

## EXPERIMENT NO – 1

**OBJECT:** To determine the ferrous ion ( $\text{Fe}^{+2}$ ) content in given sample solution of iron (Mohr's salt,  $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ) by titrating it against standard N/30 potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) solution by using potassium ferricyanide  $\text{K}_3[\text{Fe}(\text{CN})_6]$  as an external indicator.

### REQUIRMENTS:

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

**CHEMICALS:** Standard N/30 Potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) solution, Unknown sample solution of Iron (Mohr's salt), dilute  $\text{H}_2\text{SO}_4$

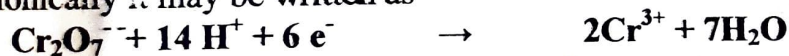
**INDICATOR:** Potassium ferricyanide  $\text{K}_3[\text{Fe}(\text{CN})_6]$  IUPAC name potassium hexa cyano ferrate (III).

### PRINCIPLE:

Potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) acts as an oxidizing agent in acidic medium and each molecule of it gives three atoms of nascent oxygen. The following reaction takes place



Ionicly it may be written as



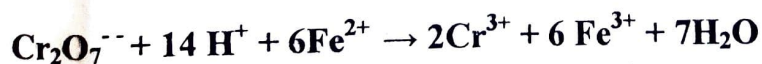
When acidic potassium dichromate is added to (standard solution), of iron ore solution, only  $\text{FeSO}_4$  is oxidized and ammonium sulphate remains unchanged. The following reaction takes place:



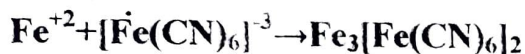
or



The overall reaction and ionic reaction is:

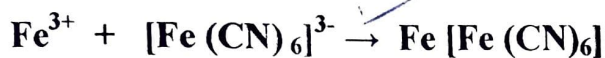


In this titration potassium ferricyanide  $\text{K}_3[\text{Fe}(\text{CN})_6]$  is used as an external indicator, if ferrous ion ( $\text{Fe}^{+2}$ ) are present a strong blue color is developed due to the following reaction :



**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.



**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  $\text{H}_2\text{SO}_4$  to it.
- ii) Fill the burette with standard N/30  $\text{K}_2\text{Cr}_2\text{O}_7$  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  $\text{K}_2\text{Cr}_2\text{O}_7$  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 $\text{K}_2\text{Cr}_2\text{O}_7$ 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

$$\begin{aligned}(N_1 V_1) &= (N_2 V_2) \\ N_1 \times 10 &= (N/30 \times V_2) \\ N_1 &= N/30 \times V_2/10 = V_2/300\end{aligned}$$

$$\begin{aligned}\text{Strength in gram per litre} &= \text{Normality} \times \text{Equivalent weight} \\ &= N_1 \times 56 \text{ gm/litre}\end{aligned}$$

### RESULT:

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

### PRECAUTIONS:

- 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.