Salahaddin University – Erbil
College of Science
Chemistry Department

Laboratory Manual

# Basic Organic Chemistry - Practical

## First Year Chemistry Students

Lab Instructors:

Hero Sabah Rahman

Pshtiwan Adel Masum

Dr Karzan Khaleel Hameed

Dr Dotsha Jaleel Raheem

Student Name _____

Class/Group _____

**2023 - 2024**

## Contents



# Before you come to the lab

1. Read about your experiment, so that you have a good understanding of what it entails, steps and techniques involved.
2. Take note of safety precautions associated with the glassware, chemicals and equipment used in the experiment by preparing and filling a Risk Assessment form as outlined below and hand to your laboratory supervisor before starting the experiment:

# Laboratory Safety Rules

1. Always wear a lab coat inside the laboratory.
2. Wear lab specs or safety glasses whenever your lab instructors advice you to.
3. Always wear gloves in the laboratory.



4. Tie loose hair and tuck scarf ends under your lab coat.


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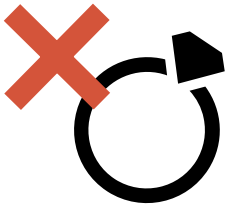
5. Do not wear long, loose dresses. Equally, shorts and short skirts are not allowed as they expose your skin to hazardous chemicals.
6. Do not wear jewelry (rings, bracelets, dangling necklaces and earrings) as they can cause injury when chemicals get trapped under them. They are also difficult to remove in case of chemical spills and accidents.



7. Open-toe shoes, high heels and sandals are not permitted in the lab. Wear shoes that are comfortable and can protect your feet from chemical spills.

8. Do not wear contact lenses (medical or otherwise) in the lab.

9. Drinking and eating is prohibited in the lab.

10. Never taste, smell or inhale chemicals in the lab. If smelling the chemical is required then gently waft the air above the container towards your nose.
11. All containers should be labeled properly. Never use chemicals with missing or damaged label.

# Risk Assessment

Read about the chemicals, glassware and equipment used ***Before*** you start the experiment and make yourself familiar with the risks associated with them.

Fill in this form about hazards involved in the experiment and how to safely handle them.

Experiment name: …….

| | Items | Risk | Safety Measures |
|---|---|---|---|
| **Chemicals** | | | |
| | | | |
| | | | |
| | | | |
| **Glassware** | | | |
| | | | |
| | | | |
| | | | |
| **Equipment** | | | |
| | | | |
| | | | |
| | | | |

References:

Student name: …… Signature: ……

Date of Experiment: ……

# Preparation of Laboratory Reports

Writing a report is an essential part of conducting experiments. It is necessary for the student to write a clear introduction about the experiment, list the chemicals, glassware and equipment used in the experiment. Re-writing the procedure is not required. However, it is necessary to discuss the results obtained and their meaning and significance. Within each experiment in this manual, a number of questions and requirements are put in order to guide students towards the type of information they need to include in each section of the report. Students must write their lab reports independently from their lab partners. Apart from the results section which is often the same for lab partners working on the same experiment, copying is considered plagiarism and similar reports will receive lowered or no points.

General outline of a lab report:

### First page:

| | |
|---|---|
| Student Name | |
| Experiment Number and Title | |
| Class and Group | |
| Date of Experiment | |
| Lab partner(s) Names | |

### Following pages:

Introduction and Background

Results (Signed on the day of experiment)

Chemicals, Glassware and Equipment

Discussion

References

# Experiment (1): Physical Properties of Organic Compounds: Determination of Melting Point (m.p.)

Melting is the change from the highly ordered arrangement of particles (molecules, atoms) in the crystalline structure to the more random arrangement in a liquid state. Melting occurs when enough thermal energy is provided to overcome the intermolecular forces that hold these particles in position. Melting point gives a clue about the strength of intermolecular forces: the higher the m.p. of a compound the stronger its intermolecular forces and vice versa.

Melting point is a physical property used for:

- Identification of unknown compounds
- As an indication of purity

Pure organic compounds often melt within a temperature range of 1 – 2 °C

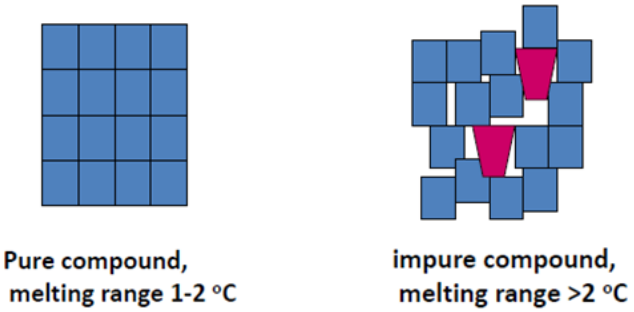



**Figure 1: The crystalline structure of molecules and the effect of impurities on disrupting it**

## Aim of the experiment:

1. Understand the technique used in measuring melting points of organic compounds
2. Finding melting point of an unknown compound and determining its identity using melting point tables.

## Procedure:

1. Seal one end of a capillary tube by rotating it in the edge of a burner flame.
2. Place small amount of the solid material (completely dry and finely powdered) in the capillary tube.

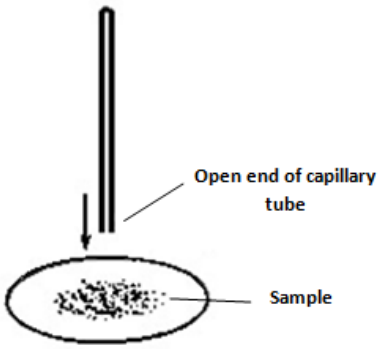



**Figure 2: Inserting a sample into a capillary tube**

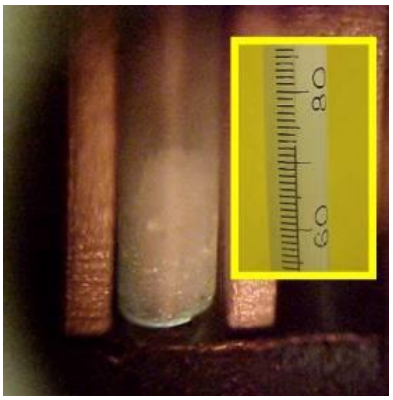
3. Tap the closed end gently on the bench to pack the solid at the bottom (note that the column of solid should be tightly packed and not more than 2-3 mm in height).
4. Place the capillary tube in melting point measuring instrument and start heating it.
5. The increase in temperature should be slow and gradual to get an accurate m.p. reading.
6. Record the temperature at both start and end of melting.

## Note:

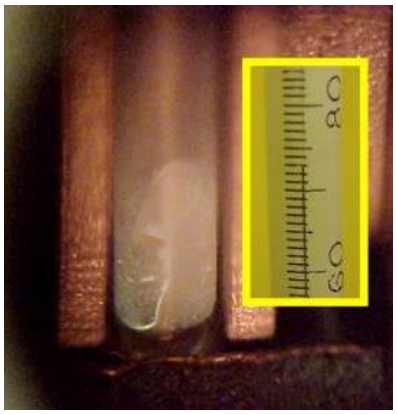
The end of the capillary tube should be aligned with the bulb of the thermometer in order to get accurate reading

**How to record melting point correctly:**

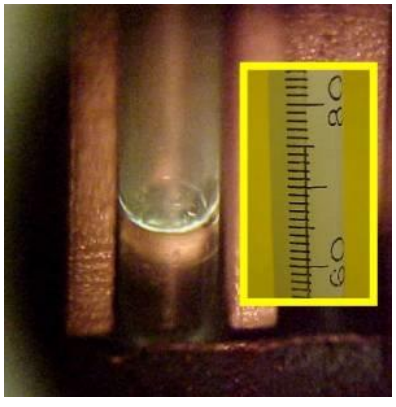
Take this sample as an example to explain the changes you may see during a melting process.




Tiny droplets appear at the start of melting. Here, the sample has started to melt just above 72°.




It becomes easier to see the liquid phase as temperature rises to 73 $^{o}$C.




Finally, at a temperature of 75°C, the entire solid has melted and a clear liquid can be seen.

Melting point of this compound is therefore recorded as a range: 72 - 75 °C.

## Introduction:

Define melting point

What is the importance of measuring melting point?

Why different compounds have different melting points?

Why pure and impure samples of a compound have different melting points?

How melting is different in organic and ionic compounds?

What are the factors that affect melting point?

## Results

Record the start of melting as $T_1$

Record the end of melting as $T_2$

Report the melting point of your compound as a range: $T_1$ - $T_2$ $^oC$ (e.g., m.p. = 62 - 65 $^oC$)

Ask your instructor for the class of your unknown. Using melting point tables of organic compounds, make a list of possible compounds based on the measured melting point.

## Chemicals, Glassware and Equipment

Draw the set up for your experiment then name and annotate all chemicals and glassware used.

## Discussion

Describe what happened to the unknown substance during melting.

What information about the unknown compound do you get from its m.p.?

What points should you consider in order to get accurate melting point results?

## References

What sources did you use in writing this report?

# Experiment (2): Physical Properties of Organic Compounds: Determination of Boiling Point (b.p.)

Boiling point (b.p.) of a liquid represents the energy required to overcome the various intermolecular attractions binding its molecules (e.g., dipole-dipole attraction and hydrogen bonding) and therefore undergo a phase change into the gaseous phase. Therefore, the boiling point of a liquid is also an indicator of the strength of the attractive forces between its molecules.

Boiling point is a physical property used for

1. Identification of a liquid organic compound
2. Separation and purification

## Aim of the experiment:

1. Understand the techniques used in measuring boiling points of organic compounds
3. Finding boiling point of an unknown compound and determining its identity using boiling point tables.

## Procedure:

1. Attach a test tube to a thermometer by means of a rubber band see (Figure 3).
2. Place 2 mL of the unknown liquid in the test tube
3. Immerse the open end of a sealed capillary tube (about 4 cm long) in the liquid
4. Put the thermometer and tube in a water (or oil) bath
5. Start heating with a burner or on a heater until a rapid stream of bubbles starts coming out of the capillary tube. At this point remove the burner. The stream of bubbles becomes slower and the temperature drops until a point is reached when the bubbling stops, and the liquid starts to rise in the capillary tube. Record this temperature as the boiling point of the liquid

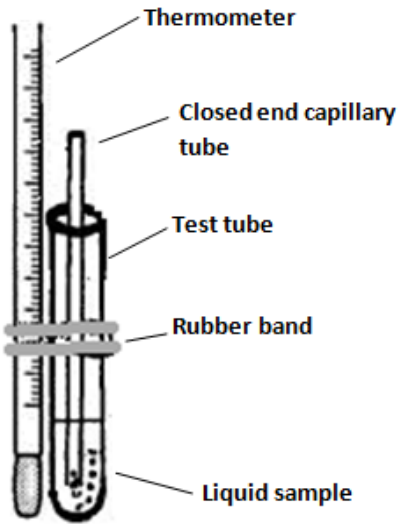



**Figure 3: Sample, capillary tube and thermometer set-up for measuring boiling point**

# Lab report:

## Introduction:

What is meant by boiling? and what happens to an organic compound at boiling point?

What is the importance of measuring boiling point?

Why different compounds have different boiling points

What are the factors that affect boiling point?

## Results

Record the temperature at which the liquid enters the capillary tube as boiling point in $^{o}C$

Ask your instructor for the class of your unknown. Using boiling point tables of organic compounds, make a list of possible compounds based on the measured boiling point.

## Chemicals, Glassware and Equipment

Name and annotate all chemicals, glassware used in the experiment and draw the set up for your experiment

## Discussion

Can you tell the identity of the unknown liquid from its boiling point?

What other information about the unknown compound do you get from its b.p.? for example what can you infer about a compound with low boiling point?

What are the sources of error when measuring boiling point?

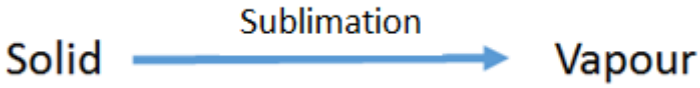
When do you use water bath and oil bath for heating? Is there any difference between the two?

## References

Sources used in writing this report

# Experiment (3): Purification of Organic Compounds by Sublimation

Sublimation is the transition of a substance from the solid phase to the gas phase without passing through an intermediate liquid phase




This phenomenon could be seen in compounds as carbon dioxide (dry ice), iodine and naphthalene. This characteristic can be used to separate compounds that can sublime from non-volatile impurities.

Sublimation is used for the purification of solid organic compounds. At this process the compound is volatilized by heating at a temperature below its melting point. The vapour condenses back to the solid state on a cold receiving surface.

Organic compounds with relatively high vapour pressure at temperatures below their m.p. can undergo sublimation at atmospheric pressure. The technique works Best on non-polar compounds, which are generally more volatile than polar ones of similar molecular weight.

## Aim of the experiment:

Separation of benzoic acid from non-volatile impurity (charcoal) based on their volatility differences

## Procedure:

1. Place (1.0 g) of the impure compound in a crucible.
2. Take a filter paper that is large enough to cover the crucible. Use a pin (or a pencil tip) to make holes in the paper.
3. Place a funnel upside down on the crucible and paper as shown in Figure 4. Record $Wt_1$ (as explained in Results section)
4. Start heating the sample gently with a Bunsen burner.
5. Weigh the pure compound $Wt_2$ (as explained in Results section) and calculate its ratio in the mixture

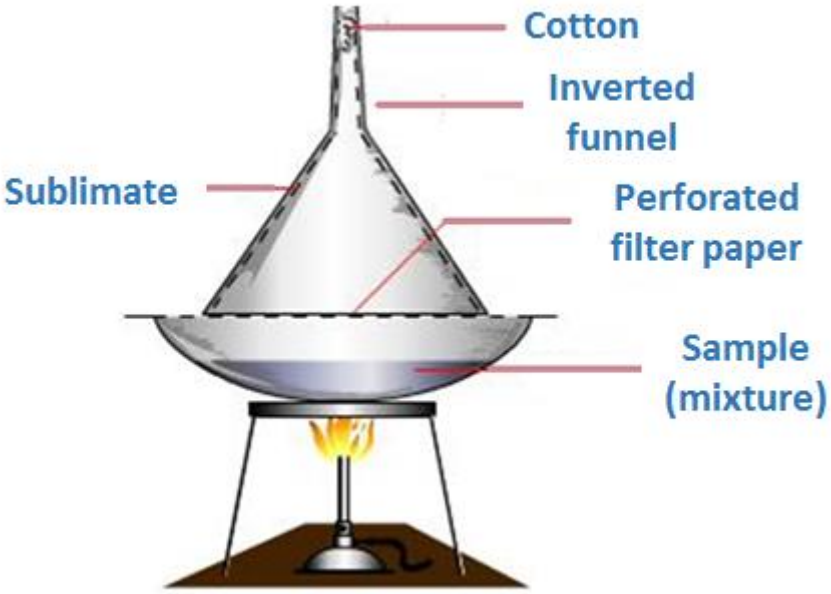



**Figure 4: Experimental set up for sublimation**

## Preparation of Lab Report:

### Introduction and Background

What is meant by sublimation and what is its principle?

Give examples on compounds undergoing sublimation

### Results

$Wt_1$: weight of filter paper + glass funnel + cotton

$Wt_2$: weight of filter paper + glass funnel + cotton + pure benzoic acid

$Wt_2$ - $Wt_1$ = weight of pure benzoic acid

Calculate % of benzoic acid in the mixture as follows:

$$\% \text{ Benzoic acid in the mixture} = \frac{Weight\ of\ pure\ benzoic\ acid\ (g)}{Weight\ of\ mixture\ (g)} \times 100$$

## Chemicals, Glassware and Equipment

Name and annotate all chemicals, glassware used in the experiment and draw the set up for your experiment

## Discussion

What does your experimental result tell you?

What are the advantages and disadvantages of sublimation?

Are there other methods (apart from sublimation) that could be used in purification of organic compounds?

What is the name of the process where a compound is converted from vapour to solid without going through a liquid state (note: this process is opposite to sublimation)?

## References

What sources did you use in writing this report?

# Experiment (4): Purification of Organic Compounds by Recrystallisation

Recrystallisation is a technique used to purify solids. The process relies on the fact that solubility increases with increasing temperature. As a hot saturated solution cools, it becomes supersaturated and the solute precipitates (crystallises) out. In a recrystallisation procedure, an impure (crude) solid is dissolved in a hot solvent. As this solution is cooled, the pure product crystallises out and the impurities stay dissolved.

**Effect of temperature on solubility of benzoic acid**

Solubility of benzoic acid in water increases with increasing temperature. This characteristic can be used to clean benzoic acid from impurities whose solubility do not change with temperature.

**Table 1: Increase in solubility of benzoic acid in water with increasing temperature**

| Temperature $^{o}C$ | Solubility<br>g $_{\text{Benzoic Acid}}$ /100mL $_{\text{water}}$ |
|---|---|
| 0 | 0.17 |
| 18 | 0.27 |
| 25 | 0.34 |
| 40 | 0.55 |
| 75 | 2.15 |
| 100 | 5.63 |

**Main steps in a recrystallization experiment:**

- Choose a suitable solvent.
- Dissolve the impure solid in the minimum volume of the hot solvent.
- Remove insoluble impurities by hot filtration.
- Slowly cool the hot solution to recrystallise the desired compound from the solution.
- Filter the solution to isolate the purified solid compound.
- Dry the purified crystals.

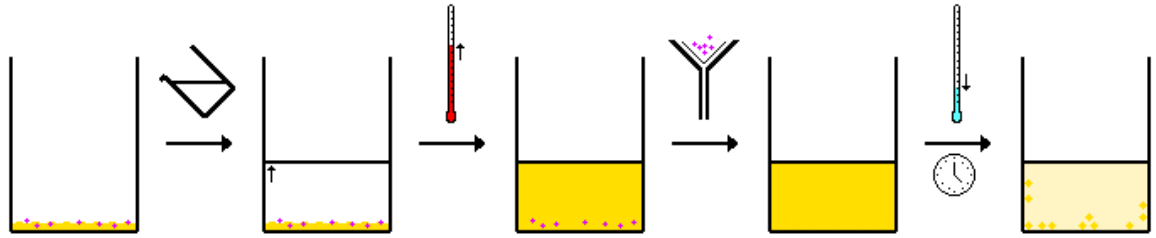


Figure 5: The main steps for purification by recrystallisation

## Aim of the experiment:

Purification of benzoic acid from charcoal is taken as an example on separation of organic compounds from impurities based on solubility differences at different temperatures.

## Procedure:

1. Weigh (0.5 g) of the impure sample and place it in a conical flask.
2. Place 20 mL of the solvent in a beaker and heat it to boiling.
3. Add the hot solvent to the impure mixture. Start by adding 1 mL portions and note the solubility and stop the addition once the desired compound is dissolved.
4. Heat briefly to ensure that the desired compound is dissolved completely (at or near the b.p. of the solvent).
5. Filter the hot solution and let the filtrate cool down slowly to allow crystallisation of the pure compound.
6. Separate the pure crystalline solid by filtration (weight of filter paper is required in this step).
7. Let the crystals dry in an oven at 60 – 70 $^{o}$C or at room temperature (ask your instructor)
8. Weigh the dried crystals and calculate the percentage of the pure compound.

## Preparation of Lab report:

Make use of the following in building your report:

### Introduction

What is meant by recrystallisation?

What is the purpose of recrystallisation?

What are the desired characteristics of a recrystallisation solvent?

What is the effect of temperature on solubility?

### Results

$Wt_1$: weight of dry filter paper

$Wt_2$: weight of Filter paper + pure benzoic acid

Find weight of pure benzoic acid = $Wt_2$ - $Wt_1$

Find the % of pure benzoic acid in the original mixture as follows:

$$\%\ \text{Benzoic acid in the mixture} = \frac{Weight\ of\ pure\ benzoic\ acid\ (g)}{Weight\ of\ mixture\ (g)} \times 100$$

### Chemicals, Glassware and Equipment

Draw the set up for your experiment, then name and annotate all chemicals, glassware that you used in the experiment.

### Discussion

What was the percentage of pure benzoic acid in the experiment? Do you think you could get higher % or better crystalline solid by doing somethings differently?

What factors affected your result and what are the main sources of error in the experiment?

Why is it necessary to keep water volume to minimum during the experiment?

What techniques can be followed to induce crystallisation of solute from a solution?

What is the difference between “crystallization” and “recrystallisation”?

**References** What sources did you use in writing this report?

# Experiment (5): Simple Distillation

Simple distillation is a method for purification of liquids from soluble solids or mixture of compounds with boiling point differences of at least 40-50°C. In this method the liquid to be purified is placed in a distillation flask and heated. The liquid is then converted to vapor (gas), this vapor turns back into liquid when reaching the condenser's cool surface. A receiver flask is placed at the end of the condenser to collect the pure liquid.

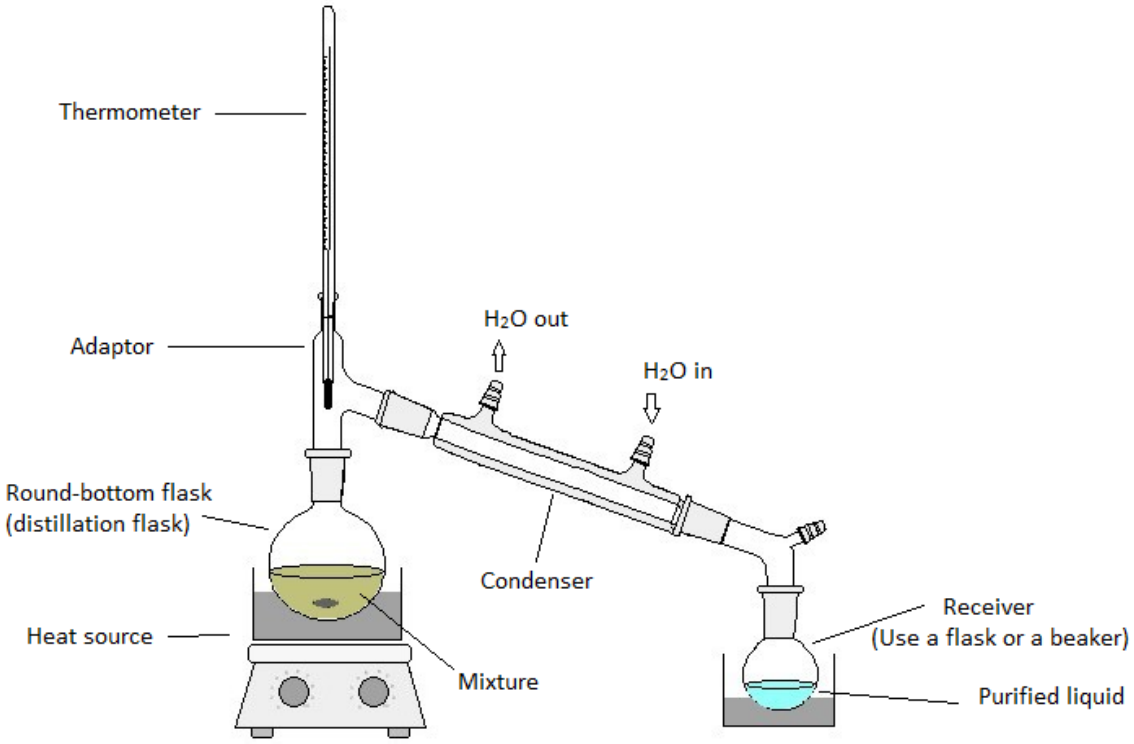



**Figure 6: Experimental set-up for simple distillation**

## Aim of the experiment:

1. Separation of pure water from a water-salt solution using simple distillation.
2. Familiarise the student with using condensers in organic chemistry laboratory as well as safe and correct set up of experiments that involve simultaneous heating and cooling.

## Procedure:

1. Take a clean dry round-bottom flask and add few boiling chips to it. Measure their weight together using an electrical balance. Record this as **$Wt_1$**
2. Measure 20 mL of the water-salt solution and transfer it to the round-bottom flask.
3. Place an adaptor and a thermometer on the flask and connect it to a condenser as shown in Figure 6 (ask your instructo for help). Note that the system should not be completely closed (why?).
4. Connect the condenser to a water tap and let water flow throw the condenser (note the correct position of **in** and **out** water lines)
5. Start heating the mixture
6. When the liquid reaches a temperature close to its boiling point, vapor starts to condense and collect at the receiver. The thermometer reading remains constant at this stage (**record this as distillation temperature for the liquid**)
7. Turn off the heater when there is only 1-2 mL of liquid remaining in the distillation flask (why?).
8. Let the flask dry completely either by leaving it on the heater (turned off or on low temperature) or placing it in an oven.
9. Weigh the flask again (after it has cooled down) to find out the amount of remaining solid. Record this as **$Wt_2$**

## Preparation of Lab report

### Introduction and Background

What is the principle of simple distillation?

What other types of distillation are there? What are they used for and how does their experimental set-up differ from simple distillation?

Distillation is a separation method? Can you give examples on other types of separation methods?

### Results

Record the temperature at which distillation occurred and annotate it as distillation temperature (is this temperature a range or a single distinct value?)

Calculate the weight of the salt remaining in the flask as follows:

$Wt_1$: weight of flask + boiling chips

$Wt_2$: weight of flask + boiling chips + salt

Weight of salt = $Wt_2 - Wt_1$

Calculate the % of NaCl (weight/volume) in the mixture as follows:

$$\%\ of\ NaCl \quad = \frac{Weight\ of\ NaCl\ (g)}{Volume\ of\ original\ mixture\ (mL)} \times 100$$

Measure the volume of the pure liquid collected at the end of the experiment

Calculate the % of the pure liquid (volume/volume) in the mixture as follows:

$$\%\ of\ pure\ liquid \quad = \frac{Volume\ of\ pure\ liquid\ (mL)}{Volume\ of\ original\ mixture\ (mL)} \times 100$$

## Chemicals, Glassware and Equipment

Name and annotate all chemicals, glassware used in the experiment and draw the set up for your experiment

## Discussion

What was the % of solid and liquid in the mixture? Was the separation process successful, was there any loss of material at any stage of the experiment? could the method be improved in any way?

Is there a specific correct position for the thermometer on top of the flask?

Was the distillation temperature the same as the boiling point of the liquid in the experiment?

What is the benefit of using boiling chips in the experiment?

Will there be any problem if water entrance and exit were reversed in the condenser?

How can you separate a mixture of two liquids with less than 20 $^{o}$C difference in boiling point? Will the experimental set-up be any different? Draw the set.

## References

Sources used in writing this report

# Experiment (6): Extraction of Caffeine from Tea Leaves

Caffeine belongs to a class of naturally occurring compounds known as alkaloids. It is a compound with mental stimulating effects that can be found in tea and coffee. It is also added to other beverages such as cola and energy drinks. It is estimated that caffeine makes up about 3% of the tea leave's dry weight.

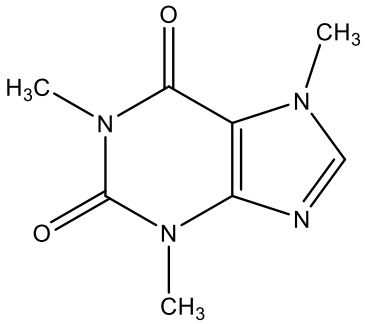



**Figure 7: The structure of caffeine**

In order to separate caffeine from tea leaves, an extraction procedure is followed using aqueous and organic solvents. Extraction of a compound means its isolation from a matrix. The most common type of technique followed in such cases is solid-liquid and liquid-liquid extraction. For solid-liquid extraction tea leaves are heated in water then a filtration or decantation step is used to separate the solid from liquid extract. For liquid-liquid extraction an organic solvent ($CHCl_3$ or $CH_2Cl_2$) is used to extract caffeine from water then the two liquids are separated from one another using a separating funnel (Figure 8 and Figure 9).

**The effect of tannins on the extraction process:**

Tannins are acidic compounds found in a variety of plant-based food and drinks including tea. They are responsible for the bitter taste and brown-red colour of tea. They are also responsible for a number of health benefits as well as some undesired effects associated with drinking tea.

Some tannins might be soluble in organic solvents such as $CHCl_3$ and $CH_2Cl_2$ used in the experiment and therefore can interfere with the extraction of caffeine. To overcome this problem, sodium carbonate ($Na_2CO_3$) is added. This has the effect of reacting with the acidic tannins and other acidic compounds in the extract and changing them to an

ionic form that is water soluble and remain in the aqueous phase during the extraction. This results in higher purity of the extracted caffeine.

**Figure 8: A separating funnel used to separate immiscible liquids with different densities**

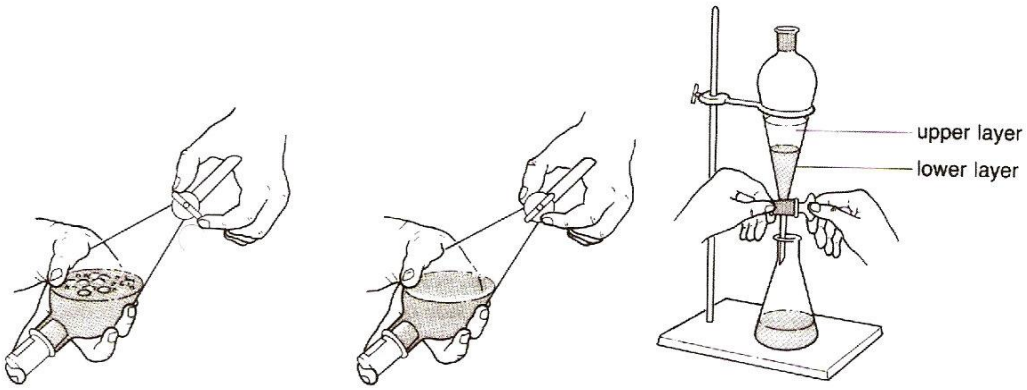



**Figure 9: The correct way to use a separating funnel**

## Procedure:

### Solid – liquid extraction stage

1. Weigh 4g of tea (or take 2 tea bags) into a 250 mL beaker and add 0.5 g of $Na_2CO_3$ and 40 mL of water.
2. Heat the mixture to boiling then steep for 3-5 minutes
3. Filter the mixture and discard tea leaves. The remaining solution should be around 5 mL (if it is more than 5 mL, heat it again to evaporate excess water)

**Liquid – liquid extraction stage**

4. Let the solution cool down to room temperature.
5. Transfer into a separating funnel then add 5 mL of $CHCl_3$. Remove the organic layer into a clean dry beaker.
6. Repeat the previous step one more times and combine the collected organic fractions (also called chloroform extract)

**Drying of the product**

7. Transfer the chloroform extract to a clean dry crucible (previously weighed) and place it in the fume hood for the solvent to evaporate.
8. Measure the weight of the collected caffeine (by subtracting the crucible weight)

## Results and calculations

Calculate the theoretical weight of caffeine in your sample knowing that tea leaves contain 3% caffeine as follows:

| | Weight of caffeine (g) | Weight of tea (g) |
|---|---|---|
| Expected value in 1oo g | 3 | 100 |
| Expected value in 4 g | X | 4 |

$$\% \text{ Theoritical weight} = \frac{4\ (g)\ \times 3\ (g)}{100\ (g)}$$

This will theoretically give the caffeine weight contained in 4 g tea sample

Record the actual weight of caffeine obtained in your experiment

Calculate % Recovery as follows:

$$\% \text{ Recovery} = \frac{Actual\ weight}{Theoritical\ weight} \times 100$$

## Discussion

How much caffeine could you extract in the experiment?

How does your result compare to the expected theoretical value?

The obtained caffeine may be impure how do you purify it? what are possible impurities?

How can you prove the identity of the obtained crystals to be caffeine?

What are the main extraction techniques used in this experiment?

What makes caffeine transfer into the $CHCl_3$ layer from the aqueous layer?

What is the reason for adding $Na_2CO_3$ in the experiment?