH}added (ml)(A) V_{NaoH}added (B)

EXP. (10): Titration of a strong Acid (HCl) Vs strong base (NaOH)

Requirements: Saturated calomel electrode, platinum electrode, quinhydrone, potentiometer, beakers, burette, pipette, stirrer, salt bridge. etc.

Chemicals: the following solutions are required:

1) 0.1 N NaOH (N/10)
2) 0.05 N HCl (N/10)
3) Quinhydrone.

Procedure:

Part A: Preparation of standard Oxalic acid solution, $C_2H_2O_4.6H_2O$

Weigh about 0.63 g of oxalic acid, in a weighing bottle. Transfer it into a clean 100 ml std. flask through funnel. Dissolve it in distilled water and make a homogeneous solution upto the mark. From the wt. of Oxalic acid calculate the normality of std. solution.

Part B: Standardization of NaOH solution.

Take NaOH(~N/10) solution in burette. Pippete 20ml of standard Oxalic acid solution in a clean conical flask. Titrate it with NaOH using phenolphthalein indicator till colourless solution changes to pale pink in colour. From the titration data normality of NaOH solution is calculated.

Part C: Estimation of HCl in the given test solution

1. Make the given test HCl solution homogeneous by adding distilled water upto the mark.
2. Connect the Potentiometer to A/C mains and Switch it on.
3. Pipette 20ml of the test soln. in a clean 100 ml beaker. Add a pinch of Quinhydrone to it and dip a platinum electrode in the solution. Connect the electrode to pH terminals.

4. In another 100 ml beaker, a saturated calomel electrode is placed in a solution of KCl(Sat.). The electrode is connected to RE terminals.

5. The two solutions are connected by means of salt bridge to form a Galvanic cell.
   $^{(-)}\text{Pt} \;\Big|\; \text{Hg}_{(1)}, \text{Hg}_2\text{Cl}_2(s) \;\Big|\; \text{KCl}_{(sat)} \;\Big\|\; \text{H}^+ \text{ (unknown) } \Big|\; \text{Q}, \text{QH}_2\text{Pt} \Big|^{(+)}$

6. Switch on the Instrument; keep the mv/current RED switch in pressed position (mv mode). Display reads the EMF of cell in milli volts. Adjust the initial EMF to a present value.

7. Add NaOH from burette in 1 ml portions to the acid, stir it and note the EMF.

8. Continue the titration till a sudden inflexion in EMF occurs (the value changes to –ve), then take about 6 to 8 readings after inflexion in **1 ml intervals.**

9. From the titration, approximate volume of NaOH required is found out. (Table-1)

10. The titration is repeated with addition of NaOH in 0.1 ml lots in the vicinity of end point (In 2 ml range) (Table -2)

11. Draw a graph between E cell Vs volume of NaOH. Added. The intersection point at Ecell = Zero may give an approximate end point.

12. Another graph is obtained by plotting $\Delta E/\Delta V$ (y-axis) vsV_{NaOH} (x-axis) to get a sharp peak, which corresponds to the equivalence point.

Questions:

1. What is the relation between emf of cell and pH of solution?
2. Why cell potential decreases with addition of alkali to the test acid solution?
3. What is the use of quinhydrone in this experiment?

Expt (10) POTENTIOMETRY ACID-BASE TITRATION

Date:

Expt No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.				

Table.2.Weight of empty weighing bottle

	.				

Wt. of Oxalic Acid = W_1-W_2 =

$N_{(std)}$ Oxalic Acid = $\frac{\text{wt}}{\text{eq.wt}} \text{ x } \frac{1000}{\text{Vml}}$ =

(V_{ml} = 100ml), Eq Wt of Oxalic acid=126/2=63

Part B : Standardization of NaOH

Titration of Std. Oxalic. Vs. NaOH (link)

Sl No.	V_{oxalic} (Std) (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

$$N_{NaOH} = \frac{N_{Oxalic} \text{ x } V_{Oxalic}}{V\ NaOH} = - =$$

POTENTIOMETRY ACID-BASE TITRATION (HCl Vs. NaOH)

PART-C: Estimation of HCl in the given test solution:

Table : 1.

S.No.	Vol of NaOH added (ml)	Ecell (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

Table : 2.

S.No.	V_{NaOH} (ml)	Ecell (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
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16				
17				
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20				
21				
22				
23				
24				
25				

Calculations:

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

(^{V}NaOH = Peak from differential graph –(B)

Wt of HCl = N_{HCl} x 36.5 = ______________gpl

Results:

1. Weight of Oxalic Acid = ____________g
2. N_{std}. Oxalic Acid = _____________
3. N_{NaOH} (Link) = _____________

4.V_{NaOH} @ end point (from graph) = _____________ (ml)

5.N_{HCl} (Test) = ______________

6. Weight of HCl in test solution = _____________ gpl

Marks

Observations and calculations (20):_______________

Results & Graphs (10):______________

Discussion of results (5):________________________

Record (15):________________________

Total(50):________________________

Signature of Faculty

*A lot of what passé for depression these days is nothing more than the body saying that it needs work.

pH Metry

Glass Electrode

THEORY:-Glass electrode is a pH indicating electrode, which is H^+ ion sensitive.

Construction:

A special type of soft-glass of the soda-lime type, with a low melting point, relatively high electrical conductivity, is blown into a thin walled bulb. This bulb is fused to an ordinary glass-tube. Aqueous solutions of 0.1N HCl and saturated KCl are sealed within the bulb. A platinum wire coated with Ag-AgCl, immersed in this solution, is an internal reference electrode that provides the electrical contact.

When the glass electrode, containing 0. 1N HCl is placed in a solution of a different pH, will develop a difference in potential at the interface between the glass membrane and the solutions containing the H^+ ions. The magnitude of this difference of potential depends upon the difference in the concentrations of H^+ ions in both the solutions.

Cell Notation:-

(-) Pt | $Hg_{(1)}$, $Hg_2\ Cl_2(s)$ | Sat. KCl || Test soln of | Glass | 0.1NHCl | AgCl, Ag+ | $Pt^{(+)}$
Unknown pH membrane l

Cell Reaction :-

Anode:- $2Hg + 2Cl^- \rightarrow Hg_2\ Cl_2 + 2e^-$

Cathode:- $2AgCl + 2e^- \rightarrow 2Ag + 2Cl^-$

The EMF of cell is given as

$E_{cell} = E_{Glass} - E_{SCE}$

The potential of Glass electrode is given by

$E_{Glass} = [E^o_G - 0.0591pH]$ E^o_G = SRP of Glass electrode

$= E_{cell} = E^o_G - 0.0591pH - 0.242$

$$pH = \frac{E_G{}^o - E_{cell} - 0.242}{0.0591}$$

$(P^H = {}^-\log[H^+])$

pH Meter: The glass membrane of the glass electrode offers a high resistance in measuring circuit potential and even a highly sensitive galvanometer fails to detect currents flowing due to potential imbalance. For null point detection, and for measuring the small current, amplification of signals is necessary. pH meter is the instrument used for this purpose. It is an electronic voltmeter of requisite sensitivity and stability, and it provides a scale calibrated directly in pH units.

P^H Scale = Acidic = 1 to 7, Basic = 7 to 14

ACID –BASE pH-METRIC TITRATION

THEORY:

1) Titration of strong acid (HCl) against a strong base (NaOH):

In this titration, as the base is added to the acid, the OH^- ions of the base react with the H_3O^+ ions of the acid forming water.

$$H_3O^+ + OH^- \rightleftharpoons 2H_2O$$

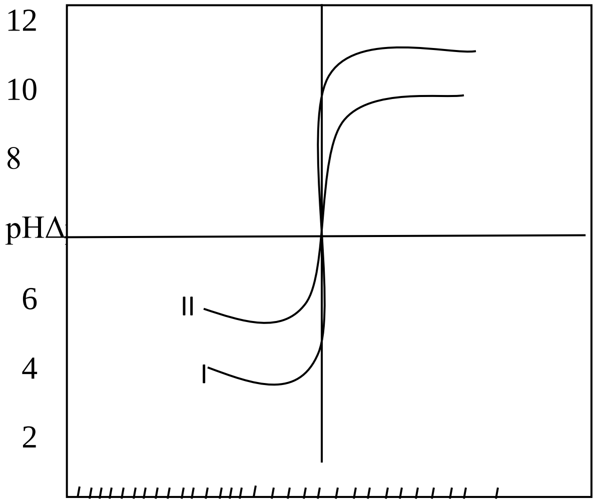
Consequently, the H_3O^+ ion concentration in the acid will decrease, i.e., the pH of the acid will progressively increase. Initially the change in the pH will be gradual, however near the neutralization point we will have a sharp increase in the pH from about 4 to 10 within 0.2 ml range of the base added, around the end point.

From the plot of the pH against the volume (V) of the base added, we will obtain the end point of the titration.

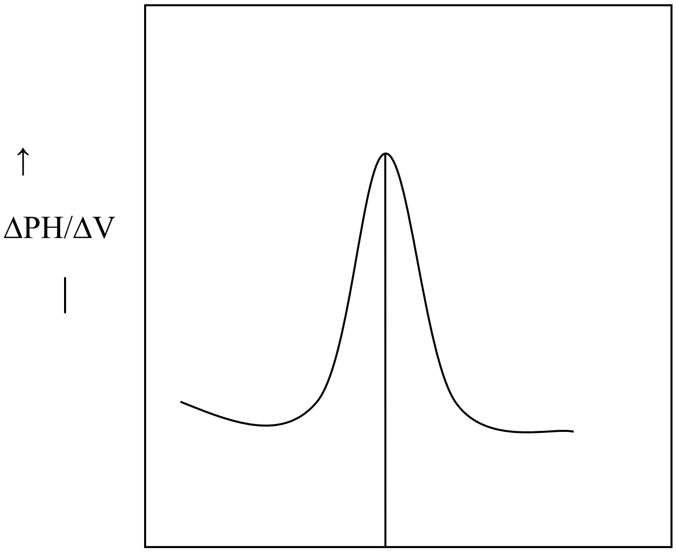
Plot of $\frac{\Delta pH}{\Delta V}$ Vs V in the vicinity of the equivalence point (V=0.1 ml) will give a curve whose peak indicates a sharp end point.

Titration of a weak acid (CH_3COOH) against a strong base (NaOH):

Here the initial concentration of H_3O^+ ions in acid solution will be low, hence the high pH. As the titration proceeds the pH of the acid will gradually increase. At the equivalence point, change in the pH will be sharp, as in titration of a strong acid and strong base, but the slope of the curve, obtained in the plot of pH vs Volume of the base added, is not as steep as in the strong acid strong base titration curve. Another point to be noted in this titration is that the ionic (CH_3COO^-) hydrolysis of the salt formed, will make the pH of the reaction mixture higher than 7 at the equivalence point.



Volume of base added→(A)



V_{NaOH} added (ml)—(B)

I - Curve for Neutralization of Strong acid and strong base. Initially P^H is low for strong acid.

II- Curve for Neutralisation of Weak acid and strong base.Initially P^H is higher for weak acid.

Experiment (11): PH Metry Titration of HCl vs NaOH.

Requirements: pHmeter, glass electrode, burette, pipette, beakers.

Procedure:

Part 'A': Preparation of standard Oxalic acid solution.

Weigh about gms of oxalic acid in a weighing bottle Transfer it into a clean 100 ml standard flask through funnel. Dissolve it in distilled water to make a homogeneous solution upto the mark. From the wt of oxalic acid calculate the normality of standard solution.

Part 'B': Standardization of NaOH solution.

Take NaOH 0.1N Solution in burette. Pipette out 20 ml of standard oxalic acid solution into a clean conical flask. Titrate it with NaOH using phenolphthalein indicator till colourless soln. changes to pink in colour. From titration data, normality of NaOH solution is calculated.

Part 'C': Estimation of HCl in the given test solution

1.Switch on the pH meter by placing the plug in the A.C. mains socket.
2.Keep the Temp at 30^oC and place both the switches in pressed position.
3.Place the combined Glass-calomel electrode setup in distilled water and check for display of pH=7.0 otherwise adjust with pH 7.0 preset control to get 7.0 pH (right side knob).
4. Take out the electrode from water, clean it with tissue paper and dip it in a 4.0 pH buffer.
5.Release the check / Read switch and adjust the SET Buffer control to get a display of pH=4.0 (mv=176). Once calibrated, this knob should not be disturbed.
6.Take out the electrode from Buffer soln., wash the electrode with distilled water, and clean it with tissue paper.
7.Make the given HCl test soln. homogeneous upto the mark by adding distilled water.
8.Pipette out 40ml of HCl in a clean 100ml beaker. Dip the electrode in the solution and measures the pH of solution by releasing the Check/Read Switch.
9.Add(~N/10) Soln. of NaOH from burette into the acid, in 1 ml intervals, stir the soln. in beaker with glass-rod and measure its pH.
10.Continue the titration till a sudden inflexion in pH occurs, then take about 6 to 8 readings after inflexion in 1 ml intervals.
11.From the titration approximate volume of NaOH required is found out. (Table-1).
12.The titration is repeated with addition of NaOH in 0.1 ml lots in the vicinity of end point (In 2ml range) (Table-2).
13.Draw a graph of pH Vs. Volume of NaOH added. The intersection point pH=7.0. may give a rough end point.
14.Another graph is obtained by plotting $\Delta pH/\Delta V$(Y- axis) Vs. VNaOH (X-axis) to get a sharp peak, which corresponds to the equivalence point. (Differential Graph)

Exp. No. (11) pH- METRIC TITRATION (STRONG ACID VsSTRONG BASE)

Date:

Exp. No:

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.				

Table.2.Weight of empty weighing bottle

	.				

Wt. of FAS = W_1-W_2 = __________ =__________

$N_{(std)}$ Oxalic acid = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ = ______________=______________

(V_{ml}=100ml).

Part B :Standardisation of NaOH

Titration of Std. Oxalic Vs. NaOH (link)

Sl No.	V_{Oxalic} (Std) (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

$N_{NaOH} = \frac{N_{Oxalic} \text{ x } V_{Oxalic}}{VNaOH}$ = ____________=_________________

PART-C: Estimation of HCl in the given test solution:

Table : 1.

S.No.	Vol of NaOH added (ml)	pH	Ecell (mv)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			

Table : 2.

S.No.	VNaOH (ml)	pH	ΔpH	$\frac{\Delta pH}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$ = ________________ = ________________________

(V_{NaOH} = Peak from differential graph –(B)

Wt of HCl = N_{HCl} x 36.5 = _______________ = _____________gpl

Application of pH measurements

- Food processing, dairies, breweries, distilleries.
- Purification of drinking water, pollution control, sewage treatment.
- Aquaculture and agricultural soil testing. Pharmaceuticals, cosmetics.
- Electroplatings, cement, fuels etc.

Questions:

1. What do you mean by combined electrode?
2. Which electrode acts as anode and which acts as cathode?
3. Why should we calibrate the instrument using a buffer solution?
4. At neutralization point what is the emf of the cell?
5. Why is salt bridge not used in this experimental set up?

Results:

1. Weight of Oxalic Acid = ____________g

2. N_{std}. Oxalic Acid = _____________

3. N_{NaOH} (Link) = _____________

4.V_{NaOH} @ end point (from graph) = _____________ (ml)

5.N_{HCl} (Test) = ______________

6. Weight of $_{HCl}$ in test solution = _____________ gpl

Marks

Observations and calculations (20):_______________

Results & Graphs (10):______________

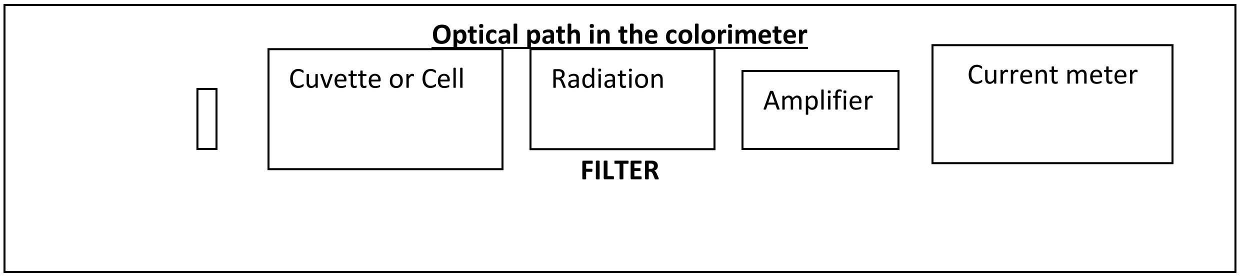
Discussion of results (5):_______________________

Record (15):_______________________

Total(50):_______________________

COLORIMETRY

1. **Introduction:** In colorimetry, the light absorptive capacity of a system (coloured solution) is measured and this measurement is related to the concentration of the coloured substance in the solution. The absorbance is governed by "Beer-Lambers" Law.



Optical path in the colorimeter

2. **Colorimeter:**
A colorimeter (absorptiometer) consists of the following parts:
1) light source 2) light filter 3) cell for coloured solution 4) photocell 5) amplifier
3. **Colour Filters:**
The filter is a coloured glass plate or coloured gelatin coated on glass plate.
Property of a filter is that when while light is passed through the filter, it transmits light from a specified region of spectrum, preferentially absorbing the other parts of the spectrum.
4. **BEER'S LAW:** It states that when monochromatic light passes through a transparent medium the rate of decrease in intensity with the concentration of the medium is directly proportional to the intensity of the light. This is equivalent to stating that the intensity of the transmitted light decreases exponentially as the concentration of the absorbing medium increases arithmetically.
It can be expressed as
$\frac{-\mathrm{dI}}{\mathrm{dc}}$ = KI, on Integration between limits Io—It& C=0—c, gives
$\frac{\mathrm{Io}}{\mathrm{It}}$ = Kc (or) $I_t = I_o.10^{-kc}$ (1)

Where $I_0{}^o$ = Intensity of the incident light,
I_t= Intensity of transmitted light,
c= Concentration of the medium and
K= proportionality factor.
k= constant for the wave length & the absorbing medium
$K=\frac{k}{2.303}$
5. **LAMBERT'S LAW:-**It states that when monochromatic light passes through a transparent medium the rate of decrease in intensity with the thickness of the medium is directly proportional to the intensity of the light (or) The intensity of the transmitted light decreases exponentially as the thickness of the absorbing medium increases arithmetically.

It can be expressed as $\frac{-\mathrm{dI}}{\mathrm{dc}}$ = KI

On integration between limits Io----Itand dx-0-x, we get(Where ‘x’ is thickness of the medium.)

$$I\,n\,\frac{Io}{It} = Kx \qquad \text{(OR)} \qquad I_t = I_o.\,10^{-kx} \qquad - \qquad (2)$$

6. **BEER-LAMBERT’S LAW:-** It is a combination of the above two laws relating intensity of light with concentration as well as thickness of absorbing medium. It states that

“ when monochromatic light passes through a transparent medium the rate of decrease in intensity with the**concentration and thickness of the medium** is directly proportional to the intensity of the light.”

Combination of equations 1 & 2 is Beer-Lamberts’ law, which can be expressed as

$$I_t = I_o.10^{-€cx} \qquad \text{(OR)} \quad \log\frac{Io}{It} = €cx \qquad - \qquad (3)$$

Where € is molar absorption coefficient or molar absorptivity, or molar extinction coefficient ‘c’ is expressed in moles / litre, and x in centimeters.

Equation (3) is the fundamental equation of colorimetry& spectrophotometry, and is often termed as the Beer-Lambert’s Law.

The relation between absorbance A, the transmittance T and the molar absorption coefficient (€) is given

$A = €cx = \log \frac{Io}{It}$

We know that $A = \log \frac{I}{T} = -\log T$

$$\therefore \log T \quad = \quad \log\frac{I_t}{I_o} \qquad - \qquad (4)$$

Transmittance, T is given as $T = \frac{It}{Io}$

$\%T = 100 \times I_t/I_o$

7. CONDITIONS FOR BEER-LAMBERT’S LAW TO BE APPLICABLE:.

1) Solution should be coloured.

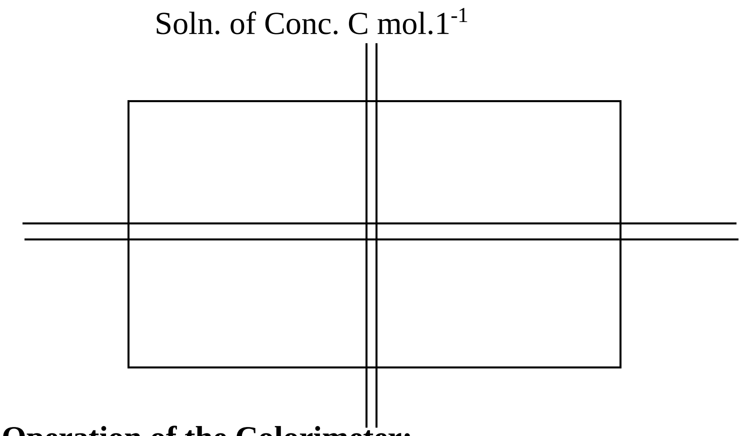
2) Incident radiation should be monochromatic.

3) Solution should be homogeneous

4) Solution should be dilute.

5) Each molecular or ion species should absorb independently.

Principle of absorbance of radiation



8. Principle of Operation of the Colorimeter:

A low voltage lamp forms the light source. This light passes through a select filter. The light transmitted by the filter passes through the cell containing the coloured solution, and falls on a sensitive photocell, An amplifier amplifies the current generated by the photocell. The amplifier output drives a current meter calibrated in optical density and % transmittance.

Complementary Colours for Colorimetry

Wavelength of light Transmitted by the filter (nm)	Colour (Absorbed)	Complementary Colour (Transmitted)
400-435	Violet	Yellowish green
435-480	Blue	Yellow
480-490	Greenish Blue	Orange
490-500	Bluish Green	Red
500-560	Green	Purple
560-580	Yellowish green	Violet
580-595	Yellow	Blue
595-610	Orange	Greenish Blue
610-750	Red	Bluish Green

9.Operation of the Colorimeter:

(Note: Before operation of the colorimeter, make sure that all switches are in 'off' position.

i) Connect the colorimeter to the A/C Mains and Switch it on.
ii) 'On' the switch located at the back panel of the colorimeter. (The 'read out' will show 1.00)
iii) By rotating the disc 'B' (see diagram on Front Panel of the colorimeter), any colour filter that gives the desired wavelength range of transmitted light, can be brought in the path of the light. Select a colour filter which gives maximum optical density for a solution of a given solution under measurement.

(For the expt. With $KMnO_4$, the wavelength of transmitted light used is 490 nm or 4900 A^o (colour filter 52).

Place the solution cell with blank (distilled water is the blank for this expt.) in the housing'C'.

(Note: The solution cell should be filled to a height not exceeding ¾ and outside dried with a tissue paper)

Adjust the disc A for zero O.D, for distilled water (blank).

iv) Replace Blank in the solution cell with various sample solutions prepared(test tube-1 to 10), one after the other (as indicated in the table), after rinsing with the respective solution.
v) Record the O.D., for each of the sample solutions.
(The O.D. value should be between 0.3. and 0.7 or else the sample solutions required for any species should be brought in this range by suitable dilution.)

The graph between the optical density, taken on the y-axis and concentration on the x-axis will give a straight line passing through the origin, with a slope of Ix. (select a suitable scale on the graph paper).

EXPT (12) COLORIMETRY: VERIFICATION OF BEER-LAMBERT'S LAW & ESTIMATION OF Mn in $KMnO_4$

PROCEDURE:

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

Weigh accurately about 0.98gm of Mohr's salt and transfer it into a clean 100 mil flask using funnel. Dissolve it in distilled water, add-10 ml dil H_2SO_4 and make the solution homogeneous upto the mark.

PART B: STANDARDIZATION OF $KMnO_4$ USING MOHR'S SALT:

Fill the burette with $KMnO_4$ soln. Pipetteout 20 ml of Mohr salt soln. in a clean conical flask. Add ¾ test tube of dil H_2SO_4 and titrate the contents of flask with $KMnO_4$ till a light pink colour appears. From titration data, calculate the Normality of $KMnO_4$ (Link) solution.

Convert the concentration from Normality to Molarity.(this gives Molarity of (Link solution)

Let $M_{KMnO4} = N_{KMnO4}/5$

M_{KMnO4} = 0.00048 M = 4.8×10^{-4} M. $\frac{0.024}{5}$

PART C: ESTIMATION OF $KMnO_4$BY COLORIMETRY:

1)**Preparation of $KMnO_4$ stock solution:**10ml of link KMnO4 is taken in standard volumetric flask and diluted to 100 ml., made upto the mark and homogenous to get stock solution .

2) **Preparation of sample solutions**: The above stock solution from volumetric flask is pipettd out in test tubes from 1 to 10 respectively and diluted by adding distilled water, to make 10 ml solution in each test tube. Dilution is done as given in Table 2.of the experiment.

3) **Selection of Filter No for KMnO4 solution:** The colorimeter is calibrated by taking blank solution. For each filter No. 45 to 67 the OD of stock solution is measured in Table 1.The filter No. which gives maximum OD is selected.(forKMnO4 the filter No.52 gives maximum OD.

4)VERIFICATION OF BEER-LAMBERTS LAW:

The selected filter no for $KMnO_4$ solution is fixed, The caliberation is done by taking(distilled water) blank and adjusting OD to zero, The cuvvete is filled with each of the ten sample solutions prepared, one after the other. After rinsing with respective solution. The OD is recorded for each of the sample solution. OD Values are noted in Table-2. OD of test solution is also noted.

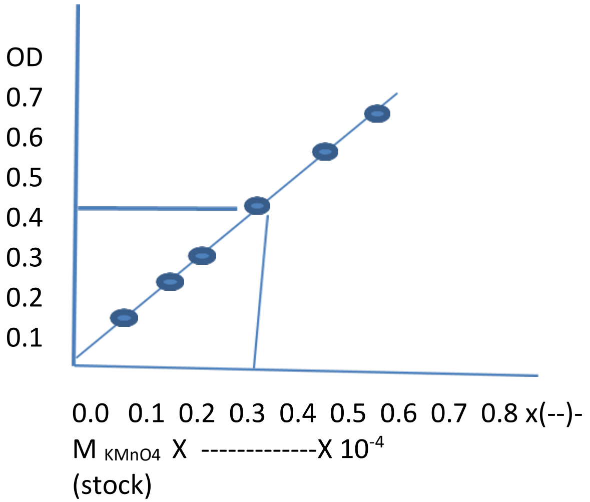
5)Calibration Graph:A graph is plotted between optical density against concentration of $KMnO_4$. it gives a straight line passing through origin. It is called calibration graph. The graph passing through origin is a proof for verification of Beer-Lambert's Law.

This graph can be used for estimation of the concentration of a given species ($KMnO_4$ in this case) in any Test solution.

6)**Concentration of test solution**:OD of test solution is marked on y-axis of the gragh. The concentration of test solution is obtained by intrapolation on x-axis.

Molarity of test solution is calculated and test solution is estimated.

COLORIMETRY:- Calibration Curve of Concentration of $KMnO_4$ versus O.D.[Verification of Beer-Lambert's Law]

	<u>Test Solution</u> O.D = ____________ Conc. Of $KMnO_4$ = __________ x10^{-4}M

Note the O.D. of test solution and mark it on the graph. The concentration of the test solution would be arrived at by multiplying the concentration as read on the calibration graph with the dilution factor.

Applications of Colorimetry:-

1. Estimation of steel analysis, fluoride, nitrate analysis, biochemical/laboratories, pharmaceutical industry, textile industry etc.

Questions

1. For the following transmittance what is the absorbance or optical density.

   0% ; 1% ; 10% ; 100%

2. What type of solutions are analyzed in Colorimetry?
3. How can Colorimetry be used to find out concentration of species in solutions which are not colored?
4. Why only dilute solutions are used in Colorimetry?
5. What is the role of filters in Colorimetry and how do you select a proper filter.

Exp. No. (12) COLORIMETRY: VERIFICATION OF BEER-LAMBERT'S LAW

Date:

Exp. 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 =

$N_{(std)}$ FAS = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ =

(V_{ml} = 100ml)

Part B : Standardization of $KMnO_4$ solution.

Titration of Std. FAS. Vs. $KMnO_4$ (link)

Sl No.	V_{Fe}^{2+} (ml)	Burette Reading		V_{KMnO4} ml
		Initial	Final	

Marks

Observations and calculations (20):_______________

Results & Graphs (10):______________

Discussion of results (5):________________________

Record (15):________________________

Total(50):________________________

$$N_{KMnO4} = \frac{N_{FAS} \times V_{FAS}}{V\ KMnO4}$$

Table : 1. :Selection of filters

S.No.	Filter No	OD.
1	45	
2	47	
3	51	
4	52	
5	54	
6	57	
7	60	
8	67	

Table – 2 : Concentration $KMnO_4$ Vs Optical Density

S.No.	Std. Soln V(KMnO4 +distilled water) (in ml)	Concentration $KMnO_4$(Molar)]	Optical Density
1	1+9	0.1 x---x10^{-4}	
2	2+8	0.2 x---x10^{-4}	
3	3+7	0.3 x---x10^{-4}	
4	4+6	0.4 x---x10^{-4}	
5	5+5	0.5 x---x10^{-4}	
6	6+4	0.6 x---x10^{-4}	
7	7+3	0.7 x---x10^{-4}	
8	8+2	0.8 x---x10^{-4}	
9	9+1	0.9 x---x10^{-4}	
10	10+0	1.0 x---x10^{-4}	
11	Test Solutions		

CALCULATIONS:

$N_{KMnO4\ (stock)} = N_{(Link)} \times \frac{10}{100} =$

$M_{KMnO4\ (stock)} = \frac{N(Stock)}{5} =$

O.D. of test soln =

M solution (Test) =

(From graph)

Wt. of $KMnO_4$ = -----------------g/l

$M_{(test)} \times 158 =$

Results:

1. Weight of FAS = ___________g
2. N_{std}. FAS = ____________
3. N_{KMnO4} (Link) = ____________
4. M_{KMnO4} == ____________ (stock solution)
5. Beer-Lambert’s Law verified
6. Test Solution : OD=____________: Conc of $KMnO_4$= ____________M
7. Wt of KMnO4 in test solution = _____________gpl.

DEMO EXPERIMENT OF Conductometric Titration

Part – C: Estimation of HCl in the test solution:

S.No.	V_{NaOH} (ml)	Conductivity ($mscm^{-1}$)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		

$$N_{HCl} = \frac{N_{NaOH} \text{ x } V_{NaOH}}{V_{HCl}}$$

V_{NaOH} = Inflection point from the graph (V_1) =

Wt_{HCl} = N_{HCl} x 36.5

Results:

1. Weight of Oxalic Acid = ____________g
2. $N_{std Oxalic Acid}$ = _____________
3. N_{NaOH} (Link) = _____________
4. V_{NaOH} end point (from graph) = _____________ (ml)
5. N_{HCl} (Test) = _____________
6. Weight of HCl in test solution = _____________ gpl

DEMO EXP- OF POTENTIOMETRIC TITRATION

POTENTIOMETRY REDOX TITRATION (Fe^{+2}Vs. KMnO4)

PART-C: Estimation of Fe+2 in the given test solution

Table : 1.

S.No.	Vol of KMnO4 added (ml)	Ecell (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

Table : 2.

S.No.	V $KMnO4_7$ (ml)	Ecell (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{Fe^{2+}} = \frac{N\ KMnO4_{X}\ V_{KMnO4}}{V_{Fe^{2+}}}$$

($^{V}{}_{K2Cr2O7}$ = Peak from differential graph –B)

Wt of $_{Fe^{2+}}$ = $N_{Fe^{2+}}$ x56 = _______________gpl

DEMO EXPERIMENT OF pH – METRIC TITRATION

Date :

PART-C: Estimation of HCl in the given test solution

Table : 1.

S.No.	Vol of NaOH added (ml)	pH	Ecell (mv)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			

Table : 2.

S.No.	V NaOH (ml)	pH	ΔpH	$\frac{\Delta pH}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

(V_{NaOH} = Peak from differential graph –B)

Wt of HCl = N_{HCl} x 36.5 = ______________gpl

DEMO EXPERIMENT COLORIMETRY: VERIFICATION OF BEER-LAMBERT'S LAW

Table:1.Selection of filtersTable: 2.

S.No.	Filter No	OD.
1	45	
2	47	
3	51	
4	52	
5	54	
6	57	
7	60	
8	67	

S.No.	Std. Soln V(KMnO4 +distilled water) (in ml)	Concentration (Molar)]	Optical Density
1	1+9	0.1 x---x10^{-4}	
2	2+8	0.2 x---x10^{-4}	
3	3+7	0.3 x---x10^{-4}	
4	4+6	0.4 x---x10^{-4}	
5	5+5	0.5 x---x10^{-4}	
6	6+4	0.6 x---x10^{-4}	
7	7+3	0.7 x---x10^{-4}	
8	8+2	0.8 x---x10^{-4}	
9	9+1	0.9 x---x10^{-4}	
10	10+0	1.0 x---x10^{-4}	
11	Test Solutions		

CALCULATIONS:

$\mathbf{N_{KMnO4\ (stock)}} = N_{(Link)} \times \frac{10}{100} =$

$\mathbf{M_{KMnO4\ (stock)}} = \frac{N(Stock)}{5} =$

O.D. of test soln = M solution (Test) =

(From graph)

Wt. of $KMnO_4$ = $M_{(test)}$ x 158 = =…………………..gpl

Results:

1. Weight of FAS = ___________g
2. N_{std}. FAS = ____________
3. N_{KMnO4} (Link) = ____________
4. M_{KMnO4} == ____________ (stock solution)
5. Beer-Lambert's Law verified
6. Test Solution : OD=____________: Conc of $KMnO_4$= ____________M
7. Wt of KMnO4 in test solution = _____________gpl.

CHEMICAL KINETICS

EXPERIMENT No. 13: Determination of Rate Constant of acid catalyzed Hydrolysis of Methyl acetate - (First Order reaction)

AIM: To determine the rate constant for the acid-catalyzed hydrolysis of methyl acetate**.**

THEORY

Methyl acetate undergoes hydrolysis, in the presence of an acid (HCl, for example), to give acetic acid and methyl alcohol.$\mathbf{H^+}$

$$CH_3COOCH_3+H_2O\rightarrow CH_3COOH+CH_3OH$$

In the presence of an acid, this reaction should be of second order, since two molecules are reacting.But, it is found to be first order. This may be explained in the following way:The rate of the reaction is given by

$$\mathbf{dx / dt = k' [CH3COOCH3]\ [\ H2O\ ]}$$

where k' is the rate constant (or specific rate constant).

Since water is present in large excess, its active mass (molar concentration) virtually remains constant during the course of the reaction. Therefore, its active mass gets included in the constant, and the above equation reduces to :

$$\mathbf{dx / dt = k1[CH3COOCH3]}$$

Thus, the rate of the reaction is determined by one concentration term only (that is, by a single power of the concentration term only).

Hence, the reaction is first order. Such reactions are also referred to as pseudo first order reactions.The progress of the reaction (hydrolysis of ester) is followed by removing a definite volume of the reaction mixture, at definite intervals of time, cooling it in ice, and titrating the acetic acid formed against alkali, which has already been standardized.

The amount of alkali used is equivalent to the total amount of hydrochloric acid present originally and the amount of acetic acid formed in the reaction.

The amount of acetic acid formed (x), at definite intervals of time (t), can be obtained. The amount of acetic acid formed, at the end of the reaction, is equivalent to the initial concentration (a) of the ester.Suppose the volumes of the sodium hydroxide solution (titre value) required for neutralization of 5 mlof the reaction mixture are:

(i) At the commencement of the reaction is V_0

(ii) After time (t) is V_t

(iii) At the end of the reaction is V_∞

Then:

x (amount of acetic acid formed after time) is proportional to (Vt-Vo).

a (initial concentration of ester) is proportional to (V∞-Vo).

[a – x] (Concentration of ester present after time t) is proportional to

$$\mathbf{(V\infty\text{-}Vo) - (Vt\text{-}Vo) = (\ V\infty\text{-}Vt)}$$

The first order rate expression given by:

$$\boxed{Ln\ (a / a\text{-}x\ ) = kt \qquad or \qquad Ln\ (\ V_\infty / V_\infty - V_t) = kt}$$

Hence, the rate constant (k1) could be calculated.

Procedure:

1- Take (5)ml from stock solution of methyl acetate, put it in the beaker then add (100)ml of (0.1N) HCl but not opposite (be sure not losing any volume of methyl acetate on the wall of glass) and start recording the time at the beginning of the reaction.

2- After (5)min of the reaction beginning withdraw (10)ml from the mixture to the conical flask, add (20)ml of distilled water (D.W.) (slows down the reaction)and add (2) drops of phenolphthalein indicator.

3- Titrate with (0.1N) NaOH and record the volume of NaOH that descending from the burette immediately after appearance of pink color.

4- Repeat steps (2and 3) every (5)min to calculate (V_t) according to these times (5, 10, 15, 20, 25)min.

5- To calculate (V_∞) these are two ways:

a- (Condensation): Take (10)ml from the reaction mixture from the beginning reaction in to dry conical flask, connected to condenser, start the reaction for 1hr, then rise the condenser from up end of the conical flask, add (2) drops of phenolphthalein indicator and titrate against (0.1N) NaOH.

b- Leave the reaction mixture for 24hr, then add (2) drops of phenalphalein indicator and titrate against (0.1N) NaOH.

Calculations and graph:

V_o : volume of NaOH descending from the burette at (t = 0)

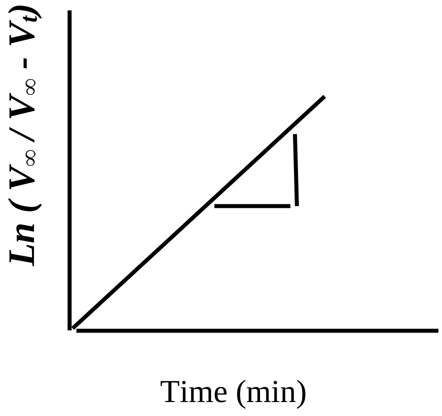
V_t : volume of NaOH descending from the burette at(t = t)

V_∞ : volume of NaOH descending from the burette at the end of reaction

1- Arrange the results according to the following table:

Time (min)	V_t (ml)	V_∞ (ml)	($V_\infty - V_t$) (ml)	$V_\infty / V_\infty - V_t$) (	Ln ($V_\infty / V_\infty - V_t$)
5					
10					
15					
20					
25					

2- Plot a graph of Ln ($V_\infty / V_\infty - V_t$) against (time in min) and from the slope determine the rate constant (k), then determine the half-life time($t_{1/2}$).



SLOPE = K

Results-

i) The plot gives a straight line passing through origin. Hence hydrolysis of ester in acidic medium is found to follow First Order of Kinetics.
ii) The rate constant K reaction is --------------min-1, from gragh.
iii) The rate constant K reaction is --------------min-1, theoretical value.

Questions –.

1)Define order and molecularity of reaction, Give one example.

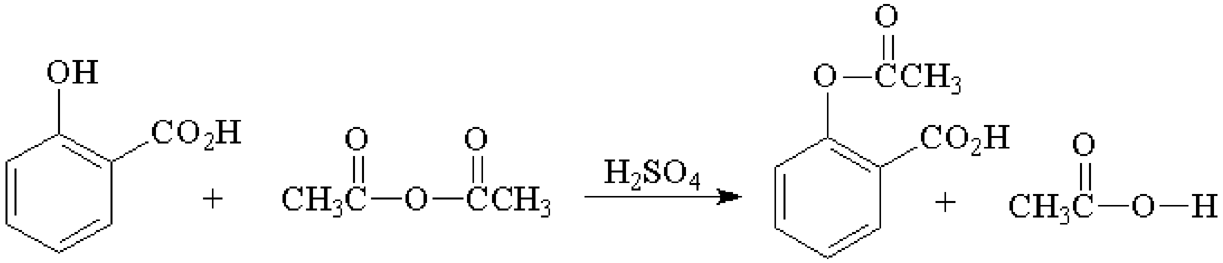
2) What is the difference between Order and Molecularity of reaction.
3) Can we study the kinetics of redox reaction like $KMnO_4$ Vs Mohrs salt.

DRUG SYNTHESIS

EXPERIMENT NO. (14): Preparation of Aspirin

Aim: To Study the Acetylation of Salicylic Acid (Aspirin)

Principle: Salicylic acid undergoes acetylation with acetic anhydride in presence of acid catalyst which generates acetyl carbonium or acetyl carbocation. The acetylation takes place selectively at weakly acidic group i.e. phenolic group in preference to strong carboxylic group to form Aspirin.



Aspirin (molar mass of 180.2 g/mol) is prepared by reacting salicylic acid (molar mass of 138.1 g/mol) with acetic anhydride (molar mass of 102.1 g/mol). Aspirin is a weak monoprotic acid.

Materials Needed

Salicylic acid, Acetic anhydride, Sulfuric acid, concentrated, Pipettes, Erlenmeyer flask, 250-mL Beakers, 2 600-mL, 25 or 80-mL, Graduated cylinders, 10-mL, Stirring rod, Ring stand.
Clamp, Ice, Ice water, 1% Ferric Chloride, 2 test tubes

Procedure

1. Prepare a hot water bath in a 600ml beaker using 300 ml of water.
2. Weigh out 4.0 g of salicylic acid. Place it in a 250-mL Erlenmeyer flask.
3. In a fume hood, add 8 mL of acetic anhydride. Swirl the flask to wet the salicylic acid crystals.
4. In a fume hood, add 4 drops of concentrated sulfuric acid, H_2SO_4, to the mixture.
5. At your lab bench, gently heat the flask in a boiling water bath for about 10-15 minutes.
6. Remove the flask from the hot water bath and add 20 mL of deionized ice water to decompose any excess acetic anhydride.

7. Make an ice bath by placing some ice and 200ml of cold water in a 600ml beaker. Chill the solution in an ice bath until crystals of aspirin no longer form, stirring occasionally to decompose residual acetic anhydride. If an "oil" appears instead of a solid, reheat the flask in the hot water bath until the oil disappears and again cool.

Determine the mass of a piece of filter paper to the nearest 0.1 g and setup a filtration apparatus. Carefully filter the contents of the flask and wash out the flask using l-2ml of ice water at a time. Do not exceed 20 ml of water. Be sure to gently mix around your crystal to help remove excess water.

9. At the end of the period gently remove your filter paper and product and allow it to dry on a clean watch glass over night.
10. Determine the mass of the dry filter paper and crystals.

FERRIC CHLORIDE TEST FOR SALICYLIC ACID

Take 5ml of distilled water and add 5mL of ferric chloride.

Dissolve a small amount the product you made in 2 ml of distilled water. Add 5 mL of aqueous 1% ferric chloride solution to the test tube and note the colour.

Now perform this same test with salicylic acid.

Formation of an iron-phenol complex with Fe(III) gives a definite color ranging from red to violet, depending upon the particular phenol present. A strong purple colour suggests the strong presence of the salicylic acid.

RESULT: White solid of aspirin is prepared.

INSTRUMENTATION CHEMICAL ANALYSIS

VIVA QUESTIONS

CONDUCTOMETRY

1. What is cell constant . What are its units ?

2. Mention the factors affecting conductivity of a solution ?

3. Name the type of cell and the electrodes used in conductivity cell ?

4. Why measured conductivity of Hcl is more than measured conductivity of CH_3COOH .

5. Conductivity of HCl test solution decreases when NaOH is added to it . Explain ?

6. Explain why conductivity increases after the end point in acid base conductometric titration ?

7. Represent the graph for Conductometric Titration of

(a) Strong Acid vs Strong Base (b) weak Acid vs Strong Base

(c) mixture of acid vs Strong Base

8. What is the change in measured conductivity when a solution is diluted ?

9. A water sample with high total dissolved solids (TDS) will have high or low conductivity ? Give reason.

POTENTIOMETRIC REDOX TITRATIONS

1. Name the electrodes in this experiment..

2. Name the reference Electrode and write its electrode reaction

3. what is the role of salt bridge in the cell .

4. Represent the cell and indicate anode and cathode .

5. What is the use of plotting differential graph.

6) Why Ecell increases during titration?

7. Calculate the emf of cell having $E^o_{SCE} = 0.242$ v and $E^o_{Fe^{+3} / Fe^{+2}} = 0.771$v

8. Write Nernst equation for Potentiometric Redox Titration .

POTENTIOMETRICACID – BASE TITRATIONS

1 .Represent the electrochemical cell for calomel and Quinhydrone electrodes.

2. Name the anode and cathode in this experiment .

3. Write the electrode reactions at anode and cathode

4. What is Quinhydrone electrode , what is its composition ?

5. Why E_{cell} decreases in this titration ?

6. Represent and Explain the graph E_{cell} vs NaOH.

7. What is the relation between E_{cell} and pH at neutralization point

8. Write any two advantages of Instrumental chemical analysis over volumetric analysis ?

pH – METRY

1. Represent the cell for pH metric acid base titration

2. What is pH, write the relation between P^H , write the Relation between P^H and { H^+ }

3. What is pH Scale ?Explain .

4. What are the anode and cathode reactions in this experiment.

5. What is glass Electrode ?

6. Represent the graph pH vs V_{NaoH} and Explain

7. Why pH increases during titrations , and what is the change in E_{cell} . Explain ?

8. What is the appropriate value of pH and E_{cell} at neutralization point

9. What is a combined electrode.

10. Write any two applications of pH measurements

11. What is the indicator electrode in this experiment . It is reversible to which ion ?

COLORIMETRY

1. What is the range of visible radiation in (A^o or nm)

2. Write the expression for Beer Lamberts Law ?

3. Write any three condition for Beer – Lamberts law .

4. Why optical Density of Solution from Test tube no : 1 to Test tube no : 10 increases?

5. What is the role colored filters in colorimeter .

6. What is the relation between absorbance (A) and Transmittance (T) .

7. At the end of experiment , how can you say that Beer Lamberts Law is verified ?

8. Which of the following solutions can be estimated by colorimeter

(a) HCl (b) $CuSo_4$ (c) NaoH (d) $K_2Cr_20_7$

9. A Solution shows 0 % absorbance , then its transmittance = _______

10. Write the Statement of Beer Lamberts law .