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Date : 31st March 2022

ORGANIC CHEMISTRY

Experiment - 1

AIM:

Preparation of benzoic acid from toluene

CHEMICALS REQUIRED

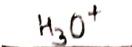
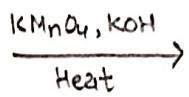
- Toluene (2 mL)
- Potassium permanganate (2.5 g)
- Sodium carbonate (2 g)
- Hydrochloric acid (conc.)
- Sodium bisulphite (saturated solution)

PROCEDURE

1. In a clean 250 mL conical flask, we take 2 mL of toluene and 2 g of sodium carbonate and dissolve in 30 mL of distilled water.
2. In a 100 mL beaker, we prepare a saturated solution of potassium permanganate (2.5 g).
3. We add the aqueous solution of permanganate to the mixture of toluene and sodium carbonate slowly till the purple colour of permanganate persists.
4. We add some pieces of pumice stones in the conical flask and cover the mouth of flask using a funnel.
5. We place a piece of marble inside the funnel.

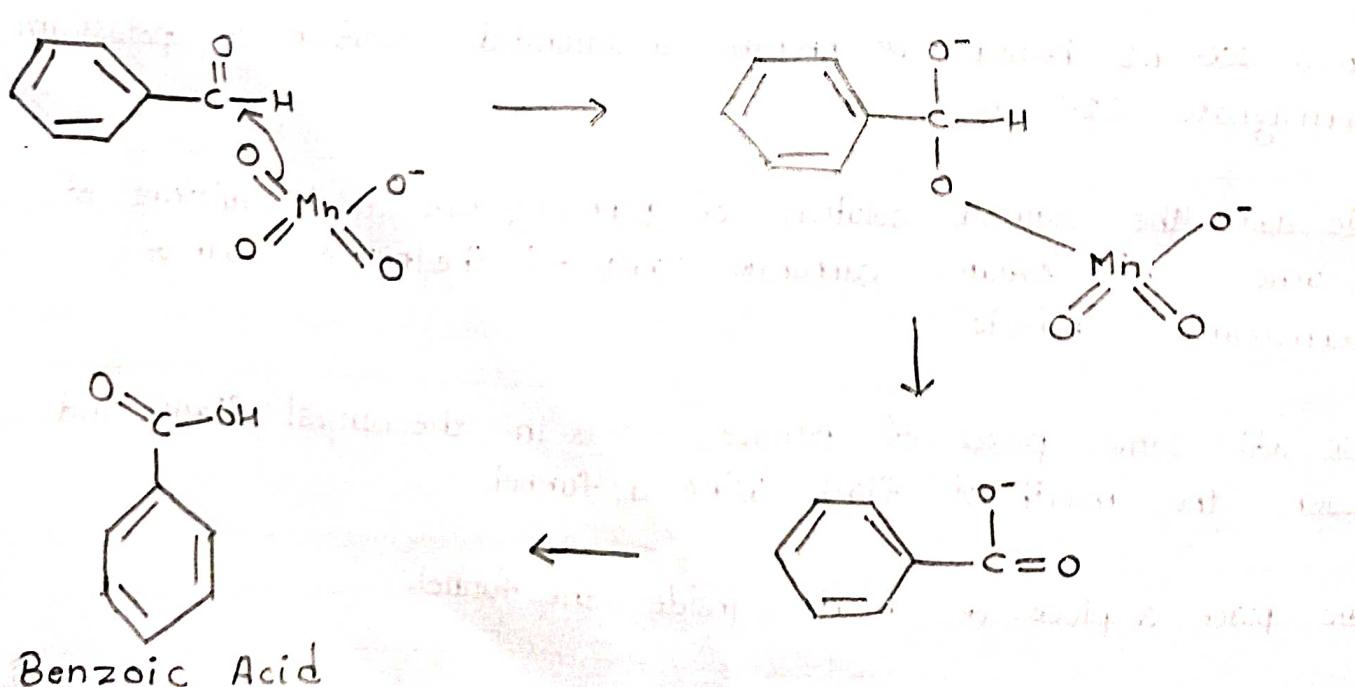
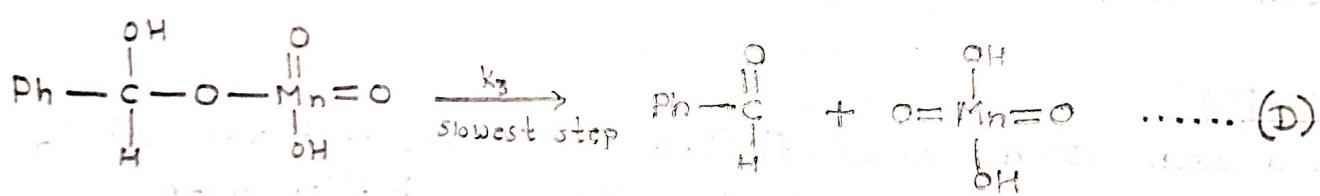
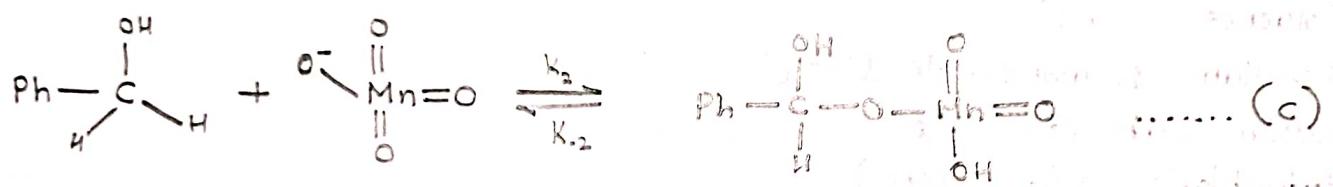
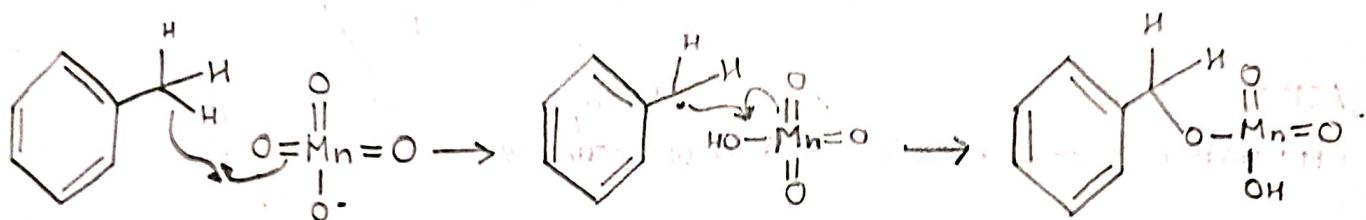
6. We gently boil the solution for about 40 minutes.
7. We cool the solution and pour it in a 500 mL beaker.
8. We add a saturated solution of sodium bisulphite till brown precipitate of manganese dioxide dissolves.
9. We acidify the contents of the beaker using concentrated hydrochloric acid (check with a piece of pH paper)
10. White ppt. of benzoic acid separates out. We filter the crude product at the sunction pump, wash with cold water and dry it.
11. We record the yield.
12. We recrystallize a small amount of crude sample with hot water.
13. We determine the melting point of the recrystallized product.

Reactions involved:



benzoic acid

Oxidation mechanism by KMnO_4



Experiment - 2

AIM

separation of amino acids from their mixture by paper chromatographic technique.

PRINCIPLE

Paper chromatography is an inexpensive and powerful analytical technique, which requires a piece of paper or strips serving as an adsorbent in the stationary phase across which a particular solution is allowed to pass. One phase is the stationary, which is held in the pores of the filter paper used and other is the mobile phase which moves over the paper.

Capillary action: the movement of liquid within the spaces of a porous material due to the forces of adhesion, cohesion, and surface tension. The liquid is able to move up the filter paper because its attraction to itself is stronger than the force of gravity.

Solubility : The degree to which a material (solute) dissolves into a solvent. Solute dissolves into solvents that have similar properties. This allows different solutes to be prepared by different combinations of solvents.

separation of components depends on both their solubility in the mobile phase and their differential affinity to the mobile phase and the stationary phase.

$R_f = \frac{\text{distance travelled by the compound}}{\text{distance traveled by the solvent front}}$

Retention factor is inversely proportional to polarity in this case.

CHEMICALS REQUIRED

- Amino acids (L-Lysine, L-Alanine, L-Leucine)
- Acetic acid
- 1-Butanol
- Distilled water
- Spraying agent : Ninhydrin (0.3% solution in rectified spirit)

APPARATUS

- Measuring cylinder
- Test tubes
- Electric air oven
- Solvent chamber (20 cm x 4 cm)
- Sprayer
- Whatman No. 1 chromatography paper (20 cm x 4 cm)

PROCEDURE

- 1) Preparation of solutions (10-15 mg of each amino acid is mixed and dissolved in 1 mL distilled water) and unknown amino acid in a separate test tube.
- 2) The chromatographic paper strip is taken and a base line is drawn above 4 cm from the lower end. Spots are given with the help of the capillary tubes.
- 3) Developing solvent - Butanol : AcOH : H₂O = 12 : 3 : 5 and poured into the jar before 1 hr. of developing and lid is placed properly.

- 4) Development of the chromatogram.
- 5) Drying in the oven
- 6) Spraying
- 7) Location of the spots with pencil.

CALCULATIONS :

The movement of any substance relative to the solvent front in a chromatographic system is Constant. In paper chromatography it is defined as, RF value - Distance moved by the substance / distance moved by the solvent front.

After completion, we get a figure such a given in fig. 1.

Polarity order :

L-lysine > L-alanine > L-leucine

R_f order :

L-leucine > L-alanine > L-lysine

α-amino acids

These are carboxylic acids which contain an amino group at the α position.



Chromatography is a method of separating mixtures based on their solubility and adsorption properties.

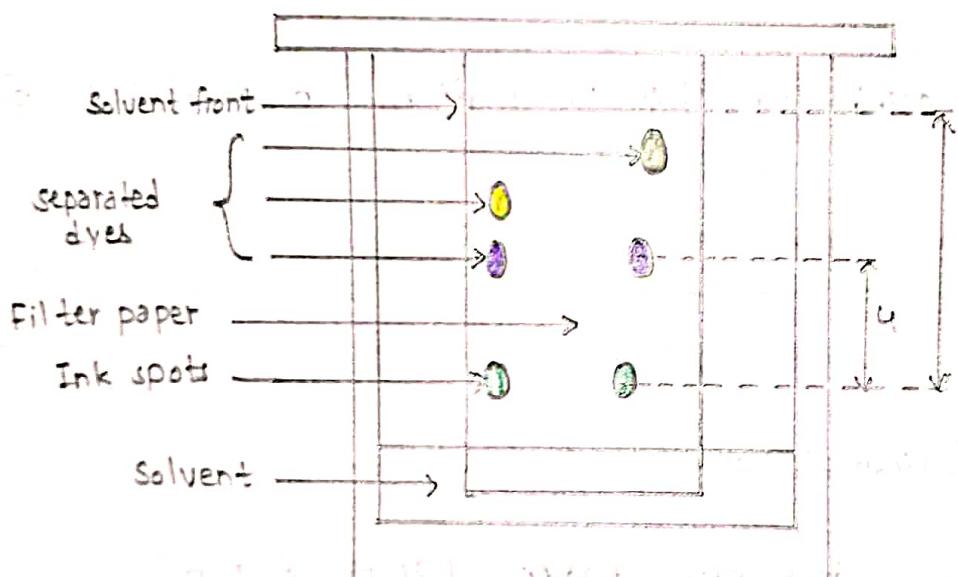
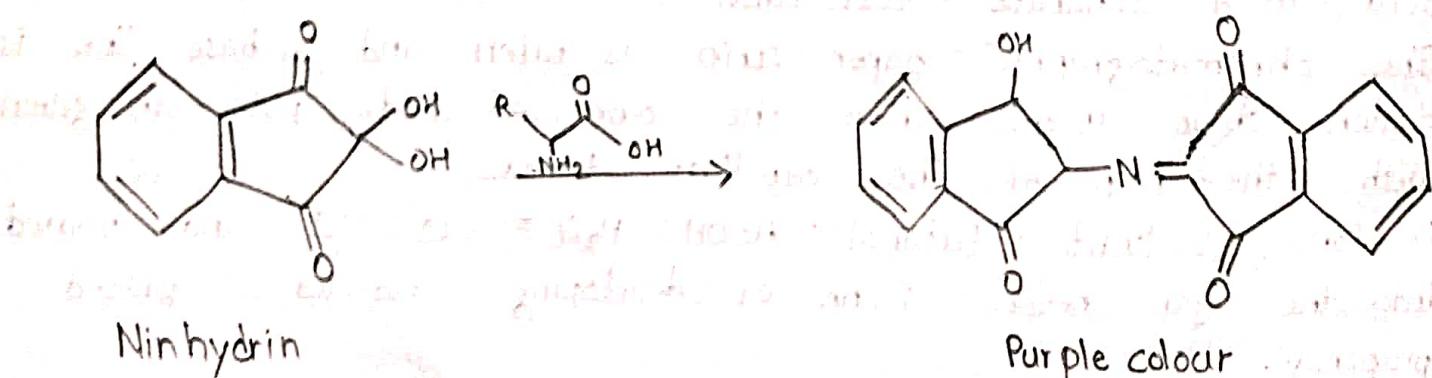


Fig. 1

Chemical Reaction:



INORGANIC CHEMISTRY

EXPERIMENT No. 1

Estimation of ferrous ion (Fe^{2+}) in a given solution by permanganometry

Chemicals required

1. Oxalic acid
2. KMnO_4
3. Mohor salt solution
4. H_2SO_4
5. Phosphoric acid

Apparatus required

1. Conical flask
2. Burette
3. Pipette
4. Glass rod

Procedure

STEP-1: Standardisation of KMnO_4 Solution by standard (N/10)
Oxalic acid solution :

Take 10ml of (N/10) Oxalic acid solution in a conical flask with the help of pipette and add 15ml of 1:4 H₂SO₄ solution and 50ml (approx.) of distilled water, heat the mixture just to boiling (i.e. very hot state but not boiling) and then titrate the mixture in warm condition against KMnO₄ solution till the colour changes from colourless to pink. Take the burette reading and repeat the experiment after washing the conical flask properly.

Observation Table:

No. of observation	Vol. of oxalic acid taken (ml)	Vol. of KMnO ₄ sol. Required	Average vol. of KMnO ₄
1.	10	10	<u>10 + 10 + 10 = 30</u>
2.	10	10	3
3.	10	10	

Calculation :

$$V_1 S_1 = V_2 S_2$$

V₁ = vol. of oxalic acid (10 ml)

V₂ = Average vol. of KMnO₄ sol.

S₁ = Strength of Oxalic acid
= 0.1 N (N/10)

S₂ = Strength of KMnO₄ sol. in,

$$N = \frac{10 \times 0.1}{10} = 0.1 N$$

STEP-2: Estimation of Fe^{+2} in a given sample

Sample No.:

Write down the Sample no. from the volumetric flask body of the given solution.

Add distilled water within the volumetric flask containing ferrous ion sample and make up the volume to 100 ml, shake the solution well and make ~~exp~~ the solution homogeneous.

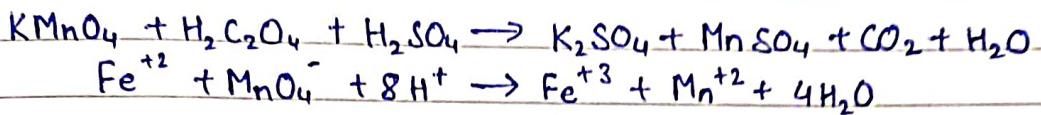
Take 10 ml of this diluted Fe^{+2} solution with the help of pipette (after cleaning it properly) in a conical flask, add 15 ml of 1:4 H_2SO_4 and add 2-3 ml of glacial Phosphoric acid and titrate the mixture against KMnO_4 , till pink colour appears.

Note the burette reading and repeat the experiment

Observation table:

No. of Observation	Vol. of Fe^{+2} (ml)	Vol. of KMnO_4	Average vol. of $\text{KMnO}_4 (V_3)$
1	10	10	<u>$10 + 10 + 10 = 30$</u>
2	10	10	3
3	10	10	

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Reactions :Calculation :

Equivalent weight of Fe^{2+} = 56

Thus, 1000 mL of 1N KMnO_4 solution = 56 g of Fe

Hence, Amount of Fe^{2+} in 10 mL of dil. sol. = $(V_3 \times S_2 \times 0.056)$ g

Amount of Fe^{2+} in a given sample (i.e. total 100 mL) = $(V_3 \times S_2 \times 0.56)$ g

Normality of KMnO_4 solution = 0.1

Volume of KMnO_4 solution = 10

We know, 1000 mL of KMnO_4 = 0.005585 g Fe^{2+}

Therefore, amount of Fe^{2+} in 10 mL KMnO_4 sol. (N_2) = 0.00005585 g

Amount of Fe^{2+} in 250 mL sol. of Mohr's salt = 0.00139625 g of Fe^{2+}

250 mL Mohr's salt contains = 0.00139625 g

1 mL Mohr's salt contains = 0.000005585 g

1000 mL Mohr's salt contains = 0.005585 g

Result :

The strength of Fe^{2+} in the Mohr's salt solution is 0.005585 g/L

EXPERIMENT NO. 2

Estimation of total hardness of water sample by complexometric EDTA titration

Chemicals Required

1. 100 ml tap water
2. pH = 10 buffer solution (Dissolve 17.5 g of A.R. NH_4Li in 142 ml concentrated NH_3 (sp. gr. 0.88-0.90) and dilute it to 250 mL)
3. EBT indicator solution (0.4% methanolic solution)
4. 0.01 (M) di-sodium salt of EDTA

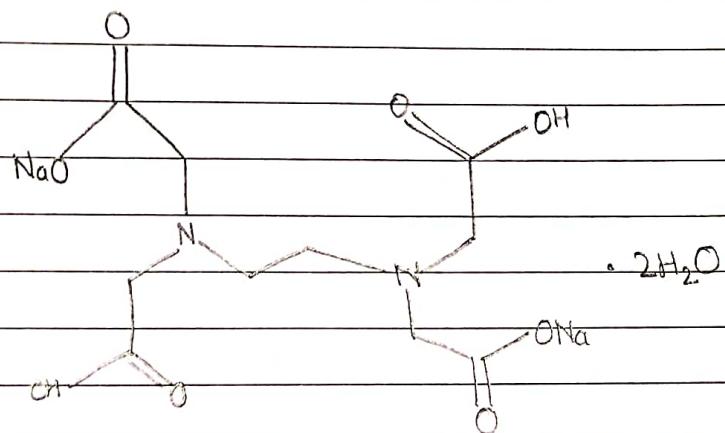
Apparatus Required

1. Conical flask (250 mL)
2. Burette
3. Pipette
4. Glass rod

Theory

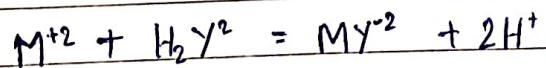
The hardness of water is due to the presence of dissolved calcium and magnesium salts. Disodium

Ves versenate dihydrate (m.w = 372.24), the disodium salt of versine or EDTA (Ethylene diamine tetra - acetic acid), is used to estimate the hardness of water. The structure of the salt is given below:



EDTA has four or six atoms which available for the formation of co-ordination bonds with a metal cation in such a way tha a stable 1:1 co-ordination complex is formed.

The reaction between a metal ion, M^{+2} with EDTA, (H_2Y^{2-}) is represented below:

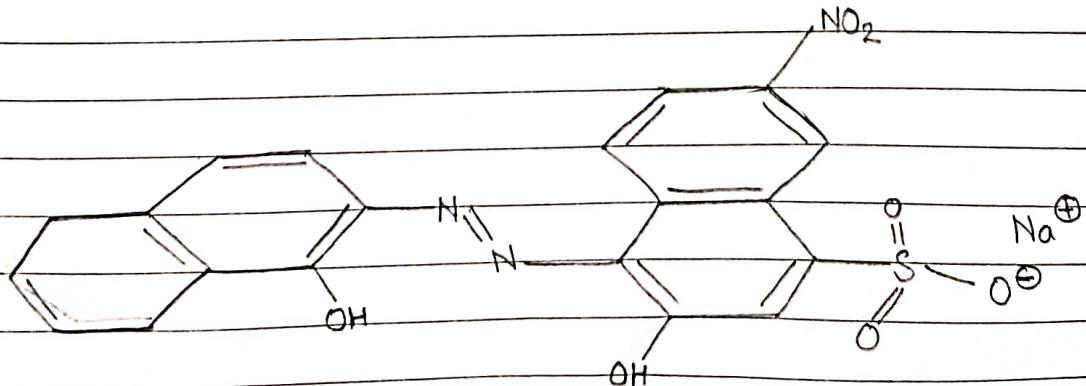


The reaction shows that one gm-ion of the complex

compound forming $\text{H}_2\gamma^2$ reacts in all cases with one gm-ion of the metal ion and in each case two gm-ions of H^+ are produced.

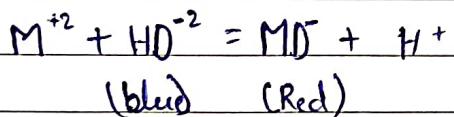
The sample solution (containing the metal ion that is to be estimated) is to be buffered to a desired pH = 10. It is then titrated directly with 0.01 M EDTA solution using Eriochrome Black-T (EBT) indicator. The colour of the solution changes from wine / purple-red to pure blue.

Eriochrome Black-T (EBT) is Sodium-1-(1-hydroxy-2-naphthylazo)-6-nitro-2-naphthal-4-sulphonate. This is also known as Solochrome Black-T or WDFA. In strongly acidic solution this azo-dye tends to polymerise to a red brown product, and consequently the indicator is rarely applied in the EDTA titration of solutions more acidic than pH = 6.5. The structure of the indicator is shown below:

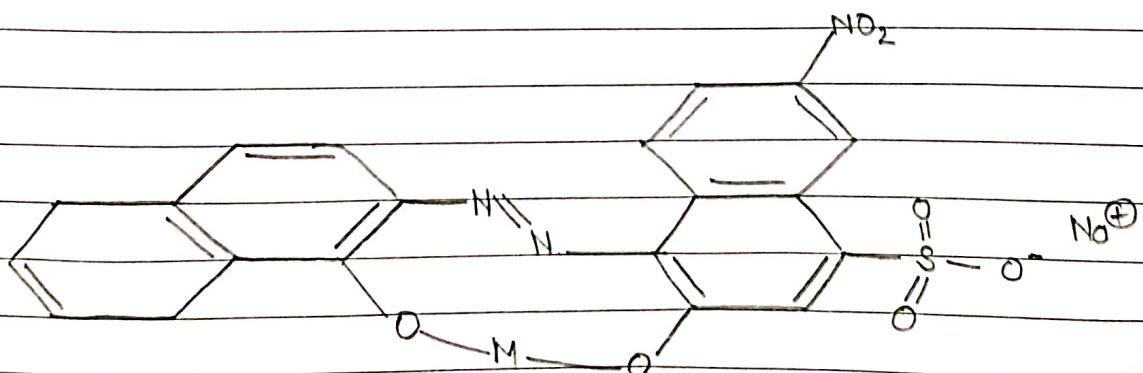


The sulphonic acid group gives up its proton long before the pH range of 7-12, which is of immediate interest for metal - ion indicator use. Only the dissociation of the phenolic H - atoms need be considered, and so the dyestuff may be represented by H_2D . The two - pK values of these two H-atoms are 6.3 and 11.5 respectively. Below $\text{pH} = 5.5$, the solution of Eriochrome Black T is red due to H_2D^- , between $\text{pH} 7$ and 11 it is blue due to HD^{-2} and above $\text{pH} = 11.5$ it is yellowish - orange due to D^{-3} .

In the pH range 7-11, the addition of metallic salts produces a brilliant change in colour from blue to red.



The structure of metal EBT complex is shown below



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Procedure

Part - I: Estimation of hardness of water using EDTA solution.

Accurately 100-ml tap water is taken in a conical flask using a measuring cylinder. Approx. 10 drops of ammonia - buffer solution ($\text{pH} = 10$) is added to the conical flask such that the smell of ammonia becomes persistent.

1 drops of EBT indicator is added to the conical flask. A 50 - ml burette is filled up to the zero mark with 0.01 M EDTA solution is added drop - wise to the conical flask with constant swirling during the titration till the colour change from wine / purple - red to blue. The titration is repeated twice to obtain concordant values. The readings are given in table 1.

Results & Discussions

Table 1 : Estimation of hardness of water using EDTA solution

Sl. NO.	Vol. of hard water sample(mL)	Burette Reading Initial	Burette Reading Final	Vol. of EDTA consumed (mL)	Mean vol. of EDTA (V mL)	Hardness of water sample (ppm)
1	25	0.0	7.1	7.1	7.1	300
2	25	0.0	7.0	7.0	7.1	996
3	25	0.0	7.2	7.2	7.1	300

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Calculations :

1000 mL of 1(M) EDTA solution = 100 g of CaCO_3

1 mL of 0.01M EDTA solution = 1 mg of CaCO_3

V mL of 0.01M EDTA solution = V mg of CaCO_3

7.1 mL of 0.0350 M EDTA = $\frac{7.1 \times 0.35 \times 100}{1000}$ of CaCO_3

$$= 0.0249 \text{ g of } \text{CaCO}_3$$

Now since 25 mL of Hard water contains 0.024 g of CaCO_3
eq of Hardness.

So, 10^6 cm^3 of Hard water contains 0.024×10^6 g of CaCO_3 eq hardness.

\therefore Total Hardness in given water sample $10V = \frac{0.0249 \times 10^6}{25} = 996 \text{ ppm}$

Result :

The Hardness of the water sample is 996 ppm.

Conclusion :

1. The total hardness of tap water tested is in the range of 100 mg/l - 150 mg/l in terms of CaCO_3 equivalent.
2. EDTA is stronger ligand than EBT towards the bivalent metal cations e.g. Ca^{2+} & Mg^{2+}

PHYSICAL CHEMISTRY

EXPERIMENT NO: 1

Aim : To determine the strength of HCl solution by conductometric titration with NaOH solution

Theory:

Conductometry is a measurement of electrolytic conductivity to monitor a progress of chemical reaction. Conductometry has notable application in analytical chemistry, where conductometric titration is a standard technique.

Electrolytic conductivity is a measure of the ability of a solution to carry electric current. Electric solutions conduct electric current by the migration of ions under the influence of electric field. According to Ohm's law, the current strength (I) flowing through a conductor is directly proportional to the potential difference (E) and inversely proportional to the resistance (R) of the conductor.

$$\text{ie. } I = E/R \text{ or, } R = E/I$$

Where the resistance (R) is the hindrance provided by the solution. The resistance of any conductor varies directly with the length and inversely with its area of cross-section.

$$R = \rho \times (l/a)$$

Where, ρ is the specific resistance and it is the resistance of a unit length of conductor of unit

Cross-section (l/a) is called cell constant.

The reciprocal of specific resistance is called specific conductance or conductivity.

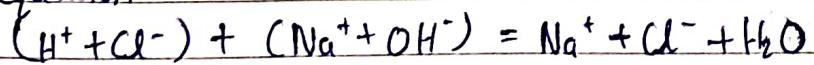
Specific conductance $(k) = \frac{1}{\rho} = \frac{l}{R} \times \frac{A}{l} = \frac{A}{R}$ with unit $\text{ohm}^{-1} \text{cm}^{-1}$.

Conductance of electrolyte depends upon i) no. of free ions.
ii) Charges on the free ions and
iii) mobility of the ions on the substitution of one ion by another of different mobility (speed of ions).

So, conductometric method can be used to determine the end point of ionic titration like :

- i) Acidimetric titration
- ii) Precipitation titration
- iii) Titration involving the formation of complex ion.

Hence there is a net change in conductance of a solution during titration. When a strong acid (e.g. HCl) is titrated against a strong base (e.g. NaOH), the neutralization reaction is the replacement of H^+ ions by an equal number of Na^+ ions, according to the equation :



Initially the conductance of a solution of HCl remains very high due to presence of highly mobile H^+ ions.

But with addition of NaOH, the conductance gradually decreases along a straight line. Since H⁺ ions combine with OH⁻ ions to form undissociated water, the faster moving H⁺ ions are replaced by relatively slower moving Na⁺ ions in aqueous solution. In this way, when all the H⁺ ions have been removed, the conductance will reach its lowest limit. Further addition of NaOH will cause the solution to have an excess of the fast moving OH⁻ ions with the result that its conductance increases linearly with the addition of excess NaOH.

Therefore the nature of the plot (conductance of the solution versus volume of base added) will be as given below (figure 1.1)

Materials :

Apparatus : Conductometer, conductivity cell, beaker, pipette, conical flask.

Chemicals : Hydrochloric acid (HCl), sodium hydroxide (NaOH), conductivity water.

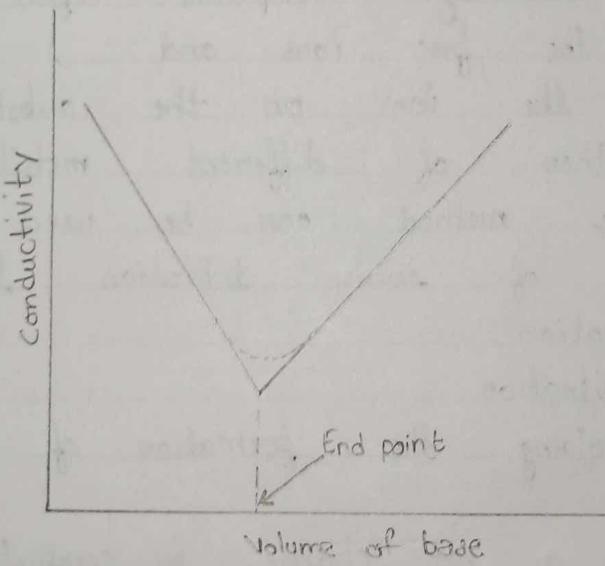


fig (1.1)

Procedure :

1. HCl solution of unknown strength {approx. $(1/200) N$ } is provided.
2. Standardized 0.1 (N) NaOH solution is provided.
Strength of NaOH should be at least 10 times higher than that of HCl, to avoid any volume effect on conductometric measurement.
3. Calibration of the instrument done at room temp.
4. Conductometric Titration :
 - i) Rinse the conductivity cell a number of times with conductivity water or double distilled water.
 - ii) Pipette out 150 mL of approx. ($N/200$) HCl in a beaker and dip the conductivity cell in it, so that the cell should dip completely in solution.
 - iii) Rinse the burette with NaOH solution and fill with it
 - iv) Add small amount of NaOH (at an interval of 0.5 mL) from burette, stir it, give time to settle down and measure the conductance after each addition.
 - v) Go on adding NaOH (at an interval of 0.5 mL) and measure conductivity value. Initially, conductivity will increase, then it will show a rise. This indicates the attainment of end point.

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vi) Take at least five readings beyond the end point.

Observation:

1. Temperature of the experiment:

2. Table 2: Conductometric titration :

Observation	Volume of HCl taken (V ₁) ml	Total volume of drops of NaOH solution added (ml)	Conduction (ms) S in (Ohm) ⁻¹
1.	150	0	4.32
2.	150	0.5	4.13
3.	150	1	4
4.	150	1.5	3.88
5.	150	2	3.75
6.	150	2.5	3.6
7.	150	3	3.48
8.	150	3.5	3.35
9.	150	4	3.2
10.	150	4.5	3.12
11.	150	5	3
12.	150	5.5	2.87
13.	150	6	2.7
14.	150	6.5	2.6

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15.	150	7	2.45
16.	150	7.5	2.3
17.	150	8	2.4
18.	150	8.5	2.55
19.	150	9	2.7
20.	150	9.5	2.83
21.	150	10	3
22.	150	10.5	3.2
23.	150	11	3.37
24.	150	11.5	3.55
25.	150	12	3.72
26.	150	12.5	3.95
27.	150	13	4.2

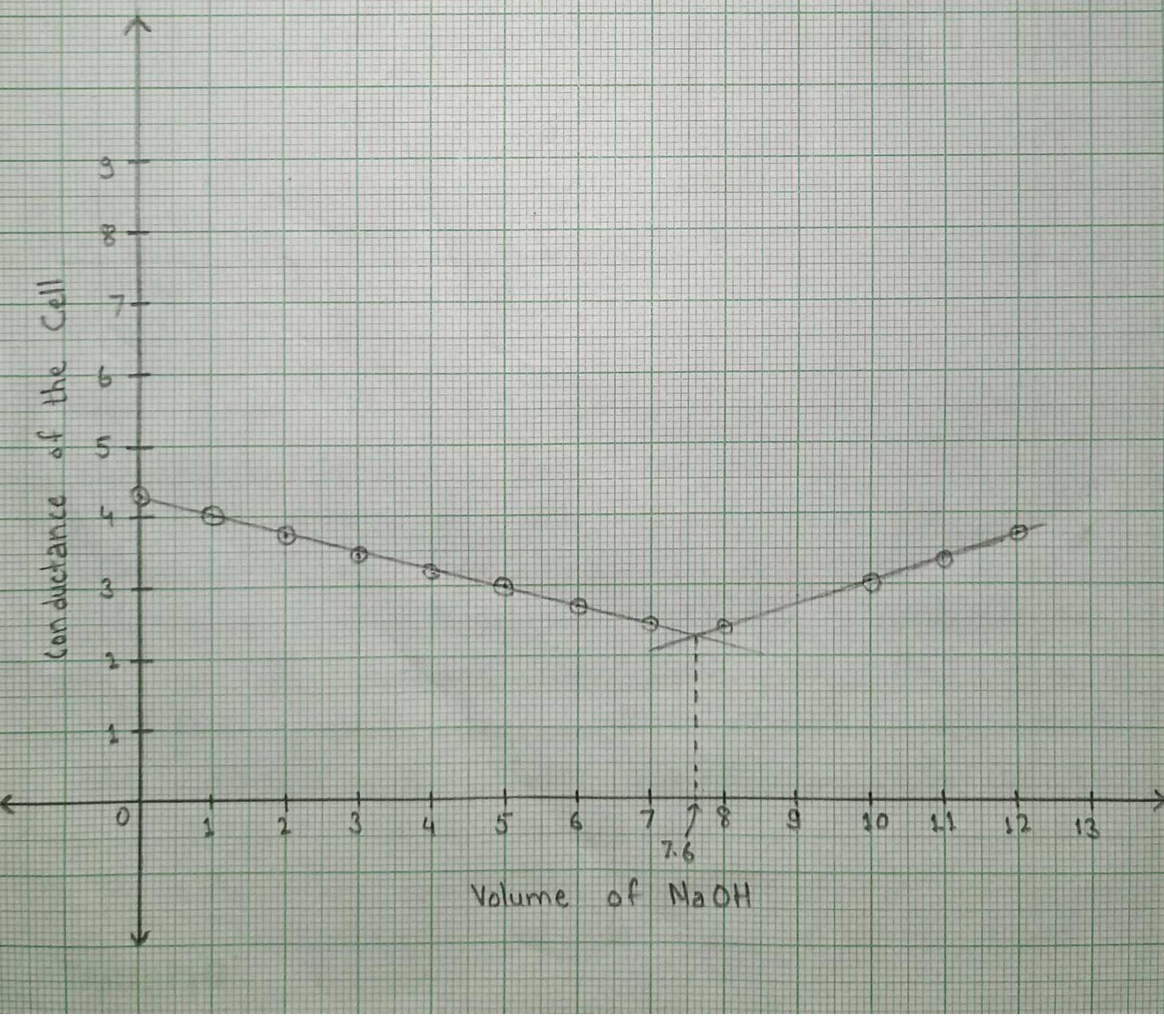
Plot a graph between conductance and volume of titrant (NaOH solution). Two intersecting lines will be obtained (as given in the figure 1) and the points of intersection of these lines represent the equivalent point (volume of NaOH required for neutralization).

Let, V_e be the volume of NaOH at the equivalent point (from graph) and the strength of acid is S_1 and strength of NaOH solution is $S_2 = 0.1 \text{ (N)}$.

Then, $V_e \times S_1 = V_2 \times S_2$ Here, $V_2 = 7.5$

$$\text{So, } S_1 = (V_2 \times S_2) / V_e = (7.5 \times 0.1) / 150 = 0.005$$

Conclusion : The strength of the acid (S_1) is 0.005 (N).



Discussions :

- i) Normally, the coloured solution which cannot be titrated with volumetric method using indicator can be titrated by the conductometric method.
- ii) The conductometric titration method can be used in case of weak acid vs. weak base and also in case of very dilute solutions.
- iii) Near the end point, no special care is necessary as it is determined graphically.

Precautions :

- i) Electrical connection should be made carefully.
- ii) Temperature during the experiment should be kept constant as conductance depends on temperature.
- iii) Stirring should be done after each addition of titrant.
- iv) To avoid the dilution effect, the concentration of the titrant should be 10 times more than that of the solution to be titrated.

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Experiment - 2

AIM:

Verification of Beer-Lambert Law and determination of amount of iron present in a supplied solution spectrophotometrically.

Theory:

When the light of a particular wavelength passes through transparent medium, intensity of the transmitted light of the same wavelength generally decreases, as the medium absorb some amount of light.

Absorbance (A) is defined as

$$A = \log_{10} \frac{I_0}{I}$$

where, I_0 is the intensity of the incident light and I is that of the transmitted light of same wavelength.

Absorbance is proportional to the concentration of the light absorbing medium (sample) (c), as well as path length (l), which is equal to the width of the cuvette (in which the sample of solution is taken).

Thus we can write,

$$A = \epsilon cl$$

where, ϵ is the proportionality constant known as molar adsorption coefficient, or molar ad absorptivity, with unit of $L \text{ mol}^{-1} \text{ cm}^{-1}$.

This equation is known as Beer-Lambert Law.

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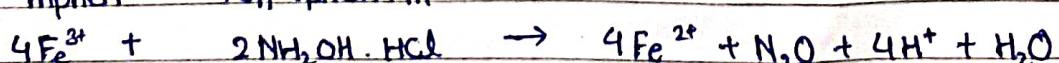
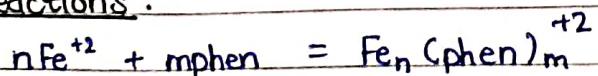
Iron present in aqueous samples can be determined spectrophotometrically by complexation with a suitable complexing agent. The absorbance of the metal-ligand complex is usually measured in the visible region and is related to metal ion concentration.

Colorimetric determination of iron can be done using several known complexing agents. Among the routinely used is 1,10-phenanthroline (phen) which reacts with Fe^{3+} to form an orange-red complex in the pH range from 2-9. Therefore, the first step involves the reduction of any Fe^{3+} present to Fe^{2+} using hydroxylamine hydrochloride.

The procedure depends on the construction of a calibration curve from standard Fe^{2+} , followed by measurement of the unknown Fe^{2+} concentration from the curve.

The first step of an analytical spectrophotometric procedure for quantitative determination of analytes is to find the wavelength at which analyte complex has maximum absorption (it will be around 510 nm). At this wavelength, the molar absorptivity is a maximum and precision is greater. This allows for more precise and sensitive determination. The absorption spectra of the iron-phen colored complex solutions with known ferrous ion concentrations are to be determined first. With those data, we will have to construct the calibration curve. A linear calibration curve will prove the Beer-Lambert law.

Reactions:



Apparatus :

1. A spectrophotometer
2. Sample cells or cuvettes

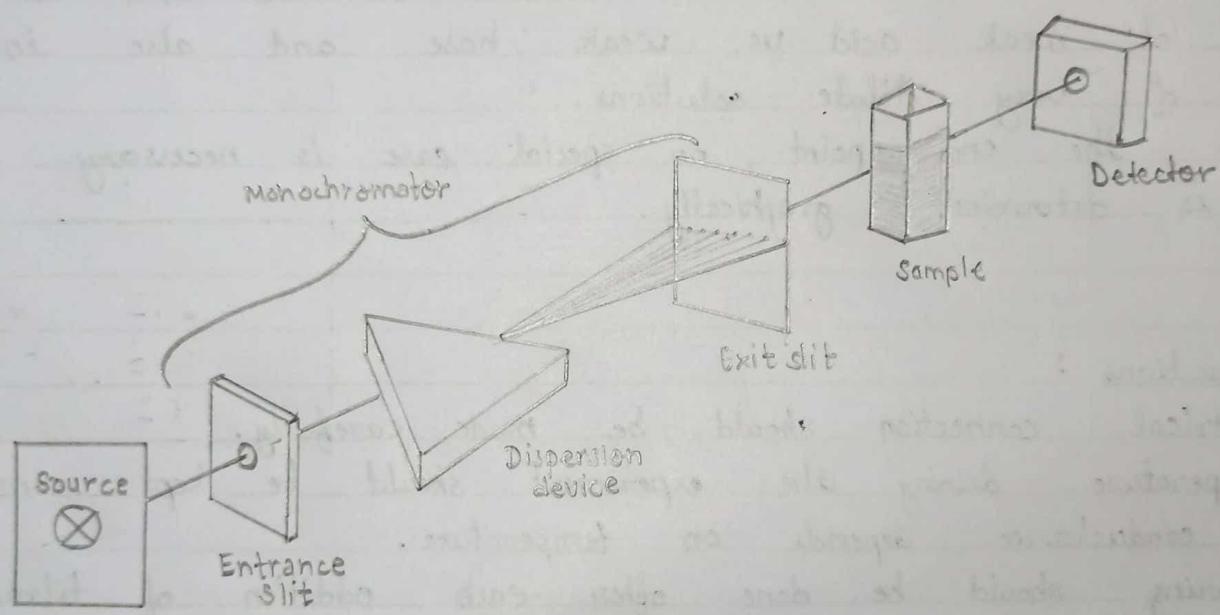
Chemical reagents :

1. Stock phenanthrolin solution : ($M_r = 180.21$) Dissolve 2.50 g in 100 mL of ethanol and complete to 1.0 L with dist. water. Store the solution in an amber bottle.
2. 0.5 M Hydroxylamine hydrochloride:
3. Standard 0.1 mM Fe^{+2} solution: Prepared by dissolving 0.0392 g of reagent-grade $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \times 6\text{H}_2\text{O}$ (ferrous ammonium sulphate hexahydrate, $M_r = 392.14$) in water in a 1-L volumetric flask containing 1 mL of H_2SO_4 98% (w/w)

Procedure :

1. Accurately transfer 0 (blank), 1, 2, 3, 4. and 5 mL of standard Fe^{+2} solution (solution 3) into separate 100 mL volumetric flask.
2. To each solution in step 1 add 2 mL hydroxylamine hydrochloride solution.
3. Add 1 mL phenanthrolin solution to each volumetric flask.
4. Complete to mark with distilled water and shake well.
5. Allow 10 min. for colour development.
6. Place the blank in the absorption cells provided and make the absorbance value zero (transmittance 100%) reading at 510 nm.
7. Remove one blank in the absorption cells provided and
8. Remove one blank cell and place one coloured sample in the cell and put in its proper place and take the absorbance reading at 510 nm.

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Spectrometer

a. Measure the absorbance of each solution at the $\lambda_{max} = 510 \text{ nm}$ and draw the calibration curve (Absorbance vs actual concentration of Fe^{2+}) to verify L-B law.

With unknown supplied sample:

1. Take the supplied solution in 100 ml volumetric flask, repeat the step 2-5. Measure the absorbance at 510 nm.
2. From the calibration curve determine the concentration of Fe^{2+} .

Calculation and discussion:

Table:1

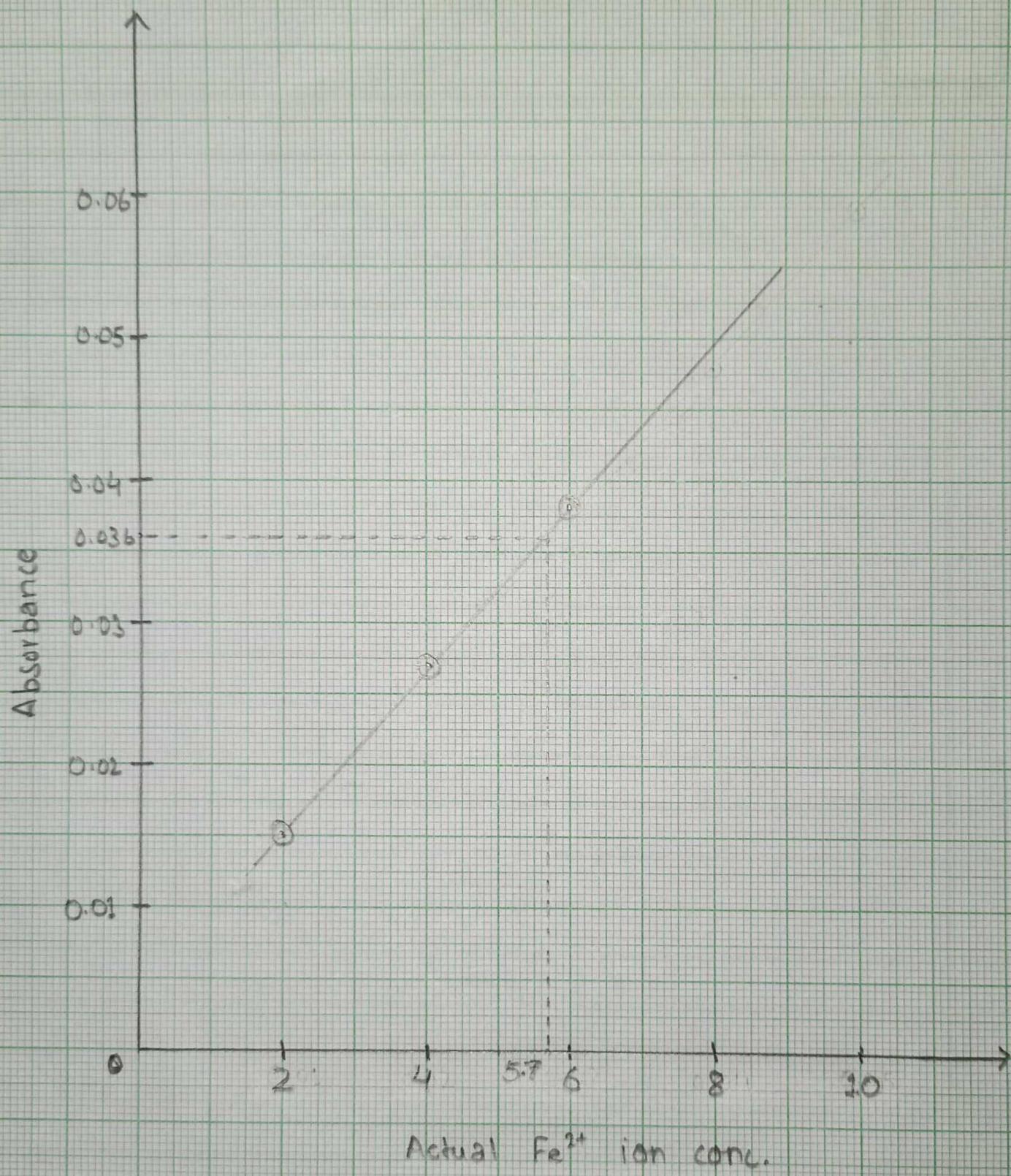
Flask no.	Vol. of 10^{-4} M Mohr Salt sol' taken (ml)	Actual conc. of Fe^{2+} solution in 100 mL sol. (i.e. $V_1 S_1 = V_2 S_2$ calculation)	OD at 510 nm	Absorbance at 510 nm
1.	1	2	0.010	0.015
2.	2	4	0.022	0.027
3.	3	6	0.033	0.038
4.	4	8	0.043	0.048
5.	5	10	0.054	0.059

(OD values are corrected for instrumental artifacts)

We plot the absorbance versus actual concentration of the known solutions (i.e., conc. of each five number of Fe^{2+} solutions prepared in step 1)

We draw the best fitting straight line through the points - this called the Beer-Lambert Law plot.

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Actual Fe^{2+} ion conc.

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We place the best absorbance value of the unknown solution onto this plot and determine its concentrations.
Hence beer lambert law is verified.

Conclusion:

From the graph we can see that concentrations and optical density are in the same straight line. So that we can say this follows the Beer - Lambert Law.

From the graph we can see that if we take the value of $OD = 0.035964$ the concentration of Fe^{+2} will be $3.33 \times 10^{-6} M$

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