EXPERIMENTAL PROCEDURES

KI1201 - General Chemistry IIA &
KI1202 - General Chemistry IIB

GENERAL CHEMISTRY LABORATORY
INSTITUT TEKNOLOGI BANDUNG
2025

Table of Contents

REGULATIONS & INTRODUCTION TO GENERAL CHEMISTRY LABORATORY

Welcome to The General Chemistry Laboratory of ITB!

The chemistry lab is a great place to study and understand chemistry through experiments. Basically, chemistry is a descriptive/real science that studies change in natural phenomena. By conducting a chemistry lab in the laboratory, one is expected to better understand the phenomena that arise in chemical reactions, which so far can only be read or imagined while studying theory in lectures.

A chemistry laboratory is a place that is very different from other places because you will be dealing directly with a wide variety of substances (**including dangerous chemicals**) and a wide variety of equipment. However, it has been specially designed so that we can work safely, with conditions that we must understand and follow the laboratory regulations.

Below, we explain the regulations of working in the General Chemistry Laboratory and introduce some basic chemical laboratory equipment. Before you begin the practical course, you must first read, study, and understand these terms.

General Requirements

- The practicum is divided into 2 periods in 1 day, namely:
  1st period 08:00 - 12:00 WIB
  2nd period 13:00 - 17:00 WIB
- The General Chemistry Laboratory is located at The Labtek 1A building 1st floor and 2nd floor.
- Check your practicum schedule.

Chronological activity:

- Following a predetermined schedule, students are welcome to enter through the east door of the General Chemistry Laboratory on the first floor/ground floor in an orderly manner, may not wear sandals, don’t wear a t-shirt, and must immediately wear a laboratory coat, identification card, and protective glasses.
- **Scan the barcode** on your ID Card immediately when entering the laboratory before the practicum begins to record your attendance.
- Collect practicum notebooks (practicum journals) on the table provided.
- Immediately gather at the front in the General Chemistry Laboratory on the first floor/second floor for listening to explanations from the practicum leader while carrying practicum guides and other notebooks (if needed)
- After that, the students return to each respective workplace. Each student will work individually under the supervision of an assistant.
- Before starting the practicum, check the equipment that has been provided, the number and integrity of the equipment, and whether it is under the existing "tool inventory list". If not, complete it immediately by asking the laboratory staff. If it matches, don't forget to sign the receipt of each inventory form. Learn the names, uses, and how to use the equipment (see basic chemical laboratory equipment).
- Students will be divided into several groups, each of which will be led or supervised by an assistant. Based on several considerations, the assistant will arrange the implementation of the work. The assistant's name must be recorded in the notebook. Groups will be announced before the first practicum is held.
- Practicum notebooks (practicum journals) must be done before the practicum begins (DO NOT work around the laboratory) and must be brought during the practicum. If this assignment is not made, the student is not given value for the experiment or is not allowed to take part in the practicum.

- Aspects assessed from the implementation of the experiment include readiness, skills, answers to questions/discussions given by assistants, neatness, and arrangement of workplaces, ability to work independently, correctness/honesty in recording data, obedience to instructions or regulations, and mastery of practical material. Observations are immediately recorded in a notebook. Other data can be asked of the assistant or practicum leader.
- After finishing work, wash each practicum equipment and it will be examined by laboratory staff.
- At the end of the practicum period, a practicum test will be carried out for around 15-20 minutes. Make sure the equipment has been washed and the table has been cleaned before the practical test is carried out.
- The staff will record the shortage or breakdown of the equipment, witnessed by the student, ending with affixing the signature.
- Students must sign the handover of this equipment. Do not leave the laboratory before the staff signs your equipment inventory list.

NOTE: For certain experiments, you will be asked to make a FULL practicum REPORT. In addition to working individually, students are also trained to work in groups. Under these circumstances, the responsibility for the success of the experiment is shared. The same goes for shared equipment. If there is damage or loss, it must be shared. ENJOY YOUR WORK!

Practicum Handbook (Practicum Module), Practicum Notes (Journal of Practicum) & Reports

- Each practicum has its practicum manual. Complete with a practicum notebook and writing materials. Keep the notebook on the desk but safe enough, do not get splashed with substances or damaged.
- The practicum manual consists of a code of conduct, work and safety regulations, and experimental modules 1 to 5.
- Each experiment will consist of module title, introduction, materials and equipment, how it works, and practicum preparation assignment questions (if any).
- Practical notes to be made include:
  - Day and date
  - Assistant name
  - Module number and the module title
  - The purpose of the experiment
  - The principle of the experiment
  - Tools and materials
  - Material safety data sheet (brief data regarding the nature and method of handling the chemicals to be used)
  - Workflow chart and observation column
  - Calculations/formulas used in the experiment
  - Discussion Column
  - Bibliography
- Each experiment will be equipped with a data sheet (which will contain observations and be signed by the assistant concerned) and a practicum test sheet. These sheets will be distributed during the practicum and when the practicum test is carried out (usually at the end of the practicum time).

Safety Regulations

• General Regulations

- Before working in the laboratory, prepare thoroughly regarding the regulations in the laboratory and master the practicum material as well as possible, starting from the objectives, basic concepts, procedures and working techniques to be carried out.
- Do not work alone in the laboratory, at least together, and for general chemistry, practicum must be accompanied by a laboratory assistant or instructor, according to the schedule provided.

- In the laboratory room, it is not allowed: smoking, eating, and drinking. Required to wear neat clothes (not t-shirts), wear a long-sleeved lab coat that meets the requirements, and wear closed shoes (not sandals). This is for the safety and health of your work.
- Always maintain the cleanliness of the worktable, sink, and surroundings. Throw garbage in its place.
- If you throw away concentrated liquid, pour it into the sink while pouring lots of water. Be careful with concentrated H_2SO_4, it has its way.
- Dispose of solids and metals into the provided container (do not throw them into the sink)!
- Solutions containing heavy metals (such as Pb, Cd, Cu, Cr, Hg, Ag, As, Zn, and Ni) must be disposed of in a separate container/bottle provided. Never throw it in the sink!
- When working with gases or smoky/concentrated substances, work in a fume hood, and do not inhale toxic gases. Never leave a running experiment, wait for the process to stop.
- Chemistry Laboratory is a seriously special place to study and work. Do not chat, joke, or play games with friends. Don't waste your time.
- Work diligently, be confident, and do not hesitate. Record every incident and experimental observation thoroughly and carefully, because one of the most important activities in practicum is observation and data collection. Don't hesitate to ask the assistant and answer any questions the assistant asks briefly and clearly.

• Tackling accidents/fires

- Accidents are unexpected events. However, the laboratory is a place with many hazards, both poisoning and fire hazards. If an accident or fire occurs, the first and foremost thing to do is: DON'T PANIC!
- If your skin is exposed to chemicals, wash it immediately with tap water and use laundry soap. If it hits your eyes or face, spray directly with tap water over the sink. Never rub your hands, especially before washing your hands. Immediately contact the officer/assistant to ask for emergency treatment.
- If the limbs are affected, especially in large numbers, use a large shower or tap water, and immediately remove the laboratory gown or other covering on the part affected by the substance. Immediately report to the officer to get further treatment.
- If there is a fire on the worktable, for example, a solution in a beaker, first of all, don't panic, don't try to extinguish it yourself, or slam the burning glass. Get away from the table and immediately report to the officer/assistant. If nothing helps, cover the glass that is burning with a wet cloth or wet mat, let it die alone, or spray it with an existing fire extinguisher.
- If the hands or skin are burned (small amount), keep ice water around the burn, then treat it with an analgesic drug, for example, an ointment or rivanol solution. Ask the clerk/assistant.

• Chemicals & Reagents

- Chemical substances and reagents needed for this General Chemistry Practicum are generally provided.
- If the user is left to each student, then these substances and reagents will be stored on a special table for this. Usually placed on the side tables of the laboratory near the window.
- Every student is MANDATORY to maintain the cleanliness of the table of this substance, and the most important thing is to keep the reagents from being damaged or contaminated due to careless taking. For example, incorrectly using a pipette to take a substance. Each reagent is equipped with its pipette (pipettes may not be exchanged), or if the reagent bottle does not have a pipette, it means taking it by pouring it into a measuring cup.
- If you are going to do a reaction test, bring a clean test tube on the test tube rack to the reagent table. Mixing is done here as well, with notes that you have to work in an orderly manner, find an empty place, and don't mix the dropper mixture.
- For each bottle of substance and reagent, there is a clear label containing the name, chemical formula, and concentration or another identity. Read it carefully before you use it. It is not allowed to exchange bottle caps.
- Concentrated chemicals such as HCl, H_2SO+, and NaOH, must be stored in a fume cupboard. When working with these substances, it must be done in a fume hood.
- Use special laboratory gloves, for example, latex or nitrile gloves, when working with materials that are irritating to the skin.

Basic Techniques in The Laboratory

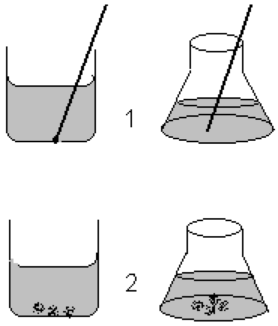
Basic Chemical Laboratory Equipment

Simple laboratory equipment commonly used in the General Chemistry Laboratory generally consists of glassware which is often used and is indispensable as a means and aids to conduct (simple) experiments. Some of the equipment commonly used in laboratories are:

- **Beaker glass**, various sizes which are written on the outside, this size is in accordance with the holding capacity. Used to hold liquids or solutions, as well as heat them, are made of strong glass heating materials such as Pyrex.
- **Erlenmeyer Flask**, like a beaker, because it is in the form of an Erlenmeyer flask, it can be used to stir liquids through shaking, as well as to perform titrations. For this titration there is a flask called the titration flask, which looks like an Erlenmeyer only with a wider neck.
- **Graduated cylinder**, to measure the volume of liquid contained in it (size), also consists of various sizes/capacities.
- **Pipette**, to measure the volume of liquid that we take or need. There are several types, the first type is volumetric pipette that can only take a certain amount of liquid (correctly), the second type is graduated measuring pipette that can adjust the volume (with care) of the liquid we take, the third type is medicine dropper (Pasteur pipette) which can take a small amount of liquid.
- **Burette**, just like a sized pipette, only because the burette has a valve to regulate the discharge of the liquid, we don't need to read the size every time. This tool is used to perform titrations.
- **Test tubes**, made of glass, of various sizes that indicate their capacity, are used to carry out chemical reactions in small amounts.
- **Watch glass**, made of clear glass, varying in diameter, used for simple reactions or evaporation
- **A funnel**, made of glass or porcelain, is used to filter by gravity, there are long and short rods.
- **The Buchner funnel**, also a type of funnel made of porcelain, the difference is that this funnel is used for rapid filtration by means of suction through a vacuum, also equipped with a suction flask. Widely used in organic chemistry laboratories.
- **A separating funnel**, made of glass, is used to separate two or more layers of liquid, in the way of extraction separation.
- **The evaporating dish**, made of porcelain, of various capacities, is used to evaporate the solution.
- **A crucible**, like a porcelain cup, only has a higher size, is used for evaporation followed by an annealing of the solid.
- **Spatulas**, of various sizes, made of iron and glass, are used to extract solids.
- **A stirring rod**, made of glass, is used to stir the solution in the flask.
- **Wire gauze/screen with asbestos center**, a wire coated with asbestos, is used to hold and spread the heat that comes from a Bunsen fire.
- **Tripod stand**, made of iron that supports the ring, is used for heating.

➢ How to Heat Liquid/Solution

In general you have to be very familiar with the aspect of security which includes the workplace, equipment, substances, people around and of course yourself. The problem is how to heat the liquid to be safe? One thing that should be avoided as far as possible in heating the liquid is bumping (sudden bubbling).



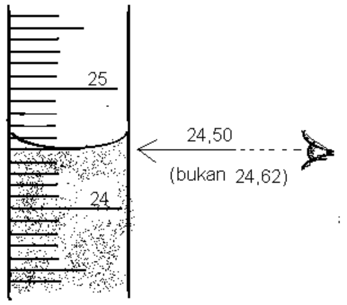
a) Heating the liquid in a test tube:

- Do not point the test tube mouth at your neighbors or yourself!
- Clamp the tube near its mouth!
- Tilt in a safe direction, heat it while shaking it intermittently.
- Keep mixing for a while after the fire is put away/not heated again.

b) Heating the liquid in a beaker or Erlenmeyer, must use: (1) **<u>stirring rod</u>**; or (2) **<u>boiling stone.</u>**

For heating using an Erlenmeyer flask, it can be done by heating directly over the fire (for a non-flammable solvent), while the liquid is shaken/rotated, occasionally removed when it feels like it will boil.

➢ How to read volume (Graduated cylinder)



A graduated cylinder or measuring flask is a tool for measuring the amount of liquid contained in it. Therefore the 0 scale (in milliliters, mL) will be at the bottom. Enter the amount of liquid that will be measured in volume, then right with a dropper to the desired scale. What is important here is how to read the scale to be read tangent to the scale with the bottom of the fluid meniscus. Meniscus is a curved line (for water to be concave) the surface of the liquid due to the adhesion force or cohesion of the liquid with the glass. In the example image, it reads **24.50 mL** instead of **24.62 mL**.

➢ How to use a pipette

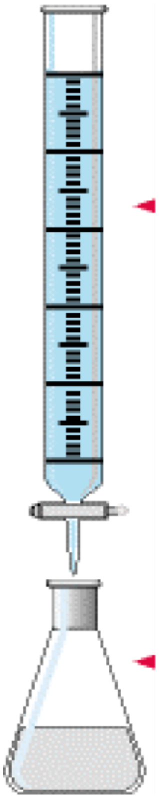
A pipette is a tool for moving a certain amount of substances from one place to another. In general, there are 3 types of pipettes, namely a drop pipette, a volumetric pipette and a measuring pipette.

Dropper drops, are used to help certain areas where the volume is out of control. Rubber is used to extract fluids. The difference between a pipette is determined by the tip of the pipette, some are tapered or long (capillary), some are large (ordinary).

A pipette the size or also called a goiter pipette, the size of which is printed on the surface of the glass, is used to move **a certain volume (with care)** of liquid. How to use a sized pipette: dip the bottom of the pipette into the liquid (until submerged), then the liquid is sucked in with **a rubber aspirator** (see picture) until it exceeds the boundary line, showing that it should not open and then move to another place while the tip of the pipette is stuck to the glass. Do not remove the remainder at the tip of the dropper. **Note:** for practice, suction is done by mouth - not to drink it - then when holding the fluid used forefinger, not thumb.

Pipette sized, used to move **a certain volume** (with care) of liquid. As the name implies, this pipette has a size scale where 0 is at the top (the hand). The way it works is in accordance with the size, this bed pipette is filled until it is exactly on a scale of 0, then issue with the index finger, and the permit to be issued must be adjusted at the speed so that the volume issued is according to what is needed.

➢ How to use a burette



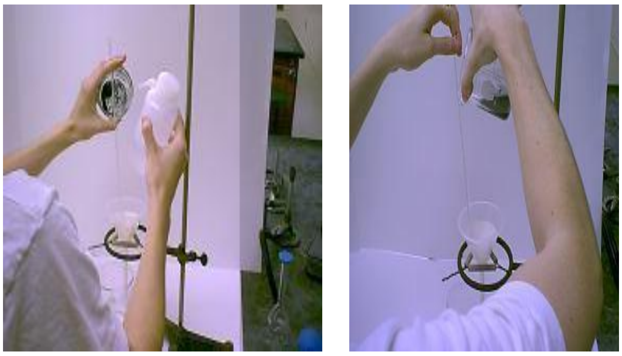
Burette, is a special tool in chemical laboratories because in terms of use it is a combination of all pipettes, in fact it has advantages over sized pipettes because at the time they are issued they do not need to be monitored for the scale. The tool used to carry out titration work, namely how to determine the concentration of a solution with another solution whose concentration has been known, with the equivalent method, for example acid-base or redox. To see that the equivalence point has been reached, an indicator substance is used, which is usually dye such as phenolphthalein. For this titration work, a tool is needed to be able to accurately measure the amount of solution that has been removed, without having to read every expenditure. Which is used, because this tool has a volume size (mL) and for its removal a faucet that regulates the settings is used.

How to prepare the burette: the inside of the burette pipe must be clean and free of grease, for which special washing is required. The faucet is closed by storing the liquid/solution from above through a glass funnel. Pay attention to whether the faucet is leaking, if it leaks, it must be opened and smeared with a little Vaseline. Fill it until it exceeds a scale of 0, then by opening the tap slightly on the surface of the meniscus the liquid offends the 0 mL line (at the top of the burette).

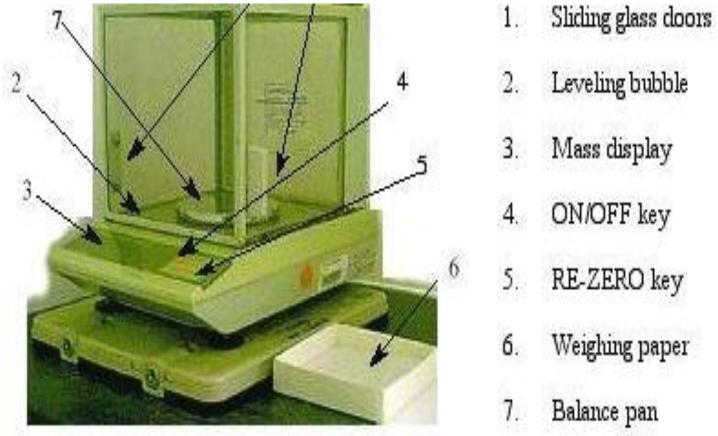
How to use a burette (in a titration): **prepare a flask that has been filled with a certain value of solution to which the concentration will be determined, as well as two or three indicator drops, under the burette valve. Hold the burette tap with the left hand (not the right hand) where the palm of the hand grasps all the faucets, and the index-thumb can turn the faucet from the inside. The titration flask is held by the neck with the right hand. While shaking the bottom of the titration flask, the burette valve is opened slowly until it stops the equivalence point. If it is near the equivalence point, spend a little until just before the indicator color changes, because even half a drop will be very meaningful in determining the end point of the titration.**

➢ How to Perform Filtering

Filtering is a method for separating and purifying a mixture. A good filtering method will produce a good product. In many Chemical experiments, the separation and purification step is an important one. Therefore, the skill of doing screening is something that must be mastered by the practitioner. The equipment that must be prepared includes a filter funnel and filter paper. There are several types of filter funnels, but the ones that are commonly used for ordinary filtering are the **funnel** and **the Buchner funnel** (see picture beside). There is also another type of funnel called **a separatory funnel**, which is commonly used for separation by extraction methods instead of ordinary filtering. How to fold the filter paper will determine whether the filtering process is good or not. Try not to have the filter paper size bigger than the size of the funnel.



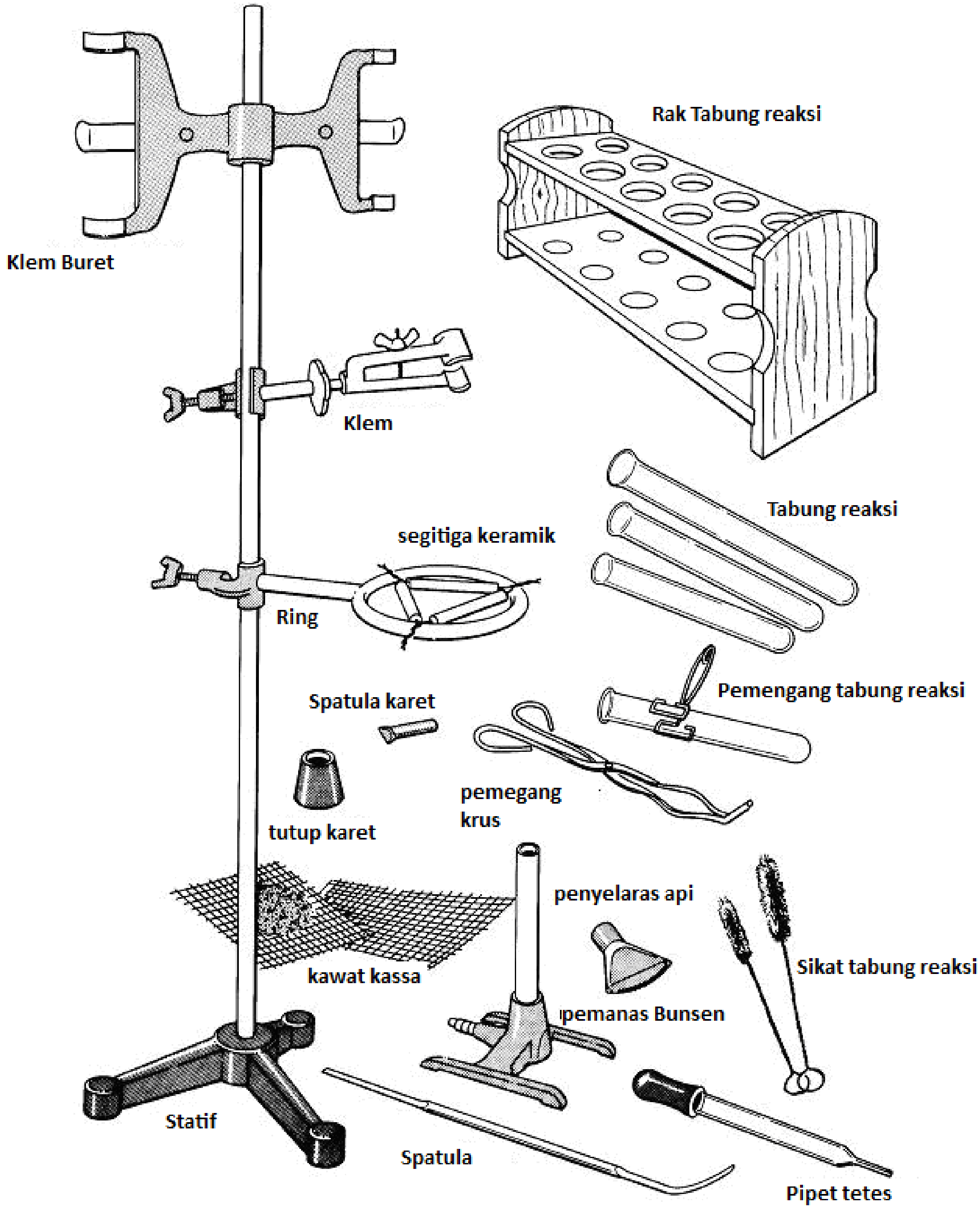
Balance or scales is a tool for measuring mass or weight. The working principle is the balance between two discs. The type of balance is generally determined by the sensitivity and accuracy of weighing, the technical balance is 0.01 to 0.001 grams, while the analytical balance is <0.0001 gram. Technically, the balance is now divided into two types, namely: triple-beam balance (swing, image on the top and bottom side) and top-loader balance (torque), and the reading is electric or digital (figure below).

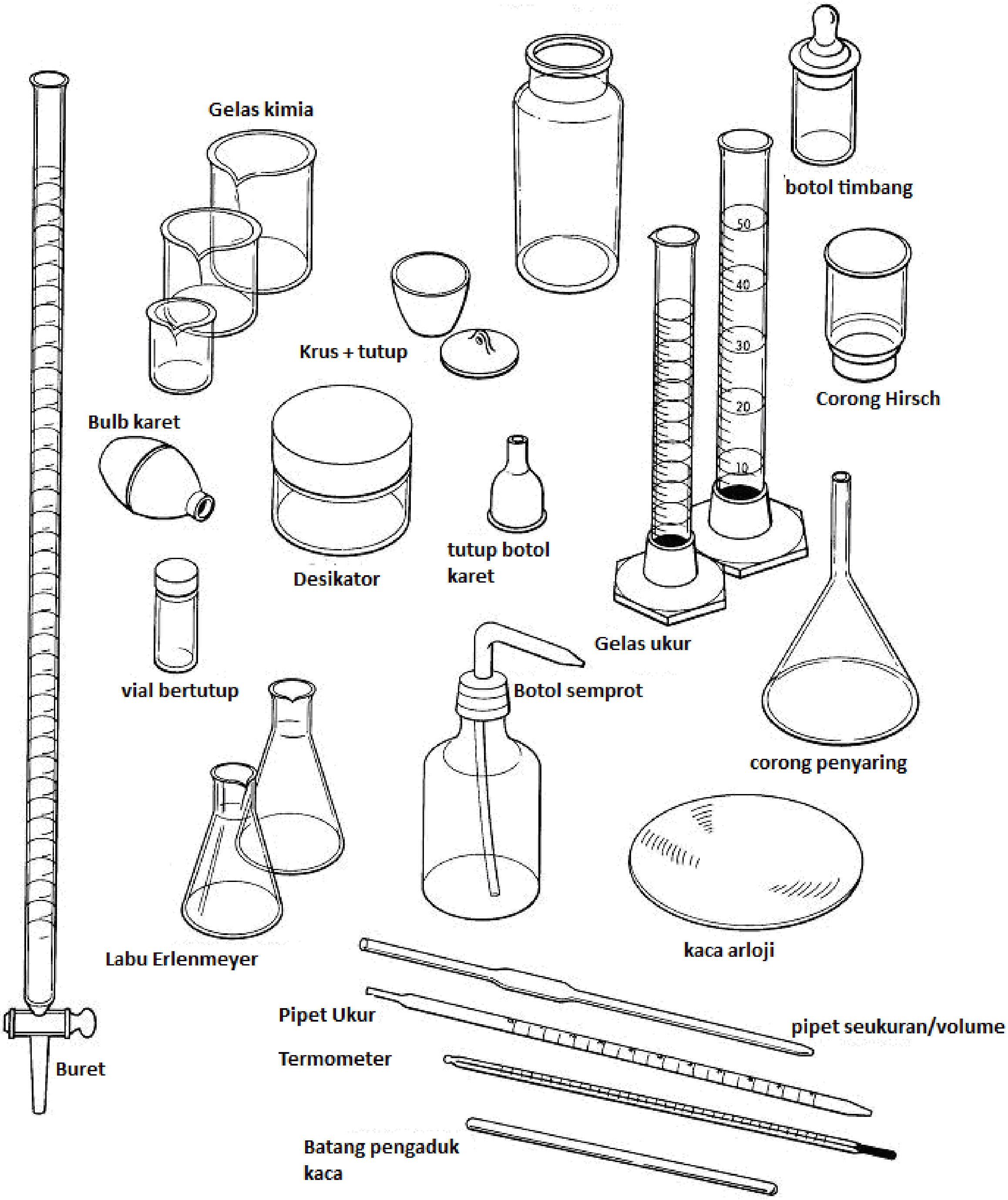


The basic principle of weighing:

1. Prepare a balance sheet in a state/position of balance/load is empty, meaning that the balance is zeroed first.
2. Save the object you want to weigh in the left arm of the balance, and the right arm to place the weights.
3. Restore the balance by keeping the weights on the right. Systematics of balancing starts with large weights approaching the weight of the object, continues with smaller weights and so on.

Chemical Laboratory General Equipment




Gelas kimia
botol timbang
50
40
30
20
10
Corong Hirsch
Krus + tutup
Bulb karet
Desikator
tutup botol karet
Gelas ukur
vial bertutup
Botol semprot
corong penyaring
Labu Erlenmeyer
kaca arloji
Pipet Ukur
pipet seukuran/volume
Termometer
Buret
Batang pengaduk kaca

Experiment I Chemical Kinetics

Introduction

The chemical reaction proceeds at a specific rate. The reaction rate can be determined by how long it takes for the reaction to complete. Some reactions required a very short time to reach, which was technically difficult to measure. However, a number of organic and inorganic reactions proceed at measurable rates within a certain temperature range, which makes it possible to study its kinetics, including its reaction rate, factors that affect the reaction rate, and its mechanism. The reaction rate can be considered as a decrease in the amount of reactants or an increase in the amount of products. Generally, the rate of a reaction depends on the concentration of its substrates, temperature at which the reaction is conducted, presence of the catalyst, and physical form of the reactants.

The reaction rate can be expressed as a function of the concentration of a substance participating in a chemical reaction. This function is commonly known as the rate law or rate equation of a reaction. The rate law can only be determined precisely through experiments; it is not deductible solely through its chemical equation.

A chemical reaction may depend on the concentration of one or more of its reactants, which is expressed as the reactant's concentration raised to a power, commonly known as its order. The total order of the reaction is defined as the sum of the orders of each reactant.

For the reaction $A + 2B \rightarrow 3C + D$, the rate law may be defined as:

$$r = -\frac{d[A]}{dt} = -\frac{1}{2}\frac{d[B]}{dt} = +\frac{1}{3}\frac{d[C]}{dt} = +\frac{d[C]}{dt} = k \cdot [A]^n \cdot [B]^m$$

where:

t = time, k = rate constant, n = rate order for A's concentration, m = rate order for B's concentration, $-\frac{d[A]}{dt}$ and $-\frac{d[B]}{dt}$ as the reaction rate with respect to A and B (reactants) with negative sign indicating that the amount of A and B are decreasing, $+\frac{d[C]}{dt}$ and $+\frac{d[D]}{dt}$ as the reaction rate with respect to C and D (products) with positive sign indicating that the amount of C and D are increasing.

A chemical reaction will occur when 1) molecules of the reactants collide at the right orientation, and 2) when the activation energy is reached. Activation energy is the amount of energy required to overcome steric effect and to start breaking bonds of the reactant's molecule. It is needed to transform the reactants into an activated complex (transition state). Activation energy may be symbolized by $\Delta H^{\ddagger}, \Delta H^{*}, E_a$, or when it is seen as a function of free energy, $\Delta G^{\ddagger}$ and ΔG^{*} may also be used. The Arrhenius equation is used to correlate activation energy to rate constant (k):

$$k = Ae^{-\frac{E_a}{RT}}$$

The equation may also be expressed using its logarithmic form:

$$\ln k = -\frac{E_a}{RT} + \ln A$$

with R being the universal gas constant $8.314\ Jmol^{-1}K^{-1}$ and T being the absolute temperature in kelvins (K).

For this experiment, the rate law for the reaction between persulfate ion ($S_2O_8^{2-}$) and iodide ion (I^-) will be determined, by calculating the reaction orders and its rate constant. Complex reaction mechanism and catalyzed hydrogen peroxide decomposition will also be observed. Determination of activation energy for a reaction will also be observed by correlating reaction rate to temperature.

Persulfate and iodide ions will undergo redox reaction to produce sulfate and triiodide ions as follows:

$$S_2O_8{}^{2-}{}_{(aq)} + 3\,I^-{}_{(aq)} \rightarrow 2\,SO_4{}^{2-}{}_{(aq)} + I_3{}^-{}_{(aq)} \qquad (1)$$

The dissolved iodine, in the form of triiodide ion, will react subsequently with a known amount of thiosulfate ion ($S_2O_3^{2-}$), regenerating iodide ion.

$$2\,S_2O_3{}^{2-}{}_{(aq)} + I_3{}^-{}_{(aq)} \rightarrow S_4O_6{}^{2-}{}_{(aq)} + 3\,I^-{}_{(aq)} \qquad (2)$$

The thiosulfate ion is added to:

- react with the produced triiodide ion, so that if reaction (1) is in equilibrium state, the reverse reaction is prevented and the regeneration of iodide ions will shift the equilibrium towards product formation.
- eliminate free iodine in the solution. When there are thiosulfate ions in the solution, there will be no free dissolved iodine. Once all thiosulfate ions have reacted, the subsequently-formed free iodine will form a blue-black complex with starch in the solution.

For each runs, there will be different amounts of $S_2O_8^{2-}$ and I^- ions, along with identical amount of $S_2O_3^{2-}$ and starch solutions. Since the amount of thiosulfate ion is identical for every run, the amount of triiodide ions formed before the solution turns blue is also identical. The time spent between mixing the reactants together to the moment the solution turns blue is considered as the time it takes to complete the reaction, thus be used to measure reaction rate, which is inversely proportional to the time measured.

Chemicals and Equipment

The following chemicals are needed for this experiment:
$Na_2S_2O_3$ 0,01 M; KI 0,40 M; KI 0,20 M; KI 0,10 M; KI 0,05 M; $(NH_4)_2S_2O_8$ 0,40 M; $(NH_4)_2S_2O_8$ 0,20 M; $(NH_4)_2S_2O_8$ 0,10 M; $Cu(NO_3)_2$ 0,1 M; Fe^{3+} 0,05 M; $S_2O_3^{2-}$ 0,1 M; and starch solution.

The following equipment are needed for this experiment:
Standard glassware, thermometer, stopwatch, 10 mL volumetric pipette, 25 mL volumetric pipette, pipette pump and electric heater.

Procedures

Part 1. Rate Law Determination for the Reaction between $S_2O_8^{2-}$ and I^-

Part 1.1 The effect of I^- Concentration on Reaction Rate

For this part, the concentration of $S_2O_8^{2-}$ is made constant while I^- varies.

1. Run 1. Take 5,0 mL of the starch solution using a cylinder, pours it into 250 mL beaker glass. Add 10 mL of $Na_2S_2O_3$ 0,01 M solution using graduated pipette or burette. Add 25 mL of KI 0,40 M solution, measured using volumetric pipette or burette. Stir the solution.
2. Take 25 mL of $(NH_4)_2S_2O_8$ 0,2 M using volumetric pipette into dry 150 mL beaker glass.
3. Equalize the temperature for both solution using a water bath.
4. Pour the $(NH_4)_2S_2O_8$ solution to KI-$Na_2S_2O_3$-starch mixture, stirring immediately until the solution turns blue. Measure the elapsed time between the moment of mixing to the moment the whole solution turns blue, and also the final temperature.
5. Repeat step 1-3 using all the available KI solution (0,20 M; 0,10 M; 0,4 M). Run 2-4.

Summary for the solution composition for run 1-3 in part 1.1:

Run	**Concentration of Reactants**	
	KI (M)	**$(NH_4)_2S_2O_8$ (M)**
1	0,40	0,20

2	0,20	0,20
3	0,10	0,20

Part 1.2 The effect of $S_2O_8^{2-}$ Concentration on Reaction Rate

For this part, the concentration of I^- is made constant while $S_2O_8^{2-}$ varies.

1. Run 1. Take 5,0 mL of the starch solution, then pours it into 250 mL beaker glass. Add 10 mL of $Na_2S_2O_3$ 0,01 M solution, measured using graduated pipette or burette. Add 25 mL of KI 0,20 M solution, measured using volumetric pipette or burette. Stir the solution.
2. Take 25 mL of $(NH_4)_2S_2O_8$ 0,4 M using volumetric pipette into dry 150 mL beaker glass.
3. Equalize the temperature for both solutions using a water bath.
4. Pour the $(NH_4)_2S_2O_8$ solution to KI-$Na_2S_2O_3$-starch mixture, stirring immediately until the solution turns blue. Measure the elapsed time between the moment of mixing to the moment the whole solution turns blue, and also the final temperature.
5. Repeat step 1-3 using all the available $(NH_4)_2S_2O_8$ solution (0,20 M; 0,10 M; 0,4 M). Run 2-4.

Summary for the solution composition for run 1-3 in part 1.1:

Run	**Concentration of Reactants**	
	KI (M)	**$(NH_4)_2S_2O_8$(M)**
1	0,20	0,40
2	0,20	0,20
3	0,20	0,10

Part 1.3 The Effect of $Cu(NO_3)_2$ Addition Towards Reaction Rate

For this part, the concentration of $S_2O_8^{2-}$ is made constant while I^- varies.

1. Run 1. Take 5,0 mL of the starch solution, then pour it into 250 mL beaker glass. Add 10 mL of $Na_2S_2O_3$ 0,01 M solution, measured using graduated pipette or burette. Add 25 mL of KI 0,40 M solution, measured using volumetric pipette or burette. Add 1 drop of $Cu(NO_3)_2$ 0,10 M solution. Stir the solution.
2. Take 25 mL of $(NH_4)_2S_2O_8$ 0,2 M using volumetric pipette into dry 150 mL beaker glass.
3. Equalize the temperature for both solutions using a water bath.
4. Pour the $(NH_4)_2S_2O_8$ solution to KI-$Na_2S_2O_3$-starch mixture, stirring immediately until the solution turns blue. Measure the elapsed time between the moment of mixing to the moment the whole solution turns blue, and also the final temperature.
5. Repeat step 1-4 using all the available KI solution (0,20 M; 0,10 M; 0,05 M). Run 2-4. Remember to add $Cu(NO_3)_2$ to KI solutions.

Summary for the solution composition for run 1-3 in part 1.1:

Run	**Concentration of Reactants**	
	KI (M)	**$(NH_4)_2S_2O_8$ (M)**
1	0,40	0,20
2	0,20	0,20
3	0,10	0,20

Part 2. Activation Energy Determination for the Reaction between Fe^{3+} and $S_2O_3^{2-}$

1. Prepare 3 250 mL beaker glasses. Fill glass 1 with room-temperature water, glass 2 with ±45 °C water, and glass 3 with ±65 °C water.

2. Prepare 3 reaction tubes, then put 2 mL of Fe^{3+} 0,05 M solution into each one. Label them as tube 1, 2, and 3. Put tube 1 to beaker glass 1, tube 2 to beaker glass 2, and tube 3 to beaker glass 3.
3. Prepare 3 reaction tubes, then put 2 mL of $S_2O_3{}^{2-}$ 0,10 M solution into each one. Label them tube A, B, and C. Put tube A to beaker glass 1, tube B to beaker glass 2, and tube C to beaker glass 3.
4. Let all the tubes sit inside the beaker glass for a while, to allow the solution's temperature to equalize
5. with the water's temperature. Once the tubes are ready, measure exactly the final temperature of the water in beaker glass.
6. This experiment is to be done in a team of 2 people. One person pours the solution in tube A to tube 1, while the other start the stopwatch exactly when the two solutions are mixed. Stop the stopwatch when the mixture turns from purple to colorless. Note down the elapsed time.
7. Repeat step 5 for tubes 2 and B (45 °C), and tubes 3 and C (65 °C).

Data Processing and Calculation

Part 1. Rate Law Determination for the Reaction between $S_2O_8{}^{2-}$ and I^-

A reaction's rate is inversely proportional to the time elapsed:

$$rate = r = k.\frac{1}{t}$$

A reaction's rate can also be expressed as its rate law, in the case of this reaction we can express it as:

$$r = k \cdot [I^-]^x \cdot [S_2O_8{}^{2-}]^y$$

For two experiments in which the concentration of $S_2O_8{}^{2-}$ is made constant, and the concentration of I^- in increased by two-fold:

$$[I^-]_2 = 2[I^-]_1$$

$$[S_2O_8{}^{2-}]_1 = [S_2O_8{}^{2-}]_2$$

$$\frac{r_2}{r_1} = \frac{k \cdot [I^-]_2{}^x \cdot \left[S_2O_8{}^{2-}\right]_2{}^y}{k \cdot [I^-]_1{}^x \cdot \left[S_2O_8{}^{2-}\right]_1{}^y}$$

$$\frac{r_2}{r_1} = \frac{(2[I^-]_1)^x \cdot [S_2O_8{}^{2-}]_1{}^y}{[I^-]_1{}^x \cdot [S_2O_8{}^{2-}]_1{}^y} = \left(\frac{2[I^-]_1}{[I^-]_1}\right)^x = 2^x$$

$$\log\left(\frac{r_2}{r_1}\right) = x \log 2$$

Thus, we can look for x, which is the reaction order in respect to the concentration of I^-, by using the mathematical derivation above. Should the value for r_2 be twice bigger than r_1, then the order for I^- would be 1; should the value for r_2 be 4 times bigger, then the order for I^- would be 2.

After calculating the value for y (the reaction order in respect to the concentration of persulfate ion), the rate constant can then be calculated:

$$k = \frac{r}{[I^-]^x[S_2O_8{}^{2-}]^y}$$

with $[I^-]$ and $[S_2O_8{}^{2-}]$ being the initial concentration of each reactant by the time of mixing.

The reaction rate for each run can be approached using the following equation, which considers the average reaction rate instead of the instantaneous reaction rate:

$$r = -\frac{\Delta[S_2O_8{}^{2-}]}{\Delta t}$$

For this experiment, the value of $\Delta[S_2O_8{}^{2-}]$ is kept constant for all runs by setting $Na_2S_2O_3$, the reagent for the subsequent reaction, as the limiting reagent for all reactions.

$$S_2O_8{}^{2-}{}_{(aq)} + 3\,I^-{}_{(aq)} \rightarrow 2\,SO_4{}^{2-}{}_{(aq)} + I_3{}^-{}_{(aq)} \qquad (1)$$

$$2\,S_2O_3{}^{2-}{}_{(aq)} + I_3{}^{-}{}_{(aq)} \rightarrow S_4O_6{}^{2-}{}_{(aq)} + 3\,I^{-}{}_{(aq)} \qquad (2)$$

The solution only turns blue when all $S_2O_3^{2-}$ ions have reacted completely with the triiodide ions. According to equation (2), 1 mole of I_3^- is reacted with 2 moles of $S_2O_3^{2-}$ ions, while according to equation (1) 1 mole of I_3^- produced from 1 mole of $S_2O_8^{2-}$. Therefore, for every 2 moles of $S_2O_3^{2-}$ used, 1 mole of $S_2O_8^{2-}$ is consumed, and we can calculate the amount of $S_2O_8^{2-}$ that reacted by calculating the amount of $S_2O_3^{2-}$ used. Every run uses 10 mL of $Na_2S_2O_3$ 0,01 M, which equates to $1{,}0 \times 10^{-4}$ mol of $S_2O_3^{2-}$ ions in the solution. Hence, to calculate the amount of $S_2O_8^{2-}$ used:

$$n_{S_2O_8{}^{2-}} = \frac{1}{2} \times n_{S_2O_3{}^{2-}} = \frac{1}{2} \times 1{,}0 \times 10^{-4}\,mol = 5 \times 10^{-5}\,mol$$

The concentration used to calculate the reaction rate is the concentration after mixing, which means the total volume of the solution is needed.

$$V_{total} = V_{KI} + V_{Na_2S_2O_8} + V_{Na_2S_2O_3} + V_{starch} = 25\,mL + 25\,mL + 10\,mL + 5\,mL = 65\,mL$$

$$V_{total} = 6{,}5 \times 10^{-2}\,L$$

Thus, the concentration changes for $S_2O_8^{2-}$ is:

$$\Delta[S_2O_8{}^{2-}] = \frac{n_{S_2O_8{}^{2-}}}{V_{total}} = \frac{5 \times 10^{-5}\,mol}{6{,}5 \times 10^{-2}\,L}$$

Afterwards, the reaction rate can be calculated.

Part 1.1 The effect of I^- Concentration on Reaction Rate

1. Calculate the initial concentration of KI in the reaction mixture (under assumption that at t = 0 s no reaction has occurred):

   $$[KI]_1 \times V_{total} = [KI]_{i,1} \times V_{KI}$$

   $[KI]_1$ = KI concentration AFTER mixing for run 1
   $[KI]_{i,1}$ = initial KI concentration used in run 1 BEFORE mixing
   V_{KI} = the volume of KI added to the mixture

2. Calculate the initial concentration of $(NH_4)_2S_2O_8$ in the reaction mixture (under assumption that at t = 0 s no reaction has occurred):

   $$[(NH_4)_2S_2O_8]_1 \times V_{total} = [(NH_4)_2S_2O_8]_{i,1} \times V_{(NH_4)_2S_2O_8}$$

   $[(NH_4)_2S_2O_8]_1$ = $(NH_4)_2S_2O_8$ concentration AFTER mixing for run 1
   $[(NH_4)_2S_2O_8]_{i,1}$ = initial $(NH_4)_2S_2O_8$ concentration used in run 1 BEFORE mixing
   $V_{(NH_4)_2S_2O_8}$ = the volume of $(NH_4)_2S_2O_8$ added to the mixture

3. Calculate the reaction rate for each run:

   $$r_1 = -\frac{\Delta[S_2O_8{}^{2-}]}{\Delta t_1}$$

4. Calculate the reaction order with respect to I- concentration (x) using data from run 1 and 2, 2 and 3

   $$\frac{r_2}{r_1} = \frac{k \cdot [KI]_2{}^{x} \cdot [(NH_4)_2S_2O_8]_2{}^{y}}{k \cdot [KI]_1{}^{x} \cdot [(NH_4)_2S_2O_8]_1{}^{y}} \qquad x_{1,2}$$

   $$\frac{r_3}{r_2} = \frac{k \cdot [KI]_3{}^{x} \cdot [(NH_4)_2S_2O_8]_3{}^{y}}{k \cdot [KI]_2{}^{x} \cdot [(NH_4)_2S_2O_8]_2{}^{y}} \qquad x_{2,3}$$

   $$\frac{r_4}{r_3} = \frac{k \cdot [KI]_4{}^{x} \cdot [(NH_4)_2S_2O_8]_4{}^{y}}{k \cdot [KI]_3{}^{x} \cdot [(NH_4)_2S_2O_8]_3{}^{y}} \qquad x_{3,4}$$

5. Find the average value for x

$$\bar{x} = x_{1,2} + x_{2,3} + x_{3,4}$$

Part 1.2 The effect of $S_2O_8^{2-}$ Concentration on Reaction Rate

1. Calculate the initial concentration of KI in the reaction mixture (under assumption that at t = 0 s no reaction has occurred):
2. Calculate the initial concentration of $(NH_4)_2S_2O_8$ in the reaction mixture (under assumption that at t = 0 s no reaction has occurred):
3. Calculate the reaction rate for each run:
4. Calculate the reaction order with respect to $S_2O_8^{2-}$ concentration (y) using data from run 1 and 2, 2 and 3, 3 and 4:

$$\frac{r_2}{r_1} = \frac{k \cdot [KI]_2{}^x \cdot [(NH_4)_2S_2O_8]_2{}^y}{k \cdot [KI]_1{}^x \cdot [(NH_4)_2S_2O_8]_1{}^y} \qquad y_{1,2}$$

$$\frac{r_3}{r_2} = \frac{k \cdot [KI]_3{}^x \cdot [(NH_4)_2S_2O_8]_3{}^y}{k \cdot [KI]_2{}^x \cdot [(NH_4)_2S_2O_8]_2{}^y} \qquad y_{2,3}$$

$$\frac{r_4}{r_3} = \frac{k \cdot [KI]_4{}^x \cdot [(NH_4)_2S_2O_8]_4{}^y}{k \cdot [KI]_3{}^x \cdot [(NH_4)_2S_2O_8]_3{}^y} \qquad y_{3,4}$$

5. Find the average value for y

$$\bar{y} = y_{1,2} + y_{2,3} + y_{3,4}$$

The Rate Law Expression

1. Calculate the rate constant k for every run in part 1.1 and 1.2 using the average order that have been previously calculated:

$$k_1 = \frac{r_1}{[KI]_1{}^{\bar{x}} \cdot [(NH_4)_2S_2O_8]_1{}^{\bar{y}}}$$

2. Express the rate law for the reaction.

$$r = \bar{k} \cdot [I^-]^{\bar{x}} \cdot [S_2O_8{}^{2-}]^{\bar{y}}$$

Part 1.3 The Effect of $Cu(NO_3)_2$ Addition Towards Reaction Rate

1. Calculate the initial concentration of KI in the reaction mixture (under assumption that at t = 0 s no reaction has occurred).
2. Calculate the initial concentration of $(NH_4)_2S_2O_8$ in the reaction mixture (under assumption that at t = 0 s no reaction has occurred).
3. Calculate the reaction rate for each run.
4. Calculate the reaction order with respect to I- concentration (x) using data from run 1 and 2, 2 and 3, 3 and 4.
5. Find the average value for x.
6. Calculate the rate constant k for every run in part 1.3, then find the average value (use the average x and y values from part 1.1 and 1.2).
7. Compare the average value for x and k in part 1.1 and 1.3.

Part 2. Activation Energy Determination for the Reaction between Fe^{3+} and $S_2O_3^{2-}$

The redox reaction between Fe^{3+} and $S_2O_3^{2-}$ is a (-4)th order reaction, with each Fe^{3+} and $S_2O_3^{2-}$ having the order of -2.

$$2\,Fe^{3+}{}_{(aq)} + 2\,S_2O_3{}^{2-}{}_{(aq)} \rightarrow 2\,Fe^{2+}{}_{(aq)} + S_4O_6{}^{2-}{}_{(aq)}$$

Its rate law can be expressed as:

$$-\frac{d[Fe^{3+}]}{dt} = k \cdot [Fe^{3+}]^{-2} \cdot \left[S_2O_3{}^{2-}\right]^{-2}$$

$$-[Fe^{3+}]^{-2} \cdot \left[S_2O_3{}^{2-}\right]^{-2} \cdot d[Fe^{3+}] = k \cdot dt$$

Note that Fe^{3+} and $S_2O_3^{2-}$ have the same reaction coefficient, therefore we do not need to multiply the rate by ½. Under that note as well, we can simplify the chemical equation, by substituting Fe^{3+} as A and $S_2O_3^{2-}$ as B:

	A	$+$	B	$\rightarrow$	$product$
Initial	A_0		B_0		
Reacted	$-x$		$-x$		$+x$
Equilibrium	$A_0 - x$		$B_0 - x$		x

with A_0 and B_0 represent the initial amount of Fe^{3+} and $S_2O_3^{2-}$ respectively and x represents the amount that reacted.

Now we can express the reaction rate in reactant concentration:

$$-\frac{d[A]}{dt} = k[A]^{-2}[B]^{-2}$$

or in product concentration:

$$+\frac{dx}{dt} = k[A_0 - x]^{-2}[B_0 - x]^{-2}$$

By integrating the rate law in product concentration and rearranged, we can calculate k using the following equation:

$$k = \frac{\left[B_0^{\,2}\left(A_0^2 x - A_0 x^2 + \frac{x^3}{3}\right) - A_0^2\left(B_0 x^2 - \frac{x^3}{3}\right) + A_0\left(\frac{4B_0 x^3}{3} - \frac{x^4}{2}\right) - \frac{B_0 x^4}{2} + \frac{x^5}{5}\right]}{t}$$

Using the aforementioned equation, calculate the value of k for each run (at room temperature, ±45 °C, ±65 °C) then construct a graph of 1/T (x axis) against ln k (y axis) according to the logarithmic form of Arrhenius equation, then calculate the activation energy in either J or kJ for the reaction. Use T in kelvins (K).

$$\mathbf{\ln k = -\frac{E_a}{R} \times \frac{1}{T} + \ln A}$$

Please bring them together:

- Lab journal
- Long-sleeved lab coat
- Tissue
- Rubber gloves (latex/nitrile)
- Glasses/goggles
- Calculator
- Graphical paper
- Ruler

Experiment II Chemical Equilibrium

Introduction

Chemical equilibrium can be recognized from its constant macroscopic properties in a closed system (or can be classified as a closed system) at certain temperature. As an example of equilibrium condition is the solvation reaction of iodine crystal in the water. The I_2 crystal starts to dissolve in the water by forming yellowish solution. After a while, the solution will turn brownish and finally becomes dark brown, while the amount of I_2 crystal is still the same. When the color of the solution is not changing anymore, the reaction has reached its equilibrium state. Based on that example, we can conclude that chemical equilibrium in a closed system at certain temperature has constant macroscopic properties.

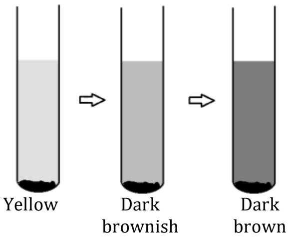


Figure 2.1 Iodine equilibrium in the water

But how do we explain the equilibrium state? We can explain that by observing the molecular effects of those molecules when they reached its equilibrium state. Radioactive form of I_2 was added to a saturated I2 solution that contains no radioisotope. After a short time, it was found that not only the undissolved I_2 crystal became radioactive but also the solution. This phenomenon showed that there are some radioactive I_2 that are dissolved in the solution meanwhile some non-radioactive I_2 are precipitated with the same density and velocity, showing no color change of the solution observed. Therefore, the chemical equilibrium is a dynamic condition, where the process in molecular level is still occurring but there are no changes observed in macroscopic level. Another example of macroscopic properties that can be observed based on its color solution is a reaction below:

An Iron (III) Nitrate solution is reacted with thiocyanate ions generating a red solution. This color changes occurs due to formation of ion complexes. Based on the concentration of the reactants, the reaction between Iron (III) and thiocyanate ions produce colored complex compound $Fe(SCN)^{2+}$

$$Fe^{3+}_{(aq)} + SCN^{-}_{(aq)} \leftarrow\rightarrow Fe(SCN)^{2+}_{(aq)}$$

To determine the concentration of a colored solution, we can use an instrument called spectrophotometer. The portion of a light beam's intensity that are transmitted or absorbed by the solution can be determined by using Beer-Lambert law. Beer-Lambert law is best used for dilute solution and use monochromatic beam as the light source.

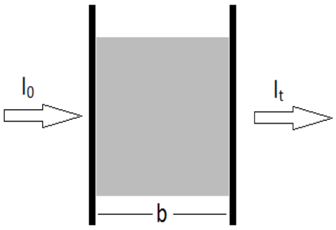


Figure 2.2 Beam intensity pass through the solution

I_0 : the intensity of the incident beam,

I_t : the intensity of the transmitted beam,

b : the path length.

The Beer-Lambert law is derived as A = a.b.c, where A is the absorbance; a is the absorption coefficient of the sample; b is the path length and c is the concentration of the sample. The ratio of concentration between two samples is equivalent to the ratio of their respective absorbance values. The absorption coefficient of $Fe(SCN)^{2+}$ is 4,70 x 10^3 M^{-1} cm^{-1} when the beam's wavelength is 447 nm. Absorbance can be defined as A = log (I_0/I_t). Beside absorbance, another unit that can be used is transmittance, which can be defined as the percentage ratio of I_t/I_0

$$\mathrm{A} = \log\frac{\mathrm{I_o}}{\mathrm{I_t}} = -\log\frac{\mathrm{I_t}}{\mathrm{I_o}} = -\log\mathrm{T} = \mathrm{a.b.c}$$

Chemicals and Equipment

The chemicals that are needed in this experiment is listed below:
KSCN 0,002 M; $Fe(NO_3)_3$ 0,002 M; $Fe(NO_3)_3$ 0,2 M, concentrated KSCN; solid Na_2HPO_4; Pb(II) solution; alcohol; H_2SO_4 2 M; NH_4CH_3COO; concentrated H_2SO_4; concentrated HCl, Ni(II) solution; NH_3 2M; dimethylglyoxime; Mg(II) solution; NH_4Cl, NaOH 2 M; $AgNO_3$; NH_3 6 M, and distilled water.

The equipment that are needed in this experiment is listed below:
Test tubes, a tube rack, Pasteur pipette, spatula, beaker glass 250 mL, burette 25 mL, wash bottle, cuvette, spectrophotometer, and electric heater.

Procedures

Section I: The equilibrium of Iron (III) - thiocyanate

a. Put 10 mL of KSCN 0,002 M into a beaker glass. Add two or 3 drops of $Fe(NO_3)_3$ 0,2 M
b. Divide the mixture into 4 different tubes
c. Use the first tube as a reference
d. Add 1 drop of concentrated KSCN solution into the second tube
e. Add 3 drops of $Fe(NO_3)_3$ 0,2 M into the third tube
f. Add 1 piece of Na_2HPO_4 into the fourth tube
g. Observe and write down your observation
h. What happen in the second, third and fourth tube?

Section II: Determination of Equilibrium constant formula and its Equilibrium constant

a. Prepare 3 250 mL beaker glasses and label them A, B, and C. Fill beaker A with 30 mL of $Fe(NO_3)_3$ 0,002 M, beaker B with 20 mL of KSCN 0,002 M and beaker C with 25 mL distilled water.
b. Prepare 5 50 mL beaker glasses and label them 1-5.
c. Use the burette to take all the solutions.
d. Fill each beaker glass with the composition listed below:

Composition	Total volume (mL)	Volume of $Fe(NO_3)_3$ 0,002 M (mL)	Volume of KSCN 0,002 M (mL)	Volume of distilled water
1	10,00	5,00	1,00	4,00
2	10,00	5,00	2,00	3,00
3	10,00	5,00	3,00	2,00
4	10,00	5,00	4,00	1,00
5	10,00	5,00	5,00	0,00

e. Fill in the mixture into the cuvette and measure the % Transmittance or absorbance of each mixture (mixture 1-5) using spectronic 20D. Use distilled water as a blank. Set the wavelength to 447 nm and write down the temperature in which the experiment is conducted.
f. By Using Beer-Lambert law, determine the concentration of $Fe(SCN)^{2+}$ when the equilibrium state is reached. Use a = $4{,}70 \times 10^3\ M^{-1}\ cm^{-1}$
g. Determine the concentration Fe^{3+} and SCN^- at the equilibrium condition
h. Determine the mathematical formula for the equilibrium constant
i. Determine the equilibrium constant of this reaction

Section III: Equilibrium of Ions

Section 3.1

a. Put 10 drops of Pb(II) solution into 3 test tubes then add 5 drops of H_2SO_4 2 M and alcohol into the three test tube.
b. Precipitates will form; leave the tube for a while to allow all precipitate to drop to the bottom of the tube, then separate the remaining solution above the precipitate using a pipette.
c. Into the 1st test tube add 10 drops of ammonium acetate to the remaining solids. Observe whether the precipitate are dissolving into the solution.
d. Into the 2nd test tube add 10 drops of concentrated HCl to the remaining solids. Observe whether the precipitate are dissolving into the solution.
e. Into the 3rd test tube add 10 drops of concentrated H_2SO_4 to the remaining solids. Observe whether the precipitate are dissolving into the solution.

Section 3.2

a. Put 10 drops of Nickel (II) solution into a test tube
b. Add 3 drops of NH_3 2 M. Write down your observation.
c. Add 3 drops of dimethylglyoxime (DMG). Write down your observation.

Section 3.3

a. Put 10 drops of Mg (II) solution into 3 test tubes
b. Into the 1st tube, add 10 drops of NH_3 2 M. Write down your observation.
c. Into the 2nd tube, add 10 drops NH_3 2 M and 10 drops of NH_4Cl. Write down your observation.

Section 3.4

a. Put 5 drops of $AgNO_3$ 0,1 M into 2 test tubes and add 5 drops of HCl 0,1 M each. Let them in the tube rack for a while to allow precipitate gather at the bottom of the tubes. If the precipitate has separated from the supernatant, decant the supernatant off from the tube.
b. Add NH_3 6 M drop by drops to the precipitate in the 1st tube. Write down your observation.
c. Add HCl 12 M drop by drops to the precipitate in the 2nd tube. Observe what happen after addition of HCl. Write down your observation.
   For your information, $[AgCl_2]^-$ and $[Ag(NH_3)_2]^+$ are silver ion complexes that dissolves in water. Write down the formation reaction of those ion complexes from $AgCl_{(s)}$. What are the colors of each ion complexes compounds?

DATA ANALYSIS

Section II: Determination of Equilibrium constant formula and its Equilibrium constant

a. Determine the absorbance and initial concentration of each composition

Composition	% Transmittance	Absorbance	[Fe(NO$_3$)$_3$)] (M)	[KSCN] (M)
1				
2				
3				
4				
5				

b. Determine the absorption coefficient of the solution based on calibration curve. Compare the absorption coefficient generated from your experiment and the literature.

c. Determine the concentration of the $Fe(SCN)^{2+}$ formed by each combination using Beer-Lambert law.

d. Determine the concentration of Fe^{3+} and SCN^- at equilibrium for all the combinations using equation below

ICE	$Fe^{3+} + SCN^- \leftrightarrow Fe(SCN)^{2+}$		
Initial	$[Fe^{3+}]_{intial}$	$[SCN^-]_{initial}$	0
Change	-x	-x	+x
Equilibrium	$[Fe^{3+}]_{eq} = [Fe^{3+}]_{initial} - x$	$[SCN^-]_{eq} = [SCN^-]_{initial} - x$	$[Fe(SCN)^{2+}]_{eq} = x$

e. The equilibrium constant is always the same in constant temperature. Determine the mathematical formula that suitable to describe the K

   a. $K = [Fe^{3+}].[\mathrm{SCN}^-].[\mathrm{Fe(SCN)}^{2+}]$
   b. $K = \frac{[\mathrm{Fe(SCN)}^{2+}]}{[Fe^{3+}].[\mathrm{SCN}^-]}$
   c. $K = \frac{[Fe^{3+}].[\mathrm{SCN}^-]}{[\mathrm{Fe(SCN)}^{2+}]}$
   d. $K = \frac{[\mathrm{SCN}^-].[\mathrm{Fe(SCN)}^{2+}]}{[Fe^{3+}]}$
   e. $K = \frac{[Fe^{3+}].[\mathrm{Fe(SCN)}^{2+}]}{[\mathrm{SCN}^-]}$

f. Determine the value of K for reaction above.

Please bring them together:

- Lab journal
- Long-sleeved lab coat
- Tissue
- Rubber gloves (latex/nitrile)
- Glasses/goggles
- Calculator

Experiment III Acid-Base Reactions

Introduction

The acid-base theory began by classifying compounds as acid or base based on their properties as aqueous solution. Acids have properties such as having sour taste, turning a blue litmus paper to red, and produce salt when reacts with base, whereas bases have bitter taste, are slippery and turns red litmus paper to blue.

There are three acid-base theory suggested:

1. Arrhenius (this theory explains that acid is a substance that when dissolved in water, dissociates as hydrogen ion (H^+) and an anion whilst a base dissociates to hydroxide ion (OH^-) and metal ion)
2. Bronsted-Lowry (this theory describes that acid is a substance that can donate a proton while base is a substance that can accept a proton)
3. Lewis (this theory explains that acid is any species that can accept lone- pair electrons while base is any species that can donate lone-pair of electrons)

In 1909, Sørensen suggested the pH concept could be used to explain the concentration of hydrogen ion. The pH of a solution is defined as the negative logarithmic value of the concentration of hydrogen ion in a solution.

$$\mathrm{pH} = -\log[\mathrm{H}^+]$$

One of methods that are commonly used to determine the concentration of hydrogen ion or concentration of acidic or basic solution is titration method. Titration is a technique to determine concentration of a substance by using another substance with known concentration. There are four types of titrations based on the type of reaction: acid - base titration, oxidation - reduction titration, complexes formation titration, and precipitation titration. Two important points that are crucial to understand are **end point** and **equivalent point**. The equivalent point is a condition in which the amount or mole of acid is equal to amount or mole of base. Whilst the end point defines as the point where the indicator has changed its color as a respond of pH changes. The difference between equivalent and end point called as titration error. In this experiment, we will conduct acid-base titration using visual indicator and pH meter (potentiometer).

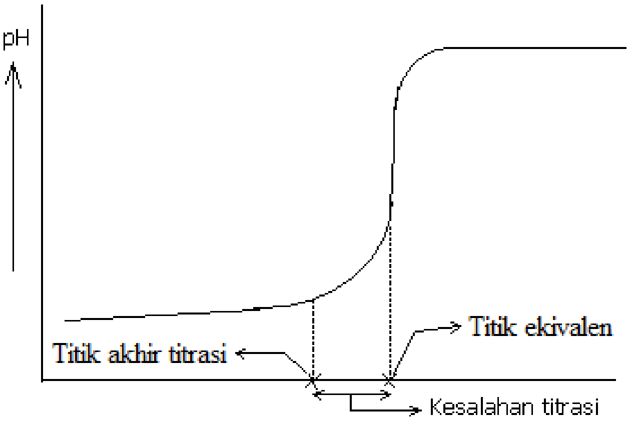


Figure 1.1 Acid-base titration curve

Acid-base indicator is a species/substance that can give different responses depending on the acidity of the solution. If the response can be observed using naked eyes, it is classified as a visual indicator. Acid-base visual indicators that are widely used are phenolphthalein, methyl red, and methyl orange. Each indicator has unique color changes in certain pH range. Choosing the right indicator before titration is a crucial aspect to minimize titration error.

An instrument that can measure pH level is pH meter. The pH meter is created based on the principles of potentiometer. It measures the concentration of hydrogen ion based on the difference of electrical potential produced by reference electrode and pH electrode. In this experiment, you will be asked to compare the concentration of the sample determined by visual titration and pH meter.

In titration, there are several terminologies that are usually used: analyte, primary and secondary standard solution. An analyte is the species/substance whose concentration will be determined. The primary standard solution is a solution whose concentration is accurately known, usually made by dissolving an exact known mass of a compound with an exact amount of solvent. The primary standard solution has to be a highly stable compound, not hygroscopic, its concentration is stable for a long period, and has a relatively big molecular weight. A secondary standard solution is a solution in which the concentration is not accurately determined. The secondary standard solution can be used for titration but its concentration are required to be determined correctly using the primary standard solution.

Chemicals and Equipment

List of chemicals that are needed in this experiment:
$H_2C_2O_4$ solution, NaOH solution pro-titration, a weak acid sample for one valence, phenolphthalein indicator, and distilled water.

List of equipment for this experiment:
Standard glassware, 250 mL Erlenmeyer flask, 25 mL volumetric pipette, pipette pump, 100 mL volumetric flask, 50 mL burette, magnetic stirrer and pH meter.

Procedures

Section 1: Acid-base titration using visual indicator

Section 1.1: Determination of NaOH solution concentration using Oxalic acid

a. Rinse the burette with small volume of distilled water to ensure that the burette is in a good condition
b. Rinse the burette using 5 mL NaOH solution pro-titration 3 times and then fill up the burette fully with NaOH solution.
c. Assemble the stand, clamp and burette so that the burette is perpendicular to the table.
d. Pipette 25,0 mL of oxalic acid solution using volumetric pipette and put it into 250 mL Erlenmeyer flask. Do it twice (duplo), so you will have 2 flasks each containing 25,0 mL oxalic acid.
e. Add 3 drops of phenolphthalein indicator into each flask.
f. Write down the initial volume of NaOH solution in the burette. Begin the titration by flowing NaOH solution drop by drops into the flask and continuously stir it slowly till the color changes from colorless to soft pink (the pink color should stable for 30 seconds). Stop the titration as soon as the soft pink color remained. Make sure not to add too much NaOH solution.
g. Note the scale of NaOH solution in the burette. Calculate the final volume of NaOH solution being used.
h. Determine the concentration of NaOH solution

Section 1.2: Determination the concentration of weak acid sample

a. Fill up the burette with NaOH solution.
b. Pipette 25 mL of acid sample into a 100 mL volumetric flask. Dilute them with distilled water till the volume sign (solution A).
c. Pipette 25 mL of diluted acid sample (solution A) into 250 ml Erlenmeyer flask. Do it twice (duplo).
d. Add 3 drops of phenolphthalein.
e. Write down the initial volume of NaOH solution in the burette. Begin the titration by flowing NaOH solution drop by drops into the flask and continuously stir it slowly till the color changes from colorless to soft pink (the pink color should stable for 30 seconds).
f. Note the scale of NaOH solution in the burette. Calculate the final volume of NaOH solution being used.
g. Determine the concentration of acid sample.

Section 2: Acid-base titration using pH meter

a. Fill up the burette with NaOH solution
b. Pipette 25 mL of solution A (from section 1.2) and put it into 100 mL of beaker glass. Then, add 25 mL of distilled water.
c. Standardize the pH meter by immersed the electrode of pH meter into standard buffer pH 4
d. Rinse the electrode using distilled water. Then put the electrode inside the beaker glass.
e. Put the magnetic stirrer into the beaker and start stirring at low speed, be careful not to destroy the electrode when it stirring. **Electrodes are very expensive.**
f. Begin the titration by adding NaOH solution as indicated

Addition of NaOH (mL)	0	2	2	2	2	2	1	0,5	0,5	0,5	0,5	1	1	1
pH														

g. Make a titration curve on a graphical paper by plotting the pH point against the volume of NaOH solution added (volume of NaOH solution as x-axis and pH as y-axis) then connecting the dots created.
h. Determine the equivalent point and the acid sample concentration
i. Determine the K_a of weak acid sample
j. Compare the concentration of acid sample from this experiment and the concentration generated from section 1.2

Data Analysis

Section 1: Acid-base titration using visual indicator

Section 1.1: Determination of NaOH solution concentration using Oxalic acid

a. Write down the reaction of this experiment
b. Calculate the volume of NaOH solution needed
c. Determine the concentration of NaOH solution using predetermined oxalic acid (note the stoichiometry of this reaction)

Section 1.2: Determination the concentration of acid sample

a. Write down the reaction of this experiment
b. Calculate the volume of NaOH solution needed
c. Determine the actual acid sample concentration (before dilution, note the dilution step)

Section 2: Acid-base titration using pH meter

a. Write down the reaction of this experiment
b. Draw the titration curve
c. Determine the equivalent point based on your graph
d. Determine the acid sample concentration
e. Determine the K_a of the sample based on your graph

Please bring them together:

- Lab journal
- Long-sleeved lab coat
- Tissue
- Calculator
- Ruler
- Graphical paper (millimeter block)

Experiment IV Oxidation-reduction reaction and Electrochemical cell

Introduction

Oxidation-reduction (redox) reaction is recognized by the changes of oxidation number between the reactant and the product. The method to balance the chemical reaction formula has been explained comprehensively in stoichiometry chapter. Therefore, in this experiment, we will only discuss several redox reactions as example.

$$\overbrace{Cl^{-}_{(aq)} + \mathrlap{\underbrace{MnO_{2\,(s)} \rightarrow Cl_{2\,(g)} + Mn^{2+}_{(aq)}}_{\text{Reduction (-2)}}}\phantom{MnO_{2\,(s)} \rightarrow Cl_{2\,(g)}}}^{\text{Oxidation (2) x (+1)}}\phantom{{} + Mn^{2+}_{(aq)}} \qquad \text{(acidic condition)} \qquad (1)$$

$$2\ Cl^{-}(aq) + MnO_2\ (s) + 4\ H^{+}(aq) \rightarrow Cl_2(g) + Mn^{2+}(aq) + 2H_2O \qquad (2)$$

In the second reaction, the balanced reaction has fulfilled the law of conservation of charge and law of conservation of mass. Based on reaction above, the Cl atom has increased the oxidation number as Cl_2 while the Mn oxidation number in MnO_2 form is reduced to Mn^{2+}.

Electrochemical cell is classified into two different types, which is Voltaic/Galvanic Cell and electrolytic cell. Galvanic cell is an electrochemical cell that consists of two electrodes and be able to produce electric current due to spontaneous redox reaction between two electrodes. One of the most important formulas for galvanic cell is Nernst formula. Daniel cell, as an example of galvanic cell, consists of zinc electrode, $Zn/Zn^{2+}_{(aq)}$ and copper electrode, $Cu/Cu^{2+}_{(aq)}$ using $ZnSO_4$ and $CuSO_4$.

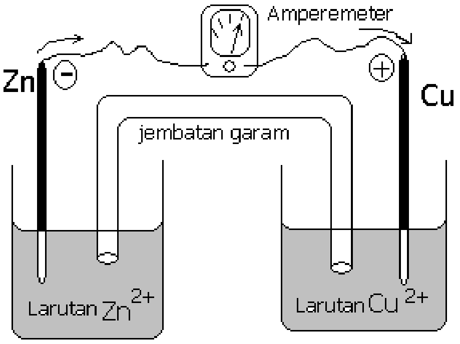


Figure 4.1 Galvanic cell

Electrolysis is a process in which electrical energy is required to drive a nonspontaneous chemical reaction to occur. The electrolysis apparatus is an electrolytic cell consisting of electrolyte (solution or molten salt) and two electrodes (anode and cathode). Oxidation reaction occurs at anode while reduction reaction occurs at cathode.

Chemicals and Equipment

Chemicals required in this experiment is listed below:
$CuSO_4$ 0,5 M; $ZnSO_4$ 0,5 M; $Pb(NO_3)_2$ 0,5 M; $Zn(NO_3)_2$ 0,5M; $NaNO_3$ 0,5 M; H_2SO_4 1 M; H_2SO_4 3 M; H_2O_2 0,1 M; KI 0,1 M; KI 0,25 M; starch solution, $FeCl_3$ 0,1 M; solution; Cu^{2+}; Zn^{2+}; Pb^{2+}; Sn^{2+}; phenolphthalein; chloroform; Metal: Zn, Cu, Mg; MnO_2 powder; Electrode: Cu, Zn, Pb, Sn, calomel and distilled water.

Equipment required in this experiment is listed below:
Standard lab glassware, U-shape tube, electric heater, adaptor, carbon electrode and potentiometer.

Procedures

Section 1: Oxidation- Reduction reactions

a. Put 2 mL of $CuSO_4$ 0,5 M into a test tube. Add 1 piece of Zn metal granule. Let it react and write down your observation. Repeat the procedure on the reverse way by adding Cu metal into $ZnSO_4$ 0,5 M solution. What happens? Write down your observation. Explain this phenomenon using the electrode potentials.
b. Put 1 piece of Mg metal into each of the three test tubes, each of them containing $Pb(NO_3)_2$ 0,5 M, $Zn(NO_3)_2$ 0,5 M and $NaNO_3$ 0,5 M respectively. Observe the changes that occurs, then arrange the metals according to its reactivity from the lowest to highest.
c. **Disproportionation reaction**. Put 10 drops of H_2O_2 0,1 M and add a very small amount of MnO_2 powder to catalyze the reaction.
d. Add 5 drops of H_2SO_4 1 M into a test tube containing 5 drops of H_2O_2 0,1 M and 10 drops of KI 0,1 M. Add 1 drop of starch solution.
e. Add 5 drops of $FeCl_3$ 0,1 M, 10 drops of H_2SO_4 1 M and 10 drops KI 0,1 M into a test tube. Warm it up and add a drop of starch solution. Observe the reaction.

Section 2 : Voltaic Cells

2.1 Determination of Electrode Potential

a. Fill up the beaker glass with $CuSO_4$ 0,1 M solution up to half of the beaker.
b. Immerse the Cu electrode and a saturated calomel electrode into this solution.
c. Connect the two electrodes to a multimeter. Note the position of the poles (for examples, the calomel electrode is on the (+) pole and the Cu electrode is on the (-) pole).
d. Note the potential of the cell including its sign (+ or -)
e. Note the room temperature and calculate the potential of saturated calomel electrode (E^o calomel = 0,242 volt)
f. Determine the $Cu|Cu^{2+}$ electrode potential and repeat it for $Pb|Pb^{2+}$, $Sn|Sn^{2+}$ and $Zn|Zn^{2+}$
g. Arrange all the metals in order of increasing potential. Does it follow the Volta series?

2.2 Determination of Electrochemical Cell Potential

Electrochemical cell is a voltaic cell consisting of two different electrodes, for example a cell consisting $Cu|Cu^{2+}$ and $Pb|Pb^{2+}$ electrode

a. Determine the voltaic cell potential of
    - $Cu|CuSO_4$ (0,1 M) and $Pb|Pb(NO_3)_2$ (0,1 M)

- Repeat the procedure for different possible combination ($Cu|Cu^{2+}$, $Pb|Pb^{2+}$, $Sn|Sn^{2+}$ and $Zn|Zn^{2+}$)

b. Note the electrodes that acts as cathode and anode during measurement

c. Determine the metal reactivity series based on your data. Compare your data collection and arrange it according the level of reactivity from high to low

Li K Ba Sr Ca Na Mg Al, Mn Zn Cr Fe Cd Co Ni Sn Pb H Sb Bi Cu Hg Ag Pt Au

Write down the cell notation and cell reaction in every cell combination above.

Section 3 : Electrolytic cell

a. Put KI 0,25 M solution in a U-shaped tube until 2 cm below the tube edge
b. Immerse the graphite electrode and connect it to the electrical source for 5 minutes, then disconnect the electrical current
c. Write down the reaction occurs at the anode and cathode electrode
d. Take 2 mL of solution from the cathode using a pipet into the 1st test tube and add a drop of phenolphthalein.
e. Take 2 mL of solution from the cathode using a pipet into the 2nd test tube and add 3 drops of $FeCl_3$ 0,1 M solution
f. Take 2 mL of solution from the anode using a pipet into the 3rd test tube. Add 2 mL of $CHCl_3$ and shake it. Observe the color of $CHCl_3$ phase.
g. Write down your observation and the entire chemical reactions.

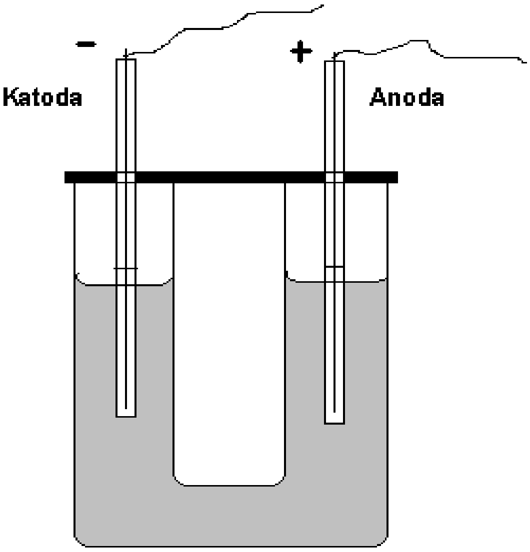


Figure 4.2 Electrolytic cell

Standard reduction potential electrode at 25°C

Electrode	E (volt)
$Zn^{2+}\mid Zn$	-0,76
$Fe^{2+}\mid Fe$	-0,44
$Sn^{2+}\mid Sn$	-0,19
$Pb^{2+}\mid Pb$	-0,13
$H^{+}\mid H_2\mid Pt$	0,00
$Cu^{2+}\mid Cu$	+0.34
$Pt\mid Fe^{3+}.Fe$	+0,77
$Ag^{+}\mid Ag$	+0,8
$Li^{+}\mid Li$	-3,04
$Al^{3+}\mid Al$	-1,66

Please bring them together:

- Lab journal
- Long-sleeved lab coat
- Tissue
- Rubber gloves (latex/nitrile)
- Glasses/goggles
- Calculator

Experiment V Organic compounds

Introduction

Organic chemistry is a branch of chemistry that studied structure, properties, and reaction of carbon atom and carbon-containing compounds. At the beginning, the scope of organic chemistry was limited to chemical compounds found in the living organism due to a point of view that showed carbon-containing compounds to be found only in living organisms or things that were once alive. Facts have shown that living organisms are not the only sources of carbon-containing compounds.

Since 1940s, organic chemistry has developed as a branch of science splendidly and has separated from inorganic chemistry. The development of organic chemistry is triggered by the needs of countries during World War II. The production of synthetic rubber, nylon, and usage of plastic were 3 examples of “new” products creation in 1940s that have greatly developed and attracted scientists to study more about organic chemistry.

A lot of organic chemicals are found in daily life, such as vitamins, plastics, detergents, cloths, inks, paints, natural crude oil, and natural gas, film, audio, and video tape, medicines, perfume, cosmetics, fertilizer, food products, and glue. In a living body there are main organic compounds such as fat, carbohydrate, and proteins as explained below.

Fat is glyceryl ester of a long chain organic acid such as:

$(C_{17}H_{35}COO)_3C_3H_5$	$(C_{17}H_{33}COO)_3C_3H_5$	$(C_{17}H_{31}COO)_3C_3H_5$
Glyceryl stearate (stearin)	Glyceryl oleate (Olein)	Glyceryl palmitate (palmitin)

If a fat is heated with addition of NaOH, a substitution reaction occurs and sodium salt and glyceryl alcohol (glycerin) are formed. This type of reaction is called saponification reaction.

$$(C_{17}H_{35}COO)_3C_3H_5 \quad + \quad 3\ NaOH \quad \rightarrow \quad 3\ C_{17}H_{35}COONa \quad + \quad C_3H_5(OH)_3$$

Glyceryl stearate Sodium hydroxide Sodium stearate glycerin

Sodium stearate is one of the molecules that may constitute a soap.

Carbohydrate is a class of compounds that consisted of carbon, hydrogen and oxygen, usually has 6 carbon atoms or multiplication of 6 carbon atoms in a molecule. The ratio of hydrogen and oxygen usually 2:1 similar like in the water molecules; hence the name carbohydrates (they are formerly thought to be hydrates of carbon). The molecular weight of polysaccharides such as starch and cellulose are quite big approximately 10.000 – 30.000. Sugar is a type of carbohydrate, which has sweet taste.

Protein is a class of compounds that consisted mainly of carbon, hydrogen, oxygen, nitrogen and few of sulfur and phosphor atoms. It has enormous molecular weight. Hydrolyzing protein will generate 15 – 20 amino acids. An amino acid consists of amine (-NH2) and carboxylic acid (COOH) group within its molecule. Glycine (CH_2NH_2COOH) is the simplest of the amino acids. Protein itself, therefore, is a polymer of amino acids that are connected to each other by peptide bonds (-CO-NH-).

Polymer is a class of macromolecular chemical compounds that are formed by repeating units of monomers. Polysaccharides and proteins are examples of polymer. Polysaccharides consist of monosaccharides as the monomer (for example, amylose in starch is formed by repeating glucose monomers) while proteins are built by amino acids as monomers. Other examples of polymer include rubber, plastic, glue and nylon.

Chemicals and Equipment

Chemicals that are required in this experiment is listed below:
40% NaOH solution, palm oil, ethyl alcohol, saturated NaCl solution, $CaCl_2$ solution, commercial detergent, egg white, $CuSO_4$ 1 % solution, NaOH 6 M solution, concentrated HNO_3, lakmus paper, $Pb(OAc)_2$ solution, transparent glue, sodium tetraborate, distilled water, tap water, and demineralized water.

Equipment that are required in this experiment is listed below:
Standard glassware, plastic cup, electric heater, evaporation dish, tube rack and test tube.

Procedures

Section 1: Soap formation and Saponification reaction.

a. Mix 5 mL of 40% NaOH solution, 5 mL of palm oil, and 5 mL of ethyl alcohol in evaporation dish.
b. Heat it up carefully and stir it continuously approximately 15 minutes.
c. When all the liquid has evaporated and the residue has solidified, add a bit of distilled water.
d. Cool it down and add 40 mL of saturated NaCl.
e. Filter the mixture then rinses once with cold water.
f. Make soap solution using your soap and dissolve it in 100 mL of distilled water.
g. Take 10 mL of your soap solution and add 5 mL of $CaCl_2$, shake and observe it.
h. Dissolve 1 g of commercial detergent in 10 mL of distilled water. Add 2-3 drops of this solution into
   - $CaCl_2$-containing distilled water
   - Tap water
   - Distilled water

   Write down your observation. Repeat this experiment with your soap and compare the result.

Section 2: Protein

Mix 2 mL egg white with 10 mL of demineralized water and stir it slowly. If the mixture is formed non-transparent solution, add a bit of salt. Prepare 5 reaction tubes and put 2 mL of egg white solution into each tube and give different treatment as explained below:

a. Into the 1st tube, add 1 mL of 1% $CuSO_4$ and a drop of NaOH 6 M into the first tube
b. Into the 2nd tube, add 1 mL of concentrated HNO_3 into the second tube. Heat it up. Cool it down and adds NaOH 6 M, shake it.
c. Into the 3rd tube, add 1 mL NaOH 6 M. Heat it up carefully using a water bath. Smell the vapor that come out from the tube. Check its acidity using litmus paper
d. Into the 4th tube, Add drops of $Pb(OAc)_2$ and 1 mL of NaOH 6 M. Heat it up carefully

Observe and note any changes that occur in the four experiment

Section 3: Polymer

a. Put 9 mL of transparent glue in a clean plastic cup (first cup)
b. Dissolve 1,2 g sodium tetraborate with 5 mL distilled water in another clean plastic cup (second cup). Mix well. Alternatively, if there is premix sodium tetraborate solution, then use that and pour it to another cup.
c. Pour the solution in the second cup into the first cup and mix it together with glass stick.
d. What happen to the mixture? Compare the characteristic of the glue before and after added with sodium tetraborate solution.

Please bring them together:

- Lab journal
- Long-sleeved lab coat
- Rubber gloves (latex/nitrile)
- Glasses/goggles
- 2 eggs (per group)
- 2 clean plastic cups (per group)
- glass detergent