Elemental Microanalysis

2006 AH MC37 (68%)

37. An analysis of an organic compound found in meteorite rocks shows the following percentage composition by mass.

C = 37·5%   H = 12·5%   O = 50%

The empirical (simplest) formula for the compound is

A CH_4O

B C_3HO_4

C $C_3H_{12}O_3$

D CH_2O_2.

2012 AH MC37 (20%)

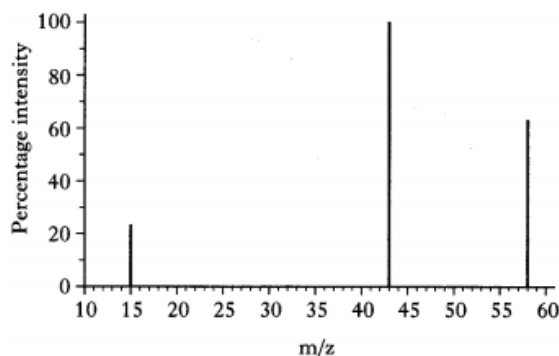**37.** Combustion analysis of hydrocarbon **X** showed that it contained 82·7% carbon and 17·3% hydrogen.

The molecular formula for **X** could be

A CH_3

B C_2H_6

C C_2H_5

D C_4H_{10}.

Mass Spectrometry

2006 AH MC38 (72%) and 2007 AH MC40 (71%) and 2014 AH MC38 (75%) and 2014 revAH MC26 (79%)

38. A simplified mass spectrum of an organic compound is shown.

Which of the following compounds gave this spectrum?

A Propane

B Propan-1-ol

C Propan-2-ol

D Propanone

2011 AH MC38 (74%)

38. The mass spectrum of an organic compound, empirical formula C_2H_4O, shows a peak for the parent ion at mass/charge ratio of 88.
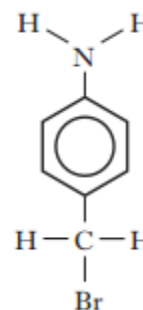
The organic compound could **not** be

A   ethanal

B   butanoic acid

C   ethyl ethanoate

D   methyl propanoate.

2014 AH MC37 (46%) and 2014 revAH MC28 (49%)

37. The two isotopes of bromine have mass numbers 79 and 81.

In the mass spectrum of

the ion fragment with a mass/charge ratio of 92 could be caused by

A $[CHBr]^-$

B $[CH_2Br]^+$

C $[C_6H_4NH_2]^-$

D $[C_6H_4NH_2]^+$.

2010 AH MC39 (42%)

39. Which of the following causes the separation of the ions in a mass spectrometer?

A A magnetic field

B A vacuum pump
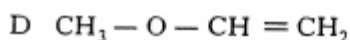
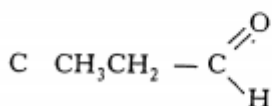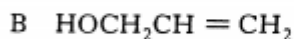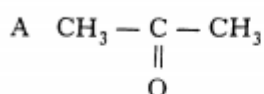C An ionisation chamber

D Electron bombardment

2004 AH MC39 (65%) and 2014 AH MC39 (80%) and 2014 revAH MC27 (80%)

39. Which of the following analytical techniques depends on the vibrations within molecules?

A   Nuclear magnetic resonance spectroscopy

B   Atomic emission spectroscopy

C   Infra-red spectroscopy

D   Mass spectrometry

2001 AH MC27 (86%)

27. Which of the following compounds is most likely to show an infra-red absorption at $2725 \, cm^{-1}$?
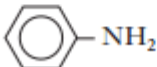
A $CH_3 - C - CH_3$
      $\overset{\|}{O}$

B $HOCH_2CH = CH_2$

C $CH_3CH_2 - C \overset{\displaystyle O}{\underset{\displaystyle H}{\diagup}}$

D $CH_3 - O - CH = CH_2$

2008 AH MC39 (58%)

39. An organic compound with empirical formula, C_2H_4O, has major peaks at $1715 \, cm^{-1}$ and $3300 \, cm^{-1}$ in its infrared spectrum.

The structural formula of the compound could be

A CH_3CHO

B CH_3COOH

C $CH_3COOCH_2CH_3$

D $CH_3CH_2CH_2COOH$.

2014 AH MC33 (83%) and 2014 revAH MC29 (90%)

33. Which of the following amines shows no infra-red absorption between $3300 \, cm^{-1}$ and $3500 \, cm^{-1}$?

A $(CH_3)_3N$

B CH_3NHCH_3
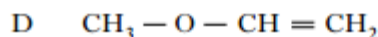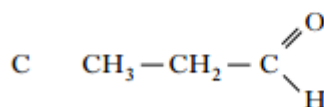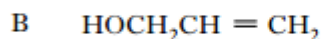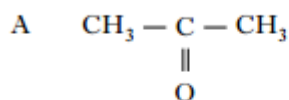
C $H_2NCH_2NH_2$

D   ⬡—NH_2

2006 AH MC39 (80%)

39. Which of the following analytical techniques depends on the vibrations within molecules?

A   Colorimetry

B   Atomic emission spectroscopy

C   Infra-red absorption spectroscopy

D   Mass spectroscopy

2010 AH MC40 (88%)

40. Which of the following compounds is most likely to show an infra-red absorption at $2725 \, cm^{-1}$?

A $CH_3 - C - CH_3$
      $\overset{\|}{O}$

B $HOCH_2CH = CH_2$

C $CH_3 - CH_2 - C \overset{\displaystyle O}{\underset{\displaystyle H}{\diagup}}$

D $CH_3 - O - CH = CH_2$

2012 AH MC38 (74%)

38. The number of waves per centimetre is known as the

A   wavenumber

B   wavelength

C   frequency

D   intensity.

2012 AH MC39 (82%)

39. Which of the following analytical techniques depends on the vibrations within molecules?

A   Colorimetry

B   Mass spectroscopy

C   Proton nmr spectroscopy

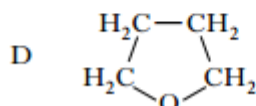D   Infra-red absorption spectroscopy

2011 AH MC39 (35%)

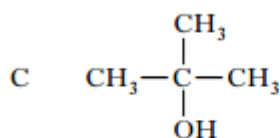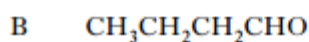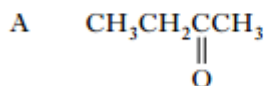39. From which region of the electromagnetic spectrum is energy absorbed in the production of proton nmr spectra?

    A   X-rays

    B   Visible

    C   Infra-red

    D   Radio waves

2011 AH MC40 (27%)
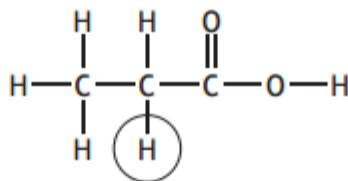
40. A compound, which has molecular formula C_4H_8O, has only 2 peaks in its low resolution proton nmr spectrum.

A possible structural formula for this compound is

    A $CH_3CH_2CCH_3$
                    ‖
                    O

    B $CH_3CH_2CH_2CHO$

                    CH_3
                    |
    C CH_3-C-CH_3
                    |
                    OH

            H_2C-CH_2
    D
            $H_2C\qquad CH_2$
                  O

2016 AH MC24 (45%)

24.

Which of the following shows the splitting pattern for the circled **H** atom above, in a high resolution proton NMR spectrum?

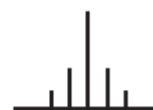A   B   C   D
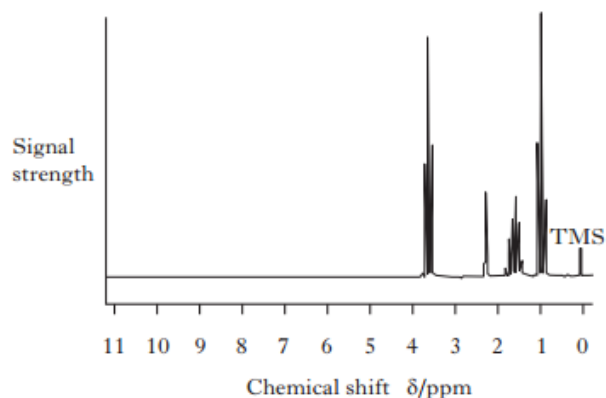

2015 revAH MC19 (89%)

19. The high resolution proton NMR spectrum of compound **X** is shown below.

X could be

    A   propanal

    B   propanone

   C   propan-1-ol

    D   propanoic acid.

2002 AH grid31b+c

31. The boxes in the grid below contain the names of techniques used in chemistry.

| A	B	C
Infra-red spectroscopy	Proton NMR spectroscopy	Visible spectroscopy
D	E	F
Ultra-violet spectroscopy	Mass spectrometry	X-ray crystallography

(b) In which technique will the test sample be destroyed?

(c) Which technique(s) will produce a diffraction pattern which enables the precise three-dimensional structure of a compound to be determined?

2001 AH L11b+c

11. The structures of the two dicarboxylic acids with molecular formula $C_4H_6O_4$ are drawn below.

A   B
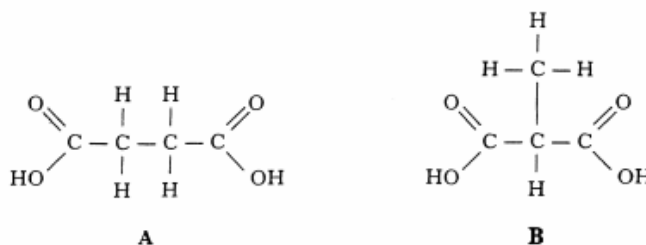
(b) The simplified mass spectrum of **A** is illustrated below.
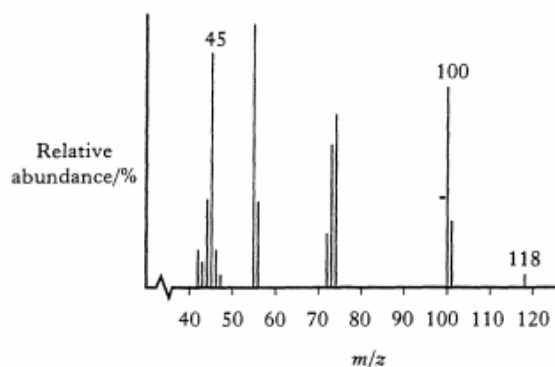
(i) Write the formula for the ion which gives the peak at $m/z = 45$.   1

(ii) What has been **lost** from molecule **A** to give the ion at $m/z = 100$?   1

(c) Copy the following diagram and complete it to show the nmr spectrum of **A**.

Chemical shift, δ/ppm   2

4
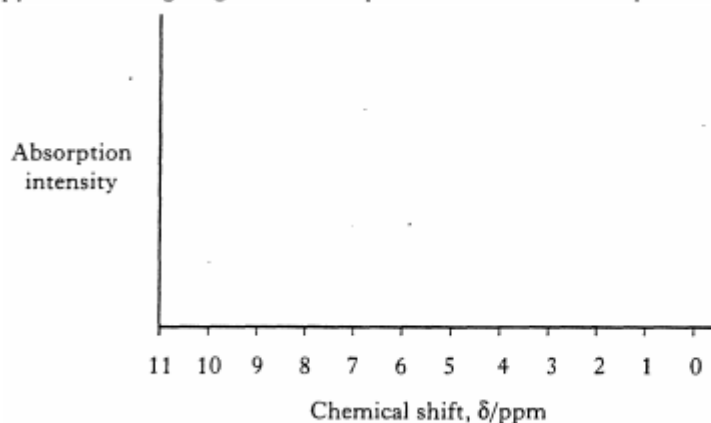
7. The following simplified spectra were obtained for a pure organic compound containing carbon, hydrogen and oxygen only.

Infra-red spectrum

Mass spectrum

Proton nmr spectrum

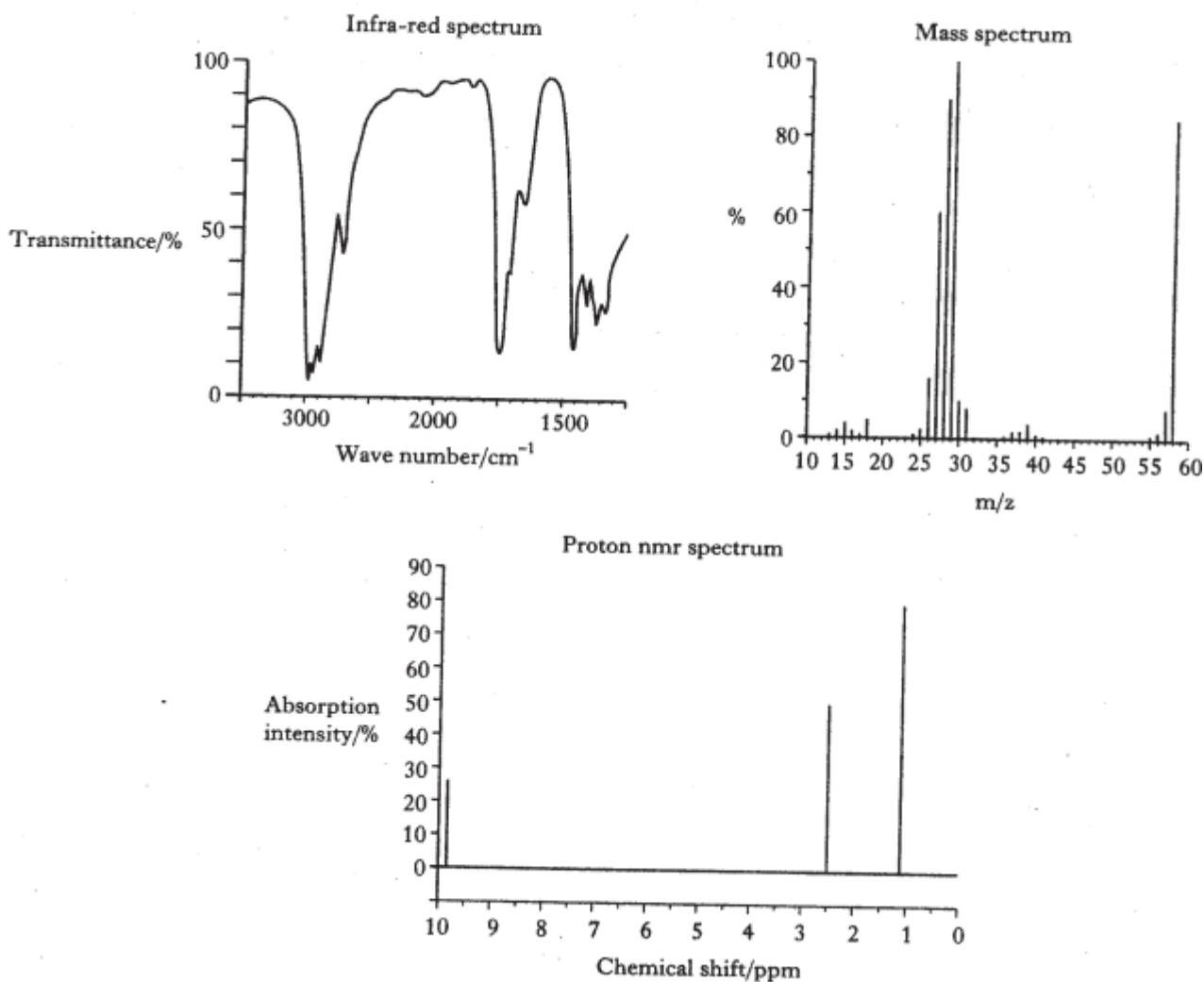The proton nmr spectrum was analysed and the data are presented in the table below.

Chemical shift	Area under the peak	H atom ratio
1·1	6·9	X
2·5	4·5	Y
9·8	2·3	Z

(a) Identify the functional group causing the absorption at $1730 \, cm^{-1}$ in the infra-red spectrum.

1

(b) Identify a possible ion fragment responsible for the peak at m/z 29 in the mass spectrum.

1

(c) From the proton nmr spectrum what is the whole number ratio of hydrogen atoms, X:Y:Z?

1

(d) From the information above draw a structural formula for this compound.

2

5

12. A chemist used a variety of techniques to identify a sweet-smelling compound, **X**.

   (a) The infra-red spectrum of **X** is shown below.

Relative transmittance vs Wavenumber/cm^{-1}
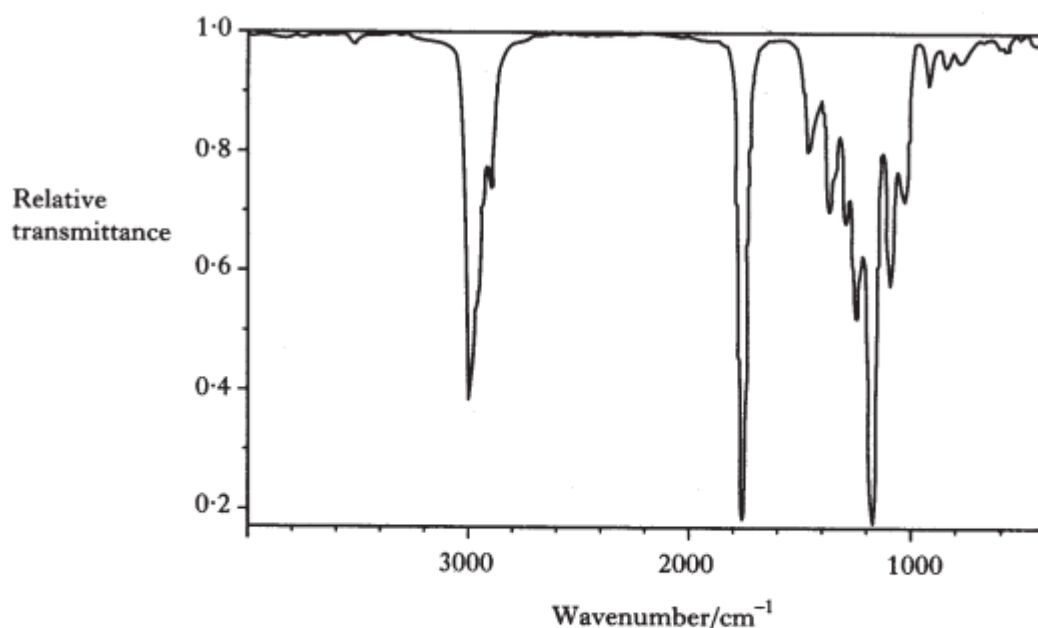
   (i) Which bond is responsible for the strong absorption at 1745 cm^{-1}? 1

   (ii) To which class of organic compounds does **X** belong? 1

   (b) Compound **X** was subjected to elemental analysis. Complete combustion of 0·210 g of **X** gave 0·478 g of carbon dioxide and 0·196 g of water. No other product was formed.

   (i) Calculate the masses of carbon and hydrogen in the original sample and hence deduce the mass of oxygen present. 2

   (ii) Calculate the empirical formula for compound **X**. 1

13. Compound **A** has molecular formula $C_4H_{10}O$.

   (a) To which two classes of organic compounds could **A** belong? 2

   (b) Compound **A** reacts with acidified potassium dichromate solution to form **B** which has molecular formula C_4H_8O.

   The proton nmr spectrum of **B** shows three peaks. Analysis of this spectrum produces the following data.

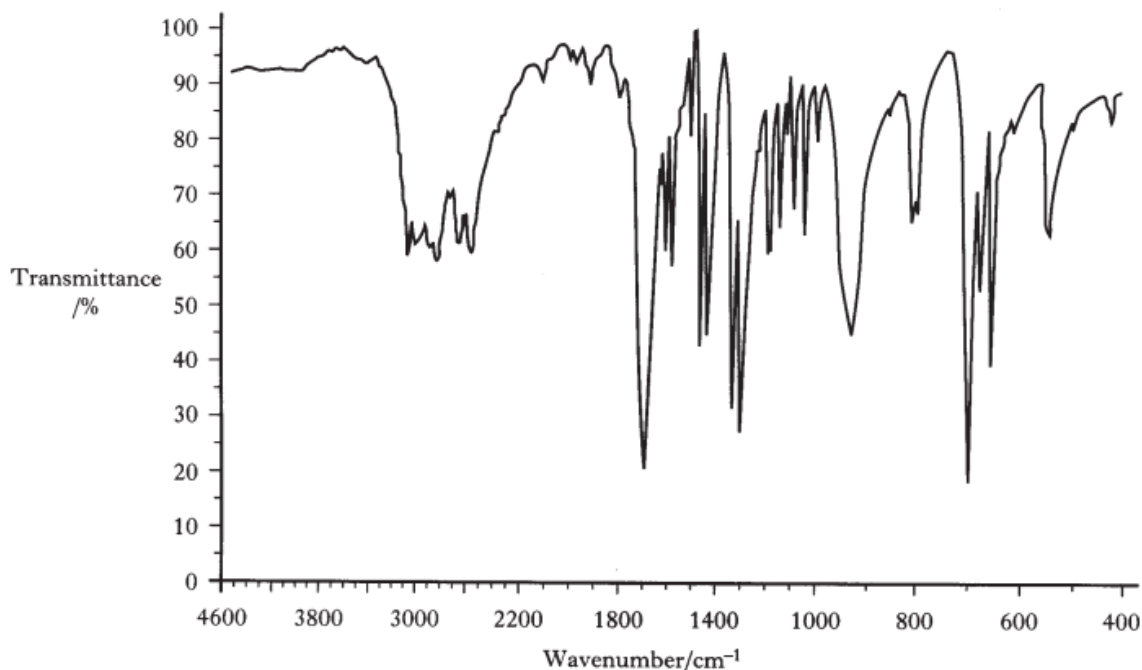| Peak	Chemical shift/ppm	Relative area under peak
1	0·95	3
2	2·05	3
3	2·35	2

   Considering all the evidence above:

   (i) draw a structural formula for **B**; 1

   (ii) name **A**. 1

6

13. A soluble white solid, **X**, does **not** react with 2,4-dinitrophenylhydrazine (Brady's Reagent).

(a) The infra red spectrum of **X** is shown.

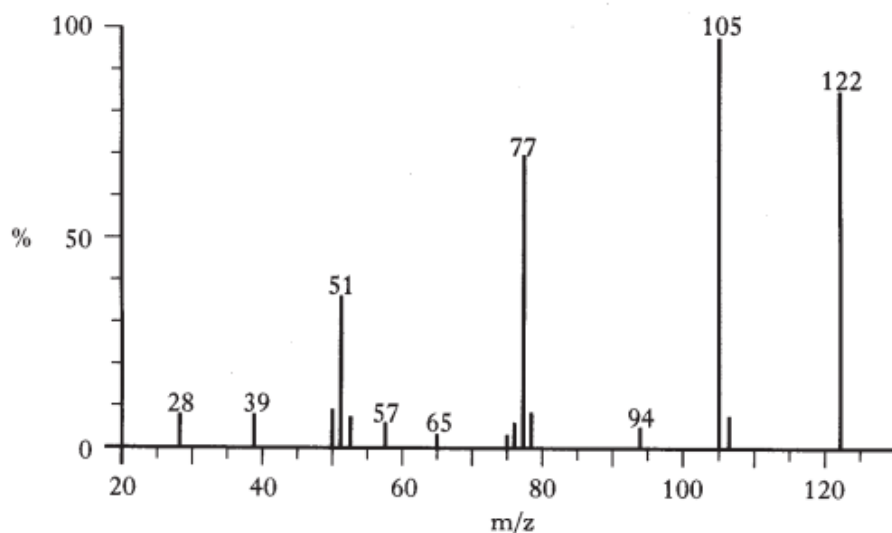Which bond causes the absorption peak at 1685 cm^{-1}?   **1**

(b) Microanalysis shows that compound **X** has a composition, by mass, of 68·9% C, 4·9% H and 26·2% O.

Show by calculation that the empirical formula for compound **X** is $C_7H_6O_2$.   **1**

(c) The mass spectrum for compound **X** is shown below.

(i) What is the molecular formula for compound **X**?   **1**

(ii) Identify a possible ion fragment responsible for the peak at m/z 77 in the mass spectrum.   **1**

(d) From the information above, draw a structural formula for compound **X**.   **1**

7

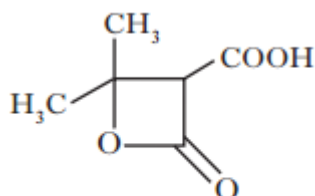**11.** Meldrum's acid is a chemical named after the Scotsman, Andrew N. Meldrum who was the first to produce it.

Microanalysis showed that Meldrum's acid has a composition, by mass, of 50% C, 5·6% H, 44·4% O.

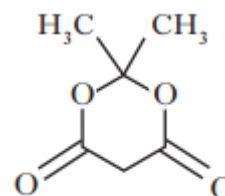(a) Use the percentage composition to calculate the empirical formula of Meldrum's acid.

(Working must be shown) **1**

(b) Meldrum initially thought the structure to be that shown as Compound A shown below. However, it was shown later that the actual structure was an isomer of A and is shown below as compound B.
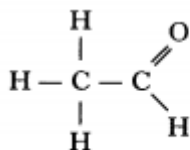
Compound A   **Compound B**

(ii) The infra-red spectrum of isomer **A** would show a strong absorbance not shown by isomer **B**.

Identify the wave number range, in cm^{-1}, where this absorbance occurs. **1**

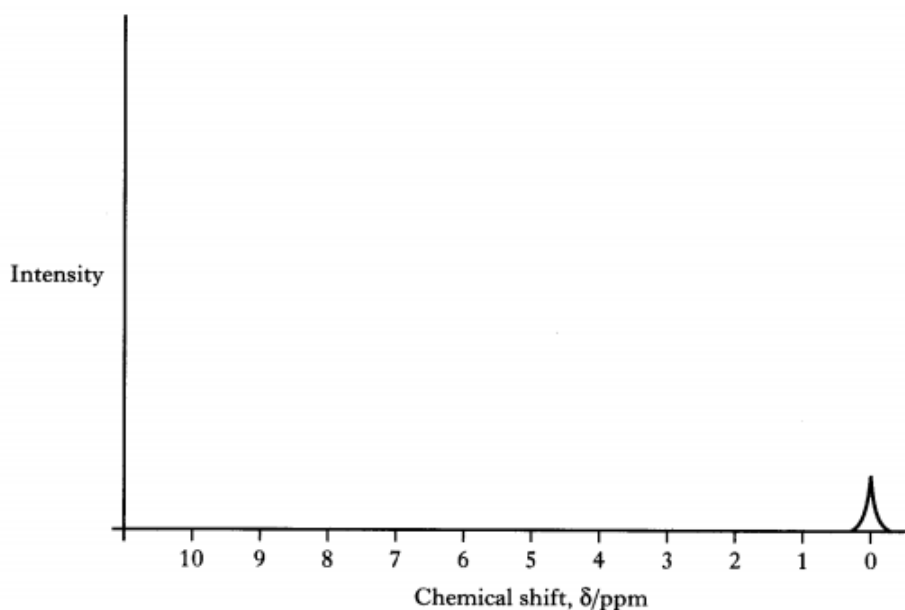**13.** A proton nmr spectrum was produced for ethanal.

ethanal

(a) Copy and complete the following diagram to show the approximate positions and relative heights of the two peaks in the proton nmr spectrum of ethanal.

Intensity

10   9   8   7   6   5   4   3   2   1   0

Chemical shift, δ/ppm **2**

(b) Which reference substance is used in proton nmr spectroscopy and causes the peak at δ = 0 ppm? **1**

13. A compound **X** containing only carbon, hydrogen and oxygen, was subjected to elemental analysis. Complete combustion of 1·76 g of **X** gave 3·52 g of carbon dioxide and 1·44 g of water. No other product was formed.

    (a)   (i)   Calculate the masses of carbon and hydrogen in the original sample and hence deduce the mass of oxygen present.   **2**

          (ii) Show, by calculation, that the empirical formula of compound **X** is C_2H_4O.   **1**

    (b) Given that the relative molecular mass of compound **X** is 88, deduce its molecular formula.   **1**

    (c) A low-resolution proton nmr spectrum of compound **X** is shown.
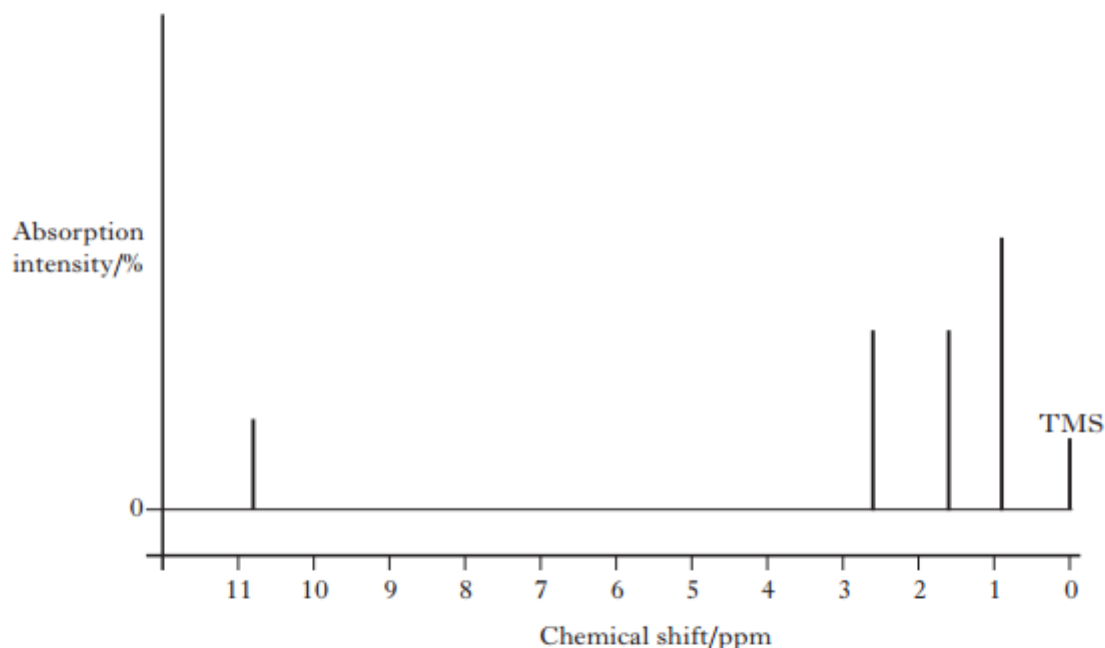
Analysis of this spectrum produced the data shown in the table below.

| Chemical shift	Relative area under the peak
0·9	3
1·6	2
2·6	2
10·8	1

          (i)   Using information from the table and your answer to (b), draw a structural formula for compound **X** and give its systematic name.   **2**

          (ii) What is the function of "TMS" (tetramethylsilane) in the proton nmr spectrum?   **1**

13. A superconductor, **X**, with a critical temperature of 95 K, was prepared by heating yttrium oxide, barium carbonate and copper oxide at high temperatures.

    (b)   (i)   **X** contains 13·4% yttrium, 41·2% barium, 28·6% copper and 16·8% oxygen.

             Assuming that the relative atomic mass of yttrium is 88·9, show by calculation that the empirical formula for **X** is $YBa_2Cu_3O_7$.   **2**

12. In a PPA, propanone reacts with 2,4-dinitrophenylhydrazine to make the 2,4-dinitrophenylhydrazone derivative as shown below.


| propanone | 2,4-dinitrophenylhydrazine | 2,4-dinitrophenylhydrazone derivative |
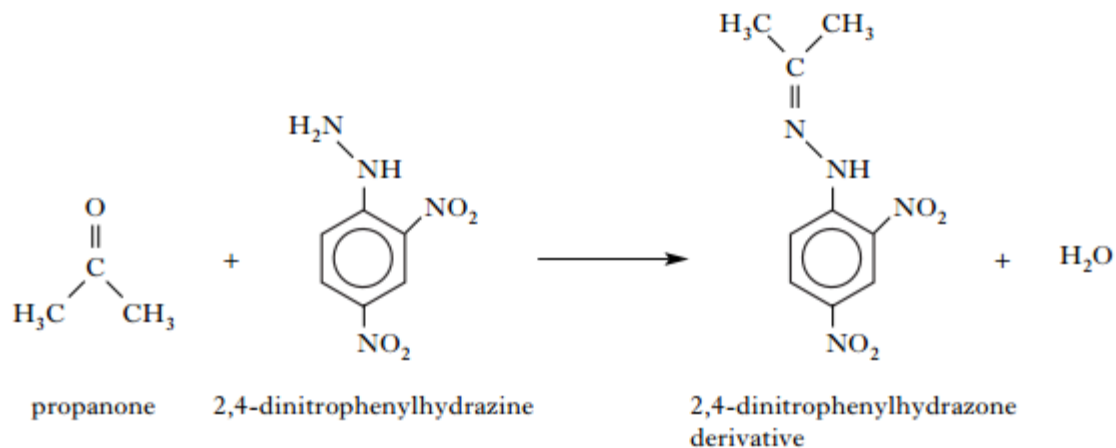
(c) Propanone has an isomer. The shortened structural formula of this isomer is CH_3CH_2CHO.

(ii) Nuclear magnetic resonance spectroscopy can also be used to distinguish between these two isomers. The proton nmr spectrum for CH_3CH_2CHO is shown.
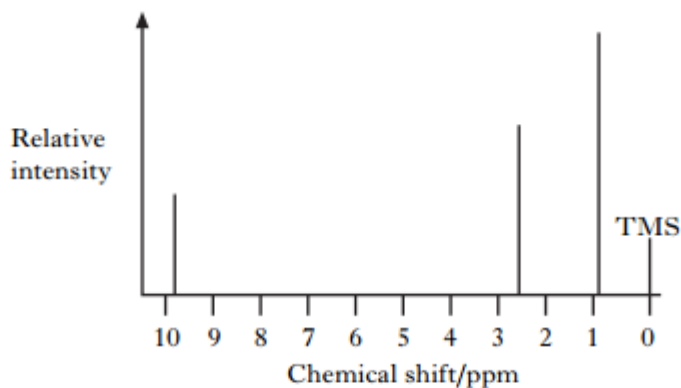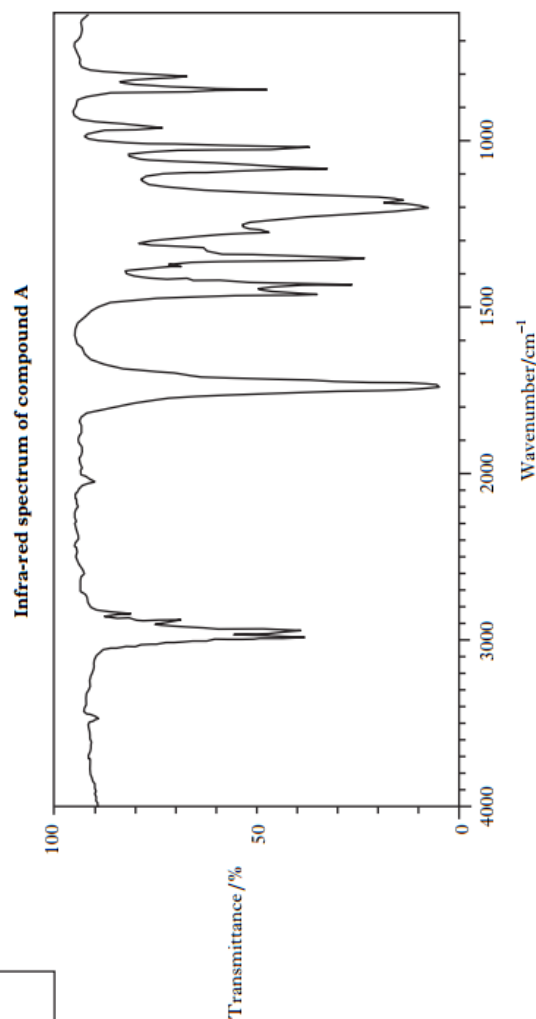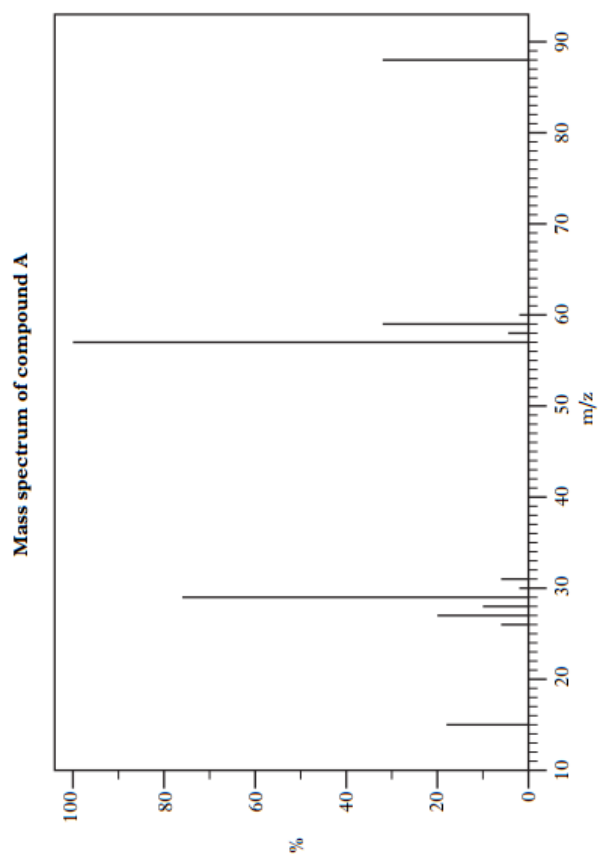

Sketch the proton nmr spectrum you would obtain for propanone. **1**

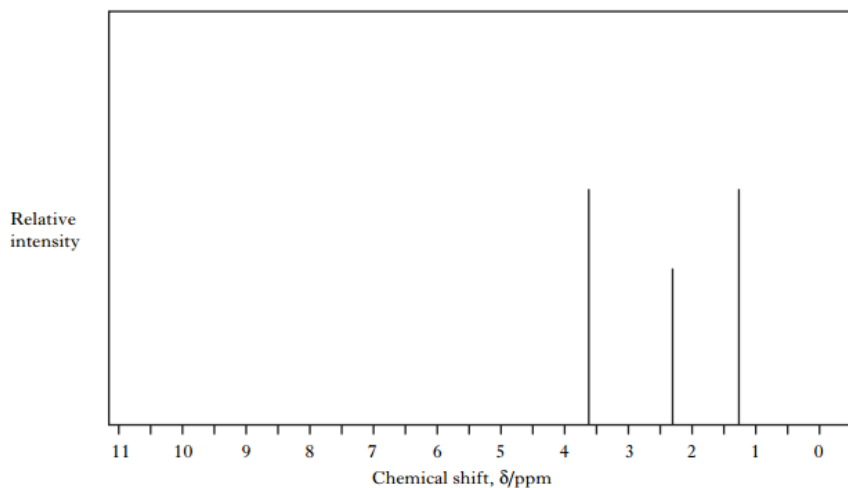(iii) A simplified mass spectrum for propanone is shown below.
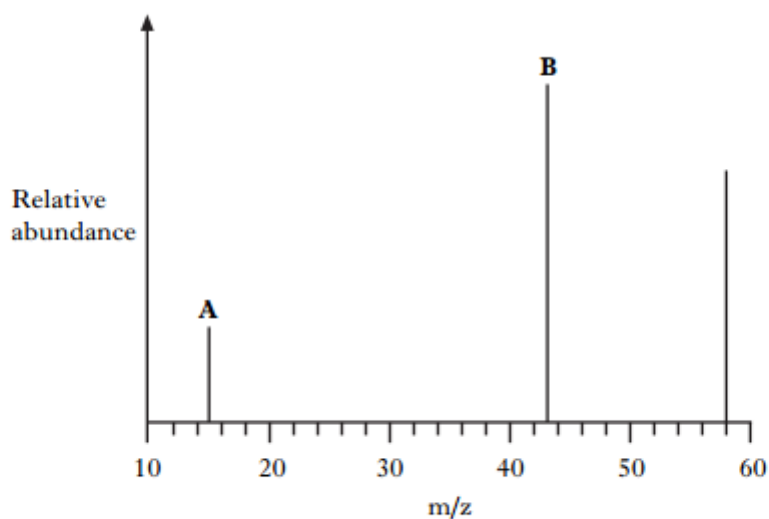
Identify the ion fragments responsible for peaks **A** and **B**. **1**

11. Spectra of an organic compound **A** are shown below.

Mass spectrum of compound A

Infra-red spectrum of compound A

Proton nmr spectrum of compound A

(a) Compound **A** has empirical formula C_2H_4O.

Using this information and the mass spectrum, deduce the molecular formula of **A**.   1

(b) The absorption peak at $1745\ cm^{-1}$ in the infra-red spectrum can be used to help identify **A**.

    (i) Which bond is responsible for this absorption?   1

    (ii) Which type of compound is **A**?   1

(c) Draw the structure of the ion fragment responsible for the peak at m/z 57 in the mass spectrum.   1

(d) Considering all the evidence, including the proton nmr spectrum, name compound **A**.   1

11. Meldrum's acid is a chemical named after the Scotsman, Andrew N. Meldrum who was the first to produce it.

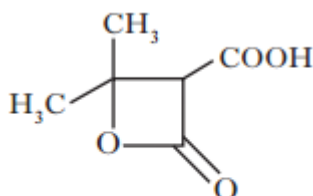Microanalysis showed that Meldrum's acid has a composition, by mass, of 50% C, 5·6% H, 44·4% O.

   (a) Use the percentage composition to calculate the empirical formula of Meldrum's acid.
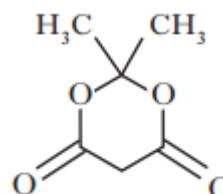
   (Working must be shown)　　　　　　　　　　　　　　　　　　　　　　　　　1

   b) Meldrum initially thought the structure to be that shown as Compound A shown below. However, it was shown later that the actual structure was an isomer of A and is shown below as compound B.

Compound A　　　　　　　　　　　　　　　　　**Compound B**

   (ii) The infra-red spectrum of isomer **A** would show a strong absorbance not shown by isomer **B**.

   Identify the wave number range, in cm^{-1}, where this absorbance occurs.　　　　1

15. Chloroalkane **A** has molecular formula C_4H_9Cl. When **A** is heated with NaOH(aq), it undergoes an S_N2 reaction to form alcohol **B**.

Alcohol **B** can be oxidised by acidified potassium dichromate solution and it can also be dehydrated to produce a mixture of two alkenes which are structural isomers.

   (c) The simplified proton nmr spectrum of one of the alkenes is shown.
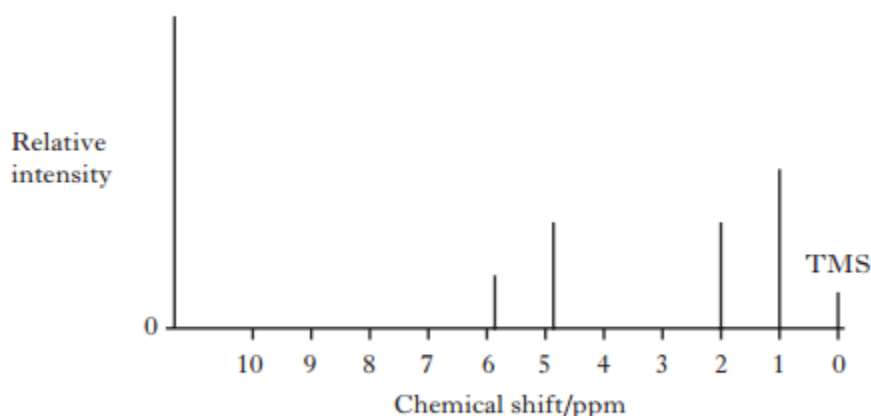
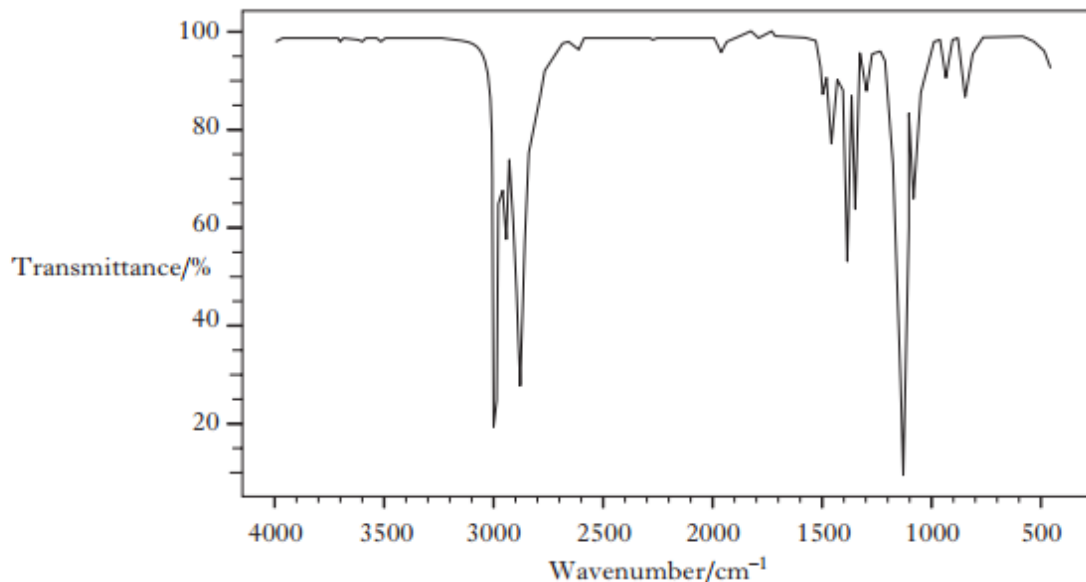

Sketch the proton nmr spectrum of the other alkene.　　　　　　　　　　　　　2

12. 5·00 g of an organic compound **A** was burned completely producing 11·89 g of CO_2 and 6·08 g of H_2O as the only products.

    (a) **Using the information above**, calculate the empirical formula of compound **A**.   **3**

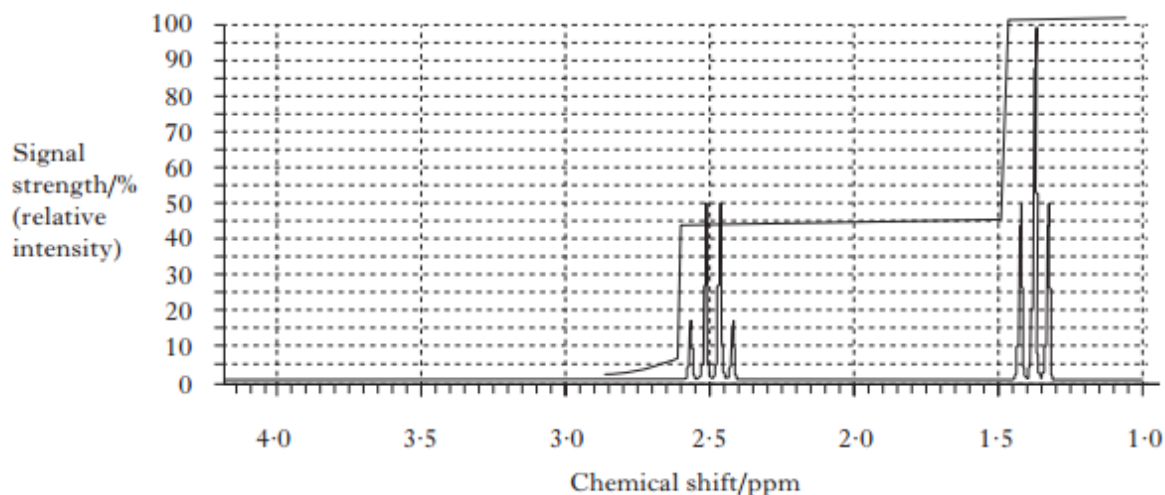   (b) The infra-red spectrum of compound **A** is shown below.

    Which bond is responsible for the peak at 1140 cm^{-1}?   **1**

    (c) The mass spectrum of compound **A** shows the molecular ion to have a mass/charge ratio of 74. Deduce the molecular formula of compound **A**.   **1**

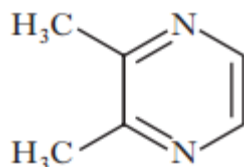   (d) The high resolution proton NMR spectrum of compound **A** is shown below.

    Using all the above information, deduce a structural formula for compound **A**.   **1**

7. The dominant flavours of chocolate are due to molecules called substituted pyrazines. These are produced when sugars and amino acids react during the roasting of cocoa beans.

An example is 2,3-dimethylpyrazine

This compound is responsible for the nutty flavour of chocolate. Other substances responsible for the distinctive smell of chocolate are aldehydes including phenylethanal, 2-methylbutanal and 3-methylbutanal.

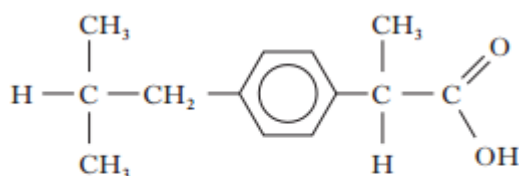(*c*) The low resolution proton NMR spectrum shown is that of one of the aldehydes in chocolate.
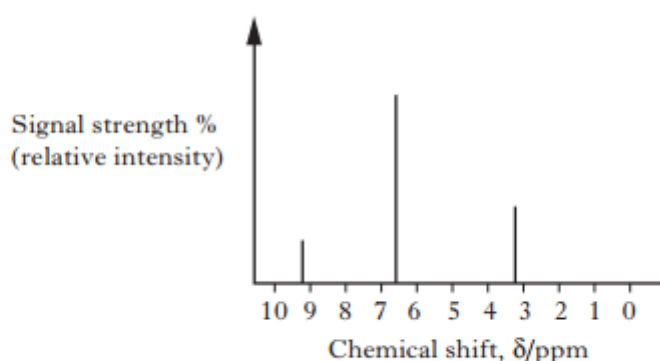
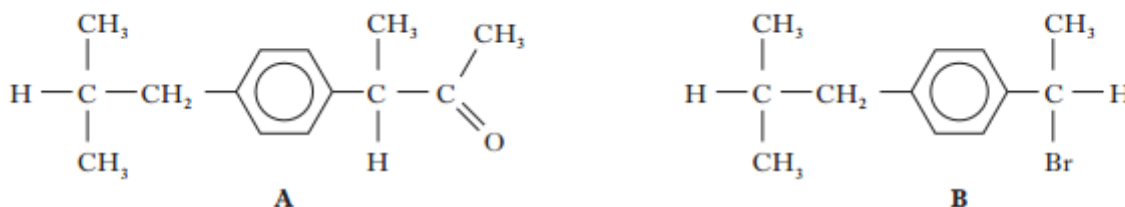Explain which of the three aldehydes above would give this proton NMR spectrum. **2**

8. Ibuprofen is one of the most commonly used non-steroidal anti-inflammatory drugs (NSAIDs).

The structure of ibuprofen is shown.

ibuprofen

(*b*) Compounds **A** and **B**, shown below, can be used to manufacture ibuprofen.

An impure sample of ibuprofen known to be contaminated with **one** of these compounds, was subjected to IR analysis and the major peaks were identified at wavenumbers 1600, 1690, 1720 and 3300 cm^{-1}.

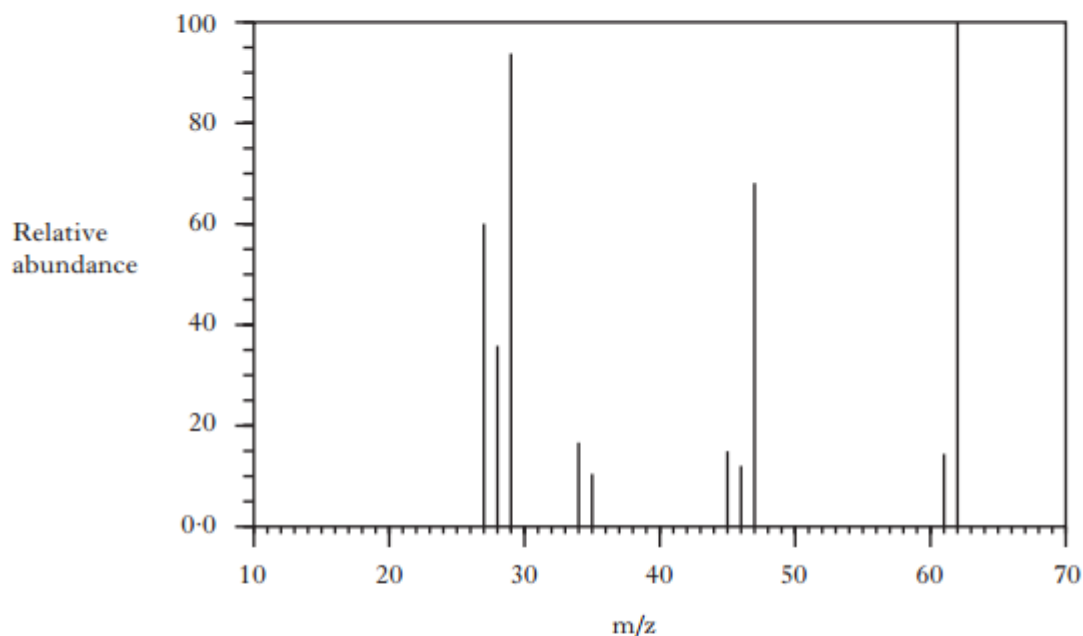(i) Explain which compound is present as an impurity. **1**

11. Compound **X** contains only carbon, hydrogen and sulphur.

(a) Complete combustion of **X** gave 3·52 g of carbon dioxide, 2·16 g of water and 2·56 g of sulphur dioxide.

Show, by calculation, that the empirical formula of compound **X** is C_2H_6S **2**

(b) The mass spectrum for compound **X** is shown below.

Suggest a possible ion fragment which may be responsible for the peak at m/z 47 in the mass spectrum.   **1**

(c) The results of the analysis of the proton NMR spectrum of **X** are shown in the table below.

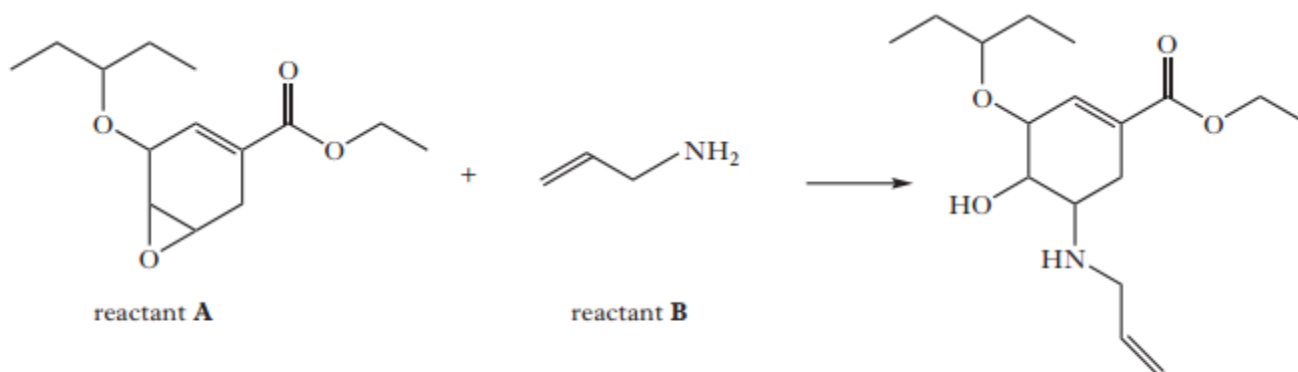| Peak	Chemical shift/ppm	Relative area under the peak
1	1·2	97
2	1·5	32
3	2·4	65

Considering all the evidence above, draw a structural formula for compound **X**.   **1**

11. (*a*) One of the main drugs used in the treatment of bird flu is Tamiflu. One step in the synthesis is shown below.

reactant **A** + reactant **B** →

In this reaction, reactant **A** has an electrophilic centre and reactant **B** has a nucleophilic centre.

(ii) Infra-red spectroscopy can be used to identify the product. The infra-red spectrum of the product has a major absorbance peak which is not present in the spectrum of either of the reactants.

In which wave number **range**, in cm^{-1}, will this absorbance peak be found? **1**

10. There are four isomers with the molecular formula C_4H_9Cl. Structural formulae for three of these isomers are

$CH_3CHClCH_2CH_3$ $(CH_3)_2CHCH_2Cl$ $(CH_3)_3CCl$
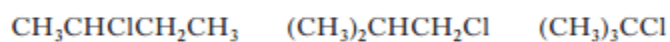
A   **B**   **C**

(*e*) Proton NMR spectroscopy can be used to distinguish between isomers **A, B** and **C** simply by counting the different numbers of peaks in each spectrum.

How many peaks would be seen in the spectrum of isomer **B**? **1**

4. During an Advanced Higher Chemistry Investigation in inorganic chemistry, a student deviates from the planned procedure and produces a pale yellow powder that the teacher suspects might be a new chemical.

Using your knowledge of chemistry suggest what the student might do to determine if it was a brand new substance. **(3)**

10. Phenylbutazone is an anti-inflammatory drug used for the short-term treatment of pain and fever in animals.

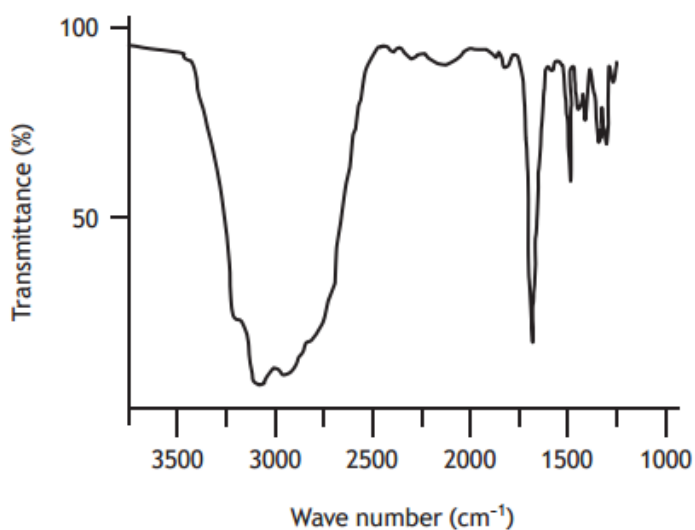(a) Phenylbutazone can be synthesised, in a multi-step process, starting from compound **A**.

Elemental microanalysis showed that compound **A** has a composition, by mass, of

$$50 \cdot 0\% \ C; \qquad 5 \cdot 60\% \ H; \qquad 44 \cdot 4\% \ O$$

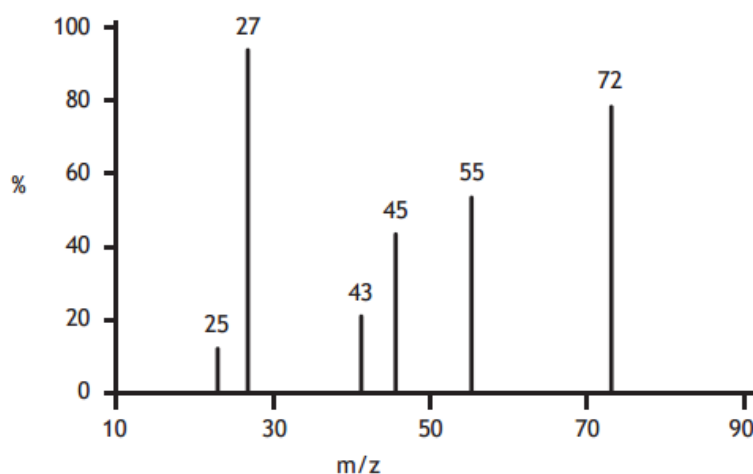Calculate the empirical formula of compound **A**.　　　　　　　**2**

(b) An infra-red spectrum for compound **A** is shown below.

Identify the functional group responsible for the peak at 1710 cm^{-1}.　　**1**

(c) The mass spectrum for compound **A** is shown below.

(i) Write the molecular formula for compound **A**.　　　　　　　**1**

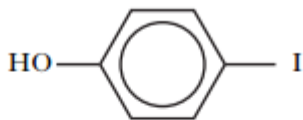(ii) Suggest a possible ion fragment that may be responsible for the peak at m/z 27.　　　　　　　**1**

(d) Considering all the evidence, draw a structural formula for compound **A**.　　**1**

17

2009 AH MC39 (60%) and 2015 AH MC38 (71%)

39.

HO———I

Which atom in the above structure would be located **most** readily using X-ray crystallography?

A   Carbon

B   Hydrogen

C   Iodine

D   Oxygen