CHAPTER III

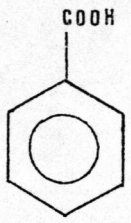
EXPERIMENTS

Equipment

1. Potentiograph E 536 (Metrohm Herisau)
2. Combined glass electrode AG CH-9101 (Metrohm Herisau)
3. 655 Multidosimat (Metrohm Herisau)
4. Automatic titrator and Exchange units model 3005(501)
5. Magnetic stirrer E649 (Metrohm Herisau)

Materials

1. Benzoic acid, AR (M&B, lot # 57518)



Empirical formular : $C_7H_6O_2$

Molecular weights : 122.12

Ka (28° c) : 7.871×10^{-5}

Description : monoclinic tablets, plates, leaflets

Solubility (Budavari, ed., 1989) : one gram dissolves in 2.3 ml ether, 3 ml acetone, 10 ml benzene, 30 ml carbon disulfide, 23 ml oil of terpentine, 250 ml water (25°c) and 230 ml water (30°c).

2. p- Nitrophenol, AR (Fluka Chemika, lot # 310855)

Empirical formular : $C_6H_5NO_3$

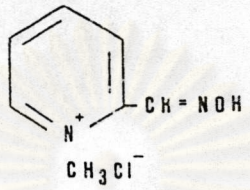
Mol. wt. : 139.11

Description : colorless to slightly yellow, odorless crystals

Ka (at 28°c) : 1.004×10^{-7}

Solubility (Budavari, ed.) : Moderately sol.in cold water, freely in alcohol, chloroform, ether, also sol.in solution of fixed alkali hydroxides and carbonates.

3. Pralidoxime chloride, STD (P.V.U-Hamburg, lot # 4711)



Empirical formular : $C_7H_9ClN_2O$

Mol. wt. : 172.63

Ka (at 28°c) : 1.240×10^{-8}

Solubility (Budavari, ed.) : one gram dissolves in 2 ml water, 12 ml methanol, 100 ml ethanol and 1000 ml isopropanol.

4. Boric acid, AR (E.Merck, lot # 5690020)

Empirical formular : H_3BO_3

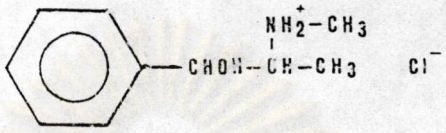
Mol. wt. : 61.84

Description : colorless, odorless, transparent crystals, or white granules or powder

Ka (at 28°c): 8.017×10^{-10}

Solubility (Budavari, ed.) : one gram dissolves in 18 ml cold water, 4 ml boiled water, 6 ml boiled alcohol and 4 ml glycerol.

5. Ephedrine hydrochloride, USP (RM 1059301)



Empirical formular : $C_{10}H_{15}NO.HCl$

Mol. wt. : 201.70

Description : orthorhombic needles, affected by light

Ka (at 28°c) : 2.153×10^{-13}

Solubility (Budavari, ed.) : one gram dissolves in 3 ml water, 14 ml alcohol, practically insoluble in ether and chloroform.

6. Potassium hydrogenphthalate, AR (Riedel-de Haen, lot # 90530)

$HOOC-C_6H_4-COOK$

Empirical formular : $C_8H_5KO_4$

Mol. wt. : 204.22

Description : orthorhombic crystals, stable in air

Ka (at 28°c) : 9.156×10^{-6}

Solubility (Budavari, ed.) : one gram dissolves in about 12 parts cold water and 3 parts boiling water.

Solvent and Other Reagents

1. Sodium hydroxide AR (E.Merck, lot#015c763598)
2. Potassium chloride AR (E.Merck, lot#208TA253636)
3. Distilled water
4. Potassium chloride solution, 3 M, electrolyte for combined glass electrode (Metrohm AG CH-9901 Herisau, lot # 62308020)
5. Standard buffer solution pH 4 (E.Merck, lot # 22370165)
6. Standard buffer solution pH 7 (E.Merck, lot # 22259244)
7. Standard buffer solution pH 10 (E.Merck, lot # 22328010)

Preparation of Sodium Hydroxide Standard Solution 0.1 N

Sodium hydroxide was dissolved in an equal weight of water and allowed to stand overnight. Taking precaution to avoid absorption of carbon dioxide, siphon off or decant the clear supernatant liquid and dilute 5.5 ml with carbon dioxide-free water to produce 1000 ml (Recommendation of The Medicine Commission, 1988).

Standardization of Sodium Hydroxide Solution

As certain it exact concentration immediately before use by titrating with it a solution of 0.3 g of potassium hydrogenphthalate, previously crushed lightly and dried at 120°c for 2 hrs, in 50 ml of carbon dioxide-free water, and titrate with the sodium hydroxide standard solution. Calculate the normality of solution which each ml of 0.1000 N sodium hydroxide is equivalent to 204.22 mg of potassium hydrogenphthalate (Recommendation of The Medicine Commission).

Preparation of Weak Acidic Compounds (0.005 M) in 0.10 M Potassium Chloride

Potassium chloride was weighed and dissolved in the distilled deionized water, and diluted with the same

solvent to give a final concentration about 0.10 M.

Each of weak acidic compound was accurately weighed in suitable quantities (according to its molecular weights) to produce about 0.01 M. They were dissolved in the solution of 0.10 M potassium chloride in order to control the ionic strength as the same through all step of the titration, warmed if necessary to effect solution. Pipette the weak acidic compound solution (~0.01 M in 0.10 M potassium chloride) 25.0 ml and 0.10 M potassium chloride solution 25.0 ml to a 100 ml beaker to produce about 0.005 M in the solution of 0.10 M potassium chloride.

Preparation of Two-Mixed Weak Acidic Compounds in 0.10 M Potassium Chloride

Transfer each 25.0 ml of two weak acidic compound solution which the concentration is about 0.01 M to a 100 ml beaker to produce about 0.005 M (each of weak acidic compounds) in the solution of 0.10 M Potassium chloride.

Titration of Weak Acidic Compounds and Two-Mixed Weak Acidic Compounds with 0.1 N Sodium Hydroxide

Fifty milliliters of the pipetted solutions were titrated with 0.1 N sodium hydroxide standard solution. The electrodes were submerged into titrated solution for 5 minutes prior to commencement of titration to assure that electrodes were in equilibrium with titrated solution. The beaker was placed on a magnetic stirrer and the combined glass electrode and a magnetic bar were inserted. The sample solution was mixed with a magnetic bar and magnetic stirrer after each addition of titrant and the pH value was measured after the stirrer off.

The precaution was not to rinse the sides of the beaker with distilled water because of the importance of knowing the exact solution volume at all times.

Determination of Equivalent Volumes

Volume of the titrant added and pH that measured for each of adding the titrant were the raw data which was gave from titration of the weak acid solution and the two mixed weak acid solution, example for the mixture of weak acid,HA and weak acid B, HB. The solution of each weak acid and the mixture were five-replicately titrated.

The Steps of Analyzing Data to determine the Equivalent Volumes

1. From the raw data, obtained from the titration of single weak acid solution, the equivalent volume could be determined by these methods; titration curve, the extrapolation of linear plot of Gran's method (G plot and V plot, according to Equation 9 and 21 for G plot and Equation 27 and 30 for V plot) and the modified equation (Equation 53 with only one variable in the equation). The simple linear regression analysis (Anderson, 1991 and Byrkit, 1987) was used in order to solve this equation.

2. From the raw datas, obtained from the titration of the two-mixed weak acids solution, they were applied to the modified equation, which was derived in the term of $Y = a_1X_1 + a_2X_2$ according to Equation 53. The multiple linear regression analysis and program SPSS/PC$^+$, were used in order to solve this equation and could determine the equivalent volume from the partial regression coefficient.

3. The equivalent volume, obtained from the modified equation for the titration of weak acidic mixture, were then compared with that obtained from G plot for the titration each of weak acid solution. To determine whether

there was a statistical difference between these results, the student t test was employed at 95% confidence interval.