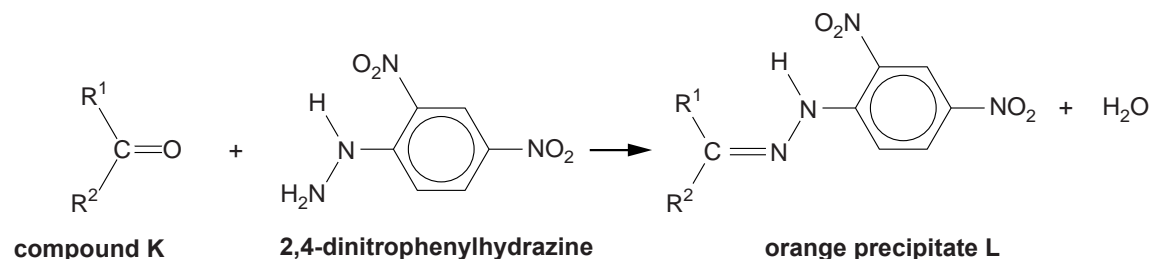


- 1 A student reacts compound **K** with 2,4-dinitrophenylhydrazine. An orange precipitate, **L**, was formed.



The student purifies the orange precipitate, **L**, and sends the sample for analysis using ^1H NMR and ^{13}C NMR spectroscopy.

- (a) Describe a use for NMR spectroscopy in medicine.

.....
 [1]

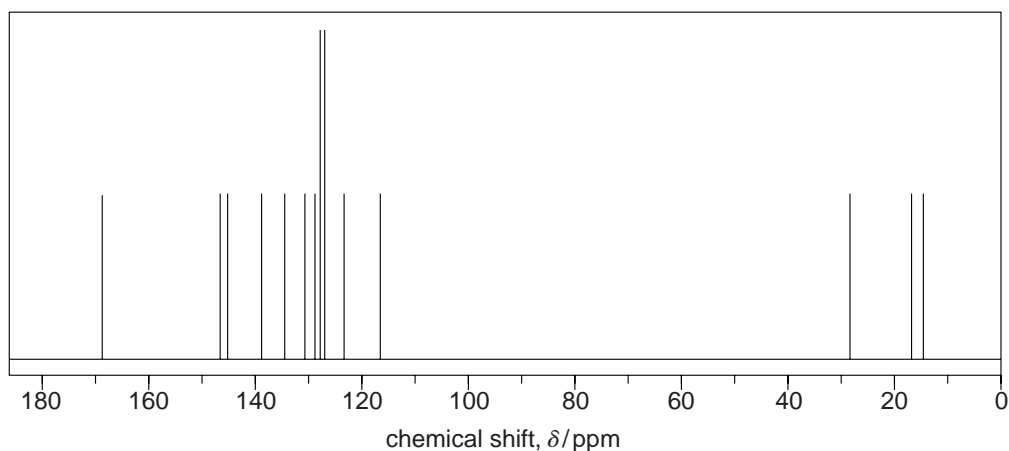
- (b) (i) State the region of the electromagnetic spectrum used in ^1H NMR spectroscopy.

..... [1]

- (ii) Explain why CDCl_3 is used as a solvent in ^1H NMR spectroscopy.

.....
 [1]

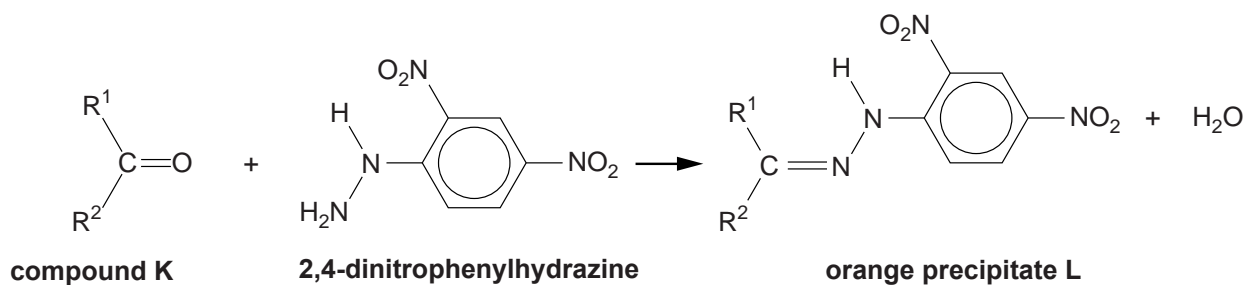
- (c) The ^{13}C NMR spectrum of **L** is shown below.



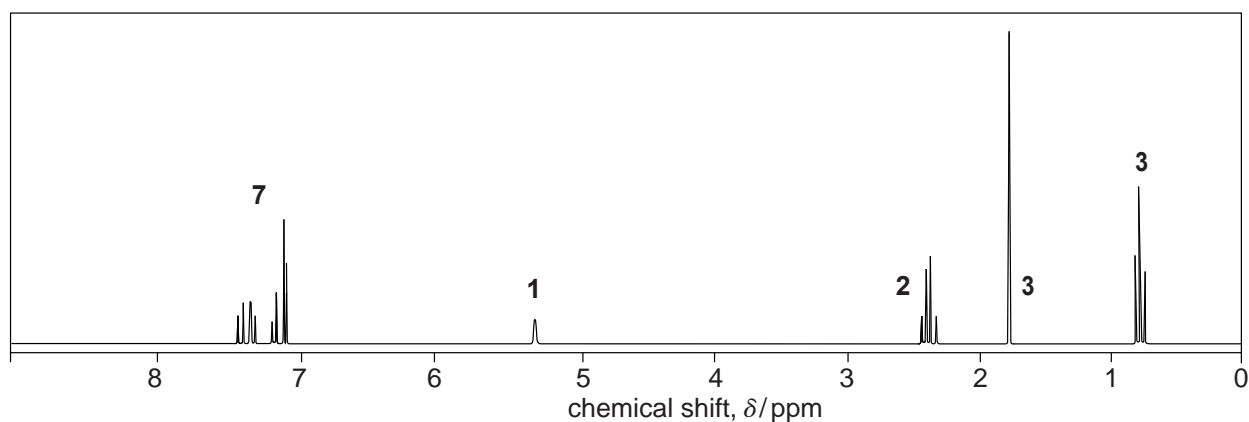
How many different carbon environments (types of carbon) are present in a molecule of compound **L**?

..... [1]

(d) The reaction of **K** to form **L** is repeated below.



The ¹H NMR spectrum of **L** is shown below.



Use your answer to (c) and the data given to identify R¹, R² and the structure of compound **L**.

Explain how you used the chemical shifts and splitting patterns in the ¹H NMR spectrum and any evidence from the ¹³C NMR spectrum.

In the ¹H NMR spectrum, HC–C=N– would have a peak in the range $\delta = 1.6\text{--}2.2$ ppm.



In your answer, you should use appropriate technical terms, spelled correctly.

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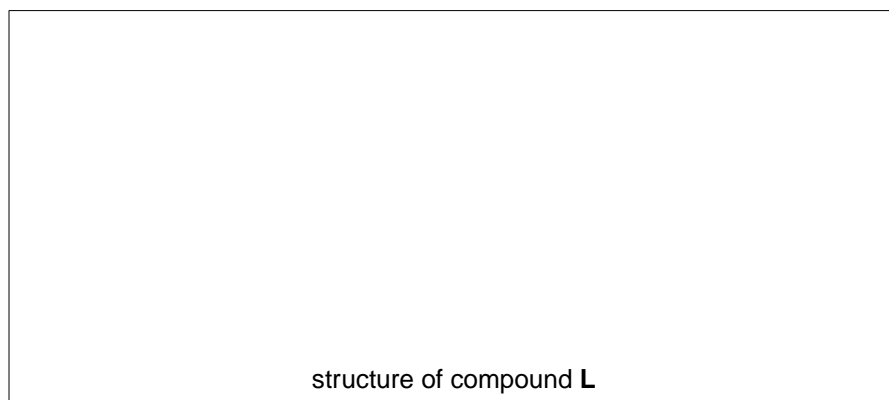
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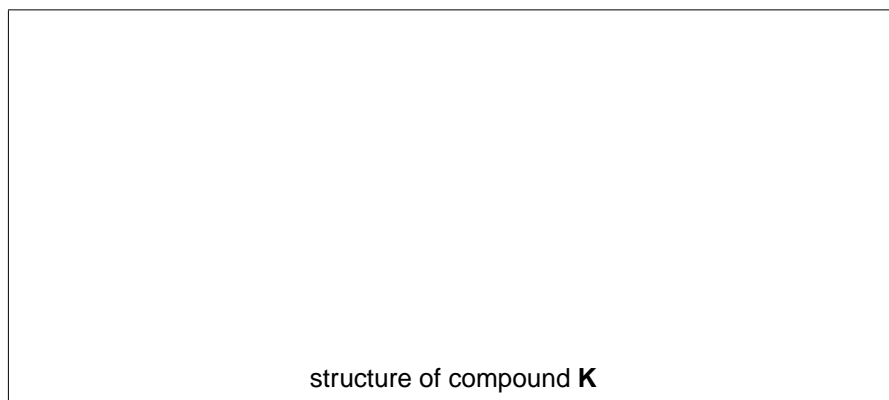
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[7]

(e) Draw the structure of compound **K**.



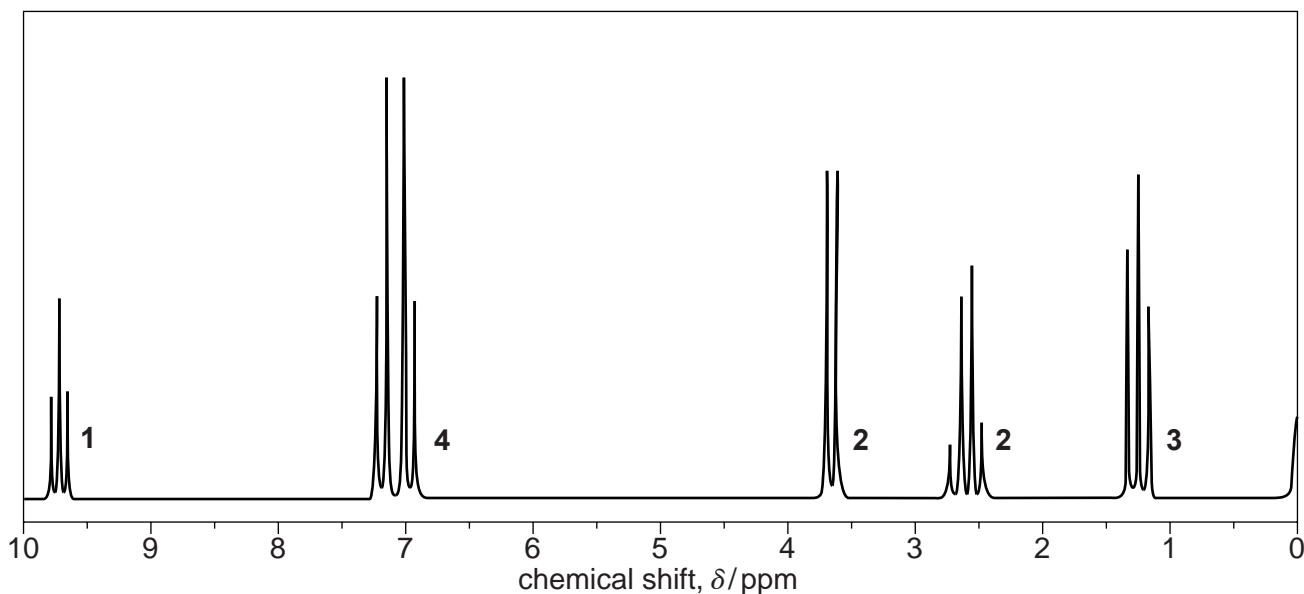
[1]

[Total: 12]

2 A chemist isolates compound **H** from a mixture and sends it for
Initial analysis shows that the ^{analysis}molecular formula of compound **H** is C₁₀H₁₂O.
The ¹³C NMR spectrum of compound **H** contained eight separate peaks.
The ¹H NMR spectrum of compound **H** is shown below.

¹H NMR spectrum

The numbers by each peak are the relative peak areas.



(a) The ¹H NMR spectrum contains a peak at $\delta = 0$ ppm resulting from a chemical added to the sample.

State the chemical responsible for the peak at $\delta = 0$ ppm, and state why this chemical was added to the sample.

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..... [1]

3 A chemist isolated compound **L** from the leaves of a common garden herb. He analysed the compound using a number of techniques.

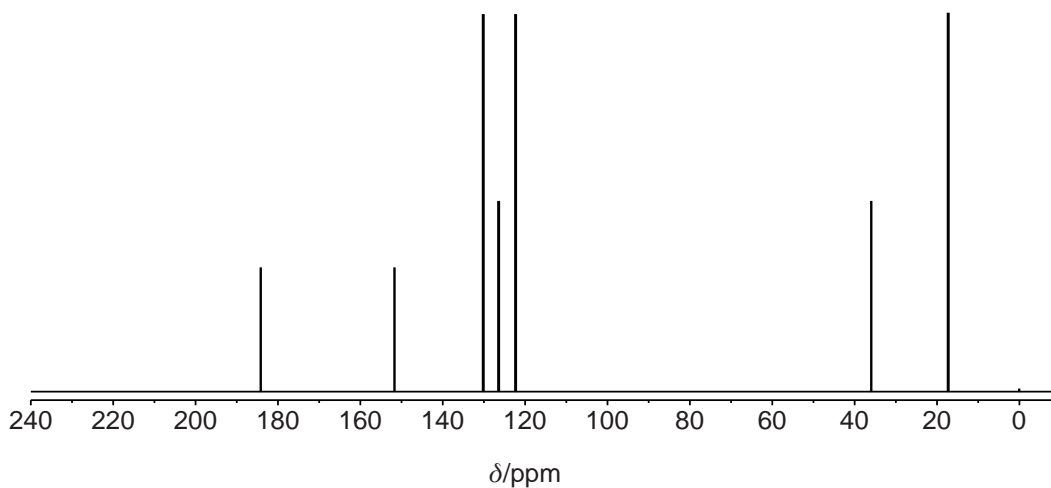
(a) Compound **L** contains C, H and O only. Elemental analysis shows that **L** has the percentage composition by mass: C, 73.15%; H, 7.37%.

The mass spectrum of **L** has a molecular ion peak at $m/z = 164$.

Show that the molecular formula of **L** is $C_{10}H_{12}O_2$.

[2]

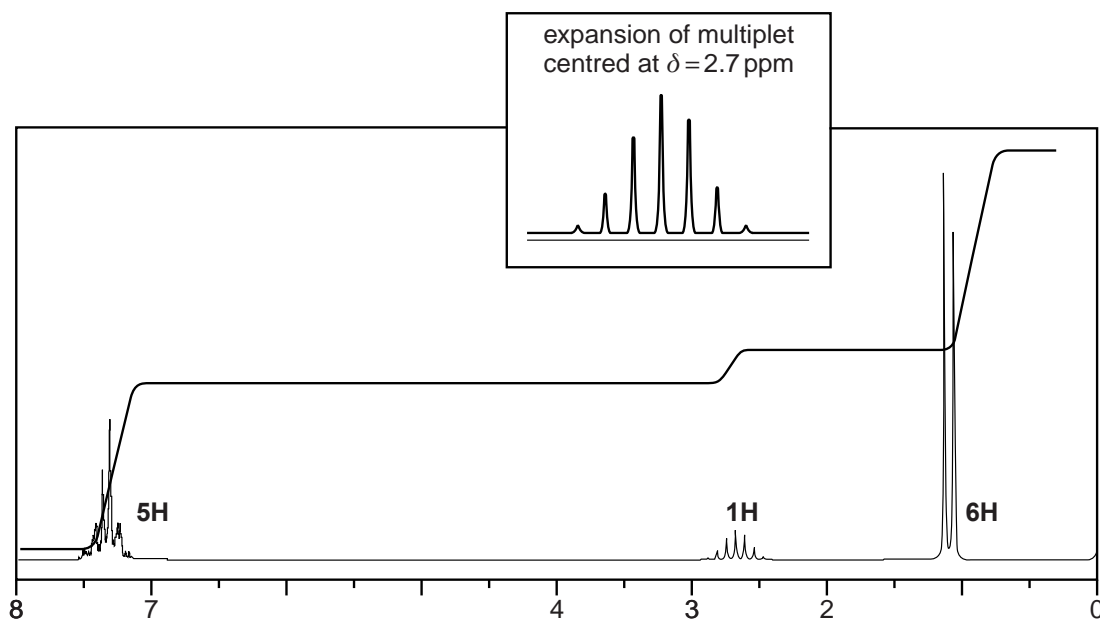
(b) The ^{13}C NMR spectrum of compound **L** is shown below.



How many different carbon environments (types of carbon) are present in a molecule of compound **L**?

..... [1]

(c) The ^1H NMR spectrum of compound **L**, $\text{C}_{10}\text{H}_{12}\text{O}_2$, is shown below. One of the signals has been enlarged to help its analysis.



- (i) A signal at $\delta = 0$ ppm results from tetramethylsilane (TMS). TMS had been added to the sample of compound **L** before the NMR spectrum was run.

State why TMS was added.

..... [1]

- (ii) The ^1H NMR spectrum includes an integration trace.

What information can be deduced about compound **L** from the integration trace?

..... [1]

- (iii) Using the information given in parts (a), (b) and (c) of this question, determine the structure of compound **L**.

Show all your reasoning.



In your answer, you should use appropriate technical terms, spelled correctly.

structure of compound L

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4 A chemist uses gas chromatography, GC, to separate the esters in a mixture. The esters are then analysed using different spectroscopic techniques.

(a) (i) How could the chemist use the results from GC to predict the number of esters in the mixture and their relative proportions?

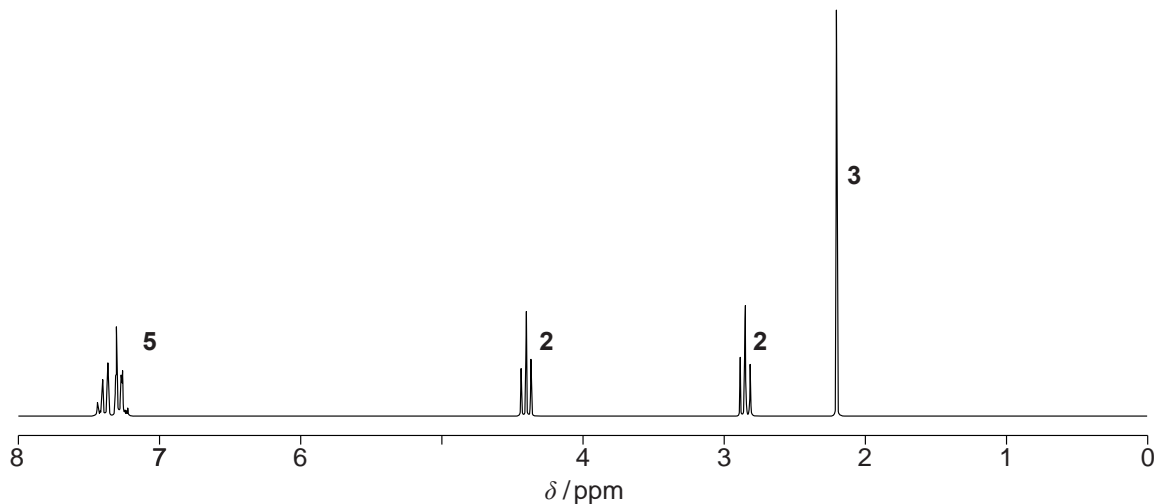
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..... [1]

(ii) Why would there be some uncertainty about using GC alone to predict the number of esters in a mixture?

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

(b) The chemist obtains a mass spectrum and a proton NMR spectrum of one of the esters separated by GC.

- The mass spectrum has a molecular ion peak at $m/z = 164$.
- The proton NMR spectrum is shown below.
The numbers on the NMR spectrum represent the relative peak areas.



Analyse this information to identify the ester.

Include full details of your analysis of the proton NMR spectrum.



In your answer, you should use appropriate technical terms, spelled correctly.

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5 Forest fires release a large number of organic compounds into the atmosphere, many in very small quantities.

(a) Compounds in the smoke from forest fires can be analysed using GC-MS.
Explain how GC-MS enables the compounds to be identified.

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..... [2]

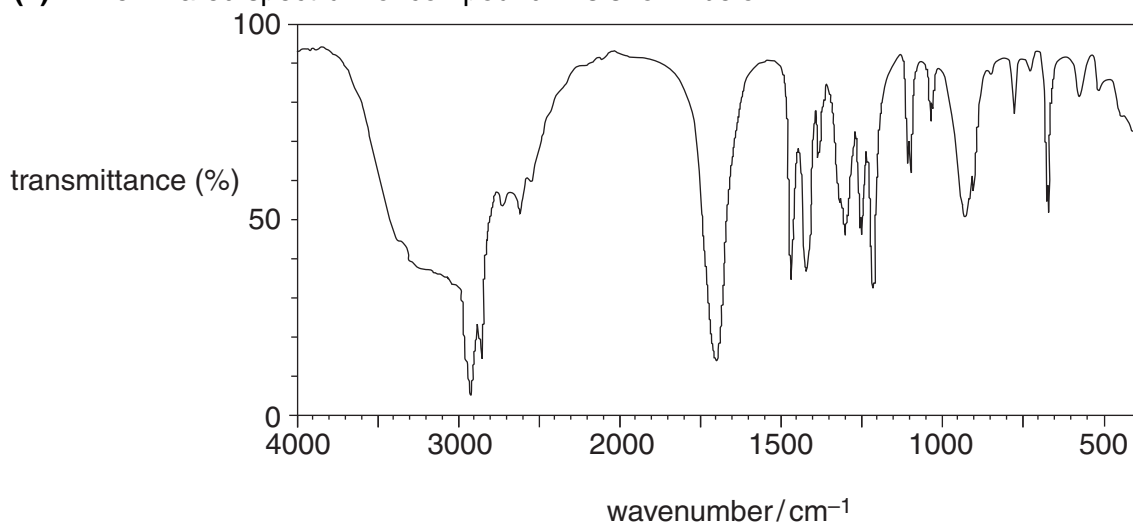
(b) Compound **F** was found to be present in the smoke.
Compound **F** contains C, H and O only and contains 54.2% oxygen by mass.
The molar mass of compound **F** is 118.0 g mol^{-1} .

(i) Using the information, show that the molecular formula of compound **F** is $\text{C}_4\text{H}_6\text{O}_4$.

Show all of your working.

[2]

(ii) The infrared spectrum of compound **F** is shown below.



Using this spectrum, name the functional group present in compound **F**.

..... [1]

(ii) Explain why deuterated dimethylsulfoxide, $(\text{CD}_3)_2\text{SO}$, is used as the solvent rather than $(\text{CH}_3)_2\text{SO}$.

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..... [1]

(iii) State why TMS was added.

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

(iv) A second proton NMR spectrum of compound **F** was obtained after adding a few drops of D_2O .

What difference would you expect to see between the proton NMR spectra of compound **F** obtained with and without D_2O ?

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..... [1]

[Total: 12]