Qualitative analysis of organic compounds

Solutions

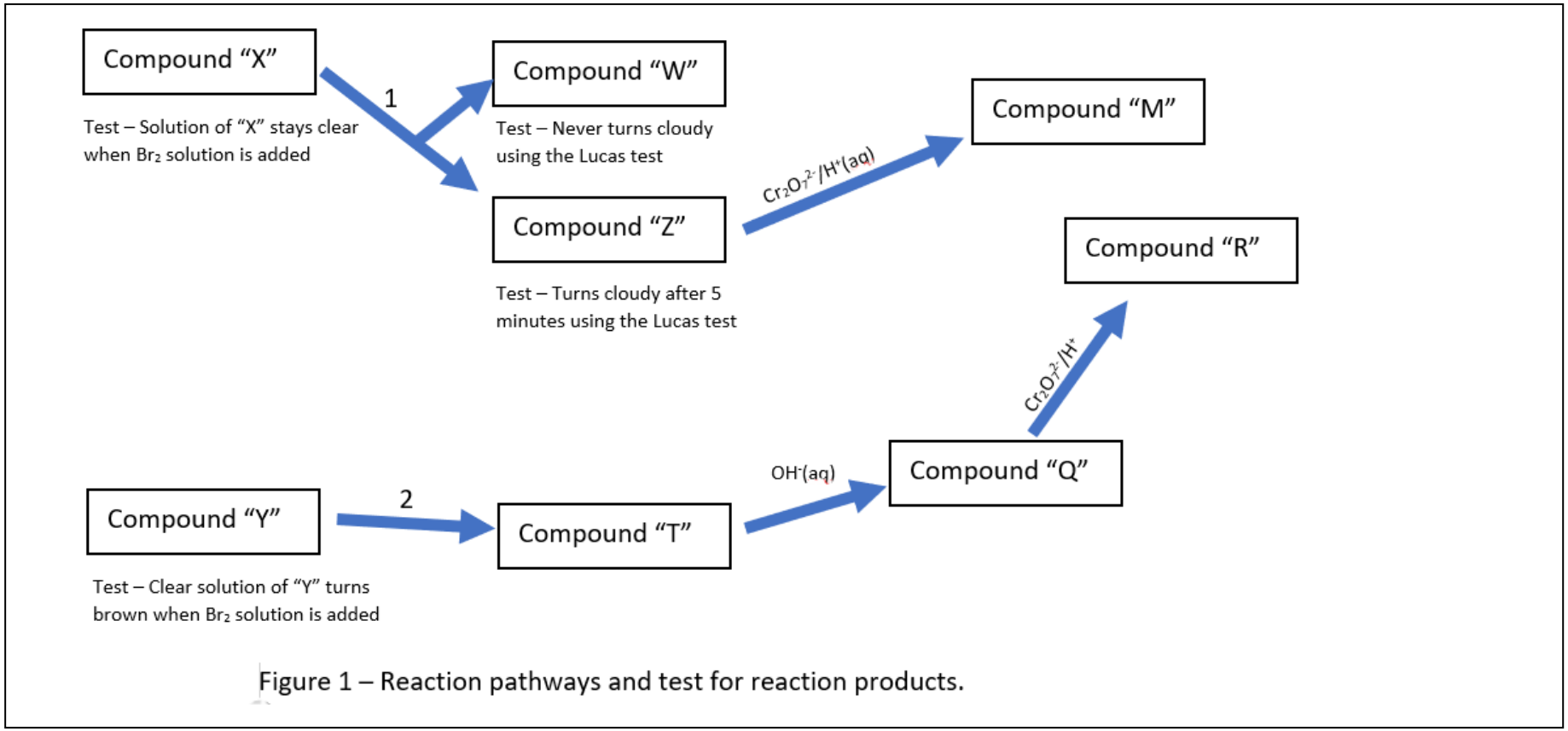
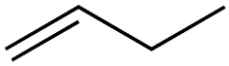
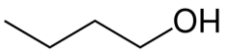
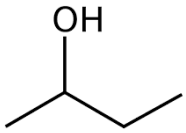
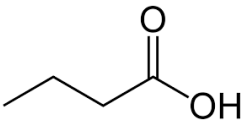
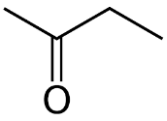
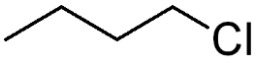
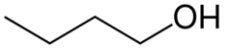


Figure 1 – Reaction pathways and test for reaction products.

Consider the reaction pathways in fig 1, above.

1. Both X and Y are straight-chain hydrocarbons containing 4 carbon atoms.
   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	*But-1-ene*	*The product is the only reactant for the formation of a secondary and primary alcohols. Most likely an alkene undergoing an addition reaction.*	
W	*Butan-1-ol*	*Is a primary alcohol and does not turn cloudy with the reagent.*	
Z	*Butan-2-ol*	*Is a secondary alcohol and turns cloudy with the reagent after a brief period.*	
R	*Butanoic acid*	*It reacts reacts vigorously with Na_2CO_3 to produce CO_2. It is an organic acid.*	
M	*Butanone*	*Oxidation of a secondary alcohol produces a ketone*	
Y	*Butane*	*It does not react with Br_2 hence it is a straight chain saturated hydrocarbon with 4 carbons.*	
T	*1-chlorobutane*	*"T" is formed from a hydrocarbon and ultimately forms a primary alcohol when undergoing a substitution reaction with OH^- ions and then oxidised to an acid.*	
Q	*Butan-1-ol*	*Primary alcohol, undergoes oxidation to an acid.*	

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

- 1 _____*addition*_____

- 2 _____*Substitution*_____

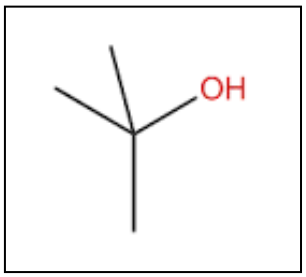
c. What are the reagents and conditions for reaction 2? ***1 mark***

_____*Cl_2/U.V. light*_____

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".
"P" is a tertiary alcohol . 2-methylpropan-2-ol ***1 mark***

ii. Draw its skeletal structure in the box below.



e. The formation of what product/s takes place using a redox reaction?
_____*T, R and M*_____ ***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***
$6e^- + 14H^+(aq) + Cr_2O_7^{2-} + \rightarrow 2Cr^{3+} + 7H_2O(l)$ for the formation of R and M
$Cl_2(g) + 2e^- \rightarrow Cl_2(g)$ for the formation of T

g. What class of reaction is responsible for the formation of compound Q?
Substitution ***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 was 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

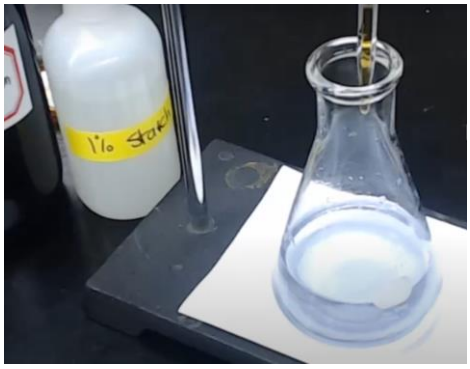


Figure 3 – colour change at endpoint.

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***

Mol = C X V ------- 1 mark
=> mol of I_2 = 1.00 M X 0.02361 = 0.0236 mol ----- 1 mark (correct and to three sig figs)

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

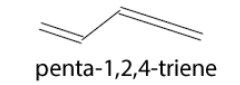
Mol = 0.520 / 66 = 0.0079 ------- 1 mark

c. Give the IUPAC name/s for all the possible isomers of “K”. ***3 marks***
Ratio alkene : I_2 => 0.0079 : 0.0236 => 1 : 3 hence 3 double bonds ---- 1 mark
Possible isomers :
- penta-1,2,4-triene ----- 1 mark
-penta-1,2,3-triene ------1 mark
-

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

2 marks

1 mark for each correct isomer



penta-1,2,4-triene

penta-1,2,3-triene

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

_____*1.00 M I_2*_____ ***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.

2 marks

Figure 4 – end point of group 2

The deep colour change of the solution in the conical flask indicates an overshot of the endpoint. Hence too much I_2 was added. --------- 1 mark
This would lead to a higher value for the amount of I_2 added and hence a higher degree of unsaturation. Eg possibly 4 double bonds. ------- 1 mark

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. *2 marks*

If water was present in the conical flask is would, if anything, dilute the colour of the indicator as it changes to blue rather than make it a deeper blue ------- 1 mark
Water can be used to rinse the conical flask and this will not impact the results. ------ 1 mark

3. Consider the experiment for the determination of the degree of unsaturation of an alkene by titration with iodine solution

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
- Pipette
- Conical flask
- White tile
- Measuring cylinder
- Safety goggles
- Lab coat
- Gloves

Procedure:

Setup:

- Rinse the burette with a small amount of the iodine solution, then fill it with the iodine solution, ensuring there are no air bubbles. Note the initial volume.
- Rinse a pipette with the alkene solution and then pipette 25.0 mL of the alkene solution into a clean conical flask.

Titration:

- Add 3-5 drops of starch solution to the alkene solution in the conical flask. The solution will remain colourless.
- Place the conical flask on a white tile to better observe the colour change.
- Slowly add the iodine solution from the burette to the alkene solution while constantly swirling the flask.
- 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.
- At this point, add the iodine drop by drop until the blue-black colour persists for at least 30 seconds, indicating the endpoint.

2. **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***

Three concordant results 25.00 + 24.90 + 25.00 -------- 1 mark
Average accurate value = 24.97 mL ------ 1 mark

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

Mol of iodine = Molarity × Volume (L)

mol of I_2 = 0.15 M X 0.02497 = 3.8 X 10^{-3} (2 sig figs)
--- 1 mark for correct mol
--- 1 mark for 2 sig figs

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

mol of alkene = 0.05 M X 0.025 = 1.3 X 10^{-3} (2 sig figs)

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

Ratio of mol of alkene to I_2
=> 1.3 : 3.8
=> simplest ratio = 1.3
three double bonds, the alkene is a triene.

e. Give the IUPAC names of all the possible isomers of the alkene given that it is an unbranched hydrocarbon of 6 carbons. ***2 marks***
Hexa-1,2,4-triene, hexa-1,2,3-triene, hexa-1,3,5-triene, hexa-1,2,5-triene, Hexa-2,3,4-triene
------ 1 mark for correct naming
------ 1 mark for correct number of isomers.
Click to revise naming of compounds with more than one double bond

Conclusion: ***1 mark***

The degree of unsaturation of the alkene was determined to be 3 by titration with a standard iodine solution using starch as an indicator.

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.

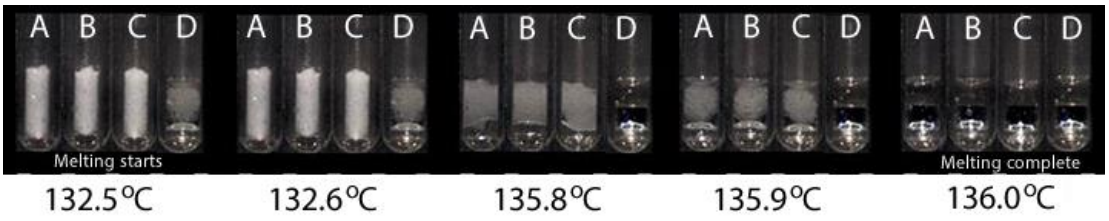


Figure 5. Melting point test of four unknown white powders

a. Which bottle/s contains pure aspirin? Justify your answer. ***2 marks***

Bottle A, B, and C contain pure samples of aspirin ---------- 1 mark
The melting point of each sample was in a narrow range between 135.8 °C and 136.0 °C consistent with the literature value. ---------- 1 mark

b. Which bottle/s contained an impure sample of aspirin or a totally different compound? Justify your answer. ***2 marks***

Bottle C ---------- 1 mark

MP of the compound in bottle C has a broad range MP from 132.5 °C to 135.8 °C. This indicates that the sample contains impurities. ---------- 1 mark

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

a. Explain the steps and apparatus required for this separation. ***2 marks***

- *Since the BPs of both methanol and ethanol are relatively close use a distillation apparatus, with a fractionating column. --------- 1 mark*
- *Gradually heat the mixture to 65.0 °C. Methanol, with the lower boiling point, will vaporize first, condense and collect in the collecting flask. The ethanol should remain in the original flask -------- 1 mark*

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

The fractionating column provides a large surface area for compounds to undergo repeated vaporisation-condensation cycles. This increases the purity of the compound being separated. ---------- 1 mark

It is used when liquids in a mixture differ in BP by a small margin, usually less than 10°C. ------- 1 mark

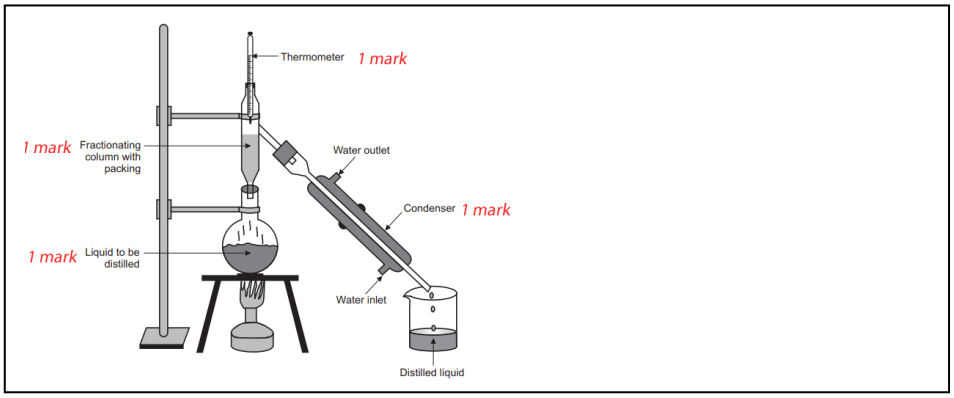
6. You are provided with a mixture of three white solid compounds, compound A, compound B and compound C. These compounds are known to be volatile with low boiling points. The boiling points of the compounds are as follows.

- **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.

- Using a fractional distillation apparatus, gradually heat the mixture in the distillation flask. -------- *1 mark*
- Heat the mixture to 50°C and keep the temperature steady. Compound A with a boiling point of 50°C will vaporise and condense first. ------- *1 mark*
- *When no more distillate is seen to collect increase the temperature to 75°C and collect compound B in a separate collecting flask* ----- *1 mark*
- *When no more distillate is seen to collect increase the temperature to 80°C and collect compound C in a separate collecting flask* -------- *1 mark*
- *The vapour rises through the fractionating column, undergoing multiple vaporization-condensation cycles, enhancing separation.*

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. ***2 marks***

The pure compounds can be cooled back to a powder form and their MP measured. --------- 1 mark
If the MP is in the specified literature value within a range of 0-2 °C then the sample is pure. -------- 1 mark

Or

They can be distilled once again. ---------- 1 mark
If the temperature in the flask containing the sample does not increase but remains at the theoretical value of the compound until no more distillate can be collected, then the sample is pure. If the temperature fluctuates then the sample is impure. ------ 1 mark