Ex. No. 3
Date:

DETERMINATION OF pK$_a$ VALUE OF GLYCINE BY pH METRY

AIM:

To determine the titration curve of the given amino acid, glycine and to use this curve to estimate its pK$_a$ values.

PRINCIPLE:

Amino acids have the general formula NH$_2$-CHR-COOH. They can act as both acids and bases. In water, most amino acids exist as zwitter ions, i.e., a dipolar ion. Thus, they are amphoteric molecules that can be titrated with both acid and alkali. Glycine, the simplest amino acid, has one acid and one amine group and is diprotic in nature. Isoelectric point, pI, is the pH of an amino acid at which the net charge on the molecule is zero. When such an amino acid is titrated against a base, it acts as an acid, and with an acid, it acts as a base.

The pH of such a weak acid or base can be calculated from the Henderson-Hasselbalch equation

$$pH = pK_a + \log \frac{[basic\ species]}{[acidic\ species]}$$

APPARATUS AND REAGENTS:

0.1 N glycine, 0.1 N HCl, 0.1 N NaOH, pH meter, pH glass electrode, burettes, beaker and buffer solutions (4, 7 and 9) for calibrating pH meter.

PROCEDURE:

TITRATION I: Glycine vs HCl

10 ml of the given 0.1 N amino acid, glycine is pipetted out into a clean 100 ml beaker followed by the addition of 20 ml of distilled water. A pH glass electrode is rinsed in de-ionized water and immersed in the glycine solution. The pH of the mixture is recorded using a calibrated pH meter. A clean burette is taken and filled with 0.1 N HCl.

Initially, 1 ml of HCl is added to the mixture. The solution is stirred well using a glass rod and the change in pH is recorded. The acid is added in increments of 1 ml until the pH falls to 3. Between pH 3 and 2, HCl is added in increments of 0.5 mL. Beyond pH 2, HCl is added in increments of 1 ml until the pH reaches 1.5. The readings are recorded in a tabular column as shown in Table 1.

TITRATION II: Glycine vs NaOH

10 ml of the given 0.1 N amino acid, glycine is pipetted out into a clean 100 mL beaker followed by the addition of 20 mL of distilled water. A pH glass electrode is rinsed in de-ionized water and immersed in the glycine solution. The pH of the mixture is recorded using a calibrated pH meter. A clean burette is taken and filled with 0.1 N NaOH.

Initially, 1 ml of NaOH is added to the mixture. The solution is stirred well using a glass rod and the change in pH is recorded. The base is added in increments of 1 mL until the pH increases to 9. Between pH 9 and 10, NaOH is added in increments of 0.5 mL. Beyond pH 10, NaOH is added in increments of 1 mL until the pH reaches 11.5. The readings are recorded in a tabular column as shown in Table 2.

The titration curve is plotted with Volume of HCl and NaOH on the x axis and pH on the y axis. The two plateaus on the curve obtained is noted where despite the addition of the acid or base, the pH of the solution does not vary significantly. The pKa values of glycine are estimated from the graph. These values are then compared with that reported in literature.
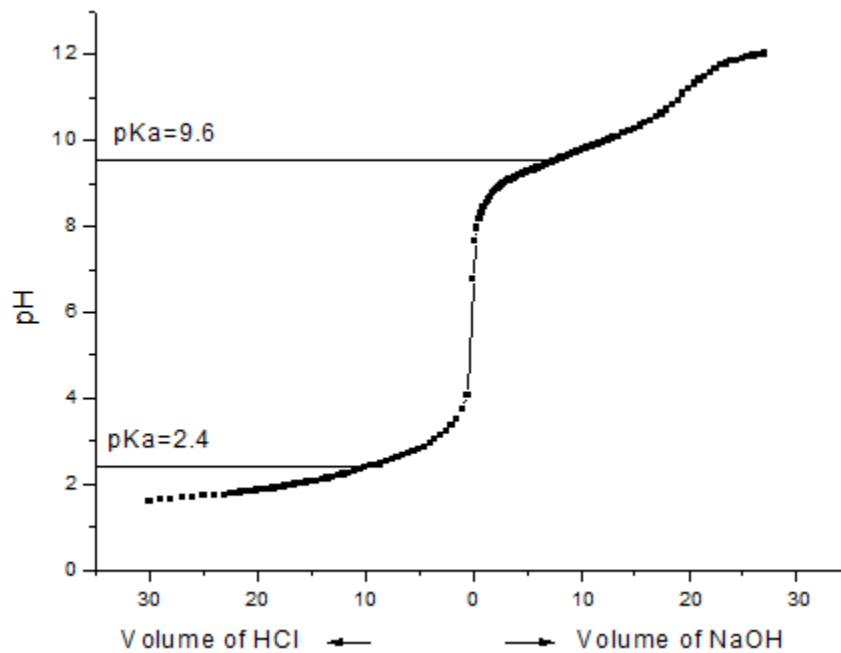
RESULT: The two pK_a values of glycine are _____ and _____.

OBSERVATION:

TABLE 1: Glycine vs. HCl				TABLE 2: Glycine Vs. NaOH		
Sl. No.	Volume of 0.1 N HCl	Observed pH		Sl. No.	Volume of 0.1 N NaOH	Observed pH
1	1			1	1	
2	2			2	2	
3	3			3	3	
4	4			4	4	
5	5			5	5	
6	6			6	6	
7	7			7	7	
8	8			8	8	
9	9			9	9	
10	10			10	10	
11	11			11	11	
12	12			12	12	
13	13			13	13	
14	14			14	14	
15	15			15	15	
16	16			16	16	
17	17			17	17	
18	18			18	18	
19	19			19	19	
20	20			20	20	
21	21			21	21	
22	22			22	22	
23	23			23	23	
24	24			24	24	
25	25			25	25	
26	26			26	26	
27	27			27	27	
28	28			28	28	
26	26			26	26	
30	30			30	30	

MODEL GRAPH:

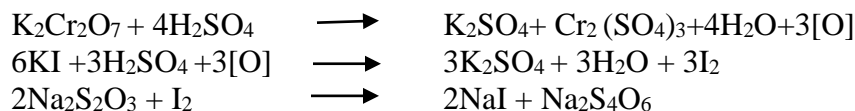**Ex. No. 6**
Date:

ESTIMATION OF COPPER IN BRASS

AIM:

To estimate the percentage of copper present in the given ore. You are supplied with an accurately 0.5 N potassium dichromate solution and an approximately 0.05 N sodium thio sulphate solution.

PRINCIPLE:

The estimation is based on iodometry titration. In iodometry, a strong oxidizing agent such as potassium dichromate is treated in neutral or acid solution with a large excess or iodide ions the latter reacts as a reducing agent and the oxidant will be quantitatively reduced .in such cases an equivalent amount of iodine is liberated and the titrated with a solution of a reducing agent, which is usually sodium thiosulphate freshly prepared starch solution is used as an indicator which given intense blue colour with iodine due to the formation of an adsorption complex. The end point is the disappearance of the blue colour.

A solution of potassium dichromate acidified with dilute sulphuric acid is treated with excess of 10% KI solution. The liberated iodine is treated against sodium thiosulphate using starch indicator until the blue colour disappears.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$
$$6KI + 3H_2SO_4 + 3[O] \longrightarrow 3K_2SO_4 + 3H_2O + 3I_2$$
$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

On adding potassium iodine to a finely ground ore dissolved in perchloric acid, copper reacts to liberate iodine

$$2Cu^{2+} + 4KI \longrightarrow 4K^+ + Cu_2I_2 + I_2 \uparrow$$
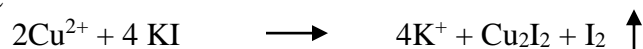
The liberated Iodine is titrated against sodium thiosulphate using starch indicator. The disappearance of blue colour is the end point.

PROCEDURE:

The given copper ion solution is made upto 100ml in a standard flask and is prepared in the following manner.0.5gm of the ore is accurately weighed and dissolved in 15ml of 75% perchloric acid in an iodine flask. The mixture is refluxed without allowing the various to escape. The contents are cooled and 15ml of water is added and again boiled for 5 mintues. After cooling 1:1 ammonium hydroxide solution is added to precipitate ion hydroxide.

TITRATION I: Standardization of sodium thiosulphate

Pipette out 20ml of standard potassium dichromate into clean conical flask. Add one test tube of dilute sulphuric acid and 10ml of 10% KI solution. Titrate the liberated iodine immediately against sodium thiosulphate taken in the burette. When the solution turns pale yellow, add 1ml of starch indicator and continue the titration. The end point is the disappearance of blue colour. Repeat the titration for concordant titre value and calculate the strength the sodium thiosulphate solution.

TITRATION II: Estimation of copper

Pipette out 20ml of the made up copper solution into a clean conical flask. Add liquor ammonia drop by drop until a deep blue precipitate is formed. Dissolve the precipitate with slight excess of dilute acetic acid and add 10ml of 10% KI solution. Titrate the liberated iodine against sodium thiosulphate taken in the burette. When the solution becomes straw yellow, add 1ml starch indicator and continue the titration. The end point is the disappearance of blue color. Repeat the titration for concordant titre value and calculate the strength of copper in the solution. From this, the amount of copper is estimated.

RESULT:

The percentage of copper present in the given ore =_____%

OBSERVATION AND CALCULATION:

TITRATION I: Standardization of sodium thiosulphate

Burette Solution	:	Sodium thiosulphate
Pipette solution	:	Std. potassium dichromate + 20ml dil.H_2SO_4 +10ml of 10% KI
Indicator	:	Starch
End Point	:	Disappearance of blue colour (Appearance of pale green colour)

S. No.	Volume of $K_2Cr_2O_7$ (ml)	Burette Reading		Volume of thiosulphate (ml)	Concordant value
		Initial	Final		

Volume of potassium dichromate,	V_1	=	ml
Normality of potassium dichromate,	N_1	=	N
Volume of Sodium thiosulphate,	V_2	=	ml
Normality of sodium thiosulphate,	N_2	=	?

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

TITRATION II: Estimation of copper
Burette Solution   :   Std. sodium thiosulphate
Pipette solution   :   20ml of copper ion solution +10ml of 10% KI
Indicator   :   Starch
End Point   :   Disappearance of blue color (Appearance of white percipitate)

S. No	Volume of copper ion solution (ml)	Burette Reading		Volume of thiosulphate (ml)	Concordant value
		Initial	Final		

CALCULATION:

Volume of sodium thiosulphate   V1   =   ml
Normality of sodium thiosulphate   N1   =   N
Volume of copper ion solution   V2   =   ml
Normality of copper ion solution   N2   =   ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

The amount of copper present in one liter of the solution $=$ Normality $\times$ 63.54
         $= x\ g/lit.$

The amount of copper present in the whole of the given solution $= \dfrac{x \times 100}{1000}$

         $= y\ g$

The percentage of copper in the given ore $= \dfrac{y \times 100}{0.5}$

         $=$ _____%

Ex. No. 8
Date:

DETERMINATION OF HIGH MOLECULAR WEIGHT OF A POLYMER BY USING OSTWALD VISCOMETER

AIM:

To determine the average molecular weight of given polymer by U-tube viscometer method and you are provided with a 2.5 wt% of polyvinyl alcohol solution.

PRINCIPLE:

The viscosity of a polymer solution is high compared to that of the pure solvent. The molecular weight of a polymer is proportional to the intrinsic viscosity of its solution (η_i) when the polymer molecular chain is extended to its full contour length. The value of intrinsic viscosity is proportional to the square root of the molecular weight M, when the polymer is in a solvent.

The relationship between intrinsic viscosity and molecular weight is given by Mark-Kuhn-Hownik's empirical equation as,

$$\eta_i = KM^\alpha$$

where α is a measure of the shape of the polymer molecular chain (coiled or extended form) and the value is usually from 0.5 to 0.8. The value of the constant K depends on the polymer and the solvent involved.

For a liquid flowing through a capillary tube, the time required to pass through the capillary tube is related to the absolute viscosity according to the equation:

$$\eta = \frac{\pi\, Pr^4\, t}{8Vl}$$

where V is the volume of the liquid which pass through the capillary, r is the radius of the capillary, l is the length of the capillary and P is the pressure head under which the liquid flow takes place.

If η and η_o are the absolute viscosities of the polymer solution and the pure solvent respectively and t and t_o are their corresponding flow times and if the same capillary tube is used. Therefore, the relative viscosity of polymer solution is given by:

$$\frac{t}{t_o} = \frac{\eta}{\eta_o}$$

For each concentration, calculate the value of η/η_0 and η_{sp} (specific viscosity)

$$\eta_{sp} = \left(\frac{t}{t_o} - 1\right)$$

From the plot of η_{sp}/C vs concentration of the polymer, intrinsic viscosity can be calculated.

APPARATUS & REAGENTS:

Ostwald's Viscometer, Stop Watch, 100ml Beaker, 25ml Standard Measuring Flask, 10ml Graduated Pipette; 10ml Ordinary Pipette.

PROCEDURE:

25 ml of 0.25%, 0.5%, 0.75%, 1%, and 1.25% polymer solutions are prepared as follows. Pipette out 2.5, 5, 7.5, 10, and 12.5 ml of 2.5% polymer solution in a 25 ml standard flask. Then make up the polymer solution with distilled water and shake the solution thoroughly.

Wash the viscometer with distilled water and dry it. Fix the viscometer vertically in a stand. Pipette out 10ml of the solvent into the lower bulb of the viscometer and suck the liquid carefully into the upper bulb, so that the liquid level is above the mark. Allow the liquid to flow freely. When the liquid level crosses the upper mark, start a stopwatch. When the liquid level crosses the lower mark, stop the stopwatch. Repeat it for two times. The average value is the flow time of water.

Similarly determine the flow time of given polymer solutions of different concentrations. Tabulate the results.

From the flow time we can calculate the specific viscosity.

$$\eta_{sp} = \left(\frac{\eta}{\eta_o}\right) - 1$$

$$\eta_{sp} = \left(\frac{t}{t_o}\right) - 1$$

From the plot of η_{sp}/c versus concentration, the intrinsic viscosity (Y-intercept) η_i can be determined. Using η_i, α and K values, the molecular weight of the polymer can be determined.

For polyvinyl alcohol-water system, the 'α' and 'K' values are 45.3×10^{-5} dl/g and 0.64 respectively.
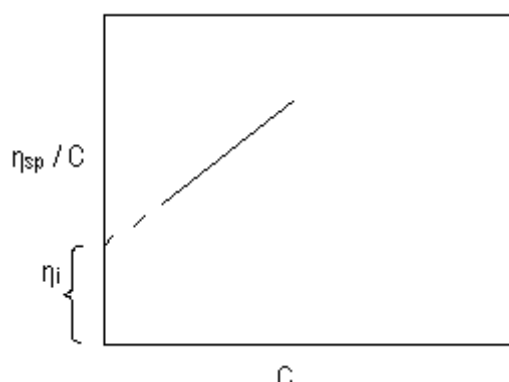
RESULT:

The molecular weight of the given polymer sample =

OBSERVATION:

S. No.	Concentration of the polymer solution, (c)	Time of flow (t)	$\eta/\eta_0 = t/t_0$	$\eta_{sp} = (t/t_0) - 1$	η_{SP}/c
1.	Distilled water	(t_o)			
2.	0.25%				
3.	0.5%				
4.	0.75%				
5.	1.0%				
6.	1.25%				

MODEL GRAPH:

CALCULATION

$$\eta_i = KM^{\alpha}$$

$$\log \eta_i = \log K + \alpha \log M$$

$$\log M = \frac{\log \eta_i - \log K}{\alpha}$$

$$\log M = \frac{\log \eta_i - \log(45.3 \times 10^{-5})}{0.64} \qquad [\eta_i \text{ - Refer graph}]$$

$$\log M = \frac{\log \eta_i - (-3.3437)}{0.64}$$

$$M = anti \log of \left[\frac{\log \eta_i - (-3.3437)}{0.64} \right]$$