Aim :- To determine the strength of Unknown oxalic acid by titrating with Potassium Permagnati solution. Prepare Standard oxalic Acid solution of N/40

Apparatus Required : Burette, Pipette, weighing bottle washing bottle, iron stand, Beaker - 400ml (2) wire gauze, tripod stand, conical flask.

Theory → It is reduction - oxidation titration where Potassium Permagnate gain 5 e⁻ while oxalic acid undergo oxidation give 2 electrons. $KMnO_4$ undergoes reduction.
The reducing agent is - - - - -
The oxidizing agent is - - - -

Molecular Equation (+5 e gained)

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O) \quad \text{—— (1)}$$

$$5(O) + 5H_2C_2O_4 \longrightarrow 10CO_2 + 5H_2O \quad \text{—— (2)}$$

(lose 2e⁻)

Ionic Equation

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 5(O) + 10CO_2$$

Since nascent (O) is not balanced in eq (1) & (2) therefore we multiply eq (2) by (5)

Ionic Equation

$2\,Mn^{+7}O_4^- + 8H^+ \longrightarrow 2Mn^{2+} + 4H_2O$ — (1)]×2

(+5e)

$C_2O_4^{2-} \longrightarrow 2CO_2 + 2e^-$ — (2)]×5

Multiply eq. (1) by 2 and eq. (2) by 5

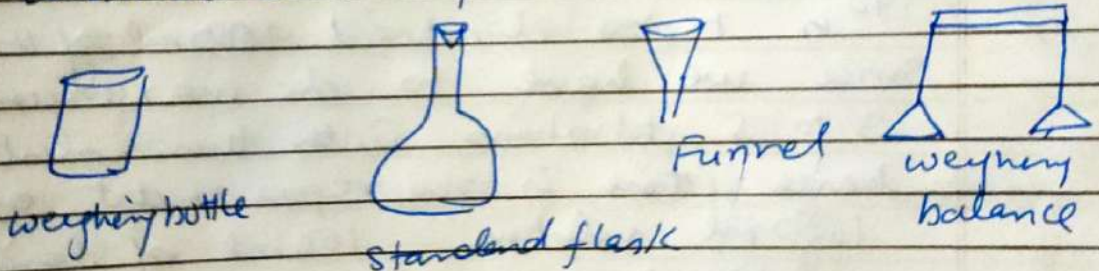
$10e^- + 2MnO_4^- + 16H^+ \longrightarrow 2Mn^{2+} + 8H_2O$

$5C_2O_4^{2-} \longrightarrow 10CO_2 + 10e^-$

$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \longrightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$

Procedure

Preparation of standard oxalic acid standard flask, weighing bottle, spatula funnel wash bottle is required



Since $\frac{N}{40}$ oxalic acid standard solution is to be prepared.

Therefore calculation of mass of oxalic acid is required for its preparation.

Molecular weight of oxalic Acid = 126 g/mole

To prepare $\frac{N}{40}$ normal solution equivalent weight of oxalic acid is required.

In the eq. (2) of O.A. there is $2e^-$

are released hence to calculate its equivalent weight the its molecular weight is devided by that number

Equivalent weight = $\frac{\text{Molecular weight}}{2}$

$= \frac{126 \text{ g/mole}}{2}$

$= 63$

To prepare 1 Normal solution ie 63 g of oxalic acid is dissolved in 1000 ml of the (H_2O)

For 1000 ml.

0.1 N — 6.3 g

0.01 N — 0.63 g

$\frac{N}{40} \longrightarrow \frac{63}{40} = 1.575$

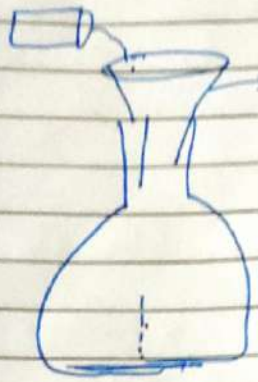
For $\frac{N}{40}$ oxalic acid in 1000 ml → 1.575 g is to be dissolved 1000 ml of H_2O Since we have to do maximum 3 to 5 titrations with this solution hence there is no requirement of 1000 ml solution. 100 ml of standard solution is sufficient

∴ 1000 ml → 1.575 g

1000 ml → $\frac{1.575}{1000} \times 100 = 0.1575$ g

First mass of empty weighing bottle is done. Let us suppose it is 5 g = m, Then in the weighing bottle 0.1575 g of oxalic is added with help of spatula & its weighed is done 5.1575 g

This mass is transferred to the standard flask with help of funnel.



After transferring the empty weighing bottle is again weighed to know the mass of weighing bottle after transference so that exact amount of oxalic acid transferred can be known.

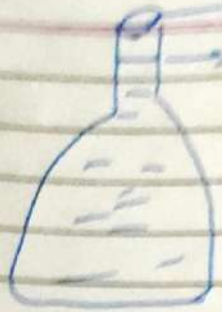
Let the mass of weighing bottle after transference is 5.0015 g.

∴ Mass of oxalic acid transferred in standard flask is = 5.1575 g − 5.0015 g
= 0.1560 g

This amt 0.1560 g of oxalic acid is transferred to standard flask.

Add 10 ml of the from the funnel so that some particles of oxalic acid is attached in the neck of funnel can be removed & go into standard flask.

Dissolve O. A. in minimum amt of the and then make the solution upto mark



Normality of the standard solution is

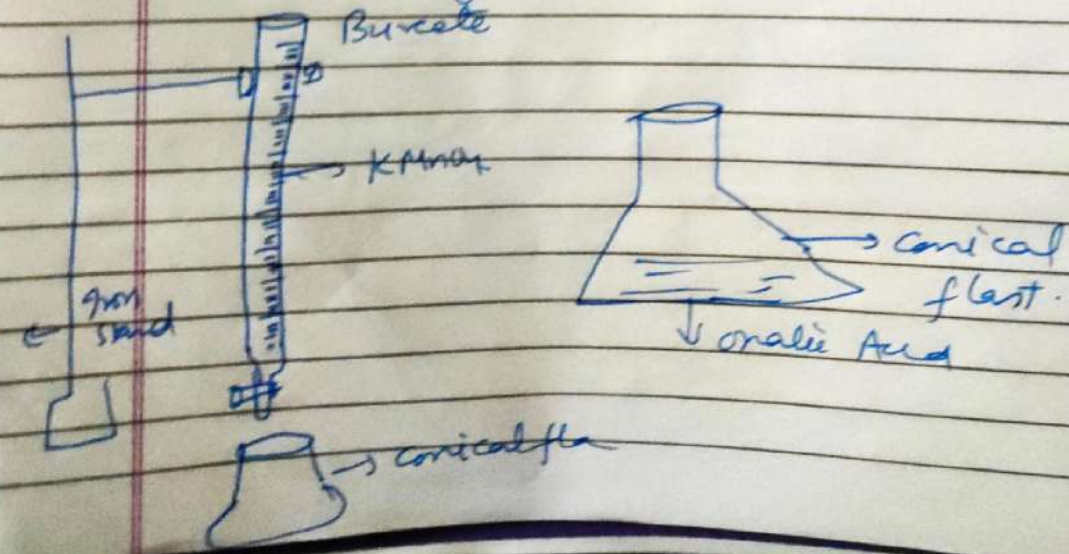
$$\frac{\text{wt. of O.A.} \times 1000}{\text{Equivalent wt} \times 100}$$

$$= \frac{0.156 \times 1000}{63 \times 100}$$

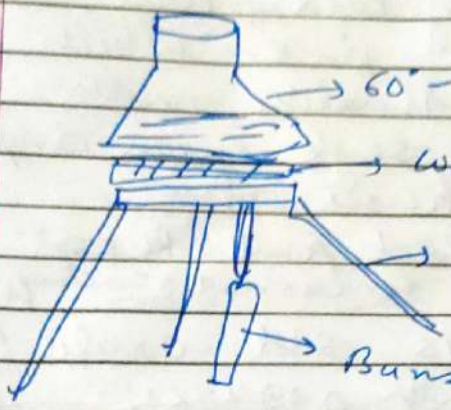
$$= \frac{1.56}{63} = 0.02476\ N$$

Step 2. Titration of Potassium Permagnate VS Standard Oxalic Acid solution

In the burette Potassium Permagnate (Prepared by lab staff) is added.



Pipette out 10 ml of standard oxalic acid solution whose normality is 0.02476 and add half T.T of H_2SO_4 sulphuric acid & then warm it 60° - 70°C and add. This temp means one can hold the neck of conical flask by bare hand if temp is higher than this then one can't



hold the conical flask, the oxalic acid is heated to fasten the reaction At room temp. titration will be not be complete since reaction of KMnO4 & oxalic acid will be very slow.

At 60° - 70°C temp conical flask is removed from the bunsen burner and initial reading of KMnO4 from burette is already written in the copy hence immediately titration of oxalic Acid with KMnO4 is done. The titration is required to be completed in few mins so that solution remains hot finally the oxidation - reduction is balanced but as it is complete

no. of moles of O.A. is balanced by no. of moles of $KMnO_4$, equivalence point is reached but we can't deduct with eyes. So the end point is performed by adding a slight excess of $KMnO_4$ that solution becomes pink which one can easily visualize with naked eye. A single excess drop of $KMnO_4$ is sufficient for observing the end point. Repeat the process to get three concurrent readings

Titration of $KMnO_4$ Vs Std. Oxalic Acid of strength 0.02476 N.

S.No	I.R. $KMnO_4$	F.R.	Vol. of $KMnO_4$ used
1	0	11.0	11.0
2	0	11.0	11.0
3	0	11.0	11.0

$(N_1V_1)_{\text{Oxalic Acid}} = (N_2V_2)_{KMnO_4}$

$0.02476 \times 10 = N_2 \times 11.0$

$\frac{0.02476 \times 10}{11} = N_2$

$0.022\,N. = N_2$ (Normality of $KMnO_4$)

Step 3 Titration of Standardised $KMnO_4$ Vs unknown oxalic Acid.

Take 10 ml of unknown strength oxalic acid solution and add 1 Test Tube of H_2SO_4. Heat the contents upto 60° – 70°C and then titrate with $KMnO_4$ present in the burette. The end point is detected by observing pink color. Then the solution is thrown into the sink, wash the conical flask with water, and then take 10ml of unknown oxalic Acid + H_2SO_4 + Warm titrate with $KMnO_4$. Repeat the procedure till three concurrent readings are obtained

SNo	IR.	FR	Vol. of standardized $KMnO_4$
1	0.0	12.0	12.0
2	0.0	12.0	12.0
3	0.0	12.0	12.0

$(N_2 V_2')_{KMnO_4} = (N_3 V_3)_{unknown\ O.A}$

$0.022 \times 12 = N_3 \times 10$

$\frac{0.022 \times 12}{10} = N_3$

$0.00.22 \times 12 = N_3$

$0.0264\ N = N_3$

Normality of unknown oxalic Acid solution = 0.0264

Strength of unknown O.A sol is

$= 0.0264 \times 63$ g/lit

$= 1.6632$ g/l

Result:- The strength of unknown oxalic acid is 1.6632 g/l