

CHAPTER I

INTRODUCTION

1.1. Background

Volumetric analysis (titration) is one way to determine the content of a compound / element in a sample. In titration by acid-base neutralization reaction, usually the end point of titration is determined using an acid-base indicator, which is a substance that has a specific color on a range of specific pH so that when the equivalence point reached, there will arise discoloration.

Potentiometry is a method of chemical analysis based on the measurement of different potential cell of an electrochemical cell. In this way the indicator for determining the equivalence point in the neutralization titration can be seen from the potential change at each additional titrant volume.

1.2. Experiment Objectives

This experiment aims to determine the concentration of chloride acid in the sample based on the neutralization (acid-base) reaction with the potentiometric method.

1.3. Experiment Benefits

The benefits of this experiment are:

1. Students can determine the content of a compound in a sample by neutralization (acid-base) reaction with a potentiometric method.
2. Students can standardize secondary standard solutions.
3. Students can determine the equivalence point using the potentiometric method.
4. Students can calculate the concentration of a compound from the data of the end point of the titration that has been obtained.

CHAPTER II

LITERATURE REVIEW

2.1. Basic Theory

Potentiometry is a method of analysis based on the measurement of different potential cell of an electrochemical cell. The potentiometric method is used to determine the concentration of an ion (ion selective electrode), the pH of a solution, and determine the end point of the titration.

Tools required in the potentiometric method are:

- (1) Reference electrode
- (2) Indicator electrode
- (3) Potential Gauge

2.2. Reference Electrode

In some use of electrochemical analysis, required an electrode with a known half-cell potential, constant, and not sensitive to the composition of the solution that is being investigated. An electrode that meets the requirements is called a reference electrode.

Some examples of reference electrodes, (Underwood, AL.& Day, RA. 1990):

1. Calomel electrode, a reference electrode-shaped tubes, made of glass or plastic. Pasta Hg / HgCl contained in the tube, which is connected with the saturated solution of KCl through a very small hole. This electrode contacted with another half-cells solution through the insulator which is made of porcelain or porous asbestos.
2. Silver / silver chloride electrode, a reference electrode that is similar to the calomel electrode which consists of an silver electrode coated with silver chloride and dipped into the KCl solution.

2.3. Indicator Electrode

The reference electrode pair is an indicator electrode (also called a working electrode) whose potential depends on the concentration of the substance being investigated. Underwood, AL. & Day, RA. (1990), explained that indicator electrodes are divided into two categories, namely: metal electrodes and membrane electrodes.

2.3.1. Metal Electrodes

Metal electrodes can be classified into the first type electrode, the second type electrode, the third type electrode.

1. The first type of electrode is an electrode that is in direct balance with the cations from the metal. For example, copper electrode.
2. The second type of electrode is an electrode whose potential value depends on the concentration of an anion with ions coming from the electrode to form stable deposits of complex ions. For example, silver electrode for halide analysis.
3. The third type of electrode is a redox electrode. For example, Mercury_EDTA electrode. Precious metals such as platinum, gold, and palladium act as indicator electrodes in redox reaction.

2.3.2. Membrane Electrodes

A membrane allows certain types of ions to pass through, but blocks others. There are two types of membrane indicator electrodes, namely ion-selective and molecular-selective electrodes. Examples of membrane indicators: glass electrodes used to determine pH.

2.4. pH Meter

pH meter is an example of a membrane electrode application that is useful for measuring the pH of a solution. The pH meter can also be used to determine the end point of the acid-base titration for replacement indicators.

A potential is generated in a thin glass membrane which separates two solutions with different hydrogen ion activity. The potential generated depends on the difference in hydrogen ion activity on each side of the membrane and is not influenced by the presence of other ions in the solution. Glass electrodes are also selective towards ions other than hydrogen. This tool is equipped with a glass electrode and a calomel electrode (SCE) or a combination of them (combination electrode).

The thing that must be considered when using the electrode is that the liquid in the electrode must always be kept higher than the solution being measured. This is intended to prevent contamination of the electrode solution or blockage of the connector due to the reaction of the analyte ions with mercury (I) ion or silver ion.

2.5. Nernst Equation

Potentiometry is a method of determining the content of a substance by measuring its potential difference. The principle is based on the Nernst Equation.

$$E = E^0 + (RT/nF) \ln(a M^{n+})$$

E^0 = standard potential electrode that is constant with metal

R = gas constant

T = absolute temperature

F = faraday constant

n = valence of ion

aM^{n+} = ionic activity

Equation above simplified into:

$$E = E^0 + \frac{0,0001983}{n} \log(a M^{n+})$$

At temperature 25°C (298 K):

$$E = E^0 + \frac{0,0591}{n} \log(a M^{n+})$$

$a M^{n+}$ can be assumed as $c M^{n+}$ (concentration of ions is in molar)

2.6. Applications of Potentiometry in Industry

In the potentiometric method, information regarding the composition contained in the sample is obtained through the potential difference between the two electrodes. This method has been known since the 20th century and its use is becoming very widespread since last 25 years and has been used for some analytic applications developed using ion selective electrode (ISE) with more sensitive and stable electronic characteristics. Potentiometry used in industrial fields such as chloride analyst in the pulp and paper, analysis of NO_3^- , F^- , Br^- , Ca^{2+} in beverages, dairy, meat or fruit juice.

CHAPTER III

RESEARCH METHODOLOGY

3.1. Materials and Instruments

3.1.1. Materials

1. Oxalic Acid ($\text{H}_2\text{C}_2\text{O}_4$)
2. NaOH
3. HCl
4. Distilled water

3.1.2. Instruments

1. pH meter
2. Magnetic stirrer
3. Burette, stative, and clamp
4. Volumetric flask
5. Erlenmeyer
6. Beaker glass
7. Dropper pipette
8. Graduated cylinder

3.2. Experimental Methods

3.2.1. Standardization of NaOH Solutions

1. Make a NaOH solution at a certain concentration, NaOH with a certain weight is put into a glass beaker then add some water and stir it until the mixture completely dissolved. Pour the NaOH solution into a 250 ml volumetric flask, add some water to the mark, and stir it.
2. Calibrate the pH meter by using distilled water until the pH constant.
3. Pour 50 mL of NaOH solution to the beaker glass and dip magnetic bar to it, put the beaker glass at the top of magnetic stirrer, set the speed at scale of 5 .
4. Pour oxalic acid solution into the burette, place the tip of the burette into the beaker glass .
5. Dip the pH electrode to the solution. (Always do the calibration before the electrode will be dipped into the new solution)
6. Record the pH value on the pH meter .
7. Drop 2 mL oxalic acid from the burette, record the pH. The addition of oxalic acid and recording the pH continues until spike of pH occur and then stable again. Record in Table 3.1.

Tabel 3.1 Data Obtained from Standardization of NaOH Solution

Volume of oxalic acid (ml)	pH of the solution	$\Delta\text{pH} / \Delta V$	$\Delta^2\text{pH} / \Delta V^2$
0			
2			
4			
....			

8. Repeat the process numbers 2-6 with a new NaOH solution but do the addition of oxalic acid every 0,1 mL in the spike of pH area. In the other area, add oxalic acid every 2 mL. Record the pH.
9. Determine the volume of oxalic acid at the end point of the titration by making a pH curve as shown in Figure 3.1.
10. Calculate the content of NaOH based on the data obtained.

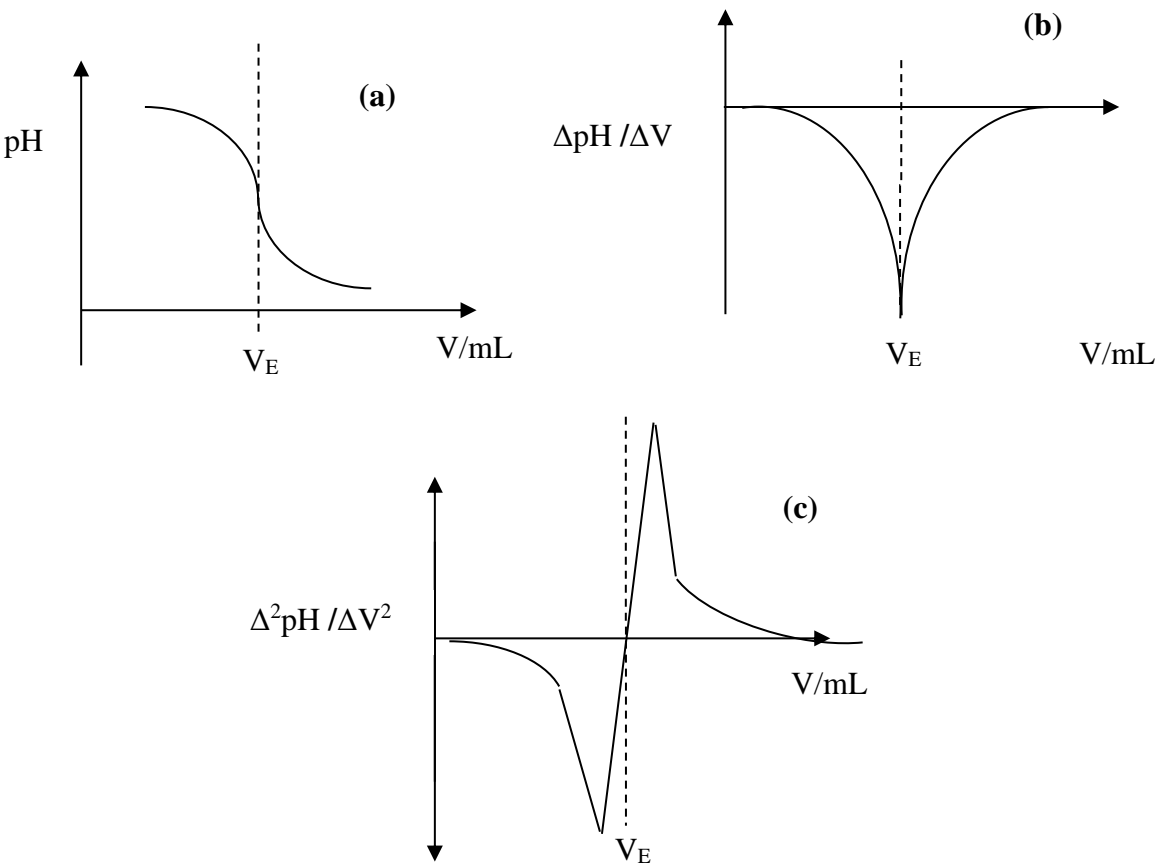


Figure 3.1 The Effect of Increasing Volume of Oxalic Acid on pH

(a) V_E vs ΔpH , (b) V_E vs $\Delta\text{pH}/\Delta V$, (c) V_E vs $\Delta^2\text{pH}/\Delta V^2$

3.2.2. Determination the Content of HCl

- 1. Calibrate the pH meter by using distilled water until the pH constant.
- 2. Pour 50 mL of HCl solution to the beaker glass and dip magnetic bar to it, put the beaker glass at the top of magnetic stirrer, set the speed at scale of 5.
- 3. Pour NaOH solution into the burette, place the tip of the burette into the Beaker glass.
- 4. Dip the pH electrode to the solution. (Always do the calibration before the electrode will be dipped into the new solution)
- 5. Record the pH value on the pH meter.
- 6. Drop 2 mL NaOH from the burette, record the pH. The addition of NaOH and recording the pH continues until spike of pH occur and then stable again. Record in Table 3.2.

Table 3.2 Data Obtained from Determination the content of HCl

Volume of NaOH (ml)	pH of the solution	$\Delta\text{pH} / \Delta V$	$\Delta^2\text{pH} / \Delta V^2$
0			
2			
4			
....			

- 7. Repeat the process numbers 1-5 with a new of HCl solution but do the addition of NaOH every 0,1 mL in the spike of pH area. In the other area, add NaOH every 2 mL. Record the pH.
- 8. Determine the volume of NaOH at the end point of the titration by making a pH curve as shown in Figure 1.
- 9. Calculate the content of HCl based on the data obtained.

BIBLIOGRAPHY

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