MJCET

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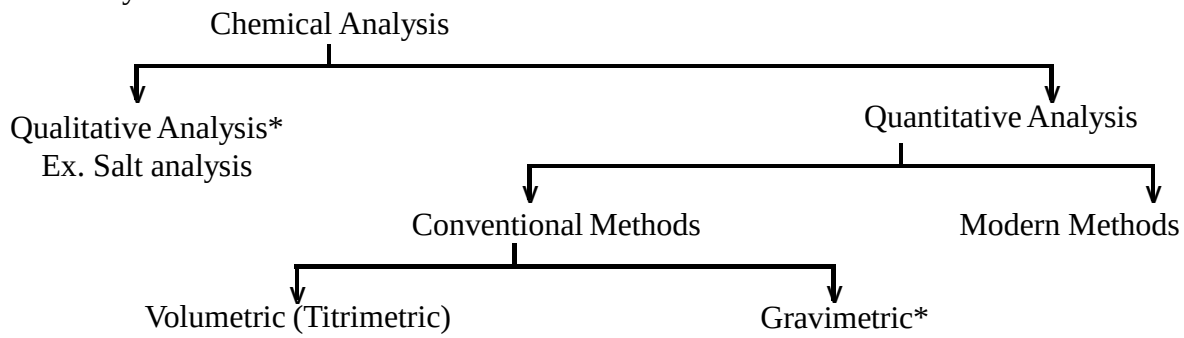
(VOL. 1 A MANUAL ON VOLUMETRIC ANALYSIS)

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



(*Not in Syllabus)

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

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

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

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

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 : $K_2Cr_2O_7$ (Potassium dichromate), Na_2CO_3 (Sodium Carbonate), $Na_2B_4O_7$ (Sodium Borate),

$H_2C_2O_4$ (Oxalic Acid), $Fe\,SO_4(NH_4)_2\,SO_4 . 6H_2O$ (Ferrous Ammonium sulphate).

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

Examples: $KMnO_4$ (Potassium Permanganate), $Na_2S_2O_3.5H_2O$(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:
Oxalic acid Vs. $KMnO_4$; Fe^{2+} Vs. $K_2Cr_2O_7$
Also Iodometry : $K_2Cr_2O_7$ Vs. Hypo ; Cu^{2+} Vs. Hypo.

iii) Precipitation titration (NaCl Vs. Ag^+; Ag^+ Vs. CNS^-)

iv) Complexometric titration (EDTA) method of estimation of hardness of water (total, permanent and temporary)

TITRATIONS :

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

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

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

Where N=Normality of the solution

V_{ml} = Volume of standard flask in ml

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

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

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

$$\mathbf{N_{KMnO_4} \times V_{KMnO_4} = N_{Oxalic\ acid} \times V_{Oxalic\ acid}}$$

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

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

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

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

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

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

Half reaction : Oxidation :

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

Half reaction : Reduction :

(Electronation) or gain of 'e' : 2x $[MnO^-_4 + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O]$ $E^0_R = 1.51$ V

Overall Reaction : $2MnO_4^- + 16H^+ + 5C_2O_4^{2-} + 10e^- \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2 + 10e^-$.
(Redox)

In the above reaction it may be noted that the oxidant $KMnO_4$ gets reduced itself because of its High Reduction Potential ($E^0_R = 1.51$V)at the same time the reductant, $H_2C_2O_4.2H_2O$ gets oxidized itself because of Less Reduction Potential ($E^0_R = 0.49$V). The equivalence established is as follows:

$2\ Mno_4^- = 10e^- = 5C_2O^2_4$ (or); $\frac{2KMnO_4}{10} = \frac{10e^-}{10} = \frac{5(H_2C_2O_4.2H_2O)}{10}$

$1 / 5KMnO_4 = 1e^- = {}^1/_2\ (H_2C_2O_4.2H_2O)$

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

31.6 g $= 1e^- = 63$ g.

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 standardisation of $KMnO_4$" (Redox Titration) the eq.wt. of Oxalic acid is 126/2=63.0 g and that of $KMnO_4$ is 158/5=31.6 g.

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

LINK TITRATION (OR) DOUBLE TITRATION

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

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

	Standard	**link**	**Test**
SYSTEM - I	$H_2C_2O_4.2H_2O$ (Reductant)	$KMnO_4$ (Oxidant)	$H_2C_2O_4.2H_2O$ (or) Fe^{2+} Amm. Sulphate (Reductant)
SYSTEM - II	$K_2Cr_2O_7$ (Oxidant)	Fe^{2+} Amm. Sulphate (Reductant)	$K_2Cr_2O_7$ (Oxidant)

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

i) Prepare a **Standard Solution** of Oxalic acid.

ii) Standardize $KMnO_4$ **Link solution** and then

iii) Determine the normality of the given Ferrous Ammonium Sulphate solution (FAS) and also estimate the amount of Fe^{2+} present in the given **test solution.**

WEIGHING

(Instructions on the use of Analytical balance and Tabulation of Data).

(Reference can be made to a sketch of the analytical balance). The analytical (Chemical) balance is rather a delicate and highly sensitive instrument and gives values to a high degree of accuracy. It is important to observe all the precautions outlined here before using.

1) Before starting to use the chemical balance, make sure it is in **rest position**. Otherwise bring it in rest position by turning the knob meant for this purpose at the bottom.

2) Do not tamper with the large levelling screws below the balance cabinet. (Generally they should have already been properly adjusted with the help of the pendulum).

3) Do not place or remove any masses from the balance pans when the balance is in 'Release' position, (i.e., when it is not 'Arrest' position). Handling of weights (gm, mg, rider) should strictly be done using a forceps. (Weights should be either in the box or on the pans).

4) In order to start using the balance, make sure, there is nothing in the balance pans (also that there is no rider on the balance beam arms). Then slowly operate the 'Knob' to 'release' (or Swing) position so that it starts swinging; watch the swings on either side of indicator. Should these be unequal, adjust for equal or 'zero' swing by the screws meant for this purpose on the extreme of the balance beam arms.

   (Turning a screw on any side **away** from mid-point of the balance (Anti-clockwise movement of the screw) would make that side heavier and conversely, taking it closer **towards** the mid-point (clockwise movement of the screw) would make it lighter.

5). Changing of weight should be done in a step-wise manner.

6). Fractional weights i.e. milligram weights from a good quality fractional weight box purchased by the student should be correctly read on the embossed side.

   Milligram weights, below 10mg (i.e. 1 to 9mg.) from the box need not be used. Rider should be used instead, for weights in this range, since it gives both unit and fractional mg. Rider should be placed on the rider carrier hook, using forceps and then placement of the rider on the balance beam on either side, should be done as required by the rider carrier.

Wt. of Rider in position 10 = Wt. of rider when placed in the pan

= 10 mg. = 0.01 g.

Then, based on the moment of force, rider at any other position indicates the weight as given by the main divisions (m.d.) and sub-divisions (s.d).

10 m.d. = 10 mg = 0.01 g.

Therefore 1 m.d. = 1 mg. = 0.001 g.

and since 1 m.d. or 1 milligram difference is further sub-divided into 5 sub-division :

1 s.d. = 1/5 m.d. = 1/5 mg = 0.2 mg. = (0.0002 g.)

It is suggested that recording of weights be strictly done as follows :

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d.
10 2	5 2	5 1	3	2 (2)
12	7	6	0	0
R(+)0	0	0	3	4
12	7	6	3	4

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d
10 1	0	5 2	8	1 (2)
11	0	7	0	0
R(+)0	0	0	8	2
11	0	7	8	2

Wt. of the Substance = 12.7634 - 11.0782 = 1.6852g.

TITRATION DATA, TABULATION AND CALCULATIONS

Titration data should essentially be recorded as follows :

The following example is for **single Titration :** std. $H_2C_2O_4 . 2H_2O$ Vs. Fe^{2+} (test solution).

i. Normality of prepared std. Oxalic acid solution :

Wt . $H_2C_2O_4 . 2H_2O$ = (12.7634 - 11.0782) g = 1.6852 g.

(from tables of weight above)

supposing solution is made in 250 ml

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

ii. Titration Data :

Titration of Std. $H_2C_2O_4 . 2H_2O$ Vs. $KMnO_4$ (link)

Sl. No.	Voxalic (Std.) ml	Burette Reading		V_{KMnO_4} 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

i) $N_{KMnO_4\,(Link)} \times V_{KMnO_4\,(Link)} = N_{\text{oxalic acid}\,(Std.)} \times V_{\text{oxalic acid}\,(Std.)}$

$$N_{KMnO_4} = \frac{N_{\text{oxalic acid}} \times V_{\text{oxalic acid}}}{V_{KMnO_4}} = \frac{0.1070 \times 20}{19.8} = 0.1080$$

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

2. N std. oxalic acid = ...

3. N $KMnO_4$ = ...

* Education is what remains after one has forgotten what one has learned in school
- Einstein

Date :

Weighing, Demonstration and Titration

TABULATION AND CALCULATIONS

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

Table.1. Weight of bottle + FAS

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d.

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

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

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

Part B : Standardisation of $KMnO_4$:

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

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

RESULTS :

1. Wt. of FAS =
2. $N_{std.\ FAS\ Solution}$ =
3. $N_{KMnO_4\ Solution}$ =

$$N_{KMnO_4} = \frac{N_{FAS} \times V_{FAS}}{V_{KMnO_4}} =$$

Signature of Faculty

Standard FAS x $KMnO_4$(Link) x Fe^{2+}(Test solution) (Instrumental)

Date:

Expt. No.

TABULATION AND CALCULATIONS

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

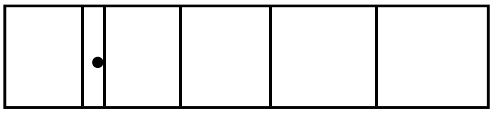
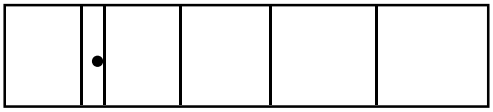
Table.1. Weight of bottle + Substance  g.

Table.2. Weight of empty weighing bottle  g.

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

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

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

Part B : Standardisation of $KMnO_4$:

Titration of Std. F.A.S. Vs. $KMnO_4$ (link)

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

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

$$N_{KMnO_4} = \frac{N_{FAS} \times V_{FAS}}{V_{KMnO_4}} =$$

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	

$$N_{Fe^{2+}}\ (Test) = \frac{N_{KMnO_4} \times V_{KMnO_4}}{V_{Fe^{2+}}} = \ldots\ldots\ldots$$

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

Results:

1. Weight of FAS = _____g
2. $N_{\text{std. FAS}}$ = _____
3. N_{KMnO_4} (Link) = _____
4. $N_{Fe^{2+}}$ (Test) = _____
5. Weight of Fe^{2+} present in test solution = _____gpl

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Experiment (1) : PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION & STANDARDISATION OF GIVEN $KMnO_4$ SOLUTION.

Expt. No. **Date :**

TABULATION AND CALCULATIONS

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

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d.

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

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

(V_{ml} = 100 ml)

Part B : Standardisation of $KMnO_4$:

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

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

$$N_{KMnO_4} = \frac{N_{FAS} \times V_{FAS}}{V_{KMnO_4}} =$$

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

RESULTS :

1. Wt. of FAS =
2. ..... $_4$ Solution =

Signature of Faculty

'REDOX' TITRATION'
(PERMANGANATOMETRY)

Experiment (1) : PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION & STANDARDISATION OF GIVEN $KMnO_4$ 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 acid solution, Ferrous ion (Fe^{2+}) is readily oxidized by $KMnO_4$; the redox reactions can be represented by the equation given below :

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 instead of $FeSO_4$ for the standardization of $KMnO_4$.

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

(Partial Ionic equation.)

Reduction : $MnO_4^- + 8H^+ + 5e^-$	$\rightarrow$	$Mn^{2+} + 4H_2O$	$E^0_R = 1.51$ V
Oxidation : $5 \times [Fe^{2+}$	$\rightarrow$	$Fe^{3+} + e^-]$	$E^0_R = 0.77$ V

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 : (or) No. of e^- transfer)

Equivalent weights : $KMnO_4 / 5 = 158/5 = 31.6$;

$FeSO_4 . (NH_4)_2 SO_4\ 6H_2O/1 = 392.1 / 1 = 392.1$

Procedure : A : Preparation of standard Ferrous Ammonium Sulphate $Fe SO_4 (NH_4)_2 SO_4 \cdot 6H_2O$ solution : Weigh out in a clean weighing bottle accurately close to 3.9210 g Ferrous Ammonium sulphate $FeSO_4 . (NH_4)_2 SO_4 . 6H_2O$; transfer it to 100 ml std. standard Flask through a funnel ; dissolve it by adding distilled water including 10 ml dil. $H_2 SO_4$ (6N) (to prevent hydrolysis) and make the solution up to the mark. Shake it well to make it homogeneous.

Hydrolysis Reactions :

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

B. STANDARDIZATION OF GIVEN $KMnO_4$ SOLUTION :

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

i) Pipette out 20 ml of the prepared std. Fe^{2+} Amm. Sulphate solution into a clean conical flask.

ii) Add 10 ml of dilute H_2SO_4 (6N) solution (1/2 Test Tube)

iii) Titrate with $KMnO_4$ (taken in the burette) to a faint pink colour which persists for at least a minute. This is the end point. (last addition of $KMnO_4$ should be dropwise with particular care to allow each drop to become decolorized before the next is added).

iv) Repeat the process till at least two concordant values are obtained.

RESULTS :

1. Wt. of FAS = ... g
2. $N_{\text{std. FAS Solution}}$ = ...
3. $N_{KMnO_4\ \text{Solution}}$ = ...

Questions :

1. What is the colour of Mn^{2+} ion ?
2. Why no indicator is added in this experiment ?
3. Why is dilute H_2SO_4 added in the standard flask while making the standard solution ?
4. Which substance gives the pink colour at the end point ?
5. Can we use dilute HCl instead of dilute H_2SO_4 ?
6. Why is dilute H_2SO_4 added to Fe^{+2} solution in the conical flask, during titration with $KMNO_4$?

* Knowledge is the true organ of sight, not the eyes.
- Panchatantra

'REDOX' TITRATION'

(PERMANGANATOMETRY)

Experiment (2) : PREPARATION OF STANDARD OXALIC ACID OR OXALATE SOLUTION & STANDARDISATION OF GIVEN $KMnO_4$ SOLUTION.

Object : To standardize $KMnO_4$ solution using Std. Oxalic acid solution.

Introduction : (*Theory*) : Although Oxalic acid is potentially an active reducing agent, the rate of its reaction is slow in the absence of catalyst. The only reaction of analytical importance is that with permanganate in acid solution. Therefore, the reaction is initiated by heating Oxalic acid and subsequently it is catalysed by Mn^{2+} ions, (one of the products).

Oxalic acid, as also sodium oxalate, which can be used for this standardization is obtainable in a state of high purity and therefore used as primary standard.

(Partial Ionic equation.)

Reduction : $2 \times [MnO^-_4 + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O]$ $E^0_R = 1.51$ V

Oxidation : $5 \times [C_2O^{2-}_4 \rightarrow 2CO_2 + 2e^-]$ $E^0_R = 0.49$ V

Overall Ionic equation :

Redox : $2MnO^-_4 + 16H^+ + 5C_2O^{2-}_4 + 10e^- \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2 + 10e^-$

Species:	$(KMnO_4)$	(H_2SO_4)	$(H_2C_2O_4)$	$(MnSO_4)$	
Color Profile :	(Purple)	(X)	(X)	(X)	X= Colorless

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

Equivalent weights $KMnO_4 / 5 = 158 / 5 = 31.6$; $H_2C_2O_4 . 2H_2O/2 = 126/2 = 63$

Procedure A : Preparation of Std. Oxalic acid ($H_2C_2O_4 . 2H_2O$) Solution :

Weigh out in a clean weighing bottle accurately close to 1.5750 g. $H_2C_2O_4 \cdot 2H_2O$; transfer it to a 250 ml std. Vol. flask through a funnel; dissolve it in distilled water and make up to the solution to the mark. Shake well to make it homogeneous.

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

Experiment (2) : PREPARATION OF STANDARD OXALIC ACID OR OXALATE SOLUTION & STANDARDISATION OF GIVEN $KMnO_4$ SOLUTION.

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic acid ($H_2C_2O_4 . 2H_2O$) Solution :

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d.

Wt. of Oxalic Acid= W_1-W_2

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

(V_{ml} = 250 ml)

Part B. Standardisation of $KMnO_4$

Titration of Std. $H_2C_2O_4.2H_2O$ Vs. $KMnO_4$

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

$$N_{KMnO_4} = \frac{N_{oxalic} \times V_{oxalic}}{V_{KMnO_4}} =$$

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

RESULTS :

1. Wt. of Oxalic acid = ...g
2. $N_{std.}$ Oxalic Solution = ...
3. N_{KMnO_4} Solution = ...

Signature of Faculty

B. STANDARDIZATION OF GIVEN $KMnO_4$ SOLUTION :

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

i) Pipette out 20 ml. of the prepared solution (standard Oxalic acid) into a clean conical flask.

ii) Add approximately (1/2 Test tube) 10 ml. of dilute H_2SO_4 (6N).

iii) Heat the mixture to ~ 70^0C (as observed by bubbling).

iv) While the solution is hot, titrate with $KMnO_4$ (taken in a burette) to a **Faint Pink colour** which persists for at least a minute. This is the end point. (last additions of $KMnO_4$ should be drop-wise, with particular care to allow each drop to become decolorized before the next is introduced).

v) Repeat the process till at least two concordant values are obtained.

Questions :

1. What is the difference in nature of Mohr's salt and oxalic acid ?
2. Why is heating required in this experiment ?
3. While adding $KMnO_4$ to oxalic acid solution, the decolouration is slow initially, why ?
4. At room temperature which reaction has higher activation energy ? FAS vs $KMnO_4$ or oxalic acid vs $KMnO_4$. Justify the answer.

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

Expt. No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic acid ($H_2C_2O_4 . 2H_2O$) Solution :

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d.

Wt. of Oxalic Acid= W_1-W_2

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

(V_{ml} = 250 ml)

Part B : Standardisation of $KMnO_4$ (link)

Titration of Std. $H_2C_2O_4.2H_2O$ Vs. $KMnO_4$ (link)

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

$$N_{KMnO_4} = \frac{N_{oxalic} \times V_{oxalic}}{V_{KMnO_4}} =$$

'REDOX LINK TITRATION'

(PERMANGANATOMETRY)

Experiment (3): Preparation of Standard Oxalic Acid, Standardisation of $KMnO_4$ (Link) Solution & Estimation of Fe^{2+} in the given Test Solution.

[Note : Typical Link Titration Experiment (as a model for writing in the record]

Aim: To estimate the amount iron of Fe^{2+} in test solution by preparing a Standard Oxalic acid solution & using link $KMnO_4$ solution.

Principle:

1. Reaction between Oxalic and Permanganate

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

Oxidation : $5 \times [C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-]$ $E^0_R = 0.49$ V

Overall Ionic equation :

Redox : $2MnO_4^- + 16H^+ + 5C_2O_4^{2-} + 10e^- \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2 + 10e^-$

Species:	$(KMnO_4)$	(H_2SO_4)	$(H_2C_2O_4)$	$(MnSO_4)$	
Color Profile:	(Purple)	(X)	(X)	(X)	X= Colorless

2. Reaction between Ferrous and Permanganate

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

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

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

$KMnO_4$ a powerful oxidising agent, oxidises Oxalic acid to CO_2 in acidic medium in hot condition while itself undergoes reduction to $MnSO_4$. In estimation part it oxidises Fe^{2+} to Fe^{3+} in acidic medium.

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

Equivalent weights $KMnO_4$ / 5 = 158 / 5 = 31.6; $H_2C_2O_4 . 2H_2O/2$ = 126/2 = 63; Fe^{2+} = 55.8

Procedure

Part A : Preparation of Std. Oxalic acid ($H_2C_2O_4 . 2H_2O$) Solution :

Exact □.□□□□gm of Oxalic acid is weighed out accurately up to 4th decimal place in a clean weighing bottle. It is transferred to a 250 ml standard Volumetric flask through a funnel then dissolved in distilled water and the solution is made upto the mark. It is shaken well to make it. The Normality of Standard Solution is calculated from the weighing data.

Part C: Estimation of Fe^{2+} in the given Test Solution.

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

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

$$N_{Fe^{2+}}\ (Test) = \frac{N_{KMnO_4} \times V_{KMnO_4}}{V_{Fe^{2+}}} = ...$$

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

=.....gpl

Results:

1. Weight of Oxalic Acid = _____g
2. $N_{\text{std. Oxalic acid}}$ = _____
3. N_{KMnO_4} (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

Part B. Standardisation of $KMnO_4$ (Link Solution)

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

20 ml. of the prepared solution (standard Oxalic acid)is pippetted out into a clean conical flask. Approximately (1/2 Test tube) 10 ml. of dilute H_2SO_4 (6N) is added to it. The mixture is heated to ~ 70^0C (as observed by bubbling).While the solution is hot, it is titrated with $KMnO_4$ (taken in a burette) till a **Faint Pink colour** which persists for at least a minute. This is the end point. (last additions of $KMnO_4$ should be drop-wise, with particular care to allow each drop to become decolorized before the next is introduced).

The process is repeated till two concordant titre values are obtained. From the titration data, normality of $KMnO_4$ is calculated.

Part C:Estimation of Fe^{2+} in the given Test Solution.

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

Other Possible Link Experiments:

	Std. Soln.	Link Soln.	Test Soln.
(i)	Oxalic	$KMnO_4$	Oxalic
(ii)	FAS	$KMnO_4$	Oxalic
(iii)	FAS	$KMnO_4$	Fe^{2+}
(iv)	FAS	$K_2Cr_2O_7$	Fe^{2+}
(v)	$K_2Cr_2O_7$	Hypo	$Cr_2O_7^{2-}$
(vi)	$K_2Cr_2O_7$	Hypo	Cu^{2+}

Experiment (4) : PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION AND ESTIMATON OF Fe^{2+} (FERROUS) IRON

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. $K_2Cr_2O_7$

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	s.d.

Wt. of $K_2Cr_2O_7 = W_1 - W_2 =$

$$N_{(std)}\ K_2Cr_2O_7 = \frac{wt}{Eq.wt} \times \frac{1000}{V_{ml}} = \ldots\ldots\ldots\ldots$$

(V_{ml} = 250 ml)

Part B: Estimation of Fe^{2+} in the given Test Solution.

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

$$N_{Fe^{2+}}\ (Test) = \frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{Fe^{2+}}} = \ldots\ldots\ldots$$

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

$$= \_\_\_\_\_ \text{ gpl}$$

'REDOX' TITRATION'

(DICHROMATOMETRY)

Experiment (4) : PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION AND ESTIMATON OF Fe^{2+} (FERROUS) IRON

Object : To estimate Iron Fe^{2+} using standard $K_2Cr_2O_7$ solution.

Introduction : (*Theory*) : Ferrous ammonium sulphate, ferrous alum (Mohr's salt) is a double salt of the composition $FeSO_4 \cdot (NH_4)_2 SO_4 \cdot 6H_2O$. This when dissolved in water breaks up into simple salts and water. Ferrous sulphate is a reducing substance. In the titration with a standard solution of $K_2Cr_2O_7$, the volume of the oxidizing substance to completely oxidize ferrous iron Fe^{2+} to Ferric Iron (Fe^{3+}) is found out. Potassium dichromate undergoes reduction because of high reduction potential. It oxidizes Fe^{2+} (ous) to Fe^{3+} (ic) and in the process gets converted to Cr^{3+} which is green in colour even after equivalance point. In order to identify the equivalance point 3-5 drops of diphenyl-amine is added as an internal indicator (E^0_{Red} = 0.76V). In order to prevent the oxidation of diphenyl amine before the oxidation of Fe^{2+}, phosphoric acid is added which reduces the reduction potential of iron couple from 0.77V to 0.44V.

(Partial Ionic equation) :

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

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

Overall Ionic equation :

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

Species: $(K_2Cr_2O_7)$ (H_2SO_4) $(FeSO_4)$ $(Cr(SO_4)_3$

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

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

(Eq. Wts.: $K_2Cr_2O_7/6 = 294.2/6 = 49.0$;

$FeSO_4 . (NH_4)_2 SO_4 . 6H_2O$ / 1= 392.1; Fe^{2+} = 55.8)

PROCEDURE : A : PREPARATION OF STD. $K_2Cr_2O_7$ SOLUTION :

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

B. ESTIMATON OF Fe^{2+} (FERROUS) IRON :

(Note : wash all glass apparatus with tap water, then rinse with distilled water, subsequently rinse the pipette and burette with their respective solutions).

* The important thing in science is not so much to obtain new facts as to discover new ways of thinking about them.

i) Make up the given Fe^{2+} Amm. Sulphate test solution up to the mark with distilled water. Shake the solution thoroughly to make it homogeneous. Pipette out 20 ml. of this made up Fe^{2+} Ammonium sulphate test solution into a conical flask.

ii) Add 10 ml of dilute H_2SO_4 (6N) and then 3 to 5 drops of internal indicator diphenylamine; (1% solution in concentrated H_2SO_4), followed by about 5 ml (1/4th Test Tube) of syrupy phosphoric acid which lowers the reduction potential of Iron couple and facilitates the initial oxidation of Ferrous to Ferric Ions.

iii) Now titrate very slowly with std. $K_2Cr_2O_7$. The solution initially acquires green colour due to formation of Cr^{+3} , its intensity increases the solution acquires a bluish green tint which shows that the end point is near. The Titration is continued now with dropwise addition of $K_2Cr_2O_7$ and good stirring until addition of a drop causes the formation of an intense **bluish violet colour** at the end point. Bluish violet colour is due to oxidation of DPA by $K_2Cr_2O_7$ which is stable. Repeat the titration to obtain at least two concordant values.

RESULTS :

1. Wt. of $K_2Cr_2O_7$ = _____g
2. ^{N} Std. $K_2Cr_2O_7$ solution =_____
3. ^{N} Fe^{2+} (test) solution =_____
4. Wt. of Fe^{2+} in the test solution =_____gpl

Questions :

1. Why is $K_2Cr_2O_7$ not a self indicator ?
2. Why is the colour of the contents of conical flask green before the end point ?
3. Which substance gives the bluish violet colour at the end point ?
4. What is the change in oxidation number of chromium in this reaction ?
5. What is the role of phosphoric acid in this experiment ?

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

Signature of Faculty

'REDOX' TITRATION'

(DICHROMATOMETRY)

Experiment (5) : Preparation of standard Ferrons Ammonium Sulphate, Standardisation of $K_2Cr_2O_4$ (Link) Solution & Estimation of Fe^{+2} in given test solution :

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

Theory : Ferrons Ammonium Sulphate, Ferrons alum (Mohr's Salt) is a double salt of the combination $FeSO_4$ $(NH_4)_2SO_4.6H_2O$ this when dissolved in water breaks up into simple salts and water. Ferrous sulphate is a reducing substance. In the titration with a standard solution of $K_2Cr_2O_7$, the volume of oxidant ($K_2Cr_2O_4$) completely oxidizes ferrous iron Fe^{+2} to Ferric Iron (Fe^{+3}) is found out $K_2Cr_2O_7$ undergo reduction because of high reduction potential. It oxidizes Fe^{+2} (ous) to Fe^{+3}(ic) and in the process gets converted to Cr^{+3} which is green in colour even after equivalence point. In order to identify the equivalence point 3-5 drops of diphenylamine added as an internal indicator (E^0_{Red} = 0.76v). In order to prevent the oxidation of diphenyl amine before the oxidation of Fe^{+2}, Phosphoric acid is added which reduces the reduction potential of iron couple from 0.77v to 0.44v.

Partial Ionic Equation :

Reduction : $Cr_2O_7^{-2} + 14H^+ + 6e^- \rightarrow 2Cr^{+3} + 7H_2O$ $E_R^o = 1.33V$

Oxidation : $6 \times [Fe^{+2} \rightarrow Fe^{+3} + e^-]$ $E_R^o = 0.77V$

Overall Ionic Equation

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

Species :	$(K_2Cr_2O_7)$	(H_2SO_4)	$(FeSO_4)$	$Cr_2(SO_4)$	$Fe_2(SO_4)_3$
Colour Profile :	(Orange)	(X)	(X)	(Greenish)	(X)

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

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

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

Experiment (5) : Preparation of standard Ferrons Ammonium Sulphate, Standardisation of $K_2Cr_2O_4$ (Link) Solution & Estimation of Fe^{+2} in given test solution :

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. FAS Solution :

Table.1. Weight of bottle + Substance

Grams	Milligrams			
			Rider (+/-)	
	100's	10's	m.d.	s.d.

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
			Rider (+/-)	
	100's	10's	m.d.	s.d.

Wt. of FAS $= W_1 - W_2 =$

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

(V_{ml} =100ml)

Part B. Standardisation of $K_2Cr_2O_7$

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

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

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

Procedure :

Part A : PREPARATION OF STD. F.A.S. SOLUTION :

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

PART B : Standarisation of $K_2Cr_2O_7$:

20ml of the prepared solution (standard FAS) is pippetted out into a clean conical flask. Approximately (1/2 Test Tube) 10 ml of dilute H_2SO_4 (6N) is added to it, and then 3 to 5 drops of internal indicator diphenylamine and about 8ml (1/3 T. T) of Syrupy Phosphoric acid ..... is added which lowers the reduction potential of Iron couple and facilitates the initial oxidation of Ferrons to Ferric. It is titrated with $K_2Cr_2O_7$ (taken in Burette) very slowly until the solution acquires a bluish greentint which shows that the end point is near. The titration is continued now with dropwise addition of $K_2Cr_2O_7$ and good stirring until addition of a drop causes the formation of bluish colour , which is stable.

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

PART C. Estimation of Fe^{+2} (Ferrous) Iron in given test solution :

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

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

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	

$$N_{Fe^{2+}}\ (Test) = \frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{Fe^{2+}}} = ...$$

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

Results:

1. Weight of FAS = _____g
2. $N_{\text{std. FAS}}$ = _____
3. $N_{K_2Cr_2O_7}$ (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

Expt. Std. FAS X K MnO_4 (Link) X Fe^{2+} (Test Soln.) (Volumetric Lab)

Date :

Expt. No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. FAS Solution :

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d

Table.2 Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d.

Wt. of FAS = W_1-W_2 =

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

(V_{ml} =100 ml)

Part B. Standardisation of $KMnO_4$ (link)

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

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

$$N_{KMnO_4} = \frac{N_{FAS} \times V_{FAS}}{V_{KMnO_4}} =$$

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	

$$N_{Fe^{2+}\ (Test)} = \frac{N_{KMnO_4} \times V_{KMnO_4}}{V_{Fe^{2+}}} = \ldots\ldots\ldots$$

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

=

Results:

1. Weight of FAS = _____g
2. $N_{\text{ std. FAS}}$ = _____
3. N_{KMnO_4} (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

'REDOX' TITRATION'

(IODOMETRY) **(DICHROMATOMETRY)**

Experiment (6) : PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION AND STANDARDIZATION OF $Na_2S_2O_3.5H_2O$ SOLUTION AND ESTIMATION OF Cr^{+3} FROM THE GIVEN TEST SOLUTION

Object : To Estimte Cr^{+3} using standard $K_2Cr_2O_7$ solution & (link) hypo solution .

Introduction :(Iodometry deals with the titration of iodine liberated in a chemical reaction).

In this titration potassium dichromate $K_2Cr_2O_7$ is the oxidising substance and $Na_2S_2O_3.5H_2O$ sodium thiosulphate **(Hypo)** is the reducing substance. The titration is done by adding to the solution of the oxidant, a large excess (roughly measured) of potassium iodide. The oxidant is reduced, liberating an equivalent amount of iodine, and the liberated iodine is titrated with thiosulphate. The direct oxidation of the thiosulphate by dichromate cannot be carried out because of lack of suitable internal indicators.

The number of equivalents of thiosulphate required for reducing liberated iodine would be equal to the number of equivalents of $K_2Cr_2O_7$ taken. Hence, by measuring the volume of thiosulphate, its normality can be determined.

(Partial ionic equation)

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

Oxidation : $3 \times [2I^- \rightarrow I_2 + 2e^-]$

Overall Ionic equation : $E^0_R = 0.62V$

Redox : $Cr_2O_7^{2-} + 14\,H^+ + 6I^- + 6e^- \rightarrow 2Cr^{3+} + 7H_2O + 3I_2 + 6e^-$

Species:	$(K_2Cr_2O_7)$	(HCl)	(KI)	$(CrCl_3)$	I_2
Color Profile:	(Orange)	(X)	(X)	(Green)	(Redish Brown)

X = Colourless

The reaction (Redox) between liberated Iodine and thiosulphate is

$$I_2 + 2S_2O_3^{2-} + 2e^- \rightarrow 2I^- + S_4O_6^{2-} + 2e^- \text{ (tetrathionate) ;}$$

Species :	I_2	$Na_2S_2O_3$	NaI	$Na_2S_4O_6$	
Color Profile:	(Redish Brown)	(X)	(X)	(X)	X = Colourless

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

Equivalent weights : $K_2Cr_2O_7 / 6 = 294.2 / 6 = 49.03$; $Na_2S_2O_3.5H_2O / 1 = 248.1$,

$Cr^{+3} = 52/3 = 17.3$

Experiment (6) : PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION AND STANDARDIZATION OF $Na_2S_2O_3.5H_2O$ SOLUTION AND ESTIMATION OF Cr^{+3} FROM THE GIVEN TEST SOLUTION

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. $K_2Cr_2O_7$:

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d.

Wt. of $K_2Cr_2O_7 = W_1 - W_2 =$

$$N_{(std)}\ K_2Cr_2O_7 = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = ...$$

(V_{ml} = 250 ml)

Part B. Standardisation of Hypo Soln:

Titration of Std. $K_2Cr_2O_7$ Vs. Hypo

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

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

PROCEDURE : A : PREPARATION OF STD. $K_2Cr_2O_7$ SOLUTION :

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

B. STANDARDISATION OF HYPO SOLUTION :

(i) Pipette out 20 ml of the prepared std. $K_2Cr_2O_7$ solution into a conical flask.

(ii) Add 20 ml. of KI (10%) solution and then 10 ml. of dilute HCl. Also add a pinch of $NaHCO_3$ to prevent oxidation by air by providing a CO_2 atomosphere. Mix the contents and keep the covered conical flask in dark for 5 minutes (to avoid photochemical reaction). Take out the flask. Spray-wash the inner walls with distilled water & shake well.

(iii) While swirling the liquid in the conical flask, add thiosulphate solution from a burette very slowly until the Reddish Brown colour changes to faint Yellow with greenish tint.

(iv) Add at this stage 5 - 6 ml. (2 - 3 droppers) of freshly prepared (1%) starch solution. The colour should change to blue. Continue addition of thiosulphate until one drop changes the colour from **blue** to **light green** (due to chromium salt) stable upto a minute. Repeat the titration to get at least two concordant values.

C. Estimation of Cr^{+3} (Chromium) in given test solution :

The given **Cr^{+3}** test solution is made upto the mark with distilled water. The solution is thoroughly shaken to make it homogenous. 20ml of the test **Cr^{+3}** solution is pipetted out in a clean conical flask. Follow the procedure mentioned in Part B The process is repeated till two concordant titre values are obtained. From the data Normality of **Cr^{+3}** and then weight of **Cr^{+3}** in test solution is

Questions :

1. Does $K_2Cr_2O_7$ react with hypo directly ?
2. What is the oxidation number of sulphur in tetrathionate ion ?
3. What is the amount of chromium in gpl in a solution of 0.1 N $K_2Cr_2O_7$?
4. What is the oxidation number of Cr in $K_2Cr_2O_7$?

Part C: Estimation of Cr^{+3} in the given Test Solution :

Titration : Cr^{+3} (Test Soln.) Vs. Hypo (Link)

Sl. No.	$V_{Cr^{+3}}$ (test Sol.) ml	Burette Reading		V_{Hypo} ml
		Initial	Final	

$$N_{Cr^{+3}}\ (Test) = \frac{N_{Hypo} \times V_{Hypo}}{V_{Cr^{+3}}} = ...$$

$$\text{Wt.of } Cr^{+3} \text{ (in Test solution)} = N_{Cr^{+3}\ (Test)} \times \text{Eq. wt.} =$$

=

RESULTS :

1. Wt. of $K_2Cr_2O_7$ =----------g
2. ^{N} $K_2Cr_2O_7$ =----------
3. ^{N} Hypo =----------
4. $^{N}Cr^{+3}$ (Test)=----------
5. Weight of Cr^{+3} present in test solution =_____gpl

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

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Experiment (7) : **PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION, STANDARDIZATION OF $Na_2S_2O_3.5H_2O$ SOLUTION, ESTIMATION OF COPPER Cu^{2+} IN THE GIVEN TEST SOLUTION**

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. $K_2Cr_2O_7$:

Table.1. Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d.	s.d.

Wt. of $K_2Cr_2O_7 = W_1 - W_2 =$

$$N_{(std)}\ K_2Cr_2O_7 = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = ...$$

(V_{ml} = 250 ml)

Part B : Standardisation of Hypo Soln (Link) :

Titration of Std. $K_2Cr_2O_7$ Vs. Hypo (Link)

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

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

'REDOX' TITRATION'

(IODOMETRY)

Experiment (7) : PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION, STANDARDIZATION OF $Na_2S_2O_3.5H_2O$ SOLUTION, ESTIMATION OF COPPER Cu^{2+} IN THE GIVEN TEST SOLUTION

Object : To Estimate Cu^{2+} (ic) using standard hypo.

Introduction : (*Theory*) : In this titration copper sulphate $CuSO_4$ is the oxidizing substance and sodium thiosulphate $Na_2S_2O_3.5H_2O$ (hypo) is reducing substance. The titration is done by adding to the solution of the oxidant, a large excess of potassium iodide. The oxidant is reduced, liberating an equivalent amount of iodine, and the liberated iodine is titrated with Std. Sodium thiosulphate solution ($Na_2S_2O_3.5H_2O$).

(Partial ionic equation)

Reduction : $2Cu^{2+} + 2e^- + 2I^- \rightarrow Cu_2I_2$ $\quad E^0_R = 0.86V$

Oxidation : $2I^- \rightarrow I_2 + 2e^-$ $\quad E^0_R = 0.62V$

Overall Ionic equation :

Redox : $2Cu^{2+} + 4I^- + 2e^- \rightarrow Cu_2I_2 + I_2 + 2e^-$

Specimen (CuSO4)	KI	Cu_2I_2	I_2	X = Colourless
Colour Profile	Blue	(X)	Cream/Buff	Reddish Brown

The reaction (Redox) between liberated iodine and thiosulphate is :

Reduction : $I_2 + 2e^- \rightarrow 2I^-$ $\quad E^0_R = 0.62V$

Oxidation : $2S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2e^-$ $\quad E^0_R = 0.09V$

Overall Ionic Equation :

$$I_2 + 2S_2O_3^{2-} + 2e^- \rightarrow 2I^- + S_4O_6^{2-} + 2e^-$$

(Thiosulphate) (Tetrathionate)

Species :	I_2	$Na_2S_2O_3$	NaI	$Na_2S_4O_6$	
Color Profile:	(Redish Brown)	(X)	(X)	(X)	X = Colourless

Part C: Estimation of Cu^{2+} in the given Test Solution.

Titration : Cu^{2+} (Test Soln.) Vs. Hypo (Link)

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

$$N_{Cu^{2+}} = \frac{N_{Hypo} \times V_{Hypo}}{V_{Cu^{2+}}} =$$

Wt.of Cu^{2+} = NCu^{2+} x Eq.Wt. =

Results:

1. Weight of $K_2Cr_2O_7$ = _____g
2. $N_{std.}$ $K_2Cr_2O_7$ = _____
3. N_{Hypo} (Link) = _____
4. $N_{Cu^{2+}}$ (Test) = _____
5. Weight of Cu^{2+} present in test solution = _____gpl

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

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

Equivalent weights : $CuSO_4 \cdot 5H_2O/1 = 249.7$; $Na_2S_2O_3, 5H_2O/1 = 248.1$

$Cu^{2+}/1 = 63.5$

PROCEDURE : A : PREPARATION OF STD. $K_2Cr_2O_7$ SOLUTION :

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

B. Standardization of HYPO :

(i) Pipette out 20 ml of the prepared std. $K_2Cr_2O_7$ solution into a conical flask.

(ii) Add 20 ml. of KI (10%) solution and then 10 ml. of dilute HCl. Also add a pinch of $NaHCO_3$ to prevent oxidation by air by providing a CO_2 atomosphere. Mix the contents and keep the covered Conical flask in dark for 5 minutes (to avoid photochemical reaction). Take out the flask. Spray-wash the inner walls with distilled water and shake well.

(iii) While swirling the liquid in the conical flask, add thiosulphate solution from a burette very slowly until the Reddish Brown colour changes to faint Yellow with greenish tint.

(iv) Add at this stage 5 - 6 ml. (2 - 3 full droppers) of freshly prepared (1%) starch solution. The colour should change to blue. Continue addition of thiosulphate until one drop changes the

colour from **blue** to **light green** (due to chromium salt) stable upto a minute. Repeat the titration to get at least two concordant values.

C. Estimation of Cu^{2+} :

(i) First make up the given Copper solution to 100ml in the Std. Vol. flask.

(ii) Pipette out 20 ml. of this made up copper solution into a conical flask.

(iii) Add dil. NH_4OH solution drop by drop until a faint permanent blue ppt. appears

(iv) Now add dil. acetic acid to just dissolve the ppt. the turbidity must disappear. The purpose of this procedure is to remove the mineral acid from the solution; the presence of such acid causes an indistinct end point because in case of mild oxidizing substance ($CuSO_4$) Iodine will be liberated not by $CuSO_4$ alone but also by the acid according to the following equation.

$KI + H_2SO_4 \rightarrow KHSO_4 + HI$; $2HI + H_2SO_4 \rightarrow I_2 + SO_2 + 2H_2O$

The excess of NH_4OH is neutralized by acetic acid.

(v) Add 20 ml of KI (10%) solution. Mix the Contents and then keep the conical flask covered with a watch glass in dark for 5 minutes. Take out the flask.and then add 25 ml of distilled water, (Spray wash).

(vi) Run in very slowly thiosulphate solution from the burette until the yellow colour of iodine has almost (not completely) disappeared.

(vii) Add at this stage 5 - 6 ml. of freshly prepared starch solution. There would be a **violet blue** colour. Also add a pinch of NH_4SCN solid . The blue colour would instantly become more intense (or greyish blue) continue the titration dropwise until the colour changes, into a **white** or **creamy** precipitate. This is the end point.

(**Note :** NH_4 SCN addition is recommended shortly before the end point. This serves to convert the cuprous iodide to cuprous thiocyanate at least on the surface of the particles)

$$Cu.I + CNS^- \rightarrow Cu.CNS + I^-$$

Cuprous iodide has a marked tendency to hold iodine molecules to its surface whereas cuprous thiocynate has this property to a much smaller extent. Therefore, the absorbed iodine is made available for titration with hypo; it leads to a sharper end-point and, accurate results.

Questions :

1. Why should we use KI in this titration ?
2. What is the change in oxidation state of copper in this reaction ?
3. What is the purpose of addition of NH_4OH and CH_3COOH ?
4. What is the role of starch ?
5. What happens when NH_4SCN is added to the contents of the flask just before the end point ?

* The scientist is not a person who gives the right answers, he the one who asks the right question.

WATER ANALYSIS

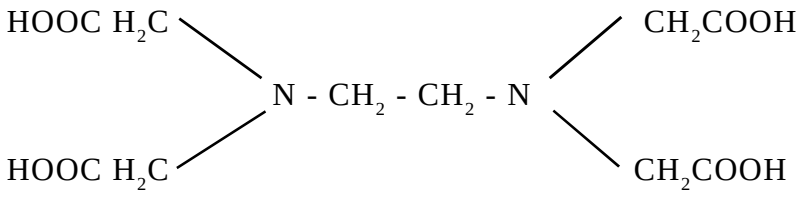
'COMPLEXOMETRIC' TITRATION

Experiment (8): Estimation of Temporary & Permanent Hardness of water using EDTA Solution.

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

EDTA = Ethylene Di ammine Tetra Acetic acid

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



A useful abbreviation is H_4Y. For practical purposes the disodium salt Na_2H_2Y is preferred as a reagent; it affords the complex - forming ion H_2Y^{2-} in aqueous solution. The reaction with cations, eg M^{2+} may be written as:

$$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+$$

(the disodium salt of EDTA available commercially is $CH_2.N(CH_2.COOH)_2\ CH_2.\ N(CH_2.COONa)_2.\ 2H_2O$ with formula wt = 372.24. It is also written as Na_2H_2 versenate $2H_2O$ i.e. disodium dihydrogen versenate).

* They are ill discoverers that think there is no land, when they can see nothing but the sea.

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

The order of stability is as follows:

Ca - EDTA > Mg - EDTA > Mg - Indicator

(Colourless) (Wine-red)

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

Mg - Indicator + EDTA $\rightarrow$ Mg - EDTA + Indicator

(Wine-red) (colorless) (blue)

Preparation of solutions:

1. **Standard hard water :** weigh out accurately 1.0000 g. dried and pure (A.R.) $CaCO_3$. Convert it to $CaCl_2$ by dissolving in minimum dilute (~6N) HCl. Make it up to 1 litre solution in distilled water. The strength is 1000 ppm.
2. **Buffer Solution :** 67.5 g. of NH_4Cl + 570 ml of conc. NH_4OH, made up to 1 litre with distilled water.
3. **Indicator :** Dissolve 0.5 g. of Eriochrome black 'T' in 100 ml of alcohol or methanol.
4. **EDTA Solution :** 4.0 g. of disodium salt EDTA (Na_2H_2 ver. $2H_2O$) dissolved in distilled water and made to 1 litre.

Procedure : Standardization of EDTA :

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

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

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

Total Hardness (Determination) : Repeat the above process except that in step (i) sample hardwater is used instead of std. hard water.

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

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

A simplified calculation would be as follows:

CALCULATIONS

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

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

$$\text{Temporary Hardenss (difference)} = \frac{V_2 - V_3}{V_1} \times 1000 = \text{ppm.}$$

(Round off the results to the nearest integer)

[**Note :** In this experiment, the standardization of EDTA could also be done by preparing a standard hard water solution using $MgSO_4.7H_2O$, M.W. = 246.5 g]

Questions :

1. To a 100ml water sample 58. 5mg of NaCl is added. What would be the hardness of sample water ?
2. How would you differentiate between distilled water and hard water sample ?

Expt. (8) Estimation of Hardness of Water - EDTA method

Date :

Expt. No.

Part A : Standardisation of EDTA

Titration of Std. hard water Vs EDTA

Sl. No	V Std.hard water ml	Burette Reading		V_{EDTA} ml (V_1)
		Initial	Final	

V_1ml of EDTA = 20ml Std. hard water

1ml EDTA = ? 1ml EDTA = $\frac{20}{V_1}$ = ...mg $CaCO_3$

Part B : Estimation of total hardness of sample water

Titration of sample hard water Vs EDTA

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

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

Part C : Estimation of permanent hardness

Titration of boiled sample water Vs EDTA

Sl. No	V boiled water ml	Burette Reading		V_{EDTA} ml (V_3)
		Initial	Final	

Permanent hardness $= \frac{V_3}{V_1}$ X 1000 = ...=.....ppm

Temporary hardness = (Total - Permanent) hardness .= .....=.....ppm

Part D : Estimation of total hardness of municipal / distilled water

Sl. No	V muncipal water ml	Burette Reading		V_{EDTA} ml (V_4)
		Initial	Final	

Marks

Observations and calculations (20) : _____

Results (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

Hardness $= \frac{V_4}{V_1}$ X 1000 =..... = ...ppm

RESULTS:

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

Signature of Faculty

Experiment (9): (Estimation of Total Hardness of Water using Std. Mg SO_4 and EDTA solution).

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. $MgSO_4$

Table.1. Weight of bottle + $MgSO_4$

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	Rider (+/-) s.d

Table.2. Weight of empty weighing bottle

Grams	Milligrams			
	100's	10's	Rider (+/-) m.d.	Rider (+/-) s.d.

Wt. of $MgSO_4$ = W_1-W_2 =

$$N_{(std)}\ MgSO_4 = \frac{wt}{eq.wt} \times \frac{1000}{V_{ml}} = ...$$

(V_{ml} = 100 ml)

Part B : Standardisation of EDTA

Titration of Std. $MgSO_4$ Vs EDTA

Sl. No	V Std. $MgSO_4$ Soln. ml	Burette Reading		V_{EDTA} ml (V_1)
		Initial	Final	

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

WATER ANALYSIS

'COMPLEXOMETRIC' TITRATION

Experiment (9): (Estimation of Total Hardness of Water using Std. Mg SO_4 and EDTA solution).

Procedure :

Part-A : Preparation of Std. $MgSO_4$ solution.

Weigh accurately, the given $MgSO_4.7H_2O$ (Epson salt) in a weighing bottle and let its weight be W_1 gms. Transfer the salt into a clean 100ml standard volumetric flask using funnel. Take the weight of empty bottle and note down its weight as W_2 gms. Dissolve the substance in minimum quantity of distilled water and make the solution upto the mark with distilled water. Shake the solution thoroughly to get uniform concentration. Calculate the normality of standard $MgSO_4$ solution.

Part-B : Standardisation of EDTA solution :

Pipette out 20 ml of the standard hardwater into a clean conical flask. Add about 5ml of (NH_4Cl+NH_3) buffer solution of pH = 10 and 3-4 drops of EBT indicator to it. The color of the solution changes to wine red. Titrate the solution against EDTA solution taken in the burette until the wine red colour of the solution changes to blue. Note down the burette reading and repeat the titration to get concordant titre values.

Part-C :

a) **Estimation of total hardness of water :** Pipette out 20 ml of given sample of water into a clean conical flask. Add about 5 ml of (NH_4Cl+NH_3) buffer solution of pH = 10 and 3 - 4 drops of EBT - indicator to it. Titrate the contents of the conical flask against the EDTA solution taken in the burette until the wine red color solution changes to blue. Note down the burette reading and repeat the titration to get concordant titre values.

b) **Estimation of Permanent Hardness of Water :** Take a 100ml given water sample in a beaker. Boil it (to remove temporary harndess) to around one-fourth of this volume. Filter it (through Whatman 42 fitter paper) to remove insoluble $CaCO_3$ and $MgCO_3$ collect the filterate and make up the volume to the original 100 ml by adding distilled water. Make the solution homogenous. Now pipette out 20 ml of this solution into a conical flask. Then repeat the process of titration steps as in Part- C (a). Note down the burette reading and repeat the titration to get concordant titre values.

Part C : Estimation of total hardness of sample water

Titration of sample hard water Vs EDTA

Sl. No	$V_{sample\ water}$ ml	Burette Reading		V_{EDTA} ml (V_2)
		Initial	Final	

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

Wt interms of $CaCO_3$ equivalents = N x Eq. wt $CaCO_3$

= x 50 = ...gpl.

Total hardness of sample water = x 1000 = mg/lit. / ppm

RESULTS:

1. Source: B/W or Tap water =_____
2. Depth (approximately) =_____
3. Locality =_____
4. N Samplewater = _____
5. Total Hardness =_____ ppm
6. N Boiled water =_____
7. Permanent Hardness =_____ ppm
8. 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 (10): ESTIMATION OF CARBONATE AND BICARBONATE ALKALINITY IN WATER

Introduction:

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

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

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

Principle:

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

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

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

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

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

Reagents required:

(i) Standard HCl (N/50)

(ii) Phenolphthalein indicator

(iii) Methyl orange indicator

Procedure:

A. Preparation of standard N/50 Na_2CO_3 solution :

Weigh out in a clean weighing bottle accurately to about 0.2622g of (A.R.) Na_2CO_3; transfer it to a clean 250ml. standard volumetric flask through a funnel; dissolve it in distilled water and make up the solution to the mark. Shake well to make it homogeneous - Calculate the normality of the standard Na_2CO_3 solution .

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

B. Standardization of HCl (~ N/50)

Pipette out 20ml of the prepared std. 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 (taken in the burette) to a light pink colour; this is the end point. Repeat the process of titration till at least two concordant values are obtained).

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

C. Estimation of CO_3^{2-} & HCO^-_3 Alkalinity in sample water

(i) Transfer 100 ml of the water sample into a clean conical flask, add 2 drops of phenolphthalein indicator, and titrate the sample with the standard HCI until the pink colour just disappears. Note the titre value as the phenolphthalein end-point [P].

(If the water sample does not give pink colour, on additon of pehnolphthalein, check the pH of water. Treat [P] = 0 and proceed for next step.)

(ii) Add 2 to 3 drops of methyl orange indicator to THE SAME solution and continue the titration until a sharp colour change from yellow to red takes place.

Note the total titre value from the beginning of the experiment as methyl orange end-point, [M].

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

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

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

Now, [P] = 10.4 ml; [M] = 25.7 ml,

2 [P] should correspond to CO_3^{2-} and [M] - 2 [P] should correspond to HCO_3^-. Thus, vol. of N/50 HCl equivalent to CO_3^{2-} = 2[P] = 2 x 10.4 ml = 20.8 ml and vol. of N/50 HCl equivalent to HCO_3^- = [M] – 2 [P] = 25.7 – 20.8 ml = 4.9 ml.

Note - Instead of methyl orange, a mixture of bromocresol green and methyl red indicators can be used for better contrast at the end point.

CALCULATION:

(i) $\mathbf{CO_3^{2-}}$ **:**

$$N_{CO_3^{2-}} = \frac{N_{HCl} \times V_{HCl}}{V_{CO_3^{2-}}} = \frac{0.02 \times 20.8}{100} = 0.00416$$

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

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

(ii) $\mathbf{HCO_3^-}$ **:**

$$N_{HCO_3^-} = \frac{N_{HCl} \times V_{HCl}}{V_{HCO_3^-}} = \frac{0.02 \times 4.9}{100} = 0.00098$$

$$\text{Wt. of } HCO_3^- = \frac{N_{HCO_3^-} \times \text{eq.wt.}}{1000} \times 1000 \text{ g/1} = 0.00098 \times 61 \text{ g/1} = 0.05978 \text{ g/1}$$

$$\text{Wt. of } CaCO_3 \text{ eqt.} = 0.05978 \times \frac{50}{61} \text{ g/l} = 0.049 \text{ g/1} = 49 \text{ mg/1} = 49 \text{ ppm}$$

D. Estimation of CO_3^{2-} & HCO^-_3 Alkalinity in test solution

Make up the given test solution upto the mark by adding distilled water. Make the solution homogeneous. Pipette 20 ml of test solution containing both CO_3^{2-} & HCO^-_3 in a clean conical flask. Follow the procedure as given in Part C for the estimation of CO_3^{2-} & HCO^-_3 alkalinity in the given test solution.

Table: Estimation of CO_3^{2-} & HCO^-_3 Alkalinity in test solution

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

Note: 1. In the calculation part $V_{CO_3^{2-}}$ & $V_{HCO_3^-}$ are taken as 20 ml

2. The results are tabulated as given below

EXPT (10): ESTIMATION OF CARBONATE AND BICARBONATE ALKALINITY IN WATER

Expt. No. **Date :**

Table: Estimation of CO_3^{2-} & HCO^-_3 Alkalinity in test solution

S.No	Vol. of test solution (ml)	Vol. of N/50 HCI run down (ml)			
		Phenolphthalein end-point, [P]	2[P]	Methyl Orange end-point, [M]	M-2[P]
1	20				
2	20				

CALCULATION:

(i) $\mathbf{CO_3^{2-}}$ **:**

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

Wt. of CO_3^{2-} = $\frac{N\ CO_3^{2-} \times \text{eq.wt.}}{1000}$ x 1000 g/1 = = g/l

Wt. of $CaCO_3$ eqt. = Wt. of CO_3^{2-} x $\frac{50}{30}$ g/l = x 1000 = ppm

(ii) $\mathbf{HCO_3^-}$ **:**

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

Wt. of HCO^-_3 = $\frac{N_{HCO_3^-} \times \text{eq.wt.}}{1000}$ x 1000 g/1 = = g/1

Wt. of $CaCO_3$ eqt. = Wt. of HCO_3^- x $\frac{50}{61}$ g/l = x 1000 = ppm

Table: Estimation of CO_3^{2-} & HCO^-_3 Alkalinity in sample water

S.No	Vol. of sample water (ml)	Vol. of N/50 HCI run down (ml)			
		Phenolphthalein end-point, [P]	2[P]	Methyl Orange end-point, [M]	M-2[P]
1	100				
2	100				

CALCULATION:

(i) CO_3^{2-} :

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

$$\text{Wt. of } CO_3^{2-} = \frac{N\ CO_3^{2-} \times \text{eq.wt.}}{1000} \times 1000 \text{ g/l} = \quad \text{g/l}$$

$$\text{Wt. of } CaCO_3 \text{ eqt.} = \text{Wt. of } CO_3^{2-} \times \frac{50 \text{ g/l}}{30} = \quad \times 1000 = \quad \text{ppm}$$

(ii) HCO_3^- :

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

$$\text{Wt. of } HCO^-_3 = \frac{N_{HCO_3^-} \times \text{eq.wt.}}{1000} \times 1000 \text{ g/1} = \quad = \quad \text{g/1}$$

$$\text{Wt. of } CaCO_3 \text{ eqt.} = \text{Wt. of } HCO_3^- \times \frac{50 \text{ g/l}}{61} = \quad \times 1000 = \quad \text{ppm}$$

Signature of Faculty

RESULTS:

I For Test Solution:-

1. [P] = ... ml; [M]=..... ml ; [M-2(P)]=.....ml
2. $N_{CO_3^{2-}}$ =..... 3. Wt. of CO_3^{2-} in mixture = ...gpl
4. Carbonate Alkalinity = ... ppm
5. $N_{HCO_3^-}$ =..... 6. Wt. of HCO^-_3 in mixture=.....gpl
7. Bicarbonate Alkalinity = ...ppm
8. Total Alkalinity in the given test soln.= .....ppm

II For Sample Water:-

1. Source O/W, B/W _____
2. Locality = _____
3. [P] = ... ml; [M]=..... ml ; [M-2(P)]=.....ml
4. Carbonate Alkalinity = ... ppm
5. Bicarbonate Alkalinity = ...ppm
6. Total Alkalinity = ...ppm
7. pH = ... 8. Conductivity = ... ms

Questions :

1. Suggest a better alternative indicator to methyl orange in this experiment.
2. Total alkalinity of a sample water is 860 ppm while temporary hardness is 120 ppm. What is alkalinity due to HCO^-_3 ?

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

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PRECIPITATION TITRATION

EXPERIMENT (11) Estimation of chloride In Water Sample

THEORY:

The titrations accompanied by the formation of sparingly soluble salts, when the solutions of two reacting substances are brought into contact with each other, are called Precipitation titrations.

Chlorides are present in water usually as NaCl, $MgCl_2$ and $CaCl_2$. The concentrations of chlorides over 250ppm impart a peculiar taste to the water, thus rendering the water unpalatable for drinking purposes. Further, existence of unusually high concentrations of chlorides in a water sample indicates pollution from domestic sewage or from industrial waste-waters. Presence of chlorides is also undesirable in boiler-feed water, under high pressure and temperature in boiler, generating hydrochloric acid which causes corrosion in boiler parts.

==========

AIM: Estimation of the amount of chloride present in a water sample by Mohr's method.

REAGENTS:

1. Standard 0.05 N NaCl solution : Weigh 2.925 g of pure NaCl (mol wt. 58.5). Dissolve it in distilled water and make-up to the volume of 1 liter.
2. Silver Nitrate Solution (0.05N) : A neutral $AgNO_3$ solution is prepared by dissolving 8.493 g of pure solid $AgNO_3$ (mol. Wt. = 169.87) in 1 liter of distilled water.
3. Potassium Chromate indicator : ($K_2Cr_2O_4$) Dissolve 5g of $k_2Cr\text{-}_2O_4$ in 100ml of distilled water. Use 1ml of the indicator for each titration.

PRINCIPLE:

Chlorides ions can be determined by titration with standard Silver Nitrate solution using Potassium Chromate as indicator. When silver nitrate solution is added to a solution of sodium chloride containing a few drops of Potassium chromate, white Silver chloride is precipitated out; even a drop of silver nitrate added in excess gives a red precipitate of silver chromate. Thus, the change of color from bright yellow to Brick red color marks the end-point.

$Cl^- + Ag^+ \rightarrow$ AgCl "! (White Precipitate)

$2\,Ag^+ + CrO_4^{-2} \rightarrow Ag_2CrO_4$ "! (Brick Red Precipitate)

Note: The water sample used for chloride determination should be practically neutral or slightly alkaline (pH 7 – 8) since Silver chromate is readily soluble in dilute acids.

Eq wt of NaCl =58.5, Cl^- =35.5

PROCEDURE:

PART – A: PREPARATION OF STANDARD NaCl SOLUTION.

Weigh accurately the given NaCl salt (H" 0.725 g) in a weighing bottle and let its weight be W_1 gm. Transfer the salt into a clean 250ml standard flask using funnel. Take the weight of empty weighing bottle and note down its weight as W_2 gm. Dissolve the salt in minimum quantity of distilled water and make up the solution up to the mark with distilled water, shake the solution thoroughly to get uniform concentration. Calculate the normality of standard NaCl solution.

PART – B: STANDARDISATION OF SILVER NITRATE ($AgNO_3$ Link) SOLUTION.

Pipette out 20ml of standard NaCl solution into a Conical flask. Add 1ml of the $K_2Cr_2O_4$ indicator. Add $AgNO_3$ link solution slowly from the burette, shaking the flask constantly. A white precipitate of AgCl is obtained. After the addition of few ml of $AgNO_3$ a red color appears in the flask but disappears quickly upon shaking. Continue the addition drop by drop till a permanent Brick Red color is obtained. Take twoconcordant readings.

PART – C: ESTIMATION OF CHLORIDES IN GIVEN TEST SAMPLE.

First make-up the given test solution to 100ml in the standard volumetric flask. From this Pipette out 20ml of test solution into a conical flask. Add 1ml of the $K_2Cr_2O_4$ indicator. Add $AgNO_3$ link solution slowly from the burette, shaking the flask constantly. A white precipitate of AgCl is obtained. After the addition of few ml of $AgNO_3$ a red color appears in the flask but disappears quickly upon shaking. Continue the addition drop by drop till a permanent Brick Red color is obtained. Take two concordant readings.

TABULATION AND CALCULATION

EXPERIMENT (11) Estimation of chloride In Water Sample

Expt. No : **Date :**

PART – A: Preparation of standard NaCl solution :

Table 1: Weight of bottle + Substance

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d	s.d
R( )				

Table 2: Weight of empty bottle

Grams	Milligrams			
	100's	10's	Rider (+/-)	
			m.d	s.d
R( )				

Calculations:

Weight of NaCl = $W_1 - W_2$ = _____ gm.

$N_{(Std)\ NaCl}$ = $\frac{Wt}{Eq.Wt} \times \frac{1000}{Vml}$ = _____ N.

PART – B: Standardization of silver nitrate ($AgNO_3$ link) solution.

S.No.	V_{NaCl} (ml)	Burette reading		V_{AgNO3} (ml)
		Initial	Final	
1.				
2.				
3.				
4.				

Calculations:

(Std. NaCl Vs. $AgNO_3$ link)

$$N_{NaCl} \times V_{NaCl} = N_{AgNO3} \times V_{AgNO3}$$

$$N_{AgNO3} = \frac{N_{NaCl} \times V_{NaCl}}{V_{AgNO3}}$$

PART – C: Estimation of chlorides in given test sample.

Table 4: Titration of Test sol (Cl's) Vs $AgNO_3$ (Link)				
S.No.	V_{Test} (ml)	Burette reading		V_{AgNO3} (ml)
		Initial	Final	
1. 2. 3. 4.				

Calculations:

(Test Sol. Vs. $AgNO_3$ link)

$N_{Test} \times V_{Test} = N_{AgNO3} \times V_{AgNO_3} =$

$N_{Test\ Sol} = \dfrac{N_{AgNO3} \times V_{AgNO_3}}{V_{Test}} =$

=

Therefore, the amount of Chlorides (Cl^-) Present in the given test solution

= $N_{Test\ solution}$ **x 35.5 g / lit.**

= _____ **gpl**

= _____ x $\frac{1}{1000}$ mg/L (or) ppm

RESULTS:

1. Weight of NaCl = _____ g.
2. $N_{Std\ NaCl}$ Solution = _____ N.
3. N_{AgNO3} (Link) = _____ N.
4. N_{Cl-} (Test Sol) =' _____ N.
5. Weight of Cl^- in test sol= _____ g/L = _____ mg/L (or) ppm

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

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CHEMICAL KINETICS

Chemical Kinetics is the branch of chemistry, which deals with the study of rates of chemical reactions. The rate of a reaction depends on factors like concentration of reactants, nature of reactants, catalyst etc. In chemical kinetics we conduct the experiment to study the effect of various parameters mentioned on the rate of reaction and elucidate the mechanism of reaction.

Rate of reaction is defined as the change of concentration of any of the reactant or product of reaction in unit time. The rate of reaction, however, decreases as the concentration of reactants decreases. Hence, we consider the rate of reaction at particular instant of time during the course of reaction.

t=o	**a**		**o**
	A	$\longrightarrow$	**Products**
at 't'	**(a-x)**		**x**

Rate of reaction $= -\frac{dx}{dt}$

dx, the small magnitude of change in concentraction of reactant in a small interval of time dt. The negative sign indicates decrease in concentration of reactant.

In Chemical Kinetics the terms "Order of reaction" and " Molecularity of reaction" are important.

For example, if the Rate equation is given by $Rate = k\,[A]^m\,[B]^n$

The order of reaction is the sum of powers of concentration terms that appear in rate equation (m+n). It can also be given as the number of molecules of reactants whose concentration changes during the reaction. The reactions are classified as first, second or third order when the concentration of only one molecule, two molecules or, three molecules change during the reaction. The sum of individual order of all reactants gives the overall order of given reaction. Molecularity of a reaction is defined as "The number of molecules taking part in the rate determining step (slowest step) of a reaction.

The rates of reactions that are too fast (ionic reactions) and too slow (corrosion) cannot be measured hence only reactions with moderate speed are discussed here.

* I have no special talents. I am only passionately curious
- Einstein.

EXPERIMENT (12) :

KINETICS OF FIRST ORDER REACTION

HYDROLYSIS OF METHYL ACETATE IN ACIDIC MEDIUM.

Aim : To study the kinetics of hydrolysis of Methyl acetate in presence of hydrochloric acid at room temperature.

Apparatus : Thermostat, Stop watch, Hot water bath, Burette, Pipette, Beaker, Crushed Ice.

Chemicals : dilute HCl, Methyl acetate, NaOH Solution, Phenolphthalein indicator.

Theory : Methyl acetate an ester undergoes hydrolysis in acidic medium as follows :

$$CH_3COOCH_3 + H_2O \xrightarrow{H^+} CH_3COOH + CH_3OH.$$

Since this reaction takes place in presence of large excess of water, its rate depends only on the concentration of ester. So it is a first order reaction.

Rate = k [ester]^1

Let (i) 'a' moles litre^{-1} is the initial conc. of methyl acetate.

(ii) After a time 't', 'x' mole litre^{-1} of ester have undergone hydrolysis.

(iii) At the given instant of time 't', the conc. of ester will be (a-x) mole. litre^{-1}.

Mathematically the rate of expression is now written as

$$-\frac{dx}{dt} \propto (a - x)$$

$$-\frac{dx}{dt} = k\,(a-x)\ ;\ k = \text{rate constant.}$$

$$-\frac{1}{a - x}.dx = k.dt$$

On Integrating the equation between the limit t = 0, t=t and x=0, x=x, we obtain

$$k = \frac{2.303}{t} \log \frac{a}{a - x}$$

The progress of the reaction is followed by estimating the acetic acid formed (one of the products) through titration of reaction mixture against a standard NaOH solution at different intervals of time.

Procedure :

1. 50 ml of 1N Hydrochloric acid is taken in a clean 250 ml conical flask. About 10 ml of methyl acetate is taken in a test tube. Both flask and test tube containing acid and ester are kept in thermostat maintained at room temperature for 15 minutes.

2. Now, 5ml of methyl acetate is pipetted out, poured quickly in the flask containing HCl and immediately the stop watch is started.

3. The reaction mixture is shaken well, and 2 ml of reaction mixture is transferred as quickly as possible into a conical flask containing about 25 ml Ice cold water. The ice chilled water arrests reaction instantaneously.

4. The solution is titrated quickly against a standard (0.1M) Sodium Hydroxide solution, using phenolphthalein indicator. The volume of causitc soda (NaOH) consumed is noted in the tabular form.

5. The process is repeated at every ten minutes to get about 6 more readings.

6. The contents of the flask are heated to about 60^0C on a hot water bath for about 15-20 minutes,to make the completion of reaction. The contents of flask are cooled to room temperature, 2 ml of this solution is pipetted out in a conical flask 5 drops of phenolphthalein indicator is added and titrated against standard NaOH solution.

S. No	Time in Minutes (t)	Volume of NaOH consumed (ml)	$\frac{V_\infty - V_0}{V_\infty - V_t}$	$\log \frac{V_\infty - V_0}{V_\infty - V_t}$	$\log (V_\infty - V_t)$	$k = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$
1	0					
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					
8	α					

V_o = Volume of NaOH consumed at t = 0 .....

V_t = Volume of NaOH consumed at a given time 't' .....

V_∞ = Volume of NaOH consumed when the reaction in completed.....

V_o is proportional to the amount of HCl present in 2 ml of reaction mixture at t = 0

V_∞ is proportional to the amount of HCl and CH_3COOH produced from the ester after completion of reaction. Thus V_∞ - Vo is proportional to the amount of CH_3COOH formed at ~ time.which in turn is proportional to initial concentration of ester $V_\infty - V_o = a$

Vt is proportional to the amount HCl and CH_3COOH produced from the ester at time t and thus $(V_t - V_o)$ is proportional to the amount of CH_3COOH formed at time t, which in turn is proportional to number of moles of ester hydrolysed, $(V_t - V_o) = x$

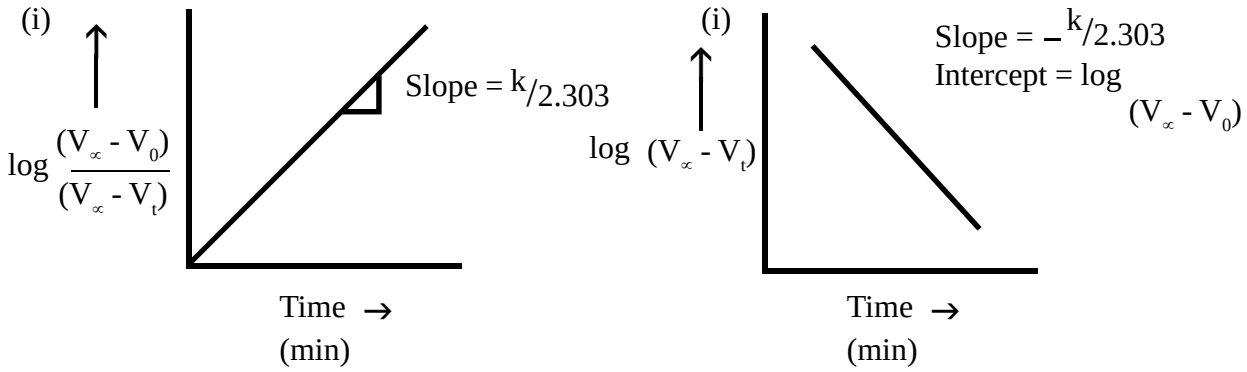
Thus $(V_\infty - V_o) - (V_t - V_o) = (V_\infty - V_t)$ would be proportional to (a-x), the number of moles of ester remaining.

The k, first order rate constant.

$$k = \frac{2.303}{t} \log \frac{a}{a - x} \quad \text{which in turns becomes} \quad k = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$$

The value of 'k' is calculated for different values of 't' from the tabulated data.

The value of 'k' can also be obtained graphically as follow :



Result : (i) The plot of $\log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$ Vs time gives a straight line passing through the origin.

Hence hydrolysis of ester in acidic medium is found to follow first order kinetics.

(ii) The rate constant 'k' reaction is .....min^{-1}.

Questions :

1. What is the difference between order and molecularity ?
2. How is the molecularity of a reaction determined ?
3. Can we study kinetics of redox reaction like $KMn0_4$ vs Mohr's salt ?

* Curiosity is a willing, a proud, an eager confession of ignorance.
I keep six honest serving-men,
They taught me all I knew,
Their names are what & why & when
And How & where & who

- Rudyard Kipling.

EXPERIMENT (12) : KINETICS OF FIRST ORDER REACTION

Expt. No. : **Date :**

CALCULATIONS : $k = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$

1.

2.

3.

4.

5.

Result : (i) The plot of log $\frac{(V_\infty - V_0)}{(V_\infty - V_t)}$ Vs time gives a straight line passing through the origin. Hence hydrolysis of ester in acidic medium is found to follow first order kinetics.

(ii) The rate constant 'k' reaction is .....min^{-1}. (from calculation)

(iii) The rate constant 'k' reaction is .....min^{-1}. (from garph)

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

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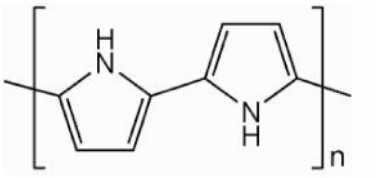
PREPARATION OF POLYMERS

EXPERIMENT (13-A) Preparation of Polypyrrole (a Conducting polymer)

Introduction: **Polypyrrole** (**PPy**) is a type of organic polymer formed by polymerization of pyrrole. Polypyrroles are conducting polymers, related members being polythiophene, polyaniline, and polyacetylene. The Nobel Prize in Chemistry was awarded in 2000 for work on conductive polymers including polypyrrole.
Structure of Polypyrrole:



Preparation of Polypyrrole:

REAGENTS:

1) 10ml of pyrrole dissolved in 25ml of CCl_4 (carbon tetra chloride)
2) 10% $FeCl_3$ (Ferric chloride) solution (Covered with watch glass)
3) Conc. HCl (To Increase the conductivity and as a doping agent).

PROCEDURE:

Dissolve 10 ml of pyrrole in 25 ml of CCl_4. Then add 10% of acidified $FeCl_3$ of aqueous solution drop by drop with stirring in a beaker . A black coloured polypyrrole polymer is formed at the junction of both the layers.When few drops of conc.HCl is added, it increases the conductivity by acting as doping agent.

Properties

Polypyrrole conducts electricity due to presence of conjugation in its structure. Films of PPy are yellow but darken in air due to some oxidation. Doping makes the materials brittle. They are stable in air up to 150 °C .PPy is an insulator, but its oxidized derivatives are good electrical conductors. The conductivity of the material depends on the conditions and reagents used in the oxidation. Conductivities range from 2 to 100 S/cm.

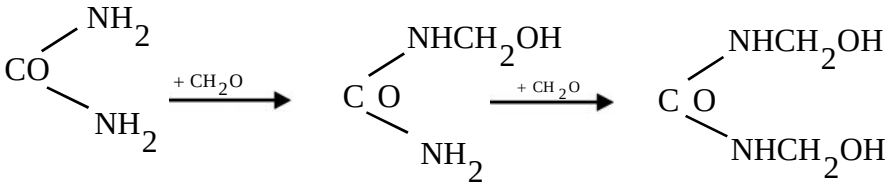
Applications

Polypyrrole and related conductive polymers have two main applications in electronic devices and for chemical sensors. PPy is also potential vehicle for drug delivery. The polymer matrix serves as a container for proteins.

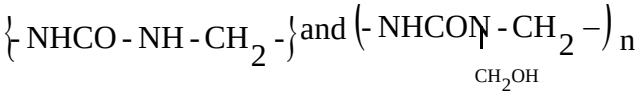
Preparation of Polymers

Experiment (13-B): Condensation of Urea with Formaldehyde

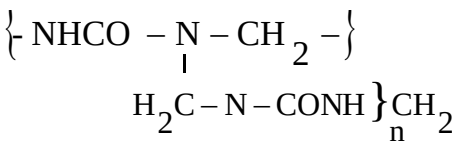
Theory: The character and rate of the reaction between urea and formaldehyde in aqueous solution depend on the reaction conditions. In the presence of alkalis and excess formaldehyde, urea gives predominantly monomethylol and dimethylol urea:



Ammonia also acts as alkaline catalyst. Methylol ureas containing hydroxyl groups are soluble in water to give viscous solutions. Heating of such solutions condenses the molecules of methylol ureas with elimination of water and formation of linear macromolecules having the structural units:

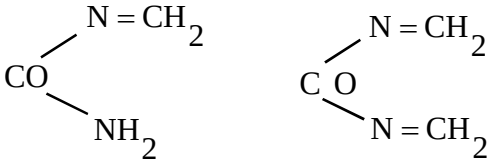


Next formed are more complicate crosss - linked macromolecules with methylene links:



or cyclic molecules, presumably with 6 - and 8- membered cycles. Because of their enlarged and complicated molecules and the absence of hydroxyl groups, these polymers are insoluble in water. In industry such a deepened condensation is attained by heating the dry product with a filler (for instance wood flour) in a mould under pressure. Linear polymers soften and then set again to convert into thermosetting colourless or white urea-formaldehyde resins (carbamide or amino resins). Similar conversation in our experiment can also be attained with acid catalysts. The hardness of the end products depends on the intensity of heating of the reaction mixture.

If the reaction between urea and formaldehyde proceeds from the very beginning in the presence of an acid catalyst (for instance, if formaldehyde is not neutralized), the first reaction products, even in dilute solutions, are predominantly water - insoluble methylene ureas:



that are polymerized immediately. The obtained product does not contain hydroxyl groups, has very large macromolecules and is insoluble in water.

In the absence of catalysts, urea reacts with formaldehyde in dilute solutions (Test C) very slowly; the reaction in concentrated solution (Test B) is faster and liberates much heat that accounts for boiling of the mixture even when the source of external heat is removed. The condensation proceeds simultaneously in several directions but the products insoluble in water are formed slowly.

Reagents: 1. Urea 2. Formaldehyde (35 - 40%) - Neutral Solution

3. Oxalic Acid (Saturated Solution)

A. Place 2 g of urea, 8 ml of formaldehyde, 1 ml of conc. ammonia solution and a boiling stone into a test tube and fix it in the stand in the inclined position (in the HOOD). Heat the test tube over a burner and boil its contents fo 10 - 15 minutes to reduce the liquid volume by 1/3. Discontinue heating, allow the thickened liquid to cool slightly and transfer half of the solution into another test tube. Add 2 - 3 drops of oxalic acid into the second test tube.

Mix well the contents in the both test tubes by shaking and keep them on a water bath (50 - 60^{o}C) for 5 - 10 minutes. During this time the contents of one test tube usually vitrifies into a transparent or white mass. cool the test tubes add 3 - 4 ml of water and compare the solubility of the products at room temperature and with heating.

The insoluble vitreous product of condensation can be removed from the test tube by breaking the the latter.

B. Carefully heat a mixture of 2 g of urea and 3 ml of formaldehyde in a test tube over a burner to dissolve urea. Transfer part of the solution to other test tubes.One of them should contain a drop of conc. HCl and the other 2 - 3 drops of oxalic acid solution. Observe the changes. Heat the remaining part of the solution until it start boiling, and also note the changes in the appearance of the mixture.

C. Place 1 g of urea, 1.5 ml of formaldehyde and 10 ml of water in test tube and shake the mixture to dissolve urea. Transfer half of the obtained solution into another test tube, add 0.5 ml of Conc. HCl, shake and keep both test tubes on a hot water bath for a few minutes. Note the different behaviour of the test tube contents.

Test the obtained white product for solubility in boiling water.

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VOLUME - II

INSTRUMENTAL CHEMICAL ANALYSIS

INSTRUMENTAL CHEMICAL ANALYSIS

Introduction :

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

Advantages of instrumental analysis :

- Low concentartion of sample required
- Highly sensitive and selective
- Reliable measurements.
- Fast determination
- Indicators are not required

Method	Instrument	Phenomenon underlying the method	Quantity measured
Optical methods			
1. Colorimetry 2. SpectroPhoto-metry	1. Colorimeter 2. Spectrophoto-meter	Interaction of matter with electromagnetic radiation	Absorbance or Trasmittance
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 the flow/movement of the ions followed by their electrode reaction at the anode and cathode respectively. This amounts to indirect transfer of 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 the 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 welded to a plantium 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.

$$x = \text{Cell constant} = \frac{\text{Distance between the electrodes}}{\text{Cross - sectional area of elecrode}} = \frac{\ell}{a}$$

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

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 specfic conductivity of an electrolyte is expressed in $ohm^{-1}\ cm^{-1}$, mho cm^{-1}, $S.cm^{-1}$, $S.m^{-1}$

Measuring ℓ and **a** of a cell for determining the cell constant is very inconvenient, and therefore the cell constant is usually determined by measuring the conductance of a solution whose specific conductance is exactly known. The solution used for this purpose is potassium chloride.

Cell Constant = Specific conductance of KCl x Measured Resistance of KCl

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

* The important thing is not to stop questioning.

- Einstein.

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 gradient of 1 Volt cm^{-1}. But the ionic mobilities of H^+ and OH^- are abnormally high. ($H^+ = 36.8 \times 10^{-4}$, $OH^- = 20.5 \times 10^{-4}$ cm^2 sec^{-1} $Volt^{-1}$). Thus the conductance of an electroyte is quite sensitive to the concentration and ionic mobilities in acid-base titrations of H^+ and OH^- ions.

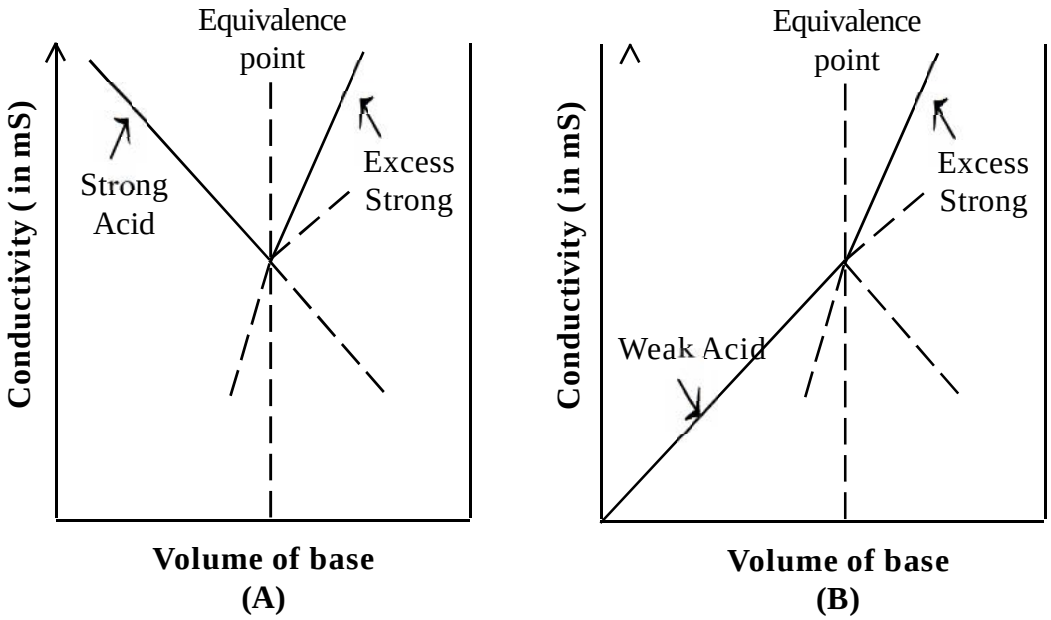
In the conductometric acid-base titrations the course of the titration as base is added to the acid or acid to the base, is followed from the gradual change in the conductivity of the titrand.

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

In 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.

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

In solution water is little ionised 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 the equivalence 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 (B) Weak acid Vs Strong base.



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 intrapolated lines is the equivalence point of the titration.

2. Titration of Weak Acid (CH_3COOH) 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 ionised. As NaOH is added highly ionised sodium acetate is formed and effectively the net result of the titration is the replacement of unionised CH_3COOH molecules by the highly ionised sodium acetate.Therefore there will be a gradual increase in the conductance.

$$CH_3COOH + Na^+ + OH^- \rightleftharpoons CH_3COO^- + Na^+ + H_2O$$
$$(aq) \quad (aq) \quad (aq) \qquad (aq) \quad (aq)$$

After the equivalence point, any further addition of NaOH will produce a more rapid increase in conductance due to the 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.

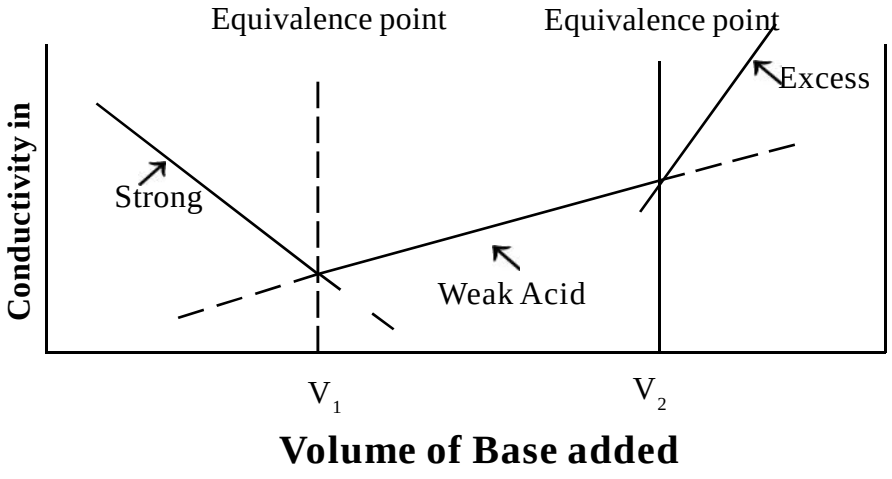
3. Titration of a mixture of strong and weak acids

(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 titration of HCl Vs NaOH and the second one for the end point of HCl + CH_3COOH Vs NaOH.

V_1 = Volume of N aOH required for neutralisation of HCl.

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



EXPERIMENTS (14, 15, 16 and 17) :

A. Preparation of Std. Oxalic Acid Solution :

Weigh about 0.63 gr. 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.

B. 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 poisition (pressed).

C. Estimaiton 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 HCI + 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 are obtained.

Application of conductometric measurements of aqueous and non aqueous solutions :

Used in cooling towers, reverse osmosis, steam boilers, acid, salt and alkali concentrations, laboratory analysis, fruit peeling, salinity, chemical streams etc.

Industries which use conductance :-

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

Questions :

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 acid?
7. Is it possible to determine the volume ratio of strong and weak acids in a mixture?

Expt(14). Conductometric Titration of Strong Acid Vs Strong Base

Date :

Expt. No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

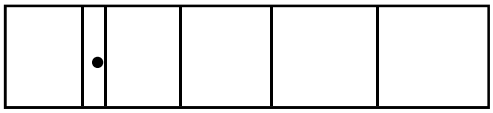
Table.1. Weight of bottle +Oxalic Acid  g.

Table.2. Weight of empty weighing bottle  g.

Wt. of Oxalic Acid = $W_1 - W_2$ =

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

(V_{ml} = 100 ml)

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	

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

Part - C Estimation of HCl in the test solution :

Table : Conductometric titration of HCl x NaOH

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

$$N_{HC1} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HC1}}$$

=

V_{NaOH} = Inflection point from the graph,

$$Wt_{HC1} = N_{HC1} \times 36.5$$

=

Results:

1. Weight of Oxalic Acid = _____g
2. $N_{\text{std. Oxalic acid}}$ = _____
3. N_{NaOH} (Link) = _____
4. V_{NaOH} end point (from graph) = _____ (ml)
5. N_{HCl} (Test) = _____
6. Weight of H Cl in test solution = _____ gpl

Expt(15). 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 (ms)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

$$N_{CH_3COOH} = \frac{N_{NaOH} \times V_{NaOH}}{V_{CH_3COOH}}$$

=

V_{NaOH} = Inflection point from the graph,

$Wt_{CH_3COOH} = N_{CH_3COOH} \times 60$ gm/lit

=

Marks	
Observations and calculations (20) :	_____
Results & Graphs (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_{CH_3COOH} (Test) = _____
6. Weight of CH_3COOH in test solution= _____ gpl

Signature of Faculty

Expt (16). 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 ☐☐.☐☐☐☐ g.

Table.2. Weight of empty weighing bottle ☐☐.☐☐☐☐ g.

Wt. of Oxalic Acid= W_1-W_2

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

(V_{ml} = 100 ml)

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	

Marks

Observations and calculations (20) : _____

Results & Graphs (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 (ms)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

$$N_{HC1} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

=

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

$$Wt_{HC1} = N_{HC1} \times 36.5$$

=

$$N_{CH_3COOH} = \frac{N_{NaOH} \times V_{NaOH}}{V_{CH_3COOH}}$$

=

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

$$Wt_{CH_3COOH} = N_{CH_3COOH} \times 60 \text{ gm/lit}$$

=

Results:

1. Weight of Oxalic Acid = _____g
2. $N_{\text{std. Oxalic acid}}$ = _____
3. N_{NaOH} (Link) = _____
4. V NaOH@ end point (from graph) for HCl = _____ (ml)
5. N HCl (Test) = _____
6. Weight of HCl in test solution = _____ gpl
7. V NaOH@ end point (from graph) for CH_3COOH = _____ (ml)
8. N_{CH_3COOH} (Test) = _____
9. Weight of CH_3COOH in test solution = _____ gpl

Signature of Faculty

EXPERIMENT(17) CONDUCTOMETRIC PRECIPITATION TITRATION

AIM: To determine the amount of $BaCl_2$ present in the given solution by Conductometric titration.

Theory:-

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

In the precipitation titration, the ions are converted to insoluble precipitate, which will not contribute in the conductance. When Na_2SO_4 is added slowly from the burette to the solution of $BaCl_2$, Barium ions are substituted by relatively slow moving Na^+ ions. This decreases the conductance gradually and Barium sulphate ($BaSO_4$) gets precipitated while chloride ions are liberated, as shown in the equation;

$$[Ba^{+2} + 2Cl^-] + [2Na^+ + SO_4^{-2}] \rightarrow BaSO_4 \downarrow + 2Na^+ + 2Cl^-$$

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

Eq wts :- $BaCl_2$ =208.23/2=104.1 , Na_2SO_4 = 142.04/2 = 71.02

PROCEDURE:

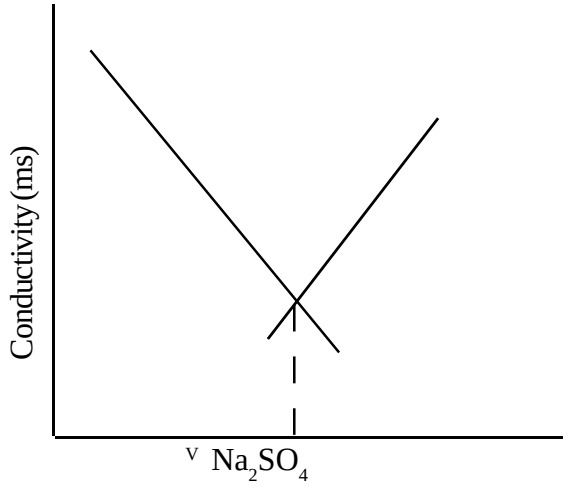
PART – A: PREPARATION OF STANDARD (0.2N) Na_2SO_4 SOLUTION:

Weigh accurately about 1.42g of the given amount of Na_2SO_4 into a 100ml standard flask, dissolve in small quantity of distilled water and makeup using distilled water, and shake for uniform concentration.

PART – B: ESTIMATION OF $BaCl_2$:

Make up the given $BaCl_2$ solution to 100ml in a standard flask using distilled water. Pipette out 40ml of the solution into a clean 100ml beaker. Immerse a conductivity cell and note down the conductance of the solution , after calibrating the conductivity meter. Fill the burette with Na_2SO_4. Add Na_2SO_4 at intervals of 0.5ml, stir the solution well and measure the conductance of the solution on every addition. The conductance of the solution decreases gradually upto the end point and increases sharply later. The conductometric titration is continued for further 4-5 ml after getting the end point. Conductance is plotted against the volume of Na_2SO_4 added. The point of intersection gives the end point.

Graph :- $^V Na_2SO_4$ **Vs conductivity (ms)**



MJCET

TABULATION AND CALCULATION

PART – A: PREPARATION OF STANDARD (0.2N) Na_2SO_4 SOLUTION:

Weight of weighing bottle + Na_2SO_4 (W_1) = _____

Weight of empty weighing bottle (W_2) = _____

Therefore, Weight of salt Na_2SO_4 ($W_1 - W_2$) = _____

And $^{N}Na_2SO_4$ = $\frac{Wt}{Eq.Wt} \times \frac{1000}{V_{ml}}$ _____

PART – B: ESTIMATION OF $BaCl_2$: = ----------

S.No.	Volume of Na_2SO_4 (ml)	Conductance in ms
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

Calculations:

$$^{N}BaCl_2 = \frac{^{N}Na_2SO_4 \text{ x } ^{V}Na_2SO_4}{^{V}BaCl_2}$$

Weight of $BaCl_2$ = N_{BaCl2} x eq wt of $BaCl_2$ = _____ g/l

RESULTS:

1) Wt of Na_2SO_4 = _____g
2) $^{N}Na_2SO_4$ = _____
3) $^{V}Na_2SO_4$ at end point from the graph = -_____ml
4) $^{N}Ba_2Cl_2$ =_____
5) The weight of of $BaCl_2$ present in 1 lit of solution =_____g/l

Marks

Observations and calculations (20) : _____

Results & Graphs (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

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POTENTIOMETRY

THEORY :

In potentiometry, cell potential measurements 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, a 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 (CH_3COOH) x Strong Base (NaOH)

iii) Mixture of Acids (HCl+ CH_3COOH) 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.

EXPERIMENT - 18

POTENTIOMETRY REDOX TITRATION

Estimation of Fe^{2+} Vs $K_2Cr_2O_7$.

Aim : To titrate potentiometrically Fe^{2+} against standard $K_2Cr_2O_7$.

Theory : The **reference electrode** used here is saturated calomel electrode (SCE). It consists of mercury metal covered with a paste of Hg + Hg2 Cl2 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 develops a 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 :

$$(-)\ Pt \mid Hg_{(l)}, Hg_2Cl_{2\,(s)} \mid KCl_{(Sat)} \parallel Fe^{3+}, Fe^{2+} \mid Pt\ (+)$$

Cell Reaction :

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

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

Cell E.m.f. :

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

* We should not only use the brains we have, but all that we can borrow.

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 dichromate (~N/40) solution .

PROCEDURE :

PART A: Preparation of Std. F.A.S. Solution.

Weigh accurately to about 0.98 gr. of Mohr salt (F.A.S.), transfer it in a clean 100 ml standard flask through funnel. Dissolve it in distilled water, add ~ 1/2 Test tube (~ 10 ml) of dil H_2SO_4 and make the solution up to the mark. Make it Homogeneous. From the weight of FAS, Calculate the Normality of Standard solution.

PART - B : Standardization of $K_2Cr_2O_7$ solution.

20ml of the prepared solution (standard FAS) is pippetted out into a clean conical flask. Approximately (1/2 Test Tube) 10 ml of dilute H_2SO_4 (6N) is added to it, and then 3 to 5 drops of internal indicator diphenylamine and about 8ml (1/3 T. T) of Syrupy Phosphoric acid ..... is added which lowers the reduction potential of Iron couple and facilitates the initial oxidation of Ferrons to Ferric. It is titrated with $K_2Cr_2O_7$ (taken in Burette) very slowly until the solution acquires a bluish green tint which shows that the end point is near. The titration is continued now with dropwise addition of $K_2Cr_2O_7$ and good stirring until the color of solution changes from green to parmanenet blue which is stable.

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

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

1. Make the given test solution of Fe^{+2}, upto 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 soln in a clean 100 ml beaker, place the platinum electrode in the soln, 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

$$\textbf{(-)}\ Pt \left| Hg_{(l)}, Hg_2Cl_{2(s)} \right| KCl_{(Sat.)} \Big\| Fe^{+3}, Fe^{+2} \Big| Pt\ \textbf{(+)}$$

6. Switch on the Instrument. Keep the mV current RED switch in Pressed position (mV mode). The display reads EMF of cell in millivolts. Adjust the initial EMF to a preset value with right side knob.
7. Add $K_2Cr_2O_7$ 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 inflexion in EMF occurs, then take about 6 to 8 readings after inflexion in 1 ml INTERVALS.
9. From the titration approximate volume of $K_2Cr_2O_7$ required is found out.
10. The titration is repeated with addition of $K_2Cr_2O_7$ 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 $K_2Cr_2O_7$ added; the Inflexion point gives an approximate equivalence point.

12. Differential graph is drawn by plotting $\frac{\Delta E}{\Delta V}$ (Y - axis) Vs $V_{K_2Cr_2O_7}$ (X - axis) to get a sharp peak, which corresponds to the precise equivalence point of titration.

TABULATION

Table : 1

S.No.	Vol. of $K_2Cr_2O_7$ added (ml)	Ecell (mv)
1.		
2.		
3.		

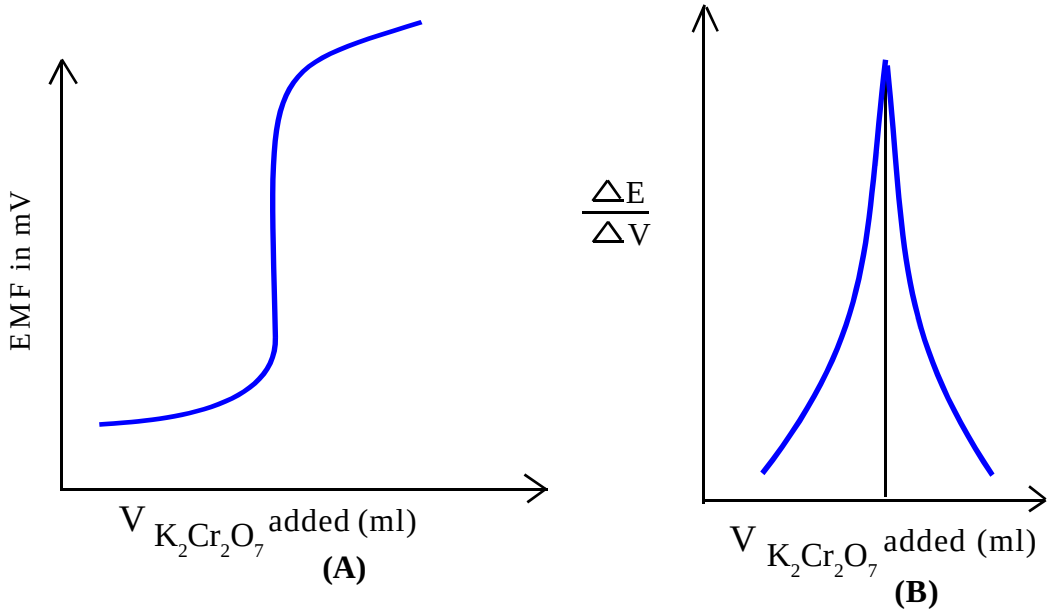
[Draw 17 lines in full page]

Table : 2

S.No.	$V_{K_2Cr_2O_7}$ (ml)	Ecell (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				

[Draw 25 lines in full page]

GRAPHS:



EXP. No. (18) POTENTIOMETRY REDOX TITRATION

Date :

EXP. NO. :

TABULATION AND CALCULATIONS

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

Table.1. Weight of bottle + FAS ☐☐.☐☐☐☐☐ g.

Table.2. Weight of empty weighing bottle ☐☐.☐☐☐☐☐ g.

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

$$N_{(std)}\ F.A.S = \frac{Wt}{Eq.Wt} \times \frac{1000}{Vml} = ...$$

(V=100 ml)

Part B : Standardisation of $K_2Cr_2O_7$:

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

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

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

POTENTIOMETRY REDOX TITRATION (Fe^{+2} Vs. $K_2Cr_2O_7$)

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

Table : 1

S.No.	Vol. of $K_2Cr_2O_7$ 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.	$V_{K_2Cr_2O_7}$ (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 :

$$N_{Fe^{2+}} = \frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{Fe^{2+}}} =$$

($V_{K_2Cr_2O_7}$ = Peak from differential graph - B)

Wt of Fe^{2+} = $N_{Fe^{2+}}$ x 56 = gpl

Results:

1. Weight of FAS = _____g
2. $N_{\text{std. FAS}}$ = _____
3. $N_{K_2Cr_2O_7}$ (Link) = _____
4. V $K_2Cr_2O_7$@ end point (from graph)= _____ (ml)
5. $N_{Fe^{2+}}$ (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 addtion of $K_2Cr_2O_7$ 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.

EXPERIMENT - 19

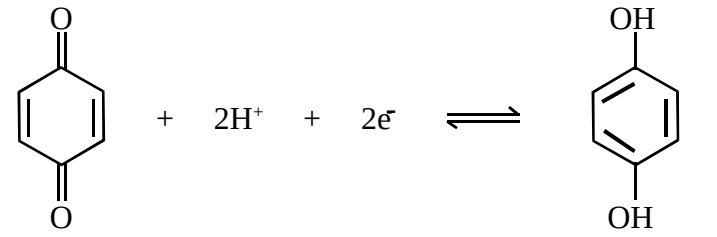
POTENTIOMETRY ACID-BASE TITRATION

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

Aim : To titrate potentiometrically HCl against standard NaOH.

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

The indicator electrode is 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

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

Where E^{o}_{Q/QH_2} (SRP of Quinhydrone electrode) is 0.6996V at 25^0C. 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).

Cell Representation : -

(-) Pt | $Hg_{(l)}$, $Hg_2Cl_{2\,(s)}$ | $KCl_{(Sat)}$ || H^+ [Unknown] | Q, QH_2 | $Pt^{(+)}$

Cell Reaction: -

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

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

Cell E.m.f. :-

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

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

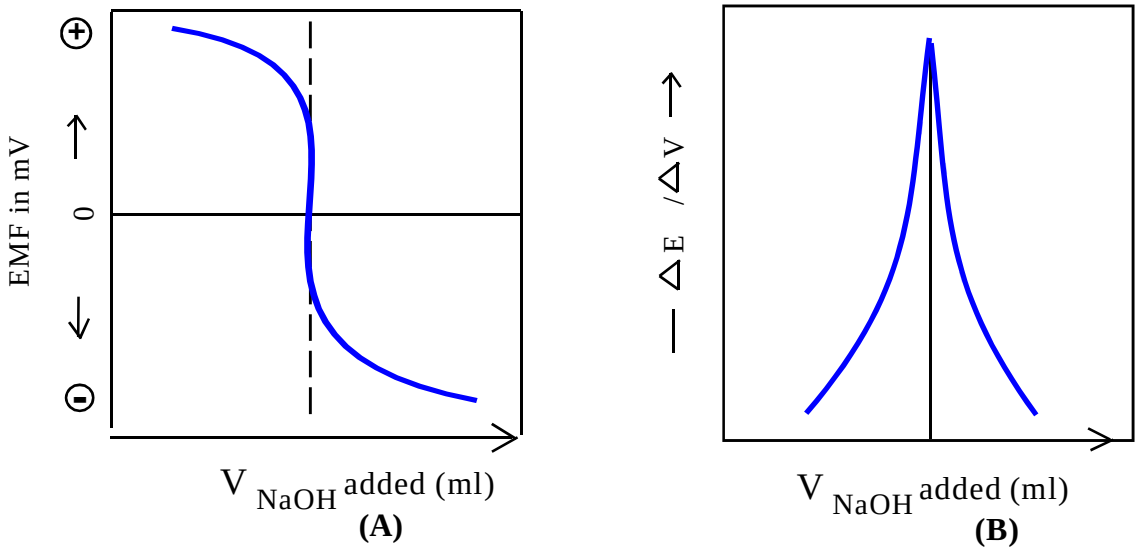
$$E_{cell} = 0.6996 - 0.0591pH - 0.242$$

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

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. Ecell values are noted down, graph is plotted , **end point** is located and concentration is calculated.

Potentiometric Acid - Base Titrations: Graphs



EXPERIMENT *: 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/20)

3) Quinhydrone

Procedure :

Part A : Preparation of standard Oxalic acid solution

Weigh about 0.63 gr. 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 up to 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 soln. 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.

   (-) $Pt \mid Hg(l), Hg_2Cl_2(s) \mid KCl\ (sat.) \parallel H^+\ (unknown) \mid Q, QH_2 \mid Pt$ **(+)**

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 preset 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) vs V_{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 soluiton ?
2. Why cell potential decreases with addition of alkali to the test acid solution ?
3. What is the use of quinhydrone in this experiment ?

Exp. (19) POTENTIOMETRY ACID-BASE TITRATION

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1. Weight of bottle + Oxalic Acid ____.____ g.

Table.2. Weight of empty weighing bottle ____.____ g.

Wt. of Oxalic Acid= W_1-W_2

$$N_{(std)}\text{Oxalic acid} = -\frac{Wt}{Eq.Wt} \times \frac{1000}{Vml} = ...$$

(V_{ml} = 100 ml)

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	

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

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.				
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21.				
22.				
23.				
24.				
25				

Calculations :

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

(V_{NaOH} = Peak from diff. 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) : _____

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pH Metry

Glass Electrode ----- pH Meter

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, it 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	$Hg_{(l)}$, $Hg_2Cl_{2\,(s)}$	Sat. KCl	Test soln of Unknown pH	Glass membrane	0.1N HCl	AgCl, Ag	Pt (+)

Cell Reaction : -

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

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

$E_{Glass} = E^o_G - 0.0591\ pH$ $\quad$ E_G^o = SRP of Glass electrode

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

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

$\left(P^H = -\log [H+] \right)$

pH Meter : The glass membrane of the glass electrode offers a high resistance in measuring circuit of the potentiometer 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 = 1 to 14, Acadic = 1to 6, Neutral = 7, Basic = 8 to 14

ACID - BASE pH - METRIC TITRATION

THEORY :

1) Titration of a 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.

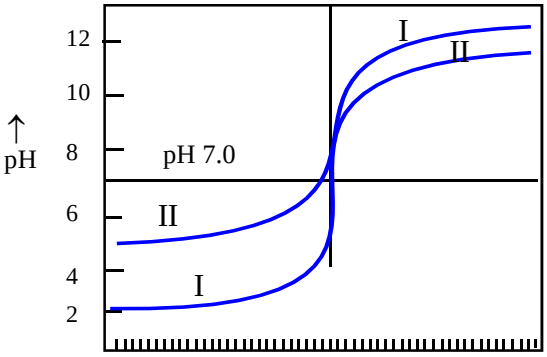
Plot of $\frac{\Delta pH}{\Delta V}$ Vs V in the vicinity of the equivalence point (V=0. 1 ml) will give

a curve whose peak indicates a sharp end point.

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

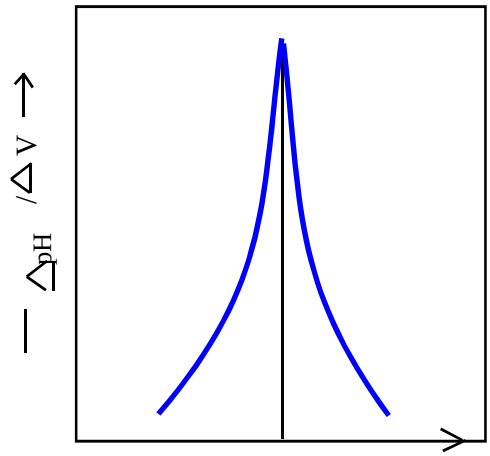
Here the initial concentration of H_3O^+ ions in acid solution will be low, hence the

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 the 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 anionic (CH_3COO^-) hydrolysis of the salt formed, will make the pH of the reaction mixture higher than 7 at the equivalence point.



Neutralization of I strong acid and strong base

II Weak acid and strong base



□ □□□□

—

* It's not that I'm so smart, it's just that I stay with problems longer
- Einstein

Experiment : (20) Titration of strong acid vs. strong base.

Requirements : pH meter, glass electrode, burette, pipette, beakers.

Chemicals : HCl (N/40) ; NaOH (N/10)

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 0.1 N Solution in burette.Pipette out 20 ml of standard oxalic acid solution in 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°C 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 = 178). Once calibrated, this knob should not be disturbed.
6. Take out the electrode from Buffer soln., wash the electrode with distilled water, 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 100 ml beaker. Dip the electrode in the solution and measure 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 2 ml range). (Table-2)
13. Draw a graph of pH Vs. Volume of NaOH added. The intersection point at pH=7.0 may give a rough end point.
14. Another graph is obtained by plotting $\Delta pH/\Delta V$ (Y-axis) Vs. V_{NaOH} (X-axis) to get a sharp peak, which corresponds to the equivalence point. (Differential Graph)

EXP. (20) pH - METRIC TITRATION (STRONG ACID Vs STRONG BASE)

Expt. No. **Date :**

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1. Weight of bottle + Oxalic acid ☐☐.☐☐☐☐ g.

Table.2. Weight of empty weighing bottle ☐☐.☐☐☐☐ g.

Wt. of Oxalic Acid= W_1-W_2

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

(V_{ml} = 100 ml)

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	

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

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 diff. 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 the salt bridge not used in this experimental set up ?

Results:

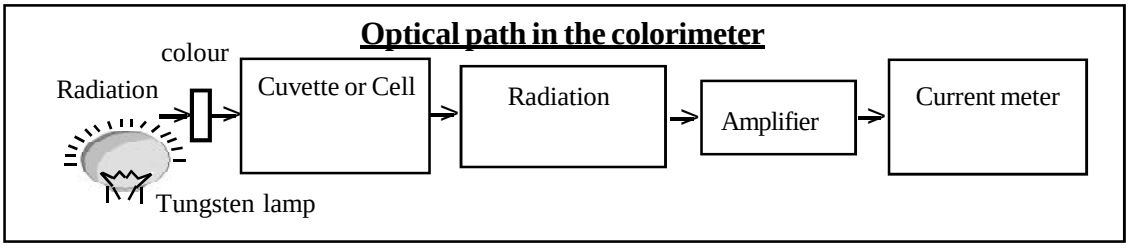
1. Weight of Oxalic Acid = _____g
2. $N_{(std)}$ Oxalic Acid = _____
3. N_{NaOH} (Link) = _____
4. V_{NaOH} @ end point (from graph) = _____ (ml)
5. N_{HCl} (Test) = _____
6. Weight of $_{HCl}$ in test solution = _____ gpl

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

Signature of Faculty

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.



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 white 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 arithmatically. It can be expressed as

$$\frac{-dI}{dc} = KI,$$ on Integration between limits Io—It & C =0— c, gives

$$\ln \frac{Io}{It} = Kc \quad \text{(or)} \qquad I_t = I_o.10^{-kc} \quad (1)$$

Where I_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 $\frac{-dI}{dx} = KI$

Where 'x' is thickness of the medium. On integration between limits Io --- It and x = 0--- x, we get

$$\ln \frac{Io}{It} = Kx \qquad (OR) \qquad I_t = I_o.10^{-kx} \quad - \quad (2)$$

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 the absorbing medium.

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

$$I_t = I_o.10^{-\epsilon c x} \qquad (OR) \qquad \log \frac{I_o}{I_t} = \epsilon cx \qquad - \qquad (3)$$

where Č is molar absorption coefficient or molar absorptivity, or molar extinction coefficient 'c' is expressed in moles / litre, and x in centimetres.

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(ϵ) is given as $T = \frac{It}{Io}$

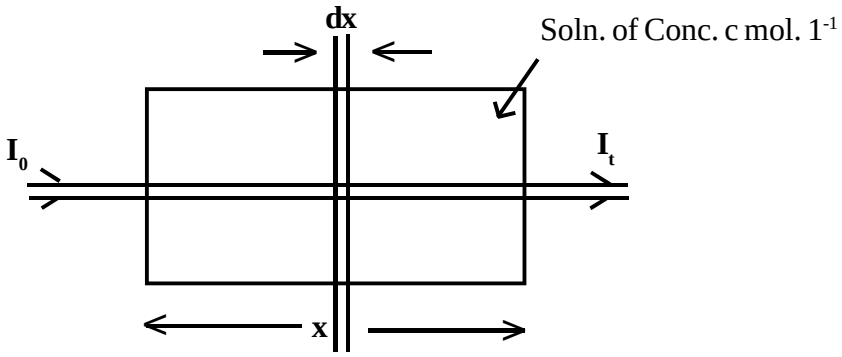
$$A = \epsilon cx = \log \frac{I_o}{I_t} \qquad \text{We Know that } A = \log \frac{1}{T} = -\log T$$

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

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 molecule 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 selected 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 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 species under measurement.

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

iv) 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 3/4 and outside dried with a tissue paper)

v) Adjust the disc A for zero O.D.

vi) Replace Blank in the solution cell with various sample solutions prepared, one after the other (as indicated in the table), after rinsing with the respective solution.

vii) 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 $\hat{I}x$. (select a suitable scale on the graph paper).

EXPT (21) COLORIMETRY : VERIFICATION OF BEER-LAMBERT'S LAW & ESTIMATION OF Mn IN $KMnO_4$

REQUIREMENTS :

Colorimeter, set of colour filters, burettes, standand volumetric flasks, graduated 5 ml and 10 ml pipettes. Test Tubes, Cuvettes, Test Tube stand.etc

CHEMICALS (REAGENTS) :

(i) A stock solution of $KMnO_4$ ~ N/40 = ~ M/200 ($\because$ M = N/5)

(ii) Mohr's Salt [Fe SO_4. $(NH_4)_2$ SO_4. $6H_20$]

PROCEDURE :

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

Weigh accurately about 0.98 gm of Mohr's salt and transfer it into a clean 100 ml flask using funnel. Dissolve it in distilled water, add ~ 10 ml dil H_2 SO_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. Pipette out 20 ml of Mohr salt soln. in a clean conical flask. Add 3/4 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.

Tilrate it with $KMnO_4$ stock solution,

Convert the concentration from Normality to Molarity.

$$\text{Let } N_{KMnO_4} = 0.024, \text{ then its } M_{KMnO_4} = \frac{0.024}{5} = 0.0048 \text{ M}$$

10ml of link $KMnO_4$ is taken in standard volumetric flask and diluted to 100 ml . we get **stock** solution having

$$M_{KMnO_4} = 0.00048 = 4.8 \times 10^{-4} \text{ M}$$

From this stock solution the following samples solutions can be prepared as indicated in the following Table-2 (Page No. 114) and their optical density (O.D.) recorded using the colorimeter, and a suitable colour filter.

Selection of Filter No for $KMnO_4$ solution :

The cuvvete containing stock $KMnO_4$ solution is placed (sockets) and the optical density (O . D) is noted , for a givven filter no. 45 the process is repeated i.e the o.d of stock $KMnO_4$ solution is noted for each of the other seven filters as given in table-1 (Page no . 114) . The instrument should be set to zero o.d using distilled water for each of the filters . The filter no. which gives maximum o.d is selected for taking the readings (O.D) of the ten sample solutions prepared

VERIFICATION OF BEER-LAMBERT'S LAW :

The filter no for $KMnO_4$ solution is fixed the cuvvete is filled with distilled water and o.d is set to zero the cuvvete is filled with each of the ten sample solutions is prepared one after the other , After with rinsing with respective solution the o.d is recorded for each of the sample solution .

Calibration Graph :

A graph is ploted 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 .

Experiment (22) : Estimaiton of $KMnO_4$ in test Solution

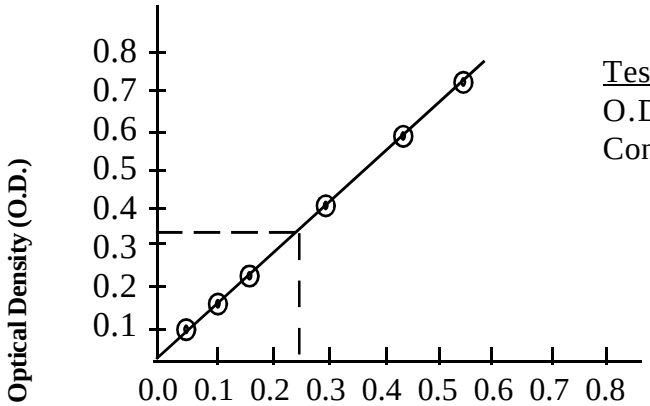
The given Test solution ($KMnO_4$ in this expt.) is taken in the rinsed cuvette.Wipe the outside of the cuvette dry with a tissue paper. Insert the cuvette in the Colorimeter.

Record the optical density of the test solution on the colorimeter and read the corresponding concentration on the calibration graph (obtained in the above).

(As an example a calibration graph prepared is shown below with; 1 cm on x axis (concentration) = 0.5 x 10^{-4} M and 1 cm on y axis (O.D.) = 0.1. units O.D.)

Using th graph from the obtained o.d of test solution the concentration of test solution is calculated by the method of inter polation .

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



Test Solution
O.D = _____
Conc. of $KMnO_4$ = _____ x 10^{-4} M

Concentration of $KMnO_4$ **(_____ x 10^{-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 analysed in colorimetry ?
3. How can colorimetry be used to find out concentration of species in solutions which are not coloured?
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 ?

MJCET

EXP. N0. (21 & 22) COLORIMETRY : VERIFICATION OF BEER-LAMBERT'S LAW
ESTIMAITON OF $KMNO_4$ IN TEST SOLUTION

Expt No : **Date :**

TABULATION AND CALCULATIONS

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

Table.1. Weight of bottle + FAS | | | | | | . | | | | g.

Table.2. Weight of empty weighing bottle | | | | | | . | | | | g.

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

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

(V_{ml}=l00ml)

Part B. Standardisation of $KMnO_4$

Titration of Std. F.A.S. Vs. $KMnO_4$ (link)

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

Marks

Observations and calculations (20) : _____

Results & Graphs (10) : _____

Discussion of results (5) : _____

Record (15) : _____

Total (50) : _____

$$N_{KMnO_4} = \frac{N_{FAS} \times V_{FAS}}{V_{KMnO_4}} =$$

Table 1 : Selection of Filters.

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

Table 2: Concentration of $KMnO_4$ vs Optical Density

Sl. No	Std. Soln. V($KMnO_4$ + distilled water) (in ml)	Concentration (Molar)] ------- x 10^{-4}	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 :

$N\ KMnO_{4\ (Stock)} = N_{(Link)} \times \frac{10}{100} = ...$

$MKMnO_{4\ (Stock)} = \frac{N\ (Stock)}{5} .....$

O,D of test sol^n = Msolution (Test) = (From graph)

Wt. of $KMnO_4$ = $M_{(Test)}$ X 158

=.....gpl

Results :

1. Wt. of FAS = ...g
2. N_{std} FAS = ...
3. N_{KMnO_4}(link) = ...
4. M_{KMnO_4} = ... (stock solution)
5. Beer - Lambert's Law verified.
6. Test Solution : OD = _____ : Conc. of $KMnO_4$ = _____ M
7. Wt of $KMnO_4$ in test solution = _____ gpl.

Signature of Faculty

EXPERIMENT NO (23) : VERIFICATION OF BEER-LAMBERTS LAW & ESTIMATION OF COPPER BY COLORIMETRIC METHOD

Exp. No. : **Date :**

AIM: To estimate the amount of copper present in the given sample by colorimetry and to verify Beer-Lamberts law.

Apparatus : Standard flask , Burette , Pipette ,colorimeter ,beaker.

Chemicals : Potassium ferrocyanide , Acetic acid ,Ammonium Hydroxide solution & sulphuric acid.

Principle:

Copper yields a red brown colour with potassium ferrocyanide in acetic acid and ammonium acetate buffer. Intensity of the colour is measured in the colorimeter. Its optical density is measured and concentration of Cu^{2+} ions is found from standard calibration curve.

Mol wt $CuSO_4.5H_2O$ =250, at wt Cu^{2+} = 63.5

Procedure:

Preparation of $CuSO_4$ Stock solution:

Weigh accurately 0.9818gm of pure $CuSO_4.5H_2O$ is and transfer in 250ml standard flask using funnel.Dissolve it in distilled water , add approximately 5ml dilute H_2SO_4 and make the solution homogeneous upto the mark.This is the stock solution.

Preparation of testing standards:

From the above stock solution take separately 10ml,15ml, 20ml, 25ml ,30mland 35ml into 6 standard flasks. Add NH_4OH drop by drop to all the six flasks to neutralize $CUSO_4$ till the solution produces turbidity. Then add 5ml of 2% acetic acid solution and 5ml of 0.2% potassium ferrocyanide to each of the flasks(appox NH_4OH : CH_3COOH = 4 drops : 14 drops, to get proper buffer solution) . Now the solutions are made upto the mark with distilled water. Select the suitable filter with the 6^{th} solution of $CuSO_4$ and then with selected filter no.,The optical densities of all the solutions are determined using colorimeter.

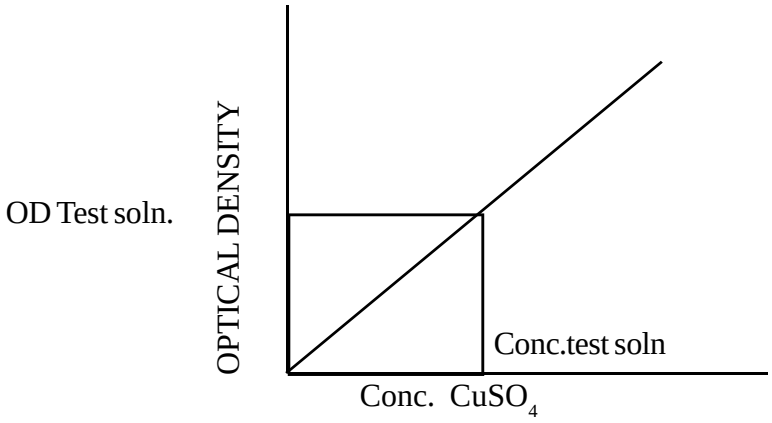
Estimation of $CuSO_4$ in the given test solution.

Take a small quantity of the given test solution in the cuvette.Find the OD of test solution and mark it on the graph and read the concentration from the calibration curve.

A graph is plotted by taking the concentration of Cu^{2+} on X axis and optical density on Y axis. The curve so obtained is called calibration curve. The curve is used for determination of concentration of Cu^{2+}present in the unknown sample.

CALIBRATION CURVE OF CONENTRATION. $CuSO_4$ Vs OD

VERIFICATION OF BEER-LAMBERTS LAW



EXPERIMENT NO (23) : VERIFICATION OF BEER-LAMBERTS LAW & ESTIMATION OF COPPER BY COLORIMETRIC METHOD

TABULATION AND CALCULATIONS

TABLE-1 SELECTION OF FILTERS

S.NO:	FILTER NO:	O.D
1	45	
2	47	
3	51	
4	52	
5	54	
6	57	
7	60	
8	67	

S.NO:	Stock. soln.+NH_4OH+CH_3COOH +distilled water(ml) = 100ml	M x Concentration in each flask --------	Optical Density
1	10+	MX0.1	
2	15+	MX0.15	
3	20+	MX0.2	
4	25+	MX0,25	
5	30+	MX0.3	
6	35+	MX0.35	
7	Test solution.		

TABLE-2 CONCENTRATION OF $CuSO_4$ vs. OPTICAL DENSITY.

Calculations:

Concentration of Stock solution(molarity of $CuSO_4$)= $M = \frac{Wt.of\ CuSO4}{MolWt.of\ CuSO4} X \frac{1000}{V\ ml}$

Vml = 250 ml

Optical density of the test solution =

Concentration of copper in the test solution from graph =

Wt of Cu^{2+} = conc. of test x 63.5 = _____ gpl

Results:

1. M_{CuSO4} (stock) = _____
2. O.D of test solution = _____
3. Conc. of $CuSO_4$ in test (from graph) = _____
4. Wt of Cu^{2+} in the test solution = _____gpl
5. Beer-lamberts law verified

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

Signature of Faculty

EXP. (24) SPECTROPHOTOMETRY

The Spectrophotometer is an instrument for measuring the transmittance or absorbance of a sample as a function of wavelength. The basic principles of Colorimetry are also applicable to the Spectrophotometry (i.e. Beer-Lambert's Law). In addition to coloured samples even colourless samples can be analyzed where absorption lies in the ultra violet region.

1. **Monochromators:** This is an optical device for isolating any desired wavelength of high spectral purity from a continuous source of a beam of radiation.

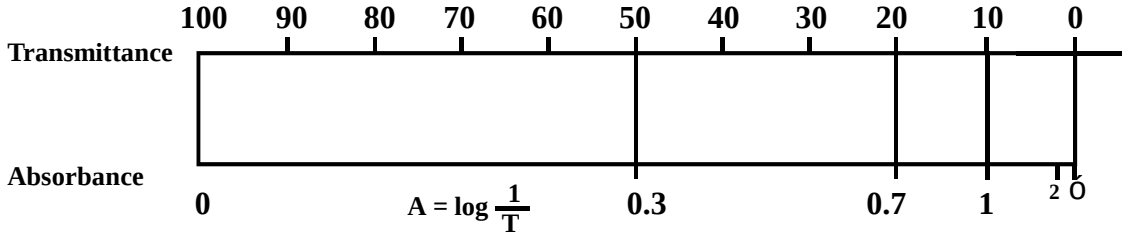
   In the Colorimetry a broad band of radiation in the visible region is incident on the sample where as in Spectrophotometry the source of monochromatic radition is resolved to a band width of less than a nanometer (10^{-9} m = 10^{-7} cm = 10 A^0) with the help of a diffraction grating or prism.

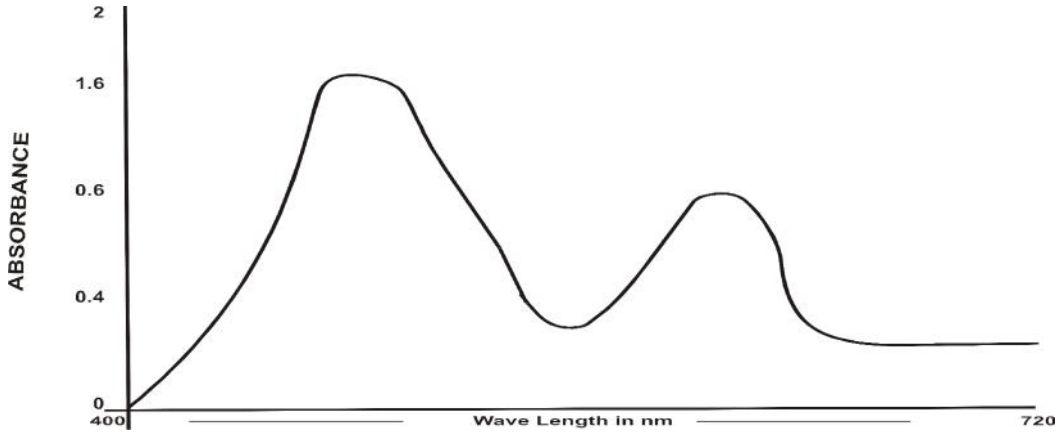
2. **Source of radiation:** Tungsten Lamp for visible region (400 nm - 800 nm).
   Deuterium Lamp for Ultra-violet region (200 nm - 400 nm).

3. **Cells:** The Cells hold the liquid sample in the beam of the Spectrophotometer. The cell must transmit radiant energy in the spectral region of interest. The glass cells serve in the visible region whereas quartz in the U.V. region. The cell must have uniform diameter so that the optical path length remains the same.

4. **Photometer:** Photometer is a device for measuring the intensity of transmitted radiation. In Sepctrophotometer a signal is produced due to the difference between the transmitted radition of a reference material (blank) and that of sample at selected wavelength.

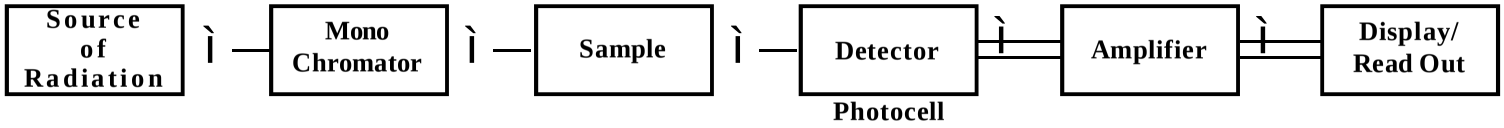
TYPICAL SPECTRUM



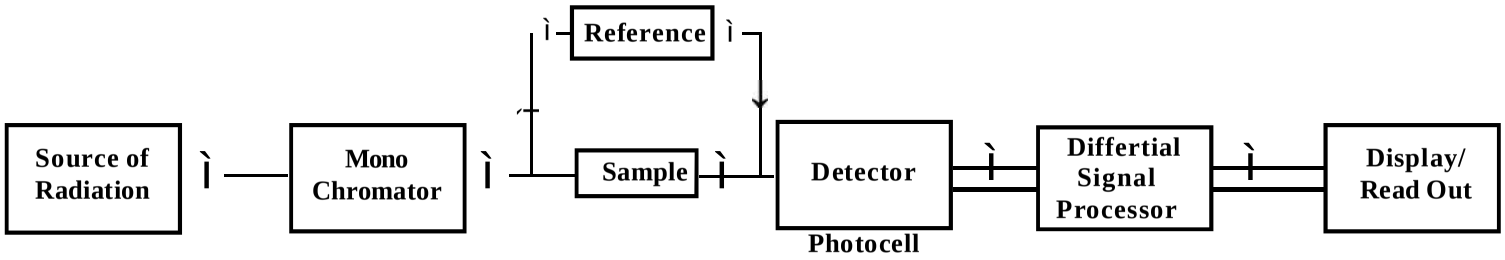


SCHEMATIC DIAGRAM

SINGLE BEAM INSTRUMENT



DOUBLE BEAM SPECTROMETER



EXP. (24) SPECTROPHOTOMETRY

Recording of Cu^{+2} spectrum, determination of maximum wavelength and molar absorptivity

Aim : To determine maximum wavelength of absorption of Cu^{+2} solution and to calculate molar absorptivity.

Requirements : Spectrophotometer, Cuvettes, standard flask.

Reagents : Aqueous copper sulphate solution of 0.01M concentration.

Procedure :

1. Preparation of aqueous copper sulphate solution :

Weigh about 0.25 g of $CuSO_4$ in weighting bottle and transfer through funnel into a 100ml volumetric flask, make the solution upto the mark and shake it well for homogeneity.

Calculate its concentration

$$M_{CuSO_4} = \frac{\text{Wt of } CuSO_4 \times 1000}{\text{Mol. Wt. of } CuSO_4 \times \text{Vol. in ml}}$$

$$= \frac{\text{Wt of } CuSO_4 \times 1000}{250 \times 100}$$

2. Procedure to record the spectrum :

Clean the cuvettes with distilled water. Fill one cuvette upto 3/4th level with distilled water and other cuvette with solution under test ($CuSO_4$ Sol.).

Distilled water is used as reference, as it is solvent for making the solution.

Place the cuvettes holding the opaque sides in the blank of the instrument.

Distilled water cuvette is kept in blank for reference and $CuSO_4$ solution cuvette in sample 1. Light passes through the solution, through transparent sides of cuvettes.

Connect the spectrophotometer to PC for recording the spectrum. [Follow the instructions for operation of spectrophotometer]. The range of visible spectrum for recording the spectrum is from 400 to 1000 nm. The graph of absorbance Vs wavelength is displayed on the PC. From the graph the peak (maximum wavelength) is obtained.

Calculations :

According to Beer Lamberts Law.

$A = \in C x$

A = absorbance
ε = molar absorptivity
C = molar concentration
x = thickness of Cuvettes

Maximum wavelength = 803 nm

Optical density or absorbance = 0.126

x = 1cm. C = 0.01 M

$$\in = \frac{A}{C x} = \frac{0.126}{0.01 \times 1} = 12.6 \text{ lit mole}^{-1} \text{ cm}^{-1}$$

Operation of Spectrophotometer :

1. Connect the spectrophotometer and PC to AC mains switch on the mains.
2. 'ON' the switch located at the back panel of spectrophotometer.
3. Press <ENTER> key of spectrophotometer.
4. SL 159 ELICO UV - vis spectrum is displayed or LCD of instrument.
5. Press <ENTER> key.
6. INITIALIZATION UNDER PROGRESS PLEASE WAIT.
   followed by :-
   LAMP TESTING UNDER PROGRESS
   PLEASE WAIT
   followed by
   LAMP TEST OVER
   AUTOCAL UNDER PROGRESS
   followed by
   (1) Scan (2) M.WL (3) CONC (4) LAMPS
   (5) TIME SCAN (6) PC
7. **Lamp Selection :**
   Press <ENTER> key, as it is desired to select 'W' lamp for recording only visible spectrum
   1. D2LAMP ON 2. W Lamp ON
   is displayed.
8. Press <1> key, as it is not desired to use D2 lamp which is used for UV - Spectrum.
   Press <ENT> key and wait for about 15 min. to warm up the instrument.
   (1). Scan (2). MWL (3). CONC (4). Lamps

(5). Time Scan (6). PC
will be displayed.
Press <6> key to connect to PC.

9. **Step for setting on PC :**
Click on spectra treats, ICON
Click on FILE New
Click on SETTINGS
Select 'COMMUNICATION PORT'
Click 'OK'
'CONNECTION SUCCESSFULY ESTABLISHED' is displayed on PC.

10. Clean the Cuvettes with tissue paper and place them holding the rough side, in the respective blanks (Ref = Water, Sample 1 = Cu Solution)

11. Click on '**UV- VIS Spectrophotometer**' a drop down menu appears.
select 'Scan Spectrum' and click.
Enter the range of wavelength 400 to 900 nm
click on 'NEW REFERENCE' and then click 'START SCAN'
Absorbance vs wavelength graph is displayed.

12. To note the maximum wave length peak select 'peak picking' from 'view'.
Red colour marker appears on the graph.

13. To observe the value for maximum wave length click 'view' and then click 'Data Table'. Data table of recorded range of wave length with absorbance values appears. Search for the maximum wavelength value (with red marker). From this value calcualte molar absorptivity.

14. To close the spectrophotometer press Esc, then remove and wash the cuvettes. Switch off the button at the back of panel and switch off the mains.

Result :

1. Maximum wavelength of absorption for Cu^{+2} = _____nm.
2. Absorbance = _____.
3. Molar absorptivity = _____ lit $mole^{-1}$ cm^{-1}.

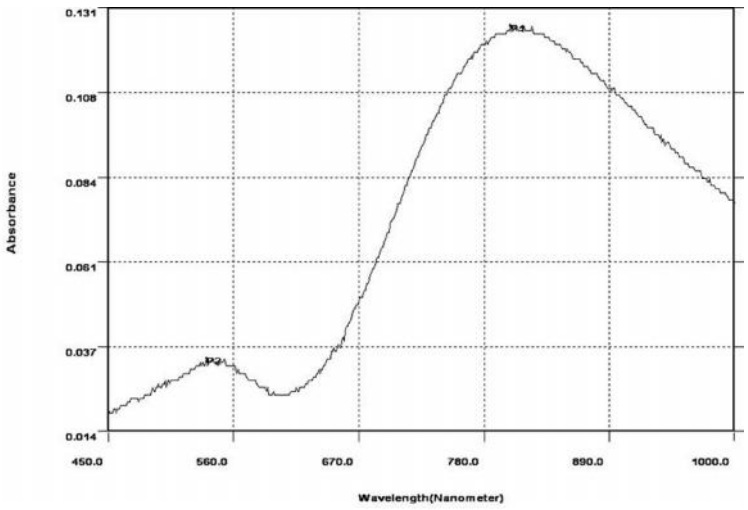
Questions :

1. What is the range of Wave length in visible radiation of Spectrum ?
2. Which lamp is used for UV Light and for Visible Light in the Spectrophotometer ?
3. What is the difference between Colorimeter and Spectrophotometer in terms of source of radiation ?
4. What are the applications of Spectrophotometer ?

A Typical Data of Wavelength vs. Absorbance recorded for $CuSO_4$ Solution.

Wavelength $=^a$ (nm)	Absorbance A
703	0.075
708	0.079
713	0.087
723	0.091
728	0.095
733	0.098
738	0.101
743	0.105
748	0.108
753	0.111
758	0.113
763	0.115
768	0.117
773	0.119
778	0.121
783	0.121
788	0.123
793	0.124
798	0.124
803	**0.126**
808	0.125
813	0.125
818	0.125
823	0.124
828	0.124
833	0.123
838	0.122
843	0.121
848	0.120
853	0.120
858	0.117
863	0.117
868	0.115

A Typical Graph for the Absorbance of Cu^{2+} solution vs Wavelength recorded on Spectrophotometer
^{a}max = 803 nm



SPECTROPHOTOMETRY

Date :

Expt No. :

Tabulation and Calculation :

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

Signature of Faculty

MUFFAKHAM JAH COLLEGE OF ENGINEERING & TECHNOLOGY
(SULTAN-UL-ULOOM EDUCATION SOCIETY)
B.E. I/IV - CHEMISTRY SECTION
INSTRUMENTAL CHEMICAL ANALYSIS

SCHEDULE OF PRACTICALS

Seat Nos	Group	W	E	E	K		S
		1st	2nd	3rd	4th	5th	6th	7th
1, 2	A	C1	C1	P1	P1	pH1	Col1	S
3, 4	B	C2	C2	P2	P2	pH2	Col2	P
5, 6	C	C3	C3	P3	P3	pH3	Col3	E
7, 8	D	C4	C4	P4	P4	Col1	pH1	C
9, 10	E	C5	C5	P5	P5	Col2	pH2	T
11, 12	F	P1	P1	pH1	Col1	C1	C1	R
13, 14	G	P2	P2	pH2	Col2	C2	C2	O
15, 16	H	P3	P3	pH3	Col3	C3	C3	P
17, 18	I	P4	P4	Col1	pH1	C4	C4	H
19, 20	J	P5	P5	Col2	pH2	C5	C5	O
21, 22	K	pH1	Col1	C1	C1	P1	P1	T
23, 24	L	pH2	Col2	C2	C2	P2	P2	O
25, 26	M	pH3	Col3	C3	C3	P3	P3	M
27, 28	N	Col1	pH1	C4	C4	P4	P4	E
29, 30	O	Col2	pH2	C5	C5	P5	P5	T
31, 32	P	Col3	pH3	C6	C6	P6	P6	R
33, 34	Q	C6	C6	P6	P6	Col3	pH3	Y
35, 36	R	P6	P6	Col3	pH3	C6	C6	

C1 - C6 - Conductometer 1 to Conductometer 6

P1 - P6 - Potentiometer 1 to Potentiometer 6

pH1 - pH3 - pH Meter 1 to pH Meter 3

Col 1 to Col 3 - Colorimeter 1 to Colorimeter 3