Exp → Estimation of Iron by Potientiometric titration

Aim

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

Apparatus required

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

Reagents required

Ferrous ammonium sulphate, dil. H_2SO_4, Std. $K_2Cr_2O_7$

Principle

Measurement of Emf of a electrochemical cell using potentiometer and change in Emf due to the chemical (redox) reaction is monitored. In this potentiometric titration setup an indicator electrode (Pt electrode) & reference electrode (calomel) is coupled to form electrochemical cell for Fe^{2+} to Fe^{3+}.

Fe^{2+} is oxidizing to Fe^{3+} as $K_2Cr_2O_7$ as progressively added. Platinum electrode which is kept in contact with a mixture of Fe^{2+} & Fe^{3+} ions acts as a redox electrode (indicating redox rxn)

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The reduction potential of this single electrode depends on the ratio of $[Fe^{2+}/Fe^{3+}]$ initially. During the titration of Fe^{2+} in H_2SO_4 medium with $K_2Cr_2O_7$, this ratio varies to a little extent at the beginning and suddenly near the end point. After the end point it the ratio changes very little. It can be noted that there is a sudden change in the ratio of $[Fe^{3+}/Fe^{2+}]$ as the equivalence point is reached. This causes a sudden increase in the EMF of the cell at equivalence point.

Procedure

(i) The given ferrous iron solution is made upto a known volume (say 100 ml) in a SMF following the standard procedure with usual precautions.

(ii) Exactly 10 ml of the made up Fe^{2+} solution is pipette out into a clean 100 ml beaker. About 10 ml of dilute H_2SO_4 and 100 ml of distilled water are added to it.

(iii) A platinum electrode is dipped into this solution & coupled with the standard calomel electrode. The resultant cell is then incorporated into the potentiometric

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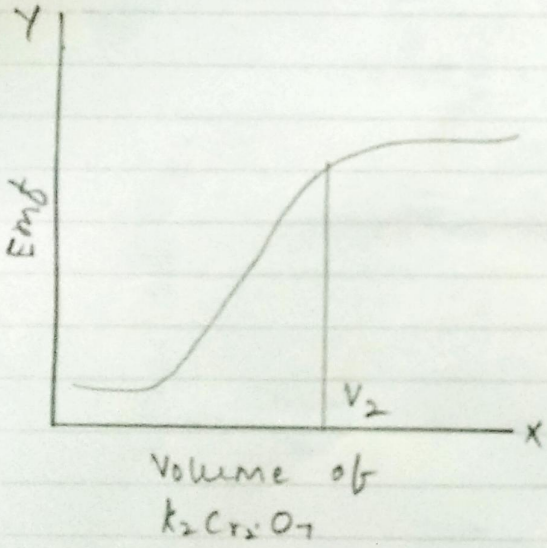
The cell setup:

$$Hg | Hg_2Cl_2(s), KCl(1N) || Fe^{2+} | Fe^{3+}, Pt$$

The chemical reaction:

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

Pilot titration



Fair titration

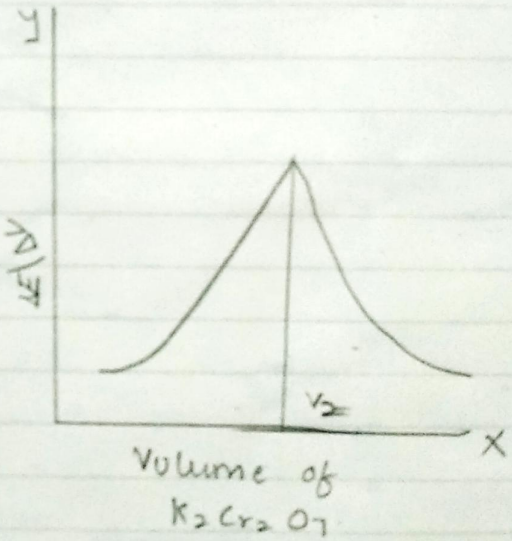


Table 1 : Pilot titration : FAS vs $K_2Cr_2O_7$

FAS → Ferrous Ammonium Sulphate

Sr. No.	Volume of $K_2Cr_2O_7$ (ml)	EMF (mV)	ΔE (mV)
1.	0	364	- Nil -
2.	1	380	16
3.	2	400	20
4.	3	423	23
5.	4	440	17
6.	5	460	20
7.	6	490	30
8.	7	530	40
9.	8	670	40
10.	9	690	20
11.	10.	701	11
12.	11	720	19
13.	12	740	20
14.	13	745	5
15.	14	760	15
16.	15	772	12

Pilot titration

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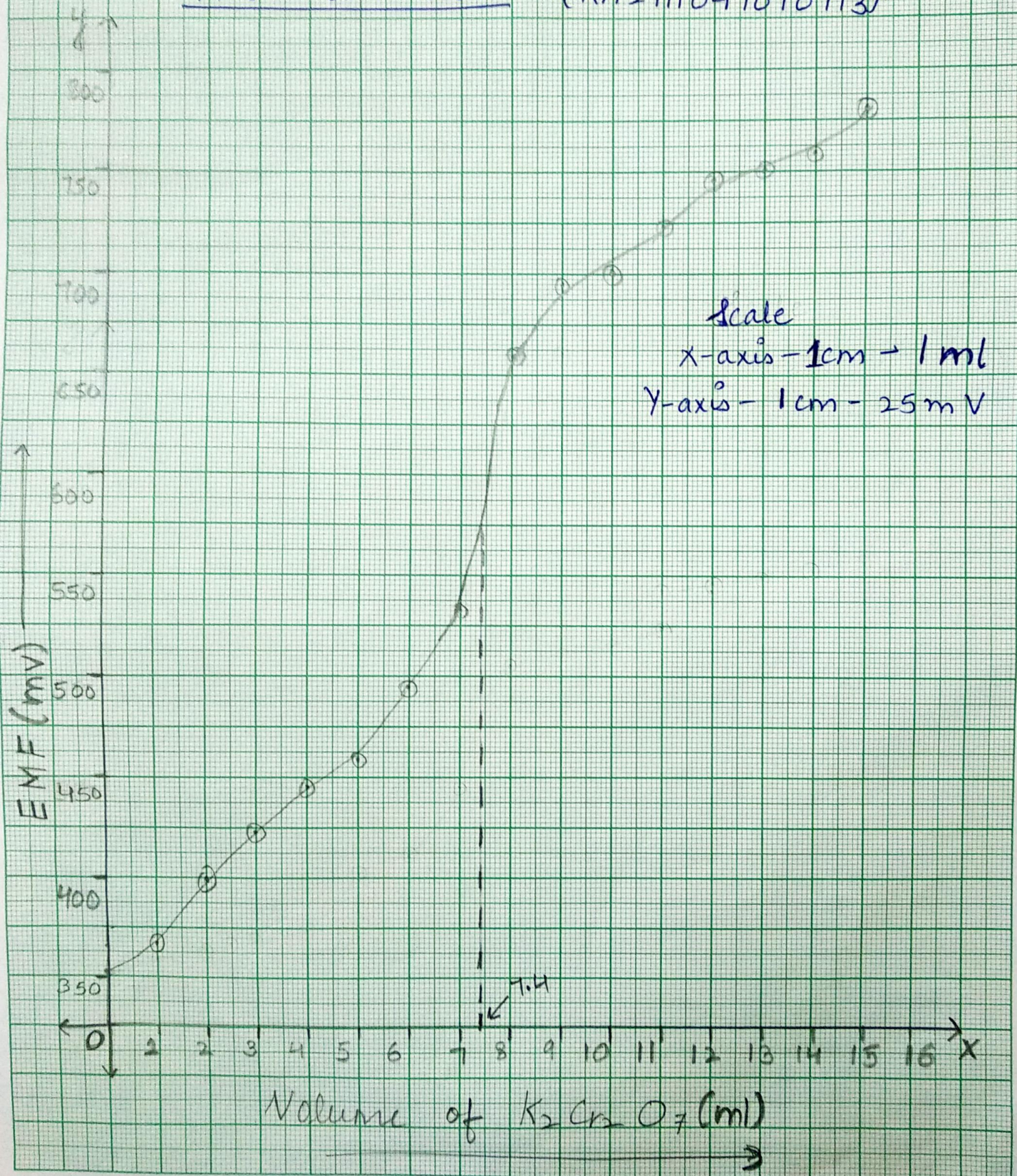

Table 2 : Fair Titration - FAS vs $K_2Cr_2O_7$

Sr. No.	Volume of $K_2Cr_2O_7$ (ml)	EMF (mV)	ΔE (mV)	ΔV (mL)	$\Delta E / \Delta V$
1.	5	465	—	—	—
2.	5.2	470	5	0.2	25
3.	5.4	480	10	0.2	50
4.	5.6	491	11	0.2	55
5.	5.8	496	5	0.2	25
6.	6	500	4	0.2	20
7.	6.2	510	10	0.2	50
8.	6.4	521	11	0.2	55
9.	6.6	533	12	0.2	60
10.	6.8	543	10	0.2	50
11.	7	550	7	0.2	35
12.	7.2	560	10	0.2	50
13.	7.4	630	70	0.2	350
14.	7.6	650	20	0.2	100
15.	7.8	670	20	0.2	100
16.	8	680	10	0.2	50
17.	8.2	685	5	0.2	25
18.	8.4	690	5	0.2	25
19.	8.6	694	4	0.2	20
20.	8.8	704	10	0.2	50
21.	9	710	6	0.2	30

Fair titration

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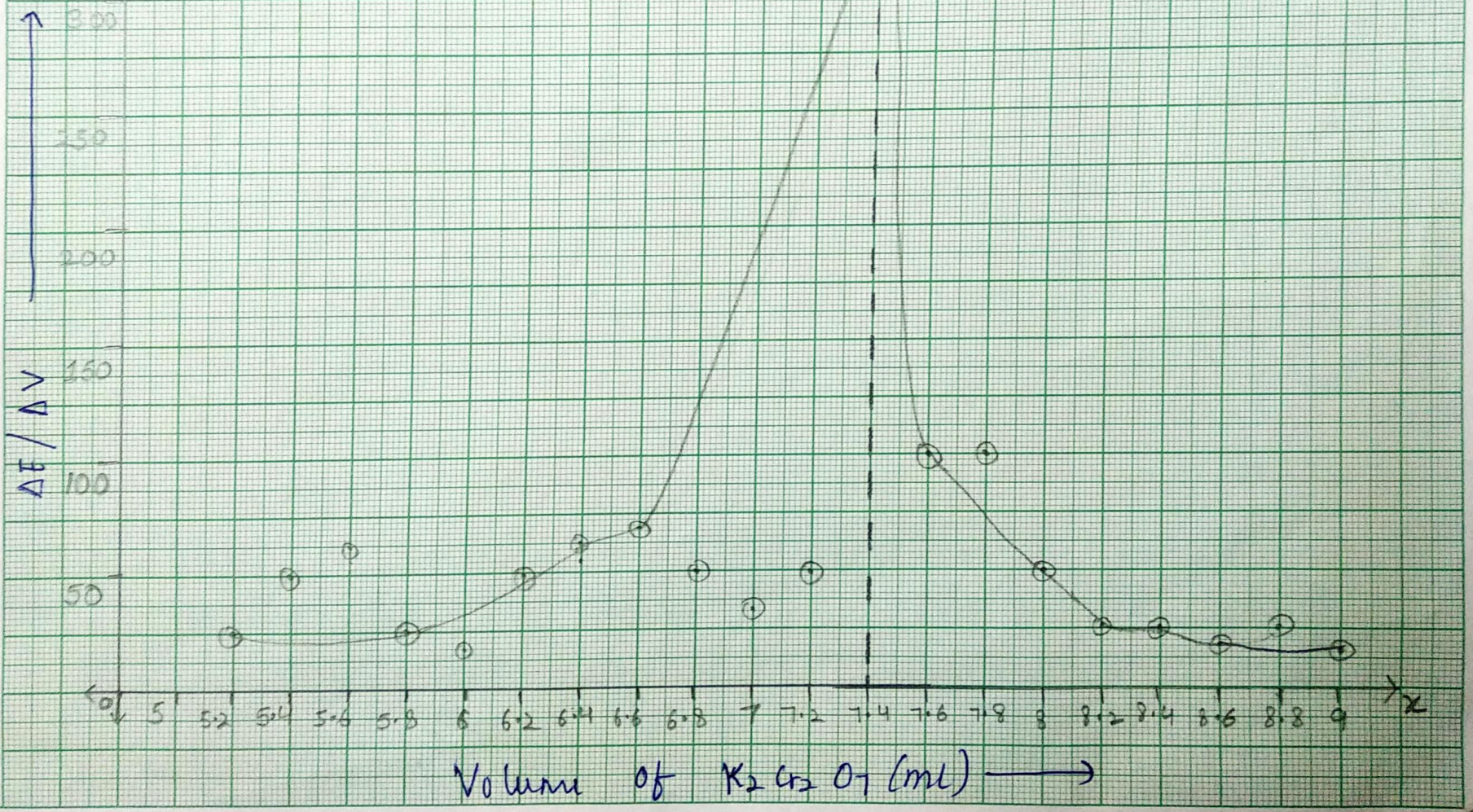
Scale

X-axis - 1 cm - 0.2 ml

Y-axis - 1 cm - 50 unit



* Calculation.

V_1 = Volume of pipette solution (FAS) = 10 ml

V_2 = Volume of $K_2Cr_2O_7$ = 7.41 ml

N_2 = Normality of $K_2Cr_2O_7$ = 0.1 N

Normality of FAS (N_1) = ?

Strength of FAS = $N_1 = \frac{V_2 N_2}{V_1}$

$= \frac{7.41 \times 0.1}{10} = 0.074\ N$

Normality = 0.074N

- Amount of Fe^{2+} (g/lit) = Eq. wt. × Normality of Fe^{2+}
  $= 55.85 \times 0.074$ (g/lit)

Amount of Fe^{2+} = 4.132 g/lit

- Amount in Fe^{2+} in 100 ml
  $= \frac{\text{eq. wt.} \times \text{Normality of } Fe^{2+}}{10}$
  $= \frac{55.84 \times 0.074}{10} = 0.0413$ grams.

* Result

Weight of iron in given solution = 0.0413 grams

circuit.

(iv) Standard $K_2Cr_2O_7$ solution, which is taken in a burette, is added in installments of 1ml into the beaker and the cell e.m.f. is measured after each addition by proper mixing.

(v) The process is continued still & also well beyond the neutralization point as indicated by an abrupt change in the e.m.f.

The cell setup → $Hg | Hg_2Cl_2(s), KCl(1N) || Fe^{2+} | Fe^{3+}, Pt$

The chemical reaction:

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

*(i) Note the volume $K_2Cr_2O_7$ solution required for complete oxidation of Fe^{2+} solution from the plot of e.m.f. vs the volume of the standard $K_2Cr_2O_7$ solution added. The range at which the endpoint lies may be evaluated.

(ii) Calculate the normality of given Fe^{2+} solution using the formula $N_1V_1 = N_2V_2$

(iii) One more similar titration is performed using 0.1 ml portions of $K_2Cr_2O_7$ (std.)

solution close to the end point (1ml on either side of the range) and tabulating the measured emf corresponding to each addition.

(iv) Graph is plotted (i) Emf vs Volume of $K_2Cr_2O_7$ addition

(ii) $\Delta E / \Delta V$ vs volume of $K_2Cr_2O_7$ addition

(v) The exact end point can be determined from the plot of $\Delta E / \Delta V$ versus volume of standard $K_2Cr_2O_7$ solution.

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