Week 55

A-Level Chemistry

Homework bundle for this week

本周作业合集

exercises.lol

Contents 目录

A-Level Chemistry Name: _____ Score: _____

1. In TLC, the stationary and mobile phases are:
   - ☐ the plate coating (stationary) and the solvent moving up it (mobile)
   - ☐ two gases
   - ☐ two solids
   - ☐ a carrier gas and a liquid
2. In GLC, the mobile phase is:
   - ☐ an unreactive carrier gas
   - ☐ a solid plate
   - ☐ a polar solvent
   - ☐ water
3. In carbon-13 NMR, the number of peaks tells you:
   - ☐ how many different carbon environments there are
   - ☐ the number of hydrogen atoms
   - ☐ the molar mass
   - ☐ the boiling point
4. In proton NMR, the number of groups of peaks tells you:
   - ☐ the number of different proton (hydrogen) environments
   - ☐ the molar mass
   - ☐ the number of carbons
   - ☐ the boiling point
5. The Rf value is:
   - ☐ distance moved by the spot ÷ distance moved by the solvent front
   - ☐ distance moved by the solvent ÷ distance moved by the spot
   - ☐ the retention time
   - ☐ the peak area
6. Equivalent carbons (in the same environment) give a single shared peak.

   ☐ True ☐ False

7. In thin-layer chromatography, the ratio of distances moved by a spot and the solvent is the _____ value.

_____

8. In gas chromatography, the time a component takes to pass through the column is its _____ time.

_____

9. The number of peaks in a carbon-13 NMR spectrum shows the number of different carbon _____.

_____

10. A more soluble component travels further up a TLC plate.

☐ True ☐ False

37. Analytical techniques

Answers · 答案

A-Level Chemistry

1. **the plate coating (stationary) and the solvent moving up it (mobile)**

   The plate coating stays still; the solvent (mobile phase) travels up, carrying the spots.

2. **an unreactive carrier gas**

   A carrier gas moves the components past the liquid stationary phase.

3. **how many different carbon environments there are**

   Each distinct carbon environment gives one peak; equivalent carbons share a peak.

4. **the number of different proton (hydrogen) environments**

   Protons in different environments give peaks at different chemical shifts.

5. **distance moved by the spot ÷ distance moved by the solvent front**

   Rf = spot distance / solvent-front distance; it lies between 0 and 1.

6. **True**

   Carbons in identical environments are not distinguished, so they appear as one peak.

7. **Rf**

   Always between 0 and 1.

8. **retention**

   Used to identify components.

9. **environments**

   Equivalent carbons give one peak.

10. **True**

    It spends more time in the moving solvent.

A-LEVEL CHEMISTRY • EXERCISE SHEET

37.1 Thin-layer chromatography

Name: _____ Class: _____ Date: _____ **Total: 9 marks**

KEY WORDS stationary phase 固定相 • mobile phase 流动相 • thin-layer chromatography 薄层色谱 • chromatography 色谱 • solvent front 溶剂前沿

Objective

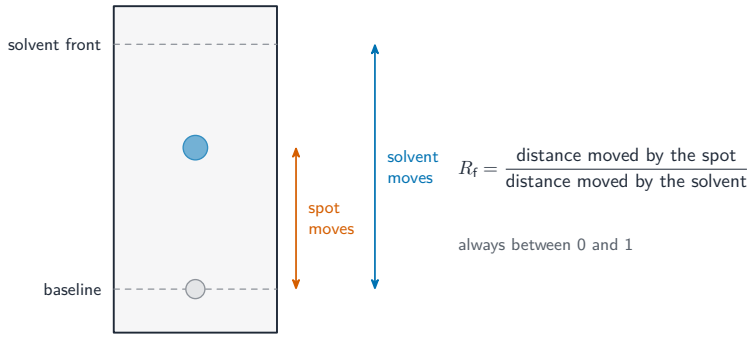
Build the skills to answer exam questions on **thin-layer chromatography** 薄层色谱 (TLC) — the two phases, the R_f **value**, and what it tells you.

You must be able to:

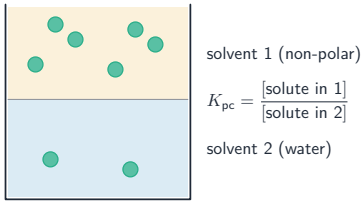
- identify the stationary and mobile phases in TLC
- calculate an R_f value
- explain why one component has a smaller R_f than another

1 Worked examples

■ **The R_f value**



The R_f value is how far the spot moved divided by how far the solvent front moved



TLC separates by partition between phases

$$R_f = \frac{\text{distance moved by the spot}}{\text{distance moved by the solvent front}}$$

- **stationary phase** stays still (e.g. aluminium oxide on a plate); **mobile phase** is the solvent that moves up.
- R_f is always between 0 and 1.
- a substance that sticks more strongly to the stationary phase (or is less soluble in the mobile phase) moves **less** → **smaller** R_f.

2 Practice

2.1 Name the stationary phase commonly used in TLC. [1]

2.2 A spot moves 3.6 cm; the solvent front moves 4.8 cm. Calculate its R_f. [1]

3 Exam-style questions

3.1 An R_f value can never be: [1]

- **A** 0.2
- **B** 0.85
- **C** 1.4
- **D** 0.5

3.2 A component with a **smaller** R_f value: [1]

- **A** is more soluble in the mobile phase
- **B** sticks more strongly to the stationary phase
- **C** moves further up the plate
- **D** has no interaction with the plate

3.3 On a TLC plate, a spot travels 2.1 cm while the solvent front travels 6.0 cm.

(a) Calculate the R_f value. [2]

(b) State what a low R_f suggests about how strongly the component binds to the stationary phase. [1]

3.4 Explain why two components of a mixture separate on a TLC plate. [2]

Solutions

2.1 aluminium oxide (alumina) / silica on a plate.

2.2 $R_f = 3.6/4.8 = 0.75$.

3.1 C. R_f is always between 0 and 1, so 1.4 is impossible.

3.2 B. A smaller R_f means it sticks more strongly to the stationary phase and moves less.

3.3 (a) $R_f = \dfrac{2.1}{6.0} = 0.35$.
(b) it binds (adsorbs) strongly to the stationary phase.

3.4 the components have different affinities for the stationary and mobile phases; so they travel up the plate at different rates and end at different heights, giving different R_f values.

A-LEVEL CHEMISTRY • EXERCISE SHEET

37.2 Gas/liquid chromatography

Name: _____ Class: _____ Date: _____ **Total: 21 marks**

KEY WORDS retention time 保留时间 • mobile phase 流动相 • stationary phase 固定相 • gas/liquid chromatography 气液色谱 • chromatography 色谱

Objective

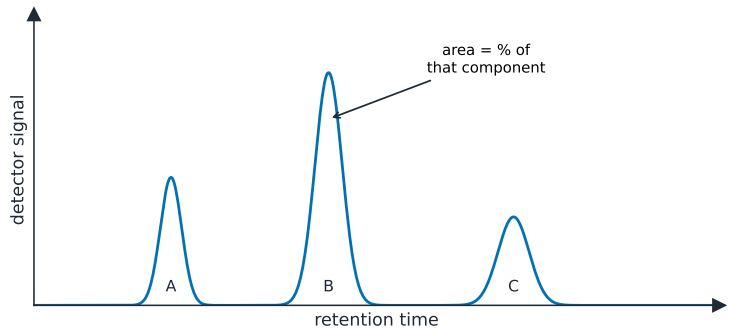
Build the skills to answer exam questions on **gas/liquid chromatography** 气液色谱 (GLC) — the phases, **retention time** 保留时间, and reading peak areas.

You must be able to:

- identify the stationary and mobile phases in GLC
- explain what retention time depends on
- use peak areas to find the composition of a mixture

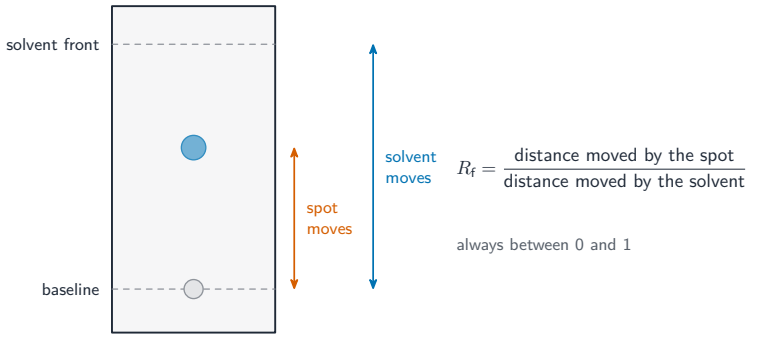
1 Worked examples

Reading a chromatogram



t_R identifies · peak area ≈ amount

Each component gives a peak at its own retention time; the peak area is its percentage in the mixture



GLC alongside TLC as a chromatographic method

- **stationary phase:** a high-boiling non-polar liquid on a solid support; **mobile phase:** an unreactive carrier gas.
- **retention time** = how long a component takes to pass through; more interaction with the stationary phase → **longer** retention time.
- **peak area** = the proportion (percentage) of that component.

2 Practice

2.1 Name the mobile phase in GLC. [1]

2.2 State what the **area** of a GLC peak tells you. [1]

3 Exam-style questions

3.1 In GLC, a component that interacts **more** with the stationary phase has a: [1]

- **A** shorter retention time
- **B** longer retention time
- **C** larger R_f
- **D** smaller peak area

3.2 The **percentage** of each component in a mixture is found from the: [1]

- **A** retention time
- **B** number of peaks
- **C** area under each peak
- **D** height of the baseline

3.3 A GLC trace of a three-component mixture has peak areas 5, 3 and 2 units.

(a) Calculate the percentage of the first component. [2]

(b) State which component left the column **last**, given it interacted most with the stationary phase. [1]

3.4 Explain why different components of a mixture have different retention times. [2]

3.5 A mixture of three alcohols is analysed by gas/liquid chromatography. The chromatogram shows three peaks.

Peak	Retention time / min	Peak area / arbitrary units
1	1.8	132
2	3.4	396
3	5.9	132

(a) Name the mobile phase and the stationary phase in GLC, and state what each does. [3]

(b) Calculate the percentage composition of the mixture, stating the assumption you are making. [3]

(c) Explain why the component of peak 3 has the longest retention time. [3]

(d) The three peaks alone cannot identify the alcohols. Explain why, and name one technique that can be coupled to GLC to solve this. [3]

Solutions

2.1 an unreactive carrier gas.

2.2 the percentage (proportion) of that component in the mixture.

3.1 B. More interaction with the stationary phase → longer retention time.

3.2 C. The area under each peak gives the percentage.

3.3 (a) total area $= 5 + 3 + 2 = 10$; first component $= \frac{5}{10} \times 100 = 50\%$.
(b) the component with the **longest** retention time / smallest R_f-type behaviour — the one that interacts most strongly.

3.4 each component interacts to a different extent with the stationary phase; the more it interacts, the more it is held back, so it takes longer to pass through.

3.5 (a) The mobile phase is an **inert carrier gas** such as nitrogen or helium, which sweeps the vaporised sample through the column; the stationary phase is a high-boiling **liquid** coated on an inert solid support; components separate because each spends a different fraction of its time dissolved in the liquid rather than moving with the gas.
(b) Total area $= 132+396+132 = 660$; peak $1 = \frac{132}{660} = 20\%$, peak $2 = \frac{396}{660} = 60\%$, peak $3 = 20\%$. The assumption is that the **detector responds equally** to each component, so peak area is proportional to the amount present.
(c) That component is the most soluble in the liquid stationary phase, or has the strongest intermolecular attraction to it; it therefore spends the greatest fraction of its time dissolved and the least moving with the carrier gas; so it takes longest to reach the detector.
(d) A retention time is only characteristic **under identical conditions** — column, temperature and flow rate — and two different compounds can share one, so a peak alone names nothing. Coupling the column to a **mass spectrometer** (GC–MS) gives each peak a fragmentation pattern that identifies it.

6 **(a)** Thin-layer and gas/liquid chromatography can be used to separate mixtures into their individual components.

(i) Define the following terms used in chromatography.

R_f value .....

.....

retention time .....

.....

[2]

(ii) Each type of chromatography makes use of a stationary phase and a mobile phase.

Complete Table 6.1 with a description of each of these.

Table 6.1

	stationary phase	mobile phase
thin-layer chromatography		
gas/liquid chromatography		

[1]

(b) A mixture of two substances **A** and **B** is analysed by thin-layer chromatography.

The R_f value of substance **A** is larger than that of substance **B**.

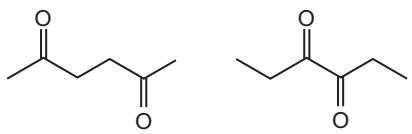
Suggest why substance **A** has a larger R_f value.

.....

..... [1]

(c) The two isomeric compounds **Y** and **Z** are analysed by proton (^{1}H) NMR spectroscopy.

Y **Z**



(i) Complete Table 6.2 to predict the number of peaks observed in the proton (^{1}H) NMR spectra for **Y** and **Z**.

Table 6.2

compound	number of peaks observed
Y	
Z	

[1]

(ii) Name **all** the different splitting patterns observed in the proton (^{1}H) NMR spectra for **Y** and **Z**.

Y .....

Z .....

[2]

[Total: 7]

A-LEVEL CHEMISTRY • EXERCISE SHEET

37.3 Carbon-13 NMR spectroscopy

Name: _____ Class: _____ Date: _____ **Total: 9 marks**

KEY WORDS carbon-13 nmr 碳 • nmr 核磁共振 • chemical shift 化学位移 • chemical environment 化学环境

Objective

Build the skills to answer exam questions on **carbon-13 NMR** 碳-13 核磁共振 — using the number and position of peaks to count carbon **chemical environments** 化学环境 and suggest structures.

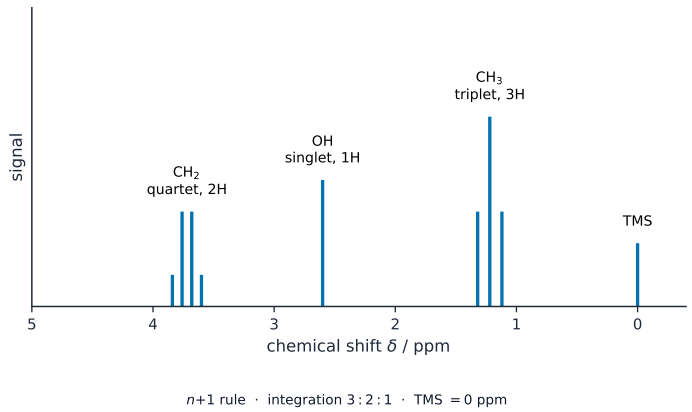
You must be able to:

- state what the number of peaks in a ^{13}C NMR spectrum tells you
- use chemical shift to suggest the type of carbon
- deduce possible structures from a ^{13}C spectrum

1 Worked examples

Counting carbon environments

NMR uses a very strong magnetic field (from a superconducting magnet) to study how nuclei behave



An NMR spectrum from a carbon environment count

- in ^{13}C NMR, each different **carbon environment** gives **one peak** (equivalent carbons share a peak).
- **number of peaks** = number of different carbon environments.
- **chemical shift** (peak position) suggests the type of carbon (e.g. C=O is far downfield).

Example: ethanol CH_3CH_2OH has 2 carbon environments → **2 peaks**.

2 Practice

2.1 State what the number of peaks in a ^{13}C NMR spectrum tells you. [1]

2.2 State how many peaks propanone, CH_3COCH_3, gives in its ^{13}C spectrum. [1]

3 Exam-style questions

3.1 In ^{13}C NMR, two carbon atoms in the **same** environment give: [1]

- **A** two separate peaks
- **B** a single peak
- **C** no peak
- **D** a split peak

3.2 How many peaks does propan-1-ol, $CH_3CH_2CH_2OH$, give in its ^{13}C spectrum? [1]

- **A** 1
- **B** 2
- **C** 3
- **D** 4

3.3 A compound C_3H_6O gives **two** peaks in its ^{13}C NMR spectrum, one far downfield (a C=O).

(a) Deduce a possible structure. [2]

(b) Explain how the number of peaks supports your structure. [1]

3.4 Explain why ethanoic acid, CH_3COOH, gives **two** peaks in its ^{13}C NMR spectrum. [2]

Solutions

2.1 the number of different carbon (chemical) environments in the molecule.

2.2 2 peaks (the two CH_3 carbons are equivalent; the C=O is the second).

3.1 B. Equivalent carbons in the same environment give a single peak.

3.2 C. Propan-1-ol has three different carbon environments → 3 peaks.

3.3 (a) propanone, CH_3COCH_3 — the C=O gives the downfield peak and the two equivalent CH_3 give the other.
(b) the two equivalent methyl carbons share one peak and the C=O gives the second, so two peaks fit.

3.4 ethanoic acid has two different carbon environments — the CH_3 carbon and the COOH carbon; each gives one peak, so there are two peaks.

37.4 Proton (1H) NMR spectroscopy

Name: _____ Class: _____ Date: _____ **Total: 28 marks**

KEY WORDS splitting 裂分 • tetramethylsilane 四甲基硅烷 • chemical shift 化学位移 • chemical environment 化学环境 • proton 质子

Objective

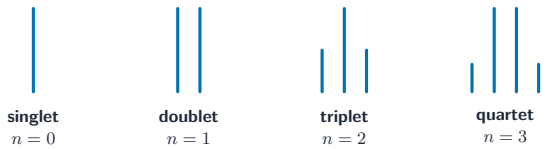
Build the skills to answer exam questions on **proton (^{1}H) NMR** 质子核磁共振 — chemical environments, peak **area** ratios, **splitting** 裂分 by the $n+1$ rule, and the use of TMS and D_2O.

You must be able to:

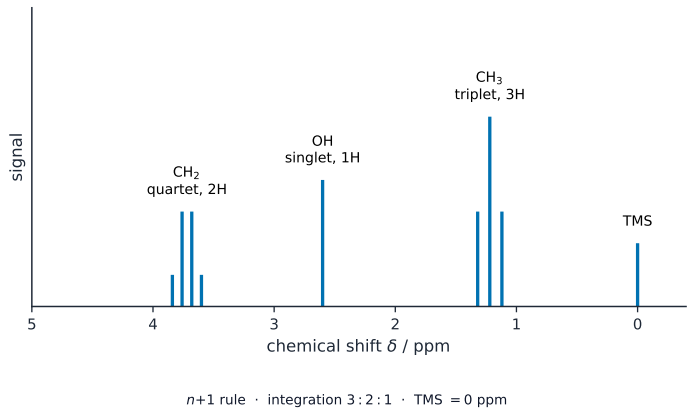
- count proton environments and use area ratios
- apply the $n+1$ rule to predict splitting patterns
- explain the roles of TMS, a deuterated solvent and D_2O

1 Worked examples

Splitting and areas



The $n+1$ rule: n equivalent neighbouring protons split a peak into $n+1$ lines



Ethanol: three environments give a CH_3 triplet, a CH_2 quartet and an OH singlet, areas in ratio 3:2:1

- **number of peaks** = number of proton environments; **area ratio** = ratio of protons.
- $n+1$ **rule:** n equivalent neighbours → $n+1$ lines (0→singlet, 1→doublet, 2→triplet, 3→quartet).
- **TMS** sets the shift = 0; a **deuterated solvent** ($CDCl_3$) gives no signal; shaking with **D_2O** removes O–H/N–H peaks.

■ Reading a proton NMR spectrum, in four passes

Work through the spectrum in this order; each pass answers a different question and each carries its own marks.

1. **Number of signals = number of chemical environments.** Protons are equivalent if swapping them leaves the molecule identical. Ethanol, CH_3CH_2OH, has three environments, so three signals.
2. **Area (integration) ratio = ratio of the numbers of protons.** Ethanol integrates 3 : 2 : 1.
3. **Chemical shift δ says which environment**, read off the data booklet. Roughly: CH_3 next to carbon 0.9; next to C = O 2.2; CH next to O 3.5; aromatic H 7.0; aldehyde H 9.5; carboxylic acid H 11.5.
4. **Splitting = the $n+1$ rule.** A signal is split into $n+1$ peaks by n protons on the **adjacent** carbon: 0 neighbours → singlet, 1 → doublet, 2 → triplet, 3 → quartet.

Two things the exam tests every session.

- **–OH and –NH protons do not split and are not split**, because they exchange rapidly. They give a broad singlet at a variable shift.

- **Adding D_2O makes that peak disappear**, because the labile H is exchanged for D, which does not appear in a ^1H spectrum. That is how an –OH or –NH is identified, and it is a favourite one-mark question.

Worked example. A compound $C_3H_6O_2$ gives three signals: δ 11.5 (1H, singlet, lost on shaking with D_2O), δ 2.4 (2H, quartet), δ 1.1 (3H, triplet). Deduce its structure.

1. The δ 11.5 singlet lost with D_2O is a carboxylic acid –OH.
2. The 3H triplet at δ 1.1 is a CH_3 with two neighbours, so CH_3CH_2–.
3. The 2H quartet at δ 2.4 is that CH_2, with three neighbours, next to the C = O.
4. Assembling: CH_3CH_2COOH, propanoic acid, which matches $C_3H_6O_2$.

The solvent is usually $CDCl_3$ or another deuterated solvent, chosen because it contains **no ^1H** and so adds no signals of its own. **TMS** is the reference at $\delta = 0$: inert, volatile, and its twelve equivalent protons give one strong sharp peak away from everything else.

2 Practice

2.1 State what the relative **areas** of ^1H NMR peaks tell you. [1]

2.2 Using the $n+1$ rule, state the splitting of a CH_3 next to a CH_2. [1]

2.3 State why a deuterated solvent is used, and name the compound used as the $\delta = 0$ reference. [2]

2.4 Explain what happens to the spectrum of ethanol when a few drops of D_2O are added. [2]

2.5 Give the splitting pattern of the CH_2 signal in CH_3CH_2Br, with your reasoning. [2]

3 Exam-style questions

3.1 A proton with **two** equivalent neighbours appears as a: [1]

- **A** singlet
- **B** doublet
- **C** triplet
- **D** quartet

3.2 Shaking a sample with D_2O causes a peak to **disappear**. This peak is due to: [1]

- **A** a CH_3 group
- **B** an O–H or N–H proton
- **C** a CH_2 group
- **D** the TMS reference

3.3 The ^{1}H NMR spectrum of ethanol, CH_3CH_2OH, has three groups of peaks.

(a) Give the splitting pattern of the CH_3 and the CH_2 peaks, with reasons. [2]

(b) State the ratio of the peak areas. [1]

3.4 A compound has two ^{1}H environments with area ratio 3:2; the larger peak is a triplet and the smaller a quartet. Deduce the two groups present and explain the splitting. [3]

3.5 Compound X has the molecular formula $C_4H_8O_2$. Its proton NMR spectrum shows four signals.

δ	**relative area**	**splitting**
4.1	2	quartet
2.0	3	singlet
1.3	3	triplet

(a) State what the number of signals tells you about compound X. [1]

(b) Explain what the relative areas represent, and give the total number of hydrogen atoms they account for. [2]

(c) Use the splitting patterns to deduce which groups are adjacent to each other. [3]

(d) Deduce the structure of X and name it. [3]

(e) A different compound Y, also $C_4H_8O_2$, gives a broad singlet at δ 11.4 that disappears when D_2O is added. Identify the group responsible and explain why the peak disappears. [3]

Solutions

2.1 the ratio of the numbers of protons in each environment.

2.2 a triplet ($n = 2$ neighbours $\rightarrow 2 + 1 = 3$ lines).

2.3 A deuterated solvent contains no ^{1}H, so it adds no signals of its own to the spectrum; the reference is tetramethylsilane, TMS.

2.4 The –OH peak disappears; the labile hydrogen is exchanged for deuterium, which does not appear in a ^{1}H spectrum.

2.5 A quartet; the adjacent CH_3 has three protons, and $n + 1 = 4$.

3.1 C. Two equivalent neighbours give $2 + 1 = 3$ lines — a triplet.

3.2 B. D_2O exchange removes the O–H / N–H peak.

3.3 (a) the CH_3 is a triplet (its 2 CH_2 neighbours give $2 + 1 = 3$); the CH_2 is a quartet (its 3 CH_3 neighbours give $3 + 1 = 4$).
(b) $CH_3 : CH_2 : OH = 3 : 2 : 1$.

3.4 the triplet (area 3) is a CH_3 with 2 neighbours, and the quartet (area 2) is a CH_2 with 3 neighbours; so the groups are CH_3 and CH_2 next to each other, i.e. an ethyl group; each splits the other by the $n + 1$ rule.

3.5 (a) X has three chemical environments for its protons — one for each signal.
(b) The areas are in the ratio of the numbers of protons in each environment; $2+3+3 = 8$ hydrogen atoms, which matches $C_4H_8O_2$.
(c) The quartet at δ 4.1 has three neighbours, so it is a CH_2 next to a CH_3; the triplet at δ 1.3 has two neighbours, so it is that CH_3 next to the CH_2; the singlet at δ 2.0 has no neighbours, so its CH_3 is attached to a carbon carrying no hydrogens, the C = O.
(d) The δ 4.1 shift shows the CH_2 is attached to oxygen; so X is $CH_3COOCH_2CH_3$, ethyl ethanoate.
(e) A carboxylic acid –OH; the labile proton exchanges rapidly with deuterium from the D_2O, and deuterium gives no signal in a proton NMR spectrum, so the peak vanishes.

7 **P**, **Q**, **R**, **S**, **T**, **U**, and **V** are the seven structural isomers with molecular formula $C_5H_{10}O$ that have a carbonyl group.

P $CH_3(CH_2)_3CHO$ **Q** $CH_3CH_2CH(CH_3)CHO$ **R** $(CH_3)_2CHCH_2CHO$

S $(CH_3)_3CCHO$ **T** $CH_3CH_2CH_2COCH_3$ **U** $CH_3CH_2COCH_2CH_3$

V $(CH_3)_2CHCOCH_3$

(a) Only one of these seven compounds has stereoisomers.

Draw three-dimensional diagrams of the **two** stereoisomers of this compound.

[2]

(b) **P**, **Q**, **R**, **S**, **T**, **U**, and **V** are treated separately with alkaline I_2(aq) and the product mixture is acidified.

(i) Identify the **two** compounds that give a positive result with alkaline I_2(aq).

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

(ii) Describe the observations when **one** of the compounds you have identified in **(b)(i)** is treated with alkaline I_2(aq) and give the structural formulae of the **two** carbon-containing products of this reaction.

observations .....

two carbon-containing products .....

and .....

[2]

P $CH_3(CH_2)_3CHO$ **Q** $CH_3CH_2CH(CH_3)CHO$ **R** $(CH_3)_2CHCH_2CHO$

S $(CH_3)_3CCHO$ **T** $CH_3CH_2CH_2COCH_3$ **U** $CH_3CH_2COCH_2CH_3$

V $(CH_3)_2CHCOCH_3$

(c) The proton (^1H) NMR spectra of **P**, **Q**, **R**, **S**, **T**, **U**, and **V** are compared.

(i) Identify the only compound that gives a spectrum with two singlets and no other peaks.

..... [1]

Fig. 7.1 shows the spectrum obtained from one of the compounds.

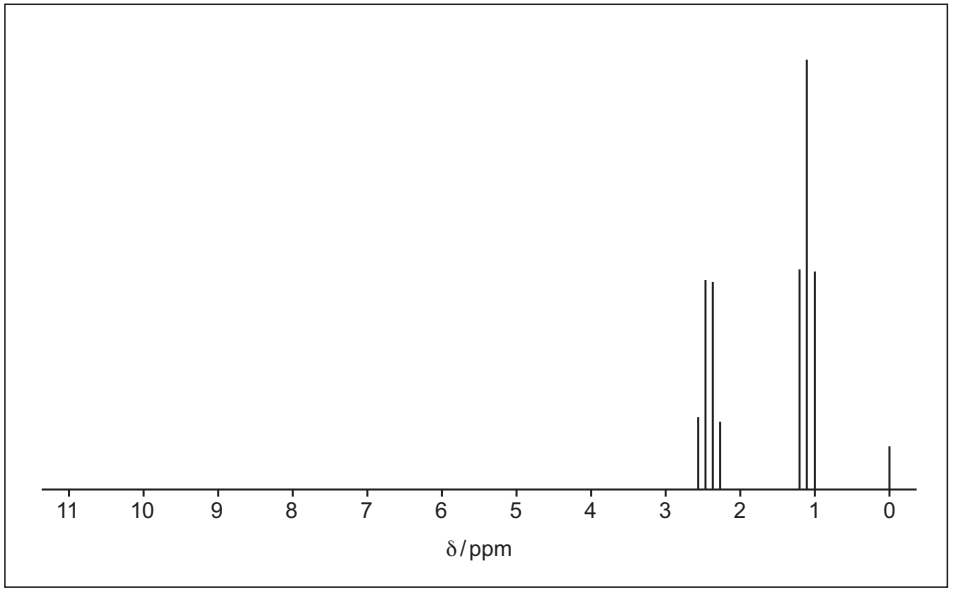


Fig. 7.1

(ii) Identify the compound that gives this spectrum.

..... [1]

(iii) Name the splitting pattern of the peak at δ = 1.1 in Fig. 7.1.

Give the reason for this splitting.

name .....

reason .....
[1]

(iv) Identify the substance that gives the small peak at δ = 0 in Fig. 7.1.

..... [1]

(d) The carbon-13 NMR spectra of **R**, **S**, **T** and **U** are compared.

Complete Table 7.1 to state the number of peaks in the spectrum of each compound.

Table 7.1

compound		number of peaks
R	$(CH_3)_2CHCH_2CHO$	
S	$(CH_3)_3CCHO$	
T	$CH_3CH_2CH_2COCH_3$	
U	$CH_3CH_2COCH_2CH_3$	

[2]

[Total: 11]