

CHAPTER I

INTRODUCTION

1.1 Background

Science and technology that continue to develop, especially in the industrial sector in Indonesia, encourage industries in various sectors to continue to innovate and develop themselves, one of which is in the chemical industry sector. This development increases the need for production in the chemical industry, both in terms of raw materials and other supporting materials. The chemical industry has a variety of raw materials and supporting materials used, including ethyl acetate which is a type of solvent with the molecular formula $\text{CH}_3\text{COOC}_2\text{H}_5$.

Esterification is a reaction to form an ester from a carboxylic acid and an alcohol. The reaction products are esters and water. The general equation for this reaction can be determined as follows:



The esterification reaction is a reversible, exothermic reaction, and runs very slowly but when using a mineral acid catalyst such as sulfuric acid (H_2SO_4) or hydrochloric acid (HCl) equilibrium will be achieved in a fast time (Susanti, 2019). Therefore, it is necessary to study the influencing factors and conduct various experiments to determine the various process variables that affect the esterification process.

1.2 Practical Purpose

1. Knowing the effect of reaction time on the conversion of esterification reactions.
2. Knowing the effect of the variable on the conversion in the esterification process.
3. Knowing the effect of the variable on the reaction rate constant (k) in the esterification process.
4. Knowing the effect of the variable on the direction of equilibrium (K) in the esterification process.

1.3 Practicum Benefits

1. Students can understand how the effect of reaction time on conversion in the esterification process.
2. Students can determine the effect of a certain variable on the conversion

of the ester formed.

3. Students can find out how to observe the effect of a certain variable on the direction of the equilibrium (K) and the reaction rate constant (k).
4. Students can conduct numerical studies based on the experiments carried out.

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CHAPTER II

LITERATURE REVIEW

2.1 Ester

Ester is one of the compounds resulting from the condensation reaction between carboxylic acid and alcohol with the help of a catalyst. These compounds are commonly used in the chemical, pharmaceutical, cosmetic, and food industries. Catalysts that can be used in ester synthesis are homogeneous and heterogeneous catalysts (Leite *et al.*, 2023).

The most widely used homogeneous catalysts in ester synthesis are chloric, sulfuric, phosphoric, and p-toluene sulfonic acids. The advantages of using homogeneous catalysts are low cost and high conversion. However, the use of homogeneous catalysts has several disadvantages, including that they cannot be reused, can increase the corrosion of the equipment used, and require more stages of purification (Leite *et al.*, 2023).

Heterogeneous catalysts that can be used in ester synthesis are zeolites and silica. The use of heterogeneous catalysts in ester synthesis has several advantages and disadvantages. The advantages of using heterogeneous catalysts are that they are easily separated from the reaction medium and can be recycled. However, heterogeneous catalysts show lower catalytic activity compared to homogeneous catalysts (Leite *et al.*, 2023).

2.2 Kinetics Reaction

Esterification or ester production is a reaction between a carboxylic acid and an alcohol with the product are ester and water. An example is the reaction between acetic acid and ethanol. The esterification reactions include the following:



Chemical reaction rate equation:

$$-r_A = \frac{dC_A}{dt} = k_1[A][B] - k_2[C][D]$$

With:

$-r_A$ = reaction rate of formation ester

$[A]$ = acetic acid concentration $[\text{CH}_3\text{COOH}]$

$[B]$ = ethanol concentration $[\text{C}_2\text{H}_5\text{OH}]$

$[C]$ = ethyl acetate concentration $[\text{CH}_3\text{COOC}_2\text{H}_5]$

- [D] = water concentration [H₂O]
 k₁ = reaction rate constant to the right (products direction)
 k₂ = reaction rate constant to the left (reactants direction)
 t = time reaction

Based on the kinetics reaction, the reaction speed of ester formation will increase with increasing temperature, stirring, and adding a catalyst. This can be explained by the Arrhenius equation:

$$k = Ae^{-E_A/RT}$$

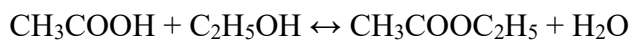
With:

- k = reaction rate constant (L/mol.waktu)
 A = collision frequency factor
 E_A = activation energy (J/mol)
 R = universal gas constant (8,314 J/mol.K)
 T = temperature (K)

Based on the Arrhenius equation, it can be seen that the reaction rate constant is influenced by the values of A, E_A, and T, the increase of the collision factor (A), will increase the reaction rate constant (k). The value of activation energy (E_A) is influenced by the use of a catalyst, catalyst will reduce the activation energy so that the value of k will increase. The higher the temperature (T), the value of k also getting bigger.

2.3 Thermodynamics Overview

Based on the thermodynamics overview, we can find out whether the reaction is reversible or irreversible by looking at the change in Gibbs energy (ΔG°). The esterification reaction between acetic acid and ethanol occurs according to the following reaction:



$$\Delta H^\circ_{298} = \Delta H^\circ_f \text{ produk} - \Delta H^\circ_f \text{ reaktan}$$

Known standar ΔH°_f data (Smith *et al.*, 2001) :

$$\Delta H^\circ_f 298 \text{ CH}_3\text{COOH} = -484500 \text{ J/mol}$$

$$\Delta H^\circ_f 298 \text{ C}_2\text{H}_5\text{OH} = -277690 \text{ J/mol}$$

$$\Delta H^\circ_f 298 \text{ CH}_3\text{COOC}_2\text{H}_5 = -480000 \text{ J/mol}$$

$$\Delta H^\circ_f 298 \text{ H}_2\text{O} = -285830 \text{ J/mol}$$

So :

$$\begin{aligned} \Delta H^\circ_{298} &= (\Delta H^\circ_f 298 \text{ CH}_3\text{COOC}_2\text{H}_5 + \Delta H^\circ_f 298 \text{ H}_2\text{O}) - (\Delta H^\circ_f 298 \text{ CH}_3\text{COOH} + \\ &\quad \Delta H^\circ_f 298 \text{ C}_2\text{H}_5\text{OH}) \\ &= (-480000 \text{ J/mol} - 285830 \text{ J/mol}) - (-484500 \text{ J/mol} - 277690 \text{ J/mol}) \end{aligned}$$

$$= -3640 \text{ J/mol}$$

Based on the thermodynamics overview, it can also be seen that the reaction is endothermic or exothermic by observing the enthalpy changes. From the calculation, the enthalpy change (ΔH) is negative which indicates that the esterification reaction of acetic acid with ethanol is exothermic. The Gibbs energy value (ΔG°) can be calculated using the following equation:

$$\Delta G^\circ_{298} = \Delta G^\circ_f \text{ products} - \Delta G^\circ_f \text{ reactants}$$

Known standar ΔG°_f data (Smith *et al.*, 2001):

$$\Delta G^\circ_{f298} \text{ CH}_3\text{COOH} = -389900 \text{ J/mol}$$

$$\Delta G^\circ_{f298} \text{ C}_2\text{H}_5\text{OH} = -174780 \text{ J/mol}$$

$$\Delta G^\circ_{f298} \text{ CH}_3\text{COOC}_2\text{H}_5 = -332200 \text{ J/mol}$$

$$\Delta G^\circ_{f298} \text{ H}_2\text{O} = -237129 \text{ J/mol}$$

So :

$$\begin{aligned} \Delta G^\circ_{298} &= (\Delta G^\circ_{f298} \text{ CH}_3\text{COOC}_2\text{H}_5 + \Delta G^\circ_{f298} \text{ H}_2\text{O}) - (\Delta G^\circ_{f298} \text{ CH}_3\text{COOH} + \\ &\quad \Delta G^\circ_{f298} \text{ C}_2\text{H}_5\text{OH}) \\ &= (-332200 \text{ J/mol} - 237129 \text{ J/mol}) - (-389900 \text{ J/mol} - 174780 \text{ J/mol}) \\ &= -4649 \text{ J/mol} \end{aligned}$$

From the Van't Hoff equation :

$$\Delta G^\circ_{298} = -R \cdot T \cdot \ln K$$

$$\ln K = \frac{-\Delta G^\circ_{298}}{R \cdot T}$$

$$\ln K = -\frac{(-4649) \text{ J/mol}}{8,314 \text{ J/mol.K} \cdot (298 \text{ K})}$$

$$K = 6,5240$$

If in this practicum an operating temperature of 54°C is used, then the value of K at 54°C can be calculated:

$$\ln \frac{K}{K_{298}} = -\frac{\Delta H^\circ_{298}}{R} \left(\frac{1}{T} - \frac{1}{T_{298}} \right)$$

$$\ln \frac{K_{327}}{6,5240} = -\frac{(-3640) \text{ J/mol}}{8,314 \text{ J/mol.K}} \left(\frac{1}{327} - \frac{1}{298} \right) \text{K}$$

$$K_{327} = 5,7277$$

From the Gibbs energy calculation, the K value at an operating temperature of 54°C is obtained and a value of 5,7277 is obtained. So, it can be concluded that the esterification reaction of acetic acid with ethanol is a reversible reaction.

At equilibrium with an operating temperature of 54°C and a K value of 5.7277, the theoretical conversion value can be calculated as follows:

$$K = \frac{C_C C_D}{C_A C_B}$$

$$K = \frac{(C_{A0}X_A)(C_{A0}X_A)}{C_{A0}(1-X_A)(C_{B0}-(C_{A0}X_A))}$$

$$K = \frac{(X_{Ae})^2}{(1-X_{Ae})(1,5-X_{Ae})}$$

$$5,7277 = \frac{(X_{Ae})^2}{(1-X_{Ae})(1,5-X_{Ae})}$$

$$X_{Ae} = 0,8273$$

So that at equilibrium with an operating temperature of 54°C theoretically a conversion value of 82.73% is obtained (The above calculations are only examples, the practicum must adjust the reaction temperature and the mole ratio of reactants according to the temperature variable obtained in the practicum).

2.4 Reaction Mechanism

The esterification reaction is a reversible reaction which means that this reaction can proceed in the direction of ester formation or undergo ester breakdown back into acids and alcohols (Jyoti & Soni, 2023). Esterification reactions are characterized by the formation of esters from the reaction of carboxylic acids and alcohol (methanol or ethanol). Esterification reactions are commonly used to treat biodiesel production feedstocks to reduce the concentration of free fatty acids (Almeida *et al.*, 2018).

This reaction takes place slowly at room temperature so heating and the use of catalysts are required to accelerate the reaction rate. The catalyst used in esterification can be either an acid or base catalyst. This practicum uses carboxylic acid in the form of acetic acid reacted with an alcohol in the form of ethanol using an acid catalyst. In the manufacture of ethyl acetate, the esterification reaction that occurs in this practicum and the mechanism of acid catalysts in ester hydrolysis are as follows:

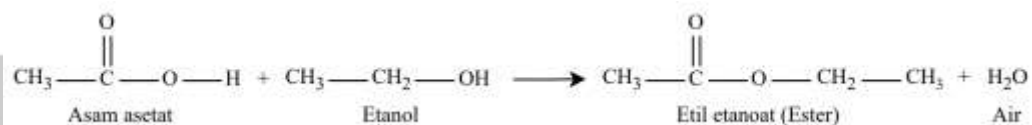


Figure 2. Esterification reaction

The mechanism of the esterification reaction is a substitution reaction between an acyl nucleophile and an acid catalyst (usually HCl or H₂SO₄). The carbonyl group of the carboxylic acid is not strong enough as an electrophile to be attacked by the alcohol. The acid catalyst will protonate the carbonyl group and activate it towards attacking the nucleophile. The release of the proton will

produce the hydrate of the ester and then proton transfer occurs.

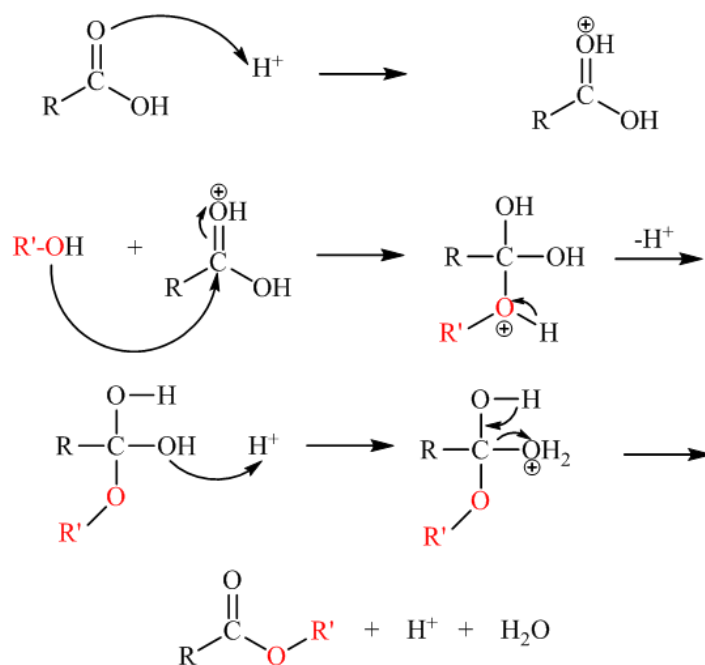


Figure 2.2 Mechanism of esterification reaction

The mechanism of esterification with an acid catalyst includes:

1. In the first step, the carbonyl group will be protonated by the acid. Transfer of protons from the acid catalyst to the carbonyl oxygen atom, resulting in an increase in the electrophilicity of the carbonyl carbon atom.
2. The second step involves the addition of a nucleophile, i.e. the OH group on the alcohol attacks the protonated carbonyl carbon. So a new C-O bond (ester bond) is formed.
3. The third stage is the equilibrium stage in which the H^+ group is removed from the new ester bond. Deprotonation is carried out to form stable C-O bonds.
4. In the fourth step, one of the hydroxyl groups must be protonated, because the two hydroxyl groups are identical.
5. The fifth stage, involves breaking the C-O bonds and releasing water. In order for this event to occur, the hydroxyl group must be protonated so that its ability as a free group is better.
6. In the last stage, the protonated ester releases its proton.

2.5 Influential Variables

The esterification reaction is influenced by several variables. These variables include:

1. Reaction time

According to Lestari *et al.* (2020), the longer the reaction time, the more conversion will be produced. This is due to the greater contact between substances (colliding with each other). However, when the process conditions reach equilibrium, the increasing reaction time can cause the reaction to reverse back towards the reactants so that the length of time the reaction will not affect the results obtained (Mirzayanti *et al.*, 2023).

2. Reagent ratio

The mole ratio of acid and alcohol has a direct impact on the conversion rate of esterification (Kastratovic & Bigovic, 2018). This is due to the reversible nature of the reaction so that one of the reactants must be made in excess so that the reaction tends to move towards the product to produce more esters.

3. Stirring

Stirring in the reaction process has a positive impact on increasing the reaction speed where by stirring, the tendency of contact between reactants will be higher so that the reaction speed increases (Nuryoto *et al.*, 2020). The optimum stirring speed for various raw materials needs to be adjusted based on their different physical properties (Panchal *et al.*, 2020).

4. Temperature

The conversion value will increase as the temperature increases. This is in accordance with the Arrhenius equation where if the temperature rises, the price of the reaction rate constant will be greater so that the reaction runs faster. However, because the esterification reaction is exothermic, the increase in temperature will not always increase the conversion. There is an optimum point where the increase in temperature actually decreases the equilibrium, so it does not increase the conversion value (Fakhry & Rahayu, 2016). In addition, too high reaction temperatures are also avoided due to the possibility of increased and excessive ethanol evaporation, shifting the reaction equilibrium towards the substrate (Piotrowski & Kubica, 2021).

5. Catalyst

According to Nuryoto *et al.* (2020), if the esterification process is carried out without a catalyst, the reaction will not be effective and efficient so that the presence of a catalyst can accelerate the reaction rate and maximize the conversion of acetic acid. Increasing the amount of catalyst results in an increase in conversion yield during the reaction time. The use

of catalysts accelerates the reaction by lowering its activation energy
(Jusniar *et al.*, 2020).

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CHAPTER III
PRACTICUM METHOD

3.1 Experimental Design

3.1.1 Practical Design

3.1.2 Variable

A. Fixed variable

1. Types of carboxylic acid :
2. Total volume :
3. Titration sample volume :
4. Sampling time :

B. Changed Variable

3.2 Materials and Tools Used

3.2.1 Materials

1. Acetic acid
2. Alcohol
3. Catalyst
4. NaOH
5. PP indicator

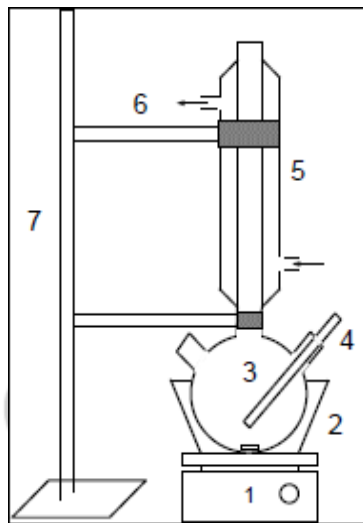
3.2.2 Tools

1. Scale
2. Three-neck flask
3. Leibig condensor
4. Electric stove
5. Thermometer
6. Burette 50 mL
7. Measuring pipette
8. Drop pipette
9. Stative
10. Clamp
11. Erlenmeyer
12. Beaker glass
13. Volumetric flask
14. Aspirator

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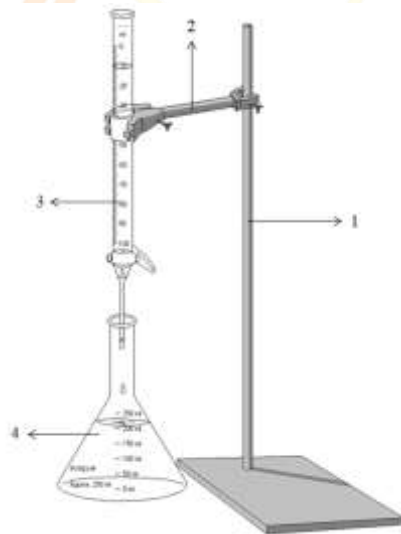
3.3 Toolkit Images



Where:

1. Magnetic Stirrer + heater
2. Waterbath
3. Three-neck flask
4. Thermometer
5. Leibig condenser
6. Clamp
7. Stative

Figure 3.2 Series of hydrolysis tools



Where:

1. Stative
2. Clamp
3. Burette
4. Erlenmeyer

Figure 3.3 Series of titration tools

3.4 Procedure

A. Preparation

1. Calibrate the pycnometer by weighing the empty pycnometer and recording its mass, then filling it with aquadest, weighing it, and recording its mass.
2. Measuring the density of acetic acid, catalyst, and alcohol using a pycnometer. The pycnometer is filled with the reagent whose density you want to calculate, then weighing and recording its mass.
3. Calculate the density with this equation :

$$\rho = \frac{(mass\ pycno + reagent) - (mass\ empty\ pycno)}{volume\ pycno}$$

4. Weighing ... gram of NaOH, then dissolve into 250 mL of aquadest. After dissolving, put it in the burette to use as titrant.

B. Main Experiment

1. Assemble the tools as depicted in the figures above.
2. Mix ... mL of acetic acid, ... mL of catalyst, and ... mL of alcohol in a beaker glass. Take 5 mL of the sample as t_0 . Then, add 3 drops of PP indicator and titrate with NaOH ... N. Reminder that total volume for the t_0 experiment is only 1/10 of the total volume in t_1 , t_2 , t_3 , and t_4 experiment.
3. Mix ... mL of acetic acid and ... mL of catalyst. Then, heat it up to ... °C in a three-neck flask.
4. In a different place, heat the alcohol to a temperature of ... °C in beaker glass.
5. After two reactants reach the variable temperature, mix alcohol into acetic acid solution in the three-neck flask.
6. Observe the temperature of mixture. After reaching the temperature according the variable, take 5 mL of the sample start from t_1 with a sampling time of every ... minutes until the time reached ... minutes.

C. Analytical Method

1. Take 5 mL of the sample, add 3 drops of PP indicator, then titrate with NaOH ... N.
2. Observe the color change that occurred from colorless to weak pink. Note the amount of titrant used. Stop sampling after reaching the time of ... minutes.
3. Repeat the above steps for the second variable.

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BIBLIOGRAPHY

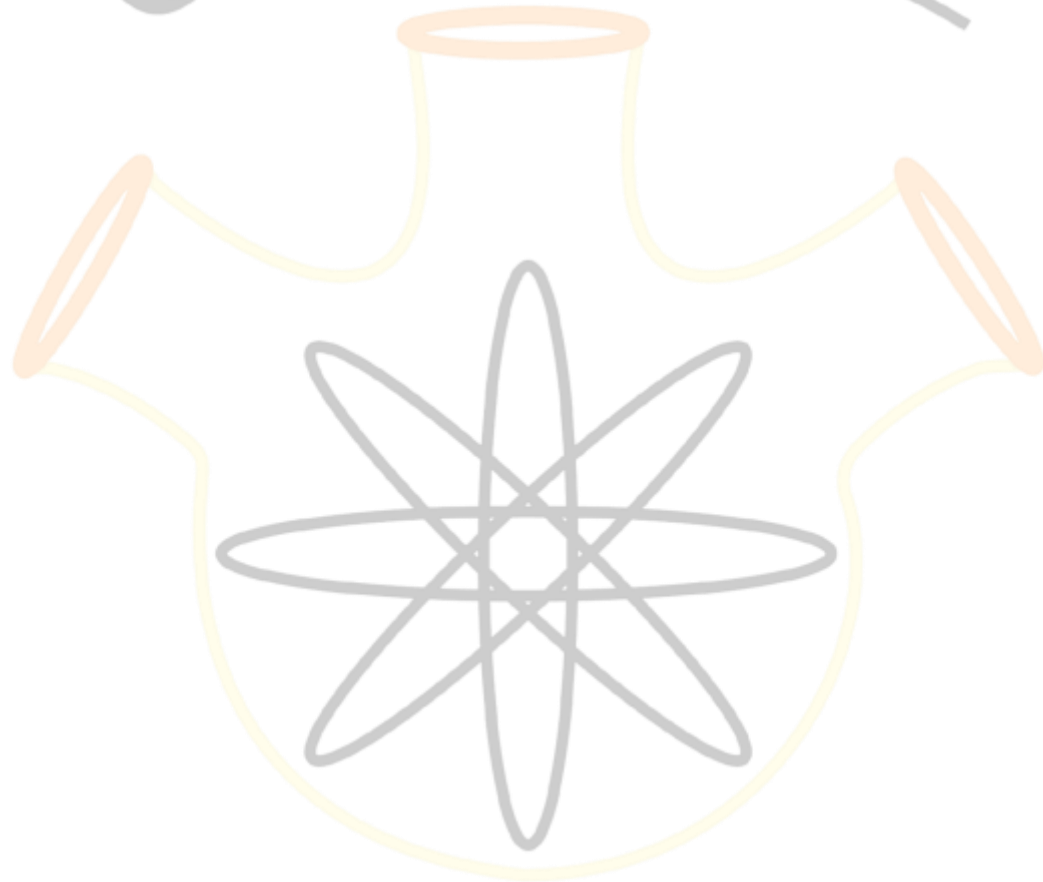
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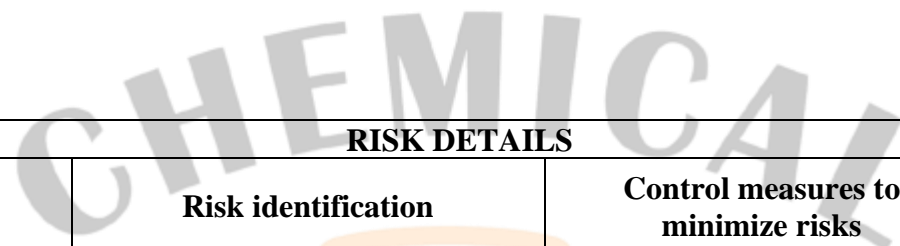
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HAZARD IDENTIFICATION (HI)											
A	Mechanic		D	Environment		E	Chemicals		G	Other dangers	
A1	Manual handling		D1	Noise		E1	Poison	√	G1	Compressed gas	
A2	Moving parts		D2	Vibration		E2	Irritant	√	G2	Ionizing radiation	
A3	Rotating parts		D3	Lighting		E3	Corrosive	√	G3	UV radiation	
A4	Cutting		D4	Moisture		E4	Carcinogenic	√	G4	Fatigue	
B	Biology		D5	Temperature	√	E5	Flammable	√	G5	Narrow space	
B1	Bacteria		D6	Travel hazards		E6	Explosive		G6	Overcrowded	
B2	Virus		D7	Slippery surface	√	E7	Cryogenics		G7	Thermometer	
B3	Mushroom		D8	Solid waste		F	Equipment				
C	Electricity		D9	Air quality	√	F1	Pressure vessels				
C1	High voltage		D10	Solitary work		F2	Hot equipment	√			
C2	Static electricity		D11	Sparks/droplets/floods	√	F3	Laser				
C3	Cable	√	D12	Powder spills	√	F4	Glass vessels				

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RISK DETAILS							
HI	Risk (after control measures)				Risk identification	Control measures to minimize risks	First aid measures
	High	Medium	Low	Minimum			
1. PREPARATION / INITIAL STAGE							
		√			<ul style="list-style-type: none"> - Reagents spill when measuring density using picnometers - Reagents spilled when weighing - Reagent spilled when inserting the titrant into the burette 	Using complete personal protective equipment such as lab coats, latex gloves, and wearing shoes	<ul style="list-style-type: none"> - Stop the source of the spill - Stay away from reagent spills - Clean spills with proper absorbents and use full personal protective equipment - Rinse under running water if the reagent gets on the skin
			√		Picnometers, measuring cups and pipettes fall during sampling	<ul style="list-style-type: none"> - Using complete personal protective equipment such as lab coats, latex gloves, and wearing shoes - Be careful when moving or moving tools so they don't get scuffed 	<ul style="list-style-type: none"> - Away from the place where the tool fell - Cleaning tool fragments with a broom - When cleaning the fragments of the tool need to use complete personal protective equipment
		√			The tip of the pipette breaks when taking reagents or samples	Be careful when taking the reagent so that the tip of the pipette does not come into contact with the lip of the reagent bottle	<ul style="list-style-type: none"> - Using full personal protective equipment - If it breaks and comes into contact with droplets, rinse immediately under running water - Removing clothes affected by droplets
		√			<ul style="list-style-type: none"> - Exposed to droplets or acid spills when take from acid chamber 	<ul style="list-style-type: none"> - Using full personal protective equipment such as lab coats, latex gloves, 	<ul style="list-style-type: none"> - Rinse under running water if the reagent gets on the skin - Removing clothing affected by

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RISK DETAILS							
HI	Risk (after control measures)				Risk identification	Control measures to minimize risks	First aid measures
	High	Medium	Low	Minimum			
					<ul style="list-style-type: none"> - Exposed to acid droplets from a pipette that breaks the tip 	<ul style="list-style-type: none"> - as well as wearing shoes - The presence of supervision when taking reagents from the acid chamber 	reagents
			√		Installation of the tool <ul style="list-style-type: none"> - The tool is dropped because it is not firmly attached - Thermometer rupture due to forcing in the installation 	<ul style="list-style-type: none"> - Ensuring clamps firmly hold the tool - Installation of the tool should not be oblique - Installing the tool according to the available sizes 	<ul style="list-style-type: none"> - Away from the place where the tool fell - Cleaning tool fragments with a broom - When cleaning the fragments of the tool need to use complete personal protective equipment
2. MAIN EXPERIMENTS							
				√	Electric shock when connecting an electric stove to a power source	<ul style="list-style-type: none"> - Make sure the power source cables and plugs are not wet - Wearing full personal protective equipment 	<ul style="list-style-type: none"> - Turn off the source of electric current - Push the victim's body with an insulator object - Seek medical attention if there are burns
			√		The temperature of the three-neck flask and the temperature of the alcohol are too hot so that excessive evaporation occurs	<ul style="list-style-type: none"> - Controlling the flow of water in the reverse cooler - Setting the temperature of the electric stove so that it 	Regulating the temperature in the device circuit with isolator protection

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RISK DETAILS							
HI	Risk (after control measures)				Risk identification	Control measures to minimize risks	First aid measures
	High	Medium	Low	Minimum			
						is not too high but still according to variables	
3. ANALYSIS / FINAL STAGE							
		√			<ul style="list-style-type: none"> - Exposed to droplets of the solution when taking samples - Exposed to drops of the solution when adding indicators - Exposed to titrant droplets when titrating 	Using complete personal protective equipment such as lab coats, latex gloves, and wearing shoes	<ul style="list-style-type: none"> - Rinse under running water if the reagent gets on the skin - Removing clothing affected by reagents
			√		When washing tools that have been used, the tool falls and breaks because it uses laundry soap that can make it slippery	<ul style="list-style-type: none"> - Be careful when washing so that the tool does not slip out of the hands - Be careful when washing so that the tool does not get hit by the sink 	<ul style="list-style-type: none"> - Away from the place where the tool fell - Cleaning tool fragments with a broom - When cleaning the fragments of the tool need to use complete personal protective equipment