

- Aim: Conductometric titration to determine the strength of strong acid (HCl) by strong base (NaOH)
- Procedure :
- 1. Rinse the conductivity cell with double distilled water.
- 2. Pipette out 20mL HCl (unknown conc.) in a beaker & dip the conductivity cell in it.
- 3. Add small amount of NaOH (0.1N) from burette, stir & measure the conductance.
- 4. Measure the conductance after each addition.
- 5. Take at least 5 readings after the end pt.

• Results :

$$\text{Normality of HCl} = 0.045\text{N}$$

$$\text{Strength of HCl} = 1.6425 \text{ g/L}$$

**OBSERVATIONS
TABLE**

NO.	Volume of NaOH added V_2 (mL)	Conductance
1	0	3.34×10^{-3}
2	1	3.15×10^{-3}
3	2	2.69×10^{-3}
4	3	2.43×10^{-3}
5	4	2.07×10^{-3}
6	5	1.87×10^{-3}
7	6	1.51×10^{-3}
8	7	1.29×10^{-3}
9	8	1.08×10^{-3}
10	9	0.84×10^{-3}
11	10	0.91×10^{-3}
12	11	1.05×10^{-3}
13	12	1.15×10^{-3}
14	13	1.28×10^{-3}
15	14	1.37×10^{-3}

End Point: 9mL

$$\rightarrow \text{Volume} = 9 \text{mL} (V_2) \quad | \quad N_2 = 0.1 \text{N}$$

$$\rightarrow V_1 = 20 \text{mL}$$

Calculations:

$$N_1 V_1 = N_2 V_2 \quad \Rightarrow \quad N_1 = \frac{N_2 V_2}{V_1}$$

$$\text{Strength (g/L)} = N_1 \times E_g \text{ wt} \quad | \quad \therefore N_1 = \frac{0.1 \times 9}{20} = 0.045 \text{N}$$

$$= N_1 \times 36.5$$

$$\text{Strength} = \frac{1.6425 \text{ g/L}}{(g/L)}$$

- Aim: To potentiometrically do redox titration of Fe^{2+} by standard Ce^{4+} soln.

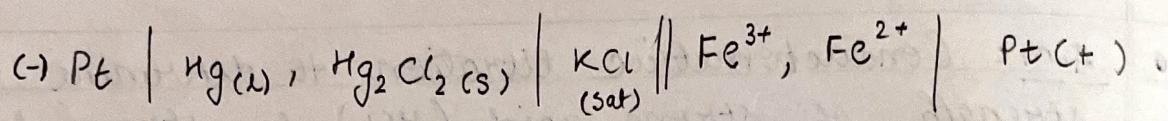
- Apparatus & Chemicals Required -

Potentiometer, Pt electrode, saturated calomel electrode (SCE), Ferrous ammonium sulphate & 0.25 M Ceric ammonium sulphate (both prepared in 2N H_2SO_4)

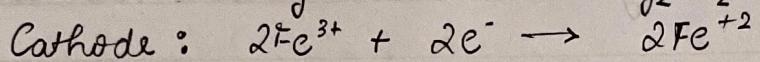
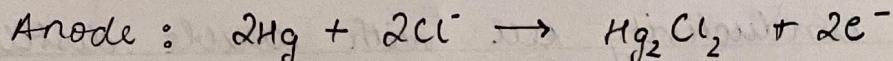
- Procedure:

1. Pipette out 25ml of test solution (ferrous solution) in a clean 100ml beaker, place the platinum electrode & SCE in the solution, which creates a $\text{Fe}^{2+}/\text{Fe}^{3+}$ couple. Connect the electrodes to the potentiometer & measure the EMF of the cell.
2. Add ceric sulfate from burette in small portions to the ferrous solution, stir it and note the EMF : $\text{Fe}^{2+} + \text{Ce}^{4+} \rightarrow \text{Fe}^{3+} + \text{Ce}^{3+}$
3. Continue the titrations till a sudden inflection in EMF occurs. Then take about 6 to 8 readings after inflection.
4. Draw a graph of E_{cell} vs Volume of ceric sulfate added; the inflection point gives an approximate equivalence.
5. Differential graph is drawn by plotting $\Delta E/\Delta V$ (Y-axis) vs volume of Ceric sulfate (X-axis)

Cell Representation -



Cell reaction -



Cell EMF -

$$E_{\text{cell}} = E_{\text{Fe}^{3+}/\text{Fe}^{2+}}^{\circ} + \frac{2.303RT}{F} \log \frac{[\text{Fe}^{3+}]}{[\text{Fe}^{2+}]} - E_{\text{SCE}}$$

Observation Table

Sr No.	Vol. of Corrige Soln (VmL)	EMF (in V)	ΔE (in V)	ΔV_E (in mV)	PILOT READING	
					ΔE ΔV	
1	1	0.375	-	-	-	
2	2	0.412	0.037	1	0.037	
3	3	0.433	0.021	1	0.021	
4	4	0.462	0.029	1	0.029	
5	5	0.680	0.218	1	0.218	
6	6	0.806	0.126	1	0.126	
7	7	0.832	0.026	1	0.026	
8	8	0.845	0.013	1	0.013	
9	9	0.855	0.01	1	0.01	
10	10	0.860	0.005	1	0.005	
ACTUAL READING	1	0.394	-	-	-	
	2	0.416	0.022	1	0.022	
	3	0.435	0.019	1	0.019	
	4	0.463	0.028	1	0.028	
	5	0.664	0.201	1	0.201	
	5.1	0.734	0.07	0.1	0.7	
	5.2	0.744	0.01	0.1	0.1	
	5.3	0.763	0.019	0.1	0.19	
	5.4	0.78	0.017	0.1	0.17	
	5.6	0.789	0.002	0.1	0.02	
	5.7	0.800	0.011	0.1	0.11	
	5.8	0.812	0.006	0.1	0.06	
	5.9	0.814	0.002	0.1	0.02	
	6.0	0.819	0.005	0.1	0.05	
	6.1	0.827	0.008	0.1	0.08	
	6.2	0.834	0.001	0.1	0.04	

to get a sharp peak, which corresponds to the precise equivalence point of titration.

6. From the titration curve, volume of ceric sulfate required is found out and conc. of ferrous sulphate can be calculated.

• Result:

1. Amount of Ferrous sulphate / Ferrous ammonium sulphate in the given solution = 19.992 g/L
2. Vol. of ceric sulphate (ceric ammonium sulphate) for the end pt. = 5.1 mL

Calc for
Ep 8
on Pg 11
Blank

Calculations

From the graph —

• Vol. at equivalent point = 5.1 mL

Vol. of $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4$ = $N_1 V_1 = N_2 V_2$

$$\therefore N_2 = \frac{N_1 V_1}{V_2} = 0.051 N$$

N_1 = Normality of $\text{Ce}(\text{SO}_4)_2$ = 0.25 N

N_2 = Normality of $\text{Fe}(\text{SO}_4)_2(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ = 0.051 N

V_1 = Volume of $\text{Ce}(\text{SO}_4)_2$ = 5.1 mL (from the graph)

V_2 = Volume of $\text{Fe}(\text{SO}_4)_2(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ = 25 mL

Amt. of $\text{Fe}(\text{SO}_4)_2(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ = Normality \times Eq. wt

$$= \text{Normality} \times \text{Mwt} = 0.$$

$$= 0.051 \times 392$$

$$= \underline{19.992 \text{ g/L}}$$

- Aim: To perform pH-metric titration of acidic (HCl) water by standard base (NaOH)

- Apparatus & Chemicals Required -

Standard NaOH solution, standard buffer solⁿ, glass electrode, saturated calomel electrode (SCE), HCl solⁿ (of unknown conc. - analyte), pH meter.

- Procedure -

1. Switch ON the pH-meter & allow 10 mins warming up time. Keep the instrument in 'stand by' mode.
2. Set the temp. knob to room temp.
3. Connect the two electrodes in their proper places to form a complete cell. Wash the glass & calomel electrodes with distilled water & blot dry with paper. Immerse them in a beaker containing distilled water.
4. With the help of the knot named power on off brings the needle to pH scale 7.0
5. Remove the electrodes from distilled water, dry them & immerse them in a buffer solution of known pH. Change the instrument from 'stand by' mode 'Read'. The needle will deflect to show some pH. With the help of calibrating knob during the needle to the correct pH of buffer solⁿ. The instrument is thus calibrated

Observation

Burette : 0.25N NaOH solⁿ

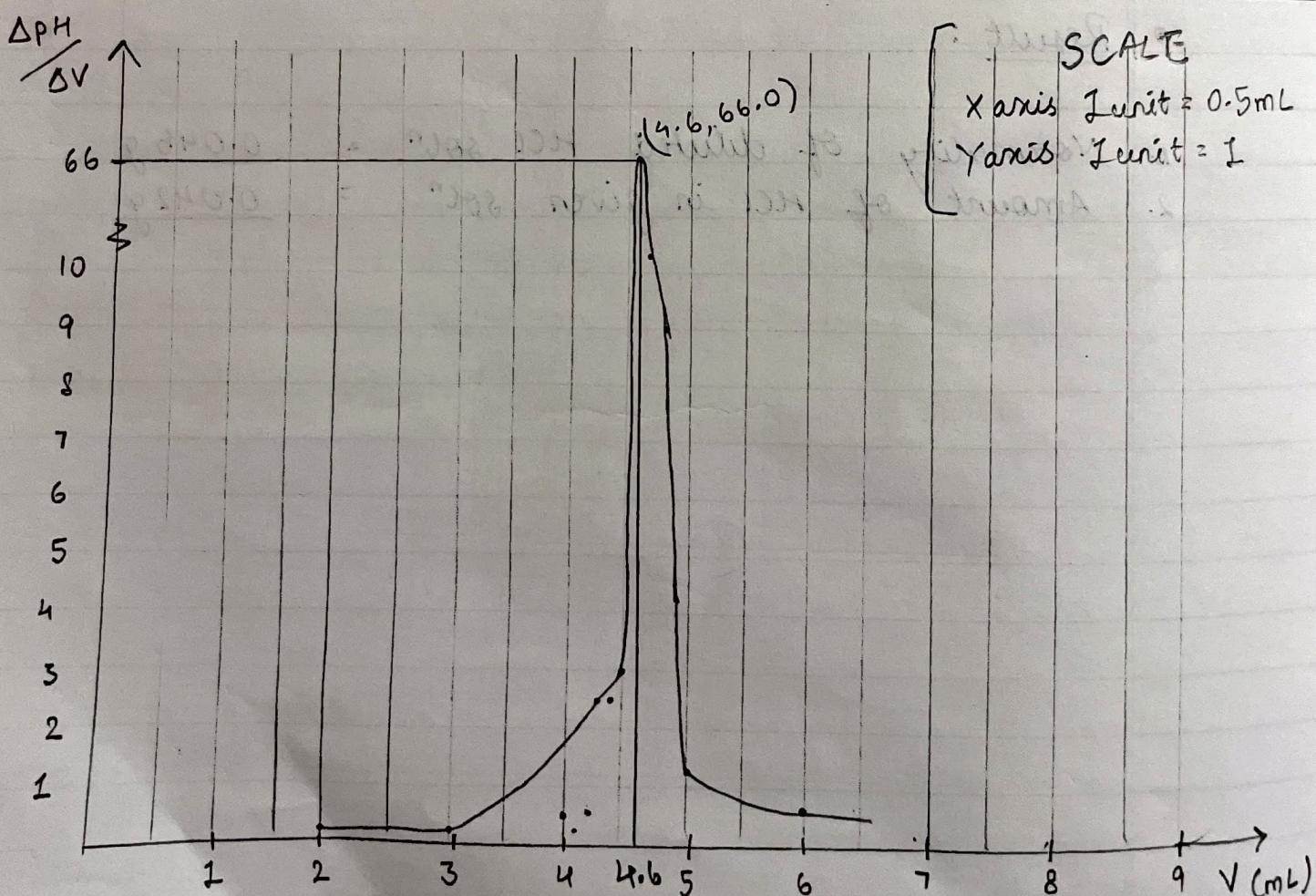
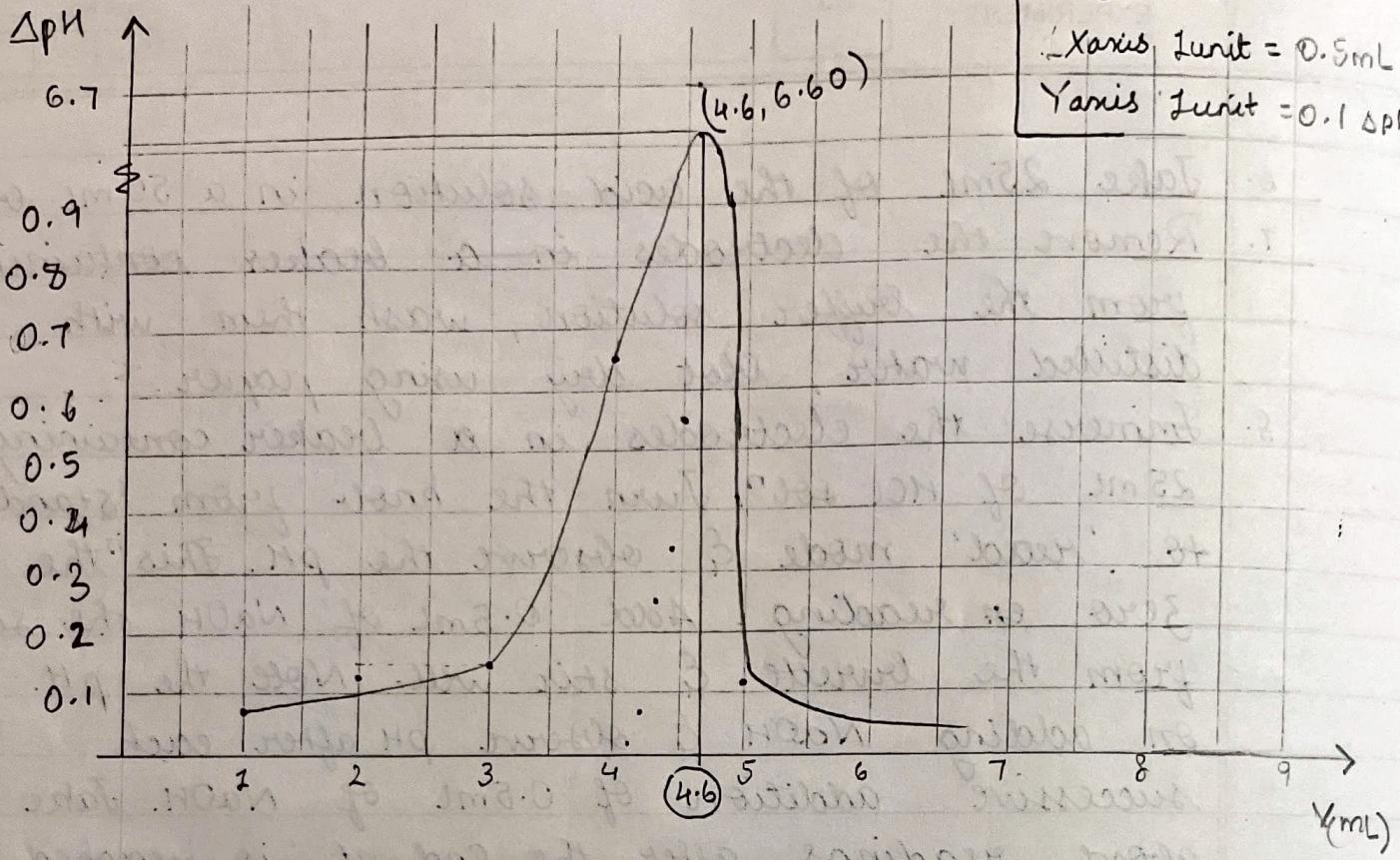
Beaker : 25ML dil. HCl solⁿ

Sr no.	Vol. of 0.25N NaOH - V (mL)	pH	ΔpH	ΔV	$\Delta pH / \Delta V$
1	0	0.68	-	-	-
2	1	0.76	0.08	1	0.08
3	2	0.89	0.13	1	0.013
4	3	1.08	0.19	1	0.19
5	4	1.05	0.42	1	0.42
6	5	11.78	10.28	1	10.28
7	6	12.48	0.7	1	0.7
8	7	12.67	0.19	1	0.19
9	8	12.82	0.15	1	0.15
10	9	12.91	0.09	1	0.09

PILOT
READING

1	0	0.69	-	-	-
2	1	0.76	0.07	1	0.07
3	2	0.89	0.13	1	0.013
4	3	1.14	0.15	1	0.25
5	4	1.80	0.66	1	0.66
6	4.1	1.82	0.02	0.1	0.20
7	4.2	1.88	0.06	0.1	0.6
8	4.3	2.14	0.26	0.1	2.6
9	4.4	2.40	0.26	0.1	2.6
10	4.5	2.70	0.30	0.1	3.0
11	4.6	9.30	6.60	0.1	66.0
12	4.7	10.33	1.03	0.1	10.3
13	4.8	11.23	0.90	0.1	9.0
14	4.9	11.66	0.43	0.1	4.3
15	5.0	11.79	0.13	0.1	1.3
16	6	12.48	0.69	1	0.69
17	7	12.70	0.22	1	0.22
18	8	12.82	0.12	1	0.12
19	9	12.90	0.08	1	0.08

ACTUAL
READING



Calculations:

$$(2) \text{ Normality of diluted HCl} = \frac{\text{Normality of NaOH} \times \text{Vol. NaOH}}{\text{Vol. HCl}}$$

(A) $= \frac{0.25 \times 10}{25} = \frac{0.25 \times 4.5}{25}$

$A = 0.046 N$

$$\begin{aligned}
 (2) \text{ Amount of HCl in } 25 \text{ mL diluted sol?} - \\
 (B) &= \text{Normality of dil. HCl} \times \text{Equivalent wt. HCl} \times (25 \text{ mL})
 \end{aligned}$$

6. Take 25ml of the acid solution in a 50 ml beaker.
7. Remove the electrodes in a beaker containing from the buffer solution, wash them with distilled water, blot dry using paper.
8. Immerse the electrodes in a beaker containing 25 ml of HCl soln. Turn the knob from 'stand by' to 'read' mode & observe the pH. This is the zero ~~or~~ reading. Add 0.5 ml of NaOH the soln from the burette & stir well. Note the pH. Keep on adding NaOH & observe pH after each successive addition of 0.5 ml of NaOH. Take 4 or above readings after the end pt. is reached.

• Result :

1. Normality of diluted HCl soln = 0.046 g.
2. Amount of HCl in given soln = 0.042 g

- Aim: To determine the conc. of Co^{2+} ion in water sample by spectrophotometer.

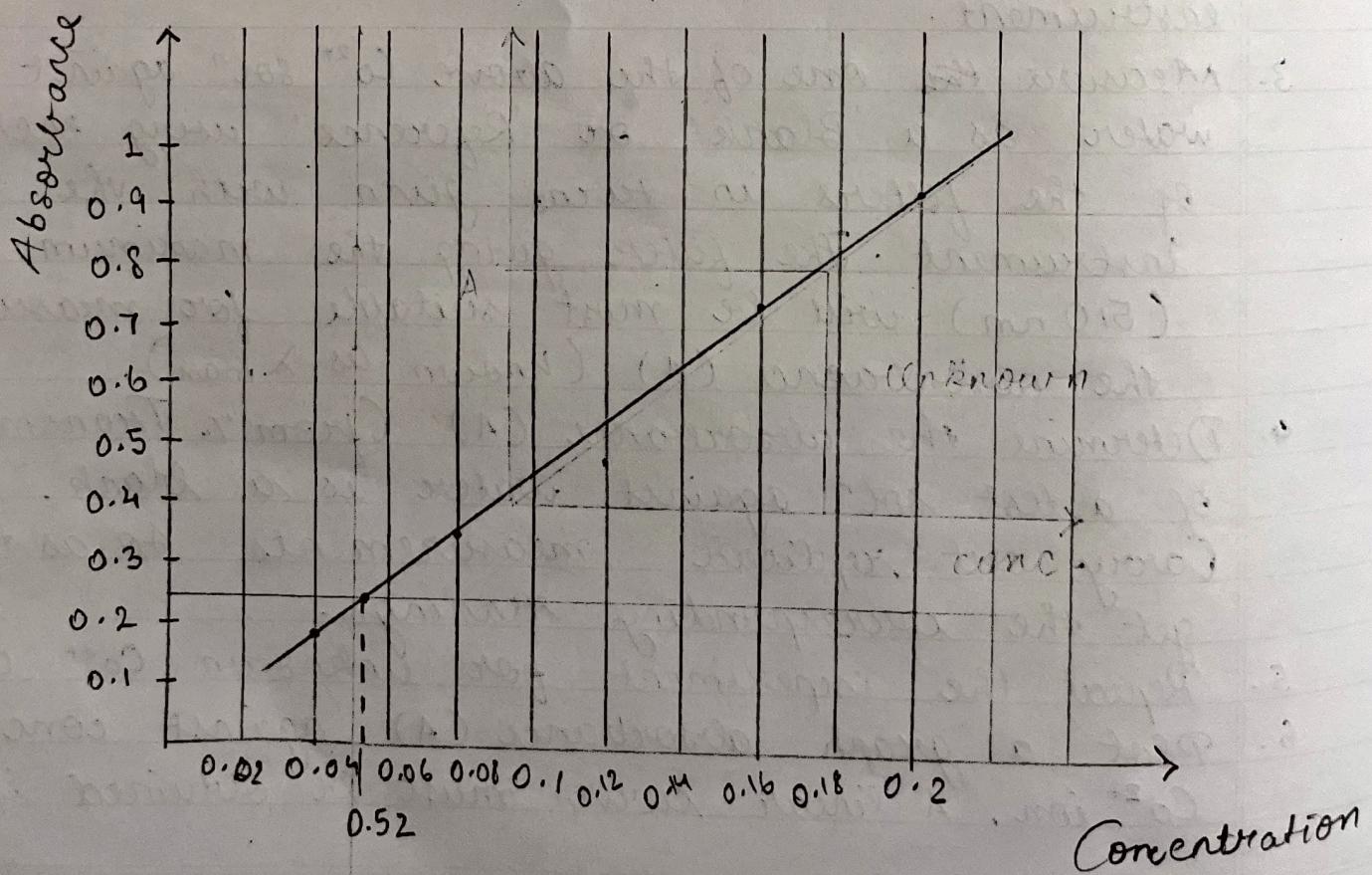
- Apparatus & Chemicals Required:

~~Spectrometer~~ Spectrophotometer, Filter, Cobalt Nitrate.

- Procedure:

1. Preparation of 0.2M standard cobalt nitrate solⁿ: Prepare a stock solution of 0.2M cobalt nitrate (5.82g in 100mL distilled water). Dilute this stock solution quantitatively as to get desired conc. of 0.04M, 0.08M, 0.12M, 0.16M solⁿ in 100mL SME.
2. Strictly follow the instructions given on the instrument.
3. Measure ~~the~~ one of the above Co^{2+} solⁿ against water as a 'Blank' or 'Reference' using each of the filters in turns given with the instrument. The filter giving the maximum (510 nm) will be most suitable for measuring the absorbance (A) (known as λ_{max}).
4. Determine the absorbance (A) (from % Transmittance) of a test solⁿ against water as a blank. Carry out replicate measurements so as to get the corresponding readings.
5. Repeat the experiment for unknown Co^{2+} conc.
6. Plot a graph absorbance (A) against conc. of Co^{2+} ion. A linear curve must be obtained if the

Sr no.	Concentration 'V' (M)	% Transmittance (T%)	Absorbance (A) (From Table)
1	0.04	70%	0.18
2	0.08	46%	0.36
3	0.12	36%	0.48
4	0.16	21%	0.75
5	0.20	14%	0.93
6	Unknown	58%	0.25



Beer - Lambert's Law is valid. Find the conc. of the unknown Co^{2+} solution from this curve from its absorbance.

1. Tabulate the observations.

• Result :

1. The straight-line nature graph of conc. vs absorbance at $\lambda_{\text{max}} 510\text{nm}$ which confirms the Beer - Lambert's law.
2. From the graph the conc. of unknown Co^{2+} solⁿ
= 0.52 M

- Aim: To estimate COD of waste water.
- Procedure:

BLANK -

1. Take 10mL of standard 0.1N $K_2Cr_2O_7$ and 10mL distilled water in conical flask.
2. Add carefully $\frac{1}{4}$ TT conc. H_2SO_4 & place a clean glass funnel. Boil the soln for five minutes.
3. Cool & titrate against 0.1N $FeSO_4$, $(NH_4)_2SO_4$, using 1-2 drops of Ferroin as indicator.
4. Sharp colour change from (Green to) blue to wine-red colour indicates the end pt.
5. Repeat the titration.

SAMPLE -

1. Take 10mL of given polluted water sample in a conical flask.
2. Add 10mL standard 0.1N $K_2Cr_2O_7$ & $\frac{1}{4}$ TT conc. H_2SO_4 . Mix well.
3. Place clear glass funnel on the conical flask.
4. Boil the solution for 5 mins with occasional shaking till solution turns to green.
5. Now cool the flask under tap water & titrate against 0.1N $FeSO_4$, $(NH_4)_2SO_4$, using 1-2 drops of Ferroin indicator as done for blank.
6. Repeat the titration.

Observations

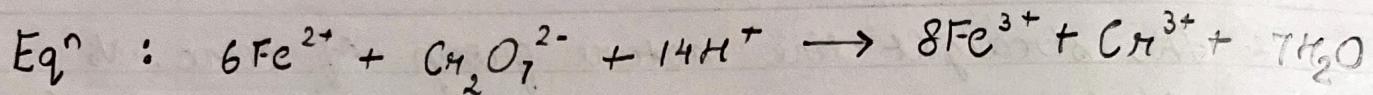
BLANK -

Burette : 0.1N FeSO_4 (NH_4)₂ SO_4 soln

Flask : 10mL distilled water + 10mL 0.1N $\text{K}_2\text{Cr}_2\text{O}_7$ soln + $\frac{1}{4}$ TT conc. H_2SO_4

Indicator : 1-2 drops of Ferroin indicator

Colour Change : Green to blue to wine-red colour.



Observations Table

BLANK

Sr no.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Concurrent Reading (mL)
1	0	20.1	20.1	
2	0	20	20	20
3	0	20	20	

[a = 20]

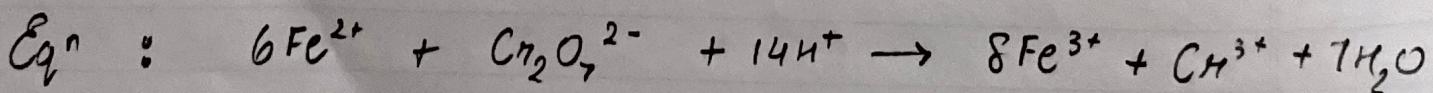
SAMPLE -

Burette : 0.1N FeSO_4 (NH_4)₂ SO_4 soln

Flask : 10mL polluted water + 10mL 0.1N $\text{K}_2\text{Cr}_2\text{O}_7$ soln + $\frac{1}{4}$ TT conc. H_2SO_4

Indicator : 1-2 drops of Ferroin indicator

Colour change: (Green to) Blue to wine-red colour



SAMPLE -

Sr no.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Concurren cent Reading (mL)
1	0	12.1	12.1	
2	0	12.2	12.2	
3	0	12.1	12.1	12.1

$$[b = 12.1]$$

Calculations -

$$COD \text{ (mg/L)} = \frac{(a-b) N \times 8000}{\text{mL of sample}}$$

[Where, Vol. of $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4$ for blank is 'a' mL
 & that for sample is 'b' mL, Normality of $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4$ soln is 'N']

$$\therefore COD = \frac{(20-12.1) \times 0.1 \times 8000}{10}$$

$$COD = 632 \text{ ppm}$$

- Result :

1. Vol. of Fe^{2+} sol. required for blank = 20 mL
2. Vol. of Fe^{2+} sol. required for sample = 12.1 mL
3. COD in the given polluted water sample = 632 ppm