

VSM COLLEGE OF ENGINEERING

RAMACHANDRA PURAM - 533 255



DEPARTMENT OF S&H

ENGINEERING/APPLIED CHEMISTRY LAB MANUAL

FOR I B.TECH STUDENTS

(AS PER JNTU-KAKINADA)

(R16)

PREPARED BY

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**ENGINEERING / APPLIED CHEMISTRY
LABORATORY
(Common to all branches)**

1. Introduction to Chemistry laboratory – Molarity, Normality, Primary, secondary standard solutions, Volumetric titrations, Quantitative analysis, Qualitative analysis, etc.
2. Trial experiment - Determination of HCl using standard Na_2CO_3 solution.
3. Determination of alkalinity of a sample containing Na_2CO_3 and NaOH.
4. Determination of KMnO_4 using standard Oxalic acid solution.
5. Determination of Ferrous iron using standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution.
6. Determination of Copper using standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution.
7. Determination of temporary and permanent hardness of water using standard EDTA solution.
8. Determination of Copper using standard EDTA solution.
9. Determination of Iron by a Colorimetric method using thiocynate as reagent.
10. Determination of pH of the given sample solution using pH meter.
11. Conductometric titration between strong acid and strong base.
12. Conductometric titration between strong acid and weak base.
13. Potentiometric titration between strong acid and strong base.
14. Potentiometric titration between strong acid and weak base.
15. Determination of Zinc using standard EDTA solution.
16. Determination of Vitamin – C.

Outcomes: The students entering into the professional course have practically very little exposure to lab classes. The experiments introduce volumetric analysis; redox titrations with different indicators; EDTA titrations; then they are exposed to a few instrumental methods of chemical analysis. Thus at the end of the lab course, the student is exposed to different methods of chemical analysis and use of some commonly employed instruments. They thus acquire some experimental skills.

Reference Books

1. A Textbook of Quantitative Analysis, Arthur J. Vogel.
2. Dr. Jyotsna Cherukuris (2012) *Laboratory Manual of engineering chemistry-II*, VGS Techno Series
3. Chemistry Practical Manual, Lorven Publications
4. K. Mukkanti (2009) *Practical Engineering Chemistry*, B.S. Publication

EXPERIMENT-1

1. CHEMICAL ANALYSIS

Analysis: Chemical analysis of the compounds is carried out in two ways.

1. **Qualitative Analysis:** It shows the elements present in a given substance.
2. **Quantitative Analysis:** The determination of the quantity of a particular component present in a substance. This analysis can be carried out in two ways i.e.,
 - a. Gravimetric Analysis
 - b. Volumetric Analysis
- a. **Gravimetric Analysis:** It involves the estimation of the amount of a given compound from the result of weighing.
- b. **Volumetric Analysis:** It is based on measuring the volume of the solution of a substance.

The term volumetric analysis is formerly used for this form of quantitative chemical analysis carried out by determining the volume of solution of accurately known concentration which is required to react quantitatively with a measured volume of a solution of the substance to be determined.

The volumetric analysis is a technique of finding the volume of a solution of unknown concentration which will exactly react with a definite volume of another solution of known concentration in this method. A known volume of one solution is taken in a conical flask and the solution of another substance with known concentration is added very slowly to it till the reaction is completed. This is indicated by certain substances known as indicators. This process is called titration. The stage at which the reaction is completed is called End point.

From the volume of the unknown solution which is required to completely react with definite volume of the other solution of known strength. The strength of the unknown solution can be calculated based on normality i.e.,

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2}$$

N_1 = Normality of Known solution

V_1 = Volume of known solution

N_2 = Normality of unknown solution

V_2 = Volume of unknown solution

1. Terms used in Volumetric Analysis

- a. **Titration:** The drop wise addition of one solution to the other solution from burette to conical flask, to find the unknown volume or concentration by using known volume or concentration is called titration.
- b. **Titrant:** The reagent from the burette is called titrant.
- c. **Titrate:** The substance being titrated is termed as titrate.
- d. **Equivalent Point (or) Stoichiometric End Point:** It is the point at which the amounts of reagent or substance bring determined and chemically equivalent or it is the exact stage at which the chemical reaction involved in the titration is just completed.
- e. **Indicator:** The substance which helps in the visual detection of the completion of the titration is known as indicator.
- f. **End Point:** The point at which the colour change of the indicator is apparent to the eye is called End Point.

Standard Solution:

A solution whose concentration is known is called a standard solution. There are two types of standard solutions depending on the nature of the substance.

- a. **Primary Standard Solution:** Substance which is stable, pure, readily soluble in water, with high equivalent weight and the composition of its solution should not change on standing or during storage is called standard solution.

Eg: Oxalic acid, Potassium dichromate, Anhydrous Na_2CO_3 etc.

- b. **Secondary Standard Solution:** The substance which donot fulfil the above mentioned requirements of primary standard substances, their solutions are not directly prepared by weighing and the exact strength of the solution is found by titrating it against some primary standard is called secondary standard substance and the process is called standardization.

Eg: NaOH, KOH, KMnO_4 , HCL, H_2SO_4 etc.

Molarity:

The number of moles of solute present per litre of the solution is called molarity. It is indicated by 'M'. If 'n' moles of solute dissolved 'v' litres of solution, then molarity of solution is $M = n/v$ moles/litre

But

$$n = \frac{\text{weight of the solute (G)}}{\text{Gram molecular weight of solute (GMW)}}$$

Then

$$M = \frac{G}{\text{GMW}} \times \frac{1}{V(\text{in lit})} \text{ or } \frac{G}{\text{GMW}} \times \frac{1000}{V(\text{in ml})}$$

Normality:

The number of gram equivalent weights of the solute present in one Liter of the solution is known as Normality. It is indicated by 'N'

$$N = \frac{\text{Weight of Solute}}{\text{Gram Equivalent weight of Solute}} \times \frac{1000}{V(\text{in ml})}$$

The Gram Equivalent Weights are different for different solutes. Now let us know how to calculate the equivalent weight of different types of solutes.

- i. **For Acids:** Equivalent weight of an acid is given by the formula:

$$E = \frac{\text{Molecular weight of an acid}}{\text{Basicity of an acid}}$$

Basicity: The number of replaceable hydrogen atoms by metal ions in a molecule of the acid is known as basicity of the acid.

Eg: HCl Eq. wt = $\frac{36.5}{1} = 36.5$

H_2SO_4 Eq. wt = $\frac{98}{2} = 49$

H_3PO_4 Eq. wt = $\frac{97.97}{3} = 32.66$

- ii. **For Bases:** The number of replaceable hydroxyl groups present in a molecule of the base is known as its acidity.

$$E = \frac{\text{Molecular weight of the base}}{\text{Acidity of the base}}$$

Eg: NaOH Eq. wt = $40/1 = 40$

Ba(OH)_2 Eq. wt = $171.3/2 = 86.5$

- iii. **For salts:**

$$\text{Eq. wt} = \frac{\text{Molecular weight of salt}}{\text{Total No. of +ve charges on cations} - \text{charges on anions}}$$

Eg: NaCl

Total No. of Cations $\text{Na}^+ = 1$

Total No. of Anions $\text{Cl}^- = 1$

Eq.wt = $58.5 / 1 = 58.5$

iv. For Oxidant / Reductant:

Depends on the redox reaction, the equivalent weight of these changes with the nature of the reaction.

The No. of moles of e^- transferred to 1 mole of oxidant or released from 1 mole of oxidant or released from 1 mole of reductant indicates the equivalent weight of the oxidant or reductant respectively.

Eg: i) KMnO_4 is an oxidizing agent in acidic medium

The reduction half reaction is



The oxidation state of manganese changes from +7 to +2 the No. of moles of e^- transferred to KMnO_4 per mole is 5

$$E = \frac{\text{Molecular weight of } \text{KMnO}_4}{5} = \frac{158.04}{5} = 31.61$$

Types of Titrations and Indicators:

Depending upon the nature of chemical reaction involved, the volumetric titrations are classified into 4 types.

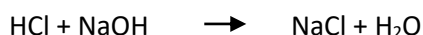
- 1) Acid – Base Titration
- 2) Oxidation – Reduction titration
- 3) Complex metric titration
- 4) Precipitation titration

In the above titrations acid-base titration, complex metric and precipitation titrations are no change in oxidation state as they depend upon the combination of ions. But the oxidations reduction reactions involve a change of oxidation state or a transfer of e^- .

1) Acid – Base Titration

This Acid – Base titrations are also known as neutralization reactions or acidimetric or alkalimetric. This volumetric determination of an acid with alkali to produce salt with unionized water i.e., neutralization.

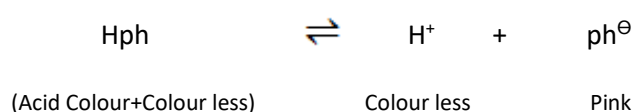
For e.g., the titration of NaOH against HCl



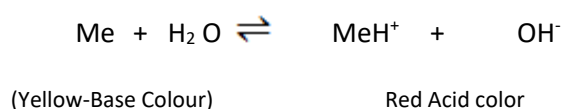
These include the titration of free bases, or those formed from salts of weak acids by hydrolysis with a standard acid called Acidimetry and the titration of free acids, or those formed by the hydrolysis of salts of weak acids by hydrolysis of salts of weak bases, with a standard base called Alkalimetry. The reactions, involve the combination of hydrogen and hydroxide ions to form water.

The indicators used for neutralization titration are called, which themselves are weak acids and weak bases. They have different colours in ionized and unionized forms.

Eg: Phenolphthalein is a weak acid and its unionized molecules are colourless while on ionization it gives pink colour due to $\text{Ph}^\ominus$ ions. Hence it gives pink colour in alkaline medium, colourless in acid medium and the P^{H} range is 8.0 to 9.6



Methyl orange is a weak base. It changes colour at P^{H} range 3.1 to 4.4 from yellow to red in basic to acidic medium respectively.



Because these indicators change their colour depending on P^{H} of the medium they are called P^{H} indicators. The selection of proper indicator for a given titration is very important one should select an indicator which changes colour approximately at P^{H} of the equivalent point of titrations.

2) Oxidation – Reduction Titration/ Oxidometry

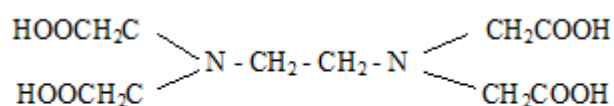
The titration in which reducing agent is titrated against an oxidizing agent and vice-versa are called redox titrations. In this different types of titrations are present i.e.,

- Permanganometry:** Potassium permanganate is the oxidizing agent and titrated against a reducing agent like Fe^{+2}
- Chromatometry:** Potassium dichromate is the oxidizing agent and titrated against a reducing agent like Fe^{+2}
- Iodometry:** It is based on oxidation by the action of free iodine generated from KI.
- Bromometry:** It involves the oxidation with potassium bromated. Other redox titrations include cerimetry (Reduction by Ce^{+4} ions), vanadometry (oxidation by VO_3^- ions), Titanometry (Reductions by Ti^{+3} ions)

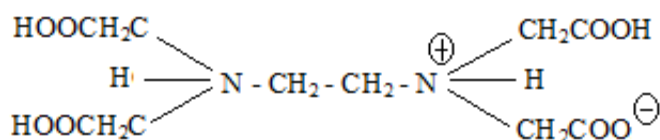
The indicators used are called redox indicators whose oxidized form is of different colour from that of reduced form. The change of colour of the redox indicator depends upon the electrode potential of the system. While choosing the redox indicator for a particular titration, it is necessary to ensure that its potential lies within that of the system and colour change should occur as close to the equivalent point as possible.

3) Complexometric Titration: These titrations involve the formation of a stable soluble complex between the metal and complexing reagent. The metal ion is called central atom and the complexing reagent is called ligand.

/the most remarkable ligand used widely in complexometric titration is EDTA (Ethylene Diamine Tetra-acetic Acid), because it forms stable complexes with a number of metal atoms at a definite P^H range

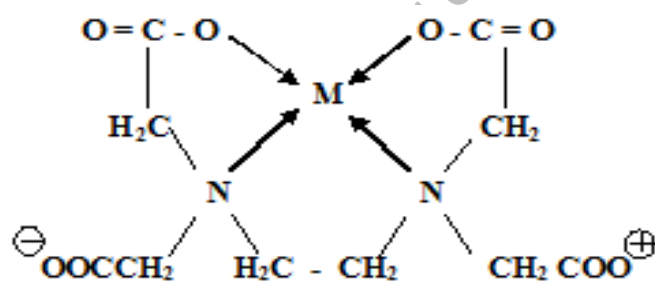


EDTA Structure



Zwitter ion

It has six potential sites (hexa-dentate) for bonding a metal ion. The Four Carboxy groups and two Amino groups. When EDTA is dissolved in water it forms Zwitter ions Disodium salt of EDTA is used for titrations because it combines with metal ion in a 1:1 ratio regardless of the charge on the cation to form stable cage like structure.



The complexes of divalent metals like Ca^{+2} and Mg^{+2} form stable complexes at P^H 9-10. Ammonical buffer is used to maintain the P^H

The indicators used in these titrations undergo colour change as they form less stable complexes with metal ions. EDTA replaces the indicator from [Indicator-M] complex and forms colourless, soluble stable complex as [M-EDTA] complex. The colour of the indicator is the end point.

Eg: Eriochrome Black-T indicator

Viva Questions

1. What is Qualitative & Quantitative analysis?
2. What is Volumetric analysis? Write its use
3. What is Gravimetric analysis?
4. What is Titration? Mention the terms involved in it?
5. How many types of titrations are there?
6. What is concentration? Write the terms expressed for the Concentration?
7. What is primary standard solution & what is secondary standard solution?
8. Define end point?
9. What is indicator? Mention its purpose? Give some indicators.
10. What is the use of standard solution?
11. What is the formula for estimation of unknown concentration of a solution?
12. Define (i) Molarity (ii) Normality

EXPERIMENT-2**Estimation of HCl by using standard Na₂CO₃ Solution**

Aim: To estimate the amount of HCl present in the given solution by using Na₂CO₃.

Apparatus: Burette, Pipette, Conical flask, volumetric flask, beaker.

Chemicals required: Hydrochloric acid, Sodium Carbonate, Methyl orange indicator

Principal: The amount of HCl can be estimated by titration with a standard Na₂CO₃ solution by using acid base indicator like methyl orange. The following neutralization reaction takes place during the titration.



At neutralization point the solution is neither acidic nor basic and when a drop of HCl is added, the solution becomes acidic indicated by a colour change from yellow to red at the end point.

Step1: Standardization of HCl:

First wash the burette with tap water and then with distill water and fill the known HCl solution it. Pipette out 10ml of Standard Na₂CO₃ solution into a clean conical flask and add 1 or 2 drops of methyl orange indicator. Then the solution appears as pale yellow. This solution is titrated against the standard HCl solution until a colour change from yellow to pink is obtained. Then note the burette reading. Repeat the titration until we get concurrent readings.

Standardization of HCl:

S.No	Volume of Standard Na ₂ CO ₃ solution (ml)	Burette Reading		Volume of HCl Rundown (in ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

Formula: $N_1V_1 = N_2V_2$

N_1 : Normality of HCl =.....

V₁: Volume of HCl =

N₂: Normality of Na₂CO₃ =

V₂: Volume of Na₂CO₃ =

$$N_1 = \frac{N_2 V_2}{V_1}$$

Normality of HCl (N₁) = ----- N

Step 2: Estimation of HCl:

The given unknown HCl solution is make up in a 100ml volumetric flask up to the mark and fill it in a burette. Pipette out 10ml of Standard Na₂CO₃ solution into a clean conical flask and add 1 or 2 drops of methyl orange indicator. Then the solution appears as pale yellow. This solution is titrated against the standard HCl solution until a color change from yellow to pink is obtained. Then note the burette reading. Repeat the titration until we get concurrent readings.

Estimation of HCl:

S.No	Volume of Std Na ₂ CO ₃ solution (ml)	Burette Reading		Volume of unknown HCl Rundown (in ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

Formula $N_2 V_2 = N_3 V_3$

N₂: Normality of Na₂CO₃ =

V₂: Volume of Na₂CO₃ =

N₃: Normality of unknown HCl =

V₃: Volume of unknown HCl =

$$N_3 = \frac{N_2 V_2}{V_3}$$

normality of Unknown HCl = ----- N

$$\text{Normality} = \frac{\text{Weight}}{\text{Gram equivalent weight}} \times \frac{1000}{V \text{ (in ml)}}$$

=

Amount of HCL = -----

Precautions:

1. All the apparatus must be washed with distilled water only because tap water contains hardness
2. All the reagents must be prepared with distilled water

Result: The amount of HCl presents in given 100 ml solution _____ Gms

Viva Questions

1. To what category of titration does this experiment belongs?
2. What is Acid-Base titration?
3. Which indicator is used in this titration?
4. Why did you choose methyl orange?

5. What is the color of methyl orange in basic medium?
6. What is the color of methyl orange in acidic medium?
7. What is the standard solution in this experiment?
8. Is it a primary standard solution? Why so?
9. What do you mean by normality?
10. How can you calculate the strength of the given solution?
11. Why should you carry out the titration for 3 or 4 times?

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EXPERIMENT : 3**Alkalinity of a sample containing Na_2CO_3 and NaOH .****AIM AND OBJECTIVE:**

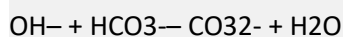
To determine the alkalinity of a given water sample.

THEORY:

Alkalinity is a measure of the capability of water to absorb H^+ ions without significant change of pH.

In other words, alkalinity is a measure of the acid buffering capacity of water. The determination of alkalinity of water is necessary for controlling the corrosion, to calculate the amount of lime and soda needed for water softening; in conditioning the boiler feed water, etc.

Alkalinity of a sample of water is due to the presence of OH^- (hydroxide ion), HCO_3^- (bicarbonate ion) and CO_3^{2-} (carbonate ion) or the mixture of two ions present in water. The possibility of OH^- and HCO_3^- ions together is not possible since they combine together to form CO_3^{2-} ions.



PRINCIPLE: – Alkalinity of water means the total content of those substances in it which causes an increased

OH^- ion concentration up on dissociation or due to hydrolysis. The alkalinity of water is attributed to the presence of (i) Caustic alkalinity (Due to OH^- and CO_3^{2-})

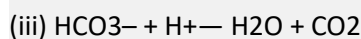
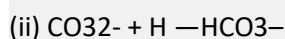
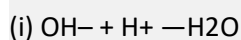
(ii) Temporary hardness (Due to HCO_3^-)

Alkalinity is a measure of ability of water to neutralize the acids

Determination of alkalinity

OH^- , CO_3^{2-} and HCO_3^- can be estimated separately by titration against standard acid using phenolphthalein and methyl orange as indicators

The determination is based on the following reactions



The titration of water sample against a standard acid up to phenolphthalein end point (P) marks the completion of reaction (i) and (ii) only. This amount of acid used thus corresponds to OH⁻ plus one half of the normal CO₃²⁻ present. On the other hand, titration of the water sample against a standard acid to methyl orange end point (M) marks the completion of reaction (i), (ii) and (iii). Hence the total amount of acid used represents the total alkalinity.

Thus,

$$P = \text{OH}^- + \frac{1}{2} \text{CO}_3^{2-}$$

$$M = \text{OH}^- + \text{CO}_3^{2-} + \text{HCO}_3^-$$

METHODOLOGY:

APPARATUS AND MATERIALS

- Burette
- Pipette
- Conical flask
- Beakers
- Funnel and
- Dropper
- Burette stand
- Clamp

CHEMICALS REQUIRED AND MATERIALS

- phenolphthalein
- methyl orange indicator
- Distilled Water
- Standard sulphuric acid
- Ethyl alcohol

PROCEDURE

1. Firstly, the pH value for both water samples was measured.

2. 100ml of the first sample AB was measured into a conical flask
3. Then 2-3 drops of phenolphthalein was added to the conical flask
4. When there was no color change was observed, 2-3 drops of methyl orange was added to it
5. The burette was the filled with the sulphuric acid, noted the initial reading and began titrating it against the water sample AB
6. The titration continued until there was a colour change observed in the water sample, which indicates the end point
7. The titration was stopped, noting the final reading and the water sample was then discarded.

S.No	Volume of standard MgSO ₄ solution (V ₁ ml)	Burette Reading		Volume of EDTA consumed (V ₂ ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

$$M_1V_1 = M_2V_2$$

M₁= Molarity of H₂SO₄ solution

V₁ = Volume of H₂SO₄ solution

M₂= Molarity of watersample

V₂= Volume of watersample

$$M_2 = \text{Molarity of watersample} = \frac{M_1V_1}{V_2}$$

PRECAUTION

- All the glass apparatus was washed thoroughly with distilled water before use
- Before use, the burette and pipette was rinsed properly.

- It was ensured that the burette has no leakage.
- The conical flask was placed on white paper to identify the color change at the end point.

OBSERVATION

During the experiment, it was observed that after adding the phenolphthalein indicator to both water samples, there was no form of change in their colour. Methyl orange changed their colour to yellow, after titration with the standard sulphuric acid, their end point colour was pink. But the average titre value of the water sample AB was relatively higher than that of the water sample AC.

CONCLUSION

At the end of the experiment and from the analysis of the results obtained, the following conclusion can be made;

- Sample AB= ppm (mg/L) CaCO_3
- Sample AC= ppm (mg/L) CaCO_3
- Total Alkalinity = mg/L CaCO_3
- The Phenolphthalein Alkalinity was zero, this implies that the alkalinity due to Hydroxyl and Carbonate ions is zero hence, making the Alkalinity due to Bicarbonate equal to the Total Alkalinity for both water samples.

RECOMMENDATION

Alkalinity is not considered detrimental to humans but is generally associated with pH values, hardness and excess dissolved solids. High alkalinity waters may also have a distinctly flat, unpleasant taste. Based on the experiment carried out above, it was found that the alkalinity of the samples is 61.00 ppm (61.00 mg/L) and 40.00 ppm (40.00 mg/L). As per the provisional code, alkalinity should not exceed 200 ppm (200 mg/L) for potable water. For the fresh water alkalinity ranges from 20 – 100 ppm (20 – 100 mg/L). The Alkalinity of the given samples are within the limits specified in the standards.

EXPERIMENT : 4**Estimation of Permanganate by using Standard Oxalic acid solution**

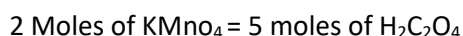
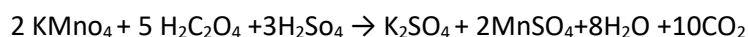
Aim: - To estimate the amount of KMnO_4 by using standard Oxalic acid solution.

Apparatus:-

Burette, Pipette, Conical flask, volumetric flask, Retard stand.

Required chemicals:-

- 1) Potassium permanganate (KMnO_4)
- 2) Oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$)
- 3) Sulphuric acid (H_2SO_4 -4N)

Chemical Reaction:-**Procedure:-**

All the apparatus are washed with tap water first, after washed with distilled water. Then the burette is filled with Potassium permanganate (KMnO_4) solution without any air bubbles. 20 ml of oxalic acid is pipette out into the conical flask without any bubbles and add 10 ml 4 N H_2SO_4 . Then heat the flask about 5mnts. Then titrate this mixture against with KMnO_4 with careful until the colorless solution turns into (light) pale pink. Then titration is stopped and notes the end point. Repeat the titration until concurrent values are obtained.

Estimation of KMnO_4

S.No	Volume of Oxalic acid (ml)	Burette reading		Volume of KMnO_4 (in ml)
		Initial	Final	
1				
2				
3				

Formula & Calculation:

$$N_1V_1 = N_2V_2$$

N_1 : Normality of KMnO_4 = _____?

V_1 : Volume of KMnO_4 = _____

N_2 : Normality of oxalic acid = -----

V_2 : Volume of oxalic acid = _____

The volume of KMnO_4 present in 1Ltr solution

(X) = Molarity of KMnO_4 X Equivalent weight of KMnO_4 .

= _____ grams

Result: The amount of the given KMnO_4 present in 100 ml solution

= X/10-----Gms

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Viva Questions

1. Which type of titration involved in this experiment?
2. What is oxidation? What is oxidizing agent?
3. What is Reduction? What is reducing agent?
4. Name some oxidizing agents.
5. Which indicator is used in this titration?
6. Why this titration is called as Redox titration?

EXPERIMENT: 5**Estimation of Ferric iron using standard $K_2Cr_2O_7$ solution**

Aim: To estimate the amount of ferric iron (Fe^{+3}) in the given 100 ml of the solution using a standard potassium dichromate solution.

Apparatus:

Pipette, Burette, Conical Flask, Retard Stand, Bunsen burner, Volumetric Flask.

Required chemicals:-

1. 0.05 N standard $K_2Cr_2O_7$ solution
2. $SnCl_2$ solution.
3. $HgCl_2$ solution.
4. 1:3 ratio of H_3PO_4 and H_2SO_4 acid mixture.
5. Diphenylamine internal indicator.

Standardization of given Fe^{+3} solution:

Make up the given Ferric iron solution up to the mark with distilled water and shake well for uniform concentration. Pipette out 20 ml of it into a clean conical flask, add 10 ml of concentrated HCl and heat the solution to boiling until the colour of the solution changes to yellow. Add $SnCl_2$ solution drop by drop to the hot solution till the yellow colour disappears. Cool the solution under the tap and add 10 ml of $HgCl_2$ solution, in one portion. Similarly, white precipitate of $HgCl_2$ is obtained. Add 5ml of acid mixture and 3-4 drops of Diphenyl amine indicator. Titrate the solution against standard $K_2Cr_2O_7$ solution taken in a burette till blue-violet colour is obtained. This is the end point. Repeat the titration until the concurrent values are obtained. Tabulate the readings

Table:

S.No	Volume of Fe^{+3} solution (in ml)	Burette reading (in ml)		Volume of $K_2Cr_2O_7$ (in ml)
		Initial	Final	
1				
2				
3				

Calculations:

$$N_1V_1 = N_2V_2.$$

Where

$$N_1 = \text{Normality of } K_2Cr_2O_7 =$$

$$V_1 = \text{Volume of } K_2Cr_2O_7 =$$

$$N_2 = \text{Normality of } Fe^{+3} \text{ solution.}$$

$$V_2 = \text{Volume of } Fe^{+3} \text{ solution.}$$

$$N_2 = \frac{N_1V_1}{V_2}$$

$$= \frac{(N_1 \times \text{End point})}{20}$$

Amount of Fe^{+3} present in given 100 ml of the solution

$$\text{Weight} = \frac{N_2 (\text{Normality of } Fe^{+3}) \times 55.85}{10} \text{ gms}$$

*** Preparation of standard $K_2Cr_2O_7$ solution:**

For preparing of 0.05 N Potassium dichromate solution weight out accurately about 0.245 gms of $K_2Cr_2O_7$ into 100ml of standard volumetric flask and dissolve the solid in a small amount of distilled water. Make up the resulting solution with distilled water up to the mark and shake the flask well for uniform concentration.

Normality of $K_2Cr_2O_7$ (N_1)

$$(N_1) = \frac{\text{weight}}{\text{gram equivalent}} \times \frac{1000}{\text{value in ml}}$$

$K_2Cr_2O_7$ gram equivalent weight = 49

Precautions:

1. Titration should be slow. i.e., drop by drop at the end point.
2. Through shaking slowly should be required for every drop.

Result: The amount of the (Fe^{+3}) in given sample _____ gr.

Viva Questions

1. Which type of titration involved in this experiment?
2. What is oxidation? What is oxidizing agent?
3. What is Reduction? What is reducing agent?
4. Name some oxidizing agents.
5. Which indicator is used in this titration?
6. Why this titration is called as Redox titration?
7. Why do you have to heat the contents in the conical flask to 70°C?
8. What happens when heated to higher temperatures?
9. Why do you have to use only H_2SO_4 and why not HCl or HNO_3 ?
10. Is use of acid is necessary in this redox titration?

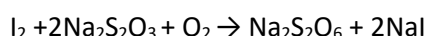
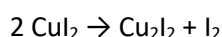
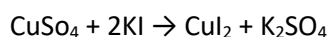
EXPERIMENT: 6**ESTIMATION OF COPPER BY IODOMETRY**

Aim: To estimate the amount of Copper present in the given solution using a standard solution of Potassium Dichromate and Hypo as the link solution.

Apparatus: 100 ml standard flask, Burette, Iodometric Flask, Conical Flask, Pipette, Simple balance with weights,

Chemicals required: $K_2Cr_2O_7$, Hypo ($Na_2S_2O_3$), KI, Conc. HCl, dil. Acetic acid, $NaHCO_3$, H_2SO_4 , $CuSO_4$, Ammonia solution, Starch & distilled water.

Principle: Any cupric salt in neutral medium when treated with potassium Iodide forms a white precipitate of cuprous iodine is set free quantitatively. The liberated Iodine is treated against Hypo using starch as the indicator.

Chemical reactions:**Procedure:**

1. **Preparation of standard potassium dichromate:** Weigh out accurately the given pure crystalline sample of potassium dichromate and transfer into 100 ml standard (volumetric) flask provided with a funnel. Dissolve the dichromate in a small quantity of distilled water, and make up to the mark. The contents in the flask are shaken well for uniform concentration. Calculate the normality of potassium dichromate.
2. **Standardization of sodium thiosulphate:** Rinse the burette and fill it up with hypo solution without any air bubbles. Note the burette reading. Take about 20 ml of 10% KI solution in a clean conical flask and add 2 grams of sodium bicarbonate followed by 5 ml of concentrate HCl gently rotate the flask for mixing the liquids. Rinse the pipette with a little of potassium dichromate solution and then transfer 20 ml of the same to the conical flask. Shake it well, stopper it, and keep it in dark place for 5 minutes. Titrate the liberated iodine by running down hypo from the burette with constant stirring. When the solution attains a pale yellow colour add 2 ml of freshly prepared starch solution. The colour changes to blue. Continue the titration drop-wise till the colour changes from blue to light green indicating the end point. Repeat the titration for concurrent values

3. **Estimation of copper:** Make up the given solution of copper to the 100ml volumetric flask with distilled water and shake well for uniform concentration. Pipette out 20 ml of this solution into a redissolved the precipitate in dilute acetic acid. Now add 10 ml of 5% KI, When iodine is liberated giving a brown colour. Titrate this solution against standard hypo solution till light yellow colour is obtained. Now add 2 ml of starch solution and continue the titration till blue colour changes to creamy white, which is the end point. Repeat the titration for concurred values and calculate the amount of copper.

Preparation of standard solution:

W_1 = Weight of bottle + substance = _____ gms

W_2 = Weight of bottle = _____ gms

Weight of substance = $(W_1 - W_2)$ = _____ gms.

$$\text{Normality of the solution} = \frac{(w_1 - w_2) \times 10}{\text{Equivalent Weight}} = \frac{(w_1 - w_2) \times 10}{49} =$$

S.No	Volume of standard ($K_2Cr_2O_7$) Solution (V_1)	Burette Reading		Volume of consume (V_2)
		Initial	Final	
1	20 ml			
2	20 ml			
3	20 ml			
	$V_1 =$			$V_2 =$

N_1 : Normality of Potassium dichromate =

V_1 : Volume of Potassium dichromate = 20 ml

N_2 : Normality of Hypo = ?

V_2 : Volume of Hypo =

$$N_1 V_1 = N_2 V_2$$

N_2 : Normality of Hypo =

$$N_2 = \frac{N_1 V_1}{V_2} =$$

S.No	Volume of Copper Solution (V_3)	Burette Reading		Volume of consume (V_4)
		Initial	Final	
1	20 ml			

2	20 ml			
3	20 ml			
	$V_3 = 20 \text{ ml}$			$V_4 =$

N_3 = Normality of Copper Solution =?

V_3 = Volume of Copper Solution = 20 ml

N_4 = Normality of Hypo =

V_4 = volume of Hypo =

$$N_3 V_3 = N_4 V_4$$

$$\text{Normality of Copper solution} = N_3 = \frac{N_4 V_4}{V_3}$$

$$\text{Amount of Copper present in the whole of the given solution (10 ml)} = \frac{N_3 \times 63.54}{10}$$

Result: Amount of Copper Present in the whole of the given solution (100 ml) = _____ gm.

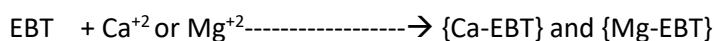
EXPERIMENT-7**DETERMINATION OF HARDNESS OF WATER**

AIM: To determine the amount of hardness present in a given water sample by using standard EDTA.

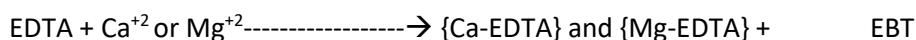
Chemicals required: 1) Zinc sulphate solution, 2) Standard disodium salt of ethylene di amine tetra acetic acid (Na_2EDTA) 0.01 M, 3) Ammonia buffer solution (pH 10), 4) Sodium hydroxide solution, 5) Erichrome black T solution,

Apparatus required: Burette, pipette, volumetric flask, conical flask, stirrer, wash bottle.

Principle: EDTA molecule forms a stable complex with Ca and Mg ions at a pH of 8-10. Erichrome black-T too can form a complex with these ions but it is not as stable as complexed formed with EDTA. At a pH 8-10 Erichrome black T imparts wine red color at complexed state in un complexed state it imparts sky blue color.



Unstable complex (wine red)



Stable complex (Colorless)

sky blue

Preparation of Reagents:

Sample water: ordinary tap water can be used as sample water.

0.01M EDTA solution: Dissolve 3.72 grams of EDTA di sodium salt in 1litre of distilled water. Standardise this against potassium biphthalate which is prepared by dissolving 4.085 grams of Potassium biphthalate in 1000ml of distilled water

0.01M Standard MgSO_4 solution: Dissolve 2.465 grams of Magnesium sulphate in 1Lt of distilled water up to the mark.

Step-I: Standardization of EDTA Solution:**Procedure:**

20 ml of Magnesium sulphate solution is pipetted out in to a clean conical flask. To this solution, 2ml of buffer solution, 3 or 4 drops of EBT indicator are added. Then there develops a wine red colour.

This solution is titrated against EDTA solution taken in the burette. The end point of the titration is conversion of wine red colour into blue. Repeat the titrations till you get the concordant values. By knowing the volume of the EDTA consumed, the molarity of EDTA is calculated

S.No	Volume of standard MgSO ₄ solution (V ₁ ml)	Burette Reading		Volume of EDTA consumed (V ₂ ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

$M_1V_1 = M_2V_2$

M₁= Molarity of MgSO₄ solution V₁ = Volume of MgSO₄ solution

M₂= Molarity of EDTA solution V₂= Volume of EDTA solution

M₂= Molarity of EDTA solution= $\frac{M_1V_1}{V_2}$

Step-II: Estimation of Hardness of water

Rinse and fill the burette with the given EDTA solution. Pipette out 20ml of sample water solution into a conical flask. Add 5ml of Ammonia buffer solution and a pinch of EBT indicator. Titrate the solution against EDTA drop wise with shaking till the wine red color changes to sky blue. Note the volume of EDTA used. Repeat the titration for concordant readings.

Estimation of Hardness

S.No	Volume of sample Hard water (V ₃ ml)	Burette Reading		Volume of EDTA consumed (V ₂ ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

DETERMINATION OF HARDNESS OF WATER:

Volume of Hard water taken for each titration (V₃) =

Volume of 0.01 M EDTA used (burette reading) = V_2 ml

100ml of 1M EDTA = 100mg of CaCO_3

1ml of 0.01M EDTA = 1 mg of CaCO_3

V ml of 0.01M EDTA = 1V mg of CaCO_3 = x mg of CaCO_3

20ml of hard water contains = x mg of CaCO_3

1000ml of hard water contains = $x/20 \times 100$ mg of CaCO_3 = y mg /L

Result: The Total Hardness of water = y mg/L

Viva Questions

1. (a) What is Hardness? (b) Why it is due to? (c) Why soaps are not effective in hard water?
2. What is the principle involved in this experiment?
3. Which type of titration involved in this experiment?
4. What is the full form of EDTA & EBT?
5. Why EBT indicator should be used in basic medium?
6. Which buffer is used in this titration? What is its p^{H} value?
7. How many complexes are formed during this titration?
8. What is color of the unstable complex? What is the color of the stable complex?
9. What is the indicator used in this titration?

Experiment-8**ESTIMATION OF COPPER BY EDTA BY COMPLEXOMETRY**

Aim: To estimate the Copper in the given solution by preparing a standard solution of EDTA.

Apparatus: 100 ml standard flask, Burette,
250 ml Conical Flask,
20 ml Pipette,
Simple balance with weights,

Chemicals Required: Ethylene Di-amine tetra acetic acid (EDTA),
Fast sulphone Black-F Indicator,
Ammonia solution,
Copper solution,
Distilled water.

Principle: Fast sulphone black F indicator is specific in its reaction with copper in ammonia solution, it forms red complex with copper. In the titration of copper in ammonical solution the colour changes at the end point from pale blue to bright green depend upon the concentration of Cu^{+2} ions.

Procedure:

1. **Preparation of standard solution of EDTA:** Weigh out accurately about 0.3722 Gms of EDTA sample into 100 ml standard flask and dissolve minimum quantity of distilled water make up the solution up to the mark with distilled water and shake the flask well for uniform concentration.
2. **Estimation of copper:**
Make up the given solution of copper up to the mark and shake the flask well for uniform concentration, rinse the pipette with the copper solution and pipette out 20 ml of copper solution into a clean 250 ml conical flask. Add 20 ml of distilled water and 5 ml of ammonia solution and 5 drops of the Fast sulphon black F indicator solution titrate with standard EDTA solution until the colour changes of from blue to dark green. Repeat the titration to get the concurrent values.

Preparation of Standard solution:

W_1 = Weight of bottle + substance = _____ gms

W_2 = Weight of bottle=-----gms

Weight of substance ($W_1 - W_2$) = -----gms

Molarity of the solution = $(W_1 - W_2) \times 10 \div \text{molecular weight} =$

Calculation:

S.NO	Volume of standard solution (V ₂)	Burette reading		Volume of consumed (V ₁)
		Initial	Final	

M₁ = Molarity of EDTA =

V₁ = volume of EDTA solution =

M₂ = Molarity of copper solution =?

V₂ = Volume of Copper solution = 20 ml

$$M_1 V_1 = M_2 V_2$$

$$M_2 = \text{Molarity of copper solution} = \frac{M_1 V_1}{V_2}$$

1 ml of 0.05 M EDTA = 3.177 mg of Cu²⁺

Amount of copper present in the given 100ml of solution

$$= \frac{\text{Titrate value} \times \text{Molarity of EDTA} \times 3.177 \times 1000}{0.05 \times 20}$$

=

Result: Amount of copper present in the given 100ml of solution =

-

Viva Questions

1. What is the full form of EDTA & EBT?
2. What is the principle in EDTA titrations?
3. Give the structures of EDTA & EBT
4. Which buffer is used in this titration?
5. What are the constituents of the ammonium buffer?
6. What is the indicator used in this titration?
7. Write the structure of fast sulphone black-F?
8. How the end point is indicated in this estimation of copper?
9. What is Ligand?

Experiment: 9**ESTIMATION OF COPPER BY COLORIMETRIC METHOD****AIM:** Estimation of Copper by Colorimetry**Apparatus:** - Colorimeter, Test tubes, Burettes.

Principal: - Colorimeter measures the optical density of absorbing substance where optical

Density (O.D) is defined as $O.D = \log I_0/I \rightarrow$ Where I_0 = Intensity of incident light I = Intensity of transmitted light

As per beers law, optical density of an absorbing substance is related to the concentration by the equation.

$$O.D = E.C.I$$

$$O.D = (E.I).C.....Eq.(2)$$

Where 'C' is the concentration of the substance, is the path length, which represents the width of the cell used and is constant for a given cell used, E is the molar absorption coefficient and is a Constant for given substance. Equation (2) may be written as

$$O.D. \propto CEq.(3)$$

Equation (3) represents the quantitative from of Beer's law. If the optical density of a substance is

Determined at varying concentration. A plot of O.D. Vs C gives a straight line.

PROCEDURE: - Take the sample solution of $CuSO_4$ and prepare the following 10 sample solutions in test tubes as 1 to 10 and note the optical density values.

1. 1 ml 0.1 M $CuSO_4$ + 4 ml Distilled water
2. 2 ml 0.1 M $CuSO_4$ + 3ml Distilled water
3. 3 ml 0.1 M $CuSO_4$ + 2ml Distilled water
4. 4 ml 0.1 M $CuSO_4$ + 1 ml Distilled water
5. 5 ml 0.1 M $CuSO_4$ + 0 ml Distilled water

S.NO	Volume of the 0.1M $CuSO_4$ solution(ml)	Volume of the Distilled water(ml)	Optical Density (O.D)
------	--	--------------------------------------	-----------------------

1	1	4	
2	2	3	
3	3	2	
4	4	1	
5	5	0	

GRAPH: - plot a graph of O.D Vs [CuSo₄]. A straight line passing through origin is obtained the slope of which gives EI, E is the molar absorption coefficient.

$$\text{Slope} = \frac{y_2 - y_1}{x_2 - x_1}$$

$$\text{Slope} - EI \rightarrow E = \frac{\text{slope}}{I}$$

$$E = \text{-----mole}^{-1}\text{cm}$$

Result: - The amount of Copper present in the given CuSo₄ solution is ____grams

Viva Questions

1. What do you mean by Colorimetry?
2. What is a Colorimeter?
3. What is Beer's law?
4. Express Beer's law in the form of equation?
5. What are $A = \epsilon l c$?
6. What is the common oxidation state of copper?
7. What is the principle involved in colorimetric determination of copper?
8. What is calibration curve?
9. What is the nature of the calibration curve?
10. Why it should be so?

Experiment: 10

Determination of P^H of given sample

Aim: To determine the P^H of the given sample.

Apparatus:

1. A P^H meter with glass and reference electrode with temperature compensation.
2. Beaker.
3. Electrode.

Reagents required:

1. PH 1.68 (at 20⁰c) : Dissolve 12.7 gm of potassium tetroxalate dehydrate in 1 L (0.05M)
2. PH 4.0 (at 20⁰c) : Dissolve 10.21 gms of potassium hydrogen phthalate in 1 L (0.05M)
3. PH 6.88 (at 20⁰c) : Dissolve 3.40 gm of potassium hydrogen phosphate or 3.55 gm of disodium hydrogen phosphate in 1 L
4. PH 9.2 (at 20⁰c) : Dissolve 3.18 gm of sodium tetra borate decahydrate in 1 L

Sample preparation: (Sample solutions)

Appropriate concentrations for test solutions are as follows.

Sugar and sugar containing products -25%

Powdery materials -25%

Liquids, normal consistency -at sample strength

Liquids, highly viscous -50%

For apparently non – soluble materials-Shake for 30 minutes before testing

Theory:

PH is a measure of the acidity of a solution. It is the negative logarithm of H⁺ ion concentration.

$$PH = -\log [H^+]$$

Definition of PH is not restricted to H⁺ ion concentration but it also represents the tendency of hydrogen ions to interact to other components of the solution. It is also affected by the ionic strength of solution. Hence even on the addition of KCl also alters the PH of a solution

pure water with Ph close to 7.0 is said to be neutral solution, with less than 7 is said to be acidic nature solution and greater than 7 is said to be basic nature of solution.

The normal range for PH value of water on surface water systems is 6.5 to 8.5 and ground water systems 6 to 8.5. Water with a low PH (<6.5) could be acidic, soft and corrosive. Water with a PH > 8.5 could indicate that the water is hard.

Procedure:-

Standardize the P^H meter using the two buffer solutions nearest to the expected P^H of test solution measure the P^H of the test solution. Recheck the standardization of the P^H meter using the appropriate buffer solutions.

S. NO	Sample name	P ^H of sample
1		
2		
3		
4		
5		

Result:

Sample name _____

P^H of the given sample _____

Viva Questions

1. What is P^H ? Mention its scale?
2. What is the reason for acidity in natural waters?
3. What is the concept of CaCO_3 equivalent for expressing acidity?
4. What is the range of acidity in lake, river waters?
5. Give two examples of strong and weak acids?
6. What is the P^H range of acidic solutions?
7. What is the P^H range of basic solutions?
8. How the P^H varies with H^+ ion concentration?
9. What is the P^H value of pure water?

Experiment: 11**CONDUCTOMETRY**

Aim: - To determine the strength of given HCl solution by Conductometrically

Apparatus: - Conductivity meter, conductivity cell, Beakers, Pipette, burette

Chemicals: - NaOH solution, HCl solution.

Determination of strength of given HCl solution:

The given unknown HCl solution is 100 ml volumetric flask is made up to the mark and then 10 ml of given unknown HCl solution is taken in a 100 ml beaker. To this 40 ml of distilled water is added, the electrode of the cell is dipped in the solution. The conductivity of solution is noted before adding the alkali. The standardized NaOH is added from burette with 1 ml of increasing by shaking thoroughly, the contents of the beaker and conductivity values are noted, the values of observed conductivities are plotted against volume of NaOH. The concentration of HCl is determined using the end point from the point.

S.No	Volume of the NaOH in ml	Conductivity (ohm ⁻¹)
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Volume of NaOH solution from graph V_2 =-----ml

Volume of HCl solution V_1 = 10 ml

Normality of NaOH solution $N_2 = \text{-----}N$

Normality of HCl solution $N_1 = \text{-----}N$

FORMULA:

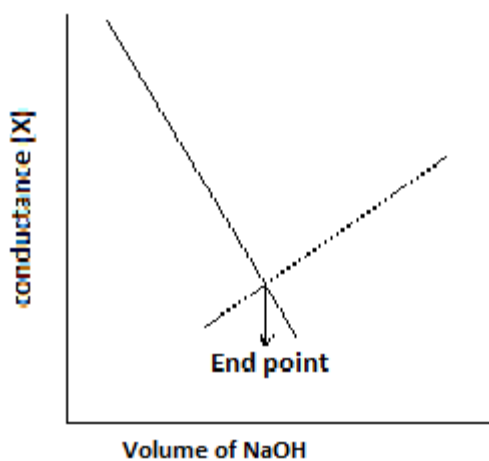
$$N_1 V_1 = N_2 V_2$$

Result:

Strength of HCl = Normality of HCl (N_1) $\times$ equivalent weight of HCl

= ----- gm/L

Model Graph:



RESULT: Strength of HCl solution = Normality of HCl solution $\times$ *eg. wt of HCl*

$$= N_3 \times 36.5$$

$$= \text{-----} g/$$

Viva Questions

1. What is the principle involved in this titration?
2. Explain conductivity, molar conductivity & Specific conductivity.
3. What do you mean by cell constant?
4. What could be the shape of the graph?
5. What are the units of conductivity in CGS and SI systems?
6. When strong acid combines with a strong base what type of reaction occurs?
7. How conductance is related to the concentration of the ions?
8. Why conductance decreases on addition of NaOH to HCl?
9. Name the apparatus used for this method?
10. What are the equivalent masses of HCl & NaOH?

EXPERIMENT-12**Conductometric titration of strong acid vs weak base**

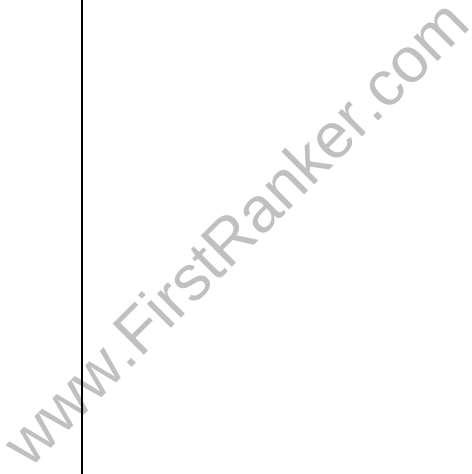
Aim: To determine the end point of strong acid with weak base titration by conductometrically.

Apparatus: conductivity meter, conductivity cell, beakers, pipette, burette.

Chemicals required: HCl, NH_4OH , and phenolphthalein indicator.

Determination of strength of given HCl solution:

The given unknown HCl solution is 100 ml volumetric flask is made up to the mark and then 10 ml of given unknown HCl solution is taken in a 100 ml beaker. To this 40 ml of distilled water is added, the electrode of the cell is dipped in the solution. The conductivity of solution is noted before adding the alkali. The standardized NH_4OH is added from burette with 1 ml of increasing by shaking thoroughly, the contents of the beaker and conductivity values are noted, the values of observed conductivities are plotted against volume of NH_4OH . The concentration of HCl is determined using the end point from the point.

S.No	Volume of the NaOH in ml	Conductivity (ohm^{-1})
		

Volume of NaOH solution from graph $V_2 = \text{-----ml}$

Volume of HCl solution $V_1 = 10 \text{ ml}$

Normality of NaOH solution $N_2 = \text{-----} N$

Normality of HCl solution $N_1 = \text{-----} N$

FORMULA:

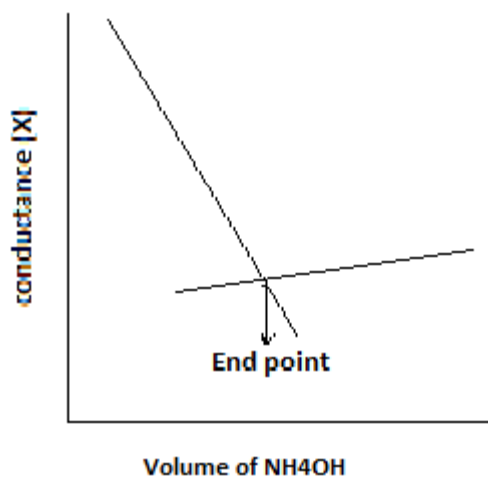
$$N_1 V_1 = N_2 V_2$$

Result:

Strength of HCl = Normality of HCl (N_1) $\times$ equivalent weight of HCl

= ----- gm/L

Model graph:



Viva Questions

1. Why the shape of the graph is so in this experiment?
2. What is the principle involved in this titration?
3. Explain conductivity, molar conductivity & Specific conductivity.
4. What do you mean by cell constant?
5. What could be the shape of the graph?
6. What are the units of conductivity in CGS and SI systems?

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EXPERIMENT-13**POTENTIOMETRY TITRATION OF STRONG ACID Vs STRONG BASE**

AIM: - To Determine the concentration of a strong acid by a Potentiometric titration with a given strong base of known concentration.

APPARATUS: - Potentiometer, Pt-electrode, Calomel electrode, Salt bridge, 150 ml beakers, Stirrer, Pipette, Burette.

CHEMICALS: - 0.1M NaOH, 0.1M HCL, Quinhydrone solid, KCl Solution.

PRINCIPLE: - The quinhydrone electrode is prepared by taking saturated solution of quinhydrone in a known quantity of strong acid whose concentration is to be determined. This electrode is combined with the calomel electrode to make the cell. Pt, Hg, Hg₂Cl₂(s)/Saturated//H⁺/QH₂/Pt

In acid medium quinhydrone exist in two forms.

As NaOH is added to a solution of H⁺ ions with quinhydrone the H⁺ ions are consumed steadily and the relative concentration of QH₂ and Q changes with the titration. At the end point where all the ions are consumed, QH₂ gets completely converted to Q. the electrode potential of the electrodes is given as per the nearest equation.

$$E_{QH_2/Q} = E^0_{QH_2/Q} - \frac{0.059}{n} \log \frac{a_{QH_2}}{a_Q \cdot a_{H^+}}$$

$$= E^0_{QH_2/Q} - \frac{0.118}{n} \log aH \quad [\because a_Q \text{ And } a_{CH_2} = 1]$$

At the endpoint, which $aH^+ \rightarrow 0$ there is a sudden fall in the cell emf $E_{cell} = E_{calomel} + E_{aH_2/Q}$

Since calomel is constant the variation of E_{cell} is a measure of the variation of $E_{aH_2/Q}$. The endpoint is the titration is the point where there is a maximum fall in potential with a small addition of NaOH.

Procedure: - Pipettes out 10 ml of the given HCl solution into a clean 100ml beaker add a pinch of quinhydrone and stir it well to make a saturated solution of quinhydrone is H^+ . Dip a platinum electrode. Take a second beaker with saturated KCl solution in it. Dip the calomel electrode in it. Connect two beakers with a salt bridge; connect the terminals of the potentiometer with the two electrodes. Note the emf of the cell. Fill a burette with 0.1 NaOH, add 0.5ml of NaOH into the beakers containing acid, stir well for equilibrium to establish. Note the emf of the cell once again. The variation of emf by successive addition of 0.5 ml of NaOH. Each time will be about 10-20mv, as the endpoint approaches, the fall in the emf would increase from 10-20 mv to 40-50 mv. At this stage add NaOH in batches of 0.2 ml and note emf after each addition. At the end point the emf falls suddenly by about 100-150 mv. Continue the titration with 0.2 ml of NaOH and note 5-6 emf values. Now the variation is emf gets smaller and takes 4-5 emf readings, after the endpoint by adding batches of 1ml of NaOH each time, tabulate the results.

GRAPH: - Plot a graph of emf Vs volume of NaOH added and a second graph of $\Delta E / \Delta V$ Vs Volume of NaOH added. Note the point of Neutralization from both the graph and calculate the conc of given HCl using the concn of given NaOH as 0.1M

Sl.No	Volume of Alkali (ml)	Emf (mv)	$\xrightarrow{\text{Poly'n}} \frac{\Delta E}{\Delta V}$
1			
2			
3			
.			
.			
.			
30			

CALCULATION: -

HCl

NaOH

 $V_1 = 10ml$
 $V_2 = \text{_____}$ (From Graph)

 $N_1 = ?$
 $N_2 = 0.1$
RESULT: - At the Neutralization point 10 ml of given HCl =? And concern of given HCl=_____

Viva Questions

1. What is Potentiometric titration?
2. What is electrode potential?
3. What is Reduction potential?
4. What is Oxidation Potential?
5. What is Reference electrode?
6. What is indicator electrode?
7. What is the effect of temperature on EMF of an unknown solution?
8. What is the effect of concentration on EMF of an unknown solution?
9. Write Nernst equation for single electrode potential?
10. Which electrode is employed to find out the pH of an unknown solution?
11. What is the EMF of standard hydrogen electrode?
12. What is the shape of the graph?

EXPERIMENT: 14**POTENTIOMETRY Titration of Strong acid Vs Weak Base**

Aim: to determine the concentration of strong acid by a potentiometer titration gives a weak base of known concentration.

Apparatus: Potentiometer, Pt –electrode, Calomel electrode, Salt Bridge, 100ml beakers, stirrer, Pipette, Burette.

Chemicals: 0.1 M NH₄OH, 0.1 m HCl, Quinhydrone solid, Cu solution.

Principle: The Quinhydrone electrode is prepared by taking saturated solution of Quinhydrone in a known Quantity of strong acid whose concentration is to be determined. This electrode is combined with the calomel electrode to make the cell. Pt, Hg, Hg₂Cl₂(S)/saturated/H⁺/QH₂/Pt.

In acid medium quinhydrone exist in two forms.

As NH₄OH is added to a solution of H⁺ ions with quinhydrone the H⁺ ions are consumed steadily and the relative concentration of QH₂ and Q changes with the titration. At the end point where all the ions are consumed, QH₂ gets completely converted to Q. the electrode potential of the electrodes is given as per the nearest equation.

$$E_{QH_2/Q} = E^0_{QH_2/Q} - \frac{0.059}{n} \log \frac{aq. a^{r+} qh^{+}}{aqH_2}$$

$$= E^0_{QH_2/Q} - \frac{0.18}{n} \log a_h [aq \text{ and } a_{h_2} = 1]$$

At the end point, which aH⁺ there is a sudden fall in the cell enf $E_{cell} = E_{calomel} + E_{ah_2/Q}$.

Since calomel is constant the variation of E_{cell} is a measure of the variation of $E_{QH_2/Q}$. the end point is the titration is the point where there is a maximum fall in potential with a small addition of Noah.

Procedure: Pipettes out 10 ml of the given HCl solution into a clean 100 ml beaker add a pinch of Quinhydrone and stir it well to make a saturated solution of Quinhydrone is H⁺ dip a platinum electrode. Take a second beaker with saturated Cu solution in it .Dip the calomel electrode in it .Connect two beakers with a salt bridge. Connect the terminals of the potentiometer with the two electrodes. Note the enf of the cell. Fill a burette with 0.1 NH₄OH add 0.5 ml of NH₄OH into the beakers containing acid, stir well for equilibrium to establish. Note the enf of the cell one again.

The variation of enf by successive addition of 0.5 ml of NH₄OH.Each time will be about 10-20 ml, as the end point approaches, the fall in the enf would increase from 10-20 ml to 40-50 ml. At this tage add NH₄OH in batches of 0.2 ml and note enf after each addition. At the end point the enf falls suddenly by about 10-150 mv. Continue the titration with 0.2 ml of NH₄OH and note 5-6 enf values. Now the variation is EMF gets smaller and takes 4-5 enf readings. After the end point by adding batches of 1 ml of now each time, tabulate the results.

Table:

S.NO	Volume of	Enf (mv)	
1			
2			
3			

Calculation:

HCL

 $V_1 = 10\text{ml}$ $N_1 = ?$ NH_4OH $V_2 = \text{_____}$ (From graph) $N_2 = 0.1$ $V_1N_1 = V_2N_2$

$$N_1 = \frac{V_2N_2}{V_1}$$

Result: Concentration of given HCL (N_1) = _____ N.

Graph: plot a graph of enf V_8 volume of NH_4OH added and see and graph of E/V V_8 volume of NH_4OH added. Hole the point of neutralization from both the graph and calculate the concentration of given Hcl using the concern of given NH_4OH as 0.1M

Viva Questions

1. How many types of potentiometric titrations are there? What are they?
2. How the end point is determined using this potentiometric titration method?
3. What is the shape of the graph?
4. What is the use of Nernst equation?
5. What is the difference between potentiometry and conductometry?
6. What are the advantages of potentiometric over normal titrations?

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Experiment-15

ESTIMATION OF ZINC BY EDTA BY COMPLEXOMETRY

Aim: To estimate the Zinc in the given solution by preparing a standard solution of EDTA.

Apparatus: 100 ml standard flask, Burette,
250 ml Conical Flask,
20 ml Pipette,
Simple balance with weights,

Chemicals Required: Ethylene Di-amine tetra acetic acid (EDTA),
Eriochrome Black-T Indicator,
Ammonia solution,
Zinc solution,
Distilled water.

Principle: Eriochrome Black-T indicator is specific in its reaction with zinc in ammonia solution, it forms red complex with zinc. In the titration of zinc in ammonical solution the colour changes at the end point from pale blue to bright green depend upon the concentration of Cu^{+2} ions.

Procedure:

3. **Preparation of standard solution of EDTA:** Weigh out accurately about 0.3722 Gms of EDTA sample into 100 ml standard flask and dissolve minimum quantity of distilled water make up the solution up to the mark with distilled water and shake the flask well for uniform concentration.
4. **Estimation of zinc:**
Make up the given solution of zinc up to the mark and shake the flask well for uniform concentration, rinse the pipette with the zinc solution and pipette out 20 ml of zinc solution into a clean 250 ml conical flask. Add 20 ml of distilled water and 5 ml of ammonia solution and 5 drops of the Eriochromeblack-T indicator solution titrate with standard EDTA solution until the colour changes of from blue to dark green. Repeat the titration to get the concurrent values.

Preparation of Standard solution:

W_1 = Weight of bottle + substance = _____ gms

W_2 = Weight of bottle=-----gms

Weight of substance ($W_1 - W_2$) = -----gms

Molarity of the solution = $(W_1 - W_2) \times 10 \div \text{molecular weight} =$

S.NO	Volume of standard solution (V ₂)	Burette reading		Volume of consumed (V ₁)
		Initial	Final	

Calculation:

M₁ = Molarity of EDTA =

V₁ = volume of EDTA solution =

M₂ = Molarity of zinc solution = ?

V₂ = Volume of Zinc solution = 20 ml

$$M_1 V_1 = M_2 V_2$$

$$M_2 = \text{Molarity of zinc solution} = \frac{M_1 V_1}{V_2}$$

1 ml of 0.05 M EDTA = 3.177 mg of Cu²⁺

Amount of zinc present in the given 100ml of solution

$$= \frac{\text{Titrate value} \times \text{Molarity of EDTA} \times 3.177 \times 1000}{0.05 \times 20}$$

=

Result: Amount of zinc present in the given 100ml of solution =

-

Viva Questions

10. What is the full form of EDTA & EBT?
11. What is the principle in EDTA titrations?
12. Give the structures of EDTA & EBT
13. Which buffer is used in this titration?
14. What are the constituents of the ammonium buffer?
15. What is the indicator used in this titration?
16. Write the structure of fast sulphone black-F?
17. How the end point is indicated in this estimation of zinc?
18. What is Ligand?

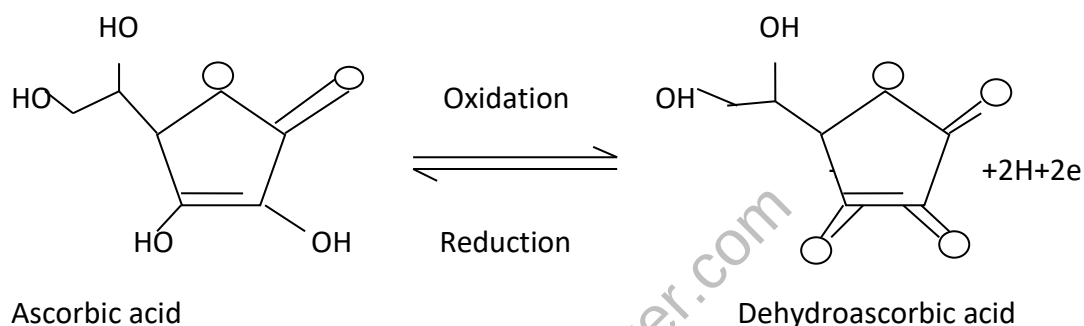
Experiment: 16**DETERMINATION OF VITAMIN C****AIM**

To determine the Vitamin C concentration in fruit juices.

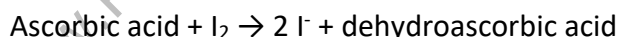
INTRODUCTION

Vitamin C or ascorbic acid is a water soluble antioxidant that plays a vital role in protecting the body from infection and disease. It is an essential antioxidant that plays a vital role in protecting the body from infection and disease. It is an essential antioxidant needed for the human body. It is not synthesized by the human body and therefore must be acquired from dietary sources- Primarily fruits and vegetables.

The chemical structure and antioxidant (reducing) action of ascorbic acid are illustrated in the redox half equation below:



This method determines the vitamin C concentration in a solution by redox titration using iodine. As the iodine is added during the titration, the ascorbic acid is oxidized to dehydroascorbic acid, while the iodine is reduced to iodide ions.



Once all the ascorbic acid has been oxidized, the excess iodine is free to react with the starch indicator, forming the blue-black starch-iodine complex. This is the endpoint of the titration. The method is suitable for use with Vitamin C tablets, fresh or packaged fruit juices and solid fruits and vegetables.

CHEMICALS REQUIRED**0.005 M Iodine solution**

Weigh 2 gm of potassium iodide into a 100 ml beaker. Weigh out 1.3 gm of iodine and add it into the same beaker. Add a few ml of distilled water and swirl for a

few minutes until iodine is dissolved. Transfer iodine solution to a 1 L volumetric flask, making sure to rinse all traces of solution into the volumetric flask using distilled water. Make the solution up to the 1 L mark with distilled water. The concentration of the prepared iodine solution can be more accurately determined by titration with a standard solution of potassium thiosulphate using a starch indicator.

0.5% Starch Indicator Solution

Weigh 0.25 gm of soluble starch and add it to 50 ml of near boiling water in a 10 ml conical flask. Stir to dissolve and cool before using.

SAMPLE PREPARATION

For Vitamin C tablets: Dissolve a single tablet in 10 ml of distilled water (in a volumetric flask if possible).

For fresh fruit juice: Strain the juice through cheese cloth to remove seeds and pulp which may block pipettes.

For packaged fruit juice: This may also need to be strained through cheese cloth if it contains a lot of pulp or seeds.

For fruits and vegetables: cut a 100 gm sample into small pieces and grind in a mortar and pestle. Add 10 ml portions of distilled water several times while grinding the sample, each time decanting off the liquid extract into a 10 ml volumetric flask. Finally, strain the ground fruit/vegetable pulp through cheese cloth, rinse the pulp with 10 ml portion of water and collect all filtrate and washings in the volumetric flask. Make the extracted solution up to 100 ml with distilled water.

PROCEDURE

Pipette out 20 ml aliquot of the sample solution into a 250 ml conical flask and add about 150 ml of distilled water and 1 ml of starch indicator solution. Titrate the sample with 0.005 M iodine solution. The end point of the titration is identified as the first permanent trace of a dark blue-black colour due to the starch-iodine complex. Repeat the titration with further aliquots of sample solution until you obtain concordant results (titres agreeing within 0.1 ml). Calculate the average volume of iodine solution used from concordant titres.

The average titre volume should ideally be in the range of 10-30 ml. If the titre required for a 20 ml aliquot of sample solution is well outside this range then a larger or smaller aliquot volume should be chosen. If the volume of the titre is too low, dilute the standard. If the volume of the titre is too high, dilute the sample.

CALCULATIONS

Volume of iodine solution consumed in ml =

Molarity of iodine solution (M) =

Volume of sample solution taken (x ml) =

CALCULATIONS

Volume of iodine solution consumed in ml =

Molarity of iodine solution (M) =

Volume of sample solution taken (x ml) =

Molarity of sample solution = $\frac{\text{molarity iodine} \times \text{volume of iodine}}{\text{Volume of sample solution}}$

=

Amount of Vitamin C present in 100 ml = $\frac{\text{molarity} \times \text{Eq. Wt. of ascorbic acid}}{10}$

=

Then calculate the concentration of Vitamin C (ascorbic acid) in percentage of weight.

RESULT

Sample name:

The concentration of Vitamin C in sample (fruits or fruit juices) =%

.....

Viva Questions

1. What is the chemical name of vitamin C?
2. What is the main function of vitamin C in human beings?

3. What happens when a person suffers from the deficiency of vitamin C?
4. What is the molecular formula and molecular mass of vitamin C?
5. What are the melting and boiling points of vitamin C?
6. What is the principle involved in the estimation of vitamin C?
7. Basically vitamin C is an acid. Then why it cannot be determined by acid-base titrations?
8. How can you estimate the amount of vitamin C present in the given fruit juice?

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