CHAPTER I
INTRODUCTION

1.1 Background

Starch and its derivatives are versatile materials widely used in various industries, including beverages, processed foods, paper, animal feed, pharmaceuticals, chemicals, and non-food industries such as textiles, detergents, packaging, and others. In the food industry, starch functions as a gel-forming agent and an encapsulating agent. In the paper industry, it is used as an additive such as wet-end surfactant size and coating binder, adhesive material, and glass fiber sizing (Chiu & Solarek, 2009).

Hydrolysis reactions are generally carried out using acid catalysts such as HCl (hydrochloric acid). The main material used in the hydrolysis process is starch. In Indonesia, many starch-producing plants can be found, such as rice, corn, cassava, tubers, palm (aren), and others (Baskar & Muthukumaran, 2008). Starch and its derivatives are widely utilized in both food and non-food industries. In non-food industries, starch is used in metal processing, textiles, cosmetics and pharmaceuticals, paper, construction, and mining industries. In the textile industry, starch is used as an adhesive. It can also be applied as a wrinkle-reducing agent for fabrics. In the chemical sector, starch and its derivatives are applied in the production of biodegradable plastics, surfactants, polyurethane, resins, chemical compounds, and pharmaceuticals (Yetti et al., 2007).

In other sectors, starch and its derivatives are utilized as non-toxic and skin-safe detergent ingredients, binders, solvents, biopesticides, lubricants, colorants, and flavoring agents. In the food industry, starch is commonly used as a thickener, colloid stabilizer, gel-forming agent, adhesive, and water-retaining agent. Specifically, in the food sector, starch plays an important role in the production of baby food, cakes, pudding, milk thickeners, jelly candies, and dextrin (Hill, 1997).

1.2 Problem Formulation

Starch provides significant benefits in human life. However, starch requires proper treatment before processing or utilization due to its relatively complex composition. Therefore, it is important for a Chemical Engineering student to understand how to modify starch composition so that it can be utilized optimally for human needs. In this experiment, the effect of hydrolysis temperature on the starch hydrolysis reaction and the reaction rate constant will be studied.

1.3 Experiment Goals

1. To study the effect of variables on the starch hydrolysis reaction.
2. To calculate the reaction rate constant and analyze the effect of variables on the reaction rate constant.
3. To compare the calculated reaction rate constant with values reported in the literature.

1.4 Experiment Benefits

1. Students can understand the effect of variables on the starch hydrolysis reaction.
2. Students can calculate the reaction rate constant and analyze the effect of variables on the reaction rate constant.
3. Students can compare the calculated reaction rate constant with literature values.

CHAPTER II
LITERATURE REVIEW

2.1 Starch

Starch is a homopolymer of glucose composed of dehydrated glucose units linked together through glycosidic bonds. Each dehydrated glucose unit contains three hydroxyl groups located at positions C-2, C-3, and C-6. Among these, the primary hydroxyl group at carbon 6 exhibits the highest reactivity, followed by the secondary hydroxyl groups at carbons 2 and 3. Starch consists of two main fractions: amylose and amylopectin. The degree of polymerization of native starch ranges from approximately 160 to 6,000, with a relative molecular weight of about 25,000 to 1,000,000. Starch is insoluble in water and has limited intermolecular macromolecular interaction capability. In addition, starch has limitations in terms of processability, dimensional stability, and the mechanical properties of its final products. Therefore, native starch is generally not used directly without modification (Ozkan et al., 2019).

2.2 Amylose and Amylopectin

Starch belongs to the group of polysaccharides, which are glucose polymers composed of amylose and amylopectin. Amylose is a linear long-chain molecule with α-(1,4) glycosidic bonds and contributes to the amorphous matrix of starch. Amylopectin is a branched molecule with a double-helix structure and α-(1,6) branching points, which give starch its semi-crystalline structure. Amylopectin accounts for approximately 70–85% of total starch content and has a much larger molecular size (10^7–10^9 Da) compared to amylose (10^5–10^6 Da) (Compart et al., 2023).

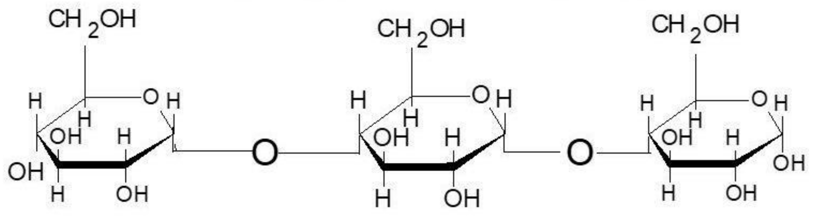

Figure 2.1 Structure of Amylose

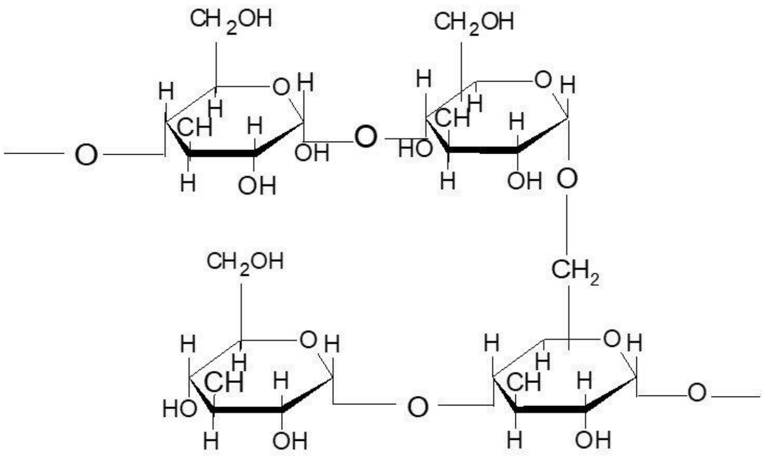

Figure 2.2 Structure of Amylopectin

2.3 Starch Hydrolysis

2.3.1 Kinetics of Starch Hydrolysis Reaction

Hydrolysis is a reaction involving the attachment of a hydroxyl group (–OH) to a compound. The –OH group is generally derived from water molecules. Hydrolysis can be classified into pure hydrolysis, acid-catalyzed hydrolysis, base-catalyzed hydrolysis, combined alkali-water hydrolysis, and enzymatic hydrolysis. Based on the reaction phase, hydrolysis is categorized into liquid-phase hydrolysis and vapor-phase hydrolysis.

Starch hydrolysis is the process of breaking down starch molecules into simpler components such as dextrin, isomaltose, maltose, and glucose. This reaction occurs between starch as the reactant and water as the co-reactant. The reaction is considered first-order because water is present in excess, so its concentration change can be neglected. The overall reaction is:

$$(C_6H_{10}O_5)_X + H_2O \rightarrow x\ C_6H_{12}O_6$$

Based on reaction rate theory:

$$-r_A = k.C_{starch}.C_{water} \qquad (2.1)$$

Since the water volume is sufficiently large, the water concentration during the reaction can be considered constant and incorporated into a new constant k′:

$$k^{'} = k.C_{water} \qquad (2.2)$$

Thus, Equation (2.1) can be rewritten as $-r_A = k.C_{starch}$. From this rate equation, starch hydrolysis is classified as a first-order reaction. If $-r_A = -\frac{dC_A}{dt}$ then Equation (2.2) becomes:

$$\frac{-dC_A}{dt} = k'C_A \qquad (2.3)$$

$$\frac{-dC_A}{C_A} = k'dt \tag{2.4}$$

If $C_A = C_{A0}(1 - X_A)$ and the equation is solved by integration with the boundary conditions t_1: C_{A0} dan t_2: C_A, the following equation is obtained:

$$-\int_{C_{A0}}^{C_A} \frac{dC_A}{C_A} = k' \int_{t_2}^{t_1} dt \tag{2.5}$$

$$\ln \frac{C_{A0}}{C_A} = k'(t_2 - t_1) \tag{2.6}$$

$$\ln \frac{1}{(1-X_A)} = k'(t_2 - t_1) \tag{2.7}$$

Where X_A is the reaction conversion after one second.

Equation (2.7) can be solved using a linear regression approach $y = mx + c$, where $y = \ln \frac{1}{(1-X_A)}$ and $x = t_2$.

2.3.2 Starch Hydrolysis Methods

The process of starch decomposition is known as starch hydrolysis. Starch hydrolysis is divided into two methods: acid hydrolysis and enzymatic hydrolysis.

1. Acid Hydrolysis

   Acid hydrolysis is a process that uses acids to convert polysaccharides (such as starch and cellulose) into sugars. This hydrolysis is carried out using H^+ catalysts derived from acids such as HCl, H_2SO_4, and HNO_3 (Wang & Copeland, 2015). The acid hydrolysis method is relatively simpler because it does not require multiple stages, as in enzymatic hydrolysis. In addition, acid hydrolysis requires a relatively shorter processing time, simpler technology, easier process condition control, and lower costs (Devitria & Sepriyani, 2018).

2. Enzymatic Hydrolysis

   Enzymatic hydrolysis is the process of converting cellulose and hemicellulose into reducing sugars using enzymes. The enzyme used to convert hemicellulose into glucose is xylanase, while the enzyme used to convert cellulose into glucose is cellulase. Enzymatic hydrolysis can specifically cleave glycosidic bonds without leaving harmful residues (Salsabilla & Fahruroji, 2021).

2.4 Starch Modification

The application of native starch is often limited due to its low solubility, low water absorption capacity, and the turbidity of the gel produced. Amylose tends to undergo retrogradation, forming rigid gels, while amylopectin forms softer gels, resulting in low thermal resistance and increased thermal decomposition rates. In addition, native starch has limited functional stability against variations in pH and temperature during processing. Some starch granules are inert, insoluble in water at room temperature, and highly resistant to enzymatic degradation, preventing them from performing their functional applications optimally (Mauro et al., 2023).

Various limitations of native starch can be overcome through starch modification using conventional methods such as thermal, chemical, and enzymatic treatments, as well as modern non-thermal methods including high-pressure treatment, ultrasonic waves, plasma treatment, and electric field application. Starch is generally modified to improve its physical and chemical properties in order to obtain the desired functional characteristics. The physicochemical properties of starch that may be affected after modification include solubility, light transmittance value, rheological properties, thermal properties, and the crystalline structure of starch granules (Hutabarat & Stevensen, 2023).

2.5 Fehling’s Reagent in the Analysis of Sugars from Hydrolysis

2.5.1 Composition and Function of Fehling’s Reagent

Fehling’s reagent is a chemical solution used to detect the presence of reducing sugars such as glucose in a solution. This reagent consists of two components, namely Fehling A and Fehling B, which are mixed immediately before use to maintain solution stability. Fehling A is a copper(II) sulfate ($CuSO_4$) solution, blue in color, and serves as the source of Cu^{2+} ions. Fehling B is a mixture of sodium potassium tartrate (Rochelle salt) and sodium hydroxide (NaOH), which functions as an alkaline medium and stabilizes Cu^{2+} ions to keep them soluble under alkaline conditions (Aljamali et al., 2025).

2.5.2 Reaction Mechanism

The Fehling method relies on the reduction of copper(II)-tartrate complex in a strongly alkaline medium using methylene blue as an indicator. The titration endpoint is determined when the blue color of methylene blue disappears. When Fehling A and Fehling B are mixed, a light blue precipitate of copper(II) hydroxide ($Cu(OH)_2$) is initially formed. This precipitate then reacts with sodium potassium tartrate to form a deep blue copper–tartrate complex.

When the Fehling solution is heated in the presence of a reducing sugar, the copper–tartrate complex is reduced to a brick-red precipitate of copper(I) oxide (Cu_2O). The bis-tartratocuprate(II) complex oxidizes the aldehyde group into a carboxylate ion, while Cu^{2+} ions are reduced to Cu^{+} ions. The formation of the brick-red Cu_2O precipitate indicates a positive result, meaning that the reducing sugar has been oxidized. The color change from blue to brick-red indicates that the titration endpoint has been reached. The chemical reactions involved are as follows (Zhang & Chen, 2020):

$$CuSO_4 + 2NaOH \rightarrow Cu(OH)_2 \downarrow \text{(light blue)} + Na_2SO_4$$

$$CH_2OH(CHOH)_4CHO + 2Cu(OH)_2 \rightarrow CH_2OH(CHOH)_4COOH + Cu_2O \downarrow \text{(brick red)} + H_2$$

2.6 Influential Variables

The variables that influence the starch hydrolysis reaction include:

1. Catalyst

Hydrolysis is a relatively slow reaction; therefore, a catalyst is required to accelerate the reaction. The catalyst used can be either an acid or an enzyme due to their faster performance. Various acids can be used, ranging from HCl and H_2SO_4 to HNO_3 (Nasution et al., 2023). The reaction rate is influenced by the concentration of H^+ ions rather than the type of acid used. However, in industrial applications, hydrochloric acid is commonly used (Zuhair Ds., 2022). This selection is based on the fact that the salt formed during neutralization does not cause significant problems, except for a salty taste if present in high concentration. Therefore, the acid concentration in the hydrolysis medium is kept as low as possible. Generally, acid solutions with higher concentrations than those used in syrup production are applied. Hydrolysis at atmospheric pressure (1 atm) requires a much more concentrated acid.

2. Temperature

   The effect of temperature on the reaction rate follows the Arrhenius equation, where a higher temperature results in a faster reaction rate. The optimum temperature in the hydrolysis reaction leads to lower activation energy, allowing for higher hydrolysis conversion (Milek & Lamkiewicz, 2022). To achieve a certain conversion, approximately 48 minutes are required to hydrolyze cassava starch at 100°C (Ardiansyah et al., 2018). Meanwhile, the hydrolysis of wheat and corn starch using an H_2SO_4 catalyst requires a temperature of 160°C.

3. Mixing (Agitation)

   Mixing is required to enhance contact between reactants and accelerate the hydrolysis rate. At the initial stage, agitation helps reduce mass transfer resistance, especially in systems with high solid concentrations. An appropriate stirring speed can improve reaction efficiency, whereas excessive agitation may reduce efficiency due to excessive shear forces. Increasing the agitation speed from 0 to 150 rpm can enhance glucose yield; however, increasing it further to 200 rpm may reduce hydrolysis efficiency (Guo et al., 2015).

4. Reactant Ratio

   If one of the reactants is present in excess, the equilibrium can shift favorably toward the products. Therefore, starch suspensions with lower concentrations provide better results compared to higher concentrations. When the starch suspension concentration is reduced from 40% to 20% or 1%, the conversion increases from 80% to 87% or 99%, respectively (Groggins, 1958). At high suspension concentrations, reactant molecules have limited mobility due to crowding effects. To produce glucose, a starch suspension of approximately 20% is generally used.

CHAPTER III

EXPERIMENTAL METHOD

3.1 Experimental Design

3.1.1 Experimental Setup

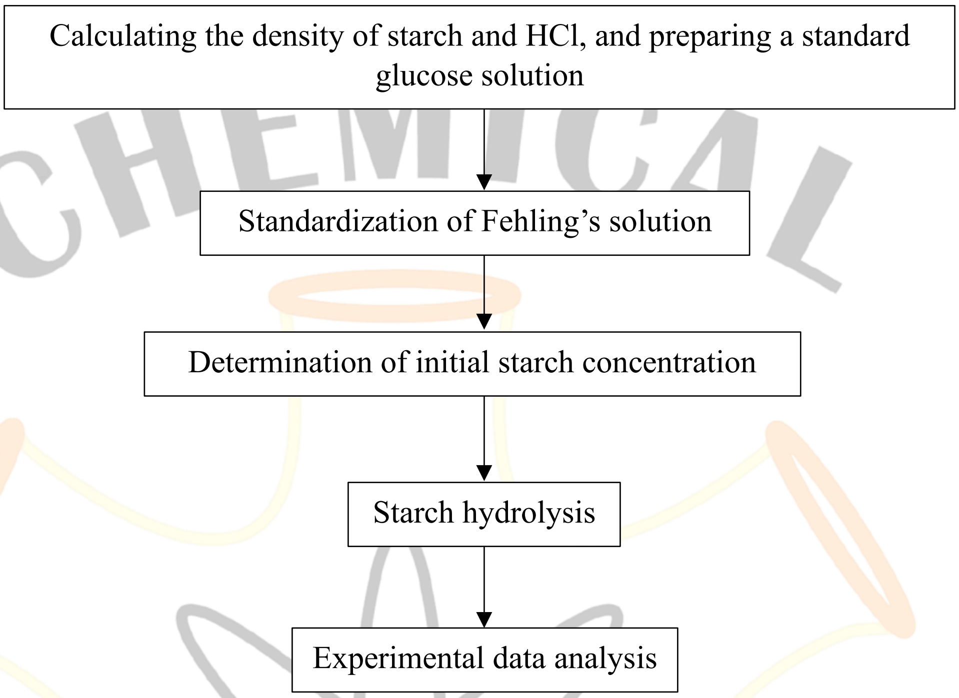

Figure 3.1 Experimental Setup Scheme

3.1.2 Determination of Variables

a. Controlled Variables :

b. Independent Variables :

3.2 Materials and Equipment

3.2.1 Materials

1. Anhydrous glucose
2. Starch powder
3. NaOH
4. HCl
5. Methylene blue (MB) indicator
6. Fehling A solution
7. Fehling B solution
8. Aquadest

3.2.2 Equipment

1. Graduated cylinder
2. Thermometer
3. Erlenmeyer flask

4. Retort stand and clamp
5. Burette
6. Three-neck round-bottom flask
7. Volumetric flask

3.3 Apparatus Diagram

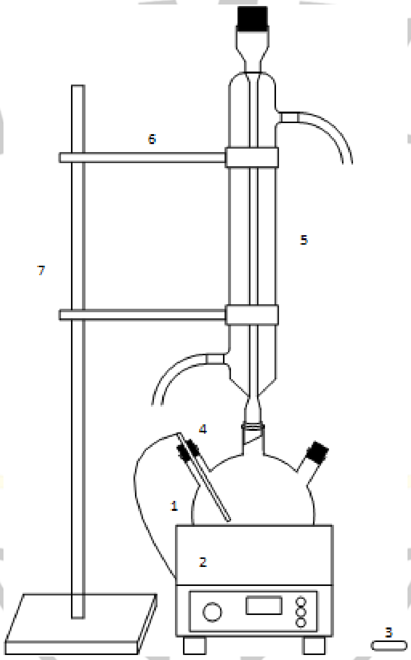

Figure 3.2 Hydrolysis Apparatus Setup

Description:

1. Three-neck round-bottom flask
2. Heating Mantle
3. Stirrer
4. Thermocouple
5. Reflux condenser
6. Clamp
7. Retort stand

3.4 Experimental Procedure

1. Initial Preparation
    a. Determination of Starch Density

    Add 1 gram of starch into a graduated cylinder containing 5 mL of distilled water. Record the change in volume.

$$\rho \text{ starch} = \frac{\text{m starch}}{\Delta \text{V}} \tag{3.1}$$

    b. Determination of HCl Density

Weigh the empty pycnometer (m_1). Fill the pycnometer (with known volume, V) with HCl solution and weigh it again (m_2). Calculate the density of HCl as follows:

$$\rho\ \text{HCl} = \frac{m_2 - m_1}{\Delta V} \tag{3.2}$$

c. Preparation of Standard Glucose Solution

Dissolve 2 grams of anhydrous glucose in 1000 mL of distilled water.

2. Determination of Starch Content

a. Standardization of Fehling's Solution

Mix 10 mL of Fehling A, 10 mL of Fehling B, and 15 mL of standard glucose solution. Heat the mixture until boiling. After boiling, add 3 drops of methylene blue (MB) indicator. Titrate with the standard glucose solution until the color changes to brick red. Record the volume of titrant required (F). The titration must be carried out under boiling conditions (on a hot plate or stove).

b. Determination of Initial Starch Content

Weigh … grams of starch and add … mL of HCl/H_2SO_4 catalyst and … mL of distilled water into a three-neck round-bottom flask. Heat the mixture to … °C for 1 hour. After cooling, dilute the solution with distilled water to 500 mL. Take 20 mL of the solution and neutralize with NaOH until pH = 7. Take 5 mL of the neutralized solution and dilute to 100 mL. Then take 10 mL of this diluted solution. Into an Erlenmeyer flask, add 10 mL sample solution, 10 mL Fehling A, 10 mL Fehling B, and 15 mL standard glucose. Heat to 60 °C and add 3 drops of MB indicator. Titrate with standard glucose solution until the color changes to brick red. Record the required titrant volume (M). The titration must be conducted under boiling conditions. Repeat the same procedure for other variables.

c. Starch Hydrolysis

Weigh … grams of starch and add … mL of HCl/H_2SO_4 catalyst and … mL of distilled water into a three-neck round-bottom flask. Heat the mixture to … °C. After 5 minutes, withdraw a 20 mL sample. Neutralize the sample with NaOH until pH = 7. Take 5 mL of the neutralized solution and dilute to 100 mL. Then take 10 mL of the diluted solution. Into an Erlenmeyer flask, add 10 mL sample solution, 10 mL Fehling A, 10 mL Fehling B, and 15 mL standard glucose. Heat to 60 °C and add 3 drops of MB indicator. Titrate with standard glucose solution until the color changes to brick red. Record the titrant volume required (M). Sampling is conducted every 5 minutes for a total of 25 minutes (t_1 = 5 min, t_2 = 10 min, t_3 =15 min, t_4 = 20 min, t_5 = 25 min). Repeat the same procedure for the second variable.

Determination of initial starch content:

$$X_{P0} = \frac{(F-M) \times N\ \text{glucose} \times \frac{500}{\text{basis}} \times \frac{100}{5} \times 0{,}9}{W} \tag{3.3}$$

Where,

N = 0,002 gr/mL

W = weight of starch

Reagent requirement calculations:

a) Calculation of HCl requirement

$$V_{HCl} = \frac{N\ \text{HCl} \times \text{MW HCl} \times \text{V Solution}}{\rho\ \text{HCl} \times \text{HCl content} \times 1000 \times \text{greq}} \tag{3.4}$$

b) Calculation of starch requirement

$$\%\text{suspension} = \frac{X_p \times \text{W starch}}{\text{W starch} + \text{W HCl} + \text{W aq}} \tag{3.5}$$

Where,

$$W_{starch} = \rho_{starch} \times V_{starch} \tag{3.6}$$

$$W_{HCl} = \rho_{HCl} \times V_{HCl} \tag{3.7}$$

$$W_{aq} = \rho_{aq} \times (V_{solution} - V_{starch} - V_{HCl}) \tag{3.8}$$

$$X_{P0} = \frac{(F-M) \times N\ \text{glucose} \times \frac{500}{\text{basis}} \times \frac{100}{5} \times 0{,}9}{W} \tag{3.9}$$

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HAZARD IDENTIFICATION AND RISK ANALYSIS

MATERIAL : STARCH HYDROLYSIS

HAZARD IDENTIFICATION

A	**Mechanical**		**D**	**Environment**		**E**	**Chemical**		**G**	**Other risks**	
A1	Manual handling		D1	Noise		E1	Toxicant	√	G1	Compressed gas	
A2	Moving parts		D2	Vibration		E2	Irritant	√	G2	Ionising radiation	
A3	Rotating parts		D3	Lighting		E3	Corrosive		G3	UV radiation	
A4	Cutting		D4	Humidity		E4	Carcinogenic		G4	Fatigue	
B	**Biology**		D5	Temperature	√	E5	Flammable		G5	Cramped space	
B1	Bacteria		D6	Dangers of travel		E6	Explosive		G6	Crowded	
B2	Virus		D7	Slippery surface	√	E7	Cryogenics		G7	Thermometer	
B3	Mold		D8	Solid waste		**F**	**Equipment**				
C	**Electrical**		D9	Air quality		F1	Pressure vessel				
C1	High voltage	√	D10	Solitary work		F2	Heating equipment	√			
C2	Static electricity		D11	Splashes/drops/floods	√	F3	Laser				

C3	Cable	√	D12	Pile of powder	√	F4	Glass vessel			

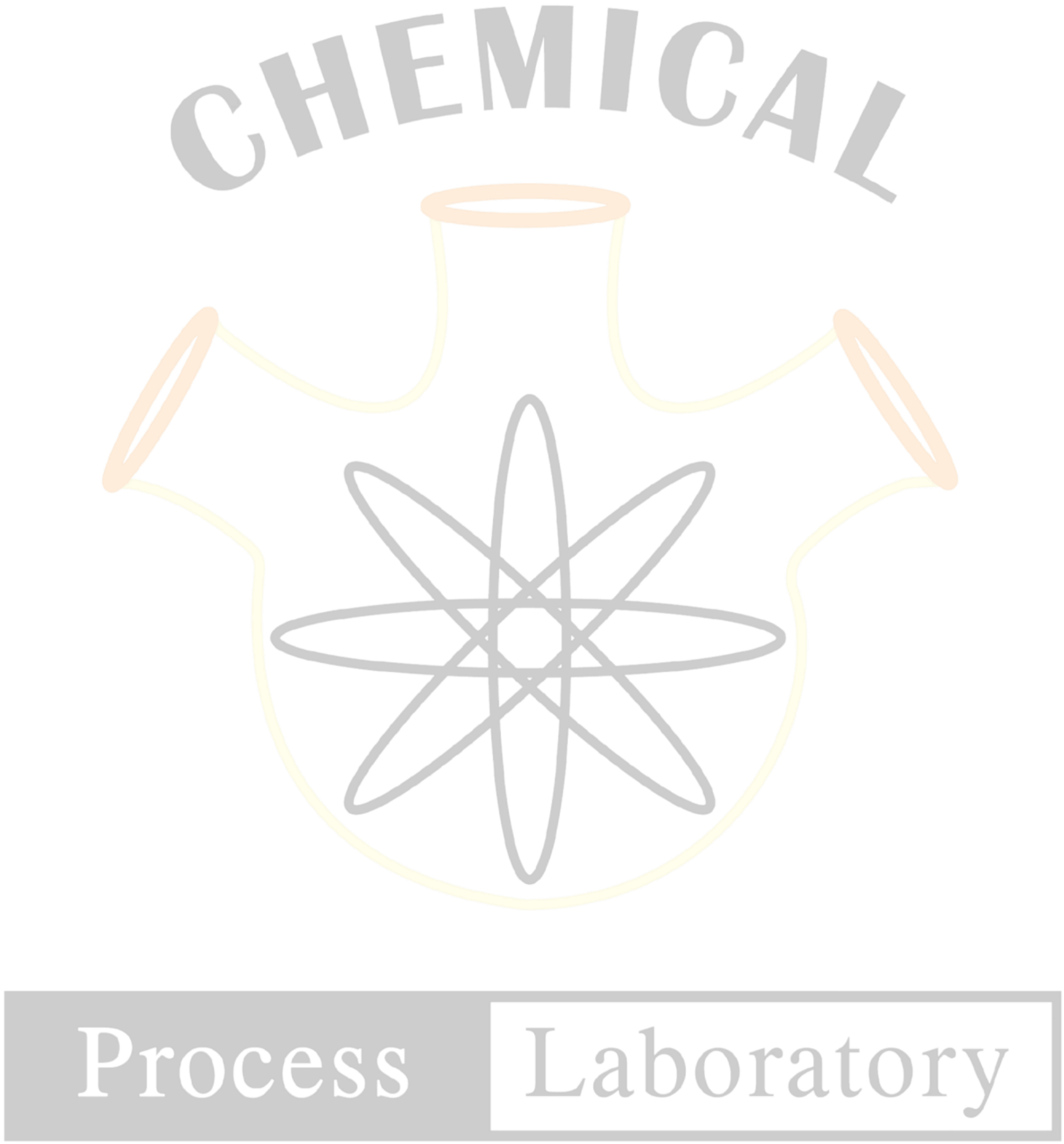
RISK ANALYSIS

IB	**Risks (after control measures)**				**Risk identification**	**Control measures to minimise risk**	**First aid measures**
	High	**Medium**	**Low**	**Minimal**			
1. PREPARATION / INITIAL PRACTICAL							
D7, D11				√	During pycnometer calibration and reagent preparation, there is a risk of distilled water (aquadest) spilling, which may cause the working surface or floor to become slippery.	Exercise caution when handling distilled water. If transferring distilled water to containers or areas prone to spillage, use a funnel to minimize the risk of creating slippery surfaces.	If a slip occurs, examine the injured area and provide appropriate first aid treatment. If the injury is severe or worsens, immediately take the victim to the nearest hospital or clinic.

D12				√	During the starch density calculation, there is a risk that spilled starch powder may contaminate the laboratory area and cause respiratory irritation or obstruction if inhaled.	Exercise caution when transferring starch powder to prevent spillage.	Clean up any spilled starch powder immediately. If inhaled, remove the exposed person from the contaminated area and ensure the airway is clear. If the condition worsens, take the victim to the nearest hospital or clinic for further medical attention.
2. MAIN PRACTICAL							
C1, C3		√			The presence of damaged or exposed electrical cables may result in short circuits or electric shock.	Conduct proper inspection and ensure that all equipment is in good and safe working condition before use.	Disconnect the power source immediately to stop the flow of electricity. .

D5, F2		√			There is a risk of contact with the electric stove or equipment heated on the electric stove, which may cause burns.	Exercise caution with hot surfaces during the heating process and ensure that the apparatus is positioned in a secure location to avoid any accidental contact.	Discontinue the heating process, cool the burn, and administer an analgesic. If the condition persists, proceed to the nearest clinic or hospital
RISK ANALYSIS							
IB	**Risks (after control measures)**				**Risk identification**	**Control measures to minimise risk**	**First aid measures**
	High	**Medium**	**Low**	**Minimal**			

E1, E2		√			The use of acidic and basic reagents in this laboratory practice carries the risk of skin contact, which may cause irritation, and accidental ingestion may result in poisoning.	Wear latex gloves during the laboratory session and replace them if torn or punctured, while exercising caution when handling acidic and basic reagents.	If the reagent comes into contact with the skin, immediately wash the affected area thoroughly with running water. If ingested, promptly drink mineral water and rinse the mouth to minimize the hazard. If the condition worsens, take the individual to the nearest hospital or clinic.
3. ANALYS / FINAL PRACTICAL							
D7, D11				√	During titration, there is a risk of solution spillage, which may result in slippery surfaces.	Exercise caution when handling distilled water. If directing it toward an area prone to spillage, use a funnel to minimize the risk of creating slippery surfaces.	If a slip occurs, examine the injured area and provide appropriate treatment. If the injury is severe or worsens, take the individual to the nearest hospital or clinic.


CHEMICAL
Process Laboratory