



**JHARKHAND**  
**Rai University**  
RANCHI

**LAB MANUAL**  
**CHEMISTRY FOR MLT**

## LIST OF EXPERIEMNTS

S.NO.	Name of experiment	Page no.
<b>Unit-I</b>	<b>Quantitative Analysis</b>	
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3	To Prepare and Standardize Sodium Hydroxide Solution by acid – base titration.	
<b>Unit-II</b>	<b>Water Analysis</b>	
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5	To determine the Carbonate Hardness of a given sample of water	
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7	Estimation of Mohr's salt using standard KMnO <sub>4</sub> .	
8	To determine the strength of a given Potassium dichromate solution with N/20sodium thiosulphate (Hypo) solution	
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<b>Unit IV</b>	<b>Gravimetric Practical</b>	
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	Estimation of Barium as Barium Sulphate	
<b>Unit V</b>	<b>Qualitative Analysis</b>	
	Determination of one anion and one cation in a given salt. +Cation- Pb <sup>2+</sup> , Cu <sup>2+</sup> , Al <sup>3+</sup> , Fe <sup>3+</sup> , Mn <sup>2+</sup> , Ni <sup>2+</sup> , Zn <sup>2+</sup> , Co <sup>2+</sup> , Ca <sup>2+</sup> , Sr <sup>2+</sup> , Ba <sup>2+</sup> ,Mg <sup>2+</sup> , NH <sub>4</sub> Anion- (CO <sub>3</sub> ) <sup>2-</sup> , S <sup>2-</sup> , NO <sub>2</sub> <sup>-</sup> , SO <sub>3</sub> <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , NO <sub>3</sub> <sup>-</sup> , Cl <sup>-</sup> , Br <sup>-</sup> , I <sup>-</sup> , PO <sub>4</sub> <sup>3-</sup> , C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> , CH <sub>3</sub> <sup>-</sup>	

## EXPERIMENT NO. 1

Aim: To prepare the standard solution M/10 of oxalic acids.

**Theory:**

Hydrated oxalic acid =  $C_2H_2O_4 \cdot 2H_2O$

Molecular mass of Oxalic Acid = 126.

12.6 g of oxalic acid per liter of the solution should be dissolved to prepare M/10 oxalic acid solution.

On the other hand,

$12.6 / 4 = 3.15$  g of crystals of oxalic acid should be dissolved in water and precisely 250 ml of the solution should be produced.

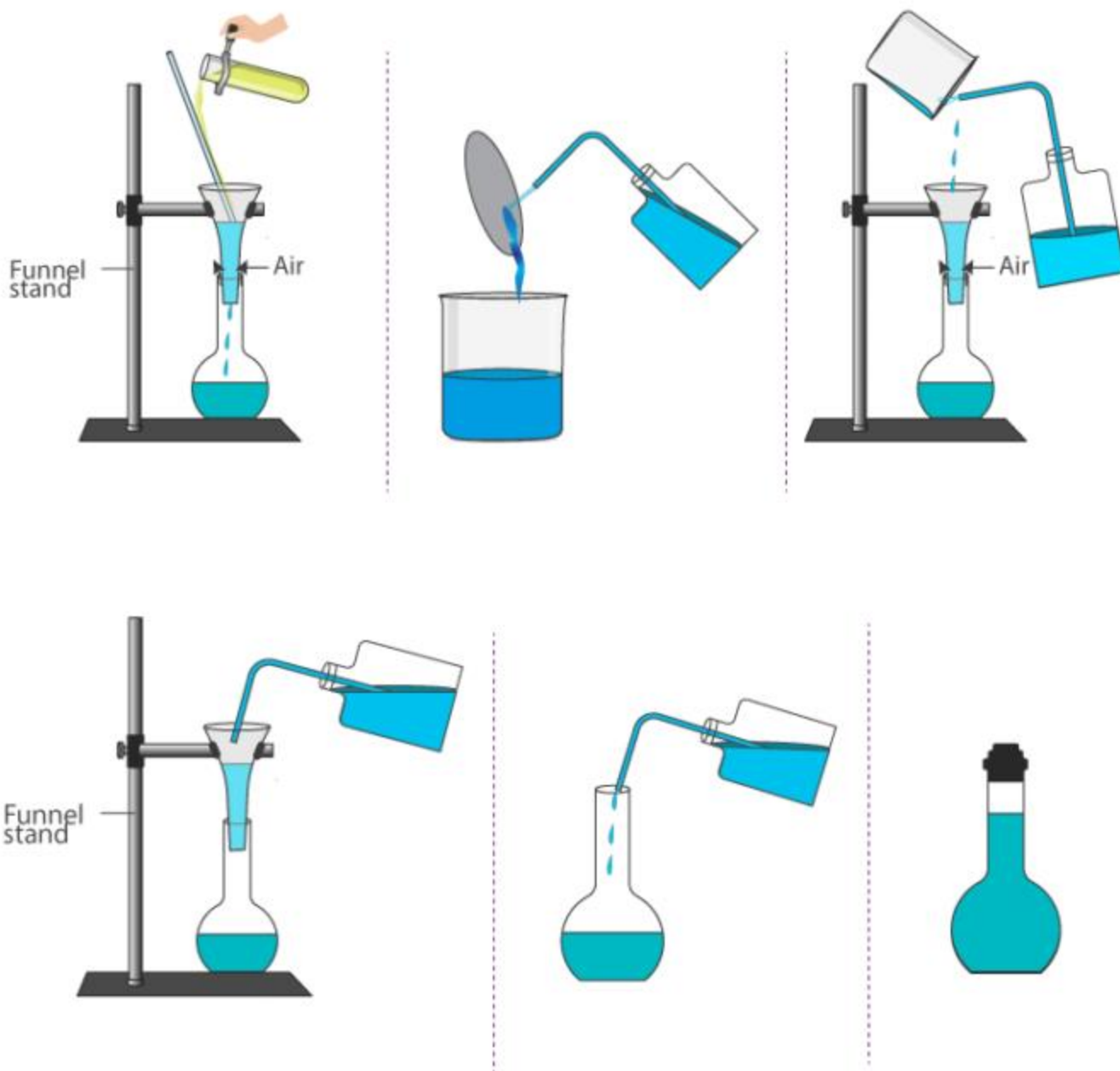
**Apparatus Required:**

1. Chemical balance
2. Watch glass
3. Weight box
4. 250ml beaker
5. Glass rod
6. 250ml measuring flask
7. Wash bottle
8. Weighing tube
9. Funnel
10. Funnel stand
11. Distilled water

**Chemical required:**

1. Distilled water
2. Oxalic acid

## Apparatus Setup:



Step by Step Laboratory Preparation of Standard Solution of Oxalic Acid

**Procedure:**

1. Take a watch glass, wash it with distilled water and dry it.
2. Weigh the exact amount of clean and dried watch glass and record its weight in the notebook.
3. Weigh correctly on the watch glass 3.15 g of oxalic acid and record this weight in the notebook.
4. Using a funnel, transfer oxalic acid softly and carefully from the watch glass into a clean and dry measuring flask.
5. Wash the watch glass with distilled water to move the particles that stick to it into the foam with the assistance of a wash bottle.
6. For this purpose, the volume of distilled water should not exceed 50 ml.
7. Wash funnels several times with distilled water to move the sticking particles into the measuring flask using a wash bottle. Add water in tiny quantities while washing the funnel. The distilled water quantity used for this purpose should not exceed 50 mL.
8. Using a wash bottle, wash the funnel carefully with distilled water to pass the solution attached to the funnel into the measuring flask
9. Turn the flask of measurement until the oxalic acid dissolves.
10. Using a wash bottle, thoroughly add enough distilled water to the measuring flask just below the etched mark on it.
11. Add the last few mL of distilled water drop into the measuring flask until the reduced meniscus level just touches the mark.
12. Put the stopper on the mouth of the flask and shake softly to make the entire solution uniform. Calculate it as a solution of oxalic acid M/10.

**Results:**

250cm<sup>3</sup> of decimolar or (M/10) solution of oxalic acid is prepared.

**Precautions:**

1. Weighing of oxalic acid crystals need weights of 2g + 1g + 100mg + 5mg.
2. Wash the watch glass carefully so that even a single crystal of oxalic acid is not left on the watch glass.
3. Last few drops should be added using a pipette to avoid extra addition of distilled water above the mark on the neck of the measuring cylinder.
4. If it is necessary to titrate oxalic acid or oxalate, add the required dilute H<sub>2</sub>SO<sub>4</sub> amount and heat the flask to 60 °-70 ° C.

## EXPERIMENT NO. 2

**Aim:** To prepare the 250 cm<sup>3</sup> of N/10 standard solution of sodium carbonate.

**Theory:** Sodium carbonate is essentially insoluble in nearly saturated sodium hydroxide. The insoluble sodium carbonate will settle to the bottom of the container after the saturated NaOH has equilibrated for a couple of days. The supernatant can be withdrawn carefully to prepare a diluted NaOH solution free of sodium carbonate. The water for preparing NaOH standard solution should be boiled to remove any dissolved CO<sub>2</sub>, because dissolved CO<sub>2</sub> can cause a titration error. To prepare the standard solution of sodium carbonate, the equivalent weight of sodium carbonate =

$$h=2=106/2=53$$

1000cm<sup>3</sup> of normal sodium carbonate solution requires 53g of sodium carbonate. Therefore, 250cm<sup>3</sup> of 10

Sodium carbonate requires

$$53/1000 \times 250 = 1.325$$

### **Apparatus Required:**

1. Chemical balance
2. Watch glass
3. Weight box
4. 250ml beaker
5. Glass rod
6. 250ml measuring flask
7. Wash bottle
8. Weighing tube
9. Funnel
10. Funnel stand

### **Chemical required:**

1. Distilled water
2. Sodium Carbonate

**Procedure:**

1. Take a watch glass, wash it with distilled water and dry it.
2. Weigh the exact amount of clean and dried watch glass and record its weight in the notebook.
3. Weigh correctly on the watch glass with 1.325 g of sodium carbonate and record this weight in the notebook.
4. Using a funnel, transfer sodium carbonate softly and carefully from the watch glass into a clean and dry measuring flask.
5. Wash the watch glass with distilled water to move the particles that stick to it into the foam with the assistance of a wash bottle.
6. For this purpose, the volume of distilled water should not exceed 50 ml.
7. Wash funnels several times with distilled water to move the sticking particles into the measuring flask using a wash bottle. Add water in tiny quantities while washing the funnel. The distilled water quantity used for this purpose should not exceed 50 mL.
8. Using a wash bottle, wash the funnel carefully with distilled water to pass the solution attached to the funnel into the measuring flask
9. Turn the flask of measurement until the sodium carbonate dissolves.
10. Using a wash bottle, thoroughly add enough distilled water to the measuring flask just below the etched mark on it.
11. Add the last few mL of distilled water drop into the measuring flask until the reduced meniscus level just touches the mark.
12. Put the stopper on the mouth of the flask and shake softly to make the entire solution uniform. Calculate it as a solution of sodium carbonate N/10.

**Observations:**

Weight of the watch glass	W1g
Weight of the watch glass + Sodium carbonate	W1 + 1.325 g
Weight of Sodium carbonate	1.325 g
Volume of distilled water	250 cm <sup>3</sup>

**Results and Discussion:**

250cm<sup>3</sup> of decimolar or N/10 standard solution of sodium carbonate is prepared.

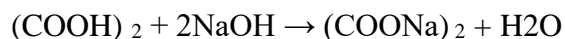
**Precautions:**

1. While weighing do not spill the substance on the balance pan
2. While preparing the laboratory reagent apron should be used.
3. Bases are highly corrosive so they should be handled with extreme care.
4. Watch glass must be dry.
5. Funnel should be washed thoroughly several times.
6. Shaking of solution should be done thoroughly so that it may become uniform.
7. While making the solution the solvent should be added such that the lower meniscus of the solvent must be on the mark of the measuring flask.
8. Last few drops should be added using a pipette to avoid extra addition of distilled water above the mark on the neck of the measuring cylinder.

### EXPERIMENT NO. 3

**Aim:** To Prepare and Standardize Sodium Hydroxide Solution by acid – base titration.

**Theory:** Standardization of sodium hydroxide is done by acid base titration. When oxalic acid is allowed to react with sodium hydroxide, sodium oxalate and water are obtained. In this titration, to locate the end point phenolphthalein indicator is used. Appearance of the pale pink colour which persists for 30 sec is the endpoint.



#### **Apparatus Required:**

Conical flask,

Burette,

Pipette,

Standard flask

#### **Chemical required:**

NaOH,

Oxalic acid,

Phenolphthalein

#### **PROCEDURE:**

Preparation:

1.4 gm of NaOH is accurately weighed and transferred in 1000 ml volumetric flask. The volume is made up to mark with distilled water to get 0.1 N solution of NaOH Weigh accurately.

2.0.63 g of oxalic acid into a 100 ml volumetric flask and make up to 100 ml volume with distilled water.

3. Pipette out 20 ml solution and titrated 0.1 NaOH using phenolphthalein as indicator.

4. Continue the titration to get the concordant value.

$$N_1 V_1 = N_2 V_2$$

$$N_2 = N_1 V_1 / V_2$$

**Observation:**

Sl. NO	Content of the Flask	Burette Reading (ml)				Indicator
		Initial reading	Final reading	Difference	Precision	
1	20ml				19.8	Phenolphthalein
2	20ml			19.8		Phenolphthalein
3	20ml			19.8		Phenolphthalein

**Calculation:**

$$N_1 V_1 = N_2 V_2$$

$N_1 = 0.1$  N = Normality of Oxalic acid Solution,

$N_2 = ?$  = Normality of NaOH

$V_1 = 20$  ml = Volume of Oxalic acid Solution,

$V_2 = 19.8$  ml = Volume of NaOH

$$N_2 = N_1 V_1 / V_2$$

$$N_2 = 20 \text{ ml} \times 0.1 / 19.8 \quad N_2 = 0.10 \text{ N}$$

**Result:**

The strength of the given solution of sodium hydroxide is...0.10 N.

**Precautions:**

1. Distilled water was used for washing and rinsing of glass apparatus.
2. Measurements is done with accuracy.
3. Same amount of indicator was added each time.
4. . End point was observed carefully

## **EXPERIMENT NO. 4**

**Aim:** To determine the Non-Carbonate Hardness of a given sample of water.

Theory: To determine permanent water hardness in a laboratory, you first need to remove temporary hardness by boiling a water sample and then titrate it with EDTA (ethylenediaminetetra acetic acid) using Eriochrome Black T as an indicator. The volume of EDTA used to reach the endpoint (wine-red to blue color change) is then used to calculate the permanent hardness.

### **Apparatus required:**

Burette

Glass funnel

Pipette

Flask Dropper

Measuring cylinder

Conical flask

Filter paper

### **Chemical required:**

EDTA,

Eriochrome Black-T,

NH<sub>2</sub>CL,

Ammonia Buffer,

Magnesium Carbonate,

90% ethyl alcohol,

Distilled water.

Water sample

### **Procedure:**

**Remove Temporary Hardness:**

1. Boil a known volume of the water sample (e.g., 100 ml) until approximately half the volume remains.
2. This boiling process precipitates calcium and magnesium carbonates, which cause temporary hardness.
3. Cool the solution and filter it to remove the precipitated carbonates.
4. Make up the volume of the filtrate to the original volume (100 ml) with distilled water.

**Titration:**

1. The burette is filled with standard EDTA solution to the zero level.
2. Take 50ml of the treated (boiled) sample water in flask. If sample having high Calcium content then take smaller volume and dilute to 50ml.
3. Add 1ml Ammonia buffer.
4. Add 5 to 6 drop of Ericrome black – T indicator. The solution turns into wine red colour.
5. Note the initial reading.
6. Titrate the content against EDTA solution. At the end point colour change from wine red to blue colour.
7. Note the final reading and record it. Repeat the process till we get concordant value. 8. Take 50ml sample in another flask and boiled it. (Add distilled water to get final volume of water.)
9. Repeat step 3-7.

**Calculations:**

S.No	Water sample (50ml)	Initial Value	Final Value	MI of EDTA
1				x
2				
3				

Permanent Hardness (mg/L as CaCO<sub>3</sub>) = (Volume of EDTA used x Molarity of EDTA x 100,000) / Volume of water sample (in ml)

**Result:** Permanent Hardness (mg/L as CaCO<sub>3</sub>)=.....

**Precaution:**

1. Distilled water was used for washing and rinsing of glass apparatus
2. Same amount of indicator was added each time
3. pH 10 was maintained during the titration by adding buffer
4. End point was observed carefully

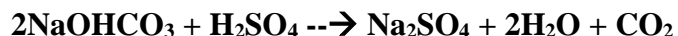
## EXPERIMENT NO. 5

**Aim:** To determine the Carbonate Hardness of a given sample of water.

**Theory:** Carbonate and bicarbonate ions in the sample can be determined by titrating it with against slandered sulphuric acid ( $\text{H}_2\text{SO}_4$ ) using phenolphthalein and methyl orange as indicators. Addition of phenolphthalein gives pink red colour in presence of carbonates and titration with  $\text{H}_2\text{SO}_4$  convert  $\text{CO}_3^{2-}$  into  $\text{HCO}_3^-$  and decolorise the red colour as shown below



Thus the carbonate neutralization is only half way these carbonate along with already present ones are then determined by continuing the titration using methyl orange indicator which gives yellow colour in presence of bicarbonate. On complete neutralization of bicarbonate the yellow colour will change to red.



The bicarbonate titre value will be less since carbonate is not present.

### **Apparatus required:**

Burette

Glass funnel

Pipette

Flask Dropper

Measuring cylinder

Conical flask

### **Chemical required:**

Sulphric acid (.1N)

Water sample

Phenol naphthalene

Methyl orange

### **Procedure:**

1. Pipette out 10 ml of sample water in conical flask.
2. Add 1 or 2 drop of phenol naphthalene, the solution will turn pink colour
3. Titrate the contents against the 0.1N H<sub>2</sub>SO<sub>4</sub> filled in burette, the end point is indicated by change in pink colour to colourless. Record the burette reading.
4. If on addition of phenol naphthalene the sample does not change to pink, it indicates the absence of CO<sub>3</sub><sup>2-</sup>.
5. In such case add methyl orange indicator in the same aliquot and titrate against the 0.1N H<sub>2</sub>SO<sub>4</sub> till the straw yellow colour changes to rose red.
6. Note the burette reading.

**Table:**

S.N.	Volume of H <sub>2</sub> SO <sub>4</sub> consumed for neutralization of CO <sub>3</sub> <sup>2-</sup> (ml)	Volume of H <sub>2</sub> SO <sub>4</sub> consumed for neutralization of HCO <sub>3</sub> <sup>-</sup> (ml)	meq/l HCO <sub>3</sub> <sup>-</sup>	meq/l CO <sub>3</sub> <sup>2-</sup>
1				
2				
3				
4				

**Calculation:**

Amount of 0.1N H<sub>2</sub>SO<sub>4</sub> consumed in 1<sup>st</sup> titration = A ml

Amount of 0.1N H<sub>2</sub>SO<sub>4</sub> consumed in 2<sup>nd</sup> titration = B ml

Amount of carbonate ion meq/l = 20 x A

Amount of bicarbonate ion meq/l = 20x (A-B)

**Result:**

The carbonate and bicarbonate in given water ----- meq/l

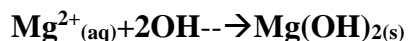
**Precautions:**

1. Distilled water was used for washing and rinsing of glass apparatus.
2. Same amount of indicator was added each time.
3. pH 10 was maintained during the titration by adding buffer .
4. End point was observed carefully.

## EXPERIMENT NO. 1

**Aim:** To determine the total Hardness of a given sample of water by EDTA method.

**Theory:** Hardness in water is due to the presence of dissolved salts of calcium and magnesium. Hardness is caused by polyvalent metallic cations, though the divalent cations, such as calcium and magnesium cations are usually the predominant cause of hardness. In addition, hardness is also caused by Fe<sup>2+</sup> and Mn<sup>2+</sup> ions. For example, when hard water is heated, Ca<sup>2+</sup> ions react with bicarbonate (HCO<sub>3</sub><sup>-</sup>) ions to form insoluble calcium carbonate (CaCO<sub>3</sub>).



Depends on ability of ethylenediamine tetra acetic acid (C<sub>10</sub>H<sub>16</sub>O<sub>8</sub>N<sub>2</sub>) or its disodium salt to form stable complexes with calcium and magnesium ions. When the dye eriochrome black T (EBT) (C<sub>20</sub>H<sub>13</sub>N<sub>3</sub>O<sub>7</sub>S) is added to solution containing calcium and magnesium ions at pH 10.0, a Wine red complex is formed, this solution is titrated with standard solution of disodium salt of EDTA, which extracts calcium and magnesium from the dye complex and the dye is changed back to its original blue colour. Eriochrome black T is used to indicate the end point for the titration of calcium and magnesium together.

### **Apparatus required:**

Burette 25-30ml

Glass funnel

Pipette 1ml

Flask Dropper

Measuring cylinder

Conical flask

### **Chemical required:**

EDTA,

Eriochrome Black-T,

NH<sub>2</sub>CL,

Ammonia Buffer,

Magnesium Carbonate,

90% ethyl alcohol,

Distilled water.

Water sample

**Reagent preparation:**

1. EDTA solution: 4gm EDTA and 0.1gm magnesium bicarbonate dissolve in 800 ml distilled water.

2. Eriochrome Black-T: 0.4gm Eriochrome Black T, 4.5 gm hydroxylmine hydrochloride add in 100ml 95% ethyl alcohol.

3. Ammonia Buffer:

Stock A: 16.9gm of NH<sub>4</sub>CL in 143ml of conc. NH<sub>4</sub>OH,

Stock B: 1.25gm magnesium salt of EDTA dissolve in 50 ml distilled water. Mix both stock solutions and dilute to 250ml with DDW. Dilute 10ml of the solution to 100ml with DDW.

**Procedure:**

1. The burette is filled with standard EDTA solution to the zero level.

2. Take 50ml sample water in flask. If sample having high Calcium content then take smaller volume and dilute to 50ml.

3. Add 1ml Ammonia buffer.

4. Add 5 to 6 drop of Eriochrome black – T indicator. The solution turns into wine red colour.

5. Note the initial reading.

6. Titrate the content against EDTA solution. At the end point colour change from wine red to blue colour.

7. Note the final reading and record it. Repeat the process till we get concordant value. 8. Take 50ml sample in another flask and boiled it. (Add distilled water to get final volume of water.)

9. Repeat step 3-7.

**Calculations:**

S.No	Water sample (50ml)	Initial Value	Final Value	MI of EDTA
1				x
2				
3				

Total hardness of water mg/L (CaCO<sub>3</sub> Scale) = ml of EDTA used (unboiled) \*1000 /ml of sample

Or,

$$\text{Hardness (in mg/L as CaCO}_3) = (V \times N \times 50 \times 1000) / (SV)$$

Where:

V = volume of titrant (mL); N = normality of EDTA; 50 = equivalent weight of CaCO<sub>3</sub>; SV = sample volume (mL)

Permanent hardness of water mg/L (CaCO<sub>3</sub> Scale) = ml of EDTA used (boiled) \*1000/ml of sample

Temporary hardness of water mg/L (CaCO<sub>3</sub> Scale) = Total hardness of water - Permanent hardness of water

1000 ml of 1(M) EDTA  $\equiv$  100 gm CaCO<sub>3</sub>

1 ml of 1(M) EDTA  $\equiv$  (100/1000) gm CaCO<sub>3</sub>

1 ml of 1(M/50) EDTA  $\equiv$  (100/1000)  $\times$  (1/50) gm

CaCO<sub>3</sub>  $\equiv$  (100/1000)  $\times$  (1/50)  $\times$  1000 mg CaCO<sub>3</sub>  $\equiv$  2 mg CaCO<sub>3</sub>

x ml of EDTA required for titration So,

x ml (M/50) EDTA  $\equiv$  2  $\times$  x = 2x mg CaCO<sub>3</sub>

Now Volume of water taken = 50 ml so,

50 ml water contains = 2xmg CaCO<sub>3</sub>

1000 ml water contains =  $[2x/50 \times 1000]$  mg CaCO<sub>3</sub> = y ppm or mg/l

**Observation:** The colour of soluble distilled water and R.O water instantly changed into blue while tap water and pond water turned wine red when Ericrome black T was added and therefore after turned blue when titrated against EDTA solution.

**Precautions:**

1. Distilled water was used for washing and rinsing of glass apparatus.
2. Same amount of indicator was added each time.
3. pH 10 was maintained during the titration by adding buffer .
4. End point was observed carefully.

## EXPERIMENT NO. 1

**Aim:** Estimation of Mohr's salt using standard  $\text{KMnO}_4$ .

**Theory:** Potassium permanganate is a strong oxidant in the presence of sulfuric acid. Mohr salt is a double salt forming a single crystalline structure having the formula  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ . The chemical name for Mohr's salt is ferrous ammonium sulfate.

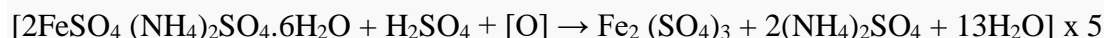
Mohr salt acts as a reducing agent and potassium permanganate acts as an oxidizing agent. Acid Base Titration with Mohr's Salt against  $\text{KMnO}_4$  is an example of redox reaction. In this redox reaction, ferrous ion from Mohr's salt gets oxidized and pink colored of manganese present in potassium permanganate, which is in the +7-oxidation state gets reduced to colorless  $\text{Mn}^{2+}$  state.

The chemical reaction and the molecular chemical equation is given below.

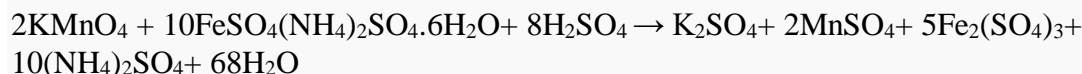
Reduction half reaction –



Oxidation half reaction –



Overall reaction –



### **Apparatus required:**

1. Burette
2. Conical Flask
3. Beakers
4. Pipette
5. Funnel
6. Retort Stand with Clamp
7. Measuring flask

## 8. Volumetric flask

### Chemical Required:

1. Mohr's salt
2. Potassium permanganate
3. Sulfuric acid

### Procedure:

Preparation of 250ml of M/20 solution of Mohr's salt:

1. The molar mass of Mohr's salt is  $392\text{g mol}^{-1}$ . It is a primary standard. Since  $1000\text{cm}^3$  of 1M potassium permanganate require Mohr's salt of  $=392\text{g}$  So,  $250\text{cm}^3$  of M/20 potassium permanganate require Mohr's salt of  $= (392 / 20) / 1000 \times 250 = 4.9\text{g}$
2. Accurately weigh 4.9g of Mohr's salt using a chemical balance and watch glass.
3. Now put weighed Mohr's salt in a volumetric flask using a funnel.
4. Now add 5ml of dilute sulfuric acid and distilled water in the same flask and dissolve Mohr's salt.
5. Now fill the volumetric flask with distilled water according to the required volume.

### Titration

1. Wash the pipette and the burette with distilled water and then rinse it with the corresponding solution which needs to be filled in them.
2. Rinse the burette with potassium permanganate solution and then fill the potassium permanganate solution in it.
3. Fix the burette in the burette stand and then place a white tile beneath it so that the endpoint can easily be traced.
4. Rinse the pipette as well as the conical flask with Mohr's salt.
5. Take out 10 ml of standard more mohr salt solution in the conical flask.
6. Add 1 + 2 full of sulfuric acid so as to prevent the oxidation of manganese to form manganese dioxide.
7. Take the initial reading of the burette before starting that experiment.
8. Now against the potassium permanganate solution start the titration and slowly still the flask gently.

9. Initially, the purple colour of  $\text{KMnO}_4$  will be discharged with ferrous ammonium sulfate. The appearance of the permanent pink colour indicates the endpoint. Repeat the experiment unless 3 concordant value is obtained.
10. It is important to note down the upper meniscus reading of the burette

**Observation Table:**

S.N.	Vol. of Mohr's salt solution(ml)	Strength of $\text{KMnO}_4$ (N)	Burette reading of $\text{KMnO}_4$ (ml)			concordant value
			Initial (ml)	Final (ml)	Difference	
1						
2						
3						
4						

**Calculation:**

Strength of  $\text{Fe}^{2+}$  in Mohr's salt solution: Volume of  $\text{KMnO}_4$  solution required for Mohr's salt solution = a ml = .....ml.

Strength of  $\text{KMnO}_4$  solution = y (N) = ..... (N).

a ml of y(N)  $\text{KMnO}_4$  solution  $\equiv 55.85 \times a \times y / 1000$  gm of  $\text{Fe}^{2+}$

$\equiv z$  gm of  $\text{Fe}^{2+}$  = .....gm.

1000ml of Mohr's salt contain =  $z \times 1000 / 5$  gm of  $\text{Fe}^{2+}$  = ..... gm. = w gm of  $\text{Fe}^{2+}$   
 Amount of  $\text{Fe}^{2+}$  = w gm / lit = ..... gm./ lit

**Conclusion:**

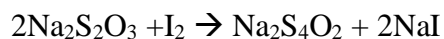
The amount of  $\text{Fe}^{2+}$  estimated in the supplied Mohr's salt solution is ..... gms. /lit at ..... $^{\circ}\text{C}$ .

## EXPERIMENT NO. 1

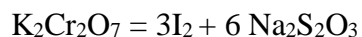
**Aim:** To determine the strength of a given Potassium dichromate solution with N/20 sodium thiosulphate (Hypo) solution

**Theory:** This is an iodometric titration, where liberated iodine is titrated with thiosulphate. The reaction involves potassium dichromate oxidizing iodide ions to iodine, which is then reduced by thiosulphate. Potassium dichromate is a strong oxidizing agent. In acidic medium, it liberates iodine from potassium iodide (KI). The liberated iodine is then titrated against standard sodium thiosulphate solution.

Reaction involve:



Hence



Apparatus required:

Burette

Pipette (25 mL)

Conical flask

Measuring cylinder

Beaker

Funnel

**Chemicals required:**

Given potassium dichromate solution

N/20 sodium thiosulphate solution

Potassium iodide (KI)

Dilute sulphuric acid or hydrochloric acid<sup>5</sup>. \* Freshly prepared starch solution (indicator)

Distilled water

**Procedure:**

1. Rinse and fill the burette with N/20 sodium thiosulphate solution.
2. Pipette out 25.0 mL of the given potassium dichromate solution into a clean conical flask.
3. Add about 10 mL of 10% KI solution and 5–10 mL of dilute H<sub>2</sub>SO<sub>4</sub> to the conical flask.
4. Allow the solution to stand for 1–2 minutes in the dark (to ensure complete liberation of iodine).
5. Titrate the liberated iodine with sodium thiosulphate solution from the burette.
6. When the solution becomes pale yellow, add 2–3 mL of starch solution (the solution turns deep blue).
7. Continue titrating until the blue color just disappears (end point).
8. Note the burette reading.
9. Repeat the experiment to obtain concordant value (i.e., at least 3 consistent readings).

**Observations Table:**

S.No	Volume of Dichromate (mL)	Burette Reading (ml)			Concordant reading
		Initial Reading (mL)	Final Reading (mL)	Difference	
1	25.0				
2	25.0				
3	25.0				

**Calculations:**

Let the average volume of sodium thiosulphate used = V mL

Normality of sodium thiosulphate ( $N_1$ ) =  $1/20 = 0.05$  N

Volume of sodium thiosulphate = V mL

Volume of potassium dichromate = 25.0 mL

Normality of potassium dichromate =  $N_2$

$$N_1V_1 = N_2V_2 \Rightarrow 0.05 \times V = N_2 \times 25$$

$$\Rightarrow N_2 = 0.05 \times V/25$$

Strength (g/L) = Normality  $\times$  Equivalent weight of  $K_2Cr_2O_7$

Equivalent weight of  $K_2Cr_2O_7 = 49.03$  g/eq

So,

$$\text{Strength} = N_2 \times 49.03 = 0.05 \times V/25 \times 49.03$$

**Result:**

The strength of the given potassium dichromate solution is= ..... g/L

**Precautions:**

Use freshly prepared starch solution.

Perform titration immediately after adding KI to prevent iodine loss.

Rinse the pipette, burette, and conical flask properly before use.

Avoid direct sunlight during titration (iodine is volatile).

Use white tile to observe the color change clearly.

**Aim:** Preparation of Buffer Solution and Standardization of pH Meter

**Theory:** A buffer solution resists changes in pH when small amounts of acid or base are added. It usually consists of:

Type of Buffer solution

A weak acid and its salt (acidic buffer)

A weak base and its salt (basic buffer)

Examples:

Acidic buffer: Acetic acid + Sodium acetate

Basic buffer: Ammonium hydroxide + Ammonium chloride

pH Meter Standardization:

A pH meter must be standardized before use to ensure accurate pH measurements. It is calibrated using standard buffer solutions of known pH (commonly pH 4.00, 7.00, and 9.20 or 10.00).

**Apparatus required:**

Beakers (100 mL, 250 mL)

Volumetric flasks

Measuring cylinder

Pipettes

Glass rod

pH meter with electrode

Distilled water

**Chemicals required:**

Glacial acetic acid

Sodium acetate

Ammonium hydroxide

Ammonium chloride

Standard buffer tablets/solutions (pH 4.00, 7.00, 10.00)

Distilled water

**Procedure:**

**A. Preparation of Buffer Solutions**

**Acidic Buffer (pH ~4.5):**

1. Take 25 mL of 0.2 M acetic acid in a beaker.
2. Add 25 mL of 0.2 M sodium acetate.
3. Mix thoroughly and transfer to a 100 mL volumetric flask.
4. Make up the volume to 100 mL with distilled water.

**Basic Buffer (pH ~9.2):**

1. Take 25 mL of 0.2 M ammonium hydroxide.
2. Add 25 mL of 0.2 M ammonium chloride.

Mix and make up to 100 mL in a volumetric flask with distilled water.

**B. Standardization of pH Meter**

**Procedure:**

1. Switch ON the pH meter and allow it to warm up (if necessary).
2. Rinse the electrode with distilled water and blot dry with tissue (don't rub).

3. Immerse the electrode in pH 7.00 buffer solution.
4. Adjust the meter reading to 7.00 using the calibration knob (or auto-calibrate, if digital).
5. Rinse the electrode again and immerse it in pH 4.00 or pH 10.00 buffer.
6. Adjust the second calibration knob if available (slope).
7. Rinse and store electrode in electrode storage solution or distilled water after use.

**Observations Table:**

A. Buffer Preparation Table:

Type of Buffer	Chemicals Used	Volume Taken	Final Volume
Acidic Buffer	0.2 M Acetic Acid + Sodium Acetate	25 mL + 25 mL	100 mL
Basic Buffer	0.2 M NH <sub>4</sub> OH + NH <sub>4</sub> Cl	25 mL + 25 mL	100 mL

B. pH Meter Calibration Table:

Standard Buffer Used	Actual pH	pH Meter Reading	Adjustment Done
pH 7.00	7.00		Yes/No
pH 4.00 or 10.00	4.00/10.00		Yes/No

**Result:**

Buffer solutions were prepared successfully.

The pH meter was standardized using pH 7.00 and pH 4.00/10.00 buffer solutions.

The meter is now ready for accurate pH measurements.

**Precautions:**

Use freshly prepared standard buffer solutions or certified buffer tablets.

Always rinse the electrode with distilled water before and after immersion.

Do not touch the glass bulb of the pH electrode.

Buffers and solutions should be at room temperature for accurate calibration.

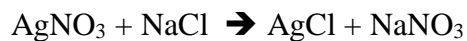
Calibrate the meter at least twice with pH 7 and either 4 or 10.

## EXPERIMENT NO. 1

**Aim:** To Determine the Strength of Silver Ions ( $\text{Ag}^+$ ) in a Given  $\text{AgNO}_3$  Solution Using Chloride Ions (Mohr's Method)

**Theory:** This is a precipitation titration method called Mohr's method. It is based on the formation of a white precipitate of silver chloride ( $\text{AgCl}$ ) when silver nitrate reacts with sodium chloride. Once all  $\text{Ag}^+$  has reacted, the first excess of  $\text{Cl}^-$  reacts with chromate ions to form a \*\*reddish-brown precipitate of silver chromate ( $\text{Ag}_2\text{CrO}_4$ ), indicating the end point.

Titration reaction



End point reaction:



### **Apparatus required:**

Burette

Pipette (25 mL)

Conical flask

Measuring cylinder

Beakers

Funnel

### **Chemicals required:**

Given  $\text{AgNO}_3$  solution (unknown concentration)

Standard  $\text{NaCl}$  solution (e.g., N/50 or 0.02 N)

Potassium chromate indicator ( $\text{K}_2\text{CrO}_4$ )

Distilled water

**Procedure:**

Rinse and fill the burette with standard NaCl solution.

Pipette out 25.0 mL of the Ag NO<sub>3</sub> solution into a conical flask.

Add 1–2 mL of potassium chromate solution as the indicator (solution turns yellow).

Titrate slowly with NaCl solution from the burette.

Near the endpoint, a reddish-brown precipitate of Ag<sub>2</sub>CrO<sub>4</sub> appears and persists (does not disappear on shaking).

Note the burette reading.

Repeat the titration to obtain concordant readings (at least 2-3 consistent values).

**Observations Table:**

S.No	Volume of AgNO <sub>3</sub> (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume of NaCl used (mL)
<b>1</b>	25.0			
<b>2</b>	25.0			
<b>3</b>	25.0			

Calculations

Let the average volume of NaCl used = V mL

Normality of NaCl (N<sub>1</sub>) = e.g., 0.02 N

Volume of NaCl = V mL

Volume of AgNO<sub>3</sub> = 25.0 mL

Normality of AgNO<sub>3</sub> = N<sub>2</sub>

$N_1 \times V = N_2 \times 25 \rightarrow N_2 = N_1 \times V / 25$

Strength (g/L) = Normality  $\times$  Equivalent weight of AgNO<sub>3</sub>

Equivalent weight of AgNO<sub>3</sub> = Molar mass = 169.87 g/mol (since it gives 1 Ag<sup>+</sup> ion per mole)

Strength = N<sub>2</sub>  $\times$  169.87 = N<sub>1</sub> $\times$ V/25  $\times$  169.87

**Result:**

The strength of silver ions (Ag<sup>+</sup>) in the given AgNO<sub>3</sub> solution is = ..... g/L

**Precautions:**

Perform the titration in neutral to slightly alkaline medium (Mohr's method is pH-sensitive).

Use freshly prepared potassium chromate indicator.

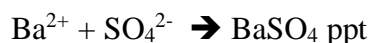
Rinse the pipette and burette properly before use.

The reddish-brown Ag<sub>2</sub>CrO<sub>4</sub> precipitate should appear gradually and persist.

Avoid over titration; stop at first permanent pink/brown color.

Aim: Estimation of Barium as Barium Sulphate (Gravimetric Method)

**Theory:** This is a gravimetric analysis method. Barium ions (Ba<sup>2+</sup>) are quantitatively precipitated from a barium salt solution (like BaCl<sub>2</sub>) by adding excess sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) or sulphate ions (e.g., from H<sub>2</sub>SO<sub>4</sub> or Na<sub>2</sub>SO<sub>4</sub>) to form barium sulphate (BaSO<sub>4</sub>), a white insoluble precipitate.



The precipitate is filtered, washed to remove impurities, dried, ignited (if necessary), and weighed. From the mass of  $\text{BaSO}_4$ , the amount of  $\text{Ba}^{2+}$  can be calculated using stoichiometry.

**Apparatus required:**

Beakers

Glass rod

Measuring cylinder

Funnel

Filter paper (Whatman No. 42 or equivalent)

Gooch crucible or sintered glass crucible (or porcelain crucible)

Balance

Oven and desiccator

**Chemicals required:**

Barium chloride ( $\text{BaCl}_2$ ) solution (given sample)

Dilute sulphuric acid ( $\text{H}_2\text{SO}_4$ ) or sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) solution

Distilled water

**Procedure:**

Take 50 mL of the barium chloride solution in a beaker.

Heat the solution to about 60–70°C.

Slowly add dilute sulphuric acid (or  $\text{Na}_2\text{SO}_4$  solution) dropwise with constant stirring until precipitation is complete.

Allow the precipitate of  $\text{BaSO}_4$  to settle.

Digest the precipitate for 30 minutes on a water bath or low flame to ensure complete and compact precipitation.

Filter the precipitate using pre-weighed filter paper or crucible.

Wash the precipitate several times with **hot distilled water** to remove chloride/sulphate ions (test the washings with  $\text{AgNO}_3$  for  $\text{Cl}^-$  or  $\text{BaCl}_2$  for  $\text{SO}_4^{2-}$ ).

Dry the filter paper with precipitate in an oven at **110°C** until constant weight is obtained.

Cool in a desiccator and weigh accurately.

Record the mass of  $\text{BaSO}_4$  obtained.

### Observations and Calculations:

Description	Mass (g)
Weight of filter paper/crucible ( $W_1$ )	
Weight of filter paper + $\text{BaSO}_4$ ( $W_2$ )	
Mass of $\text{BaSO}_4 = W_2 - W_1$	

Molar Masses:

Molar mass of  $\text{BaSO}_4 = 233.39 \text{ g/mol}$

Molar mass of  $\text{Ba} = 137.33 \text{ g/mol}$

Amount of  $\text{Ba} = 137.33 / 233.39 \times$  times mass of  $\text{BaSO}_4$

Strength of  $\text{Ba}^{2+}$  in g/L (if solution volume used =  $V \text{ mL}$ ):

Strength (g/L) = mass of  $\text{Ba}$  in g/  $V \times 1000$

### Result:

Mass of Barium Sulphate ( $\text{BaSO}_4$ ) obtained = ..... g

Amount of Barium ( $\text{Ba}^{2+}$ ) present in the given solution = ..... g

Strength of  $\text{Ba}^{2+}$  in the sample solution = ..... g/L

### Precautions:

Add sulphuric acid slowly to avoid super-saturation and colloidal precipitate.

Ensure complete precipitation by allowing the mixture to stand or digest.

Use hot water for washing to avoid dissolution of  $\text{BaSO}_4$ .

Dry the precipitate completely to obtain accurate weight.

Handle the filter paper carefully to avoid tearing and loss of precipitate.

**Aim:** Qualitative Analysis of One Cation and One Anion in a given salt:

**Theory:**

Qualitative analysis is based on the selective precipitation and characteristic reactions of ions. The analysis involves:

Preliminary tests (physical properties, solubility, flame test, etc.)

Systematic group analysis for cations

Confirmatory tests for anions

**Cations and Anions under Study**

**Cations**

$\text{Pb}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$

**Anions**

$\text{CO}_3^{2-}$ ,  $\text{S}^{2-}$ ,  $\text{NO}_2^-$ ,  $\text{SO}_3^{2-}$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{PO}_4^{3-}$ ,  $\text{C}_2\text{O}_4^{2-}$ ,  $\text{CH}_3\text{COO}^-$

**Apparatus required:**

Test tubes, test tube holder, dropper

Glass rod, beaker, burner

Filter paper, watch glass

**Chemicals required:**

Dil. and conc. HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, NaOH, NH<sub>4</sub>OH, BaCl<sub>2</sub>, AgNO<sub>3</sub>, KMnO<sub>4</sub>, Nessler's reagent, etc.

Group reagents: HCl, H<sub>2</sub>S, NH<sub>4</sub>Cl, NH<sub>4</sub>OH, Na<sub>2</sub>CO<sub>3</sub>

Known reagents for confirmatory tests

**Procedure**

**Step A: Preliminary Examination**

1. Physical appearance – Note the color, smell, and texture of the salt.
2. Flame test (for cations like Ba<sup>2+</sup>, Sr<sup>2+</sup>, Ca<sup>2+</sup>) – Observe flame color.
3. Dry heating test – Heat a small quantity of salt in a dry test tube:  
Observe evolution of gases or color change.

**Step B: Detection of Anion (Acid Radicals)**

1. Prepare sodium carbonate extract:

Boil 1g salt with 10 mL distilled water + sodium carbonate.

Filter and collect the filtrate for anion testing.

2. Perform group tests for anions:

Add dil. HCl: Effervescence → CO<sub>3</sub><sup>2-</sup>

Add BaCl<sub>2</sub> solution: White ppt → SO<sub>4</sub><sup>2-</sup> (insoluble in HCl), SO<sub>3</sub><sup>2-</sup> (soluble in HCl)

Add AgNO<sub>3</sub>: White ppt → Cl<sup>-</sup>, Yellow ppt → I<sup>-</sup>

Add FeSO<sub>4</sub> + conc. H<sub>2</sub>SO: Brown ring → NO<sub>3</sub><sup>-</sup>

Add KMnO<sub>4</sub> : Decolorization → S<sup>2-</sup>, SO<sub>3</sub><sup>2-</sup>, NO<sub>2</sub><sup>-</sup>, C<sub>2</sub>O<sub>4</sub><sup>2-</sup>

Add ammonium molybdate + HNO<sub>3</sub>: Yellow ppt → PO<sub>4</sub><sup>3-</sup>

Add ferric chloride: Blood red color → C<sub>2</sub>O<sub>4</sub><sup>2-</sup> or CH<sub>3</sub>COO<sup>-</sup>

3. Confirmatory tests based on suspected anion.

### Step C: Detection of Cation (Basic Radicals)

1. Prepare original solution:

Dissolve a pinch of salt in distilled water or dil. HCl.

2. Group-wise cation analysis:

Group	Group Reagent	Radicals Detected
I	Dil. HCl	Pb <sup>2+</sup> , Ag <sup>+</sup>
II	H <sub>2</sub> S in acidic medium	Cu <sup>2+</sup> , Pb <sup>2+</sup>
III	NH <sub>4</sub> Cl + NH <sub>4</sub> OH + H <sub>2</sub> S	Al <sup>3+</sup> , Fe <sup>3+</sup> , Mn <sup>2+</sup> , Cr <sup>3+</sup>
IV	NH <sub>4</sub> OH + H <sub>2</sub> S	Ni <sup>2+</sup> , Co <sup>2+</sup> , Zn <sup>2+</sup>
V	(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> + NH <sub>4</sub> OH	Ca <sup>2+</sup> , Ba <sup>2+</sup> , Sr <sup>2+</sup>
VI	No group ppt	Mg <sup>2+</sup> , NH <sub>4</sub> <sup>+</sup>

3. **Confirmatory tests for identified cation:**

Pb<sup>2+</sup>: Yellow ppt with KI

Cu<sup>2+</sup>: Blue ppt with NH<sub>4</sub>OH

Fe<sup>3+</sup>: Blood red with KSCN

Al<sup>3+</sup>: White gelatinous ppt with NH<sub>4</sub>OH

Mn<sup>2+</sup>: Pink ppt with NaOH

Ni<sup>2+</sup>: Red ppt with dimethylglyoxime

Zn<sup>2+</sup>: White ppt with NaOH, dissolves in excess

Ca<sup>2+</sup>: White ppt with (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>

Ba<sup>2+</sup>: White ppt with H<sub>2</sub>SO<sub>4</sub> (BaSO<sub>4</sub>)

Sr<sup>2+</sup>: White ppt with (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>

Mg<sup>2+</sup>: White ppt with NaOH

NH<sub>4</sub><sup>+</sup>: Ammonia gas with NaOH + heat → pungent smell

**Observation Table:**

Test	Observation	Inference
Physical appearance		
Dry heating test		
Flame test		
Anion group test		Suspected anion
Confirmatory test for anion		Anion confirmed
Cation group test		Suspected cation group
Confirmatory test for cation		Cation confirmed

**Result:**

Cation detected: .....

Anion detected: .....

Hence, the given salt is [name of salt]

**Precautions:**

Use dilute acids carefully to avoid excessive reaction.

Wash test tubes properly between tests to avoid contamination.

Handle toxic gases (like H<sub>2</sub>S) in a fume chamber.

Use freshly prepared reagents for accurate results.

Record observations carefully and do not assume results.