EXPERIMENT NO. 3 (a)

Date :

STANDARDISATION OF $KMnO_4$

AIM OF THE EXPERIMENT : To determine the strength of $KMnO_4$ solution by using standard sodium oxalate solution.

APPARATUS REQUIRED

1. Burette
2. Pipette
3. Conical flask
4. Burette Stand
5. Marble sheet / Asbestos Pad
6. Measuring cylinder

CHEMICALS REQUIRED

1. N/10 sodium oxalate ($Na_2C_2O_4$)
2. $KMnO_4$
3. H_2SO_4 (6N)

THEORY

When $KMnO_4$ is standardised against sodium oxalate in the presence of sulphuric acid, $KMnO_4$ oxidises oxalate ion into CO_2 while $KMnO_4$ gets reduced to $MnSO_4$. Mn^{+7} is reduced to Mn^{+2} at a temperature of 60^0-70^0C. The end point is detected when permanent pink colour develops in the solution.

CHEMICAL EQUATION

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$[Na_2C_2O_4 + H_2SO_4 + [O] \rightarrow Na_2SO_4 + 2CO_2 + H_2O] \times 5$$

$$2KMnO_4 + 8H_2SO_4 + 5Na_2C_2O_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Na_2SO_4 + 8H_2O + 10CO_2$$

IONIC EQUATION

$$[MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O] \times 2$$

$$[C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-] \times 5$$

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

PROCEDURE :

1. Thoroughly wash the glassware with water.
2. Rinse the burette with supplied $KMnO_4$ solution and pipette with given sodium oxalate solution.
3. Fill the burette with the supplied $KMnO_4$ solution upto a convenient mark, Remove any air gap and note the initial burette reading.
4. Pipette out 10 ml of standard sodium oxalate solution into a conical flask and add approximately 10 ml of 6N H_2SO_4 to it.
5. Warm the solution upto 60^0C to 70^0C.
6. Carry out the titration by adding $KMnO_4$ solution from the burette till the colour changes from colourless to light pink. Note the final burette reading.
7. Repeat the process to get the concordant reading.

CALCULATION :

At the end point

$N_1V_1 = N_2V_2$

Where N_1 = Normality of $KMnO_4$ solution.

V_1 = Volume of $KMnO_4$ solution required for titration = V ml

N_2 = Normality of sodium oxalate solution (N/10)

V_2 = Volume of sodium oxalate solution required for titration = 10 ml

So $$N_1 = \frac{N_2V_2}{V_1} = \frac{N}{10} x \frac{10}{V}$$

Strength of $KMnO_4$ = N_1 x Eq. wt. of $KMnO_4$

= N_1 x 31.6 = ... g/l

Questions for Discussion.

1. What is redox titration ?
2. Which indicator is used in this experiment ?
3. What do you mean by primary standard and secondary standard ?
4. What do you mean by oxidation and reduction ?
5. What do you mean by oxidising agent and reducing agent ?
6. Why heating is necessary in the experiement ?
7. What is the colour change of $KMnO_4$ in acidic, alkaline and neutral medium titration ?
8. What is the equivalent weight of $KMnO_4$ in acidic, alkaline and neutral medium ?
9. Why $KMnO_4$ is added slowly in the titration ?
10. What is the equivalent weight of $Na_2C_2O_4$?

Rough Work

DEPARTMENT OF CHEMISTRY

Roll No. :

Branch :

Date :

AIM OF THE EXPERIMENT :

OBSERVATION TABLE

No.of Obs.	Vol. of $Na_2C_2O_4$ solution (ml)	Burette Reading (ml)		Difference V (ml)	Remark
		Initial	Final		
1.					
2.					
3.					
4.					

CALCULATION :

CONCLUSION :

EXPERIMENT NO. 3 (b)

Date :

ESTIMATION OF Fe^{2+}

AIM OF THE EXPERIMENT : To determine the amount of Ferrous (Fe^{2+}) ions present in the Mohr's salt solution by using standard $KMnO_4$ solution

APPARATUS REQUIRED

1.Burette 2. Pipette 3. Beaker 4. Conical flask

5. Burette Stand 6. Marble sheet / Asbestos Pad 7. Measuring cylinder

CHEMICALS REQUIRED

1. Standard $KMnO_4$ solution
2. Mohr's salt solution
3. H_2SO_4 (6N)

PRINCIPLE

To estimate the amount of ferrous (Fe^{2+}) ions in supplied Mohr's salt solution, it is titrated against a standard $KMnO_4$ solution. In the presence of H_2SO_4, $KMnO_4$ oxidises ferrous ions to ferric ions, and it self get reduced to $MnSO_4$ i.e. Mn^{+7} reduced to Mn^{+2}. The end point is detected when a faint permanent pink colour is developed in the solution.

CHEMICAL EQUATION

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$[2FeSO_4 + H_2SO_4 + [O] \rightarrow Fe_2(SO_4)_3 + H_2O] \times 5$$

$$2KMnO_4 + 8H_2SO_4 + 10\ FeSO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$$

IONIC EQUATION

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

$$[Fe^{2+} \rightarrow Fe^{3+} + e^-] \times 5$$

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

PROCEDURE :

1. Thoroughly wash the glassware with water.
2. Rinse the burette with supplied $KMnO_4$ solution and pipette with the given Mohr's salt solution.
3. Fill the burette with the supplied standard $KMnO_4$ solution upto a convenient mark, remove any air gap and note the initial burette reading.
4. Pipette out 10 ml of Mohr's salt solution into a conical flask and add approximately 10ml of 6N H_2SO_4 to it by using a measuring cylinder.
5. Carry out the titration by adding $KMnO_4$ from the burette till the colour changes to light pink. Note the final burette reading.
6. Repeat the process to get the concordant reading.

CALCULATION :

At the end point

$N_1V_1 = N_2V_2$

Where N_1 = Normality of standard $KMnO_4$ solution.

V_1 = Volume of $KMnO_4$ solution required for titration.

N_2 = Normality of Mohr's salt solution.

V_2 = Volume of Mohr's salt solution required for titration.

So $$N_2 = \frac{N_1V_1}{V_2}$$

Strength of Fe^{+2} ion in Mohr's salt solution

= N_2 x Eq. wt. of Fe^{2+} in Mohr's salt

= N_2 x 55.85 = ... g/l

DEPARTMENT OF CHEMISTRY

Roll No. :

Branch :

Date :

AIM OF THE EXPERIMENT :

OBSERVATION TABLE :

No.of Obs.	Vol. of Mohr's salt solution (ml)	Burette Reading (ml)		Difference (ml)	Remark
		Initial	Final		
1.					
2.					
3.					
4.					

CALCULATION :

CONCLUSION :

Questions for Discussion.

1. Which is more stable between Fe^{2+} or Fe^{3+} and why ?
2. What are internal, external and self indicators ? Give one example of each.
3. Why $KMnO_4$ is not a primary standard ?
4. Why H_2SO_4 is used for acidification of $KMnO_4$ solution and not HCl or HNO_3 ?
5. Why ferrous sulphate is not used in titration instead of Mohr's salt ?
6. What is the formula of Mohr's salt ?
7. Which meniscus is to be seen in the burette containing $KMnO_4$ solution and why ?
8. Which indicator is used during standardization of $KMnO_4$ solution ?
9. What acts as catalyst in this reaction ?
10. Why $KMnO_4$ is coloured ?

Rough Work