10/2013 EXPERIMENT NUMBER: 7

TEST FOR PHENOLIC GROUP

EXPERIMENT	OBSERVATION	INFERENCE
1. LITMUS TEST: Add 1 drop of the organic compound on a blue litmus paper.	Blue litmus changes to red.	Phenolic group is present.
2. FERRIC CHLORIDE TEST: To an aqueous solution of the compound, add 1ml of neutral ferric chloride solution.	Violet colouration.	Phenolic group is present.
3. BROMINE WATER TEST: To the aqueous solution of the organic compound, add bromine water.	White precipitate formed.	Phenolic group is present.

→ RESULT:

The given organic compound contains phenolic group.

10/14

EXRIMENT NUMBER : 8.

TEST FOR ALDEHYDIC GROUP.

EXPERIMENT	OBSERVATION	INFERENCE.
1. 2,4-DNP TEST: To a little of the organic compound, add 1ml of 2,4-DNP reagent and shake well.	Red, yellow or orange ppt formed.	Carbonyl group is present.
2. TOLLENS TEST: To a few ml of the organic compound, add 1-2 ml of Tollen's Reagent. Shake well and heat it in a water bath for 2-3 minutes.	Silver mirror or grey precipitate formed.	Aldehydic group is present.
3. FEHLING'S TEST: Mix equal volumes of Fehling's solution A and B and add a few ml of organic compound into it. Shake well and keep the test tube in a boiling water bath for 2 minutes.	Red precipitate of Cu_2O formed.	Aldehydic group is present.

→ RESULT: Given organic compound contains aldehydic group.

EXPERIMENT NUMBER : 9

TEST FOR CARBOXYLIC ACID GROUP.

EXPERIMENT	OBSERVATION	INFERENCE.
1. LITMUS TEST: Dip the blue litmus paper into the organic compound	Blue litmus changes to red.	Carboxylic acid group is present.
2. $NaHCO_3$ TEST: To the aqueous solution of the organic compound, add a little $NaHCO_3$ powder.	Brisk effervescence of a colourless, odourless gas which turn limewater milky.	Carboxylic acid group is present.
3. ESTER FORMATION TEST: To 1ml of the organic compound in a clean dry Test tube, add 1ml of ethanol and 2 drops of conc : H_2SO_4. Warm the mixture in a water bath for 10-15 minutes. Cool and pour the contents to an aqueous solution of Na_2CO_3 in a beaker. Smell it.	Pleasant fruity smell due to the presence of an ester.	Carboxylic acid group is present.

→ RESULT: The given compound contains carboxylic acid group.

VOLUMETRIC

ANALYSIS

2013 EXPERIMENT NUMBER: 10.

PREPARATION OF STANDARD SOLUTION OF MOHR'S SALT.

→ **AIM:**

To prepare standard solution of $\frac{M}{20}$ ferrous ammonium sulphate.

$\frac{M}{20}$ = 0.05 Molar.

→ **APPARATUS REQUIRED:**

watch glass, beaker, standard flask, funnel, glass rod.

→ **CHEMICALS REQUIRED:**

Mohr's salt (Ferrous Ammonium sulphate hexahydrate), Distilled water, dil: Hydrochloric acid.

→ **CALCULATION OF WEIGHT OF THE SALT:**

Molarity of the solution = 0.05 = M.

Weight of Mohr's salt required to make 0.05 Molar Solution = $W = \frac{M \times M_r \times V(mL)}{1000}$.

M_r = 392 g mol^{-1}.

V(mL) = 250 mL.

$\therefore W = \frac{0.05 \times 392 \times 250}{1000}$

$W = 4.9\,g$

$= g$

→ PROCEDURE:

1. Weigh accurately 4.9 g of Mohr's salt crystals on a watch glass.
2. Transfer carefully, the crystals of Mohr's salt to a clean, dry beaker.
3. Add 20 ml of dil: H_2SO_4 to the Mohr's salt in the beaker to prevent hydrolysis of ferrous ions to ferric ions.
4. Add 20 ml of water into the beaker. Dissolve the Mohr's salt completely in the solution.
5. Transfer the solution using glass rod and funnel to a standard flask.
6. Add water to the standard flask to make up the volume to 250 ml.
7. Shake the standard flask well so that the solution becomes homogeneous

→ RESULT:

The standard solution of Mohr's salt is prepared.

20/10/14

2023 EXPERIMENT NUMBER: 11

STANDARDISATION OF $KMnO_4$ USING 0.05M MOHR'S SALT SOLUTION.

→ AIM:

To standardise the given $KMnO_4$ using 0.05M Mohr's salt solution.

→ APPARATUS REQUIRED:

Burette, pipette, standard flask, conical flask, funnel, glass rod.

→ CHEMICALS REQUIRED:

Mohr's salt, $KMnO_4$, 4N H_2SO_4, distilled water.

→ PRINCIPLE:

1. This is an example of a redox reaction.
2. Acidified $KMnO_4$ is the oxidising agent.
3. Mohr's salt is the reducing agent.
4. $KMnO_4$ is the self indicator.
5. End point is the colour change from colourless to permanent pink.
6. During the reaction, the oxidation state of Mn changes from +7 to +2. The oxidation state of Fe changes from +2 to +3.
7. $KMnO_4$ is in the burette and Mohr's salt is in the conical flask taken by the pipette.

→ EQUATION:

Ionic Equations:

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

Overall Equation: $2KMnO_4 + 10FeSO_4 + 8H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$.

→ PROCEDURE:

1. Standard solution of 0.05M Mohr's salt is prepared accurately by weighing 4.9g of Mohr's salt using a chemical balance.
2. Rinse the burette with water and then with $KMnO_4$.
3. Fill up the burette with $KMnO_4$.
4. Rinse the pipette with Mohr's salt solution. Pipette out 20mL of mohr's salt solution. Transfer it into the conical flask.
5. Add 10mL of 4N H_2SO_4 into the conical flask to make the medium acidic.
6. Titrate Mohr's salt solution against $KMnO_4$ solution till end point is reached.
7. Note down the initial burette reading and the final burette reading.
8. Repeat the titration till you get concordant value.

→ RESULT:

1. Molarity of $KMnO_4$ = 0.0102 $mol\,L^{-1}$

2. Strength of $KMnO_4$ = 1.611 $g\,L^{-1}$.

→ PRECAUTIONS:

1. The burette & pipette must be washed well.
2. For colourless solutions, lower meniscus must be checked while for coloured solutions, upper meniscus must be considered while measuring the level.

10/14

→ CALCULATION:

Standardisation of $KMnO_4$.

SR. No.	Initial Burette Reading	Final Burette Reading	Volume of $KMnO_4$ (mL)	Volume of Mohr's Salt Soln (mL)
1.	0	19.5	19.5	20
2.	0	19.5	19.5	20

$KMnO_4$ × Mohr's Salt

$$\frac{M_1V_1}{N_1} = \frac{M_2V_2}{N_2}$$

M_1 = Molarity of $KMnO_4$ = ?
V_1 = Volume of $KMnO_4$ used = 19.5mL.
N_1 = No. of moles of $KMnO_4$ = 1.

M_2 = Molarity of Mohr's Salt = 0.05 M
V_2 = Volume of Mohr's Salt used = 20mL
N_2 = No. of moles of Mohr's Salt = 5

$$M_1 = \frac{M_2V_2N_2}{N_2V_1} = \frac{0.05 \times 20 \times 1}{5 \times 19.5}$$

$$M_1 = 0.0102 \text{ mol } L^{-1}.$$

Strength or wt/litre of $KMnO_4$:

Strength = M × Molar mass
= 158 g mol^{-1} × 0.0102 mol L^{-1}
= 1.611 g L^{-1}.

29/2013 EXPERIMENT NUMBER : 12

PREPARATION OF STANDARD SOLUTION OF OXALIC ACID

→ **AIM:**

To prepare a standard solution of $\frac{M}{40}$ oxalic acid.

$\frac{M}{40} = 0.025M$.

→ **APPARATUS REQUIRED:**

Watch glass, beaker, standard flask, funnel, glass rod.

→ **CHEMICALS REQUIRED:**

Oxalic acid, distilled water.

→ **CALCULATION OF WEIGHT OF THE SALT:**

$Wt = M \times M_r \times V$

$Wt = (90+36) \times \frac{1}{40} \times 250.$ $(H_2C_2O_4 \cdot 2H_2O)$

$Wt = \frac{126}{4} \times 25.$

$Wt = 0.7875g$

→ **PROCEDURE:**

1. Dissolve the oxalic acid in minimum quantity of water in a beaker
2. Transfer the solution to a standard flask from the beaker using funnel and glass rod. Make the solution to 250ml in the standard flask.

→ RESULT:

$\frac{M}{40}$ oxalic acid solution (250 mL) is prepared.

25/10/14

09/2018 EXPERIMENT NUMBER: 13

STANDARDISATION OF $KMnO_4$ USING 0.025 M OXALIC ACID.

→ **AIM:**

To standardise the given $KMnO_4$ using $\frac{M}{40}$ oxalic acid solution.

→ **APPARATUS REQUIRED:**

Burette, pipette, standard flask, conical flask, funnel, glass rod.

→ **CHEMICALS REQUIRED:**

Oxalic acid, $KMnO_4$ solution, 4N H_2SO_4.

→ **PRINCIPLE:**

1. This is an example of a redox reaction.
2. Acidified $KMnO_4$ is the oxidising agent.
3. Oxalic acid is the reducing agent.
4. $KMnO_4$ is the self-indicator.
5. End point is the colour change from colourless to permanent pink.
6. During the reaction, the oxidation state of Mn changes from +7 to +2. T
   oxidation state of C changes from +3 to +4.
7. $KMnO_4$ is in the burette and oxalic acid is in the conical flask. It
   by the pipette.

→ **EQUATION:**

Ionic Equation:

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

Overall Equation: $2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 10CO_2$

→ PROCEDURE:

1. Prepare a standard solution of 0.025 M oxalic acid by accurately weighing 0.7875 g of oxalic acid using a chemical balance.
2. Fill the burette with $KMnO_4$ after rinsing well.
3. Pipette out 20 mL of oxalic acid from the standard flask to the conical fla
4. Add 1 test tube full of 4N H_2SO_4 into the oxalic acid solution of the conical flask.
5. Heat the solution in the conical flask to 60–70°C
6. Titrate the hot solution of oxalic acid against the $KMnO_4$ till end point is reached [colourless to permanent pink].

→ RESULT:

1. Molarity of $KMnO_4$ = 0.0102 mol L^{-1}.
2. Strength of $KMnO_4$ = 1.611 g L^{-1}.

→ PRECAUTIONS:

1. The burette and pipette must be washed well.
2. For colourless solution, lower meniscus must be considered while for coloured solutions, upper meniscus must be considered while measuring the level of liquid.

30/10/18

→ OBSERVATION AND CALCULATION:

SR. No.	Initial Burette Reading	Final Burette Reading	Volume of KMnO4 (mL)	Volume of Oxalic acid sol
1.	0	19.5	19.5	20
2.	0	19.5	19.5	20.

$KMnO_4$ × Oxalic Acid

$$\frac{M_1V_1}{N_1} = \frac{M_2V_2}{N_2}$$

M_1 = Molarity of $KMnO_4$ = ?
V_1 = Volume of $KMnO_4$ = 19.5mL
N_1 = No. of moles of $KMnO_4$ = 2.

M_2 = Molarity of Oxalic Acid = $\frac{M}{40}$ = 0.025
V_2 = Volume of oxalic acid = 20mL
N_2 = No. of moles of oxalic acid = 5.

$$M_1 = \frac{M_2V_2N_1}{N_2V_1}$$

$$M_1 = \frac{0.025 \times 20 \times 2}{5 \times 19.5}$$

$$M_1 = 0.0102$$

Strength or wt/Litre of $KMnO_4$:

$$M = \frac{Wt}{M_r \times V} \Longrightarrow \frac{Wt}{V} = M \times M_r.$$

Strength = $0.0102\ mol\ L^{-1} \times 158\ g\ L^{-1}$
= $1.611\ g\ L^{-1}$

INORGANIC QUALITATIVE ANALYSIS

→ PRELIMINARY TESTS:

	EXPERIMENT	OBSERVATION	INFERENCE.
1.	Colour and appearance.	Colourless, crystalline salt.	absence of Cu^{2+}, Mn^{2+}.
2.	Odour	slight smell of ammonia	NH_4^+ may be present.
3.	Solubility	Soluble in water.	No need to make Na_2CO_3 extract.
4.	Flame Test: make a paste of a little salt in conc: HCl. Introduce it to a flame on a platinum loop.	No observation	absence of Ca^{2+}, Cu^{2+}, Ba^{2+}, Sr^{2+}.

	EXPERIMENT	OBSERVATION	INFERENCE.
5.	Dry Heating: Take a little salt in a dry test tube and heat it directly over a flame.	Strong smell of ammonia. White sublimate.	NH_4^+ may be present.

→ TEST FOR ANIONS:

	EXPERIMENT	OBSERVATION	INFERENCE.
	TEST FOR CO_3^{2-} To a little of the salt, add a few drops of dil: H_2SO_4	No characteristic observation.	CO_3^{2-} is absent.
2.	TEST FOR CH_3COO^- To a little of the salt, add a few drops of conc: H_2SO_4	No characteristic observations	CH_3COO^- is absent.
3.	TEST FOR HALIDES:	OBSERVATION	INFERENCE

	a few drops of conc: H_2SO_4		
	To a little of the salt, add a few drops of conc: H_2SO_4.	No characteristic observations.	Cl^-, Br^-, I^- are absent.
4.	TEST FOR NO_3^-:		
	To a little of the salt, add a few drops of conc: H_2SO_4 and heat. Add a small paper ball to it and heat again	No characteristic observations.	NO_3^- is absent.
5.	TEST FOR SO_4^{2-}		
	To a little of the salt solution, add a little $BaCl_2$ solution.	No characteristic observations	SO_4^{2-} is absent.
6.	TEST FOR PO_4^{3-}		
	To a little of the salt, add conc: HNO_3. Heat, add powdered Ammonium Molybdate and heat again.	Canary yellow precipitate	PO_4^{3-} is present.

CONFIRMATION TEST FOR PO_4^{3-}

EXPERIMENT	OBSERVATION	INFERENCE.
To a little of the salt, add cobalt nitrate solution	Violet ppt soluble in dil: CH_3COOH.	PO_4^{3-} is confirmed.

RESULT: The given salt has PO_4^{3-} as anion.

→ TEST FOR CATIONS:

EXPERIMENT	OBSERVATION	INFERENCE.
GROUP 0 ELEMENTS.		
TEST FOR NH_4^+. To a little of the salt, add NaOH solution and heat.	Smell of ammonia.	NH_4^+ may be present.

CONFIRMATION TEST FOR NH_4^+.

To a little of the salt solution, add a little Nessler's Reagent.	Camel brown precipitate obtained.	NH_4^+ is confirmed.
To a little of the salt solution, add a little Na_2CO_3 solution.	No precipitation.	NH_4^+ is confirmed.

RESULT: The given salt has NH_4^+ as cation.

CONCLUSION: The given salt is Ammonium Phosphate, $(NH_4)_3PO_4$.

30/5/13