

# Chromatography

## Question Paper 2

<b>Level</b>	International A Level
<b>Subject</b>	Chemistry
<b>Exam Board</b>	CIE
<b>Topic</b>	Analytical techniques
<b>Sub-Topic</b>	Chromatography
<b>Paper Type</b>	Theory
<b>Booklet</b>	Question Paper 2

**Time Allowed:** 60 minutes

**Score:** /50

**Percentage:** /100

**Grade Boundaries:**

A*	A	B	C	D	E	U
>85%	77.5%	70%	62.5%	57.5%	45%	<45%

- 1 Different analytical techniques are used to build up a picture of complex molecules. Each technique on its own provides different information about complex molecules but together the techniques can give valuable structural information.

- (a) Complete the table, identifying the technique which can provide the appropriate structural information.

structural information	analytical technique
three-dimensional arrangement of atoms and bonds in a molecule	
chemical environment of protons in a molecule	
identity of amino acids present in a polypeptide	

[3]

- (b) One general method of separating organic molecules is chromatography. Briefly explain the chemical principles involved in each of the following techniques.

- (i) paper chromatography

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- (ii) thin-layer chromatography

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[2]

- (c) A combination of mass spectrometry and NMR spectroscopy is often enough to determine the structure of a simple organic compound.  
The organic compound **N** produced a mass spectrum in which the ratio of the M:M+1 peaks was 5.9:0.20, and which had an M+2 peak of similar height to the M peak.

(i) Calculate how many carbon atoms are present in one molecule of **N**.

(ii) Deduce which element, other than carbon and hydrogen, is present in **N**.

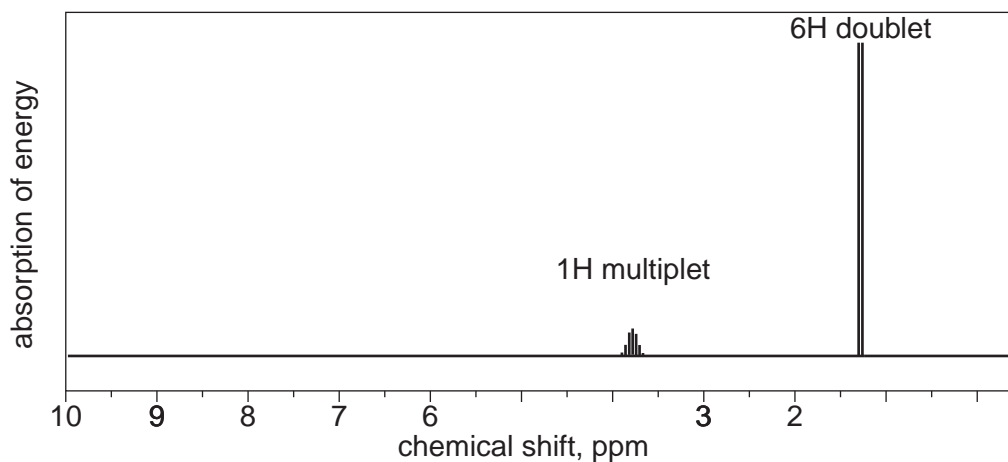
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(iii) Explain how many atoms of this element are present in one molecule of **N**.

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The NMR spectrum of **N** is shown.



(iv) State the empirical formula of **N** and, using the NMR data, suggest the structural formula of **N**, explaining your reasons.

[6]

[Total: 11]

2 Instrumental methods of analysis have become increasingly important in recent years. The use of chromatography to separate substances, and NMR spectroscopy to identify them, has become routine in many laboratories.

(a) Chromatography relies on either partition or adsorption to help separate substances.

(i) Briefly explain how each method brings about separation.

partition .....

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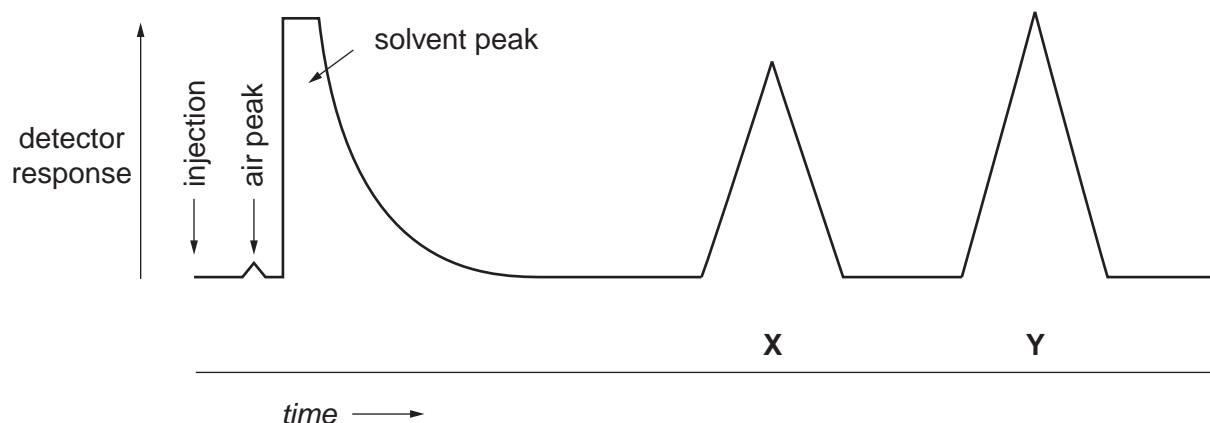
adsorption .....

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(ii) The table shows three different techniques of chromatography. Identify which separation method, *partition* or *adsorption*, applies to each.

technique	separation method
paper chromatography	
thin-layer chromatography	
gas/liquid chromatography	

(iii) The diagram represents the output from gas/liquid chromatography carried out on a mixture.



Determine the percentage of each of the two components **X** and **Y** in the mixture.

(b) NMR spectroscopy is a very important analytical technique for use with organic compounds.

(i) Why is NMR spectroscopy particularly useful for organic compounds?

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(ii) Two molecules, propanal and propanone, have the same molecular formula,  $C_3H_6O$ . Draw the displayed formula of each compound and explain briefly how NMR spectroscopy can distinguish between the two structures.

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[4]

[Total: 9]

3 A large number of organic compounds are soluble in both water and non-aqueous solvents such as hexane. If such a compound is shaken with a mixture of water and the non-aqueous solvent, it will dissolve in both solvents depending on the solubility in each.

(a) (i) State what is meant by the term *partition coefficient*.

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(ii) When  $100\text{ cm}^3$  of an aqueous solution containing  $0.50\text{ g}$  of an organic compound **X** was shaken with  $20\text{ cm}^3$  of hexane, it was found that  $0.40\text{ g}$  of **X** was extracted into the hexane.

Calculate the partition coefficient of **X** between hexane and water.

(iii) If **two**  $10\text{ cm}^3$  portions of hexane were used instead of a single  $20\text{ cm}^3$  portion, calculate the total amount of **X** extracted and compare this with the amount extracted using one  $20\text{ cm}^3$  portion.

**(b)** PCBs are highly toxic compounds released into the atmosphere when some plastics are burned at insufficiently high temperatures. In recent years PCB residues have been found in the breast milk of Inuit mothers in northern Canada. Foods, such as oily fish, seal and whale meat, which are high in fat, form an important part of the Inuit diet.

**(i)** Suggest why berries and drinking water are not contaminated by PCBs in the same way that oily fish, seal and whale meat are.

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**(ii)** Based on the information provided, what can you say about the partition coefficient between fat and water for PCB residues?

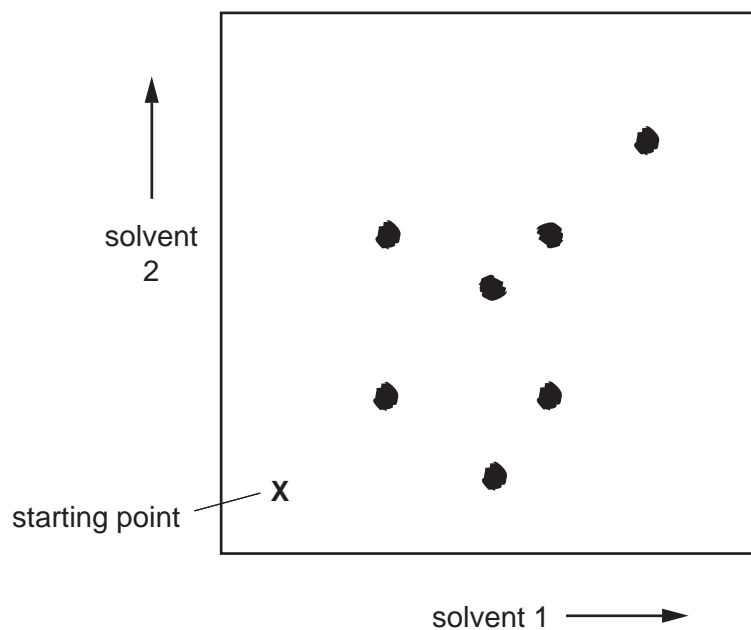
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[3]

(c) The diagram shows the result of two-way paper chromatography.



(i) How many spots were there after the first solvent had been used?

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(ii) Circle the spot that moved very little in solvent 2, but moved a greater distance in solvent 1.

(iii) Draw a square around the spot that could be separated from the rest by using **only** solvent 1.

[3]

[Total: 11]



4 Much of the preparation of evidence to solve crimes now relies on instrumental analysis. This question deals with some of the techniques used.

(a) Electrophoresis can be used to separate amino acids produced by hydrolysing proteins. The amino acids are placed in a buffered solution in an electric field. In a solution of given pH, what **two** factors affect the movement of a given amino acid?

(i) .....

(ii) .....

[2]

(b) Nuclear magnetic resonance (NMR) spectroscopy and mass spectrometry are also used in the detection of certain molecules, particularly those containing hydrogen atoms.

(i) Explain how and why the NMR spectrum of propanal,  $\text{CH}_3\text{CH}_2\text{CHO}$ , would be different from that of propanone,  $\text{CH}_3\text{COCH}_3$ , which contains the same atoms.

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(ii) Explain how and why the mass spectrum of the two compounds in (i) would be different.

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[4]

- (c) At one time, bromomethane,  $\text{CH}_3\text{Br}$ , was widely used to control insect pests in agricultural crops and timber. It is now known to break down in the stratosphere and contribute to the destruction of the ozone layer.

Samples can be screened for traces of bromomethane by subjecting them to mass spectrometry.

- (i) Which peak(s) would show the presence of bromine in the compound?

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- (ii) How could you tell by studying the M and M+2 peaks that the compound contained bromine rather than chlorine?

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[3]

[Total: 9]

- 5 (a) Electrophoresis can be used to separate amino acids which are produced by the hydrolysis of a polypeptide.

Using glycine as an example, explain why the result of electrophoresis depends on pH.

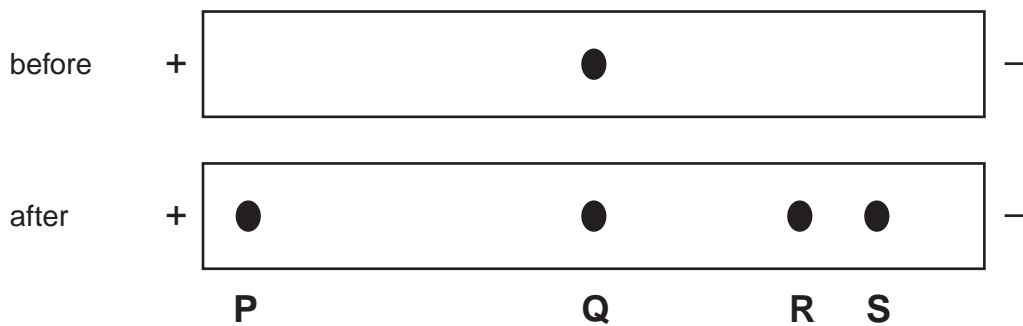
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- (b) The diagram below shows the results of electrophoresis in neutral solution. At the start of the experiment a spot of a solution containing a mixture of amino acids **P**, **Q**, **R** and **S** was placed in the middle of the plate. Following electrophoresis the amino acids had moved to the positions shown in the lower diagram.



- (i) Which amino acid existed mainly as a zwitterion in the buffer solution? Explain your answer.

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- (ii) Assuming amino acids **R** and **S** carry the same charge when in this buffer solution, which is likely to be the larger molecule? Explain your answer.

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[2]

- (c) Amino acids may also be separated by using two-dimensional paper chromatography. This involves putting a spot of the mixture on the corner of a piece of chromatography paper and allowing a solvent to soak up the paper. The paper is then dried, turned through 90° and placed in a second solvent. This method gives better separation than a one solvent method.

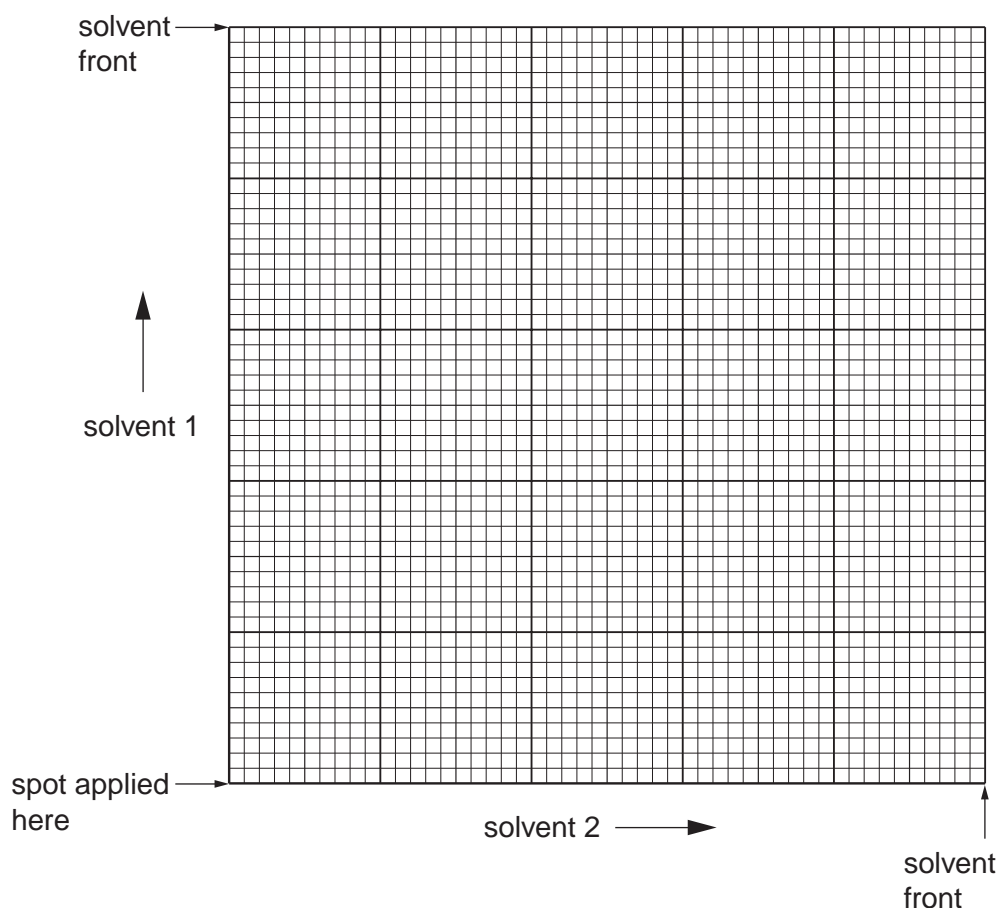
- (i) Paper chromatography relies on partition between the solvent applied and another phase.

What is this second phase? .....

- (ii) The table below shows the  $R_f$  values for some amino acids in two different solvents.

amino acid	$R_f$ solvent 1	$R_f$ solvent 2
<b>A</b>	0.1	0.2
<b>B</b>	0.0	0.4
<b>C</b>	0.3	0.0
<b>D</b>	0.8	0.9
<b>E</b>	0.6	0.5

Use the grid below to plot the positions of the amino acids after two-dimensional paper chromatography using solvent 1 followed by solvent 2.



- (iii) Which amino acid travelled fastest in **both** solvents? .....
- (iv) Which amino acid did not move at all in solvent 2? .....

[5]

[Total: 10]