LABORATORY GUIDE

CHEMICAL SEPARATION METHODS (CHEM 4141)

By:

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LABORATORY RULES

These rules have been established to ensure order and smooth running during the practical session.

1. Students must be present in the laboratory no later than 5 (five) minutes before the practical session begins.
2. Before entering the laboratory, students must have prepared the necessary equipment, such as: fine laboratory tools, coarse laboratory tools, test tube brushes, cleaning fluid (detergent), and writing materials.
3. Upon entering the laboratory, lab coats must be worn.
4. During the practical session, eating, drinking, smoking, chatting and leaving the laboratory (pacing about) without the permission of the lecturer or assistant on duty are not permitted.
5. The borrowing of laboratory equipment must always be accompanied by a borrowing slip signed by the lecturer or assistant on duty.
6. Any equipment that is damaged or broken must be replaced no later than before the final examination takes place.
7. If unable to attend, students must notify the laboratory coordinator of their absence. Should a student miss three practical sessions, they will not be permitted to attend subsequent practical sessions.
8. Students are not permitted to join other classes or groups during the time allocated for each student, unless authorised by the practical coordinator.
9. Matters not covered in these regulations will be addressed at a later date.
10. Violators of these regulations will be subject to sanctions in the form of cancellation of the experiment.

Kendari,

Practical Coordinator

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EXPERIMENT I
SIMPLE DISTILLATION

A. Basic Theory

Distillation is used to purify liquids based on differences in boiling points. In this process, the liquid is converted into vapour, which is a pure substance. The vapour is then cooled. During cooling, the vapour condenses into a pure liquid known as the distillate. Distillation can be used to obtain a pure solvent from a solution containing a solute.

Distillation is a separation method based on Raoult's Law and the difference in boiling points. To discuss distillation, one must also study the vapour-liquid phase equilibrium; this equilibrium depends on the vapour pressure of the solution. Raoult's Law is used to explain the phenomena occurring in separation processes that utilise the distillation method; it states that the vapour pressure of a component evaporating from a solution is equal to the vapour pressure of the pure component multiplied by the molar fraction of the component evaporating from the solution, at the same temperature. If the solution contains component A, which

has a pure vapour pressure P^o, then$_A$ the vapour pressure of component A in the solution

is:

$P_A = P^oA.X_A$

Where:

P_A = the vapour pressure above the solution

P^o_A = vapour pressure of pure A

X_A = Molar fraction of A

If the solution is a binary mixture and is an ideal solution, then the vapour pressure of the binary vapour mixture of A and B is the sum of their vapour pressures.

$P_{total} = P_A + P_B = P°_A X_A = P°_A X_A$

B. Objective: To understand the basic principles of the distillation process in a simple way

C. Apparatus and Materials

Apparatus : distillation flask, cork/rubber stopper with a hole, *thermometer*, *electric heating mantle*, measuring cylinder, stand and clamp, condenser, boiling stones, Erlenmeyer flask, beaker, adapter, and rubber tubing.

Materials : sample to be distilled (e.g. ethanol-distilled water) and paraffin.

D. Procedure

1. Set up the distillation apparatus as shown in the diagram.
2. Fill the distillation flask with 50 ml each of distilled water and ethanol
3. Then add a few boiling stones.
4. Pass water through the condenser and then heat the distillation flask until the mixture boils.
5. Observe the rise in temperature on the thermometer and read the boiling point of the distillate.
6. Measure the volume of the distillate obtained.

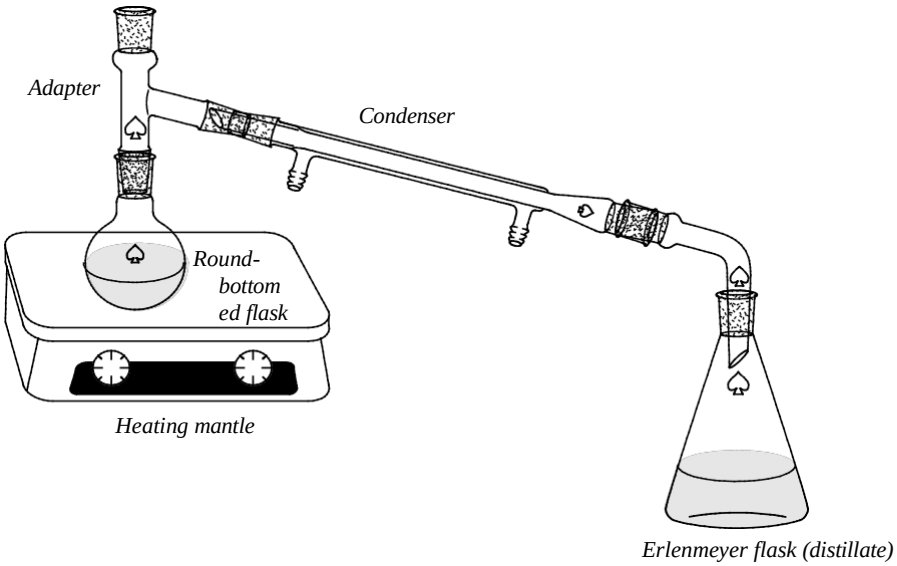

Figure 1. Simple distillation apparatus

EXPERIMENT II

EXTRACTION OF NATURAL SUBSTANCES

Basic Theory:

Natural materials are still used in medicine today and are even used as a source of study in drug development (Luo et al., 2014). The use of natural materials as medicines began as early as 2600 BC. The use of natural materials as traditional medicines has also pioneered studies on the active compounds found in natural materials that can be utilised to treat various diseases.

Extraction is a process of obtaining the desired active components from a material by separating one or more components from the material that is the source of those components (Ahmad, 2006). The extraction method depends on the polarity of the compound to be extracted. Materials and chemical compounds will dissolve easily in solvents of relatively similar polarity. The principle of solubility applied is 'like dissolves like', meaning that a polar solvent will dissolve polar compounds and a non-polar solvent will dissolve non-polar compounds (Khopkar, 2009).

Maceration is the simplest extraction method; it is carried out by soaking the sample in a solvent. In maceration extraction, the solvent penetrates the cell walls and enters the cell cavities containing the active substances, which then dissolve into the solvent. Due to the difference in concentration of the active substances within the cells, the concentrated solution will be released. The advantage of this maceration extraction method is that the procedure is simple and the equipment required is readily available (Ahmad, 2006).

Aims of the Experiment:

1. To determine how to obtain an extract from a natural material
2. To obtain an extract from a natural material using the maceration method

Equipment and Materials:

1. Equipment:

1. 150 mL beaker
2. 150 mL measuring cylinder
3. Jar
4. Dropper
5. Spatula

6. Stirring rod
7. Funnel
8. Analytical balance
9. Blender
10. *Cutter*
11. *Hot plate*

2. Material

1. Natural material sample (part of a plant, such as a leaf)
2. Methanol
3. Distilled water
4. Filter paper
5. Gauze
6. Tissues
7. *Aluminium foil*

Procedure:

1. Sample Preparation

The sample, consisting of leaves from a natural source, is washed thoroughly and then dried in the sun to remove moisture. The dried leaves are then cut into small pieces and ground using a blender to facilitate the extraction process.

2. Maceration Process

A 25-gram portion of the ground sample is weighed out and macerated with 100 ml of methanol. The maceration process is carried out for 1–2 hours, stirring occasionally. The filtrate is then collected via filtration. The resulting filtrate is heated to evaporate the solvent. It is then left to stand for a short while before being weighed, and the extraction yield is calculated.

EXPERIMENT III
PAPER CHROMATOGRAPHY

A. Basic Theory

Gordon, Martin and Synge have carried out partition chromatography using paper as the adsorbent. Consequently, this method of chromatography is known as paper chromatography.

A mixture of substances to be separated, in the form of a small drop, is placed a few centimetres from the edge of a strip of filter paper (Figure 6). Then, the edge (tip) of the filter paper closest to the drop of the mixture is dipped into a solvent system in such a way that the drop itself does not become submerged.

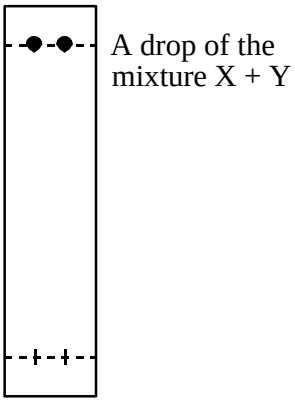

Figure 6. A simple paper chromatography technique

The solvent system (developing solution) usually consists of an organic solvent saturated with water. The solvent system is absorbed by the filter paper and moves along the paper, passing through the droplet of the substance to be separated. The water from the developing solution absorbed by the filter paper is adsorbed by the paper and becomes the stationary solvent or stationary phase. Meanwhile, the organic solvent from the developing solution acts as the mobile solvent or mobile phase.

After sufficient *development* time has elapsed to allow the solvent to travel a certain distance along the filter paper, the chromatogram is dried and then sprayed with a reagent. This reagent will produce a colour reaction with the various dissolved substances that have been separated from one another. The locations of these separated substances are indicated by specific coloured spots.

An important parameter in paper chromatography is the R_f value, given by the formula:

$$R_f = \frac{distance\ travelled\ by\ the\ solute}{solvent\ distance} = \frac{x}{p} = \frac{y}{p}$$

This Rf value is specific to each substance that has been separated in a particular solvent.

B. **Objective**: to determine the Rf value of a specific sample using paper chromatography

C. Apparatus and Materials

Apparatus : glass cylinder, Whatman filter paper, glassware

Materials : - samples containing Pb^{2+}, Ag^{+}, and Hg^{2+} ions

- standard solutions (4 mg/ml) in the form of chlorides: Pb^{2+} , Ag^{+} , and Hg^{2+}

Solvent : a mixture of 15% water, 10% ethyl acetoacetate, 75% n-butanol, and glacial acetic acid as required to achieve a pH range of 3.5–5.

D. Procedure

Place 50 ml of the solvent in a glass cylinder. Prepare a 25x25 cm sheet of Whatman paper. 2 cm from one edge, draw a line (with a pencil) divided into 6 sections and marked from number 1 to number 6. At marks 1, 3, and 5, apply drops of the standard solutions (Pb^{2+} , Ag^{+} , and Hg^{2+} respectively). At marks 2, 4, and 6, apply drops of the sample solution. Using tweezers, roll the paper into a cylinder. Place this paper cylinder into the provided glass cylinder, with the side of the paper containing drops of the substances at the bottom. Ensure the paper cylinder does not touch the glass walls. Seal the glass cylinder. Once the solvent level has risen to approximately three-quarters of the paper's height, remove the paper cylinder. Mark the solvent level with a pencil, release the paper clip and allow to dry. Spray this sheet with dilute K_2CrO_4. The colours of $PbCrO_4$, $AgCrO_4$, and $HgCr_4$ can be used to identify the respective ions in the solution. Mark the resulting stains and calculate R_f for the standard substance and the sample.

EXPERIMENT IV

SEPARATION OF *AMOXICILLIN* CONTAMINANTS USING THE ADSORPTION METHOD

Basic Theory:

The long-term presence of antibiotic residues in the environment will lead to bacterial resistance, posing a serious threat to public health. Harmful contaminants do not originate solely from industry; some also stem from medicines, one example being the use of amoxicillin. The inappropriate use of amoxicillin can result in a number of negative consequences. One of these is the emergence of resistant bacteria, necessitating the use of antibiotics with greater potency than this particular type. The issue of bacterial resistance to antibiotics has become a serious problem in the healthcare sector.

Adsorption is a method frequently used to remove contaminants from chemical industrial waste. Adsorption is considered highly competitive, cost-effective, and an efficient process for removing hazardous substances or other compounds.

Objective of the Experiment: to determine the effect of pH on the adsorption of amoxicillin using Ti_{O_2}.

Equipment:

1. 50 mL beaker
2. 150 mL and 5 mL measuring flasks
3. 1,000 mL volumetric flask
4. Dropper pipette
5. Analytical balance
6. Spatula
7. Stirring rod
8. Funnel
9. Stopwatch
10. UV-Vis spectrophotometer

Materials:

1. Titanium dioxide (TiO_2) *anatase*
2. *Amoxicillin* 30 ppm
3. Sodium hydroxide (NaOH) 0.1 N
4. Hydrochloric acid (HCl) 0.1 N
5. Distilled water
6. filter paper

Procedure:

1. Weigh 0.1 grams of TiO_2 and place it into 5 separate beakers
2. Add 50 mL of a 30 ppm amoxicillin solution, the pH of which has been adjusted to pH 3, 5, 7, 9 and 11 using HCl and NaOH solutions in each beaker
3. Homogenise the solutions by stirring for 30 minutes
4. Filter the homogenised solution using filter paper
5. Measure the absorbance of the filtrate using a *UV-Vis* spectrophotometer at a wavelength of 240 nm.

EXPERIMENT V
SOLID-LIQUID EXTRACTION

A. Basic Theory

If one component of a mixture is a solid that is highly soluble in a specific solvent and the other component is specifically insoluble, the separation process can be carried out by simple stirring with a specific solvent, followed by filtration. However, if the soluble component is only slightly soluble or, due to its form, the dissolution process is very slow, separation must be carried out using Soxhlet extraction.

B. **Objective:** to determine the fat and oil content in fish or candlenuts

C. Apparatus and Materials

Equipment : solid-liquid extraction apparatus (Soxhlet)

Materials : petroleum ether, anhydrous$_{CaCl_2}$, fish or candlenut samples

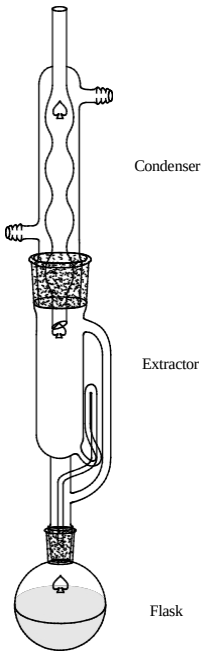

Figure 2. Solid-liquid extraction apparatus (Soxhlet)

D. Procedure

a. Fish oil extraction

Weigh approximately 50 grams of fish meat that has been cut into pieces and dried in an oven for 1 day at a temperature of 110°C. The dried fish meat

is then weighed to determine its moisture content. The fish meat is subsequently ground with a mortar until soft. Carefully transfer the softened fish meat into a filter, add 3 grams of $CaCl_2$and seal the filter with cotton wool on both sides. If the Soxhlet chamber is too large, fill the Soxhlet extraction chamber with marbles so that when the filter containing the dried fish is placed in the Soxhlet extraction chamber, its top is slightly higher than the circulation tube. Fill the heating flask with petroleum ether to approximately two-thirds of its volume. Heat until the petroleum ether boils completely (*a water bath may also be used for heating*). Circulate cooling water through the condenser. Extract the fish meat for approximately 2 hours or for at least 6 circulation cycles. Once finished, attach the flask containing the petroleum ether to *the* rotary *evaporator* and evaporate until all the petroleum ether has been removed. (Evaporation can also be carried out in the open in a fume cupboard, but care must be taken as petroleum ether is highly flammable). Wipe the outside of the flask with a tissue, then weigh the flask containing the fish oil inside. Measure the volume of fish oil obtained, determine the fat content as a percentage, and calculate its specific gravity.

b. Extraction of candlenut oil

Grind a sufficient amount of candlenut seeds with a mortar until fine, then weigh out approximately 5 grams. Carefully transfer the ground candlenuts into a filter, add the preheated sand and cover the filter with cotton on both sides; carry out the extraction as per procedure a

EXPERIMENT VI

ANALYSIS OF TS, TDS AND TSS IN WATER SAMPLES

Objective of the Experiment:

1. To understand the principles of measuring solids using the gravimetric method
2. To measure the concentrations of TS (Total Solids), TDS (Total Dissolved Solids) and TSS (Total Suspended Solids)

Equipment:

1. Petri dish
2. Whatman filter paper
3. Desiccator
4. Oven
5. Beaker
6. Stirring rod
7. Analytical Balance
8. Water Bath
9. Glass Funnel

Materials:

1. Distilled water
2. Water sample

Procedure:

a. Preparation

1. First clean the porcelain beakers and filter paper using distilled water.
2. The clean evaporating dishes and filter paper are heated to 105°C in an oven for one hour.
3. Place the evaporating dishes and filter paper in a desiccator for 30 minutes.
4. Weigh until constant, and record the weight of the porcelain dish and filter paper.

   The weight of the porcelain dish 1 = A gram

   The weight of the porcelain dish 2 = B gram

   The weight of the porcelain dish 3 = C gram

Weight of filter paper = D gram

b. Total Solids (TS) Measurement

1. Add 10 mL of the water sample gradually to a 100 mL porcelain beaker
2. Evaporate on a water bath until dry
3. Once dry, place evaporating dish 1 containing the sample in an oven at 105°C for 1 hour
4. Cool porcelain dish 1 in a desiccator for 30 minutes.
5. Weigh until constant and record the weight = E grams

c. Total Dissolved Solids (TDS) Measurement

1. Filter 10 mL of the water sample using ash-free filter paper, and transfer the filtrate to porcelain dish 2.
2. The filtrate in porcelain dish 2 is evaporated on a water bath until dry
3. Once dry, place porcelain dish 2 containing the filtrate in an oven at 150°C for 1 hour.
4. Cool porcelain dish 2 in a desiccator for 30 minutes.
5. Weigh until constant and record the weight = F grams

d. Measurement of Total Suspended Solids (TSS)

1. The filter paper containing the sediment is placed in evaporating dish 3.
2. Evaporation dish 3 containing the filter paper is placed in an oven at 150°C for 1 hour.
3. Cool evaporating dish 3 containing the filter paper in a desiccator for 30 minutes.
4. Weigh until constant and record the weight = G grams

Calculation basis for TS, TDS and TSS concentrations

- TS = $1000/V \times (E - A) \times 1000$ = …. mg/L
- TDS = $1000/V \times (F - B) \times 1000$ = …. mg/L
- TSS = $1000/V \times \{G - (C + D)\} \times 1000$ = …. mg/L

Notes:

A = weight of evaporating dish 1 (g)

B = weight of evaporating dish 2 (g)

C = mass of evaporating dish 3 (g)

D = weight of filter paper (g)

E = weight of evaporating dish 1 + total residue (g)

F = weight of evaporating dish 2 + soluble residue (g)

G = weight of evaporating dish 3 + filter paper (g)

V = volume of water sample (mL)

EXPERIMENT VII

PURIFICATION OF PHENOL BY DISTILLATION

B. Basic Theory

The purification of a volatile substance can be carried out using the distillation method. In this experiment, phenol is distilled from non-volatile impurities at a constant rate. The rate of phenol evaporation is slow, so the volume of the distillate and the sample being distilled must be equal. The use of CuSO4during the distillation of the acidified sample allows the formation of copper sulphide without subsequent decomposition into H_2S. The acidic solution also prevents the precipitation of copper hydroxide, which acts as an oxidizing agent for phenol.

C. Objective: To purify phenol

D. Apparatus and Materials

Apparatus :
- distillation apparatus, all made of glass, comprising a with a 1-litre capacity and a Graham condenser
- pH meter

Materials : all materials were prepared using phenol- and chlorine-free distilled water as follows:

- Copper sulphate solution

  Solution of 100 g $_{CuSO_4}$.$5H_2O$ in distilled water, diluted to 1 litre
- Phosphoric acid solution, 1+9

  Dilute 10 ml $_{of}$ 85%$_{H_3PO_4}$to 100 ml with distilled water
- Orange metal indicator

  Dissolve 0.5 grams of orange metal in 1 litre of distilled water
- Special reagents for turbid distillates

  1. 1 N sulphuric acid
  2. Sodium chloride
  3. Chloroform or ethyl ether

4. 2.5 N sodium hydroxide

   Dilute 41.7 ml of 6 N NaOH to 100 ml, or dissolve 10 g of NaOH in 100 ml of distilled water

E. Procedure

1. Place 500 ml of the sample in a beaker. Lower the pH to approximately 4.0 using a 1:9$_{H_3PO_4}$ solution. Measure the pH using orange metal indicator or a pH meter. Add 5 ml of $CuSO_4$ solution. Transfer the solution to a distillation flask, using a 500 ml measuring flask as the receiver.
2. If the volume of the distillate has reached 450 ml, stop the distillation. If boiling ceases, add 50 ml of phenol-free distilled water to the distillation flask, then continue the distillation until 500 ml is obtained
3. A single distillation process should purify the sample sufficiently. Occasionally the distillate is turbid; in this case, the distillate must be acidified with H_3PO_4 1:9 and 5 ml of$CuSO_4 \cdot 5H_2O$ solution added, then redistilled as per procedure 2 above. If the second distillate is still turbid, an extraction process as described in procedure 4 below is carried out before distillation.
4. If the second distillate is turbid, the original 500 ml sample is extracted first, as follows:

   The sample is acidified by adding 4 drops of orange metal indicator and sufficient 1 N H_3PO_4. Transfer to a separating funnel and add 150 g of NaCl. Shake with five additions of chloroform. The first addition is 40 ml and the subsequent additions are 25 ml each. Transfer the chloroform layer to a second separating funnel, shake whilst adding 2.5 N NaOH solution three times in succession, the first addition being 4.0 ml and the subsequent additions 3.0 ml each. Combine the alkaline extracts and heat in a water bath until the chloroform has evaporated. Cool and dilute to 500 ml with distilled water, then carry out distillation as described in procedures 1 and 2 above.

EXPERIMENT VIII
THIN-LAYER CHROMATOGRAPHY

I. OBJECTIVE

Students will be able to determine the separation of the polarity properties of extracted compounds based on the Rf value of the spots using the TLC method.

II. THEORY

Chromatography is a method of separating dissolved substances based on the migration of substances in a two-phase or multi-phase system; these substances exhibit differences in mobility due to variations in adsorption capacity, partition coefficient, solubility, vapour pressure, molecular size, or ionic charge density, and each substance can subsequently be identified and quantified using analytical methods.

Thin-layer chromatography is based on the adsorption of substances onto a stationary phase (solid) coated onto a plate (glass, metal). The substances to be separated are applied as spots or bands; the plate is then placed in a tightly sealed vessel containing a developing solution, whereupon the substances migrate due to capillarity, resulting in separation in the form of spots.

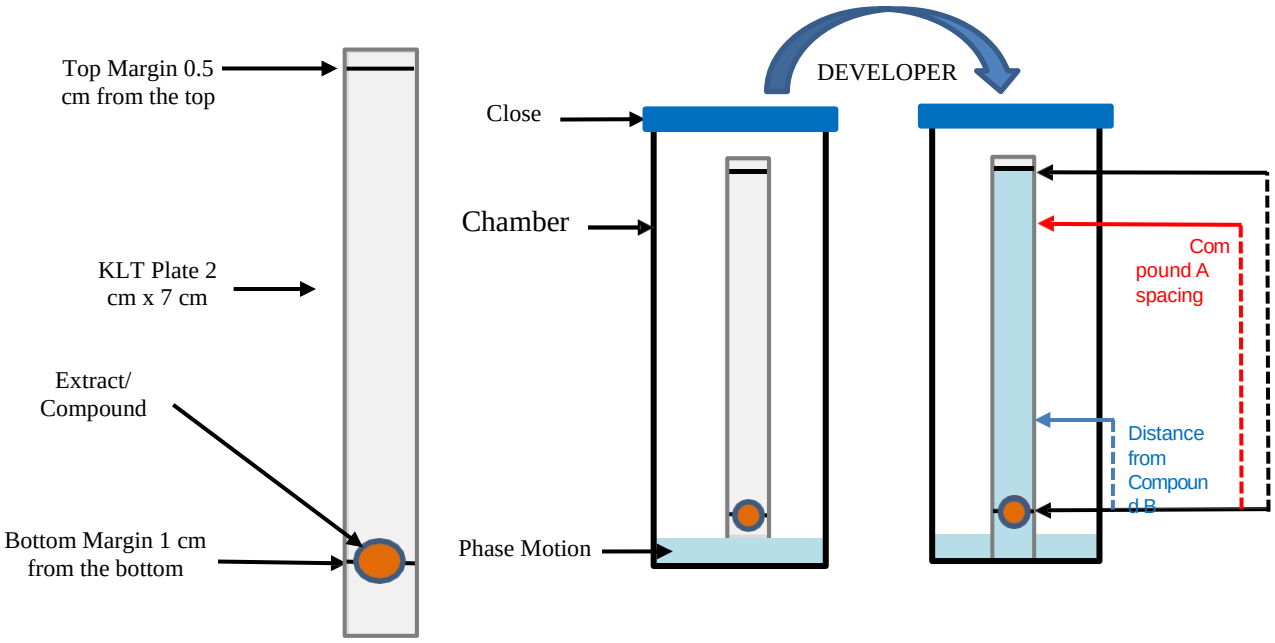

Figure 1. Illustration of Thin-Layer Chromatography (Yodha, 2021)

The stationary phase consists of a fine powder that acts as an adsorbent.

Adsorbent powders commonly used in TLC include: silica gel (silicic acid), alumina (aluminium oxide), kieselguhr (diatom earth) and cellulose. Meanwhile, the mobile phase mobile phase/developing solvent is a single solvent or a mixture of solvents. This developing solvent moves through the porous stationary phase due to capillary forces. The substances to be separated are first dissolved in a small amount of volatile solvent at a concentration of 5–10%. Thin-layer chromatography can be used for the following purposes: 1.) obtaining quantitative results (preparative chromatography); 2.) qualitative identification (Rf of the spot is compared with the Rf of a reference compound; the spot is identified using a specific reagent); and 3.) exploring solvent systems to be used in column chromatography, preparative TLC, or high-performance liquid chromatography.

III. EQUIPMENT AND MATERIALS

Equipment	**Materials**
Developing chamber	Extract from experiment 2
Capillary tube	Ethanol
Evaporation dish	n-hexane
	LC plate Silica gel GF254, 2 x 7 cm (2 plates)

IV. METHOD

- Prepare 10 mL of each developing solution with a solvent ratio of n-hexane : ethanol of 1:3 and 3:1.
- Place the developing solution into the chamber and seal tightly; allow the saturation process to take place for 15 minutes. (To help speed up saturation, filter paper may be placed on one side or around the walls of the chamber until all the filter paper is wet).
- Prepare the sample extracts: a) initial extract; b) extract dissolved in n-hexane; c) extract dissolved in ethanol
- Prepare a 2 x 7 cm TLC plate, carefully marking a 1 cm lower margin and a 0.5 cm upper margin with a pencil (taking care not to scratch the surface).
- Apply ± 5 µl of extracts a, b, and c to the starting line using a capillary tube and allow to dry.
- Carefully place the plate into the chamber and close it quickly

- Leave until the developing front reaches the end line
- Remove the tray and let it dry.
- Observe the spots visually and under UV lamps at 254 nm and 366 nm; if necessary, spray with a chemical reagent
- calculate the Rf value for each spot, and plot the chromatogram for each view (visual, UV light 254, and UV light 366).

V. OBSERVATION DATA

No.	LC Plate	Eluent ratio (n-hexane : ethanol)	Elution distance (cm)	Extract travel distance (cm)			Rf		
				a	b	c	a	b	c
1	Plate 1	1:3							
2	Plate 2	3:1							

VI. PRE-LAB ASSIGNMENT

1. Explain the principle of separation by chromatography!
2. What is meant by Rf in chromatographic analysis and explain the importance of the Rf value!
3. If you are going to perform separation by TLC and there is no literature information available regarding the appropriate eluent for separating the analytes, what steps would you take?

EXPERIMENT IX
DISTRIBUTION CONSTANT (K_D) OF IOD IN LIQUID-LIQUID EXTRACTION

A. Basic Theory

In principle, the value of KD(distribution constant) can be determined at a constant temperature if the concentrations of each substance (analyte) are known in both the aqueous and organic phases.

Iodine (I_2) is soluble in water but is far more soluble in organic solvents, such as chloroform ($CHCl_3$) and carbon tetrachloride (CCl_4). If one of these organic solvents is added to a solution of iodine (in an aqueous solvent), and the mixture is shaken vigorously, the iodine will distribute between the two solvents. The iodine dissolves in the organic solvent, whilst the remainder remains in the water.

Through titration, the residual iodine concentration in the water and the concentration of iodine transferred to the organic solvent can be determined. These results can then be used to calculate the KDvalue for iodine in the organic/aqueous system.

When carrying out the extraction, it is necessary to find the most efficient conditions, whether the extraction is to be performed once or repeatedly. A common piece of equipment used in solvent extraction is a separating funnel. The question that arises is: to what minimum extent should the volume of solvent be used in the extraction, or into how many portions should the solvent be divided for use in repeated separations to achieve optimal conditions? The answer to this problem can be explained as follows:

For example, in a volume of V ml of aqueous (polar) solvent containing W0grams of soluble compound that can be extracted or removed by adding S ml of organic (non-polar) solvent, if W1grams is the weight of the soluble substance remaining in the aqueous phase after the first separation, then the concentration of the soluble substance in the aqueous phase is W1 /V g/ml and in the organic phase is (W0 – W1)/S g/ml, with the distribution coefficient given by:

$$K_D = \frac{W_1/V}{(W_0 - W_1/S} \; atau \; W_1 = \frac{K_d V}{K_d V + S}$$

Furthermore, suppose W1 grams remain in the second layer, then:

$$K_D = \frac{W_1/V}{(W_0 - W_1/S} \; atau \; W_2 = W_1 \frac{K_d V}{K_d V + S}$$

$$= W_0 \left(\frac{K_d V}{K_d V + S} \right)$$

The same applies if Wn grams remain in the water layer after the nth separation, then:

$$W_n = W_n \left(\frac{K_d V}{K_d V + S} \right)$$

Good results will be obtained if the extraction is carried out several times by dividing the extraction solvent into several portions, rather than performing a single extraction using the entire amount of solvent.

B. Objective: To determine the K_D value for iodine in an organic/iodine system

C. Apparatus and Materials

Apparatus: 50 ml or 100 ml separating funnel, 100 ml beaker, 50 ml measuring cylinder, 25 ml measuring pipette, Erlenmeyer flask, 50 ml acid burette, stand and clamp

Materials: solid iodine, 0.01 M $Na_2S_2O_3$, 2 M H_2SO_4, 0.2% starch solution, CCL_4 or $CHCl_3$, starch solution, and distilled water.

D. Procedure

Weigh 0.125 g of solid iodine accurately, then dissolve it in 50 ml of water. Transfer 25 ml of the solution into a 50 ml or 100 ml separatory funnel. Add 5 ml of chloroform or carbon tetrachloride (either one) and shake for a few minutes. Allow to stand for a short while until complete separation of the layers occurs. Drain the organic layer through the bottom of the separatory funnel and pour the aqueous layer into an Erlenmeyer flask through the top of the separatory funnel. Acidify the aqueous solution with 4 ml of 2 M H_2SO_4 solution. Add 1 ml of 0.2% starch solution and titrate immediately with 0.01 M $Na_2S_2O_3$ standard solution until the blue colour of the solution disappears completely. Calculate the grams of iodine remaining in the water. By knowing the original amount of iodine in

grams, the amount of iodine extracted into the organic solvent can be calculated. Calculate K_D iodine.

EXPERIMENT X

Separation of Mixtures by Sublimation

Basic Theory:

Sublimation is a process of purifying a substance by heating a mixture, thereby producing a sublimate (a sublimate is a collection of material in a specific location formed upon heating a substance that can change directly from the solid phase to the gaseous phase and back to the solid phase). Heating organic compounds causes a phase change; for instance, if a substance is in a solid state at room temperature, at a certain temperature it will directly transform into a gaseous phase without first passing through a liquid phase (Syafurjaya, 2011).

The principle of sublimation is that differences in vapour pressure are used to separate or purify solid compounds that can sublimate at room pressure; the sublimation process is very easy to carry out at room pressure without reducing the pressure; simply heating the substance is sufficient for it to sublimate immediately.

Aim of the Experiment: to understand and practise the separation of mixtures using the sublimation method.

Apparatus:

1. Porcelain dish
2. Watch glass
3. Mortar and pestle
4. *Hotplate*
5. Wire Mesh
6. Dropper
7. Spatula
8. Analytical balance

Materials:

1. Solid iodine
2. Activated carbon
3. Ice cubes

Procedure:

1. Weigh 3 grams of solid iodine and activated carbon using an analytical balance
2. Grind the solid iodine using a mortar and pestle until fine
3. Place into a porcelain dish
4. Add the activated carbon impurity
5. Homogenise the iodine and activated carbon
6. The porcelain dish was heated on *a hotplate*
7. Cover with a watch glass
8. Add ice on top of the watch glass
9. Leave the sample in the porcelain dish until it evaporates
10. Switch off *the hotplate*
11. Observe what happens to the porcelain dish; a crust will form on the underside of the watch glass and the sides of the porcelain dish