

AQA A-LEVEL CHEMISTRY • 7405

CHEMISTRY



EXAM QUESTION PACK

3.3.16 CHROMATOGRAPHY

SCORE

/84

PERCENTAGE

%

STEMUP

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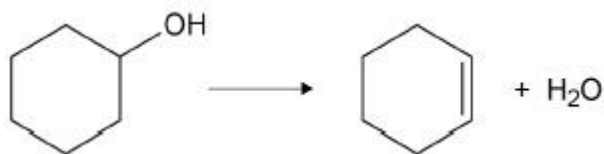
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Q1.

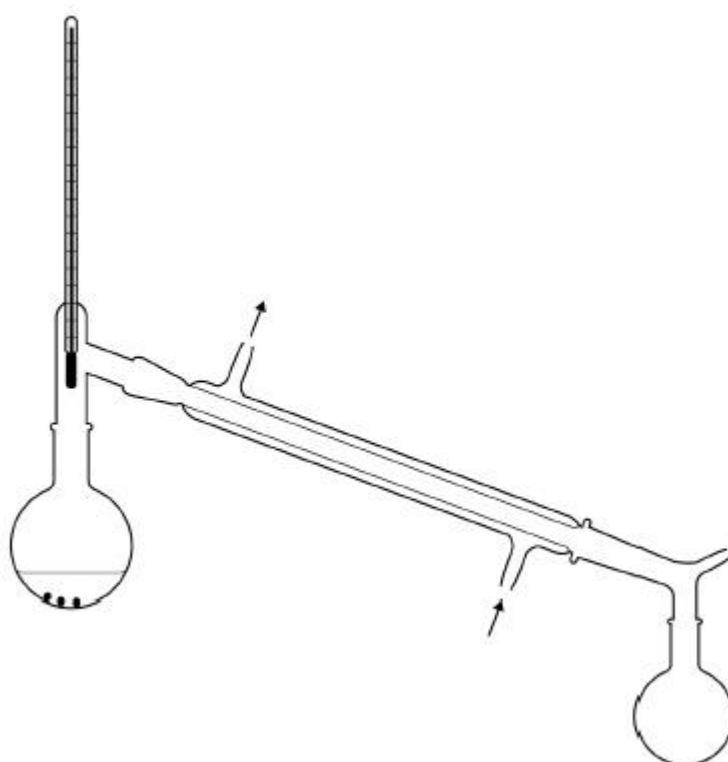
Cyclohexene (boiling point = 83 °C) can be prepared by the dehydration of cyclohexanol (boiling point = 161 °C) using concentrated phosphoric acid.



A student prepared cyclohexene by placing 10 cm³ of cyclohexanol (density = 0.96 g cm⁻³) into a round-bottomed flask.

3 cm³ 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.

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. _____

(2)

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

(1)

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

(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.

(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.

(2)

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

(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.

(1)

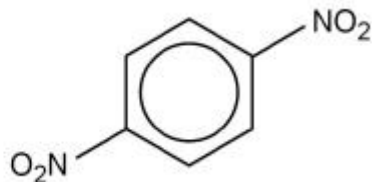
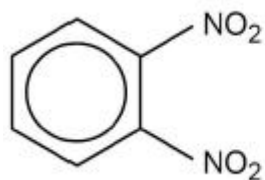
- (b) Name and outline a mechanism for the reaction of this species with nitrobenzene to form 1,3-dinitrobenzene.

Name of mechanism

Mechanism

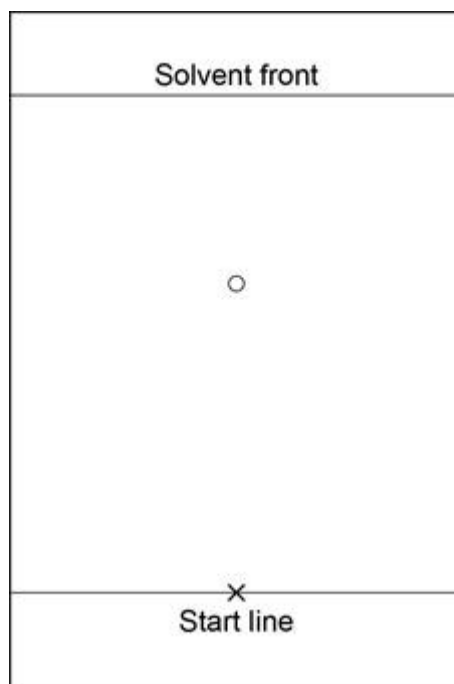
(4)

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



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.

(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 R_f 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.

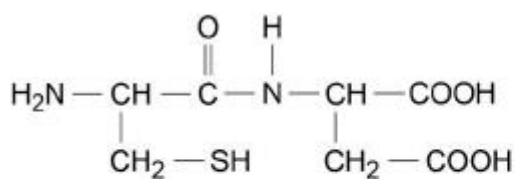
(2)

(Total 12 marks)

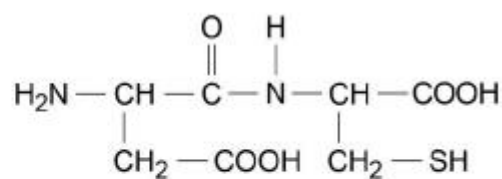
Q3.

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.

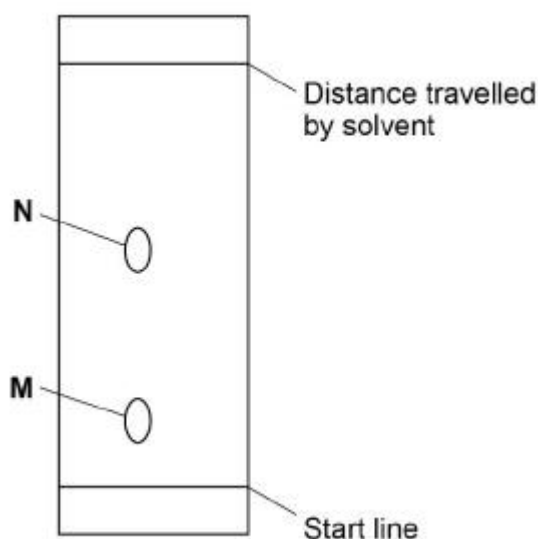
Gas chromatography explanation _____

Mass spectrometry explanation _____

(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).

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

- 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)

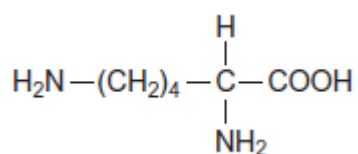
- (c) Explain why different amino acids have different R_f values.

(2)

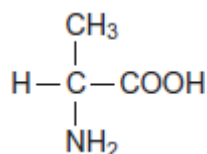
(Total 10 marks)

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

(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.

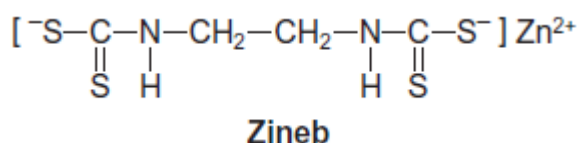
Suggest why lysine leaves the column after alanine.

(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.



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

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)

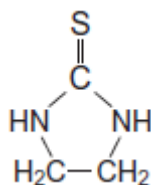
- (ii) Calculate the M_r of Zineb. Give your answer to the appropriate precision.

(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.

(4)

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.

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.

- 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.

(4)

(Total 12 marks)

Q8.

- (a) Name compound **Y**, $\text{HOCH}_2\text{CH}_2\text{COOH}$

(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)

- (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)

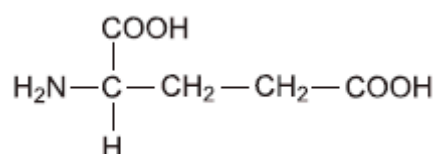
- (iii) The organic product from part (ii) can also be polymerised.
Draw the repeating unit of the polymer formed from this organic product.

(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

(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.

(3)

(Total 13 marks)

Mark schemes

Q1.

- (a) **M1** Moles of cyclohexanol = $(10 \times 0.96)/100.0 = 0.096$

Correct answer scores all 3 marks

1

- M2** Max mass of cyclohexene = $0.096 \times 82.0 = 7.87(2)$
= $M1 \times 82.0$ (process mark)

1

- M3** % yield = $(5.97 / 7.87) \times 100 = 76\%$ (Allow range 75.8 – 76)
= $(5.97 / M2) \times 100$ (process mark)

1

Alternative method

- M1** Moles of cyclohexanol = $(10 \times 0.96)/100.0 = 0.096$

- M2** Moles of cyclohexene = $5.97/82.0 = 0.0728$

- M3** % yield = $0.0728 / 0.096 \times 100 = 76\%$ (allow range 75.8 – 76)
= $(M2 / M1) \times 100$
Allow 1/3 for 62(.2)%

- (b) Add bromine (water)

If M1 not correct then only allow M2 if reagent involves bromine (water)

1

Would turn (from orange to) colourless / decolourise

Do not allow incorrect starting colour, but allow brown/red/yellow

Not discolour.

Ignore clear

1

- (c) Na_2CO_3 would neutralise/react with/remove (phosphoric) acid/ $\text{H}_3\text{PO}_4/\text{H}^+$

1

- (d) avoid pressure build-up / release pressure / release CO_2 /air/gas / prevent stopper blowing out

Ignore explosion

Do not allow an incorrect named gas

Allow idea that build-up of gas/ CO_2 would lead to increased pressure/stated effect of increased pressure

1

- (e) Does not dissolve in/react with the cyclohexene

Allow remains a solid/is inert in cyclohexene

Allow organic product/organic compound formed/ organic layer/distillate instead of cyclohexene

Do not allow if answer implies cyclohexanol

Do not allow if answer says does not react with products
Ignore references to filtration
Do not allow insoluble/unreactive unless qualified by implied reference to cyclohexene

1

(f) If diagram drawn:

M1 diagram of basic set up to include flask or tube with side-arm/Buchner flask, flat-bottomed funnel/Buchner funnel, filter paper

M2 apparatus should work, flow through, air-tight connection between flask and funnel, arrow/label/description (to vacuum pump)

Do not allow "standard" Y-shaped funnel

1

If description given:

M1 Buchner funnel/flat-bottomed funnel containing filter paper

M2 Buchner flask/side-arm flask connected to vacuum pump

Do not allow just "funnel"

Penalise M2 if described apparatus would not actually work.

1

(g) Cyclohexene is less polar than cyclohexanol / cyclohexanol is more polar than cyclohexene

It = cyclohexene

Allow cyclohexene is non-polar and cyclohexanol is polar

1

Cyclohexene has a greater affinity/attraction for the mobile phase/hexane / cyclohexanol has a greater affinity/attraction for the stationary phase/silica

Allow cyclohexanol held in the stationary phase for longer

Allow cyclohexene is more soluble in the mobile phase/hexane or converse for cyclohexanol

Allow references to hydrogen bonds between cyclohexanol and silica

1

(h) Would be no peak at 3230 – 3550 cm⁻¹ due to O—H((alcohol))

OR

There would be no additional peaks in the fingerprint region compared to a pure sample / fingerprint region exactly matches cyclohexene

Need wavenumber and bond for mark

1

[13]

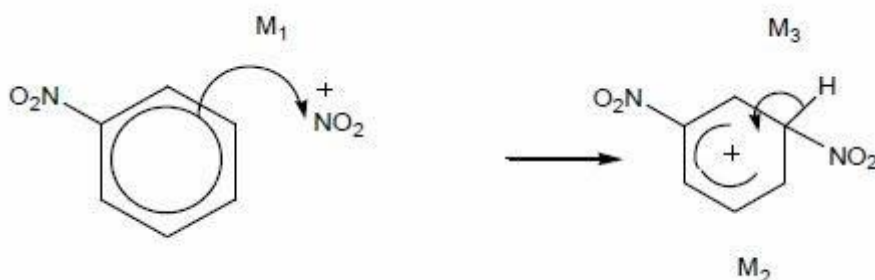
Q2.

(a) $\text{HNO}_3 + 2\text{H}_2\text{SO}_4 \rightarrow \text{NO}_2^+ + \text{H}_3\text{O}^+ + 2\text{HSO}_4^-$

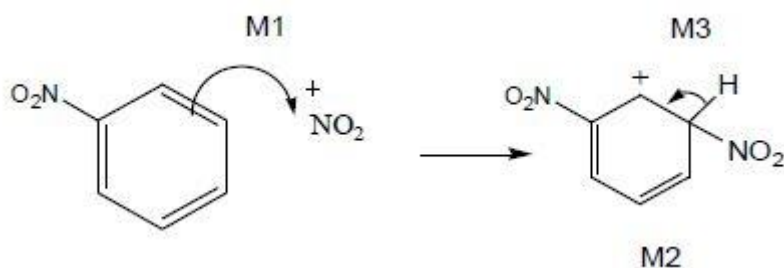
Allow $\text{H}_2\text{SO}_4 + \text{HNO}_3 \rightarrow \text{NO}_2^+ + \text{HSO}_4^- + \text{H}_2\text{O}$

Allow a combination of equations which produce NO_2^+

- (b) Electrophilic substitution.
Ignore nitration



OR Kekule



M1 Arrow from inside hexagon to N or + on N (Allow NO_2^+)

M2 Structure of intermediate

- horseshoe centred on C1 and must not extend beyond C2 and C6, but can be smaller
- + in intermediate not too close to C1 (allow on or "below" a line from C2 to C6)

M3 Arrow from bond into hexagon (Unless Kekule)

- Allow M3 arrow independent of M2 structure
- + on H in intermediate loses M2 not M3

- (c) D

- (d) (Balance between) solubility in moving phase and retention by stationary phase

OR (relative) affinity for stationary / solid and mobile / liquid / solvent (phase)

- (e) Solvent depth must be below start line
Ignore safety

- (f) 1,2- is more polar **OR** 1,4- is less polar
OR 1,2 is polar, 1,4- is non-polar

1,4- (or Less/non polar is) less attracted to (polar) plate / stationary phase / solid
OR (Less/non polar is) more attracted to / more soluble in (non-polar) solvent /
 mobile phase / hexane

1

M2 dependent on correct M1

If M1 is blank then read explanation for possible M1 and M2

Allow converse argument for 1,2

(g) No CE = 0

Yes - mark on but there is **NO MARK FOR YES**

Mark independently following yes

Solvent (more) polar or ethyl ethanoate is polar

1

Polar isomer more attracted to / more soluble in / stronger affinity to the solvent
 (than before)

Penalise bonded to mobile phase in M2

1

[12]

Q3.

(a) **Gas chromatography explanation**

Different retention times / dipeptides appear at different times.

1

Different balance between solubility in the moving phase / gas carrier **and** retention
 by the stationary phase / column **OR** different relative affinity for mobile and
 stationary phases.

1

Mass spectrometry explanation

Same m/z values.

1

(Both) dipeptides / **J** and **K** have same molecular formula / M_r .

1

(b) ser-ala

1

ala-lys

1

ser-ala-lys

This order only.

1

[7]

Q4.

(a) **Wear plastic gloves:**

Essential – to prevent contamination from the hands to the plate

1

Add developing solvent to a depth of not more than 1 cm³:

Essential – if the solvent is too deep it will dissolve the mixture from the plate

1

Allow the solvent to rise up the plate to the top:

Not essential – the R_f value can be calculated if the solvent front does not reach the top of the plate

1

Allow the plate to dry in a fume cupboard:

Essential – the solvent is toxic

Allow hazardous

1

- (b) Spray with developing agent or use UV

1

Measure distances from initial pencil line to the spots (x)

1

Measure distance from initial pencil line to solvent front line (y)

1

R_f value = x / y

1

- (c) Amino acids have different polarities

1

Therefore, have different retention on the stationary phase or different solubility in the developing solvent

1

[10]

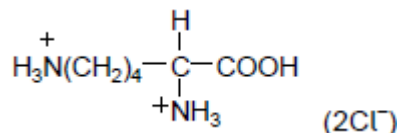
Q5.

- (a) 2,6-diaminohexanoic acid

Ignore additional , or – or spaces.

1

- (b) (i)



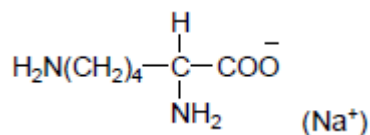
NB both N must be protonated.

Allow $-\text{NH}_3^+$ allow CO_2H Allow $-\text{H}_3\text{N}$.

Penalise – C_4H_8 – here.

1

- (ii)



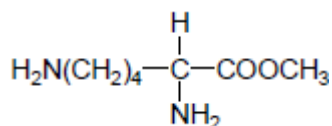
Allow CO_2^- .

Allow $-\text{H}_2\text{N}$.

Allow $-\text{COONa}$ but penalise $\text{O}-\text{Na}$ bond shown.

1

(iii)

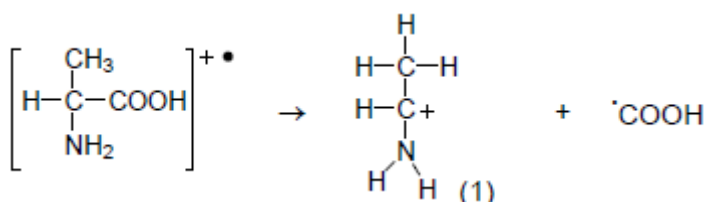


Allow CO_2CH_3 .

Allow $-\text{NH}_3^+$ or $-\text{H}_2\text{N}$.

1

(c)



1 for displayed formula of fragment ion.

1 for molecular ion of alanine AND radical.

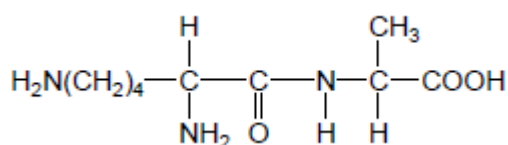
Allow molecular ion without brackets and fragment ion in brackets with outside +.

Allow dot anywhere on radical.

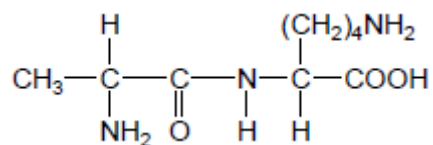
Allow $[\text{C}_3\text{H}_7\text{NO}_2]^+ \cdot$ for molecular ion.

2

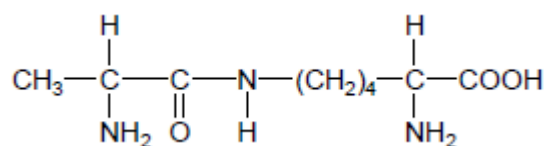
(d)



OR



OR



Dipeptide, not repeating unit /.

Allow CO_2H Allow $-\text{H}_2\text{N}$.

Allow $-\text{CONH}-$.

- (e) M1 In acid lysine has double positive or more positive charge 1
- M2 (Lysine ion) has greater affinity / greater attraction / adheres better / sticks better to polar / stationary phase 1
- M2 only scores after a correct M1.
- Ignore greater retention time. 1

[9]

Q6.

- (a) (i) Two rings only around nitrogen or sulfur 1
- Lose this mark if more than 2 atoms are ringed.
- Do not allow two atoms at the same end of the ion. 1
- (ii) 275.8 1
- Accept this answer only. Do not allow 276 1
- (iii) Carboxylate / COO^- 1
- Allow salt of carboxylic acid or just carboxylic acid. 1
- (b) $(32.1 / 102.1) = 31.4\%$ 1
- Do not penalise precision but do not allow 1 significant figure. 1
- (c) Zineb is mixed with a solvent / water 1
- Max=2 if M1 missed 1

Use of column / paper / TLC 1

Lose M1 and M2 for GLC 1

Appropriate collection of the ETU fraction 1

OR Appropriate method of detecting ETU

Allow ETU is an early fraction in a column or collecting a range of samples over time, lowest retention time / travels furthest on paper or TLC (allow 1 mark for having the longest retention time in GLC). 1

Method of identification of ETU (by comparison with standard using chromatography) 1

If method completely inappropriate, only M1 is accessible 1

[8]

Q7.

- (a) **If 2 stage test for one compound, award no marks for that compound, eg no mark for ROH or RX to alkene then Br₂ test. If reagent is wrong or missing, no mark for that test; if wrong but close/incomplete, lose reagent mark but can award for correct observation. In each test, penalise each example of wrong chemistry, eg AgClr₂**

propan-1-ol

acidified
potassium
dichromate

sodium

Named acid + conc H₂SO₄

named acyl chloride

PCl₅

1 M1

(orange) turns green

effervescence

Sweet smell

Sweet smell /misty fumes

Misty fumes

1 M2

propanal

add Tollens or Fehlings / Benedicts

acidified
potassium
dichromate

Bradys or 2,4-dnph

if dichromate used for alcohol cannot be used for aldehyde

1 M3

Tollens: silver mirror or Fehlings/ Benedicts: red ppt

(orange) turns green

Yellow or orange ppt

1 M4

propanoic acid

Named carbonate/ hydrogencarbonate

water and UI (paper)

Named alcohol + conc H_2SO_4

sodium or magnesium

PCl_5

if sodium used for alcohol cannot be used for acid

1 M5

effervescence

orange/red

Sweet smell

effervescence

Misty fumes

if PCl_5 used for alcohol cannot be used for acid

1 M6

1-chloro propane

NaOH then acidified AgNO_3

AgNO_3

*If acidification missed after NaOH ,
no mark here but allow mark for observation*

1 M7

white ppt

white ppt

1 M8

(b) oxidation (of alcohol by oxygen in air)

1 M1

absorption at 1680 -1750 (due to $\text{C}=\text{O}$)

Must refer to the spectrum

1 M2

comparison of polarity of molecules or correct imf statement:
 propanone is less polar OR propan-2-ol is more polar
 OR propanone has dipole-dipole forces
 OR propan-2-ol has hydrogen bonding

M3

1

about attraction to stationary phase or solubility in moving phase
 Propan-2-ol has greater affinity for stationary phase or vice versa
 OR propanone is more soluble in solvent/moving phase or vice versa

M4

1

[12]

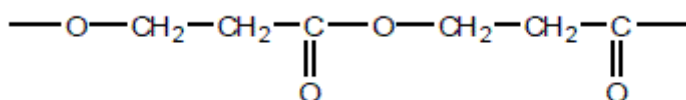
Q8.

- (a) 3-hydroxypropanoic acid

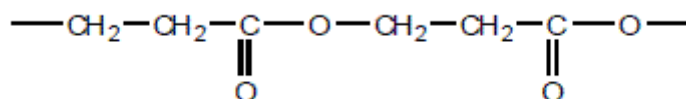
*allow 3-hydroxypropionic acid
 must be correct spelling*

1

- (b) (i) must show trailing bonds



or can start at any point in the sequence, e.g.



not allow dimer

allow -O-CH₂CH₂COOCH₂CH₂CO-

or -CH₂CH₂COOCH₂CH₂COO-

ignore () or n

NB answer has a total of 6 carbons and 4 oxygens

1

- (ii) condensation (polymerisation)

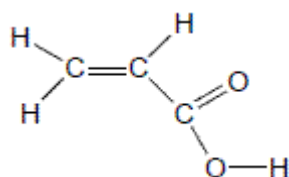
Allow close spelling

1

- (c) (i) C=C or carbon-carbon double bond

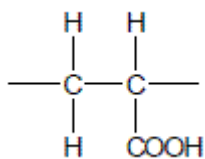
1

- (ii)



*must show **ALL** bonds including O-H*

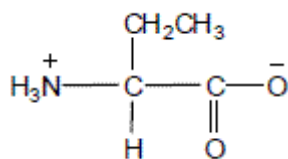
- (iii) must show trailing bonds



allow polyalkene conseq on their c(ii)
ignore n

1

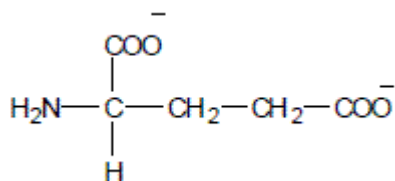
- (d)



allow $\text{NH}_3^+ -$
allow COO^-

1

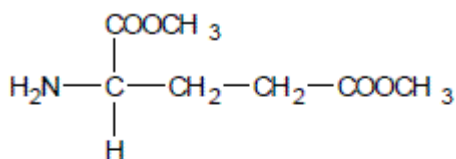
- (e) (i)



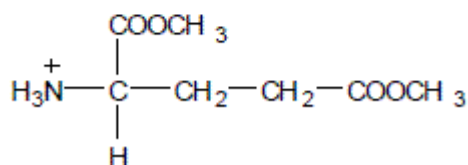
In (e), do not penalise a slip in the number of carbons in the $-\text{CH}_2\text{CH}_2-$ chain, but all must be bonded correctly
NB two carboxylate groups
Allow COONa or $\text{COO}^- \text{Na}^+$ but not covalent bond to Na
allow NH_2-

1

- (ii)



OR

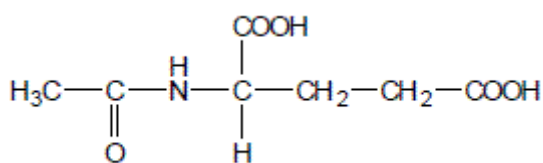


In (e), do not penalise a slip in the number of carbons in the $-\text{CH}_2\text{CH}_2-$ chain, but all must be bonded correctly
NB two ester groups

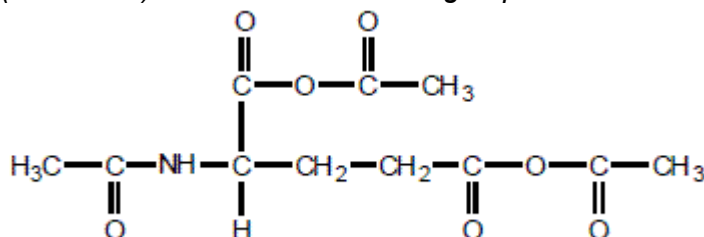
allow NH_2^- or $^+\text{NH}_3^-$

1

(iii)



*In 4(e), do not penalise a slip in the number of carbons in the -CH₂CH₂- chain, but all must be bonded correctly
allow anhydride formation on either or both COOH groups
(see below) with or without amide group formation*



1

(f) **M1** phase or eluent or solvent (or named solvent) is moving or mobile

1

M2 stationary phase or solid or alumina/silica/resin

1

M3 separation depends on balance between solubility or affinity
(of compounds) in each phase

OR

different adsorption or retention

OR

(amino acids have) different R_f values

OR

(amino acids) travel at different speeds or take different times

1

[13]

Examiner reports

Q1.

- (a) Most students coped with the need to convert volume to mass and gave 9.6 g of cyclohexanol at the start, but too many then assumed that this meant the maximum possible yield of cyclohexene would be 9.6 g and so divided 5.97 by this. 47.5% of students completed the calculation successfully.
- (b) Most students (70.2%) were able to suggest the use of bromine water, with only very few getting the colour change the wrong way round. The most common wrong answer was the suggestion of a test for the absence of alcohol rather than a positive test for the presence of an alkene.
- (c) This was not answered as well as expected (42% correct), with many vague references to 'removing impurities' or 'separating the layers'.
- (d) The key to questions like this about understanding the method of a practical is to relate the answer to the position of the process in the overall method. The reference to periodic opening of the tap preceded the actual separation of the layers in the method, yet many answers referred to opening the tap to run off one of the layers. It was interesting that more students (57.4% correct) were able to suggest that this was to release pressure of CO₂ (due to reaction with acid) than were able to suggest, in part (c), that the carbonate had been added in order to react with acid.
- (e) The low success rate in this question (28.2% correct) is explained by a lack of specificity in students' answers; the mark required a reference to a lack of reactivity with, or solubility in, **cyclohexene**.
- (f) Most students chose to draw a diagram rather than write a description; the former is the recommended approach. However, clear, simple cross-sectional line diagrams of a Buchner funnel and filter paper on top of a Buchner flask (with side-arm attached to a vacuum pump) were seldom seen. Tubes were often shown as sealed, bungs and side-arms were missing and the side-arm was often drawn on the funnel rather than the flask. Only 28.7% of students gained both marks; 37.1% failed to score.
- (g) This part gave numerous examples of repeating the question, with many answers seen along the lines of "the retention time is shorter because the retention time is shorter". Some students also inverted the question and tried to describe why cyclohexene had a longer retention time, while others thought incorrectly that cyclohexene is polar due to the C=C bond. An inversion of reasoning that was often seen was the incorrect idea that cyclohexanol has hydrogen bonding, which is why it is polar. 34.9% of students scored zero here.
- (h) Too many students failed to remember the need always to mention both the wavenumber range **and** the bond responsible in questions of this type. Most correctly stated that a peak in the range 3230–3550 would not be visible, but many then failed to state that this peak relates to the O–H bond in an alcohol. As a result, only 43.7% of students gained the mark.

Q2.

- (a) This was well known; most students gave an equation for a 1:1 mole ratio, although a 1:2 ratio and a two-step process were also seen and credited.

- (b) This was also well known and 35% of students scored full marks. In the mechanism, some students incorrectly showed a lone pair on the electrophile, or the horseshoe was sometimes drawn in an incorrect orientation relative to C1. Some students started from benzene and formed nitrobenzene first.
- (c) Only the very weakest 7% of students failed to calculate the correct R_f value.
- (d) This question tested understanding of the balance between solubility in the moving phase and retention by the stationary phase. Both parts were needed in the answer.
- (e) This question was found very easy by most, although weaker students discussed safety issues.
- (f) A third of students scored both marks, but sadly 7% of students made no attempt at the question. They were asked to deduce the answer to an unfamiliar practical situation, but one which is similar to those they will have met in Required Practical Activity 12. Many deduced that the 1,4 isomer was less polar, but then attempted to explain this relative polarity rather than answering the question asked.
- (g) This final part to the question was not attempted by 10.2% of students. The best answers demonstrated the ability of candidates to deduce that the student was correct, and to explain their deduction.

Q5.

Parts (a), (b)(i), (b)(ii) and (d) were very well answered, but formation of the ester in part (b)(iii) was less well understood. Just over a third of students scored both marks in part (c), but a mark was often lost where a displayed formula for the fragment ion was not shown. Other errors included omitting the charge and the radical dot on the molecular ion or including two positive charges on the fragment ion, ie writing C^+ in the structure and also writing + outside brackets around the ion. There were very few correct answers to part (e). Only a small number of students recognised that the amino acids would become protonated in acidic solution and so the protonated lysine would have twice the charge of the protonated alanine.

Q6.

- (a) In part (i), most students were able to identify two appropriate atoms in the structure, but the ionic nature of Zineb resulted in many students failing to calculate its M_r correctly in part (ii). In part (iii) proved to be demanding too, ester being the most common error.
- (b) The simple calculation in this part proved straightforward for most students.
- (c) This part highlighted the difficulties students face when applying theoretical ideas. Many students ignored the specific references to Zineb and ETU and wrote at length about the theory of chromatography. It was not uncommon to see long answers that did not specify the type of chromatography to be used, or make any mention of Zineb or ETU. Students were similarly reluctant to use the information in the questions to predict the outcomes for this particular separation. Such non-specific answers received few marks. A significant number of students chose gas chromatography, and even then could not make any use of the information at hand.

Q7.

There were many excellent answers to part (a) of this question. Common errors in what were otherwise correct answers for part (a) included incomplete reagents such as

omission of the acid with potassium dichromate or simply stating “carbonate” rather than the full name or formula of a carbonate such as sodium carbonate or Na_2CO_3 .

The haloalkane proved to be the most difficult to identify, with students often treating it as an acyl chloride and assuming that addition of water would give misty fumes.

A single test was required for each compound so answers which involved a preliminary step before a test did not score.

In part (b), the first two marks were often gained but few students gained both of the other two. This was often because of incorrect or missing statements about polarity in propanone or propan-2-ol or because of a lack of knowledge of what is happening in this type of chromatography. Many students thought that the substances would rise in the column while others thought that the M_r would affect retention time.

Q8.

Part (a) was answered well by two thirds of the candidates. In part (b)(i), half gave a correct structure; many wrong answers showed too many repeating units or included an extra oxygen atom. The type of polymerisation in part (b)(ii) and the required bond in part (c)(i) were both very well known. In part (c)(ii) the answer was required as a displayed formula, ie it must show **all** the bonds. Many candidates have still not realised that all includes the O–H bond. Part (d) was correctly answered by fewer than a fifth of the candidates. Many simply drew 2-aminobutanoic acid rather than its zwitterion. In parts (e)(i) and (e)(ii), many candidates did not recognise that both acid groups in glutamic acid would react. Two thirds of candidates answered part (e)(i) correctly, but parts (e)(ii) and (e)(iii) were found more difficult. In part (f), some candidates explained clearly how column chromatography worked and just under a third gained full marks in this section. Others described paper chromatography or were obviously unfamiliar with the topic.