C 141 (Experiment No.____)
NAME : ______________________________ **BATCH : _______**
ROLL No. : ___________________________
SIGNATURE : ________________________ **DATE : ________**

pH - METRIC TITRATION OF A POLYPROTIC ACID Vs BASE

AIM

To determine the volume of a polyprotic acid (H_3PO_4) in the given unknown solution and to estimate the amount of H_3PO_4 present in 100ml of given acid solution by titrating against a strong base (NaOH), using pH – meter.

THEORY

In order to express the acidity or alkalinity of a solution, one can use the hydrogen ion concentration. The ionic product of water is 10^{-14} .
Therefore, in water (it is neutral), we have : $[H^+] = [OH^-] = 10^{-7}$
The pH of a solution is defined as the logarithm of the reciprocal of hydrogen ion concentration (or) negative logarithm of hydrogen ion concentration.
That is :

$$pH = \log \frac{1}{[H^+]} \quad \text{(or)} \quad pH = -\log [H^+]$$

We can use glass electrode as indicator electrode and calomel electrode as reference electrode or sometimes combined electrode may be used. When glass electrode is in contact with the solution, it acquires some potential which depends upon the concentration of H^+ ion in solution i.e., thereexist a potential difference at the interface between the glass electrode containing H+ and the solution.
The determination of pH of the solution can be represented as:

pt/0.1N H_3PO_4/glass electrode/test solution/KCl saturated with $HgCl_2$/Ag^+

The e.m.f of this cell may be calculated potentiometrically. The potential of the calomel and glass electrodes are known, so the pH of the solution can be calculated using the following equation.

$pH = E_0 – (0.2242 / 0.0591) + \text{potential of calomel electrode}$

When an alkali is added to an acid solution, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution increases very rapidly.
From the sharp jump in the curve, we can find the equivalence point, from which the strength can be calculated.

MATERIALS REQUIRED

pH meter, electrodes (combined glass electrode or indicator and reference), beaker (100 ml), burette (50 ml), pipette (10 ml), stirrer / glass rod, conical flask (100 ml), volumetric flask (100 ml), oxalic acid solution (0.1 N), H_3PO_4 solution, NaOH solution, buffer solutions (pH – 4.01, 7.0, 9.18).

PROCEDURE

PART-I: Standardization of NaOH against standard Oxalic acid (0.1N)

1. 10ml of given 0.1N standard Oxalic acid is pipetted out into a 100ml conical flask.
2. This solution is titrated against the given unknown concentration of NaOH using 1-2 drops of phenolphthalein indicator until the end point is colorless to pale pink.
3. Tabulate the values and repeat the titration for concurrent readings and determine the concentration of supplied NaOH solution.

Table 1

S.No	Volume of Oxalic Acid taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)
1				
2				
3				

$(HCOO)_2$ $N_1 V_1 = N_2 V_2$ (NaOH)

PART-II: Measurement of pH

1. The pH meter is standardized or calibrated by using the buffer solutions of appropriate pH.
2. 10 ml of the given H_3PO_4 solution is taken in a 100 ml beaker and add 40ml of distilled water using burette.
3. Insert the electrode in the beaker containing above solution, so that they are dipped in the acid solution.
4. Stir the solution well and measure the initial pH of the solution.
5. Add 0.5 ml or 1.0 ml portion of NaOH solution from the burette, and stir the contents thoroughly.
6. Measure the pH of the solution for each addition of base. This is known as pilot titration for equivalence point determination.
7. On continuing the titration, there will be 2 pH-metric jumps will be observed. The same procedure will be adopted for the accurate titration by adding 0.1 ml portions of strong base from the burette, where exact end point is to be determined.
8. Repeat this procedure for the unknown volume of solution supplied in the 100ml volumetric flask and calculate the amount of H_3PO_4 present in it.
9. Plot the graph(s) for the known and unknown solutions. From the sudden rise in pH on the S-curve of the graph, plot the line to the x-axis which gives you the volume of base consumed. And also, for the difference in pH with respect to the difference in volume gives the volume of NaOH consumed in ml.
10. From the known solution, calculate the strength of H_3PO_4 solution and the amount of H_3PO_4 present in 100 ml of the original solution.
11. From the unknown solution, calculate the volume of H_3PO_4 present in the solution.
12. Theoretical and Graphical calculations are to be shown in separate & proper format for the known and unknown solutions with all formulas, units etc.

Note

Handle the instrument, electrodes(cell), glassware with care.
Avoid parallax error while taking the readings.
The electrode should not touch the walls of the beaker.

Table 2

Sl. No.	Volume of NaOH consumed, v (ml) Pilot		Accurate		Δv		pH		pH		pH / Δv	
	known	unk	known	unk	known	unk	known	unk	known	unk	known	unk
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

$$(NaOH))\ N_2 V_2 = N_3 V_3\ (H_3PO_4)$$

OBSERVATIONS AND CALCULATIONS

Room Temperature = ... ^{o}C.

1. The values of the observed pH are plotted as ordinate (y - axis) against the volume of NaOH solution added as abscissa (x - axis).

   And also, the values of the ΔpH / Δv are plotted as ordinate (y - axis) against the volume of NaOH solution added as abscissa (x – axis).

2. Find out the end point from both the graphs by plotting a line on S-curve to the x-axis. This gives the volume of NaOH solution at the equivalence point.

3. The strength of the unknown acid solution and the amount of H_3PO_4 present in the given unknown solution is calculated.

RESULTS

1. Strength of NaOH solution = ...
2. Strength of known acid (H3PO4) = ...
3. Volume of unknown acid solution = ...
4. Amount of H_3PO_4 present in 100 ml of original solution = ...
5. Comment on the nature of the graph(s).

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