

## ESTIMATION OF IRON BY POTENTIOMETRIC TITRATION

Aim:

To estimate the amount of  $\text{Fe}^{2+}$  ion present in the given solution.

Apparatus Required:

Potentiometer assembly, 25ml of burette, 10ml pipette, 250 ml beakers, standard flask, calomel and platinum electrodes.

Reagents Required:

Ferric Ammonium Sulphate, dil  $\text{H}_2\text{SO}_4$ , standard  $\text{K}_2\text{Cr}_2\text{O}_7$ .

Principle:

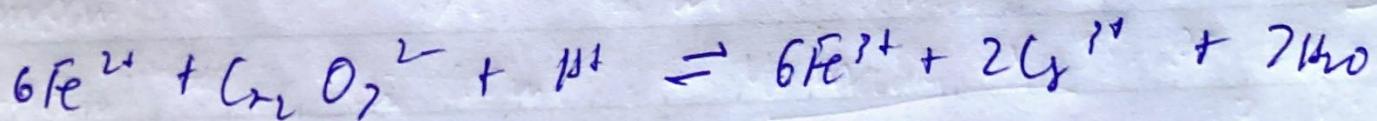
Measurement of one of electrochemical all using potentiometer and change in EMF due to the chemical (redox) reactions are monitored. In this potentiometric titration set up an indicator electrode (Pb electrodes) & reference electrode (calomel) is coupled to form electrochemical cell for  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$ .

FAS    v/s     $\text{H}_2\text{Cr}_2\text{O}_7$     (Pilot)    Titration

| Sr No | Vol of $\text{H}_2\text{Cr}_2\text{O}_7$ (mL) | GMF (v) | DE (v) |
|-------|---|---------|--------|
| 1     | 0.0   | 328     | 0      |
| 2     | 1.0   | 358     | 30     |
| 3     | 2.0   | 375     | 17     |
| 4     | 3.0   | 389     | 14     |
| 5     | 4.0   | 399     | 10     |
| 6     | 5.0   | 410     | 11     |
| 7     | 6.0   | 420     | 10     |
| 8     | 7.0   | 437     | 17     |
| 9     | 8.0   | 454     | 17     |
| 10    | 9.0   | 492     | 38     |
| 11    | 10.0  | 646     | 154    |
| 12    | 11.0  | 660     | 14     |
| 13    | 12.0  | 669     | 09     |
| 14    | 13.0  | 673     | 4      |
| 15    | 14.0  | 676     | 3      |
| 16    | 15.0  | 680     | 5      |

The cell set-up:  $\text{Hg} | \text{HgCl}_2(\text{s})$ ,  $\text{KCl}$  (1M) ||  $\text{Fe}^{2+} | \text{Fe}^{3+}$ ,  $\text{PQ}$

The chemical reaction:



$\text{Fe}^{2+}$  is oxidized to  $\text{Fe}^{3+}$  as  $\text{K}_2\text{Cr}_2\text{O}_7$  progressively added. Platinum electrode which is kept in contact with a mixture of  $\text{Fe}^{2+}$  &  $\text{Fe}^{3+}$  ions at a redox electrode (indicating the redox reaction). The reduction potential of the right electrode depends on the ratio of  $[\text{Fe}^{2+}/\text{Fe}^{3+}]$  initially. During the titration of  $\text{Fe}^{2+}$  in  $\text{H}_2\text{O}_2$  medium with  $\text{K}_2\text{Cr}_2\text{O}_7$ , this ratio varies to a little extent at the beginning and suddenly near the end point. After the end point in the ratio changes very little. It can be noted that there is a sudden change in the ratio of  $[\text{Fe}^{2+}/\text{Fe}^{3+}]$  at the equivalent point reached. This causes a sudden increase in the emf of the cell at equivalent point.

### PROCEDURE →

- 1) The given ferrous iron solution is made up to a known value in a SMT following the standard procedure using usual procedure.
- 2) roughly 10 ml of the mixed up  $\text{Fe}^{2+}$  solution is pipetted out into a clean 100 ml beaker about 10 ml of dil  $\text{H}_2\text{O}_2$  and 100 ml of distilled water are added to it.

F<sub>113</sub> V/V K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> (Ferric Titration)

| Sr.No. | Vol of K <sub>2</sub> C <sub>2</sub> O <sub>4</sub> (mL) | GMF(V) | ΔE(OV) | OV(mV) | ΔE/ΔOV(mV) |
|--------|--|--------|--------|--------|------------|
| 1      | 9.0  | 492    | 0      | 0      | 0          |
| 2      | 9.2  | 497    | 5      | 0.2    | 25         |
| 3      | 9.4  | 521    | 24     | 0.2    | 120        |
| 4      | 9.6  | 640    | 119    | 0.2    | 595        |
| 5      | 9.8  | 654    | 14     | 0.2    | 70         |
| 6      | 10.0   | 661    | 7      | 0.2    | 35         |
| 7      | 10.2   | 666    | 5      | 0.2    | 25         |
| 8      | 10.4   | 667    | 1      | 0.2    | 5          |
| 9      | 10.6   | 669    | 2      | 0.2    | 10         |
| 10     | 10.8   | 670    | 1      | 0.2    | 5          |
| 11     | 11.0   | 671    | 1      | 0.2    | 5          |
| 12     | 11.2   | 674    | 3      | 0.2    | 15         |
| 13     | 11.4   | 675    | 1      | 0.2    | 5          |
| 14     | 11.6   | 676    | 1      | 0.2    | 5          |
| 15     | 11.8   | 678    | 2      | 0.2    | 10         |
| 16     | 12.0   | 678    | 0      | 0.2    | 0          |

CALCULATION -

Mol of titrant mol<sup>-1</sup>(PAS) = 10 mL (V<sub>1</sub>).

Mol of K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> = 96 mL (V<sub>2</sub>).

Normality of K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> = 0.1N      Normality of PAS (M)  
= 0.096M.

Strength of PAS = V<sub>2</sub>N<sub>2</sub>/V<sub>1</sub> = 0.096 N.

Amount of Fe<sup>2+</sup> (g/L) = G<sub>2</sub> wt × Normality of Fe<sup>2+</sup> = 0.53616 g

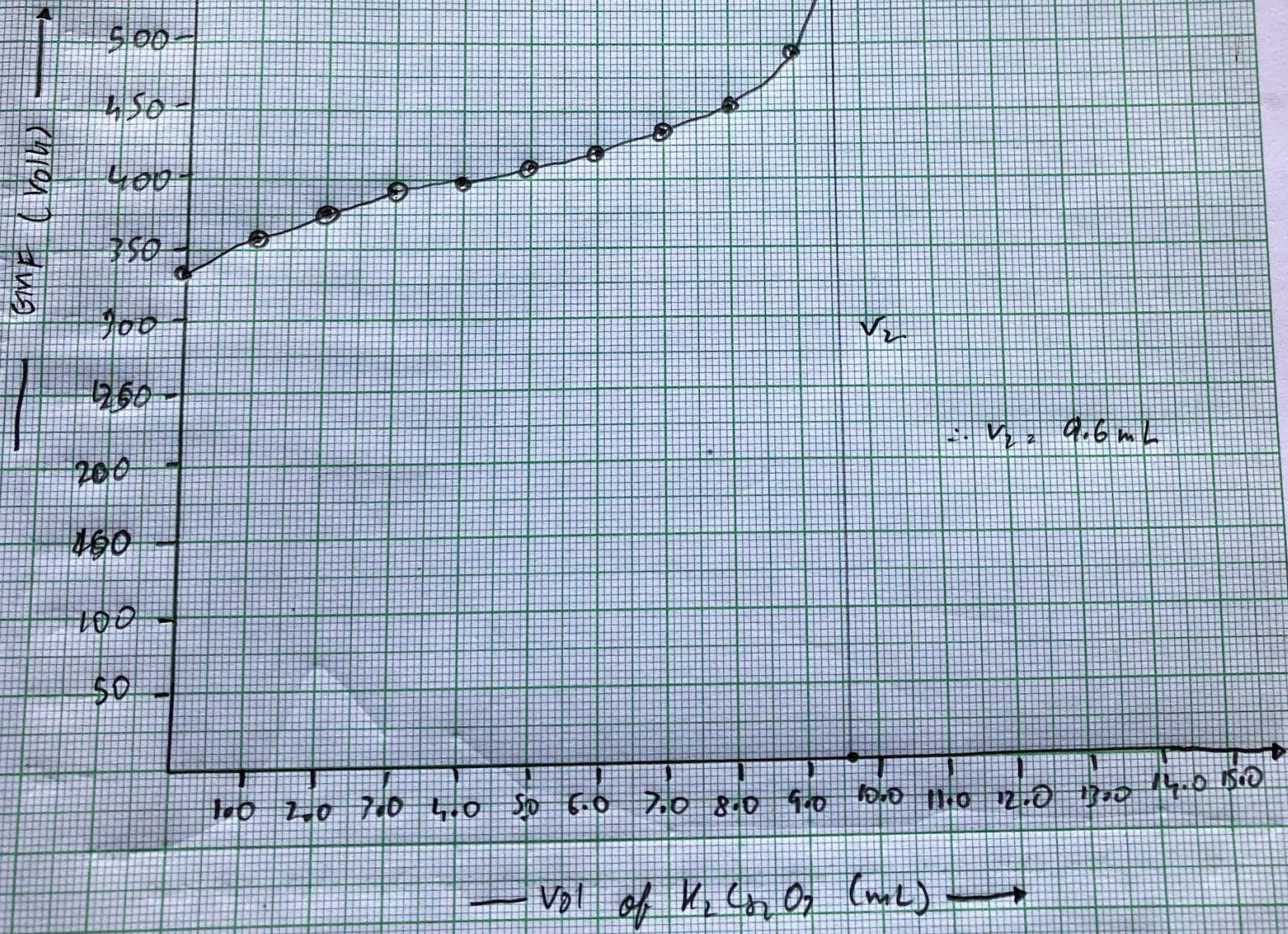
Amount of Fe<sup>2+</sup> in 100mL = Normality × 55.85  
= 2.994 g.

- 3) A platinum electrode is dipped into this solution and coupled with the standard calomel electrode. The resulting cell is then incorporated in the potentiometric circuit.
- 4) Standard  $K_2Cr_2O_7$  solution, which is taken in a burette, is added in installments of 1 mL into the beaker and the cell EMF is measured after each addition of reagent mixing.
- 5.1 The process is continued till and also well beyond the neutralization point as indicated by an abrupt change in the EMF.
- 6) Note the volume  $K_2Cr_2O_7$  solution required for complete oxidation of  $Fe^{2+}$  solution from the plot of EMF versus the volume of the standard  $K_2Cr_2O_7$  solution added. The range at which the endpoint lies may be evaluated.
- 7) Calculate the normality of given  $Fe^{2+}$  solution using the formula
- $$N_1 V_1 = N_2 V_2$$
- 8) Graph plotted in (i) EMF vs Volume  $K_2Cr_2O_7$  added  
(ii)  $\Delta E/\Delta V$  vs volume  $K_2Cr_2O_7$

Piloted Graph

Scale

$X_{axis} = 1 \text{ big unit} = 1 \text{ mL}$   
 $Y_{axis} = 1 \text{ big unit} = 50 \text{ V.}$

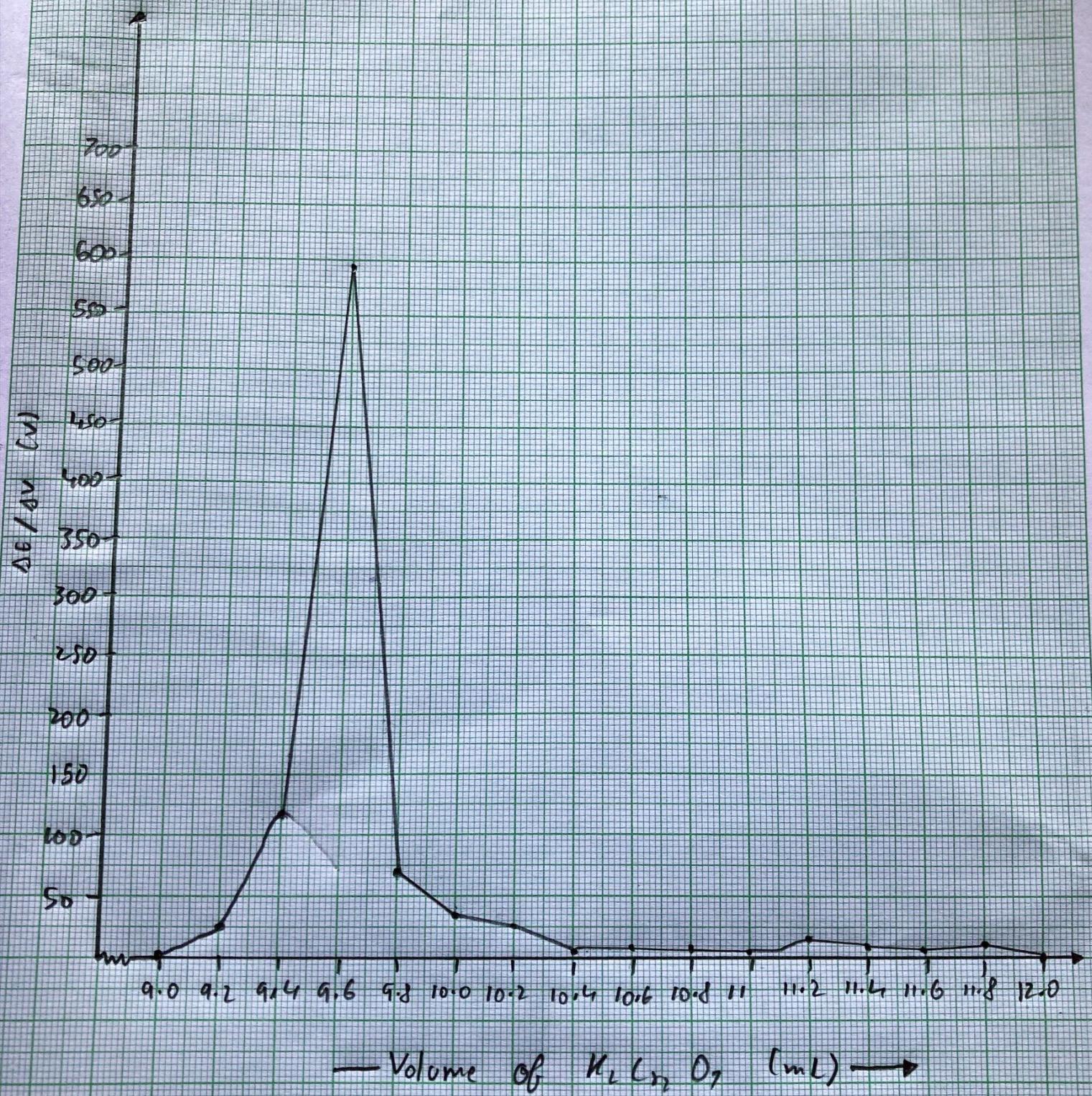


Fair Graph

Scale

X axis = 1 big Unit = 0.2 mL

Y axis, 1 big Unit = 0.2 mL



ii) The end point can be determined from the plot of  $\text{mV}$  versus volume of standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution.

### RESULTS -

The weight of iron present in the given solution = 0.53616 g.