Date 02/12/12

Expt. No. 1

Page No. 1

Experiment -1

Objective: To determine the amount and constituents in the given water sample.

Apparatus and Reagents :- Burette, pipette, volumetric flask, conical flask, standard N/10 HCl solutions, phenolphthalein indicator, methyl orange indicator and water sample.

Theary:

$$OH^- + HCO_3^- \longrightarrow CO_3^{-2} + H_2O$$

(i) (P): phenolphthalein end point.

$$\left.\begin{aligned} OH^- + H^+ &\longrightarrow H_2O \\ CO_3^{-2} + H^+ &\longrightarrow H_3O_3^- \end{aligned}\right\} (P)$$

(ii) (M): methyl orange end point

$$HCO_3^- + H^+ \longrightarrow H_2O + CO_2 \quad \} (M)$$

Teacher's Signature :

Date

Expt. No. 1 Page No. 2

Observation Table.

S.No	Vol of water sample taken (m.l)	Volume of HCl used (ml)				
		I.R	Reading at first end point	Reading at second end point	phenolph-thalein end point (P)	methyl orange end point (M)
1.	10 ml	0	4.1	14.7	4.1	14.7
2.	10 ml	30.4	34.4	~~49~~ 14.6	4	14.6
3.	10 ml.	0	4	14.6	4	14.6
	Concordant Readings.				4 ml (P)	14.6 ml (M)

Calculations:-

Formula to find Normality.

$N_1V_1 = N_2V_2$

N_1: Normality (unknown sol.)
N_2: Normality (known sol.)
V_1: Volume of sol.
V_2: volume of sol.

Formula to find Strength.

= Normality × Equivalent weight

Given: Normality of HCl is $\frac{1}{10}$

From Observation: P (4 ml)
M (14.6 ml)

I. Normality & Strength of OH^-

Teacher's Signature : _____

Date

Expt. No. 1 Page No. 3

(a) Normality of OH^-

$N_1V_1 = N_2V_2$
$N \times 10 = 0.1 \times P$
$N \times 10 = 0.1 \times 4$
$N = 0.04\ eq\,L^{-1}$

(b) Strength of OH^-

Strength = N× eq. weight of OH^-
$= 0.04 \times 17$
$= 0.68\ g\,L^{-1}$

II Normality & Strength of CO_3^{--}

(a) Normality of CO_3^{--}

$N_1V_1 = N_2V_2$
$N \times 10 = 0.1 \times 2P$
$N \times 10 = 0.08 \times 8$
$N = 0.08\ eq\,L^{-1}$

(b) Strength of CO_3^{-2}

Strength = N× eq. weight of OH^-
$= 0.08 \times 30$
$= 2.4\ g\,L^{-1}$

III Normality & Strength of HCO_3^{-2}

(a) Normality of HCO_3^{-2}

$N_1V_1 = N_2V_2$
$N \times 10 = 0.1 \times (M \times 2P)$
$N \times 10 = 0.1 \times (14.6 - 8)$
$N = 0.066$

(b) Strength of HCO_3^{-2}

Strength = N× eq. weight of HCO_3^{-2}
$= 0.066 \times 61$
$= 4.03\ g\,L^{-1}$

Teacher's Signature : _____

Date

Expt. No. 1 Page No. 4

Result

The given water sample contains :

OH^- alkanity = $0.68\ g\ L^{-1}$

CO_3^{-2} alkanity = $2.4\ g\ L^{-1}$

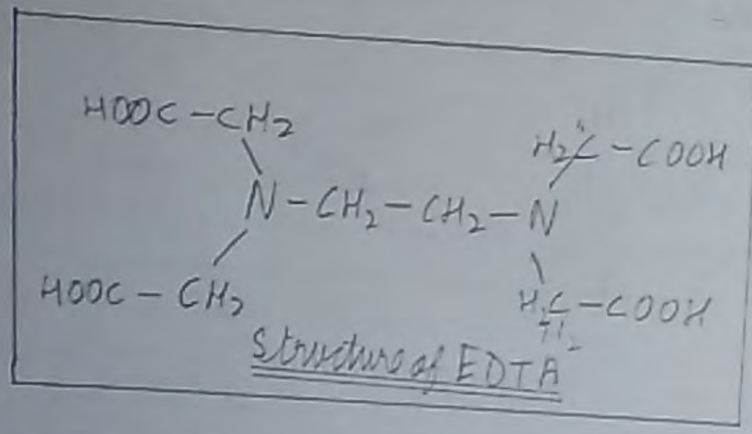
HCO_3^{-1} alkanity = $4.03\ g\ L^{-1}$

Total alkanity = $7.11\ g\ L^{-1}$

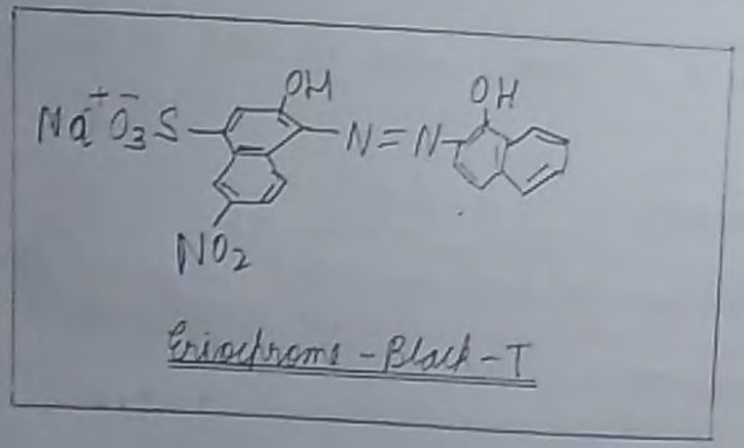
Precautions

1. The apparatus should be cleaned before the start of the experiment.
2. Use indicator carefully (Only 2-3 drops).
3. Note the phenolphthalein end point and methyl orange end-point carefully.

Teacher's Signature : _____



Structure of EDTA



Eriochrome - Black - T

Date 12/12/22

Expt. No. 2 Page No. 5

Experiment - 2

Objective: To determine temporary and permanent hardness in water by EDTA as standard solution.

Apparatus and Reagents: 0.1M EDTA, Eriochrome Black-T, Ammonia Buffer, Burette, Pipette, Volumetric flask, conical flask, dropper and water sample.

Principle: EDTA (Ethylenediamine tetra acetic acid) forms colorless stable complexes with Ca^{2+} and Mg^{2+} ions present in water at pH = 9-10. To maintain the pH of the solution at 9-10, buffer solution ($NH_4Cl + NH_4OH$) is used. E.B.T (Eriochrome Black-T is used as an indicator)

$$\begin{bmatrix} Ca^{+2} \\ Mg^{+2} \end{bmatrix} \xrightarrow[\text{Buffer solution (pH=9-10)}]{\text{E.B.T}} \underbrace{\begin{bmatrix} Ca-EBT \\ Mg-EBT \end{bmatrix}}_{\text{wine red colour, unstable complex}} \xrightarrow{EDTA} \underbrace{\begin{bmatrix} Ca-EDTA \\ Mg-EDTA \end{bmatrix}}_{\text{colourless stable complex}} + \underbrace{\text{E.B.T}}_{\text{Blue}}$$

Observations:-

(i) With Standard (known) hard water.

Date 12/12/22

Expt. No. 2 Page No. 5

Experiment - 2

Objective: To determine temporary and permanent hardness in water by EDTA as standard solution.

Apparatus and Reagents: 0.1 M EDTA, Erichrome Black-T, Ammonia Buffer, Burette, Pipette, Volumetric flask, conical flask, dropper and water sample.

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$$\begin{bmatrix} Ca^{+2} \\ Mg^{+2} \end{bmatrix} \xrightarrow[\text{Buffer solution (pH = 9-10)}]{\text{E.B.T}} \begin{bmatrix} Ca\text{-EBT} \\ Mg\text{-EBT} \end{bmatrix} \xrightarrow{\text{EDTA}} \begin{bmatrix} Ca\text{-EDTA} \\ Mg\text{-EDTA} \end{bmatrix} + \text{E.B.T}$$

{wine red colour, unstable complex} → {colourless stable complex} + {Blue}

Observations:-

(i) With standard (known) hard water.

Teacher's Signature : _____

Date

Expt. No. 2 Page No. 6

S.No	Vol of Standard (Known) hard water (ml)	Burette Reading		Volume of EDTA sol^n used (ml)
		Initial Reading	Final Reading.	
1	10 ml	0	5.2	5.2
2	10ml	5.2	10.3	5.1
3.	10 ml.	11	16.1	5.1
		Concordant Reading = 5.1 (A)		

(ii) With Boiled Water (hard) sample.

S.No	Vol of Boiled water sample (ml)	Burette Reading (ml)		Vol of EDTA sol^n used (ml)
		Initial Reading	Final Reading	
1	10 ml	17	19	2
2	10ml	19	20.9	1.9
3	10ml.	20.9	22.8	1.9
		Concordant Reading = 1.9. (B)		

Calculations

Given: Normality of EDTA = 1/50

From observation: EDTA consumed by Standard water sample = A = 5.1 ml

EDTA consumed by Boiled water sample = B = 1.9 ml

Teacher's Signature : _____

Date

Expt. No. 2 Page No. 7

I For standard Water sample:

10ml of Water sample consume Aml of $\frac{N}{50}$ EDTA = A mg eq to $CaCO_3$

1ml of Water sample = $\frac{A}{10}$ eq to $CaCO_3$

1000ml of Water sample = $\frac{5.1}{10} \times 1000$

$= 510$ mg / ltr.

II For Boiled Water Sample.

10ml of Water sample consume B ml of $\frac{N}{50}$ EDTA = B mg eq to $CaCO_3$

1 ml of B.W = $\frac{B}{10}$ mg

1000ml of B.W = $\frac{B}{10} \times 1000$

$= \frac{1.9}{10} \times 1000$

$= 190$ mg / ltr.

Total Hardness = 510mg / ltr. ∴ Hardness in standard water sample.
Permanent Hardness = 190mg / ltr. ∴ Hardness in Boiled water sample.
Temporary Hardness = Total Hardness - Permanent hardness
$= 320$ mg / ltr.

Teacher's Signature : _____

Date

Expt. No. 2 Page No. 8

Result.

The given water sample contains,

Total Hardness = 510 mg/ltr

Permanent Hardness = 190 mg/ltr

Temporary Hardness = 320 mg/ltr.

Precautions:

(i) The apparatus should be cleaned properly with distilled water.

(ii) Reaction content should be continuously shaken.

(iii) The pH should be maintained during titration.

(iv) The end point should be observed correctly.

Teacher's Signature : _____

Date 3/1/23

Expt. No. 3 Page No. 9

Experiment - 3

Objective:- To determine the percentage of available chlorine in the given sample of bleaching powder. ($\frac{250}{3}$)

Apparatus and Reagents:- Burette, pipette, volumetric flask, conical flask, bleaching powder, sodium thiosulphate N/10, potassium iodide, acetic acid, freshly prepared starch solution (indicator).

Principle:-

$$CaOCl_2 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ca + H_2O + Cl_2$$

$$Cl_2 + 2KI \longrightarrow 2KCl + I_2$$

$$Starch + I_2 \longrightarrow Starch\ Iodide\ complex\ (dark\ blue\ colour)$$

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI\ (colourless)$$

(Sodium tetrathionate)

Observation Table:-

S. No.	Vol. of Bleaching Powder sol taken	Burette Reading		Volume Used.
		I.R	F.R.	
1	10 ml	0	6	6.
2	10 ml	6	11.8	5.8
3	10 ml.	11.8	17.6	5.8
		Concordant Reading = 5.8. (A)		

Teacher's Signature : _____

Expt. No. 3 Date

Page No. 10

Calculations :-

Normality of Bleaching powder

$$N_1V_1 = N_2V_2$$

$$N \times 10 = \frac{1}{10} \times A$$

$$N = \frac{5.8}{100} \text{ eq. ltr.}$$

Strength = N × Equivalent weight of Chlorine.

$$= \frac{5.8}{100} \times 35.5$$

$$= 2.059 \text{ gm/ltr}$$

Percentage of Chlorine in given sample. $= \frac{250}{3} \times 100 \times \frac{2.059}{1000}$

$= 17\%$

Result :-

The percentage of available Chlorine in Bleaching powder sample is 17%.

Precautions:

1. All the reagents should be freshly prepared
2.

Teacher's Signature : _____

Expt. No. 3 Date

Page No. 11

2. The amount of starch indicator should be the same in all the titrations.

3. The end point of the titration should be carefully observed.

4. The glass apparatus should be washed first with chromic acid and then with distilled water.

Teacher's Signature : _____

Expt. No. 4 Date 9/1/23

Page No. 12

Experiment-4.

Objective: To determine the ferrous content in given iron ore sample using Potassium ferrocyanide as external indicator [$K_3Fe(CN_6)$]

Apparatus and Reagents:- Burette, pipette, volumetric flask, Conical flask, Standard N/10 potassium dichromate solution, Iron ore sample, potassium ferrocyanide as an external indicator.

Principle:

$$Cr_2O_7^{-2} + 14H^+ + 6e^- \longrightarrow 2Cr^{+3} + 7H_2O$$

$$[Fe^{2+} \longrightarrow Fe^{3+} + e^-] \times 6$$

$$Cr_2O_7^{-2} + 14H^+ + 6Fe^{+2} \longrightarrow 2Cr^{+3} + 7H_2O + 6Fe^{3+}$$

$$3Fe^{+2} + 2[Fe(CN)_6]^{-3} \longrightarrow Fe_3[Fe(CN)_6]_2$$

(ferrous ferric cyanide)

Blue complex.

Teacher's Signature : _____

Date

Expt. No. 4. Page No. 13

Observation Table.

S. No.	Vol of Iron Sample taken.	Burette Reading Initial	Burette Reading Final	Vol of $K_2Cr_2O_7$ Used.
1	10 ml	0	3	3
2	10 ml	3	6.2	3.2
3	10 ml	6.5	9.5	3
		Concordant Reading = 3, (A)		

Calculations:

$N_1V_1 = N_2V_2$

(Iron ore sol.) ($K_2Cr_2O_7$ sol)

$N_1 \times 10 = \frac{1}{10} \times A$

$N = \frac{3}{100}$

Amount of Fe^{2+} ions in iron ore sol = $N_1 \times 28 = 0.03 \times 28 = 0.84$ gm/ltr.

Amount of ~~Fe~~ iron in iron ore sol = $N \times 56 = 1.68$ gm/ltr.

Teacher's Signature : _____

Expt. No. _____ Date _____

Page No. 14

Result:

The ferrous content in the supplied sample of iron ore is 0.84 gm/ltr.

Precautions:

1. Titration is to be carried out at room temperature.

2. $K_2Cr_2O_7$ acts as an oxidising agent in acidic medium, therefore add dil H_2SO_4 in the iron ore sol.

Teacher's Signature : _____

Date 16/1/23

Page No. 15

Expt. No. _____

Experiment-5

Aim: To determine the chloride content in given water sample by Mohr's method.

Apparatus: Burette, pipette, conical flask, beaker, volumetric flask, dropper.

Reagents & Indicator: (N/10) Silver Nitrate solution, NaCl solution, distilled water.

Theory:

$$NaCl + AgNO_3 \longrightarrow \underset{\text{(white ppt.)}}{AgCl\downarrow} + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \longrightarrow \underset{\text{(Reddish brown)}}{Ag_2CrO_4} + 2KNO_3$$

$$Ag_2CrO_4 + 2Cl^- \longrightarrow AgCl\downarrow + CrO_4^{2-}$$

$$2AgNO_3 + K_2CrO_4 \longrightarrow \underset{\text{(Reddish Brown)}}{Ag_2CrO_4} + 2KNO_3$$

Teacher's Signature : _____

Date _____

Expt. No. _____ Page No. 16

Observation Table:-

S.no.	Vol of Water sample. (ml)	Burette Reading		Vol of $AgNO_3$ Used. (A)
		Initial Reading	Final Reading	
1.	10	0	9	9
2.	10	9	17.8	8.8
3.	10	18	27	9

Concordant Reading = 9

Calculations:

Volume of Water sample = 10 ml
Volume of $AgNO_3$ used = 9 ml

$N_1V_1 = N_2V_2$

$N_1 \times 10 = \frac{1}{10} \times 9$

$N_1 = 0.09$

Strength of Cl^- ions = N_1 × equivalent weight of Chlorine.
= 0.09×35.5
= 3.195 gm/lth. or 3195 ppm

Teacher's Signature : _____

Expt. No. _____

Result: The chloride content in the given water sample is 3195 ppm.

Precautions

1. The glass apparatus should be cleaned with distilled water.
2. The pH of the solution must be maintained between 7-8.
3. Equal drops of indicator should be used in all titrations

Teacher's Signature : _____