MORADABAD INSTITUTE OF TECHNOLOGY
MORADABAD

Student Name :- Aryabh Prajapati

Father's Name :- Mr. Sanjay Babu

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Subject Teacher :- Dr. Harendra ~~Sharma~~ Kumar

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General instructions

1. laboratory coat must be worn at all times during the laboratory period.
2. Do not perform unauthorized experiment.
3. Never work alone in the laboratory.
4. At once report all incidents to your teacher.
5. Never taste a chemical or soln unless specifically directed to do so.
6. Be alert to what your laboratory neighbour is doing your neighbour accidents may be your injury.
7. Read the label carefully before removing a chemical from its shelf and keep the bottle at its appropriate place.
8. If any of your glass equipment is cracked or broken, do not use it.
9. If you need to observe the odour of a substance, gently fan a little of the vapour toward with your hand, but keep your face at a safe distance.
10. When you are heating a test tube, never point its mouth towards yourself or anyone else.
11. When diluting concentrated acid pour the acid slowly into water with stirring. Never add water to the acid.
12. Do not casually dispose of chemicals down the drain
13. Protect your hands if you have to pick up hot objects.
14. Do not force ~~computers~~ stoppers on the glass tubings.
15. Use no open flames near inflammable solvents.

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(16) Use fire extinguisher at the time of fire. Before you leave the laboratory make certain that any gas line you used in shut tightly.

Laboratory accidents and first aid :-

⇒ Fire :- ① For large fires get yourself and others away from burning area.
② Notify your instructor immediately.
③ Remove container or flammable solvent from the immediate area and turn off burners.
④ If your clothing on fire, do not run, roll on the floor to smother the fire and to help keep flames away from your hands.

⇒ Small Burns :- Immediately plunged the burned area into cold water preferably with ice in it.
⇒ Severe Burns :- Do not apply any ointment but seek professional medical treatment at once.

⇒ Chemical Burns :- ① General Burns :- Immediately wash of it with plenty of water.
② Acids = Wash with plenty of water then with very dilute sodium bisulphite solution.
③ Bases = Wash with plenty of water then with very dilute acetic acid solⁿ.

$$1-\ OH^- + H^+ = H_2O$$

$$2-\ CO_3^{--} + H^+ = HCO_3^-$$

$$3-\ HCO_3^- + H^+ = H_2O + CO_2$$

A = P

P = M

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Experiment - 1

- **Object** = To determine the amount of alkalinity of supplied water sample.

- **Apparatus used** = Burette, pipette, conical flask, funnel Beaker (250 ml).

- **Chemicals used** = N/20 HCL, phenopthalein & methyl orange.

- **Theory** - "The alkalinity of water may be defined as its capacity to neutralize acids". The alkalinity of water is mainly due to presence of hydroxide ions (OH^-), carbonate ions (CO_3^{2-}) & bicarbonate ions (HCO_3^-) in water.

These can be obtained separately by the titration against standard acid using phenopthalein & methyl orange as indicators. The various rxn involved are:

The titration of water samples against standard acid up-to phenopthalein end point marks the completion of rxn (i) and (ii) only. The volume of acids (A ml) used up-to this point corresponds to the complete neutralization of OH^- ions and one half of the normal CO_3^{2-} ions present. The alkalinity up-to phenopthalein end point is known as phenopthalein alkalinity [P].

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The titration of water sample against the standard acid up-to methyl orange end point shows the completition of rxn^s (i) (ii) and (iii). ∴ the amount of acid (B ml) carbonate and all the Bicarbonates present. The total amount of acid [(A+B) ml] in two titrations represents the totali alkalinity (M)

→ The possible combinations of ions causing alkalinity in water are :-

1- OH^- only
2- CO_3^{2-} only
3- HCO_3^- only
4- OH^- and CO_3^{2-} only
5- CO_3^{2-} and HCO_3^- only

The possibility of OH^- and HCO_3^- together is not possible since they combine together to form CO_3^{2-} and H_2O

$$OH^- + HCO_3^- = CO_3^{2-} + H_2O.$$

After knowing the values of P and M the nature of alkalinity can be determined by using the table

Results of titration to phenopthalein end point [P] & Methyl orange end point [M]	OH^-	CO_3^{2-}	HCO_3^-
[P] = 0	Nil	Nil	[M]
[P] = [M]	[P] or [M]	Nil	Nil.

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$[P] = 1/2\ [M]$	Nil	$2[P]$ or $[M]$	Nil
$[P] > 1/2\ [M]$	$2[P] - [M]$	$2\{[M] - [P]\}$	Nil
$[P] < 1/2\ [M]$	Nil	$2[P]$	$[M] - 2[P]$

→ Alkalinity is generally expressed as parts per million (ppm) in terms of $CaCO_3$.

• Observations -:

S.no	Volume of water Sample (ml)	Vol. of acid used upto phenopthalein End point [P]	Vol. of acid used upto methyl orange End point [M]
1 -	25 ml	3.9 ml	9.5 ml
2 -	25 ml	3.6 ml	8.5 ml
3 -	25 ml	3.5 ml	8 ml

Calculations -:

(i) Phenopthalein alkalinity [P] in terms of $CaCO_3$ equivalent

$$N_1V_1 = N_2V_2$$

(Acid) (Water Sample)

$$\frac{1}{20} \times 3.6 = N_2 \times 25$$

$$N = \frac{1}{500} \times 3.6$$

Strength in terms of $CaCO_3$ equivalent

$= N_2 \times$ Equivalent weight of $CaCO_3$

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$= \frac{3.6}{500} \times 50$ gm/l

$= .36$ gm/l

$= .36 \times 1000$ mg/l or ppm

$= 360$ mg/l or ppm

(ii) Methyl orange alkalinity [M] in terms of $CaCO_3$ equivalent.

$N_3V_3 = N_4V_4$

(Acid) (Water sample)

$\frac{1}{20} \times 8 = N_4 \times V_4$

$\frac{1 \times 2}{5} = N_4 \times 25$

$\frac{2}{125} = N_4$

Strength in terms of $CaCO_3$ equivalent -

$= N_4 \times$ Eq. weight of $CaCO_3$

$= \frac{2}{\cancel{125}\,5} \times 50$

$= \frac{4}{5}$ gm/l

$= 800$ mg/l or ppm.

- Important Note = To calculate the alkalinity in terms of individual ions, find out ~~the~~ to which cases

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the values of P and M falls from table A.

(i) If $P = 0$ → Alkalinity is only due to bicarbonate ions
→ Bi-carbonate alkalinity in terms of $CaCO_3$ eq. = M

(ii) If $P = 1/2\ M$ → Alkalinity is only due to carbonate ions
→ Carbonate alkalinity in term of $CaCO_3$ eq. = 2P or M

(iii) If $P = M$ → Alkalinity is only due to hydroxide ions
→ Hydroxide alkalinity in terms of $CaCO_3$ eq = P or M.

(iv) If $P > 1/2\ M$ → Alkalinity is due to hydroxide and carbonate ions
a) Hydroxide alkalinity in terms of $CaCO_3$ eq. = (2P-M)
b) Carbonate alkalinity in terms of $CaCO_3$ eq. = 2(M-P)

(v) If $P < 1/2\ M$ → Alkalinity is due to ~~hydroxide~~ carbonate & bicarbonate ions
a) Carbonate alkalinity in terms of $CaCO_3$ eq. = 2P
b) Bicarbonate alkalinity in terms of $CaCO_3$ eq. = [M]-2[P]

- **Result** = The given water sample has:
  * alkalinity due to OH^- = Nil
  * alkalinity due to CO_3^{2-} = 2 [P] = 2×360 = 720

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As $P < \frac{1}{2}M$; * Alkalinity due to HCO_3^- = 800 - 2×360
= 80

→ Total alkalinity = Alkalinity due to ($CO_3^{2-} + HCO_3^- + OH^-$)
= ~~640~~ 720 + 80
= 800 ppm

• Precautions =

1) The glass apparatus should be cleaned and properly rinsed before the start of experiment.
2) HCL level in burette must be read carefully.
3) Volume of HCl should be same in all titrations.

• Viva Voice = Q1- What is alkalinity?
Ans = Alkalinity is the measure of the ability of a solution to neutralize acids.

Q2- Why alkalinity is not due to the simultaneously presence of OH and HCO_3^- ?
Ans- The alkalinity is not due to the OH^- and HCO_3^- because they combine together to form CO_3^{2-} and H_2O.

$$OH^- + HCO_3^- = CO_3^{2-} + H_2O$$

Q3- Write the PH range of Phenop. & methyl orange?
Ans- Phenopthalein = 8.3 – 10.0
Methyl orange = 3.1 – 4.4

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Experiment - 2

- **Object** = To determine the temporary or permanent hardness of water by using complexometric titration.

- **Apparatus used** = Burette, conical flask, funnel, pipette Beaker, etc.

- **Chemicals used** = EDTA solⁿ (0.01)M, Buffer Solution (NH_4OH + NH_4Cl, Eriochrome Black-T (EBT), water sample.

- **Theory** = The hardness of water can be determined by complexometric titration. EDTA is used as complexing ~~-ometric ometric~~ reagent. The Ca^{2+} and Mg^{2+} present in water are titrated with EDTA using eriochrome Black-T as indicator.

Estimation of hardness by EDTA method is based on the following principle.

First, the indicator eriochrome Black-T which is Blue coloured dye, from an unstable complex with calcium or magnesium ions in hard water at PH of 9 to 10. The complex is wine red in colour.

$$Mg^{2+}/Ca^{2+} + EBT \longrightarrow [Mg^{2+}/Ca^{2+} - EBT]$$

unstable complex (wine red)

As this solⁿ is titrated against EDTA, the free Ca^{2+} and Mg^{2+} ions in water form stable metal

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ion EDTA complex, which is colourless.

$$[Mg^{2+} / Ca^{2+}] + EDTA \rightarrow [Mg^{2+} / Ca^{2+} - EDTA]$$

stable complex (colourless)

Once the free metal ions are complexed, the EDTA replaced Ca^{2+} and Mg^{2+} ions from the unstable indicator complex also, to form a stable complex with the result, the indicator is set free. Since the free indicator is blue in colour at the above at the above mentioned PH, the end point is the appearance of blue colour.

$$[Mg^{2+}/Ca^{2+}-EBT] + EDTA \rightarrow [Mg^{2+}/Ca^{2+} - EDTA] + EBT \text{ (Blue)}$$

Thus the amount of EDTA used corresponds to the hardness of water. The temporary hardness of water is removed by boiling and after the removal of ppt by filtration, the permanent hardness in the filtrate is determined by titration with EDTA as above. Temporary hardness will be given by the difference of total hardness and permanent hardness.

Observations :-

- Total hardness (before boiling)

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S.no	Volume of water sample taken (ml)	Burette Reading			Actual volume of EDTA used
		Initial	Final	Difference	
1	25 ml	0	16	16	
2	25 ml	16	32	16	a = 16 ml
3	25 ml	32	48.2	16.2	

- Permanent hardness (after boiling)

S.no	Volume of water sample taken (ml)	Burette Reading			Actual volume of EDTA used.
		Initial	Final	Difference	
1	25 ml	0	14	14	
2	25 ml	14	28	14	b = 14 m
3	25 ml	28	40.5	12.5	

- <u>Calculations</u> = EDTA forms 1:1 complex with Ca^{2+} and Mg^{2+}

$\therefore$ 1000 ml of 1M EDTA = 1 mole of $CaCO_3$
= 100 gm of $CaCO_3$

$\therefore$ 1 ml of 0.01 EDTA = $100 \times \frac{1}{1000} \times \frac{1}{100}$ gm of $CaCO_3$

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= 1mg of $CaCO_3$.

Total hardness = 16 ml of 0.01 EDTA = 1×16
= 16mg of $CaCO_3$

This hardness is present in 25 ml of sample water.
So, hardness present in 1000 ml of sample water
$= 1000 \times \frac{16}{25}$

$= 40 \times 16 = 640$ gm of $CaCO_3$

∴ Total hardness = 640 mg/lit or ppm

Permanent hardness = 14ml of .01 EDTA = 1×14 = 14mg of $CaCO_3$

This hardness is present in 25 ml of sample water
So, hardness present in ~~25 ml~~ 1000ml of sample water
$= 1000 \times \frac{14}{25}$

$= 40 \times 14 = 560$ gm of $CaCO_3$

Temporary hardness = (Total − Permanent) hardness
= 640 − 560
= 80 mg/lit or ppm.

- **Result** Total hardness = 640 ppm
  Permanent hardness = 560 ppm
  Temporary hardness = 80 ppm

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- **Precautions**

1) The glass apparatus should be cleaned before starting the experiment.
2) The pH should be maintained during titration.
3) Hard water sample should be boiled properly.

- **Viva Voice :-**

Q1- How will you define hardness?
Ans- The hardness of water is defined as the presence of (Ca^{2+}) and (Mg^{2+}) in water.

Q2- What is the cause of temporary hardness in water?
Ans Temporary hardness is due to presence of $Ca(HCO_3)_2$ and $Mg(HCO_3)_2$ in water

Q3- How will you remove the temporary hardness?
Ans- It is removed by heating the water as calcium & magnesium carbonates are precipitated out of water and are collected as residue.

Q4- What do you understand by permanent hardness?
Ans- It is the presence of chlorides, nitrates and sulphates of calcium and magnesium in water.

Q5- What is the effect on the end point with eriochrome black-T indicated, if the hard water sample does

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not contain Mg^{2+} / Ca^{2+} ?

Ans- EBT will not change its colour to wine red. The Sol^n will be colour blue. Titration will not occur.

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

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

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

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

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Experiment - 3

Object - To determine iron (ferrous) content in the supplied sample of iron ore by titrimetric analysis against standard $K_2Cr_2O_7$ solution using $K_3[Fe(CN)_6]$ as an external indicator.

Apparatus used :- Burette, pipette, conical flask, beaker, glass rod, watch glass, funnel.

Chemicals used - Iron ore sample, standard potassium dichromate solution (N/10), ~~pottast~~ potassium ferricyanide

Theory = Potassium dichromate solution in presence of H_2SO_4 is a strong oxidizing agent which converts ferrous ions (Fe^{2+}) present in the iron ore into ferric ions (Fe^{3+}). The end point can be determined by using potassium ferricyanide as an external indicator as after the completion of rxn, no Fe^{2+} will be present in the rxn thereby no greenish blue coloration is produced.

Observations :-

S.no	Vol. of Sample soln	Reading of Burette (ml)			Actual volume of $K_2Cr_2O_7$ used (ml)
		Initial	Final	Difference	
1	25ml	0	12	12	
2	25 ml	12	24.5	12.5	12.5
3	25 ml	24.5	37	12.5	

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Calculations :-

$$N_1 V_1 = N_2 V_2$$

$$\left(\text{Mohr Salt Soln}\right) = K_2Cr_2O_7$$

$$N_1 \times 25 = \frac{N}{10} \times 12.5$$

$$N_1 = \frac{12.5}{25 \times 10}$$

Strength of given iron ore soln = Normality × Eq. weight of mohr salt.

$$= \frac{12.5}{25 \times 10} \times 392.12 \text{ gm/lit.}$$

$$= 19.606 \text{ gm/Lit.}$$

As 392.12 gm of Mohr salt contain = 56 gm fe
So, 19.606 gm of Mohr salt contain $= \frac{56 \times 392.12}{19.606}$ gm
$= 2.8$ gm

Result = The ferrous content in ~~the~~ supplied sample of iron ore is 2.8 gm.

Precautions ① Potassium dichromate acts as an oxidising agent in acidic medium, ∴ add dilute H_2SO_4 in mohr salt soln.

② The vol. of soln taken out for checking end point should not be more otherwise the result

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will not give accurate value.

(3) The glass rod should be washed each time before withdrawing a drop from the conical flask.

(4) Rinse the burette and pipette properly before starting the titration.

Viva Voice

Q1- What is the formula of ferrous ammonium sulphate?

Ans- $(NH_4)_2 Fe(SO_4)_2 \cdot 6H_2O$

Q2- What are the main ores of iron?

Ans- Haematite, Magnetite, limonite, siderite.

Q3- What types of indicator are using in this titration?

Ans- $K_3[Fe(CN)_6]$ as an external indicator.

(8)

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Experiment - 4

Object = To determine the viscosity of a liquid with the help of a ostwald's viscometer.

~~Theory~~ **Apparatus Used** = Ostwald's viscometer, Specific gravity bottle, stopwatch, beaker, etc.

Theory = Viscosity of liquid is a measure of its frictional resistance. let 'l' be length of the capillary and r its radius, then the volume of liquid flowing through the capillary tube for difference of pressure P is given by the formula.

$$V = \frac{\pi P r^4}{8 n l}$$

where n is the viscosity of a liquid taken in viscometer.

For first liquid $V_1 = \frac{\pi P_1 r^4}{8 n_1 l}$ — (1)

For second liquid $V_2 = \frac{\pi P_2 r^4}{8 n_2 l}$ — (2)

i.e. $\frac{V_1}{V_2} = \frac{P_1}{P_2} \times \frac{n_2}{n_1}$ — (3)

Now the pressure of the liquids $P = hdg$ or $h\rho g$ Since h and g are same for two liquids.

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Hence $P \propto \rho$

Pressure of the liquid in each case is proportional to the density of liquid. $P_1 \propto \rho_1$ $P_2 \propto \rho_2$

$$\frac{P_1}{P_2} = \frac{\rho_1}{\rho_2} \quad \text{--- (4)}$$

Let Q be the volume of each liquid flowing through the capillary tube in time t_1 and t_2 Then -

$$V_1 = \frac{Q}{t_1} \quad \& \quad V_2 = \frac{Q}{t_2}$$

$$\frac{V_1}{V_2} = \frac{t_2}{t_1} \quad \text{--- (5)}$$

Substituting the value of $\frac{P_1}{P_2}$ and $\frac{V_1}{V_2}$ from (4) & (5) respectively in the equation (3) we get :-

$$\frac{t_2}{t_1} = \frac{\rho_1}{\rho_2} \times \frac{\eta_2}{\eta_1}$$

$$\boxed{\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2}}$$

Thus the comparision of viscosities is made by knowing the times for corresponding flow and the densities of the liquids.

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Observations :- (a) Table of measurement of flow time [t_1 and t_2]

S.no	Water: Flow time b/w A and B	Water: Mean	Liquid: Flow time b/w A and B	Liquid: Mean
1	53 sec		65 sec	
2	50 sec	50.2	65 sec	63.8
3	50 sec		63 sec	
4	49 sec		64 sec	
5	49 sec		62 sec	

(b) Table for measurement of density (ρ_2)

Weight of empty RD bottle → 21 gm
Weight of RD bottle + water → 50.14 gm
Weight of RD bottle + liquid → 46.30 gm
Weight of water filled → 29.14
Weight of liquid filled → 25.3

Density of liquid $\rho_2 = \frac{25.3}{29.14} = .86$

(c) Room temperature = 20°c
(d) Density of water = $\rho_1 = 1.00$
(e) Viscosity of water at 20°C = 0.0101 poise

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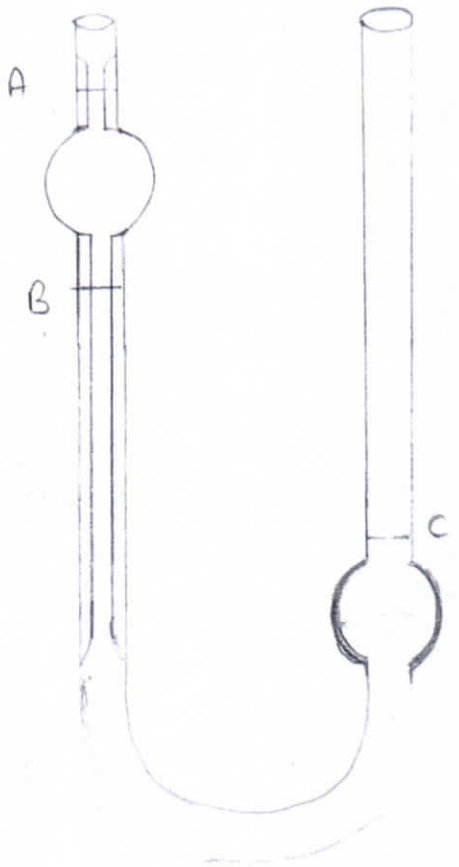

Ostwald's Viscometer

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Calculations :- The viscosity of liquid is calculated from the following formula assuming η_1 and ρ_1 of water 0.0101 and 1.00 respectively at 20° c.

$$\frac{\eta_1}{\eta_2} = \frac{t_1}{t_2} \times \frac{\rho_1}{\rho_2}$$

viscosity of liquid $\eta_2 = \frac{\eta_1 \times t_2 \times \rho_2}{t_1 \times \rho_1}$

$$= \frac{0.0101 \times 63.8 \times .86}{50.2 \times 1.00}$$

$$= \frac{.55}{50.2}$$

$\eta_2 = 0.0109$ poise.

Result → Viscosity of liquid at 20°c is 0.0109 poise.

Precautions →

① The flow time of a liquid filled in viscometer should be between 1 to 10 minutes.

② The viscometer and RD bottle should be thoroughly cleaned

③ The viscometer should be adjusted in any accurately vertical position.

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(4) Five or six observations should be recorded in each case.

Viva Voice :-

Q1- What is viscosity ?
Ans- The viscosity of a fluid is a measure of its resistance to flow at a given rate.

Q2- What is unit of viscosity ?
Ans- Poise (dyne s/cm²)

Q3- What is the effect of temperature on viscosity ?
Ans- On high temperature viscosity increases in gases but decreases in liquids.

Q4- Which is more viscous honey or water ?
Ans- Honey because it has more strong intermolecular attractive force than water.

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Experiment - 5

Object = To study variation of surface tension of liquid on addition of soap or surfactant of known concentration

Apparatus Required = Stalagometer.

Chemicals Used = (i) Reference liquid (H_2O)
(ii) Experimental liquid (Ethyl alcohol)

Theory = Surface tension = The force in newton acting at right angles along the surface of a liquid one metre in length.
Units = N/m SI system or dynes/cm in CGS system.

~~Procedure~~ = Observations :-

Density of water (25°c) =
Density of liquid (25°c) =
Surface tension of water (25°c) =

Observation table :-

S.no	Water		Given liquid	
	No. of drops	Mean	No. of drops	Mean
1)	40		72	
2)	36	38	71	71
3)	38		67	
	38		71	

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Calculations :-

Surface tension of experimental liquid

$$= \frac{\text{No. of drops of } H_2O}{\text{No. of drops of exp. liq.}} \times \frac{y}{x} \times \text{Surface tension of water (dyne/cm)}$$

$$= \frac{38}{71} \times \frac{1.0021089}{1} \times 72.14$$

$$= 38.6915 \text{ dynes/cm}$$

Result :-

The surface tension of given liquid are found to be 38.6915 dyne/cm

Precautions :-

1. Before use, the stalagmometer should be cleaned and dry.
2. No air bubble should be formed while sucking the liquid into the stalagmometer.
3. Throughout the drop counting the process, the stalagmometer should be held in a vertical position.
4. Drop formation should be adjusted at a slower rate and should not exceed fifteen drops per min.

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Viva- Voce :-

Q2- What is surface energy?
Ans- Potential energy per unit area of the liquid surface under isothermal conditions is known as surface energy.

Q3- What is the effect of temperature on surface tension?
Ans- Surface tension decreases when temp^r increases and vice-versa.

Q4- Give some applications of surface tension?
Ans- ① A needle placed on water can be made to float due to the surface tension of water.

② Surface tension prevents water from passing through the pores of an umbrella.

③ Antiseptics like dettol have low surface tension so that the spread faster.

Q1- What are the effects of surface tension?
Ans- The effect is an inward force at its surface that causes the liquid to behave as if its surface were covered with a stretched elastic membrane.

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$$NaCl + AgNO_3 \longrightarrow AgCl\downarrow + NaNO_3$$
(White ppt)

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$

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

$$2\,AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$
(Reddish brown)

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Experiment - 6

Object = To determine the chloride content in given water sample by Mohr's method.

Apparatus used = Burette, pipette, conical flask, Beaker, volumetric flask.

Chemicals used = Standard (N/20) $AgNO_3$ Sol^n, Water sample, potassium chromate.

Theory - Chlorides usually occur in water as NaCl, $CaCl_2$, $MgCl_2$. Chloride content in water sample is determined by Mohr's method. A weakly alkaline ~~method~~ sol^n (7-8) pH of the water sample is titrated against standard silver nitrate solution using K_2CrO_4 as internal indicator. Cl^- ions present in water sample react with Ag^+ ions (coming from $AgNO_3$ sol^n) forming insoluble white PPT of AgCl. CrO_4^{2-} ions form from indicator also react with Ag^+ ions giving transient red colour, which immediately disappears due to presence of greater concentration of Cl^- ions.

As the titrations proceeds all the chlorides ions are removed as AgCl, any extra drop of $AgNO_3$ reacts with ~~potas~~ potassium chromate giving reddish brown colour of Ag_2CrO_4. The formation of reddish brown colour indicates the end point.

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Observations :- • Titration with water sample -

S.no	Vol. of Water Sample(ml)	Burette Reading (ml)			Vol. of $AgNO_3$ used
		Initial	Final	Difference	
	25	0	6.5	6.5	
1	25	6.5	13	6.5	
2	25	13	19.5	6.5	6.5 ml
3	25	19.5	26	6.5	
4	~~25~~	~~26~~			

• Titration with Distilled water (Blank titration)

S.no	Volume of water Sample ml	Burette Reading (ml)			Volume of $AgNO_3$ used
		Initial	Final	Difference	
1	25	26	27	1.0	
2	25	27	27.5	.5	.5 ml
3	25	27.5	28	.5	
4	25	28	28.8	.8	

Calculations = $N_1 V_1 = N_2 V_2$
(Water sample) ($AgNO_3$ sol^n)

$\Rightarrow$ where $V_2 = 6.5 - .5 = 6$ ml

$\Rightarrow N_1 \times 25 = \frac{1}{20} \times 6 = .012$

$\Rightarrow$ Strength of Cl^- ions = ~~.012~~ $N_1 \times$ Eq. weight
= $.012 \times 35.5$ gm/l
= .426 gm/l
= 426 mg/l or ppm

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Result = The chloride ion conc. in the given water sample is 426 mg/l or ppm

Precautions = ① The glass apparatus should be cleaned with distilled water.

② The rxn content should be continously shaken during titration.

③ Equal volume of water sample and distilled water should be taken.

④ Equal drops of indicator should be used in all titration

⑤ The PH of soln must be maintained b/w 7-8.

Viva Voce

Q1- Name the forms in which Cl^- ions are present?
Ans- NaCl, $CaCl_2$, $MgCl_2$

Q2- Name the indicator used in this titration?
Ans- K_2CrO_4

Q3- What is conc. of Cl^-, which gives unacceptable taste of water?
Ans- Above 250 mg/l conc. of Cl^- it gives salty taste to water.

Q4- What is equivalent weight of Cl^- ions?
Ans- 35.5 gm/l.

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$$Ca(OCl)Cl + 4HCl \longrightarrow CaCl_2 + 2H_2O + 2Cl_2$$

$$Ca(OCl)Cl + H_2SO_4 \longrightarrow CaSO_4 + 2H_2O + Cl_2$$

$$Ca(OCl)Cl + 2CH_3COOH \longrightarrow Ca(CH_3COO)_2 + H_2O + Cl_2$$

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Experiment - 7

Object = To determine the percentage of available chlorine in a given sample of bleaching powder.

Chemicals used = Bleaching powder potassium iodide, dilute ~~acid~~ acetic acid, N/10 sodium thiosulphate soln, distilled water and freshly prepared starch soln.

Apparatus = Burette, pipette, measuring flask, conical flask, funnel, pastel and mortar.

Theory = Bleaching powder is famous for its bleaching action. It is also used as disinfectant. The main constituents of bleaching powder are calcium hypochloride [$Ca(OCl)Cl.4H_2O$], the basic calcium chloride [$CaCl_2.Ca(OH)_2.H_2O$] and some free calcium Hydroxide. [$Ca(OH)_2$]. Out of these constituents, the active constituents of bleaching powder is hypochloride. Hypochloride may be represented as:

$$OCl-Ca-Cl.$$

When dilute acid reacts with bleaching powder then free chlorine is liberated acc. to the following rxn:-

The amount of chlorine that can be obtained by action of dilute acids on bleaching powder is known as available chlorine. It is expressed as %.

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weight of bleaching powder.

Observations :

(a) Weight of empty weight tube = 3.150 gm
(b) Weight of weighing tube + weight of bleaching powder = 6.75 gm
(c) Weight of bleaching powder = 3.6 gm

S.no	Vol. of Bleaching Powder.	Burette Reading (ml)			Vol. of Hypo used (ml)
		Initial	Final	Difference	
1	25 ml	0	38.5	38.5	
2	25 ml	0	38	38	38 ml
3	25 ml	0	37.5	37.5	
4	25 ml	0	38	38	

Calculations $N_1V_1 = N_2V_2$
(Bleaching powder) (Sodium thiosulphate)

Normality of available chlorine = $N_1 = (N_2 \times V_2)/V_1$

$N_1 = \frac{N}{10} \times \frac{38}{25} = 0.152$

z = Amount of available chlorine per 250 ml of solⁿ = $N_1 \times 35.5 = 35.5 \times 0.152$
= 5.396 gm/250ml

Amount of avail. chlorine / litre of solⁿ = $\frac{z \times 250/1000}{4}$ = 1.349 gm/lit

Percentage of available chlorine = $\frac{z}{4}(100/w) = \frac{1.349}{3.6} \times 100 = 37.47\%$

[w = (weight of bleaching powder)]

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Result = The percentage of available chlorine present in given sample of bleaching powder sample is 37.47%

Precautions : ① Sample of bleaching powder must not be kept as open as it absorbs moisture from the atmosphere.

② No lumps of bleaching powder should remain while making the paste.

③ Rinse the burette with standard N/10 sodium thiosulphate soln.

④ The starch soln and acetic acid must be added in equal amount in each experiment.

⑤ Titration must be started immediately after the addition of acid because chlorine is liberated instantly.

Viva Voce

Q1- What do you mean by available chlorine?

Ans- The amount of chlorine that can be obtained by the action of dilute acids on bleaching powder is known as available chlorine.

Q2- What do you understand by iodometry?

Ans- It is a iodometric titration, a redox titration where the appearance or disappearance of elementary

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iodine indicates the end point.

Q3- What is the other name of bleaching powder?
Ans- Calcium Hypochloride

Q4- What is the app. percentage of available chlorine in commercial bleaching powder?
Ans- ~~35%~~ 10 - 40%

Q5- Give the names of main constituents of bleaching powder?
Ans- Calcium ~~hypochloroid~~ chloride [$Ca(OCl)Cl.4H_2O$]
Basic calcium chloride [$CaCl_2 . Ca(OH)_2 . H_2O$]
free calcium hydroxide [$Ca(OH)_2$]

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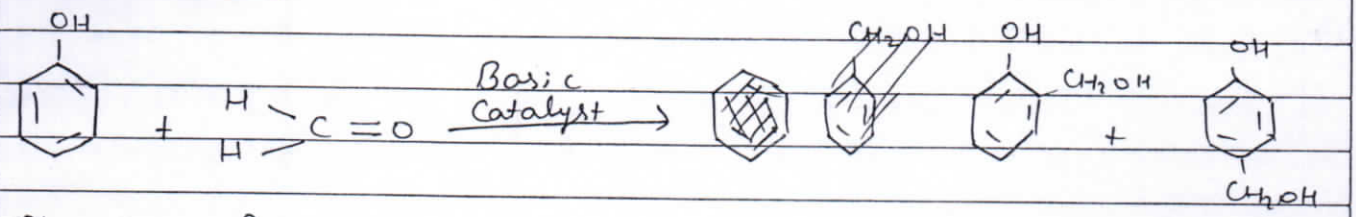
Experiment - 8

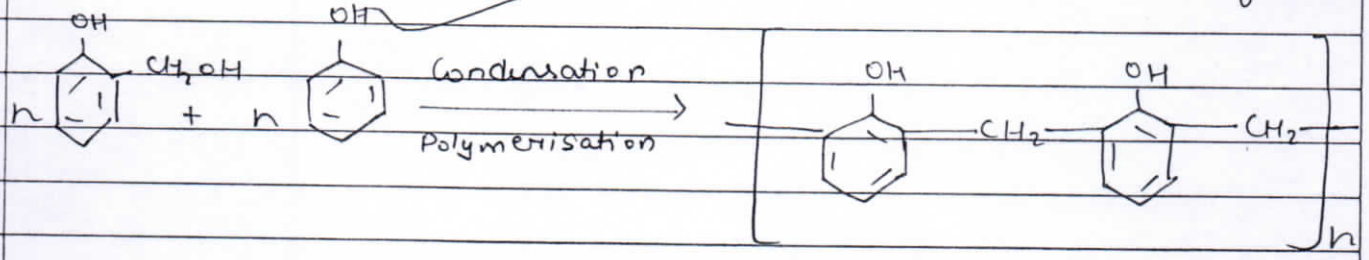
Object - To prepare phenol formaldehyde resin bakelite.

Requirements - Beaker, measuring cylinder, weight box, chemical balance, Filter paper, Formaldehyde, Glacial acetic acid, Phenol, conc. HCl

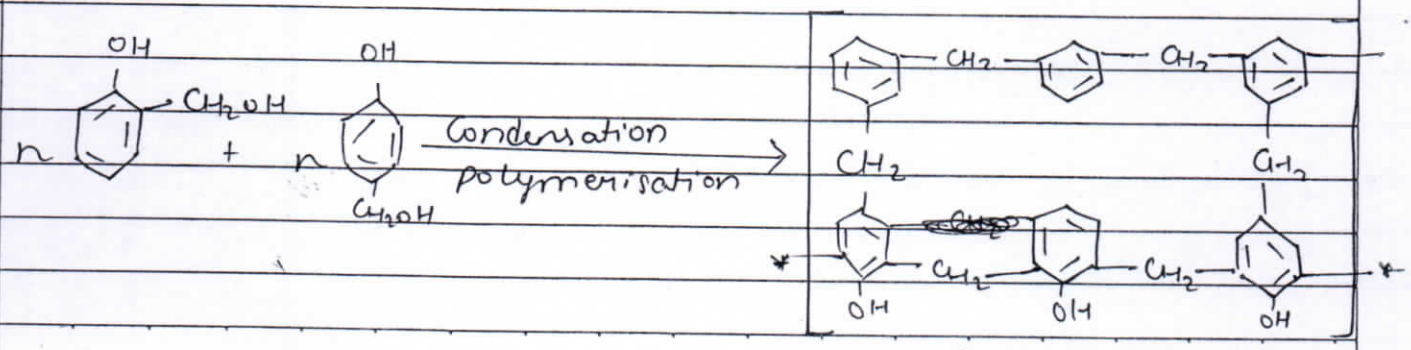
Reactions



Phenol Formaldehyde O-Hydroxy methyl Phenol P-Hydroxy Methylphenol



Novalac linear polymer



Bakelite (cross linked polymer)

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~~Result~~ = ~~The pink material is obtained bakelite resin.~~

~~Yield~~ Procedure = Place 5 ml of glacial acetic acid and 2.5 ml of formaldehyde solution in a 500 ml beaker and add 2gm of Phenol. Then add conc. HCL dropwise into the mixture carefully. Keep the above mixture for 5 minutes. Firstly it will form white ppt later on pink colour hard mass is obtained. Wash the hard mass with distilled water to remove excess of impurities. Then remove the hard mass into the watch glass, dry and weight.

Result = Pink material is obtained

yield = 30 gm

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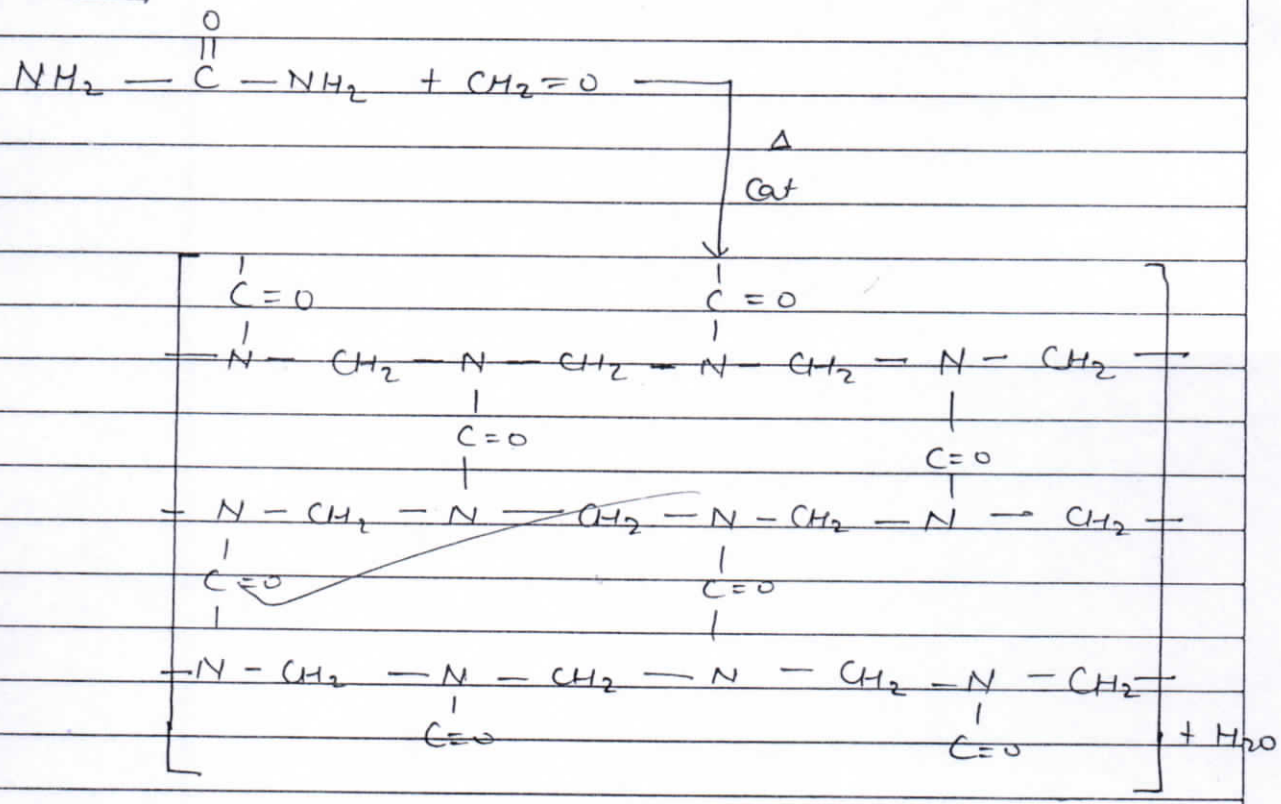
Object = To prepare urea formaldehyde (UF) resin

Reagent and apparatus = Urea formaldehyde, conc. H_2SO_4, etc.

Reactions -



Urea formaldehyde (UF) resin

Procedure = Place 20 ml of 40% formaldehyde soln in 500 ml Beaker. Add about 10gms of urea while stirring until a saturated soln is obtained. Add few drops of conc. H_2SO_4. Stir continously during the addition. All the

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voluminous white solid mass appears in the beaker. When rxn is complete, wash the residue with water and dry the product.

Result = white colour solid plastic.

yield = 28.18 gm

Precautions = ① The glass apparatus should be cleaned and rinsed properly.

② H_2SO_4 should be mixed drop wise and stir continously during the addition.

③ The reaction is sometimes vigourous and it better to be a few feet away from the beaker while adding the HCl and untill the rxn is complete.

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Experiment - 9

Object = To detect the elements and functional groups in given organic compound.

Detection of elements - Generally the organic compⁿ have, C, H and oxygen. In addition to these three elements halogen, Nitrogen, and sulphur may also be present. Firstly these elements are converted are converted into ionisable inorganic salts by fusion of the organic compound with metallic sodium. Detection of Nitrogen, sulphur, and halogens by this method is known as Lassaigne's test.

Apparatus

~~Chemicals~~ Used = Test tube ignition tube, tong and porcelain dish, beaker, funnel etc.

Chemicals used = Sodium metal, ferrous sulphate solⁿ, dil. H_2SO_4, $FeCl_3$ solution, Sodium nitro nitroprusside solⁿ, dilute acetic acid, lead acetate solⁿ, Conc. HNO_3, $AgNO_3$ solⁿ, dil HNO_3, chloroform, carbon tetra chloride, chlorine water dil. HCl., ethyl ~~HCl~~ alcohol, acetone, cobalt nitrate solution.

Lassaigne's Test = Take a piece of dry sodium metal into a clean ignition tube and cover it with a piece of organic compound. Heat

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$Na + C + N \longrightarrow NaCN$ (Sodium Cyanide)

$Na + X \longrightarrow NaX$ (Sodium Halide)

$(X = Cl, Br, F)$

$2Na + S \longrightarrow Na_2S$ (Sodium Sulphide)

If N and S are both present together

$Na + C + N + S \longrightarrow NaCNS$

(Sodium thiocyanate)

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gently in the flame initially and then strongly untill the bottom of the tube becomes red hot. Plunge the hot tube into a porcelain dish containing 15 ml of cold distilled water. Grind the contents with a pestle, boil the mixture for 5 ~~mixture~~ minutes and filter. The filtrate is known as sodium extract, which is then used for elemental detection.

← Reactions Involved =

Reactions involved

Nitrogen:

$FeSO_4 + 2NaOH \longrightarrow Fe(OH)_2 + Na_2SO_4$

Ferrous hydroxide (dirty green)

$Fe(OH)_2 + 6NaCN \longrightarrow Na_4[Fe(CN)_6] + 2NaOH$

Sodium Ferrocyanide

$3Na_4[Fe(CN)_6] + 4FeCl_3 \longrightarrow Fe_4[Fe(CN)_6]_3 + 12NaCl$

Ferric ~~ferry~~ ferrocyanide (Prussian blue)

Both N and S:

$3NaCNS + FeCl_3 \longrightarrow Fe(CNS)_3 + 3NaCl$

Ferric Sulphocyanide (blood red)

$Na_2S + Na_2[Fe(CN)_5NO] \longrightarrow Na_4[Fe(CN)_5]\cdot NoS$

Sodium Nitroprusside Sodium ~~So~~ Sulphonitroprusside (violet)

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Halogens:

① Chlorine -

$AgNO_3 + NaCl \longrightarrow AgCl\downarrow + NaNO_3$
(white ppt)

$AgCl + 2NH_4OH \longrightarrow [Ag(NH_3)_2]Cl + 2H_2O$
(Soluble)

② Bromine:

$AgNO_3 + NaBr \longrightarrow AgBr\downarrow + NaNO_3$
(pale yellow ppt)

$AgBr + 2NH_4OH \longrightarrow [Ag(NH_2)_2]Br_2 + 2H_2O$
(partially soluble)

③ Iodine:

$AgNO_3 + \overset{NaI}{\cancel{AgI}} \longrightarrow AgI\downarrow + NaNO_3$
(yellow ppt)

$AgI + NH_4OH \longrightarrow$ Insoluble.

④ Iodine ~~and~~ Bromine =

$2NaI + Cl_2 \longrightarrow 2NaCl + I_2$
(violet)

$2NaBr + Cl_2 \longrightarrow 2NaCl + Br_2$
(brown)

Detection of functional group : In the identification of organic compound the detection of functional groups is an imp. step. The organic compounds.

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are divided into groups that exhibit similar chemical reactions and each group has a structural feature common to all organic molecules. This common structural feature is known as functional group.

Apparatus used = Beaker, test tube, boiling tube, funnel, burner, tripod stand, wire gauge, etc.

Chemicals used = Sodium bicarbonate solution, natural ferric chloride solution, sodium nitrite, sulphuric acid, sodium hydroxide, pthalic anhydride, sodium metal, Na_2SO_4 Glacial acetic acid, ceric ammonium nitrate, ethanol, 2-4,-dinitrophenyl hydrazine, β-napthol, $CHCl_3$, alc. KOH, phenol, etc.

Table: 1

S.no	Experiment	Observation	Inference
	Carboxylic Group (-COOH)		
i)	Litmus test = Place a crystal or solid sample or a drop of the given liquid sample on moist blue litmus paper.	Turns red	-COOH group present.
ii)	Sodium bicarbonate test = take 2-3 ml of 10% solution of	Brisk effervescence	-COOH group present.

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	Sodium bicarbonate in a dry test tube add a pinch of given compound.		
(iii)	Ester formation = Take small amount of sample compound in a dry test tube, add 2ml of ethyl alcohol and add 2-3 drops of conc. H_2SO_4 and warm on a water bath	A fruity smell	-COOH group present.

Reactions involved :-

(i) Litmus test : Blue → Red

(ii) Sodium Bi-carbonate test :

$$RCOOH + NaHCO_3 \longrightarrow RCOONa + H_2 + CO_2$$

Effervescence

(iii) Ester formation :

$$RCOOH + R'OH \longrightarrow RCOOR' + H_2O$$

Ester fruity smell.

	Phenolic Group -		
i)	Neutral $FeCl_3$ test - Take 1 ml of neutral ferric chloride solution in cleaned and dry test tube and add 3-4 drops of a little bit of the organic compound shake well.	Violet / red / blue / Green / Colour	Phenolic Group present.

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ii)	Litmus test: put a pinch of the organic compound on a moist blue litmus paper.	Turns red	Phenolic group present
iii)	Pthalein test: Take .5g of organic compound in a cleaned and dry test tube, add 1 gm of pthalic anhydride and 2 drops of concentrated H_2SO_4 heat gently for 1 minute. Cool and pour the mixture into a beaker containing 400ml of water and make it alkine by adding NaOH	Red / pink blue Resorcinol α-napthol β-napthol	phenol, o-cresol, catechol m-cresol Fluroscent Green, Green, Faint Green.
iv)	Liberman's Nitroso test: Heat a small amount of the organic compound and few crystals of $NaNO_2$ Cool and add 1ml of concentrated H_2SO_4 and excess of water and excess of NaOH solution	A deep green to blue solⁿ is formed which turns red. Deep green to blue colour appears	

Reactions involved:

i) Litmus test: Blue → Red

ii) Neutral $FeCl_3$ test →

$$6\,C_6H_5OH + FeCl_3 \longrightarrow [(C_6H_5O)_6Fe]^{3-} + 3H^+ + 3HCl$$

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Table 2

S.No	Experiment	Observation	Inference
	Alcoholic group (OH)		
i)	Ceric ammonium nitrate test : Take 1 ml of a organic compound in a cleaned and dry test tube, add 3-4 drops of ceric ammonium nitrate solution and shake well.	Red colour	Alcoholic group present.
ii)	Ester test - Heat 1 ml of organic compound, 1 ml of glacial acetic acid and 3-4 drops of conc. H_2SO_4 in a dry test tube for 10 minutes. Now pour the reactions content into a beaker containing 20 ml of cold water	A fruity smell	Alcoholic group present

Reactions Involved :

i) Na Metal test :-

$$2ROH + 2Na \longrightarrow 2RONa + H_2$$ (Brisk Effervescence)

ii) Ester test :-

$$ROH + CH_3COOH \xrightarrow{\text{Conc. } H_2SO_4} RCOOCH_3 + H_2O$$

(glacial acetic acid) (Ester, fruity smell)

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Table-3

S.no	Experiment	Observation	Inference
	Aldehyde group (-CHO)		
i)	Schiff's Solution test = Take .5gm of solid compound or 4 drops of liquid Compound in a test tube, add 1ml of Schiff's reagent, shake vigorously.	Red or Violet colour	-CHO Group present
ii)	Tollen's Reagent test = Take a little quantity of the organic compⁿ in a test tube, add 2ml of Tollen's reagent, shake warm and allow the content to stand for 3-4 minutes	Silver mirror or grey ppt	-CHO Group present
iii)	Fehling's Solution test = Take 1ml of a mixture of equal amounts of Fehling solⁿ A and B, add 4 drops or 0.2 gm of organic compound, boil for about 5minutes	Red ppt	-CHO Group present

Reactions Involved :-

i) Tollen's Reagent test = $AgNO_3 + NaOH \rightarrow AgOH + NaNO_3$

$2AgOH \rightarrow Ag_2O\downarrow + H_2O$ (white ppt)

$Ag_2O + NH_4OH \rightarrow Ag(NH_3)_2OH$ (Tollens Reagent)

$RCHO + 2Ag(NH_3)_2OH + H_2O \rightarrow RCOONH_4 + 2NH_4OH + NH_3 + 2Ag$

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Table - 5

S.no	Experiment	Observation	Inference
	Ketonic group -		
i)	Schiff's Solⁿ test = Take .5 g of solid compⁿ or 4 drops of liquid compⁿ in a test tube add 1ml of Schiff Reagent, shake vigorously.	No Colour	Ketonic Group present.
ii)	2,4, Dinitrophenyl hydrazine test = Take a little quantity of the organic compⁿ in a test tube add 2ml of 2,4. dinitrophenyl hydrazine reagent, shake, heat & cool.	A red, yellow or orange coloured ppt	Ketonic Group present.

Table - 6

S.no	Experiment	Observation	Inference.
	Nitro Group (-NO₂)		
i)	NaOH solⁿ test - Take .5 gm of solid compⁿ or 4 drops of liquid compⁿ in a test tube add 1ml of aq. solⁿ of NaOH and warm.	Yellow Colour	Nitro Group present.
ii)	Mulliken's test - Take a little quantity of org. compⁿ in the dry test tube add 2ml of ethanol, 1ml of $CaCl_2$ or NH_4Cl solⁿ & a pinch of zn dust, boil rxn content for 5 min. Cool & filter into a test tube contain 2ml of toderis reagent.	Grey or black ppt	Nitro Group present.

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Table - 7

S.No	Experiment	Observation	Inference
	Primary amines ($-NH_2$)		
i)	Litmus test - Put a pinch of the organic comp^n on a moist red litmus paper.	Turns blue	Amines Group present.
ii)	Nitrous acid test = Dissolve a small quantity of organic comp^n in dil. HCl and Cool. Add 10% aq. $NaNO_2$ Solution.	Brisk effervescence	Aliphatic ~~amines~~ primary amines Group present
iii)	Diazotisation test - Dissolve ~~about 2~~ gm of the organic comp^n in dil HCl and Cool. Add 10% aq. $NaNO_2$ sol^n. ~~Poo~~ Pour the reaction content in a Beaker containing alkaline β-naphthol solution	Red or orange dye	Aromatic primary amines Groups present

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~~Experiment - to~~

~~Object~~ Result = We have detect the following functional groups in the given organic compⁿ

1. Carboxylic group (-COOH)
2. Alcoholic group (-OH)
3. Phenolic group
4. Aldehydic Group (-CHO)
5. Ketonic Group present
6. Nitro group ($-NO_2$)
7. Primary amines ($-NH_2$)

Precautions =

1. Acid burns = Wash with plenty of water then with very dilute Sodium bisulphite solution
2. Bases = Wash with plently of water then with very dilute acetic acid solⁿ.
   When you are heating a test tube, never point its mouth towards yourself or anyone else.
3. Protect your hands if you have to pick up hot objects.
4. Wash test tube before adding.

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Experiment -10

Object = To determine the strength of given hydrochloric acid solution by titrating it pH metrically against Sodium Hydroxide.

Chemicals / Apparatus Required = pH meter, glass electrode reference electrode, burette, stirrer, NaOH, HCl.

Principle = When an alkali is added to an acid solution the pH of the solution increases slowly. But about the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated by Normality equation.

Procedure = First standardize the PH meter against a buffer solⁿ of known PH. Now first wash the glass electrode and reference electrode with distilled water and then rinse with the acid solution. Take 50ml of HCl solution in a 500 ml of beaker, Add sufficient water so that the glass electrode as well as reference electrode is completely dipped. Note the pH of pure acid solⁿ. Now add 1ml of .1 N NaOH from the burette in the beaker. Stir the

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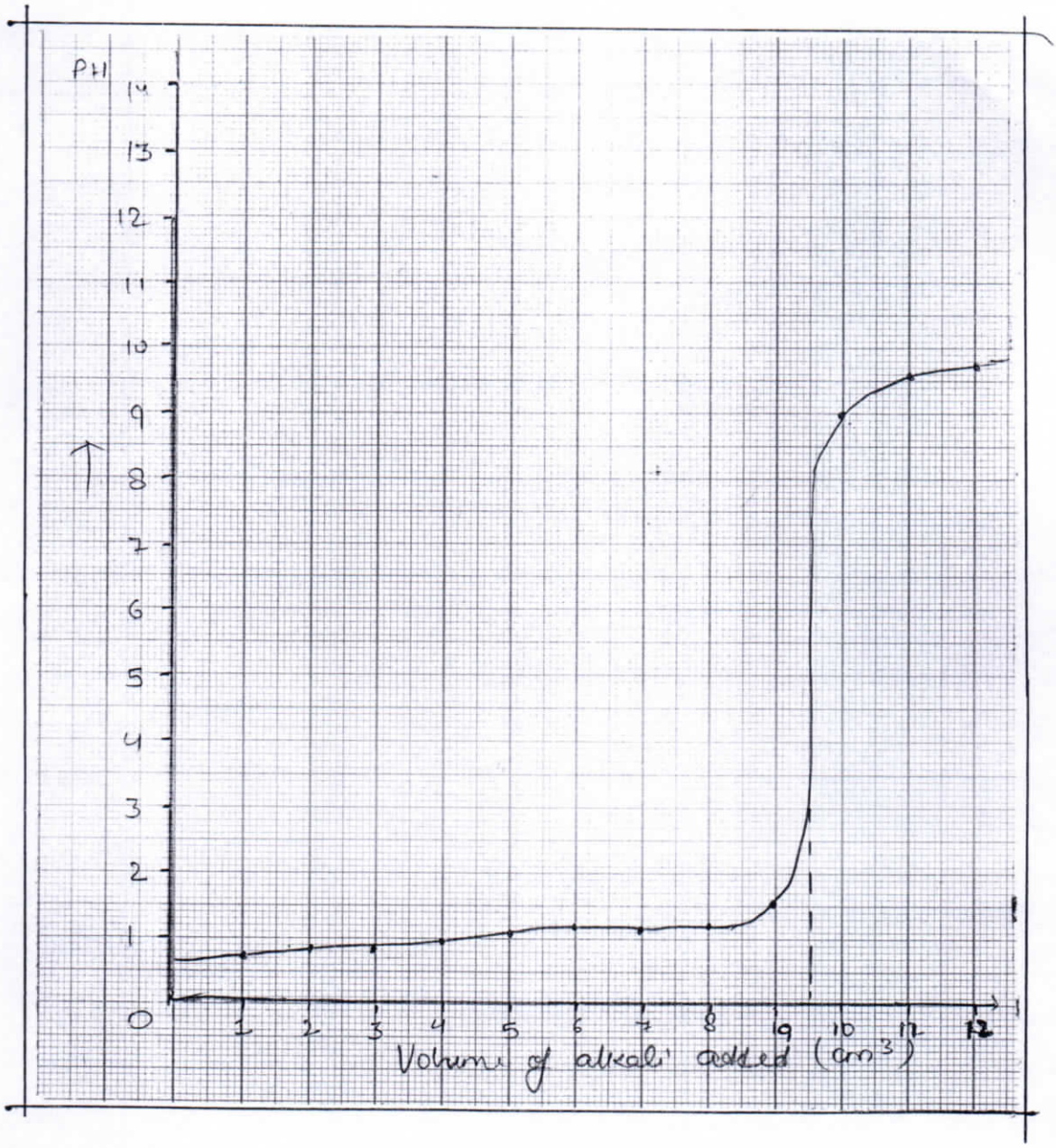
pH
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
1
2
3
4
5
6
7
8
9
10
11
12
Volume of alkali added (cm3)

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the contents well and note the pH of the solution. Continue adding NaOH soln from the Burette and note down the pH after each addition. Near the equivalence point the change in pH occurs rapidly then add alkali in fraction.

Observations = Volume of acid taken = 50ml (say)

Volume of NaOH added (ml)	0	1	2	3	4	5	6	7	8	9	10	11	12	13
pH	0.60	0.62	0.64	0.68	0.72	0.78	0.86	0.98	1.14	1.60	9.14	9.80	9.92	

Calculations = A curve is plotted with pH values as ordinate and volume of NaOH added ml as abscissa. The sharp break in the curve corresponds to the equivalence points. Suppose vol. of 0.1 NaOH used at end point = V_2 ml

(Acid) (alkali)

$$N_1 V_1 = N_2 V_2$$

$$N_1 \times 50 = \frac{1}{10} \times V_2$$

$$N_1 = \frac{1 \times V_2}{10 \times 50} = \frac{6.5}{500} = .013$$

Strength = N × Eq. wt.

= 0.013 × 36.5 gm/L = 0.4745 gm/lit.

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Result = The strength of given HCl Solⁿ is 47.45 gm/lit

Precautions = The temperature of central knob of the pH meter should be adjusted to the room temperature.

- After the addition of alkali, the Solⁿ should be thoroughly stirred
- The pH meter should be first standardized by taking a buffer of known PH.

Viva Voce

Q1- Define pH?

Ans- PH is the scale to specify how acidic or basic a water - solⁿ is.

Q2- Do you use any indicator in pH titration- No.

Q3- What is the desirable pH range for drinking water?

Ans- 6-7.5 desirable range.

Q4- What is effect of temperature on pH?

Ans- PH decreases with increase in temperature

Q5- Who suggested the pH value

Ans- Danish chemist Soren peder Lauritz Sorenson

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