

Chapter 8

Analysis

Print out and complete this worksheet to generate a summary for Chapter 8.

Chromatography

1 a This question is about thin-layer chromatography, TLC, and gas chromatography, GC. Identify the mobile and the stationary phases.

(i) TLC:

Mobile phase:

.....

Stationary phase:

.....

(ii) GC:

Mobile phase:

.....

Stationary phase:

.....

b Explain what is meant by:

(i) R_f value

.....

.....

(ii) retention time

.....

.....

c Explain the limitations of chromatography alone as an analytical tool.

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d (i) Explain the benefit of using gas chromatography in conjunction with mass spectrometry, i.e. GC-MS.

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(ii) State two uses of GC-MS.

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Chapter 8 Analysis

Nuclear magnetic resonance spectroscopy, NMR

2a Identify the region of the electromagnetic spectrum that is used in NMR spectroscopy.

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b Describe the use of tetramethylsilane in NMR. Give three reasons why it is suitable for its role.

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c Explain why ^{13}C and ^1H can both be detected by NMR.

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d Explain the need to use deuterated solvents such as CDCl_3 when running an NMR spectrum.

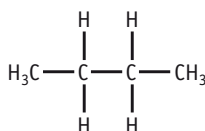
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 ^{13}C -NMR

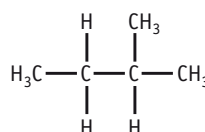
e Identify the number of peaks you would expect in the ^{13}C NMR spectrum of each of the following compounds:

(i)



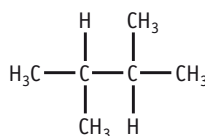
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(ii)



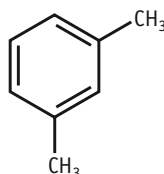
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(iii)



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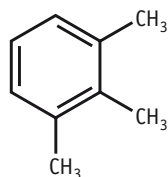
(iv)



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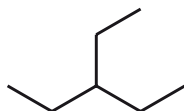
Chapter 8 Analysis

(v)



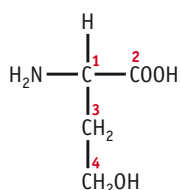
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(vi)



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f Predict the chemical shift values/ranges for each carbon atom in the following compound:

C₁: ppmC₂: ppmC₃: ppmC₄: ppm¹H NMR

g Explain what is meant by:

(i) chemical shift

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(ii) the (*n* + 1) rule

.....

(iii) relative peak areas

.....

(iv) labile protons

.....

h Identify **three** different functional groups that have labile protons.

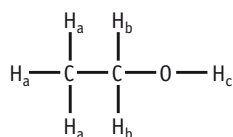
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Chapter 8 Analysis

- i Complete the table below, identifying the chemical shift range for each of the protons in bold.

| | | | | |
|----------------------|--------------------------------------------------------------------------|------------------------------------------------------------------------|-----------------------------------------------------------------------------|--------------------------------------------------------------|
| Chemical environment | CH₃ in CH ₃ CH ₂ CH ₃ | CH₃ in C ₆ H ₅ CH ₃ | CH₃ in CH ₃ CHO | CH₃ in CH ₃ COOH |
| δ/ppm (range) | | | | |
| Chemical environment | H in CH ₃ CH ₂ OH | H in CH ₃ COCH ₂ CH ₃ | H in CH ₃ COOH | H in C ₆ H ₅ OH |
| δ/ppm (range) | | | | |
| Chemical environment | CH₂ in CH ₃ CH ₂ CH ₃ | CH₂ in CH ₃ CH ₂ OH | CH₂ in CH ₃ COOCH ₂ CH ₃ | CH₂ in CH ₃ CH ₂ CHO |
| δ/ppm (range) | | | | |
| Chemical environment | H in CH ₃ CHO | CH₃ in CH ₃ OH | H in C ₆ H ₆ | H in CH ₃ CONH ₂ |
| δ/ppm (range) | | | | |

- j The different hydrogen environments in ethanol have been labelled H_a, H_b and H_c. Relevant data are given in the table and on the OCR data sheet.



| | Relative peak area | Splitting | Chemical shift/δ/ppm (range) |
|----------------|--------------------|-----------|------------------------------|
| H _a | 3 | Triplet | 0.7–1.6 |
| H _b | 2 | Quartet | 3.3–4.3 |
| H _c | 1 | Singlet | 1.0–5.3 |

- (i) Draw the structure of butan-2-ol, label the different hydrogen environments as H_a, H_b etc., and then complete the table.

| | Relative peak area | Splitting | Chemical shift/δ/ppm (range) |
|----------------|--------------------|-----------|------------------------------|
| H _a | | | |
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Chapter 8 Analysis

- (ii) Draw the structure of propyl methanoate, label the different hydrogen environments as H_a , H_b etc., and then complete the table.

| | Relative peak area | Splitting | Chemical shift/ δ /ppm (range) |
|-------|--------------------|-----------|---------------------------------------|
| H_a | | | |
| | | | |
| | | | |
| | | | |
| | | | |

- (iii) Draw the structure of 3-ethylpentane, label the different hydrogen environments as H_a , H_b etc., and then complete the table.

| | Relative peak area | Splitting | Chemical shift/ δ /ppm (range) |
|-------|--------------------|-----------|---------------------------------------|
| H_a | | | |
| | | | |
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Chapter 8 Analysis

- (iv) Draw the structure of penta-1,4-diene, label the different hydrogen environments as H_a , H_b etc., and then complete the table.

| | Relative peak area | Splitting | Chemical shift/ δ /ppm (range) |
|-------|--------------------|-----------|---------------------------------------|
| H_a | | | |
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