

2013 EXPERIMENT NUMBER: 10.

PREPARATION OF STANDARD SOLUTION OF MOHR'S SALT.

→ AIM:

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

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

→ APPARATUS REQUIRED:

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

→ CHEMICALS REQUIRED:

Mohr's salt (Ferric 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

$$\text{make 0.05 Molar Solution} = W = \frac{M \times M_r \times V(\text{mL})}{1000}.$$

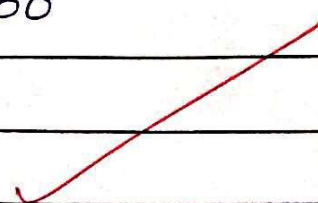
$$M_r = 392 \text{ g mol}^{-1}.$$

$$V(\text{mL}) = 250 \text{ mL}.$$

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

$$W = 4.9 \text{ g}$$

= 0



→ 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~~

2013 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:

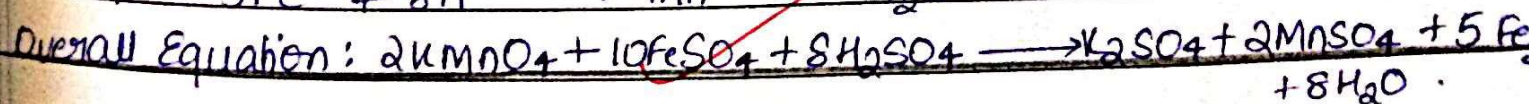
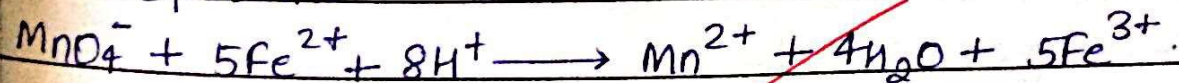
Mohr's salt, $KMnO_4$, 4N H_2SO_4 , distilled water.

→ 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:



→ 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 9N 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 $\text{KMnO}_4 = 0.0102 \text{ mol L}^{-1}$
2. Strength of $\text{KMnO}_4 = 1.611 \text{ g L}^{-1}$.

→ PRECAUTIONS:

1. The burette or 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.

30/10/14

→ CALCULATION:

Standardisation of KMnO_4 .

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

$$\text{KMnO}_4 \quad \times \quad \text{Mohr's Salt}$$
$$\frac{M_1 V_1}{N_1} = \frac{M_2 V_2}{N_2}$$

$$M_1 = \text{Molarity of } \text{KMnO}_4 = ?$$

$$V_1 = \text{Volume of } \text{KMnO}_4 \text{ used} = 19.5 \text{ mL}$$

$$N_1 = \text{No. of moles of } \text{KMnO}_4 = 1$$

$$M_2 = \text{Molarity of Mohr's Salt} = 0.05 \text{ M}$$

$$V_2 = \text{Volume of Mohr's Salt used} = 20 \text{ mL}$$

$$N_2 = \text{No. of moles of Mohr's Salt} = 5$$

$$M_1 = \frac{M_2 V_2 N_2}{N_2 V_1} = \frac{0.05 \times 20 \times 1}{5 \times 19.5}$$

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

Strength or wt/ditre of KMnO_4 :

$$\text{Strength} = M \times \text{Molar mass}$$

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

$$= \underline{\underline{1.6116 \text{ g L}^{-1}}}$$

29/2012 EXPERIMENT NUMBER: 12

PREPARATION OF STANDARD SOLUTION OF OXALIC ACID

→ AIM:

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

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

→ APPARATUS REQUIRED:

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

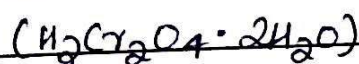
→ CHEMICALS REQUIRED:

Oxalic acid, distilled water.

→ CALCULATION OF WEIGHT OF THE SALT:

$$WT = M \times M_r \times V$$

$$WT = (90 + 36) \times \frac{1}{40} \times 250.$$



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

$$WT = \underline{\underline{0.7875g}}$$

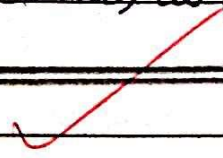
→ PROCEDURE:

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:

M oxalic acid solution (250 ml) is prepared.

40



~~Q2~~
20/10/14

09/2018 EXPERIMENT NUMBER: 13

STANDARDISATION OF $KMnO_4$ USING 0.085 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:

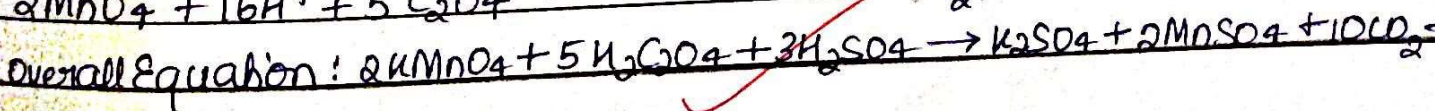
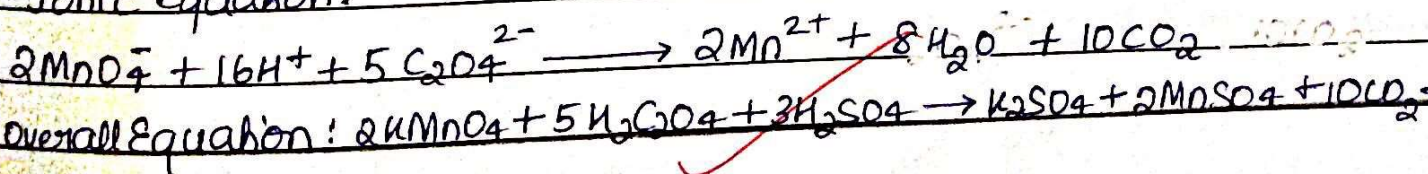
oxalic acid, $KMnO_4$ solution, 4N H_2SO_4 .

→ 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. The oxidation state of C changes from +3 to +4.
7. $KMnO_4$ is in the burette and oxalic acid is in the conical flask. To be added by the pipette.

→ EQUATION:

Ionic Equation:



→ PROCEDURE:

1. Prepare a standard solution of 0.025 M oxalic acid by accurately weighing 0.7875g of oxalic acid using a chemical balance.
2. Fill the burette with KMnO_4 after rinsing well.
3. Pipette out 20ml of oxalic acid from the standard flask to the conical flask.
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 $\text{KMnO}_4 = 0.0102 \text{ mol L}^{-1}$.
2. Strength of $\text{KMnO}_4 = 1.611 \text{ g L}^{-1}$.

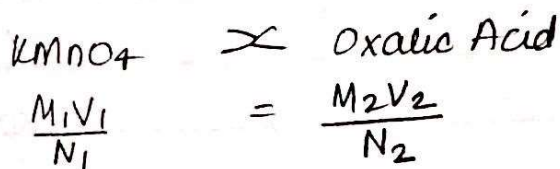
→ PRECAUTIONS:

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 $KMnO_4$ (mL)	Volume of Oxalic acid
1.	0	19.5	19.5	20
2.	0	19.5	19.5	20.



$M_1 = \text{Molarity of } KMnO_4 = ?$

$V_1 = \text{Volume of } KMnO_4 = 19.5 \text{ mL}$

$N_1 = \text{No. of mols of } KMnO_4 = 2$

$M_2 = \text{Molarity of Oxalic Acid} = \frac{M}{40} = 0.025$

$V_2 = \text{Volume of oxalic acid} = 20 \text{ mL}$

$N_2 = \text{No. of mols of oxalic acid} = 5$

$$M_1 = \frac{M_2 V_2 N_1}{N_2 V_1}$$

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

$$M_1 = 0.0102$$

Strength or wt/litre of $KMnO_4$:

$$M = \frac{Wt}{M_r \times V} \implies \frac{Wt}{V} = M \times M_r$$

$$\begin{aligned} \text{Strength} &= 0.0102 \text{ mol } L^{-1} \times 158 \text{ g } L^{-1} \\ &= 1.611 \text{ g } L^{-1} \end{aligned}$$

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+} .

INFERENCE:

NH_4^+ may be present.

OBSERVATION

Strong smell of ammonia.
White sublimate.

INFERENCE:

OBSERVATION

CO_3^{2-} is absent.

No characteristic observation.

EXPERIMENT

5. Dry Heating:

Take a little salt in a dry test tube and heat it directly over a flame.

TEST FOR ANIONS:

EXPERIMENT

TEST FOR CO_3^{2-}

To a little of the salt, add a few drops of

dil: H_2SO_4

a. TEST FOR CH_3COO^-

To a little of the salt, add a few drops of conc. H_2SO_4

No characteristic observation

CH_3COO^- is absent.

3. TEST FOR ANIONS:

OBSERVATION

INFERENCE

0. Few drops of conc: H_2SO_4

TEST FOR NO_3^-
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_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. No yellow precipitate. PO_4^{3-} is present.

CONFIRMATION TEST FOR PO_4^{3-}

EXPERIMENT

To a small of the salt,
add cobalt nitrate solution

OBSERVATION

Violet ppt soluble in
dil. H_2CO_3 .

INFERENCE.

PO_4^{3-} is confirmed.

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

→ TEST FOR CATIONS:

EXPERIMENT

GROUP 0 ELEMENTS.

OBSERVATION

INFERENCE.

TEST FOR NH_4^+ .

To a small of the salt,
add NH_4OH solution and
heat.

Smell of ammonia.

NH_4^+ may be present.

CONFIRMATION TEST FOR NH_4^+

NH_4^+ is confirmed.

Camel brown precipitate obtained.

To a small of the salt solution, add a little Nessler's Reagent.

NH_4^+ is confirmed.

No precipitation.

To a small of the salt solution, add a little Na_2CO_3 solution.

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

$(\text{NH}_4)_3\text{PO}_4$.

CONCLUSION: The given salt is Ammonium Phosphate,

~~20/5/13~~
20/5/13

