AQA A-LEVEL CHEMISTRY · 7405

CHEMISTRY



EXAM QUESTION PACK

3.3.16 CHROMATOGRAPHY

SCORE	PERCENTAGE
/84	%

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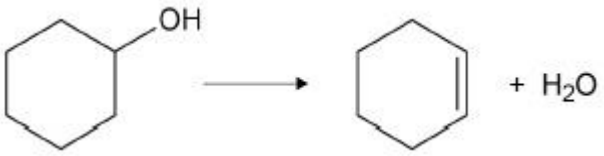
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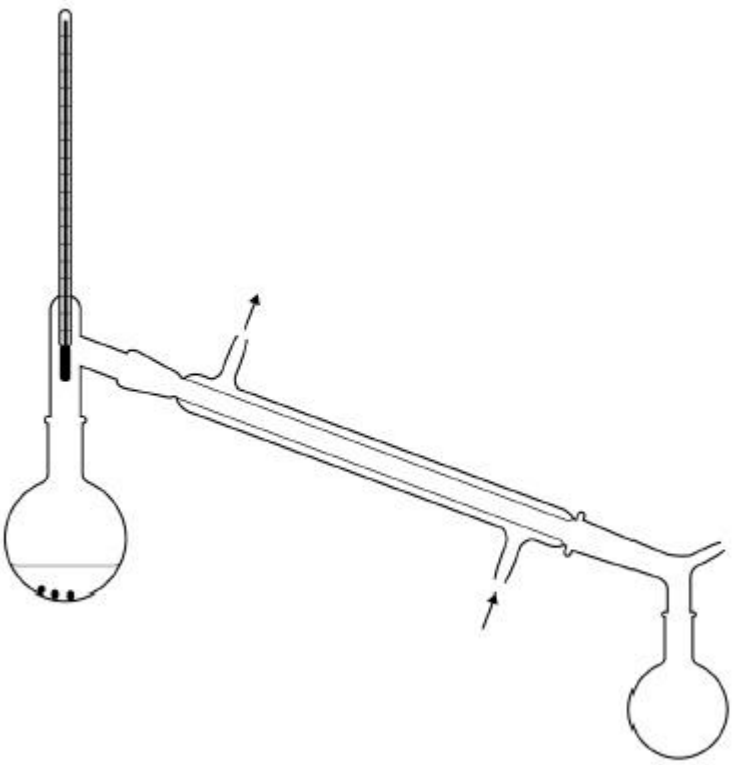
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Q1.

Cyclohexene (boiling point = 83 °C) can be prepared by the dehydration of cyclohexanol (boiling point = 161 °C) using concentrated phosphoric acid.



A student prepared cyclohexene by placing 10 cm^3 of cyclohexanol (density = 0.96 g cm^{-3}) into a round-bottomed flask.
3 cm^3 of concentrated phosphoric acid were then carefully added to the flask.
The student added a few anti-bumping granules and set up the apparatus shown in the diagram.



- The student heated the mixture and collected the liquid that distilled at temperatures below 100 °C
- The distillate was poured into a separating funnel and washed by shaking with sodium carbonate solution.
- Periodically, the separating funnel was inverted and the tap opened.
- The aqueous layer was discarded and the final organic product was dried using anhydrous calcium chloride.
- After the product was dried, the drying agent was removed by filtration under reduced pressure.

(a) The student collected 5.97 g of cyclohexene in the experiment.

Calculate the percentage yield of cyclohexene.

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Percentage yield _____ %

(3)

(b) Describe a test-tube reaction, on the product, to show that the cyclohexanol had been dehydrated.

State what you would observe. _____

_____

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_____

(2)

(c) Suggest why sodium carbonate solution was used to wash the distillate.

_____

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(1)

(d) Explain why it is important to open the tap of the separating funnel periodically.

_____

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(1)

(e) Give a property of anhydrous calcium chloride, other than its ability to absorb water, that makes it suitable as a drying agent in this preparation.

_____

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_____

(1)

(f) Describe the apparatus used to remove the drying agent by filtration under reduced pressure. Your description of the apparatus can be either a labelled diagram or a description in words.

(2)

(g) A sample of cyclohexene has been contaminated with cyclohexanol. The cyclohexene can be separated from the cyclohexanol by column chromatography. Silica gel is used as the stationary phase and hexane as the mobile phase.

Explain why cyclohexene has a shorter retention time than cyclohexanol.

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(2)

(h) Explain how an infrared spectrum would confirm that the cyclohexene obtained from the chromatography column did not contain any cyclohexanol.

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(1)

(Total 13 marks)

Q2.

This question is about nitrobenzenes.

(a) Nitrobenzene reacts when heated with a mixture of concentrated nitric acid and concentrated sulfuric acid to form a mixture of three isomeric dinitrobenzenes.

Write an equation for the reaction of concentrated nitric acid with concentrated sulfuric acid to form the species that reacts with nitrobenzene.

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(1)

(b) Name and outline a mechanism for the reaction of this species with nitrobenzene to form 1,3-dinitrobenzene.

Name of mechanism

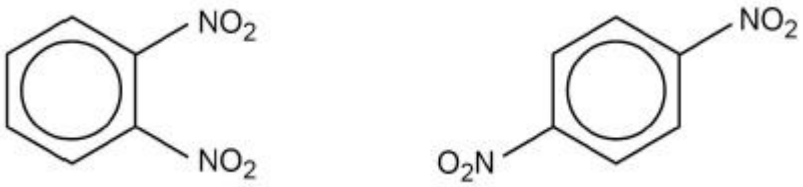
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Mechanism

(4)

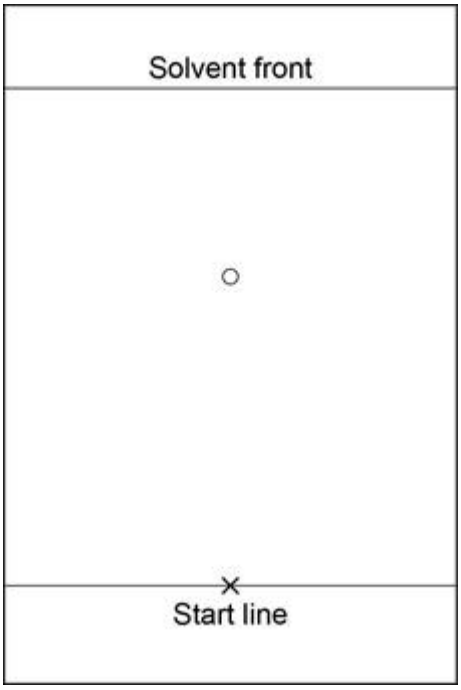
(c) The dinitrobenzenes shown were investigated by thin layer chromatography (TLC).

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In an experiment, carried out in a fume cupboard, a concentrated solution of pure 1,4-dinitrobenzene was spotted on a TLC plate coated with a solid that contains polar bonds. Hexane was used as the solvent in a beaker with a lid.

The start line, drawn in pencil, the final position of the spot and the final solvent front are shown on the chromatogram in the diagram below



Use the chromatogram in the diagram above to deduce the R^f value of 1,4-dinitrobenzene in this experiment.

Tick (✓) **one** box.

A 0.41 ⬭

B 0.46 ⬭

C 0.52 ⬭

D 0.62 ⬭

(1)

(d) State in general terms what determines the distance travelled by a spot in TLC.

_____

_____

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_____

(1)

(e) To obtain the chromatogram, the TLC plate was held by the edges and placed in the solvent in the beaker in the fume cupboard. The lid was then replaced on the beaker.

Give one other practical requirement when placing the plate in the beaker.

_____

_____

_____

(1)

(f) A second TLC experiment was carried out using 1,2-dinitrobenzene and 1,4-dinitrobenzene. An identical plate to that in part (c) was used under the same conditions with the same solvent. In this experiment, the Rf value of 1,4-dinitrobenzene was found to be greater than that of 1,2-dinitrobenzene.

Deduce the relative polarities of the 1,2-dinitrobenzene and 1,4-dinitrobenzene and explain why 1,4-dinitrobenzene has the greater R_f value.

Relative polarities

_____

Explanation

_____

_____

_____

(2)

(g) A third TLC experiment was carried out using 1,2-dinitrobenzene. An identical plate to that in part (c) was used under the same conditions, but the solvent used contained a mixture of hexane and ethyl ethanoate.

A student stated that the R_f value of 1,2-dinitrobenzene in this third experiment would be greater than that of 1,2-dinitrobenzene in the experiment in part (f)

Is the student correct? Justify your answer.

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(2)

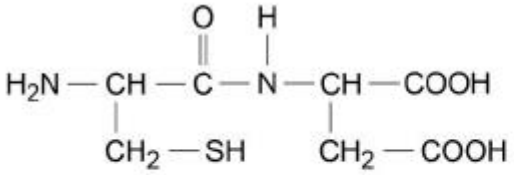
(Total 12 marks)

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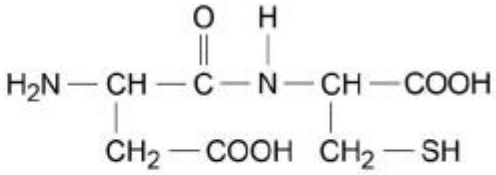
Q3.

Chromatography is used to identify amino acid sequences in compounds.

The dipeptide cysteine-aspartic acid (cys-asp), **J**, and the dipeptide aspartic acid-cysteine (asp-cys), **K**, are shown.



J (cys–asp)



K (asp–cys)

(a) A mixture of the two dipeptides **J** and **K** is analysed by gas chromatography followed by mass spectrometry (GC-MS).

Explain why **J** and **K** can be separated by gas chromatography and why mass spectrometry using electrospray ionisation does **not** enable you to identify them.

Gas chromatography explanation _____

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Mass spectrometry explanation _____

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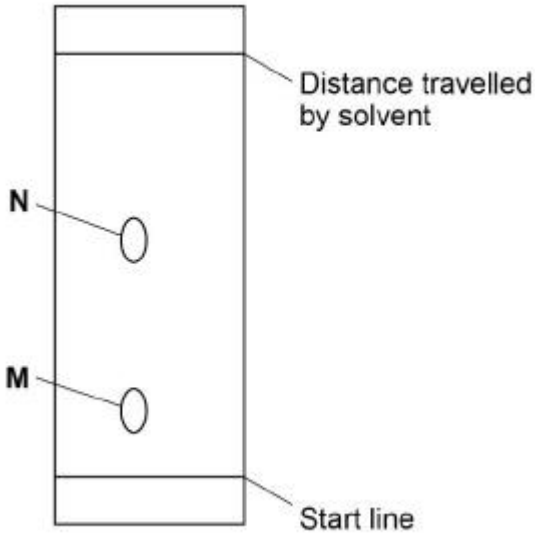
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(4)

(b) A tripeptide, **L**, is partially hydrolysed with concentrated hydrochloric acid to produce two dipeptides and the amino acids alanine (ala), lysine (lys) and serine (ser).

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The two dipeptides are separated by chromatography. The diagram below shows the chromatogram.



The table below contains the R_f values of some dipeptides.

Dipeptide	ala-lys	ala-ser	lys-ser	lys-ala	ser-ala	ser-lys
R_f value	0.55	0.85	0.10	0.20	0.15	0.45

Use the chromatogram in the diagram above and the R_f values in the table to identify the two dipeptides present in spots **M** and **N**.

Use your answers to deduce the order of the amino acids in the tripeptide **L**.

Dipeptide responsible for spot **M** _____

_____

Dipeptide responsible for spot **N** _____

_____

Order of amino acids in tripeptide **L** _____

_____

(3)

(Total 7 marks)

Q4.

A peptide is hydrolysed to form a solution containing a mixture of amino acids. This mixture is then analysed by silica gel thin-layer chromatography (TLC) using a toxic solvent. The individual amino acids are identified from their R_f values.

Part of the practical procedure is given below.

1. **Wearing plastic gloves to hold a TLC plate**, draw a pencil line 1.5 cm from the

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bottom of the plate.

2. Use a capillary tube to apply a very small drop of the solution of amino acids to the mid-point of the pencil line.
3. Allow the spot to dry completely.
4. In the developing tank, add the developing solvent to **a depth of not more than 1 cm**.
5. Place your TLC plate in the developing tank.
6. Allow the developing solvent to rise up the plate **to the top**.
7. Remove the plate and quickly mark the position of the solvent front with a pencil.
8. Allow the plate to dry **in a fume cupboard**.

(a) Parts of the procedure are in bold text.

For each of these parts, consider whether it is essential and justify your answer.

(4)

(b) Outline the steps needed to locate the positions of the amino acids on the TLC plate and to determine their R_f values.

(4)

(c) Explain why different amino acids have different R_f values.

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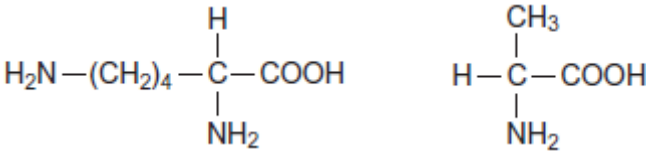
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(2)

(Total 10 marks)

Q5.

Lysine and alanine are two amino acids.



lysine

alanine

(a) Give the IUPAC name of lysine.

_____

(1)

(b) Draw structures to show the product formed in each case when lysine reacts with

(i) an excess of aqueous HCl

(1)

(ii) an excess of aqueous NaOH

(1)

(iii) methanol in the presence of a small amount of concentrated H_2SO_4

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(1)

(c) The mass spectrum of alanine gives a major peak at $m / z = 44$

Write an equation for the fragmentation of the molecular ion of alanine to give an ion that produces this peak.
In your answer, draw the displayed formula for this fragment ion.

(2)

(d) Draw a dipeptide formed from one molecule of lysine and one molecule of alanine.

(1)

(e) The dipeptide in part (d) is hydrolysed in acid conditions and the mixture produced is analysed by column chromatography. The column is packed with a resin which acts as a polar stationary phase.

Suggest why lysine leaves the column after alanine.

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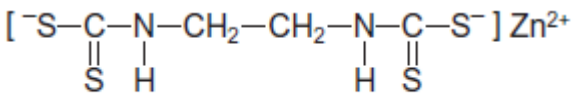
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(2)

(Total 9 marks)

Q6.

(a) Because of the toxic nature of the copper(II) ion, a wide range of alternative anti-fungal drugs has been developed for use in agriculture. One example is Zineb.



Zineb

(i) The negative ion in Zineb could act as a bidentate ligand.

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On the structure above, draw a ring around each of **two** atoms that could provide the lone pairs of electrons when this ion acts as a bidentate ligand.

(1)

(ii) Calculate the M_r of Zineb. Give your answer to the appropriate precision.

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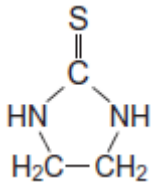
(1)

(iii) Name the functional group formed at each end of the negative ion when all the sulfur atoms in the structure of Zineb are replaced by oxygen atoms.

_____

(1)

(b) Zineb has been investigated for harmful effects. Generally, Zineb has been found to be safe to use in agriculture. It is only slightly soluble in water and is sprayed onto plants. A breakdown product of Zineb is ethylene thiourea (ETU), which is very soluble in water. The structure of ETU is shown below.



Determine the percentage, by mass, of sulfur in ETU (M_r = 102.1).

_____

_____

_____

(1)

(c) Chromatography is a technique used to show the presence of a small amount of ETU in Zineb.

Outline how this technique is used to separate and identify ETU from a sample of Zineb powder.

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(4)
(Total 8 marks)

Q7.

(a) A chemist discovered four unlabelled bottles of liquid, each of which contained a different pure organic compound. The compounds were known to be propan-1-ol, propanal, propanoic acid and 1-chloropropane.

Describe four **different** test-tube reactions, one for each compound, that could be used to identify the four organic compounds.
Your answer should include the name of the organic compound, the reagent(s) used and the expected observation for each test.

(8)

(b) A fifth bottle was discovered labelled propan-2-ol. The chemist showed, using infrared spectroscopy, that the propan-2-ol was contaminated with propanone.

The chemist separated the two compounds using column chromatography. The column contained silica gel, a polar stationary phase.

The contaminated propan-2-ol was dissolved in hexane and poured into the column. Pure hexane was added slowly to the top of the column. Samples of the eluent (the solution leaving the bottom of the column) were collected.

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- Suggest the chemical process that would cause a sample of propan-2-ol to become contaminated with propanone.
- State how the infrared spectrum showed the presence of propanone.
- Suggest why propanone was present in samples of the eluent collected first (those with shorter retention times), whereas samples containing propan-2-ol were collected later.

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(4)

(Total 12 marks)

Q8.

(a) Name compound **Y**, $HOCH_2CH_2COOH$

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(1)

(b) Under suitable conditions, molecules of **Y** can react with each other to form a polymer.

(i) Draw a section of the polymer showing **two** repeating units.

(1)

(ii) Name the type of polymerisation involved.

_____

(1)

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(c) When **Y** is heated, an elimination reaction occurs in which one molecule of **Y** loses one molecule of water. The organic product formed by this reaction has an absorption at 1637 cm^{-1} in its infrared spectrum.

(i) Identify the bond that causes the absorption at 1637 cm^{-1} in its infrared spectrum.

_____

(1)

(ii) Write the displayed formula for the organic product of this elimination reaction.

(1)

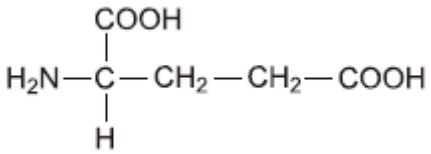
(iii) The organic product from part (ii) can also be polymerised.
Draw the repeating unit of the polymer formed from this organic product.

(1)

(d) At room temperature, 2-aminobutanoic acid exists as a solid.
Draw the structure of the species present in the solid form.

(1)

(e) The amino acid, glutamic acid, is shown below.



Draw the structure of the organic species formed when glutamic acid reacts with each of the following.

(i) an excess of sodium hydroxide

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(1)

(ii) an excess of methanol in the presence of concentrated sulfuric acid

(1)

(iii) ethanoyl chloride

(1)

(f) A tripeptide was heated with hydrochloric acid and a mixture of amino acids was formed. This mixture was separated by column chromatography.
Outline briefly why chromatography is able to separate a mixture of compounds.
Practical details are **not** required.

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(3)

(Total 13 marks)

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