A-Level Chemistry

Thin Layer Chromatography

Mark Scheme

Time available: 58 minutes
Marks available: 50 marks

1. (a) **M1** $\frac{27}{80}$ = 0.34

1

M2 glycine

M1 *some relevant working is needed to arrive at 0.325 - 0.35*

no ECF based on ***M1***

1

(b) use uv lamp or ninhydrin

allow *developing / locating agent / iodine*

1

(c) each amino acid has different (relative) affinity/attraction to/solubility in stationary and mobile phases

allow *reference to different solubility in solvent OR different affinity for stationary phase*

1

[4]

2. (a) <u>Conc</u> HCl

Allow concentrations of 5M or higher

Allow <u>conc</u> sulfuric or <u>conc</u> strong alkalis

1

(b) Using ninhydrin or ultraviolet light

Allow I_2 (vapour)

1

(c) 7 or seven

1

(d) Some of the amino acids did not separate/dissolve with the first/either solvent

OR

Some amino acids have the same Rf value or have the same affinity with the first/either solvent

Not amino acids have different Rf values in different solvents

1

[4]

3. (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 cm^3:

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]

4. (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]

5. (a) $HNO_3 + 2H_2SO_4 \longrightarrow NO_2^+ + H_3O^+ + 2HSO_4^-$

Allow $H_2SO_4 + HNO_3 \longrightarrow NO_2^+ + HSO_4^- + H_2O$

Allow a combination of equations which produce NO_2^+

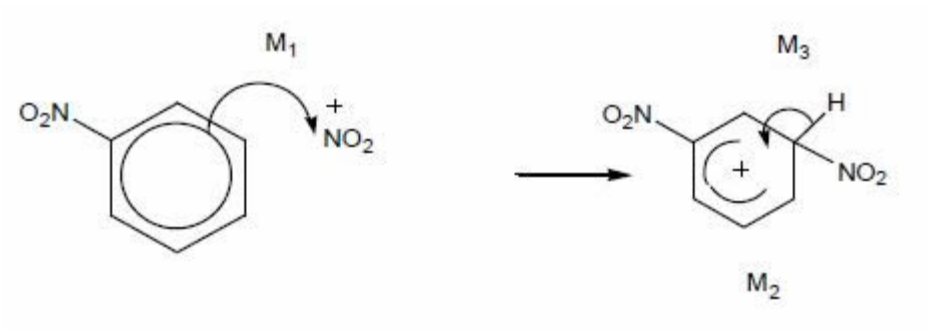
Penalise equations which produce SO_4^{2-}

1

(b) Electrophilic substitution.

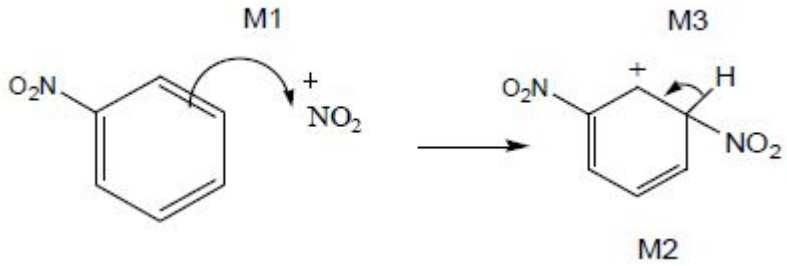
Ignore nitration

1



3

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

1

(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

1

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

1

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]

6. (a) **M1** Moles of cyclohexanol = (10 × 0.96)/100.0 = 0.096

Correct answer scores all 3 marks

1

M2 Max mass of cyclohexene = 0.096 × 82.0 = 7.87(2)

= *M1 × 82.0 (process mark)*

1

M3 % yield = (5.97 / 7.87) × 100 = 76% (Allow range 75.8 – 76)

= *(5.97 / M2) × 100 (process mark)*

1

Alternative method

M1 Moles of cyclohexanol = (10 × 0.96)/100.0 = 0.096

M2 Moles of cyclohexene = 5.97/82.0 = 0.0728

M3 % yield = 0.0728 / 0.096 × 100 = 76% (allow range 75.8 – 76)

= *(M2 / M1) × 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/H_3PO_4/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 cylcohexanol 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]