

LAB EXPERIMENT – 5

Course: Engineering Chemistry

Course Code: CHY1701

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Material Analysis: Iron in Carbon Steel by Potentiometry

1. Importance of the experiment: Steel production is an index of national prosperity and economy, and is globally used in variety of industrial sectors like shipbuilding, automobiles, construction, machinery and tools. Carbon steel is one variety in which nearly 96% of iron is alloyed with nearly 2% of carbon and other elements like manganese, chromium, nickel and copper. The composition is varied for achieving desired strength, ductile and long-term wearing properties. Thus, qualitative determination of iron in steel is very important.

2. Concept: Potentiometric titration is a process of determining the quantity of a sample by adding measured increments of a titrant until the end-point. The potential difference between indicator and reference electrodes is measured under conditions where the current passed is sufficiently small to maintain thermodynamic equilibrium. Potentiometric titrations provide reliable data than conventional titrations with chemical indicators especially with coloured or turbid solutions. In this experiment, Fe^{2+} is oxidised to Fe^{3+} by KMnO_4 as a redox titration.



Change in the concentration of Fe^{2+} ions during the addition of KMnO_4 is monitored by measuring the solution potential which is the basis for this experiment. From Nernst equation, a measurable quantity - voltage or potential is related to the concentration of species ($\text{Fe}^{3+}/\text{Fe}^{2+}$) in the solution.

$$E = E_0 + \frac{RT}{nF} \ln\left(\frac{\text{Fe}^{3+}}{\text{Fe}^{2+}}\right)$$

At the end point, a rapid change in the potential would be observed indicative of the complete conversion of Fe^{2+} to Fe^{3+} . A plot of observed potential vs volume of KMnO_4 consumed or its first derivative graph ($\Delta E/\Delta V$ vs average volume of KMnO_4) is used to detect the titration end point, which in turn, is used to qualitatively measure the amount of Fe^{2+} .

3. Applications: Potentiometry method is an electroanalytical technique which can be used to determine accurately the iron content in steel samples for industrial applications without using any indicator. This method is also useful for dilute or unknown samples or compositions for which identification of appropriate chemical indicators are challenging.

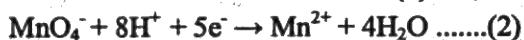
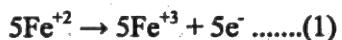
Expt. No.: 5

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Experiment	Material Analysis: Iron in Carbon Steel by Potentiometry
Problem definition	Mechanical properties of steel depend on its composition. Hence, it is important to analyze the amount of Iron in steel for industrial applications.
Methodology	Potentiometric method using KMnO_4 (oxidizing agent) to oxidize $\text{Fe}(\text{II})$ in steel to $\text{Fe}(\text{III})$ facilitates the estimation of Iron in steel.
Solution	Estimation of iron (%) in different steel samples.
Student learning outcomes	Students will learn to a) perform potentiometric method b) analyze the composition of iron in different grades of steel

Principle:

Potassium permanganate (KMnO_4) oxidizes ferrous ion to ferric ion in the presence of acid as per the reaction:



Electrode potential (oxidation potential) in the titration depends upon the concentration of Fe^{2+} , Fe^{3+} and H^+ ions. To avoid the effect of the change in H^+ ion concentration, the titration is usually carried out in large excess of acid. Oxidation potential of this redox system is given by

$$E = E_0 + \frac{RT}{nF} \ln\left(\frac{\text{Fe}^{3+}}{\text{Fe}^{2+}}\right)$$

Connecting the redox electrode (Platinum) with a saturated calomel electrode (SCE) completes the necessary cell as indicated below:



When KMnO_4 is added, Fe^{2+} is oxidized to Fe^{3+} whose concentration increases with progressive addition of KMnO_4 . The observed EMF gradually increases. At the end point, there will be a sharp increase due to the sudden removal of all Fe^{2+} ions. Plot-1: EMF measured (E) vs Volume of KMnO_4 added and Plot-2: $\Delta E/\Delta V$ vs Average volume of KMnO_4 was drawn. End point of the titration is measured from the Plot-2 graph.

Requirements:

Reagents and solutions: 100 mL of KMnO_4 (0.05 N) solution, 100 mL of steel solution, 2 N H_2SO_4 .

Apparatus: Calomel electrode, Platinum electrode, Potentiometer, Volumetric flasks, Burette, Pipette, Beakers.

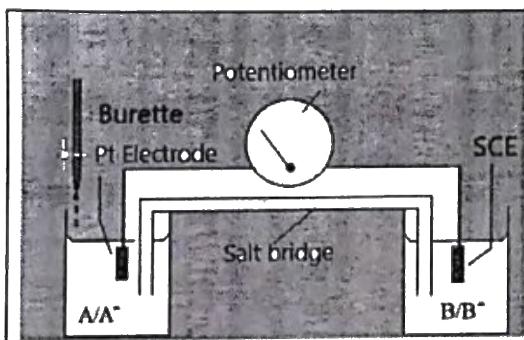


Fig. 1: Typical apparatus for potentiometric titrations.

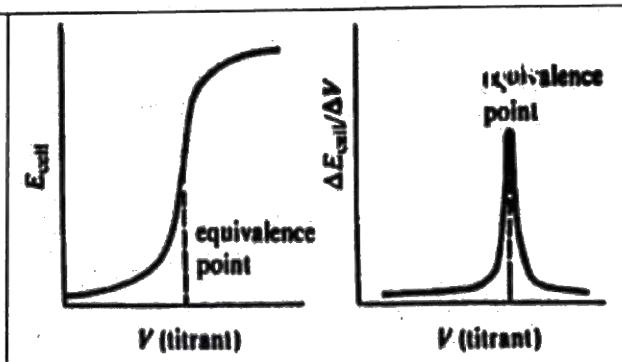


Fig. 2: Typical Potentiometric titration curves. (a) Normal curve and (b) First derivative curve.

Procedure:

Calibration of Potentiometer: Switch on the potentiometer and connect the standard cell terminals to either channel A (move channel switch to position A) or channel B (move the channel switch to position B). The meter should read 1.018 V. In case it is not 1.018 V, adjust the std. knob to obtain reference value.

Estimation of Fe(II) in steel: Transfer the given unknown steel [containing Fe(II)] solution into a clean 100 mL standard flask and make the solution up to the mark with distilled water and mix well. Pipette out 20 mL made up steel sample solution into a clean 100 mL beaker and add one test tube of dil. H_2SO_4 (2 N). Place Pt electrode in the beaker and connect to the +ve terminal of the potentiometer. In another beaker, place 50 mL of saturated KCl solution and dip the SCE in the solution and connect to the -ve terminal of the potentiometer. Place a salt bridge to complete the cell. Read the EMF of the cell and note down the value. Add 1 mL of $KMnO_4$ solution from the burette to the beaker containing steel sample solution. Stir the solution carefully and measure the EMF. Continue the addition of $KMnO_4$ solution and record the EMF for every 1 mL addition as per procedure till the potential shows a tendency to increase rapidly. After the abrupt change in cell EMF is observed, continue the titration to take 5 more reading by adding 1 mL burette solution every time. Plot EMF (ordinate) vs. volume of $KMnO_4$ added (abscissa) to get S-shaped curve which indicate the volume range of the end point.

To find out the volume of end point more precisely, carry out the 2nd titration in similar way but by adding 1 mL aliquots of $KMnO_4$ initially and then 0.1 mL aliquots between the two volumes where the end point is detected. Continue the titration beyond the end point as done above. The exact end point is determined by differential method i.e. by plotting $\Delta E/\Delta V$ vs average volume of $KMnO_4$ added. Calculate the normality strength of the Fe(II) in the given solution.

OBSERVATION AND CALCULATIONS

Potentiometric Titration-I:

Burette: KMnO₄ solution (0.05 N)

Beaker: 20 mL of steel solution containing Fe(II) + 20 mL (one test tube) of dil. H₂SO₄

Electrodes: Indicator electrode (Pt) to red terminal and SCE to black terminal

S. No.	Volume of KMnO ₄ (mL)	EMF (volts)	S. No.	Volume of KMnO ₄ (mL)	EMF (volts)
1	0	0.222	11		
2	1	0.306	12		
3	2	0.315	13		
4	3	0.369	14		
5	4	0.745	15		
6	5	0.915	16		
7	6	0.954	17		
8			18		
9			19		
10			20		

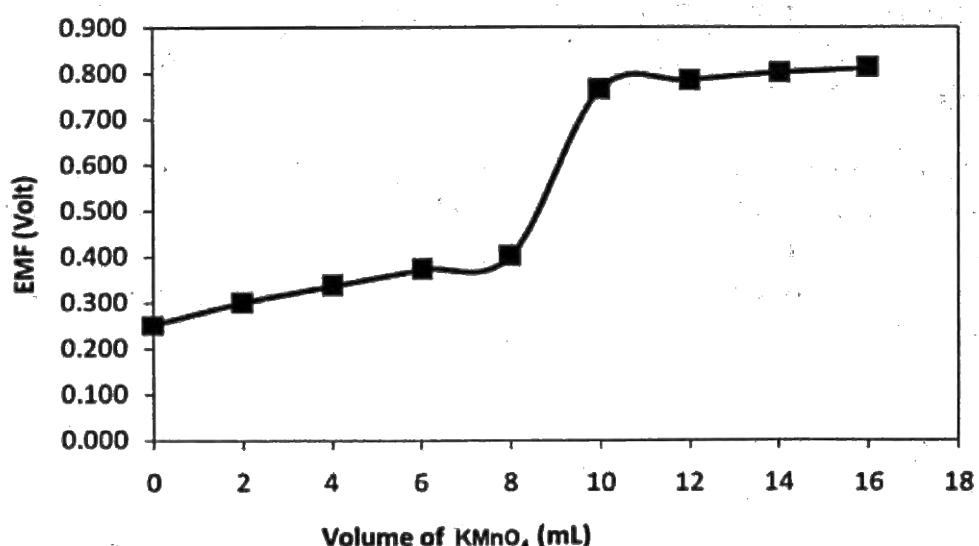
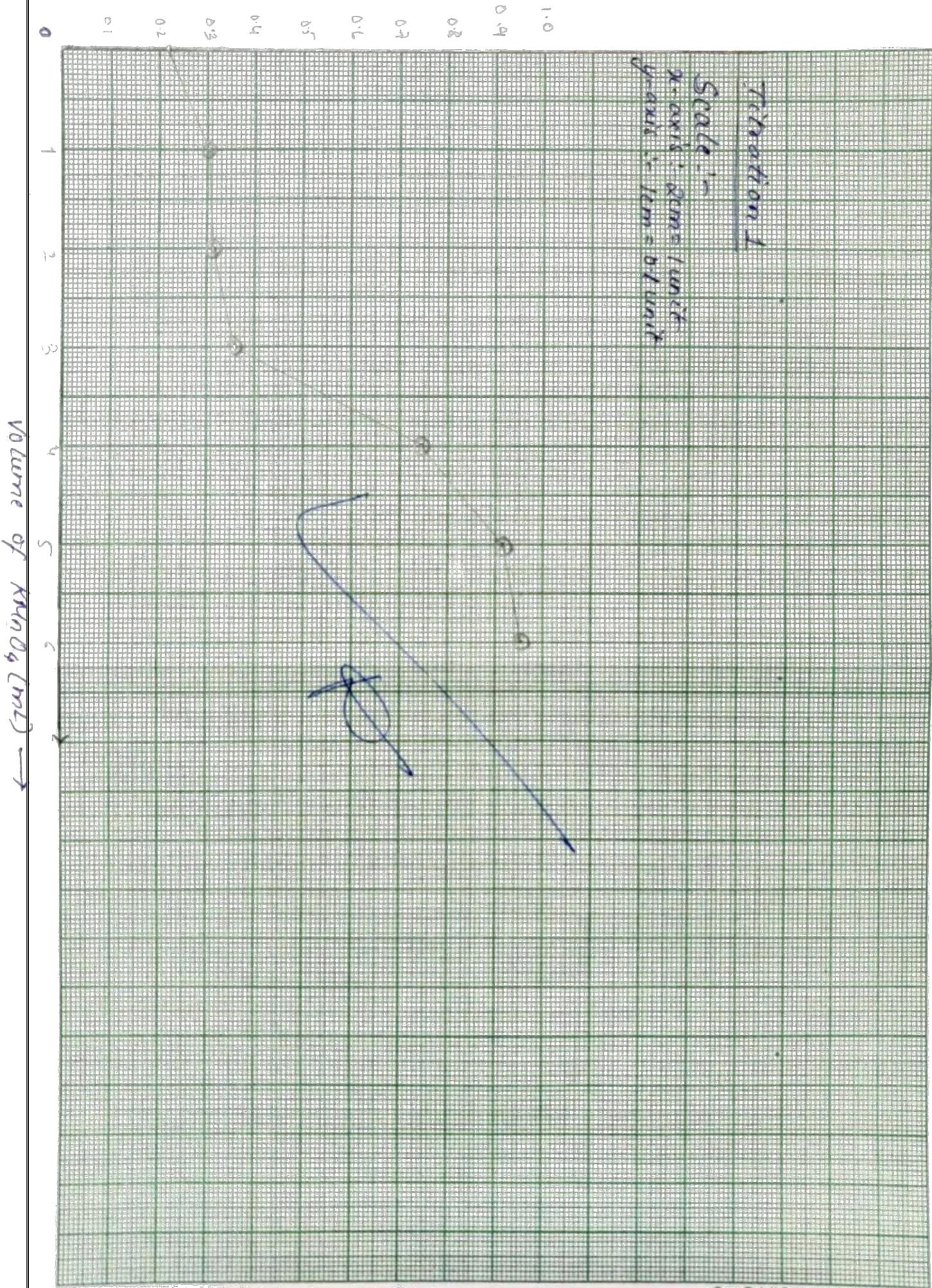


Fig. 3: Plot of EMF vs Volume of KMnO₄ added (mL)

Sample - ⑥

$E_{M.F.}$ (volt) \rightarrow



Potentiometric Titration-II:

Burette: KMnO₄ solution (0.05 N)

Beaker: 20 mL of steel solution containing Fe(II) + 20 mL (one test tube) of dil. H₂SO₄

Electrodes: Indicator electrode (Pt) to red terminal and SCE to black terminal

Sl. No.	Vol. of KMnO ₄ (mL)	EMF (Volt)	ΔE (Volt)	ΔV (mL)	ΔE/ΔV (Volt/mL)	Average Volume (mL)
1	2.0	0.337	-	-	-	- 2.1 -
2	2.2	0.342	0.005	0.2	0.025	2.1
3	2.4	0.345	0.003	0.2	0.015	2.3
4	2.6	0.355	0.010	0.2	0.050	2.5
5	2.8	0.376	0.021	0.2	0.105	2.7
6	3.0	0.392	0.016	0.2	0.080	2.9
7	3.2	0.410	0.018	0.2	0.090	3.1
8	3.4	0.420	0.010	0.2	0.050	3.3
9	3.6	0.441	0.021	0.2	0.105	3.5
10	3.8	0.515	0.074	0.2	0.370	3.7
11	4.0	0.869	0.354	0.2	1.77	3.9
12	4.2	0.964	0.095	0.2	0.475	4.1
13	4.4	0.990	0.026	0.2	0.130	4.3
14	4.6	0.998	0.008	0.2	0.040	4.5
15	4.8	1.005	0.007	0.2	0.035	4.7
16	5.0	1.009	0.004	0.2	0.020	4.9

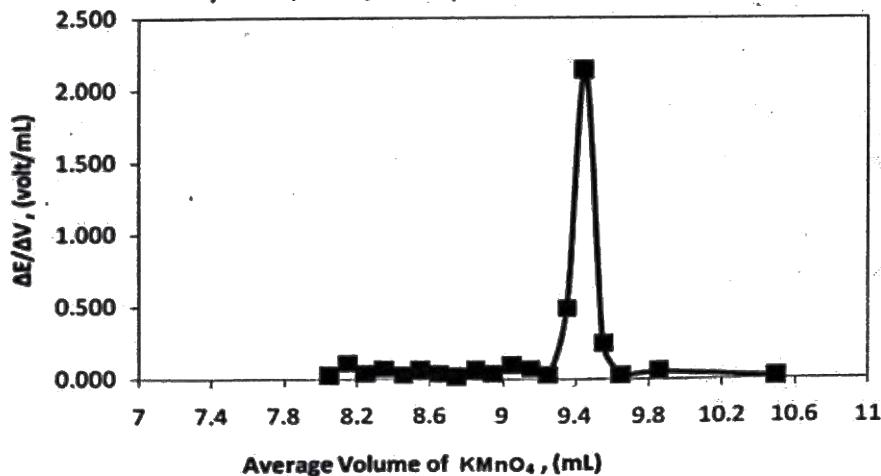


Fig. 4: Plot of $\Delta E/\Delta V$ vs Average volume of KMnO₄ added.

Sample ⑧

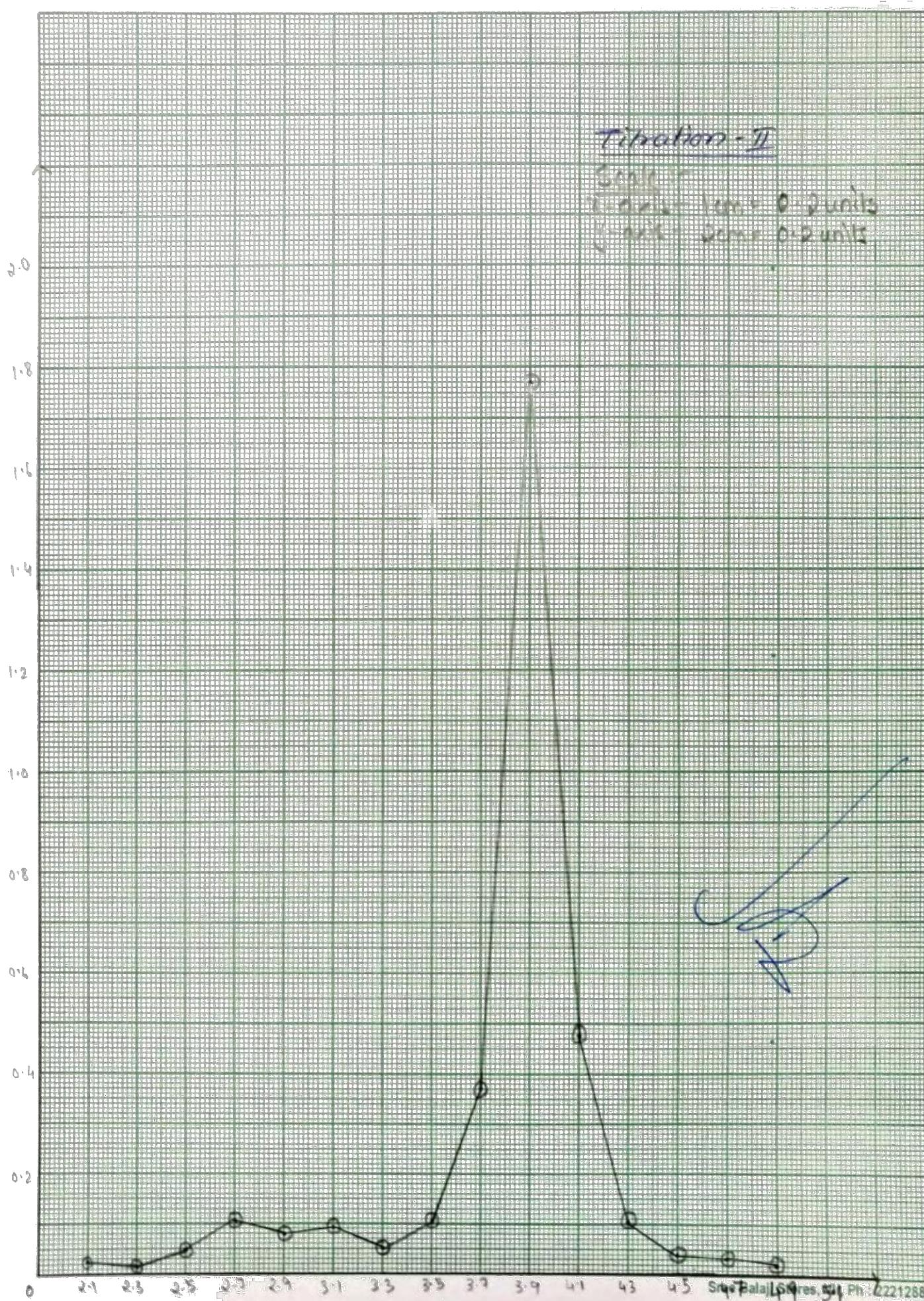
Titration - II

Graph

Initial volume = 0.0 ml

Final volume = 0.2 ml

$\Delta E/\Delta V$ (volt/mL) →



Average Volume of $KMnO_4$ (mL) →

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Calculation:

$$(N \times V) \text{ of steel sample solution} = (N \times V) \text{ of KMnO}_4$$

$$N \text{ of steel sample solution} = \underline{0.05 N \times \text{Volume of KMnO}_4 \text{ from Plot-2}}$$

$$\begin{aligned} & 20 \text{ mL of steel sample} \\ & = \underline{0.00975 \text{ N}} \end{aligned}$$

$$\text{Amount of Fe present in 1L of sample solution} = \text{Normality of steel sample} \times \text{Atomic weight of Fe (55.85)} = 0.545$$

$$\text{Amount of Fe present in given sample solution} = \underline{\text{Normality of steel sample} \times 55.85 \times 100}$$

$$\begin{aligned} & = \underline{0.0545 \frac{1000}{\text{grams in 100 mL}}} \\ & \quad \text{Revised value} \\ & \quad \text{1000} \\ & \quad \text{grams in 100 mL} \end{aligned}$$

Result: The amount of Iron present in given steel sample is found to be 0.0545 grams.

Evaluation of Result:

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded
8				