

# Introduction to IR Spectroscopy

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A Level



IR (infra-red) spectroscopy is a useful tool both for detecting previously studied substances in a sample, and for identifying the presence of functional groups in a compound.

## Part A Compound detection

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When detecting the presence of a particular compound, we compare the details of absorptions falling in the region below  $1500\text{ cm}^{-1}$  to reference spectra. This part of the spectrum often contains a great number of absorptions corresponding to different vibrational modes of the molecule, and is called the ...

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## Part B Bond identification

Another way of using IR spectroscopy is to identify the presence of particular functional groups. While the absorptions seen correspond to vibrational modes of the whole molecule, we often instead think of the vibrations as occurring in isolated parts of the molecule. Absorptions that occur at wavenumbers above  $1500\text{ cm}^{-1}$  can allow us to identify characteristic functional groups, as there is much less overlap between absorptions in this region of the spectrum, and it is only stretching modes that appear here. The stretching frequency of a bond depends both on the mass of the atoms and the strength of the bond in question.

We see absorptions due to the stretches of  bonds to hydrogen above or around  $3000\text{ cm}^{-1}$ . This is because hydrogen is a very  element, and the stretching frequency of its bonds is therefore much higher than that of other  bonds, which usually appear below  $1500\text{ cm}^{-1}$ . We see  bonds typical in organic chemistry (e.g. in alkenes or carbonyls) correspond to stretching frequencies between  $1500\text{ cm}^{-1}$  and  $2000\text{ cm}^{-1}$ , while the even   bonds typically have stretching frequencies between  $2000\text{ cm}^{-1}$  and  $2500\text{ cm}^{-1}$ .

Items:

heavy

stronger

triple

double

light

single

weaker

hydrogen



# IR Isomers

A Level



The two spectra **P** and **Q** below were obtained from compounds with the formula  $C_4H_{10}O$ .

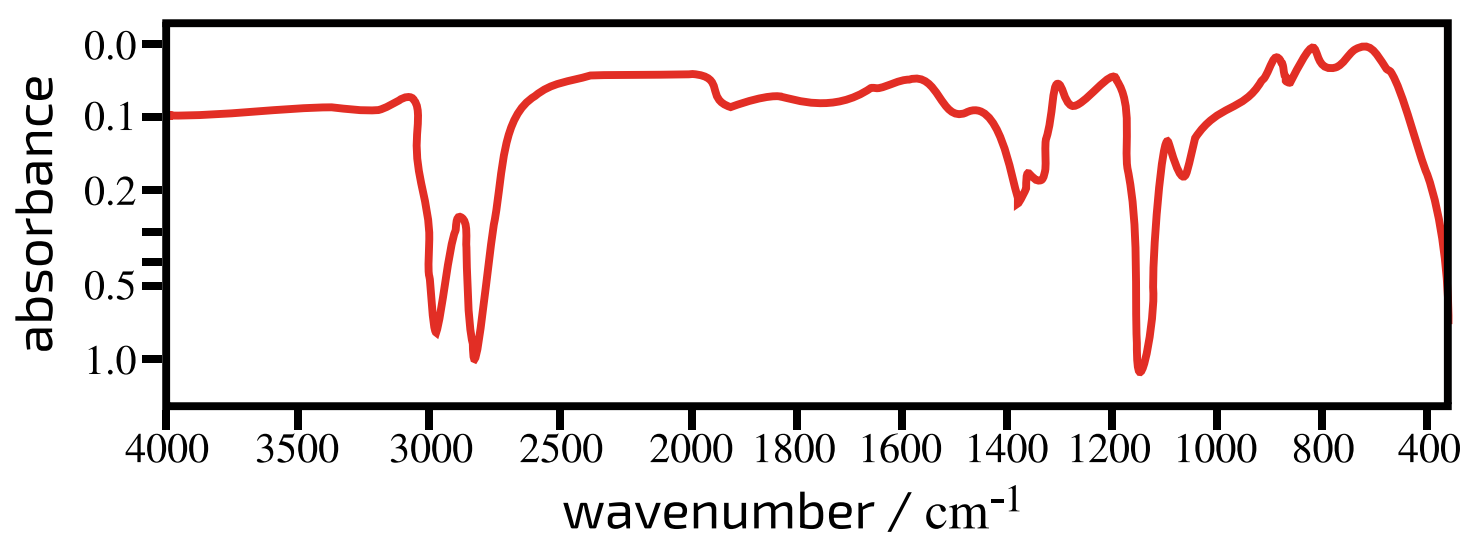


Figure 1: Infrared spectrum **P**

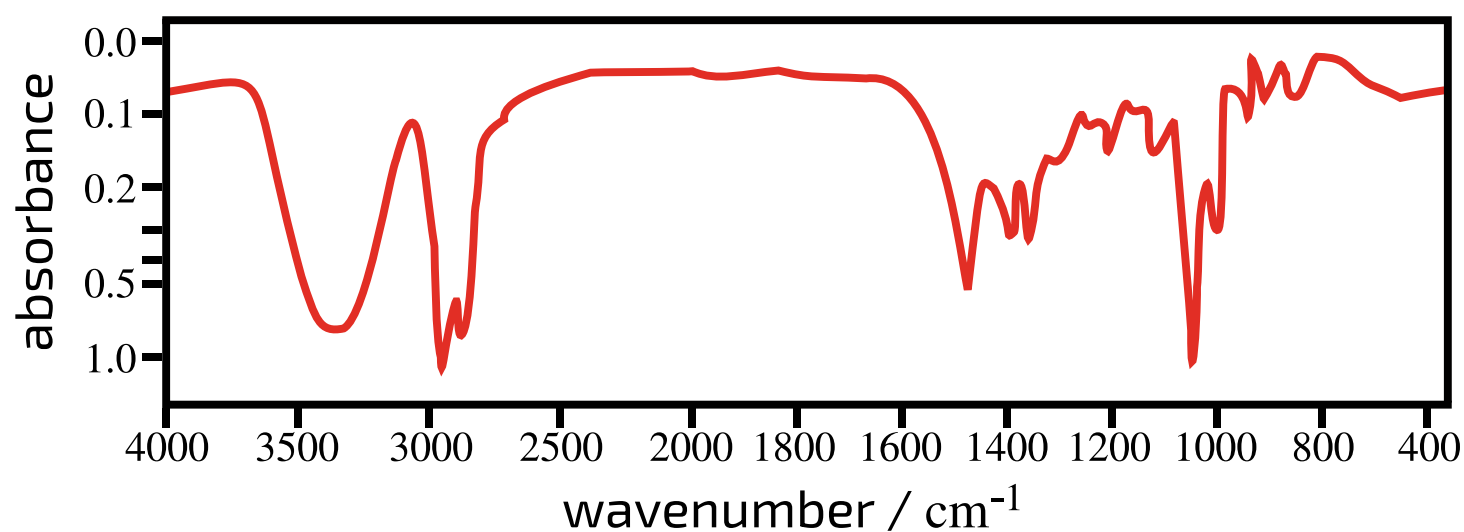


Figure 2: Infrared spectrum **Q**

## Part A Molecular formula

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Which of the following statement(s) is/are true for a compound with molecular formula  $C_4H_{10}O$ ?

- ☐ It cannot contain a double bond.
  - ☐ It cannot contain a ring.
  - ☐ It has to contain a double bond.
  - ☐ It has to contain a ring.
  - ☐ It has to contain either a double bond or a ring.
  - ☐ It may contain a carbonyl group ( $C=O$ ).
  - ☐ It may contain an alcohol group ( $OH$ ).
- 

## Part B Spectrum P

Based on absorptions seen outside of the fingerprint region, suggest a possible structure with molecular formula  $C_4H_{10}O$  that would give spectrum **P**.

Enter your answer as a SMILES string.

Use the [structure editor](#) to draw the structural formula of the molecule. Click on the round, yellow smiley face to generate a SMILES string. Copy the SMILES string and paste it in the answer box. For more detailed help, visit our [instructions for using the structure editor](#).

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## Part C Spectrum Q

Based on absorptions seen outside of the fingerprint region, suggest a possible structure with molecular formula  $\text{C}_4\text{H}_{10}\text{O}$  that would give spectrum **Q**.

Enter your answer as a SMILES string.

Use the [structure editor](#) to draw the structural formula of the molecule. Click on the round, yellow smiley face to generate a SMILES string. Copy the SMILES string and paste it in the answer box. For more detailed help, visit our [instructions for using the structure editor](#).

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## IR Oxidation

A Level



The following three infrared spectra were obtained at different stages of an oxidation reaction of an organic compound.

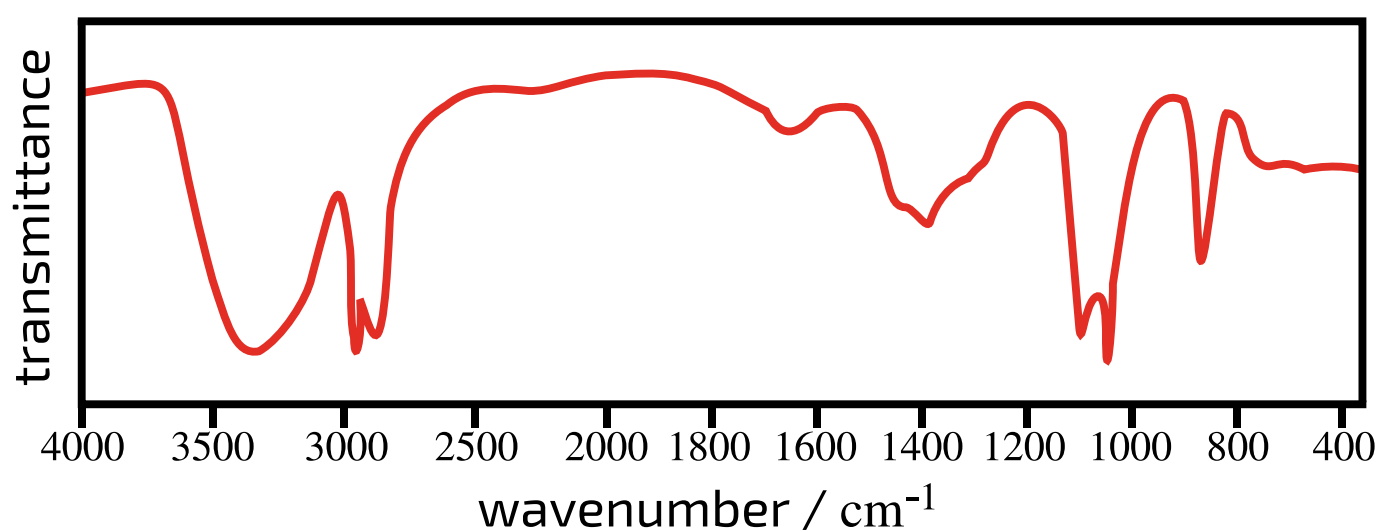


Figure 1: Spectrum for compound P

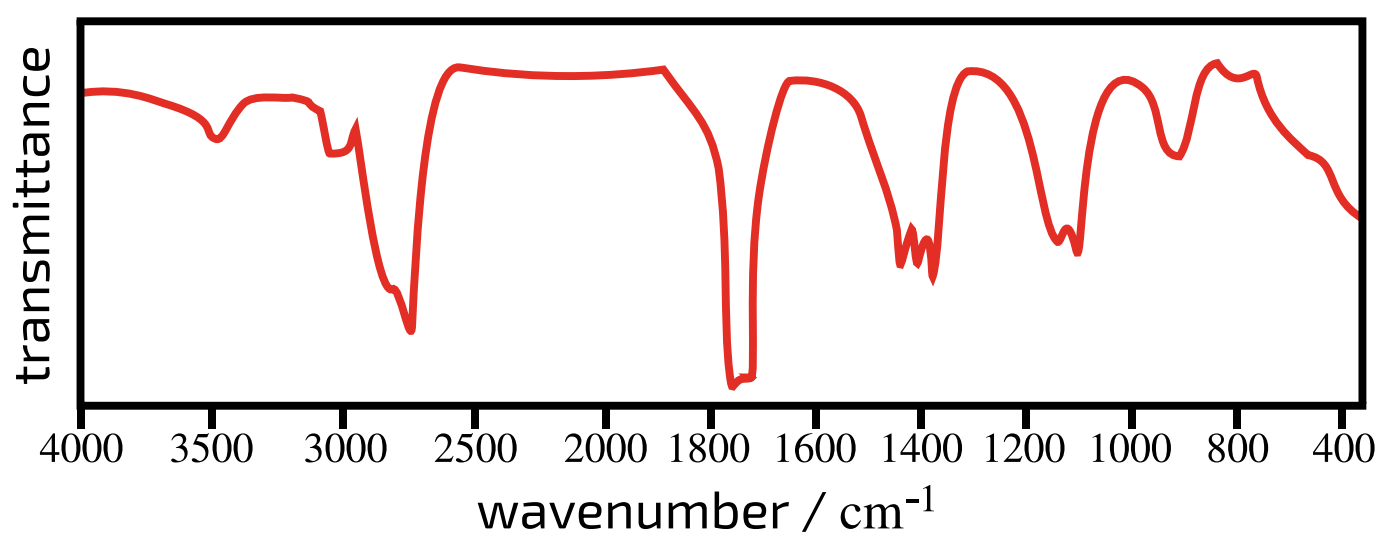


Figure 2: Spectrum for compound Q

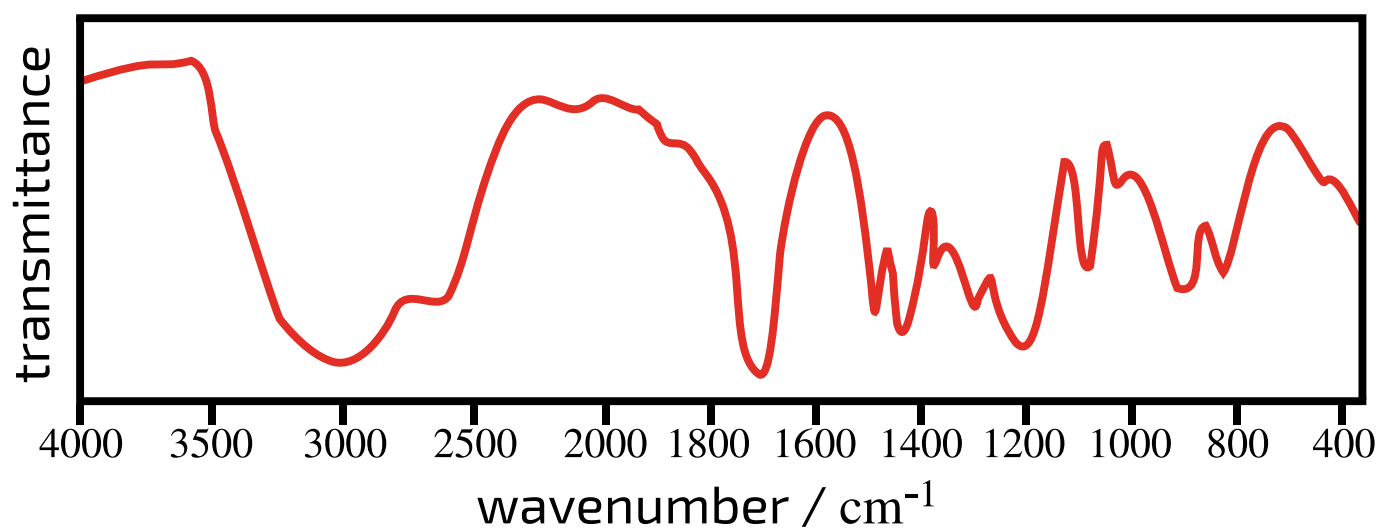


Figure 3: Spectrum for compound R

### Part A Functional group in P

What is the functional group in P that gets oxidised over the course of the reaction?

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### Part B Spectrum of P

In which parts of the spectrum can we see characteristic absorptions based on the presence of the functional group from the previous part?

☐  $1600\text{ cm}^{-1}$  to  $1800\text{ cm}^{-1}$

☐  $1800\text{ cm}^{-1}$  to  $2000\text{ cm}^{-1}$

☐  $2000\text{ cm}^{-1}$  to  $2500\text{ cm}^{-1}$

☐  $2500\text{ cm}^{-1}$  to  $3000\text{ cm}^{-1}$

☐  $3000\text{ cm}^{-1}$  to  $3500\text{ cm}^{-1}$

### Part C Bond in Q

What characteristic bond appears during the oxidation, with a characteristic absorption in the spectra of Q and R?

A  bond between  and  with a stretching frequency that falls in the   $\text{cm}^{-1}$  region.

Items:

single

double

triple

hydrogen

carbon

nitrogen

oxygen

1600-1800

1800-2000

2000-2500

2500-3000

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## Part D Functional group in R

What is the functional group formed in the oxidation and present in compound R?

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## Part E Spectrum of R

In which parts of the spectrum can we see characteristic absorptions based on the presence of the functional group from the previous part?

Around , we see a broad absorption due to the vibration of a  bond between  and . The broadening is a result of  bonding. Around , we see a sharper absorption due to the vibration of a  bond between  and .

Items:

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# Ammonia Reactions

A Level



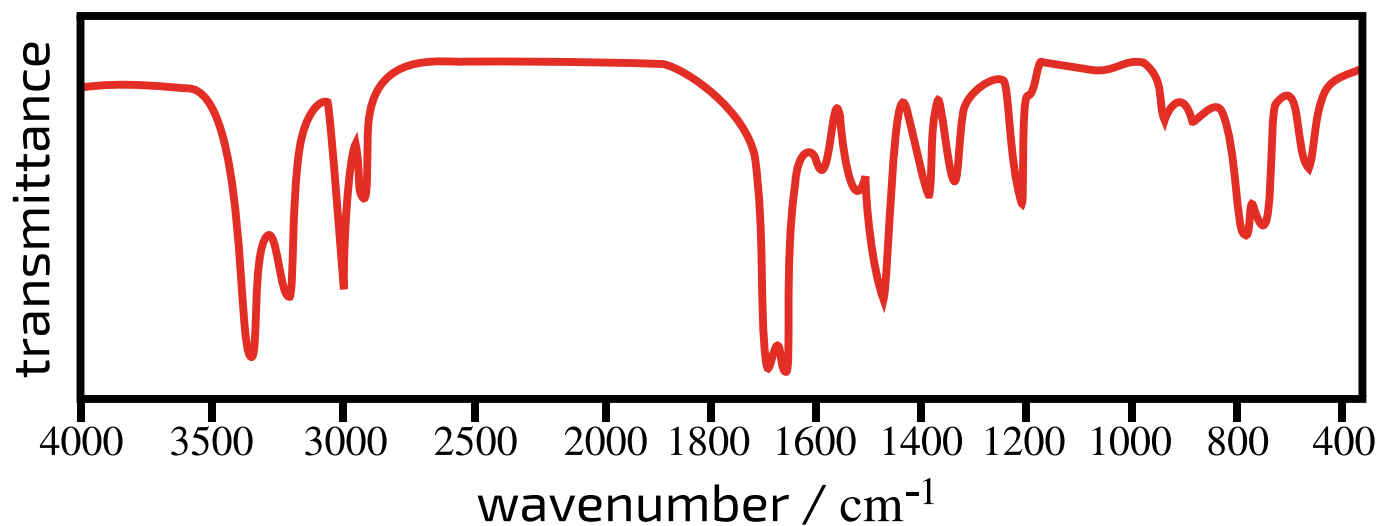
This question is about two reactions of ammonia.

## Reaction 1:

Excess ammonia is reacted with 4.77 g of copper(II) oxide. The reaction produces 3.81 g of solid **A**, liquid **B** and 0.560 g of gas **C**, which has a volume of 480 cm<sup>3</sup> at RTP.

## Reaction 2:

Ammonia reacts with compound **D** to form compound **E**, C<sub>2</sub>H<sub>5</sub>NO, and chloride salt **F**. The IR spectrum of **E** is shown below.



Identify **A**, **B**, **C**, **D**, **E** and **F**, and write equations for the two reactions.

**Part A**    **A**

Identify **A**.

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**Part B    B**

Identify **B**.

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**Part C    C**

Identify **C**.

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**Part D    D**

Identify **D**, writing the formula in a way that makes the functional group present clear, and places it on the right-hand side of the molecule.

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**Part E    E**

Identify **E**, writing the formula in a way that makes the functional group present clear, and places it on the right-hand side of the molecule.

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**Part F    F**

Identify **F**, listing the cation before the anion in the molecular formula you provide.

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### Part G    Reaction 1

Write an equation for Reaction 1 (do not include state symbols).

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### Part H    Reaction 2

Write an equation for Reaction 2, making sure to write the formulae the same way as in the previous parts of this question (do not include state symbols).

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# Introduction to NMR Spectroscopy



NMR (nuclear magnetic resonance) spectroscopy is a very powerful spectroscopic technique that helps with the characterisation of substances and structure determination. In this question, we will focus in on carbon NMR specifically.

## Part A Spin-active nuclei

NMR can only be used on nuclei that have a non-zero value of a property called *spin*. In particular, atoms containing even numbers of both protons and neutrons have a spin of zero, and so are not spin-active and will not be visible to our NMR instrument. All other nuclei have a non-zero spin, though not all are equally suitable for NMR spectroscopy.

The most common isotope of carbon is  with  protons and  neutrons in its nucleus. It is therefore . A less common isotope of carbon, with about 1% abundance, is , which has  protons and  neutrons in its nucleus and is therefore . It is only the  isotope that we will see in NMR spectroscopy.

Items:

## Part B Chemical environments

When placed in a magnetic field, nuclei with non-zero spin can take on different energy levels, and transitions between these energy levels can occur. It is the transition between these energy levels that NMR spectroscopy relies on. The differences in energy depend on the magnetic field: the stronger the field, the larger the energy differences and vice versa. To a large part, the magnetic field experienced by the nucleus is determined by the external magnetic field it is placed in. However, the field is modified somewhat by the electrons around the nucleus in question, giving rise to different nuclei within an atom potentially experiencing a slightly different magnetic field. Frozen in time, the electron distribution around any given nucleus would be different, but what matters is the average environment experienced by a given nucleus on the NMR timescale.

Rotations around single bonds and bond vibrations take place fast enough on the NMR time scale for the two carbons in an ethane molecule to be equivalent, so ethane only has  carbon environment(s). In propane, the two  carbons are equivalent, as we can draw a plane of symmetry through the molecule, and the  carbon is different, so propane has  carbon environment(s). Straight-chain butane has  carbon environments while straight-chain pentane has  carbon environments.

Items:

## Part C Shifts

Carbons in different environments will give rise to signals at different *chemical shift* values in the NMR spectrum. A higher shift corresponds to more *deshielded* nuclei that experience a higher magnetic field, as they are surrounded by less electron density.

Saturated carbons have the lowest shifts, falling into the  region. When saturated carbons are attached to electronegative atoms, the electron density around their nuclei is  meaning they have slightly higher shifts, falling into the  region. At the higher end of this is also where we usually find triple-bonded carbons. Double-bonded (and aromatic) carbons actually appear at a slightly higher shift, in the  region. The highest shift region of  is where we find carbons that are double-bonded and have bonds to particularly electronegative atoms, most notably the carbonyls.

Items:

increased

reduced

0–50 ppm

50–100 ppm

100–150 ppm

150–200 ppm

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# Counting Environments



For each of the molecules below, specify how many signals you expect to see in the  $^{13}\text{C}$  and  $^1\text{H}$  NMR spectra.

## Part A Ethanol

Specify the number of  $^{13}\text{C}$  NMR environments in ethanol.

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Specify the number of  $^1\text{H}$  NMR environments in ethanol.

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## Part B Benzene

Specify the number of  $^{13}\text{C}$  NMR environments in benzene.

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Specify the number of  $^1\text{H}$  NMR environments in benzene.

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**Part C**    **1,4-dichlorobenzene**

Specify the number of  $^{13}\text{C}$  NMR environments in 1,4-dichlorobenzene.

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Specify the number of  $^1\text{H}$  NMR environments in 1,4-dichlorobenzene.

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**Part D**    **4-nitrophenol**

Specify the number of  $^{13}\text{C}$  NMR environments in 4-nitrophenol.

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Specify the number of  $^1\text{H}$  NMR environments in 4-nitrophenol.

---

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**Part E**    **2-methylbut-2-ene**

Specify the number of  $^{13}\text{C}$  NMR environments in 2-methylbut-2-ene.

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Specify the number of  $^1\text{H}$  NMR environments in 2-methylbut-2-ene.

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# Propanal Spectrum

A Level

Fill in the gaps in the table with details of the signals you expect to see in the  $^1\text{H}$  NMR spectrum of propanal. Order the signals based on the expected chemical shift, starting with the *highest expected shift at the top*.

Expected shift	Relative integral	Splitting pattern
<input type="text"/>	1	<input type="text"/>
<input type="text"/>	<input type="text"/>	<input type="text"/>
<input type="text"/>	<input type="text"/>	<input type="text"/>

Items:

0 to 2 ppm
2 to 4 ppm
4 to 6 ppm
6 to 8 ppm
8 to 10 ppm
1
2
3
singlet
1 : 1 doublet

1 : 2 : 1 triplet
1 : 3 : 3 : 1 quartet
1 : 4 : 6 : 4 : 1 quintet
other

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# Proton NMR

$^1\text{H}$  NMR (commonly referred to as proton NMR) is the most commonly used type of NMR spectroscopy. A key feature in proton NMR is that we not only see chemical shift values, but that the spectra are quantitative: the *integral*, i.e. the area under the trace of a given signal, is proportional to the number of protons corresponding to that environment. We also see *splitting* of signals by protons coupling to one another, which means that a given signal may consist of multiple lines, giving characteristic *splitting patterns*.

## Part A Shifts

$^1\text{H}$  NMR spans a different range of shift values compared to  $^{13}\text{C}$  NMR. Match the hydrogen highlighted in bold in each case to its approximate expected shift value.

Proton type	Expected shift
$\text{CH}_3\text{CHO}$	<input type="text"/>
$\text{CH}_3\text{CHO}$	<input type="text"/>
$\text{C}_6\text{H}_6$ (benzene)	<input type="text"/>
$\text{C}_6\text{H}_5\text{CH}_3$ (methylbenzene)	<input type="text"/>
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$	<input type="text"/>
$\text{C}(\text{CH}_3)_2=\text{CH}_2$	<input type="text"/>

Items:

0 to 2 ppm

2 to 3 ppm

3 to 4 ppm

4 to 6 ppm

6 to 8 ppm

8 to 9 ppm

9 to 10 ppm

## Part B Integrals

In some compounds, it is possible to tell the proton environments purely based on the integral trace on the  $^1\text{H}$  NMR spectrum. The integrals are usually more reliable than shift values when it comes to assignments (as there are several more complicated effects that can affect shift).

Unbranched pentane contains  different proton environments. The end carbons have a total of  hydrogens in one environment. The middle carbon is bonded to  equivalent hydrogens. The remaining  hydrogens all form another environment. Since the numbers of hydrogens in each environment are all different, it is easy to tell the environments apart simply based on the integral trace, as the ratio of the integrals will match the ratio of hydrogens present in each environment.

Items:

one

two

three

four

five

six

## Part C Splitting patterns

Match the hydrogen highlighted in bold in each case to its expected splitting pattern appearance in a  $^1\text{H}$  NMR spectrum.

Proton type	Expected splitting pattern
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$	<input type="text"/>
$\text{CH}_3\text{CHCl}_2$	<input type="text"/>
$\text{CH}_3\text{CHCl}_2$	<input type="text"/>
$\text{CH}_3\text{CCl}_2\text{CBr}_2\text{CH}_3$	<input type="text"/>
$\text{CHCl}=\text{CHF}$	<input type="text"/>

Items:

singlet

1:1 doublet

1:2:1 triplet

1:3:3:1 quartet

1:4:6:4:1 quintet

# Identifying C<sub>5</sub>H<sub>10</sub>O

A Level

Compounds **A**, **B** and **C** are isomers with the molecular formula C<sub>5</sub>H<sub>10</sub>O. One of the compounds is alicyclic.

A student carries out test-tube tests on the compounds. The observations are shown below.

Compound	Brady's reagent	H <sup>+</sup> / Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup> , reflux	Bromine water
<b>A</b>	No change	Green solution	No colour change
<b>B</b>	Orange precipitate	No colour change	No colour change
<b>C</b>	Orange precipitate	No colour change	No colour change

<sup>13</sup>C NMR spectrum of **A** shows 3 peaks at  $\delta$ /ppm : 24, 36, 73.

<sup>1</sup>H NMR spectrum of **B** shows a triplet (1.0 – 1.1 ppm) and a quartet (2.4 – 2.5 ppm).

<sup>1</sup>H NMR spectrum of **C** shows a doublet (1.0 – 1.1 ppm), a singlet (2.0 – 2.1 ppm) and a septet (2.5 – 2.6 ppm)

Analyse the observations and results to identify the structures of **A**, **B** and **C**.

Use the [structure editor](#) to generate a SMILES string for each compound. In the editor, after drawing your structure, click on the 😊 to generate a SMILES string. Copy the SMILES string and paste it in the answer boxes below. For more advice on the structure editor, see [our guidance](#).

## Part A    **A**

Structure of **A**:

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---

**Part B    B**

Structure of **B**:

---

---

**Part C    C**

Structure of **C**:

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# Life on Mars

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A Level



Is there life on Mars? In 1976, two spacecraft landed on the planet. They were equipped with apparatus for carrying out experiments to try to find out the answer.

Three experiments were performed; two showed no evidence for life, but some scientists think the third experiment did. This experiment was designed to detect the presence of micro-organisms in Martian soil.

A solution of compounds, known to be used in metabolism by micro-organisms on Earth, was added to a sample of Martian soil. The compounds were labelled with  $^{14}\text{C}$ , a radioactive isotope of carbon.

The chemists who designed the experiment argued that, if micro-organisms were present in the soil sample, there was a chance that they would metabolise the labelled compounds and break them down into small molecules, some of which would contain labelled carbon atoms, and might be released as gas. The experiment therefore included a Geiger counter to measure the radioactivity of the gas contained in the apparatus.

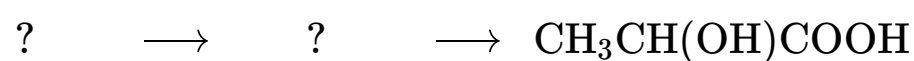
## Part A 2-hydroxypropanoic acid

One of the compounds used in this experiment was sodium 2-hydroxypropanoate (sodium lactate). This was made from a sample of 2-hydroxypropanoic acid in which each molecule contained a  $^{14}\text{C}$  atom.

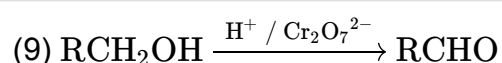
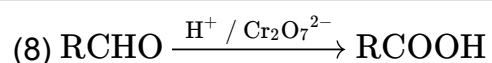
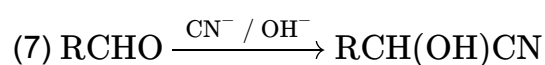
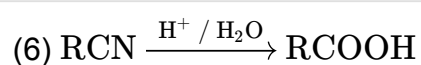
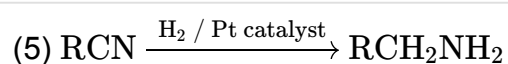
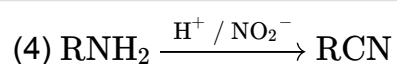
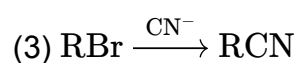
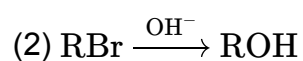
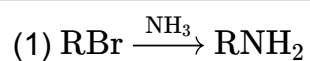
Molecules of this kind were not already available and had to be prepared from a source of  $^{14}\text{C}$  by appropriate chemical reactions. One such source is  $^{14}\text{C}$ -labelled cyanide ions,  $^{14}\text{CN}^-$ . A list of potentially useful reactions is given below; in these reactions, R represents an alkyl group.



Choose reactions from this list to suggest a two-step route which could be used to prepare 2-hydroxypropanoic acid. Record your chosen route by completing the sequence below.



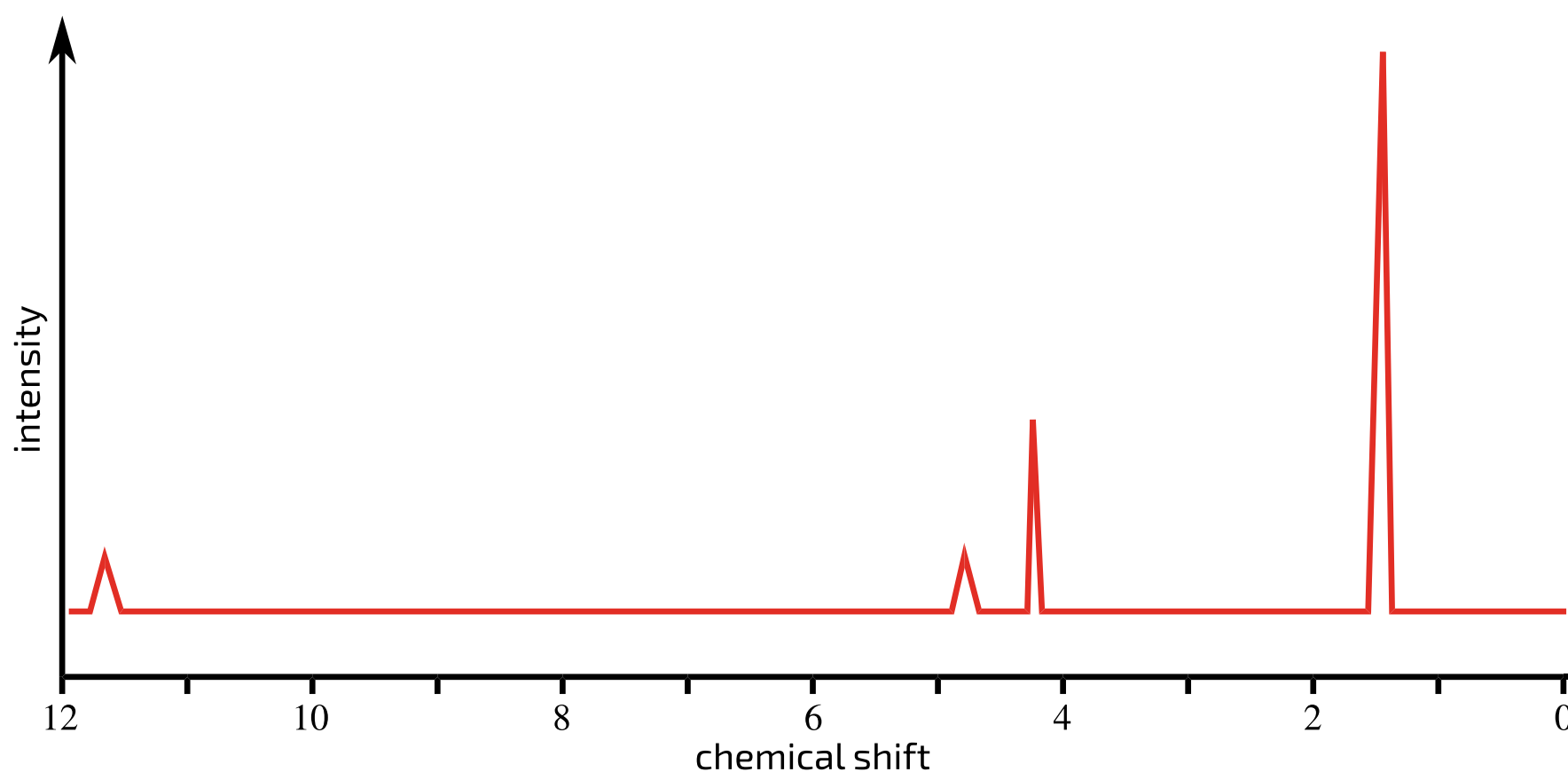
### Available items



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**Part B**  $^1\text{H}$  NMR

The proton NMR spectrum of the product of the reaction sequence was recorded and is shown in **Figure 1**.



**Figure 1:** Proton NMR spectrum of the product, with chemical shift in ppm.

As well as showing that 2-hydroxypropanoic acid has been obtained, the NMR spectrum also shows that the reaction product is free from traces of starting material. Give a value for the chemical shift of one signal which is absent from this spectrum but which would be seen if traces of starting material were present.

- ☐ 0 - 2 ppm
  - ☐ 2 - 4 ppm
  - ☐ 4 - 6 ppm
  - ☐ 6 - 8 ppm
  - ☐ 8 - 10 ppm
  - ☐ 10 - 12 ppm
-



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### Part C    Type of proton

State the type of proton which would give rise to the signal you have chosen.

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