EXPERIMENTAL DESIGN FOR ACID CONTENT DETERMINATION IN ORGANIC ACID

KUMARAGURU A/L GUNASEGARAN

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by

Kumaraguru A/L Gunasegaran

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Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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(Dr. Shuhaimi Mahadzir)

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TRONOH, PERAK

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Kumaraguru

KUMARAGURU A/L GUNASEGARAN

ABSTRACT

My research is on acid content determination in the organic acid. The primary objective of the project is to design and develop the experimental procedures to determine the acid content of organic acid. Before proceeding with the experimental design, I will utilize the existing methods of determining the acidity of organic acid. The already available methods are using the pH probe and also performing the Total Acid Number (TAN) measurement. For the TAN measurement, specifically the Potentiometric Titration will be utilized to evaluate the acid content of the acid sample.

The second main task will be the identification of the indirect measurement parameters of the organic acid. This is to correlate those parameters' values with the acidity of the organic acid.

The main experiment, which is Potentiometric Titration by using the auto-titrator, already been conducted. Several runs of experiments were preformed and the results were analyzed in order to achieve the primary objective of this project. The other indirect measurement parameters, were identified upon thorough research on the properties of the organic acid (acetic acid) and the relevant experiments were conducted sequentially, after the Potentiometric Titration experiment.

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CHAPTER 1
INTRODUCTION

1.1 Background

Organic acid's acidity determination has to be designed with proper experimental procedures. It is an organic source, and it has slightly lower acidity compared to the inorganic acids. The instantaneous method of measuring the acidity of a liquid source is by inserting the pH probe into the sample, and read the measured value in pH meter. The examples of organic acids are acetic acid, formic acid and bio-oil. Bio-oil consists of both formic acid and acetic acid, and since it has a pH range between 1.8 and 3.0, thus it is known as an organic acid. With plenty of methods are available for the acid content determination, nothing has not been specific for organic acids. This project is primarily concentrating on the experimental design of acid content measurement for organic acid. Apart from it, some other parameters of the organic acid will be investigated to correlate it with the acid content.

1.2 Problem Statement

At the moment, there is no specific acid content determination method for organic acids. The only acidity measuring method is by using the pH probe. The other acid content determination methods such as Total Acid Number (TAN), Nitrogen Content and Chloride Content have not been applied for organic acids. The reason being the contamination of the organic acids by char content and other impurities. The chosen organic acid was Acetic acid. The acetic acid's acidity level will be tested by applying the Total Acid Number (TAN) method. To proceed further with the acidity measurement of acetic acid, the availability of equipments in UTP's chemical laboratory has to be taken into consideration.

1.3 Objectives

- To develop experimental designs of acidity determination in organic acid.
- To identify the correlation between several parameters of the acetic acid and its acidity.
- To evaluate the effectiveness of the available methods of acid content determination with the new experimental designs.

1.4 Scope of Study

This project if fully experimental basis. It starts from a very basic understanding of what is all about an organic acid. Specifically, to dig and research further on the acidity level of this organic source. Firstly, few of the existing methods of determining the acidity of petroleum products will be tested to identify the most effective and accurate method. Based on the evaluation of these methods, research will be further intensified in order to design a new experiment to determine the acidity level of the acetic acid. At the same time, several parameters of the acetic acid will be identified in order to determine the correlation between those parameters and its acidity.

CHAPTER 2
LITERATURE REVIEW AND THEORY

2.1 Acetic acid

Acetic acid or Ethanoic acid, CH_3CO_2H, is a colorless liquid that has a characteristic pungent odor, boils at 118 degrees Celsius, and is miscible with water in all proportions. It is a weak organic carboxylic acid. Glacial acetic acid is concentrated, 99.5% pure acetic acid. It solidifies at about 17 degrees Celsius to a crystalline mass resembling ice. In a dilute form, mixed with water, it is the acid found in vinegar. Vinegar contains 5% or more ethanoic acid, produced by fermentation (*The Columbia Encyclopedia, 2004*). Thus, it is widely used as a food preservative and condiment. For industrial use concentrated acetic acid is prepared from the oxidation of acetaldehyde. Acetic acid is also a product in the destructive distillation of wood. It reacts with other chemicals to form numerous compounds of commercial importance. These include cellulose acetate, used in making acetate rayon, nonflammable motion-picture film, lacquers, and plastics. There are also various inorganic salts such as lead, potassium, and copper acetates. Meanwhile, amyl, butyl, ethyl, methyl, and propyl acetates are used as solvents, chiefly in certain quick-drying lacquers and cements. Amyl acetate is sometimes called banana oil because it has a characteristic banana odor. Acetic acid's acidity is different from the inorganic acid. The hydrogen (H) atom in the carboxyl group (−COOH) in carboxylic acids such as acetic acid can be given off as an H^+ ion proton, giving them their acidic character. Acetic acid is a weak, effectively monoprotic acid in aqueous solution, with a pK_a value of 4.75. Its conjugate base is acetate (CH_3COO^-). A 1.0 M solution (about the concentration of domestic vinegar) has a pH of 2.4, indicating that merely 0.4% of the acetic acid molecules are dissociated (*G. K. Jayaprakasha and K. K. Sakariah, 1998*).

The dissociation reaction can be illustrated as below:

Figure 1: Dissociation of Acetic acid with the aid of water

Table 1: Various concentration of acetic acid and the corresponding pH values

[Acetic acid]	pH
1.0 M	2.4
0.1 M	2.8-2.9
0.01 M	3.4

The crystal structure of acetic acid (in **figure 1**) shows that the molecules pair up into dimers connected by hydrogen bonds. The dimers can also be detected in the vapour at 120 °C. They also occur in the liquid phase in dilute solutions in non-hydrogen-bonding solvents, and to a certain extent in pure acetic acid, but are disrupted by hydrogen-bonding solvents.

Weak acids have small K_a values. The dissociation of this organic acid is only partial in aqueous phase, and thus only modest levels of H_3O^+ are present in the solution. An acid in solution sets up this equilibrium:

$$AH + H_2O \rightleftarrows A^- + H_3O^+$$

A hydroxonium ion is formed together with the anion from the acid. This equilibrium can be simplified by leaving out the water to emphasis the ionization of the acid:

$$AH(aq) \rightleftarrows A^-(aq) + H^+(aq)$$

Writing $H^+(aq)$ implies that the hydrogen ion is attached to a water molecule as H_3O^+. Hydrogen ions are always attached to something during chemical reactions.

Organic acids are weak in the sense that its ionization is very incomplete where the dissociation is about 20%, compared to the dissociation of strong acids at 100%.
At any one time, most of the acid will be present in the solution as un-ionised molecules. For example, in the case of dilute ethanoic acid, the solution contains about 99% of ethanoic acid molecules - at any instant, only about 1% have actually ionised. The position of equilibrium therefore lies well to the left.

The strengths of weak acids are measured on the pKa scale. The smaller the number on this scale, the stronger the acid is. The larger the value of pKa, the weaker is the acid. pKa can be calculated based on the formula illustrated below:

$$\boxed{pKa = -\log [Ka]}$$

where Ka is known as the acidity constant. The Ka value can be calculated from the acid's dissociation or ionization equation at equilibrium.

$$Ka = \frac{[H^+][A^-]}{[AH]}$$

Based on the formulas illustrated above for both pKa and Ka, it can be interpreted that:

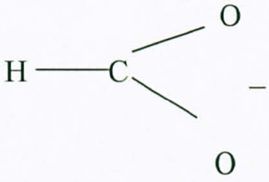
- When the ionization or dissociation of the acid is 100%, the concentration of H^+ will be higher, thus the acidity constant, Ka will be higher. This results in the pKa value to be low, indicating that the acid is stronger.
- When the ionization or dissociation of the acid is incomplete (~20%), the concentration of H^+ will be lower, thus the acidity constant, Ka will be lower. This results in the pKa value to be higher, indicating that the acid is weaker.

  The typical pKa values pf the organic acids are given below:

Table 2: The list of weak organic acids and their respective pKa values

Acid	pKa
$HCOOH$	3.75
CH_3COOH	4.75
C_2H_5COOH	4.87
C_3H_7COOH	4.82

The methanoic acid is stronger than the ethanoic acid. It depends on the stability of the anions formed – on how much it is possible to delocalize the negative charge. The less the charge is delocalized, the less stable the ion and the weaker the acid. The methanoate ion (from methanoic acid) is:



The only difference between this and the ethanoate ion is the presence of the CH_3 group in the ethanoate. But that is important. Alkyl groups have a tendency to "push" electrons away from themselves. That means that there will be a small amount of extra negative charge built up on the $-COO^-$ group. Any build-up of charge will make the ion less stable, and more attractive to hydrogen ions (*science and engineering encyclopedia, 1999*). Ethanoic acid is therefore weaker than methanoic acid, because it will re-form more easily from its ions.

2.2 Acid content measurement

2.2.1 Total Acid number (TAN)

The Total Acid Number (TAN) is the quantity of potassium hydroxide in milligrams that is required to neutralize the acids in one gram of oil. The specific unit for TAN is mg KOH/g oil. It is a crucial quality measurement of crude oil. The TAN value indicates to the crude oil refinery the potential of corrosion problems. It is usually the naphthenic acids in the crude oil that causes corrosion problems. This type of corrosion is referred to as naphthenic acid corrosion. In my research, the TAN method will be applied to Acetic acid. The method has not been tested for organic acids. The TAN value can be deduced by a couple of different methods such as color indicator titration and potentiometric titration. The potentiometric titration method is applicable and suitable for both organic acids such as formic acid, acetic acid and bio-oil and also inorganic acids such as hydrochloric acid, nitric acid and sulphuric acid. The TAN will indicate directly on the acidity level of each acid. When the sample is highly acidic, the TAN will be higher and the reverse is observed if the acidity is lower (*Neto, J.M., 1996*). The TAN have not been tried for organic acids, and the method will be tested on one particular organic acid to obtain the desired results.

2.2.2 Density of Acetic acid

One of the physical properties of acetic acid that has been taken into research is its density. The typical value for the density of acetic acid is 1.0496 g/cm^3.at 25°C. The density varies accordingly with the temperature change. The acetic acid's phase of liquid verifies that, when the concentration of acetic acid is decreased, the density will decrease. The reverse result is obtained if the concentration is high (*The Columbia Encyclopedia, 2004*). The density of acetic acid will vary accordingly to the temperature. The liquid phase of acetic acid indicates that, if the acetic acid is heated up, the lesser will be the resultant density. It will be discussed in detail in discussion part.

2.2.3 Viscosity of Acetic acid

Viscosity is a measure of the resistance of a fluid which is being deformed by either shear stress or extensional stress. Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction. The typical viscosity value of acetic acid is 1.22MPa.s at 20°C. The acetic acid's viscosity is directly proportional to its concentration. When the concentration is high, the viscosity will be at a higher value as well. Meanwhile, the reverse relation is identified between the viscosity and temperature. As the acetic acid's temperature is increased through heating, the viscosity will be decreasing gradually (*Bleazard, J. G.; Sun, T. F.; Teja, A. S., 1996*). The significant changes can only be verified via the experimental results.

2.2.4 Refractive Index of Acetic acid

Refractive Index is a measure of the bending of a ray of light when passing from one medium into another. If **i** is the angle of incidence of a ray in vacuum (angle between the incoming ray and the perpendicular to the surface of a medium, called normal) and **r** is the angle of refraction (angle between the ray in the medium and the normal), the refractive index, **n** is defined as the ratio of the sine of the angle of incidence to the sine of the angle of refraction. Thus, $n = \sin i / \sin r$. Refractive index is also equal to the velocity of light, c of a given wavelength in empty space divided by its velocity, v in a substance. Thus, $n = c / v$. The typical refractive index value of Acetic acid is between 1.37100 and 1.37500 at 20°C. The relationship between refractive index and temperature is indirect proportion, and the relationship between the refractive index and concentration of a liquid substance is direct proportion (*Encyclopedia Britannica, 2002)*.

2.2.5 Energy content measurement

The acidity measurement of acetic acid can also be done by measuring the energy content of it, specifically by measuring the molar enthalpy change of neutralization. The base that will be used is Potassium Hydroxide, dissolved in methanol, ethanol or propanol. The energy content measurement will be carried out by using different concentration of acetic acid as the quantity of base required to neutralize the acid sample will be differing too. Since the neutralization reaction is exothermic mostly, thus the final temperature of the neutral solution will be lower than that of the initial reading. If it is an endothermic reaction, thus the final temperature of the neutral solution will be higher than the initial temperature reading. If the acid is concentrated, the neutralization reaction will be slower compared to the use of diluted acid for neutralization reaction (*Heller, Gregory A, 1998*).

CHAPTER 3

METHODOLOGY

3.1 Project activities

3.1.1 Tasks completed

- Thorough research on the topic. Currently, I am reading through all the available online articles and journals on the acid content measuring methods for Acetic acid.
- Visited the UTP laboratory and sought the lab assistants' help on how the acid content measurement is conducted in laboratory.
- Consulted and sought advice from the supervisor in order to get thorough understanding on what are the tasks to be done at the moment and also in near future.
- Preparation of standard samples. 7 samples have been prepared with the pH ranging from 1 to 7.
- Several runs of Potentiometric Titration.
- Viscosity measurement for Acetic acid at different concentration and temperature.
- Density measurement for Acetic acid at different concentration and temperature.
- Energy content measurement for Acetic acid.

3.2 Acid Content measurement

3.2.1 Potentiometric Titration

This test method expresses the quantity of base as milligrams of potassium hydroxide (KOH) per gram of sample (Acetic acid), that is required to titrate a sample in a mixture of toluene and propan-2-ol to which a small amount of water has been added from its initial meter reading in milivolts to a meter reading in milivolts corresponding to an aqueous basic buffer solution or a well-defined inflection point as specified in the test method. The causes and effects of the so-called strong acids and the causes and effects containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide using a glass indicating electrode and a reference electrode or a combination electrode.

- The meter readings are plotted manually or automatically against the respective volumes of titrating solution and the endpoints are taken only at well-defined inflections in the resulting curve.
- When no definite inflections are obtained and for used Acetic acid, endpoints are taken at meter readings corresponding to those found for aqueous acidic buffer solutions.

3.3 Chemicals required

- **Acetic acid, Potassium Hydroxide, Toluene, Methanol, Ethanol Propan-2-ol.**

3.4 Tools and equipments required:

- **variable speed mechanical stirrer** – a suitable type, equipped with a propeller-type stirring paddle. The rate of stirring shall be sufficient to produce vigorous agitation without spattering and without stirring air into the solution. A propeller with blades 6 mm in radius and set at a pitch of 30 to 45° is satisfactory. A magnetic stirrer is also satisfactory.
- **sensing electrode** - standard pH, suitable for non-aqueous titration.
- **reference electrode** - Ag/AgCl reference electrode, filled with 1M-3M LiCl in ethanol.
- **combination electrodes** – sensing electrodes may have the Ag/AgCl reference electrode built into the same electrode body, which offers the convenience of working and maintaining only one electrode. The combination electrode shall have a sleeve junction on the reference compartment and shall use an inert ethanol electrolyte, for example 1M-3M LiCl in ethanol. These combination electrodes shall have the same response or better response than a dual electrode system. They shall have removable sleeves for easy rinsing and addition of electrolyte.
- **voltmeter** – that operates with an accuracy of ± 0.005 V and a sensitivity of

± 0.002 V over a range of at least ± 0.5 V when the meter is used with the electrodes specified above (sensing electrode and reference electrode stated above) and when the resistance between the electrodes falls within the range from 0.2 to 20 MΩ. The meter shall be protected from stray electrostatics fields so that no permanent change in the meter readings over the entire operating range is produced by touching, with a grounded lead, any part of the exposed surface of the glass electrode, the glass electrode lead, the titration stand or the meter.

- **burette**
- **pipette**
- **titration beaker and titration stand**
- **stop watch**
- **thermometer**
- Personal protective equipments (PPEs) required: goggles, laboratory coat and face shield (optional but recommended).

3.5 Experiments

3.5.1 Experiment 1: Dilution of Hydrochloric Acid using cold tap water

- Objective: To prepare standard samples of pH 1 to pH 7.
- Apparatus required: beaker, pH meter and conical flask.
- Chemicals required: Hydrochloric acid
- Procedures:

1. A sample of Hydrchloric acid of concentration 0.1M was to be prepared. The theoretical pH of this sample will be 1.0.
2. The available HCl acid in laboratory was of concentration 1 M. It can be diluted to achieve a concentration of 0.1M.
3. 25 mL of HCl acid of concentration 1M was diluted with 250 mL of cold tap water. The ratio of acid to water is 1:10. The concentration was expected to reduce to 0.1M, and the theoretical pH of the sample was expected to be 1.0. At the same time, the actual pH of the sample was measured using the pH meter. It was done by placing the probe in the sample, and record the stabilized pH reading which was displayed.

4. Secondly, 25 mL of the diluted acid was collected in a beaker. It was further diluted with 250 mL of cold tap water. The concentration of the acid was expected to reduce further to 0.01M and its pH at 2.0. The actual pH of the sample was measured using the pH meter and recorded.
5. A factor of ten dilution of a strong acid changes the [H^+ (aq)] by a factor of 10, and thus the pH by one unit.
6. The dilution of strong acid will result in the pH to increases towards the neutral level.
7. The above steps were repeated to achieve samples of pH 3, 4, 5, 6 and 7.
8. The acid was always added to the cold tap water, not the reverse way.
9. A graph of actual pH values against the theoretical pH values was plotted.

3.5.2 Experiment 2: Preparation of alcoholic Potassium Hydroxide solution

- Objective: To prepare an alcoholic Potassium Hydroxide solution of concentration 0.1 M.
- Apparatus required: conical flask and weighing machine.
- Chemicals required: Potassium Hydroxide and Methanol.
- Procedure:

1. 5.61 g of Potassium Hydroxide was measured using the weighing machine.
2. 1000 mL of Methanol was poured into a conical flask.
3. The potassium hydroxide was then added to the 1000 mL of Methanol.
4. The mixture was boiled gently for 10 minutes to affect the solution. Hot plate was used for the boiling process.
5. The solution was allowed to stand for two days.
6. After two days, the supernatant liquid was filtered through a fine sintered – glass funnel.
7. The solution was then transferred into a conical flask, covered with parafilm and kept in fume chamber.

- Concentration of KOH (mol/dm^3), [KOH] = n / V

  where n = no.of moles = mass of KOH (in gram)/molecular weight of KOH (in g/mol), V = volume of alcohol required to dissolve the solid KOH (in dm^3).

- For instance, an alcoholic KOH solution with a concentration of 0.1 M, is equivalent to 0.1 mol/ dm^3.
- Thus, 0.1 mol/ dm^3 = (x / 56.11 g/mol) / (1.0 dm^3) where x = mass of KOH required to prepare a KOH solution of concentration 0.1 M.
  By solving for x, the required mass of KOH = 5.611 g.
- From the above formula for the concentration of KOH:
  a) [KOH] is directly proportional to the mass of KOH.
  b) [KOH] is inversely proportional to the volume of alcohol, in our experiment the alcohol used was Methanol.
- Therefore,
  a) 1M of KOH = 5.611 g of KOH dissolved in 100 mL (0.1 dm^3) of Methanol.
  b) 2M of KOH = 11.222 g of KOH dissolved in 100 mL (0.1 dm^3) of Methanol or
  5.611 g of KOH dissolved in 50 mL (0.05 dm^3) of Methanol.
  c) 0.1M of KOH = 0.5611 g of KOH dissolved in 100 mL (0.1 dm^3) of Methanol
  or
  5.611 g of KOH dissolved in 1000 mL (1 dm^3) of Methanol.
- For further runs of experiments, Ethanol and Propan-2-ol were used to dissolve Potassium Hydroxide.
- The same calculations above can be applied to prepare a NaOH solution of various concentrations.

3.6 Potentiometric Titration

Before proceeding with the Potentiometric titration, there are some important pre-tasks to be done in order for the experiment to be run smoothly. The tasks are as follows:

3.6.1 Preparation of Electrodes:

For the auto-titrator system, the combination electrodes are used. It has to be calibrated before running the experiment. The procedures are as follows:

- Firstly, 50 mL of pH 4.01 buffer solution was added to a titration beaker and fastened to the titration head.

- The titrator was run.
- The stir function was executed. The solution was stirred for 60 seconds to condition the electrode. The elapsed time was displayed.
- Then, the measure function was executed. The changing potential value of the buffer solution was displayed. As soon as the measured value was stable, it was acquired.
- The measured potential of the first buffer solution was displayed as the result.
- The titration beaker was removed.
- The electrode and the stirrer were rinsed with deionized water.
- Secondly, 50 mL of pH 7.00 buffer solution was added to the titration beaker and fastened to the titration head.
- The stir function reappeared, followed by the measure function. As soon as the measured value became stable, it was acquired.
- In addition to the result of the first measurement, the measured potential of the second buffer solution was displayed as the result.
- The procedures above were repeated for the final buffer solution of pH 10.00, but in the laboratory, the pH of the buffer was 9.21. Thus, there was a bit of change in the potential value from the desired reading.

3.6.2 Modification of the standard method

- Since Bio-oil was not available temporarily, the acetic acid was used as the alternative since it is an organic source. For the first trial, acetic acid was used purely without any addition of toluene and propan-2-ol into it.
- The standard method of auto-titrator was set for Hydrochloric acid. Thus, it was altered to acetic acid.
- The molar mass of acetic acid was inserted, replacing the molar mass of HCl acid previously.
- The titrant solvent used was alcoholic Potassium Hydroxide solution. The auto-titrator's default titrant of Sodium Hydroxide was altered to Potassium Hydroxide.
- The other functions such as measuring unit and stirring speed can also be adjusted.

3.6.3 Experiment 3: The Total Acid Number (TAN) measurement

- Objective: To measure the Total Acid Number (TAN) of Acetic acid.
- Apparatus: Auto-titrator
- Chemicals required: Acetic acid, Deionized water, Methanolic Potassium Hydroxide solution.
- Procedure:

1. The piston of the burette was moved upward and the air expelled. An initial small amount of methanolic Potassium Hydroxide solution was siphoned in when the piston returned to its initial position.
2. 50 mL of deionized water was added to the titration beaker.
3. Then, 5 mL of Acetic acid (M=1 mol/dm^3) was added to the deionized water in titration beaker.
4. The beaker was then fastened to the titration head.
5. The titrator was run.
6. The stir function was executed and the solution was stirred for about 10 minutes.
7. After the titration was completed, the stirrer was stopped.
8. To obtain the value of alcoholic Potassium Hydroxide required to titrate the Acetic acid, the readings were obtained from the report which was printed.
9. The E-V curve obtained had illustrated the required volume of Methanolic Potassium Hydroxide solution to titrate the Acetic acid.
10. The experiment was repeated with the addition of toluene and propanol to acetic acid. The added amount of toluene and propanol was 50% of the quantity of acetic acid used. The titrant solvent used was still methanolic Potassium Hydroxide.
11. After repeating the experiment with impure Acetic acid and methanolic Potassium Hydroxide, the further experiment was conducted with the use of Ethanolic Potassium Hydroxide and Propanolic Potassium Hydroxide as the titrant solvent.
12. The obtained results were recorded and tabulated.

3.6.4 Safety precautions

- It must be ensured that we plug the power cable supplied into a receptacle outlet that is grounded. Occurrence of a technical fault could be lethal (Risk of electric shock).
- The instrument must be switched off and disconnected of the power cable before we open the housing. The sudden electric shock could be lethal (Risk of electric shock).
- We must not work in an environment which is subject to explosion hazards. The housing of the instrumentation is not gas tight, thus there is an explosion hazard due to spark formation (Risk of Explosion).
- Corrosion might be caused by the ingress of gases (Risk of Corrosion).
- The titration vessel must be tested regularly for firm seating in the titration head. If there is a possibility of it falls off, we might get injured, working with toxic titrants and solvents or strong acids or bases (Risk due to hazardous chemical substances).
- We must be fully covered with out personal protective equipments (PPEs) such as lab coat, safety goggles, mask and gloves while handling the chemicals and solvents throughout the experiment.
- We must always ensure that the splashed liquids are wiped off immediately, as the instrument is not water – proof.

3.7 Experiment 4: Density measurement

- Objective: To measure the densities of various concentrations of Acetic acid.
- Apparatus required: pycnometer, weighing machine, filter paper.
- Chemicals required: Acetic acid, distilled water
- Procedure:

1. The weight of empty and dry pycnometer was measured as m_0.
2. Then, 1/3 of the pycnometer was filled with objects made of examined materials (glass beads were used) and the mass was recorded as m_1.
3. Water was added such that pycnometer as well as capillary hole in the stopper was filled with water.
4. The spare water that leaked through the capillary hole was dried with a filter paper and the total weight was measured as m_2.
5. The pycnometer was emptied and filled with distilled water only.

6. The filter paper was used to dry the spare water again and the weight was measured as m_3.
7. The pycnometer was emptied again. It was rinsed with Acetic acid whose density is to be determined. The pycnometer was filled with the Acetic acid as previously and the weight was measured as m_4.

- Calculations:

1. The weight of water, $m_{H2O} = m_3 - m_0$ (in unit g)
2. The weight of Acetic acid, $m_{CH3COOH} = m_4 - m_0$ (in unit g)
3. The density of Acetic acid, $\rho_{CH3COOH} = (m_{CH3COOH} / m_{H2O}) \times \rho_{H2O}$ (in unit g/cm^3).

3.8 Experiment 5: Energy content measurement

- Objective: To measure the molar enthalpy change of neutralization.
- Apparatus required: polystyrene cup (beaker), accurate thermometer (±0.2°C), burette (insulation is optional), glass rod.
- Chemicals required: Acetic acid, Potassium Hydroxide solution, Phenolphthalein.
- Procedure:

1. Firstly, 50cm^3 of Acetic acid with a concentration 0.1M was pipetted into the insulated polystyrene cup.
2. Then, the burette was rinsed with Potassium Hydroxide solution of concentration 0.1M and filled up to the level of 50cm^3.
3. 3 drops of phenolphthalein was added to the insulated polystyrene cup.
4. The initial temperature of the solution was measured and recorded as T_o.
5. The Potassium Hydroxide solution was added to the polystyrene cup gradually, until the Phenolphthalein changed color to purple.
6. The maximum or minimum temperature of the final solution was observed on the temperature and recorded as T_f.

- Calculations:

1. The density was assumed close with that of water, thus the total mass, m_T = volume of acetic acid (initial) + volume of potassium hydroxide used

= X g

2. $Cp = 4.2$ J/K.g (specific heat capacity of water).
3. Temperature difference, $dT = T_f - T_o$
4. Exothermic or Endothermic neutralization reaction (depends on the outcome of the experiment).
5. $Q = m_T CpdT$
6. Number of moles of Acetic acid = MV / 1000, where M = concentration (mol/cm3)

   V = volume consumed (cm3)
7. $\Delta H_{neutralization}$ = Q / no.of moles / 1000 = Y kJ/mol

3.9 Experiment 6: Refractive Index measurement

- Objective: To measure the refractive index of Acetic acid at different concentration and temperature readings.
- Apparatus: Refractometer, Hot plate, Thermometer, Beaker, Syringe.
- Chemicals required: Acetic acid
- Procedure:

1. 50cm3 of 0.1M Acetic acid was poured into a beaker.
2. Its initial temperature was measured using thermometer and recorded.
3. The prisms were cleaned by wiping with a tissue moistened with methanol.
4. Few drops of acetic acid were placed on the prisms by using the syringe.
5. The refractive index was measured using the refractometer.
6. The measured refractive index was at initial temperature and concentration of acetic acid.
7. The acetic acid solution was heated up to certain temperature by using the hot plate and the temperature was recorded.
8. The steps above were repeated for various concentrations and temperatures of acetic acid and the respective refractive indexes of the solutions were measured and recorded.

3.10 Experiment 7: Viscosity measurement

- Objective: To measure the viscosity of Acetic acid at different concentration and temperature.
- Apparatus: Capillary viscometer in constant temperature bath, stop watch.
- Chemicals required: Acetic acid
- Procedure:

1. The acetic acid was allowed to flow through a capillary of a known diameter of a certain factor between 2 marked points.
2. The time taken by the liquid to flow was measured.
3. The time recorded was multiplied with the factor of the viscometer. The resultant value is the kinematic viscosity.
4. The temperature of the water bath was increased and the viscosity measurement was conducted for several times for different temperatures.
5. The experiment was repeated for Acetic acid with different concentrations.

3.11 Project Gantt Chart

No.	Detail / Work	1	2	3	4	5	6	7	8	9	10	M	11	12	13	14	15	16	17	18	19
1	Briefing & Update on student progress	▨										I									
2	Project work commences		▨	▨								D									
3	Submission of Progress Report 1				▨							S									
4	Project work continues					▨	▨	▨	▨	▨		E									
5	Submission of Progress Report 2										▨	M									
6	Poster Exhibition / Pre-EDX											B	▨								
7	EDX											R		▨							
8	Submission of Final Report											E			▨	▨					
9	Delivery of Final Report to External Examiner											A					▨				
10	Final Oral Presentation											K								▨	▨

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Results for Dilution of Hydrochloric Acid using cold tap water

For the preparation of standard samples, the dilution of HCL acid method was used. The samples prepared have their own theoretical pH values. At the same time, their actual pH values can be measured using the pH meter, by placing the probe in the solution and record the displayed and stabilized readings. The results obtained are as in **Appendix 1.0**. The graph below illustrates the obtained results.

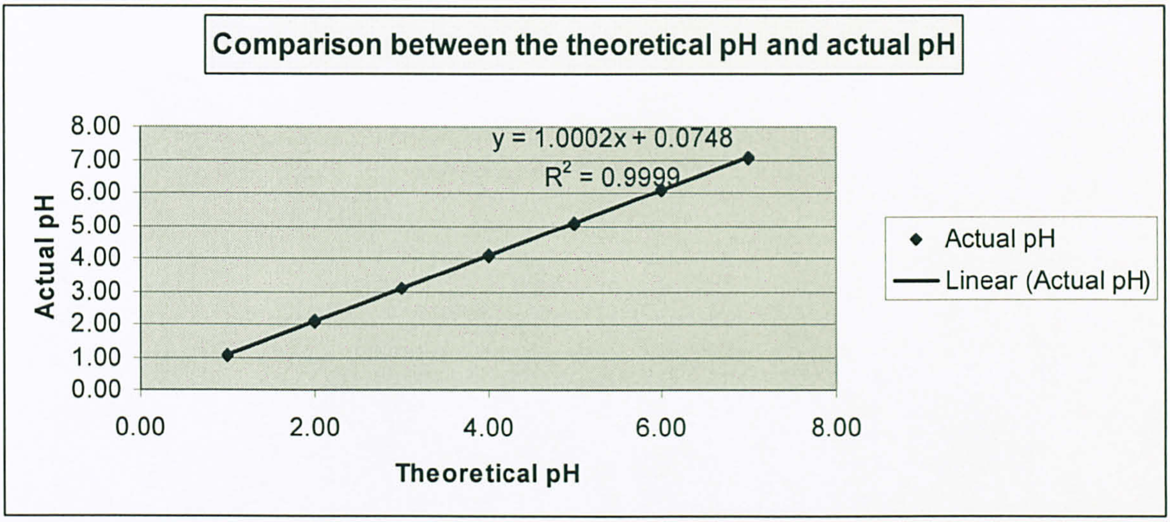


Figure 2: Comparison between the theoretical pH of the samples and the actual pH of the samples.

4.2 Results for Potentiometric Titration

4.2.1 Calibration of Electrode

Based on table in **Appendix 2.0**, for each buffer solution (with different pH), the desired value of the potential is significantly different. The graph of the potential (in mV) versus pH was plotted.

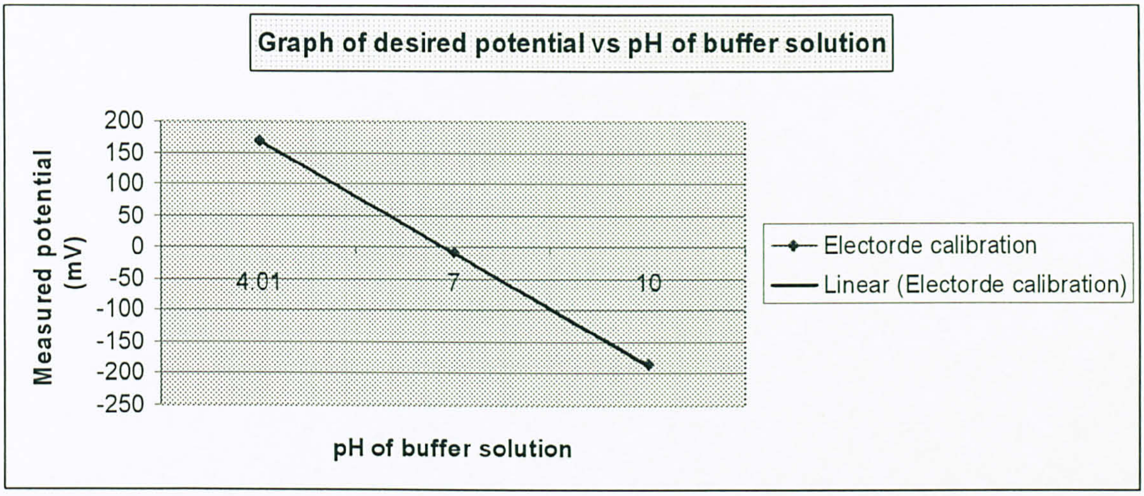


Figure 3: Graph of desired potential (in mV) against pH of the buffer solution

Based on the above graph (**Figure 3**), the slope of the graph was calculated by using the linear regression.

$$\text{Slope} = \frac{y1 - y2}{x1 - x2}$$

Thus, the calculated slope of the graph using linear regression is:

$$\text{Slope} = \frac{168.142 - (-185.913)}{4.01 - 10.00}$$
$$= -59.11 \text{ mV/pH}$$

Based on table in **Appendix 2.1**, for each buffer solution (with different pH), the measured value of the potential is significantly different. The graph of the potential (in mV) versus pH was plotted by the system.

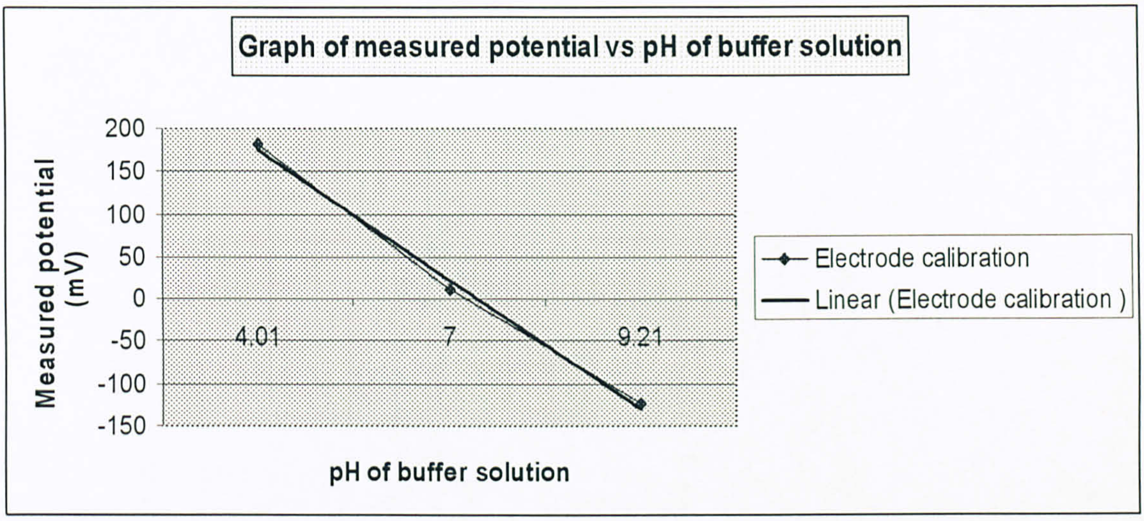


Figure 4: Graph of measured potential (in mV) against pH of the buffer solution

Based on the above graph (**Figure 4**), the slope of the graph was calculated by using the linear regression.

$$\text{Slope} = \frac{y1 - y2}{x1 - x2}$$

Thus, the calculated slope of the graph using linear regression is:

$$\text{Slope} = \frac{181.232 - (-123.022)}{4.01 - 9.21}$$
$$= -58.51 \text{ mV/pH}$$

4.2.2 Acid Content Measurement

For acid content measurement, the acid was fixed as Acetic acid. But, different bases were tried to titrate it. For the first three runs, Potassium Hydroxide in Methanol was used as the titrant. The obtained results for first run, second run, third run and fourth run are illustrated in **Appendix 2.2, Appendix 2.3, Appendix 2.4 and Appendix 2.5**. The plotted trends for the results are illustrated in **Figure 5, Figure 6, Figure** 7 and **Figure 8**.

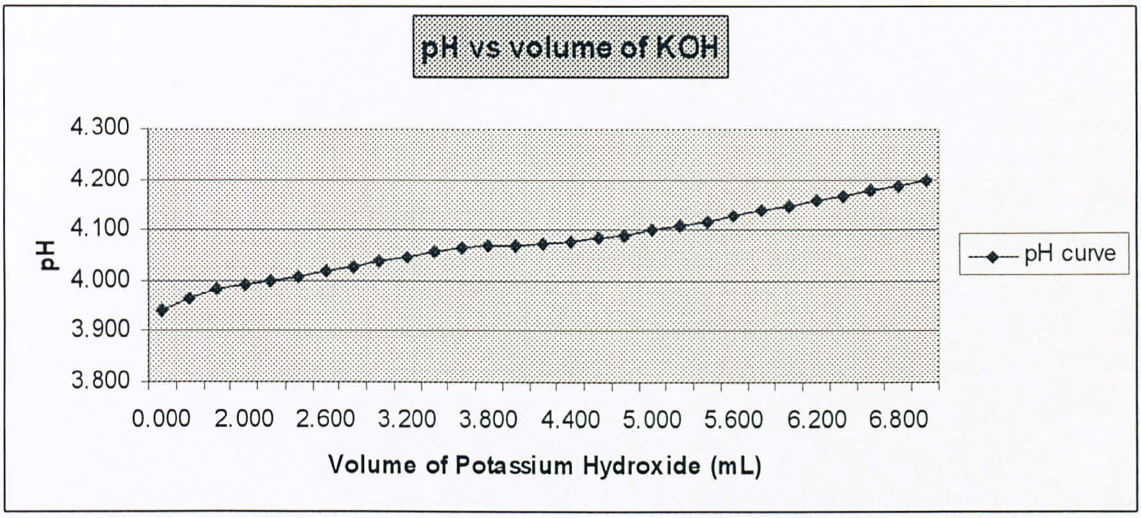


Figure 5: pH of the sample against the volume of Potassium Hydroxide for first run

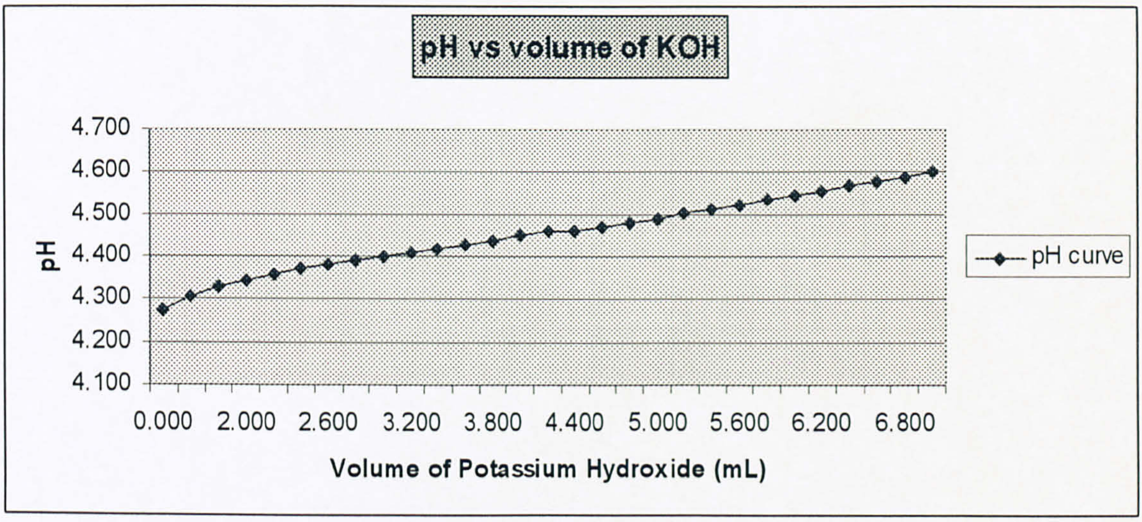


Figure 6: pH of the sample against the volume of Potassium Hydroxide for second run

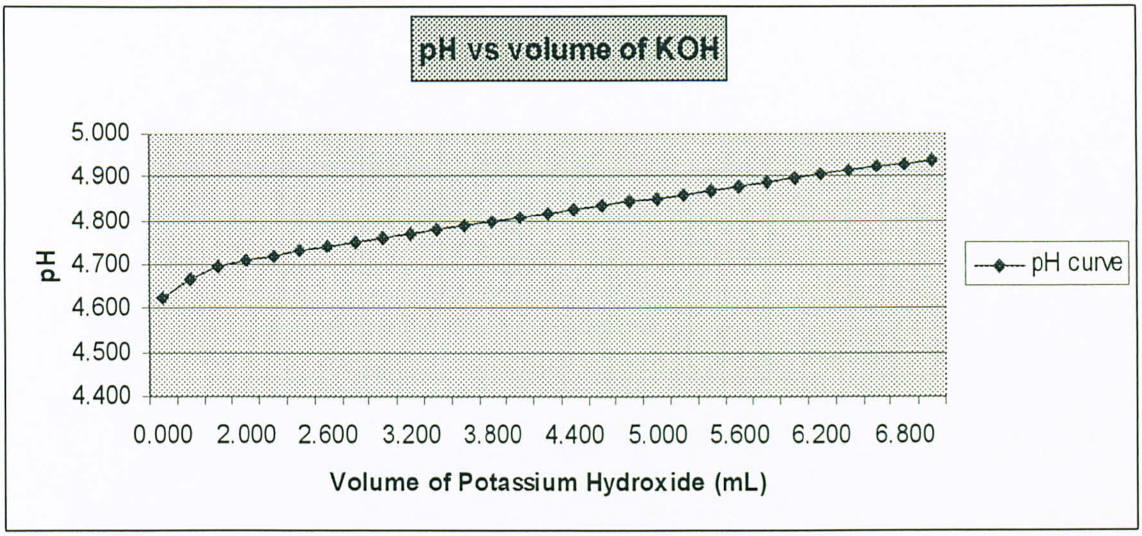


Figure 7: pH of the sample against the volume of Potassium Hydroxide for third run

After titrating the acetic acid with methanolic potassium hydroxide, the experiment was proceeded with Potassium Hydroxide in Propan-2-ol being the titrant. The sample used remained as pure Acetic acid. For previous three runs, the maximum volume for the titration to be terminated was set at 7.00 mL. For the new run, with Potassium Hydroxide as the titrant, the maximum volume for titration termination was increased to 30.00 mL. The obtained results are as in **Appendix 2.5.** The graph in **Figure 8** illustrates the trend.

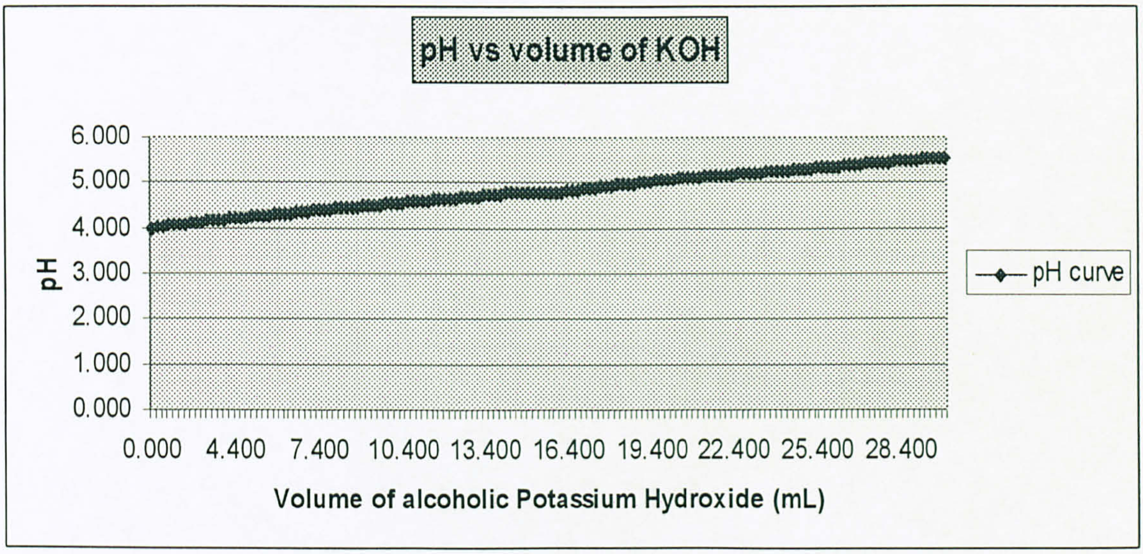


Figure 8: pH of the sample against the volume of Potassium Hydroxide for fourth run

The accurate amendments made while conducting the potentiometric titration enabled to measure the Total Acid Number for Acetic acid of concentration 0.1M. The Acetic acid used in the experiment was of various concentrations and purity level. The obtained Total Acid Number (TAN) values at various conditions are as in **Appendix 2.6, Appendix 2.7, Appendix 2.8, Appendix 2.9, Appendix 2.10** and **Appendix 2.11.** Meanwhile, the trend of the Total Acid Number values with respect to different purity of Acetic acid are illustrated in **Figure 9, Figure 10, Figure 11, Figure 12, Figure 13** and **Figure 14**.

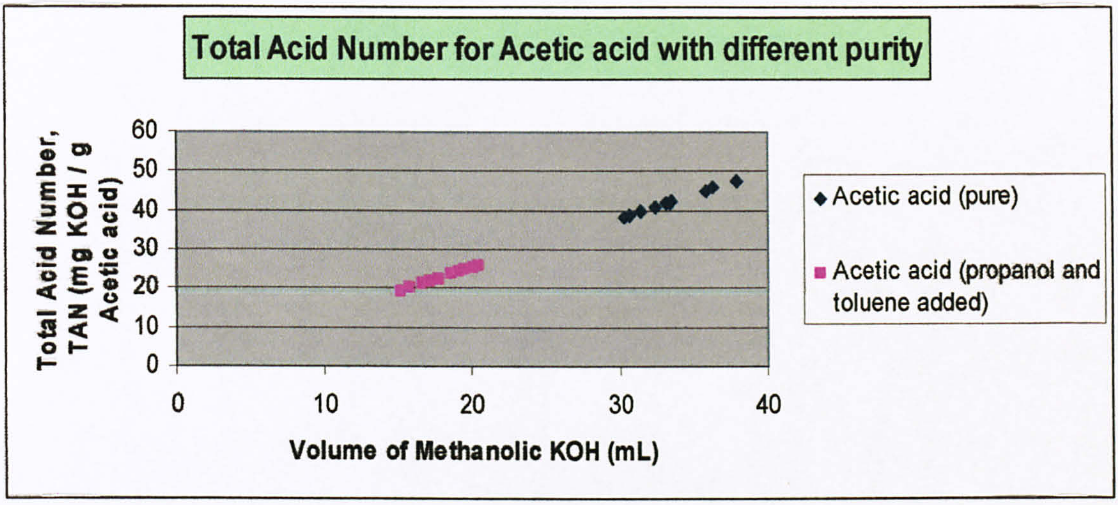


Figure 9: Total Acid Number against the volume of methanolic KOH solution.

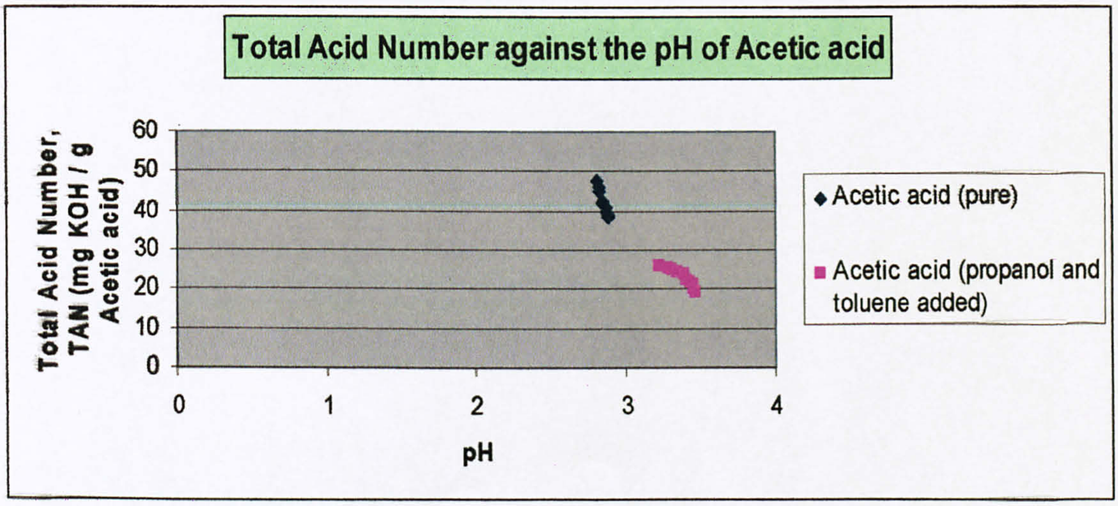


Figure 10: Total Acid Number against the pH of Acetic acid solution.

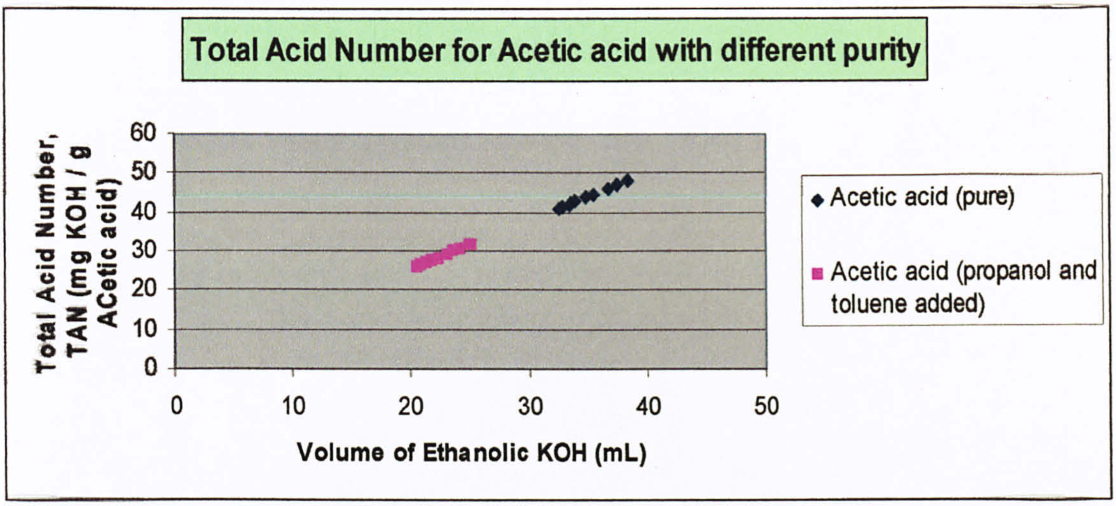


Figure 11: Total Acid Number against the volume of ethanolic KOH solution.

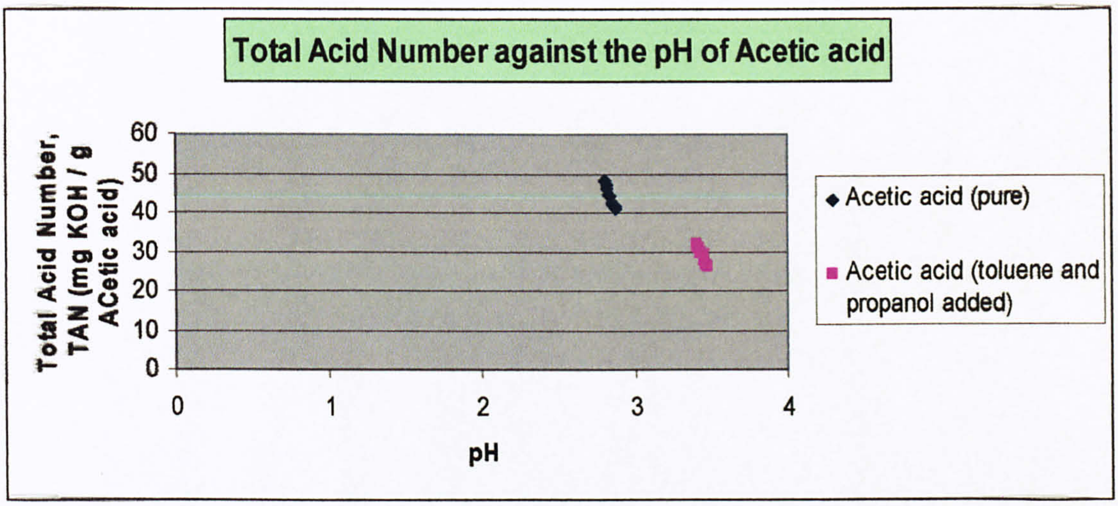


Figure 12: Total Acid Number against the pH of Acetic acid solution.

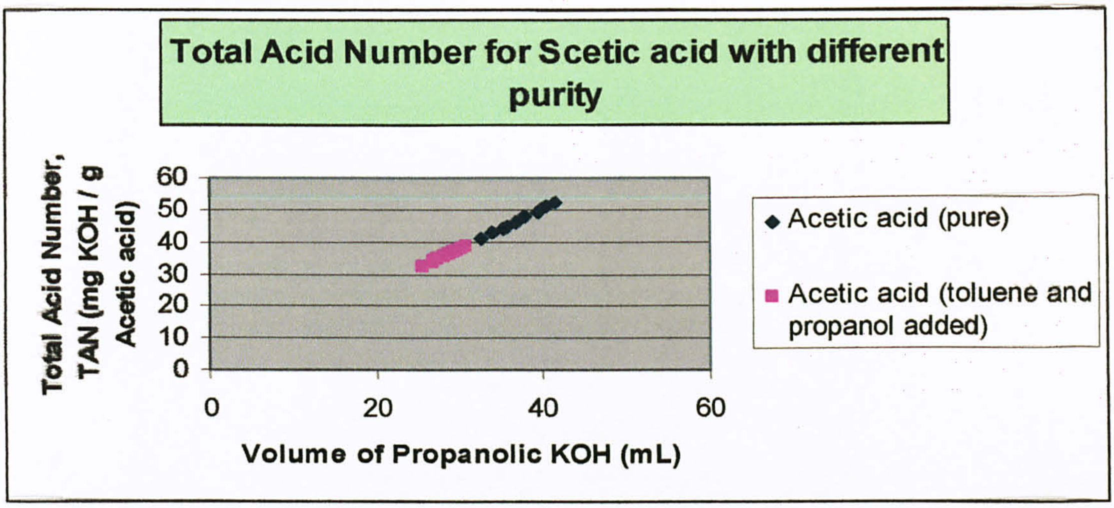


Figure 13: Total Acid Number against the volume of propanolic KOH solution.

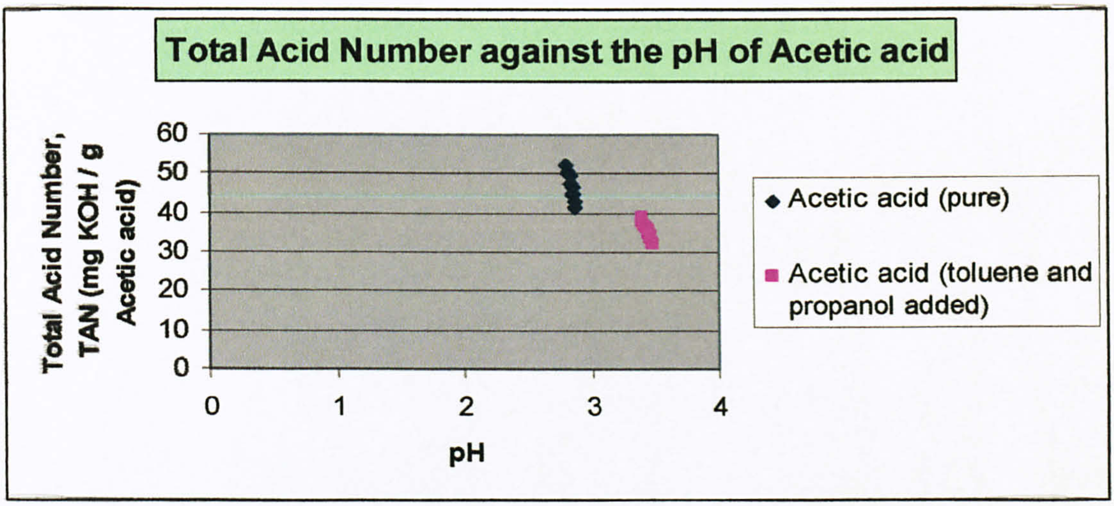


Figure 14: Total Acid Number against the pH of Acetic acid solution.

4.3 Results for density measurement

After completing the experiment for Total Acid Number (TAN) determination, the next phase involved the density measurement of Acetic acid. The density measurement, meanwhile was conducted for Acetic acid various concentrations. Specifically, the fixed concentrations were 0.01M, 0.1M and 1.0M. The obtained density values from the pycnometer, are illustrated in **Appendix 3.0, Appendix 3.1** and **Appendix 3.2**. Meanwhile, the plotted trend for the density and pH correlation is illustrated in **Figure 15**.

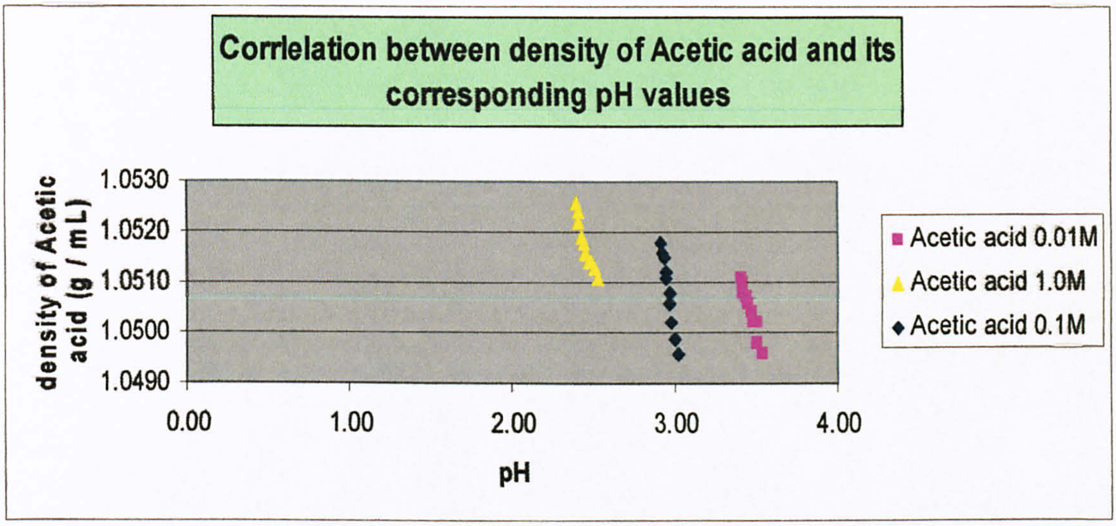


Figure 15: Density of Acetic acid with different concentrations against its corresponding pH values.

4.4 Results for viscosity measurement

The density measurement was the first indirect measurement parameter which was identified and the respective experiment was conducted. The viscosity measurement was the second indirect measurement parameter. The experiment was conducted by using the capillary tube viscometer and the obtained results are as in **Appendix 4.0**. The plotted trend of the viscosity of Acetic acid at different concentrations and temperatures is illustrated in **Figure 16**.

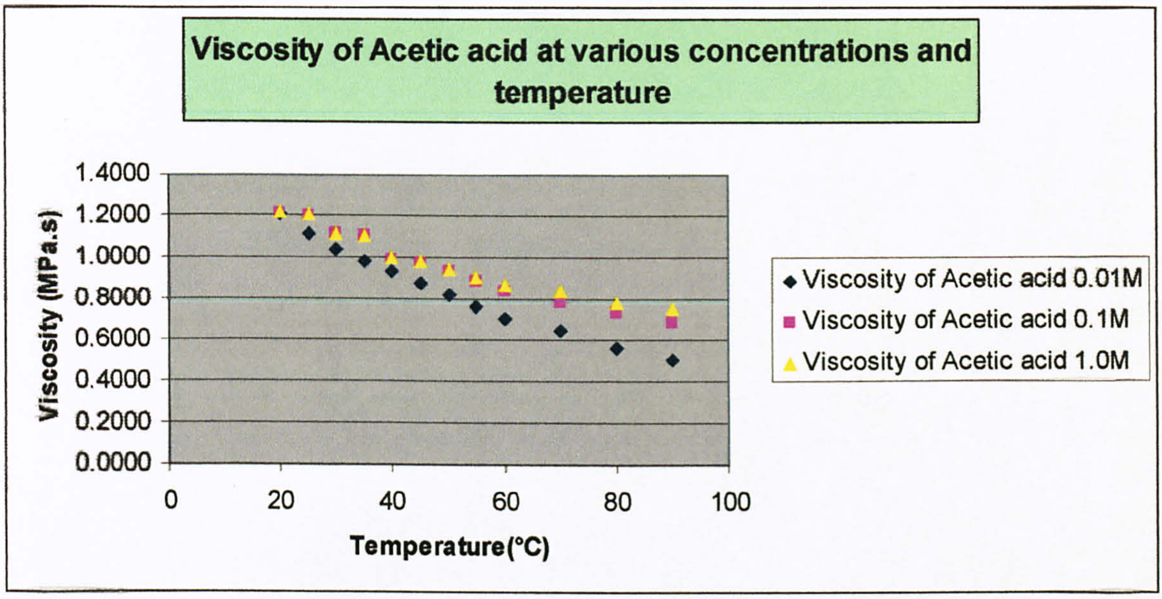


Figure 16: Viscosity of Acetic acid at various concentrations and temperatures.

4.5 Results for Refractive Index measurement

The second last indirect measurement parameter was the refractive index of acetic acid. This experiment was conducted with the help of refractometer. The obtained results are as in **Appendix 5.0**. The plotted trend of Refractive index of Acetic acid at various concentrations and temperatures is illustrated in **Figure 17**.

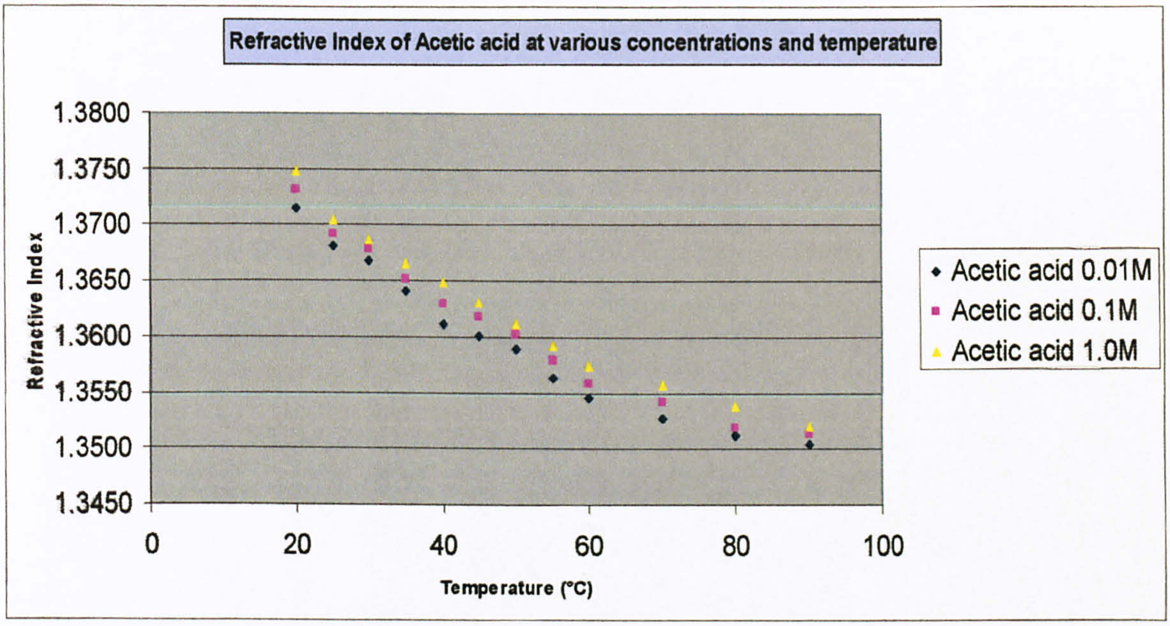


Figure 17: Refractive Index of Acetic acid at various concentrations and temperatures.

4.6 Discussion

4.6.1 Discussion for Standard Samples

Based on table in **Appendix 1.0**, the deviation between the theoretical pH of the samples and the actual pH of the samples is at minimum. For the first sample (theoretical pH=1.0), the maximum deviation was 0.10. For the second sample, where its theoretical pH was 2.0, the maximum actual pH was measured to be 2.11, with a deviation of 0.11. For the remaining samples from pH 3 up to pH 7, the maximum deviations were 0.10, 0.12, 0.09, 0.10 and 0.11 respectively. Therefore, the prepared samples are fit to be used for the Total Acid Number (TAN) measurement.

The standard samples preparation is to set a benchmark. By using these samples, we will be able to proceed with the Total Acid Number (TAN) calculation. Specifically, the method will be used is Color indicating titration. The base to be used in this experiment will be Potassium Hydroxide of concentration 0.1M. The phenolphthalein will be added to one of the standard samples, and the KOH solution will be used for titration purpose. The amount of KOH solution (in mL) which is required to neutralize the sample will be recorded and its mass can be calculated. At the same time, it is necessary to measure the mass of the standard sample. Finally, the calculated mass of the KOH solution will be converted to mg KOH, and divided with the mass of the sample (in g). Thus, the end result will indicate the required mass of KOH (in mg) to neutralize 1 g of the standard sample.

4.6.2 Discussion for Potentiometric Titration

Firstly, the electrode calibration was conducted with buffer solution. The calibration process commenced with the electrode immersed and stirred in buffer solution of pH 4.01. It was followed by immersion and stirring in buffer solution of pH 7.00 and pH 9.21 eventually. The actual pH of the final buffer solution was to be 10.00, but in the lab, the maximum pH present was 9.21. Thus, it had resulted in the potential reading to deviate significantly form the desired reading (refer to **Appendix 2.0 and Appendix 2.1**). Two sets of results were compared. The desired results, in this case the measured potential (mV) and also the pH of the buffer solution were critical to determine the slope of the graph. For the desired results, the calculated slope of the graph using linear regression was -59.11 mV/pH (refer to **Figure 3**). Meanwhile, for the real experiment conducted, the measured potential deviated significantly from the desired readings. For the measured values of the potential, the slope of the graph was -58.51 mV/pH (refer to **Figure 4**). For the pH of the buffer solution, only the final buffer was deviating 0.79 from the desired pH of the buffer solution. The difference between the two slopes of the graphs was 0.6. The fixed upper limit and the lower limit of the slope of the graphs were -55.00 mV/pH and -65.00 mV/pH. Since the results obtained from the electrode calibration had resulted in the slope of the graph to lie between the upper limit and lower limit, thus the pH electrode was fit to be used for the potentiometric titration. Its deviation of 0.6 from the desired slope was neglected.

The acid chosen for the potentiometric titration was acetic acid. This proved to be the direct alternative in the absence of Bio-oil temporarily. The base that was used for the four runs of titration was Potassium Hydroxide. But, for the first three runs, the Potassium Hydroxide was dissolved using Methanol. For the fourth run, it was dissolved using Propan-2-ol. The objective of using different alcohol is to identify if there is any significant change in the titration experiment. Based on table in **Appendix 2.2** and **Figure 5**, it can be seen that as the volume of the Potassium Hydroxide added gradually to the titration beaker, the pH of the sample started to increase. Its acidity gradually reduced. The graph in **Figure 5** is in increasing trend, but the trend is just slightly steep. For the second run (refer to **Appendix 2.3** and **Figure 6**), the titration continued from the final pH of the first run.

For the second run, the trend of the curve is similar to the first run. The significant inflection point was still not identified. For the third run (refer to **Appendix 2.4** and **Figure 7**), the titration continued further with the pH of the sample increasing slightly and the volume of the Potassium Hydroxide increasing at a steady – state of 0.2 mL. After the third run of titration was completed, the desired results were not obtained as per literature review, where the significant inflection point should have been visible on the plotted graph. For the fourth run, the titrant remained as Potassium Hydroxide, but it was dissolved in Porpan-2-ol. For the fourth run, the termination volume of the titration was increased to 30.00 mL, in order to obtain the desired results. Based on table in **Appendix 2.5** and **Figure 8**, it can be seen that the pH had exceeded the value of 5.00, as more and more Potassium Hydroxide was added to the acid sample. For the third run, after the termination volume was set at 30.00 mL, there was not any significant inflection yet to determine the titration end point. From the obtained results, there were few things to be taken note of. Firstly, the potentiometric titration did not give the desired results. There might have been some inevitable errors such as the electrode calibration and the calculation of acid's concentration.. The concentration of the titrant was calculated accurately, as it was well-prepared in a 1 litre volumetric flask. But, for the acetic acid, the concentration was not calculated tediously and it was used instantly as the acid sample. Thus, the concentration of the acid had to be determined before proceeding with the titration. Besides, there might have been some faulty readings due to the instrument problem. This need to be identified and rectified. More runs of titration need to be conducted to verify this method as the best possible acid content measurement for organic acids.

4.6.3 Discussion for Total Acid Number Measurement

The Total Acid Number (TAN) were obtained successfully after overcoming and minimizing the errors while proceeding with the experiment. Based on table in **Appendix 2.6** and **Figure 9**, the required volume of Methanolic Potassium Hydroxide to neutralize the pure Acetic acid of concentration 0.1M is in increasing trend as the Total Acid Number increases (*Neto, J.M., 1996*). The Potassium Hydroxide was dissolved in alcohol in order to act as base because the conventional TAN determination method used alcoholic base as the titrant solution. The obtained values of methanolic Potassium Hydroxide volume are directly proportional to the strength of the acid. Since the acid used in the experiment was pure acetic acid, thus the required volume of methanolic Potassium Hydroxide to neutralize it is larger. When the Potassium Hydroxide is dissolved in the alcohol, its base strength is reduced slightly, and thus it must be used in excess amount to neutralize the strong acid. If pure Potassium Hydroxide solution was to be used as the titrant, thus the required volume of it will be lesser to neutralize the acetic acid. The Total Acid Number (TAN) is calculated in unit mg KOH / g acetic acid. The respective values of TAN for acetic acid are displayed in **Appendix 2.6.**

For the second run of experiment, the Acetic acid was not used purely and it was added with toluene and propanol. The added amount was 10% of the quantity of acetic acid used. When the Acetic acid became impure due to the addition of toluene and propanol, the required volume of base to neutralize it became lesser as can be seen in **Figure 9**. This is due to reduction of strength of acid as the consequence of addition of toluene and propanol. When the titrant solvent was changed to Ethanolic Potassium Hydroxide, the required volume of base to neutralize the acetic acid was larger compared to the Methanolic Potassium Hydroxide. As explained in literature review (*science and engineering encyclopedia, 1999*), the larger alkyl group will make the formed salt to be unstable. When the salt becomes unstable, it tends to bind back with the water molecule to reform both potassium hydroxide and alcohol. Thus, it reduces the strength of base. Therefore, the order of decreasing strength of titrant solvent will be Methanolic Potassium Hydroxide, Ethanolic Potassium Hydroxide and Propanolic Potassium

Hydroxide (refer to **Figure 9, Figure 11** and **Figure 13**). The statement was justified with the results obtained from the Potentiometric Titration experiment. Meanwhile, the Acetic acid's TAN is inversely proportional to its pH value. By referring to **Figure 10, Figure 12** and **Figure 14**, it can be interpreted that the TAN will be higher if the acidity level is higher for an acid, thus the pH of the acid will be of lower value.

4.6.4 Discussion for Density measurement

The obtained results for density measurement can be discussed as per the statement in literature review. Based on the table in **Appendix 3.0, Appendix 3.1** and **Appendix 3.2**, and **Figure 15**, the density values are in increasing trend with the concentration of Acetic acid (*The Columbia Encyclopedia, 2004*). The acetic acid with the concentration of 1.0M, has the lowest pH values and highest density values. For liquid phase, when the concentration is high, the available space between acid molecules is at the least, thus the arrangement of the molecules will be compact. This results in the density to be higher. As the concentration of acetic acid is reduced from 1.0M to 0.1M and eventually to 0.01M, the density values are in decreasing trend. The relationship between the pH and density is reverse, thus it can be interpreted that the higher acidity level can be indicated by the higher density values. This new finding was justified and verified after the density measurement was repeated for several times until the observed trend was obtained.

4.6.5 Discussion for Viscosity measurement

The trend of acetic acid's viscosity can be observed in **Appendix 4.0** and **Figure 16.** The viscosity is dependent on temperature variation. From the experiment conducted, the viscosity variation was observed based on the varying concentrations of acetic acid and the corresponding temperature values. By referring to **Figure 16**, the acetic acid of concentration 0.01M illustrated the least values of viscosity. Coincidently, the viscosity values started decreasing with the increment of temperature (*Bleazard, J. G.; Sun, T. F.; Teja, A. S., 1996*). When the temperature is increased, the intermolecular collision becomes vigorous extremely, and thus the arrangement of the molecules will be scattered around. It simplifies the flow of liquid. From the discussion for density measurement (in **section 4.6.4**), the higher concentration of acetic acid results in higher density. The higher density ensures compact arrangement of molecules, thus the viscosity will be higher. The overall interpretation will be, the concentrated acid will indicate higher viscosity, and thus the lowest pH values. Therefore, the measurement of viscosity can indirectly specify the acidity level of an organic acid. The relationship between the viscosity and the acidity of organic acid is directly proportional based on the experimental results obtained.

4.6.6 Discussion for Refractive Index measurement

By referring to the results and trend in **Appendix 5.0** and **Figure 17** respectively, the refractive index measurement can be correlated with the acidity level of organic acid. The acetic acid with the highest concentration (1.0M) indicates the highest refractive index values. It was followed in accordance by acetic acid of concentration 0.1M and 0.01M respectively. The refractive index is the ratio of sine of the angle of incidence, i, to the sine of the angle of refraction, r. Thus, $n = \sin i / \sin r$. When the acetic acid's concentration is high, again we discuss the arrangement of molecules. The higher concentration of acetic acid keeps the molecules intact. Thus, when the incidence light penetrates the liquid medium, the refraction will be severely limited and reduced. This results in the ratio of sine of the angle of incidence, i, to the sine of the angle of refraction, r, to be higher. Hereby, the measured refractive index will be higher for the most concentrated acid as can bee seen in **Figure 17** (*Encyclopedia Britannica, 2002).* At the same time, it can be concluded that the higher acidity level of an organic acid can be indicated by the high refractive index values. The temperature increment results in the refractive index to be of lower values.

CHAPTER 5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the conducted experiments for the acid content determination in organic acids, the Total Acid Number (TAN) had been identified and verified as the best possible method to measure the acidity. At the same time, the indirect measurement parameters, such as the density, viscosity and refractive index of acetic acid were able to show some results to be correlated with the acidity level of organic acid. The final indirect measurement parameter, which is the energy content of acetic acid, is yet to produce the desired results even tough several runs of experiments already been conducted. In overall, the identified methods and the indirect measurement parameters were successfully correlated with the acidity of the organic acid, which was Acetic acid.

5.2 Recommendation

The utmost important recommendation is to identify the drawback of the energy content measurement. Several runs of experiments did not produce any desired outcome. The energy content measurement should be able to clearly illustrate the acidity level of Acetic acid. Thus, this method is to be researched even deeper to identify and verify its correlation with the acidity of Acetic acid. Meanwhile, the tested chemical in this project was Acetic acid, which is an organic acid. From the perspective of industrial need, the identified methods are to be tested on bio-oil. At the moment, there is no specific method of acid content determination for bio-oil. Therefore, the identified and verified indirect parameters of Acetic acid can be utilized to indicate the acidity level of bio-oil. Besides, using the bio-oil model as the sample to test all these acid content determining methods will give more realistic results, since we are interested to apply these methods on the real bio-oil.

REFERENCES

1. The Columbia Encyclopedia, Sixth Edition Copyright© 2004, Columbia University Press.

2. Bleazard, J. G.; Sun, T. F.; Teja, A. S.,1996, International Journal of Thermophysics, Volume 17, Issue 1, pp.111-125

3. G. K. Jayaprakasha and K. K. Sakariah, 1998,

   "Determination of organic acids by high-performance liquid chromatography".

4. Science and Engineering Encyclopedia, 1999.
5. F. H. Mahfud, F. P. van Geel, R. H. Venderbosch and H. J. Heeres, 2008,

   "Acetic Acid Recovery from Fast Pyrolysis Oil".

6. Neto, J.M., 1996,

   "Techniques to measure the acid content in organic and inorganic sources".

7. The Encyclopedia Britannica, 2002.
8. Heller, Gregory A, 1998,

   " Analysis on physical and chemical properties of organic acids".

APPENDICES

Appendix 1.0

Table 1.0A: The theoretical pH of the samples and the measured pH of the samples

Sample no.	HCl concentration (mol/dm^3)	Theoretical pH	Actual pH (from the pH meter)		
			Set 1	Set 2	Set 3
1	0.1	1.00	1.04	1.06	1.10
2	0.01	2.00	2.05	2.06	2.11
3	0.001	3.00	3.07	3.08	3.10
4	0.0001	4.00	4.08	4.07	4.12
5	0.00001	5.00	5.05	5.08	5.09
6	0.000001	6.00	6.04	6.06	6.10
7	0.0000001	7.00	7.05	7.07	7.11

Appendix 2.0

Table 2.0A: Desired values of the potential based on the buffer solution

Sample no.	Buffer solution	Wait time (s)	Speed (%)	Measured values (mV)	Calculated slope (mV/pH)
1	4.01	60	50	168.142	-59.11
2	7.00	60	50	-9.200	
3	10.00	60	50	-185.913	

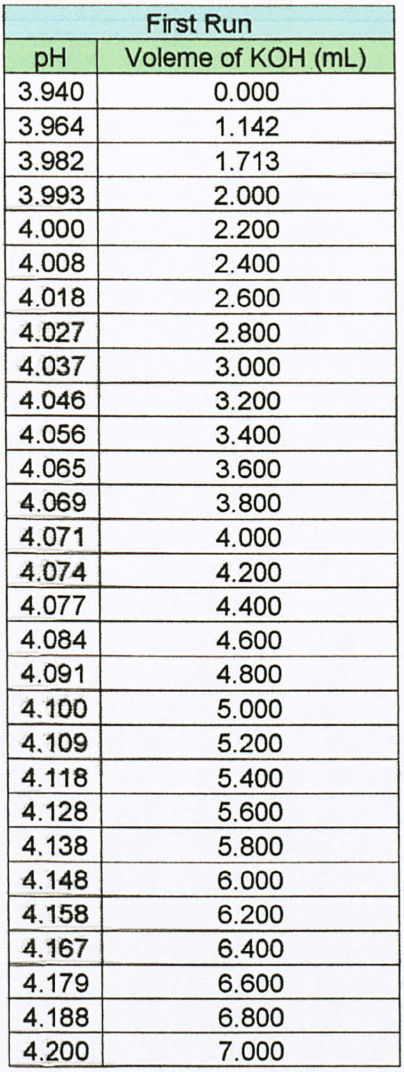
Appendix 2.1

Table 2.1A: Measured values of the potential based on the buffer solution

Sample no.	Buffer solution	Wait time (s)	Speed (%)	Measured potential (mV)	Calculated slope (mV/pH)
1	4.01	60	50	181.232	-58.51
2	7.00	60	50	11.214	
3	9.21	60	50	-123.022	

Appendix 2.2

Table 2.2A: Volume oh KOH required to neutralize the Acetic acid for first run



First Run	
pH	Voleme of KOH (mL)
3.940	0.000
3.964	1.142
3.982	1.713
3.993	2.000
4.000	2.200
4.008	2.400
4.018	2.600
4.027	2.800
4.037	3.000
4.046	3.200
4.056	3.400
4.065	3.600
4.069	3.800
4.071	4.000
4.074	4.200
4.077	4.400
4.084	4.600
4.091	4.800
4.100	5.000
4.109	5.200
4.118	5.400
4.128	5.600
4.138	5.800
4.148	6.000
4.158	6.200
4.167	6.400
4.179	6.600
4.188	6.800
4.200	7.000

Appendix 2.3

Table 2.3A: Volume oh KOH required to neutralize the Acetic acid for second run

Second Run	
pH	Voleme of KOH (mL)
4.272	0.000
4.307	1.142
4.332	1.713
4.346	2.000
4.359	2.200
4.370	2.400
4.380	2.600
4.389	2.800
4.399	3.000
4.409	3.200
4.420	3.400
4.429	3.600
4.439	3.800
4.450	4.000
4.460	4.200
4.463	4.400
4.471	4.600
4.481	4.800
4.491	5.000
4.502	5.200
4.513	5.400
4.524	5.600
4.535	5.800
4.546	6.000
4.557	6.200
4.568	6.400
4.579	6.600
4.589	6.800
4.600	7.000

Appendix 2.4

Table 2.4A: Volume oh KOH required to neutralize the Acetic acid for third run

Third Run	
pH	Voleme of KOH (mL)
4.625	0.000
4.666	1.142
4.693	1.713
4.709	2.000
4.721	2.200
4.732	2.400
4.742	2.600
4.752	2.800
4.761	3.000
4.770	3.200
4.780	3.400
4.789	3.600
4.798	3.800
4.808	4.000
4.818	4.200
4.827	4.400
4.836	4.600
4.843	4.800
4.852	5.000
4.860	5.200
4.871	5.400
4.880	5.600
4.889	5.800
4.896	6.000
4.906	6.200
4.914	6.400
4.923	6.600
4.932	6.800
4.939	7.000

Appendix 2.5 Table 2.5A: Volume oh KOH required to neutralize the Acetic acid for fourth run

Fourth Run	
pH	Voleme of KOH (mL)
3.979	0.000
4.015	1.142
4.043	1.713
4.059	2.000
4.072	2.200
4.086	2.400
4.098	2.600
4.110	2.800
4.123	3.000
4.137	3.200
4.149	3.400
4.161	3.600
4.174	3.800
4.185	4.000
4.197	4.200
4.211	4.400
4.223	4.600
4.235	4.800
4.248	5.000
4.260	5.200
4.272	5.400
4.286	5.600
4.298	5.800
4.310	6.000
4.323	6.200
4.335	6.400
4.347	6.600
4.359	6.800
4.371	7.000
4.383	7.200
4.395	7.400
4.406	7.600
4.418	7.800
4.430	8.000
4.441	8.200
4.452	8.400
4.463	8.600
4.475	8.800
4.486	9.000
4.497	9.200
4.508	9.400
4.518	9.600
4.529	9.800
4.539	10.000
4.556	10.200
4.564	10.400
4.573	10.600
4.583	10.800
4.593	11.000
4.603	11.200
4.613	11.400
4.623	11.600
4.632	11.800
4.642	12.000
4.653	12.200
4.663	12.400
4.673	12.600
4.683	12.800
4.694	13.000
4.704	13.200
4.715	13.400
4.727	13.600
4.740	13.800
4.753	14.000
4.762	14.200
4.764	14.400
4.766	14.600
4.767	14.800
4.767	15.000
4.768	15.200
4.768	15.400
4.768	15.600
4.772	15.800
4.783	16.000
4.797	16.200
4.811	16.400
4.827	16.600
4.842	16.800
4.856	17.000
4.871	17.200
4.887	17.400
4.902	17.600
4.917	17.800
4.931	18.000
4.946	18.200
4.962	18.400

4.977	18.600
4.989	18.800
5.004	19.000
5.018	19.200
5.030	19.400
5.043	19.600
5.055	19.800
5.065	20.000
5.079	20.200
5.087	20.400
5.096	20.600
5.105	20.800
5.115	21.000
5.124	21.200
5.133	21.400
5.142	21.600
5.149	21.800
5.158	22.000
5.166	22.200
5.175	22.400
5.184	22.600
5.192	22.800
5.201	23.000
5.211	23.200
5.221	23.400
5.231	23.600
5.241	23.800
5.251	24.000
5.262	24.200
5.272	24.400
5.283	24.600
5.293	24.800
5.303	25.000
5.314	25.200
5.324	25.400
5.335	25.600
5.345	25.800
5.355	26.000
5.366	26.200
5.376	26.400
5.386	26.600
5.396	26.800
5.406	27.000
5.415	27.200
5.425	27.400
5.435	27.600
5.445	27.800
5.454	28.000
5.465	28.200
5.474	28.400
5.483	28.600
5.493	28.800
5.502	29.000
5.512	29.200
5.520	29.400
5.529	29.600
5.539	29.800
5.549	30.000

Appendix 2.6

Table 2.6A: Neutralization of Acetic acid of concentration 0.1M using Methanolic Potassium Hydroxide solution of concentration 0.1M

Run	Volume consumption of Methanolic KOH (ml)	Acid Content			pH of Acetic acid
		mol/L	g/L	mg KOH / g acetic acid	
1	30.325	0.6065	36.420	38.2085	2.89
2	30.685	0.6137	36.853	38.6621	2.89
3	31.452	0.6290	37.774	39.6285	2.87
4	32.385	0.6477	38.894	40.8040	2.87
5	33.147	0.6629	39.810	41.7641	2.85
6	33.546	0.6709	40.289	42.2668	2.85
7	33.365	0.6673	40.071	42.0388	2.85
8	35.854	0.7171	43.061	45.1748	2.83
9	36.348	0.7270	43.653	45.7968	2.82
10	37.854	0.7571	45.463	47.6948	2.81

Appendix 2.7

Table 2.7A: Neutralization of Acetic acid (with the addition of Toluene and Propanol) of concentration 0.1M using Methanolic Potassium Hydroxide solution of concentration 0.1M

Run	Volume consumption of methanolic KOH (ml)	Acid Content			pH of Acetic acid
		mol/L	g/L	mg KOH / g acetic acid	
1	15.251	0.3050	18.316	19.2157	3.47
2	15.826	0.3165	19.007	19.9402	3.46
3	16.725	0.3345	20.087	21.0729	3.45
4	17.227	0.3445	20.690	21.7054	3.43
5	17.816	0.3563	21.397	22.4476	3.41
6	18.693	0.3739	22.450	23.5526	3.39
7	19.148	0.3830	22.997	24.1258	3.35
8	19.564	0.3913	23.496	24.6500	3.31
9	20.124	0.4025	24.169	25.3556	3.28
10	20.455	0.4091	24.566	25.7726	3.22

Appendix 2.8

Table 2.8A: Neutralization of Acetic acid of concentration 0.1M using Ethanolic Potassium Hydroxide solution of concentration 0.1M

Run	Volume consumption of Ethanolic KOH (ml)	Acid Content			pH of Acetic acid
		mol/L	g/L	mg KOH / g acetic acid	
1	32.568	0.6514	39.114	41.0346	2.87
2	32.857	0.6571	39.461	41.3987	2.87
3	33.451	0.6690	40.175	42.1471	2.85
4	33.582	0.6716	40.332	42.3122	2.85
5	33.874	0.6775	40.683	42.6801	2.85
6	34.846	0.6969	41.850	43.9048	2.84
7	35.357	0.7071	42.464	44.5486	2.83
8	36.689	0.7338	44.063	46.2269	2.82
9	37.354	0.7471	44.862	47.0648	2.82
10	38.287	0.7657	45.983	48.2403	2.80

Appendix 2.9

Table 2.9A: Neutralization of Acetic acid (with addition of toluene and propanol) of concentration 0.1M using Ethanolic Potassium Hydroxide solution of concentration 0.1M

Run	Volume consumption of ethanolic KOH (ml)	Acid Content			pH of Acetic acid
		mol/L	g/L	mg KOH / g acetic acid	
1	20.566	0.4113	24.700	25.9125	3.48
2	20.854	0.4171	25.046	26.2753	3.48
3	21.338	0.4268	25.627	26.8852	3.47
4	21.796	0.4359	26.177	27.4622	3.46
5	22.367	0.4473	26.863	28.1817	3.46
6	23.125	0.4625	27.773	29.1367	3.45
7	23.682	0.4736	28.442	29.8385	3.43
8	24.253	0.4851	29.128	30.5580	3.42
9	24.957	0.4991	29.973	31.4450	3.41
10	25.114	0.5023	30.162	31.6428	3.41

Appendix 2.10

Table 2.10A: Neutralization of Acetic acid of concentration 0.1M using Propanolic Potassium Hydroxide solution of concentration 0.1M

Run	Volume consumption of Propanolic KOH (ml)	Acid Content			pH of Acetic acid
		mol/L	g/L	mg KOH / g acetic acid	
1	32.639	0.6528	39.199	41.1240	2.87
2	33.886	0.6777	40.697	42.6952	2.87
3	35.245	0.7049	42.329	44.4075	2.85
4	35.836	0.7167	43.039	45.1522	2.85
5	36.745	0.7349	44.131	46.2975	2.85
6	37.863	0.7573	45.473	47.7061	2.84
7	39.298	0.7860	47.197	49.5142	2.83
8	39.857	0.7971	47.868	50.2185	2.82
9	40.362	0.8072	48.475	50.8548	2.82
10	41.523	0.8305	49.869	52.3176	2.80

Appendix 2.11

Table 2.11A: Neutralization of Acetic acid (with addition of toluene and propanol) of concentration 0.1M using Propanolic Potassium Hydroxide solution of concentration 0.1M

Run	Volume consumption of propanolic KOH (ml)	Acid Content			pH of Acetic acid
		mol/L	g/L	mg KOH / g acetic acid	
1	25.638	0.5128	30.791	32.3030	3.48
2	26.785	0.5357	32.169	33.7482	3.47
3	26.982	0.5396	32.405	33.9964	3.47
4	27.455	0.5491	32.973	34.5924	3.46
5	28.147	0.5629	33.805	35.4643	3.45
6	28.736	0.5747	34.512	36.2064	3.43
7	29.254	0.5851	35.134	36.8591	3.42
8	29.784	0.5957	35.771	37.5268	3.41
9	30.226	0.6045	36.301	38.0837	3.41
10	30.685	0.6137	36.853	38.6621	3.40

Appendix 3.0

Table 3.0A: Density measurement for Acetic acid (concentration: 0.01 M)

Acetic acid 0.01M								
trial no.	m0 (g)	m3 (g)	m4 (g)	mL (g)	mH2O (g)	ρH2O (g/mL)	ρCH3COOH (g/mL)	pH
1	19.75	45.87	47.17	27.42	26.12	1.0000	1.0496	3.54
2	19.75	45.91	47.21	27.46	26.16	1.0000	1.0498	3.51
3	19.75	45.94	47.25	27.50	26.19	1.0000	1.0502	3.50
4	19.75	45.96	47.28	27.53	26.21	1.0000	1.0502	3.48
5	19.75	46.01	47.33	27.58	26.26	1.0000	1.0504	3.47
6	19.75	46.04	47.37	27.62	26.29	1.0000	1.0506	3.45
7	19.75	46.07	47.40	27.65	26.32	1.0000	1.0507	3.44
8	19.75	46.12	47.46	27.71	26.37	1.0000	1.0508	3.42
9	19.75	46.16	47.51	27.76	26.41	1.0000	1.0510	3.42
10	19.75	46.21	47.56	27.81	26.46	1.0000	1.0511	3.41

Appendix 3.1

Table 3.1A: Density measurement for Acetic acid (concentration: 0.1 M)

Acetic acid 0.1M								
trial no.	m0 (g)	m3 (g)	m4 (g)	mL (g)	mH2O (g)	ρH2O (g/mL)	ρCH3COOH (g/mL)	pH
1	19.75	45.97	47.27	27.52	26.22	1.0000	1.0496	3.02
2	19.75	46.21	47.53	27.78	26.46	1.0000	1.0499	3.00
3	19.75	46.30	47.63	27.88	26.55	1.0000	1.0502	2.98
4	19.75	46.43	47.78	28.03	26.68	1.0000	1.0506	2.97
5	19.75	46.51	47.87	28.12	26.76	1.0000	1.0508	2.97
6	19.75	46.53	47.90	28.15	26.78	1.0000	1.0511	2.95
7	19.75	46.60	47.97	28.22	26.85	1.0000	1.0512	2.95
8	19.75	47.00	48.40	28.65	27.25	1.0000	1.0515	2.93
9	19.75	47.15	48.56	28.81	27.40	1.0000	1.0516	2.92
10	19.75	47.23	48.65	28.90	27.48	1.0000	1.0518	2.91

Appendix 3.2

Table 3.2A: Density measurement for Acetic acid (concentration: 1.0 M)

Acetic acid 1.0M								
trial no.	m0 (g)	m3 (g)	m4 (g)	mL (g)	mH2O (g)	ρH2O (g/mL)	ρCH3COOH (g/mL)	pH
1	19.75	45.99	47.33	27.58	26.24	1.0000	1.0511	2.53
2	19.75	46.04	47.39	27.64	26.29	1.0000	1.0513	2.50
3	19.75	46.11	47.47	27.72	26.36	1.0000	1.0514	2.48
4	19.75	46.14	47.50	27.75	26.39	1.0000	1.0514	2.47
5	19.75	46.23	47.60	27.85	26.48	1.0000	1.0516	2.45
6	19.75	46.29	47.66	27.91	26.54	1.0000	1.0518	2.44
7	19.75	46.34	47.72	27.97	26.59	1.0000	1.0519	2.43
8	19.75	46.42	47.81	28.06	26.67	1.0000	1.0522	2.41
9	19.75	46.50	47.90	28.15	26.75	1.0000	1.0524	2.41
10	19.75	46.64	48.05	28.30	26.89	1.0000	1.0526	2.40

Appendix 4.0

Table 4.0A: Viscosity measurement for Acetic acid with various concentrations at different temperatures

Temperature (°C)	Viscosity constant	Time taken (s)	Viscosity (MPa.s) at 0.01M
20	0.2018	6.00	1.2110
25	0.2018	5.53	1.1150
30	0.2018	5.12	1.0340
35	0.2018	4.85	0.9784
40	0.2018	4.64	0.9369
45	0.2018	4.34	0.8751
50	0.2018	4.08	0.8228
55	0.2018	3.78	0.7636
60	0.2018	3.49	0.7043
70	0.2018	3.23	0.6511
80	0.2018	2.80	0.5646
90	0.2018	2.52	0.5081

Temperature (°C)	Viscosity constant	Time taken (s)	Viscosity (MPa.s) at 0.1M
20	0.2018	6.01	1.2130
25	0.2018	5.95	1.2003
30	0.2018	5.51	1.1125
35	0.2018	5.46	1.1012
40	0.2018	4.91	0.9913
45	0.2018	4.81	0.9715
50	0.2018	4.63	0.9342
55	0.2018	4.39	0.8853
60	0.2018	4.14	0.8346
70	0.2018	3.88	0.7825
80	0.2018	3.61	0.7293
90	0.2018	3.38	0.6824

Temperature (°C)	Viscosity constant	Time taken (s)	Viscosity (MPa.s) at 1.0M
20	0.2018	6.05	1.2211
25	0.2018	5.99	1.2089
30	0.2018	5.54	1.1176
35	0.2018	5.47	1.1036
40	0.2018	4.94	0.9963
45	0.2018	4.86	0.9812
50	0.2018	4.69	0.9468
55	0.2018	4.49	0.9065
60	0.2018	4.30	0.8687
70	0.2018	4.14	0.8345
80	0.2018	3.89	0.7854
90	0.2018	3.73	0.7526

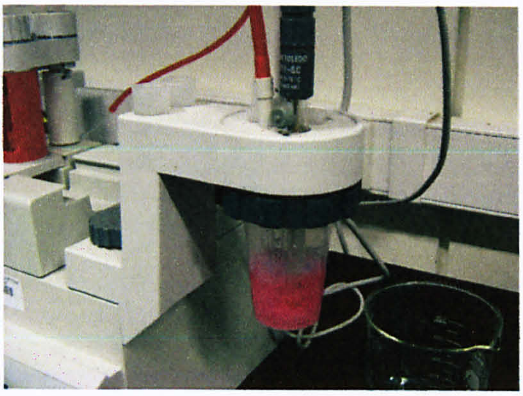
Appendix 5.0

Table 5.0A: Refractive Index measurement for Acetic acid with various concentrations at different temperatures

Temperature (°C)	Refractive Index at 1.0M	Refractive Index at 0.1M	Refractive Index at 0.01M
20	1.3748	1.3732	1.3715
25	1.3705	1.3692	1.3681
30	1.3686	1.3679	1.3668
35	1.3666	1.3652	1.3641
40	1.3649	1.3629	1.3611
45	1.3631	1.3618	1.3601
50	1.3612	1.3602	1.3589
55	1.3592	1.3579	1.3564
60	1.3574	1.3558	1.3546
70	1.3557	1.3542	1.3528
80	1.3538	1.3519	1.3512
90	1.3520	1.3513	1.3504

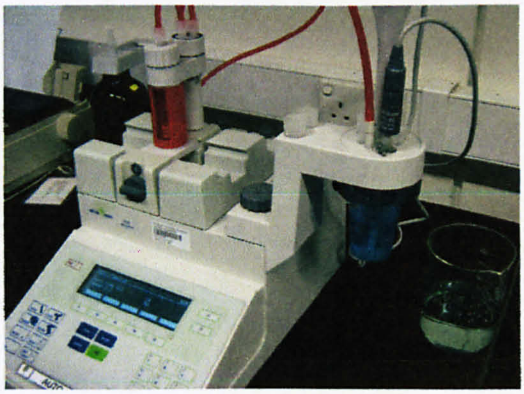
Appendix 6.0 Auto-titrator for Potentiometirc Titration

Appendix 6.1 Calibration of pH electrode



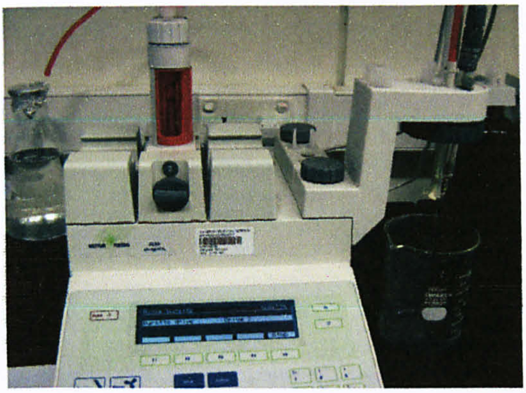
pH 4.00 buffer solution (pink)

pH 7.00 buffer solution (green)



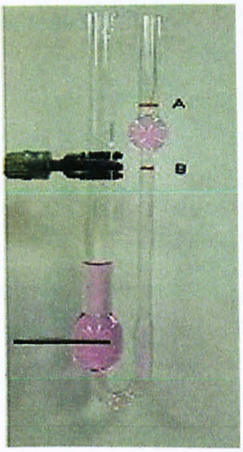
pH 9.21 buffer solution (blue color)

Appendix 6.2 Rinsing of burette with Potassium Hydroxide solution

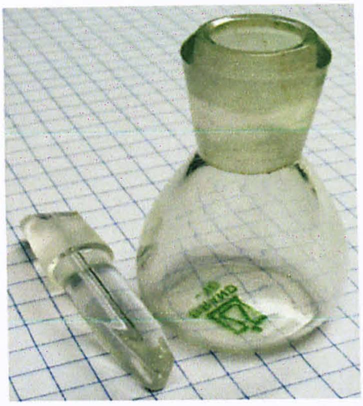


Appendix 6.3 Titration in progress

Appendix 7.0 Capillary Viscometer



Appendix 8.0 Density Pycnometer



Appendix 9.0 Refractometer