

SECTION

9

**QUALITATIVE AND
QUANTITATIVE
ANALYSIS OF ORGANIC
COMPOUNDS**



CHEMISTRY OF CARBON COMPOUNDS

Characterisation of Organic Compounds

INTRODUCTION

This section covers the **qualitative and quantitative analysis** of organic compounds. An organic compound is a compound containing carbon atoms covalently bonded to other atoms. The goal is to help you understand the processes involved in these analyses. By the end of this section, you should be able to describe both types of analysis, including:

- outlining the steps in these analyses.
- designing and conducting experiments to detect and measure carbon, hydrogen, sulphur, nitrogen, and halogens in organic compounds.

At the end of this section, you will be able to:

- Describe the qualitative and quantitative analysis of organic compounds
- Design an experiment to test for the presence and mass composition of carbon, hydrogen, sulphur, nitrogen and halogens in organic compounds

Key Ideas

- **Quantitative analysis** is a methodology used to determine the exact mass composition of elements in an organic compound, often expressed in percentages.
- **Elemental analysis** is the process of identifying which elements are present in an organic compound, typically carbon (C), hydrogen (H), oxygen (O), nitrogen (N), sulphur (S), and halogens.
- **Lassaigne's test** is a qualitative method for detecting the presence of **nitrogen, sulphur, and halogens** in organic compounds.
- **Functional group** is a specific group of atoms within a molecule that is responsible for its characteristic reactions.
- **Flame test** is a method used to detect the presence of certain metal ions based on the colour of the flame produced.

- **Chromatography** is a technique for separating mixtures into their individual components based on their relative solubility, often used to analyse and identify organic compounds.
- **Distillation** is a separation technique that uses boiling and condensation to remove and isolate a liquid part of a mixture, based upon its boiling point.
- **Condensation** is the process by which a substance in the gas state is cooled and changes to a liquid state.
- **Distillate** is the liquid that is condensed and collected from the gas state during distillation.
- An **organic compound** is a compound containing carbon atoms covalently bonded to other atoms.

METHODS OF SEPARATION AND PURIFICATION OF ORGANIC COMPOUNDS

Most substances are found as **mixtures**. These mixtures can be in different forms, depending on the **physical** and **chemical properties** of the parts that make them up.

We can separate one part of a mixture if it has a property that is very different from the others. The bigger the difference in the property, the easier it is to separate. Some properties that can help us to separate the components of mixtures are **boiling point**, **melting point**, **solubility**, **surface adsorption**, and **chemical reactivity**.

Here are some ways to **separate** and **purify** organic compounds based on these properties.

Distillation

Distillation is used to separate a volatile liquid (one that more easily turns into gas) from a mixture of volatile liquids and non-volatile liquids or solids. To use this method, the parts of the mixture must have big differences in their boiling points.

This separation method includes two steps:

1. Upon the application of heat, the more volatile liquid boils and turns into gas (liquid $\rightarrow$ gas).
2. The gas goes through a condenser, where it cools down and changes back to liquid, which is collected in a flask (gas $\rightarrow$ liquid).
3. Any dissolved solid stays in the original flask because its boiling point is too high.
4. The distillation setup has a distillation flask, a condenser, and a receiving flask. The mixture is heated using a hot plate or a Bunsen burner to start the process.

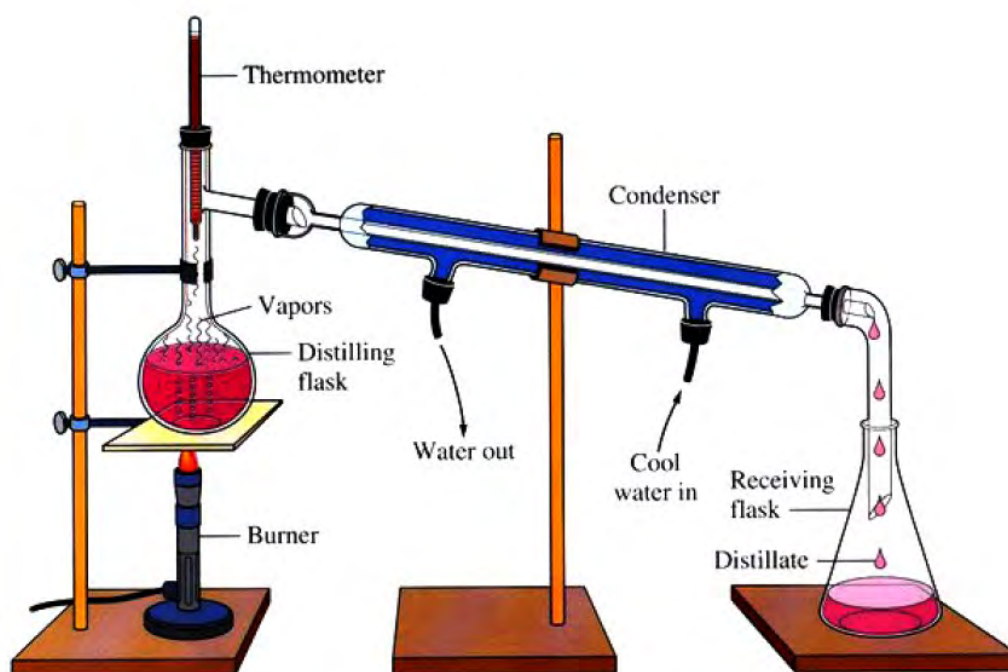


Fig .9.1: Distillation apparatus

Practical Application

- a. It is used to recover or purify seawater.
- b. It is used in the separation of ethanol from a mixture of ethanol and water (care must be taken not to use open flames as the source of heat).
- c. Purification of essential oils from plants.
- d. Production of distilled water for laboratory use.

Fractional Distillation

Fractional distillation is used to separate two or more miscible liquids (liquids that mix well) that have similar boiling points. This method works best when the boiling points are close to each other.

The process involves two main steps:

1. The mixture is heated, and all of the liquids turn into gas (liquid $\rightarrow$ gas).
2. The vapour mixture passes through a fractionating column, where it cools as it rises.
3. Each component of the mixture will then condense (gas $\rightarrow$ liquid) when it cools sufficiently to reach its boiling point.
4. Each liquid fraction is then removed at different heights up the fractionating column allowing for complete separation and isolation of each component of the mixture.
5. In the laboratory this removal step has to be done one component at a time (from the most volatile to the least), and each component is “tapped off” as the temperature at the top of the column rises.

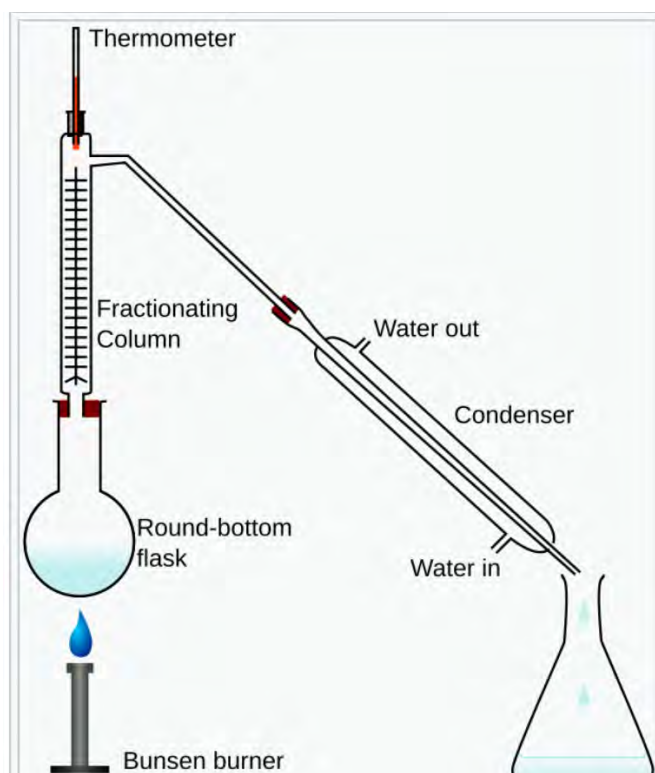


Fig. 9.2: Fractional distillation apparatus

Practical Application

- a. Distillation of methanol-ethanol mixtures.
- b. Distillation of crude oil into useful fractions.
- c. Isolating oxygen gas (or other components) from liquid air.

Crystallisation

Crystallisation is a purification technique where a solid is encouraged to come out of a solution. It is used to remove impurities from a solid compound.

Here's how it works:

1. The solid is first dissolved in a solvent, making sure the impurities also dissolve. The solution is then heated until the liquid begins to evaporate, making the solution more concentrated. This is known as a saturated (or in some cases supersaturated) solution.
2. As the solution cools, the solid becomes too concentrated to stay dissolved, so crystals of the pure substance form.
3. When the solution cools down further, more pure crystals form as the impurities stay in the solution because they are at a lower concentration.
4. The crystals are then filtered and dried using a desiccator.

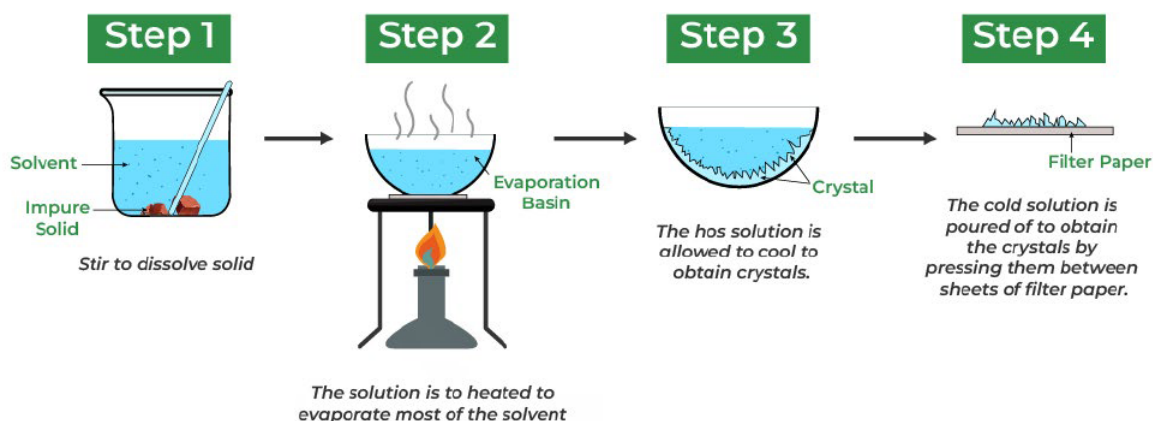


Fig. 9.3: Crystallisation apparatus

Practical Application

Substances that can be isolated by crystallisation include:

- a. Benzoic acid
- b. Glucose
- c. Paracetamol

Paper Chromatography

Paper chromatography is a method used to separate and identify different substances in a mixture. It uses two parts:

- A stationary phase (a strip of absorbent paper)
- A mobile phase (a liquid solvent that moves through the paper).

Here's how it works:

1. The substances in the mixture move at different speeds over the paper because they have different attractions to the mobile phase (the solvent) and the stationary phase (water trapped in the paper).
2. The highest point the solvent reaches on the paper is called the solvent front.
3. The final pattern of separated substances is called a chromatogram.
4. The R_f value is used to measure how far each substance moves compared to the solvent front.

$$R_f = \frac{\text{Distance moved by dissolved substance spot}}{\text{Distance moved by solvent front}}$$

The conditions of temperature and solvent used must be quoted in reference to the R_f data table to identify the sample, as these can affect the end result.

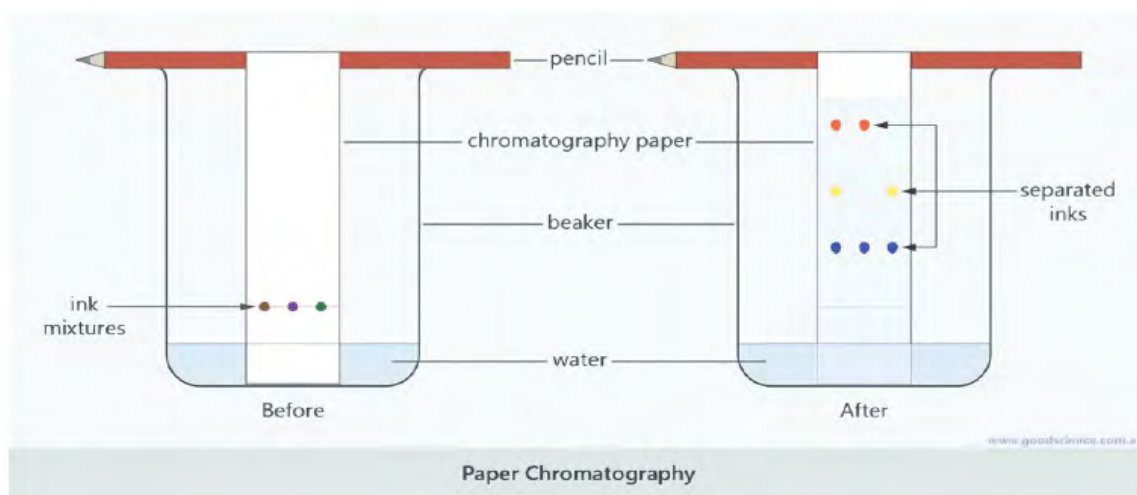


Fig 9.4: Paper chromatography

Practical Application

- a. In food dye analysis
- b. In forensic analysis
- c. To analyse and identify natural products

Activity 9.1: Live demonstration to show distillation, fractional distillation and crystallisation.

- Using the internet, click on the links below to watch videos on distillation, fractional distillation and crystallisation:

<https://www.youtube.com/watch?v=WMeVF9sLDZQ>

https://www.youtube.com/watch?v=v-wQZL_mwlo

<https://www.youtube.com/watch?v=-duFHvkbAOI>



- After watching the videos linked above, answer the following questions:
 - How can you tell when a distillation process is complete?
 - Why is simple distillation not suitable for separating two liquids with similar boiling points?
 - Why is a fractionating column important in fractional distillation?
 - How does the fractionating column help in the separation of liquids?
 - What kind of mixtures can be separated using fractional distillation?

Activity 9.2: Determining the Melting Points of Organic Solids (Benzoic Acid, Oxalic Acid, Ethanamide)

Materials needed: Samples of benzoic acid, oxalic acid, and ethanamide, capillary tubes, an oil bath with a thermometer, mortar and pestle spatula, thermometer, safety goggles and gloves.

Safety Precautions:

- Undertake this experiment under teacher supervision
- Wear goggles and gloves throughout the experiment.
- Avoid splashing the oil as this could cause burns.
- Handle heated equipment carefully to avoid burns.

Under the guidance of your teacher or technician, the below steps should be followed:

1. Finely crush the organic solids using a mortar and pestle to ensure uniform heating.
2. Take a **capillary tube** and push one end of it into the powdered solid to pick up a small amount of the compound.
3. Invert the tube so the solid is near the closed end. Tap the tube gently on a hard surface to compact the sample near the closed end.
4. Place the capillary tube with the sample into the melting point apparatus or in an oil bath setup. If using an oil bath, ensure the thermometer is positioned so it is at the same level as the sample.
5. Slowly increase the temperature of the apparatus or oil bath. Ensure adequate safety precautions are taken near a hot liquid, especially oil.
6. Watch carefully for the first signs of melting (when the sample starts to liquefy).
7. Document the temperature at which the entire sample has fully melted as the melting point.

Note:

- Begin monitoring the thermometer closely as the sample approaches its expected melting point (e.g., benzoic acid $\sim 122^{\circ}\text{C}$, oxalic acid $\sim 189^{\circ}\text{C}$, ethanamide $\sim 82^{\circ}\text{C}$).
- A pure compound will have a sharp melting range close to the literature value, while impure compounds will exhibit a broader range and a lower melting point.

Using your insights from the experiment, please respond to the following questions:

- a. Why is it important to heat the sample slowly when determining its melting point?
- b. Why is it important to use a clean and dry capillary tube when determining the melting point?
- c. How is melting point determination used in the pharmaceutical industry?

Activity 9.3: Paper Chromatography as an Analytical Technique

Materials needed: Filter paper or chromatography paper, pencil (for marking the paper), ruler, solvent (water, ethanol, or another suitable solvent), mixture to analyse (e.g., ink from different pens, plant pigments, food dyes), beakers or jars, capillary tubes or toothpicks, a pair of scissors, safety goggles and gloves. Under the guidance of your teacher or technician, the below steps should be followed:

1. Cut the chromatography or filter paper to a size that fits into your beaker or jar.
2. Draw a pencil line near the bottom (about 1-2 cm from the edge of the paper). This is your baseline, where the mixture will be applied.
3. Using a capillary tube or toothpick, apply a small dot of the mixture (e.g., ink, dye) to the baseline on the paper. Allow the spot to dry and repeat for a more concentrated spot if necessary.
4. Pour a small amount of solvent (e.g., water or ethanol) into the beaker. Ensure that the solvent level is below the baseline of the paper.
5. Carefully place the paper into the beaker, ensuring that the spot is above the solvent. Allow the solvent to travel up the paper by capillary action, carrying the components of the mixture with it.
6. Allow the solvent to move up the paper for several minutes. As it moves, the different components of the mixture will separate due to differences in their solubility and interaction with the paper and solvent.
7. Once the solvent has nearly reached the top, remove the paper and mark the solvent front with a pencil.
8. Let the paper dry completely.
9. Observe the separation of the mixture into distinct spots or bands on the paper. Measure the distance each component travelled relative to the solvent front.
10. Calculate the **R_f value** (Retention factor) for each component using the formula:

$$R_f = \frac{\text{Distance moved by dissolved substance spot}}{\text{Distance moved by solvent front}}$$

Activity 9.4: Fill in the Blanks

1. Use the words in *italics* below to complete the summary about distillation.
(*mixing, conical flask, cooled, liquid, delivery tube, gas, separation, melts, heated, condenses*)

Distillation is atechnique. A mixture is until one substance in it boils, changing to the state. This gas substance is less dense than the mixture, so rises into a, where it is transferred to another container. As the gaseous substance travels away from the heat source, it cools and back into the liquid state.

2. Define distillation and explain its purpose in the separation of mixtures.
3. Sketch the set-up of a simple distillation apparatus and explain the role of each component in the distillation process.
4. How will you recover and recycle a solvent containing dissolved solid impurities for an industrial process?
5. Consider the boiling points of the following alkanols

Alkanol	Boiling point (°C)
Methanol	65
Ethanol	79
1-propanol	97
1-butanol	117

- a. Suggest the appropriate method of separating the alkanols
 - b. Which alkanol will be collected first? Explain your answer.
6. Explain crystallisation and outline its importance
 7. What are the steps involved in crystallisation?
 8.
 - a. What is paper chromatography?
 - b. How can you identify substances in a mixture using paper chromatography?

9. Explain R_f values and their significance in paper chromatography. How can you calculate the R_f value of a particular component?
10. The green pigment chlorophyll in the leaves of plants can be obtained using the following procedures:
 Step 1: The leaf is ground with alcohol until the solution is saturated
 Step 2: The green pigment obtained is separated from the mixture
 Step 3: The colours in the pigment are then separated.
 - a. Name the apparatus used in **step 1**.
 - b. Suggest why the leaves are ground with alcohol instead of water in **step 1**
 - c. Name the type of separation method used in **step 2**.
 - d. Describe how **step 3** is carried out
11. A label on a can of green drink contains no artificial colours. Plan an investigation to show that the green colour of the drink was not a mixture of these two artificial colours. Blue (E125) Yellow (E110).

You are provided with the green colour from the drink, samples of E125 and E110 and some common laboratory apparatus.

TESTS FOR CARBON, HYDROGEN, SULPHUR, NITROGEN AND HALOGENS IN ORGANIC COMPOUNDS

The below five tests outline the procedures for testing for the presence of carbon, hydrogen, sulphur, nitrogen and halogens in organic compounds. Once you have an understanding of these procedures, you will conduct these tests, under supervision from your teacher or technician, to confirm your learning.

Testing for Carbon and Hydrogen in an Organic Compound

1. Organic sample is heated in the presence of the oxidising agent; copper (II) oxide.
2. Carbon present is converted into carbon dioxide.
3. Hydrogen present is converted into water.

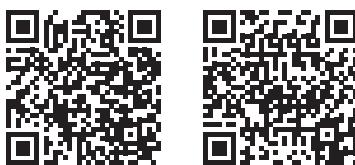
4. The presence of carbon dioxide is detected by passing it through clear lime water, which turns milky.
5. The presence of water is detected by passing it through white anhydrous copper (II) sulphate which turns blue.

Testing for Nitrogen, Sulphur and Halogens in Organic Compounds

Use the Lassaigne sodium fusion test to convert N, S and halogens into inorganic ions.

Using the internet, search for information about the Lassaigne sodium fusion test and also watch the videos on it by clicking on the links below

- <https://www.vedantu.com/jee-main/chemistry-lassaigne-test>
- <https://www.youtube.com/watch?v=jERxoPjQEiA>



Testing for sulphur

Add a few drops of freshly prepared sodium nitroprusside to the filtrate from the sodium fusion test. A purple or violet colour formation indicates the presence of sulphur in organic compounds.

Testing for nitrogen

1. Add a few drops of dilute NaOH to the filtrate of the sodium fusion test.
2. Heat and cool solution.
3. Add iron sulphate solution followed by drops of iron (III) chloride.
4. The formation of Prussian blue precipitate indicates the presence of nitrogen.

Testing for halogens

1. Add excess concentrated nitric acid to the filtrate from the sodium fusion test and heat.
2. Cool the solution and aqueous AgNO_3 solution
3. Formation of a white precipitate shows chlorine is present.

4. Formation of cream precipitate shows bromine is present.
5. Formation of yellow precipitate shows iodine is present.

Activity 9.5: Experiments to Test for Carbon, Hydrogen, Sulphur, Nitrogen, and Halogens in Organic Compounds

Materials needed: Organic sample (e.g., urea, benzene, etc.), sodium metal (for Lassaigne's test), silver nitrate solution, lead acetate solution, copper sulphate solution, concentrated nitric acid, ferrous sulphate solution, sodium hydroxide solution, calcium hydroxide (lime water), Bunsen burner, test tubes, spatula, filter paper, distilled water, Ice bath, gloves and safety goggles

Under the supervision of your teacher or technician, you will carry out the below experiments. You should follow all relevant safety guidance and wear appropriate protective clothing/items.

Procedure:

Test for Carbon and Hydrogen

1. Place a small amount of the organic compound in a dry test tube.
2. Heat the sample strongly in the presence of oxygen.
3. Pass the gases produced through limewater (Ca(OH)_2) to test for carbon dioxide.
 - A white precipitate indicates the presence of **carbon**.
4. Collect water vapour formed on the cooler part of the test tube.
 - Condensation of water indicates the presence of **hydrogen**.

Observation:

- a. White precipitate in limewater confirms carbon.
 - b. Water droplets confirm hydrogen.
5. Make a note of your findings.

Test for Nitrogen (Lassaigne's Test)

1. Heat the organic compound with sodium metal in a fusion tube (this forms sodium cyanide).
2. Cool the mixture and extract it with water to form a filtrate.

3. Add ferrous sulphate (FeSO_4) to the filtrate, followed by concentrated sulfuric acid (H_2SO_4).
4. A Prussian blue colour indicates the presence of nitrogen.
5. Make a note of your findings.

Test for Sulphur (Lassaigne's Test)

1. Perform Lassaigne's fusion as in the nitrogen test.
2. Filter the solution and add lead acetate ($\text{Pb}(\text{CH}_3\text{COO})_2$).
3. A black precipitate of lead sulphide (PbS) confirms the presence of sulphur.

Make a note of your findings.

Test for Halogens (Lassaigne's Test)

1. Perform Lassaigne's fusion to extract halogens.
2. Acidify the extract with dilute nitric acid to remove any sulphides or cyanides.
3. Add silver nitrate (AgNO_3).
 - A **white precipitate** (soluble in ammonia solution) confirms **chloride**.
 - A **pale-yellow precipitate** (insoluble in ammonia solution) confirms **bromide**.
 - A **yellow precipitate** (insoluble in concentrated ammonia solution) confirms **iodide**.

Make a note of your findings.

Self-Assessment

1. Identify the colour change when testing for chlorine in an organic compound by adding excess concentrated nitric acid to the filtrate from a sodium fusion test after heating and then AgNO_3 is added.
2. List the steps involved in the determination of nitrogen in an organic compound.
3. How can N, S and halogens be converted into inorganic ions?

4. Design an experiment to determine the presence of sulphur in an organic compound.
5. What happens when the organic compound is fused with sodium in the test?
6. What is the purpose of extracting the sodium fusion product with water?
7. What colour confirms the presence of nitrogen in the test?
8. What observation confirms the presence of sulphur?
9. Why is it important to perform each test separately for nitrogen, sulphur, and halogens after the sodium fusion?

QUANTIFICATION OF CARBON, HYDROGEN AND HALOGENS IN ORGANIC COMPOUNDS

The below outlines the procedures for testing for the mass composition of carbon, hydrogen and halogens in organic compounds. Once you have an understanding of these procedures, you will undertake some activities, to confirm your learning.

1. Convert the known mass of the dry sample to carbon dioxide and water by heating it over an oxidising agent.
2. Water produced is passed through a pre-weighed U-tube containing magnesium perchlorate and the carbon dioxide is passed through a pre-weighed U-tube containing sodium hydroxide.
3. The mass of carbon and hydrogen present are calculated as follows:
 Mass of carbon = $\frac{12}{44} \times \text{mass of CO}_2 \text{ produced}$
 Mass of hydrogen = $\frac{2}{18} \times \text{mass of H}_2\text{O produced}$
 Calculate the percentage composition by mass of C and H.
4. Determine the empirical formula and the molecular formula.

Estimation of halogen

- a. Use Carius method
- b. Use the formula:

$$\text{Mass of X} = \frac{\text{Relative atomic mass of X}}{\text{Molecular mass of AgX}} \times \text{mass of AgX produced}$$

Example 9.1

0.95 g of an organic compound on combustion in pure oxygen gave 2.85 g of CO_2 and 1.50 g of water. Calculate the percentage of carbon and hydrogen present. [C = 12, H = 1]

Answer**Use the problem-solving strategy**

- a.** Analyse the question

Known

Mass of organic compound = 0.95 g Mass of CO_2 produced = 2.85 g Mass of H_2O produced = 1.50 g

Ar. of C = 12, H = 1

Unknown

% C and % H = ?

- b.** Solve: Apply the problem-solving strategy

$$\text{Mass of carbon} = \frac{12}{44} \times \text{mass of } \text{CO}_2 \text{ produced}$$

$$\begin{aligned} \text{Mass of carbon} &= \frac{12}{44} \times 2.85 \\ &= 0.778 \text{ g of carbon} \end{aligned}$$

$$\text{Mass of carbon in the sample} = \frac{\text{mass of carbon}}{\text{mass of organic compound}} \times 100\%$$

$$\begin{aligned} \% \text{ of C} &= \frac{0.778}{0.95} \times 100\% \\ &= 81.89\% \end{aligned}$$

$$\begin{aligned} \text{Mass of hydrogen sample} &= \frac{2}{18} \times 1.50 \\ &= 0.1665 \text{ g of hydrogen} \end{aligned}$$

$$\text{Percentage of hydrogen} = \frac{\text{mass of hydrogen}}{\text{mass of organic compound}} \times 100$$

$$\begin{aligned} \% \text{ of H} &= \frac{0.1665}{0.95} \times 100\% \\ &= 17.52\% \end{aligned}$$

Activity 9.6: Determining the Mass of Elements (C, H, X) in Organic Compounds

A known mass of the compound is heated with CuO. The carbon present is oxidised to CO₂ and hydrogen to H₂O. The CO₂ is absorbed in the KOH solution, while H₂O is absorbed by anhydrous CaCl₂ and they are weighed.

$$\text{Percentage of C} = \frac{12}{44} \times \frac{\text{Mass of CO}_2}{\text{Mass of compound}} \times 100$$

$$\text{Percentage of H} = \frac{2}{18} \times \frac{\text{Mass of H}_2\text{O}}{\text{Mass of compound}} \times 100$$

Estimation of Halogens

Carius Method

A known mass of the compound is heated with Conc. HNO₃ in the presence of AgNO₃ in a hard glass tube called the Carius tube. C and H are oxidised to CO₂ and H₂O. The halogen forms the corresponding AgX. Then, it is filtered, dried and weighed.

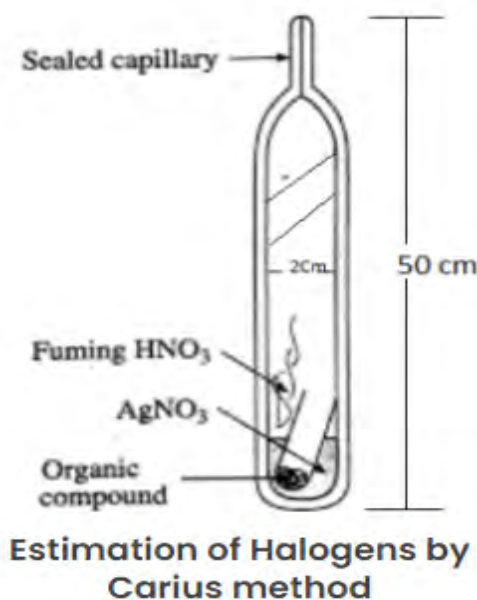


Fig 9.5: Estimation of Halogens by Carius method

$$\text{Percentage of X} = \frac{\text{Atomic mass of X}}{\text{molecular mass of AgX}} \times \frac{\text{Mass of AgX}}{\text{Mass of the compound}} \times 100$$

Calculations:

Let the mass of the given organic compound be m g.

Suppose the mass of AgX formed = m_1 g.

We know that 1 mol of AgX consists of 1 mol of X .

So, in m_1 g of AgX , mass of halogen = $\frac{\text{atomic mass of X} \times m_1 \text{ g}}{\text{molecular mass of AgX}}$

$$\text{Percentage of X} = \frac{\text{atomic mass of X} \times m_1}{\text{molecular mass of AgX} \times m} \times 100$$

Activity 9.7

- Describe how to test for carbon and hydrogen in a hydrocarbon compound.
- Outline the procedure to test for nitrogen, sulphur and halogens in an organic compound.
- Explain the process that is used for the estimation of carbon, hydrogen and halogen in organic compounds, etc.
- Name the reagents used to test for the presence of carbon in an organic compound.
 - Define the empirical formula of a compound.
- If the molar mass of a compound, X , is given, explain how to determine the molecular formula from the empirical formula.
- An organic compound, Q , contains C , H , O and N . A mass of 0.132 g of Q was burnt completely in oxygen to produce 0.072 g of water, 0.176 g of carbon (IV) oxide and 24.0 cm^3 of nitrogen.
 - Calculate the empirical formula of Q
 - If the molar mass of Q is 132 g mol^{-1} . Deduce the molecular formula of Q
 $[\text{O} = 16, \text{N} = 14, \text{C} = 12, \text{H} = 1, V_m = 22400 \text{ cm}^3 \text{ mol}^{-1}]$
- Sample Q is a crystal obtained from the bark of a tree. Plan an investigation to show that carbon, hydrogen and nitrogen are contained in the sample. You are provided with a common laboratory apparatus.

REVIEW QUESTIONS

Review Questions 9.1

1. Explain R_f values and their significance in paper chromatography. How can you calculate the R_f value of a particular component?
2. What is the difference between qualitative and quantitative analysis in organic chemistry?
3. How can we detect the presence of carbon and hydrogen in organic compounds?
4. Why is determining the purity of an organic compound important in both qualitative and quantitative analysis?
5. What are some real-world applications of qualitative and quantitative analysis of organic compounds?
6. What techniques are used to test for the presence of carbon and hydrogen in organic compounds?
7. How can sulphur be detected in organic compounds, and what reactions confirm its presence?
8. What is the procedure for detecting nitrogen in an organic compound?
9. What safety precautions are necessary when performing tests on organic compounds, particularly when using sodium metal and strong acids?

EXTENDED READING

Go online and read about how to estimate Sulphur and phosphorus in an organic compound. You can click on the links below to watch some videos.

- https://www.brainkart.com/article/Estimation-of-sulphur_36464/
- <https://www.youtube.com/watch?v=jJzWt3keHms>
- <https://www.youtube.com/watch?v=gdEpQ4jElg8>

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List of Contributors

Name	Institution
Samuel K. Agudogo	Adisadel College
Robert D. Akplai	Adidome SHS
Sylvester Bekyieriya	Lawra SHS
Christian Dzikunu	Achimota School, Accra