

GCE

Chemistry A

H432/02: Synthesis and analytical techniques

Advanced GCE

Mark Scheme for Autumn 2021

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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

Annotation	Meaning
	Correct response
×	Incorrect response
^	Omission mark
BOD	Benefit of doubt given
CON	Contradiction
RE	Rounding error
SF	Error in number of significant figures
ECF	Error carried forward
L1	Level 1
L2	Level 2
L3	Level 3
NBOD	Benefit of doubt not given
SEEN	Noted but no credit given
I	Ignore

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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

Question	Answer	Marks	AO element	Guidance
1	С	1	AO2.1	ALLOW 4 (This is the number of structural isomers)
2	В	1	AO1.2	
3	С	1	AO2.2	
4	С	1	AO2.6	
5	D	1	AO2.1	
6	В	1	AO1.2	
7	Α	1	AO1.2	
8	С	1	AO2.1	
9	С	1	AO1.2	
10	Α	1	AO2.1	
11	D	1	AO2.5	
12	В	1	AO2.1	
13	В	1	AO2.1	
14	С	1	AO1.1	
15	Α	1	AO1.2	
	Total	15		

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Q	uesti	ion	Answer	Marks	AO element	Guidance
16	(a)	(i)	 σ-bond: Overlap of orbitals between (bonding) atoms ✓ π-bond: Sideways overlap of (adjacent) p-orbitals ✓ 	2	AO1.1 ×2	ALLOW labelled diagrams IGNORE the type of orbital for σ-bond DO NOT ALLOW pi-orbital
		(ii)	σ -bonds: 9 \checkmark π -bonds: 2 \checkmark	2	AO1.2 ×2	
	(b)	(i)	H CH ₂ CH ₃ H H δ + Curly arrow from C=C bond to H of H-Br \checkmark DO NOT ALLOW partial charge on C=C Correct dipole shown on H-Br AND curly arrow showing breaking of H-Br bond \checkmark	4	AO1.2 ×2 AO2.5 ×2	NOTE: curly arrows can be straight, snake like, etc. but NOT double headed or half headed arrows 1st curly arrow must • go to the H atom of H–Br AND • start from, OR be traced back to any point across width of C=C C=C C=C C=C C=C C=C C=C C=C C=C C=

Question	Answer	Marks	AO element	Guidance
	Correct carbocation AND curly arrow from Br⁻ to C⁺ of carbocation ✓ DO NOT ALLOW δ+ on C of carbocation H CH₂CH₃ H Br⁻			 3rd curly arrow must go to the C+ of carbocation AND start from, OR be traced back to any point across width of lone pair on :Br OR start from – charge of Br ion (Lone pair NOT needed if curly arrow shown from – charge of Br ion)
	Correct product \checkmark H CH ₂ CH ₃ H C C C H H Br			ALLOW ECF for product from incorrect carbocation, i.e. H CH ₂ CH ₃ H—C—C—H Br H IF Br ₂ is used instead of HBr contact your Team Leader

Qı	uestion)	Answer	Marks	AO element	Guidance
	((ii) (major product forms from) most/more stable intermediate/carbocation ✓	2	AO1.1	For carbocation, ALLOW carbonium ion or cation	
			(major product forms from a) secondary carbocation OR carbocation bonded to more C atoms / more alkyl groups OR carbocation bonded to fewer H atoms ✓		AO1.2	IGNORE descriptions of the major/minor product in terms of Markownikoff's rule e.g. H atom joins to C with most H
						IGNORE references to stability of the product
						ALLOW ORA, i.e. (minor product forms from) least/less stable intermediate/carbocation ✓
						(minor product forms from a) primary carbocation OR carbocation bonded to less C atoms / less alkyl groups
	(i	iii)	3 ✓	1	AO1.2	OR carbocation bonded to more H atoms ✓
	(c) ((i)	Same structural formula AND Different arrangement (of atoms) in space OR different spatial arrangement (of atoms) ✓	1	AO1.1	ALLOW structure/displayed/skeletal formula DO NOT ALLOW same empirical formula OR same general formula IGNORE same molecular formula Reference to E/Z isomerism or optical
	(ii)	Student is not correct AND 2 groups on one carbon atom (of C=C) are the same OR C-C bond can rotate ✓	1	AO3.1	DO NOT ALLOW one side of C=C

Question	Answer	Marks	AO element	Guidance
(d) (1 mark for each curly arrow ✓✓	2	AO2.5 ×2	NOTE: curly arrows can be straight, snake-like, etc. but NOT half headed or double headed arrows Curly arrow from C=C bond must start from, OR be traced back to, Lower left: any part of C=C bond and go to C-C Upper left: any part of C=C bond and go to gap between C=C and C=C
	OCH ₃ OCH ₃	2	AO3.2 ×2	
		Total 17		

PMT

C	Questio	n	Answer	Marks	AO element	Guidance
17	(a)		Formation of Cl^{\bullet} $CClF_3 \rightarrow CF_3 \bullet + Cl \bullet \checkmark$	3	AO2.5	IGNORE dots for formation Cl^{\bullet} , i.e. ALLOW $CCl^{\bullet}F_3 \rightarrow CF_3 + Cl$
			Breakdown of O_3 $Cl \cdot + O_3 \rightarrow \cdot ClO + O_2 \checkmark$		AO1.1 ×2	DO NOT ALLOW ECF Dots required in this equation
			•C $lO + O \rightarrow Cl • + O_2 \checkmark$			 IGNORE O + O₃ → 2O₂ ALLOW 1 mark if both equations are correct by atom but dot(s) missing or incorrect
	(b)	(i)	F F	1	AO2.5	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous 'End bonds' MUST be shown
			Ė CI ✓			DO NOT ALLOW more than 1 repeat unit IGNORE brackets
						IGNORE n
		(ii)	More points of contact / more surface interaction (between molecules) AND Stronger/more dipole(–dipole) interactions ✓	2	AO2.1 ×2	Both answers need to be a comparison IGNORE surface area ALLOW more electrons ALLOW induced/permanent dipole interactions ALLOW London forces ALLOW van der Waals' forces (as permanent dipole-dipole and induced dipole-dipole interactions are present within this polymer)
			More energy needed to break the intermolecular forces			IGNORE IDID

Question	Answer	Marks	AO element	Guidance
(c)	$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	4	AO2.5 ×2	For polymer, DO NOT ALLOW > 1 repeat unit
	Amide link: ✓ 1 repeat unit of correct polymer: ✓		AO1.2 AO2.5	'End bonds' MUST be shown (do not have to be dotted) ALLOW -NH- at either end i.e. H H H H H O O O O O O O O O O O O O O
				IGNORE brackets IGNORE n
	Total	10		

(Quest	ion	Answer	Marks	AO element	Guidance
18	(a)	(i)	Non-superimposable mirror images (about a chiral centre) ✓	1	AO1.1	
18	(a)	(i) (ii)	Non-superimposable mirror images (about a chiral centre) Correct groups attached to chiral C of alanine seen once e.g. CH ₃ H ₂ N COOH Two 3D structures of alanine that are mirror images AND correct connectivity in both i.e. CH ₃ H ₂ N COOH COOH CH ₃ CH ₃ CH ₃ CH ₃ COOH	2	AO1.1 AO2.1 × 2	Each structure 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. CH ₃ CH
						e.g. COOH COOH R C'''' NH ₂ H ₂ N''''''C R

Question	Answer	Marks	element	Guidance
(iii)	4 ✓	1	AO2.2	
(b)	ester C ₇ H ₁₄ O ₃ H ⁺ /H ₂ O OR H ⁺ (aq) OR HCl(aq)	7	AO2.2 AO1.2 × 4 AO2.5 × 3	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous ALLOW names of reagents DO NOT ALLOW OH– for HO– but ALLOW ECF for subsequent use in (b) For hydrolysis, ALLOW dilute acid ALLOW alkaline conditions followed by protonation of carboxylate i.e. NaOH(aq)/OH¬(aq) AND H¬(aq)/HCI(aq)
	C ₅ H ₁₀ O ₃ H ₂ O acyl chloride C ₅ H ₃ O ₂ Cl NaBr/Br AND H ₂ SO ₄ /H ¹ OH OR excess NH ₃ valine haloalkane C ₅ H ₉ BrO ₂			ALLOW HBr for NaBr/H ₂ SO ₄

Questic	on	Answer	Marks	AO element	Guidance
(c)	(i)	C ₁₃ H ₁₈ O ₂ ✓	1	AO2.1	ALLOW C, H and O in any order
	(ii)	FIRST CHECK ANSWER ON THE ANSWER LINE If answer = 1.17×10^{21} award 3 marks	3	AO2.2 × 3	
		M(ibuprofen) = 206 ✓			ALLOW ECF from (c)(i)
		$n(\text{ibuprofen}) = \frac{400 \div 1000}{206} = 1.94 \times 10^{-3} \text{ (mol) } \checkmark$			Calculator: 1.941747573 × 10 ⁻³
		Number of molecules = $1.94 \times 10^{-3} \times 6.02 \times 10^{23}$ = 1.17×10^{21} to 3 SF \checkmark			ALLOW ECF from <i>n</i> (ibuprofen) 3 SF essential
(d)	(i)	O O O	2	AO3.2 × 2	IGNORE small slip in carbon chains
		O H ₃ N C CH OH (CH ₂) ₄			ALLOW H ₂ N
	(ii)	More soluble in water ✓	1	AO3.1	Answer must be a comparison ALLOW dissolve faster/quicker IGNORE absorbed more quickly (given in question)
		Total	18		

(Questic	on	Answer	Marks	AO element	Guidance
19	(a)	(i)	3-methylbut-2-enal ✓	1	AO1.2	IGNORE lack of hyphens, or addition of commas
		(ii)	O NaBH ₄ OH prenal Cr ₂ O ₇ ² -AND H ⁺ AND Ni (CH ₃) ₂ CHOH O CH ₃ COOH	7	AO1.2 ×4 AO2.5 ×3	ALLOW any combination of skeletal OR structural OR displayed formula as long as unambiguous ALLOW names of reagents and catalyst For oxidation, ALLOW K ₂ Cr ₂ O ₇ for Cr ₂ O ₇ ²⁻ ALLOW H ₂ SO ₄ for H ⁺ For left hand side esterification IGNORE C ₃ H ₇ OH IF esterification is given instead of hydrogenation contact your Team Leader

Question	Answer	Marks	AO element	Guidance
(b)*	Refer to marking instructions on page 5 of mark scheme for guidance on marking this question. Level 3 (5-6 marks) Correct calculation of the mass of $C_6H_5CH_2CI$ AND Planned synthesis to form the intermediate $C_6H_5CH_2CN$ followed by hydrolysis to form A with most of the reagents identified and equations are mostly correct. There is a well-developed line of reasoning which is clear and logically structured. The information presented is relevant and substantiated. Level 2 (3-4 marks) Correct calculation of the mass of $C_6H_5CH_2CI$ AND Planned synthesis to form the intermediate $C_6H_5CH_2CN$ with most of the reagents identified and equation is mostly correct OR Calculation of the mass of $C_6H_5CH_2CI$ is partly correct AND Planned synthesis includes formation of the intermediate $C_6H_5CH_2CN$ followed by hydrolysis to form A with some of the reagents identified OR Attempts to calculate mass of $C_6H_5CH_2CI$ but makes little progress AND Planned synthesis includes formation of the intermediate $C_6H_5CH_2CN$ followed by hydrolysis to form A with most of the reagents identified and equations are mostly correct	6	AO2.4 ×2 AO2.7 ×2 AO3.3 ×2	Indicative scientific points may include:

Question	Answer	Marks	AO element	Guidance
	There is a line of reasoning presented with some structure. The information presented is relevant and supported by some evidence. Level 1 (1-2 marks) Calculation of the mass of C ₆ H ₅ CH ₂ Cl is partly correct OR Attempts to calculate mass of C ₆ H ₅ CH ₂ Cl but makes little progress AND Planned synthesis includes formation of the intermediate C ₆ H ₅ CH ₂ CN with the reagent identified OR Planned synthesis includes both steps with some of the reagents identified OR Attempts equations for both steps but these may contain errors OR Describes one step of the synthesis with reagent(s) and equation mostly correct There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant. O marks No response or no response worthy of credit.			Synthesis: reagents and conditions Stage 1: Formation of intermediate, $C_6H_5CH_2CN$ • Reagents: $CN^-(/ethanol)$ • Equation: $C_6H_5CH_2CI + CN^- \rightarrow C_6H_5CH_2CN + CI^-$ OR $C_6H_5CH_2CI + NaCN \rightarrow C_6H_5CH_2CN + NaCI$ (OR use of KCN) Stage 2: Formation of A, $C_6H_5CH_2COOH$ • Reagents: H^+/H_2O (ALLOW 'acid hydrolysis') • Equation: $C_6H_5CH_2CN + 2H_2O + H^+ \rightarrow C_6H_5CH_2COOH + NH_4^+$ OR $C_6H_5CH_2CN + 2H_2O + HCI \rightarrow C_6H_5CH_2COOH + NH_4CI$
	Total	18		

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Question		ion	Answer	Marks	AO element	Guidance
20	(a)	(i)	Stage 1 OH H ₂ O 1 mark for each curly arrow as shown.	6	AO1.1 AO1.2 AO2.5	NOTE: curly arrows can be straight, snake-like, etc. but NOT double headed or half headed arrows Curly arrow from OHT must

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Question	Answer	Marks	AO element	Guidance
	Stage 2 Curly arrow from π-ring to C in CO₂ AND curly arrow from the C=O bond to O atom ✓		AO2.5	 1st curly arrow must go to the C of CO₂ AND start from, OR close to circle of benzene ring 2nd curly arrow must start from, OR be traced back to, any part of C=O bond and go to O
	Correct intermediate ✓ Curly arrow from C–H bond to reform π-ring AND H ⁺ formed ✓		AO2.5 AO1.2	ALLOW 2nd curly arrow from C=O to any O in CO ₂ DO NOT ALLOW the following intermediate: π-ring must cover more than half of the benzene ring structure AND the correct orientation, <i>i.e.</i> gap towards C with CO ₂ ⁻ ALLOW + sign anywhere inside the 'hexagon' of the intermediate.

Question	Answer	Marks	AO element	Guidance
	O- H COO- intermediate + H+ COO-			DO NOT ALLOW mark for intermediate if phenolic Ois missing curly arrow must start from, OR be traced back to, any part of C-H bond and go inside the 'hexagon'
(ii)	OH⁻: base ✓	2	AO2.1 ×2	ALLOW alkali IGNORE 'nucleophile', 'donates electron pair'
	CO₂: electrophile OR electron pair acceptor ✓			IGNORE lone pair acceptor (No lone pair involved)
(iii)	,o	3	AO3.1	
	OH O		AO3.2	
	2 COOH + 2H ₂ O		AO2.6	
	One ester link in organic product ✓			
	Correct structure of organic product ✓			
	Correct equation AND balanced ✓			

Quest	ion	Answer	Marks	AO element	Guidance	
(b)	(i)	Dissolve in hot water/solvent ✓ Minimum amount of solvent ✓ Cool AND Filter AND (leave to) dry ✓ All three needed	3	AO3.3 ×3	 ALLOW any solvent IGNORE Initial filtering hot filtration to remove insoluble impurities DO NOT ALLOW adding of a drying agent (e.g. MgSO₄) 	
	(ii)	C : H : N : O $31.44/12$: $1.31/1$: $18.34/14$: $48.91/16$ OR 2.62 : 1.31 : 1.31 : 3.06 \checkmark 6:3:3:7 OR $C_6H_3N_3O_7$ \checkmark Molecular formula = $C_6H_3N_3O_7$ AND use of $M=229.0$ (directly linked to molecular formula) \checkmark Any trisubstituted $-NO_2$ substituted phenol that is consistent with $M=229.0$ \checkmark Evidence for substitution $2.4.6$ OR $3.4.5$ substituted phenol AND 4 peaks/ C environments from 13 C NMR \checkmark $2.4.6$ substituted phenol AND directing effects of $-OH$ \checkmark	6	AO1.2 × 2 AO3.1 AO3.2	ALLOW alternative approach for empirical formula and evidence that 229 is equal to C ₆ H ₃ N ₃ O ₇ DO NOT ALLOW ECF from the empirical formula with the wrong molar ratio O ₂ N O ₂ N O ₂ N O ₂ O ₃ NO ₂ O ₄ NO ₂ O ₄ NO ₂ O ₅ NO ₂ O ₄ NO ₂ O ₅ NO ₂ O ₅ NO ₂ O ₆ NO ₂ O ₇ NO ₆ O ₇ NO ₇ O ₈ NO ₇	
		Total	20		1-1-	

Question	Answer	Marks	AO element	Guidance
Question 21*	Refer to marking instructions on page 5 of mark scheme for guidance on marking this question. Level 3 (5–6 marks) Compounds D, E AND F correctly identified AND Most of the observations and NMR data analysed. There is a well-developed line of reasoning which is clear and logically structured. The information presented is relevant and substantiated. Level 2 (3–4 marks) Most of compounds D, E AND F correctly identified AND	Marks 6		Indicative scientific points may include: Observations from Test-tube tests 2,4 DNP D has no C=O E and F have C=O present H*/Cr ₂ O ₇ ²⁻ D is primary OR secondary alcohol E and F are ketones (negative test shows not aldehydes) Br ₂ D, E and F have no C=C/are saturated 13 C NMR analysis D: 3 carbon environments/types of C 5 = 24, 36 ppm C-C
	Some of the observations and NMR data analysed. There is a line of reasoning presented with some structure. The information presented is relevant and supported by some evidence. Level 1 (1–2 marks) Most of compounds D, E AND F correctly identified OR Some of compounds D, E AND F correctly identified AND Analyses some of the observations or NMR data OR Analyses most of the observations from the test-tube tests. OR Analyses most of the NMR data. OR Analyses some of the observations and NMR data			• δ = 73 ppm,

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Question	Answer	Marks	AO element	Guidance
	There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant.			E F
	0 marks No response or no response worthy of credit.			
	Total	6		

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