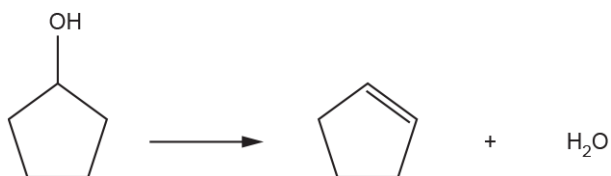


# Analytical Techniques

1. Cyclopentanol can be reacted to form cyclopentene.  
Cyclopentene is a liquid with a boiling point of 44 °C and a density of 0.74 g cm<sup>-3</sup>.  
A student plans to prepare 4.00 g of cyclopentene by reacting cyclopentanol (boiling point 140 °C) with an acid catalyst.

## Equation

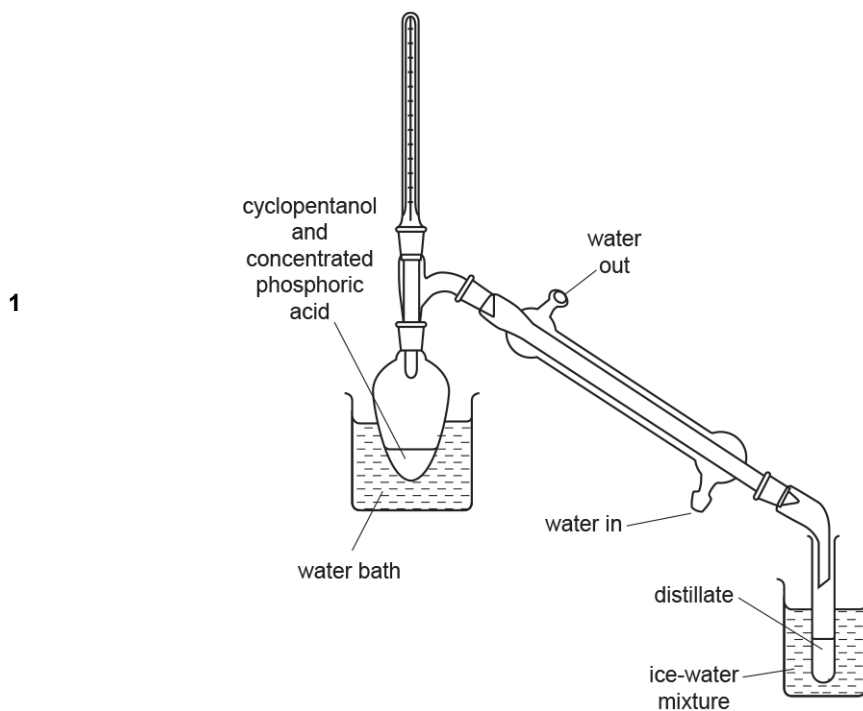


The expected percentage yield of cyclopentene is 64.0%.

## Method

The student carries out the preparation using apparatus set up for distillation, as shown below.

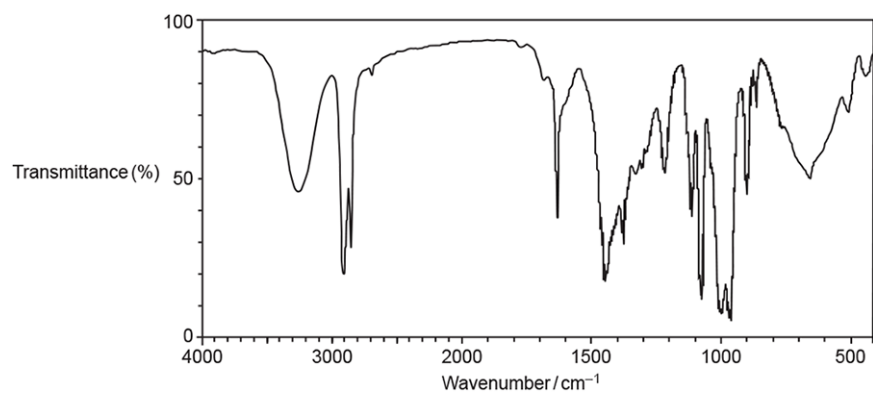
The reaction mixture is heated gently, and a distillate containing impure cyclopentene is collected.



- 2 The distillate has an aqueous layer and an organic layer.  
The student purifies the cyclopentene from the distillate.

The organic layer in the distillate was analysed by IR spectroscopy.  
The IR spectrum is shown below.

## 4.2.4 Analytical Techniques



Explain how the IR spectrum of the organic layer suggests that cyclopentene has been formed and that the reaction is incomplete.

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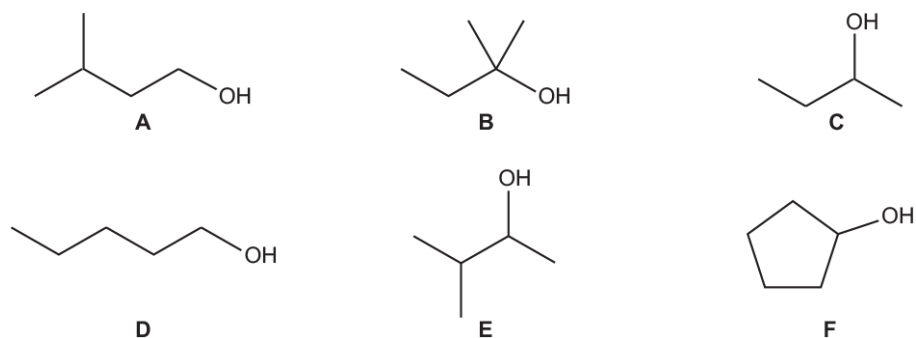
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[2]

## 4.2.4 Analytical Techniques

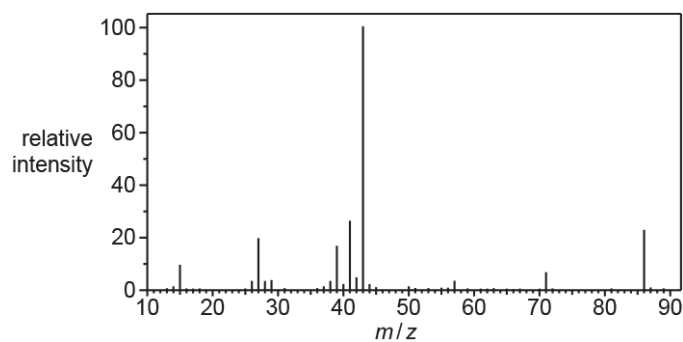
2. \* The structures of alcohols A–F are shown below.



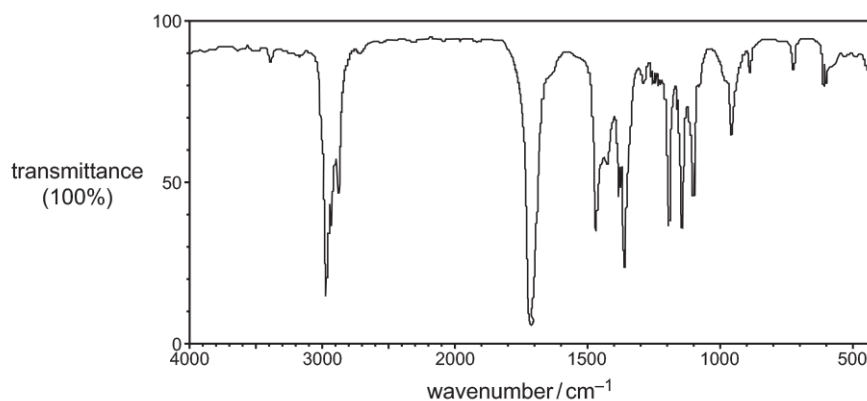
Compound **X** is one of the alcohols **A–F**.

A student refluxes compound **X** with acidified potassium dichromate(VI) as an oxidising agent. A pure sample of the organic product **Y** is obtained from the resulting mixture. The mass spectrum and IR spectrum of **Y** are shown below.

### Mass spectrum of **Y**



### IR spectrum of **Y**



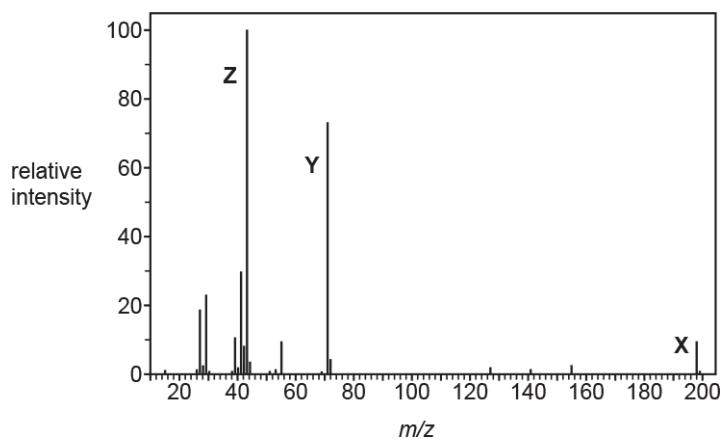
Using this information, identify compound **X** and product **Y**, and write an equation for the formation of product **Y** from compound **X**. You may use [O] to represent the oxidising agent.



## 4.2.4 Analytical Techniques

**3(a).** This question is about 1-iodopentane,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{I}$ .

The mass spectrum of 1-iodopentane is shown below.



i. What information is given by the peak labelled **X** ( $m/z = 198$ )?

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[1]

ii. Write the structural formulae of the ions responsible for the peaks labelled **Y** and **Z**.

**Y** ( $m/z = 71$ )

**Z** ( $m/z = 43$ )

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[2]

**(b).** 2-Iodo-2-methylbutane is an isomer of 1-iodopentane.

i. Draw the structure of 2-iodo-2-methylbutane.

[1]

ii. Suggest **one** similarity and **one** difference between the mass spectra of 1-iodopentane and 2-iodo-2-methylbutane.

Similarity

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Difference

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[2]

## 4.2.4 Analytical Techniques

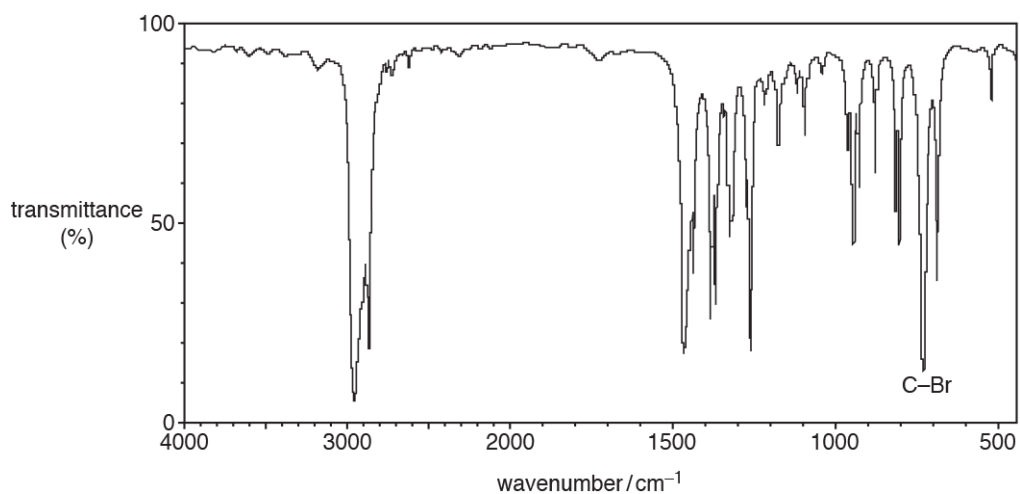
4. An alcohol can be prepared by hydrolysing the haloalkane  $C_2H_5CHBrCH_3$  with aqueous sodium hydroxide.

i. Outline the mechanism for this reaction.

Show curly arrows and relevant dipoles.

[3]

- ii. The infrared (IR) spectrum for  $C_2H_5CHBrCH_3$  is shown in **Fig. 25.2**. The C–Br bond absorption is labelled.



**Fig. 25.2**

Outline how IR spectroscopy could be used to show that the bromoalkane functional group has reacted and that the alcohol functional group has formed.

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[2]

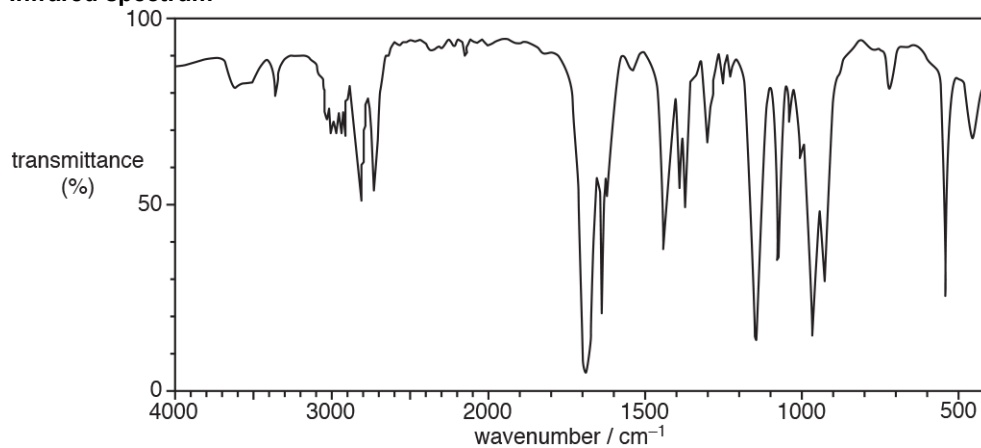
## 4.2.4 Analytical Techniques

5. \* Compound **F** is a *trans* stereoisomer which is a useful intermediate in organic synthesis.

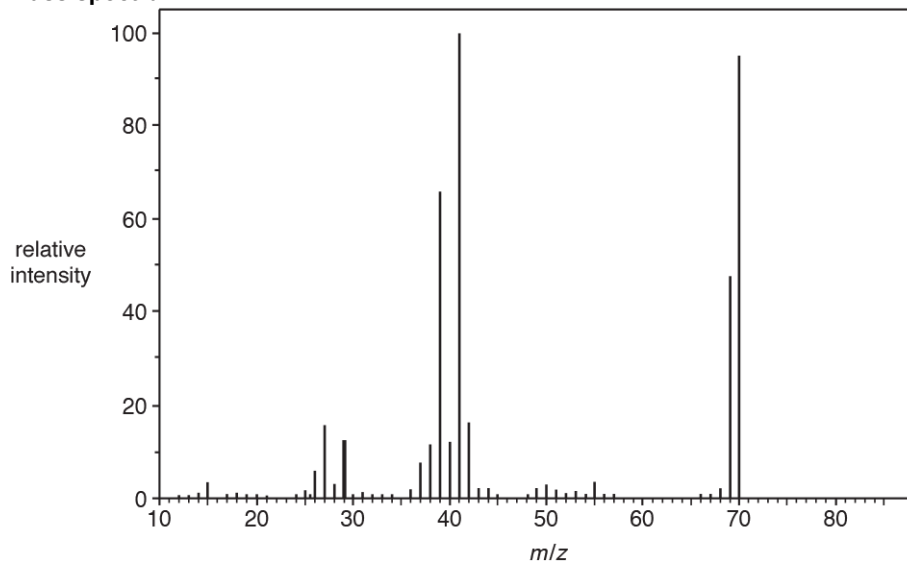
The results of elemental and spectral analysis of compound **F** are shown below.

Percentage composition by mass: C, 68.6 %; H, 8.6 %; O, 22.8 %.

### Infrared spectrum



### Mass spectrum



In the mass spectrum, the peak with the greatest relative intensity is caused by the loss of a functional group from the molecular ion of compound **F**.

Determine the structure of compound **F**.

Explain your reasoning and show your working.

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## 4.2.4 Analytical Techniques

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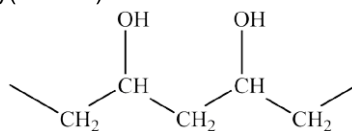
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**[6]**

6. Poly(ethenol) is used to make soluble laundry bags.

A section of the structure of poly(ethenol) is shown below.



i. Draw a structure to represent one repeat unit of poly(ethenol).

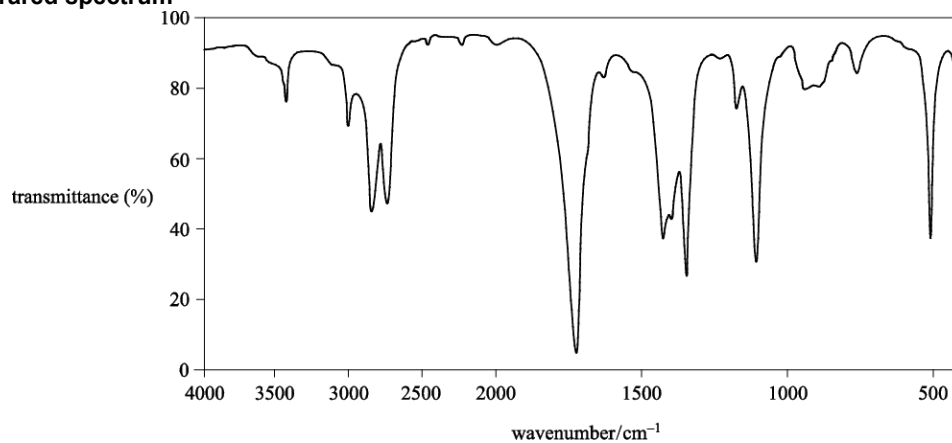
**[1]**

ii. Poly(ethenol) is not manufactured from ethenol.

Ethenol is unstable and it forms a more stable structural isomer.

Analysis of the structural isomer gave the following data.

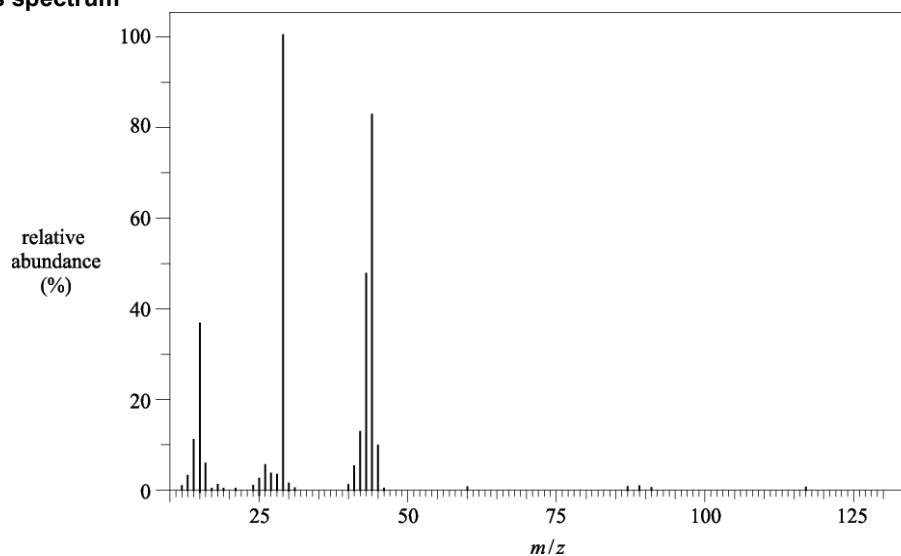
**Infrared spectrum**





## 4.2.4 Analytical Techniques

Mass spectrum



Use **all** the data to show that the isomer is **not** ethenol.

Identify the structural isomer of ethenol.

In your answer you should make clear how your explanation is linked to the evidence.

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**[4]**

7. Why are scientists concerned about the release of methane into the atmosphere?

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**[1]**

## 4.2.4 Analytical Techniques

- 8(a).** A student was provided with a mixture of two structural isomers. Each isomer has the percentage composition by mass C, 29.29%; H, 5.70%; Br, 65.01%. The relative molecular mass of each isomer is less than 150.

Determine the structures of the two structural isomers.

Show your working.



*In your answer you should link the evidence with your explanation.*

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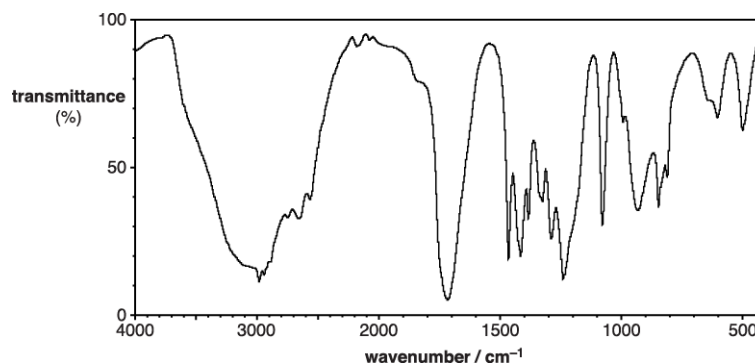
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[5]

- (b).** The student heats the mixture of the two structural isomers from **(a)** under reflux with aqueous sodium hydroxide to form two compounds, **E** and **F**. The student separates the two compounds.

Compound **E** is heated under reflux with acidified potassium dichromate(VI) to form compound **G**, which gives the infrared spectrum below.



- i. Analyse the information and spectrum to determine the structures of **E**, **F** and **G**.  
Include an equation for the formation of **G** from **E**.



## 4.2.4 Analytical Techniques

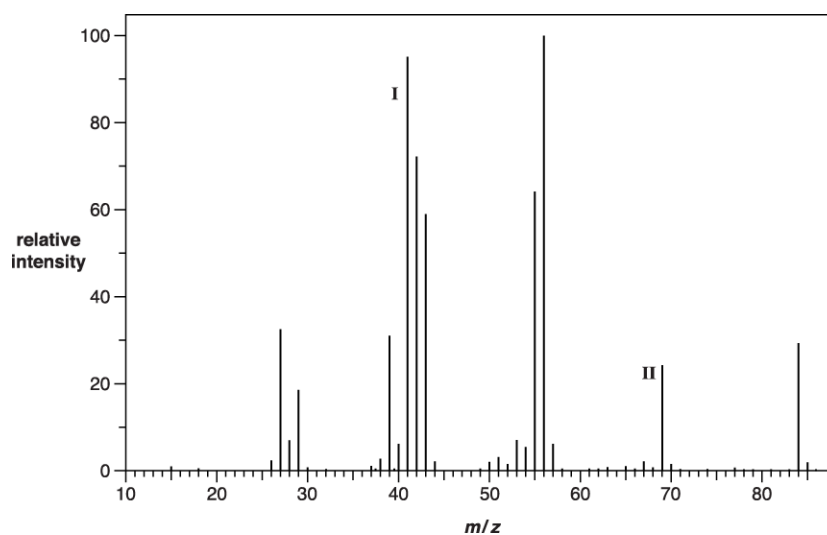
- ii. Compound **G** is heated with compound **F** in the presence of a small amount of concentrated sulfuric acid to form organic compound **H**.

Draw the structure of the organic compound **H**.

[2]

9. This question is about several unsaturated hydrocarbons.

The mass spectrum of an alkene is shown below.



- i. The empirical formula of the alkene is  $\text{CH}_2$ .

Use the empirical formula and the mass spectrum to confirm the molecular formula as  $\text{C}_6\text{H}_{12}$ .

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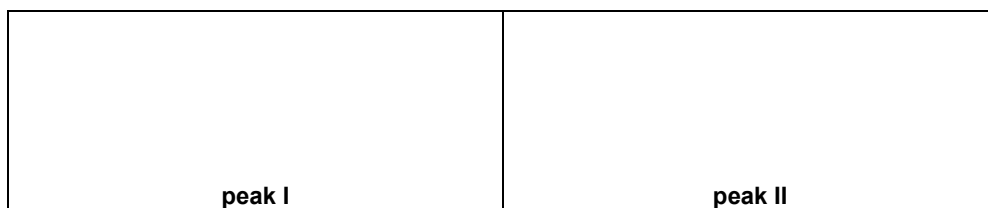
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[1]

## 4.2.4 Analytical Techniques

- ii. Further analysis showed that the alkene was hex-2-ene.

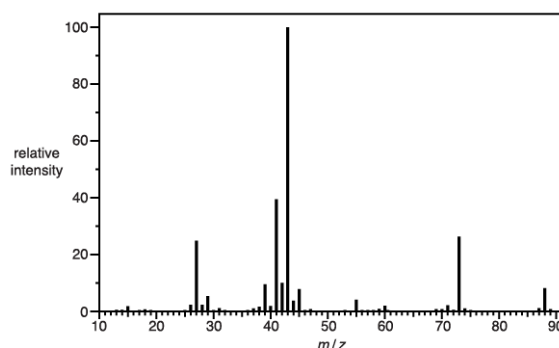
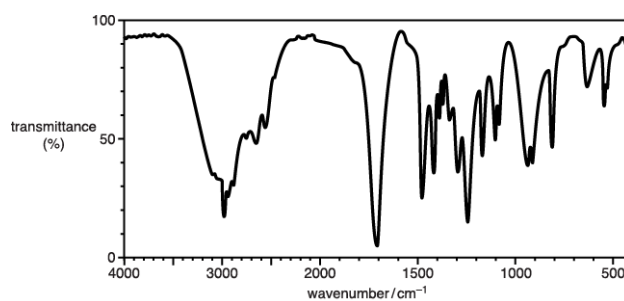
Suggest possible structures for the species responsible for the labelled peaks I and II in the mass spectrum of hex-2-ene shown opposite.



[3]

10. \* Organic compound **C** has the following percentage composition by mass: C, 54.5%; H, 9.1%; O, 36.4%.

The infrared spectrum and mass spectrum of compound **C** are shown below.



In the mass spectrum, a secondary carbocation is responsible for the peak with the greatest relative intensity.

Identify compound **C**.

In your answer you should make clear how your conclusion is linked to all the evidence.

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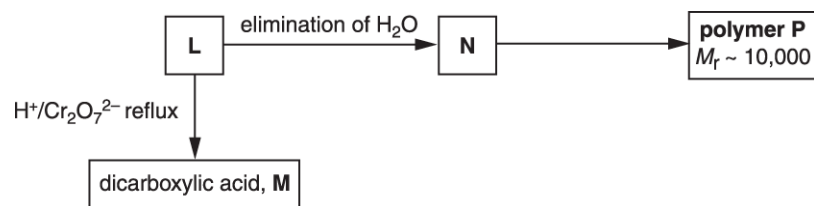
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[6]

11(a). L, M, N and P are straight-chain organic compounds containing C, H and O only.

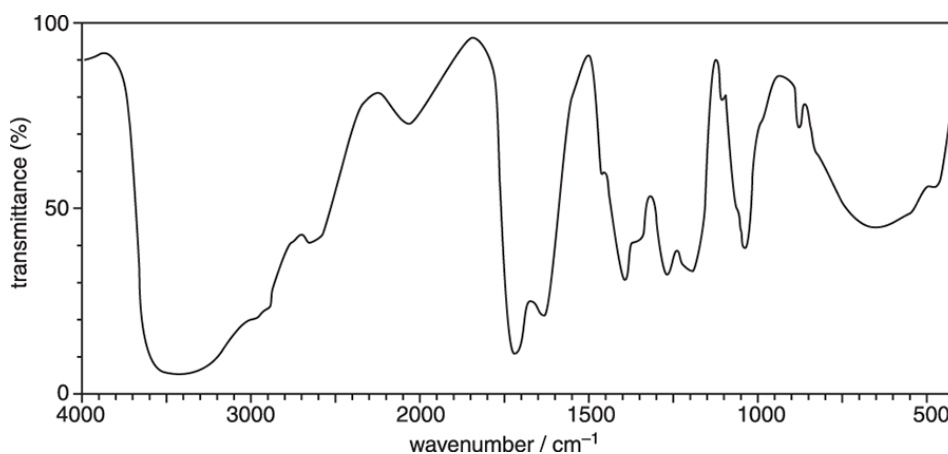
The flowchart shows reactions involving these compounds.



Analysis of compound L shows the following.

- Percentage composition by mass: C, 40.00%; H, 6.67%; O, 53.33%.
- Relative molecular mass of 90.0.
- The infrared spectrum below.

IR spectrum of L



#### 4.2.4 Analytical Techniques

Calculate the empirical and molecular formulae of compound **L**.

Show your working.

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[3]

**(b)**. Analyse the information and spectrum to determine the structures of **L** and **M**.

Include an equation for the reaction of **L** to form **M**.

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[5]

## 4.2.4 Analytical Techniques

(c). Determine the structures of compounds **N** and **P**.

Estimate the number of repeat units in polymer **P** and write the equation for the formation of **P** from **N**.

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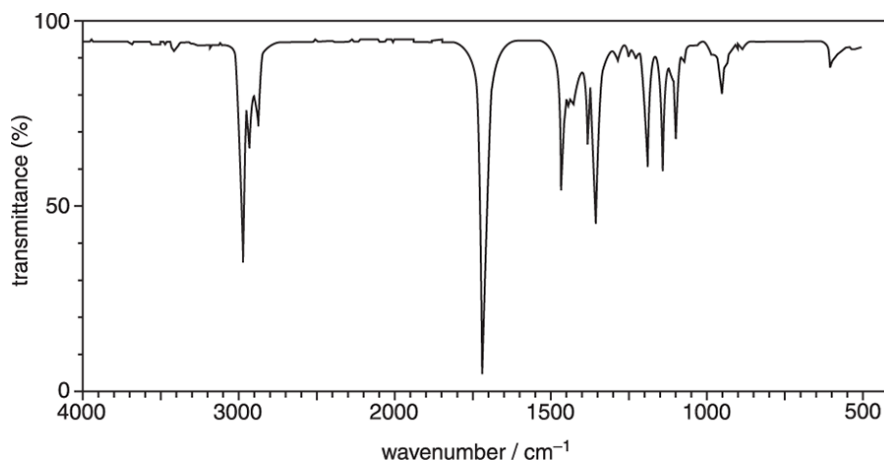
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[4]

12(a). The branched-chain alcohol **J**,  $C_5H_{12}O$ , was heated under reflux with excess  $H_2SO_4 / K_2Cr_2O_7$  to form an organic compound **K** with the infrared spectrum below.

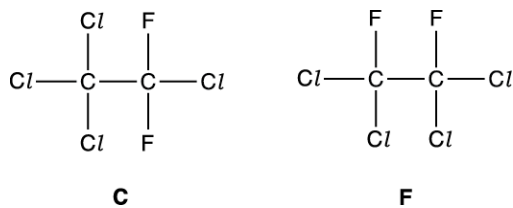


- Determine the structures for the branched-chain alcohol **J** and compound **K**. Your answer should explain all your reasoning using the evidence given.
- Write an equation for the reaction of **J** when heated under reflux with excess  $H_2SO_4 / K_2Cr_2O_7$  to form **K**. Use [O] to represent the oxidising agent.





13. Compounds **C** and **F** can be analysed to obtain infrared and mass spectra.



- i. What happens to molecules when infrared radiation is absorbed?

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----- [1]

- ii. Suggest the molecular formulae of **two** ions responsible for peaks in the mass spectrum of **C** that are **not** in the mass spectrum of **F**.

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----- [2]

14. Give chemical explanations for the following statements.

IR spectroscopy distinguishes ketones from carboxylic acids.

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----- [1]

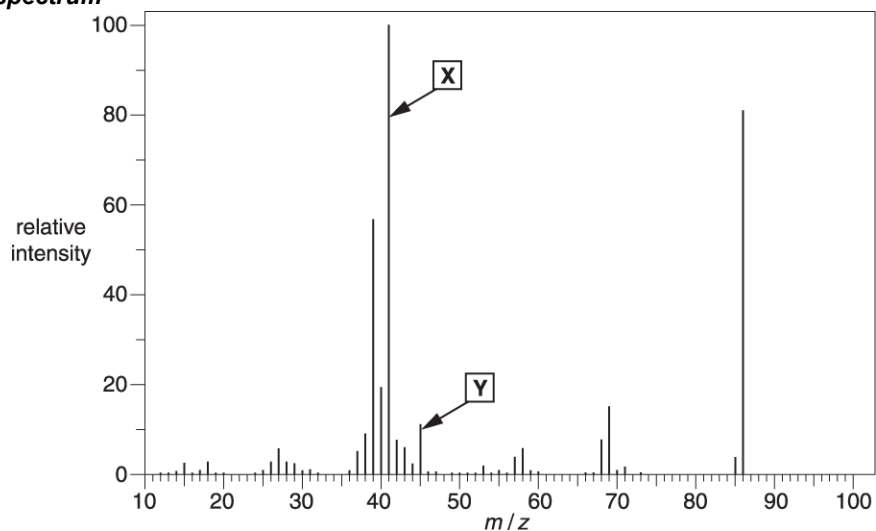
## 4.2.4 Analytical Techniques

15. Compound **G** is a branched-chain organic compound that does **not** have *E* and *Z* isomers.

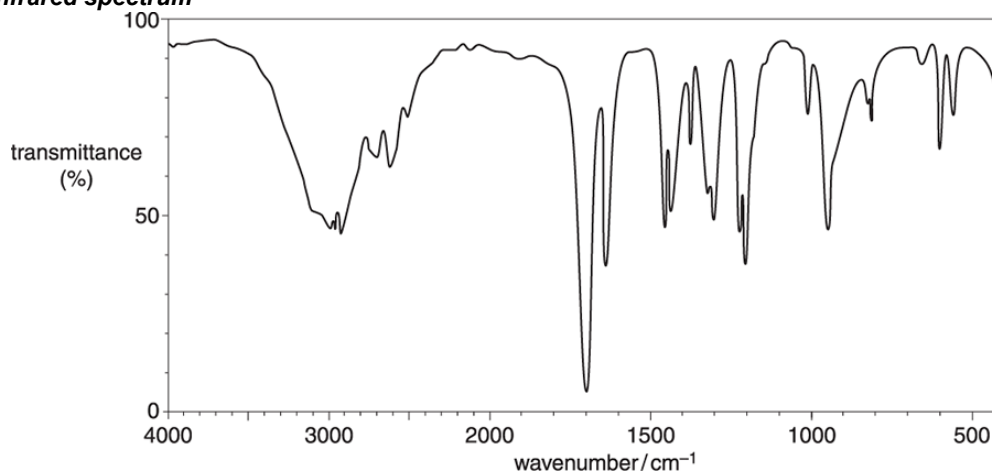
Elemental analysis of compound **G** gave the following percentage composition by mass: C, 55.8%; H, 7.0%; O, 37.2%.

The mass spectrum and infrared spectrum of compound **G** are shown below.

### Mass spectrum



### Infrared spectrum



- Calculate the empirical and molecular formulae for compound **G**.
- Write the formulae for the particles responsible for peak **X** and peak **Y** in the mass spectrum.
- Draw the structure of compound **G**.



*Explain fully how you arrive at a structure for compound **G** using all the evidence provided.*

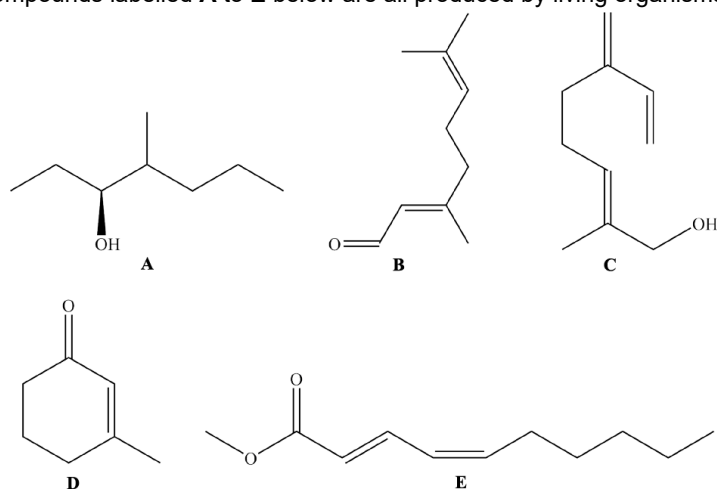
A series of horizontal dashed lines provided for writing the answer.







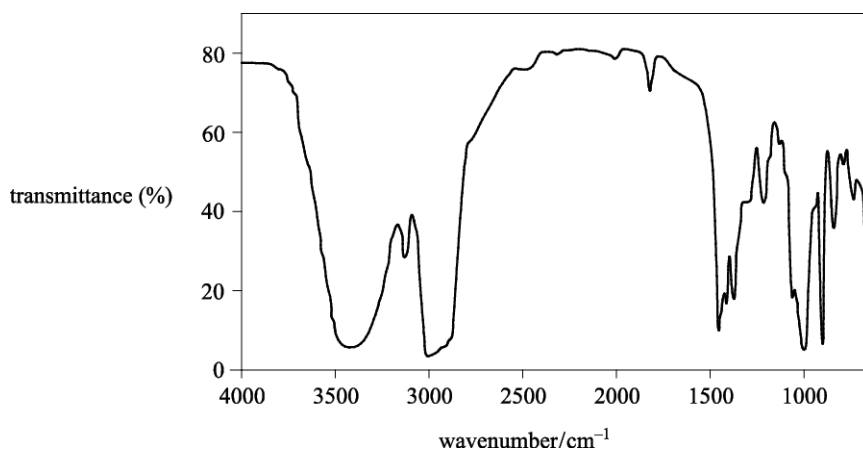
19. The organic compounds labelled **A** to **E** below are all produced by living organisms.



Analysis of one of the compounds **A** to **E** is shown below.

Percentage composition by mass: C, 78.94%; H, 10.53%; O 10.53%

Infrared spectrum:



Use this information to identify the compound.

Explain your reasoning, referring to **all** the evidence provided.

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[3]