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Analysis of Iron in Steel by Potentiometric Method

Expt No. 10

Date: 15/03/18

Principle:

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

$$5Fe^{+2} \rightarrow 5Fe^{+3} + 5e$$

$$\underline{MnO_4^- + 8H^+ + 5e \rightarrow Mn^{2+} + 4H_2O}$$

$$\mathbf{5Fe^{+2} + MnO_4^- + 8H^+ \rightarrow 5Fe^{+3} + Mn^{2+} + 4H_2O}$$

The electrode potential (oxidation potential) in the titration depends upon the concentration of Fe^{2+}, Fe^{3+} and hydrogen ions.

To avoid the effect of the change in the hydrogen ion concentration, the titration is usually carried out in large excess of an acid.

The oxidation potential of this redox system is given by

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

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

$$\mathbf{Hg\,|\,Hg_2Cl_2\,(s),\ Saturated\ KCl\,||\,Fe^{3+}, Fe^{2+}\,|\,Pt}$$

When potassium permanganate ($KMnO_4$) is added, Fe^{2+} is oxidized to Fe^{3+} whose concentration increases with progressive addition of potassium permanganate. The observed EMF gradually increases. At the end point, there will be a sharp increase due to sudden removal of all Fe^{2+} ions. Plot between EMF (E) measured *vs* volume of $KMnO_4$ added and another plot between $\Delta E / \Delta V$ *vs*average volume of $KMnO_4$are made. The end point of the titration is measured graphically.

OBSERVATION AND CALCULATIONS

Potentiometric Titration-I

Burette: Potassium Permanganate solution (0.05 N)

Beaker: 20 mL of steel solution + one test tube dilute H_2SO_4

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	0.103	11	10	0.642
2	1	0.124	12	11	0.656
3	2	0.180	13	12	0.682
4	3	0.226	14	13	0.694
5	4	0.247	15		
6	5	0.254	16		
7	6	0.269	17		
8	7	0.280	18		
9	8	0.302	19		
10	9	0.460	20		

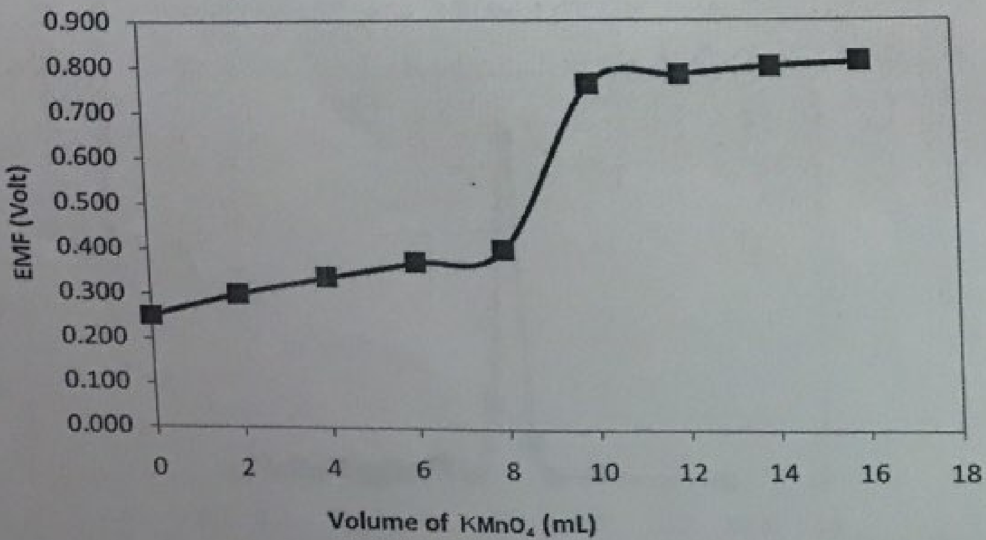

Fig. 1 : Plot of EMF vs volume of $KMnO_4$ added (mL)

Potentiometric Titration-II

20 mL of sample + 20 mL (one test tube) of 2N H_2SO_4in beaker & $KMnO_4$ in burette

Sl. No.	Vol. of $KMnO_4$ (mL)	EMF (Volt)	ΔE (Volt)	ΔV (mL)	$\Delta E/\Delta V$ (Volt/mL)	Average Volume (mL)
1	0	0.103	0.188	6	0.032	3
2	6	0.291	0.048	2	0.024	7
3	8	0.330	0.009	0.2	0.045	8.1
4	8.2	0.339	0.035	0.2	0.175	8.3
5	8.4	0.374	0.107	0.2	0.0214	8.5
6	8.6	0.481	0.059	0.2	0.295	8.7
7	8.8	0.540	0.085	0.2	0.425	8.9
8	9	0.625	0.065	0.5	0.130	9.25
9	9.5	0.640	0.095	0.5	0.190	9.75
10	10	0.785				
11						
12						
13						
14						
15						

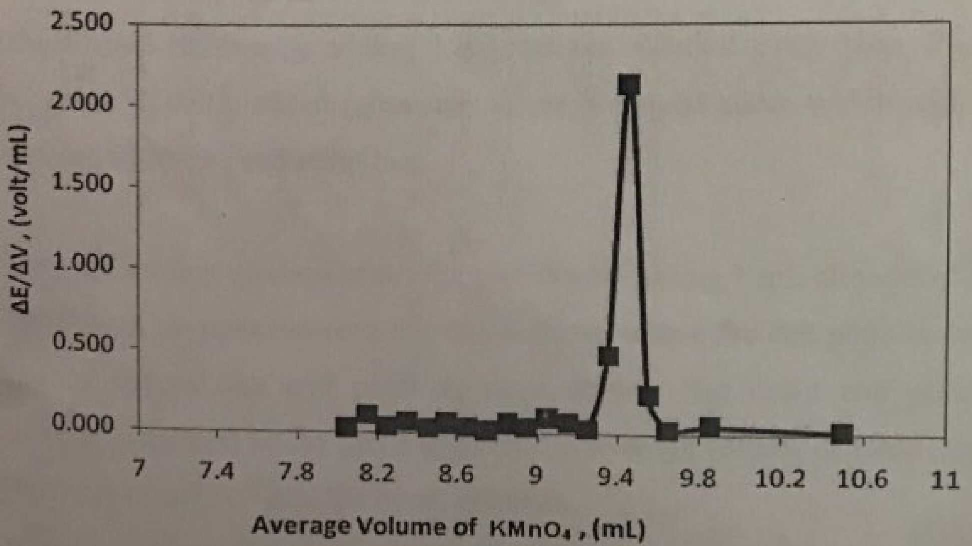

Figure II: Plot of plotting $\Delta E/\Delta V$ *versus* average volume of $KMnO_4$ added.

Requirements:

Reagents and solutions: 100 mL of $KMnO_4$ solution, 100 mL of steel solution, 2N H_2SO_4.

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

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 standard 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 up the solution with distilled water and mix well. Pipette out 20 mL made up steel sample solution into a clean 100 mL beaker. Add one test tube dilute sulphuric acid (2N). 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 a 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 will indicate the volume between which the end point lies.

Carry out another 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 of the Fe(II) in the given solution.

$$5Fe^{+2} + MnO_4^{-} + 8H^{+} \rightarrow 5Fe^{+3} + Mn^{2+} + 4H_2O$$

Calculations:

By stoichiometry, 5 moles of Fe(II) will need 1 mole of $KMnO_4$ for complete oxidation.

Hence, 279.25 g Fe(II) will need 158.04 g of $KMnO_4$

1 mL of 0.05 N $KMnO_4$ contain 0.00158g of $KMnO_4$

Therefore, 1 mL of 0.05 N $KMnO_4$ will oxidize $\frac{279.25 \text{ X } 0.00158}{158.09}$ = 0.00279 g of Fe^{2+}

= 2.79 mg of Fe^{2+}

Volume of $KMnO_4$ consumed for oxidizing 20 mL of steel solution = V mL (Taken from the second graph)

Therefore, Fe(II) present in the 20 mL steel solution = V x 2.79 mg = X mg = 23.157

This Fe(II) is present in 20 mL of steel solution given which contains Y mg/mL of steel.

Therefore, % Fe(II) in the steel sample $\frac{\text{X X } 100}{\text{Y}}$ = ------ %

Result

Percentage of Fe(II) in the given steel sample = 23.157 %

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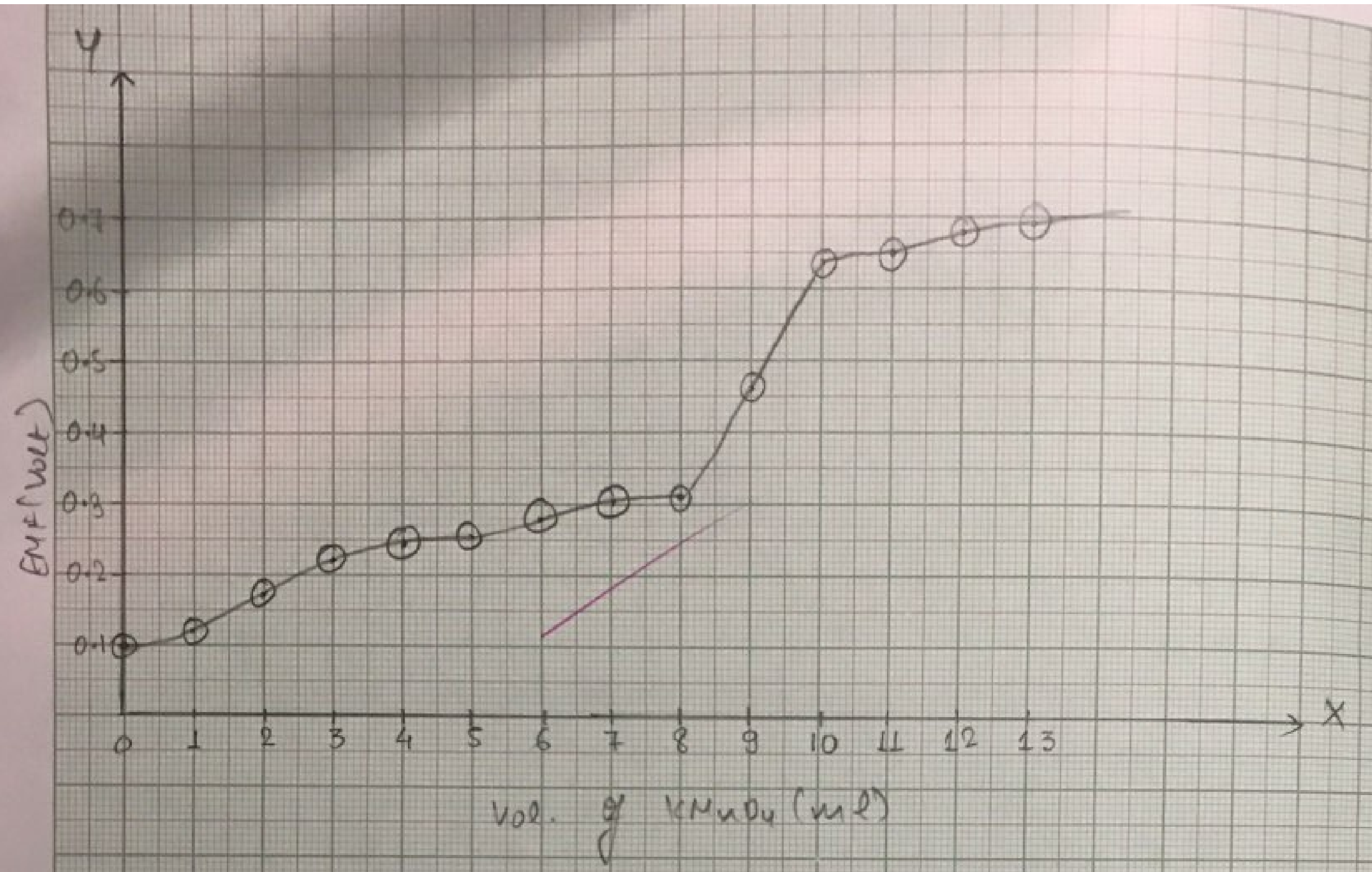
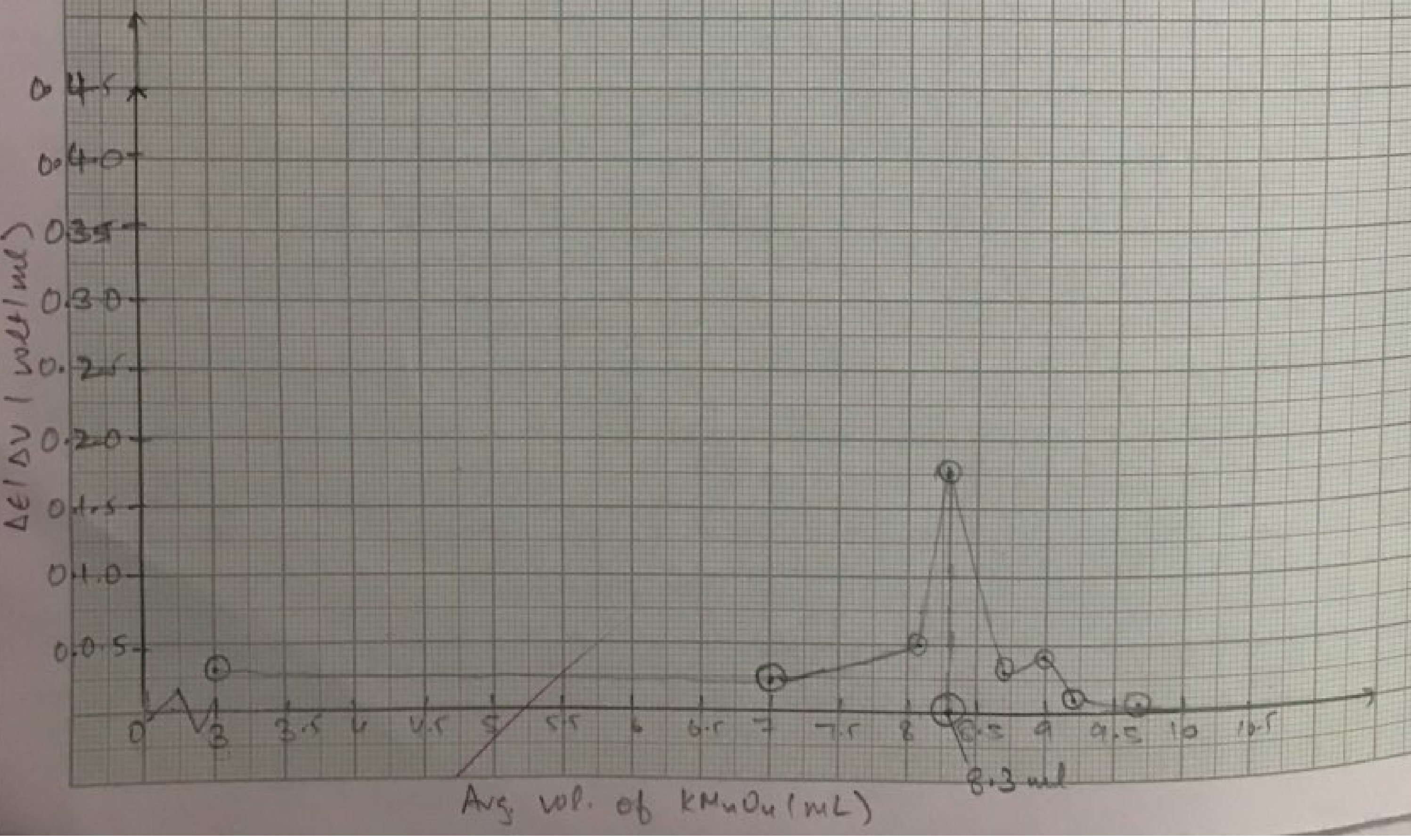


Y
X
EMF (volt)
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0
1
2
3
4
5
6
7
8
9
10
11
12
13
Vol. of KMnO4 (ml)



ΔE / ΔV (volt / ml)
0.05
0.10
0.15
0.20
0.25
0.30
0.35
0.40
0.45
0
3
3.5
4
4.5
5
5.5
6
6.5
7
7.5
8
8.5
9
9.5
10
10.5
8.3 ml
Avg. vol. of KMnO4 (mL)