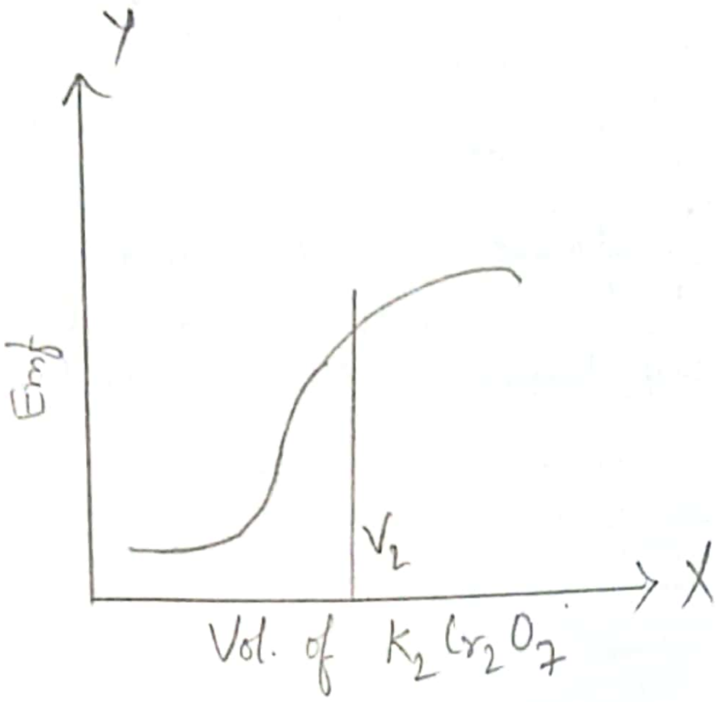
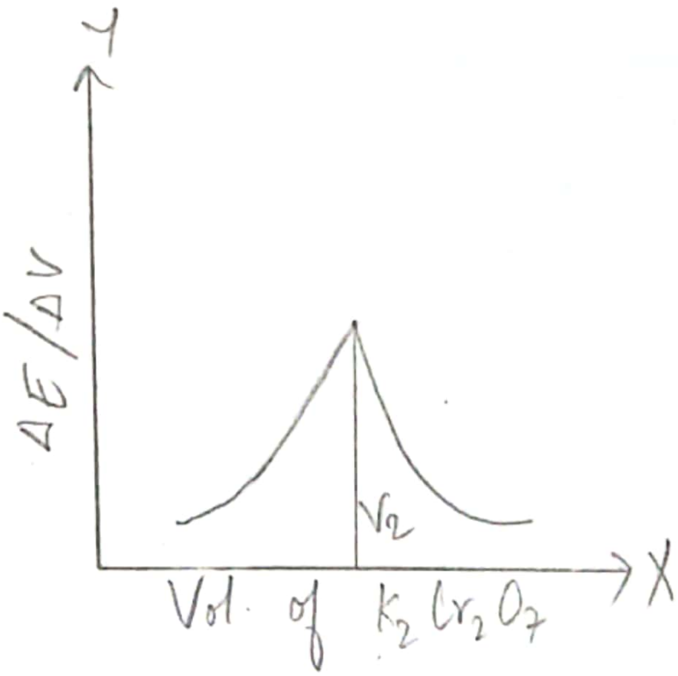


(1) Pilot titration



(2) Fair titration

Date 18·10·21

Expt. No. 4 Page No.

ESTIMATION OF IRON BY POTENTIOMETRIC 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, 250 ml beakers, standard flask, calomel and platinum electrodes.

★ REAGENTS REQUIRED:

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

★ PRINCIPLE:

→ In a titration, there is a change in ionic concentration which can be followed by measuring the potential of a suitable electrode.

→ The potential of an electrode depends upon the concentration of the ions in accordance to Nernst equation,

Eg: $O^{n+} + ne^- \longrightarrow R$

$\therefore E = E^0 - (RT/nF) \ln [R/O^{n+}]$

where E^0 is standard reduction potential.

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

→ The chemical reaction:

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

→ P.T.O

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★ OBSERVATIONS:

(1) TABLE 1:

S.No	Vol. of $K_2Cr_2O_7$ (ml)	EMF(V)	ΔE (V)	ΔV (ml)	ΔE/ΔV ($V ml^{-1}$)
1	7.0	433	–	–	–
2	7.2	441	8	0.2	40
3	7.4	452	11	0.2	55
4	7.6	464	12	0.2	60
5	7.8	516	52	0.2	260
6	8.0	599	83	0.2	415
7	8.2	622	23	0.2	115
8	8.4	632	10	0.2	50
9	8.6	640	8	0.2	40
10	8.8	645	5	0.2	25
11	9.0	649	4	0.2	20
12	9.2	652	3	0.2	15
13	9.4	655	3	0.2	15
14	9.6	658	3	0.2	15
15	9.8	660	2	0.2	10
16	10.0	667	2	0.2	10

Date

Expt. No. Page No.

* PROCEDURES :

→ The given ferrous ion solution is made up to a known volume in a SMF following the standard procedure with usual precautions.

→ 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.

→ A platinum electrode is dipped into this solution and coupled with the standard calomel electrode. The resultant cell is then incorporated into the potentiometric circuit.

→ 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 e.m.f is measured after each addition by proper mixing.

→ The process is continued till and also well beyond the neutralization point as indicated by an abrupt change in the emf.

→ Note the volume $K_2Cr_2O_7$ solution required for complete oxidation of Fe^{2+} solution from the plot of e.m.f versus the volume of the standard $K_2Cr_2O_7$ solution added. The range at which the endpoint lies maybe evaluated.

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

→ One more similar titration is performed by adding 0.1 ml portions of standard $K_2Cr_2O_7$ solution close to the end point and tabulating the measured e.m.f corresponding to each addition.

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(2) TABLE 2:

S.No.	Vol. of $K_2Cr_2O_7$ (ml)	EMF (V)	ΔE (V)	ΔV (ml)	$\Delta E/\Delta V$ ($V ml^{-1}$)
1	7.0	433	—	—	—
2	7.2	441	8	0.2	40
3	7.4	452	11	0.2	55
4	7.6	464	12	0.2	60
5	7.8	576	52	0.2	260
6	8.0	599	83	0.2	415
7	8.2	622	23	0.2	115
8	8.4	632	10	0.2	50
9	8.6	640	8	0.2	40
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13	9.4	655	3	0.2	15
14	9.6	658	3	0.2	15
15	9.8	660	2	0.2	10
16	10.0	667	2	0.2	10

Expt. No. Page No. Date

→ Graph is plotted (1) EMF vs Volume of $K_2Cr_2O_7$ addition.
(2) $\Delta E/\Delta V$ vs Volume of $K_2Cr_2O_7$ addition.

→ The exact end point can be determined from the plot of $\Delta E/\Delta V$ vs Volume of standard $K_2Cr_2O_7$ solution.

★ CALCULATIONS:

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

Volume of $K_2Cr_2O_7$ = V_2 ml (from graph) = 8 ml

Normality of $K_2Cr_2O_7$ = N_2 = 0.1 N

Normality of FAS (N_1) = ?

Strength of FAS = $N_1 = \frac{N_2V_2}{V_1} = \frac{8 \times 0.1}{10} = \frac{0.08}{10} = 0.008\ N$

Amount of Fe^{2+} (gl^{-1}) = 55.85×0.0008
= 0.4468

∴ Amount of Fe^{2+} in 100 ml = $\frac{0.4468}{10} \Rightarrow 0.04468\ g$

→

P.T.O

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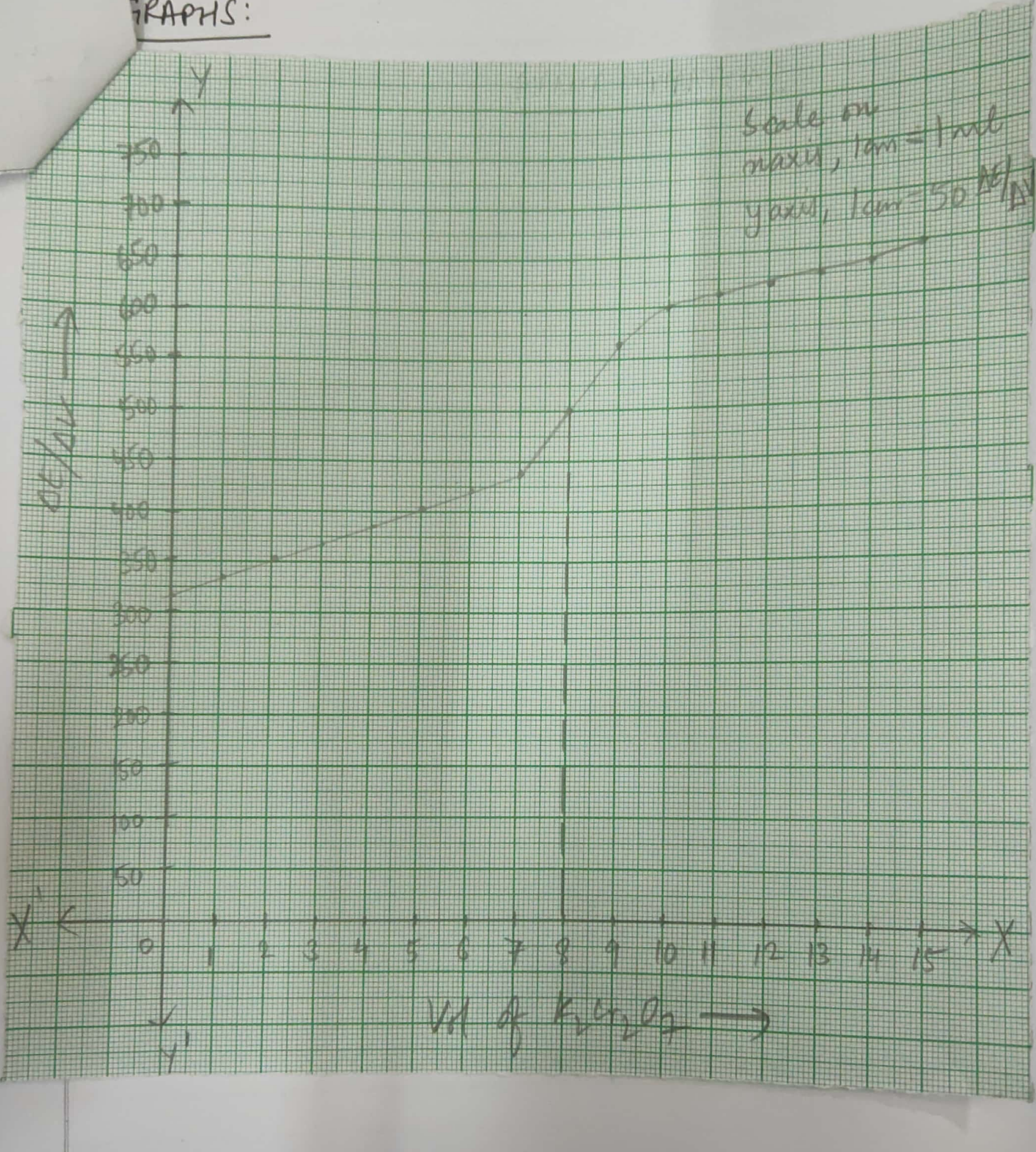
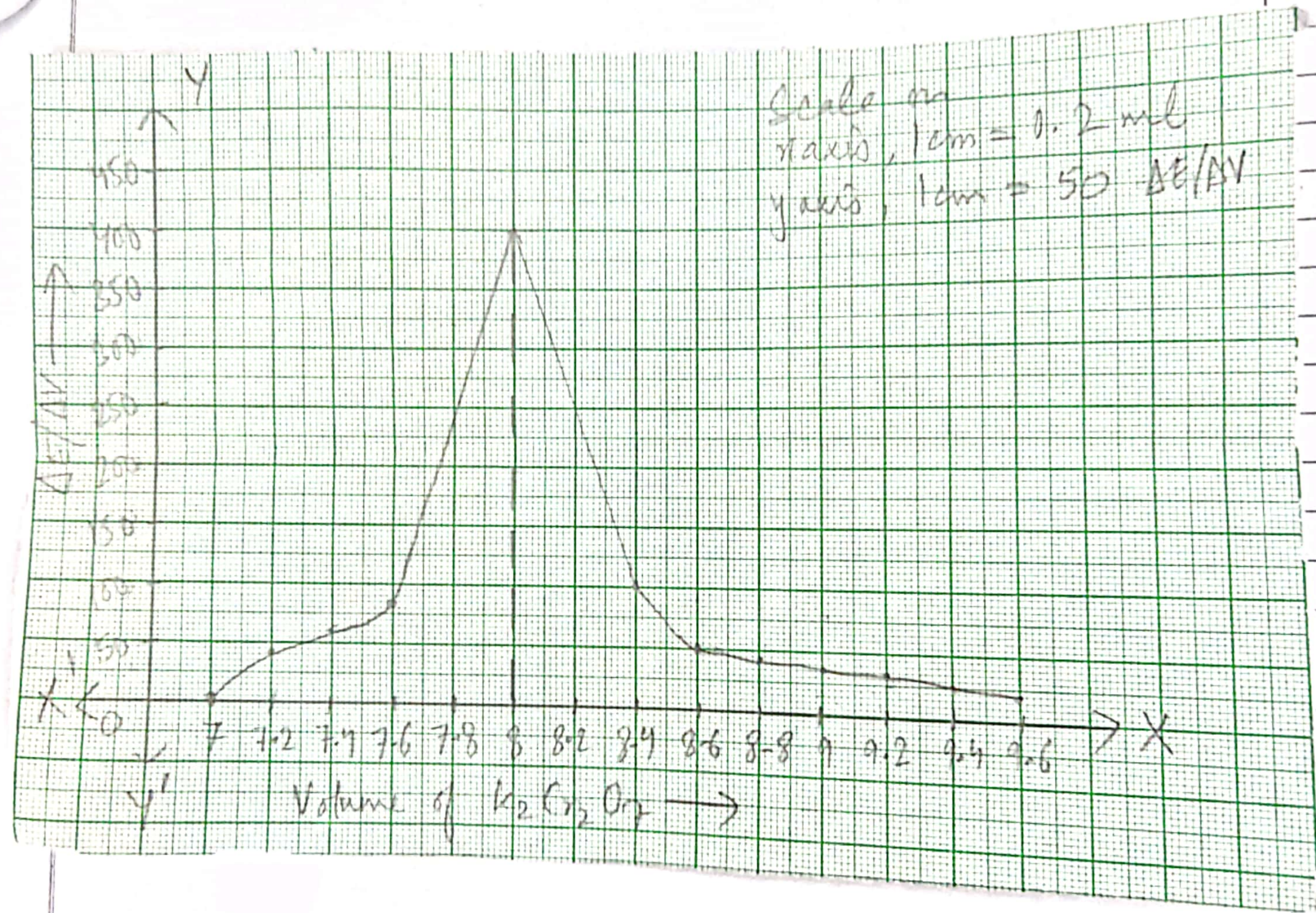
GRAPHS:


Scale on
x axis, 1cm = 1ml
y axis, 1cm = 50 ΔE/ΔV
ΔE/ΔV
Vol of $K_2Cr_2O_7$
Y
Y'
X
X'
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750

GRAPHS:



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★ RESULT:

→ The weight of Fe present in the given solution = 0.4468g

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