Date

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AIM -

To determine the amount of oxalic acid and sulphuric acid present in one litre of the given solution.

APPARATUS -

Beaker, burette, pipette, funnel & conical flask

CHEMICALS -

NaOH soln, $KMnO_4$ soln, Oxalic acid, sulphuric acid, phenolphthalein, dil. H_2SO_4 soln.

THEORY -

To determine the amount of acids i.e. oxalic acid and sulphuric acid, two titrations are carried out -

1. First Titration is mixture of acids Vs. NaOH.
2. Second Titration is mixture of acids Vs. $KMnO_4$.

In the first titration, NaOH reacts with oxalic acid as well as sulphuric acid. According to the following reactions:

$$\begin{matrix} COOH \\ | \\ COOH \end{matrix} \cdot 2H_2O + 2NaOH \longrightarrow \begin{matrix} COONa \\ | \\ COONa \end{matrix} + 4H_2O$$

Oxalic Acid. Sodium Oxalate.

$$2NaOH + H_2SO_4 \longrightarrow Na_2SO_4 + 2H_2O.$$

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OBSERVATIONS- (Part-A)

Vol. of acid mixture in conical flask = 10ml (V_2)
Soln taken in burette - NaOH soln.
Indicator - Phenolphthalein.
End Point - Colourless to light pink.

Table: Given acid solution Vs N/10 NaOH soln.

S.No.	Burette Reading		Vol. of titrant used (F-I) (in ml)
	Initial (I)	Final (F)	
1.	0	13	13
2.	8	21	13
3.	6	19	13.

Concordant reading (V_1) = 13ml.

Normality of NaOH soln (N_1) = N/10.
Vol. of NaOH soln (V_1) = 13ml
Normality of mixture (N_2) = ?
Vol. of mixture (V_2) = 10ml.

CALCULATIONS -

$$N_1V_1 = N_2V_2$$
(NaOH) (Mixture)

$$\frac{1}{10} \times 13 = N_2 \times 10.$$

$$N_2 \text{ (mixture)} = 0.13 \text{ N}$$

So, the total normality of the mixture of acids can be determined.
But in second titration, only oxalic acid (present in the given mixture of acids) reacts with $KMnO_4$ as follows,

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$
$$(H_2C_2O_4 + [O] \longrightarrow 2CO_2 + H_2O) \times 5$$

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

PROCEDURE –

A. Titration of given acid solution ~~with~~ against NaOH (N/10) ~~solution~~ –

- Rinsed and filled the burette with N/10 NaOH soln.
- Pipetted out 10ml of the given mixture soln into a conical flask.
- Added to it 2-3 drops of phenolphthalein (HPh) indicator.
- Noted initial burette reading.
- Titrated against N/10 NaOH soln till pink colour appears. This is the end point.
- Noted the final burette reading.
- Repeated the titration to get three concordant readings

OBSERVATIONS-(Part-B)

Vol. of acid mixture in conical flask (V_4) - 10ml.
Soln taken in Burette - $KMnO_4$ soln.
Indicator - $KMnO_4$ (self indicator)
End Point - colourless to light pink.

Table: Given acid mixture soln. Vs. N/20 $KMnO_4$ soln.

S.No.	Burette Reading Initial (I)	Final (F)	Vol. of titrant used (F - I) (in ml)
1.	0	4.7	4.7
2.	5	9.7	4.7
3.	10	14.7	4.7

Concordant reading, V_3 = 4.7 ml

Normality of $KMnO_4$ soln (N_3) = N/20.
Vol. of $KMnO_4$ soln (V_3) = 4.7 ml.
Normality of oxalic acid (from mixture) (N_4) = ?
Vol. of mixture (V_4) = 10 ml.

CALCULATIONS-

$$N_3V_3 = N_4V_4$$
($KMnO_4$) (Mixture)

$$\frac{1}{20} \times 4.7 = N_4 \times 10$$

$$N_4 = \frac{4.7}{200}$$

N_4 (oxalic acid) = 0.024 N

B. Titration of acid mixture solution against N/20 $KMnO_4$ solution -

- Rinsed and filled the burette with standard N/20 $KMnO_4$ solution
- Pipetted out 10ml of given mixture solution into a conical flask.
- Added to it 10 ml of dilute H_2SO_4.
- Heated the conical flask slowly until the moisture appears on the neck of conical flask or it is bearable to touch (60° - 70°C)
- Noted initial burette reading.
- Titrated it till the light pink colour appears as the end point. Noted final burette reading
- Repeated the steps to get three concordant readings.

NOTE -

NaOH reacts with oxalic acid as well as H_2SO_4, but $KMnO_4$ reacts only with oxalic acid. So, first titration A gives normality of total acid solution (N_2) while the second titration B gives normality of oxalic acid present in the acid mixture (N_4)

CALCULATIONS -

Normality (oxalic acid) = 0.024 N

Strength of oxalic acid = Normality × Eq Weight

= 0.024 × 63

= 1.48 gm/L

Normality of H_2SO_4 = Normality of acid mixture − Normality of oxalic acid

= 0.13 − 0.024

= 0.106 N

Strength of H_2SO_4 = Normality × Eq Weight

= 0.106 × 49

= 5.194 gm/L

RESULTS -

Strength of oxalic acid in given acid mixture solution is 1.48 gm/L

Strength of sulphuric acid in given acid mixture solution is 5.194 gm/L

PRECAUTIONS -

1. As rubber is attacked by $KMnO_4$, do not use rubber pinch cork burette.
2. $KMnO_4$ being of dark colour, read the upper meniscus as its lower meniscus can not be read accurately.
3. In $KMnO_4$ - Oxalic acid titration, do not overheat the flask.

VIVA -

1. $KMnO_4$ is not a primary standard Explain

Potassium Permanganate ($KMnO_4$) is not suitable to be used as a primary standard because of the following reasons-

i) it cannot be obtained in very pure form.

ii) it decomposes in presence of sunlight.

iii) it readily reacts with any traces of organic material or any other reducing substance in water.

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2. Why oxalic acid solution is heated before titration with $KMnO_4$ solution.

Oxalic acid is heated before titration with $KMnO_4$ solution because this reaction happens only at a certain temperature, this is a very slow reaction and heating increases the rate of the reaction. If not heated the light pink titrated solu turns brown soon & reaction takes longer time but overheating causes decomposition of oxalic acid to CO_2.

3. What are the types of indicators used in this reaction?

In the first titration of the acid mixture solution with NaOH, phenolphthalein is used as indicator.

In the second titration of oxalic acid with $KMnO_4$ solution, $KMnO_4$ solu acts as a self indicator.

4. Why are titrations involving $KMnO_4$ carried out in the acidic medium?

Sulphuric acid is added to prevent hydrolysis and to provide surplus H^+ ions in the solution to keep the reaction proceeding. Also sulphuric acid is stable towards oxidation.

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