TABLES ⟹

1) Ferrous Ammonium Sulphate vs std. $K_2Cr_2O_7$.
(Pilot Titration)

Sr. No.	Volume of $K_2Cr_2O_7$	EMF
	ml	mV
1.	0	310
2.	1	325
3.	2	344
4.	3	356
5.	4	369
6.	5	383
7.	6	399
8.	7	417
9.	8	507
10.	9	632
11.	10	639
12.	11	654
13.	12	660
14.	13	663
15.	14	666
16.	15	670

2) Ferrous Ammonium Sulphate vs std. $K_2Cr_2O_7$.
(Fair Titration)

Date 20/05/2021

Expt. No. 4 Page No.

ESTIMATION OF IRON BY POTENTIOMETER TITRATION.

AIM ⟹

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

APPARATUS REQUIRED ⟹

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

REAGENTS ⟹

1) Ferrous Ammonium Sulphate (FAS).
2) dil. Sulphuric Acid (H_2SO_4).
3) std. Potassium Dichromate ($K_2Cr_2O_7$).

PRINCIPLE ⟹

Measurement of EMF of an electrochemical cell using potentiometer and change in EMF due to the chemical (redoæ) reaction is monitored. In this potentiometer titration, setup an indicator electrode to Fe^{3+}.

Fe^{2+} is oæidised to Fe^{3+} as $K_2Cr_2O_7$ progressively added. Platinum electrode which is kept in contant with a miæture of Fe^{2+} and Fe^{3+} ions acts as a redoæ electrode. During the titration of Fe^{2+} in H_2SO_4 medium with $K_2Cr_2O_7$, this ratio varies to a little eætent at the beginning and suddenly

Teacher's Signature

Sr. No.	Volume of $K_2Cr_2O_7$	EMF	ΔE	ΔV	$\Delta E / \Delta V$
	ml	mV	mV	ml	
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	662	2	0.2	10

CALCULATIONS $\Longrightarrow$

V_1 = Volume of pipette (10 ml)

N_1 = Normality of FAS

V_2 = Volume of $K_2Cr_2O_7$ (from graph)

N_2 = Normality of $K_2Cr_2O_7$ (0.1 N)

$$\therefore N_1 = \frac{V_2 \times N_2}{V_1} = \frac{8 \times 0.1}{10} = 0.08\ N$$

near the endpoint. This causes a sudden increase in the EMF of two cells at equivalent point.

PROCEDURE ⟹

• PILOT TITRATION :

1) The given Fe^{2+} (Ferrous iron solution) is made up of known volume (say 100ml) in a standard volumetric flask following a standard procedure with usual precautions.

2) Exactly 10ml of made up Fe^{2+} solution is pipetted out into a clean 250ml beaker. About 10ml of dil. H_2SO_4 and 100ml of distilled water are added to it.

3) 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.

4) Standard $K_2Cr_2O_7$ solution which is taken in a burette is added in installments of 1ml into the beaker and the cell EMF is measured after each addition by proper mixing.

5) The process is continued till and also well beyond the equivalence point as indicated by an abrupt change in EMF.

6) The volume of $K_2Cr_2O_7$ solution required for complete oxidation of Fe^{2+} solution is noted from the plot of EMF versus the volume of standard $K_2Cr_2O_7$ solution added. The range at which the endpoint lies may be evaluated.

now, amount of Fe^{2+} = eq. weight × Normality of Fe^{2+}

$\therefore$ Amount of Fe^{2+} in 100ml = $\frac{N \text{ of } Fe^{2+} \times 55.85}{10}$

$= \frac{0.08 \times 55.85}{10} = \frac{4.468}{10}$

$= 0.4468$ Mg.

• FAIR TITRATION :

1) Step 2 and 3 (of pilot titration) are repeated as above and the burette is filled upto 0ml mark.

2) One more similar titration is performed by adding 0.2 ml portions of standard $K_2Cr_2O_7$ solution close to the endpoint (2ml on either side of abrupt change in EMF) and the EMF corresponding to each addition is recorded.

3) The exact endpoint can be determined from the plot of $\Delta E/\Delta V$ versus volume of standard $K_2Cr_2O_7$ solution.

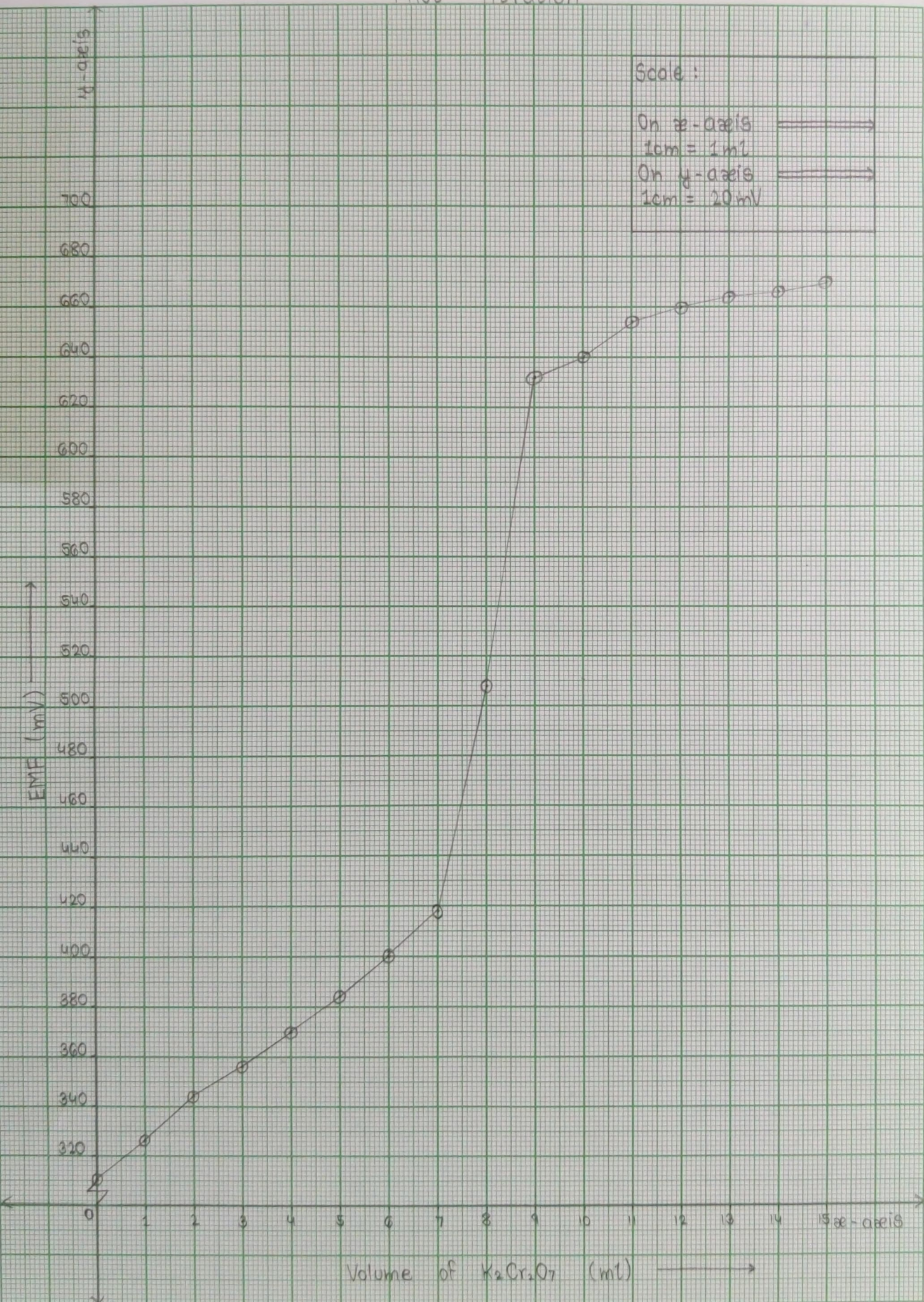
4) The normality of given Fe^{2+} solution is calculated using the formula : $V_1N_1 = V_2N_2$.

RESULT ⟹

The weight of Iron present in the given solution = 0.4468 g.

Teacher's Signature _____

Pilot Titration



Fair Titration