EXPERIMENT NO – 1

OBJECT: To determine the ferrous ion (Fe^{+2}) content in given sample solution of iron (Mohr's salt,$FeSO_4(NH_4)_2SO_4.6H_2O$) by titrating it against standard N/30 potassium dichromate ($K_2Cr_2O_7$) solution by using potassium ferricyanide $K_3[Fe(CN)_6]$ as an external indicator.

REQUIRMENTS:

APPARATUS: Burette, Pipette, Conical flask, Beaker, Funnel, Glass rod, Dropper, White glazed tile.

CHEMICALS: Standard N/30 Potassium dichromate ($K_2Cr_2O_7$) solution, Unknown sample solution of Iron (Mohr's salt),dilute H_2SO_4

INDICATOR: Potassium ferricyanide $K_3[Fe(CN)_6]$ IUPAC name potassium hexa cyano ferrate (III).

PRINCIPLE:

Potassium dichromate ($K_2Cr_2O_7$) acts as an oxidizing agent in acidic medium and each molecule of it gives three atoms of nascent oxygen. The following reaction takes place

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

Or

Ionically it may be written as

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

When acidic potassium dichromate is added to (standard solution), of iron ore solution, only $FeSO_4$ is oxidized and ammonium sulphate remains unchanged. The following reaction takes place:

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

or

$$[Fe^{++} \rightarrow Fe^{3+} + e^-] \times 6$$

The overall reaction and ionic reaction is:

$$K_2Cr_2O_7 + 7H_2SO_4 + 6\,FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3\,Fe_2(SO_4)_3 + 7H_2O$$

$$Cr_2O_7^{--} + 14\,H^+ + 6Fe^{2+} \rightarrow 2Cr^{3+} + 6\,Fe^{3+} + 7H_2O$$

In this titration potassium ferricyanide $K_3[Fe(CN)_6]$ is used as an external indicator, if ferrous ion (Fe^{+2}) are present a strong blue color is developed due to the following reaction :

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

Iron (II) hexa cyano ferrate (III)

(Turn bull's blue)

At the end point no ferrous ion are present in the solution therefore the indicator fails to produce blue colour when treated with a drop of titration mixture.

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

Iron (III) hexa cyano ferrate (III)

(Colourless)

PROCEDURE:

i) Pipette out 10 ml of iron ore sample solution in a conical flask and add half test tube of dilute H_2SO_4 to it.

ii) Fill the burette with standard N/30 $K_2Cr_2O_7$ solution up to the zero mark .

iii) Drops of indicator are placed on the white glazed tile.

iv) Add few ml of standard solution from the burette into conical flask then put one drop of solution from conical flask over the drop of indicator with the help of glass rod .

v) If the blue colour appears it indicates that the end point has not yet reached. Then again add few drops of standard solution to the conical flask solution and mix then properly by shaking.

vi) Repeat this process till the drop of indicator does not change it colour when drop of sample solution is placed over it.

vii) The reading of the burette at this point will give the volume of $K_2Cr_2O_7$ required for oxidation.

viii) Repeat the process till you get two concordant readings.

OBSERVATIONS:

S No	Volume of sample solution taken	Burette	Readings	Range	Volume of N/30 $K_2Cr_2O_7$ consumed
		Initial	Final		
1					
2					
3					
4					

CALCULATION:

Let N_1 is the Normality of Mohr's salt solution, then
Mohr's salt solution = Potassium dichromate solution

$(N1V1)$ = (N_2V_2)

$N_1 \times 10$ = $(N/30 \times V_2)$

N_1 = $N/30 \times V_2/10$ = $V_2/300$

Strength in gram per litre = Normality × Equivalent weight
= N_1 × 56 gm/litre

RESULT:

The ferrous ion content in the given iron ore solution is _____ gm/litre.

PRECATIONS:

i) Wash the glass apparatus properly before using it.
ii) $K_2Cr_2O_7$ acts as an oxidizing agent in acidic medium only so one should before titration keeping mind to add dilute H_2SO_4 solution to Mohr's salt solution.
iii) The glass rod should be washed each time before using for withdrawing a drop from the conical flask.