

EXPERIMENT-9

AIM

Perform pH metric titration of a strong acid, hydrochloric acid with a strong alkali, sodium hydroxide.

Take 25 mL of 0.05 M HCl in 50 mL beaker and titrate with 0.1M NaOH solution in an interval of 0.5 mL

APPARATUS REQUIRED

A pHmeter (an electronic meter and glass combination electrode), 50mL beaker, burette and burette stand, wash bottle, pipette, plastic funnel, glass rod.

CHEMICALS REQUIRED

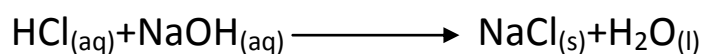
0.05 M HCl solution , 0.1 M NaOH solution, distilled water

THEORY

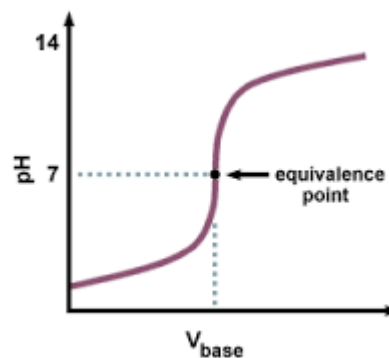
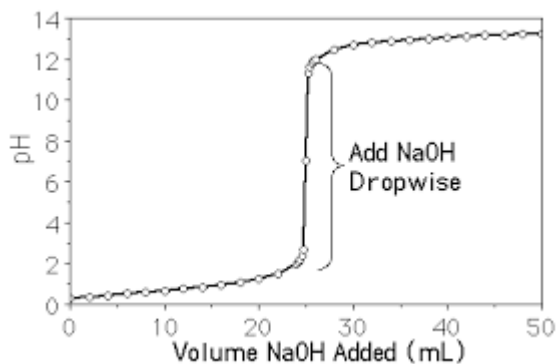
Most of the chemical and biochemical processes are profoundly affected by the acidity or alkalinity of the medium in which the reaction takes place. All acids dissociate in aqueous solution to yield H^+ ions. Some acids like HCl, H_2SO_4 , HNO_3 etc. are completely ionized in aqueous medium where as CH_3COOH , $HCOOH$ etc. ionize to a small extent only. The former are known as strong and the later as weak acids. **pH of any solution is defined as $(-\log H^+)$** and has values between 0–14. $pH < 7$ indicate acidic solution, $pH > 7$ indicate basic solution and $pH = 7$ means neutral solution.

In an acid-base titration, the important information to obtain is the equivalence point. **It is the point at which an equal amount of acid has been neutralised by equal amount of base or vice versa.** If there are a number of moles of acid in the titration flask, the equivalence point is reached when that same number of moles of base added is the same as the number of moles acid in the titration flask, and the volume of the base added is also known. Similarly, if the number of moles acid in the titration flask is unknown, it can be calculated for the equivalence point if the molarity of the base and the volume of base added are known.

The pH of a solution can be measured accurately with the help of a pH meter. Measurement of pH is employed to monitor the cause of acid-base titration. It affords a direct method of obtaining a titration curve. The titration curve is a graph of measured pH values versus the volume (ml) of titrant added. The pH values of the solution at different stage of acid–base neutralization are determined and plotted against the volume of alkali added on adding a base to an acid, the pH rises slowly in the initial stages as the concentration of H⁺ ion decreases gradually due to the consumption of some amount of HCl by NaOH resulting in the formation of NaCl (whose amount will be the same as that of NaOH added).



But, at the equivalence point (the point at which an equal amount of acid has been neutralised by equal amount of base or vice versa), pH increases rapidly as at the equivalent point H⁺ ion concentration is very small. Then it flattens out after the end point. The end point of the titration can be detected where the pH value changes most rapidly. However, the shape of the curve depends upon the ionisability of the acid and the base used and also on the acidity of base and basicity of the acid.



Once a titration curve is constructed and the equivalence point established the experimenter could then choose an indicator to perform the titration calorimetrically that would give a suitable endpoint (point at which indicator changes colour).

INDICATOR

The pH at the equivalence point is 7 and slightly before and after the equivalence point, it encompasses an interval from pH = 3 to pH = 11. Therefore, any indicator whose pH range is within this interval i.e. indicators which will change their colour within this pH range can be used. *However, the range of the steep change of pH at the equivalence point depends upon the concentrations of acid and base being titrated.* Methyl orange, methyl red and phenolphthalein, the common laboratory indicators can be used for this purpose as their respective pH ranges lie in the interval 3-11. The usual choice, however, is phenolphthalein because the colour change in this case is from colourless to a slight pink colour, a change which can be easily detected.

PROCEDURE

1. 0.1(M) NaOH solution is provided.
2. HCl solution of unknown strength is provided.
3. Calibrate the pH meter with the solutions of pH 4,7 and 10.
4. **pH-metric Titration:** Clean the electrode with distilled water and wipe them with tissue paper or filter paper. Take 25mL of HCl solution in a 50 mL beaker and immerse the electrode in it. Note down the pH range. Rinse the burette with distilled water and then with NaOH solution. After that fill the burette with NaOH solution. The reading shown on the scale of pH meter is pH value of the HCl solution. Add NaOH solution drop wise from the burette (maximum 0.5 mL at a time), shake the solution well with the help of glass rod and note the corresponding pH values. Near the end point, volume of NaOH added should be as small as possible (0.1 ml at a time, which is the least count of an burette that we use in our laboratory) because the acid is neutralized and there will a sharp increase in pH values. Further addition of even 0.01 mL of NaOH, increase the pH value to about 9–10. Put back the selector to zero position after each pH measurement, and always keep the selector at zero position when it is not in use.
Plot the graph of pH of solution v/s volume of NaOH added.

OBSERVATIONS AND CALCULATIONS

1. Preparation of 250 mL 0.05 M standard oxalic acid solution:

Mass, w of oxalic acid to be weighed for the preparation of solution

$$w = \frac{.05 \times 126 \times 250}{1000} = 1.5750 \text{ g}$$

Mass of empty weighing bottle = x =

Mass of weighing bottle and oxalic acid = $y =$

Mass of weighing bottle after transfer = $z =$

Actual mass of oxalic acid transfer = $w_1 = (y-z) =$

2. Titration of standard oxalic acid solution against NaOH:

Solution in burette = oxalic acid

Solution in titrating flask = $V_1 = 20$ mL standard oxalic acid

Indicator used = phenolphthalein

End point = colourless to pink

| S.no. | Burette reading | | Volume of HCl used |
|-------|-----------------|-------|--------------------|
| | Initial | Final | |
| 1. | | | |
| 2. | | | |
| 3. | | | |

Concordant volume(V_2)=

Table 1:- pH metric Titration

| Volume of HCl taken (V_3) (mL) | Volume of NaOH added (V) (mL) | pH |
|------------------------------------|-----------------------------------|----|
| 25 | | |

| V_{NaOH} | pH | $\Delta\text{pH} = \text{pH}_f - \text{pH}_i$ | $\Delta V = V_f - V_i$ | $\Delta\text{pH}/\Delta V$ | $V_{\text{avg}} = (V_i + V_f)/2$ | |
|-------------------|----|---|------------------------|----------------------------|----------------------------------|--|
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CALCULATIONS

1. Molarity of standard oxalic acid solution:

$$M_1 = w_1 \times 1000 / (\text{molar mass} \times V)$$

$$M_1 = w_1 \times 1000 / 126 \times 250$$

$$M_1 =$$

2. Molarity of given NaOH solution:

$$M_1 V_1 / n_1 = M_2 V_2 / n_2$$

(Standard oxalic acid) (NaOH)

$$n_1 = 1; \quad n_2 = 2$$

$$M_2 = \frac{M_1 \times V_1 \times n_2}{V_2 \times n_1}$$

$$=$$

3. Molarity of given HCl solution:

$$M_3 V_3 / 1 = M_2 V_4 / 1$$

(Given HCl) (NaOH)

$$M_3 = \frac{M_2 \times V_4}{V_3}$$

$$=$$

Strength of the given HCl will therefore be = $S_1 = M_3 \times 126 =$

Plot a graph between pH and volume of NaOH (V_{NaOH}) added. There should be a region in your graph where the slope is very steep.

Determine the mid point of this region. This is the equivalence point. Find out the corresponding volume of NaOH required (V_4 mL) for complete neutralization of HCl from the graph. Then find out the strength of HCl (S_1).

$$25 \times M_3 = V_4 \times M_2$$

(HCl) (NaOH)

Strength of HCl (S_1) = $(M_1) \times 126$ (molar mass of HCl)

Plot a graph between $\Delta\text{pH}/\Delta V$ and V_{avg} . There should be a peak in your graph which is the equivalence point. Find out the corresponding average volume of NaOH required (V_4 mL) for complete neutralization of HCl from the graph. Then find out the strength of HCl (S_1).

$$25 \times M_3 = V_4 \times M_2$$

(HCl) (NaOH)

Strength of HCl (S_1) = $(M_1) \times 126$ (molar mass of HCl)

RESULT

The strength of the unknown HCl = $S_1 =$ _____ g L^{-1}

PRECAUTIONS

- i. Electrodes must be immersed in the solution properly and sufficient time to be allowed for the electrodes to obtain the temperature of the solution.
- ii. pH meter should be calibrated before the experiment.
- iii. Magnetic stirrer may be used or the solution be stirred mechanically from time to time during pH metric titration using glass rod.
- iv. Leave the selector in zero position when it is not in use.
- v. The glass electrode should always be kept dipping in the distilled water when not in use.