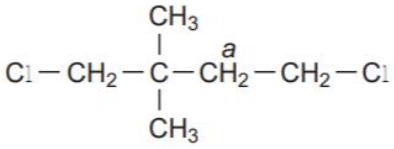
## CHAPTER 32 STRUCTURE DETERMINATION (NMR SPECTROSCOPY)

**1** N.m.r. spectroscopy can be used to study the structures of organic compounds.

**(a)** Compound **J** was studied using $^{1}H$ n.m.r. spectroscopy.



**J**

**(i)** Identify a solvent in which **J** can be dissolved before obtaining its $^{1}H$ n.m.r. spectrum.

.....

*(1 mark)*

**(ii)** Give the number of peaks in the $^{1}H$ n.m.r. spectrum of **J**.

.....

*(1 mark)*

**(iii)** Give the splitting pattern of the protons labelled *a*.

.....

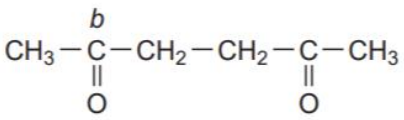
*(1 mark)*

**(iv)** Give the IUPAC name of **J**.

.....

*(1 mark)*

**(b)** Compound **K** was studied using $^{13}C$ n.m.r. spectroscopy.



**K**

**(i)** Give the number of peaks in the $^{13}C$ n.m.r. spectrum of **K**.

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*(1 mark)*

**(ii)** Use **Table 3** on the Data Sheet to suggest a $\delta$ value of the peak for the carbon labelled *b*.

.....

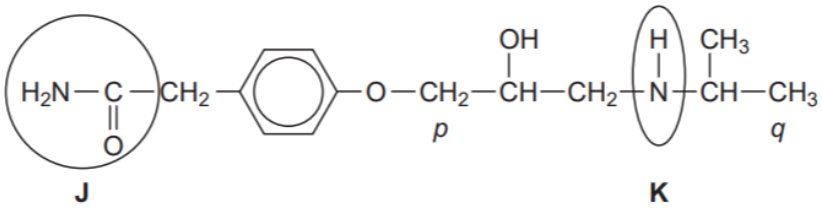
*(1 mark)*

**(iii)** Give the IUPAC name of **K**.

.....

*(1 mark)*

**2** Atenolol is an example of the type of medicine called a beta blocker. These medicines are used to lower blood pressure by slowing the heart rate. The structure of atenolol is shown below.




**(a)** Give the name of each of the circled functional groups labelled **J** and **K** on the structure of atenolol shown above.

Functional group labelled **J** .....

Functional group labelled **K** .....

*(2 marks)*

**(b)** The $^1$H n.m.r. spectrum of atenolol was recorded.

One of the peaks in the $^1$H n.m.r. spectrum is produced by the $CH_2$ group labelled *p* in the structure of atenolol.
Use **Table 2** on the Data Sheet to suggest a range of δ values for this peak.
Name the splitting pattern of this peak.

Range of δ values .....

Name of splitting pattern .....

*(2 marks)*

**(c)** N.m.r. spectra are recorded using samples in solution.
The $^1$H n.m.r. spectrum was recorded using a solution of atenolol in $CDCl_3$

**(i)** Suggest why $CDCl_3$ and **not** $CHCl_3$ was used as the solvent.

.....

.....

*(1 mark)*

**(ii)** Suggest why $CDCl_3$ is a more effective solvent than $CCl_4$ for polar molecules such as atenolol.

.....

.....

*(1 mark)*

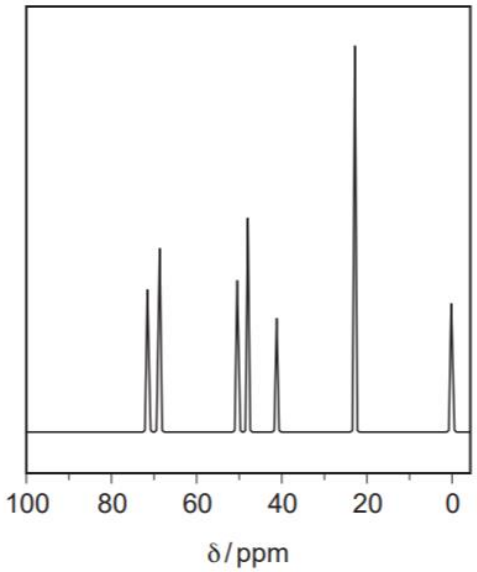
**(d)** The $^{13}C$ n.m.r. spectrum of atenolol was also recorded.

Use the structure of atenolol given to deduce the total number of peaks in the $^{13}C$ n.m.r. spectrum of atenolol.

.....

*(1 mark)*

**(e)** Part of the $^{13}C$ n.m.r. spectrum of atenolol is shown below. Use this spectrum and **Table 3** on the Data Sheet, where appropriate, to answer the questions which follow.




**(i)** Give the formula of the compound that is used as a standard and produces the peak at $\delta = 0$ ppm in the spectrum.

.....

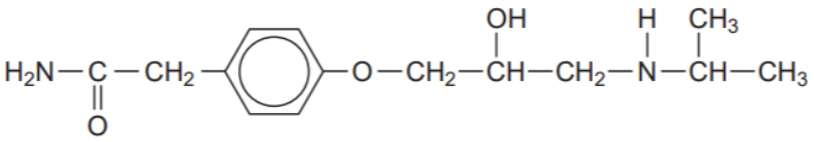
*(1 mark)*

**(ii)** One of the peaks in the $^{13}C$ n.m.r. spectrum above is produced by the $CH_3$ group labelled *q* in the structure of atenolol.
Identify this peak in the spectrum by stating its $\delta$ value.

.....

*(1 mark)*

**(iii)** There are three $CH_2$ groups in the structure of atenolol. One of these $CH_2$ groups produces the peak at $\delta = 71$ in the $^{13}C$ n.m.r. spectrum above.
Draw a circle around this $CH_2$ group in the structure of atenolol shown below.




*(1 mark)*

**(f)** Atenolol is produced industrially as a racemate (an equimolar mixture of two enantiomers) by reduction of a ketone. Both enantiomers are able to lower blood pressure. However, recent research has shown that one enantiomer is preferred in medicines.

**(i)** Suggest a reducing agent that could reduce a ketone to form atenolol.

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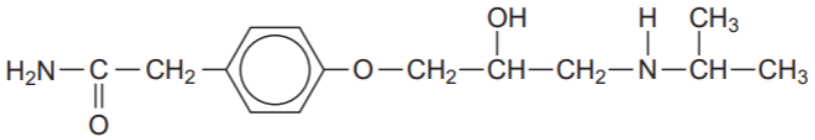
*(1 mark)*

**(ii)** Draw a circle around the asymmetric carbon atom in the structure of atenolol shown below.



*(1 mark)*

**(iii)** Suggest how you could show that the atenolol produced by reduction of a ketone was a racemate and **not** a single enantiomer.

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.....

.....

*(2 marks)*

**(iv)** Suggest **one** advantage and **one** disadvantage of using a racemate rather than a single enantiomer in medicines.

Advantage .....

.....

Disadvantage .....

.....

*(2 marks)*