

GCE

Chemistry A

H432/02: Synthesis and analytical techniques

Advanced GCE

Mark Scheme for November 2020

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This mark scheme is published as an aid to teachers and students, to indicate the requirements of the examination. It shows the basis on which marks were awarded by examiners. It does not indicate the details of the discussions which took place at an examiners' meeting before marking commenced.

All examiners are instructed that alternative correct answers and unexpected approaches in candidates' scripts must be given marks that fairly reflect the relevant knowledge and skills demonstrated.

Mark schemes should be read in conjunction with the published question papers and the report on the examination.

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1. Annotations available in RM Assessor

Annotation	Meaning
	Correct response
	Incorrect response
	Omission mark
	Benefit of doubt given
	Contradiction
	Rounding error
	Error in number of significant figures
	Error carried forward
	Level 1
	Level 2
	Level 3
	Benefit of doubt not given
	Noted but no credit given
	Ignore

2. Abbreviations, annotations and conventions used in the detailed Mark Scheme (to include abbreviations and subject-specific conventions).

Annotation	Meaning
DO NOT ALLOW	Answers which are not worthy of credit
IGNORE	Statements which are irrelevant
ALLOW	Answers that can be accepted
()	Words which are not essential to gain credit
—	Underlined words must be present in answer to score a mark
ECF	Error carried forward
AW	Alternative wording
ORA	Or reverse argument

1. Subject-specific Marking Instructions

INTRODUCTION

Your first task as an Examiner is to become thoroughly familiar with the material on which the examination depends. This material includes:

- the specification, especially the assessment objectives
- the question paper
- the mark scheme.

You should ensure that you have copies of these materials.

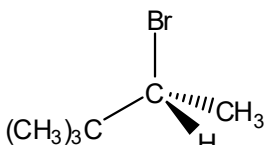
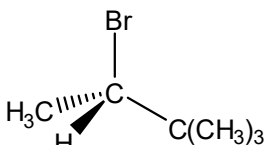
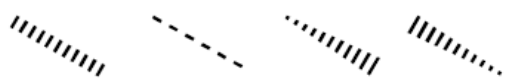
You should ensure also that you are familiar with the administrative procedures related to the marking process. These are set out in the OCR booklet **Instructions for Examiners**. If you are examining for the first time, please read carefully **Appendix 5 Introduction to Script Marking: Notes for New Examiners**.

Please ask for help or guidance whenever you need it. Your first point of contact is your Team Leader.

SECTION A

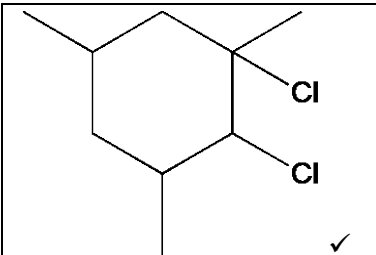
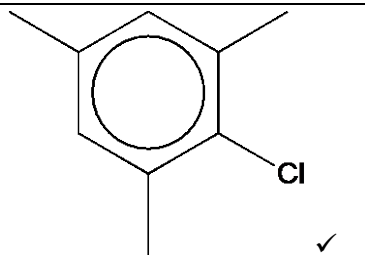
Question	Answer	Marks	AO element	Guidance
1	A	1	1.2	
2	B	1	1.1	
3	A	1	1.1	
4	C	1	2.3	
5	D	1	1.2	
6	D	1	2.1	
7	A	1	1.1	
8	C	1	2.2	
9	D	1	1.2	
10	C	1	2.5	ALLOW 5
11	A	1	2.6	
12	A	1	2.2	
13	A	1	1.1	
14	B	1	2.3	
15	C	1	1.2	

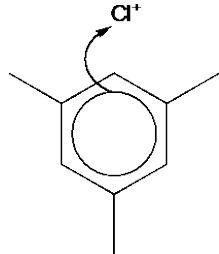
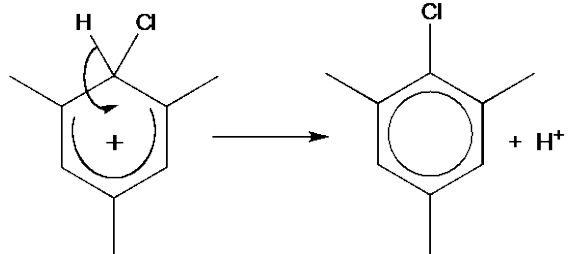
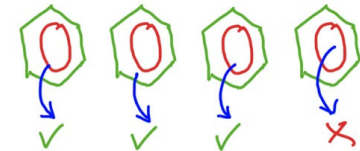
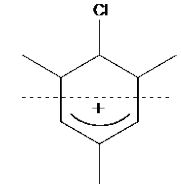
SECTION B

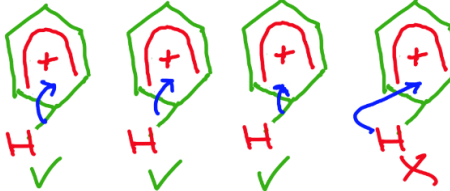
Question			Answer	Marks	AO element	Guidance
16	(a)	(i)	2-bromo-3,3-dimethylbutane ✓	1	1.2	IGNORE lack of hyphens or addition of commas ALLOW 3,3-dimethyl-2-bromobutane DO NOT ALLOW 2-bromo-3-dimethylbutane methy for methyl methly for methyl brom for bromo
	(b)	(i)	Stereoisomers Same structural formula AND Different arrangement (of atoms) in space OR different spatial arrangement (of atoms) AND Type: Optical ✓	1	1.2	ALLOW structure/displayed/skeletal formula DO NOT ALLOW same empirical formula OR same general formula IGNORE same molecular formula IGNORE references to chiral molecules/compounds
		(ii)	One 3D structure with correct groups attached to the chiral C ✓ Two 3D structures of $(\text{CH}_3)_3\text{CCHBrCH}_3$ that are mirror images AND correct connectivity in both ✓  	2	2.5 1.2	ALLOW small slip in one of the groups OR use of C_4H_9 3D structures must have four central bonds with at least two wedges. For bond into paper accept:  ALLOW two 3D structures with 2 groups swapped e.g.

[illegible]

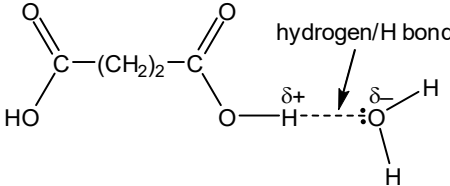
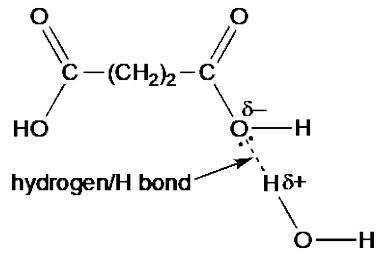
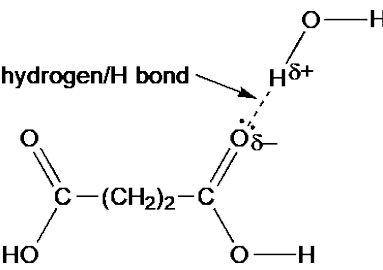
Question			Answer	Marks	AO element	Guidance
	(d)		further substitution/s OR produces different termination products OR More than one termination step OR Mixture of products are formed ✓ substitution at different positions along chain ✓	2	1.1×2	ALLOW dibromo/multibromo compounds formed OR an example of a further substitution product OR an example of a different termination product ALLOW more than one hydrogen (atom) can be replaced ALLOW radicals react with each other to form other products IGNORE references to separation of products IGNORE references to atom economy or yield ALLOW a hydrogen (atom) on a different carbon (atom) can be replaced

Question			Answer	Marks	AO element	Guidance
17	(a)	(i)	 Organic product with B  Organic product with C	2	2.5×2	
		(ii)	Reactivity of B in B electrons are localised OR in B π-bond is localised ✓ Reactivity of C in C electrons are delocalised OR In C π-system / ring is delocalised In B, electron density is higher AND B is more susceptible to electrophilic attack OR B attracts/accepts the electrophile/Cl_2 more OR B polarises the electrophile/Cl_2 more ✓ ORA	3	1.1×3	ALLOW labelled diagram to show delocalised system IGNORE charge density IGNORE electronegativity IGNORE B is more reactive/reacts more readily (no reference to electrophile) IGNORE references to electron density spread around the π-ring ALLOW chlorine

Question	Answer	Marks	AO element	Guidance
(iii)	Generation of electrophile $\text{AlCl}_3 + \text{Cl}_2 \rightarrow \text{AlCl}_4^- + \text{Cl}^+ \checkmark$ Attack of Cl^+  Curly arrow from π-bond to $\text{Cl}^+ \checkmark$ Intermediate and organic product  Correct intermediate $\checkmark$ Curly arrow from C-H bond to reform π-ring $\checkmark$ Regeneration of catalyst $\text{H}^+ + \text{AlCl}_4^- \rightarrow \text{AlCl}_3 + \text{HCl} \checkmark$	5	1.2 1.2 2.5 1.2 1.2	ANNOTATE ANSWER WITH TICKS AND CROSSES ALLOW $\text{FeCl}_3 + \text{Cl}_2 \rightarrow \text{FeCl}_4^- + \text{Cl}^+$ ALLOW use of Fe NOTE: curly arrows can be straight, snake-like, etc. but NOT double-headed or half-headed arrows 1st curly arrow must start from, OR close to, circle of benzene ring  DO NOT ALLOW following intermediate:  π-ring must cover 4 of the 6 sides of the benzene ring AND correct orientation, <i>i.e.</i> gap towards C-Cl ALLOW + sign anywhere inside the 'hexagon' of the intermediate.

Question			Answer	Marks	AO element	Guidance						
						IGNORE partial charges on the chlorine in the intermediate DO NOT ALLOW mark for intermediate if any CH₃ is missing Curly arrow must start from, OR be traced back to, any part of C-H bond and go inside the 'hexagon'  ALLOW use of AlCl₄⁻ in the mechanism ALLOW ECF for regeneration of an incorrect metal chloride catalyst e.g. AgCl₃						
	(b)		3C₃H₆O → C₉H₁₂ + 3H₂O molecular formulae of C₃H₆O AND C₉H₁₂ ✓ H₂O as by-product ✓ correct balanced equation ✓	3		2.6 2.5 2.6						
	(c)	(i)	<table><tr><td></td><td>Compound C</td><td>Compound D</td></tr><tr><td>Number of peaks</td><td>3 ✓</td><td>8 ✓</td></tr></table>		Compound C	Compound D	Number of peaks	3 ✓	8 ✓	2	3.2	
	Compound C	Compound D										
Number of peaks	3 ✓	8 ✓										

Question	Answer	Marks	AO element	Guidance
(ii)	<chem>Cc1cc(C)c(C)cc1</chem> (compound C) reagent: HNO_3 catalyst: H_2SO_4 <chem>Cc1cc(C)c(cc1[N+](=O)[O-])C(=O)O</chem> (boxed structure) 1. Sn + HCl 2. Neutralise <chem>Cc1cc(C)c(cc1N)C</chem> (boxed structure) reagent: CH_3COCl <chem>CC(=O)Nc1cc(C)c(C)cc1</chem> (compound D)	5	3.2×5	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous IGNORE names for organic intermediates (question asks for structures) ALLOW names of reagents and catalyst Around top arrow, ALLOW 1 of 2 marks if HNO_3 and H_2SO_4 swapped. i.e. reagent: H_2SO_4 catalyst: HNO_3 IGNORE references to concentration ALLOW $(\text{CH}_3\text{CO})_2\text{O}$ for left arrow IGNORE CH_3COOH IGNORE acyl chloride DO NOT ALLOW $\text{AlCl}_3/\text{FeCl}_3/\text{Fe4}$

Question	Answer	Marks	AO element	Guidance
18 (a) (i)	Reagents $\text{K}_2\text{Cr}_2\text{O}_7$ AND acid AND reflux ✓ Equation $\text{HO}(\text{CH}_2)_4\text{OH} + 4[\text{O}] \rightarrow \text{HOOC}(\text{CH}_2)_2\text{COOH} + 2\text{H}_2\text{O}$ [O] AND H_2O ✓ Correctly balanced equation ✓	3	1.1 2.5 2.6	ALLOW $\text{Na}_2\text{Cr}_2\text{O}_7$ OR $\text{Cr}_2\text{O}_7^{2-}$ ALLOW H_2SO_4 OR HCl OR H^+ ALLOW words. e.g. 'acidified dichromate' ALLOW a small slip in formula for dichromate e.g KCr_2O_7,
	(ii)  OR  Diagram showing correct dipole charges on each end of one hydrogen bond between a water molecule and a diacid ✓ Hydrogen bond between one lone pair on O atom in one of the molecules and the H atom of another AND Hydrogen bonding stated or labelled on diagram	2	2.1×2	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous DO NOT ALLOW $\delta+$ on H atoms of CH_2 group ALLOW H-bond for hydrogen bond ALLOW H bond between $\text{C}=\text{O}$ and H_2O, i.e.  IF diagram is not labelled, ALLOW hydrogen bond/H bond from text

Question		Answer	Marks	AO element	Guidance
(b)	(i)	$\text{-----C(=O)-(CH}_2\text{)}_2\text{-C(=O)-O-(CH}_2\text{)}_4\text{-O-----}$ Ester link (must be displayed) ✓ Rest of structure ✓	2	1.2 2.5	ALLOW the 'O' or C=O at either end, e.g. $\text{---O-C(=O)-(CH}_2\text{)}_2\text{-C(=O)-O-(CH}_2\text{)}_4\text{---}$ $\text{---(CH}_2\text{)}_2\text{-C(=O)-O-(CH}_2\text{)}_4\text{-O-C(=O)---}$ IGNORE brackets IGNORE n End bonds' MUST be shown (solid or dotted) DO NOT ALLOW more than one repeat unit
	(ii)	the ester/ ester bond/ ester group /polyester can be broken down ✓ OR It can be hydrolysed ✓	1	3.2	IGNORE references to photodegradable 'Bond breaks' is not sufficient – no reference to ester bond
	(iii)	$\begin{array}{c} \text{O} & & \text{O} \\ \parallel & & \parallel \\ \text{HO-C} & \text{---(CH}_2\text{)}_2\text{---} & \text{C-OH} \end{array} + 2 \text{SOCl}_2 \longrightarrow \begin{array}{c} \text{O} & & \text{O} \\ \parallel & & \parallel \\ \text{Cl-C} & \text{---(CH}_2\text{)}_2\text{---} & \text{C-Cl} \end{array} + 2 \text{SO}_2 + 2 \text{HCl}$ SOCl₂ in equation ✓ Structure of diacyl dichloride ✓ Complete balanced equation ✓	3	1.1 1.2 2.6	ALLOW alternative approach using PCl₅ or PCl₃

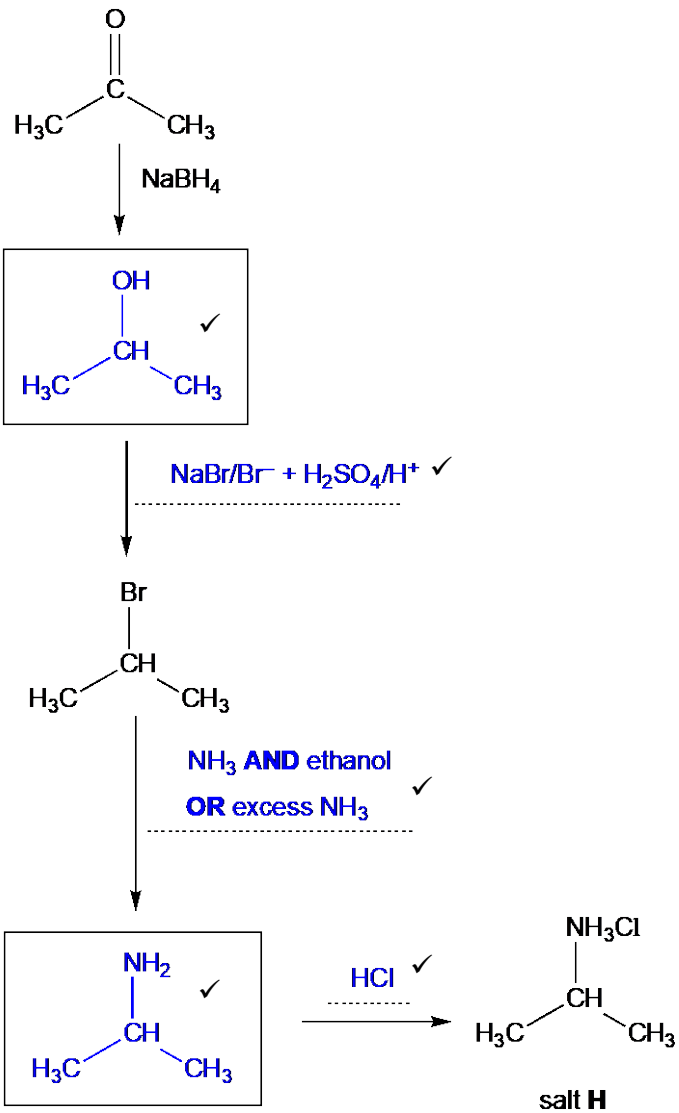
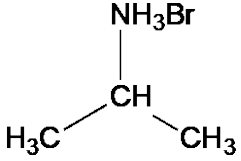
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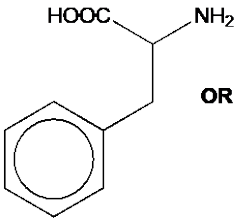
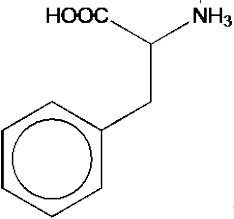
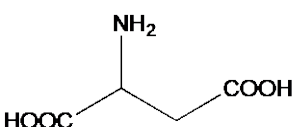
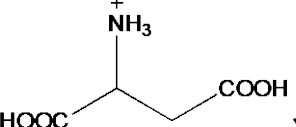
Question	Answer	Marks	AO element	Guidance
(c) (i)	Mechanism 3 marks Curly arrow from CN^- to C atom of $\text{C}=\text{O}$ ✓ Dipole shown on $\text{C}=\text{O}$ bond, $\text{C}^{\delta+}$ and $\text{O}^{\delta-}$, AND curly arrow from $\text{C}=\text{O}$ bond to O atom ✓ Curly arrow from lone pair OR – charge on O^- of correct intermediate to H^+ ✓ Product 1 mark Name of mechanism 1 mark Nucleophilic addition ✓	5		ANNOTATE ANSWER WITH TICKS AND CROSSES Curly arrow must come from lone pair on C of CN^- OR CN^- OR from minus sign on C of CN^- ion (then lone pair on CN^- does not need to be shown) Curly arrow from $\text{C}=\text{O}$ bond must start from, OR be traced back to, any part of $\text{C}=\text{O}$ bond and go to O 2.5 ALLOW curly arrow to H atom of H_2O, i.e. 2.5 IGNORE attempt to draw curly arrow showing breaking of $\text{H}-\text{O}$ in H_2O 1.1 IGNORE lack of dipole on H_2O

Question			Answer	Marks	AO element	Guidance
		(ii)	Heterolytic One (bonded) atom/O receives both/2 electrons ✓ Fission Breaking of a covalent bond ✓	2	1.2	ALLOW 2 electrons go to one (bonded) atom/O DO NOT ALLOW both pairs of electrons go to O IGNORE formation of ions/radicals For O atom, ALLOW species DO NOT ALLOW element or molecule ALLOW π bond in C=O breaks IGNORE breaking of C=O bond (no reference to only one bond breaking) 'Bond breaking' is not sufficient <i>(no reference to covalent)</i>

Question	Answer	Marks	AO element	Guidance
20 (a)*	<i>Refer to marking instructions on page 4 of mark scheme for guidance on marking this question.</i> Level 3 (5-6 marks) A correct calculation of the mass of cyclopentanol AND A detailed description of most purification steps <i>There is a well-developed line of reasoning which is clear and logically structured. The information presented is relevant and substantiated.</i> Level 2 (3-4 marks) Calculates the mass of cyclopentanol with some errors AND A detailed description of some purification steps OR A correct calculation of the mass of cyclopentanol AND A detailed description of a few purification steps <i>There is a line of reasoning presented with some structure. The information presented is relevant and supported by some evidence.</i> Level 1 (1-2 marks) Calculates the mass of cyclopentanol with some errors OR A detailed description of some purification steps <i>There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant.</i> 0 marks No response or no response worthy of credit.	6	2.8×2 3.3×4	Indicative scientific points may include: <u>Calculation of mass of cyclopentanol</u> Using moles $n(\text{cyclopentene}) = \frac{4.00}{68} = 0.0588\dots (\text{mol})$ $n(\text{cyclopentanol}) = 0.0588 \times \frac{100}{64} = 0.0919\dots (\text{mol})$ - Mass of cyclopentanol = $86 \times 0.0919 = \mathbf{7.90 \text{ g}}$ Using mass - Theoretical mass cyclopentene = $4.00 \times \frac{100}{64} = 6.25 \text{ g}$ - Theoretical $n(\text{cyclopentanol}) = \frac{6.25}{68} = 0.0919 (\text{mol})$ - Mass of cyclopentanol = $86 \times 0.0919 = \mathbf{7.90 \text{ g}}$ ALLOW for small slip in Mr / rounding errors <u>Examples of some calculation errors</u> Incorrect inverse ratio: $0.0588 \times \frac{64}{100} = 0.0376\dots (\text{mol})$ - Mass = $86 \times 0.0376 = 3.24 \text{ g}$ Ignoring % yield gives: $\frac{4.00}{68} = 0.0588\dots (\text{mol})$ - Mass = $86 \times 0.0588 = 5.06 \text{ g}$ <u>Purification</u> - Add a neutralising agent by formula or name e.g. Na_2CO_3 - In separating funnel, organic layer is on top - Drying with an anhydrous salt by formula or name, e.g. MgSO_4, Na_2SO_4, CaCl_2 - Redistil at approx. 44°C Examples of detail in bold (NOT INCLUSIVE)

Question			Answer	Marks	AO element	Guidance
	(b)		C=C/alkene peak in region $1620-1680\text{ cm}^{-1}$ ✓ O–H/alcohol peak in region $3200-3600\text{ cm}^{-1}$ ✓	2	3.2×2	LOOK ON THE SPECTRUM for labelled peaks which can be given credit IGNORE references to C-O at 1000cm^{-1}

Question	Answer	Marks	AO element	Guidance
21 (a)	 $\text{CH}_3\text{C}(=\text{O})\text{CH}_3 \xrightarrow{\text{NaBH}_4} \boxed{\text{CH}_3\text{CH}(\text{OH})\text{CH}_3} \checkmark$ $\xrightarrow{\text{NaBr/Br}^- + \text{H}_2\text{SO}_4/\text{H}^+} \text{CH}_3\text{CH}(\text{Br})\text{CH}_3 \checkmark$ $\xrightarrow{\text{NH}_3 \text{ AND ethanol OR excess NH}_3} \boxed{\text{CH}_3\text{CH}(\text{NH}_2)\text{CH}_3} \checkmark$ $\xrightarrow{\text{HCl}} \text{CH}_3\text{CH}(\text{NH}_3\text{Cl})\text{CH}_3 \text{ salt H} \checkmark$	5	2.5×5	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous ALLOW HBr ALLOW for the bottom left structure 

Question	Answer	Marks	AO element	Guidance
(b) (i)	Ester Amide Amine Carboxylic acid 4 groups correct ✓✓✓ 3 groups correct ✓✓ 2 groups correct ✓	3	1.2×3	IGNORE amino acid ALLOW carboxyl IGNORE attempt to classify amide, e.g. secondary IGNORE formulae (question asks for names) IF > 4 functional groups are shown, • Count 4 groups max but incorrect groups first IGNORE aryl OR alkyl group e.g. benzene, phenyl, aryl, arene, methyl
(ii)	Methanol 1 mark $\text{H}_3\text{C}-\text{OH}$ ✓ Amino Acids 3 marks  OR   OR  Both amino acids shown with NH_3^+ ✓	4	2.5×4	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous ALLOW + charge on H of NH_3 group, i.e. NH_3^+ If BOTH amino acids are shown with NH_3 groups (without the + charge) OR as NH_2^+ groups, award 2 of the 3 marks for the amino acids If BOTH amino acids are shown as correctly balanced salts, e.g. NH_3Cl , all marks can be awarded.

Question			Answer	Marks	AO element	Guidance
		(iii)	FIRST CHECK ANSWER ON THE ANSWER LINE If answer = 22.4 OR 22 OR 23 award 3 marks $n(\text{aspartame}) \text{ in 1 can} = 0.167 / 294 = 5.68 \times 10^{-4} \text{ (mol)} \checkmark$ $n(\text{aspartame}) \text{ limit per day} = 1.7 \times 10^{-4} \times 75 = 0.01275 \text{ (mol)} \checkmark$ $\text{number of cans} = 0.01275 / 5.68 \times 10^{-4} = 22.4 \checkmark$	3	2.2×3	If there is an alternative answer, apply ECF and look for alternative methods Alternative methods $n(\text{aspartame}) \text{ in 1 can} = 0.167 / 294 = 5.68 \times 10^{-4} \text{ (mol)} \checkmark$ $n(\text{aspartame}) \text{ per kg} = 5.68 \times 10^{-4} / 75 = 7.57 \times 10^{-6} \text{ (mol)} \checkmark$ $\text{number of cans} = 1.7 \times 10^{-4} / 7.57 \times 10^{-6} = 22.4 \checkmark$ OR $n(\text{aspartame}) \text{ limit per day} = 1.7 \times 10^{-4} \times 75 = 0.01275 \text{ (mol)} \checkmark$ $\text{mass(aspartame) limit per day} = 0.01275 \times 294 = 3.7485 \text{ (g)} \checkmark$ $\text{number of cans} = 3.7485 / 0.167 = 22.4 \checkmark$

Question	Answer	Marks	AO element	Guidance
22 (a)	CDCl ₃ used as a solvent ✓ D ₂ O used to identify OH OR NH protons ✓	2	1.1×2	Example and use required for each mark ALLOW for 1 mark, D ₂ O as a solvent
(b)*	<i>Please refer to the marking instructions on page 4 of this mark scheme for guidance on how to mark this question.</i> Level 3 (5–6 marks) Structure I has a viable chemical structure of C₆H₉NO₂ which has the key features consistent with spectral data AND Most of the data analysed <i>There is a well-developed line of reasoning which is clear and logically structured. The information presented is relevant and substantiated.</i> Level 2 (3–4 marks) Compound I has a viable chemical structure of C₆H₉NO₂ with most of the key features consistent with spectral data AND Some of the spectral data analysed. <i>There is a line of reasoning presented with some structure. The information presented is relevant and supported by some evidence.</i> Level 1 (1–2 marks) Correct determination of empirical formula and/or molecular formula. OR Analyses some of the IR and NMR data. OR Analyses most of the NMR data.	6	3.1× 4 3.2× 2	Indicative scientific points: <u>Empirical and Molecular Formulae</u> $\begin{array}{cccc} \text{C} & : & \text{H} & : & \text{N} & : & \text{O} \\ = & \frac{56.69}{12.0} & : & \frac{7.09}{1.0} & : & \frac{11.02}{14.0} & : & \frac{25.20}{16.0} \\ \text{OR} & 4.72 & : & 7.09 & : & 0.787 & : & 1.575 \\ = & 6 & : & 9 & : & 1 & : & 2 \end{array}$ - Empirical formula = C₆H₉NO₂ <i>m/z</i> = 127.0 and empirical formula mass (127) used to determine molecular formula as C₆H₉NO₂ <u>Structures of compound I</u> $\begin{array}{c} \text{H} \quad \text{O} \\ \quad \\ \text{NC}-\text{C}-\text{C}-\text{O}-\text{CH}_2\text{CH}_3 \\ \\ \text{CH}_3 \end{array}$ OR $\begin{array}{c} \text{O} \quad \text{H} \\ \quad \\ \text{CH}_3\text{CH}_2-\text{C}-\text{O}-\text{C}-\text{CN} \\ \\ \text{CH}_3 \end{array}$ OR $\begin{array}{c} \text{H} \quad \text{O} \\ \quad \\ \text{CH}_3\text{CH}_2-\text{O}-\text{C}-\text{C}-\text{CN} \\ \\ \text{CH}_3 \end{array}$ OR $\begin{array}{c} \text{O} \quad \text{H} \\ \quad \\ \text{CH}_3\text{CH}_2-\text{C}-\text{C}-\text{O}-\text{CN} \\ \\ \text{CH}_3 \end{array}$ ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous

Question	Answer	Marks	AO element	Guidance
	<i>There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant.</i> 0 marks No response or no response worthy of credit.			<u>Key features</u> • $\text{C} \equiv \text{N}$ • $\text{C}=\text{O}$ in aldehyde, ketone, ester, amide, acid anhydride • CH_3 group that would give a doublet • CH_3 group that would give a triplet • CH_2 group that would give a quartet <u>^1H NMR and IR analysis</u> ^1H NMR spectrum • $\delta = 4.2$ ppm, quartet, 2H $\text{CH}_3\text{--CH}_2\text{--O}$ • $\delta = 2.9$ ppm, quartet, 1H CO--CH--CH_3 • $\delta = 1.7$ ppm, doublet, 3H CO--CH--CH_3 • $\delta = 1.3$ ppm, triplet, 3H $\text{CH}_3\text{--CH}_2$ IR spectrum • peak at $1750\text{ (cm}^{-1}\text{)}$ is $\text{C}=\text{O}$ • peak at $2280\text{ (cm}^{-1}\text{)}$ is $\text{C} \equiv \text{N}$ ALLOW ranges from <i>Data Sheet</i> IGNORE references to C--O peaks

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