Date _____

Expt. No. _____ Page No. _____

EXPERIMENT No. 1

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

Chemicals required

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

Apparatus required

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

Procedure

STEP-1: Standarisation of $KMnO_4$ Solution by Standard (N/10) oxalic acid solution :

Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

Take 10ml of (N/10) Oxalic acid solution in a conical flask with the help of pipette and add 15 ml of 1:4 H_2SO_4 solution and 50 ml (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 $KMnO_4$ 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	$\frac{10+10+10}{3} = 10$
2.	10	10	
3.	10	10	

Calculation:

$V_1S_1 = V_2S_2$

V_1 = vol. of oxalic acid (10 ml)

V_2 = Average vol. of $KMnO_4$ sol.

S_1 = Strength of Oxalic acid
= 0.1 N (N/10)

S_2 = Strength of $KMnO_4$ sol. in,

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

Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

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 ~~up~~ 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 15ml 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 $KMnO_4$ (V_3)
1	10	10	$\frac{10+10+10}{3} = 10$
2	10	10	
3	10	10	

Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

Reactions :

$$KMnO_4 + H_2C_2O_4 + H_2SO_4 \longrightarrow K_2SO_4 + MnSO_4 + CO_2 + H_2O$$

$$Fe^{+2} + MnO_4^- + 8H^+ \longrightarrow Fe^{+3} + Mn^{+2} + 4H_2O$$

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

Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

EXPERIMENT NO. 2

Estimation of total hundress 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_4Cl 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

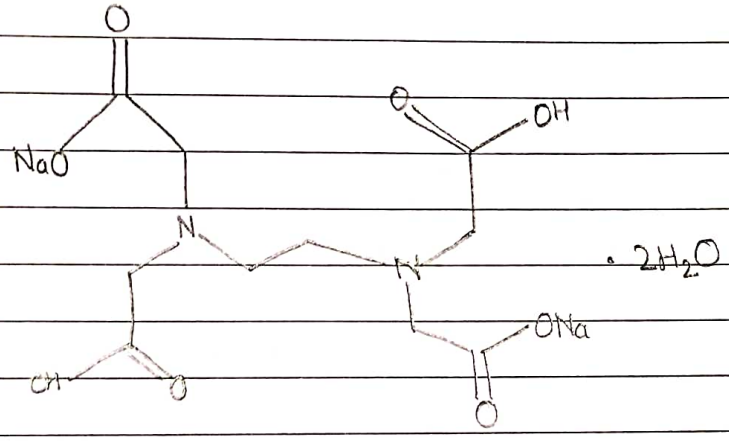
The hardness of water is due to the presence of dissolved calcium and magnesium salts. Disodium

Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

~~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:

$$M^{+2} + H_2Y^{-2} = MY^{-2} + 2H^+$$

The reaction shows that one gm-ion of the complex

Teacher's Signature _____

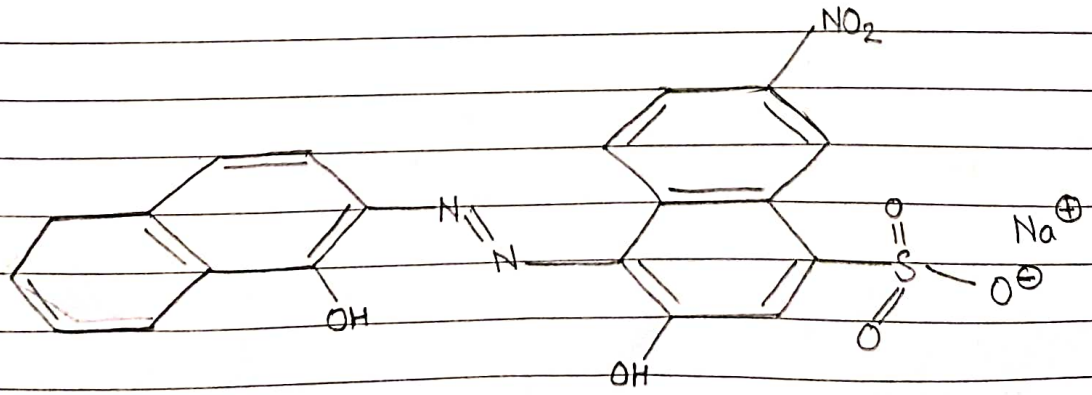
Date _____

Expt. No. _____ Page No. _____

compound forming H_2Y^{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-naphol-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:



Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

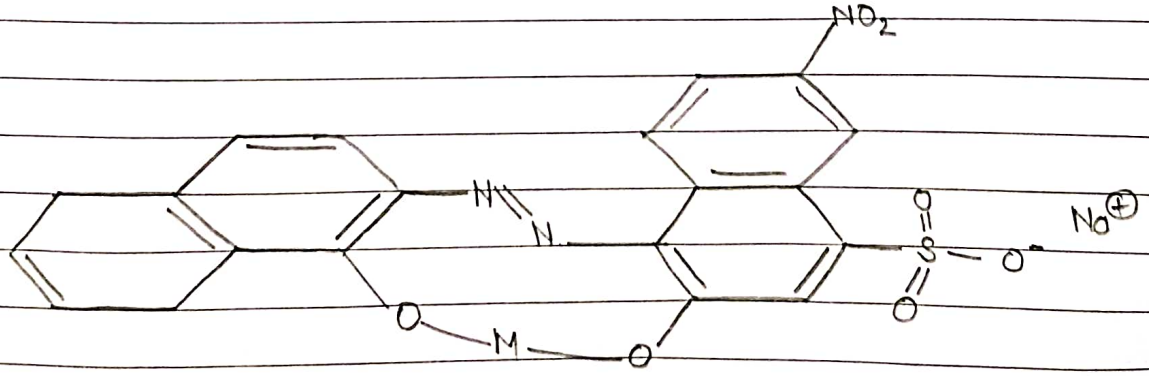
The sulphonic acid group gives up its proton long before the pH range of 7-12, which is of immediate interest for metal - ion indicator use. Only the dissociation of the phenalic H - atoms need be considered, and so the dyestuff may be represented by H_2D. The two - pK values of these two H-atoms are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

$$M^{+2} + HD^{-2} = MD^- + H^+$$

(blue) (Red)

The structure of metal EBT complex is shown below



Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

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 (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		Vol. of EDTA consumed (mL)	Mean vol. of EDAT (V mL)	Hardness of water sample (ppm)
		Initial	Final			
1	25	0.0	7.1	7.1		
2	25	0.0	7.0	7.0	7.1	996
3	25	0.0	7.2	7.2		

Teacher's Signature _____

Date _____

Expt. No. _____ Page No. _____

Calculations :

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

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

V mL of 0.01 M 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 g of $CaCO_3$

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.

∴ Total Hardness in given water sample $10\,V = \frac{0.0249 \times 10^6}{25} = 996$ 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+}

Teacher's Signature _____