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JIS College of Engineering
Department of Chemistry

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Stream: CSE (AIML)   Class Roll: 21

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5.	Determination of the concn of the electrolyte through pH measurement	23-11-23	07-11-23	07/11/23	O

Exp. No. 1

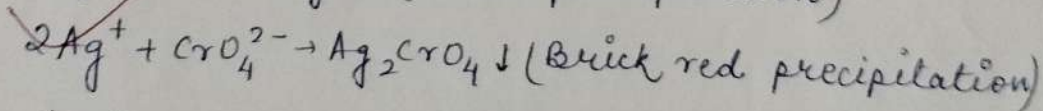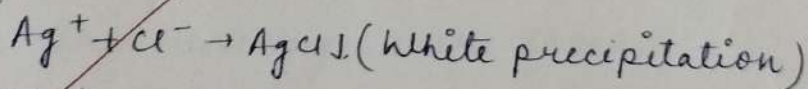## Determination of Chloride content of Water:-

Aim:-

Determination of chloride ion in a given water sample by Argentometric method (using chromate indicator solution)

Theory:-

When Silver nitrate solution is added to a given solution of chloride containing few drops of indicator ($K_2Cr_2O_4$), white silver chloride is precipitated initially [Since $K_{sp}(AgCl) < K_{sp}(Ag_2CrO_4)$, K_{sp}: Solubility product]. After finishing all the chloride ion as AgCl, Ag^+ binds CrO_4^{2-} ion and forms a brick red precipitate of Ag_2CrO_4. This brick red colour indicates the end point.

$$Ag^+ + Cl^- \rightarrow AgCl \downarrow \text{ (white precipitation)}$$
$$2Ag^+ + CrO_4^{2-} \rightarrow Ag_2CrO_4 \downarrow \text{ (Brick red precipitation)}$$

Apparatus:-

• Burette

• Pipette (25ml)

• Conical flask

Reagents:-

• $AgNO_3$ solution (N/50)

• K_2CrO_4 indicator

Determination of Water Quality Parameter
(Red-ox Titration)

AIM

Estimation of Iron present in Mohr's salt solution using standardized $KMnO_4$ solution.

THEORY

In a red-ox titration, a substance is oxidized and the other is reduced. In other words, Oxidation & reduction occurs simultaneously. Oxidation is the process of loss of one or more electrons and reduction is the gain of electrons by atoms or ions. The reagent undergoing reduction is called oxidizing agent (oxidant); and the reagent undergoing oxidation is called reducing agent (reductant). In redox titration, a reducing agent is titrated against an oxidizing agent and vice-versa. Normally potassium permanganate and iodine are commonly used as oxidizing agents and maleic acid, ferrous ammonium sulphate (Mohr's salt), sodium thiosulphate, sodium oxalate etc. are commonly used reducing agents. Potassium permanganate is a powerful and commonly available oxidizing agent
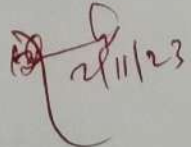
(4)

Discussion :-

1. After addition of silver nitrate from burette into the solution, silver chloride is precipitated.

2. Solubility product of Silver chromate is greater than silver chloride, thus silver chloride is precipitated until all the chloride ions are attached to the silver forming silver chloride.

3. The brick red colouration of the solution is due to formation of Silver chromate.

Conclusion :-

The amount of chloride ion (determined by Argentometric method) presents in given water sample is ___17.04___ mg/ lit.

Date of Experiment :- 05-10-23.

Date of Submission :- 27.11-23

Signature of Teacher - 27/11/23

Results and Calculations :-

Table : Determination of Chloride Ion :-

SL No.	Volume of Sample taken (ml)	Burette reading		Volume of $AgNO_3$ (ml)	Concordant volume of $AgNO_3$ (ml)	Amount of Chloride ion present (ppm)
		Initial	Final			
1	25	45.0	45.6	0.6		
2	25	45.6	46.2	0.6	0.6	17.04 mg/lit
3	25	46.2	46.9	0.7	*J. Dutta 05.10.23*	

Calculation

A.C.B. $V_1 S_1 = V_2 S_2$

where, V_1 = volume of water ~~samp~~ Sample taken (25 ml)

$$25 \times S_1 = 0.6 \times \frac{1}{50}$$

$$25 \times S_1 = 0.012$$

$$S_1 = 0.00048$$

S_1 = Strength of water sample (due to presence of Cl° ion = ?)

V_2 = volume of $AgNO_3$ sol^n (0.6)

S_2 = Strength of $AgNO_3$ sol^n N/50 or 1/50.

Hence, the strength of water sample due to presence of Cl^- is 0.00048.

1000 ml 1(N) water sample due to presence of Cl^- ions containing 35.5 gm of Cl^- ion.

1000 ml of 1 N water sample = 0.00048 (N)

$$= 0.00048 \times 35.5 \times 1000 = 17.04 \text{ mg/lit}$$

Procedure :-

1. The burette was filled with $AgNO_3$ (N/50) solution.
2. The pipette was washed with distilled water first and then rinsed with given sample of tap water. 25 ml of sample of tap water was pipetted out in a clean conical flask.
3. 2 drops of K_2CrO_4 indicator were added.
4. $AgNO_3$ was added from the burette, shaking the flask constantly. A white precipitate of $AgCl$ was obtained. After the addition of more drops of $AgNO_3$ solution, a red colour appeared in the flask as local point which disappeared upon shaking.
5. The addition of $AgNO_3$ solution was continued till solution turned reddish brown (brick red colour). The readings were taken.

we know, $V_1 S_1 = V_2 S_2$

$$10.1 \times 0.098 = 10 \times S_2$$

$$S_2 = 0.09898 \, N$$

1000 CC 1(N) Mohr's salt solution contains 56 gm of Fe.

1000 C.C 0.09898(N) Mohr's salt solution contains

$$= 56 \times 0.09898 \text{ gm of Fe}$$

$$= 5.5428 \, g \, (\underline{L})$$

Discussion :-

1. Oxidation reduction takes place simultaneously in this experiment.

2. $KMnO_4$ solution is standardized with the help of standard Oxalic acid using the formula $V_1 S_1 = V_2 S_2$

3. $KMnO_4$ acts as a self-indicator in this experiment.

4. $KMnO_4$ acts as a strong oxidizing agent in presence of H_2SO_4 thus H_2SO_4 is added in the solution.

5. Strength of Mohr's salt solution is determined with standardized $KMnO_4$ solution using formula $V_1 S_1 = V_2 S_2$

6. When all the reducing agent has been oxidized then the excess drop of $KMnO_4$ resulted in change of the colour.

Result & Calculation :-

Table :- Titration of Mohr's salt solution Against Standardized $KMnO_4$ solution (Redox - Titration):-

No of observation	Vol. of Mohr's Salt Solution (ml)	Burette Reading		Vol. of $KMnO_4$ solⁿ con-sumed (ml)	concord-ant reading (ml)	Strength of $KMnO_4$ solution (N)	Strength of Mohr's salt solution (N)	Iron present (g/l)
		Initial	Final					
1		0.0	10.1	10.1				
2	10	10.1	20.2	10.1	10.1	0.098	0.09898	5.54%
3		20.2	30.4	10.2				

Y. Dutta
12.10.23

From the normality Equation, $V_1 S_1 = V_2 S_2$ the value of S_2 is determined.

Where, V_1 = volume of $KMnO_4$ solution = 10.1 ml

S_1 = Strength of $KMnO_4$ solution = 0.098 N

V_2 = volume of Mohr's salt solution = 10 ml

S_2 = Strength of Mohr's salt solution

with the strength of the Mohr's salt solution.

Apparatus :-

- Graduated pipette (10 ml)
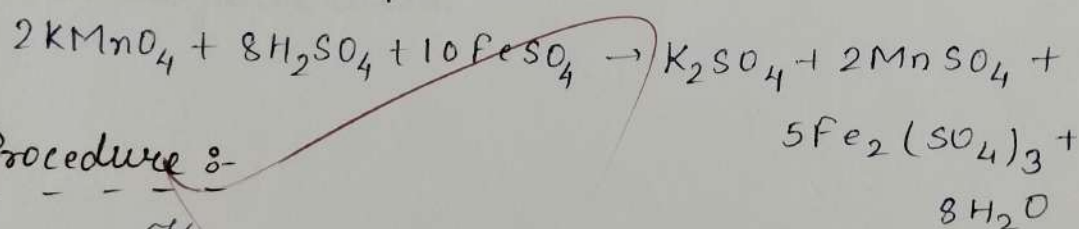- Burette
- Conical Flask (250 ml)
- Measuring cylinder (10 ml)

Reagents :-

- Mohr's salt solution [$FeSO_4$, $(NH_4)_2SO_4 \cdot 6H_2O$]
- Standardized Potassium permanganate solution
- 2 (N) Sulfuric acid

and its oxidizing power depends on the acidity of the solution. In most of these titrations, solution of reducing agents should be made acidic before carrying out the titration. In acidic solution, reduction of potassium permanganate is represented by :-

$$[MnO_4^- + 8H^+ + 5e \leftrightarrow Mn^{+2} + 4H_2O] \times 2$$
$$2[Fe^{+2} \rightarrow Fe^{+3} + e] \times 5$$

$$\overline{2MnO_4^- + 8H^- + 10Fe^{2+} \rightarrow 2Mn^{2+} + 8H_2O + 10Fe^{3+}}$$
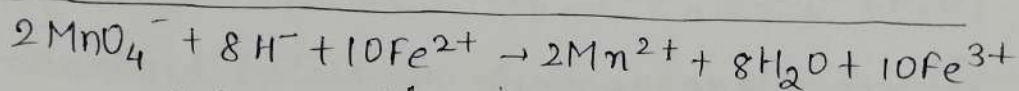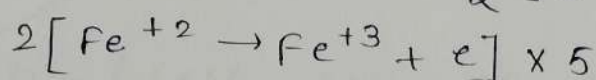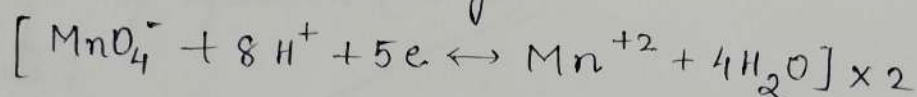
so the complete equation is

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

Procedure :-

Known strength of $KMnO_4$ solution, $2(N)$ H_2SO_4 solution and unknown strength of Mohr's salt solution are supplied. Now the burette is rinsed with $KMnO_4$ solution & then filled with it. After that, 10ml of Mohr's salt solution was pipetted out in a 250 ml conical flask & 10ml of dilute H_2SO_4 solution was added to it and then it was titrated by standardized $KMnO_4$ solution. At the end point, the colour changed from colourless to pink. Then from the burette reading the amount of iron present in Mohr's salt solution was determined along

Procedure :-

1. 0.001 M $NaBH_4$ was prepared with distilled water in a 100 ml volumetric flask.

2. 0.0001 M $AgNO_3$ was prepared with distilled water in a 100 ml volumetric flask and taken 50 ml in an Erlenmeyer flask.

3. A magnetic stirrer bar was added in the flask and stirred it with a constant speed.

4. $NaBH_4$ solution was added drop wise in the flask with stirring.

5. After yellow coloration excess $NaBH_4$ solution was added in the solution.

Observation :-

In 0.0001 M $AgNO_3$ solution, 0.001 M $NaBH_4$ sol^n will be added with dropper. After few drop of $NaBH_4$ around 7-8 drops it will turn yellow and after addition of more drops it will turn golden. Further, if more drops of $NaBH_4$ sol^n will turn into blackish colour. If it will keep for around 12 hours long it will turn into agglomeration of silver nanoparticles.

Experiment no :- 3   Date :- 02-11-23

Synthesis of Silver Nanoparticles Doped Organic Thin Film For Organic Transistors :-

AIM :- Synthesis of Silver Nanoparticles through reduction route.

Theory :- The definition of nanoparticles is an aggregate of atoms bonded together with a radius between 1 and 100 nm. There is a sudden shift of all properties of material when they just enter into the nanoscale. As material size reduces from centimeter (bulk) to nanometer scale, properties mostly decreases as much as six orders of magnitude to that at macro level. As nanostructures are having reduced dimensions, it leads to increase in surface energy via increase in surface area. If we consider bulk material the surface to volume ratio is low, whereas the surface to volume ratio is increasing enormously in case of nanomaterial. Conducting metals may show insulting behaviour in nanodimension. Hence one nanometer is a magical point on the dimensional scale.
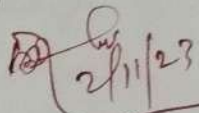
Materials needed :-

Silver nitrate $(AgNO_3)$; Sodium Borohydride $(NaBH_4)$, Distilled water.
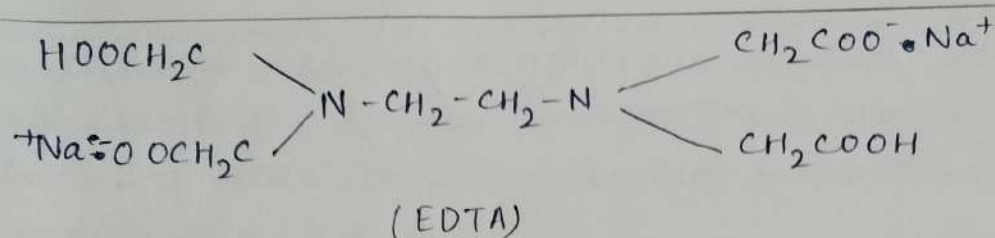
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Conclusion :-

The amount of Iron present in Mohr's salt sol^n is <u>5.5428 g/L</u>.

Date of Experiment :- 12-10-23
Date of Submission :- 2-11-23
Signature of Teacher :- 2/11/23

$$HOOCH_2C$$

$$N-CH_2-CH_2-N$$

$$CH_2COO^-\cdot Na^+$$

$$^+Na^-O\ OCH_2C$$

$$CH_2COOH$$

(EDTA)

The quick complex and one step interaction of polydentate ligands with metals to yield stable complexes, is the principle of these complexometric titrations in which metal in solutions are titrated against that of polydentate ligands.

Apparatus →

- Pipette (25 ml)
- Burette
- Beaker (250 ml)
- Conical Flask

Reagents →

- Water sample
- EDTA solution
- Eriochrome Black-T-indicator
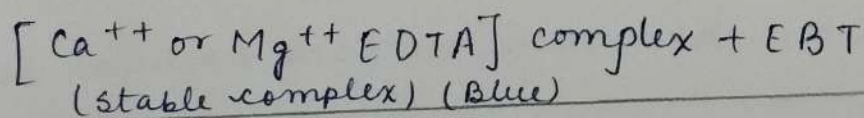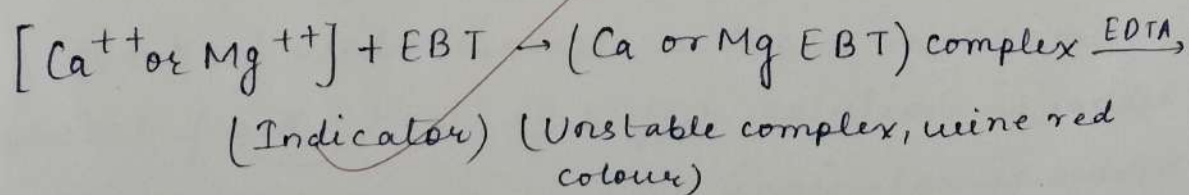- Buffer solution ($NH_4Cl + NH_4OH$)

Procedure :-

Determination of total hardness :

50 ml of water sample is taken in 250 ml conical flask. 1 ml of buffer solution is added to it. Now a pinch of EBT indicator is added. The solution becomes wine red. Now the solution

it is used to estimate Ca^{2+} & Mg^{2+} ions. Dissociation constant values indicate the EDTA behaves like a dicarboxylic acid. Two of its carboxyl group are strongly acidic the other two hydrogens are released during complex formation.

Ionisation of this complex depends on the p^H of the solution. Hence in this titration p^H of the sensitive indicators are used. In this estimation of Ca^{2+} and Mg^{2+} with EDTA, an azodye called Erichrome Black - T (EBT) is used as an indicator. This forms a metal indicator complex the stability is initially red. As the titration progress the metal ions form more stable complex with EDTA, hence the indicator anion goes to the solution and the colour changes from wine red to blue at the end point. Since the action of the indicator of formation of metal - EDTA complex is governed by p^H hence it is kept constant by adding a suitable buffer $(NH_4Cl + NH_4OH)$.

$$[Ca^{++} \text{ or } Mg^{++}] + EBT \xrightarrow{} (Ca \text{ or } Mg \text{ } EBT) \text{ complex } \xrightarrow{EDTA}$$

(Indicator) (Unstable complex, wine red colour)

$$[Ca^{++} \text{ or } Mg^{++} \text{ } EDTA] \text{ complex } + EBT$$

(stable complex) (Blue)

Experiment no → 4   Date → 09-11-23

Determination of water Quality Parameter (Hardness of water)

AIM →

Estimation of calcium and magnesium hardness seperately of a given tap water sample.

THEORY →

Hardness is defined as soap consuming capacity of water. Bicarbonates of Ca, Mg cause temporary hardness. However, chlorides, sulphates and carbonates of Ca & Mg cause permanent hardness. Hard water is not suitable for use as boiler feed water, because it leads to the formation of scales & slugs causing wastage of fuel, chocking of pipes, decreases efficiency & chances of explosion is also present. Hard water may also produce undesirable sports on the fabric due to its reaction with dyes when using in textile finishing. Thus softening of water is very important. In order to use any softening process type & extent of hardness must be known as prequesite; complexometric titration is one of the best methods for hardness estimation.

Many metals for complexes with such reagents which contain appropriate ligands. EDTA is one such reagent, which forms complexes with metals. In the form of its disodium salt,

Conclusion

At first if 0.001 M $NaBH_4$ solution will added dropwise in 0.0001 M $AgNO_3$ solution; the colour would turn out as yellow colour.

Date of Experiment : 2-11-23

Date of submission :- 9-11-23

Signature of Teacher :- J. Dutta

9/11/23

Determination of the concentration of the Electrolyte
Through pH Measurement.

AIM:

Determination of the unknown strength of HCl solution
by Standardized NaOH solution Using pH Metric
Method.

THEORY:

Most of the chemical & biochemical processes are
profoundly affected by the acidity and / or the
alkalinity of the medium in which they take
place. All acids dissociate in aqueous solution
to yield H^+ ions. Some acids such as HCl, H_2SO_4 and
HNO_3 are completely ionized in aqueous solution,
wheras most of organic acids like $HCOOH$, CH_3COOH,
ionized to a small extend only. The former are
known as 'strong' acids, while the latter are
classified as 'weak' acids. If we compare the
ionization of $1(N)$ HCl and $1(N)$ CH_3COOH, then we
will notice that, even their molar concentrations
are same. HCl is ionized extensively and forms
a strong acid solution wheras the CH_3COOH forms
a weak acid solution containing lesser
amount of H^+ ions. From these observations, we
can conclude that the acidity of a solution
does not depend upon the molarity of the
acid, but upon the concentration of H^+ ions.
We can follow from the above dissussion that

$$= 244 \text{ mg of } CaCO_3$$

Therefore the total Hardness of water sample is = 244 ppm.

Discussion

1. NH_4Cl and NH_4OH buffer solution is added to maintain the pH of the reaction medium at 10 which is a prerequisite for formation of metal – EBT complexes.

2. After addition of EDTA solution stable metal – EDTA complex is formed. Thus free EBT is responsible for the blue colouration of the solution after the titration is over.

Conclusion

Total Hardness of water sample = 244 ppm.

Date of Experiment → 09-11-23

Date of submission → 30-11-23

Signature of teacher → J. Dutta

30.11.23

⑤

Result and Calculation

Table -I → Determination of Total Hardness :-

No of Observations	Volume of water sample taken (ml)	Burette reading		Volume of EDTA solⁿ consumed (ml)	Concordant reading (ml)	Hardness in ppm
		Initial	Final			
1		0.0	12.2	12.2		
2	50	12.2	24.4	12.2	12.2	244
3		24.4	36.8	12.4		

1000 ml of 1 M EDTA solution = 100 g of $CaCO_3$

1 ml of 0.01 M EDTA = $\dfrac{100 \times 0.01}{100}$ g of $CaCO_3$.

12.2 ml 0.01 M EDTA = $\dfrac{100 \times 0.01 \times 12.2 \times 1000}{1000 \times 100}$

$= 12.2$ mg of $CaCO_3$.

50 ml of Tap water sample contains 12.2 mg of $CaCO_3$.

1000 ml " " " " " $\dfrac{12.2 \times 1000}{50}$

is titrated against 0·01(M) EDTA solution. At the
end point the colour changes from wine red to
blue. The titration is repeated three times and
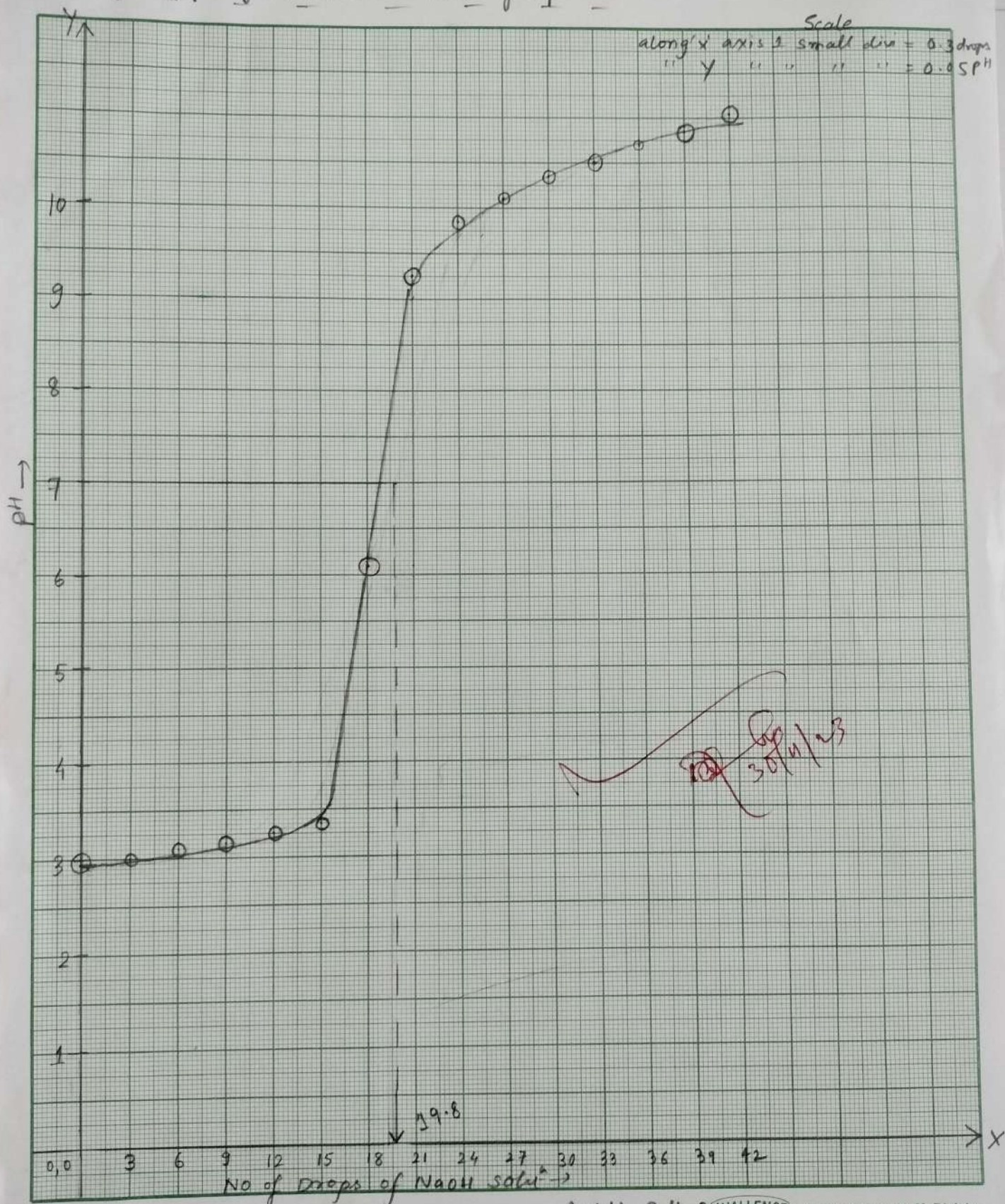reading is noted.

Table → Titration of HCl using NaOH →

Serial No.	No. of Drops of NaOH solution	pH
1	0	2.96
2	3	3.02
3	6	3.09
4	9	3.13
5	12	3.26
6	15	3.34
7	18	6.12
8	21	9.23
9	24	9.84
10	27	10.11
11	30	10.36
12	33	10.52
13	36	10.70
14	39	10.87
15	42	11.07

Drop counting :

20 drops = 1 ml

1 " = $\frac{1}{20}$ ml

No of Drops of NaOH sol^n Vs p^H graph.

Scale
along 'x' axis 1 small div = 0.3 drops
" y " " " = 0.05 p^H

↓ 19.8

No of Drops of NaOH solu^n →

AIML, Roll, 2

whereas a_{H^+} is the activity of the H^+ ions. For dilute solutions, activity can be replaced by concentration, i.e,

$$pH = -\log_{10}[H^+]$$

All pH meters have provision for standardizing the glass electrode in a buffer solution of known pH. This is necessary because different electrodes have different assymmetry potentials. One adjustment has been made, so that the registers correctly the known pH of the buffer solution, the instrument gives the pH of other solution without any calculation.

Measurement of pH is also employed to monitor the course of acid base titrations. The pH value of the solution at different stages of acid base neutralization is determined and is plotted against the volumes of the acid / alkali added. On adding a base to an acid, the pH rises slowly in the initial stages $pH = -\log_{10}[H^+]$; then it changes rapidly. However, the shape of the inflection point (i.e, where the pH changes abruptly) and symmetry of the curve on its two sides depends upon the ionizability of the acid and the base used and on the basicity of the acid and the acidity of the base.

if we know that about the concentration of H^+ ions. i.e. $[H^+]$, for a given solution the acidity of that solution can be expressed qualititatively.

Water is weakly ionized into H^+ ions and OH^- ions according to the following reaction :-

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

$$H_2O + H_2O \rightleftharpoons H_3O + OH^-$$
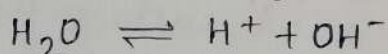
The ionic product of water, $K_W = [H^+][OH^-]$

For pure water $[H^+] = [OH^-] = 10^{-7}$

The H^+ ion concentration can be measured accurately using suitable methods. The acidic and alkaline nature of a solution can be expressed in the terms of concentration of H^+ ions. Due to some practical difficulties, the acidity or alkalinity of a solution is not expressed in terms of concentration of H^+ ions. Sorensen suggested the use of pH scale.

H^+	10^0	10^{-1}	10^{-2}	10^{-3}	10^{-4}	10^{-5}	10^{-6}	10^{-7}	10^{-8}	10^{-9}	10^{-10}	10^{-11}	10^{-12}	10^{-13}	10^{-14}
p^H	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14

$\longleftarrow$ Acidic $\longrightarrow$ Neutral $\longleftarrow$ Alkaline $\longrightarrow$

pH scale is a logarithmic scale. It is defined as the -ve logarithmic of the H^+ ion concentration. The pH scale has values between 0-14. On this scale, pH of any solution is defined as :-

$$pH = -\log_{10} a_{H^+}$$

Aparatus:-

- Burette
- Pipette (10ml)
- Digital pH Meter & pH Electrode
- Plastic Beaker (100ml)

Reagents:-

- Unknown HCl solution
- Standardized NaOH $\left(\frac{N}{10}\right)$ solution [supplied]

Procedure:-

1. Standarlized NaOH ($^N/_{10}$) solution is supplied.
2. Unknown HCl solution was supplied.
3. The pH electrode was rinsed with de-ionized water.
4. 10 C.C HCl solution was pipetted out into the plastic beaker and water was added if necessary, so that both the electrodes were completely immersed within the solution. The electrode was joined with the Digital pH Meter and the pH was measured very carefully.
5. NaOH solution was added from a buratte drop-wise (approximately 2-3 drops)
6. The pH value of the solution was measured after addition of 2-3 drops of NaOH and the pH electrode was mildly stirred. The process was repeated until you had at least

Calculation

V_1 = Volume of NaOH solution (result from graph $\times \frac{1}{20}$ ml)

V_2 = Volume of HCl = 10 ml

S_1 = Strength of NaOH = 0.11 (N)

S_2 = Strength of HCl = ?

$$V_1 S_1 = V_2 S_2$$

$$S_2 = \frac{V_1 \times S_1}{V_2}$$

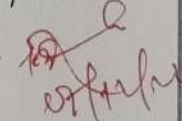$$\Rightarrow S_2 = \frac{19.8 \times \frac{1}{20} \times 0.11}{10} = \frac{0.99 \times 0.11}{10} (N)$$

$$\therefore S_2 = 0.01 (N)$$

Conclusion :

The unknown strength of HCl is determined by standardized NaOH solution with the help of a digital pH meter. Hence the unknown strength of HCl is — 0.01 (N).

Date of Experiment : 23-11-23

Date of Submission : 07-12-23

Signature of Teacher :-

five points beyond the end point.

7. A curve was drawn by plotting the pH value against the no of drops of the titrant, the end point was found and the strength of the HCl solution was calculated.

Discussion :-

1. NaOH is a secondary standard solution, so it should be standardized with primary standard Oxalic acid solution using formula

$$V_1 S_1 = V_2 S_2$$

2. No indicator is used during the titration of HCl against NaOH solution.

3. This experiment is more accurate as it is performed by a digital pH meter. Thers we can minimize human error.

4. After plotting the graph (drops of NaOH solution along x-axis and pH along y axis), we get the value of NaOH solution. So the strength of unknown HCl solution will be more accurate than other titration.

5. Titration is performed at room temperature ($25°- 30°c$)