

1. Nov/2022/Paper_41/No.9

(a) Gas-liquid chromatography involves a stationary phase and a mobile phase.

(i) Name, or describe in detail, a suitable substance that could be used for each phase.

stationary

mobile

[1]

A mixture of three organic compounds is separated by gas-liquid chromatography. The chromatogram obtained is shown in Fig. 9.1. The amount of each substance is proportional to the area under its peak.

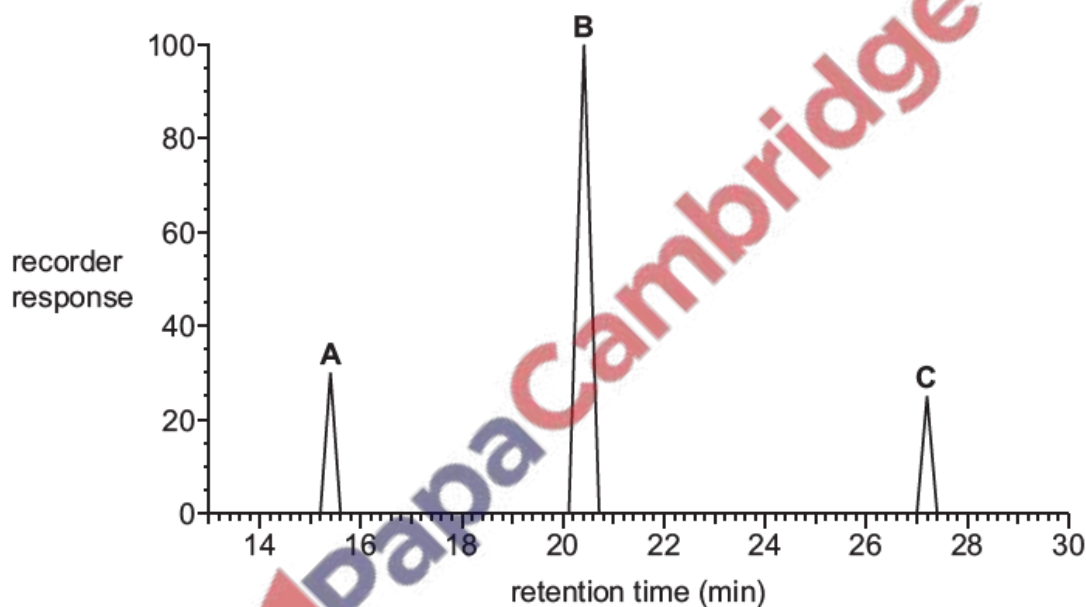


Fig. 9.1

(ii) Explain the meaning of retention time.

.....
..... [1]

(iii) Calculate the percentage of **B** in the mixture. Show your working.

percentage of **B** = % [2]

- (b) Complete Table 9.1 to give the number of peaks in the carbon-13 NMR spectrum of each of the five isomers of $C_5H_{10}O_2$ that has an ester group.

Table 9.1

structural formula	number of peaks
$CH_3CH_2CH_2CO_2CH_3$	
$CH_3CH_2CO_2CH_2CH_3$	
$CH_3CO_2CH_2CH_2CH_3$	
$(CH_3)_2CHCO_2CH_3$	
$CH_3CO_2CH(CH_3)_2$	

[2]

- (c) State the number of peaks that would be seen in the proton (1H) NMR spectrum of methyl butanoate, $CH_3CH_2CH_2CO_2CH_3$. Name all the splitting patterns seen in this spectrum.

number of peaks

splitting patterns

[2]



(d) **D** and **E** are both esters with the molecular formula $C_5H_{10}O_2$. Their proton (1H) NMR spectra are shown in Fig. 9.2 and Fig. 9.3.

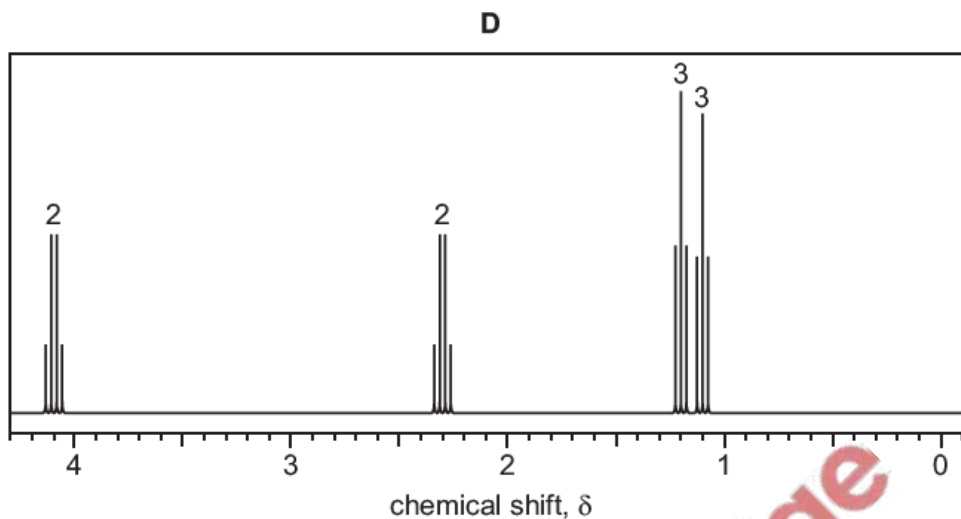


Fig. 9.2

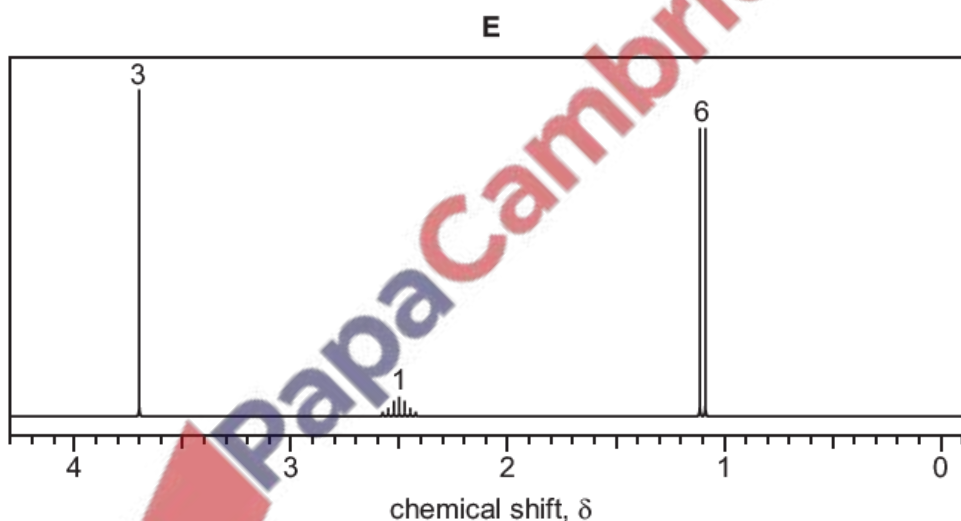
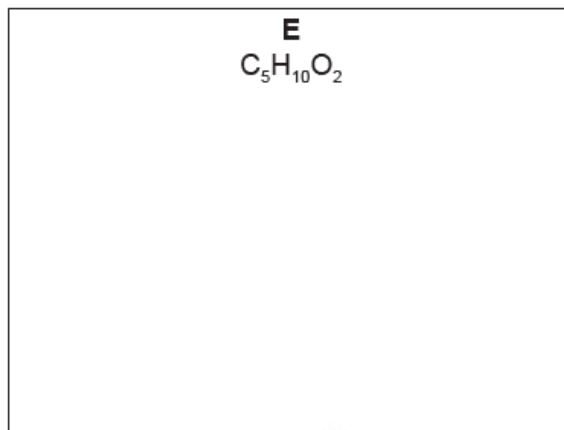
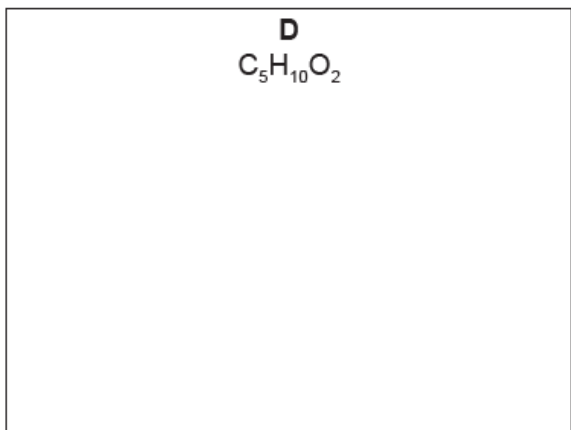


Fig. 9.3

Table 9.2

environment of proton	example	typical chemical shift range, δ /ppm
alkane	$-CH_3$, $-CH_2-$, $>CH-$	0.9–1.7
alkyl next to $C=O$	$CH_3-C=O$, $-CH_2-C=O$, $>CH-C=O$	2.2–3.0
alkyl next to aromatic ring	CH_3-Ar , $-CH_2-Ar$, $>CH-Ar$	2.3–3.0
alkyl next to electronegative atom	CH_3-O , $-CH_2-O$, $-CH_2-Cl$	3.2–4.0
attached to alkene	$=CHR$	4.5–6.0

- (i) Deduce the structures of the two esters **D** and **E** and draw their displayed formulae in the boxes below.



[2]

- (ii) The spectrum of **D** includes a quartet at δ 4.1.

Identify the protons responsible for this quartet on your structure in (i) by labelling these protons with the letter **F**.

Explain why this peak is split into a quartet.

.....
.....

[1]

- (iii) The spectrum of **E** has a doublet at δ 1.1.

Identify the protons responsible for this doublet on your structure in (i) by labelling these protons with the letter **G**.

Explain why this peak has a chemical shift of 1.1.

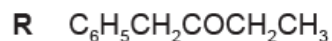
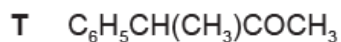
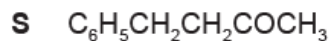
.....
.....

[1]

[Total: 12]

When answering this question it should be assumed that together all the hydrogen atoms in a benzene ring result in a single unsplit peak at $\delta = 7.2$ in a proton (^1H) NMR spectrum.

The structures of five isomeric ketones, **P**, **Q**, **R**, **S** and **T** are given.



(a) Identify all the chiral carbon atoms on the structures above. Label each chiral carbon atom with an asterisk (*). [1]

(b) The proton (^1H) NMR spectrum of one of the five isomers, **P**, **Q**, **R**, **S** or **T**, is shown in Fig. 8.1.

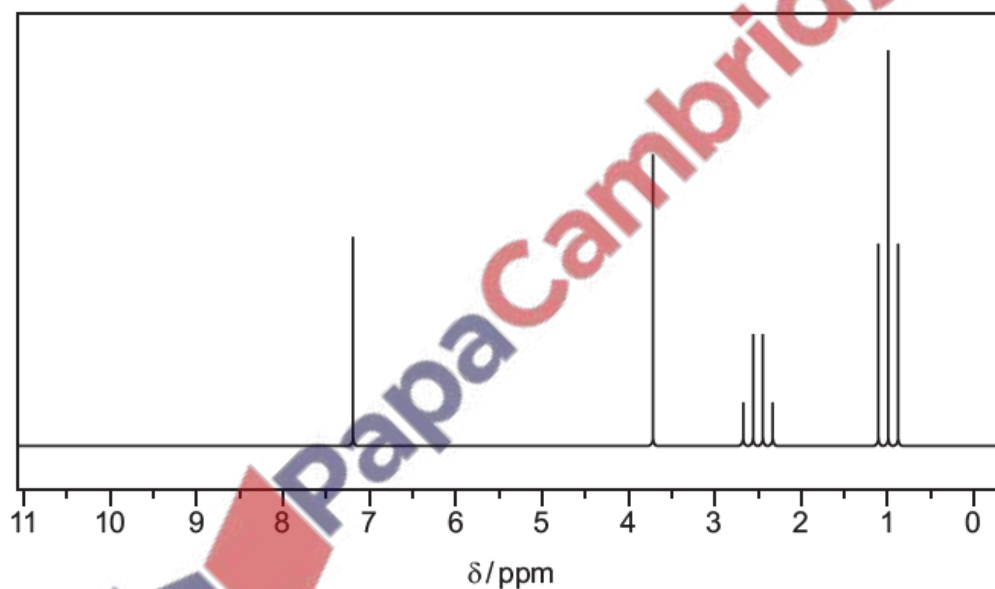


Fig. 8.1

- (i) Identify which of the compounds **P**, **Q**, **R**, **S** or **T** gives this spectrum.
Draw the displayed formula of the compound you have identified. Identify the protons responsible for the peaks at $\delta = 3.7$, $\delta = 2.5$ and $\delta = 1.0$ on the structure you have drawn.

[2]

- (ii) Name the splitting pattern of the peak at $\delta = 3.7$. Explain why it has this splitting pattern.

.....
..... [1]

- (c) Choose from the letters **P**, **Q**, **R**, **S** and **T** to identify:

- (i) the **two** compounds that each have a doublet peak in the proton (^1H) NMR spectrum

..... [1]

- (ii) the compound with only three peaks in its proton (^1H) NMR spectrum.

..... [1]

- (d) Suggest a suitable solvent that should be used for obtaining the spectrum shown in Fig. 8.1.

..... [1]


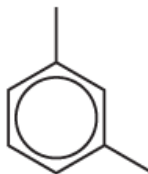
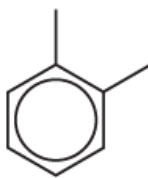
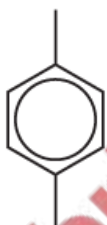
- (e) The proton (^1H) NMR spectrum of compound **T** is compared in the presence of D_2O and in the absence of D_2O .

Describe any difference between the two spectra. Explain your answer.

.....
..... [1]

- (f) Complete Table 8.1 below to give the number of peaks in the carbon-13 NMR spectrum of each compound.

Table 8.1

compound	number of peaks	compound	number of peaks
			
			

[2]

[Total: 10]

