Experiment - 8

Aim - To determine the amount of total iron in an iron ore solution by external indicator method.

Indicator - Potassium ferricyanide

End Point - Non appearance of bluish green colour externally.

Reactions Used -

$$K_3[Fe(CN)_6] \xrightarrow{Aq.} 3K^+ + [Fe(CN)_6]^{3-}$$

$$3Fe^{+2} + 2[Fe(CN)_6]^{3-} \longrightarrow Fe_3[Fe(CN)_6]_2$$

Ferro ferricyanide
(Prussian blue colour)

$$Fe^{+3} + [Fe(CN)_6]^{3-} \longrightarrow Fe[Fe(CN)_6]$$

Colourless

$$Sn^{2+} \longrightarrow Sn^{4+} + 2e^- \text{ (oxidation)}$$

$$2Fe^{3+} + 2e^- \longrightarrow 2Fe^{2+} \text{ (reduction)}$$

$$Sn^{2+} + 2Fe^{3+} \longrightarrow Sn^{4+} + 2Fe^{2+}$$

$$Sn^{2+} \longrightarrow Sn^{4+} + 2e^-$$

$$2Hg^{2+} + 2e^- + 2Cl^- \longrightarrow Hg_2Cl_2$$

$$Sn^{2+} + 2Hg^{2+} + 2Cl^- \longrightarrow Sn^{4+} + Hg_2Cl_2 \downarrow$$

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7H_2O$$

$$Fe^{2+} \longrightarrow Fe^{3+} + e^- \quad \times 6$$

$$Cr_2O_7^{2-} + 14H^+ + Fe^{2+} \longrightarrow 2Cr^{3+} + Fe^{3+} + 7H_2O$$

Dated 24/12/2020

Experiment - 8

Aim - To determine the amount of total iron in an iron ore solution by external indicator method.

Apparatus - Burette, pipette, titration flask, 250 ml volumetric flask, 500 ml beaker, test tube, measuring cylinder, filter paper, zinc dust etc.

Chemicals Required - 5% Stannous chloride solution, saturated mercuric chloride solution, standard pot. dichromate solution, conc. HCl and dil. HCl.

Indicator - Potassium ferricyanide

End Point - Non appearance of bluish green colour externally.

Theory - Determination of iron content using External Indicator -

The ore solution is reduced to slight excess of stannous chloride as in case of internal indicator. The reduced solution is then titrated against standard dichromate solution in acidic medium.

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Observations:- Volume of ore solution taken = 10 ml

S.NO.	Initial Burette Reading	Final Burette Reading	Vol. of $K_2Cr_2O_7$ used (ml)
1.	0	9.5	9.5
2.	9.5	18.9	9.4
3	18.9	28.4	9.5 ✓

Concordant reading (V) = 9.5 ml ✓

using potassium ferricyanide as an external indicator to find out end point of titration. It reacts with Fe^{+2} ions of ore solution to give prussian blue colour.
At end point, when all Fe^{2+} ions get oxidised to Fe^{3+} ions, bluish green colour does not appear with indicator.

From the volume of $K_2Cr_2O_7$, used at end point the amount of total iron in ore solution can be calculated in the form of ferrous ions.

Use of External Indicator - Place a series of small drops of freshly prepared potassium ferricyanide indicator on a white tile by means of glass rod. Run in dichromate solution from the burette in lots in iron ore solution and shake. Take out a drop of the reaction mixture from the conical flask with the help of glass rod, mix it with one drop of indicator on the tile. If a blue colour is produced, continue the process till a drop let of reaction mixture gives no blue colour on the tile. This is the end point.

Calculations :-

1. Normality of ore solution :-

Vol. of ore solution taken for titration :- 10 ml
Concordant volume of $K_2Cr_2O_7$ solution used = 9.5 ml
Normality of $K_2Cr_2O_7$ solution = $\frac{1}{10}$ N

Applying Normality equation,

$N_1 V_1 = N_2 V_2$
(ore) ($K_2Cr_2O_7$)

$N_1 \times 10 = \frac{1}{10} \times 9.5$

$N_1 = \frac{9.5}{100} N$

$\boxed{N_1 = 0.095\,N}$ ✓

2. Strength of total iron in ore solution - ✓

Strength of total iron in ore solution = Normality × Equivalent wt. of iron

$= 0.095N \times 56$
$= 5.32$ g/lt
$= 5.32 \times 10^3$ ppm ✓
$= 5320$ ppm ✓

Procedure -

Step I Reduction of ore solution -

Pipette out 10 ml of iron ore solution into a conical flask. Add 3 ml conc. HCl and heat to boiling to get yellow solution. Add 5% $SnCl_2$ solution dropwise with shaking till yellow colour just disappears. Add 2-3 drops in excess. Cool the solution to room temperature under tap water. Add 3 ml saturated $HgCl_2$ solution in one lot and shake well till silky white ppt is formed.

Step II Titration of reduced solution Vs $K_2Cr_2O_7$ sol^n

i) For long range reading :- Fill the burette with standard $K_2Cr_2O_7$ after rinsing. Add 10 ml dil. HCl to reduced iron solution. Add 5ml dichromate solution from burette in lot. Shake contents well. Take out a drop of this solution with a clean tile and check for end point. Add dichromate sol^n in 5 ml lots and check for end point till it is obtained. Record the volume of dichromate used as long range reading.

ii) For short range reading — Reduce the iron solution. Acidify it and add dichromate sol^n from burette in 1 ml lots starting from lower end of long range reading. After every addition

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Results - The amount of total iron in our solution is 5.32 g/lt.

check for the end point externally. Record the volume.

iii) For decimal reading - Reduce the iron ore. Acidify it with dil. HCl. Add dichromate solution in 0.1 ml lots starting from lower end of short range reading till end point is obtained. Record decimal reading.

iv) For concordant reading - Take three concordant readings with reduced iron ore solution.

Results - The amount of total iron in ore solution is 5.32 g/lt.

8/01/21