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S.No.	Name of Experiment	Date	Page No.	Remarks/ Signature
1.	~~TO~~ DETERMINE AND COMPARE THE ALKALINITY OF WATER SAMPLE 'A' AND 'B' BY DOUBLE INDICATOR TITRATION METHOD	08/05		08/12
2.	~~TO~~ DETERMINE HARDNESS OF WATER SAMPLE COMPLEXOMETRICALLY USING EDTA (M/100)	08/12		08/19
3.	~~TO~~ ESTIMATE THE AMOUNT OF RESIDUAL CHLORINE IN WATER BY IODOMETRIC TITRATION.	08/19.		08/26
4.	~~TO~~ PREPARE THE STANDARD BUFFER SOLUTION USING ACETIC ACID AND SODIUM ACETATE AND MEASURE THE APPROXIMATE p^H OF THE GIVEN UNKNOWN SOLUTIONS BY UNIVERSAL INDICATOR METHOD.	08/26		09/11
5.	TO PREPARE THE STANDARD BUFFER SOLUTION USING AMMONIUM HYDROXIDE AND AMMONIUM CHLORIDE AND MEASURE THE APPROXIMATE p^H OF GIVEN UNKNOWN SOLUTIONS BY UNIVERSAL INDICATOR METHOD	09-11.		26-09-24

S.No.	Name of Experiment	Date	Page No.	Remarks/ Signature
6.	TO DETERMINE THE SURFACE TENSION OF GIVEN LIQUID AT LABORATORY TEMPERATURE USING STALAGMOMETER AND COMPARE THE CLEANSING POWER OF DETERGENTS 'C' and 'D'	09/24		Kamala 10/02

TO DETERMINE AND COMPARE THE ALKALINITY OF WATER SAMPLE 'A' AND 'B' BY DOUBLE INDICATOR TITRATION METHOD.

APPARATUS REQUIRED:-

1) Burette
2) Pipette
3) Beakers
4) Conical flask
5) Stand with clamp

CHEMICALS REQUIRED:-

1) Standard HCl (N/50)
2) Water samples
3) Phenolphthalein
4) Methyl orange

THEORY:-

Alkalinity is a measure of the ability of water to neutralize acids. The alkalinity of water is mainly due to the presence of :-

a) Soluble hydroxide only (OH^- ions)
b) Soluble bicarbonate only (HCO_3^- ions)
c) Soluble carbonate only (CO_3^{-2} ions)
d) A mixture of soluble carbonate and bicarbonate (CO_3^{-2} and HCO_3^- ions)
e) A mixture of soluble hydroxide and carbonate (OH^- and CO_3^{-2} ions)

The presence of hydroxide (OH^-) and bicarbonate (HCO_3^-) ions is not possible because they combine together to form carbonate (CO_3^{-2}) ion.

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

The types and extent of alkalinity present in water sample can be determined by titrating it against standard acid using phenolphthalein and methyl orange as indicators. Hence this titration is known as double indicator titration. The chemical reactions involved can be represented by the following equations.

$$OH^- + H^+ \longrightarrow H_2O \quad — (1)$$
$$CO_3^{--} + H^+ \longrightarrow HCO_3^- \quad — (2)$$
$$HCO_3^- + H^+ \longrightarrow H_2CO_3 \rightleftharpoons 2H^+ + CO_3^{--} \quad — (3)$$

A known volume of water sample is titrated against standard acid. The volume of acid used upto phenolphthalein end point (P) corresponds to reaction (1) and (2) only i.e. complete neutralization of OH^- ions and half neutralization of CO_3^{--} ions. The volume of acid used upto methyl orange end point (M) corresponds to the reactions (1), (2) and (3) i.e. complete neutralization of OH^-, CO_3^{--} and HCO_3^- ions. The total amount of acid used from the beginning of experiment to methyl orange end point (M) corresponds to total alkalinity present in water sample.

From the respective volume of acid used in phenolphthalein end point (P) and methyl orange end point (M), the respective concentration of various ions can be determined in terms of $CaCO_3$ equivalents by using normality equation.

The results may be summarized in the following table by which the amounts of hydroxide, carbonates and bicarbonates can be calculated.

Flowchart for Alkalinity :-

100ml water sample in conical flask vs N/50 HCl in burette

↓ 1-2 drops phenolphthalein

Pink colour.

↓ Titration with HCl.

Colourless(phenolphthalein end point)

↓ 1-2 drops methyl orange.

Yellow colour

↓ continue titration with HCl

Orange colour(methyl orange end point)

↓

Repeat 3-4 times titration
upto concurrent reading.

SUMMARY OF RESULT

Conditions	Hydroxide ions (OH^-)	Carbonate ions (CO_3^{--})	Bicarbonate ions (HCO_3^-)
If $P = 0$	Nil	Nil	M
If $P = M$	M	Nil	Nil
If $P = \frac{1}{2}M$	Nil	2P	Nil
If $P < \frac{1}{2}M$	Nil	2P	M-2P
If $P > \frac{1}{2}M$	2P-M	2(M-P)	Nil.

PROCEDURE

100 ml of water sample was pipette out into 250 ml conical flask. 1-2 drops of phenolphthalein was added to the sample. The burette was rinsed and filled with standard HCl (N/50). The water sample was titrated against standard acid HCl from the burette till the pink colour just discharged or disappeared. The burette reading was noated as the phenophthalein end point (P). Now again 1-2 drops of methyl orange indicator was added to the same solution and process of titration was continued till the yellow colour changed to just orange which was noted as the methyl orange end point (M).

The whole titrate value from the begining to the end was noted. The experiment was repeated to get two concurrent readings.

OBSERVATION TABLE FOR SAMPLE 'A'

S.N	Volume of water sample 'A'	Burette reading			Concurrent Reading.
		Initial	Phenolphthalein(P)	Methyl orange(M)	
1.	100	0	24.6	38.0	
2.	100	0	24.4	38.0	P = 24.4
3.	100	0	24.4	38.0	M = 38.0

CALCULATION FOR SAMPLE 'A'

Volume of water sample taken = 100 ml
Phenolphthalein reading (P) = 24.4 ml
Methyl orange reading (M) = 38.0 ml
Normality of standard acid HCl $= \frac{N}{50}$ (F = 1)

Since, $P > \frac{1}{2}M$, the water contains OH^- and CO_3^{--} ions only.

Alkalinity due to OH^- ions.
The volume of standard acid with respect to OH^- ions in 100ml of water sample = (2 P − M)

$= (2 \times 24.4 - 38) = 10.8$ ml

The normality of water sample with respect to OH^- can be calculated by using normality equation

$$V_1N_1 = V_2N_2$$

Here; V_1 = volume of acid consumed = 10.8 ml
N_1 = Normality of Acid = N/50 (F=1) = N/50
V_2 = volume of water sample = 100 ml
N_2 = Normality of water sample.

Now;

$10.8 \times N/50 = 100 \times N_2$

or, $N_2 = \frac{10.8 \times N/50}{100}$

∴ $N_2 = 0.00216$ N

∴ Concentration in gm/l = Normality × eq. wt of OH^-

= 0.00216×17 gm/l (as OH^-)

[Eq. wt of $OH^- = 17$, Eq wt of $CaCO_3 = 50$]

= $0.00216 \times 17 \times \frac{50}{17}$ gm/l (as $CaCO_3$)

= 0.108×10^3 mg/litre

= 108 ppm

= $\frac{108}{14.3}$

= 7.55° clark [14.3 ppm = 1° clark)

Alkalinity due to CO_3^{--} ions

The volume of standard acid with respect to CO_3^{--} ions in 100ml of water sample = 2 (M-P)

= 2(38-24.4)

= 27.2 ml

The normality of water sample with respect to CO_3^{--} ions can be calculated by normality equation

$V_1N_1 = V_2N_2$

or, $27.2 \times N/50 = 100 \times N_2$

∴ $N_2 = \frac{27.2 \times N/50}{100} = 0.00544$ N

∴ Concentration in gm/litre = Normality × eq. wt of CO_3^{--}

= 0.00544×30 gm/l (as CO_3^{--})

[Eq. wt of CO_3^{--} = 30, Eq wt of $CaCO_3$ = 50]

$= 0.00544 \times 30 \times \frac{50}{30}$ gm/l (as $CaCO_3$)

$= 0.272 \times 10^3$ mg/litre (as $CaCO_3$)

= 272 ppm

$= \frac{272}{14.30}$

= 19.02 °clark

RESULT FOR SAMPLE 'A'

The alkalinity of water sample 'A' due to the presence of OH^- and CO_3^{--} ions.

The alkalinity due to OH^- ions = 7.55 ° clark

The alkalinity due to CO_3^{--} ions = 19.02° clark

∴ Total alkalinity = (7.55° + 19.02°) clark

= 26.57° clark.

OBSERVATION TABLE FOR SAMPLE 'B'

S.N	Volume of (ml) water sample (B)	Burette reading (ml)			Concurrent reading (ml)
		Initial	Phenolphthalein (P)	methyl orange (M)	
1.	100	0	14.8	33.1	
2.	100	0	14.4	33.1	P = 14.4
3.	100	0	14.4	33.1	M = 33.1

CALCULATION FOR SAMPLE 'B'

Volume of water sample 'B' taken = 100 ml

Phenolphthalein reading (P) = 14.4 ml

methyl orange reading (M) = 33.1 ml

Normality of standard acid HCl = N/50 (F = 1)

Since, $P < \frac{1}{2} M$, the water sample contains CO_3^{--} and HCO_3^- ions only.

Alkalinity due to CO_3^{--} ions.

The volume of standard acid with respect to CO_3^{--} ions in 100ml of water sample B $= 2P = 2 \times 14.4$
$= 28.8$ ml

The normality of water sample with respect to CO_3^{--} can be calculated by using normality equation.

$$V_1 N_1 = V_2 N_2$$

Here, V_1 = Volume of acid consumed
N_1 = strength of acid
V_2 = volume of water sample 'B'
N_2 = strength of water sample 'B'

Or, $28.8 \times N/50 = 100 \times N_2$

$$\therefore N_2 = \frac{28.8 \times N/50}{100} = 0.00576 \text{ N}$$

$\therefore$ Concentration in gm/litre = Normality $\times$ eq. wt of CO_3^{--}
$= 0.00576 \times 30$ gm/litre (as CO_3^{--})
$= 0.00576 \times 30 \times \frac{50}{30}$ gm/l (as $CaCO_3$)

[Eq wt of $CO_3^{--} = 30$, Eq wt of $CaCO_3 = 50$]
$= 0.288$ gm of $CaCO_3$/litre
$= 0.288 \times 10^3$ mg of $CaCO_3$/litre
$= 288$ mg of $CaCO_3$/litre
$= 288$ ppm
$= \frac{288}{14.30}$
$= 20.13^0$ clark.

Alkalinity due to HCO_3^- ions.

The volume of standard acid with respect to CO_3^{--} ions in 100ml of water sample. = (M-2P)

$= (33.1 - 2 \times 14.4) = 4.3$ ml

The normality of water sample with respect to HCO_3^- ion can be calculated by normality equation

$$V_1N_1 = V_2N_2$$

$$4.3 \times N/50 = 100 \times N_2$$

$$\therefore N_2 = \frac{4.3 \times N/50}{100} = 0.00086\ N$$

$\therefore$ Concentration in gm/litre = Normality × eq wt of HCO_3^-

$= 0.00086 \times 61$ gm/l (as HCO_3^-)

$= 0.00086 \times 61 \times \frac{50}{61}$ gm/l (as $CaCO_3$)

[Eq wt of $HCO_3^- = 61$ and Eq wt of $CaCO_3 = 50$]

$= 0.043$ gm of $CaCO_3$/litre

$= 0.043 \times 10^3$ mg of $CaCO_3$/litre

$= 43$ mg of $CaCO_3$/litre

$= 43$ ppm

$= \frac{43}{14.3}$

$= 3.006°$ clark.

RESULT FOR SAMPLE 'B'.

The alkalinity of water sample 'B' is due to the presence of CO_3^{--} and HCO_3^- ions.

The alkalinity due to CO_3^{--} ion = 20·13° clark

The alkalinity due to HCO_3^- ions = 3·006° clark

$\therefore$ Total alkalinity = (20·13° + 3·006°) clark

= 23·136° clark.

COMPARISON OF ALKALINITY OF WATER SAMPLE 'A' and 'B'

Water Sample 'A'	Water sample 'B'
- The alkalinity of water sample 'A' is due to the presence of OH^- and CO_3^{--} ions.	- The alkalinity of water sample 'B' is due to presence of CO_3^{--} and HCO_3^- ions.
- The alkalinity due to OH^- ions is 7.55° clark	- The alkalinity due to CO_3^{--} ions is 20.13° clark
- The alkalinity due to CO_3^{--} ions is 19.02° clark	- The alkalinity due to HCO_3^- ion is 3.006° clark
∴ Total alkalinity = 26.57° clark	∴ Total alkalinity = 23.136° clark.

Conclusion:-

Hence, In this way the alkalinity of water sample 'A' and 'B' can be determined by double indicator titration.

PRECAUTIONS:-

a) All the apparatus should be washed with distilled water

b) The burette and pipette should be washed with respective liquids.

c) Indicator should not be in excess

d) There should ~~not~~ be any red tinge in phenolphthalein end point.

08/12

Name of the Experiment :-2

Page No.	
Date	08/12.

To DETERMINE THE HARDNESS OF WATER SAMPLE COMPLEXOMETRICALLY USING EDTA (M/100)

APPARATUS REQUIRED

1) Burette
2) Pipette
3) Conical flask
4) Beakers
5) Stand with clamp

CHEMICAL REQUIRED

1) Standard M/100 EDTA
2) Hard water or sample water
3) EBT indicator
4) $NH_3 - NH_4Cl$ buffer ($p^H = 10$)

THEORY

Water which consumes considerable amount of soap to produce lather or foam is known as hard water. Hardness of water is caused by the presence of bicarbonates, chlorides and sulphates of calcium and magnesium. On the basis of salts present in water, the hardness of water is classified as (i) Temporary and (ii) Permanent hardness.

i) Temporary hardness:

It is due to the presence of bicarbonates of calcium and magnesium, which can be removed by boiling.

$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3 \downarrow + CO_2 \uparrow + H_2O$$

$$Mg(HCO_3)_2 \xrightarrow{\Delta} MgCO_3 \downarrow + CO_2 \uparrow + H_2O$$

ii) Permanent hardness:

It is due to the presence of sulphates and chlorides of calcium and magnesium which cannot be removed by simple boiling. The total hardness (Temporary and permanent) can be determined by complexometric titration using EDTA, in presence of EBT as an indicator. Permanent hardness can be determined by removing temporary hardness (converting bicarbonates into insoluble carbonate) by boiling followed by titration with EDTA. The difference in two titre values corresponds to temporary hardness of water.

Temporary hardness = Total hardness - Permanent hardness.

The hardness is expressed in terms of $CaCO_3$ equivalent.

The determination of hardness (total and permanent) is based on the following principle:

i) The indicator Eriochrome black T (EBT) forms a wine red unstable complex with metal ions (Ca^{+2} and Mg^{+2}) present in hard water in alkaline medium.

$$M^{+2} + EBT \longrightarrow [M-EBT]$$

Unstable complex (Wine red)

ii) As this wine red solution is titrated against standard EDTA, the M^{+2} ions (Ca^{+2} and Mg^{+2}) present in hard water form stable metal EDTA complex (Ca^{+2} and Mg^{+2})

$$Mg^{+2} + EDTA \longrightarrow [M-EDTA]$$

Stable complex (Colorless)

When all the Ca^{+2} and Mg^{+2} ions in water are complexed with EDTA, then EDTA replaces Ca^{+2} and Mg^{+2} ions from the unstable complex, as a result EBT is set free and turns blue color at the end point in alkaline medium (pH=10)

$[M-EBT] + EDTA \longrightarrow [M-EDTA] + EBT$
Blue.

Here, $M^{+2} = Ca^{+2}$ and Mg^{+2} ions.

EBT = Erichrome black T, the metal ion indicator
EDTA = Ethylene diamine tetra acetic acid, a complexing agent (a ligand)

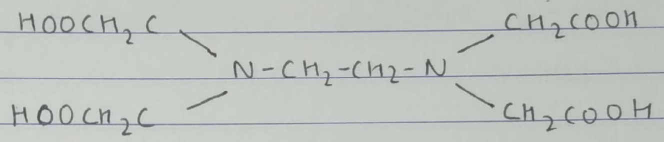

Fig:- structure of Ethylene diamine tetra acetic acid (EDTA)

Generally disodium salt of EDTA is used since EDTA is insoluble in water.

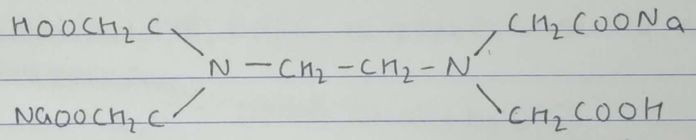

Fig:- structure of disodium salt of EDTA.

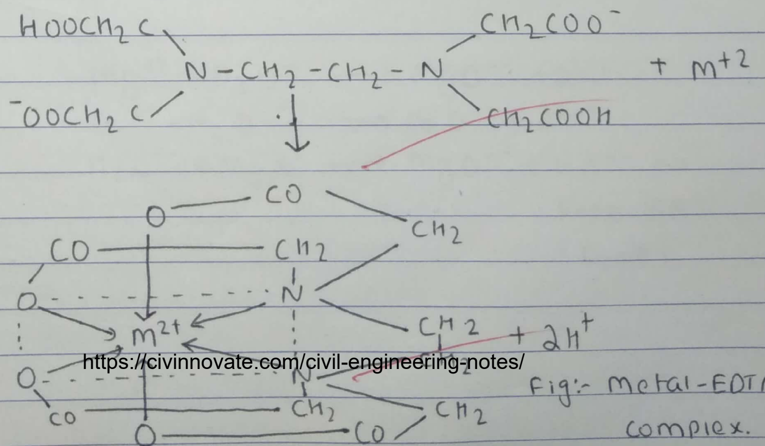

Fig:- metal-EDTA complex.

EBT is a metal ion indicator. For simplicity its sodium salt can be represented by NaH_2X which ionizes as

$$NaH_2X \rightleftharpoons Na^+ + H_2X^-$$

The anion of EBT acts as an acid-base indicator with two color changes at different medium.

$$\underset{\text{Red}}{H_2X^-} \underset{}{\overset{p^H = 6.3}{\rightleftharpoons}} \underset{\text{blue}}{HX^{--}} \overset{p^H = 11.5}{\rightleftharpoons} \underset{\text{Yellow.}}{X^{---}}$$

At p^H 8-10 the blue form of indicator HX^{--} forms a wine red color with Mg^{+2}.

$$Mg^{+2} + HX^{--} \longrightarrow \underset{\text{wine red.}}{MgX^- + H^+}$$

The disodium salt of EDTA is represented by Na_2H_2D which ionizes as,

$$Na_2H_2D \rightleftharpoons 2Na^+ + H_2D^{--}$$

The anion of EDTA (H_2D^{--}) reacts with Ca^{+2} and Mg^{+2} ions forming the stable comprexes.

$$Mg^{+2} + H_2D^{--} \longrightarrow MgD^{--} + 2H^+$$
$$Ca^{+2} + H_2D^{--} \longrightarrow CaD^{--} + 2H^+$$
$$H_2D^{--} + MgX^- \longrightarrow MgD^{--} + \underset{\substack{\text{Free EBT} \\ \text{(Blue)}}}{HX^{--}} + H^+$$

PROCEDURE

For determination of total hardness.

First of all, all the apparatus required were taken and washed with water as well as rinsed with respective acid or sample water. Burette was rinsed using EDTA solution where as pipette was rinsed using water sample. The two beakers were filled with water sample and EDTA solution respectively. 50 ml of water sample was pipette out into 250 ml conical flask where 2ml of Buffer ($NH_4OH - NH_4Cl$) was added. Finally, ~~250 ml~~ 2-3 drops of EBT indicator was added. The solution of water sample, buffer and indicator was titrated with EDTA solution till the wine red color changes into blue at the end point. Then, the reading was taken for burette and noted. The same process was continued for few more times to get the concurrent reading.

For determination of permanent hardness.

250 ml of water sample was taken in 250 ml volumetric flask and then it was transferred into 500 ml beaker. The beaker was boiled (heated) for half an hour till the volume reduced to 100 ml. The 500 ml beaker was then kept to cool and then it was filter through Whatman 40 filter paper in 250 ml volumetric flask. The water in volumetric flask was then made exactly 250 ml by adding distilled water and was shaked well to make it homogeneous. 50 ml of this water sample was pipette out from the volumetric flask into 250 ml conical flask. Then 2ml buffer and 2-3 drops of EBT was added in the conical flask. Finally the sample was

titrated with standard EDTA solution from burette till the wine red colour changes to clear blue at end point. the reading was taken from burette and was noted. The same process was continued for 3 or 4 times to get the concurrent reading.

OBSERVATION TABLE

For total hardness

Water sample taken for each titration = 50 ml

Solution taken in the burette = M/100 (f = 1) EDTA

Indicator used: EDT (Erichrome black T)

Titration medium: Alkaline

End point: wine red to clear blue.

No. of obs	Burette reading (EDTA)			concurrent reading (ml)
	Initial (ml)	Final (ml)	Difference (ml)	
1	0	20.2	20.2	19.5
2	0	19.5	19.5	
3	0	19.5	19.5	

For permanent hardness

Water sample taken for each titration = 50 ml

Solution taken in burette = M/100

Indicator used = EBT

Titration medium = Alkaline

End point = wine red to clean blue.

Flowchart For hardness

For total hardness

50 ml water sample in conical flask vs M/100(f=1) Standard EDTA in burette.

↓ 2 ml buffer (NH_4OH+NH_4Cl) and 2-3 drops EBT

Wine red colour

↓ Titration with EDTA

Clear blue colour.

For permanent hardness:

250 ml water sample in 500 ml beaker

↓ Boiled

100 ml water sample.

↓ Cooled and filtered through Whatman 40 filter paper.

water sample with tempory hardness removed

↓ Addition of distilled water to 250 ml in volumetric flask and pipette out

50 ml of water sample in conical flask

↓ 2 ml of buffer (NH_4OH-NH_4Cl) and 2-3 drops EBT.

Wine red colour

↓ Titration with N/100 standard EDTA

Clear blue colour.

No. of Obs	Burette reading (EDTA)			Concurrent reading (ml)
	Initial (ml)	Final (ml)	Difference (ml)	
1	0	11.7	11.7	
2	0	11.2	11.2	11.2.
3	0	11.2	11.2	

CALCULATION.

For total hardness

Water sample taken for each titration = 50 ml

Strength of EDTA solution = M/100

volume of EDTA consumed = 19.5

EDTA reacts with metal ion in the ratio of 1:1 by moles

1 mole of EDTA = 1 mole of $CaCO_3$

or, 100 ml of 1M EDTA = 40 gm Ca^{++} = 100 gm $CaCO_3$

or, 1 ml of ~~ED~~ 1M EDTA = 100 mg $CaCO_3$

or, 19.5 ml of M/100 EDTA = $\frac{100 \times 19.5}{100}$ mg $CaCO_3$

= 19.5 mg $CaCO_3$

= Hardness present in 50 ml water sample.

∴ Hardness present in 1 litre water sample = $\frac{19.5 \times 1000}{50}$ mg $CaCO_3$

= 390 mg $CaCO_3$

= Total hardness

∴ Total hardness = 390 mg/litre

= 390 PPM

= $\frac{390}{14.3}$ = 27.27° Clark.

For permanent hardness:

water sample taken for each titration = 50 ml

strength of EDTA solution = M/100

volume of EDTA consumed = 11.2 ml

EDTA reacts with metal ion in the ratio 1:1 by moles

1 mole of EDTA = 1 mole of $CaCO_3$

100 ml of 1 m EDTA = 40 gm Ca^{+2} = 100 gm $CaCO_3$

1 ml of 1 m EDTA = 100 mg $CaCO_3$

11.2 ml of M/100 EDTA = $100 \times 11.2 \times \frac{1}{100}$ mg $CaCO_3$

= 11.2 mg $CaCO_3$

= hardness present in 50 ml

∴ Hardness in 1 litre water sample = $11.2 \times \frac{1000}{50}$ mg $CaCO_3$

= 224 mg $CaCO_3$

= permanent hardness.

∴ permanent hardness = 224 mg/litre

= 224 ppm

= $\frac{224}{14.3}$ = 15.66° clark.

Finally

∴ Total hardness = Temporary hardness + permanent hardness

so, Temporary hardness = Total hardness - Permanent hardness

= 390 - 224

= 116 mg/litre

= 116 ppm

= $\frac{116}{14.3}$ = 11.60° clark.

RESULT

Total hardness of given water sample = 390 mg/l of $CaCO_3$
= 390 ppm
= 27.27° clark.

Permanent hardness = 224 mg/litre
= 224 ppm
= 15.66° clark

Temporary hardness = 116 mg/litre
= 116 ppm
= 11.60° clark.

CONCLUSION:

In this way total and permanent hardness of water can be determined by complexometric titration using of standard EDTA in presence of EBT indicator.

PRECAUTION

- All the apparatus should be washed with distilled water.
- The burette and pipette should be rinsed with corresponding solution
- The colour of water sample should be clear blue at the end point with no voilet tinge.

08/19

TO ESTIMATE THE AMOUNT OF RESIDUAL CHLORINE IN WATER BY IODOMETRIC TITRATION.

APPARATUS REQUIRED

a) Burette
b) Pipette
c) Beakers
d) 250 ml Iodine flask with stopper.

CHEMICAL REQUIRED

a) Potassium iodide
b) Sodium bicarbonate
c) Sodium thiosulphate
d) Potassium dichromate
e) conc. hydrochloric acid
f) Starch solution.

THEORY

Chlorination is addition of chlorine compounds to water for disinfection. It is the most common and effective method to disinfect drinking water supplies. Chlorine is added to water in the form of bleaching powder, chlorine either in aqueous solution or chlorine gas, chloramines etc. When chlorine is added to water each of these produces free residual chlorine that destroys pathogenic (disease-causing) micro-organism. Free residual chlorine is chlorine present in the form of dissolved chlorine gas, hypochlorous acid and hypochlorite ions. The presence of free residual chlorine in drinking water is correlated with the absence of diseases causing organisms and thus is a measure of the potability of water.

When chlorine (either gas or that released from bleaching powder) is dissolved in water, it decomposes rapidly into hypochlorous (HOCl) acid and hydrochloric (HCl) acid.

$$\underset{\text{Bleaching powder}}{CaOCl_2} + H_2O \longrightarrow Ca(OH)_2 + Cl_2$$

$$Cl_2 + H_2O \longrightarrow HOCl + H^+ + Cl^-$$

Hypochlorous acid is more reactive so it dissociates into hydrogen and hypochlorite ions.

$$HOCl \rightleftharpoons H^+ + Cl^- \quad \bar{O}Cl$$

The hypochlorite ion is responsible for killing germs. Similarly,

When chloramine is used, it forms hypochlorous acid along with ammonia

$$NH_2Cl + H_2O \longrightarrow NH_3 + HOCl$$

Hypochlorous acid is the most effective form of free residual i.e. chlorine available for disinfection. The excess or overdose of chlorine imparts an unpleasant taste to water and also injurious to health. Hence the amount of residual chlorine in water needs to be estimated. The residual chlorine in water is determined by iodometrically. Iodometry refers to the titration of iodine librated by oxidation of iodine against standard solution of reducing agent like thiosulphate.

The principle involved in the estimation of residual chlorine in water is that, the residual chlorine present in water oxidizes iodine to iodine. The liberated iodine is titrated against a standard solution of sodium thiosulphate (hypo) using starch as an indicator.

Standardization of sodium thiosulphate solution involves the reaction as follows.

$$K_2Cr_2O_7 + 6KI + 14HCl \longrightarrow 2CrCl_3 + 8KCl + 7H_2O + 3I_2$$

Estimation of chlorine in water involves the following reactions.

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

$$I_2 + \underset{\text{sod. thiosulphate}}{2Na_2S_2O_3} \longrightarrow 2NaI + \underset{\text{sod. tetrathionate.}}{Na_2S_4O_6}$$

$$\text{Starch} + I_2 \longrightarrow \underset{\text{(Deep blue color)}}{\text{starch-iodide complex}}$$

PROCEDURE

Step I: Standardization of hypo solution.

All the apparatus were washed with tap water and then rinsed with respective solution. First of all, burette was rinsed approx. N/20 hypo solution and filled with it. Then 100ml of distilled water was pipette out into iodine flask and 1gm potassium iodide (KI) was added and then 1 gm of sodium bicarbonate ($NaHCO_3$) was added on it. Then the flask was swirled slowly to dissolve the salts. And 5ml of conc. Hydrochloric acid (HCl) was added dropwis to prevent from fumming. And 25 ml of standard $K_2Cr_2O_7$ solution was added on it and

1) Flowchart for standardization of $Na_2S_2O_3$ (hypo) solution

100ml of distilled water in Conical flask
↓ 1gm $NaHCO_3$ +
1 gm KI + 5 ml conc HCl dropwise
+ 25 ml Stand. $K_2Cr_2O_7$ sol^n
lid closed and kept in dark.
↓ after 5 mins
Rinse inner wall of flask and cork with distilled water
↓ Titration with hypo sol^n
Greenish yellow colour.
↓ 1 ml starch sol^n
dark blue colour.
↓ Again titration with hypo dropwise
light blue colour (End point)

2) Flow chart for estimation of residual Chlorine in water sample.

10ml (10%) KI Sol^n in conical flask
↓ 100ml of water sample.
lid closed and kept in dark.
↓ after 5 mins
Rinse inner wall of flask and cork with distilled water
(straw yellow colour)
↓ 1 ml of starch sol^n
dark blue colour
↓ Titration with hypo sol^n sample
(colourless (End point)

then immediately it was closed by lead and was kept in dark for about 5 minutes. Then after 5 minutes, stopper(lead) was opened and rinsed with distilled water and also the inner walls of the conical flask. Then, the solution was titrated with hypo solution until the greenish yellow colour was appeared and then 1 ml of starch-iodide complex was added in it and again, it was titrate with hypo solution but that time dropewise and at the endpoint solution colour changed into sky blue colour, Burette reading was noted and titration was repeated two or more times to get two concurrent reading and then the strength of hypo solution was calculated using normality equation.

Step II: Estimation of residual chlorine in water sample.

10 ml (10%) KI solution was pipette out into the iodine flask and 100ml of chlorinated water sample was added and immediately mouth was closed with lead and plaseed in dark for 5 minutes. Then lead and inner wall of conical flask was rinsed with distilled water and the colour of solution turned into straw yellow colour. Then 1 ml of starch solution was added and titration was continued with hypo solution drop wise till the end poind where solution turned into colourless. The process was repeated until the two concurrent reading was noted.

OBSERVATION

(I) Standardization of sodium thiosulphate (Hypo) solution.

Volume of potassium dichromate solution taken in each titration = 25 ml

Strength of standard potassium dichromate solⁿ = $\frac{N}{20}$ (F = 1.01)

Indicator = starch solution

End point = Dark blue to sky blue.

No. of obs	Burette reading of hypo solⁿ			Concurrent reading (ml)
	Initial (ml)	Final (ml)	Difference (ml)	
1	0	27.5	27.5	
2	0	27.4	27.4	27.4.
3	0	27.4	27.4	

CALCULATION

Vol of $K_2Cr_2O_7$ solⁿ (V_1) = 25 ml

Strength of $K_2Cr_2O_7$ (S_1) = N/20 (f = 1.01)

Vol of $Na_2S_2O_3$ (hypo) consumed (V_2) = 27.4

Using normality equation,

Strength of $Na_2S_2O_3$ (hypo) consumed

$$S_2 = \frac{V_1 S_1}{S_2}$$

$$= \frac{25 \times N/20 \times 1.01}{27.4}$$

$$= \frac{N}{20} \; (F = 0.92)$$

Name of the Experiment

Page No.	
Date	

(II) Estimation of chlorine

vol. of chlorinated water sample taken in each titration = 100 ml

strength of hypo solution = N/20

indicator: starch solution

End point: Dark blue to colourless

No. of obs	Burette reading of hypo soln			Concurrent reading
	Initial(ml)	Final(ml)	Difference(ml)	
1	0	1.2	1.2	
2	1.2	2.3	1.1	1.1
3	2.3	3.4	1.1	

CALCULATION

Vol of hypo solution consumed (V_1) = 1.2 ml

strength of hypo soln (S_1) = N/20 (f = 0.92)

vol. of water sample (V_2) = 100 ml

Residual chlorine present in water sample (S_2) = $\frac{V_1 S_1}{V_2}$

$$= \frac{1.2 \times N}{100 \times 20} \times 0.92$$

$$= \underline{5.52 \times 10^{-4}} \text{ N}$$

gm/litre of chlorine = N × eq wt of chlorine

$= 5.52 \times 10^{-4} \times 35.5$ gm/l

= 0.55[illegible]2 × 35.5 mg

= $\underline{19.596 \text{ ppm.}}$

RESULT

The amount of residual chlorine in water sample is 19.596 ppm

CONCLUSION

The residual chlorine in water sample can be determined by iodometric titration using starch as an indicator.

PRECAUTION

a) The burette, pipette and conical flask should be washed with distilled water.
b) The titration should be done drope-wise after addition of starch solution.
c) The content of flask shoud be kept in dark before titration.

08/26

Name of the Experiment :-4

Page No.	
Date	08/26

TO PREPARE THE STANDARD BUFFER SOLUTIONS USING ACETIC ACID AND SODIUM ACETATE AND MEASURE THE APPROXIMATE p^H OF THE GIVEN UNKNOWN SOLUTIONS BY UNIVERSAL INDICATOR METHOD.

APPARATUS REQUIRED:-

a) 100 ml volumetric flasks
b) Test tubes and test tube stand
c) Beakers
d) Pipette
e) Conical flask
f) Funnel and dropper

CHEMICALS REQUIRED:-

a) Glacial acetic acid
b) Standard NaOH
c) Pure AR sodium acetate
d) Universal indicators
e) Phenolphthalein.

THEORY

A buffer is an aqueous solution which has a constant p^H value, and resists the change in p^H on addition of small amount of acid and base

$$CH_3COOH + H_2O \rightleftharpoons CH_3COO^- + H_3O^+$$

$$CH_3COONa \longrightarrow CH_3COO^- + Na^+$$

The acidic buffer consists of a mixture of a weak acid and its soluble ionic salt

The p^H of the acid buffer can be calculated by Henderson equation.

$$p^H = pKa + \log \frac{[salt]}{[acid]} \quad \text{—} (1)$$

Where $p^{Ka} = -\log Ka$ of the weak acid, Ka = ionization constant of weak acid.

[salt] = molar concentration of the soluble salt

[acid] = molar concentration of weak acid.

The approximate p^H of the solution can be determined by universal indicator, but the accurate p^H can be determined by p^H meter. The buffer solution having known p^H is called standard buffer.

Universal indicator is a mixture of several indicators. It can show several colour changes over a wide range of p^H values.

Buffer Capacity:-

The numbers of moles of acid or base added per litre of buffer require to cause a unit change in p^H.

$$\text{Buffer capacity} = \frac{\text{No. of moles of acid or base added to the buffer solution}}{\text{Change in } p^H}$$

① Prepration of 100ml of approx 0.4m acetic acid

100ml acid Soln (0.4m approx)
↓ (normality eqn)
Solution prepared
↓
2.5ml glacial acetic acid
↓
distilled water added upto mark.

2) Standardization of approx 0.4m acetic acid

100ml NaOH pipetted in 250ml Vcf
↓ (two drops phenolphthalein)
Titrated with prepared CH_3COOH from burette
↓
upto concurrent reading
↓ (normality eqn)
strength of acid calculated
↓

3) Prepration of 100ml of 0.2m acetic acid from 0.55m acetic acid.

100 ml exact 0.2M acetic acid preparation
↓ (normality eqn)
required vol of acid calculated.
↓
required vol of acid is kept in volumetric flask
↓ add distill water up to 100ml

For the prepration of standard acidic buffer solutions following steps are carried out.

i) Prepration of 100ml of approx 0.4 M acetic acid

ii) standardization of the prepared acetic acid with standard NaOH

iii) Prepration of 100 ml of exact 0.2M acetic acid from the above standard acetic acid

iv) prepration of 100ml of 0.2m sodium acetate

v) Prepration of standard acidic buffer solutions by mixing 0.2 M acetic acid and 0.2M sodium acetate in different proportions by volume in 9 test tubes

PROCEDURE:-

The process involves the following steps:-

Step I: Prepration of 100ml approx. 0.4M acetic acid.

100ml of an acetic acid solution of 0.4M was prepared by calculating the required volume of acetic acid using normality equation. (strength of glacial acetic acid = 16M)

$V_1 = 100ml$ $\quad$ $V_2 = ?$

$S_1 = 0.4\,M$ (approx) $\quad$ $S_2 = 16M$

Using

$$V_1 S_1 = V_2 S_2$$

$$\text{or, } V_2 = \frac{V_1 S_1}{S_2}$$

$$\therefore V_2 = \frac{0.4 \times 100}{16} = 2.5\,ml$$

4) Prepration of 100ml of exact 0.2M sod. acetate soln.

wt of sod. acetate calculated

↓

transferred into V.F.

↓

distilled water added upto mark.

5) Prepration of standard acidic buffer solutions by mixing 0.2M acetic acid and 0.2M sod. acetate soln.

12 test tubes named as 1,2,3,...9, A1, A2 and A3

↓ universal indicator

5-5 drops in each 12 testubes

↓ vol in each be 10ml

Diff vol of CH_3COOH and CH_3COONa added in each 1,2,...,9, testubes.

↓ A1, A2 and A3

8 unknown buffer soln taken

↓

color change studied

↓

color matched of A1, A2 and A3 with 1,...,9, testubes

↓

pH calculated using henderson's equation

2.5 ml of glacial acetic acid was taken into volumetric flask and distilled ~~with~~ water was added upto the mark and shaked well. Then the whole solution was poured into burette upto zero mark.

Step II: Standardization of the approx 0.4 m acetic acid.

10 ml of standard NaOH was pipetted out in 250 ml conical flask and two drops of phenolpthalein was added. It was titrated with prepared acetic acid from the burette till the end point (colorless). Titration was done untill the concurrent reading occured.

No. of obs	Vol of NaOH (ml)	Burette reading			concurrent reading
		Initial (ml)	final (ml)	Difference (ml)	
1	10	0	1.9	1.9	
2	10	1.9	3.7	1.8	1.8
3	10	3.7	5.5	1.8	

The strength of acid is calculated by normality eqn:-

$V_A S_A = V_B S_B$

$V_A = 1.8$ $S_A = ?$

$V_B = 10\,ml$ $S_B = M/10 \times 1.005$

$$\therefore S_A = \frac{10 \times 0.1 \times 1.005}{1.8} = 0.5583\,N$$

Step: III :- Prepration of 100ml of 0·2 M acetic acid from 0·5583 M acetic acid.

100 ml of exact 0·2 m acetic acid was prepared from the above standard acetic acid solution. By diluting the required volume of acid was calculated using normality equation.

$V_1 S_1 = V_2 S_2$

$V_1 = 100 ml \quad V_2 = ?$

$S_1 = 0{\cdot}2 M \quad S_2 = 0{\cdot}5583$

$$or, \; V_2 = \frac{100 \times 0{\cdot}2}{0{\cdot}55}$$

$$\therefore V_2 = 36{\cdot}36 \text{ ml}$$

36·36 ml of acetic acid (0·55 m) from the burette was taken into 100 ml volumetric flask and the volume was adjusted by adding distilled water up to the mark.

Step: IV: Prepration of 100ml of 0·2 M sodium acetate solution.

100ml of exact 0·2m sodium acetate solution was prepared by dissolving the calcucated amount of pure dry AR sodium acetate.

molecular weight of sod. acetate = 82·03

we, ve

$$\frac{w}{mol.wt} = \frac{M \times V ml}{1000}$$

$$\frac{w}{82{\cdot}03} = \frac{0{\cdot}2 \times 100}{1000} \Rightarrow w = 1{\cdot}64 \text{ gm.}$$

Exactly 1.64 gm of sodium acetate was weighed and transfered into 100ml volumetric flask. Distilled water was added ~~the~~ little bit first it was shaked well and then distilled water was added upto the mark.

Step VI:- Prepration of standard acidic buffer solution by mixing 0.2M acetic acid and 0.2M sodium acetate solutions.

First of all twelve test tubes were ~~tak~~ taken and each of them was washed with clean tap water. Nine of the twelve test tubes were marked as 1, 2, ..., 9 and remaining three were marked as A_1, A_2 and A_3. Five drops of universal indicator was added in each 12 clean test tubes. Then different volumes of 0.2M acetic acid was added into test tubes marked as 1, 2, ... 9 followed by 0.2M sodium acetate solution for the same test tubes marked as 1, 2, ..., 9. Total volume in each 9 test tubes is 10ml. Again 10ml of unknown buffer solutions was taken into clean test tubes marked as A_1, A_2, A_3. p^H of first nine standard buffer solutions was calculated by Henderson equation and p^H of unknown buffer solutions (A_1, A_2, A_3) was calculated using color against standard buffer solutions.

The ratios in which 0.2m acetic acid and 0.2m sodium acetate ~~should~~ was taken is shown in table:-

Test tube number	1	2	3	4	5	6	7	8	9
vol. of acetic acid(ml)	9	8	7	6	5	4	3	2	1
vol of sod.acetate(ml)	1	2	3	4	5	6	7	8	9

CALCULATIONS:

Calculation of pH of different standard buffer solutions:-

pH of standard buffer solution in:

a) Test tube no.1.

$$p^H = 4.74 + \log \frac{1/10 \times 0.2M}{9/10 \times 0.2M}$$

$$= 3.78$$

b) Test tube no.2

$$p^H = 4.74 + \log \frac{2/10 \times 0.2}{8/10 \times 0.2}$$

$$= 4.13$$

c) Test tube no.3

$$p^H = 4.74 + \log \frac{3/10 \times 0.2}{7/10 \times 0.2}$$

$$= 4.37$$

d) Test tube no.4

$$p^H = 4.74 + \log \frac{4/10 \times 0.2}{6/10 \times 0.2}$$

$$= 4.56$$

e) Testube no. 5

$$p^H = 4.74 + \log \frac{5/10 \times 0.2}{5/10 \times 0.2}$$

$$= 4.74$$

f) Test tube no. 6

$$p^H = 4.74 + \log \frac{6/10 \times 0.2}{4/10 \times 0.2}$$

$$= 4.91$$

g) Test tube no. 7

$$p^H = 4.74 + \log \frac{7/10 \times 0.2}{3/10 \times 0.2}$$

$$= 5.10$$

h) Test tube no. 8

$$p^H = 4.74 + \log \frac{8/10 \times 0.2}{2/10 \times 0.2}$$

$$= 5.34$$

i) Test tube no. 9

$$p^H = 4.74 + \log \frac{9/10 \times 0.2}{1/10 \times 0.2}$$

$$= 5.69.$$

Name of the Experiment

Test tube number	vol of 0.2 M acetic acid (ml)	vol of 0.2 M sod. acetate (ml)	$p^H = pKa + \log\frac{[Salt]}{[acid]}$	p^H of unknown buffer soln
1	9	1	3.78	A_1 =
2	8	2	4.13	
3	7	3	4.37	
4	6	4	4.56	
5	5	5	4.74	
6	4	6	4.91	A_2 =
7	3	7	5.10	
8	2	8	5.34	
9	1	9	5.69	A_3 =

RESULT :-

The p^H of unknown buffer solutions A_1, A_2 and A_3 are found to be 3.78, 4.91, 5.69 respectively.

CONCLUSION :-

The p^H of unknown buffer solutions can be determined by matching color with that of standard buffer solutions.

PRECAUTIONS :-

i) The same number of drops of universal indicator should be added

ii) There should not be any drop of water in any of the test tube

iii) matching the color of the solutions in the test tube shoud be done carefully.

TO PREPARE THE STANDARD BUFFER SOLUTIONS USING AMMONIA AND AMMONIUM CHLORIDE AND MEASURE THE APPROXIMATE p^H OF THE GIVEN UNKNOWN SOLUTION BY USING UNIVERSAL INDICATOR METHOD.

APPARATUS REQUIRED:-

a) 100ml volumetric flasks
b) Test tubes and test tube stand
c) Beakers
d) Pipette
e) Burette
f) Conical Flask
g) Funnel
h) Dropper.

CHEMICALS REQUIRED:-

a) Ammonia.
b) Standard HCl.
c) Pure AR ammonium chloride.
d) Universal indicator.
e) Methyl orange.

THEORY

A buffer is an aqueous solution which has a constant p^H value, and resists the change in p^H on addition of small amount of acids and bases.

The basic buffer consists of a mixture of a weak base and it's soluble ionic salt. The p^H of basic buffer can be calculated by Henderson equation.

$$p^{OH} = p^{K_b} + \log \frac{[salt]}{[base]} \text{ —— } (1)$$

Where $p^{K_b} = -\log K_b$ of the weak base, K_b = ionization ~~of~~ constant of weak base.

$NH_4OH \rightleftharpoons$

$NH_4Cl \rightarrow$

Buffer Capacity :

Universal indicator;

[Salt] = molar concentration of the soluble salt.
[base] = molar concentration of the weak base

From the value of p^{OH} of the basic buffer, it's p^{H} can be calculated by the relation.

$$p^{H} + p^{OH} = 14$$
$$\therefore \ p^{H} = 14 - p^{OH}$$

For the prepration of standard basic buffer solutions, following steps are ~~to~~ involved,

i) Prepration of 100ml of approx 0.4M ammonium hydroxide
ii) standardization of the prepared ammonium hydroxide with standard HCl
iii) Prepration of 100ml of exact 0.2M ammonium hydroxide from the standard ammonium hydroxide.
iv) Prepration of 100ml of 0.2M ammonium chloride.

PROCEDURE:-

The process involves the following steps:-

Step 1: Prepration of 100ml of approx 0.4M ammonia solution

100ml of ammonium hydroxide solution of approx. 0.4M was prepared by calculating the required volume of ammonium hydroxide using normality equation. (strength of ammonia solution = 12 M)

$V_1S_1 = V_2S_2$, $V_1 = 100\,ml$ $S_1 = 0.4M$ (approx)
$V_2 = ?$ $S_2 = 12M$

$$\therefore V_2 = \frac{V_1S_1}{S_2} = 3.3\ ml.$$

1) Preparation of 100 ml of approx 0.4m ammonia sol^n

100ml of ammonia soln (0.4 approx)
↓ normality eqn
Solution prepared
↓
3.3 ml of ammonia soln
↓
distilled water added upto mark and shaked well.

2) Standardization of 0.2m (approx) ammonia soln

100 ml of approx (0.4m) ammonia pipetted out in 250 C.f
↓ one drop methyl orange
Titrated with prepared N/10 HCl from burette upto concurrent reading
↓ normality eqn
Strength of base calculated.

3) Preparation of 0.2m ammonium hydroxide from 0.45m NaOH.

100ml of exactly 0.2 ammonia solution was prepared from st ammonium soln by diluting
↓ normality eqn
req vol of acid calculated.

3.3 ml of ammonia solution was taken into 100ml volumetric flask and distilled water was into it upto mark. It was shaked well.

Step: 2: Standardization of the approx 0.2M ammonia solution.

10ml of approx 0.4m ammonia was pipetted out into 250ml conical flask and 1 drop of methyl orang was added. Then it was titrated with standard N/10 HCl from the burette till the color turned into light pink (end point)

No. of obs	vol of NaOH (ml)	Burette reading.			concurrent reading ml
		Initial (ml)	Final (ml)	Difference (ml)	
1	10	0	45.9	45.9	
2	10	0	45.6	45.6	45.6
3	10	0	45.6	45.6	

From normality equation

$V_A S_A = V_B S_B$, $V_A = 45.6 ml$, $S_A = N/10$

$V_B = 10 ml$, $S_B = ?$

$$\therefore S_B = \frac{V_A S_A}{V_B} = 0.45 M$$

∴ Therefore, the exact strength of the prepared ammonia solution is 0.45 M.

4) Prepration of 0.2m ammonium chloride solution

wt of NH_4Cl was calculated and exactly weighet

↓

transferred into C.V

↓

distilled water was added initially just to dissolve the compound then added to mark and made homogeneous

5) Prepration of standard basic buffer solution by mixing 0.2m NH_4Cl and 0.2m NH_4OH solⁿ

12 test tube were named as 1,2,3,...9 and B_1, B_2, B_3

↓ universal indicator

5-5 drop in each 12 test tubes.

↓

different volumes of NH_4OH and NH_4Cl was added in T.T.S 1,2,3,...9

↓ vol in each TT be 10ml

unknown buffer solⁿ taken

↓ B_1, B_2, B_3

Color matched observed for B_1, B_2, B_3 with 1, 6, 9

↓

pH calculated using Henderson eqⁿ.

Step: 3: Prepration of 0·2M ammonium hydroxide from 0·45m ammonium hydroxide

100 ml of exactly 0·2m ammonic solution from the standard ammonium solution by diluting the required volume of acid solution was prepared

$V_1S_1 = V_2S_2$ $V_1 = 100ml$, $S_1 = 0·2m$

$V_2 = ?$ $S_2 = 0·45$

$$So,\ V_2 = \frac{100 \times 0·2}{0·45} = 43·85\ ml$$

43·85 ml of ammonium hydroxide (0·45m) was taken into 100ml volumetric flask. Distilled water was added upto the mark and shaked well.

Step: 4! Prepration of 0·2m ammonium chloride solution.

100 ml of exactly 0·2m ammonium chloride solution was prepared by dissolving the calculated amount of pure dry AR ammonium

Molecular weight of ammonium chloride = 53·5

Groom molecular weight of ammonium chloride is = 53·5 gm

100 ml of 1m NH_4Cl = 53·5 g

$$100\ ml\ of\ 0·2m = \frac{53·5}{1000} \times 0·2 \times 100\ gm = 1·07\ gm.$$

Exactly 1·07gm of NH_4Cl was weighed and transferred into 100 ml volumetric flask. Distilled water was added upto mark and shaked well.

Step: 5: Preparation of standard basic buffer solutions by mixing 0.2M ammonium hydroxide and 0.2m ammonium chloride solutions.

First of all, all twelve test tubes were taken and washed with clean tap water. Nine of the 12 test tube were named as 1, 2, 3, ..., 9 and three test tubes were marked as B1, B2 and B3. Five drops of universal indicator was added into each 12 test tubes. Then, different volumes of 0.2M ammonium hydroxide into Test tubes marked as 1, 2, 3, ..., 9 followed by 0.2m ammonium chloride solutions for the same test tubes. Total volume in each nine test tube (1, 2, ... 13) was 10ml. Again 10ml of unknown buffer solutions was taken in to clean test tubes marked as B1, B2 and B3. pH of first nine standard buffer solutions was calculated by henderson equation and pH of unknown buffer solutions (B1, B2 and B3) was calculated using color against standard buffer solutions.

The ratios in which 0.2M NH_4Cl and 0.2m NH_4OH was taken as shown down:-

Test tube number	1	2	3	4	5	6	7	8	9
vol. of ammonium hydroxide (ml)	9	8	7	6	5	4	3	2	1
vol of ammonium chloride (ml)	1	2	3	4	5	6	7	8	9

CALCULATIONS:

pOH of standard buffer solution in test tube no. 1

$$pOH = 4.74 + \log \frac{1/10 \times 0.2}{9/10 \times 0.2} = 3.78$$

$$pH = 14 \cdot 3.78 = 10.22$$

pOH of standard buffer solution in test tube no. 2.

$$pOH = 4.74 + \log \frac{2/10 \times 0.2}{8/10 \times 0.2} = 4.13$$

$$\therefore pH = 14 - 4.13 = 9.87$$

pOH of standard buffer solution in test tube no. 3

$$pOH = 4.74 + \log \frac{3/10 \times 0.2}{7/10 \times 0.2} = 4.\text{[illegible]}37$$

$$pH = 14 - 4.73 = 9.63.$$

pOH of standard buffer solution in test tube no. 4

$$pOH = 4.74 + \log \frac{4/10 \times 0.2}{6/10 \times 0.2} = 4.56$$

$$\therefore pH = 14 - 4.56 = 9.44$$

pOH of standard buffer solution in test tube no. 5

$$pH = 4.74 + \log \frac{5/10 \times 0.2}{5/10 \times 0.2} = 4.74$$

$$\therefore pH = 14 - 4.74 = 9.26$$

p^{OH} of standard buffer solution in test tube no.6

$$p^{OH} = 4.74 + \log\frac{(6/10 \times 0.2)}{(4/10 \times 0.2)} = 4.91$$

$$p^{H} = 14 - p^{OH} = 14 - 4.91 = 9.09$$

p^{OH} of standard buffer solution in test tube no.7

$$p^{OH} = 4.74 + \log\frac{7/10 \times 0.2}{3/10 \times 0.2} = 5.10$$

$$p^{H} = 14 - 5.10 = 8.9$$

p^{OH} of standard buffer solution in test tube no.8

$$p^{OH} = 4.74 + \log\frac{8/10 \times 0.2}{2/10 \times 0.2} = 5.34$$

$$p^{H} = 14 - 5.34 = 8.66$$

p^{OH} of standard buffer solution in test tube no.9

$$p^{OH} = 4.74 + \log\frac{9/10 \times 0.2}{1/10 \times 0.2} = 5.69$$

$$p^{H} = 14 - 5.69 = 8.31.$$

T.T NO	vol of 0.2m NH_4OH (ml)	vol. of 0.2 m NH_4Cl (ml)	$p^{OH} = p^{Kb} + \log\frac{[salt]}{[base]}$	$p^{H} = 14 - p^{OH}$	p^{H} of common buffe
1	9	1	3.78	10.22	B1 = 10.22
2	8	2	4.13	9.87	
3	7	3	4.37	9.63	
4	6	4	4.56	9.44	
5	5	5	4.74	9.26	
6	4	6	4.91	9.09	B 2 = 9.09
7	3	7	5.10	8.9	
8	2	8	5.34	8.66	
9	1	9	5.69	8.31	B3 = 8.31

RESULT:

The p^H of unknown buffer solution B_1, B_2 and B_3 are found to be ~~[illegible]~~, 10·22, 9·09 and 8·31 respectively.

CONCLUSION:

The p^H of unknown buffer solution can be determined by matching the colour with that of standard buffer solutions.

PRECAUTIONS:

i) The same no. of drops of universal indicator should be added

ii) There should not be any drop of water in all test tubes

iii) The distilled water should be used in the prepration of solutions.

076-09-24

~~TO~~ DETERMINE THE SURFACE TENSION OF GIVEN LIQUID AT LABORATORY TEMPERATURE USING STALAGMOMETER AND COMPARE THE CLEANSING POWER OF DETERGENTS 'C' and 'D'

REQUIREMENTS

a) Stalagmometer
b) Beaker
c) stand
d) Two detergent samples
e) Specific gravity bottle
f) Provided unknown liquid.

THEORY:-

Surface tension is defined as the force acting right angle to the surface along one unit length. The surface tension of the given liquid is determined relative to water at laboratory temperature using stalagmometer by counting the number of drops formed by same volume of liquid and water. Hence this method is also known as drop count method. The method is based on capillary action.

When the liquid flows through a capillary tube, a drop is formed at the lower end of the tube. The size of drop ~~decre~~ increases to same extent than falls off. Hence, the size of the drop depends upon the radius of the capillary tube and the surface tension of the liquid.

The determination of the surface tension of liquid is based on the fact that when the downward force due to the weight of drop is equal to the upward force of the surface tension, the drop of liquid falls down from the lower end of the capillary tube.

Downward Force = Upward Force

i.e $W = mg = 2\pi r \gamma$ — (i)

where 'W' is weight of drop, 'm' is the mass of the drop, g is acceleration due to gravity, $2\pi r$ is the circumference of the capillary tube with radius r, γ is the surface tension.

Comparing the surface tension of two liquids,

$m_1 g = 2\pi r \gamma_1$ — (2)

$m_2 g = 2\pi r \gamma_2$ — (3)

where m_1 and m_2 be the masses of two liquids (2) and (3)

The cleansing power of detergent depends upon the capacity to reduce the surface tension of water. Detergent is a chemical substance in the form of powder or liquid to remove dirt from cloths, dishes etc. by reducing surface tension of water. Detergents even at very low concentration lowers the surface tension of water to a great extent. The detergents decreases the interfacial tension between water and dirt or dust hence increases the cleansing activity. The cleansing power of detergents can be determined by the reduction they cause in the surface tension of water. Greater the reduction in surface tension, the greater will be the cleansing power of the detergent. The surface tension of the detergent can be determined using stalagmometer by counting the number of drops of distilled water and detergent solution using the formula.

$$\gamma_d = \frac{n_w \times d_d}{n_d \times d_w} \times \gamma_w$$

where γ_d, d_d, n_d are the surface tension, density and number of drops of detergent γ_w, d_w and n_w are the surface tension, density and number of drops of water respectively. The detergent solutions are very dilute so the densities of water and detergent solutions will be all most same. hence.

$$\text{Surface tension } (\gamma) \propto \frac{1}{\text{Temperature}}$$

$$\text{surface tension } (\gamma) \propto \frac{1}{\text{no. of drops}}$$

$$\text{surface tension } (\gamma) \propto \text{size of drops.}$$

$$\text{Surface tension } (\gamma) \propto \frac{1}{\text{cleansing powder.}}$$

Flowchart:-

Stalagmometer and sp. gravity bottle was washed with distilled water.

↓

Weight of empty sp gravity bottle was measured (W1)

↓

Lower end of stalagmometer was dipped in a beaker containing unknown liquid

↓

unknown liquid was sucked up to point higher than A and the count was started from point A untill it reached to point B.

↓

Same process was repeated for at least three times

↓

Unknown liquid was transferred into sp. gravity bottle upto fall and it's weight is measured (W2)

↓ Surface tension equation.

S.T of liquid was calculated

↓

Same process was repeated for distilled water, detergent 'X' and detergent 'Y'.

↓

Wt of distilled water in sp. gravity bottle calculated (W3)

↓

S.T of detergent 'X' and detergent 'Y' was calculated using surface tension eq^n.

PROCEDURE:-

First of all, stalagmometer and specific gravity bottle was washed with distilled water. The lower end of the stalagmometer was dipped in a small beaker containing unknown liquid and sucked till it rises above the mark 'A'. Then, the liquid was let to flow and brought to the level of mark 'A'. The numbers of drops was counted while the liquid flowed from mark A and reached upto mark B. The process was repeated upto three times and the mean was taken. Again, the stalagmometer was cleaned and the whole process was repeated for distilled water. The weight of empty specific gravity bottle and the weight of sp. gravity bottel was again taken with fully filled unknown liquid. Later the number of drops and the whole process ~~is~~ again carried for two detergent solutions 'c' and 'd' respectively. The weight of those two detergents were same therefore their weight inside sp. gravity bottle was not measured.

OBSERVATIONS:-

No. of obs	Liquid	No. of drops	mean	surface tension
1	unknown liquid	34	33	$\gamma_1 = 82.18$
2		33		
3		32		
1	Distilled water	34	35	$\gamma_w = 73.49$
2		35		
3		36		
1	Detergent 'C'	54	55.33	$\gamma_c = 46.48$
2		55		
3		57		
1	Detergent 'D'	62	64	$\gamma_D = 40.18$
2		64		
3		66		

Laboratory temperature (T° c) = 15°C
surface Tension of distilled water (γ_w) at 15°c = 73.49 dyne/cm.

CALCULATION:-

Weight of empty specific gravity bottle = W_1 gm = 24.24 gm
weight of specific gravity bottle with water = W_2 gm
= 49.94 gm
weight of specific gravity bottle with unknown liquid
(W_3 gm) = 51.34 gm
∴ weight of water = $(W_2 - W_1)$ gm = (49.94 − 24.24)
= 25.7 gm
∴ weight of liquid = $(W_3 - W_1)$ gm = (51.34 − 24.24)
= 27.1 gm

Weight of two detergents sample C and D

$W_C = W_D = 60$ gm

The relative density of liquid $= \frac{d_l}{d_w} = \frac{W_3 - W_1}{W_2 - W_1}$

$$\text{or, } \frac{d_l}{d_w} = \frac{27.1}{25.7}$$

$$\therefore \frac{d_l}{d_w} = 1.054$$

Density $= \frac{\text{mass}}{\text{volume}}$ [∵ vol of water and liquid are same]

$$\text{or, } \frac{\gamma_l}{\gamma_w} = \frac{n_w \times d_l}{n_l \times d_w} = \frac{35 \times 27.1}{33 \times 25.7}$$

$$\text{or, } \therefore \frac{\gamma_l}{\gamma_w} = 1.11$$

$$\Rightarrow \gamma_l = 1.11 \times \gamma_w = 1.11 \times 73.49$$

$$\therefore \gamma_l = 82.18 \text{ dynes/cm.}$$

Where γ_l, d_l and n_l are the surface tension, density and number of drops of given liquid respectively. γ_w, d_w and n_w are the surface tension, density and no. of drops of distilled water respectively.

By putting the respective values, the surface tension of the liquid can be found out.

$$\gamma_l = \frac{n_w \times d_l}{n_l \times d_w} \times \gamma_w$$

Again,

Surface tension of detergent solution C

$$\gamma_c = \frac{n_w}{n_d} \times \gamma_w$$

$$= \frac{35}{55.33} \times 73.49$$

$$\Rightarrow \gamma_c = 46.48 \text{ dynes/cm}$$

Surface tension of detergent solution D

$$\gamma_D = \frac{n_w}{n_D} \times \gamma_w$$

$$= \frac{35}{64} \times 73.49$$

$$\gamma_D = 40.18 \text{ dyne/cm.}$$

Result:-

The surface tension of the given liquid ~~is~~ was 82.18 dynes/cm.

The surface tension of detergents C and D ~~is~~ are found to be 46.48 dynes/cm and 40.18 dynes/cm. respectively. The cleansing powder of detergent D is more than detergent C.

CONCLUSION:-

The surface tension of the given liquid is determined relative to water using stalagmometer by counting the no. of drops of liquid ~~and water~~.

PRECAUTIONS:

a) The stalagmometer should be fitted vertically with same height.

b) The stalagmometer and sp. gravity bottle should not be exchanged.

c) The stalagmometer and specific gravity bottle should be cleaned with distilled water.

d) The rate of falling drops should be adjusted. The drop should be fallen from the stalagmometer under their own height.

10/02

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10/02

Page No.	
Date	

Name of the Experiment

TO STANDARDISE POTASSIUM PERMANGANATE SOLUTION AND USE IT TO ESTIMATE THE AMOUNT OF IRON AND DETERMINE THE PERCENTAGE PURITY IN THE SAMPLE OF FERROUS SALT SOLUTION.

APPARATUS REQUIRED:-

a) Burette
b) Conical flask
c) Pipette
d) Beakers.

CHEMICALS REQUIRED:-

a) $KMnO_4$ solution
b) Standard oxalic acid N/10 (f=1)
c) Mohr's salt solution
d) Bench sulphuric acid.

THEORY

The estimation of amount of iron in mohr's salt by titration against standard $KMnO_4$ is an example of redox titration (Reduction-oxidation titration). In redox titration, a reducing agent is titrated against an oxidizing agent or vice versa. The oxidation and reduction are complementary to one another and takes place simultaneously. Potassium permanganate is not primary standard substance. So it should be standardized by titration against standard oxalic acid in acidic medium and reaction involved is represented as

$$2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

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In ionic form

$$2MnO_4^- + 5C_2O_4^{-2} + 16H^+ \longrightarrow 2Mn^{+2} + 10CO_2 + 8H_2O$$

Hydrated ferrous ammonium sulphate [$FeSO_4 (NH_4)_2SO_4 \cdot 6H_2O$] is commonly known as mohr's salt in which active species is $FeSO_4$. It is a double salt and is more stable than $FeSO_4$ so generally used in qualitative analysis as reducing agent. The estimation of iron in mohr's salt can be determined by titration against standard $KMnO_4$ solution in acidic medium. During redox titration Fe^{+2} ions of mohr's salt are oxidized to Fe^{+3} ions by $KMnO_4$ and itself reduces to colorless manganous sulphate. The reaction involved is represented by following equation.

$$2KMnO_4 + 10FeSO_4 + 8H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$$

Ionically,

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{+2} + 4H_2O] \times 2$$

$$2Fe^{++} - 2e^- \longrightarrow 2Fe^{+3}] \times 5$$

$$2MnO_4^- + 16H^+ + 10Fe^{+2} \longrightarrow 2Mn^{+2} + 10Fe^{+3} + 8H_2O$$

In this titration, $KMnO_4$, the oxidizing agent is the self indicator as it is strongly colored (purple) and decolorizes after reduction. Potassium Permanganate is purple in its oxidized form (MnO_4^-) but colorless in its reduced form (Mn^{+2})

$$\underset{\text{(purple)}}{MnO_4^-} + 8H^+ + 5e^- \longrightarrow \underset{\text{(colorless)}}{Mn^{2+}} + 4H_2O$$

As the titration proceeds, the MnO_4^- will be reduced to Mn^{+2} and simultaneousy Fe^{+2} will be oxidized to Fe^{+3}. When whole of the reducing agent (Fe^{+2}) will be consumed, the oxidizing agent (MnO_4^-) cannot be reduced, then the addition of even one drop of $KMnO_4$ solution gives light pink color to the solution. The light pink indicates the end point of the titration.

The equivalent weight of an oxidizing and reducing agent can be obtained by dividing molecular weight of the compound by the total number of electrons that are gained or lost in a reaction per molecule of the substance.

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{+2} + 4H_2O$$

$$\text{Equivalent weight of } KMnO_4 = \frac{\text{molecular weight}}{\text{change in oxidation state}}$$

$$= \frac{\text{Molecular weight}}{5}$$

Similarly, for the oxidation of $Fe^{+2} \longrightarrow Fe^{+3}$

$$Fe^{+2} - e^- \longrightarrow Fe^{+3}$$

Therefore, equivalent weight of ferrous salt

$$= \frac{\text{molecular weight}}{\text{Change in oxd}^n\text{ state}}$$

$$= \frac{\text{molecular weight}}{1}$$

Step:-1: prepration of 100ml of 0.1N oxalic acid
weight of dry oxalic acid calculated
(Normality eqn)

Step 2:- Standardisation of $KMnO_4$ Solution by tiration
against caused Oxalic acid 0.1N

Burette rinsed with $KMnO_4$ and filled up to zero
mark (upper miniscus)

↓

10ml of 0.1N oxalic acid pipetted and transferred
into 250ml conical flask

↓ 10 ml bench H_2SO_4

60-70°c heated below B.P.

↓

Titraded in hot condition and end point (light pink)
was noted

↓

Repeated upto concurrent reading

↓ strength calculated

Normality eqn

Step:3:- Estimation of iron in mohr's salt solution by
titration against standard potassium permanganate

10 ml of 5% mohr's salt pipetted out in 250ml conical
flask

↓ 10ml bench H_2SO_4

Titrated with $KMnO_4$ up to end point

↓ Normality eqn

strength calculated.

PROCEDURE:-

Step: I: Prepration of 100ml of 0.1N oxalid acid $(COOH)_2 \cdot H_2O$

Mol.wt of $(COOH)_2 \cdot 2H_2O = 126$

Eq.wt of $(COOH)_2 \cdot 2H_2O = \frac{126}{2} = 63$

~~Eq. wt of $(COOH)_2 2H_2O$~~

1000 ml of 1N $(COOH)_2 \cdot 2H_2O = 63$ gm

1000ml of 0.1N $(COOH)_2 \cdot 2H_2O = \frac{63}{10}$ gm $= 6.3$ gm

100ml of 0.1N $(COOH)_2 \cdot 2H_2O = \frac{63}{10} \times \frac{1}{10}$ gm $= 0.63$ gm

0.63 gm of oxalic acid was taken acurately and kept in 100ml of volumetric flask using funnel and added little distilled water and shaked well to dissolve the acid. The the volume was adjusted upto 100ml by adding distilled water and it was shaked well to make homogenous.

Step: II: Standardisation of $KMnO_4$ solution by titration against standard oxalic acid 0.1N.

The burette was rinsed with $KMnO_4$ and filled upto zero mark and the upper meniscus was noted as $KMnO_4$ is translucent. 10ml of 0.1N oxalic acid and 10ml of bench sulphuric acid was transfereed into 250ml conical flask. Then the solution was heated at 60-70°C (below boiling point). It was titrated while in hot condition and continuous shaked but slowly. The dropwise addition of potassium permanganate was continued till a last single drop made the solution light pink. The titration was continued upto concurrent reading. The strength of solution ($KMnO_4$) was calculated by using normality equation.

Step: III: Estimation of iron in Mohr's salt solution by titration against standard potassium permanganate

10 ml of 5% Mohr's salt solution was taken into 250ml conical flask with the help of 10 ml pipette and 10ml of bench sulphuric acid was added. It was titrated with standard potassium permanganate solution till light pink color presisted. The process was repeated upto two concurrent readings.

OBSERVATION:

vol of oxalic acid taken in each titration = 10 ml

vol of bench H_2SO_4 = 10 ml

Indicator: No indicator used, $KMnO_4$ itself as an indicator

End point: colorless to light pink.

I) Standardization of $KMnO_4$

No of obs	Burette Reading			Concurrent Reading
	Initial(ml)	Final(ml)	Difference(ml)	
1	0	10.2	10.2	10.2
2	10.2	20.4	10.2	
3	20.4	30.6	10.2	

CALCULATION:

Vol of oxalic acid (V_1) = 10 ml

Strength of oxalic acid (N_1) = $\frac{N}{10}$

Vol. of $KMnO_4$ (V_2) = 10.2 ml

Strength of $KMnO_4$ (N_2) = ?

Now;

$$V_1N_1 = V_2N_2$$

or, $N_2 = \frac{V_1N_1}{V_2} = \frac{10\times0.1}{10.2}$

$\therefore N_2 = 0.098N$

: II : Estimation of iron in Mohr's salt solution

Vol. of Mohr's salt solution in each titration = 10 ml

" " Bench H_2SO_4 = 10 ml

Indicator: No indicator used, $KMnO_4$ itself acts as an indicator

End point: colorless to light pink.

No. of observation	Burette Reading			Concurrent reading (ml)
	Initial (ml)	Final (ml)	difference (ml)	
1	0	11.6	11.6	
2	11.6	23.2	11.6	11.6
3	23.2	34.8	11.6	

Calculation of the strength of mohr's salt
[calculation of practical value]

vol. of $KMnO_4$ (V_1) = 11.6
strength of $KMnO_4$ = 0.098N
vol. of mohr's salt = 10 ml
strength of mohr's salt = ? (N_2)

Now;

$$V_1N_1 = V_2S_2$$

$$\Rightarrow N_2 = \frac{V_1N_1}{V_2} = \frac{11.6\times 0.098}{10}$$

$$\therefore N_2 = 0.1136\,N$$

Again
gram/litre = strength in Normality x Eq. wt of mohr's salt
= 0.1136 x 392 = 44.56 g/l

392 gm of mohr's salt contains 56 gm of iron
[At wt of Fe = 56]

44.56 gm of " " " $\frac{56\times 44.56}{392}$

= 6.36 gm of iron

Calculation of Theoretical value [Given 5% mohr's salt w/v] and percentage purity of the salt.

100 ml of mohr's salt solution contains 5 gm of mohr's salt

1000 ml of " " " " $\frac{5 \times 1000}{100}$ = 50 gm of mohr's salt.

392 g of " " " " 56 gm of iron

50 g of " " " " $\frac{56 \times 50}{392}$ gm

= 7.14 gm of iron

percentage purity of mohr's salt solution = $\frac{\text{practical value}}{\text{Theoretical value}} \times 100$

$= \frac{6.36}{7.14} \times 100$

= 89.16 % Ans

RESULT:

The concn of $KMnO_4$ is found to be 0.098N and the amount of iron present in given sample of mohr's salt is found to be 0.1136 N

Percentage purity of the mohr's salt is found to be 89.16 %.

CONCLUSION:-

The amount of iron in given mohr's salt solution can be estimated by redox titration using standard potassium permanganate soln and percentage purity of the salt can also be determined by permanganometric titration.

PRECAUTIONS:-

i) Freshly prepared $KMnO_4$ should be used
ii) H_2SO_4 should be added in excess otherwise brown precipitate of MnO_2 may appear.
iii) The titration with oxalic should be done in hot condition
iv) $KMnO_4$ shoud be added slowly during the titration otherwise brown turbidity of MnO_2 will be formed.

PREPRATION OF INORGANIC COMPLEX

APPARATUS REQUIRED:-

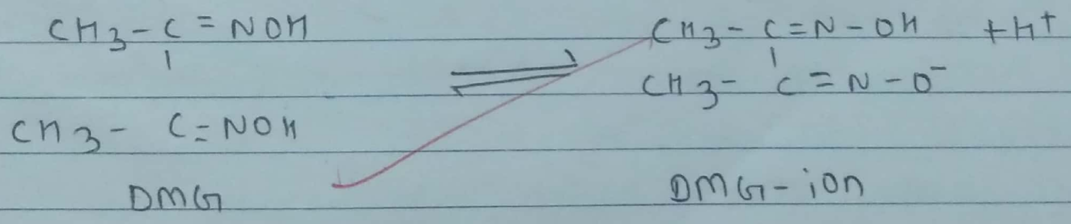
a) Beakers
b) Stirrer (Glass rod)
c) Funnel
d) Test tube

CHEMICAL REQUIRED:

a) $NiSO_4(NH_4)_2SO_4 \cdot 6H_2O$
b) Ammonia solution
c) Dimethylglyoxime (1% in ethano)
d) Barium chloride solution.

THEORY:-

Dimethyl glyoxime is a bidentate ligand. The dimethyl glyoximato ion (dmg⁻ or DMG⁻) is obtained when one OH group attached with N-atom in DMG molecules loses a proton



$$\begin{matrix} CH_3-C=NOH \\ | \\ CH_3-C=NOH \end{matrix} \rightleftharpoons \begin{matrix} CH_3-C=N-OH \\ | \\ CH_3-C=N-O^- \end{matrix} + H^+$$

DMG DMG-ion

Two molecules of dmg⁻ or DMG⁻ ions react with Ni^{+2} ion in ammoniacal solution and forms the highly insoluble neutral red chelate of bis-(dimeth glyoximato) Ni(II) which has two 5-membered and two 6-membered rings. The red precipitate of Ni-DMG complex is washed with water and dried at

Flow chart

Prepration of inorganic complex

↓

200ml water was kept in 500ml beaker

↓

100ml distilled water was taken in 250ml glass beaker

↓

250 ml glass beaker was placed in 500ml beaker

↓ warm

25ml nickel ammonium sulphate was added

↓ 60-70°C temp (bubble)

30 ml 1% DMG was added in the beaker till red colour appeared

↓ 2-3 drop NH_4OH was added to make just alkaline

Tested with red litmus paper till changed to just blue

↓

warmed in water bath to separate the ppt and heat was off

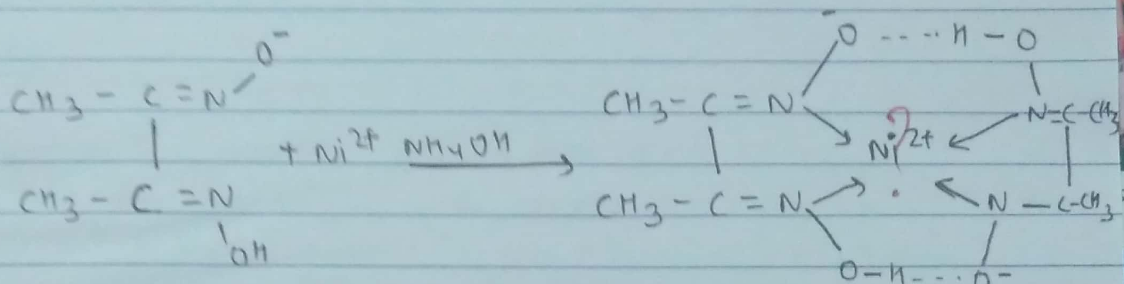
↓

Then filtered and dried it in oven

↓

Then it is weighed and calculated.

110-120°c and weighed



Bis-(dimethyl glyoximato) nickel(II)

PROCEDURE

100 ml of 1% DMG was prepared in ethanol. 25 ml of nickel ammonium sulphate was taken in 250 ml beaker and it was diluted by adding 100 ml distilled water. The solution was heated to 70-80°c and 30 ml of 1% DMG was added slowly with constant stirring till scarlet precipitate of Ni-DMG complex was formed. Then dilute ammonia solution dropwise was added with constent stiring till the precipitate was alkaline (checked with red litmus paper). A few drops of dimethyl glyoxime was added to check the completeness of precipitation. The precipitate was heated on water bath for about 30 minutes and filtered. Then the precipitate was washed with hot water till the precipitate was free from SO_4^{--} (test with barium chloride solution). The precipitate at 110-120°C in oven was dried and cool. Then, the weight of the precipitate was

determined and the weight of nickel in the complex was calculated.

OBSERVATION:-

Weight of filter paper = w gm = 1.51 gm

weight of filter paper with precipitate = w_1 gm = 1.73 gm

weight of precipitate = $w_1 - w = w_2$ gm = 0.22 gm

CALCULATION

288.91 gm of Ni-DMG complex contains 56.65 gm Ni

0.22 gm of Ni-DMG complex contains $= \frac{56.59}{288.91} \times 0.22$

$= 0.3394$ gm of Ni

Amount of Ni in Ni-DMG complex = 0.3394 gm

% of Nickel in the complex = 0.3394 ×100 = 33.94 %.

RESULT:-

The amount of Ni in Ni-DMG is found to be 0.3394 gm and the percentage of Ni in complex is found to be 33.94 %.

Conclusion:

The Ni-DMG complex is prepared from nickel salt and the amount of nickel in the complex can be determined by gravimetric method.

PRECAUTIONS

a) There should be no water during the prepration of DMG solution.

b) Excess of DMG solution should be added during the prepration of Ni-DMG complex.

c) The precipitate should be made slightly alkaline. more alkaline solution make the precipitete deposited on the wall of the beaker.

d) Nickel ammonium sulphate should not be boiled.

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