

CHAPTER I

INTRODUCTION

1.1. Background

Measurement of acids and bases is a very important environmental parameter in everyday life. Water, soil, waste and food materials such as fruits and vegetables contain both acidic and alkaline substances. The acid and base levels can be expressed in degrees of acidity (pH) or degrees of basicity (pOH). Analysis of acid and base concentrations in analytical chemistry can be carried out by acid-base titration. An acidic substance can be determined using a base as a titrant or vice versa, a substance that is alkaline can be determined using an acid as a titrant. This can be studied in acid-alkalimetry material.

Acidi-alkalimetry is a titration method based on the neutralization reaction between the titrant and the substance to be titrated. Acidimetry is the determination of the concentration of a base or an alkaline salt in a solution by using an acid solution of known concentration as a titrant. Meanwhile, alkalimetry is the determination of the level of acid or acidic salts in a solution using a known concentration of an alkaline solution as a titrant.

In this acidi-alkalimetry practicum, the practitioner determines the types of bases in the sample, such as NaOH, Na_2CO_3 , as well as NaOH and Na_2CO_3 . The type of base can be determined from the volume of titrant required in the volumetric titration. In addition, in this practicum, the acid content in the sample is also determined.

1.2. Experiment Objectives

1. Analyze the type of sample based on the volume of titrant found.
2. Determine the level of base in sample.
3. Determine the acid content in sample.

1.3. Experiment Benefits

This volumetric quantitative analysis experiment based on the neutralization reaction is useful for determining the level or concentration of a substance in the sample.

CHAPTER II

LITERATURE REVIEW

2.1 Definition

Titration is the volumetric determination of the content of a substance using other known solutions.

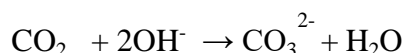
The reaction that occurs between acids and bases



Acidimetry is a titration method based on neutralization reactions between the titrant substance and the substance to be titrated.

Acidimetry: determination of the levels of bases or alkaline salts in a solution using an acidic solution that has a known concentration as a titrant.

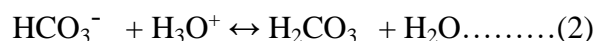
Sodium hydroxide is easily polluted to become sodium carbonate. This is because NaOH can absorb CO₂ in air and react as follows:



Often sodium carbonate and sodium bicarbonate are found together in a solution. This mixture of compounds can be found using the standard acid titration method

Carbonate Titration

The carbonate ion is titrated with a strong acid as the titrant, the reaction that occurs



$$\text{Ka}_1 = 4,6 \cdot 10^{-7} \quad \rightarrow \quad \text{pKa} = 6,34$$

$$\text{Ka}_2 = 4,4 \cdot 10^{-11} \quad \rightarrow \quad \text{pKa} = 10,36$$

PP is used as an indicator for the first reaction (first TAT) and MO is used as an indicator for the second reaction (second TAT).

Volume Relations in Carbonate Titration

In a solution of NaOH, Na₂CO₃, and NaHCO₃, they can exist as a single substance. But often together, for example, NaOH is mixed with Na₂CO₃ or NaHCO₃. This can be identified after the compound is titrated with HCl.

Table 2.1. Identification of the Bicarbonate Mixture

Substance	Relationships for qualitative identification	Millimoles of the substance present
NaOH	$y = 0$	$M \times x$
Na_2CO_3	$x = y$	$M \times x$
NaHCO_3	$x = 0$	$M \times y$
$\text{NaOH} + \text{Na}_2\text{CO}_3$	$x > y$	$M \times (x - y)$
$\text{NaHCO}_3 + \text{Na}_2\text{CO}_3$	$x < y$	$M \times (y - x)$

information:

M = molarity

X = volume needed to achieve TAT I using the PP indicator

y = volume needed to achieve TAT II using the MO indicator

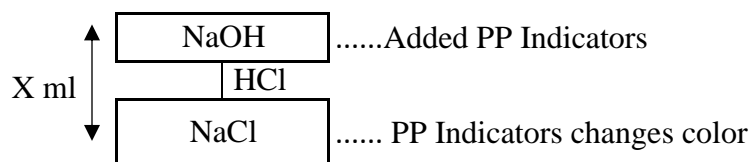


Figure 2.1 NaOH titration diagram

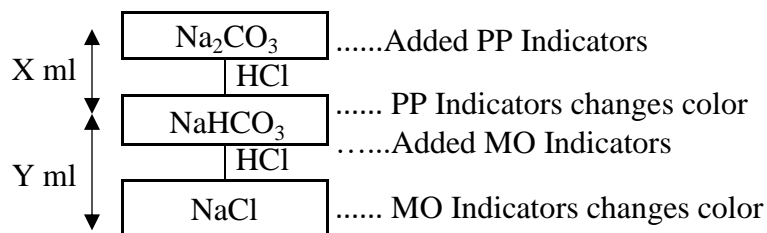


Figure 2.2 Na₂CO₃ titration diagram

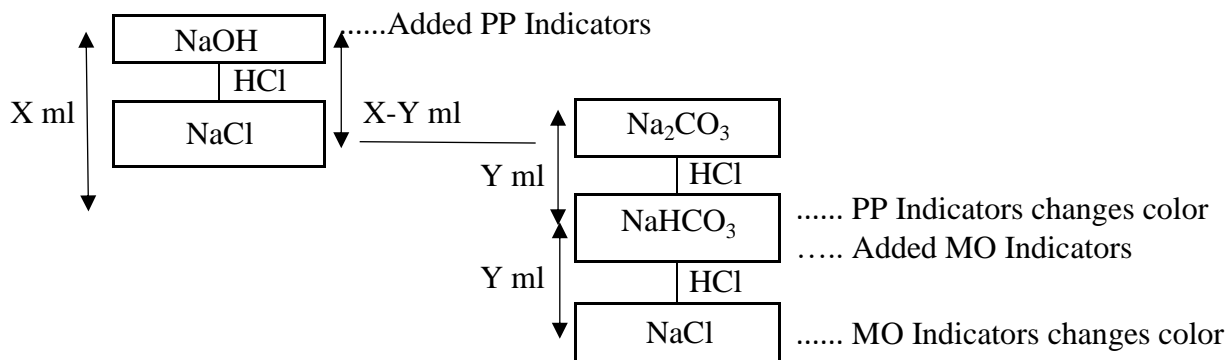


Figure 2.3 NaOH and Na₂CO₃ mixed titration diagram

Information :

| Titrated



Volume of titrant

Alkalimetry: determination of the level of acid or acidic salts in a solution using an alkaline solution whose concentration has been known as the titrant.

2.2 Indicator

According to Waty and Hasby (2020), acid-base indicators are complex compounds that can react with acids or bases accompanied by a color change. In addition, Indicators are also a substance used to determine when the end point of the titration (TAT) is reached by indicating a change in color. In acid-base analysis, the terms TAT and equivalent point (TE) are known, what is meant by TAT is a state when the indicator changes color and at that time the titration is stopped, while TE is a state where the number of moles of the equivalent of the substance titrated is the same as the number of moles of the equivalent of the titrant substance. When TAT is reached, it is expected that the number of equivalent moles of the titrated substance is equal to the equivalent mole of the titrant substance ($TAT = TE$). The indicators to be used in alkalimetric acid titration are:

a. BTB (Bromothymol Blue)

According to Islam et al. (2019), Bromothymol blue (BTB) at a very high pH can turn blue. This indicator is also known as dibromothymolsulfonephthalein which is a chemical indicator of weak acids and bases. This BTB indicator is known as an investigator that is very sensitive to changes in the structure and physical properties of proteins, phospholipids and biomembranes.

Table 2.2 pH Range of BTB Indicator

pH range	Color change
6,0 – 7,6	yellow - blue

The structure of the BTB indicators is as follows:

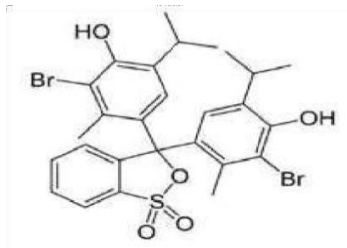


Figure 2.4 Schematic of the BTB indicator structure

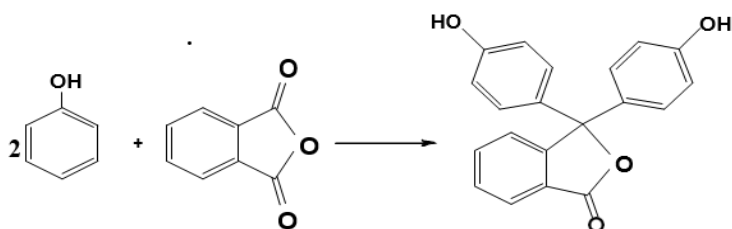
b. PP (Phenolphthalein)

According to Apriyani et al. (2016), Phenolphthalein indicator or PP indicator is one of the synthesis indicators used in strong base-strong acid titrations. Phenolphthalein 3,3bis(4-Hydroxyphenyl)phtalid has a characteristic odorless white or slightly yellow-white powder. PP can be synthesized by the condensation reaction of phenol and phtalic anhydride, as shown in Figure 2.2.

Table 2.3 pH Range of PP Indicator

pH range	Color change
8,0 – 9,6	Colorless - pink

The structure of PP indicators is as follows:



Phenol Phthalic anhydride Phenolphthalein

Figure 2.5 Schematic reaction for the formation of phenolphthalein

c. MO (Methyl Orange)

According to Madjid et al. (2015), Methyl orange is one of the anionic dyes containing azo groups. These dyes are widely used in dyeing processes and indicators in determining the end point of titrations. In an alkaline solution, methyl orange is yellow.

Table 2.4 pH Range of MO Indicator

pH range	Color change
3,1 – 4,4	Red – yellow

The structure of methyl orange is as follows :

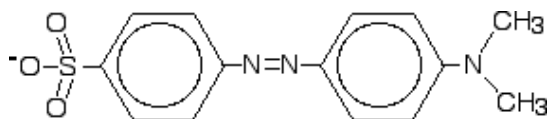


Figure 2.6 Structure of Methyl Orange (Yellow) in alkaline conditions

The hydrogen ion is attracted to one of the nitrogen ions in the nitrogen-nitrogen double bond to give a structure which can be written as follows :

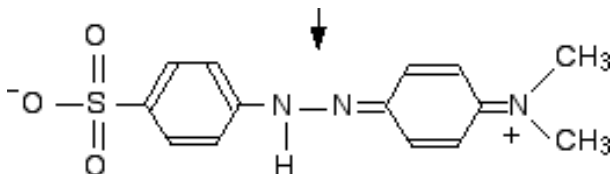


Figure 2.7 Structure of Methyl Orange (Red) in the acidic state

d. Methyl Red

Methyl Red is an indicator that changes color to red when it is in an acidic solution.

Methyl Red is an azo dye.

Table 2.5 pH Range of Methyl Red Indicator

pH range	Color change
4,4 – 6,2	Red – yellow

The structure of methyl red as follows:

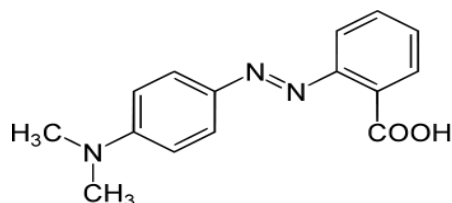


Figure 2.8. Methyl Red Structure

e. Thymol Blue

Thymol blue (thymolsulphonephthalein) is a green-brown or reddish-brown crystalline powder used as a pH indicator. It is insoluble in water but soluble in alcohol and dilute alkaline solutions.

Table 2.6 pH Range of Thymol Blue Indicator

pH range	Color change
1,2 – 2,8	Red – yellow
8,0 – 9,6	Yellow - blue

The structure of thymol blue is as follows:

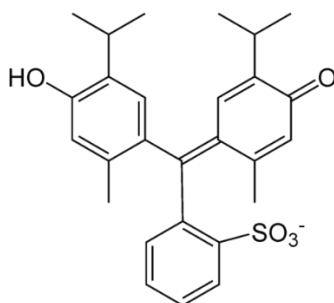


Figure 2.9. Thymol Blue Structure

f. Methyl yellow

Methyl yellow, or C.I. 11.020, is a chemical compound that can be used as a pH indicator. In aqueous solutions at low pH, methyl yellow is red.

Table 2.7 pH Range of Methyl Yellow Indicator

pH range	Color change
2,9 – 4,0	Red – yellow

The following is the structure of methyl yellow as follows:

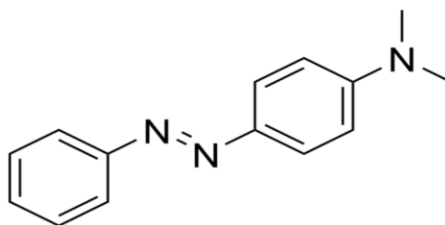


Figure 2.10. Methyl Yellow Structure

g. Methyl purple

Methyl purple is a derivative of pararosanillin, used as an anti-allergic and bactericidal, acid-base indicator, biological dye, and textile dye. Also known as crystal purple, gentian purple.

Table 2.8 pH Range of Methyl Purple Indicator

pH range	Color change
4,8 – 5,4	Purple – green

The structure of methyl purple as follows:

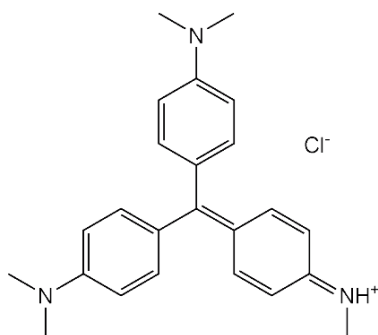


Figure 2.11. Methyl Purple Structure

h. Bromophenol Blue

Bromophenol Blue (Tetrabromophenolsulfonphthalein) is used as a pH indicator, color marker, and dye. It can be prepared by slowly adding excess bromine to a hot solution of phenolsulfonphthalein in glacial acetic acid.

Table 2.9 pH Range of Bromophenol Blue Indicator

pH range	Color change
3,0 – 4,6	Yellow – blue

The structure of bromophenol blue is as follows:

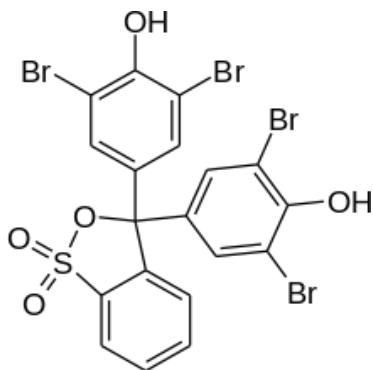


Figure 2.12. Bromophenol Blue Structure

i. Bromocresol Green

Bromocresol green (Tetrabromo-m-cresol sulfonphthalein) is a gray powder that is soluble in water or alcohol and is a dye from the triphenylmethane family (triarylmethane dyes). belongs to a class of dyes called sulfonephthaleins. used as a pH indicator in applications such as growth media for microorganisms and in titrations.

Table 2.10 pH Range of Bromocresol Green Indicator

pH range	Color change
3,8 – 5,4	Yellow – blue

The structure of bromocresol green is as follows :

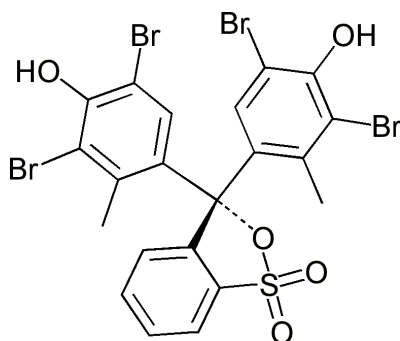


Figure 2.13 Bromocresol green structure

j. Alizarine Yellow R

Alizarine Yellow R is a yellow azo dye made by a diazo coupling reaction. It usually forms as a sodium salt. In pure form it is a rust colored solid. Its main use is as a pH indicator.

Table 2.11 pH Range of Alizarine Yellow R Indicator

pH range	Color change
10,1 – 12,1	Yellow – violet

The structure of alizarine yellow R is as follows:

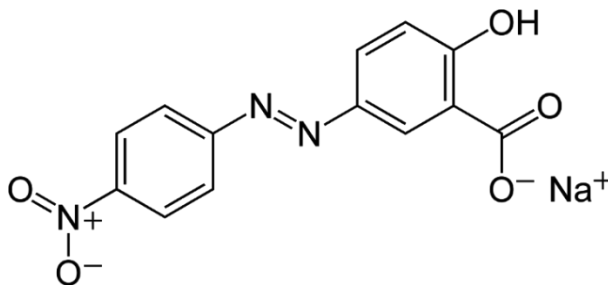


Figure 2.14 Alizarine Yellow R Structure

2.3 Titration Curve

Acid-base titration can be expressed in terms of the titration curve between pH (pOH) versus the titrant milliliter. Such a curve helps to consider the feasibility of a titration in choosing the right indicator. We will examine two cases, the titration of a strong acid with a strong base and a titration of a weak acid with a strong base.

a. Titration of Strong Acids and Strong Bases

Strong acids and strong bases do not hydrolyze completely in aqueous solutions. So the pH is the same at various points during the titration. It can be calculated directly

from the stoichiometric quantities of acids and bases that have been allowed to react. At the point of equality, the pH is determined by the extent to which water dissociates at 25 °C, the pH of pure water is 7.00.

b. Weak Acid and Strong Base

Titration On this titration curve, the curve for a weak acid starts to increase rapidly, when the base is added first. The rate of increase decreases with increasing B- concentration. This solution is said to be buffered in areas where the increase in pH is slow. Note that if the acid is neutralized $[HB^-] \approx [B^-]$

$$pH = pKa - \log \frac{[HB^-]}{[B^-]} \approx pKa$$

After the halfway point, the pH rises again slowly until a large change occurs at the point of equality

c. Titration of Strong Acids and Weak Bases

In this titration curve, the pH is first determined by the concentration of the acid (before the equivalence point). Then after reaching the equivalence point all acids react with bases. After the equivalence point in the solution there is an excess of weak bases and salt in the form of a buffer.

2.4 Application in Industry

a. In the pulping process

In the pulping process, a very important process is the bleaching process, where the purpose of bleaching is to remove the color from the lignin residue from the pulp to increase brightness, maintain brightness stability, cleanliness, and other undesirable properties, provided that it maintains the strength of cellulose. and carbohydrate regions in the pulp of unbleached fiber. In the bleaching process in pulp making, the bleaching process is stage extraction using NaOH to separate lignin from cellulose.

In this process, it is very important to pay attention to how much NaOH is used so that the bleaching process gets good results. According to Vania et al. (2022), the greater the concentration of NaOH, the more lignin will be degraded, so that the lignin content will decrease. While the pulp yield will be lower if the concentration of NaOH

is greater, because the concentration of NaOH which is too high will degrade cellulose. NaOH here is needed to separate lignin from cellulose and hemicellulose (Amelia, 2021).

b. Determination of sodium benzoate content in sweet soy sauce

According to Nurul et al. (2022), soy sauce is a food flavoring that tastes sweet and is a black liquid. Soybean is the basic ingredient for making soy sauce. In the production of soy sauce, preservatives such as sodium benzoate are added so that the soy sauce can last longer. Based on research conducted by Barita Aritonang and Yosy Silalahi in 2016, he used non-branded soy sauce samples that are commonly found in the market. The first step is to weigh the sample then add distilled water then shake and stir until evenly distributed. Next, a few drops of H₂SO₄ 4 N were added to an acidic condition. Then add ether then shake and let stand until the water layer separates from the ether. The next step, separating the ether (top) into the Erlenmeyer flask and filtration three times. Then the ether was collected in an Erlenmeyer flask, then heated over a water bath until dry (evaporation occurred). Then added neutral ethanol, then stirred until dissolved and added phenolphthalein indicator. Next, titrate with 0.1 N NaOH until the color changes to pink (Aritonang & Silalahi, 2016).

2.5 Physical and Chemical Properties of Reagents

a. NaOH

Physics :

MW = 40 g/mol

Density = 2,13 gr/cc

MP = 318,4C

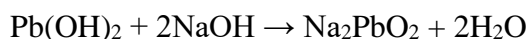
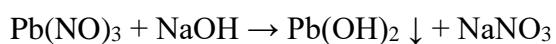
BP = 1390°C

Solubility in 100 parts of water 0°C = 82,3

Solubility in 100 parts of water 100°C = 56,3

Chemistry

- With Pb(NO₃) forms a precipitate of Pb(OH)₂ which dissolves in the excess reagent



- With $\text{Hg}_2(\text{NO}_3)_2$ forms a black precipitate of Hg_2O which dissolves in the excess reagent.
- It is a strong base.
- Easily soluble in water and hygroscopic.
- Easily absorbs CO_2 to form carbonates.

b. HCl

Physics :

MW = 36,47 g/mol

Density = 1,268 g/cc

MP = -110°C

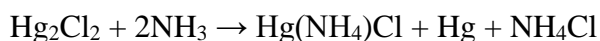
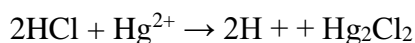
BP = 85°C

Solubility in 100 parts of water 0°C = 82,3

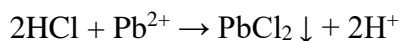
Solubility in 100 parts of water 100°C = 56,3

Chemistry

- Reacts with Hg^{2+} to form a white precipitate of Hg_2Cl_2 which is insoluble in hot water and dilute acid but dissolves in dilute ammonia, KCN solution and thioisulfate.



- Reacts with Pb^{2+} to form a white precipitate of PbCl_2



- It evaporates easily, especially when heated
- Concentration does not change easily due to air / light
- It is a strong acid because of its high degree of dissociation

c. Phenolphthalein ($\text{C}_{20}\text{H}_{14}\text{O}_4$)

Physics :

MW = 318,31 g/mol

Density = 1,299 gr/cc

BP = 261°C

pH = 8,0-9,6

Solubility in 100 parts of cold water $0,5^\circ\text{C}$ = 1,3

Chemistry

- It is a diprotic acid and is colorless.
- Initially dissociates into a colorless form and then loses H^+ to become ions in the conjugated system, resulting in a red color.

d. Methyl Orange

Physics :

Shape = Solid

Colour = Orange

pH = 3.1-4.4

BM = 327.34 gr/mol

TL = more than 300°C

Chemical:

- Chemically stable under standard room conditions (room temperature).
- Highly reactive with powerful oxidizers.

2.6 Concentration Unit

a. Molarity (M)

The molarity of a solution states the number of moles of a substance per liter of solution.

$$M = \frac{\text{mole of solute}}{\text{liter of solution}}$$

b. Molality (m)

Molality (m) represents the number of moles of the solute in 1 kg of solvent. Molality is independent of temperature, and is used in the field of physical chemistry, especially in its colligative nature.

$$\text{molality}(m) = \frac{\text{mole of solute}}{\text{kg of solvent}}$$

c. Normality (N)

Normality expresses the equivalent amount of solute in per liter of solution. The equivalent of a substance in solution depends on the type of reaction that the substance undergoes, because this unit is used to equalize the substance in the reaction.

$$\text{Normality (N)} = \frac{\text{equivalent of salute}}{\text{liter of solvent}}$$

$$\text{Normality (N)} = \text{Molarity} \times \text{valency}$$

d. Mole fraction (X)

A number that expresses the ratio of the number of moles of solute and solvent in a solution. In general, if there is a solution AB where A mole of solute and B mol of solvent, then the mole fraction A (X_A) is

$$X_A = \frac{\text{mole A}}{\text{mole A} + \text{mole B}}$$

The mole fraction of substance B (X_B) is

$$X_B = \frac{\text{mole B}}{\text{mole A} + \text{mole B}}$$

For the sum of both fractions

$$X_A + X_B = 1$$

2.7 Reagent Making

a. HCl 37% ($\rho = 1.19 \text{ gr/ml}$)

$$N = M \times \text{Valency}$$

$$M = \frac{10 \cdot \rho \cdot \%}{MW}$$

$$V1 \cdot M1 = V2 \cdot M2$$

b. NaOH (MW NaOH = 40 gr/mol)

$$N = M \times \text{Valency}$$

$$M = \frac{\text{gram}}{MW} \times \frac{1000}{ml}$$

CHAPTER III

PRACTICUM METHOD

3.1. Materials and Tools Used

3.1.1. Materials

1. Primary Standard Solution Na_2CO_3
2. NaOH
3. $\text{H}_2\text{C}_2\text{O}_4$
4. HCl
5. Phenolphthalein
6. Methyl Orange (MO)

3.1.2. Tools

1. Burette, statif, clamp
2. Erlenmeyer
3. Funnel
4. Volume pipette
5. Measuring pipette
6. Stirrer
7. Beaker glass
8. Dropper pipette
9. Measuring flask
10. Measuring cup

3.2. Practicum Procedure

3.3.1. Preparation of NaOH solution

1. Weigh the solid NaOH with an analytical balance according to the data on the LPR until the weight is constant.
2. Dissolving solid NaOH with distilled water in a 100 ml volumetric flask up to the mark.

3.3.2. Preparation of HCl solution

1. Take concentrated HCl in an acid room according to the data on the LPR.

2. Dilute concentrated HCl with distilled water in a 250 ml volumetric flask to the mark.

3.3.3. Standardization of NaOH with 0.1N H₂C₂O₄

1. 10 ml of 0.1 N oxalic acid was taken and put in an erlenmeyer.
2. Add a few drops of the PP indicator
3. Titrate with NaOH until the pink color is almost gone
4. Record the required volume of NaOH titrant (ml)
5. Calculate the concentration of NaOH in units of Normality with the following formula:

$$N_{NaOH} = \frac{(V \times N)_{H_2C_2O_4}}{V_{NaOH}}$$

Information :

N NaOH = Concentration of NaOH (N)

V NaOH = Volume of NaOH (ml)

N H₂C₂O₄ = Concentration of H₂C₂O₄ (N)

V H₂C₂O₄ = Volume H₂C₂O₄ (ml)

3.3.4. Standardization of HCl with standardized NaOH

1. 10 ml of HCl is taken and put into Erlenmeyer
2. Add a few drops of the PP indicator
3. Titrate with NaOH until the pink color is almost gone
4. Record the required volume of NaOH titrant (ml)
5. Calculate the concentration of HCl in units of Normality using the following formula:

$$N_{HCl} = \frac{(V \times N)_{NaOH}}{V_{HCl}}$$

Information :

N NaOH = Concentration of NaOH (N)

V NaOH = Volume of NaOH (ml)

N HCl = Concentration of HCl (N)

V HCl = Volume of HCl (ml)

3.3.5. Looking for NaOH levels

1. 10 ml of sample solution is taken and put into Erlenmeyer.
2. Add a few drops of the PP indicator
3. Titrate with HCl until the red color is almost gone (pink).
4. Record the need for HCl at TAT = x ml
5. Add a few drops of the MO indicator, the sample will change color to orange red
6. Calculate the NaOH content in the sample using the following formula:

$$\text{Level of NaOH} = (x) \times N \text{ HCl} \times MW \text{ NaOH} \times \frac{1000}{10} \text{ ppm}$$

Information :

NaOH content = calculated in ppm

X = volume of HCl titrant required when adding PP indicator
(ml)

N HCl = Concentration of HCl

BM NaOH = Molecular Weight of NaOH

3.3.6. Looking for levels of Na₂CO₃

1. 10 ml of sample solution is taken and put into Erlenmeyer.
2. Add a few drops of the PP indicator
3. Titrate with HCl until the red color is almost gone (pink).
4. Record the need for HCl at TAT 1 = x ml
5. Add a few drops of the MO indicator
6. Titrate with HCl until the color turns orange red.
7. Record the need for HCl for Na₂CO₃ = y ml
8. Calculate the Na₂CO₃ content in the sample using the following formula :

$$\text{Level of Na}_2\text{CO}_3 = (x + y) \times N \text{ HCl} \times \frac{MW \text{ Na}_2\text{CO}_3}{2} \times \frac{1000}{10} \text{ ppm}$$

Information :

Na₂CO₃ content = calculated in units of ppm

X = volume of HCl titrant required when adding PP indicator
(ml)

Y = volume of HCl titrant required when adding MO
indicator (ml)

N HCl = Concentration of HCl
 BM Na₂CO₃ = Molecular Weight of Na₂CO₃

3.3.7. Looking for levels of Na₂CO₃ and NaOH

1. 10 ml of sample solution is taken and put into Erlenmeyer.
2. Add a few drops of the PP indicator
3. Titrate with HCl until the red color is almost gone (pink).
4. Record the need for HCl at TAT 1 = x ml
5. Add a few drops of the MO indicator
6. Titrate with HCl until the color turns orange red.
7. Record the need for HCl for Na₂CO₃ = y ml
8. Calculate the levels of NaOH and levels of Na₂CO₃ with the following formula:

$$\text{Level of NaOH} = (x - y) \times N \text{ HCl} \times MW \text{ NaOH} \times \frac{1000}{10} \text{ ppm}$$

$$\text{Level of Na}_2\text{CO}_3 = (2y) \times N \text{ HCl} \times \frac{MW \text{ Na}_2\text{CO}_3}{2} \times \frac{1000}{10} \text{ ppm}$$

Information :

NaOH content = calculated in ppm

X = volume of HCl titrant required when adding PP indicator (ml)

Y = volume of HCl titrant required when adding MO indicator (ml)

N HCl = Concentration of HCl

BM NaOH = Molecular Weight of NaOH

BM Na₂CO₃ = Molecular Weight of Na₂CO₃

3.3.8. Find the acid level of the sample

1. Take 10 ml of sample, dilute to 100 ml of aquadest
2. Take 10 ml of the sample solution, put it in the Erlenmeyer.
3. Add a few drops of PP indicator (± 3 drops)
4. Titrate with NaOH until it is pink
5. Record the required volume of NaOH titrant (ml)

6. Calculate the acid concentration of the sample in units of Normality by using the following formula:

$$N_{Acid} = \frac{(V \times N)_{NaOH}}{V_{sample}} \times \text{dilution factor}$$

Information :

N_{Acid} = Acid concentration (N)

V_{NaOH} = Volume of NaOH (ml)

N_{NaOH} = Concentration of NaOH (N)

V_{sample} = Sample volume (ml)

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