Laboratory analysis of organic compounds

This booklet addresses the following dot points from the 2024-2027 Study Design Unit 4,
- Area of Study 2
How are organic compounds analysed and used? outcome 2

Outcome 2

On completion of this unit the student should be able to apply qualitative and quantitative tests to analyse organic compounds and their structural characteristics, deduce structures of organic compounds using instrumental analysis data, explain how some medicines function, and experimentally analyse how some natural medicines can be extracted and purified.

- qualitative tests for the presence of carbon-carbon double bonds, hydroxyl and carboxyl functional groups

- applications and principles of laboratory analysis techniques in verifying components and purity of consumer products, including melting point determination and distillation (simple and fractional)

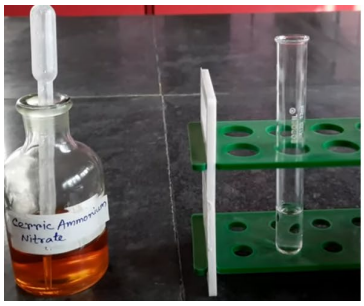
- measurement of the degree of unsaturation of compounds using iodine

Note – The Lucas test and the ceric ammonium nitrate test, both mentioned below, are classic techniques for distinguishing between primary, secondary, and tertiary alcohols. They are not explicitly required in the VCE Chemistry curriculum. The VCE Chemistry study design focuses more on the understanding of organic reactions and the identification of functional groups rather than specific qualitative tests like the Lucas test and the ceric ammonium nitrate test. The examiner my, however, decide to give the relevant information and ask students to conclude from a set of results, as per examples given here.

Testing for hydroxyl (-OH) groups

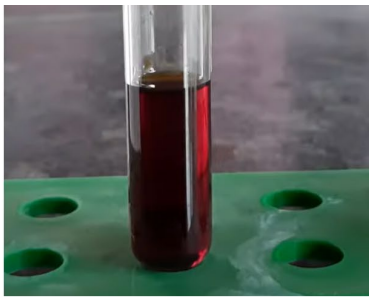
Ceric ammonium nitrate test

The ceric ammonium nitrate test is useful for detecting the presence of hydroxyl groups in alcohols, including primary, secondary, and tertiary alcohols. This test is a general qualitative



Clear pure sample

Sample with Ceric ammonium nitrate sample added



Test produces a red precipitate in the presence of hydroxyl groups

indicator of hydroxyl groups in organic molecules.

Lucas test

This test distinguishes between primary, secondary, and tertiary alcohols.

Reagent: Lucas reagent (a clear mixture of concentrated hydrochloric acid and zinc chloride). In the presence of a:

- **Primary Alcohols**: No visible reaction at room temperature.
- **Secondary Alcohols**: Cloudiness appears after a few minutes.
- **Tertiary Alcohols**: Immediate cloudiness or formation of an insoluble layer.

Three samples of three different, pure alcohols were placed in the three test tubes with Luca reagent. Immediately test tube C went cloudy indicating a tertiary alcohol.

Five minutes later test tube B also went cloudy indicating a secondary alcohol whilst test tube A did not turn cloudy at room temperature, indicating the presence of a primary alcohol.

Testing for carboxyl (-COOH) groups

Litmus test

This test identifies the presence of an acid. Blue litmus turns red in the presence of an acid.

Organic acids turn blue litmus red.

Sodium bicarbonate ($NaHCO_3$) test

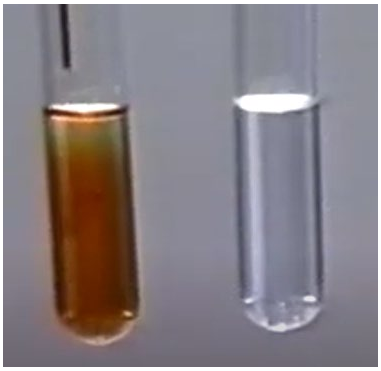
An acid when mixed with $NaHCO_3$ will react to form carbon dioxide gas according to the equation below.

$$R\text{-}COOH(aq) + NaHCO_3(aq) \rightarrow R\text{-}COONa\ (aq) + H_2O\ (l) + CO_2(g)$$

Testing for saturation (C=C)

Bromine water test

The reddish-brown colour of bromine disappears, indicating the presence of double or triple bonds (unsaturation) as bromine reacts with the C=C bonds in an addition reaction. No colour change indicates the compound is saturated (only single (C-C) bonds).



Test tube “A” contains a saturated hydrocarbon whilst test tube “B” contains an unsaturated hydrocarbon which turns bromine solution clear.

Iodine

Iodine test - this reaction is similar to the bromine test. Iodine reacts with carbon-to-carbon double bonds, to undergo a colour change, not always visible and distinct, as in the bromine water titration. Unlike the reaction with highly reactive bromine (Br_2), the reaction of iodine with unsaturated hydrocarbons is extremely slow and not feasible in analysis involving titration.
Below is a brief summary of the two tests for unsaturation.

Iodine Test:

- The reaction is similar in principle to the bromine test, where both involve addition across carbon–to-carbon double or triple bonds (C=C or C≡C).
- The brown/black colour of iodine (I_2 in solution) may fade slightly if unsaturation is present, indicating that iodine has reacted with the alkene or alkyne.
- The reaction is extremely slow and often reversible, because iodine is much less reactive than bromine.
- As a result, the iodine test is not reliable for detecting unsaturation in hydrocarbons and is not suitable for quantitative titration methods.
- The colour change is also less distinct, making end-point detection difficult.

Bromine Water Test (Bromine Titration):

- Bromine water is a more reactive halogen solution; it reacts readily with unsaturated compounds via addition reactions, breaking the C=C or C≡C bonds.
- The brown colour of bromine disappears quickly, due to bromine’s high reactivity, providing a clear and visible end point.
- Because of its reactivity and clear colour change, bromine water is widely used both qualitatively and quantitatively (in titration) to determine degrees of unsaturation.
- The reaction is fast and irreversible, making it much more feasible for analytical purposes than the iodine test.

Iodine number - also known as **iodine value,** is a measure of the degree of unsaturation (double bonds) in fats and oils. It is defined as the number of grams of iodine that react with 100 grams of the specified fat or oil. The more unsaturated the fat or oil, the higher its iodine number, because iodine reacts with the double bonds in the fatty acids. Reactions with iodine take time and are often left for several hours.

$$\text{Iodine Number} = \frac{\text{Mass of } I_2 \text{ absorbed}}{\text{Mass of fat sample}} \times 100$$

1. A sample of sunflower oil was analysed to determine its degree of unsaturation. It was found that 0.120 mol of iodine (I_2) reacts completely with 100 g of the oil.
   Calculate the iodine number of sunflower oil. (The iodine number is the mass of iodine, in grams, that reacts with 100 g of fat or oil.)

2. . A 0.500 g sample of an unknown lipid is found to absorb 0.00440 mol of iodine, I_2, in an addition reaction.
   a. What is the iodine number of this lipid?

   b. Given that sunflower oil has an iodine number of 130 discuss its suitability as a biodiesel in cold climates with clear reference to intermolecular forces.

   _____

   _____

   _____

   _____

   c. Given that atmospheric oxygen (O_2) reacts with carbon-to-carbon double bonds to form peroxides that can undergo polymerisation, suggest one limitation for this fuel.

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Testing for purity

Melting point determination

Pure substances have uniform intermolecular forces acting between all the particles of the pure compound in the solid state. A specific amount of heat energy will break all the intermolecular bonds instantly. Pure substances, therefore, have a well-defined melting point and complete the transition from solid to liquid in a very small temperature range, usually withing 0.2 ^{o}C to 2.0 ^{o}C. For example, pure benzoic acid melts sharply at 122°C and pure aspirin at 136 ^{o}C.

Impurities disrupt the regular lattice structure of a crystalline solid, making it easier to overcome the intermolecular forces holding the solid together. As a result, an impure sample melts over a broader range and at a lower temperature compared to the pure substance. This phenomenon is known as ***melting point depression***. For instance, if benzoic acid is contaminated with a small amount of salicylic acid, the melting point may drop and spread over a range of several degrees, say from 118°C to 121°C.

Chemistry Behind Melting Point Depression

Intermolecular Forces:

- Pure substances have uniform intermolecular forces. During melting, these forces need to be overcome, which occurs at a specific temperature.

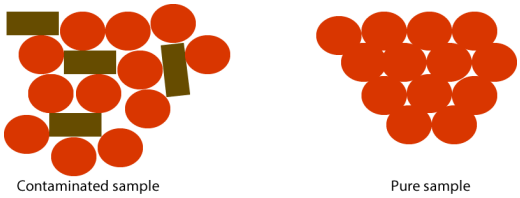


Diagram 1.

- Impurities introduce different intermolecular interactions, between the particles of the solid, which alter the energy required for the phase transition.

- Different shaped particles also change the packing structure within the solid leading to weaker intermolecular forces as shown above in diagram 1.

Testing for purity- simple and fractional distillation

Both simple and fractional distillations separate liquids from a mixture using the boiling temperature of each component in the mixture. Pure substances have a sharp and distinct boiling point.

Simple Distillation: - Simple distillation is ideal for separating a liquid from a solution or a mixture of liquids with significantly different boiling points (greater than 25-30°C apart). Simple distillation involves 3 steps.

1. Heating the mixture gradually allows the component with the lower boiling point to vaporise first.
2. As the vapour passes through the condenser it cools and condenses back into a liquid.
3. The pure liquid is then collected in the collecting flask as the distillate.

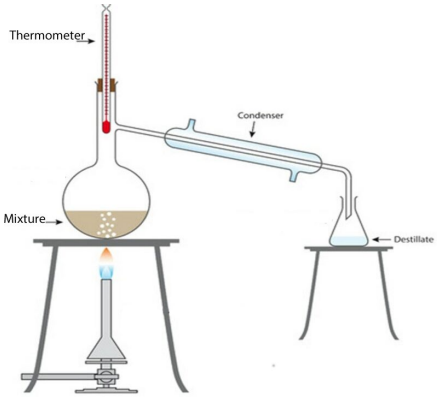


Diagram 2- simple distillation setup.

Pure compounds have a sharp boiling point. By monitoring the temperature at which the distillate is collected, you can assess its purity. A pure compound will distil over a narrow temperature range. If the compound is pure, the temperature should remain constant throughout the distillation of that particular component.

Fractional Distillation - Fractional distillation is used for separating mixtures of liquids with closer boiling points (less than 25-30°C apart). It provides better separation than simple distillation by using a fractionating column. The process is similar to simple distillation but with the exception of a fractionating column between the distillation flask and the condenser. The fractionating column provides a larger surface area where multiple vaporisation-condensation cycles occur. As the vapours of different compounds rise up the fractionating column the components with the higher boiling points condense and return to the flask, whilst the lower melting point components continue up the fractionating column and pass into the condenser.

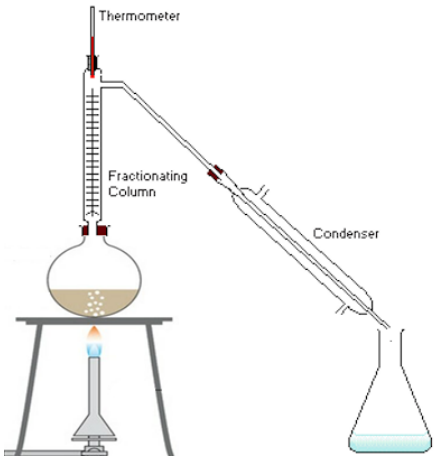
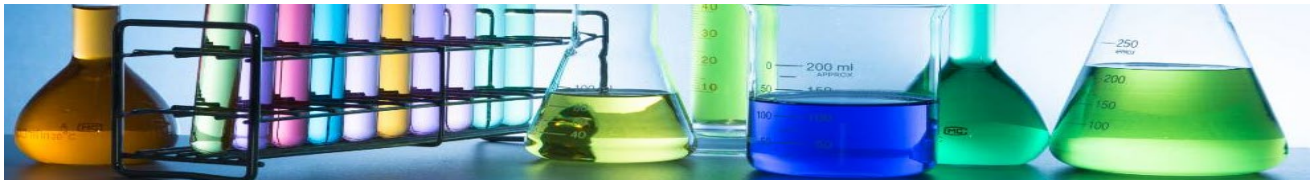


Diagram 3 – fractional distillation

Purity can be verified by distilling within a narrow temperature range, according the literature values of boiling points of the components of the mixture under investigation. If impurities are present they will distil over a range of temperatures resulting in a number of distinct fractions.



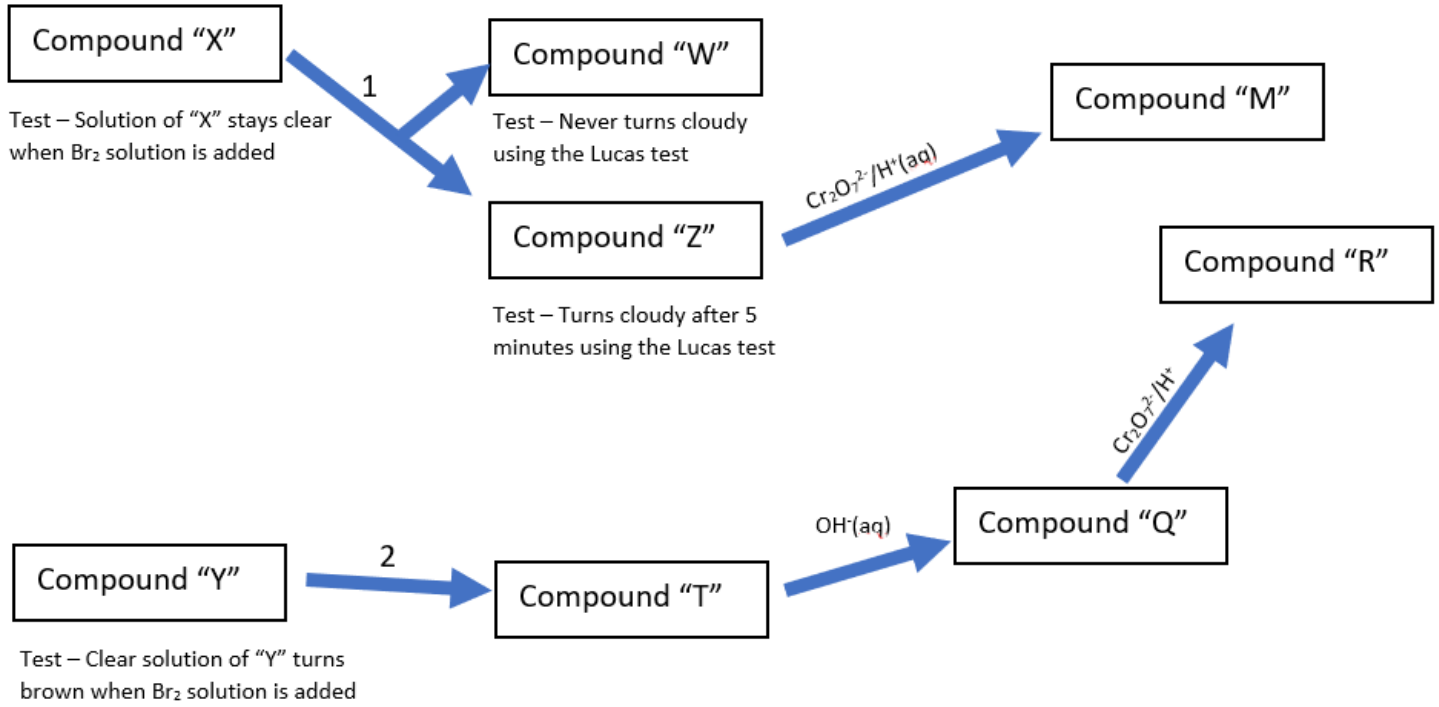


Figure 1 – Reaction pathways and test for reaction products.

Consider the reaction pathways in fig 1, above.

1. Both X and Y are unbranched hydrocarbons, 4 carbon atoms long.
    a. Given that compound "R" reacts vigorously with a solution of Na_2CO_3 to produce CO_2 gas, complete the table below. ***24 marks***

Compound	IUPAC name	Criteria used to identify the compound	Skeletal structure
X			
W			
Z			
R			
M			
Y			
T			
Q			

b. Identify the class of reaction represented by reaction: ***2 marks***

- 1 _____

- 2 _____

c. What are the reagents and conditions for reaction 2? ***1 mark***

_____

d. An Isomer of compound "X" was the new reactant for reaction 1 and produced compound "P". Compound "P" was subsequently tested using the Lucas reagent and found that it turned cloudy immediately.

i. Give the IUPAC name for compound "P".

_____ ***1 mark***

ii. Draw its skeletal structure in the box below.

e. The formation of what product/s takes place using a redox reaction?

_____ ***3 marks***

f. Give a balanced chemical half equation (states included) for the reduction reaction taking place during one of the redox reaction/s mentioned in e. above. ***2 marks***

g. What class of reaction is responsible for the formation of compound Q? ***1 mark***

2. Compound "K" is a straight-chained hydrocarbon containing 5 carbon atoms. A pure, 0.520 grams sample of compound "K" (molar mass 66 amu) was dissolved in 100 mL using an appropriate solvent and the resulting clear solution placed in a 200 mL conical flask. A few drops of a colourless starch solution were added to the flask and the clear solution titrated against a 1.00 M I_2. The experimental setup is shown in fig. 2.

Figure 2 – experimental setup

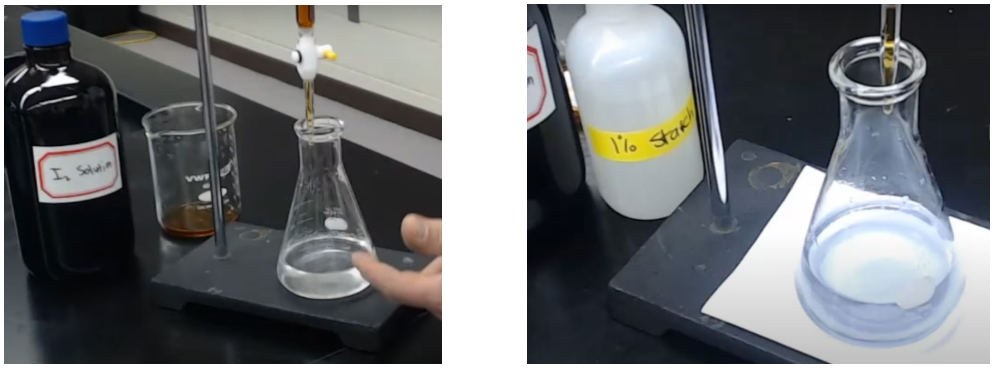


Figure 3 – colour change at endpoint.

The endpoint was reached when the solution turns permanently blue (fig. 3), where upon an average titre of 23.61 mL was obtained using 3 concordant results

a. Calculate the mol of I_2 needed to react with 0.520 grams of the hydrocarbon. ***2 marks***

b. Calculate the mol of hydrocarbon in the conical flask ***1 mark***

c. Give the IUPAC name/s for all the possible isomers of "K".

_____ ***3 marks***

d. Draw the skeletal formulae of all the possible isomers of compound "K".

2 marks

e. What should the burette be washed with prior to starting the titration?

_____ ***1 mark***

f. Another group conducted the same experiment and got the results shown in fig. 4.
Indicate how the average titre for group 2 would differ from the previous group's results and give a detailed explanation.

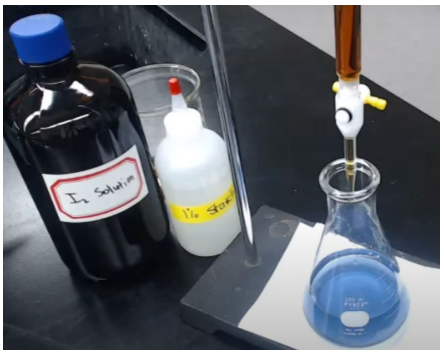


Figure 4 – end point of group 2

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_____ *2 marks*

g. The difference in the colour change of group 2 was explained by one student in their logbook. Below is the extract from the booklet.
"*The difference in colour can be explained by the fact that our group (group 2) washed the conical flask with distilled water and did not dry the flask before the titration procedure. This also diluted the contents of the flask and a greater volume of titre was needed*"
Comment on the validity of this explanation.

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_____ *2 marks*

3. Consider the experiment for the determination of the degree of unsaturation of a straight-chain alkene by titration with iodine solution

* Note - This method is not practical for a quick titration, as the addition reaction with iodine is very slow and often requires several hours to reach completion. The degree of unsaturation refers to the total number of carbon-to-carbon double or triple bonds and ring structures present within a molecule.

Objective - To determine the degree of unsaturation (number of double bonds) in an unknown alkene by titrating it with a standard iodine solution using starch as an indicator.
Safety and Disposal:

- Wear safety goggles, gloves, and a lab coat at all times.
- Handle iodine solution with care as it can stain and is harmful if ingested or comes into contact with skin.
- Dispose of all chemical waste according to specified safety protocols.

Materials:

- Iodine solution (0.15 M)
- 0.05M alkene solution
- Starch solution (indicator)
- Distilled water
- Burette (50mL)
- Pipette (25mL)
- Conical flask (200 mL)
- White tile
- Safety goggles
- Lab coat
- Gloves

Procedure:

1. Rinse the burette with a small amount of the iodine solution.
2. Fill the burette with the iodine solution, ensuring there are no air bubbles. Note the initial volume.
3. Pipette 25.0 mL of the alkene solution into a clean conical flask.
4. Add 3-5 drops of starch solution to the alkene solution in the conical flask. The solution will remain colourless.
5. Place the conical flask on a white tile to better observe the colour change.
6. Slowly add the iodine solution from the burette to the alkene solution while constantly swirling the flask.
7. As iodine is added, it will react with the alkene, causing the solution to remain colourless. Continue adding iodine until the solution starts to turn blue-black, indicating the presence of unreacted iodine.
8. At this point, add the iodine drop by drop until the blue-black colour persists for at least 30 seconds, indicating the endpoint.

Recording Results:

Burette reading	Trial 1	Trial 2	Trial 3	Trial 4
Start (mL)	0.00	12.88	5.00	21.01
Finish (mL)	25.01	37.78	30.50	46.01
Total (mL)	25.00	24.90	25.50	25.00

a. Determine the average titre (mL) ***2 marks***

b. Determine the moles of iodine (I_2) used in the titration. Giv ethe answer to the right number of significant figures. ***2 marks***

c. Determine the moles of alkene. Give the answer to the right number of significant figures. ***2 marks***
Mol of alkene = Molarity × Volume (L)

d. Calculate the degree of unsaturation (number of double bonds) in the alkene: ***1 mark***

e. Give the IUPAC names of all the possible isomers of the alkene given that it is a straight chain hydrocarbon of 6 carbons. ***2 marks***

Conclusion:

1 mark

4. The melting point of aspirin is given as 136 °C. Four bottles were left on the table in the laboratory marked A, B, C and D. Each bottle contained a white powder but no indication whether it was aspirin or not. The results of a melting point test of the four powders is shown below in fig. 5.

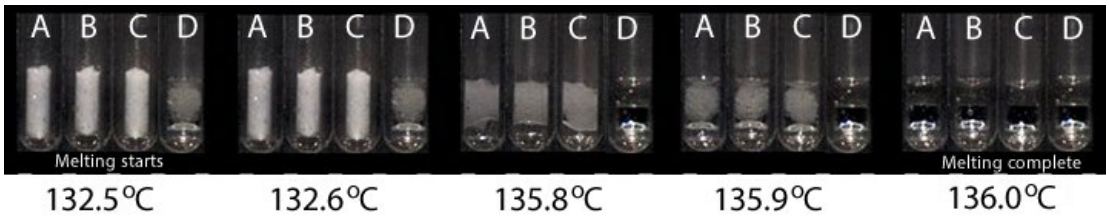

Figure 5. Melting point test of four unknown white powders

a. Which bottle/s contains pure aspirin? Justify your answer.

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_____ ***2 marks***

b. Which bottle/s contained an impure sample of aspirin or a totally different compound? Justify your answer.

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_____ ***2 marks***

5. A liquid mixture containing methanol (BP 64.7°C) and ethanol (BP 78.4°C) needs to be separated.

a. Explain the steps and apparatus required for this separation.

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_____***2 marks***

b. What is the purpose of a fractionating column and when is it used.

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_____***2 marks***

6. You are provided with a mixture of three white solid compounds, compound A, compound B and compound C. These compounds are known to be volatile with low boiling points. The boiling points of the compounds are as follows.

- **Compound A**: 70°C
- **Compound B**: 75°C
- **Compound C**: 80°C

a. Describe a detailed, step-by-step experimental procedure to separate these compounds that can be performed in the school laboratory.

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_____ ***4 marks***

b. Include a detailed, labelled setup, in the box provided below. ***4 marks***

c. Provide a qualitative analysis method to verify the purity of each component.

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_____***2 marks***

7. A student is given 2.50 grams of a brand of butter, and it absorbs 0.015 moles of iodine (I_2) , 253.8 g/mol, during the iodine number determination. Given that the iodine number of a fat or oil is given as the mass, in grams, of iodine that reacts with 100 grams of the fat or oil, calculate the iodine number of the butter, to the right number of significant figures. ***2 marks***

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