

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 -

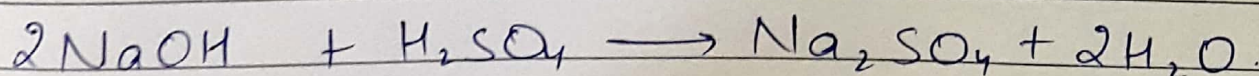
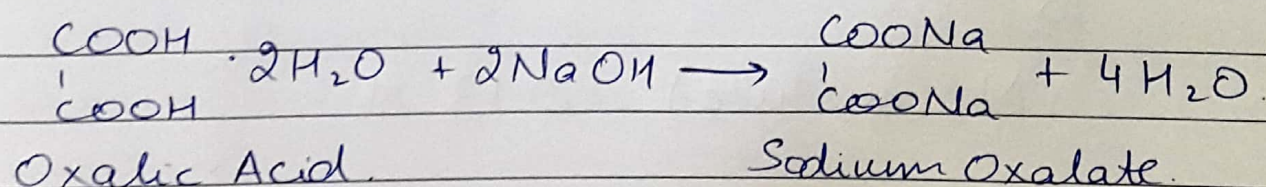
NaOH soln, KMnO_4 soln, Oxalic acid, Sulphuric acid, phenolphthalein, dil. H_2SO_4 soln.

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:



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 -

$$N_1 V_1 = N_2 V_2$$

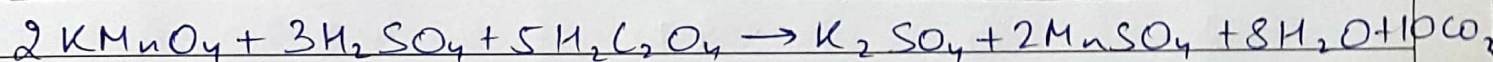
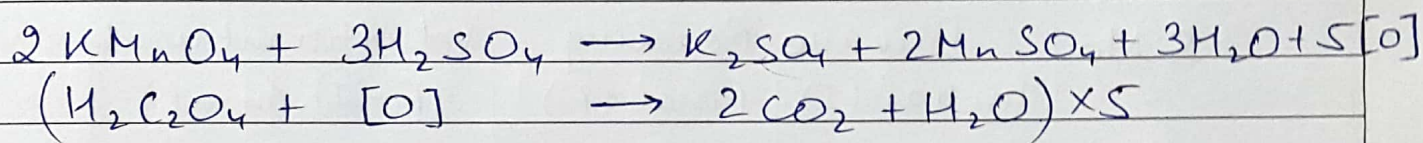
(NaOH) (Mixture)

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

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

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,



PROCEDURE -

A. Titration of given acid solution ~~with~~ against NaOH ($\text{N}/10$) solution -

- Rinsed and filled the burette with $\text{N}/10$ 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 $\text{N}/10$ 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. $\text{N}/20$ KMnO_4 soln.

S.No.	Burette Reading		Vol. of titrant used (F - I) (in ml)
	Initial (I)	Final (F)	
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) = $\text{N}/20$.

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 -

$$\begin{aligned} N_3 V_3 &= N_4 V_4 \\ (\text{KMnO}_4) & \quad (\text{Mixture}) \\ \frac{1}{20} \times 4.7 &= N_4 \times 10 \end{aligned}$$

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

$$N_4 (\text{oxalic acid}) = 0.024 \text{ N}$$

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 10 ml 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 ($60^\circ - 70^\circ\text{C}$)
- 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 gives normality of total acid solution (N_2) while the second titration B. gives normality of oxalic acid present in the acid mixture (N_1)

CALCULATIONS -

$$\text{Normality (oxalic acid)} = 0.024 \text{ N}$$

$$\begin{aligned}\text{Strength of oxalic acid} &= \text{Normality} \times \text{Eq Weight} \\ &= 0.024 \times 63 \\ &= 1.48 \text{ gm/L.}\end{aligned}$$

$$\begin{aligned}\text{Normality of H}_2\text{SO}_4 &= \text{Normality of acid mixture} - \text{Normality of oxalic acid} \\ &= 0.13 - 0.024 \\ &= 0.106 \text{ N.}\end{aligned}$$

$$\begin{aligned}\text{Strength of H}_2\text{SO}_4 &= \text{Normality} \times \text{Eq Weight} \\ &= 0.106 \times 49 \\ &= 5.194 \text{ gm/L.}\end{aligned}$$

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 soln 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 soln 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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