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Practical Chemistry
For Health Specialties
CHEM 105-4

1441 / 1442 H

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Chemical analysis

Chemical analysis: is the study of the chemical composition and structure of substances. There are two kinds of analysis: qualitative analysis and quantitative analysis.

1- Qualitative Analysis

It is a chemical analysis which aims to identify the constituents of the substance, whether it is a pure substance (a sample salt) or a mixture of several substances.

2- Quantitative Analysis

It is a chemical analysis which aims to estimate the percentage of each essential components of substance.

Qualitative Analysis of ionic compounds

When a metal (on the left side of the periodic table) transfers one or more electrons to a non-metal (on the right side), ionic bonds are formed. Ionic compounds are composed of cations (basic radicals) and anions (acidic radicals). For example, NaCl salt is composed of sodium cation and chloride as anion.

Detection of the anions (acidic radicals) in ionic compounds

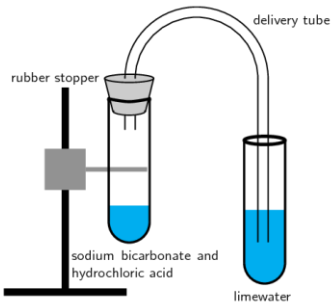
Anions are divided into three groups; each group has a certain reagent which the group is named after.

I) Anions of dilute hydrochloric acid group

Carbonate anion CO_3^{2-}

1- Reaction with group reagent (dil. HCl):

Take 0.1 g of solid salt of Na_2CO_3 in a test tube; add dilute HCl. Effervescence occurs and carbon dioxide gas is evolved which turns clear lime water milky by passing in it for short time.



$$Na_2CO_3 + 2\ HCl \longrightarrow 2\ NaCl + H_2O + CO_2 \uparrow$$

$$CO_2 + Ca(OH)_2 \longrightarrow CaCO_3 + H_2O$$

2- Reaction with magnesium sulphate:

To a solution of solid sodium carbonate in water add 1 ml of $MgSO_4$ solution. A white precipitate of magnesium carbonate $MgCO_3$ is formed on cold.

$$Na_2CO_3 + MgSO_4 \longrightarrow Na_2SO_4 + MgCO_3 \downarrow$$

white ppt.

3- Reaction with mercuric chloride:

To a solution of solid sodium carbonate in water, add 1 ml of $HgCl_2$. A reddish-brown precipitate of basic mercuric carbonate is formed on cold.

$$4\ Na_2CO_3 + 4\ HgCl_2 \longrightarrow HgCO_3 \cdot 3HgO + 8\ NaCl + 3\ CO_2$$

Reddish brown ppt.

Lab Report:

If you are provided with a sample of sodium carbonate salt:

Physical properties:

Color : Shape: Solubility in water:

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Chemical properties:

Experiment	Observation	Results
Solid + dil. HCl	Effervescence and evolution of CO_2 gas which turbid lime water.	CO_2 gas evolved
Salt solution + $MgSO_4$	White precipitate is formed on cold	$MgCO_3$
Salt solution + $HgCl_2$	Reddish brown precipitate is formed on cold.	$HgCO_3$. 3HgO

Bicarbonate anion HCO_3^-

1- Reaction with group reagent (dil. HCl):

Take 0.1 g of solid salt of $NaHCO_3$ in a test tube; add dilute HCl. Effervescence occurs and carbon dioxide gas is evolved which turns clear lime water milky by passing in it for short time.

$$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

$$CO_2 + Ca(OH)_2 \longrightarrow CaCO_3 + H_2O$$

2- Reaction with magnesium sulphate:

To a solution of solid sodium bicarbonate in water, add 1 ml of $MgSO_4$ solution. A white precipitate of magnesium carbonate $MgCO_3$ is formed after boiling.

$$2\ NaHCO_3 + MgSO_4 \longrightarrow Na_2SO_4 + Mg(HCO_3)_2$$

Magnesium bicarbonate (soluble)

$$Mg(HCO_3)_2 \xrightarrow{\text{boiling}} MgCO_3 + H_2O + CO_2$$

Magnesium carbonate white ppt.

3- Reaction with mercuric chloride:

To a solution of solid sodium bicarbonate in water add 1 ml of $HgCl_2$. No precipitate is formed on cold.

Lab Report:

If you are provided with a sample of sodium bicarbonate salt:

Physical properties:

Color: Shape: Solubility in water:

Chemical properties:

Experiment	Observation	Results
Solid + dil. HCl		
Salt solution + $MgSO_4$		
Salt solution + $HgCl_2$		

Thiosulphate anion $S_2O_3^{2-}$

1- Reaction with group reagent (dil. HCl):

In a test tube, add a few drops of dil. HCl to 0.1 g of solid sodium thiosulphate salt. Sulfur dioxide gas evolved and a yellow precipitate is formed due to the formation of suspended sulfur in the solution.

$$Na_2S_2O_3 + 2\,HCl \longrightarrow 2\,NaCl + H_2O + SO_2 + S\downarrow$$

2- Reaction with iodine solution:

On adding iodine solution to thiosulphate salt solution, the brown color of iodine solution disappears due to the formation of sodium tetra thionate which is colorless.

$$2\,Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2\,NaI$$

3- Reaction with barium chloride solution:

To a solution of solid sodium thiosulphate in water add 1 ml of $BaCl_2$. A white precipitate of BaS_2O_3 is formed.

$$Na_2S_2O_3 + BaCl_2 \longrightarrow 2NaCl + BaS_2O_3$$

Lab Report:

If you are provided with a sample of sodium thiosulphate salt:

Physical properties:

Color : Shape: Solubility in water:

Chemical properties:

Experiment	Observation	Results
Solid + dilHCl		
Salt solution + iodine solution		
Salt solution + $BaCl_2$		

II) Anions of concentrated sulfuric acid group

Chloride anion Cl^-

1- Reaction with group reagent (Conc. H_2SO_4):

On adding concentrated sulfuric acid with heating to sodium chloride solid salt, a colorless hydrogen chloride gas is evolved.

$$2\ NaCl + H_2SO_4 \longrightarrow Na_2SO_4 + 2\ HCl\uparrow$$

The evolved hydrogen chloride gas forms white clouds of ammonium chloride when it is exposed to a glass rod wetted by ammonia solution.

$$HCl + NH_3 \longrightarrow NH_4Cl$$

ammonium chloride

2-Reaction with silver nitrate solution:

In addition of silver nitrate solution to NaCl solution, a white precipitate of silver chloride AgCl is formed. This precipitate is insoluble in water but soluble in ammonium hydroxide solution and also it turns into violet when exposure to sunlight.

$$NaCl + AgNO_3 \longrightarrow NaNO_3 + AgCl\downarrow$$

silver chloride

3-Reaction with lead acetate solution $Pb(CH_3COO)_2$**:**

To a solution of sodium chloride add 1 ml of lead acetate solution. A white ppt. of $PbCl_2$ is formed which soluble in hot water.

$$2NaCl + (CH_3COO)_2Pb \longrightarrow 2\ CH_3COONa + PbCl_2\downarrow$$

Lab Report:

If you are provided with a sample of sodium chloride salt:

Physical properties:

Color : Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results
Solid + conc. H_2SO_4		
Salt solution + $AgNO_3$		
Salt solution + $Pb(CH_3COO)_2$		

III) Anions of Miscellaneous group

Sulphate anion SO_4^{2-}

1- Reaction with barium chloride solution:

On adding barium chloride solution to Na_2SO_4 , a white precipitate of barium sulphate is formed which is insoluble in diluted hydrochloric acid.

$$Na_2SO_4 + BaCl_2 \longrightarrow 2\ NaCl + BaSO_4\downarrow$$

2- Reaction with silver nitrate solution:

To a solution of sodium sulphate in a test tube, add 1ml of $AgNO_3$ solution. A white ppt. of Ag_2SO_4 is formed which is insoluble in dil HCl or dil. HNO_3.

$$Na_2SO_4 + 2\ AgNO_3 \longrightarrow 2\ NaNO_3 + Ag_2SO_4 \downarrow$$

3- Reaction with lead acetate solution:

A white precipitate of lead (II) sulphate is formed when few drops of lead acetate solution added to a 1 ml of sodium sulphate solution.

$$Na_2SO_4 + (CH_3COO)_2Pb \longrightarrow 2\ CH_3COONa + PbSO_4 \downarrow$$

lead (II)sulphate

Lab Report:

If you are provided with a sample of sodium sulphate salt:

Physical properties:

Color : Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results
Salt solution + $BaCl_2$		
Salt solution + $AgNO_3$		
Salt solution + $Pb(CH_3COO)_2$		

Phosphate anion PO_4^{3-}

1- Reaction with barium chloride solution:

On adding barium chloride solution to Na_3PO_4 , a white precipitate of barium phosphate is formed which is dissolve in diluted hydrochloric acid.

$$2\ Na_3PO_4 + 3\ BaCl_2 \longrightarrow 6\ NaCl + Ba_3(PO_4)_2 \downarrow$$

barium phosphate

2- Reaction with silver nitrate solution:

To a solution of sodium phosphate in a test tube, add 1ml of $AgNO_3$ solution. A yellow precipitate of silver phosphate is formed which is dissolves in dil. HNO_3.

$$Na_3PO_4 + 3\ AgNO_3 \longrightarrow 3\ NaNO_3 + Ag_3PO_4 \downarrow$$

silver phosphate

3- Reaction with ammonium molybdate solution $(NH_4)_2MoO_4$:

On adding ammonium molybdate solution to solution of Na_3PO_4 in presence of concentrated nitric acid, a canary yellow precipitate of ammonium-phosphomolybdate, $(NH_4)_3[P\ (Mo_3O_{10})_4]$ is formed after heating.

$$Na_3PO_4 + (NH_4)_2MoO_4 \xrightarrow{HNO_3} (NH_4)_3[P\ (Mo_3O_{10})_4] \downarrow$$

ammonium phosphomolybdate
(Canary yellow precipitate)

Lab Report:

If you are provided with a sample of sodium phosphate salt:

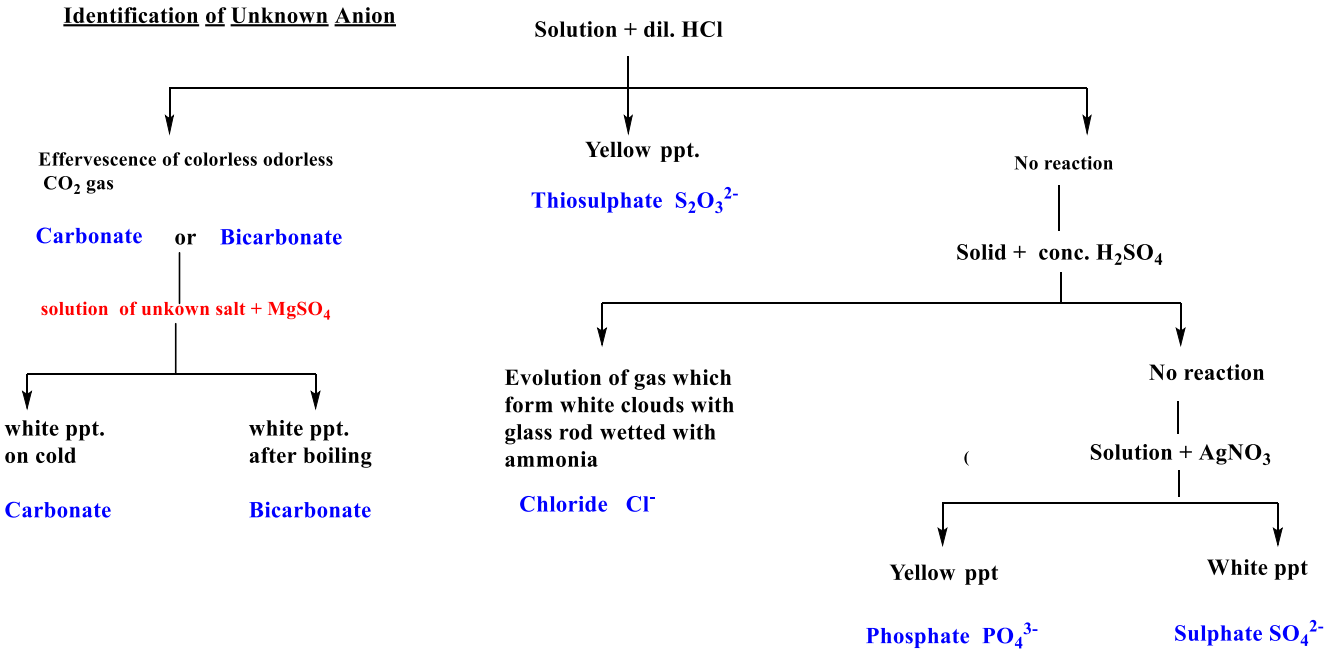
Physical properties:

Color: Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results
Salt solution + $BaCl_2$		
Salt solution + $AgNO_3$		
Salt solution + ammonium molybdate solution		

Scheme for identification of unknown acid radical



Identification of the cations (basic radicals) in the ionic compounds

The basic radicals are divided to 6 groups, called analytical groups, each group has its certain reagent which is called group reagent.

Group	Group reagent	Ions	Precipitated as	Formula of precipitate
First analytical group	dil. HCl	Pb^{2+}	Chlorides	$PbCl_2$
Second analytical group	H_2S + dil. HCl	Cu^{2+}	Sulphides	CuS
Third analytical group	NH_4OH + NH_4Cl	Al^{3+} ,	Hydroxides	$Al(OH)_3$
Fourth analytical group	H_2S + NH_4OH + NH_4Cl	Mn^{2+}, Zn^{2+}	Sulphides	MnS ZnS
Fifth analytical group	$(NH_4)_2CO_3$ + NH_4OH + NH_4Cl	Ba^{2+} & Ca^{2+}	Carbonates	$BaCO_3$ $CaCO_3$
Sixth analytical group	No particular reagent	NH_4^+		Different precipitated form

First analytical group

Group reagent: Diluted hydrochloric acid (dil. HCl)

Lead (II) Cation : Pb^{2+}

1- Reaction with group reagent:

On adding few drops of diluted hydrochloric acid to a solution of lead (II) nitrate, A white precipitate of $PbCl_2$ is formed which soluble in hot water and insoluble in NH_4OH.

$$Pb(NO_3)_2 + 2\ HCl \longrightarrow 2\ HNO_3 + PbCl_2 \downarrow$$

2- Reaction with potassium iodide solution:

Mix drops of KI solution with solution of $Pb(NO_3)_2$ to give yellow ppt. of PbI_2 soluble in hot water.

$$Pb(NO_3)_2 + 2\ KI \longrightarrow PbI_2 \downarrow + 2\ KNO_3$$

3- Reaction with potassium chromate K_2CrO_4

A yellow ppt. of $PbCrO_4$ was formed and it is insoluble in NH_4OH.

$$Pb(NO_3)_2 + K_2CrO_4 \longrightarrow PbCrO_4 + 2\ KNO_3$$

Lab Report:

If you are provided with a sample of lead nitrate salt:

Physical properties:

Color: Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results
Salt solution + dil HCl		
Salt solution + KI		
Salt solution + K_2CrO_4		

Second analytical group

Group reagent: Hydrogen sulphide in acidic medium (dil. HCl + H_2S)

Copper (II) Cation : Cu^{2+}

1- Reaction with group reagent:

On adding drops of diluted hydrochloric acid to a solution of copper (II) sulphate then passing hydrogen sulphide gas through the solution (or used solution of sodium sulphide), a black precipitate is formed of copper (II) sulphide which dissolve in hot nitric acid.

$$CuSO_4 + H_2S \xrightarrow{\text{dil. HCl}} H_2SO_4 + \underset{\text{copper (II) sulphide}}{CuS}$$

2- Reaction with sodium hydroxide solution:

A blue precipitate of $Cu(OH)_2$ is formed which turns black precipitate after boiling due to formation of CuO (cupric oxide).

$$CuSO_4 + NaOH \xrightarrow{\text{dil. HCl}} Cu(OH)_2 + Na_2SO_4$$

$$Cu(OH)_2 \xrightarrow{\text{heat}} CuO + H_2O$$

3- Reaction with ammonium hydroxide solution:

A blue ppt. of $Cu(OH)_2$ is formed which soluble in excess of NH_4OH forming a deep blue color solution.

$$CuSO_4 + 2\ NH_4OH \longrightarrow Cu(OH)_2 + (NH_4)_2SO_4$$

$$Cu(OH)_2 \xrightarrow{\text{excess of } NH_4OH} \text{soluble}$$

Lab Report:

If you are provided with a sample of copper sulphate salt:

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Physical properties:

Color : Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results
Salt solution + dil. HCl + H_2S		
Salt solution + NaOH		
Salt solution + NH_4OH		

Third analytical group

Group reagent: Ammonium hydroxide solution in presence of ammonium chloride Solution (NH_4Cl is used to prevent the precipitation of hydroxides of other metals e.g Mn, Zn, Co, Ni and Mg).

Aluminum Cation : Al^{3+}

1- Reaction with group reagent:

On adding ammonium hydroxide solution to a solution of aluminium sulphate, a gelatinous white precipitate of aluminium hydroxide is formed which dissolves in all dilute acids.

$$Al_2(SO_4)_3 + 6\,NH_4OH \xrightarrow{NH_4Cl} 3\,(NH_4)_2SO_4 + 2\,Al(OH)_3$$

Aluminum hydroxide

2- Reaction with sodium hydroxide solution:

Gelatinous white precipitate of $Al(OH)_3$ is formed.

$$Al_2(SO_4)_3 + 6NaOH \longrightarrow 3\,Na_2SO_4 + 2\,Al(OH)_3$$

Aluminium hydroxide

Aluminium hydroxide precipitate dissolves in excess NaOH forming sodium meta-aluminate.

$$Al(OH)_3 + NaOH \longrightarrow NaAlO_2 + 2\,H_2O$$

3- Reaction with sodium phosphate solution:

A white precipitate of aluminium phosphate is formed which is dissolves in ammonium hydroxide and diluted acids.

$$Al_2(SO_4)_3 + 2Na_3PO_4 \longrightarrow 2AlPO_4 + 3Na_2SO_4$$

Lab Report:

If you are provided with a sample of Alumium sulphate salt:

Physical properties:

Color : Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results
Salt solution + NH_4OH+ NH_4Cl		
Salt solution+ NaOH		

Fourth analytical group

Group reagent:

Hydrogen sulphide in basic medium ($NH_4OH + NH_4Cl + H_2S$)

Zinc Cation : Zn^{2+}

1- Reaction with group reagent:

On adding drops of NH_4OH solution and NH_4Cl solution to a solution of zinc sulphate then passing hydrogen sulphide gas through the solution (or used solution of sodium sulphide), a white precipitate of zinc sulphide (ZnS) which soluble in mineral acids.

$$ZnSO_4 + H_2S \longrightarrow ZnS + H_2SO_4$$

2- Reaction with sodium hydroxide solution:

Gelatinous white ppt. of $Zn(OH)_2$ is precipitated which soluble in excess of sodium hydroxide forming sodium zincate Na_2ZnO_2.

$$ZnSO_4 + NaOH \longrightarrow Zn(OH)_2 + Na_2SO_4$$

$$Zn(OH)_2 + 2NaOH \longrightarrow Na_2ZnO_2 + 2\,H_2O$$

sodium zincate

3- Reaction with ammonium hydroxide solution:

A white ppt. of $Zn(OH)_2$ is precipitated which insoluble in excess of NH_4OH.

$$ZnSO_4 + 2NH_4OH \longrightarrow Zn(OH)_2 + (NH_4)_2SO_4$$

4- Reaction with sodium phosphate solution:

A white precipitate of zinc phosphate is formed which is dissolves in ammonium hydroxide and diluted acids.

$$3\,ZnSO_4 + 2Na_3PO_4 \longrightarrow Zn_3(PO_4)_2 + 3Na_2SO_4$$

Lab Report:

If you are provided with a sample of zinc sulphate salt:

Physical properties:

Color: Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results

Manganese Cation : Mn^{2+}

1- Reaction with group reagent:

A pale brown (buff) ppt. of Manganese sulphide (MnS) is formed which soluble in dil. acids.

$$MnCl_2 + H_2S \longrightarrow MnS + 2HCl$$

2- Reaction with ammonium hydroxide solution:

White precipitate of manganese (II) hydroxide is formed.

$$MnCl_2 + 2\ NH_4OH \longrightarrow Mn(OH)_2 + 2\ NH_4Cl$$

Lab Report:
If you are provided with a sample of $MnCl_2$ salt:
Physical properties:
Color : Shape: Solubility in water:
Chemical properties

Experiment	Observation	Results

Fifth analytical group

Group reagent: Ammonium carbonate solution $(NH_4)_2CO_3$) in the presence of NH_4OH and NH_4Cl.

Calcium Cation : Ca^{2+}

1- Reaction with group reagent:

On adding ammonium carbonate solution to calcium chloride solution, a white precipitate of calcium carbonate is formed which dissolves in mineral acids.

$$CaCl_2 + (NH_4)_2CO_3 \longrightarrow 2NH_4Cl + CaCO_3$$

2- Reaction with diluted sulphuric acid:

A white precipitate of calcium sulphate is formed.

$$CaCl_2 + H_2SO_4 \longrightarrow 2HCl + CaSO_4$$

3- Flame Test

At expose the solid of calcium salt to flame, calcium cations turn the non-luminous part of Bunsen flame into brick red.

Lab Report:
If you are provided with a sample of calcium chloride salt:
Physical properties:

Color : Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results

Barium Cation: Ba^{2+}

1- Reaction with group reagent:

On adding ammonium carbonate solution to barium chloride solution, a white precipitate of barium carbonate is formed which dissolves in mineral acids.

$$BaCl_2 + (NH_4)_2CO_3 \longrightarrow BaCO_3 + 2NH_4Cl$$

2- Reaction with calcium sulphate:

A white precipitate of barium sulphate is formed.

$$BaCl_2 + CaSO_4 \longrightarrow BaSO_4 + CaCl_2$$

3- Reaction with potassium chromate:

A yellow precipitate of barium chromate is formed which insoluble in acetic acid.

$$BaCl_2 + K_2CrO_4 \longrightarrow BaCrO_4 + 2KCl$$

Lab Report:

If you are provided with a sample of calcium chloride salt:

Physical properties:

Color : Shape: Solubility in water:

Chemical properties

Experiment	Observation	Results

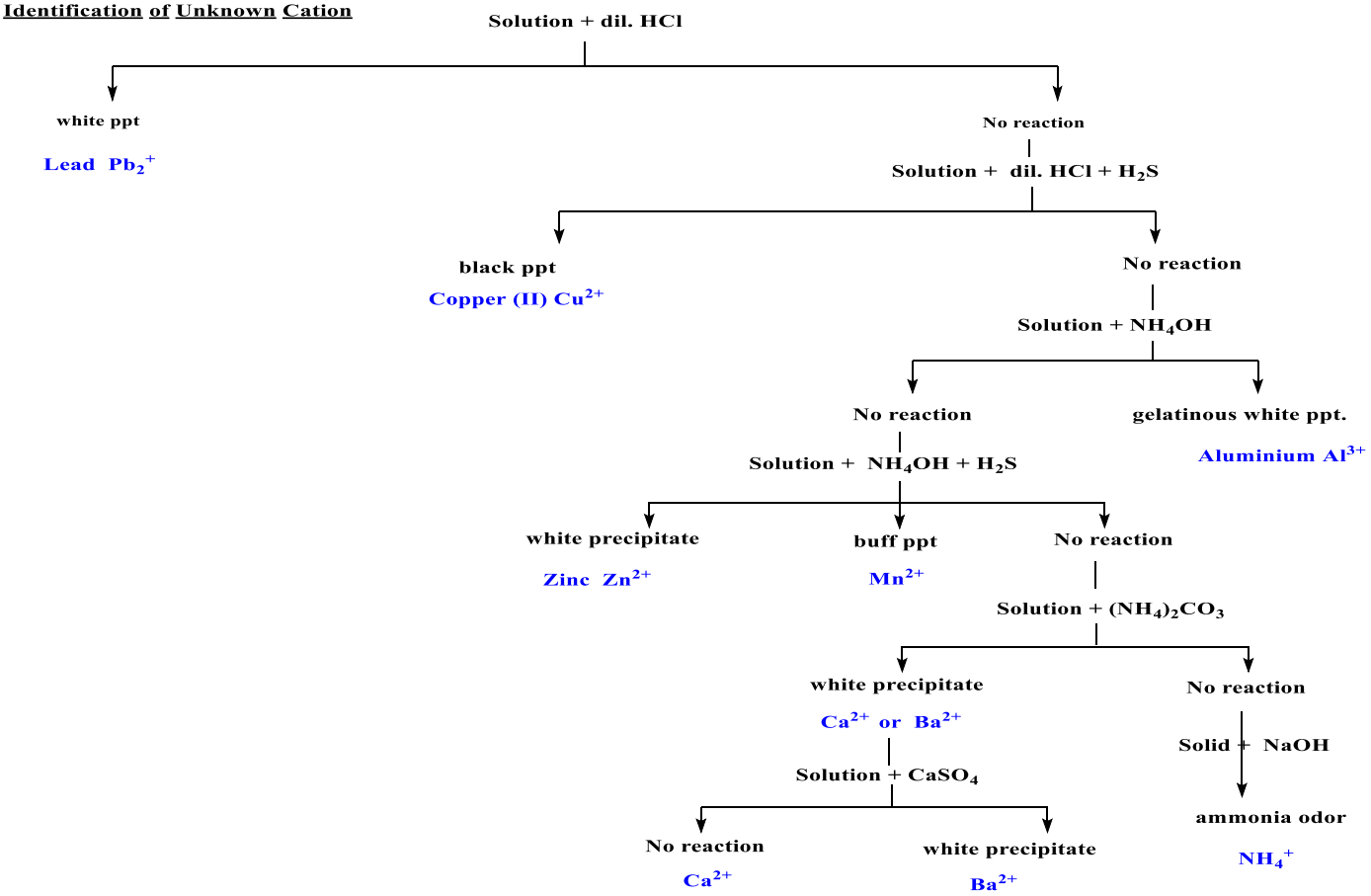
Sixth analytical group

Ammonium Cation NH_4^+

At treatment of solid ammonium chloride with sodium hydroxide solution, ammonia odor is produced.

$$NH_4Cl + NaOH \longrightarrow NH_3 + NaCl + H_2O$$

Identification of Unknown Cation



Quantitative Analysis

Quantitative analysis is classified into two main categories which are volumetric analysis and gravimetric analysis.

Volumetric analysis:

It is a kind of quantitative analysis based on measurement of volumes of the substances to be analyzed. It is commonly used to determine the unknown concentration of a known reactant. Volumetric analysis is often referred to as titration.

Titration:

A process of determining the concentration of an acidic (or basic) solution by knowing the volume of which that is required to neutralize another basic (or acidic) solution of known volume and concentration.

In this process, the consumed volume of a solution of known concentration will measuring through titration with a known volume of another solution which is of unknown concentration.

Titrant (the standard solution):

Solution of a known concentration, which is added to another solution whose concentration, has to be determined. On the other words, it's a solution of accurately known concentration.

Titrand or analyte:

It's the solution whose concentration has to be determined.

Equivalent point:

Point in titration at which the amount of titrant added is just enough to completely neutralize the analyte solution. At the equivalent point in an acid –

base titration, moles of base equal moles of acid and the solution only contains salt and water.

Indicator:

It's a weak acid or base that is added to the analyte solution and it changes color when the equivalent point is reached.

End point:

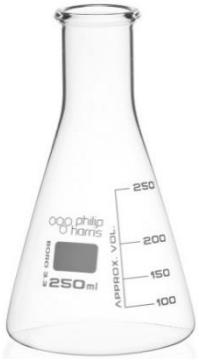
Refer to the point at which the indicator changes color in an acid – base titration.

Acid base titration

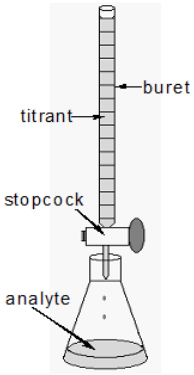
These titrations are based on the neutralization reaction that occurs between an acid and a base.

Tools used in titration:

- Burette
- Pipette
- Funnel
- Erlenmeyer or conical flask
- White tile (used to see a color change in the solution)
- Titrant (a standard solution of known concentration)
- Analyte, or titrand (the solution of unknown concentration)
- Indicators



Conical flask



Titration set

Pipette Funnel

Standardizations of a hydrochloric acid solution using a standard solution of sodium carbonate

Principle:

A two-stage reaction occurs when you add a hydrochloric acid (HCl) solution to a solution of sodium carbonate (Na_2CO_3):

1- The hydrogen ion in HCl switches places with one of the sodium ions in Na_2CO_3 to produce sodium hydrogen carbonate, also known as sodium bicarbonate (baking soda), and sodium chloride (salt).

$$Na_2CO_3 + HCl \longrightarrow NaHCO_3 + NaCl$$

2- Sodium bicarbonate is basic, and it reacts with the HCl still in solution to produce sodium chloride, carbon dioxide and water.

$$NaHCO_3 + HCl \longrightarrow NaCl + CO_2 + H_2O$$

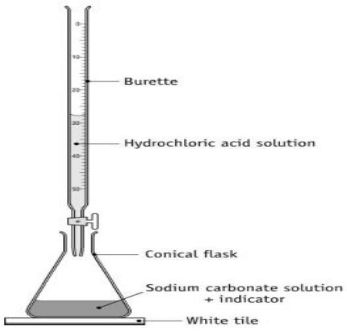
- Phenolphthalein indicator is a good indicator for the first stage because it responds to the pH change caused by the formation of sodium bicarbonate (pH change from 11 to 8). It is pink in basic solutions and turns colorless as soon as the solution becomes acidic.

- Methyl orange indicator, in the second stage, responds to pH changes associated with the formation of NaCl (pH change from 8 to 3), changing from yellow to red as the solution becomes more acidic.

- When ph.ph. is used in the neutralization of HCl with sodium carbonate, the volume of acid used will be equivalent to half of the carbonate, while when methyl orange is used in this titration the volume of acid used will be equivalent to all carbonate.

Chemicals and tools:

1	50-mL Burette	6	Pipette pump.
2	250-mL conical flasks	7	HCl (aq)
3	Burette funnel	8	0.1 N Na_2CO_3 (aq)
4	Two 250-mL beaker	9	M.O indicator
5	10-mL pipette	10	Phenolphthalein



Procedure:

1- Wash the tools by distilled water.

2- Fill the burette with the hydrochloric acid.

3- Draw 10 mL of 0.1N of the sodium carbonate solution by a pipette and transfer it to clean conical flask

Using phenolphthalein indicator*:*

4- Add 2-3 drops of phenolphthalein to the sodium carbonate solution in the flask and note the pink color.

5- Place the flask under the burette and start adding the acid solution to the Erlenmeyer flask, until the color change from pink to colorless. Record the volume consumed from the burette.

6- Repeat step 5, three times.

Using methyl orange *indicator:*

7- Using the clean pipette and transfer 10 mL of the o.1 N sodium carbonate solution into the clean conical flask. Add 2-3 drops of methyl orange indicator. Note the color of the solution.

8- Carry out a titration by adding hydrochloric acid solution from the burette until the color of the solution in the conical flask changes from yellow to red. Record the volume consumed from the burette. 9- Repeat step 8, three times.

Results and Calculations:

1. In Case of using ph.ph

$$\text{The volume of HCl } V_1 = \frac{1}{2} \text{ of carbonate}$$

$$\text{So, } \quad 2 \times V_1 = \text{all carbonate}$$

$$(N_1 \times 2V_1)\ HCl = (N_2 \times V_2)\ Na_2CO_3$$

$$N_1 \text{ of HCl} = \frac{N_2 \times V_2}{2V_1}$$

strength in gm/L = normality x eq. wt.

Strength of HCI = N_1 x 36.5 g/L

2. In case of using M.O

Start point	End point	Volume of HCl consumed (V_1)
		Average V_1 =

$$\text{The volume of HCl } V_1 = \text{all carbonate}$$

$$(N_1 \times 2V_1)\ HCl = (N_2 \times V_2)\ Na_2CO_3$$

$$N_1 \text{ of HCl} = \frac{N_2 \times V_2}{V_1}$$

strength in gm/L = normality x eq. wt.

Strength of HCI = N_1 x 36.5 g/L

Estimation of the normality of sodium hydroxide using a standard hydrochloric acid

The principle:

Aim: To estimate the amount of sodium hydroxide dissolved in 250 ml of the given unknown solution volumetrically. For this, you are given with a standard solution of sodium carbonate solution of normality 0.1 N (standard solution) and hydrochloric acid solution.

In the first step: Neutralization of sodium carbonate by HCl is given below to indicate the end point, methyl orange is used as an indicator.

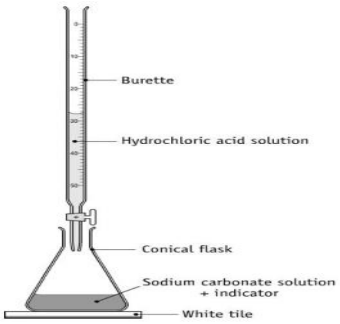
$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2$$

In the second step: Neutralization of sodium hydroxide by HCl is given below. To indicate the end point, phenolphthalein is used as an indicator.

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

Chemicals and tools:

1	50-mL Burette	7	HCl (aq)
2	250-mL conical flasks	8	0.1 N Na_2CO_3 (aq)
3	Burette funnel	9	NaOH solution
4	Two 250-mL beaker	10	M.O indicator
5	10-mL pipette	11	Phenolphthalein
6	Pipette pump.	12	Distilled water



Procedures:

a) Standardization of HCl:

1. Wash the tools by distilled water.
2. Fill the burette with the hydrochloric acid.
3. Draw 10 mL of 0.1N of the sodium carbonate solution into the volumetric pipette and transfer this solution into an Erlenmeyer flask.
4. Add 2-3 drops of methyl orange indicator. Note the color of the solution.

5. Carry out a titration by adding hydrochloric acid solution from the burette until the color of the solution in the conical flask changes from yellow to red. Record the volume consumed from the burette.
6. Repeat step 5, three times.

b) <u>Standardization of NaOH:</u>

1- Wash all tools by distilled water.
2- Put 10 ml of NaOH solution in conical flask (using pipette) and then add 2 drops of phenolphthalein indicator. The color of the indicator turns to pink color.
3- Fill the burette with HCl and titrate NaOH solution until the color of indicator is discharged.
4- Repeat step 9, three times.

<u>Calculations:</u>

<u>Standardization of HCl</u>

Start point	End point	Volume consumed (V_1)
		Average V_1 =

Suppose that the volume of HCl is V_1 and its normality is N_1 while V_2 is the volume taken from sodium carbonate and N_2 is its normality.

The volume of HCl (from burette) ≡ all carbonate = V_1

$$(N_1 \times V_1)\ HCl = (N_2 \times V_2)\ Na_2CO_3$$

$$N_1\ HCl = \frac{(N_2 \times V_2)\ Na_2CO_3}{V_1\ HCl} = N$$

Estimation of the normality of sodium hydroxide

For HCl:

N_1 = Known from the previous step, V_1 = the average consumed volume

For NaOH:

N_2 = Unknown V_2 = 10 ml

Start point	End point	Volume consumed (V_1) ml
		Average V_1 = ml

Thus, we can calculate the normality of NaOH from the following relation:

$$N_1V_1 = N_2V_2$$

We can calculate the strength of NaOH from the following equation:

Eq. wt. of NaOH = 40

S = Normality x Equivalent weight

= N2 × 40

Practical Organic Chemistry

Liquids

I) Alcohols

a) Required Chemicals:

Alcohols: Methanol (CH_3OH), ethanol (CH_3CH_2OH), glycerol, ($CH_2OHCHOHCH_2OH$)

Reagents:

Potassium dichromate ($K_2Cr_2O_7$), potassium permanganate ($KMnO_4$), concentrated sulfuric acid (H_2SO_4), salicylic acid, sodium bicarbonate ($NaHCO_3$), iodine solution (I_2), sodium hydroxide (NaOH).

Indicators:

Phenolphthalein (Ph. Ph) and borax solution.

b) Apparatus:

Test tubes, test tubes rack, test tubes holders, clamps, beakers and Bunsen flame.

c) Experimental:

(1) Methyl Alcohol (CH_3OH)

Physical Properties:
Color: Colorless liquid
Miscibility: Miscible with water
Effect on L.P: Neutral

Chemical Properties:

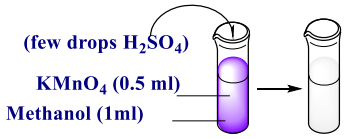
1 - Oxidation by $K_2Cr_2O_7$:

In a dry test tube 0.3 ml of methyl alcohol was taken with 0.3 ml of $K_2Cr_2O_7$ followed by the addition of few drops of conc. H_2SO_4 with gentle heating. The color changes to green.

$$CH_3OH + K_2Cr_2O_7 \xrightarrow[\triangle]{H_2SO_4\ conc.} HCHO + Cr_2(SO_4)_3 + K_2SO_4$$

2 - Oxidation by $KMnO_4$:

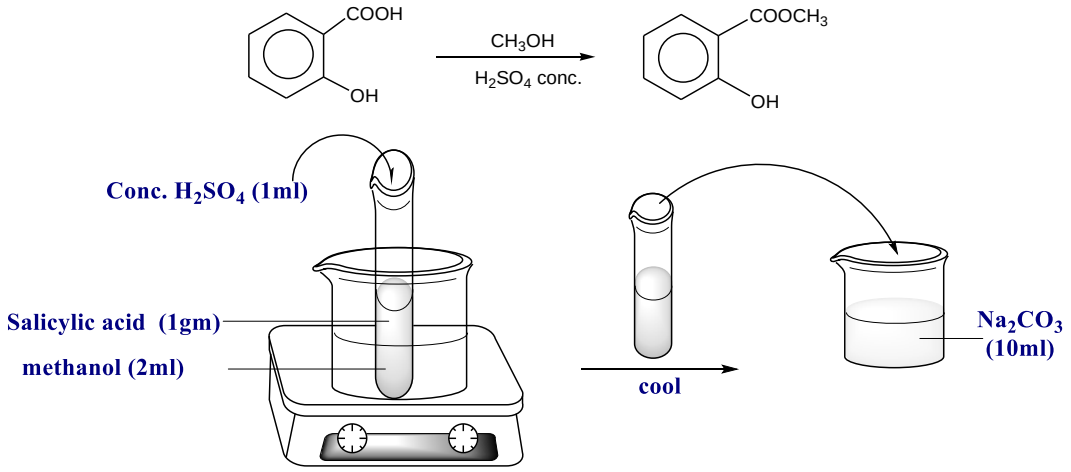
In a dry test tube 0.3 ml of methyl alcohol was taken with 0.2 ml of $KMnO_4$ followed by the addition of few drops of conc. H_2SO_4. The violet color of $KMnO_4$ disappears.



$$CH_3OH + KMnO_4 + H_2SO_4 \rightarrow HCHO + MnSO_4 + K_2SO_4$$

3 -Esterification:

In a dry test tube 1ml methanol, 0.2 gm of salicylic acid and few drops of concentrated H_2SO_4 were heated to boiling with continuous shaking for 5 minutes. After cooling the mixture is poured onto a solution of 10 ml of sodium bicarbonate in a beaker. Effervescence with characteristic odor of methyl salicylate was observed.



Laboratory report

Physical Properties: -

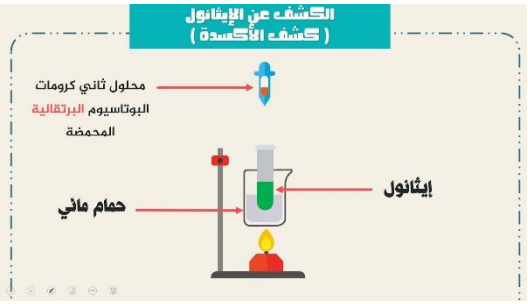
Color:
Miscibility:
Effect on L.P.

Chemical Properties: -

No.	Experiment	Observation	Results
1	$K_2Cr_2O_7$		
2	$KMnO_4$		
3	Esterification		

(2) Ethyl alcohol, (C_2H_5OH)

Physical Properties:
Color: Colorless liquid
Miscibility: Miscible with water
Effect on LP: Neutral



Chemical Properties:

1 - Oxidation by $K_2Cr_2O_7$:

In a dry test tube take 1 ml of ethyl alcohol, 0.5 ml of $K_2Cr_2O_7$ followed by the addition of few drops of conc. H_2SO_4. With gentle heating, the color changes to green.

$$CH_3CH_2OH + K_2Cr_2O_7 \xrightarrow[\triangle]{H_2SO_4} HCHO + Cr_2(SO_4)_3 + K_2SO_4$$

2- Oxidation by $KMnO_4$:

In a dry test tube take 1 ml of ethyl alcohol, 0.5 ml of $KMnO_4$ followed by the addition of few drops of conc. H_2SO_4. The violet color of $KMnO_4$ disappears.

$$C_2H_5OH + K_2MnO_4 + H_2SO_4 \rightarrow CH_3CHO + MnSO_4 + K_2SO_4$$

3- Esterification:

In a dry test tube, a mixture of 1 ml ethanol, 1 ml of acetic acid and few drops of conc. H_2SO_4 were heated to boiling with continuous shaking for 5 minutes. After cooling, the mixture is poured onto a solution of 10 ml of sodium bicarbonate in a beaker. Effervescence with characteristic odor of ethyl acetate was observed.

$$C_2H_5OH + CH_3COOH \xrightarrow[heat]{H_2SO_4\ conc} CH_3COOC_2H_5 + H_2O$$

4- Iodoform Test:

In a clean test tube, a mixture of 1 ml ethyl alcohol, 3 ml iodine solution and about 1 ml of diluted NaOH was heated on water bath (W.B.) till the color becomes yellow. Yellow crystals of iodoform were separated after cooling.

$$CH_3CH_2OH + I_2 \xrightarrow{\text{Oxidation}} CH_3CHO + 2HI$$

$$CH_3CHO + 3\,I_2 \xrightarrow{\text{Replacement}} CI_3CHO + 3HI$$

$$CI_3CHO + NaOH \xrightarrow{\text{hydrolysis}} CHI_3 + HCOONa$$

Laboratory report

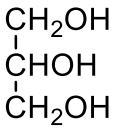
Physical Properties: -

Color:
Miscibility:
Effect on L.P.

Chemical Properties: -

No.	Experiment	Observation	Results
1	$K_2Cr_2O_7$ + conc. H_2SO_4		
2	$KMnO_4$ + conc. H_2SO_4		
3	Esterification		
4	Iodoform		

(3) Glycerol



Physical Properties:
Color: Colorless liquid
Miscibility: Miscible with water
Effect on LP: Neutral

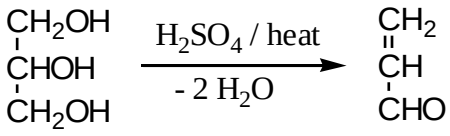
Chemical Properties:

1- Borax Test:

In a test tube take 1 ml of borax solution and two drops of phenol phthalein (Ph. Ph.) a pink color appears. Few drops of glycerol were added till the pink color disappears. Warm the solution the pink color reappears while disappears on cooling.

2- Acrolein Test:

In a dry clean tube, a mixture of glycerol 0.2 ml and 2-3 drops of concentrated H_2SO_4 was heated vigorously with shaking in a fume cupboard. An acrolein odor was observed.



Laboratory report

Physical Properties: -
Color:
Miscibility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Borax test		
2	Acrolein test		

II) Aldehydes

(1) Formaldehyde (HCHO)

Physical Properties: -
Color: Gas (40% in water)
Miscibility: Soluble in water
Effect on LP: Neutral
Toxicity: Highly toxic

Chemical Properties: -

1– $AgNO_3$ test:

In a test tube a mixture of 0.5 ml solution of formaldehyde (formalin) and 0.5 ml of ammoniacal silver nitrate is heated on a water bath for about 5 minutes silver mirror appears on the wall of the test tube.

$$HCHO + \text{amm. } AgNO_3 \xrightarrow{\triangle} HCOOH + Ag$$

2- Fehling's test:

In a test tube, a mixture of 0.5 ml of Fehling A, 0.5 ml of Fehling B and 1 ml HCHO is heated on water bath for few minutes. a reddish-brown precipitate of cuprous oxide is formed.

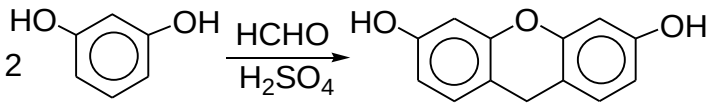
$$HCHO + 2\ CuO \rightarrow HCOOH + Cu_2O$$

3– Schiff's test:

In a test tube, shake 0.2 ml of formaldehyde solution and 0.1 ml of Schiff's reagent. A purple color is appeared.

4-Xanthene test:

In a test tube, shake a mixture of 0.2 ml of formaldehyde solution and 0.1 ml of resorcinol and then add 0.2 ml of conc. H_2SO_4 A red violet ring with reddish white precipitate is formed.



Laboratory report

Physical Properties:-

Color:
Miscibility:
Effect on LP:

Chemical Properties:-

No.	Experiment	Observation	Results
1	Amm.$AgNO_3$ Test		
2	Fehling's Test		
3	Schiff's reagent		
4	Xanthene Test		

(2) Acetaldehyde (CH_3CHO)

Physical Properties: -

Color: liquid
Miscibility: Miscible with water
Effect on LP: Neutral

Chemical Properties: -

1– Amm. $AgNO_3$ test:

In a test tube take 0.5 ml of acetaldehyde solution and 0.5 ml of ammoniacal silver nitrate then heat on water bath for about 5 minutes a silver mirror was appeared on the wall of the test tube.

$$CH_3CHO + \text{Amm. } AgNO_3 \xrightarrow{\triangle} CH_3COOH + Ag$$

2- Fehling's test:

In a test tube, heat 0.5 ml of Fehling A, 0.5 ml of Fehling B and 1 ml CH_3CHO on water bath for few minutes. A reddish-brown precipitate of cuprous (Cu^{+2}) complex is formed.

$$CH_3CHO + 2\ CuO \rightarrow CH_3COOH + Cu_2O$$

3– Schiff's test:

In a test tube, a mixture of 0.2 ml of acetaldehyde solution and 0.1 ml of Schiff's reagent was shaken to give purple color.

4- Iodoform test:

In a clean test tube, a mixture of 0.3 ml acetaldehyde, 1 ml iodine solution and about 1 ml of diluted NaOH was heat on water bath (W.B.) till the color changes to yellow and pale yellow precipitate was formed.

$$CH_3CHO + 3\ I_2 \xrightarrow{\text{replacement}} CI_3CHO + 3\ HI$$

$$CI_3CHO + NaOH \xrightarrow{\text{hydrolysis}} CHI_3 + HCOONa$$

5- Sodium nitroprusside test:

In a clean test tube, shake 0.2 ml of acetaldehyde, 0.2 ml of sodium nitroprusside and a solution of sodium hydroxide to give deep red color.

Laboratory report

Physical Properties: -

Color:

Miscibility:

Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Amm.$AgNO_3$ Test		
2	Fehling's Test		
3	Schieff's Reagent		
4	Iodoform Reaction		
5	Condensation Reactions		
6	Sodium nitroprusside		

(3) Chloral hydrate, ($CCl_3CHO.\ H_2O$)

Physical Properties: -

Color: Crystalline solid

Solubility: Soluble in water

Effect on LP: Neutral

Chemical Properties: -

1-Effect of NaOH

In a test tube, 0.2 gm of chloral hydrate ($CCl_3CHO.H_2O$) is heated with 0.5 ml of NaOH to give chloroform odor ($CHCl_3$), (two layers separated).

$$CCl_3CHO.\ H_2O + NaOH \xrightarrow{\triangle} CHCl_3 + HCOONa + H_2O$$

2- Effect of conc. H_2SO_4

In a test tube, add 0.2 gm of chloral hydrate and 0.2 ml of conc. H_2SO_4, shake well then leave the tube. An oily layer of chloral appears in the solution.

$$CCl_3CHO.\ H_2O + \text{conc. } H_2SO_4 \rightarrow CCl_3CHO + H_2O$$

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Laboratory report

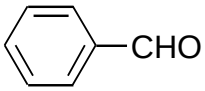
Physical Properties: -

Color:
Solubility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	NaOH Test		
2	H_2SO_4 Test		

(4) Benzaldehyde,



Physical Properties: -

Color: liquid
Miscibility: Immiscible with water, miscible with organic solvents
Effect on LP: Neutral

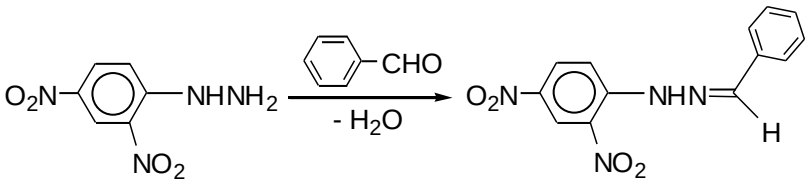
Chemical Properties: -

1– Schiff's test:

In a test tube, a mixture of 0.2 ml of benzaldehyde and 0.1 ml of Schiff's reagent is shaken vigorously to give purple color.

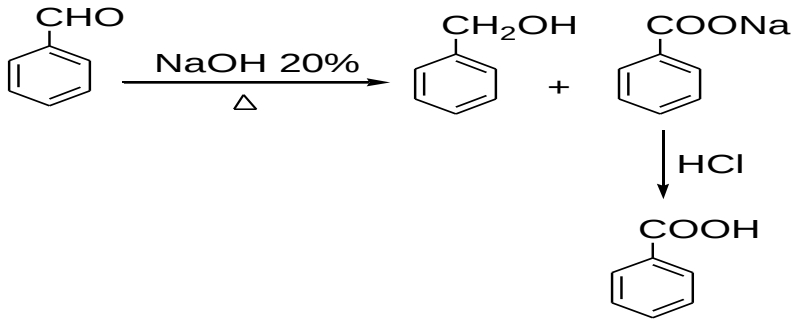
2- Condensation test:

A solution of 0.2 ml benzaldehyde and 0.2 ml of 2,4-dinitrophenyl hydrazine is heated on water bath to give yellow precipitate of the hydrazone derivative.



3- Cannizzaro test:

In a dry test tube, heat gently 0.1 ml of benzaldehyde with 0.2 ml of 20% NaOH then cool followed by addition of drops of conc. HCl. A white precipitate of benzoic acid is formed.



Laboratory report

Physical Properties:-
Color:
Miscibility:
Effect on LP:
Chemical Properties:-

No.	Experiment	Observation	Results
1	Schiff's Reagent		
2	Condensation Reactions		
3	Cannizzaro Reaction		

III) Ketones

(5) Acetone (CH_3-CO-CH_3)

Physical Properties:-
Color: Colorless liquid
Miscibility: Miscible with water
Effect on LP: Neutral
Chemical Properties:-

1- Iodoform test:

In a clean test tube, add 0.3 ml of acetone, 1 ml of iodine solution and about 1 ml of conc. NaOH. The color of the solution turns yellow followed by yellow crystals separation.

$$CH_3COCH_3 + 3I_2 \rightarrow CI_3COCH_3 + 3HI$$

$$CI_3COCH_3 + NaOH \rightarrow CHI_3 + CH_3COONa$$

2- Sodium nitroprusside test:

In a clean test tube a mixture of 0.2 ml of acetone, 0.2 ml of sodium nitroprusside and an excess solution of sodium hydroxide is shaken to give deep red color.

3- Deing's test:

In a clean test tube 0.3 ml of acetone heated with 0.2 ml of Deing's (1) followed by addition of 0.2 ml of Deing's (2) the color is discharged and a heavy white precipitate is formed.

4- Schiff's test:

In a test tube, a mixture of 0.2 ml of acetone and 0.1 ml of Schiff's reagent gives no color.

Acetone

Physical Properties:-

Color:
Miscibility:
Effect on LP:

Chemical Properties:-

No.	Experiment	Observation	Results
1	Iodoform Reaction		
2	Sodium nitroprusside Test		
3	Deing's Test		
4	Schiff's Reagent		

Carboxylic Acids

IV) Aliphatic Carboxylic Acids

Carboxylic acids are organic compounds containing one or more of carboxylic group attached to an alkyl or aryl group. They have a general formula (Ar) R-COOH. Carboxylic acids are classified as mono- , di and tricarboxylic acids:

a) Chemicals Required:

Formic acid, acetic acid, oxalic acid, tartaric acid, succinic acid, citric acid, sodium carbonate, ferric chloride ($FeCl_3$), $AgNO_3$, $KMnO_4$, ethanol, conc. sulfuric acid, Ammonium hydroxide, calcium chloride, urea, resorcinol and Deing's (1) and (2).

b) Equipments:

Test tubes, test tube rack, test tube holder clamps, beakers and Bunsen burner.

c) Experimental:

Preparation of Neutral Solution (N.S):

In a beaker, take about 1 ml (or 1 g) of the free acid and about 10 ml of H_2O followed by addition of NH_4OH solution till becomes neutral (check with red and blue litmus papers). If the solution becomes alkaline boil to evaporate the excess ammonia (check with red and blue LP)

(1) Formic acid)HCOOH)

Physical Properties: -
Color: Colorless liquid
Miscibility: Miscible with water
Effect on LP: Acidic
Odor: Irritant

Chemical Properties:

1- Acidity test:

In a test tube, take few drops of formic acid and dilute with 0.5 ml of water followed by dropwise addition of sodium bicarbonate solution. A strong effervescence appeared and CO_2 evolved.

$$HCOOH + NaHCO_3 \rightarrow HCOONa + CO_2\uparrow + H_2O$$

2- $FeCl_3$ test:

In a test tube, add few drops of $FeCl_3$ solution to 0.3 ml of the neutral solution of formic acid. A red color appears turns to brown precipitate on heating.

$$\mathbf{HCOONH_4 + FeCl_3 \rightarrow (HCOO)_3Fe \xrightarrow{\triangle} (HCOO)Fe(OH)_2}$$

3- Effect of ammoniacal $AgNO_3$:

In a test tube, to 0.3 ml of the neutral solution of formic acid add 0.3 ml of ammoniacal silver nitrate then heat on water bath. A white precipitate of silver formate is formed.

$$HCOONH_4 + Ag\ NO_3 \rightarrow HCOOAg + NH_4NO_3$$

4-Effect of $KMnO_4$:

In a test tube, to 0.3 ml of the neutral solution of formic acid add few drops of $KMnO_4$, the violet color discharge.

5- Esterification:

In a dry test tube, 0.4 ml formic acid, 1 ml of ethanol and few drops of concentrated H_2SO_4 were heated to boiling with continuous shaking for 5 minutes. After cooling, the mixture is poured onto a solution of 10 ml of sodium bicarbonate in a beaker. Effervescence with characteristic odor of ethyl formate was observed.

$$HCOOH + C_2H_5OH \xrightarrow{\text{conc. } H_2SO_4} HCOOC_2H_5 + H_2O$$

Laboratory Report

Physical Properties: -

Color:
Solubility:
Effect on LP:

No.	Experiment	Observation	Results
1	Acidity Test		
2	$FeCl_3$ Test		
3	$AgNO_3$ Test		
4	$KMnO_4$ Test		
5	Esterification		

(2) Acetic acid (CH_3COOH)

Physical Properties:-
Color: Colorless liquid
Miscibility: Miscible with water
Effect on LP: Acidic (change blue LP to red)
Odor: Irritant

Chemical Properties:

1- Acidity test:

In a test tube, take few drops of acetic acid and dilute with 0.5ml water followed by drop wise addition of sodium carbonate solution, a strong effervescence appears and CO_2 evolved.

$$CH_3COOH + Na_2CO_3 \rightarrow CH_3COONa + CO_2 \uparrow$$

2-Effect of $FeCl_3$:

In a test tube, add few drops of $FeCl_3$ solution to 0.3 ml of the neutral solution of acetic acid, a red color appears turns to brown precipitate on heating.

$$FeCl_3 \rightarrow (CH_3COO)_3Fe \xrightarrow{\Delta} (CH_3COO)\ Fe\ (OH)_2$$

3-Effect of Ammoniacal $AgNO_3$:

In a test tube, to 0.3 ml of the neutral solution of acetic acid add 0.3 ml of ammoniacal silver nitrate then heat on water bath. A white precipitate of silver nitrate is formed.

$$CH_3COONH_4 + AgNO_3 \rightarrow CH_3COOAg + NH_4NO_3$$

4-Effect of $KMnO_4$:

In a test tube, add 0.3 ml of the neutral solution of CH_3COOH to 0.3 ml $KMnO_4$ solution, then acidify by dilute H_2SO_4, the violet color does not discharge.

5- Esterification:

In a dry test tube, 0.4 ml acetic acid, 0.5 ml dry ethanol of and few drops of concentrated H_2SO_4 were heated to boiling with continuous shaking for 5 minutes. After cooling, the mixture is poured onto a solution of 10 ml of sodium bicarbonate in a beaker. Effervescence with characteristic odor of ethyl acetate was observed.

$$CH_3COOH + C_2H_5OH \xrightarrow{\text{conc. } H_2SO_4} CH_3COOC_2H_5 + H_2O$$

Laboratory Report

Physical Properties: -

Color:
Solubility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Acidity Test		
2	$FeCl_3$ Test		
3	$AgNO_3$ Test		
4	$KMnO_4$ Test		
5	Esterification		

(3) Oxalic acid

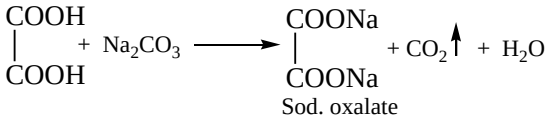
Physical Properties:-

Color: Colorless crystals
Solubility: Soluble in water
Effect on LP: Acidic (change blue LP to red)

Chemical Properties:

1- Acidity test:

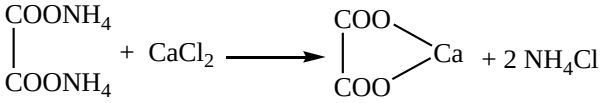
In a test tube take, dissolve few crystals of oxalic acid in water (0.5 ml), add sodium carbonate solution drop wisely, a strong effervescence appears and CO_2 evolved.



Sod. oxalate

2- Effect of $CaCl_2$:

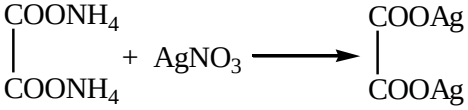
In a test tube, add 0.3 ml of $CaCl_2$ solution to 0.3 ml of the neutral solution of oxalic acid, a white precipitate of calcium oxalate is formed at once.



Calcium oxalate

3- Effect of Amm. $AgNO_3$:

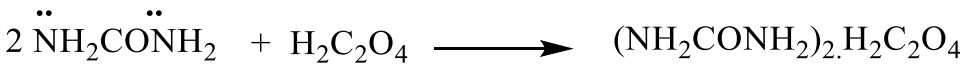
In a test tube add to 0.3 ml of neutral solution of oxalic acid 0.3 ml of ammoniacal silver nitrate, a white precipitate of silver oxalate is formed.



4- Salt formation:

Urea can be reacts as a weak base with acid to form its corresponding salts.

In a test tube, to a solution of 0.3 g of oxalic acid add a concentrated solution of urea, a whit precipitate of urea oxalate is formed.



5-Effect of $KMnO_4$

In a test tube, to 0.3 ml of N.S. of oxalic acid add a solution of $KMnO_4$ followed by few drops of dilute H_2SO_4, the violet color is decolorized on warming (at 60° C).

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Laboratory Report

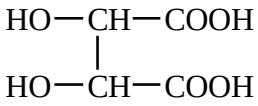
Physical Properties: -

Color:
Solubility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Acidity Test		
2	$CaCl_2$ Test		
3	$AgNO_3$ Test		
4	Urea Oxalate Salt		
5	$KMnO_4$ Test		

(4) Tartaric acid



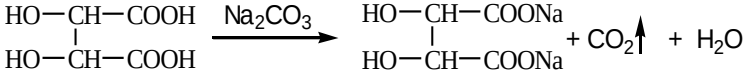
Physical Properties:-

Color: Colorless crystals
Solubility: Soluble in water
Effect on LP: Acidic (change blue LP to red)

Chemical Properties:

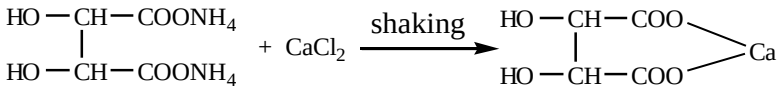
1- Acidity test:

In a test tube, take one crystal of tartaric acid and dissolve it in 0.5 ml water followed by addition of sodium carbonate solution, a strong effervescence appears and CO_2 evolved.



2- Effect of $CaCl_2$:

In a test tube add 0.3 ml of $CaCl_2$ solution to 0.3 ml of the neutral solution of tartaric acid a white precipitate of calcium tartarate is formed after shaking.



(3) Resorcinol test:

In a test tube, a mixture of 0.5 ml of N.S. of tartaric acid and 0.3 ml of resorcinol solution is warmed with drops of concentrated H_2SO_4, a red–brown ring is formed.

(4) Fenton's test

One drop of freshly prepared $FeSO_4$ solution is added to 2 ml of neutral solution of tartaric acid. Add dilute hydrogen peroxide solution dropwise until the solution changed to green color, then add an excess amount of NaOH solution. A violet color is formed

Laboratory Report

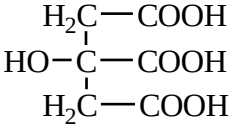
Physical Properties: -

Color:
Solubility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Acidity Test		
2	$CaCl_2$ Test		
3	Resorcinol test:		
4	Fenton's test		

(5) Citric acid



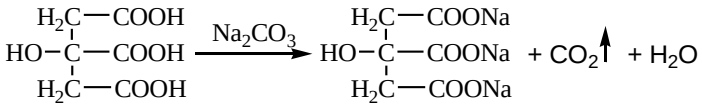
Physical Properties:-

Color: Colorless crystals
Solubility: Soluble in water
Effect on LP: Acidic (change blue LP to red)

Chemical Properties:

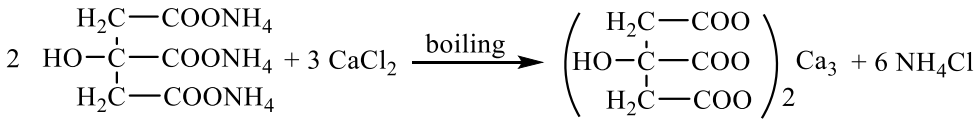
1- Acidity test:

In a test tube take one crystal of citric acid and dissolve it in water 0.5 ml followed by addition of sodium carbonate solution a strong effervescence appears and CO_2 evolved.

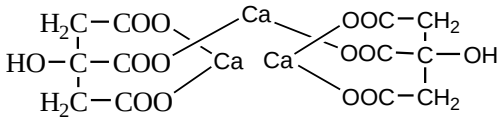


2- Effect of $CaCl_2$:

In a test tube, added 0.3 ml of $CaCl_2$ solution to 0.3 ml of the neutral solution of citric acid, a white precipitate of calcium citrate is formed after boiling.

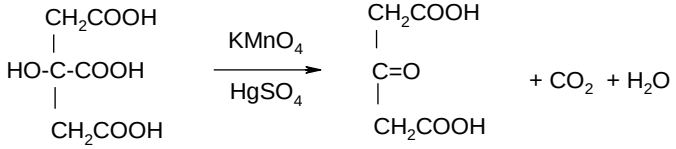


or



3- Deing's test:

In a clean test tube, 0.4 ml of the N.S. of citric acid is heated with 0.3 ml of Deing's (1) followed by addition of 0.3 ml of Deing's (2), the color discharged and a heavy white red precipitate is formed.



Acetone dicarboxylic acid

Laboratory Report

Physical Properties: -

Color:
Solubility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Acidity Test		
2	$CaCl_2$ Test		
3	Deing's test:		

V) Aromatic Carboxylic Acids

a) Chemicals required:-

Benzoic acid, Salicylic acid, Cinnamic acid, Phenylacetic acid, Phthalic acid, Sodium Carbonate, Ethanol, Ferric chloride, Ammonium hydroxide, conc. Sulfuric acid, Methanol, Resorcinol, Phenols, Sodium Hydroxide,

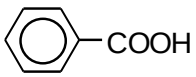
b) Apparatus:

Similar to aliphatic acids.

c) Neutral solution:

Similar to aliphatic acids.

(1) Benzoic acid



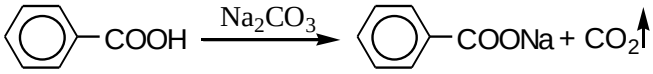
Physical Properties:-

Color: Colorless crystals
Solubility: Insoluble in cold water
Effect on LP: Acidic (change blue LP to red)

Chemical Properties:

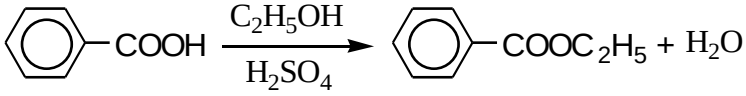
1- Acidity test:

In a test tube, take few crystals of benzoic acid in 0.5 water ml followed by addition of sodium carbonate solution, an effervescence appears and CO_2 evolved.



2- Esterification:

In a dry test tube, 0.2 g benzoic acid, 1 ml of ethanol and few drops of concentrated H_2SO_4 were heated to boiling with continuous shaking for 5 minutes. After cooling, the mixture is poured onto a solution of 10 ml of sodium bicarbonate. Effervescence with characteristic odor of ethyl benzoate is observed.

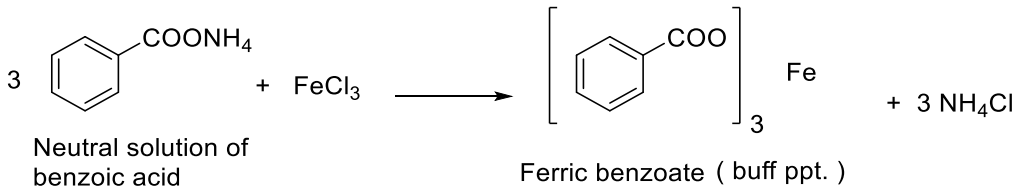


3- Sublimation test:

In a dry test tube, 0.2 gm of benzoic acid and two drops of conc. H_2SO_4 is heated on a hot plate, long needles crystals are formed on the above wall of the tube.

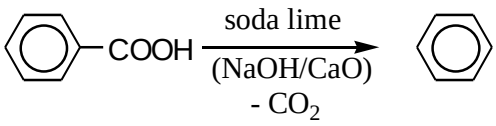
4- Effect of Fecl$_3$:

To a test tube contains 0.3 ml of the neutral solution of benzoic acid add few drops of $FeCl_3$ solution, a buff precipitate is formed.



5- Effect of soda lime:

In a dry test tube, 0.2 gm of benzoic acid is heated with 0.3 gm of soda lime (NaOH/CaO) to give benzene odor.



Laboratory report

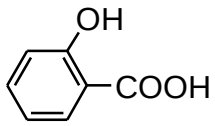
Physical Properties: -

Color:
Solubility:
Effect on LP:

Chemical Properties: -

No.	Experiment	Observation	Results
1	Acidity Test		
2	Esterification		
3	$FeCl_3$ Test		

(2) Salicylic acid



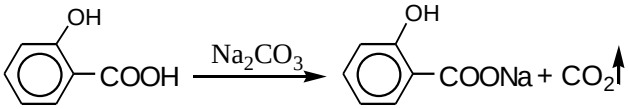
Physical Properties:-

Color: Colorless crystals
Solubility: Partially soluble in water
Effect on LP: Acidic (change blue LP to red)
Due to the presence of the phenolic OH group in salicylic acid, it gives the reactions of phenols in addition to those of acids.

Chemical Properties:

1- Acidity test:

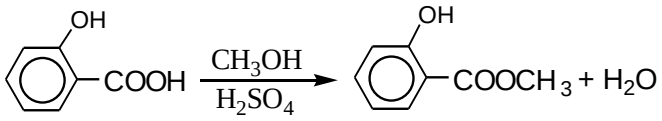
In a test tube, take few crystals of salicylic acid in 1m of water followed by addition of sodium carbonate solution, a weak effervescence appears and CO_2 evolved.



2- Esterification:

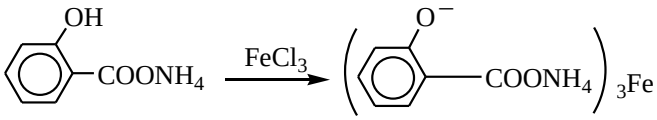
In a dry test tube, 0.2 g salicylic acid, 1 ml of methanol and few drops of concentrated H_2SO_4 were heated to boiling with continuous shaking for 5 minutes. After cooling, the mixture is poured onto a solution of 10 ml of sodium

bicarbonate in a beaker. Effervescence with characteristic odor of methyl salicylate is observed (winter green oil).



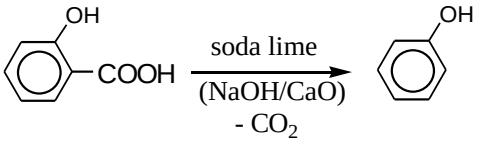
3- Effect of $FeCl_3$:

To a test tube contains 0.3 ml of the neutral solution of salicylic acid added few drops of $FeCl_3$ solution, a deep violet color appears.



5- Effect of soda lime:

In a dry test tube, 0.2 gm of salicylic acid is heated with 0.3 gm of soda lime (NaOH/CaO) to give phenol odor.



Laboratory report

Physical Properties: -

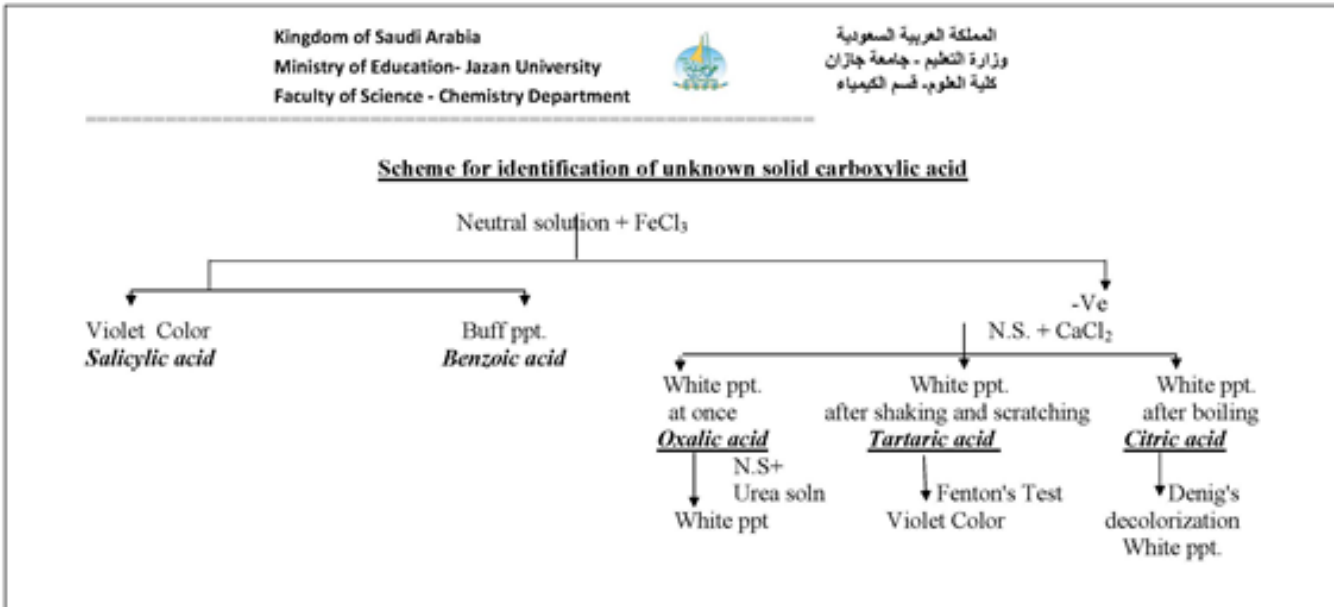
Color:

Solubility:

Effect on LP:

Chemical Properties: -

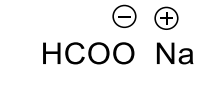
No.	Experiment	Observation	Results
1	Acidity Test		
2	Esterification Test		
3	$FeCl_3$ Test		



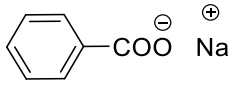
Salts of Carboxylic Acids

Salts of carboxylic acids are solid organic compounds that soluble in water. They are classified into:

1- **Metallic salts**, such as:

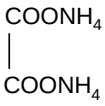


Sodium formate

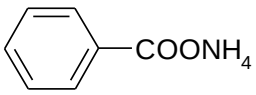


Sodium benzoate

2- **Ammonium salts**, such as:



Ammonium oxalate



Ammonium benzoate

General reactions of carboxylic acids salts

Salts of acids give all characteristic reactions of the corresponding acids without preparing neutral solution while they cannot react with sodium carbonate (Acidity test).

Differentiation between ammonium and metallic salts of carboxylic acid:

Solid salt + NaOH (20%) or rubbing with solid Na_2CO_3:

- If ammonia evolved on cold *Ammonium salt*
- If no ammonia evolved *Metallic salt*

Scheme for identification of unknown salts of carboxylic acids

a) Solid unknown + NaOH

Odor of ammonia — ***Ammonium salt***

- Ve — ***Metallic salt***

b) Solution of unknown + HCl

White ppt — ***Aromatic salt of acid***

- Ve — ***Aliphatic salt of acid***

If compound is salt of aliphatic acid:

1 ml of salt solution in water + $FeCl_3$ solution

Red Color turn to brown ppt. by boiling — ***Acetate or Formate***

Salt + Na_2CO_3 + $KMnO_4$

Decolorization — ***formate***

-ve — ***Acetate***

– Ve

solution in water + $CaCl_2$

White ppt — at once — ***oxalate***

white ppt — after scratching — ***Tartarate***

white ppt — after boiling — ***citrate***

***If compound is salt of aromatic acid:**

1 ml of solution of substance in water + $FeCl_3$ solution

Violet Color — ***Salicylate***

Buff ppt. — ***Benzoate***

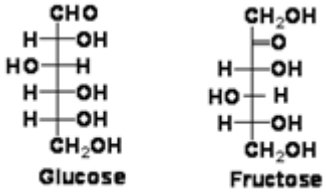
Carbohydrates

Carbohydrates are the most abundant class of organic compounds found in living organisms. They originate as products of **photosynthesis**, an endothermic reductive condensation of carbon dioxide requiring light energy and the pigment chlorophyll.

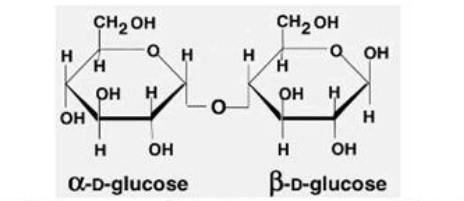
$$n\ CO_2 + n\ H_2O + \text{energy} \longrightarrow C_nH_{2n}O_n + n\ O_2$$

a) Chemical required:

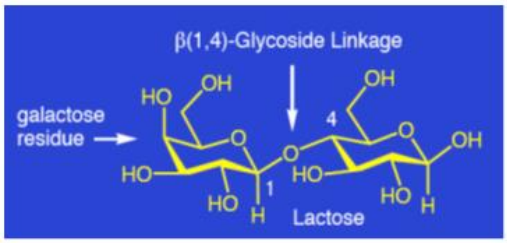
i) Monosaccharaides: Aldoses (glucose) and ketoses (fructose).



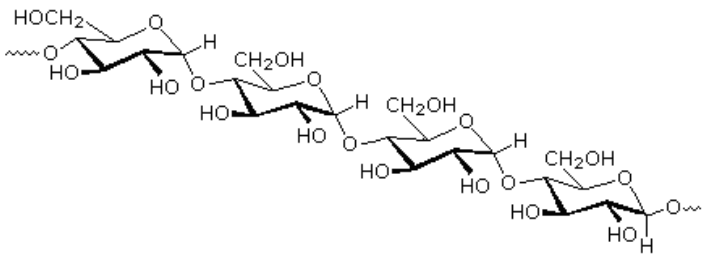
ii) Disaccharides: Maltose, Lactose



Maltose



iii) Polysaccharides: **Starch**



Equipment and reagents:

Test tubes, Racks of test tubes, test tubes holders, clamps, Bunsen flames and beakers.

Fehling's solution A: Cu_2SO_4

Fehling's solution B: KOH and Sodium Potassium Tartarate (Rochelle salt)

Fehling's reagent: Equal volumes of Fehling A and Fehling B are mixed to form a deep blue solution.

Barfoed's reagent (a solution of cupric acetate $Cu(CH_3COO)_2$ and acetic acid).

Benedict's solution ($CuSO_4$ and sod.citrate).

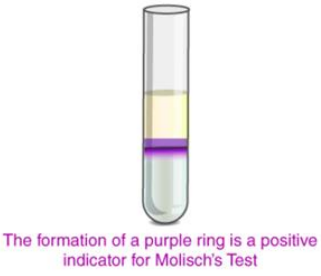
Tollen's Reagent: Ammoniacal silver nitrate solution (Ag_2O / NH_4OH).

Silwanoff's reagent [0.05 gm of resorcinol in 100 ml HCl (4 N)]:

c) Physical Properties:

Color:
Shape:
Solubility:
Effect on LP:

d) Chemical Properties:

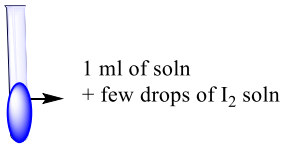

The formation of a purple ring is a positive indicator for Molisch's Test

Molisch's test:

In a test tube, place 0.5 ml of the carbohydrate solution and 0.5 ml of alcoholic α-naphthol followed by addition of few drops of concentrated H_2SO_4 on the wall of the test tube. A deep violet ring appeared and spreads in the solution by shaking: **This test is positive with all carbohydrates.**

To identify the type of carbohydrate you have to carry out the following experiments.

1- Iodine test:



To the solution of the carbohydrate add few drops of iodine solution → Blue color ⇒ The carbohydrate is starch.

2- Barfoed's Test.

One ml of solution of the carbohydrate is placed in a test tube. Three ml of Barfoed's is added. Then, the solution is heated in a boiling water bath for three minutes.

Red precipitate of Cu_2O. ⇒ The carbohydrate is glucose, fructose

→ No precipitate ⇒ The carbohydrate lactose, Maltose or Sucrose.

3- Fehling's test:

Solution of carbohydrate + 0.5 ml of Fehling A + 0.5 ml of Fehling B and heat on water bath for 5–10 minutes:

→ Red precipitate of Cu_2O. ⇒ The carbohydrate is glucose, fructose, lactose or Maltose.

→ No precipitate ⇒ The carbohydrate is sucrose

form a deep blue solution.

4-Benedict's Test:

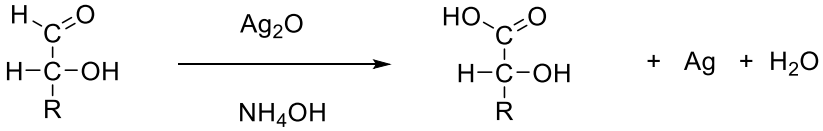
Place 1 ml of carbohydrate solution in a clean test tube. Add 1 ml of Benedict's solution, shake the tube and then set it aside in the rack. Describe and interpret the result as:

→ White precipitate ⇒ The carbohydrate is glucose, fructose, lactose or Maltose.

→ No precipitate ⇒ The carbohydrate is sucrose.

5-Tollen's Test

Reducing sugars (glucose, fructose, maltose, lactose) reduce ammoniacal silver nitrate solution (Tollen's Reagent: Ag_2O / NH_4OH) to silver mirror.



Add 5 drops of Tollen's reagent to sugar solution. Warm the mixture on boiling water bath.

→ silver mirror ⇒ The carbohydrate is glucose, fructose, lactose or Maltose.

→ No silver mirror ⇒ The carbohydrate is sucrose

6-Furfural test:

Take 2 ml of sugar solution in a test tube and, 1 ml of conc. HCl and then boil on direct flame. Expose a wetted paper with aniline acetate to the vapors evolved:

→ The paper changed to red ⇒ The sugar is fructose.

The color of the paper does not change ⇒ The sugar is glucose.

7- Rapid Furfural Test.

In a test tube, place 1ml of the carbohydrate solution, add 1ml of alcoholic α-naphthol, and then add 5 ml of conc. HCl. place the test tube in a warm water-bath for

→ The violet color appears immediately ⇒ The sugar is fructose.

→ The violet color appears after time ⇒ The sugar is glucose

8- Silwanoff test:

To the sugar solution add Silwanoff's reagent [0.05 gm of resorcinol in 100 ml HCl (4 N)]:

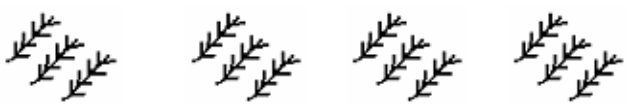
→ A red color ⇒ The sugar is fructose.

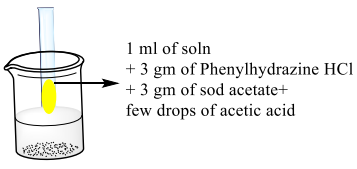
→ No a red color ⇒ The sugar is glucose.

5- Osazone formation:

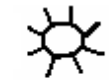
In a test tube, take 0.5 ml of concentrated solution of the carbohydrate, 0.5 gm of phenylhydrazine hydrochloride (in 1:3 ratio) and 0.5 gm of sodium acetate then heat the mixture on water bath for 10 minutes and observe the following:

→ Yellow precipitate formed on hot ⇒ Glucose or fructose. Examine the precipitate using microscope to see the crystals shape.





→ Yellow precipitate formed after cooling ⇒ maltose or lactose. Examine the crystals using the microscope

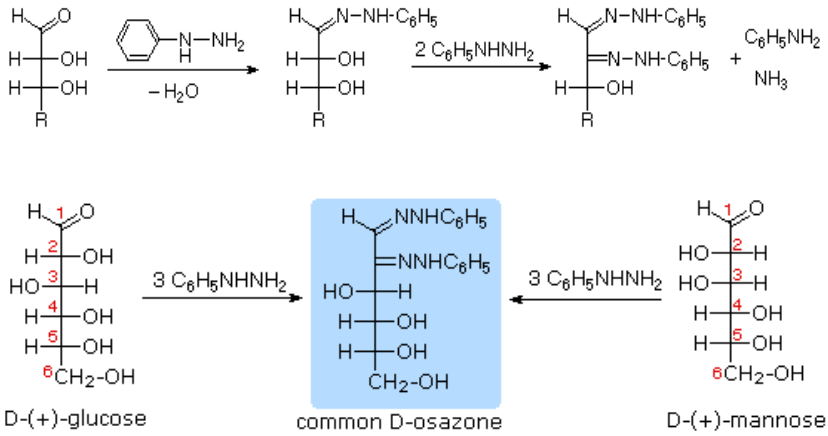


Lactose

Maltose

→ No precipitate formed ⇒ The carbohydrate is non reducing sugar (Sucrose).

Osazone Formation



To differentiate between glucose and fructose carry out the following experiments:

==========

Laboratory Report

Physical Properties:-

Color:

Solubility:

Effect on LP:

Chemical Properties:-

No.	Experiment	Observation	Results
1			
2			
3			
4			
5			
6			

Physical Properties:-

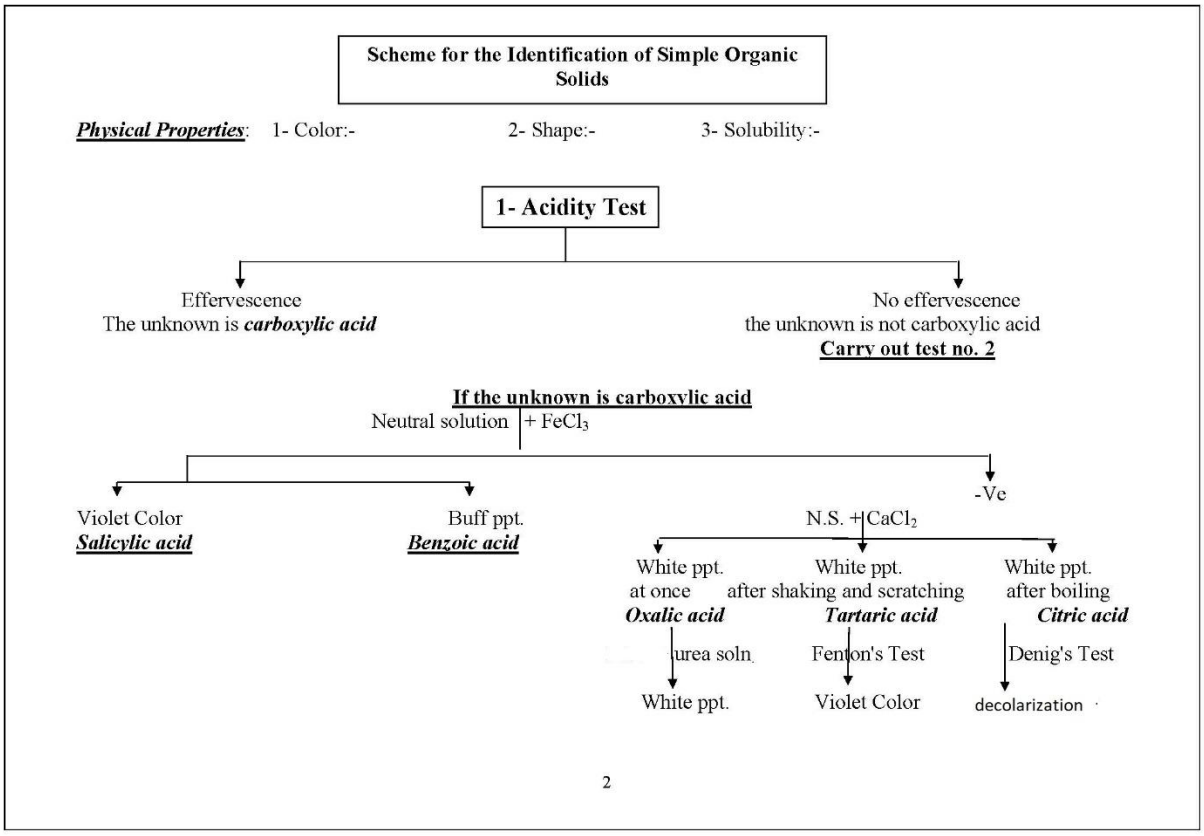
Color:

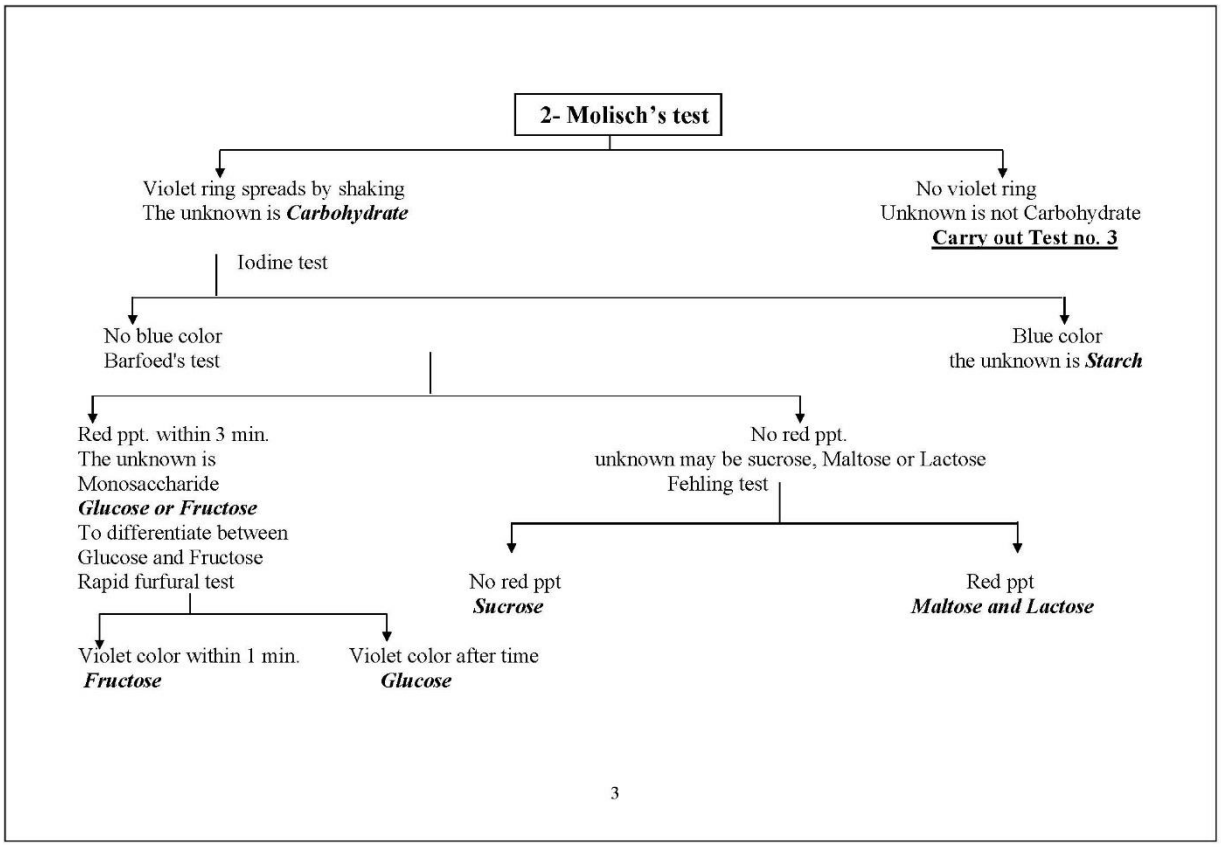
Solubility:

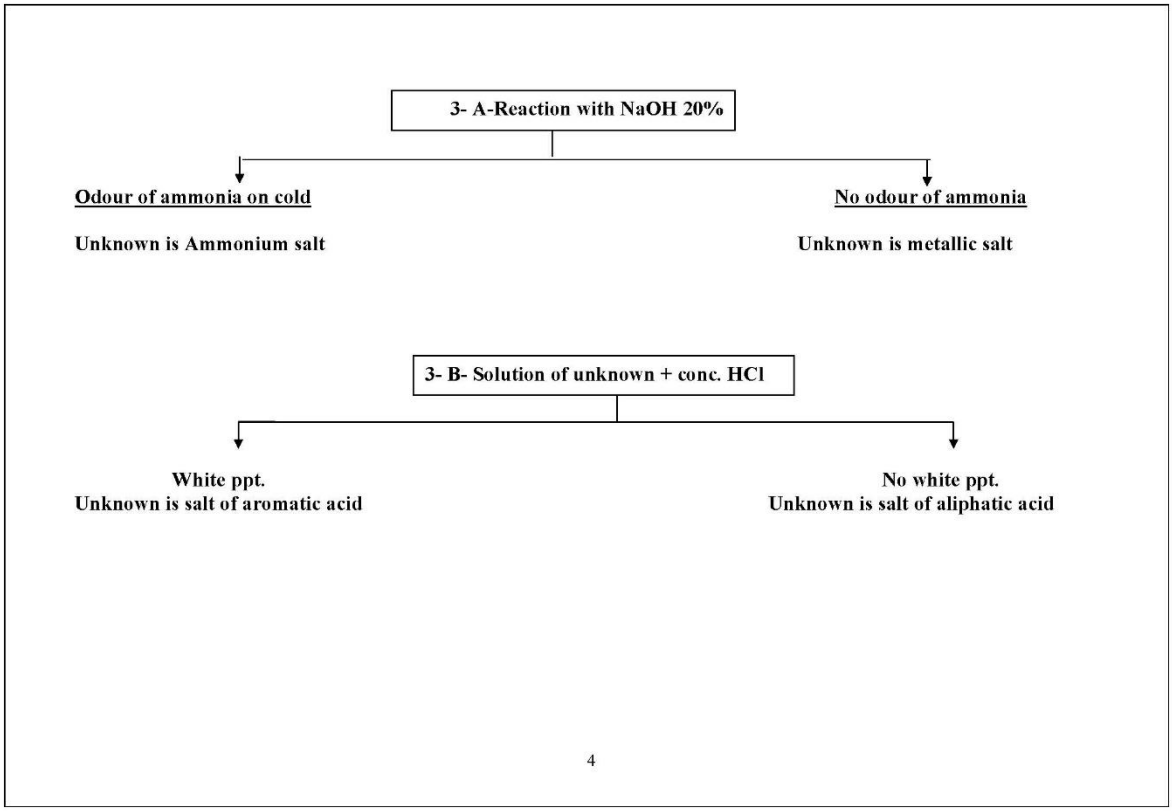
Effect on LP:

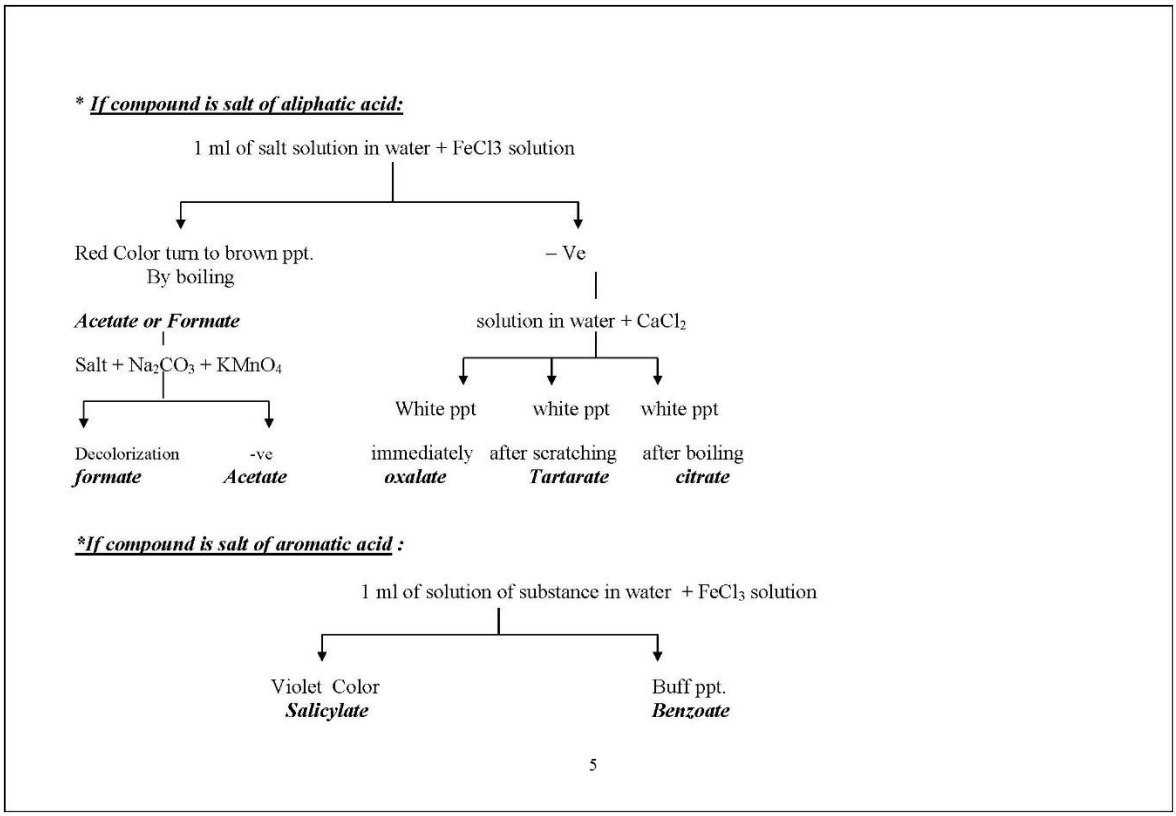
Chemical Properties:-

No.	Experiment	Observation	Results
1			
2			
3			
4			
5			
6			


Scheme for the Identification of Simple Organic Solids
Physical Properties: 1- Color:- 2- Shape:- 3- Solubility:-
1- Acidity Test
Effervescence
The unknown is carboxylic acid
No effervescence
the unknown is not carboxylic acid
Carry out test no. 2
If the unknown is carboxylic acid
Neutral solution + FeCl3
Violet Color
Salicylic acid
Buff ppt.
Benzoic acid
-Ve
N.S. + CaCl2
White ppt. at once
Oxalic acid
White ppt. after shaking and scratching
Tartaric acid
White ppt. after boiling
Citric acid
urea soln.
Fenton's Test
Denig's Test
White ppt.
Violet Color
decolarization
2


2- Molisch's test
Violet ring spreads by shaking
The unknown is Carbohydrate
No violet ring
Unknown is not Carbohydrate
Carry out Test no. 3
Iodine test
No blue color
Barfoed's test
Blue color
the unknown is Starch
Red ppt. within 3 min.
The unknown is
Monosaccharide
Glucose or Fructose
To differentiate between
Glucose and Fructose
Rapid furfural test
No red ppt.
unknown may be sucrose, Maltose or Lactose
Fehling test
No red ppt
Sucrose
Red ppt
Maltose and Lactose
Violet color within 1 min.
Fructose
Violet color after time
Glucose
3


3- A-Reaction with NaOH 20%
Odour of ammonia on cold
Unknown is Ammonium salt
No odour of ammonia
Unknown is metallic salt
3- B- Solution of unknown + conc. HCl
White ppt.
Unknown is salt of aromatic acid
No white ppt.
Unknown is salt of aliphatic acid
4


* If compound is salt of aliphatic acid:
1 ml of salt solution in water + FeCl3 solution
Red Color turn to brown ppt.
By boiling
– Ve
Acetate or Formate
solution in water + CaCl2
Salt + Na2CO3 + KMnO4
White ppt
white ppt
white ppt
Decolorization
formate
-ve
Acetate
immediately
oxalate
after scratching
Tartarate
after boiling
citrate
*If compound is salt of aromatic acid :
1 ml of solution of substance in water + FeCl3 solution
Violet Color
Salicylate
Buff ppt.
Benzoate
5