MAHARASHTRA AGRICULTUAL UNIVERSITIES

AGRICUTURAL POLYTECHNIC

(LOWER AGRICULTURAL EDUCATION)

LABORATORY MANUL

First year (Agri. Diploma)

SUBJECT – General Science - I
(Chemistry)

Prepared By

Dr.S.S.Kolape
Principal, ATS, Puntamba.
Shri. B. M. Belhekar
Agril. Asstt. ATS, Puntamba.

MAHARASHTRA AGRICULTUAL UNIVERSITIES
AGRICULTURE POLYTECHNIC
(LOWER AGRICULTURAL EDUCATION)
Practical

Course Title: General Science– I (Chemistry) First Year

Index

EXPERIMENT No. 1
ORIENTATION OF BASIC LABORATORY TECHNIQUES

A) Cutting glass tube and glass rod

Aim: To learn the technique of cutting glass tube and glass rod.

Requirements: Glass tube, glass rod, glass cutting file.

Procedure:

Hold the glass tube or the glass rod to be cut with your hand firmly keeping the thumb close to cutting point and take the support of the table. Create a scratch across the tubing or rod surface by means of cutting file. Only make one single cut with a smooth movement of the hand. Do not 'saw' the glass with the file it otherwise would give multiple scratch.

Now hold firmly the tube/rod with your both hands keeping the thumbs behind the scratch and on either side of the scratch mark and break by jerk bending facing away from you. For safety place a piece of towel to cover your hand. If the cut part bears sharp edges rub it gently on some hard flat surface like stone or cement concrete of simply heat edges.

B) Bending a glass tube

Aim: To learn the technique of bending glass tube.

Requirements: Glass tube and burner, wire gauze.

Procedure:

Take the glass tube to be bent, hold with your both hands apart leaving portion of the tube to be bent. Heat this portion using high blue flame of the burner. When this part softens apply gentle bending pressure and stop heating when desired bend angle is reached. Place on wire gauze for cooling.

C) Drawing out a glass jet

Aim: To learn the technique of drawing out a glass jet.

Requirements: Glass tube, glass cutting file, burner.

Procedure:

Take the desired glass tube, hold with your both apart leaving portion of the tube to be jetted ie narrowed. Heat this portion using high blue flame of the burner. When this part softens, take away from the flame and stretch gently up to desired length. Cut the narrowed part from the middle with the file and place on suitable place for cooling.

D) Study of burner

Aim: Study of burner.

Observe your laboratory burner carefully. It has a heavy metallic base with a nozzle for fuel gas to come out. The base bears a side tube that is connected with rubber tube which supplies the fuel gas. The base is connected with detachable hollow metallic tube, barrel, with a hole at its base to permit air to enter in. This hole can be monitored by a rotating collor to control the intake of air. When this hole is closed yellow reducing flame is obtained which produces black shoot. Therefore it is partly opened and adjusted to get oxidizing blue flame free from black it is flame is used for heating purposes.

The burner needs periodic cleaning of the nozzle with a fine metal wire. Also the rubber tube needs periodic inspection and must be replaced by a new one if required.

EXPERIMENT No. 2
PREPARATION OF LYOPHILIC SOLUTION, STARCH OR GUM.

Theory:
There are two types of solution:
i) Lyophilic Solution: When there is a great affinity between dispersed phase and dispersion medium. eg starch sol, gum sol.

ii) Lyophobic solution: When there is no or little affinity between dispersed phase and dispersion medium. e.g. $Al(OH)_3$ solution, $Fe(OH)_3$ solution, As_2O_3 solution.

Aim: To prepare lyophilic solution of starch (or gum).

Requirements: Starch or gum, distilled water, small beakers (100 ml capacity), funnel' glass rod, filter paper etc.

Procedure: [Procedure for starch solution and Gum solution is same]
1) Take about 60 ml of distilled water in a clean beaker and start heating on wire
gauze.

2) In another clean small beaker take about 200 mg (0.2g) starch. Add about 1 to
2 ml distilled water and make its paste with the help o glass rod.

3) To this paste of starch add the hot distilled water in small portions at a time and stir well with each addition. After adding about 60 ml of hot water, heat the content further to let it just boils. Then leave it for cooling.

4) After cooling, filter through filter paper to remove insoluble starch, if any. The filtrate so obtained is the solution of starch.

EXPERIMENT No. 3
STUDY OF ROLE OF EMULSIFYING AGENTS IN STABILISING THE EMULSION OF OIL.

Theory: Emulsions are lyophobic colloid in nature. Water and oil on vigorous shaking form emulsion but is unstable. Hence on standing get separated into two layers. It is called as demulsification.

However it can stabilize by the addition of stabilizing agent which forms a very thin protecting layer between water and oil, and reduce the interfacial surface tensions. Emulsifying agents differ in their ability to stabilize the emulsions.

Aim: To study the role of emulsifying agents in stabilising the emulsion of an oil.

Requirements: Synthetic detergent, Gum Arabic, Soap, Cotton seed oil, measuring pipette or cylinders, stop watch.

Procedure:
Part-I: Preparation of solutions of 'stabilising agents'.

1) Take 0.5 g of gum Arabic and dissolve in 50 ml of water, if required heat to dissolve.

2) Similarly weight 0.5 g synthetic detergent and dissolve in 50 ml of water.

3) Likewise weight 0.5g powdered soap and dissolve in 50ml of water, if required heat to dissolve.

Part-II: Main Experiment:

1) Take three test tubes and label them as T-1, T-2 and T-3. Add 5ml of oil with measuring pipette (or cylinder) in each of these tubes.

2) Add 5ml of distilled water to each of this containing oil. Two layers are seen.

3) Shake well, any one out of T-1, T-2, T-3, after closing its opening with thumb. You will notice that water and oil mix together but on standing they get
separated in short time and two layers are again seen, indicating the instability of emulsion.

4) Now add 5ml gum solution to T-1 and again shake well in the same way. Water and oil again mix together to form the emulsion. Keep T-1 on test tube stand, start the stop watch immediately and record the time for the separation of two layers. It is called demulsification time. Record it.

5) Repeat the same procedure for T-2 by adding 5ml synthetic detergent solution and record the demulsification time.

6) Similarly repeat the same procedure for T-3 by adding 5ml soap solution to T-3 and record the demulsification time.

Conclusion: Greater the demulsification time the better is the emulsifying power of the emulsifying agent.

Observation Table:

Test Tube	**Emulsifier used**	**Demulsification Time, sec.**
T-1	Gum Arabic	
T-2	Synthetic detergent	
T-3	Soap	

EXPERIMENT No. 4
DETERMINATION OF PH OF ACID, BASE, SALT AND FRUIT JUICES

Theory: pH is defined as the negative logarithm to the base 10 of the molar concentration of H^+ ions in the solutions.

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

Similarly pOH is defined as the negative logarithm to the base of molar concentration of OH ions the solution.

$$pOH = -\log_{10}[OH^-]$$

Note that pH + pOH = 14

Acidic, basic and neutral solutions: Measurement of pH indicates the acidic, basic or neutral nature of the solution.

Acidic solution	Neutral solution	Basic solution
For acidic solution $[H^+] > [OH]$. $[H^+] > 10^{-7}$ mol dm^{-3} And pH < 7	For pure water or neutral solution pH = pOH = 7	For basic solution $[H^+] < [OH]$. $[H^+] < 10^{-7}$ mol dm^{-3} and pH > 7

Aim: Determination of pH of solutions of i) Acids ii) Bases iii) Salts and iv) Fruit juices – using pH paper or universal indicator.

Requirements: Universal indicator, pH paper, distilled water, test tubes, test tube stand, glass rod etc.

Procedure;
Theoretical Aspects:
1) Wash all the apparatus and rinse with distilled water every time.

2) Some fruits are juicy like sweet lime, water melon, orange etc. Their juice can be directly taken and tested.

3) But some fruits are not juicy like apple, papaya, chiku etc. For such fruits, take their small pieces in a test tube, add few ml distilled water, crush with glass rod, filter and use the filtrate for testing their pH

4) Testing with pH paper: pH papers in the form of strips are available. Make small pieces of a strip and place them on filter paper away from each other. Dip the tip of glass rod in the solution to be

tested and touch with the pH paper to make it wet. The pH paper imparts certain colour. Match the colour with colour chart provided and note the corresponding pH.

5) Testing with Universal indicater: Take about 2 ml of solution to be tested in a test tube. Add on drop of universal indicator, shake and match the colour developed with the colour chart provided and note the corresponding pH.

Practical Aspects:
A) Learning with known solutions:

1. Acid solutions:
i) Take given 0.01N HC1 solution and find its pH as described above either by universal indicator or pH paper. We know that its pH is 2.

ii) Now take 1 ml of 0.01 N HCl on a 100 ml cylinder and dilute to 100 ml with distilled water. It becomes 10^{-4} N HCl. Find its pH. We know that its pH is 4

2. Base solutions
i) Take given 0.01 N NaOH solutions and find its pH. We know that its pH is 12

ii) Now take 1 ml of 0.01 N NaOH in a 100 ml cylinder and dilute to 100 ml with distilled water. It becomes 10^{-4} N NaOH. Find its pH. We know that its pH is 10.

3. Salt solutions:
i) Take few crystals of NaCl salt in a test tube, dissolve in few ml of distilled water and test the pH. We know that its pH is 7.

ii) Take a few crystals of ammonium chloride, NH_4Cl salt, dissolve in few ml of distilled water and test the pH. We know, on the basis of nature of salt, its pH is less than 7 ie acidic in nature.

iii) Take a few crystals of sodium acetate, CH_3COONa salt, dissolve in few ml of distilled water and test the pH. We know, on the basis of nature of salt, its pH is more than 7 ie basic in nature.

B) Use of skill learnt:

4) Testing of fruit juices: Test different fruit juices and report their pH. If juices are dark coloured, add 2 drops of universal indicator instead of one drop, to subside the interference. Test at least four seasonal fruits available like sweet lime, orange, water melon, melon,

apple, chiku, mango, grapes, papaya, banna, custard apple, pine apple etc.

Observation Table:

No.	Material	pH	Nature
1	0.01 N HCl	2	Acidic
2	0.0001 N HCl	4	Acidic
3	0.01 N NaOH	12	Basic
4	0.0001 N NaOH	10	Basic
5	NaCl	7	Neutral
6	NH_4Cl		
7	CH_3COONa		
8	Fruit (name)		
9	Fruit (name)		
10	Fruit (name)		
11	Fruit (name)		

EXPERIMENT No. 5
COMPARISON OF pH OF STRONG ACID AND WEAK ACID HAVING SAME CONCENTRATION.

Theory:

The degree of ionization of strong acid is more than that of weak acid. Therefore even if their concentrations are same their pH would not be same. Strong acid will have more $[H^+]$ ions ie less pH as compared to weak acid having same concentration.

Aim: To compare the pH of solutions of strong acid with that of weak acid having the same concentration.

Requirements: 0.01 N HCl, 0.01 N CH_3COOH, 0.0001 N HCl, 0.0001 N CH_3COOH, universal indicator, test tube, test tube stand, glass rod, 100 ml measuring cylinder.

Procedure:

1) Rinse test tube, glass rod and measuring cylinder with the distilled water every time.

2) Take 2 ml of 0.01 N HCl in one test tube and 2 ml of 0.01 N CH_3COOH in another test tube.

3) Add 2 drops of universal indicator in each of these solutions and shake. The solutions impart certain colour.

4) Match the colour of these solutions with that given on colour chart and record the pH.

5) Similarly take 2 ml 0.0001 N HCl solution in one test tube and 2 ml 0.0001 N CH_3COOH in another test tube. Add 2 drops of universal indicator in each of these solutions, shake and record the pH after matching with the colour chart.

6) Now take 1 ml 0.0001 N HCl with measuring pipette in a measuring cylinder (10 ml capacity) and dilute to 10 ml with distilled water. This gives 10^{-5} N HCl solution.

7) Take 2 ml of this 10^{-5} N HCl solution in a test tube and discard the remaining 8 ml of it. Add 2 drops of universal indicator, shake, and record the pH after matching with the colour chart.

8) In the same way 1 ml 0.0001 ml N CH_3COOH with measuring pipette in a measuring cylinder (10 ml capacity) and dilute to 10 ml with distilled water. This gives 10^{-5} N CH_3COOH solution.

9) Take 2 ml of this 10^{-5} N CH_3COOH solution in a test tube and discard the remaining 8 ml of it. Add 2 drops of universal indicator, shake, and record the pH after matching with the colour chart.

Observation Table:

No.	Acids	pH
1	Strong acid 0.01 N HCl Weak acid 0.01 N CH_3COOH	
2	Strong acid 0.0001 N HCl Weak acid 0.0001 N CH_3COOH	
3	Strong acid 10^{-5} N HCl Weak acid 10^{-5} N CH_3COOH	

Conclusion: pH of strong acid HCl is less than weak acid CH_3COOH having same concentration. This is because the degree of ionization of strong acid (eg HCl) is mote than that of weak acid (eg CH_3COOH).

EXPERIMENT No. 6
THE pH CHANGE DUE TO COMMON ION EFFECT RELATED TO WEAK ACIDS AND WEAK BASES

Theory:

The common ion effect is the shifting of equilibrium of weak acid (or weak base) by the addition of strong electrolyte having one ion common with the weak acid (or weak base).

The equilibrium of weak acid gets shifted to left when some electrolyte having common ion is added. This results in decrease in $[H^+]$ ion concentration and increase in pH. eg

$$CH_3COOH \leftrightarrows CH_3COO^- + H^+$$
$$CH_3COONa \leftrightarrows CH_3COO^- + Na^+$$

Similarly the equilibrium of weak base gets shifted to left when some electrolyte having common ion is added. This results in decrease in [OH] ion concentration and increase in pOH or decrease in pH. eg

$$NH_4OH \leftrightarrows NH_4^+ + OH^-$$
$$NH_4Cl \leftrightarrows NH_4^+ + Cl^-$$

Aim: To study the changes in pH of weak acid under the influence of common ion effect.

Requirement: 0.2 N Acetic acid, 0.2 N sodium acetate, 2 N sodium, acetate, 0.2 N NH_4OH, 0.2 N NH_4Cl, 2N NH_4Cl, universal indicator, measuring pipette, test tube, test tube stand etc.

Procedure: Part-I: Effect of common ion on weak acid, CH_3COOH

1) Take three test tubes, rinse with distilled water and mark them as T-1, T-2 and T-3.

2) In T-1 take 1 ml of 0.2 N CH_3COOH + 1 ml H_2O. Add 1 drop of universal indicator, record the pH after matching with colour chart.

3) In T-2 take 1 ml 0.2 N CH_3COOH + 1 ml 0.2 N CH_3COONa. Add 1 drop of universal indictor, record the pH after matching with colour chart.

4) In a test tube take 1 ml 0.2 N CH_3COOH + 10 ml 2 N CH_3COONa. Out of these 11 ml solution, take 2 ml in T-3. Add 1 drop of universal indicator, record the pH after matching with colour chart.

Part-II: Effect of common ion on weak base, NH_4OH.

1) Take three test tubes, rinse with distilled water and mark them as T-1, T-2 and T-3.

2) In T-1 take 1 ml of 0.2 N NH_4OH + 1 ml H_2O. Add 1 drop of universal indicator, record the pH after matching with colour chart.

3) In T-2 take 1 ml of 0.2 N NH_4OH + 1 ml 0.2 N NH_4Cl. Add 1 drop of universal indicator, record the pH after matching with colour chart.

4) In a test tube take 1 ml 0.2 N NH_4OH + 10 ml N NH_4Cl. Out of these 11 ml solution, take 2 ml in T-3. Add 1 drop of universal indicator, record the pH after matching with colour chart,

Observation Table:
Part-I: Effect of common ion on weak acid, CH_3COONa.

No.	**Test tube**	**Colour**	**pH**	**Conc. Of CH_3COONa**	**Remark**
1	T-1			Zero	No common ion effect
2	T-2			Equal to CH_3COOH	pH increased, common ion effect observed.
3	T-3			100 times more than CH_3COOH	pH increased, common ion effect further observed.

Observation Table:
Part-I: Effect of common ion on weak base, NH_4OH.

No.	**Test tube**	**Colour**	**pH**	**Conc. Of NH_4OH**	**Remark**
1	T-1			Zero	No common ion effect
2	T-2			Equal to NH_4OH	pH decreased, common ion effect observed.
3	T-3			100 times more than NH_4OH	pH decreased, common ion effect further observed.

EXPERIMENT No. 7
PREPARATION OF STANTARD SOLUTION OF OXALIC ACID

Theory: In volumetric analysis ie titration, to know the strength (concentration) of unknown solution we require a solution of known concentration. The solution of known concentration is called as **'standard solution'**.

Aim: To prepare a standard solution of oxalic acid. (Say 100 ml 0.1 N)

Requirement: Crystalline oxalic acid $H_2C_2O_4.2H_2O$, distilled water, 100 ml volumetric flask, watch glass, glass rod, 250 ml beaker etc.

Procedure:

1) Weight empty clean dry watch glass.

2) Place the crystals of oxalic acid on the watch glass and weigh again so as to get 0.63 g substance.

3) Transfer the weighed oxalic acid to a beaker.

4) Pour distilled water over the watch glass to transfer all the crystals into beaker.

5) Dissolve the substance completely in distilled water, taking care that the volume should not exceed 100 ml.

6) Transfer the solution from beaker to 100 ml volumetric flask and dilute very carefully to 100 ml mark. Stopper the flask and shake; it gives standard solution of oxalic acid having concentration 0.1 N and volume 100 ml.

Calculation:

1) Molecular weight of crystalline oxalic acid $H_2C_2O_4.2\ H_2O$ = 63 g.

2) Thus 63 g oxalic acid dissolved to final volume 1 dm^3 would be 1 N solution.

3) $\therefore$ 6.3 g oxalic acid dissolved to final volume 1 dm^3 would be 0.1 N solution.

4) 63 g oxalic acid dissolved to final volume 100 ml would be 0.1 N solution.

Observation:

1) Weight of empty watch glass = W_1=……g

2) Weight of watch glass + oxalic acid = W_2=……g

3) Weight of oxalic acid = W_2 - W_1=……g

EXPERIMENT No. 8
VOLUMETRIC ESTIMATION OF SODIUM HYDROXOIDE BY STANDARD SOLUTION OF OXALIC ACID

Aim: To determine the strength in normal terms and gram per dm^3 of sodium hydroxide by using standard solution of oxalic acid.

Given: 0.1 N $H_2C_2O_4$, Oxalic acid.

Requirement:
Burette, 10 ml pipette, conical flask, NaOH, $H_2C_2O_4.2\ H_2O$, phenolphthalein.

Procedure:
1) Wash burette, pipette and conical flask with water.

2) Rinse the burette with NaOH solution and fill with it up to zero level after removing air bubbles if present.

3) Rinse the pipette with oxalic acid. Pipette out 10 ml of the oxalic acid in the conical flask.

4) Add two drops of phenolphthalein indicator into conical flask, Titrate slowly against NaOH solution with constant shaking till pink colour is just obtained. Record the reading. Take three readings. All the readings must be same or nearly same.

Equation:

$$H_2C_2O_4 + 2NaOH \rightarrow Na_2C_2O_4 + 2\ H_2O$$

Observation: Given 0.1N $H_2C_2O_4$

No.	**Burette NaOH ml**	**Pipette $H_2C_2O_4$ ml**	**Mean burette reading**	**Indicator**	**End Point**
1	--	10	----- = V_1	Phenolphthalein	Colourless to pink
2	--	10			
3	---	10			

Calculation:

(i) NaOH : $H_2C_2O_4$

$N_1V_1 = N_2V_2$

$$N_1 = \frac{0.1 \times 10}{V_1}$$

$\therefore$ NaOH = ----------N

(ii) Strength of NaOH solution = N x Eq. wt. of NaOH

= N x 40

= ----- g dm^{-3}

Result:

1) Normally of NaOH Soln. = ---------- N

2) Strength of NaOH soln. = ---------- g dm^{-3}

EXPERIMENT No. 9
QUALITATIVE ANALYSIS OF INORGANIC COMPOUND

Qualitative analysis of inorganic compound involves detection of one **'Basic'** and **'Acidic'** radical. It is carried out in three steps.

A) Preliminary Examination:
B) Detection of Basic Radical (Cation):
C) Detection of Acidic Radical (anion):

A) PRELIMINARY EXAMINATION:

Test	Observation	Inference
1) Colour:	a) Colourless	Ca^{2+}, Pb^{2+}, NH_4^+, Al^{3+}, Zn^{2+}
	b) Blue	Cu^2
	c) Green	Ni^{2+}
	d) Pink	Co^{2+}
2) Nature:	a) Crystalline	Soluble salts
	b) Hygroscopic (moist)	$CaCl_2$, $Ca(NO_3)_2$, $Cu(NO_3)_2$
3) Dry Test: Heat a little substance in a clean dry test tube.	a) Decrepitation (crackling noise)	$Pb(NO_3)_2$
	b) Fusion	$Ca(NO_3)_2$, $Pb(NO_3)_2$
	c) Condensation of water on cooler part of test tube	Salts containing water of crystallization
	d) White sublimation	NH_4^+
	e) Brown fumes	NO_3^-, Br^-
	f) Violet fumes	I^-
	g) Colour of the residue left	
	Yellow	Pb^{2+}
	White	Ca^{2+}
	Coloured mass	Cu^{2+}, Ni^{-2+},Co^{2+}
4) NaOH Test: Sus. + dil. NaOH, boil	Smell of NH_3 NH_3 gas turns moist turmeric paper brown (do not touch the paper with mouth of test tube)	NH_4^+
5) Dry Test for Acidic Redical:		

i) Dil. H_2SO_4 Test. Sub. + dil. H_2So_4	a) Effervescence b) Light brown gas	Ca_3^{2-} NO_2^-
ii) Conc. H_2SO_4 Test. (Take cautiously) Sub.+ Conc. H_2So_4	a) Brown fumes b) Violet fumes c) Yellowish green gas	NO_3^-, Br^- I^- Cl^-
iii) Phosphate test: Sub.+ Conc. HNO_3^+ Ammonium molybadate	Yellow ppt, soluble in NaOH	PO_4^{3-}

B) DETECTION OF BASIC RADICAL:

PREPARATION OF SOLUTION:
Take ¼ spoon substance + 2 test tube distilled water, stir, heat if necessary. Filter only if solutions is turbid ie not clear. Use it as original solution, **(O.S.)**

DETECTION GROUP: (Gr. I, II, IIIA, III B, IV, V, VI)

Test	Observation	Inference
Test for Gr. I O.S. + dil. HCl	a) ppt obtained b) No ppt	a) Gr. I Present b) Gr. I Absent
Test for Gr. II O.S. + dil. HCl, warm + H_2S	a) ppt obtained b) No ppt	a) Gr. II Present b) Gr. II Absent
Test for Gr. III A O.S. + NH_4Cl + NH_4OH	a) ppt obtained b) No ppt	a) Gr. III A Present b) Gr. III A Absent
Test for Gr. III B O.S. + NH_4Cl + NH_4OH + H_2S	a) ppt obtained b) No ppt	a) Gr. III B Present b) Gr. III B Absent
Test for Gr. IV O.S. + NH_4Cl + NH_4OH + $(NH_4)CO_3$	a) ppt obtained b) No ppt	a) Gr. IV Present b) Gr. IV Absent
Test for Gr. V O.S. + NH_4Cl + NH_4OH + Na_2HPO_4	a) ppt obtained b) No ppt	a) Gr. V Present b) Gr. V Absent
Test for Gr. VI No particular test	a) If no ppt to Gr.	a) Gr.VI must be

	vi	Present

Note: After detecting the Group find the **'Basic Radical'** in the group detected only. Once basic radical is detected, confirm it by taking Confirmatory Test (C.T.)

GROUP – I

If **Gr.I** is present, then the basic radical is Pb^{2+}

Confirmatory Test (C.T.):

Test	Observation	Inference
Pb^{2+}		
1) O.S. + Kl	Yellow ppt	Pb^{2+} Confirmed
2) O.S. + dil. H_2SO_4	White ppt	Pb^{2+} Confirmed
3) O.S. + dil. K_2CrO_4	Yellow ppt	Pb^{2+} Confirmed

GROUP – II

Radicals of Gr.II: Pb^{2+}, Cu^{2+} any one radical is present.

Test	Observation	Inference
O.S. + dil. H_2SO_4	a) White ppt b) No ppt, and O.S. blue	Pb^{2+} Present Cu^{2+} Present

Confirmatory Test (C.T.):

Test	Observation	Inference
Pb^{2+}		
1) O.S. + Kl	Yellow ppt	Pb^{2+} Confirmed
2) O.S. + dil. H_2SO_4	White ppt	Pb^{2+} Confirmed
3) O.S. + dil. K_2CrO_4	Yellow ppt	Pb^{2+} Confirmed
Cu^{2+}		
1) O.S. + Kl	Brown colour (with white ppt)	Cu^{2+} Confirmed
2)) O.S. + dil. $K_4[Fe(CN)_6]$	Reddish brown ppt	Cu^{2+} Confirmed

GROUP – III A

Radicals of Gr. III A: Al^{3+}, Fe^{3+} any one radical is present.

Test	Observation	Inference
O.S. + NH_4Cl + NH_4OH	a) Reddish brown ppt b) White gelatinous ppt	Fe^{3+} Present Al^{3+} Present

Confirmatory Test (C.T.):

Test	Observation	Inference
Fe^{3+} 1) O.S. + NH_4CNS 2) O.S. + $K_4[Fe(CN)_6]$	 Blood red colour Blue colour	 Fe^{3+} Confirmed Fe^{3+} Confirmed
Al^{3+} 1) O.S. + NaOH 2)) O.S. + Na_2HPO_4	 White gelatinous ppt White ppt	 Al^{3+} Confirmed Al^{3+} Confirmed

GROUP – III B

Radicals of Gr. III B: Ni^{2+}, Co^{2+}, Zn^{2+}, Mn^{2+} any one radical is present.

Test	Observation	Inference
O.S. + NH_4Cl + NH_4OH + H_2S	a) White ppt b) Buff ppt c) Black ppt	Zn^{2+} Present Mn^{2+} Present Na^{2+} or Co^{2+} Present
O.S. Colour	a) Green b) Pink	Ni^{2+} Present Co^{2+} Present

Confirmatory Test (C.T.):

Test	Observation	Inference
Zn^{2+} 1) O.S. + $K_4[Fe(CN)_6]$ 2) O.S. + NH_4OH	 White ppt White ppt soluble in excess of NaOH	 Zn^{2+} Confirmed Zn^{2+} Confirmed
Ni^{2+} 1) O.S. + Dimethyl glyoxime + NH_4OH 2) O.S. + Na_2HPO_4	 Red ppt Pale green ppt, soluble in excess of NH_4OH	 Ni^{2+} Confirmed Ni^{2+} Confirmed
Co^{2+} 1) O.S. + NH_4CNS	 Deep blue colour	 Co^{2+} Confirmed

2) O.S. + NH_4OH	Blue ppt	Co^{2+} Confirmed
Mn^{2+} 1) O.S. + NaOH + Br_2 water 2) O.S. + PbO_2 + Conc.HNO_3, + boil + H_2O	Black or brown ppt Pink colouration	Mn^{2+} Confirmed Mn^{2+} Confirmed

GROUP – IV

Radicals of Gr. IV: Ca^{2+}, Sr^{2+}, Ba^{2+} any one radical is present.

Test	Observation	Inference
Ca^{2+} O.S. + $CaSo_4$	a) White ppt immediately obtained b) White ppt on warming and waiting c) No ppt even on warming	Ba^{2+} Present Sr^{2+} Present Ca^{2+} Present

Confirmatory Test (C.T.):

Test	Observation	Inference
Ca^{2+} 1) O.S. + $(NH_4)_2C_2O_4$ ammonium oxalate 2) O.S. + NaOH	White ppt White ppt soluble in excess of NaOH	Ca^{2+} Confirmed Ca^{2+} Confirmed
Ni^{2+} 1) O.S. + Dimethyl glyoxime + NH_4OH 2) O.S. + NH_4OH	Red ppt Pale green ppt, soluble in excess of NH_4OH	Ni^{2+} Confirmed Ni^{2+} Confirmed
Co^{2+} 1) O.S. + NH_4CNS 2) O.S. + NH_4OH	Deep blue colour Blue ppt	Co^{2+} Confirmed Co^{2+} Confirmed
Mn^{2+} 1) O.S. + NH_4OH +	Black or brown ppt	Mn^{2+} Confirmed

Br_2 water 2) O.S. + Pbo_2 + HNO_3, + boil + H_2O	Pink colouration	Mn^{2+} Confirmed

GROUP – V

If Gr. V is present, then the basic radical is Mg^{2+}.
Confirmatory Test (C.T.):

Test	Observation	Inference
Mg^{2+} 1) O.S. + NaOH	a) White ppt	Mg^{2+} Confirmed
2) O.S. + **Hypoiodide soln** *. *** Hypoiodide soln.:** ½ ml NaOH + ½ KI + I_2 till solution becomes yellow	b) Reddish Brown ppt	Mg^{2+} Confirmed

GROUP – VI

If Gr.VI is present, then the basic radical is NH_4^+
Confirmatory Test (C.T.):

Test	Observation	Inference
NH_4^+ 1) O.S. + NaOH, boil	Smell of NH_3, turns moist turmeric paper brown.	NH_4^+ Confirmed
2) O.S. + Nessler's reagent * [***Nessler's reagent:** 3 drops $HgCl_2$ + Kl with shaking till scarlet red ppt dissolves in Kl + equal volume of NaOH]	Brown ppt	NH_4^+ Confirmed

C) DETECTION OF ACIDIC RADICAL:

Hint: Take test in serial order. Once 'Acidic Radical' is detected do not take further test. Directly take the **C.T.** of acidic radical detected.

Test	Observation	Inference
1) O.S. + dil. HCl	Effervescence	CO_3^{2-} present
2) O.S. + $FeSO_4$ + Conc.H_2SO_4 shake	Brown colour	NO_3^- present
3) O.S. + $HgCl_2$	Scarlet red ppt	I^- present
4) O.S. + Conc.H_2SO_4 + Starch soln. (freshly prepared)	Yellowish brown colour	Br^- present
5) O.S. + Conc.HNO_3 + Ammonium molybdate	Yellow ppt	PO_4^{3-} present
6) O.S. + $AgNO_3$	White ppt, soluble in NH_4OH	Cl^- present
7) O.S. + $BaCl_2$ or $Ba(NO_3)_2$	White ppt, insoluble in dil. HCl	SO_4^{2-}present
8) O.S. + Kl + Starch soln. (freshly prepared)	Dark blue colour	NO_2^- present
9) O.S. + 1 drop v. dilute $KMnO_4$ warm. (1 drop $KMnO_4$ + large H_2O, Use it as v.dilute $KMnO_4$)	Pink colour disappears	$C_2O_4^{2-}$ present
10) O.S. + $FeCl_3$	Reddish colour	CH_3COO^- present

Confirmatory Test (C.T.):

Test	Observation	Inference
1) Cl^-		
i) O.S. + $AgNO_3$	White ppt, soluble in NH_4OH	Cl^- Confirmed
ii) O.S. + lead acetate $(CH_3COO)_2Pb$	White ppt	Cl^- Confirmed
2) Br^-		
i) O.S. + $AgNO_3$	Pale yellowish ppt	Br^- Confirmed
ii) O.S. + MnO_2 + conc.H_2SO_4, heat	Brown fumes	Br^- Confirmed
3) I^-		
i) O.S. + $CuSO_4$	Brown colour with white ppt	I^- Confirmed
ii) O.S. + $HgCl_2$	Scarlet red ppt	I^- Confirmed

4) CO_3^{2-} i) O.S. + dil. H_2SO_4 ii) O.S. + dil. HNO_3	 Effervescence Effervescence	 CO_3^{2-} Confirmed CO_3^{2-} Confirmed
5) SO_4^{2-} i) O.S. + $(CH_3COO)_2Pb$ lead acetate ii) O.S. + $BaCl_2$ or $Ba(NO_3)_2$	 White ppt White ppt, insoluble in HCl	 SO_4^{2-} Confirmed SO_4^{2-} Confirmed
6) NO_3^- i) Ring Test: O.S. + $FeSO_4$ + conc. H_2SO_4 slowly from sides without shaking the tube ii) O.S. + Cu filling conc. H_2SO_4, heat	 Brown ting at the bottom Brown fumes	 NO_3^- Confirmed NO_3^- Confirmed
7) NO_2^- i) O.S. 1 drop on filter paper + 1 drop KI + starch ii) O.S. + $FeSO_4$ + dil. H_2SO_4, shake	 Dark blue spot on filter paper Brown colour	 NO_2^- Confirmed NO_2^- Confirmed
8) PO_4^{3-} i) O.S. + $AgNO_3$ ii) O.S. + NaOH + $BaCl_2$	 White ppt White ppt	 PO_4^{3-} Confirmed PO_4^{3-} Confirmed
9) $C_2O_4^{2-}$ i) O.S. + $CaCl_2$ ii) O.S. + $BaCl_2$ or $Ba(NH_3)_2$	 White ppt White ppt	 $C_2O_4^{2-}$Confirmed $C_2O_4^{2-}$ Confirmed
10) $CH_3\ COO^-$ i) O.S. + $FeCl_3$ ii) O.S. + conc. H_2SO_4, heat strongly till almost dryness	 Reddish colour or ppt (To this + dil. HCl → red colour disappears) Black clour	 $CH_3\ COO^-$ Confirmed $CH_3\ COO^-$ Confirmed

Result: The given compound contains:

1) Basic radical…… 2) Acidic radical ……

EXPERIMENT No. 10
QUALITATIVE ANALYSIS FOR ORGANIC COMPOUND

DETECTION OF NITROGEN, SULPHUR, CHLORINE, BROMINE AND IODINE IN AN ORGANIC COMPOUND

Caution: ☺ Sodium metal is very active, hence preserved under Kerosene. Never bring it in contact with water.

Part – I: Preparation of Sodium Extract, (S.E.)

1) Take about two test tube of distilled water in porcelain dish.

2) Take out sodium metal from kerosene, place on filter paper. Cut the sodium metal with knife into few small pieces. Take 2 or 3 fusion tube. In each tube take 1 – 2 pieces of sodium metal.

3) Heat gently the fusion tube to melt the sodium metal. Then add the organic compound and heat again very strongly to red hot, plunge into water taken in the porcelain dish and cover immediately the dish with asbestos sheet to avoid splashing. Repeat the same procedure with remaining fusion tubes.

4) Boil the solution for few minutes along with broken fusion tubes. Filter the solution and collect the filtrate into small beaker. It is called as **'Sodium Extract' (S.E.)**

5) The elements C, N, S and X (X = Cl, Br, I halogens) gets converted into sodium compounds as.

i) $C + N + Na \rightarrow NaCN$
ii) $S + 2\ Na \rightarrow Na_2S$
iii) $X + Na \rightarrow NaX$

Part –II: Detection of N, S, halogens in Sodium Extract (S.E.)

No.	Test	Observation	Inference
1	**Test for Nitrogen, N:** S.E. 2 ml + 2 drops NaOH + 1-2 ml $FeSO_4$, boil + dil. H_2SO_4	a) Green or Blue colour b) No green or Blue colour	N Present N Absent
2	**Test for Sulphur, S:** S.E.1- 2 ml + 1 drops sodium nitroprusside	a) Violet or purple colour b) No violet or purple colour	S Present S Absent

3	**Test for Iodine, I:** S.E. 1 ml + $HgCl_2$	a) Scarlet red ppt b) No Scarlet red ppt	I Present I Absent
4	**Test for Bromine, Br:** S.E. 1 ml + Conc.+ H_2SO_4 + Starch	a) Yellowish brown colour b) No Yellow brown colour	Br Present Br Absent
5	**Test for Chlorine, Cl:** S.E. 1 ml + $AgNO_3$	a)White ppt soluble in NH_4OH b) No White ppt	Cl Present Cl Absent

Result: The given organic compound contains ..... Element/s.