Chemistry chart

STD XII Science
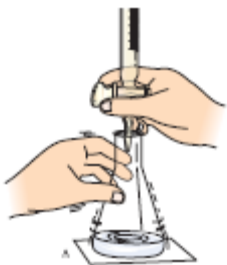
Name_____

Class_____

Div_____

Roll No_____

Batch No_____

VOLUMETRIC ANALYSIS

REDOX TITRATIONS

Experiment No:- 1   Date:--------------

Aim:- You are provided with two solutions as follows.

Container A: -----------------**M/N** Stock Solution of Hydrated Mohr's Salt.

Container B: - **KMnO$_4$** Solution.

Using the Stock Solution from container **A** Prepare 100 ml of _____
M/N Solution of Mohr's Salt in the given standard measuring flask **C.**

Using the solution in flask C determine the

1. Molarity/Normality of the solution in container B.
2. Strength of less /more concentrated solution in container B/C in terms of gms/_____ ml.
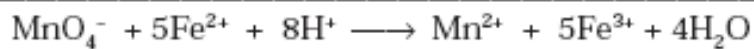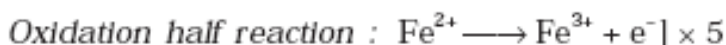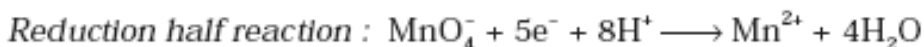3. % Purity of Solution in Container B (-----Gms of which have been dissolved per-------ml)

Apparatus & Chemicals Required:-

Burette, Pipette conical flask Burette stand white tile, KMnO$_4$ Solution , Hydrated Mohr's Salt and Dil. sulphuric acid

Theory:-

This is a Redox titration where Neutralization point is due to Reduction and Oxidation reaction occurring simultaneously. In this titration - **KMnO$_4$** undergoes reduction with change in oxidation number of manganese ion from +7 to +2 and Mohr's Salt undergoes oxidation with change in oxidation number of iron+2(Ferrous) to +3(Ferric) Thus acts as a - **KMnO$_4$** Oxidizing agent and Mohr's Salt acts as reducing agent and at the end point colour change is from colourless to light pink

(b) Ionic equation

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

Oxidation half reaction : $Fe^{2+} \longrightarrow Fe^{3+} + e^-] \times 5$

$$MnO_4^- + 5Fe^{2+} + 8H^+ \longrightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$$

Procedure:

1. Rinse the pipette with the _____ N/M Mohr's Salt solution and pipette out 10 ml of it in a washed titration flask.
2. Rinse and fill the burette with the given $KMnO_4$ solution.
3. Add one test-tube (15 ml) full of dilute sulphuric acid (2 N) to the solution in the titration flask.
4. Note the initial reading of the burette.
5. Add $KMnO_4$ solution from the burette till a permanent light pink colour is imparted to the solution in the titration flask on addition of the last single drop of $KMnO_4$ solution.
6. Note the final reading of the burette.
7. Repeat the above steps to get three concordant readings.

Result:

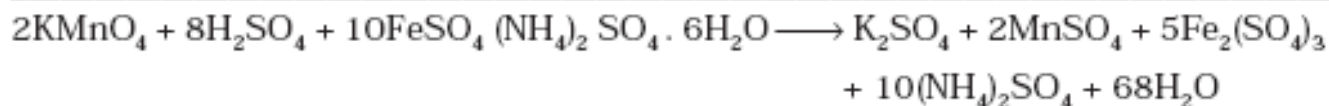| Solution	Normality	Molarity	Strength	% Purity
Mohr's Salt	---------N	..... M	..... gms	.....
$KMnO_4$	---------N	..... M	..... gms	

_____Solution is Less/More Concentrated in terms of Normality/Molarity/gms per ------ml

1. Given:-
2. To prepare:
3. To find:

Observations:

1. Solution in Burette : $KMnO_4$ solution.
2. Pipette Solution: Mohr's Salt Solution
3. Solution in Flask: 10 ml of ------ N /M Mohr's Salt+ 1test tube di!. H_2SO_4,
4. Indicator: $KMnO_4$ acts as self indicator.
5. End point: Colourless to light pink.

Chemical equations:

$$2KMnO_4 + 8H_2SO_4 + 10FeSO_4 (NH_4)_2 SO_4 . 6H_2O \longrightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3$$
$$+ 10(NH_4)_2SO_4 + 68H_2O$$

Observation Table:

Pilot Reading_____ml To _____ml.

Burette Reading	I	II	III	Constant Burette Reading (C.B.R.)
Final	... mL	.... mL	..... mL	
Initial	OmL	OmL	OmL	..... mL
Difference	.... mL	..... mL	..... mL	

Calculations:

1. To calculate volume required to prepare 100 ml of --------------N/M_____Solution

   Solution to be prepared=Stock Solution

   $$N_1V_1=N_2V_2$$

2. To calculate Normality of : $KMnO_4$ solution = $N_1V_1=N_2V_2$
3. To calculate Grams per -----ml   =N X Eq.Wt X ----------/1000
4. To calculate % Purity

   Gms/ml of pure $KMnO_4$ solution **(calculated)**
   % Purity=--   X   100
   Gms/ml of Impure $KMnO_4$ solution **(Given)**

REDOX TITRATIONS

Experiment No:- 2   Date:--------------

Aim:- You are provided with two solutions as follows.

Container A: -----------------**M/N** Stock Solution of Hydrated Oxalic Acid

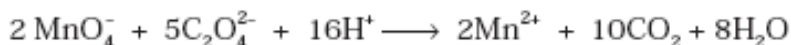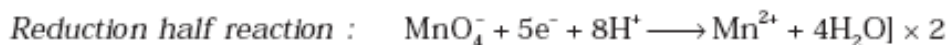**Container B:** - **KMnO$_4$** Solution.

Using the Stock Solution from container **A** Prepare 100 ml of _____ M/N Solution of Hydrated Oxalic Acid in the given standard measuring flask **C.**

Using the solution in flask C determine the

1. Molarity/Normality of the solution in container B.
2. Strength of less /more concentrated solution in container B/C in terms of gms/_____ ml.
3. % Purity of Solution in Container B (-----Gms of which have been dissolved per-------ml)

Apparatus & Chemicals Required:-

Burette, Pipette conical flask Burette stand white tile, KMnO$_4$ Solution , Hydrated Oxalic acid and Dil. sulphuric acid

Theory:- This is a Redox titration where Neutralization point is due to Reduction and Oxidation reaction occurring simultaneously. In this titration - **KMnO$_4$** undergoes reduction with change in oxidation number of manganese ion from +7 to +2 and Oxalic acid undergoes oxidation with change in oxidation number of carbon from +3to +4 Thus- **KMnO$_4$** acts as a oxidizing agent and Oxalic acid acts as reducing agent and at the end point colour change is from colourless to light pink

B. Ionic equation

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

Oxidation half reaction : $C_2O_4^- \longrightarrow 2CO_2 + 2e^-] \times 5$

_____

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

_____

In these equations, MnO_4^- is reduced to Mn^{2+} and $C_2O_4^{2-}$ is oxidised to CO_2. The oxidation number of carbon in $C_2O_4^{2-}$ changes from +3 to +4.

Procedure:

1. Rinse the pipette with the _____ N/M oxalic acid solution and pipette out 10 ml of it in a washed titration flask.
2. Rinse and fill the burette with the given $KMnO_4$ solution.
3. Add one test-tube (15 ml) full of dilute sulphuric acid (2 N) to the solution in the titration flask.
4. Note the initial reading of the burette.
5. Heat the flask to 60 -70 °C and add $KMnO_4$ solution from the burette till a permanent light pink colour is imparted to the solution in the titration flask on addition of the last single drop of $KMnO_4$ solution.
6. Note the final reading of the burette.
7. Repeat the above steps to get three concordant readings.

Result:

Solution	Normality	Molarity	Strength	% Purity
Oxalic acid	---------N	..... M	..... gms	.....
KMnO₄	---------N	..... M	..... gms	

_____Solution is Less/More Concentrated in terms of Normality/Molarity/grams per ml

1. Given:-
2. To prepare:
3. To find:

Observations:

1. Solution in Burette : $KMnO_4$ solution.
2. Pipette Solution: Oxalic acid
3. Solution in Flask: 10 ml of ------ N /M Oxalic acid + 1test tube di!. H_2SO_4, & Heat.
4. Indicator: $KMnO_4$ acts as self indicator.
5. End point: Colourless to light pink.

Chemical equations:

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

Observation Table:

Pilot reading_____ml To _____ml.

Burette Reading	I	II	III	Constant Burette Reading (C.B.R.)
Final	... mL	.... mL	..... mL	
Initial	OmL	OmL	OmL	..... mL
Difference	.... mL	..... mL	..... mL	

Calculations:

5. To calculate volume required to prepare 100 ml of --------------N/M_____Solution

   Solution to be prepared=Stock Solution

   N1V1=N2V2

6. To calculate Normality of : $KMnO_4$ solution

   N1V1=N2V2

7. To calculate Grams per -----ml   =N X Eq.Wt X ----------/1000
8. To calculate % Purity

   Gms/ml of pure $KMnO_4$ solution **(calculated)**
   % Purity=-- X 100
   Gms/ml of Impure $KMnO_4$ solution **(Given)**

Note:

1. To calculate Molarity of : $KMnO_4$ solution 1M=5N

(To convert from Normality to Molarity divide Normality by five & To convert from Molarity to Normality Multiply Molarity by five.)

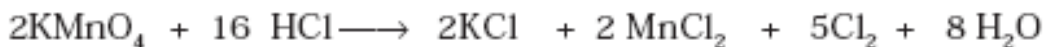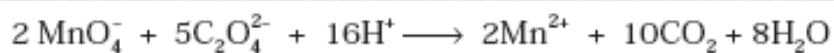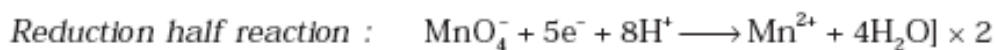2. To calculate Molarity of : Oxalic Acid solution 1M=2N

(To convert from Normality to Molarity divide Normality by two & To convert from Molarity to Normality Multiply Molarity by two.)

3. To calculate Molarity of : F.A.S solution 1M=1N ie Molarity=Normality

Additional Information:

The acid used in this titration is dilute sulphuric acid. Nitric acid is not used as it is itself an oxidising agent and hydrochloric acid is usually avoided because it reacts with $KMnO_4$ according to the equation given below to produce chlorine and chlorine which is also an oxidising agent in the aqueous solution.
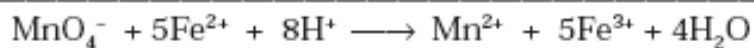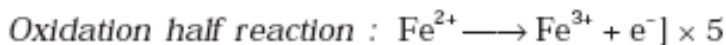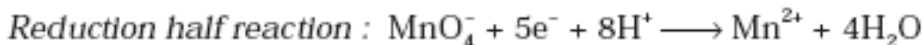
$$2KMnO_4 + 16\ HCl \longrightarrow 2KCl + 2\ MnCl_2 + 5Cl_2 + 8\ H_2O$$

In the titration of KMnO$_4$ V/S Oxalic acid

B. Ionic equation

Reduction half reaction : $\qquad MnO_4^- + 5e^- + 8H^+ \longrightarrow Mn^{2+} + 4H_2O] \times 2$

Oxidation half reaction : $\qquad C_2O_4^- \longrightarrow 2CO_2 + 2e^-] \times 5$

_____

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

_____

In these equations, MnO_4^- is reduced to Mn^{2+} and $C_2O_4^{2-}$ is oxidised to CO_2. The oxidation number of carbon in $C_2O_4^{2-}$ changes from +3 to +4.

- During the titration of oxalic acid against potassium permanganate, warming of oxalic acid solution (50°–60°C) along with dilute H_2SO_4 is required. This is essential because the reaction takes place at higher temperature. During the titration, first manganese sulphate is formed which acts as a catalyst for the reduction of $KMnO_4$ by oxalic acid. Therefore, in the beginning the reaction rate is slow and as the reaction proceeds, the rate of the reaction increases.

- **In the titration of KMn0$_4$ V/S Mohr's Salt**

(b) Ionic equation

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

Oxidation half reaction : $Fe^{2+} \longrightarrow Fe^{3+} + e^-] \times 5$

_____

$MnO_4^- + 5Fe^{2+} + 8H^+ \longrightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

_____

- The oxidation number of iron in Mohr's salt is +2. Iron is oxidised during the reaction and its oxidation number changes from +2 to +3.

- In the titration of KMnO$_4$ V/S ferrous ammonium sulphate heating of ferrous ammonium sulphate solution is not required because reaction rate is very high even at room temperature. Also, at high temperatures, ferrous ions may be oxidised to ferric ions by oxygen of air and error may be introduced in the experiment.

Precautions
(a) Always rinse the burette and the pipette with the solutions to be taken in them.
(b) Never rinse the conical flask with the experimental solutions.
(c) Remove the air gaps if any, from the burette.
(d) Never forget to remove the funnel from the burette before noting the initial reading of the burette.
(e) No drop of the liquid should hang at the tip of the burette at the end point and while noting reading.
(f) Always read the upper meniscus for recording the burette reading in the case of all coloured solutions.
(g) Never use pipette and burette with a broken nozzle.
(h) Lower end of the pipette should always remain dipped in the liquid while sucking the liquid.
(i) Do not blow out the last drop of the solution from the jet end of the pipette.
(j) The strength of the solution must be calculated up to the fourth decimal place.
(k) Do not forget to heat the mixture of oxalic acid and H$_2$SO$_4$ solutions between 50°–60° C while titrating it against potassium permanganate.

Discussion Questions???

(i) What specific name is given to the permanganate titrations?
(ii) Which indicator is used in the permanganate titration?
(iii) Why is a burette with pinch-cock regulator not used for the permanganate titration ?
(iv) Why do we heat oxalic acid solution containing sulphuric acid up to 50–60°C in the permanganate titration?
****

PHYSICAL EXPERIMENTS

EXPERIMENT NO. 1

Aim: To prepare lyophilic sol of starch.

Apparatus: 250-ml beaker, funnel, glass rod, filter paper, tripod Stand, wire gauze, etc.

Materials required: Soluble starch and water.

Theory: Starch forms a hydrophilic sol when water is used as dispersion medium. The formation of sol is accelerated by heating. The starch sol is prepared by heating starch and water at about 100° C and is quite stable.

Procedure:

1. About 1 gm of starch is taken on a watch glass.
2. The paste of starch is prepared and transferred to a 250-ml beaker.
3. Water (125ml) taken in a beaker is heated so that it starts boiling.
4. The paste is slowly poured with stirring into this boiling water in the beaker.
5. Boiling is continued for about 2-3 minutes and the beaker is allowed to cool.
6. The contents of the beaker are filtered through a filter paper in the funnel.

Precautions:

1. The apparatus used for preparing sol should be cleaned properly.
2. Starch should be converted into a fine paste before adding to boiling water.
3. It is necessary to constantly stir the contents during preparation of sol.

RESULT: The colloidal sol of starch is prepared from the given starch powder.

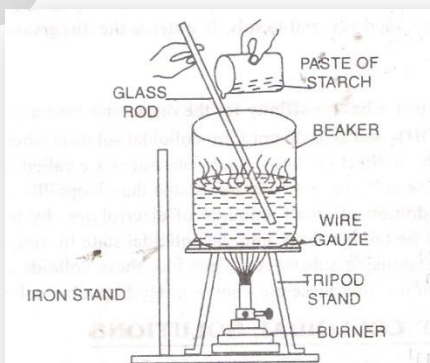*(On L.H.S of Journal)*

Diagram:

Fig. 3.1. Preparation of starch sol.

EXPERIMENT NO. 2

Aim: To determine the enthalpy of Neutralization of Hydrochloric acid with Sodium hydroxide solution.

Apparatus: a wide mouth polythene bottle, a rubber cork having two holes, thermometer

$(1 / 10^{th}$ degree), stirrer, 100ml measuring cylinder, etc.

Materials required: 0.5M Hydrochloric acid, 0.5M Sodium hydroxide solution.

Theory: Heat is evolved during neutralization of an acid with an alkali. Enthalpy of

neutralization is heat evolved when one gram equivalent of the acid is

neutralized completely by a base in dilute solution. Enthalpy of neutralization is

calculated by mixing known volume or standard solutions of acid and alkali

and observing change in temperature.

Procedure:

1. After washing rinse the measuring cylinder with 0.5M NaOH solution.
2. Measure exactly 50ml of 0.5M NaOH in measuring cylinder and transfer it to the clean polythene bottle.
3. Note down the initial temperature of the alkali in the polythene bottle.
4. After washing, rinse the measuring cylinder with 0.5M HCl.
5. Measure exactly 50ml of 0.5M HCl and transfer it to a clean and dry 250ml beaker.
6. Note down the initial temperature of HCl.
7. Add all the HCl solution to the polythene bottle and shake the mixture well.
8. Note down immediately the maximum temperature reached.

Observations:

1. The initial temperature of NaOH t_1=_____^{o}C
2. The initial temperature of HCl t_2= _____^{o}C
3. The final temperature of the mixture t_3= _____^{o}C

Calculations:

1. Heat evolved by neutralization = Heat gained by NaOH + Heat gained by HCl+ heat gained by Polythene bottle.

Heat gained by NaOH =Volume of NaOH*Sp Heat of NaOH Soln x $(t_3$-t_1)

Heat gained by HCl =Volume of HCl *Sp Heat of HCl Soln x $(t_3$-t_2) + W x $(t_3$-t_2)

Heat gained by Polythene bottle = W x $(t_3$-t_2)

W= heat gained by polythene bottle

Heat evolved by neutralization = 50 x 1x (t_3-t_1) + 50 x 1 x (t_3-t_2) +W x (t_3-t_2)

$$= 50 [(t_3-t_1) + (t_3-t_2) + 0]$$

(Heat gained by polythene bottle is assumed to be zero) ie W=0)

$$= Q \text{ cals.}$$

Note: Sp Heat of NaOH Soln & Sp Heat of HCl Soln is taken as unity for simplification.

1. When 50ml of 0.5M HCl is neutralised, heat evolved is **Q** cals.
2. Therefore When 100ml of 0.5M HCl is neutralised, heat evolved is **2Q** cals.
3. Therefore When 100ml of 1M HCl is neutralised, heat evolved is **2** x **2** x **Q** cals.
4. Therefore When 1000ml of 1M HCl is neutralised, heat evolved is **10** x **2** x **2** x **Q** cals.

====**40 Q** Cals.

= 40Q/ 1000 Kcals.
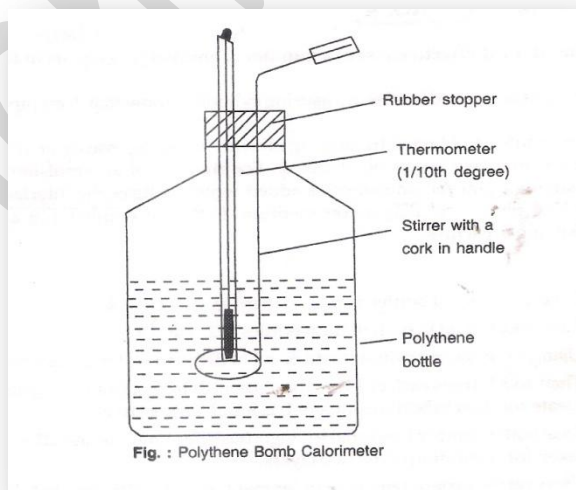
= 0.04Q Kcals.

= -ve ------------ Kcals.

Note   -ve sign because it is Exothermic Reaction.

    Result: The heat of neutralization of HCl and NaOH is found to be_--_____ Kcals.

Diagram :


Fig. : Polythene Bomb Calorimeter

EXPERIMENT- 3

Aim: - To study the effect of concentration on the rate of reaction between Sodium thiosulphate and hydrochloric acid.

Apparatus: - 10ml pipette, stop watch, two burettes and five 100ml conical flasks.

Materials required: - 0.1M $Na_2S_2O_3$ solution and 1M HCL solution.

Theory:- According to law of mass action, rate of a chemical reaction is directly proportional to product of the molar concentration of the reactants. In other words the rate of reaction increases with increase in the conc. of the reactants. The effect of concentration of reactants on rate of a reaction between sodium thiosulphate and hydrochloric acid.

$$Na_2S_2O_3 + 2HCl \rightarrow S_{(s)} + 2NaCl_{(aq)} + SO_{2(l)} + H_2O_{(l)}$$
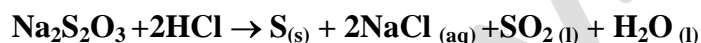
The insoluble sulphur formed during the reaction gives a milky appearance and makes the solution opaque. Therefore rate of reaction can be studied by measuring the time taken to produce enough sulphur to make some mark invisible on paper kept under the conical flask in which the reaction is carried out.

 Procedure: -

1) Wash the conical flask with water and label them as 1,2,3,4 and 5 respectively.

2) With the help of a Measuring cylinder add 10,20,30,40,50ml of 0.1M $Na_2S_2O_3$ solution to flask 1,2,3,4 and 5.

3) Now add 40,30,20 and 10ml of water to flask 1,2,3 and 4 respectively so that volume of solution in each flask is 50ml.

4) Take 10ml HCl in a test tube with the help of pipette.

5) Add 10ml of hydrochloric acid taken in test tube to conical flask no 1 containing 0.1 M $Na_2S_2O_3$ and 40 ml of water and start stop watch. Shake the contents of the conical flask and place it on the tile with a cross mark.

 6) Go on observing from top to downwards in the flask and note down the time from the stop watch when the cross mark just becomes invisible.

7) Repeat the above steps by adding 10ml of 1M HCl to flask 2, 3, 4, and 5 and record the time taken in each case for the cross to become just invisible.

Precautions: -

1) *The apparatus must be thoroughly clean. If the same conical flask is to be used again and again, it should be thoroughly washed with cone. HNO_3 and then with water.*
2) *Measures the volume of sodium thiosulphate solution hydrochloric acid and distilled water very accurately.*
3) *Use the same tile with the same cross mark for all observations.*
4) *Complete the experiment at one time only so that there is not much temperature variation.*
5) *Start the stopwatch immediately after adding HCl to sodium thiosulphate solution.*
6) *View the cross mark through the reaction mixture from top to bottom for all observation.*

Source of Error:- The conical flask may contain some solution left out due to which the time reading may change.

Result: - The graph between l/t v/s volume of $Na_2S_2O_3$ Or Concn of $Na_2S_2O_3$ is straight line. As concentration of $Na_2S_2O_3$ increases, the rate of the reaction increases.

Diagram:
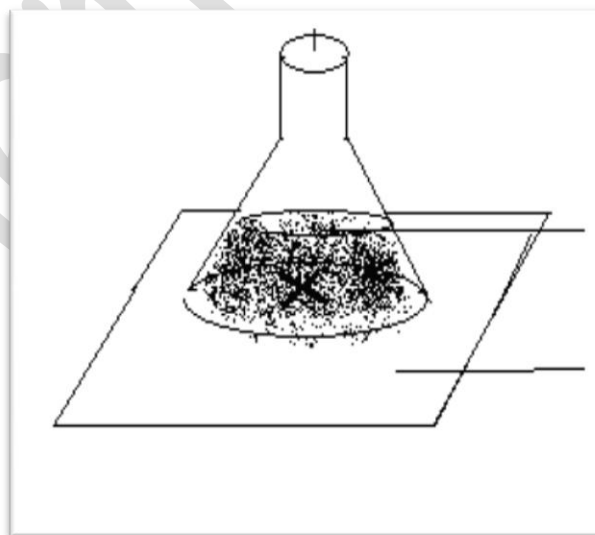
Observation Table:

Sr No	Amount of Na$_2$S$_2$O$_3$ in ml	Amount of water in ml	Initial Concentration of Na$_2$S$_2$O$_3$ in M	Time (t) in Sec	1/t Sec^{-1}
1					
2					
3					
4					
5					

Graph: Plot a Graph of l/t v/s volume of Na$_2$S$_2$O$_3$ or Conc. of Na$_2$S$_2$O$_3$
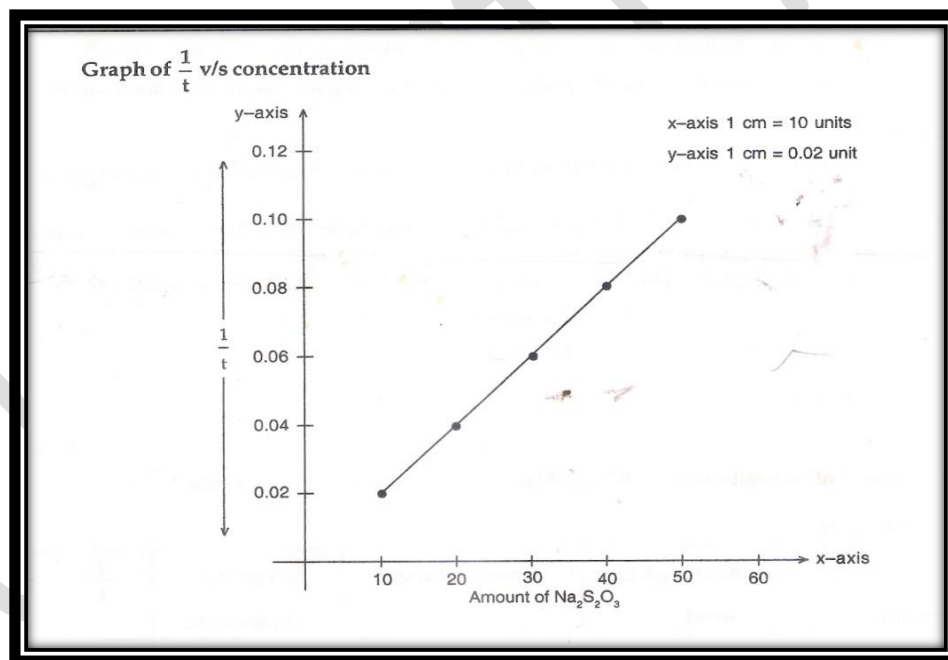
EXPERIMENT NO. 4

Role of emulsifying agent

Aim
To study the role of emulsifying agents in stabilizing the emulsions of different oils.

Apparatus Required
• Test tubes : Six • Small measuring cylinders (5 ml) : Five • Test tube stand : One • Glass rod : One • Stop watch : One

Material Required
1) 1% Soap solution   2) Mustard oil, groundnut oil, coconut oil & Palmolive oil .

Theory
Emulsion is a type of colloid in which, both the dispersed phase and the dispersion medium are liquids. Here the dispersed phase and the dispersion medium are distinguished by their relative amounts. The one, which is present in smaller proportion, is called **dispersed phase**, while the other, which is present in relatively large quantity, is known as the **dispersion medium**.When oil is shaken with water, a faint milky solution is often observed, which is unstable and is called an **emulsion of oil in water**. On standing, it gets separated into two layers, i.e. oil and water.

The stability of an oil and water emulsion is increased by the addition of a suitable emulsifying agent such as soap solution. Soap contains sodium salt of long chain aliphatic carboxylic acids with the carboxyl group as the polar group, which decreases the interfacial surface tension between oil and water. Hence oil mixes with water and emulsification takes place. In the presence of optimum amount of soap solution, oil in water emulsion is more stable and the separation of oil and water layers takes more time.

Procedure
(i) Dissolve 0.1 g of soap in 10 mL of distilled water to prepare 1% of soap solution in a test tube with vigorous shaking and heat the content of the test tube if needed. Label it as 'A'*(will be supplied to you)*.
(ii) Take four test tubes. Mark these as B, C, D & E and to each of the test tubes, add 5 mL distilled water followed by 2 ml of mustard oil in test tube B, 2 ml groundnut oil in test tube C , 2 ml coconut oil in test tube D, and 2 ml Palmolive oil in test tube E respectively.
(iii) Now add 0.5 ml of soap solution from test tube 'A' into each test tube (B, C, D and E).
(iv) Shake test tube B vigorously for few minutes, keep it in a test tube stand and simultaneously start the stopwatch. Record the time taken for the separation of the two layers.
(v) Repeat the same procedure with test tubes C ,D and E and record the time for the separation of the layers in each case.
(vi) Record your observations in a manner detailed in Table 1.1

Note:- *Rate each oil according to the time taken for separation in to two layers. The most stable emulsion takes the longest time***.**
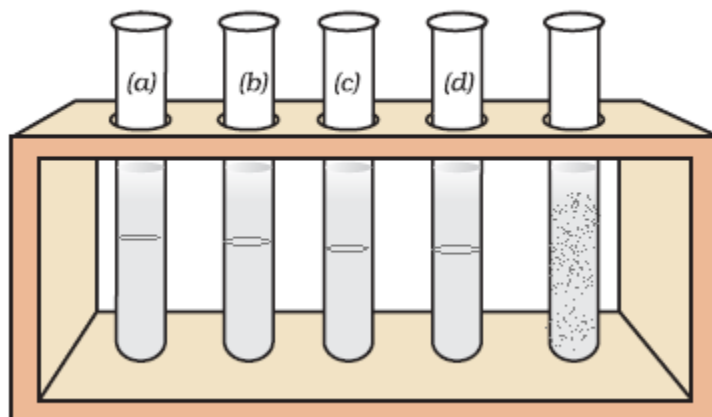
Result:-The rating of the oils on the basis of the stability of emulsion formed is as follows
       1. _____oil *(Maximum time)*
       2. _____oil
       3. _____oil
       4. _____oil *(Minimum time)*

Observation

Table 1.1 : Emulsification of different oils by 1% soap solution

Temperature:-_____K

Test tube specification	Name of oil used for emulsification	Time taken for the separation of layers	
		in minutes	in seconds
B			
C			
D			
E			

Diagram

Precautions

(a) Add equal number of drops of a soap/detergent solution to all the test tubes.

(b) To minimise the error in recording the time required for the separation of layers in different systems, shake all the test tubes for identical time span.

(c) Start the stopwatch immediately after shaking is stopped and stop it immediately when the two layers separate.

Discussion Questions

(i) Name a reagent other than soap, which can be used as an emulsifying agent in the *oil in water type emulsion.*

(ii) Milk is said to be a stable emulsion. What provides stability to milk?

(iii) Can two miscible liquids form an emulsion?

(iv) Why do separation of layers of different oils forming an emulsion with water take different time?

(v) What are the points of similarity and dissimilarity among sol, gel and emulsion?

(vi) Suggest a test to distinguish between *Oil in Water* and *Water in Oil* type of emulsions.

(vii) Give some examples of emulsions that you come across in daily life.

(viii) Dettol forms an emulsion in water. How does this emulsion get stabilised?

Demonstration Experiments

Experiment-N0-1

Cell potential in Daniel cell

Aim

To study the variation in cell potential of the cell $Zn/Zn^{2+}||Cu^{2+}/Cu$ with change in concentration of electrolytes ($CuSO_4$/$ZnSO_4$) at room temperature.

Apparatus Required

• Zinc plate : One • Copper plate : One • Beaker (50 mL) : Six • Voltmeter (Potentiometer) : One
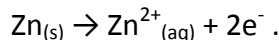• Salt bridge : One

Material Required

1.0 M & 0.1M Zinc sulphate solution: 100mL each, 1.0M & 0.1M copper sulphate solution: 100mL each
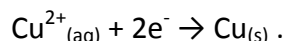
Theory

In the Daniel cell, copper and zinc electrodes are immersed in a solution of copper(II) sulfate and zinc sulfate respectively.

At the anode, zinc is oxidized per the following half reaction:

$$Zn_{(s)} \rightarrow Zn^{2+}_{(aq)} + 2e^- .$$

At the cathode, copper is reduced per the following reaction:

$$Cu^{2+}_{(aq)} + 2e^- \rightarrow Cu_{(s)} .$$

The total reaction being:

$$Zn(s) + Cu^{2+}(aq) \rightarrow Zn^{2+}(aq) + Cu(s).$$
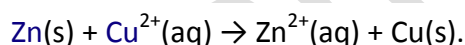
Procedure

(i) Set up the cell as given in Fig. 4.1, using 1.0 M $ZnSO_4$ and 1.0 M $CuSO_4$ solution.
(ii) Measure the potential difference of the cell using potentiometer and record the cell potential
(iii) Remove the salt bridge as soon as the cell potential measurement is over.
(iv) Repeat the procedure as per different combinations of copper sulphate & Zinc sulphateas shown in observation table
(v) Calculate the cell potential using Nernst equation and record it in the given table.

Result

The cell potential varies with change in concentration of Copper sulphate & Zinc sulphate solution and values observed are in agreement with cell potential values calculated using Nernst equation

Diagram:

Fig 4.1
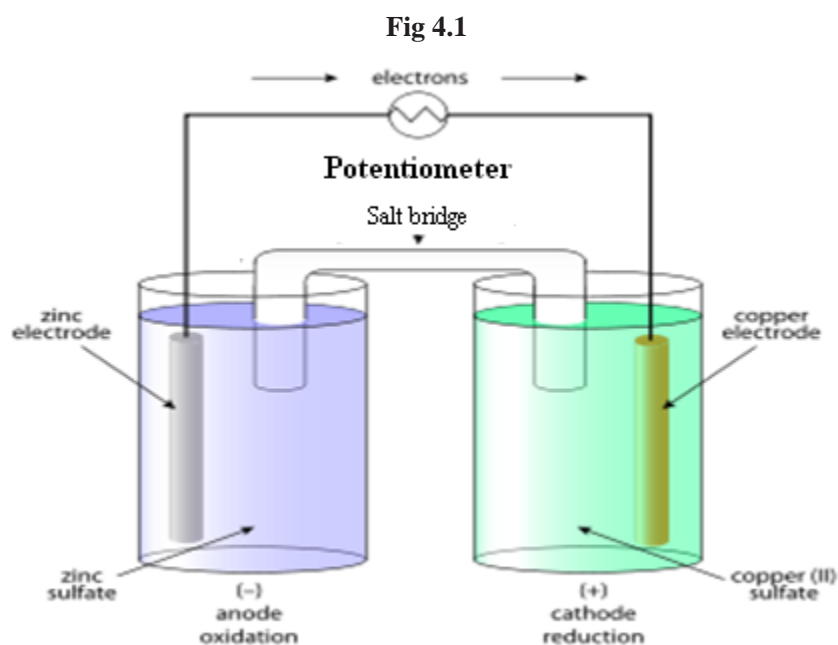
Observation:

Obs.No	Concentration of Solution		E^0 cell in Volts Std Cell Potential	E cell in Volts Cell Potential	
	Copper Sulphate	Zinc Sulphate	$E^0 = E^0_{cathode} - E^0_{anode}$	Calculated	Observed
1	1M	1M	1.10	1.10	
2	0.1M	0.1M	1.10	1.10	
3	1M	0.1M	1.10	**1.1295**	
4	0.1M	1M	1.10	**1.0705**	

Calculations:

1. $\mathbf{E^0}$= $\mathbf{E^0}$ cathode- $\mathbf{E^0}$ anode

$\mathbf{E^0}$ cathode=+0.34

$\mathbf{E^0}$ anode=-0.76

$\mathbf{E^0}$=0.34-(-0.76) =1.10 volts

2. To calculate Ecell for Obs.No 1

$$\text{Ecell= } E^0 \text{cell} - \frac{2.303RT}{nF} logQ \frac{[\text{Zinc ions}]}{[\text{Copper ions}]}$$

$logQ = \frac{[\text{Zinc ions}]}{[\text{Copper ions}]} x \frac{[\text{Copper}]}{[\text{Zinc}]}$ but $\frac{[\text{Copper}]}{[\text{Zinc}]}$=1

R=8.314J/K, T=298K and n=2

$$E = (E_R^{\circ} - E_L^{\circ}) - \frac{0.059}{2} \log \frac{\left[Zn^{2+}_{(aq)}\right]}{\left[Cu^{2+}_{(aq)}\right]}$$

$$\text{Ecell= } 1.10 - \frac{0.0591}{2} log \frac{[1]}{[1]}$$

log 1=0
Ecell= 1.10

Similarly for **Obs.No 2** also

$$\text{Ecell= } 1.10 - \frac{0.0591}{2} log \frac{[0.1]}{[0.1]}$$

Ecell= 1.10 volts

For **Obs.No 3**

$$Ecell= 1.10-\frac{0.0591}{2} \, log \, \frac{[0.1]}{[1]}$$

$$log \frac{[0.1]}{[1]} = log\,10^{-1} = -1$$

$$Ecell= 1.10 - \frac{0.0591}{2}\text{x} -1$$

Ecell= 1.10 +0.295

Ecell=1.1295 volts

For **Obs.No 4**

$$Ecell= 1.10-\frac{0.0591}{2} \, log \, \frac{[1]}{[0.1]}$$

$$log \frac{[1]}{[0.1]} = log\,10^{1} = +1$$

$$Ecell= 1.10 - \frac{0.0591}{2}\text{x} +1$$

Ecell= 1.10 -0.295

Ecell=1.0705 volts

Precautions

(a) Clean copper and zinc strips and connecting wires with sand paper before use.
(b) Place the salt bridge immediately in distilled water after its use.
(c) Carry out dilution of the solution to another concentration very carefully.

Discussion Questions????

1. What is a Electrochemical cell?

Experiment-N0-2

Paper chromatography

Aim
Separation of pigments present in the leaves (spinach) and flowers (rose, marigold) by paper chromatography and determination of R_f value of components.

Theory
In paper chromatography, water molecules present in the pores of the filter paper act as the stationary phase and the moving phase can be a solvent like hexane, toluene, acetone or a mixture of solvents such as methanol-water mixture etc. As the moving phase passes through the spot on which sample has been adsorbed, it dissolves the components more or less readily; depending upon the solubility and carries them along with it while moving on the support.

At a given temperature and for a given solvent, it is possible to determine the characteristic rate of movement of each substance on the chromotographic paper, as the moving phase moves.This is represented by relative front or **retardation factor also called R_f value.** R_f values of different compounds are different even if the mobile phase (solvent) is same. Furthermore, R_f value of a compound may be different in different solvents. R_f values can be calculated by using the following expression:

$$R_f = \frac{\text{Distance travelled by the substance from reference line (cm)}}{\text{Distance travelled by the solvent front from reference line (cm)}}$$

Since solvent front moves faster than the compounds, the R_f value of a substance will always be less than one. Also note that R_f value has no unit.

If the compound is coloured then its position on the chromatographic paper may be easily located. However, if the substance is colourless, it may be treated with a reagent, which imparts it a characteristic colour. This reagent is given the name **developer**. Iodine is the most commonly used developer in paper chromatography. Several other techniques are available for locating the spots.

Material Required

Whatman's filter paper No.1 of size 4 cm × 17 cm : One • Gas jar of size 5 cm × 20 cm: One
• Rubber cork fixed with hook in the centre : One • Test tubes : As per need
• extract of leaves : As per need • Distilled water : As per need • Methanol/Acetone : As per need
• Petroleum ether boiling range (60–80°C) : As per need • Chloroform /Acetone : As per need

Procedure
(i) Grind leaves in a mortar and transfer the paste into a test tube.
(ii) Add small amounts of methanol or acetone in the crushed material. Close the test tube with an appropriate cork and shake it well. Filter it and collect the filtrate in a test tube and cork the test tube.
(iii) Procure a Whatman filter paper No.1 of size 4 cm × 17 cm and mark a line at a distance of 3 cm from one of the ends of the paper with the help of a pencil [Fig. 5.1(a)].
(iv) Using a finely drawn capillary put one spot 'a' for the extract of leaves. Allow these spot to dry as shown in Fig. 5.1 (a).
(v) Hang the filter paper in a jar containing 20 mL mixture of petroleum ether (boiling range 60–80°C) and chloroform containing 19 mL petroleum ether and 1 mL chloroform or a mixture of petroleum ether (boiling range 60–80°C) and acetone in the ratio 9:1 (18 mL petroleum ether + 2 mL acetone)
so that the solvent does not touch the reference line as given in Fig. 5.1 (b).
(vi) Keep this jar as such till the mobile phase (solvent) rises up to 2/3 of the length of the paper [Fig. 5.1(c)].
(vii) Remove the filter paper from the jar, mark the solvent front, outline the spots with the help of a pencil and allow the filter paper to get dry.

(viii) Measure the distance travelled by the solvent front and the centre of different spots with respect to the reference line as given in Fig. 5.1 (d).

(x) Ascertain the number of pigments, which are present in the extract of leaves

(xi) Calculate the R_f value of different spots with the help of the expression mentioned earlier.

(xii) Record your observations as in Table 5.1.

Result

(i) R_f values of components of flower are _____.
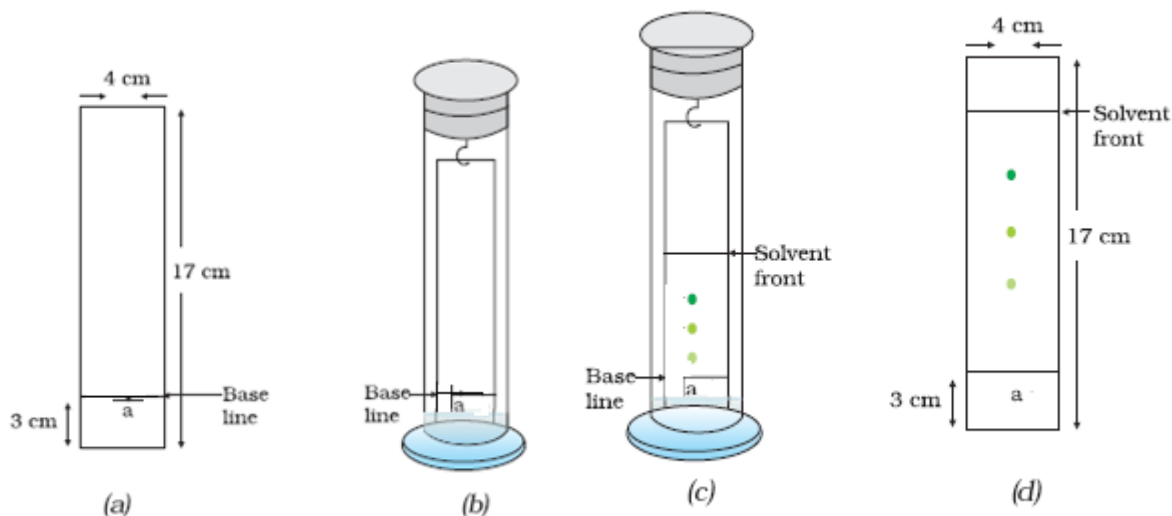
Fig. 5.1 : *(a) Marked paper; (b) Dipping the filter paper in the solvent;*
(c) Developing chromatogram; and (d) Developed chromatogram

Table 5.1: Separation of pigments of leaves

Sl. No.	Name of the extract	Colour of the spot	Distance travelled by the components of the spot 'a' from the reference line in cm	Distance travelled by the solvent from reference line in cm	R_f value
1.					
2.					
3.					
4.					

Precautions

(b) Dip the paper strip in the solvent in such a way that the spot of the mixture is above the solvent level and the movement of the solvent front is not zig-zag.

(c) While spotting the test solution on the paper, do not allow the spots to spread. Use finely drawn capillary to put the spot on the paper.

(e) Once the experiment is set, do not disturb the jar as long as the chromatogram is being developed.

(f) Keep the jar covered with the lid when the chromatogram is being developed.

PREPARATION OF INORGRGANIC COMPOUNDS

Aim
To prepare double salt: ferrous ammonium sulphate (Mohr's salt).

Material Required

Beaker (50 mL) : One • Conical flask (50 mL) : One • Trough : One • Glass rod : One • Tripod stand : One • Funnel : One • Wire gauze : One

• Ferrous sulphate : As per need • Ammonium sulphate : As per need • Dilute sulphuric acid : As per need • Ethanol : As per need
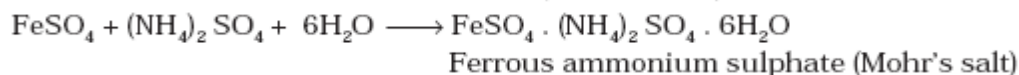
Theory
When a mixture containing equimolar proportions of ferrous sulphate and ammonium sulphate is crystallized from its solution, a double salt is formed. The formation of double salt may be shown as follows:

$$FeSO_4 + (NH_4)_2\,SO_4 + 6H_2O \longrightarrow FeSO_4 \cdot (NH_4)_2\,SO_4 \cdot 6H_2O$$
$$\text{Ferrous ammonium sulphate (Mohr's salt)}$$

Fe^{2+} and Al^{3+} ions undergo hydrolysis, therefore, while preparing aqueous solutions of ferrous sulphate and aluminium sulphate in water, 2-3 mL dilute sulphuric acid is added to prevent the hydrolysis of these salts.

Procedure
Preparation of Double Salt: Ferrous Ammonium Sulphate
(i) Dissolve 3.5 g of ferrous sulphate and 1.7 g of ammonium sulphate (weighed separately), in 5 mL of distilled water contained in a 50 mL conical flask by heating. Add about 0.5 mL of dilute sulphuric acid to the flask and concentrate the solution by heating till the crystallization point is reached.
(ii) Allow the mixture to cool to room temperature slowly.
(iii) On cooling, light green crystals of ferrous ammonium sulphate separate out.
(iv) Decant the mother liquor and wash the crystals by shaking with very small amounts of 1:1 cold water and alcohol mixture to remove sticking mother liquor.
(iv) Separate the crystals by filtration wash with alcohol, dry between the folds of a filter paper and record the yield.

Result

Yield of Mohr's salt is _____ g.

Precautions
(a) Cool the solution slowly to get good crystals. Avoid rapid cooling.
(b) Do not disturb the solution while cooling.
(c) Avoid prolonged heating while preparing crystals of ferrous ammonium sulphate, as it may oxidise ferrous ions to ferric ions and change the stoichiometry of the crystals.

Discussion Questions?????
(i) Why do we take equimolar quantities of reacting compounds in the preparation of double salts?
(ii) In the preparation of ferrous ammonium sulphate, can concentrated sulphuric acid be used in place of dilute sulphuric acid? Explain.
(iii) What is the difference between iron compounds; $K_4[Fe(CN)_6]$ and $FeSO_4.(NH_4)_2SO_4.6H_2O$?
(vi) What is the difference between a complex compound and a double salt?

PREPARATION OF ORGANIC COMPOUNDS

Aim
To prepare acetanilide.

Theory
The replacement of one hydrogen atom of the — NH_2 group of aniline by $CH_3CO–$ group in the presence of glacial acetic acid. Gives acetanilide. In the laboratory, acetylation is usually carried out with acetic anhydride. Acetyl chloride may also be used for the purpose of acetylation if acetic anhydride is not available. Acetylation with CH_3COCl is usually carried out in the presence of pyridine.
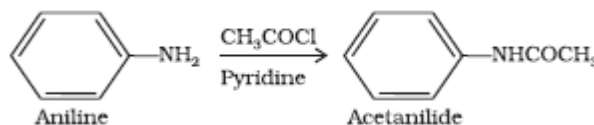
Material Required
Funnel : One • Round bottomed flask (100 mL) : One • Beaker (250 mL) : One • Air condenser : One
• Sand bath : One • Clamp and iron stand : One • Pumice stone : As per need • Melting point assembly : One

• Aniline : 5 mL • Acetic anhydride /Acetyl chloride : 5 mL • Acetic acid / Pyridine : 5 mL

Procedure
(i) Take 5 mL of aniline in a 100 mL round bottom flask and add acetylating mixture containing 5 mL acetic anhydride and 5 mL glacial acetic acid. Alternatively, you can use 5 mL of acetyl chloride and 5 mL of dry pyridine as the acetylating mixture.
(ii) Fit an air condenser on the mouth of the round bottom flask after adding a few pumice stones and reflux the mixture gently for 10-15 minutes on a sand bath.
(iii) Cool the reaction mixture and pour it slowly in 150-200 mL of ice cold water with stirring.
(iv) Filter the solid, wash it with cold water and recrystallise a small amount of sample from hot water containing a few drops of methanol or ethanol.
(v) Report the yield and the melting point of the compound.

Result

(a) Yield of acetanilide _____ g.

(b) Melting point of acetanilide is _____ °C.

Precautions
(a) Handle acetic anhydride and acetyl chloride carefully as they cause irritation to the eyes and acetyl chloride also strongly fumes in air.
(b) Store acetylchloride under dry conditions.
(c) Handle pyridine with extreme caution. Dispense it in an efficient fume cupboard and wear disposable glasses while using it.
(d) Distil pyridine before use because it absorbs mioisture and the reaction does not take place under moist conditions.
(e) Wash the solid 2-3 times with cold water till the filtrate is neutral to litmus.
(f) Determine the melting point of perfectly dried and recrystallised sample.

IDENTIFICATION OF FUNCTION AL GROUP PRESENT IN
THE GIVEN ORGANIC COMPOUND

TEST	OBSERVATION	INFERENCE
1. TEST FOR CARBOXYLIC GROUP (-COOH)		
Compound + $NaHCO_3$	Effervescence of a gas	.: Carboxylic group is present (- COOH)
	No effervescence of a gas	.: Carboxylic group is absent
2. TEST FOR PHENOLIC GROUP (Ar-OH)		
Small amount of compd + NaOH and heat	Substance is soluble and is regenerated by addition of dil HCl	.: Phenolic group is present
	Substance not soluble	.: Phenolic group is absent
OR		
Aqueous soln of a small amount of compd in a test tube + 2-3 ml of neutral $FeCl_3$ Solution	Green or violet colour obtained	.: Phenolic group is present
	No green or violet colouration obtained	.: Phenolic group is absent
3. TEST FOR AMINO GROUP (-NH$_2$)		
Small amount of compound in a test tube + dil HCl	Substance dissolves which reappears on addition of NaOH soln	.: Amino group is present (-NH$_2$)
	Substance does not dissolve	.: Amino group (-NH$_2$) is absent
4. TEST FOR CARBONYL GROUP (-CHO or >CO) (2,4-DNP) test		
Shake small amount of Organic compound with 2,4 dinitrophenyl hydrazine (2,4-DNP) in a test tube.	Orange –yellow ppt. is formed.	.: Carbonyl group (aldehydic or ketonic group) is present. I.e. Organic compound is either Aldehyde or Ketone

Test for Aldehyde (Schiff's reagent test)		
Small amount of substance + Schiff's reagent	Magenta (pink) colour	.: Aldehydic group is present
	No Magenta (pink) colour	.: Aldehydic group is absent
Test for Aldehyde (Silver mirror test)		
1ml of compd in a clean test tube + 1ml Tollen's reagent. Warm in a water bath.	Silver mirror (or a black ppt.) is formed on the side of the test tube	: Aldehydic group is present
	No silver mirror formed.	.: Aldehydic group is absent
Test for Ketone (Iodoform test)		
1 ml of compd in a test tube+ 1ml NaOH + I_2 soln till yellow. Warm on a water bath; add more I_2 till colour persent. Cool under a tap.	Light yellow crystalline solid (Iodoform) separates out	.: Ketonic group is present.
	No ppt	.: Ketonic group is absent
Test for Ketone (Sodium nitroprusside test)		
Dissolve a crystal of sodium nitroprusside in 1ml distilled water. Add 0.5 ml of compd followed by NaOH soln. dropwise.	Red colour formed	.: ketone may be present.
	No red colour	.: Ketonic group is absent

5. TEST FOR ALCOHOLIC GROUP (R-OH)

Small amount of compd in a test tube + 1ml CH_3COOH + 2-3 drops of conc. H_2SO_4. Warm this solution in a water bath, pour the contents in the test tube into a beaker containing water	Sweet smell or fruity odour	.: Alcoholic group (-OH) is present
	No sweet (fruity odour)	.: Alcoholic group is absent
OR		
Compd in a test tube + small piece of Na metal	Brisk effervescence	: Alcoholic group is present
	No brisk effervescence	.: Alcoholic group is absent

_____@@@@@@@@@@@@@@@@@_____

How to write a project report?

1. Cover page with title

2. Certificate

3. Content/Index

4. Statement of the problem/Aim

5. Introduction

6. Theory (if any)

7. Apparatus & materials required

8. Procedure +(diagram or picture if any)

9. Observations /Calculations or both

10. Results/Discussion/conclusion

11. Further investigation

12. Acknowledgement/Bibliography

13. Reference.

Note:- *The Project report is to be written by the students in their own handwriting on A4-size Bond paper & submitted in a file. Only the cover page with title may be a computer print.*

………. Following is the Content from Goa Board Circular No-30 year 2014

Suggestion regarding chemistry practicals.
1. We propose to shift the analysis of natural organic compound to Std. XI (Science).
2. Physical Chemistry experiments for demostrations only, but to be recorded in the Journal.
3. The marking scheme for the final Std. XII Sc. Examination (peocheals) may be as follows.
   Volumetric Analysis ………………………………… 7 marks
   Salt Analysis (Inorganic) onalative analysis ….. 8 marks
   Functional group………………………………………. 2 marks
   Journal + Viva ……………… 2 marks + 2 marks = 4 marks
   Project + Viva ……………… 2 marks + 2 marks = 4 marks

Scheme for allotment of marks to the different experiments.

Internal Examiner	External Examiner

1. Volumetric analysis ………… 7 marks
a) Observation …………. 1 mk
b) Calculation of Volume . 1 mk.
c) Correct titre reading ….. 3 mk
d) 2 calculations … ……..2 mk
7 mk
 	
2. Functiona Group ……………… 2 Marks
 For detailed distribution of marks refer annexure – I
 	
3. Journal ……………………………… 2 marks
   Viva on Journal experiment ..2 marks
 	
 Total marks…………………………13 marks
	1. Inorganic qualitative
	   analysis – (Salt analysis).. 8 marks
	a) Pre/Drytest -- 1/2 mark each
	          = 1/2 X 8 = 4 marks
	b) Group separation ……. 1 mark
	c) Indentsfication of cation ..1/2 mk
	c) C.T. For cation …………… 1/2 mk
	d) Identification of Onion … 1 mark
	e) C. T. For Anions …………. 1/2 mk
	f) Correct formula of salt ….. 1/2 mk
	          _____
	          8 marks
	2. Project ……………………… 2 marks
	   Viva on project …………… 2 marks
	          _____
	 Total marks = 12 marks

ANNEXURE – I

Distribution of marks for Identification of Functional Group

Functional Group	Marks to be alloted
1. Carboxylic group (- Co OH	2 marks
2. Phenolic group	If not identified correctly. 1/2 mk to be awarded for responding – Co OH as absent $ 1 and 1/2 for detecting phenolic group correctly.
3. Anino group (- NH₂)	If not identified correctly. 1 mk for recoding -Co OH $ phenolic group as absent, 1 mk for detecting - NH₂ group correctly
4. Carbinyl group	If not identified correctly. 1 mk for recoding absence of Co OH , - NH₂ $ phenolic group (1/2 mark only if only two of these groups are mentioned. 1/2 mk for detecting carbonyl group 1/2 mk for distinguishing carbenyl group either as – CHo or CO
5. Alcoholic group (- OH)	Not identified correctly, 1mk for reporting – CO OH , phenolic, - NH₂ $ carbonyl group as absent. (1/2 mk only if any of the two above groups are mentioned) 1 mk for recording alcoholic group correctly

P.S. Test for alcoholic group to be performed last.

The scheme for detection of Functional Group should be as follows.

1. Test for Co OH group
2. Test for phenolic group
3. Test for - NH₂
4. Test for Carbenyl group
5. a) Detection of -CHO
   b) Detection of – CO
6. Test for alcoholic group

@@

Analysis of Salt

PRELIMINARY TESTS:		
TEST	**OBSERVATION**	**INFERENCE**
1. Colour	(a) Blue or Bluish green	Cu^{2+} or Ni^{2+} may be present Ni^{2+} may be present Fe^{3+} may be present
	(b) Green (c) Reddish Brown or Brown	Co^{2+} may be present Mn^{2+} may be present
	(d) Pink (e) Light Pink, Flesh colour or earthy colour	$Cu^{2+}, Ni^{2+}, Fe^{3+}, Co^{2+},$ Mn^{2+} may be absents.
	(f) Colourless (white)	$Pb^{2+}, Al^{3+}, Zn^{2+}, Ca^{2+}, Sr^{2+},$ $Ba^{2+}, Mg^{2+}, K^+, NH_4^+$ may be present
2. Heating in a dry test tube Little of compound heated in a dry heating tube **N.B** *(1) Observe the evolution Of gases also*	(a) Decrepitation *(Crackling sound)* (b) Coloured residue *(including black)*	$Pb(NO_3)_2, KCl, Ba(NO_3)_2$ KNO_3 may be present $Cu^{2+}, Co^{2+}, Ni^{2+}, Fe^{3+}$ salts may be present
(2) If original compd is coloured then only a black residue obtained	(c) White infusible residue	$Ba^{2+}, Sr^{2+}, Ca^{2+}$ may be present
	(d) White sublimate	.: Ammonium halides may be present
(3) Observe the changes by first slowly heating followed by strong heating	(e) Yellow residue when hot which turns white when cold	Zn^{2+} may be present
	(f) White compd gives black residue on strong heating	.: $K_2 C_2 O_4$ may be present
	(g) **Evolution of gases** (1) Colourless gas with Smell of Ammonia turning red litmus paper blue	.: NH_4^+ may be present
	(2) Colourless gas turning blue litmus paper red	.: Cl^-, SO_4^{2-}, CH_3COO^- may be present
	(3) Colourless gas with having vinegar like smell	CH_3COO^- may be present

TEST	OBSERVATION	INFERENCE
	(4) Colourless gas turning lime water milky	$C_2O_4^{2-}$ may be present
	(5) Brown funes give out	NO_3^-, Br^- may be present
	(6) Greenish yellow gas turning starch iodide paper blue	Cl^- may be present
	(7) Reddish brown gas turning starch iodide paper orange yellow	Br^- may be present
	(8) Dark violet vapours gives out turning starch iodide paper blue	I^- may be present
3. Flame test *Clean the Pt.loop/Nichrome wire by dipping it in a t.t containing a little conc. HCl and placing it in the blue part of the flame. moisten a little compd on the watch glass with conc. HCl. Dip the Pt.loop/Nichrome wire in this mixture and hold it in the blue part of the flame. Observe the flame colour*	(1) Lilac or Violet flame	K^+ may be present
	(2) Bluish white flame	Pb^{2+} may be present
	(3) Bluish green flame	Cu^{2+} may be present
	(4) Apple green flame	Ba^{2+} may be present
	(5) Brick red (Dull red) flame	Ca^{2+} may be present
	(6) Crimson red (dark red) flame	Sr^{2+} may be present
	(7) No characteristic colour to the flame	K^+ Pb^{2+} Cu^{2+} Ba^{2+} Ca^{2+} Sr^{2+} May be absent.
4. Test for NH_4^+ Compound + dil NaOH. Warm	(1) Gas evolved with smell of ammonia forming dense white fumes with a glass rod dipped in conc. HCl.	∴ NH_4^+ may be present
	(2) No smell of ammonia	∴ NH_4^+ may be absent
DRY TEST FOR ANION (ACIDIC RADICALS) **5. Action of dil. HCl** Compd + dil HCl *(Observe without heating.)*	(1) Effervescence of a gas turning lime water milky	∴ CO_3^{2-} may be present
	(2) No Effervescence	∴ CO_3^{2-} may be Absent

TEST	OBSERVATION	INFERENCE
6. **Action of Conc.H$_2$SO$_4$** Compd + Conc.H$_2$SO$_4$ **NB**: *Test for Cl$^-$ in cold. If Cl absent, warm and observe*	(1) Colourless fumes of HCl given out forming dense white fumes with a glass rod dipped in NH$_4$OH.	.: Cl$^-$ may be present
	(2)Colourless vapours given out on warming, having the smell of vinegar.	.: CH$_3$COO$^-$ may be present
	(3) Reddish brown or yellowish brown vapour given out on warming the test tube.	.: Br$^-$,NO$_2^-$, NO$_3^-$ may be present
	(4) Brownish fumes obtained above (3) increases on addition of Cu foil.	.: Br$^-$ may be absent NO$_2^-$, NO$_3^-$ may be present
	(5) Violet vapours given out on warming the test tube.	.: I$^-$ may be present
	(6) No characteristic fumes / vapours	Cl$^-$, Br$^-$, I$^-$, NO$_3^-$, CH$_3$COO$^-$ may be absent
7.Test for PO$_4^{3-}$ Compd.+ Conc HNo$_3$ *(Boil to drive out any coloured fumes if given.)* Add excess of ammonium molybdate soln	1) Canary yellow ppt	.: PO$_4^{3-}$ may be present
	2) No Canary yellow ppt	.: PO$_4^{3-}$ absent.
8. **Test for C$_2$O$_4^{2-}$ (KMnO$_4$ Test)** Compd. + 2 mldil.H$_2$SO$_4$ Boil off any gas evolved, add little more dil.H$_2$SO$_4$ and then KMnO$_4$ soln dropwise.	KMnO$_4$ Soln is decolourised	.: C$_2$O$_4^{2-}$ may be present
	KMnO$_4$ Soln is not decolourised	.: C$_2$O$_4^{2-}$ absent.

NB: *If the tests (5),(6),(7) and (8) do not give positive results ,the compound may contain SO$_4^{2-}$ anion.*

PREPARATION OF SOLUTION (W.E)

Take a 10 ml (approx) of distilled water in a 25 ml beaker, warm and add pinch by pinch the compound , stir till it dissolves, then add more . Solution obtained is (W.E) **Water Extract** (*Use this solution to perform the wet tests for cation and anion*).

1. <u>**TEST FOR HALIDES**</u> **(Cl⁻ , Br⁻ , I⁻)** W.E + Ag NO₃	No ppt or Ppt soluble in dil HNO₃	Halides absent.
	Ppt insoluble in dil HNO₃	Halides (Cl⁻ , Br⁻, I⁻) are present
<u>**DETECTION OF HALIDES (Cl⁻, Br⁻,I⁻)**</u> *Note the colour of the appt obtained above in*	(a) White ppt soluble in NH₄OH (b) Pale yellow ppt (c) Yellow ppt	Cl⁻ is present Br⁻ is present I⁻ is present
<u>**C.T FOR Cl⁻ /Br⁻ / I⁻**</u> (i) W.E + Conc H₂ SO₄+ MnO₂. Warm gently	(a) Faint greenish yellow fumes turning moist blue litmus paper red and finally bleaches it (b) Reddish brown fumes (c) Violet fumes	Cl⁻ is confirmed Br⁻ is confirmed I- is confirmed
ii) W.E + dil. H₂SO₄ +CCl₄ or CHCl₃ + excess of strong chlorine water. Shake well and allow to stand	(a) Colourless CCl₄ /CHCl₃ layer (b) Reddish yellow CCl₄ / CHCl₃ layer (c)Violet CCl₄ /CHCl₃ layer	Cl⁻ is confirmed Br⁻ is confirmed I⁻ is confirmed
2. TEST FOR <u>**CARBONATE (CO₃²⁻)**</u> W. E + CaCl₂	White ppt soluble in dil HNO₃ with effervescence.	CO₃²⁻ is present
<u>**C.T for CO₃²⁻**</u> **(i)** W.E + dil HCl	Effervescence of a gas turning lime water milky.	CO₃²⁻ is confirmed
(ii) W.E + phenolphthalein	Pink colour	CO₃²⁻ is confirmed
3. <u>TEST FOR SULPHATE</u> (SO₄²⁻) W.E + di.l HNO₃ + Ba(NO₃)₂	White ppt	SO₄²⁻ is present
C. T FOR SULPHATE W.E + Pb (CH₃ COO)₂	White ppt insoluble in dil. HNO₃	SO₄²⁻ is confirmed

4. TEST FOR NITRATE **(NO_3^-)** W.E Conc H_2SO_4 +Cu filings. *Warm Carefully.*	Brown fumes of NO_2 and Bluish green soln	NO_3^- is present
C.T For NO_3^- W.E + Conc H_2SO_4 Cool under tap. then add freshly prepared saturated $FeSO_4$ soln Carefully along the sides of the test tube .	Brown ring (of $FeSO_4.NO$) at the junction of the two solutions	NO_3^- is confirmed
5.TEST FOR **PHOSPHATE (PO_4^{3-})** W.E + Conc. HNO_3 boil gently. Add ammonium Molybdate in excess and warm again.	Canary yellow ppt	PO_4^{3-} is present
C.T For PO_4^{3-} W.E + NH_4Cl + NH_4OH + Mg SO_4 solution	White crystalline ppt of Magnesium ammonium phosphate	PO_4^{3-} is confirmed
6.TEST FOR **ACETATE (CH_3COO^-)** W.E + neutral $FeCl_3$ soln	Reddish coloured ppt or soln	$CH_3 COO^-$ is present
C.T for $CH_3 COO^-$ To the decanted solution from above add dil HCl	Reddish colour disappears and a white ppt is formed in a yellow coloured solution	$CH_3 COO^-$ is confirmed
(2) ESTER TEST W.E + Conc. H_2SO_4 (2ml) Heat. Add ethyl alcohol (1ml). Shake warm pour this soln into a beaker containing water.	Pleasant fruity smell of ester.	$CH_3 COO^-$ is confirmed.
7. TEST FOR OXALATE **($C_2O_4^{2-}$)** W.E + dil CH_3COOH. Boil to remove CO_2. Add $CaCl_2$ soln.	White ppt formed that dissolves on adding dil HNO_3 and warming.	$C_2O_4^{2-}$ is present.
C.T FOR $C_2O_4^{2-}$ W.E + dil H_2SO_4 warm. Add 2-3 drops of $KMnO_4$	Pink colour of $KMnO_4$ is decolourised with the evolution of CO_2 gas.	$C_2O_4^{2-}$ is confirmed

WET TEST FOR CATION
DETECTION OF CATION OF GROUP ZERO (NH_4^+)

TEST	OBSERVATION	INFERENCE
W.E + 2ml dil NaOH shake well and warm gently	(1) No smell of Ammonia/ NH_3 gas not evolved	.: Group O is absent .: NH_4^+ is absent
	(2)NH_3 gas evolved recognized by its smell turning most red litmus paper blue **or** moist Turmeric Paper brown **or** Evolution of dense white fumes when a glass rod dipped in conc.HCl is held over the mouth of the test tube	.: Group O is Present .: NH_4^+ is Present
C.T. for NH_4^+ 1ml of Nessler's reagent + 2 drops of W.E	Brown ppt or Colouration	.: NH_4^+ is confirmed

SEPARATION OF CATIONS INTO GROUPS (I – VI)

TEST	OBSERVATION	INFERENCE
1)W.E + dil. HCl	White ppt	Group I is present .: Pb^{2+} is present *(Refer table No 2)*
	No white ppt	Group I is absent
2)W.E + dil. HCl + warm and add H_2S water.	Black ppt	.: Group II is present .: Cu^{2+} is present *(Refer Table No 3)*
	No black ppt	Group II is absent
3) W.E + Solid NH_4Cl + NH_4OH till alkaline	Coloured ppt *(white or Brown)*	.: Group III is present .: Al^{3+}, Fe^{3+}, Mn^{2+} are present *(Refer Table No 4)*
	No coloured ppt	.: Group III is absent

		.
4) W.E + solid NH_4Cl +NH_4OH till alkaline + H_2S gas / H_2S water	Coloured ppt *(Black, White,Pink)*	Group IV is present Co^{2+}, Ni^{2+}, Zn^{2+} and Mn^{2+} are present *(Refer Table No 5)*
	No coloured ppt	.: Group IV is absent
5) W. E + Solid NH_4Cl + NH_4OH till alkaline + $(NH_4)_2 CO_3$. Warm	White ppt	Group V is present .: Ba^{2+}, Sr^{2+}, Ca^{2+} are present *(Refer Table No 6)*
	No white ppt	.: Group V is absent .: Group VI is present .: K^+, or Mg^{2+} may be present *(Refer Table No 7.)*

TABLE NO. 2
Analysis of Group I   (Pb^{2+})
The colour of the group I ppt ($PbCl_2$) is white
.: Pb^{2+} is present.

C.T. for Pb^{2+}

TEST	OBSERVATION	INFERENCE
1) W.E $+^1/_2$ ml KI	Yellow ppt of Pb I $_2$	.: Pb^{2+} is confirmed
2) W.E + ½ ml K_2CrO_4	Yellow ppt	.: Pb^{2+} is confirmed
3) W.E + ½ ml dil H_2SO_4	White ppt of $PbSO_4$	.: Pb^{2+} is confirmed

TABLE NO 3
Analysis of Group II (Cu^{2+})
The colour of the group II ppt (CuS) is black .: Cu^{2+} is present.

C.T. for Cu^{2+}

Test	OBSERVATION	INFERENCE
1. W.E + KI soln	Brown ppt of CuI_2	.: Cu^{2+} is confirmed
2. W.E + dil NaoH	Blue ppt of $Cu(OH)_2$ turning black on heating	.: Cu^{2+} is confirmed

TABLE NO. 4
Analysis of Group III (Al^{3+}, Fe^{3+}, Mn^{2+})

The colour of the ppt of Group III hydroxides is white / Reddish brown

White gelatinous ppt. .: Al^{3+} is present. **C.T. for Al^{3+}:** 1) W.E + NaOH soln dropwise → gelatinous white ppt soluble in excess of NaOH. 2) W.E +Na_2CO_3 solution →white ppt. 3) W.E + ammonium acetate soln.→No ppt in cold but on boiling a white gelatinous ppt. **Al^{3+} is confirmed**.	Reddish brown or brown or pink ppts . :Fe^{3+} or Mn^{2+} are present.	
	: W.E +$K_4Fe(CN)_6$→ Deep blue ppt / no deep blue ppt.	
	Deep blue ppt or colouration Fe^{3+} is present.	No deep blue ppt Mn^{2+} is present
	C.T for Fe^{3+} W.E. + KSCN/NH_4CN → Blood red colouration. **Fe^{3+} is confirmed**	**C.T for Mn^{2+}** 1) W.E.+ NH_4OH till alkaline →white ppt turning brown on heating 2) W.E +5ml of dil HNO_3 +pinch of solid Sodium bismuthate. Shake, allow to stand →violet colouration. **Mn^{2+} is confirmed**

*Note: With concentrated W.E the Mn^{2+} is precipitated In Group III as Mn $(OH)_2$ (brown or buff ppt) Otherwise Mn^{2+} will be precipitated as MnS (Pinkish ppt) in Group IV. Thus, Mn can analysed either in **Group III** or in **Group IV**.*

TABLE NO 5
Analysis of Group IV $(Co^{2+}, Ni^{2+}, Zn^{2+}, Mn^{2+})$

The colour of the ppt.of Group IV sulphides is Black / White / Pinkish

Black ppt.		**White ppt.**	**Pinkish ppt.**
: Co^{2+} or Ni^{2+} are present		. Zn^{2+} is present.	. : Mn^{2+} is present.
W.E + equal volume acetone + $NH_4SCN \rightarrow$ Blue colour / no blue colour		**C.T for Zn^{2+}:** 1) W.E. + $K_4Fe (CN)_6 \rightarrow$ White ppt. W.E.+NaOH soln. dropwise $\rightarrow$ White ppt. Soluble in excess of NaOH and is reppt. as ZnS on addition of H_2S gas.	**C.T. for Mn^{2+}:** 1) W.E + $NH_4OH \rightarrow$ White ppt.turning brown on heating. 2) W.E +5ml dil HNO_3 pinch of sodium bismuthate. Shake and allow to stand $\rightarrow$ Violet colouration. 3) W.E + PbO_2 (0.1g) +1/2 ml of dil.(1:1) HNO_3; boil and allow to settle $\rightarrow$ Violet colouration.
Blue colour Co^{2+} is present. **C. T. for Co^{2+}** 1) W.E.+1ml of a-nitroso β- napthol $\rightarrow$Brown ppt. 2) W.E.+NaOH $\rightarrow$blue ppt changing to brown on heating. 3) W.E.+ NH_4Cl+NH_4OH till alkaline +$K_4Fe(CN)_6\rightarrow$ Red soln. Warm $\rightarrow$Reddish brown ppt. .:Co^{2+} is confirmed .	**No Blue colour** Ni^{2+} is present. **C.T. for Ni^{2+}** 1) W.E. + NH_4Cl +NH_4OH till alkaline + dimethyl glyoxime. $\rightarrow$Scarlet red ppt. 2) W.E. + NH_4OH in excess. $\rightarrow$ Pale green ppt. soluble in excess giving blue soln. .:Ni^{2+} is confirmed	.: Zn^{2+} is confirmed	.:$Mn^{2+\,is}$ confirmed .

40

TABLE NO 6
Analysis of Group V (Ba $^{2+}$, Sr $^{2+}$, Ca $^{2+}$)

The colour of the ppt. of Group V carbonates is white therefore Ba $^{2+}$/. Sr^{2+} or Ca^{2+} are present

W.E +Acetic acid+ $K_2CrO_4 \rightarrow$ yellow ppt / no yellow ppt.

Yellow ppt. Ba $^{2+}$ is present.	No yellow ppt. Sr^{2+} or Ca^{2+} are present . W.E +Acetic acid + NH$_4$OH till alkaline + solid (NH$_4$)$_2$ SO$_{4 +}$ boil $\rightarrow$ white ppt. / No white ppt	
C.T.for Ba^{2+}: 1) W.E +Acetic acid.+ ammonium oxalate soln. $\rightarrow$ White ppt. insoluble in acetic acid. 2) W.E +Acetic acid. + dil. H$_2$SO$_4 \rightarrow$ White ppt. 3) Flame test $\rightarrow$ Apple green flame. .:Ba^{2+} is confirmed.	**White ppt.** Sr^{2+} is present **.C.T.for Sr^{2+}:** 1) W.E +Acetic acid Ammonium oxalate $\rightarrow$White ppt. Insolube in acetic acid. 2) W.E +Acetic acid.+ dil. H$_2$SO$_4 \rightarrow$ White ppt. 3) Flame test $\rightarrow$ Crimson red flame. .:Sr^{2+} is confirmed.	**No white ppt** Ca^{2+} is present **.C.T. for Ca^{2+}** 1) W.E +Acetic acid.+ CaSO$_4 \rightarrow$ No ppt. 2) W.E +Acetic acid.+ (NH$_4$)$_2$C$_2$O$_4 \rightarrow$ White ppt. Insoluble in acetic acid. 3) Flame test $\rightarrow$ Brick red Flame. .:Ca^{2+} is confirmed.

TABLE NO 7

Analyses of Group VI (Mg$^{2+,}$ K$^+$)

| **TEST FOR Mg^{2+}**
W.E add NH$_4$Cl and NH$_4$OH till alkaline (in slight excess) and then add ammonium phosphate solution. Scratch the inner side of the test tube with a glass rod$\rightarrow$
  1. white crystalline ppt .: Mg^{2+} is present
  2. No white   ppt .: Mg^{2+} is Absent

 C.T for   Mg^{2+}
  W.E + NaOH solution
  $\rightarrow$ White ppt soluble in NH$_4$Cl   solution

  .: Mg^{2+} is confirmed | **TEST   FOR   K$^+$**
  W.E + 1-2ml of fresh and clear solution of sodium cobaltinitrite. $\rightarrow$

Yellow ppt

.: K$^+$ is present.

C. T for K$^+$
(1) W.E + picric acid $\rightarrow$ yellow ppt.
  .: K$^+$ is confirmed |

Result:-

      a. Cation_____

      b. Anion_____

      c. Formula_____

      d. Name of the Compound_____

Instructions:

1. Always be in time for your practical's.

2. Always corne prepared for the experiment. This will help in understanding the experiment better.

3. Always listen to the teacher's instructions carefully and note down the important points and precautions to be followed.

4. Do only the experiments assigned, unallotted experiments should not be done.

5. Do your experiment honestly without caring for the final result. Record the observations on a rough note-book instead of writing on pieces of paper.

6. Be economical with the reagents. Only small quantities of the reagent are to be used.

7. Handle the glass apparatus very carefully. In case of any breakage, report it to your teacher at once.

8. Dispose of all waste liquids in the sink and allow the water to run for sometime by opening the water tap.

9. Keep your work place clean. If an acid or other corrosive chemical is spilled, wash it off with water.

10. In case of any injury or accident or breakage of the apparatus, report it to the teacher immediately.

11. Wash your hands with soap after the experiment.

Precautions:

1. Do not touch any chemical with the hand as some of them may be corrosive.

2. Never taste a chemical. It may be poisonous.

3. Do not place the chemical on the palm of your hand.

4. Do not keep the reagent bottles open.

5. Do not roam here and there in the laboratory uselessly.

6. Do not put any object into the reagent bottle.

7. Do not bring inflammable liquids such as alcohol or ether near the flame.

8. Do not take the reagent from the shelf to your work place.

9. Do not disturb the arrangement of the reagents placed on the shelf.

10. Do not use cracked glass apparatus such as beakers for heating purposes.

11. Do not keep the water tap running when not required.

12. Do not throw solid waste materials like pieces of filter paper, broken test-tubes, etc in the sink. Throw them in the waste box only.

13. Do not heat beakers or a china dish directly on a flame. Always make use of wire gauze.

Wear: **1.Safety gloves 2.Safety glasses 3.Gas masks and 4.Lab coat** *(Apron)* **in the Lab**
*******************Work Safely*******************