INSTRUMENTAL ANALYSIS EXPERIMENTS

Fourth Class

2nd Course

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Experiment No. 1

Calculation of acid ionization constant (week acid) from pH measurements

Procedure:

1- Calibrate the glass electrode using a buffer solutions of pH=4 and pH=9.

2- Transfer aqueous solution of unknown acetic acid in a 100 ml beaker. Immersed the glass electrode inside the solution with the aid of distilled water and measure the pH value at this stage.

3- Add 0.1N of sodium hydroxide from the burette to the unknown aqueous solution of acetic acid gradually and record the pH after each addition. Add sodium hydroxide solution according to the following table:

Volume of NaOH (0.1N)	pH
1 mL, 1 mL	5
0.5 mL , 0.5 mL	7
0.2 mL , 0.2 mL	9
1 mL , 1 mL	12

Results and Calculations

1- Arrange and tabulate your results in a table include the added volume of 0.1 N – NaOH versus pH-value.

2- From the curve of pH-value vs. volume of 0.1 N NaOH find and allocate the volume of NaOH at equivalent point (V1).

3- Calculate the concentration of hydrogen ion (hydronium ion or Oxonium ion) from the recorded pH value.

$$pH=-\log[H^+] \quad \text{or} \quad pH=-\log[H_3O^+]$$

4- Calculation the unknown acid concentration N_2 as:

$$N_1 \times V_1 = N_2 \times V_2$$

N_1=0.1N and V_2=unknown solution volume

5- Calculate the weak acid ionization constant K_a as follows:

$$Ka = \frac{[H^+]\,[CH_3COO^-]}{[CH_3COOH]} = \frac{[H^+]^2}{[CH_3COOH]}$$

Experiment No. 2

Spectrophotometric Determination Phosphate ion

Procedure

1- introduce 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0 mL of 10 ppm phosphate ion in 50 mL volumetric flask.
2- Add 0.5 mL of ammonium molybdate (4%) with the aid of graduated cylinder to each volumetric flask.
3- Leave the solution with continuous mixing for 5 mins.
4- Add by graduated cylinder 0.5 mL of ascorbic acid (2%) to each volumetric flask.
5- Complete the solutions to 50 mL with distilled water and warm the solutions (10 mins) until the blue color of molybdenum is formed.
6- Measure the absorbance of each solution at λ_{max} of 660 nm.
7- Prepare a blank solution containing all solutions above except phosphate ion using the steps above and measure its absorbance and subtracted its value from the actual absorbance value.
8- Treat the unknown exactly the same given in steps 1-6.
9- Draw a calibration graph between absorbance (A) and concentration of phosphate ion .
10- Calculate the concentration of phosphate ion in the unknown solution with the aid of the above calibration graph.

Experiment No. 3

Spectrophotometric Determination of Fe(III) Via The Formation of Charge Transfer Complex

Procedure

1- Add to a series of volumetric flask of (25 mL) capacity (1, 2, 3, 4 and 5 mL) of iron (III) (50 ppm).
2- To each of the above volumetric flasks including the unknown solution flask add (2 mL) of potassium ferrocyanide of (2×10^{-3} M); thus, soluble Prussian blue is formed.
3- Dilute the solution to the mark with distilled water then mix well and leave for (10 mins).
4- Measure the absorbance values at (710 nm).
5- Measure the absorbance of the formed Purssian blue from a solution containing (3 mL) of (50 ppm) iron (III) ten times for calculating the standard deviation and relative standard deviation percentage of the method.

Results and Calculations

1- Arrange and tabulate your results in a table include the concentration of iron (III) with ppm concentration versus absorbance.
2- Plot the standard curve for the determination of iron (III).
3- Allocate the concentration of the unknown from the calibration graph anf from the straight line equation.
4- Calculate the percentage of relative standard deviation (RSD%) and the percentage of the relative error and the percentage of recovery (R%) for iron (III)in step no. 5.

Experiment No. 4

Determination of PH and Ionization constant of an indicator (bromo thymol blue) B.T.B.

Procedure:

1- Prepare a standard solution of bromo thymol blue indicator of pH=1; by transferring 1mL of indicator solution (0.1% wt./vol.) to a volumetric flask (25 mL). Add 4 drpps of concentrated hydrochloric acid then complete to the mark with distilled water.

2- Record absorption values for the above solution at (390-500 nm) by graded increment of (10 nm) to determine the wavelength of maximum absorption.

3- In a volumetric flask of (25 mL) prepare standard solution of bromo thymol blue of different pH values according to the following table.

4- Measure the absorbance of solutions prepared in step 3 at wavelength of maximum absorption.

Indicator solution (0.1% wt/vol.)	Volume of $H_2PO_4^-$ (O.1 M)	Volume of HPO_4^{2-} (O.1 M)	pH
1 mL	5mL	0mL	4.5
1 mL	5mL	1mL	6.2
1 mL	1mL	5mL	7.5
1 mL	0mL	5mL	9.1

Results and Calculations

1- Arrange and tabulate your results in two tables the first one includes the wavelength versus absorption for the indicator solution in its acidic form; while the second include the pH values versus absorbance of the standard indicator solution.

2- Plot the pH-values against absorbance of the prepared solution of the indicator.

3- Find the pH of the indicator from the plot, which represent the practically obtained value.

4- Calculation the pH of the indicator theoretically from the equation below:

$$pH = pKa + log\frac{[ln^-]}{[Hln]}$$

$$K_a = 1.3 \times 10^{7-}$$

Experiment No. 5

Direct and indirect Spectrophotometric method for the Determination Chromate

$$Cr_2O_7^{2-} + 2OH^- \rightleftharpoons 2CrO_4^{2-} + H_2O$$

orange color yellow color

Procedure

1- Prepare series of solution of 0.5N-$K_2Cr_2O_7$ via pipetting variable volumes (0.1, 0.15,0.2,0.25, and 0.3mL) of the standard solution in a volumetric flask of (25 mL) capacity. Calculate the concentration of each prepared solution.

2- Add to the content of each flask fixed volume of (0.5 mL) of (0.5M-NaOH); complete the volume to the mark with distilled water.

3- Measure the absorbance for each flask at (420 nm).

4- Repeat step no.2 with the unknown solution.

5- Draw a curve between [CrO_4^{2-}] vs. absorbance to find out the concentration of the unknown. (**Direct Method**).

6- Prepare six volumetric flasks (25mL); add to each flask (1mL) of the unknown sample solution (unknown concentration of [$Cr_2O_7^{2-}$]).

7- Add (0.5 mL) of (0.5M-NaOH) solution to each flask.

8- Add averiable volume of (0, 0.1, 0.15, 0.2, 0.25, and 0.5 mL) of the standard solution of (0.5N-$K_2Cr_2O_7$), complete to the mark with distilled water and measure the absorbance at (420 nm).

9- Draw a relation between [CrO_4^{2-}] vs. absorbance(A) to find out the concentration of the unknown. (**Standard Addition Method**).