

Aim -

Determination of total hardness of water sample by EDTA method

Requirements -

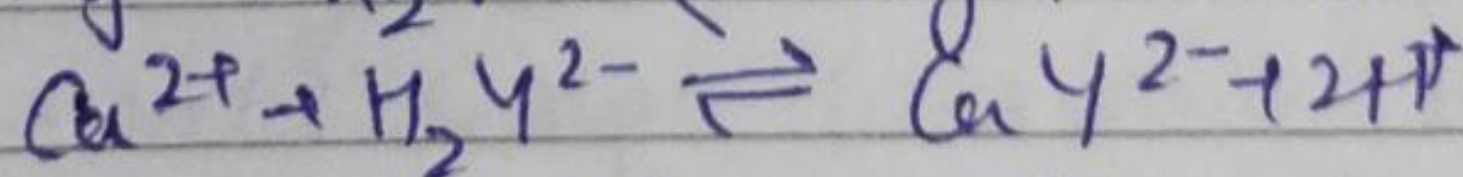
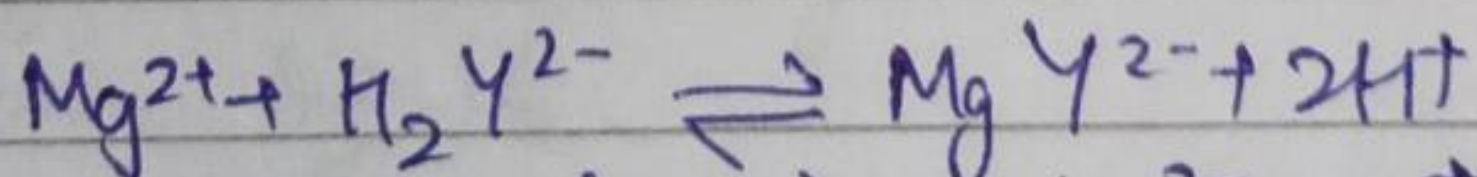
apparatus - Volumetric flask (100 mL), burette (50 mL), pipette (10 mL), weighing bottle, conical flask (100 mL), funnel

Reagents - EDTA soln, ammonia-ammonium chloride buffer (pH = 10), Eriochrome black T (EBT) Indicator

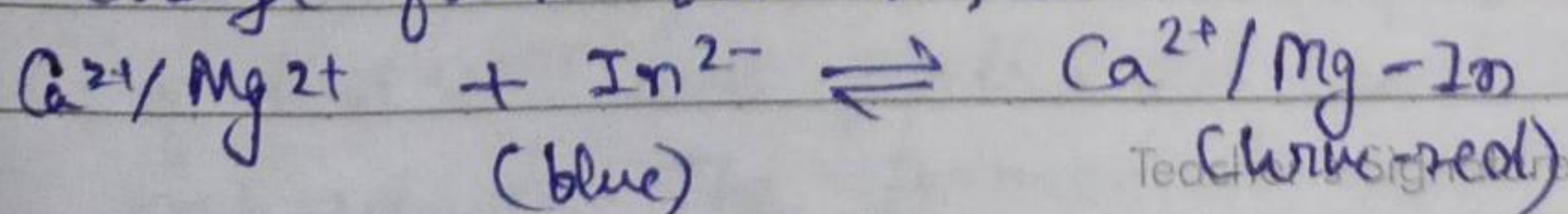
Principle - hardness can be estimated by complexometric titration using EDTA, EBT as indicator. EBT when added to water forms an unstable wine-red colored complex with calcium and Mg. As the soln is titrated with EDTA, free  $Mg^{2+}$  &  $Ca^{2+}$  present in water forms stable colourless complex with EDTA. Once all  $Mg^{2+}$  &  $Ca^{2+}$  are complexed, EDTA replaces  $Mg^{2+}$  &  $Ca^{2+}$  complexed with EBT in the unstable wine red complex. Free EBT imparts blue colour to soln. Wine red to blue colour change indicates end point.

Theory -

EDTA can combine with metal ion in 1:1 ratio chelate structure



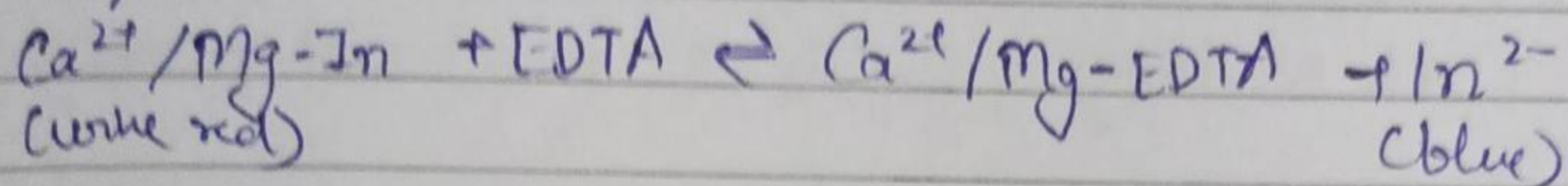
EBT is a metal ion indicator, addition of metal produces colour change from blue to red.



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At the end point, no more free metal ions are present in the solution, the free indicator is liberated hence colour changes from red to blue



### Procedure -

1. The standard solution of EDTA was prepared by dissolving known amount of EDTA (4-6g) in 100-150 mL of deionized water and 4 mL NaOH and then making up the volume in a 250 mL volumetric flask.
2. A clean burette is washed with distilled water and then rinsed with small amount of standard EDTA sol<sup>n</sup>. It is then filled with standard EDTA sol<sup>n</sup> upto the zero level ensuring that no air bubbles are formed.
3. ~~This~~ In a clean conical flask 25 mL of standard <sup>hard</sup> water was taken, 5-6 drops of EBT indicator was added with 3 mL of buffer. The color of the sol<sup>n</sup> turns wine red. Titrate the sol<sup>n</sup> against EDTA sol<sup>n</sup> from burette, till the color of sol<sup>n</sup> turned blue. Take concordant readings were taken.
4. About 25 mL of given sample was of hard water was taken in another clean conical flask and 3 mL of buffer sol<sup>n</sup> followed by 5-6 drops of EBT indicator is added to it. Titrate similarly to find volume of EDTA used by unknown hard water sample. Let the EDTA volume be  $V_2$ .
5. 100 mL of hard water sample was taken in a beaker and boil gently for an hour. Cool and filter the water and collect in a standard 100 mL flask and make up the volume to 100 mL. 25 mL of this sol<sup>n</sup> was pipetted out and 3 mL of buffer with EBT was added. The permanent hardness of water was calculated. Let volume of EDTA be  $V_3$ .



### Result-

Total hardness of water = \_\_\_\_\_ ppm  
Permanent hardness = \_\_\_\_\_ ppm  
Temporary hardness = \_\_\_\_\_ ppm

### Precautions-

- 1/ The sharp color change at the end point using EBT as indicator is observed if both Mg and Ca ions are present in the sol<sup>n</sup> of hard water. In the absence of Mg ions the color change at end pt is not sharp.
- 2/ The pH for the reaction mixture during titration should be maintained between 8 and 10. At lower pH, the bonding between Mg ions and the indicator is weak so the red colour of complex b/w them is not maintained. At higher ~~pH~~ pH the Mg ions are precipitated as  $Mg(OH)_2$ .
- 3/ The buffer sol<sup>n</sup> should not be pipetted out with the mouth because it may affect the normal pH of blood which is 7.4.

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### Observations -

a) Titration of standard hard water with EDTA) Standardisation of EDTA soln.

Sno.	Volume of std hard water (mL)	Initial reading (x mL)	Final reading (y mL)	Vol of EDTA ( $V_1 = y - x$ )
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1

2

3



## Calculation-

i) Standardisation of EDTA sol<sup>n</sup> (Titration of std hard water with EDTA)

Vol of hard water = 25 mL

Vol of EDTA used =  $V_1$  mL

$V_1$  mL of EDTA = 25 mg of  $\text{CaCO}_3$

1 mL of EDTA =  $\frac{25}{V_1}$  mg of  $\text{CaCO}_3$

ii) Calculation of total hardness of water-

Volume of hard water = 25 mL

Volume of EDTA for titration =  $V_2$  mL

25 mL of given hard water =  $\frac{25}{V_1} V_2$  mg of  $\text{CaCO}_3$

1000 mL of given hard water contains =  $\frac{25}{V_1} \times V_2 \times \frac{1000}{25}$  mg of  $\text{CaCO}_3$   
 $= \frac{V_2}{V_1} \times 1000$  mg of  $\text{CaCO}_3$

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

(iii) Calculation of Permanent hardness of water sample

Vol of hard water = 25 mL

Vol of EDTA =  $V_3$  mL

2500 mL of hard water after boiling =  $\frac{V_3}{V_1} \times 1000$  mg of  $\text{CaCO}_3$

$= \frac{V_3}{V_1} \times 1000$  ppm

(iv) Calculation of Temporary hardness-

Total - Permanent = Temporary hardness



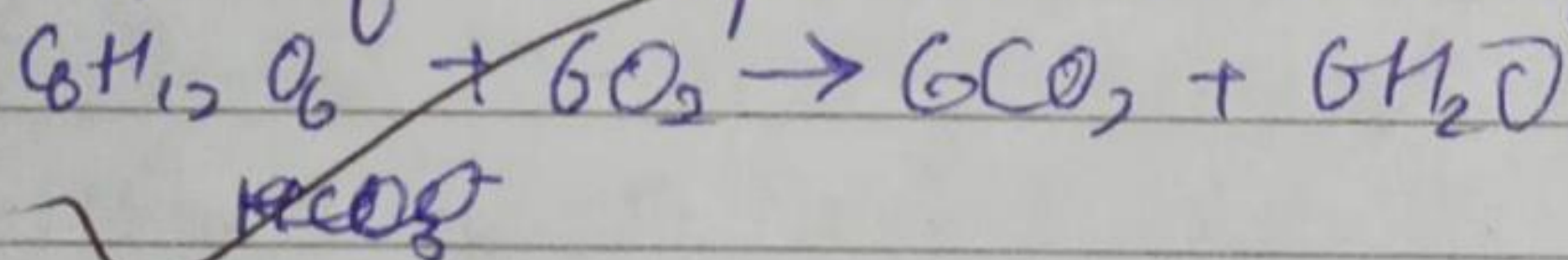
Aim -

To determine the amount of dissolved  $\text{CO}_2$  in a given water sample.

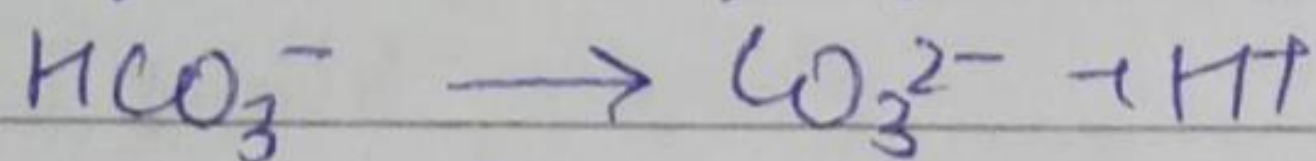
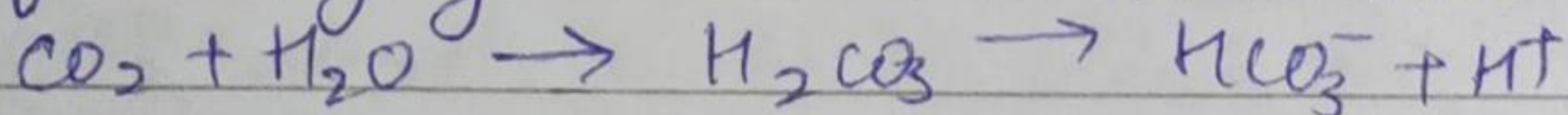
Materials Required - Burette (50 ml), Pipette (10 ml), Measuring flask (80 ml), Conical flask (100 ml), beaker, Funnel,  $\text{NaOH}$  sol<sup>n</sup> (N/40), Standard solution of oxalic acid (N/40), water sample, phenolphthalein indicator.

Theory -

- Dissolved  $\text{CO}_2$  is the amount of carbon dioxide that is present in water.
- Water bodies receive  $\text{CO}_2$  from atmosphere and from the respiratory activity of aquatic organisms. It is also released by the process of decomposition in the aquatic ecosystem.

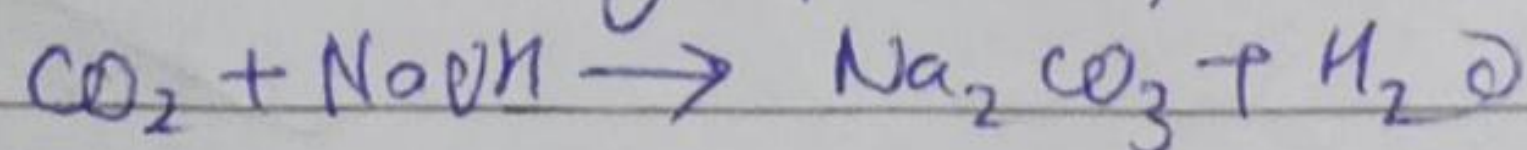


A small fraction of the  $\text{CO}_2$  that dissolves in water reacts rapidly to form carbonic acid. This, in turn, partially dissociates to form hydrogen bicarbonate and carbonate ion.



$\text{CO}_2$  will continue to dissolve until equilibrium is reached.

This free  $\text{CO}_2$  which gets dissolved in water, is determined by titrating std.  $\text{NaOH}$  using phenolphthalein indicator.



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Procedure-

1. Standardization of NaOH solution (N/40) - Standardization of NaOH solution was done using standard solution of oxalic acid. 10 mL of N/40 oxalic acid sol<sup>n</sup> was pipetted out in a conical flask followed by the addition of 2-3 drops of phenolphthalein indicator. Titration was repeated till two concordant readings were obtained.
2. Estimation of dissolved CO<sub>2</sub> in a given water sample - 10 mL of given water sample was pipetted out in a conical flask and 2-3 drops of phenolphthalein indicator were added to it. The water sample was then titrated against the standard NaOH solution. The appearance of pink colour indicated the end point. The burette reading was noted and the titration was repeated till two concordant readings were obtained.

Result-

Amount of dissolved CO<sub>2</sub> in a given sample of water is

= 11.88 mg/L

Precautions-

1. The sol<sup>n</sup> should be well shaken
2. Fixed amount of indicator should be added during each titration
3. End point should be noted properly.

Inference

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## Observation -

1) Standardization of NaOH vs Standard Oxalic acid -  
 Table-1 Titration of NaOH vs D.A (N/10)

S.N.	Volume of std. Oxalic acid Soln (mL)	Burette reading (mL)		Volume of NaOH used (mL)
		Initial	Final	
1	10.10	10.0	19.0	9.0
2	10	10	19	9
3	10	10	19	9
Concordant reading				9.0 mL

Thus, normality of NaOH =  $N_{NaOH} = \frac{N_{OA} \times V_{OA}}{V_{NaOH}} = \frac{10 \times 10}{9} N = 0.0277 N$  ✓

2) Titration of given water sample with standard NaOH -  
 table-2

S.N.	Vol of water sample	Burette reading (mL)		Volume of NaOH used (mL)
		Initial	Final	
1	10	30	30.2	0.2
2	10	30	30.2	0.2
3	10	30	30.2	0.2
Concordant reading				0.2 mL

Thus normality of water sample =  $N_{water} = \frac{N_{NaOH} \times V_{NaOH}}{V_{water}} = 0.0054 N$  ✓

3) Strength of  $CO_2$  in water sample =  $N_{water} \times \text{equivalent wt. of } CO_2$   
 $= N_{water} \times 22 \text{ g/L}$   
 $= N_{water} \times 22 \times 1000 \text{ mg/L or ppm}$   
 $= 0.0054 \times 22$   
 $= 0.1188 \text{ g/L} \times 1000 \text{ (g/L)}$   
 $= 11.88 \text{ ppm}$  ✓



Aim-

Spectrometric analysis of  $\text{KMnO}_4$  sol<sup>n</sup>

Requirement-

Test tube, test tube holder, measuring cylinder, pipette, spectrometer, Erlenmeyer flask,  $\text{KMnO}_4$ , water.

Theory-

- \* A spectrophotometer is an instrument that measure the amount of photons absorbed after it passes through sample solution.
- \* With the spectrometer, the amount of a known chemical substance can also be determined by measuring the intensity of light detected.
- \* Beer-Lambert law - states that there is a linear relationship b/w the absorbance & the concentration of sample. For this reason - Beer's law can only applied when there is a linear relation.

$$A = \epsilon_0 \times l \times c$$

A = absorbance,  $\epsilon_0$  = molar extinction coefficient  
l = is the path length, c = concentration.

Procedure -

1. Preparation of 100ml of a stock standard sol<sup>n</sup> of 0.001 M  $\text{KMnO}_4$ . Accurately weigh 126mg solid  $\text{KMnO}_4$ . Transfer quantitatively to a 100ml volumetric flask & fill the flask with water.
2. Prepare 6 standard in 100ml volumetric flask with conc. of 0.0004 M, 0.0002 M, 0.0001 M, 0.00005 M by diluting.

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- 3 Rinse one of the ~~bevette~~<sup>cuvette</sup> with distilled water & fill it with water. Put the ~~bevette~~<sup>cuvette</sup> in the sample, compare this to reference solution.
- 4 Rinse cuvette once with distilled  $H_2O$  & once with std. sol<sup>n</sup>
- 5 Repeat this procedure for the two cuvettes at  $\lambda = 420, 430, 440, 450 - 600\text{nm}$ . First setting  $A=0$  for the cuvette with water, then measuring  $A$  for cuvette with  $0.001\text{M KMnO}_4$
- 6 Prepare a graph of absorbance  $A$  vs  $\lambda$  & determine  $\lambda_{\text{max}}$
- 7 Set  $\lambda$  at  $550\text{nm}$  place the cuvette with distilled water in the cell compartment
- 8 Measure and record the absorbance of each of 8 in std. sol<sup>n</sup>.
- 9 Draw a Plot  $\lambda$  as conc<sup>n</sup> of  $\lambda$  & y - absorbance at  $\lambda_{\text{max}}$
- 10 Use Beer's Law to calculate  $\epsilon$  for  $\text{KMnO}_4$

Result -

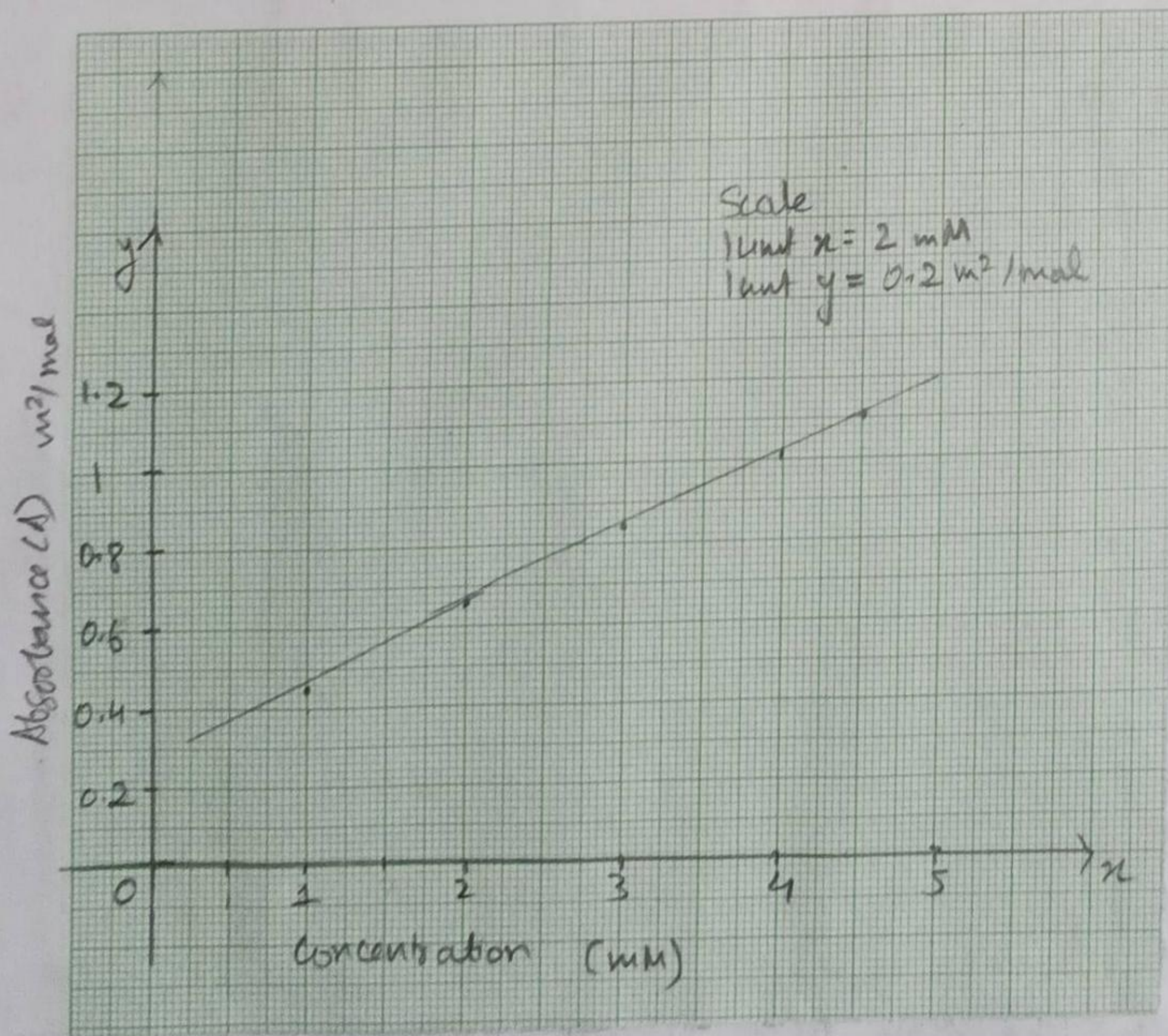
The result of the exp shows greater the conc<sup>n</sup> greater will be the absorbance. The value of absorbance increase on increasing conc<sup>n</sup> at  $\lambda_{\text{max}} = 550$  results  
Verify <sup>Beer's</sup> Lambert's Law.

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## Observation-

Concentration (mM)	Absorbance (A) $\text{m}^2/\text{mol}$
1	0.47
2	0.65
3	0.83
4	1.01
5	1.19





Aim - To determine the electrical conductivity and TDS of a given water sample using conductivity.

Materials Required -

conductivity meter, beakers (50 mL), weighing bottle, standard flask (100 mL), funnel, tissue paper, potassium chloride, distilled water.

Theory -

1. Electrical Conductivity (EC) - conductivity ( $\kappa$ ) is a measure of ability of an aqueous soln to carry an electric current. When an electrical potential difference is placed across a conductor. The its movable charges flow, giving rise to current.
2. Conductance (G) - is defined as the reciprocal of resistance ( $G = 1/R$ ).  
 $G = \kappa \left( \frac{A}{L} \right)$

The units of  $\kappa$  are  $\Omega^{-1}\text{m}^{-1}\text{cm}$  or  $\text{mho}/\text{cm}$ . Conductivity is customarily reported in  $\mu\text{mhos}/\text{cm}$

$$1 \text{ mS}/\text{m} = 10 \mu\text{mhos}/\text{cm}$$

$$1 \mu\text{S}/\text{cm} = 1 \mu\text{mho}/\text{cm}$$

Total Dissolved Solids (TDS) -

- Solid refer to matter suspended or dissolved in water.
- waters with high dissolved solids generally are of inferior ~~palatability~~ palatability & may induce an unfavourable physiological response in transient consumer. A limit of 500 mg dissolved solids/L is desirable for drinking waters.

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**Total Solids** - is the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature.

• **Total Solids** = total suspended solids + total dissolved solids

**Total suspended solids** - The portion of that solids retained by a filter of 2µm.

**Total dissolved solids** - The portion that passes through the filter of 2µm or smaller.

rel<sup>n</sup> b/w TDS & EC :

$$\text{TDS (mg/L)} = 0.6 \text{ EC (}\mu\text{S/cm)}$$

### Procedure

1. **Preparation of KCl sol<sup>n</sup> (0.1N)** - 0.7456 g of KCl was weighed and transferred to the beaker containing distilled water (50ml). The solution was mixed thoroughly with the glass rod until it dissolved completely. The solution is then transferred to the 100ml standard flask and the volume was made up to 100ml by adding distilled water. This solution was used to calibrate the conductivity meter.
2. **Calibration of conductivity meter** - 0.1N KCl solution was taken in a beaker and the probe electrode was immersed into it. The calibration button was selected on the instrument and the instrument was calibrated as conductivity of 0.1N KCl solution as 14.12 mS/cm at 30°C.
3. **Testing of water sample** - After the calibration, the electrodes were rinsed thoroughly with distilled water and carefully wiped with tissue paper. 50ml of water sample was taken in a beaker and electrode was immersed into it for one min for steady reading. The reading observed after



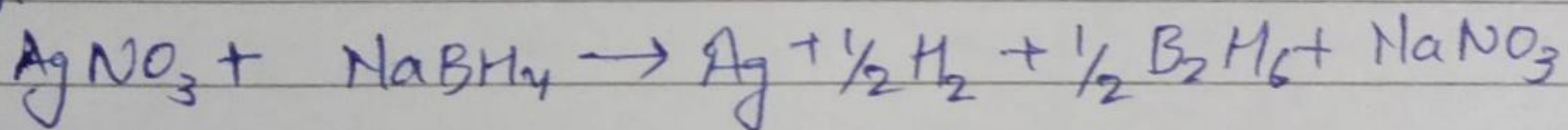
Aim-

To synthesize silver nanoparticles via chemical reduction method and estimate particle size using visible spectroscopy.

Material required - conical flasks (50 mL and 100 mL), ice bath, magnetic stirrer, silver nitrate ( $\text{AgNO}_3$ ), sodium borohydride ( $\text{NaBH}_4$ ), cuvettes, dropper, distilled water, UV-vis spectrophotometer.

Theory-

- Nanotechnology deals with processes that take place on the nanometer scale, that is 1 to 100 nm.
- properties of metal nanoparticles are different from those of bulk materials made from the same atom.
- The synthesis of silver nanoparticles (AgNPs) is being done via chemical reduction method of silver nitrate using  $\text{NaBH}_4$ .

Role of  $\text{NaBH}_4$  →

1. Acts as a reducing agent to reduce ionic silver.
2. Adsorption of borohydride plays a key role in stabilizing growing silver nanoparticle by providing a particle surface charge. There must be enough borohydride to stabilize the particles as the rxn proceeds.

Procedure-

1. A 10 mL volume of 1.0 mM silver nitrate was added dropwise

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- (about 1 drop per second) to 30 mL of 2.0 mM Sodium Borohydride solution that had been chilled in an ice bath.
- 2 The reaction mixture was stirred vigorously on a magnetic stir plate. The solution turned light yellow after the addition of 2 mL of silver nitrate and a brighter yellow when all of the silver nitrate had been added.
  - 3 The entire addition took about 3 mins, after which the stirring was stopped and the stir bar removed.
  - 4 The appearance of clear yellow colour confirmed the colloidal silver nanoparticle formation.
  - 5 Lastly, the size determination of as synthesised AgNPs was done using UV-vis spectrophotometer.

### Result -

The colour of the AgNP synthesized was yellow and  $\lambda_{\text{max}} = 400 \text{ nm}$

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