AMOGH GARG – 2020UCO1688

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EXPERIMENT-1

AIM – To estimate the amount of iron present in a given solution of Ferrous Ammonium Sulphate using potassium permanganate solution as intermediate.

MATERIAL REQUIRED – Mohr's Salt, $KMnO_4$ soln, Dil. H_2SO_4, Burette, Funnel, Stand, White tile, Pipette, Volumetric Flask, Conical Flask, Measuring Flask.

THEORY – Mohr's salt act as reducing agent and $KMnO_4$ act as O.A; so, the reaction between Mohr's Salt and $KMnO_4$ is a redox reaction. In this redox reaction ferrous ions from Mohr salt get oxidised and pink colour of Manganese present $KMnO_4$ which is the +7.

Oxidation state gets reduced to colourless Mn^{2+} state. $KMnO_4$ is powerful O.A. It helps in estimation of reducing agent like Mohr Salt in acidic medium according to –

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2$$

$$[Fe^{2+} \longrightarrow Fe^{3+} + e^-] \times 5$$

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

This titration is based upon oxidation-reduction titration. When Mohr's salt is

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titrated against $KMnO_4$ in the presence of acidic medium by H_2SO_4.

PROCEDURE – 1) 10 ml of known Mohr's salt (N/40) was taken in classical flask.
2) About half the test tube of H_2SO_4 was added to it.
3) The burette was filled with $KMnO_4$ soln.
4) The conical flask contents were titrated against.
5) 3 concordant reading were recorded.
6) The same process was repeated for an unknown soln. of Mohr's salt.

RESULT – The molarity of given Mohr's salt is 0.03 moles/L and strength is 11.92 g/L.

PRECAUTIONS – 1) $KMnO_4$ is dark in colour so always read upper miniscus.
2) Rinse the pipette and burette before using.
3) Clean the apparatus with distilled water
4) Strength of unknown soln should be taken upto 2 decimal places.

A. Observation table for known soln:

S No.	Vol of Mohr's salt soln (ml)	Burette Reading (ml)			Concordant Reading	Indicator
		Initial	Final	Volm of soln		
1.	10	0	9.1	9.1	{	KMnO4
2.	10	10	19.1	9.1	9.1	(self-ind.)
3.	10	20	29.1	9.1	}	

B. Observation table for unknown soln:

So No.	Volm of Mohr's salt	Burette Reading (ml)			Concord. Reading	Indicator
		Initial	Final	Volm used		
1.	10	0	12.5	12.5	}	KMnO4
2.	10	12.5	25	12.5	12.5	(self-ind)
3.	10	25	37.5	12.5	}	

CALLULATIONS –

A. Normality of unknown Mohr's salt –

$N_1V_1 = N_2V_2$

$\Rightarrow \frac{1}{10} \times 10 = N_2 \times 9.1$

$\Rightarrow N_2 = \frac{1}{36.1} N$

$N_2V_2 = N_3V_3 \Rightarrow \frac{1}{36.1} \times 12.5 = N_3 \times 10$

$\Rightarrow N_3 = 0.03\,N$

B. Strength of unknown Mohr's salt –

Strength = Normality × Eq. Mass

= 0.03 × 392

= 11.92 g/L

C. Molality = $\frac{\text{Normality}}{\text{No. of } e^{\ominus} \text{ gained}}$ = 0.03 moles/L

EXPERIMENT – 2

AIM – To determine the strength of Potassium Dichromate soln using Ferrous Ammonium Sulphate soln. as an intermediate.

MATERIAL REQUIRED – Mohr Salt, n-Phenyl Anthranilic Acid, Potassium Dichromate, Dil. H_2SO_4, Burette, Funnel, Stand, Measuring Flask, Pipette, Weighing bottle, Conical Flask, Volumetric Flask

THEORY – Acidic $K_2Cr_2O_7$ is a strong O.A., when it is added to Ferrous Ammonium sulphate soln., containing dil. H_2SO_4 only $FeSO_4$ is oxidised. $(NH_4)_2SO_4$ remain unchanged hence not shown in the eqn. The reaction takes place as follows –

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3O$$

$$6FeSO_4 + 3H_2SO_4 + 3O \longrightarrow 3Fe_2(SO_4)_3 + 3H_2O$$

Complete Reaction is –

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \longrightarrow 2Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O$$

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

PROCEDURE – 1.) 10 ml of Mohr's salt was taken in conical flask.
2.) About half test tube of dil. H_2SO_4 was added to the conical flask.
3.) A few drops of n-phenyl anthralic acid was added as indicator.
4.) The burette was filled with potassium dichromate.
5.) The conical flask content was titrated against known $K_2Cr_2O_7$ soln., taken in burette.
6.) The colour first turned green. On further adding $K_2Cr_2O_7$ from the burette, the colour of the soln. turned greenish purple. This was taken as end pt.
7) 3 concordant reading were taken.

RESULT – The strength of unknown $K_2Cr_2O_7$ soln. is 2.45 g/L.

PRECAUTION – 1.) Rinse the pipette and burette before use.
2.) Don't use Rubber cork.
3.) Take accurate reading and don't go with avg. reading.

A. Observation Table for unknown $K_2Cr_2O_7$ soln –

S. No.	Vol of Mohr Salt	Burette Reading (ml) Initial	Final	Volm. used	Concordant Reading	Indicator Used.
1.	10	0	16.5	16.5	} 16.5	N-Phenyl Anthranilic Acid.
2.	10	16.5	33	16.5		
3.	10	33	49.5	16.5		

B. Observation Table for unknown $K_2Cr_2O_7$ soln.

S. No.	Volm of Mohr Salt	Burette Reading (ml) Initial	Final	Volm Used	Concordant Reading	Indicator used
1.	10	0	8	8	} 8	N-Phenyl Anthranilic Acid.
2.	10	8	16	8		
3.	10	16	24	8		

CALCULATIONS –

$N_1V_1 = N_2V_2 \Rightarrow \frac{1}{10} \times 16.5 = N_2 \times 10$

$\Rightarrow N_2 = 0.041N$

$N_2V_2 = N_3V_3 \Rightarrow 0.04 \times 10 = N_2 \times 8$

$\Rightarrow N_2 = 0.05\ N$

∴ Normality of unknown $K_2Cr_2O_7$ soln = 0.05 N

→ Strength of unknown $K_2Cr_2O_7$ soln –

Strength = Normality × Eq Mass

$= 0.05 \times \frac{291}{6}$

$= 2.45\ g/L$

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EXPERIMENT-3

AIM :- To determine the strength of a given $CuSO_4$ soln iodometrically.

MATERIALS REQUIRED- $CuSO_4$ soln, KI, Sodium Thiosulphate, Burette, Measuring Flask, Stand, Volumetric Flask, Conical Flask, White Tile, Test tubes and funnel.

THEORY - When KI is added to a soln of $CuSO_4$, a white cuprous Iodide (Cu_2I_2) is precipitated and an equivalent amount of iodide is liberated. The free iodide is titrated with standard soln. of hypo, using starch as an indicator. As soon as all the liberated iodide has been reduced to NaI, the blue colour of Iodo-starch complex, will disappear and the colour of ppt. in conical flask will be white.

$$2\,CuSO_4 + 4KI \longrightarrow Cu_2I_2\downarrow + 2K_2SO_4 + I_2$$
(white ppt)

$$2\,Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$
(Sodium Tetrathionate)

PROCEDURE - 1.) 10 ml of known $CuSO_4$ soln. was taken in a conical flask.

2.) About half test tube of KI soln. was added to it.

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3.) The burette was filled with sodium Thiosulphate soln.

4.) Conical flask contents were titrated against the thiosulphate soln. taken in the

5.) At this point, 5-6 drop of starch indicator was added to it.

6.) The titration was continued with sodium Thiosulphate soln, until the blue colour of complex just disappeared.

7.) This was taken as the end point. Three concordant reading were taken.

8.) The process was repeated for unknown $CuSO_4$ soln.

RESULT - The strength of $CuSO_4$ soln is 9.32 g/L.

PRECAUTION - 1.) Iodine soln. attacks rubber, ∴ it should always be taken in glass stopper burette.

2.) Clean the apparatus with distilled water.

A. Observation Table for Known $CuSO_4$ Soln.

S. NO	Volm of $CuSO_4$ (ml)	Burette Reading (ml)			Concordant Reading	Indicator Used
		Initial	Final	Total		
1.	10	0	14.2	14.2	} 14.2	} Starch
2.	10	15	29.2	14.2		
3.	10	30	44.2	14.2	}	}

S NO.	Volm of $CuSO_4$ (ml)	Burette Reading (ml)			Concordant Reading	Indicator used
		Initial	Final	Total		
1.	10	0	9.5	9.5	}	
2.	10	10	19.5	9.5	9.5	Starch
3.	10	20	29.5	9.5	}	

CALCULATIONS –

$N_1V_1 = N_2V_2 \Rightarrow \frac{1}{10} \times 10 = N_2 \times 9.5$

$\Rightarrow N_2 = 0.025\ N$

$N_2V_2 = N_3V_3 \Rightarrow N_3 \times 10 = 0.025 \times 14.2$

$N_3 = 0.037\ N$

Strength $= N \times wt$

$= 0.037 \times 249.5$

$= 9.32\ g/L$

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EXPERIMENT-4

AIM – To calculate the concentration of unknown $KMnO_4$ solution using UV visible spectrometer.

MATERIALS REQUIRED – Test tubes, $KMnO_4$ soln of different concentrations, Light source, Monochromator, Sample, Detector

THEORY – • Beer Lambert's Law – (Also called Beer Law) It is a relationship between the attenuation of light through a substance and the properties of that substance.

• Consider monochromatic light transmitted through a solution, with an incident intensity of I_0 and a transmitted intensity of I.

Then the transmittance $T = \frac{I}{I_0}$

$$T(\%) = 100 \times \frac{I}{I_0}$$

The absorbance, $A = \log_{10} \frac{I_0}{I} = -\log_{10} T$

• The Beer-Lambert law is linear relationship b/w the absorbance and the concentration, molar absorption coefficient and optical coeff.

$$A = \varepsilon c l$$

$A \equiv$ Absorbance

$\varepsilon \equiv$ Molar absorption coeff. $(M^{-1} cm^{-1})$

$c \equiv$ Molar conc. (M) ; $l \equiv$ optical length

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PROCEDURE – 1.) Prepare $KMnO_4$ stock solution ($\lambda max = 525$ nm)
2.) Prepare five standard soln of $KMnO_4$.
3.) Measure their absorbances at 525 nm.
4.) Plot calibration curve using the 5 absorbance and conc. values.
5.) Calculate 'ε' [Slope $= \varepsilon \times l$]
6.) Measure absorbance of unknown sample.
7.) Using $A = \varepsilon c l$, calculate unknown $KMnO_4$ concentration.

RESULT – The concentration of unknown $KMnO_4$ soln is 0.344 M.

PRECAUTIONS – 1.) Clean the apparatus using distilled water.
2.) Measure the readings carefully and with precision.

CALCULATION — Unknown Absorbance = 0.64

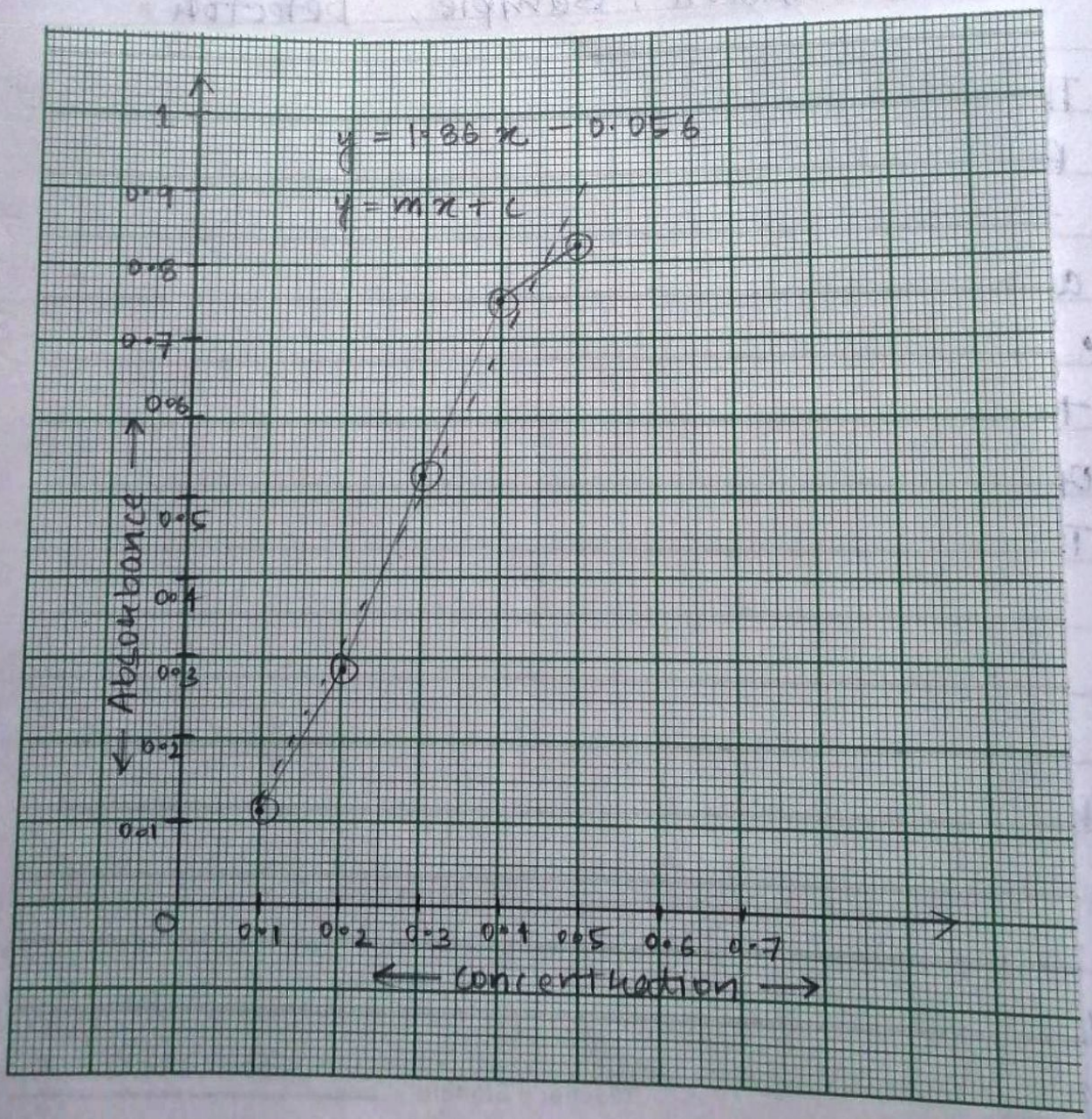
$A = \varepsilon c l \Rightarrow A = (\varepsilon l)c$

$y = mx + c$

$\varepsilon = \frac{m}{l}$

$\Rightarrow c = \frac{A}{\varepsilon l} \Rightarrow c = \frac{0.64}{1.86 \times 1}$

$= 0.344\ M$



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EXPERIMENT-5

AIM- To determine the strength of $KMnO_4$ soln with the help of potassium dichromate soln, using Mohr's salt as an intermediate.

MATERIAL REQUIRED- Mohr's salt, Potassium dichr., n-phenyl anthranilic acid, dil. sulphuric acid, $KMnO_4$, Burette, Funnel, Stand, pipette, white tile, measuring flask, conical flask, test tubes.

THEORY- Acidic $K_2Cr_2O_7$ is a strong OA. When it is added to ferrous ammonium sulphate soln containing dil. H_2SO_4, only $FeSO_4$ is oxidised.

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

$KMnO_4$ being a powerful O.A, helps in estimat R.A. like Mohr's salt in acidic medium, according to -:

$$MnO_4^- + 8H^+ + 5Fe^{2+} \longrightarrow 5Fe^{3+} + 2Mn^{2+} + 4H_2$$

PROCEDURE - 1) 10 ml of Mohr's salt soln was taken in a conical flask.
2) Half test tube of dil. H_2SO_4 was added to it.
3) The burette was filled with known $K_2Cr_2O_7$ soln.
4) The colour of first turned to green. On further addn. of $K_2Cr_2O_7$, it changed to

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greenish purple.

5.) 10 ml of Mohr's salt was taken in a conical flask and half test tube of dil. H_2SO_4 was added to it.

6.) 3 concordant readings were taken.

RESULT – Strength of given $KMnO_4$ soln. is 1.087 g/L.

PRECAUTION – 1.) $KMnO_4$ is dark coloured, so read upper miniscus.

2.) Don't use rubber cork burette.

3.) Use dil. H_2SO_4 for acidifying $KMnO_4$.

OBSERVATION – Table for known soln. –:

S No	Volm of soln taken	Burette Reading (ml)			Concordant Reading (ml)	Indicator used
		Initial	Final	Total		
1.	10	0	12.5	12.5	12.5	Diphenyl Amine
2.	10	13	25.5	12.5		
3.	10	26	38.5	12.5		

Table for unknown soln –

S No	Volm of soln taken	Burette Reading			Concordant Reading	Indicator used
		Initial	Final	Total		
1.	10 ml	0	9.2	9.2	9.2 ml	$KMnO_4$
2.	10 ml	10	19.2	9.2		
3.	10 ml	20	29.2	9.2		

CALCULATION –

$N_1V_1 = N_2V_2$ ⇒ $N_1 = \frac{N}{40}$; $V_1 = 12.5$; $V_2 = 10$ ml

⇒ $10 \times N_2 = \frac{1}{40} \times 12.5$

$N_2 = 0.03N$

$N_1V_1 = N_2V_2$

⇒ $N_1 \times 2 = 0.08 \times 10$

$N_1 = 0.03N$

Strength $= 0.03 \times \frac{158}{5} = 1.07$ g/L