Bharath

INSTITUTE OF HIGHER EDUCATION AND RESEARCH

(Declared as Deemed-to-be-University under section 3 of UGC Act 1956)

A GRADE ACCREDITED BY NAAC
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BHARATH INSTITUTE OF SCIENCE AND TECHNOLOGY

173, Agaram Road, Selaiyur, Chennai - 600 073. Tamil Nadu, India.

RECORD NOTE BOOK

Name : MATAM DHARSHAN

Reg. No : U20AE051

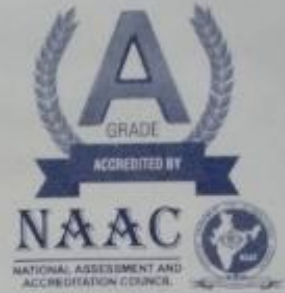Year /Sem : I-Year I/II - SEM

Branch : AERO

Subject : Chemistry Lab

⚙️ Bharath

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Name .... MATAM DHARSHAN

Course ...B. TECH........ Branch ..AEROSPACE............

Year ...2019 - 2020............ Semester ....2nd............

Register No. | U18AE051

Certified to be the bonafide Record of work done by the above student in the

Engineering...CHEMISTRY...U18BSCH2L4. laboratory during the

........2nd........ Semester in the Academic Year 2018-2019

_M 3/5/19_

Signature of the Lab-in-charge   Signature of the Head of Dept.

Submitted for the practical examination held on ...19|5|19...............

Internal Examiner   External Examiner

INSTRUCTIONS FOR MAINTAINING THE RECORD NOTE BOOK

1. The Record should be written neatly in ink on the pages of the right hand side and the diagrams / drawings to be drawn on the pages of the left hand side in pencil.

2. Every Experiment should begin on a new page.

3. The right hand side pages of the record should contain :

   I. SI. No. and date of performance of the Experiment in the margin at the top

   ii. Experiment No. and the title of the Experiment on the first line followed by

   iii. Aim of the Experiment.

   iv. A list of apparatus required.

   v. Description of the apparatus.

   vi. Theory of the Experiment in brief.

   vii. Inference of the result.

4. The left hand side pages of the Record should contain :

   I. Neat sketches of apparatus used and full page graphs wherever possible.

   ii. Diagrams of Electrical connections neatly drawn.

   iii. Observation (to be entered in a tabular form neatly wherever possible)

   iv. A detailed account of manipulation.

CONTENTS

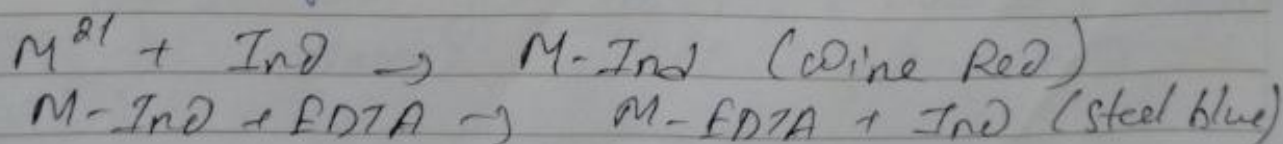| SL. No.	DATE	NAME OF THE EXPERIMENT	PAGE No.	MARKS	REMARKS
1	10.8.18	Determination of Total hardness by EDTA Method	1-5	10	Good
2	2.11.18	Estimation of copper by EDTA Method	7-9	10	Good
3	6.3.19	Conductometrically Titration of strong acid with strong base.	11-13	10	Good
4	20.3.19	Estimation of chlorides in water by argentometric Method.	15-17	10	Good
5	22-3-19	Estimation of alkalinity	19-23	10	Good
		———— Completed ————			

DETERMINATION OF TOTAL HARDNESS BY EDTA METHOD

Aim – To estimate the amount of total hardness, permanent hardness and temporary hardness of the given water sample. You are provided with a standard hard water sample and standard EDTA Solution.

Principle: Temporary hardness is largely due to the presence of bicarbonate of Calcium and Magnesium. When the sample of water is boiled, bicarbonates of calcium and Magnesium are converted to insoluble carbonates and hydroxides, which can be removed by filteration. The permanent hardness, which is due to the presence of chlorides and sulphates of Ca and Mg, cannot be removed by boiling.

$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3 + H_2O + CO_2$$
$$Mg(HCO_3)_2 \xrightarrow{\Delta} Mg(OH)_2 + 2CO_2$$
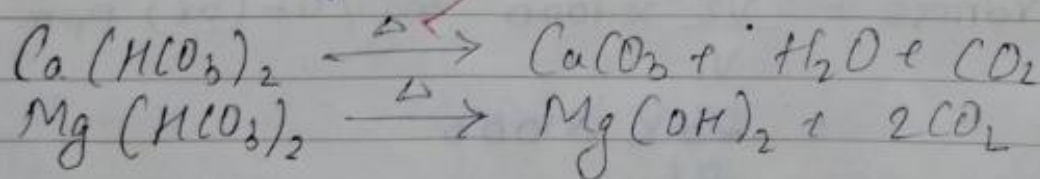
The total hardness is the sum of permanent and temporary hardness, which is determined by complexing with EDTA.

$$M^{2+} + Ind \longrightarrow M\text{-}Ind \text{ (Wine Red)}$$
$$M\text{-}Ind + EDTA \longrightarrow M\text{-}EDTA + Ind \text{ (steel blue)}$$

Simple Procedure

Content	Titration 1	Titration 11
1. Burette Solution	EDTA	EDTA
2. Pipetted Solution	20ml of Std. hardwater	20ml of Sample hard water
3. Reagent added	5ml of buffer Solution	5ml of buffer Sol".
4. Indicator	Eriochrome Black-T	Eriochrome Black-T
5. End point.	Appearance of Steel blue colour	Appearance of Steel blue colour.

Standardisation of EDTA
Standard hard water EDTA

S.No.	Volume of Std. Hand Water (ml)	Burette Reading Initial (ml)	Burette Reading Final (ml)	Volume of EDTA Consumed (ml)	Concordant Value (ml) V_1	Indicator
1.	20	0	26.0	26.0		
2.	20	0	26.0	26.0	26.0	EBT

Volume of EDTA Consumed for std. Hand Water = V_1 ml
= 26 ml

Estimation of Total Hardness - Standardised EDTA V_1
Unknown Hand Water.

S.No	Volume of std. hard water (ml)	Burette reading Initial	final	Vol^m of EDTA Consumed (ml)	Concordant Value (ml) V_2	Indicator
1	20	0	26·8	26·8		
					26·8	EBT
2	20	0	26·8	26·8		

Volume of EDTA Consumed for unknown hard water = V_2 ml
$= 26·8$ ml

Calculation

Total hardness $= \dfrac{V_2}{V_1} \times 1000$ mg/lit (or) ppm

$= \dfrac{26·8}{26} \times 1000$

$= 1030·76$

Procedure-

Titration-I :- Standarisation of EDTA.
The burette is washed with distilled water
and then risned with EDTA Solution. 20ml
of standard hard water is taken in a Conical
flask. 5ml of buffer solution is added to
maintain the pH of the mixture, followed
by the addition of 1-3 drops of EBT. The
Solution is titrated with EDTA. The end
point is the appearance of steel blue
Colour. Titration is repeated to get
concordant value. From this the Volume
of EDTA Consumed (V₁) is noted.

Titration-II: Estimation of Total hardness
The burette is filled with EDTA Solution.
20ml of unknown water sample is taken
in the Conical flask. 5ml of buffer
Solution is added to maintain the pH of
the mixture, followed by the addition of
1-3 drop of EBT. It is titration with
EDTA Solution. The end point is the
apperance of steel blue Colour. Titration
is repeated to get Concordant Value. From
this the volume of EDTA Consumed
(V₂) is noted.

Result-
Total hardness of water = 1030 ppm.

ESTIMATION OF COPPER BY EDTA METHOD

Aim - To estimate the amount of copper in the given solution using EDTA. You are provided with a 0.01M solution of EDTA. and Standard $CuSO_4$ Solution, of strength 0.01 M.

Principle - Copper forms red coloured complex with fast Sulphon Black F. indicator and the colour reaction is specific for copper xho ions in ammonical medium. In the direct titration of copper in ammonical solution, the colour change at the end point solution, the colour change at the end point is from magenta or purple to dark green.

Procedure -
Standardication of EDTA -
standard $CuSO_4$ (20 ml) solution is pipetted out into a clean conical flask and diluted to 50ml using distilled water. Five ml of Concentra -ted ammonia solution and 5 drops of fast Sulphon Black F-indicator solution are added. The solution is then titrate 1 against EDTA Solution taken in the bunette until the colour changes from purple to dark green.

Short Procedure

Content	Titration I	Titration II
Burette Solution	EDTA	EDTA
Pipette Solution	Std. CuSO₄ 20ml + 30ml Water	20 ml of Unknown Solu +30 ml water
Indicator	5ml ammonia + 5ml fast Sulphon black-F	5ml ammonia + 5m fast Sulphon black
End point	purple to dark green	purple to dark gree

Standardisation of EDTA Solution -

S.No	Vol^m of Std. CuSO₄ Solution taken (ml)	Burette reading Initial (m)	Final (ml)	Volume of EDTA Consumed (ml)	Concordant Value (ml)	Indicator
1	20	0	21.6	21.6		
2	20	0	21.6		21.6	BB-F

Calculation -

Volume of Std. Copper solution taken = 20ml

Strength of Copper Solution = 0.01 M

Volume of EDTA Consumed = V₁

Strength of EDTA X = $\frac{20 \times 0.01}{21.6}$ = 0.0092

Estimation of Copper

EDTA Vs Given Copper Solution.

S. No	Vol^m of Copper Solution taken (ml)	Burette reading Initial	Burette reading Final	Volume of EDTA Consumed (ml)	Concordant Value (ml)	Ind
1	20	0	16.4			
				16.4	16.4	PSB-
2	20	0	16.4			

Volume of EDTA Consumed $= V_2 = 16.4$

Strength of EDTA $= X = 0.0092$

Volume of Copper solution taken $= 20\,ml$

Strength of Copper Solution $= \dfrac{V_2 \times X}{20}$

$$Y = 0.007544$$

Amount of Copper present in the given solution $= \dfrac{Y \times 63.54}{10}\, g$

$$= 0.04793\, g$$

Estimation of Copper -

The given Copper Solution is made upto the mark in a 100 ml standard flask and shaken well. Twenty ml of the solution is pippetted out into a clean conical flask and diluted to 50 ml using distilled water. Five ml of Concentrated ammonia solution and 5 drop of fast sulphon black A-indicator solution are added. The solution is then titrated against EDTA solution taken in the burette until the colour changes from purple to dark green.

Result - Amount of Copper present in the given Solution = 0.04793 grams.

CONDUCTIVITY TITRATION OF STRONG ACID WITH STRONG BASE

Aim- To determine the strength of a strong acid (like HCl) conductometrically. You are provided with 0.1 N NaOH.

Procedure -

The conductivity cell is worked with water rinsed with conductivity water. Twenty ml of 0.1 N HCl is pipetted out into a clean 100 ml beaker. The conductivity cell is dipped into it. The burette is filled with NaOH solution and clamped above the beaker. One ml of NaOH solution is added from the burette and stirred well. The conductance of the solution is measured. Similarly conductance is measured from each 1 ml additions of NaOH from the burette.

Principle-

A solution of electrolytes conducts electricity due to the presence of ions. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration when sodium hydroxide is added slowly to the hydrochloric acid, it get neutralized as shown by the following equation.

Measurement of Conductance - Standard Sodium Hydroxide Vs Hydrochloric Acid.

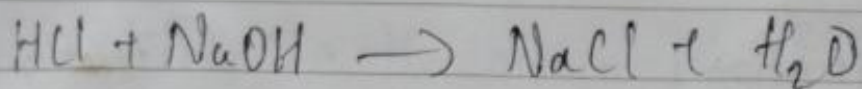| S.No	Volume of NaOH (ml)	Conductance (mh
1.	0	2.46
2.	1	1.52
3.	2	0.74
4.	3	1.33
5.	4	1.86
6.	5	2.46
7.	6	2.93
8.	7	3.41
9.	8	3.95
10.	9	4.41
11.	10	4.83
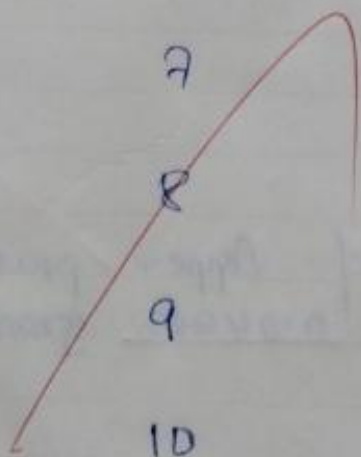
Calculation

Normality of NaOH = $N_1 = 0.1$

Volume of Sodium Hydroxide form the graph = $V_1 = 2$

Volume of HCl = V_2 ml = 20

Normality of HCl $(N_2) = \dfrac{V_1 \times N_1}{N_2}$

$N_2 = 0.01 N$

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

During the addition of the sodium hydroxide conductivity of the solution decreases slowly. This is because of the removal of fast moving H^+ ion by slow motion Na^+ ions. This decreasing trend continues till the end point is reached.

After the complete neutralization of all HCl, addition of excess of sodium hydroxide causes sudden increase in conductance. This is due to the presence of excess of hydroxide ions in solution.

Initially the measured value of conductance gradually decreases, after the equivalence point the value increases steadly. A graph is drawn by taking conductance in the Y-axis and the volume of NaOH in the x-axis. The point of intersection of the two straight lines gives the end point.

Result :

Strength of hydrochloric acid : 0.01 N

Estimation of chlorides in water By Argentometric Method.

Aim – To estimate the amount of chlorides present the given water sample. A standard solution of sodium chloride is provided with an approxima -te N/20 solution of silver nitrate.

Principle – Natural water contains chloride ions in the for of NaCl, KCl, CaCl$_2$, and MgCl$_2$. The total chlorine ion can be estimated by Argentometric method. It is known as Mohr's method. Here Ag$^+$ ion in solution in reacts with chloride ions in the presence of potassium chromat as the indicator to form sparingly soluble silver salt.

$$AgNO_3 + NaCl \longrightarrow AgCl + NaNO_3$$
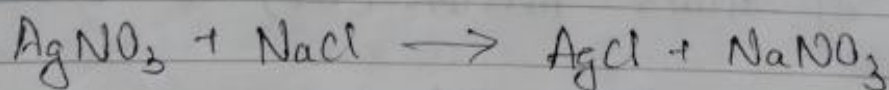
When all the chloride ions are removed, a colour change from yellow to reddish orange is noticed as end point of the titration.

$$2AgNO_3 + K_2Cr_2O_4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$
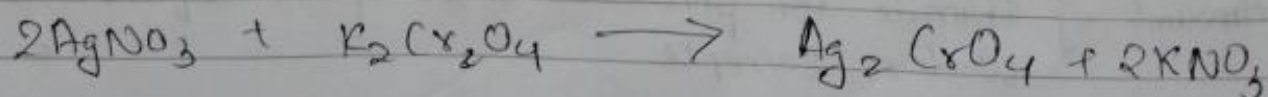
Procedure –

I. Standardardisation of silver nitrate.
The burette is washed and filled with silver nitrate. 20 ml of sodium chloride is pipetted

Short Procedure

S.No	Content	Titration 1	Titration 4
1	Burette Solution	Siluer nitrate	Siluer nitrate
2	pipette Solution	20ml of NaCl	20ml of water Sol
3	Reagent added	___	___
4	Indicator	potassium Chromate	potassium chroma
5.	End point	Appearance reddish brown	Appearance reddis brown.

Equivalent weight of chloride = 35.45

Titration I Standandization of Siluen nitrate.
Sodium chloride VS Siluer nitrate.

S.No	Volume of NaCl (ml)	Burette Reading Initial	Burette Reading Final	Vol^m of Siluer nitrate (ml)	Concordant Indica Value (ml)
1.	20	0	23.7		
				23.7	23.7 pot.Chrom
2.	20	0	23.7		- afe.

Calculation -
Volume of Sodium chloride (V_1) = 20ml
Normality of Sodium chloride (N_2) = 0.005 N
Volume of Siluer nitrate (V_2) = 23.7 ml
Normality of Siluer nitrate (N_2) = $\dfrac{V_1 N_1}{V_2}$

= 0.00421

Titration II - Estimation of chloride Water Sample V_1 Silver nitrate.

S.No	Vol^m of Water Sample (ml)	Burette reading Initial	Burette reading Final	Vol^m of Silver nitrate (ml)	Concordent (ml)	Indicator
1	20	0	13.6			Potassium chromate
				13.6	13.6	
2	20	0	13.6			

Calculation -

Volume of water sample (V_1) = 20 ml

Normality of water sample (N_1) = 0.0028

Volume of Silver nitrate (V_2) = 13.6 ml

Normality of Silver nitrate (N_2) = 0.004 N..

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

$$= 0.0028$$

Amount of chloride ion present in 1 lts of the water sample = Eq wt $\times N_2$

= X_{gm}

= 0.0028×35.45

= 0.101

Amount of chloride ion present in 100 ml of the water sample

= $\frac{X}{10}$ g

= $\frac{0.101}{10}$ gm = 0.0101 gm

out into a clonical flask. 1 ml of 2% potassium chromate indicator is added. Then the solution is titrated against silver nitrate solution. At the end point, the solution changes its colour from yellow to reddish brown changes its colour from yellow to reddish brown. The titration is repeated for concordant values.

II. Estimation of chloride.

20 ml of water is pipetted out in to clean conical flask. 1 ml of 2% potassium chromate indicator is added. Then the solution is titrated against silver nitrate solution. At the end point, the solution changes its colour from yellow to reddish brown. The titration is repeated for concordant values.

Result -
Amount of chlorides present in the given solution = 0.0101 mg/lit or ppm.

Aim- To determine the alkalinity of a given water sample.

principle- The alkalinity in water is due to the presence of caustic alkalinity - $NaOH$, KOH Na_2CO_3, K_2CO_3, $NaHCO_3$ or $KHCO_3$. Bicarbonate alkalinity is due to $Ca(HCO_3)_2$ or $Mg(HCO_3)_2$. This can be estimated by titration against acid.

When a solution containing carbonate is titrated against a strong acid like HCl, the first equivalence point of is due to the reaction.

$$CO_3^{2-} + H^+ \longrightarrow HCO_3^-.$$
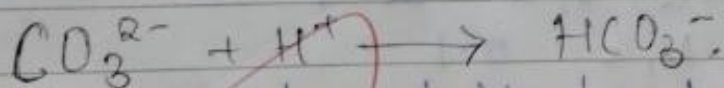
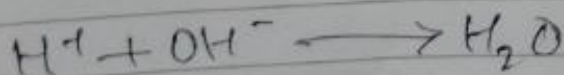This can be indicate by phenolphtalein indicator and the titrate value is termed as phenolphatein alcalinity, P. The second equivalence point corresponding to the reaction.

$$HCO_3^- + H^+ \longrightarrow H_2O + CO_3.$$
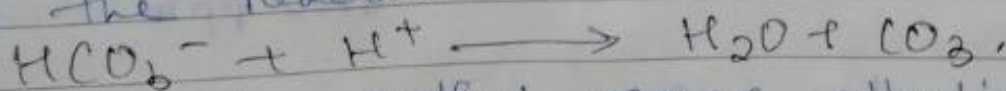
is called methyl orange alkalinity, indicated by methyl orange.

The alkalinity due to OH alone is called hydroxide alkalinity.

$$H^+ + OH^- \longrightarrow H_2O$$

Short Procedure

Content	Titration-1	Titration-11
Burette Solution	$0.1 N\ H_2SO_4$	$0.1 M\ H_2SO_4$
Pipette Solution	20 ml of given water Sample 2	20ml of given water Sample
Reagent added	—	—
Indicaton	phenolphthalein	Methyl orange
End point.	i) Disappearance of pink Colour	i) Colour changes from yellow to reddish orange.

Estimation of phenolphthalein Alkalinity :-

S.No	Vol^m of std. hand water ml	Burette reading initial	Burette reading final	Vol^m of H_2SO_4	Concordant Value (ml)	Indica... Phen d p thal
1.	80	0	19.9			
				19.7	19.7	
2.	80	0	19.9			

Volume of H_2SO_4 Consumed $= 19.7$, V_1

Normality of $H_2SO_4 = 0.1$, N_1

Volume of water sample $= V_2 = 20$

Normality of the water sample $= \dfrac{V_1 \times N_1}{N_2} = \dfrac{19.7 \times 0.1}{20}$

Phenolphthalein alkalinity interms of carbonate $P =$

$$= \frac{19.7 \times 0.1}{20} \times 50 \times 1000 \text{ ppm}$$

$$P = 4925 \text{ ppm}$$

Estimation of Total Alkalinity:

S.No	Volume of Std. hand water (ml)	Burette Reading initial	Burette Reading final	Volume of H_2SO_4 (ml)	Concordant Value (ml)	Indicator
1.	20	0	32.4			methyl
				32.4	32.4	orange
2.	20	0	32.4			

Volume of H_2SO_4 Consumed, $V_1 = 32.4$
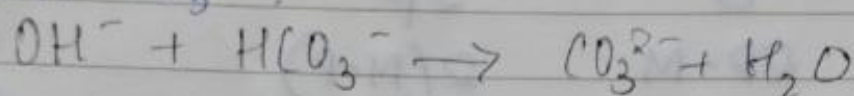Normality of H_2SO_4, $N_1 = 0.1$

Volume of water Sample = 20
Total alkalinity in terms of Carbonate M
$$= \frac{V_1 \times N_1}{V_2} \times 50 \times 1000 \text{ ppm}$$
$$= 8100 \text{ ppm}$$

$$\frac{1}{2} M = \frac{8100}{2} = 4050 \text{ ppm}$$

The total alkalinity due to all alkaline species is determined using methyl orange indicator, and is denoted as T. But OH^- and HCO_3^- cannot exist together as they simultaneously from CO_3^{2-}.

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

Hence all the three (OH^-, HCO_3^-, CO_3^{2-}) cannot exist together. Based on the titre value, different types of alkalinity are evaluated.

Procedure-

Determination of phenolphthalen Alkalinity-

80 ml of water sample is pipetted out into a clean conical flask. Two drop of phenolphalen indicator are added and titrated against standard H_2SO_4 taken in the burette. The end point is the disappearance of pink colour. Titration is repeated for Concordant Values.

Determination of Total alkalinity,

80 ml water sample is pipetted out into a clean conical flask. Two or three drops of methyl orange indicator are added and

Interpretation of Various Alkalinity :

Titrate Value	Hydroxide alkalinity	Carbonate alkalinity	Bicarbonate alkalinity
$P = 0$	0	0	M
$P < \frac{1}{2}M$	0	$2p$	$M - 2p$
$P = \frac{1}{2}M$	0	$2P$	0
$P > \frac{1}{2}M$	$2P - M$	$2(M-P)$	0
$P = M$	M	0	0

1) when $P = M$, there is only OH^- alkalinity
2) when $P = 0$, alkalinity is only due to HCO_3^-
3) when $P < \frac{1}{2}M$, present of HCO_3^-, CO_3^{2-} alkalinity
4) when $P = \frac{1}{2}M$, only CO_3^{2-} is present

5) when $P > \frac{1}{2}M$, CO_3^{2-} and OH^- are present

$P > \frac{1}{2}M$

$2P - M \Rightarrow$ Hydroxide Alkalinity

$2 \times 4925 - 8100 = 1750$ ppm

$2(M-P) \Rightarrow$ Carbonate Alkalinity

$2(8100 - 4925) = 6350$ ppm

titrated against standard H_2SO_4 taken in the
burette. The end point is the Colour
change from pale yellow to pale pink colour
Titration in repeated for Concordant val

Result :-

Bicarbonate alkalinity = 0   ppm
Hydroxide alkalinity = 1250   ppm
Carbonate alkalinity = 6350   ppm