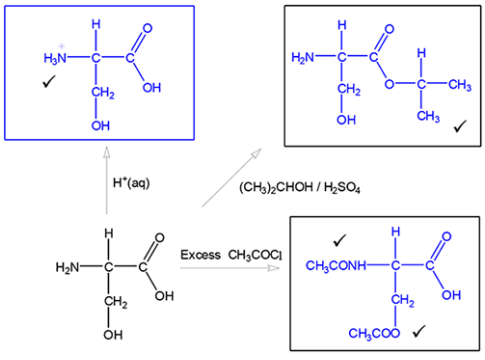
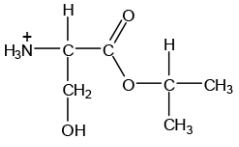
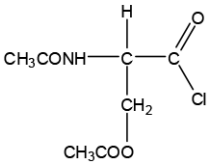
Mark scheme - Chromatography and Qualitative Analysis

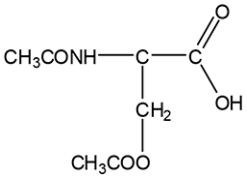
Question			Answer/Indicative content	Marks	Guidance
1			F/aldehyde **AND** Tollens' (reagent) **AND** Silver (mirror/precipitate/ppt/solid) ✓ G/alkene/C=C **AND** Bromine/Br2 **AND** goes colourless/decolourised ✓ G/ketone **AND** 2,4-dinitrophenylhydrazine **AND** orange/yellow/red precipitate ✓ G/ketone **AND** Tollens' (reagent) **AND** no silver mirror/no change/no reaction ✓	4 (AO2.3) (AO3.3) (AO3.3) (AO3.3)	**IGNORE** use of 2,4-DNP with F **ALLOW** ammoniacal silver nitrate **OR** Ag^+/NH_3 **ALLOW** black ppt **OR** grey ppt **ALLOW** bromine water/ $Br_2(aq)$ **ALLOW** errors in spelling for 2,4-DNP **ALLOW** 2,4(-)DNP **OR** 2,4(-)DNPH **ALLOW** Brady's reagent or Brady's Test **ALLOW** solid **OR** crystals **OR** ppt as alternatives for precipitate **ALLOW** ammoniacal silver nitrate **OR** Ag^+/NH_3 **ALLOW** black ppt **OR** grey ppt **ALLOW** alterative approach using acidified potassium dichromate for tests with F and/or G, with correct observations, alongside use of 2,4-DNP **Examiner's Comments** Candidates who found this question difficult often did not give a response that would identify all three of the functional groups (aldehyde, ketone and alkene). The use of Tollens' in identifying aldehydes was well demonstrated, however no reaction with Tollens' was less well demonstrated as a result for ketones.
			Total	**4**	
2	a	i		4(AO 2.5 ×4)	**ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **ALLOW** protonation of NH_2 group in reaction with $(CH_3)_2CHOH$ i.e.  **ALL** structures must be based on serine

For reaction with excess CH_3COCl,
IGNORE reaction of COOH to form an acid anhydride

ALLOW 1 mark for

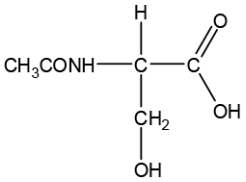


(*both NH and OH groups reacted but acyl chloride instead of COOH*)
OR

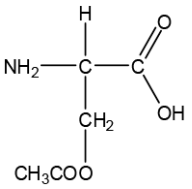


(*both NH and OH groups reacted but H missing from α C atom*)

OR



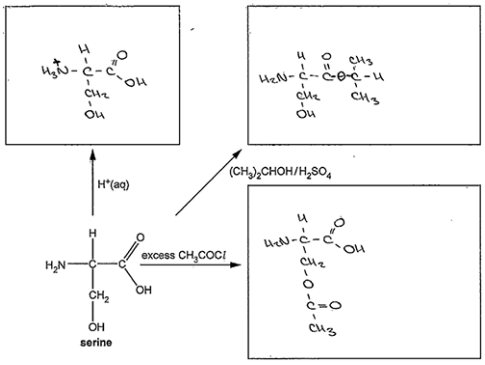
(*NH group reacted correctly but rest of serine unchanged*)
OR



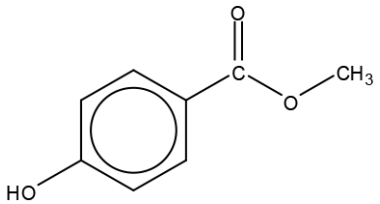
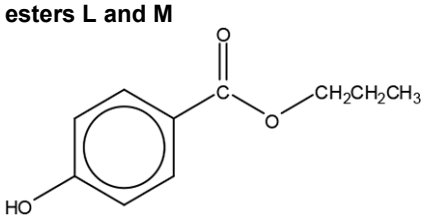
(*OH group reacted correctly but rest of serine unchanged*)

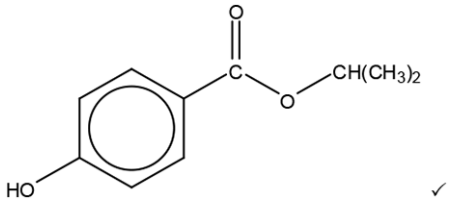
Examiner's Comments

This question focused on the reactions of the different functional groups in serine. Candidates are familiar with these reactions and most candidates scored at least two marks. These were achieved by the formation of the ammonium ion by reaction with H^+ and the formation of the ester from $(CH_3)_2CHOH$. Determining the organic product from the reaction of serine with excess CH_3COCl

					proved more difficult. Some candidates realised that the OH of serine's R group would react and others drew the amide formed from the reaction of the NH_2 group. The best responses showed both conversions and scored full marks. Lower ability responses often showed organic products containing the Cl atom from the ethanoyl chloride rather than the CH_3CO group. **Exemplar 2**  This response scored three marks. The candidate has drawn correct organic products for the reaction of serine with H^+ and $(CH_3)_2CHOH$. The product from the reaction of excess CH_3COCl shows the formation of an ester but not the formation of the amide. This product was seen often by examiners and scored one mark. If an amide group had been shown this response would have received full marks. Notice the candidate has presented their organic products clearly with the structures drawn in a similar manner to serine shown in the question. This is a good strategy to avoid errors and omissions when drawing organic structures.
		ii	**IF M_r(amino acid) = 131 from titration analysis AWARD** **first 3 marks** **ALLOW 3SF** or more throughout **IGNORE** trailing zeroes, e.g. **ALLOW** 0.044 for 0.0440 --- $n(HCl) = 0.150 \times \frac{25.0}{1000}$ **OR** 3.75×10^{-3} (mol) ✓ n(**amino acid**) in 250 cm^3 $= 3.75 \times 10^{-3} \times \frac{250.0}{21.30}$ **OR** 0.0440 (mol) ✓	4 (AO 2.8) (AO 2.8) (AO 2.8)	**ALLOW** alternative approaches **Calculator:** 0.04401408451 **ALLOW ECF** from incorrect n(HCl) **ALLOW ECF** from incorrect n(**amino acid**) **ALLOW ECF** from incorrect M(**amino acid**) **i.e. ECF** for alkyl group closest to calculated M(alkyl group), e.g. for M(alkyl group) = 15, **ALLOW** $CH_3CH(NH_2)COOH$

			M**(amino acid)** $= \frac{5.766}{0.0440} = 131$ (g mol^{-1}) ✓ Amino acid = $(CH_3)_2CHCH_2CH(NH_2)COOH$/leucine **AND** working to show R = 57 to justify choice **OR** evidence to show M_r leucine = 131 to justify choice ✓	(AO 3.2)	**Note:** evidence may be shown with table **Examiner's Comments** Candidates are confident in tackling titration analysis and the majority of responses were well structured. Most candidates used the results to determine the *Mr* of the unknown amino acid and hence identify it as leucine. However, other approaches were also seen and these were given full credit if correct. A common error was incorrect scaling to determine the amount of amino acid used to make the 250 cm^3 solution. A significant number of responses simply multiplied the number of moles of acid by ten rather than using a factor of 250/21.30. Error carried forward marks were available and so the majority of candidates scored 3 or 4 marks.
	b	i	R_f value in range 0.33 – 0.35 ✓	1(AO 1.1)	**ALLOW** 2 SF or more. But ignore digits after second sig fig **ALLOW** 0.3̇ for 0.33….. **Examiner's Comments** Candidates are familiar with the calculation of R_f values and this question was answered very well. Although almost all candidates showed appropriate working as part of their response some candidates gave the final answer to only one significant figure and did not receive credit. Candidates were expected to give their answer to a number of significant figures appropriate to the measurements they had made, so a minimum of two significant figures was expected. The mark scheme allowed a response within a range, rather than a specific value, and most candidates scored in this part.
		ii	gly(cine) ✓ Amino acid matches (leu(cine) and) glycine in Solvent **W** **AND** Amino acid matches (ala(nine) and) glycine in Solvent **X** ✓	2(AO 2.3 ×2)	**ALLOW** glycine has the same/similar Rf as the unknown in both solvents/chromatograms **ALLOW** suitable alternatives for R_f e.g. moves same distance
			Total	**11**	
3		i	**FIRST CHECK ANSWER ON ANSWER LINE IF answer = 7.5 × 10^{-4} award 2 marks** --- **[K] in mol dm^{-3}** $\frac{9.13 \times 10^{-2}}{166} = 5.50 \times 10^{-4}$ (mol dm^{-3}) ✓	**2**	**If there is an alternative answer, Apply ECF** **Alternative method** **[K] in g dm^{-3} with peak area of 5.9** $9.13 \times 10^{-2} \times \frac{5.9}{4.3}$ **OR** $9.13 \times 10^{-2} \times 1.37$

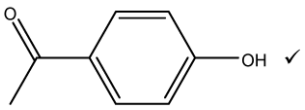
			[L] from peak areas $5.50 \times 10^{-4} \times \frac{5.9}{4.3}$ **OR** $5.50 \times 10^{-4} \times 1.37\ldots\ldots$ $= 7.5 \times 10^{-4}$ (mol dm^{-3}) ✓ ***2 SF Required***		= 0.125 **OR** 0.13 (g dm^{-3}) ✓ Calculator: 0.125272093 **[L] in mol dm^{-3}** $\frac{0.125}{166} = \mathbf{7.5 \times 10^{-4}}$ **OR** $\frac{0.13}{166} = \mathbf{7.8 \times 10^{-4}}$ (mol dm^{-3}) ✓ --- **Common errors:** **Common errors:** **Award 1 mark for:** • 0.099(from $\frac{9.13 \times 10^{-2}}{166} \times 180$) • 6.9×10^{-4} (from $\frac{0.125}{180}$) • 7.2×10^{-4} (from $\frac{0.13}{180}$) • 7.0×10^{-4} (from $\frac{0.25272093}{180}$) **Examiner's Comments** This question required candidates to apply their knowledge of gas chromatography and the mole to solve this problem. Most candidates recognised the need to use the relative peak areas to determine the relative proportion of **M**. Many also realised that division by the molar mass was required to ensure the final answer was given in mol dm^{-3}. However, some used molar mass of **M** rather than **K** in this step, leading to an answer of 7.0×10^{-4} mol dm^{-3}. Answer = 7.5×10^{-4} mol dm^{-3}
		ii	**ester J**  ✓ **esters L and M**  ✓	**3**	**ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **L** and **M** can be identified either way round **IGNORE** 'C_3H_7' in **L** and/or **M** as ambiguous *(question requires structures)* **IGNORE** connectivity of phenol OH group *(marks are for structures of alkyl groups)* **Examiner's Comments**

			 ✓		Examiners were encouraged by the number of good responses to this problem solving question. Most candidates achieved at least one mark in this part, often from a correct structure of **J**. Although many candidates deduced that the R group for both **L** and **M** consisted of 3 C atoms and 7 H atoms, only the highest ability candidates were able to join these correctly. A small but significant number of responses showed R groups that involved O atoms, despite the prompt that the R represented an alkyl group. Candidates are advised to read questions carefully.
			Total	**5**	
4		i	Bromine/ Br_2 **AND** goes colourless/decolourised ✓	**1**	**Note:** both reagent and observation are required **ALLOW** bromine water/ $Br_2(aq)$ **Examiner's Comments** Almost all candidates were able to correctly describe the use of bromine as a test for an unsaturated chain.
		ii	Tollens' (reagent) **AND** Silver (mirror/precipitate/ppt/solid) ✓	**1**	**Note:** both reagent and observation are required for the mark. **ALLOW** ammoniacal silver nitrate **OR** Ag^+/NH_3 **ALLOW** black ppt **OR** grey ppt **Examiner's Comments** Almost all candidates were able to correctly describe the use of Tollens' reagent as a test for an aldehyde functional group.
		ii i	(Add) 2,4-dinitrophenylhydrazine **AND** orange/yellow/red precipitate ✓ Take melting point (of crystals) ✓ Compare to known values/database ✓	**3**	**ALLOW** errors in spelling **ALLOW** 2,4(-)DNP **OR** 2,4(-)DNPH **ALLOW** Brady's reagent or Brady's Test **ALLOW** solid **OR** crystals **OR** ppt as alternatives for precipitate **Mark second and third points independently of response for first marking point** **DO NOT ALLOW** 2nd and 3rd marks for taking and comparing boiling points **OR** chromatograms **Examiner's Comments** The use of 2,4-dinitrophenylhydrazine as a test for the carbonyl group is well known by candidates at this level. The majority of the cohort correctly

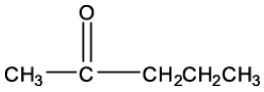
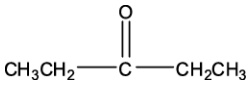
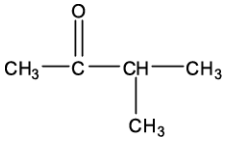
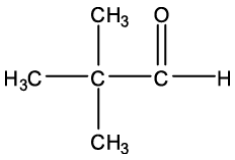
					identified this test and the subsequent analysis of the melting point of the products as a method of identifying each compound. Lower ability candidate responses made reference to analysis of the boiling points of the cinnamaldehyde and methylcinnamaldehyde as a means of identification.
			Total	**5**	
5			*Refer to the marking instructions on page 5 of the mark scheme for guidance on marking this question.* **Level 3 (5–6 marks)** Develops a plan that identifies **all** compounds by a process of elimination **AND** includes essential detail for all required tests and observations *There is a well-developed line of reasoning which is clear and logically structured* **Level 2 (3–4 marks)** Develops a plan that identifies at least half of the compounds **OR** identifies the functional groups in most of the compounds **AND** includes detail of the required tests and observations *There is a line of reasoning with some structure. The information is mostly relevant and supported by some evidence.* **Level 1 (1–2 marks)** Develops a plan that attempts to identify the compounds **OR** functional groups **AND** includes detail of the required tests and observations *There is a line of reasoning using information that is mostly relevant.* **0 marks** – *No response or no response worthy of credit with no compounds identified*	**6**	**Indicative scientific points may include:** **<u>Functional groups</u>** • **B** alkene and tertiary alcohol • **C** alkene and aldehyde • **D** alkene and primary alcohol • **E** ketone • **F** secondary alcohol • **G** alkene and ketone **<u>Tests</u>** • **B, C, D** and **G** → **Bromine** decolourises • **C, D** and **F** → **(H^+/)$Cr_2O_7^{2-}$** green • **C, E** and **G** → **2,4-DNP** orange precipitate • **C** → **Tollens** silver mirror For Tollens' **ALLOW** alternative: Fehling's solution produces a 'brown / brick red / orange' precipitate For 2,4- DNP, **ALLOW** 2,4-DNPH and Brady's **No credit for tests on products of tests, melting points, spectra, etc.** ***For other tests seen, contact TL for advice*** **Examiner's Comment:** This question required candidates to apply their knowledge of functional group tests for several naturally occurring organic compounds. Candidates were free to plan a series of chemical tests and

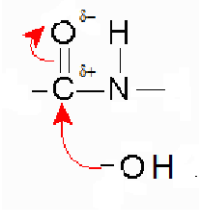
	B	C	D	E	F	G
Bromine	✓	✓	✓			✓
(H^+/)$Cr_2O_7^{2-}$		✓	✓		✓	
2,4-DNP		✓		✓		✓
Tollens'		✓				

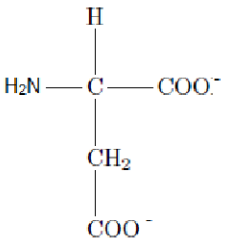
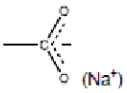
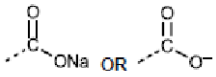
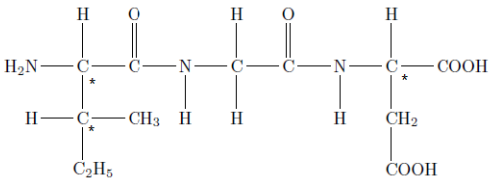
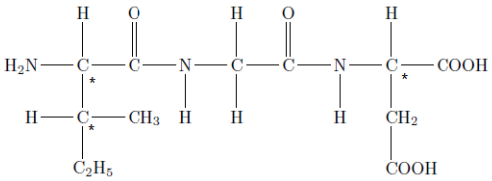
					there were many different solutions. The best responses planned to identify compounds by using tests that eliminated compounds one by one for further tests. These were often concise and very clear. Unfortunately, many responses were excessive in length with confusing plans that sometimes broke down partway through. Some candidates gave chemical tests for the functional groups in each compound in turn. They then didn't link the tests together to show how the compounds could be distinguished. Weaker candidates sometimes wrote down chemical tests for the different functional groups without linking these to the compounds. Poorly prepared candidates sometimes had the wrong observations for the tests. A significant number of candidates thought that the cyclic alcohol F was a phenol. Despite these difficulties, there were many candidates who scored all six marks by a variety of methods.
			Total	**6**	
6	a		**Empirical formula** Mole Ratio C : H : O = 5.88 : 5.92 : 1.47 ✓ Empirical formula = C_4H_4O ✓ **Molecular formula** Molecular formula = $C_8H_8O_2$ **AND** Evidence of 136 in working or from labelled peak in spectrum ✓	**3**	**ANNOTATE ANSWER WITH TICKS AND CROSSES** **ALLOW** $\frac{70.58}{12.0} : \frac{5.92}{1.0} : \frac{23.50}{16.0}$ **ALLOW** 4:4:1 if linked to C:H:O Alternative method for 3 marks: C: $\frac{136 \times 70.58/100}{12.0} = 8$ H: $\frac{136 \times 5.92/100}{1.0} = 8$ O: $\frac{136 \times 23.50/100}{16.0} = 2$ **Examiner Comments** The empirical formula was correctly calculated by all but the weakest candidates. The final mark was more difficult to obtain as it required evidence that the molar mass had been determined from the mass spectrum and used in establishing the molecular formula.
	b		**Functional groups** Phenol **AND** ketone ✓ **Explanation** Links phenol to (weak) acidity **AND** no reaction with Na_2CO_3 (so not carboxylic acid) ✓ Links 2,4-DNP(H) or Brady's reagent observation to	**3**	**DO NOT ALLOW** any other functional groups for first marking point. **ALLOW** identity of functional groups in the explanation if not stated on functional group prompt line.

			carbonyl **AND** Tollens' reagent observation (so not an aldehyde) ✓		**ALLOW** "aldehyde or ketone" in place of carbonyl **Examiner Comments** Many candidates were able to suggest that the compound contained a ketone but found it more difficult to indicate the presence of phenol. Approximately 20% of the entry obtained all three marks. When explaining the presence of the ketone some failed to indicate that the 2,4-DNP test indicated that the compound must contain a carbonyl and just focused on the lack of reactivity with Tollens'. Answers suggesting the molecule contained a ketone as no reaction was observed with Tollens' did not gain credit when no reference to carbonyl was seen. Those who recognised the presence of a phenol explained that the only acidic functional group that does not react with sodium carbonate is a phenol.
	c		**Carbon NMR analysis** Peaks between 110–160 ppm are the (four) aromatic (carbon environments) ✓ Compound contains a C=O between 190 – 200 ppm **AND** Compound contains a C-C at 20 – 30 ppm ✓ **Structure**  ✓	**3**	**ALLOW** peaks to be identified by: • Peaks labelled on spectrum • Peaks indicated on a chemical structure • Peaks indicated from within text **Note:** If identifying aromatic peaks from the spectrum all four peaks should be indicated. **ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **Examiner Comments** When interpreting a carbon-13 NMR spectrum, candidates should be advised to fully label any peaks. Many candidates failed to indicate the presence of four aromatic peaks yet produced a structure containing a benzene ring. In some cases candidates did not link their answer to part (a) of the question giving structures that did not match their molecular formula.
			Total	**9**	
7	a	i	**Reagent and observation** sodium carbonate **AND** Fizzing/effervescence/bubbling ✓ **Equation** Correctly balanced equation ✓ e.g. $2RCOOH + Na_2CO_3 \rightarrow 2RCOONa + CO_2 + H_2O$	2	**Note:** both reagent and observation are required for first mark **ALLOW** name or formula for any suitable carbonate e.g $NaHCO_3$, potassium carbonate etc. **ALLOW** reagent from equation if not stated elsewhere
		ii	**Reagent and observation** Tollens' (reagent) **AND**	2	**Note:** both reagent and observation are required for first mark

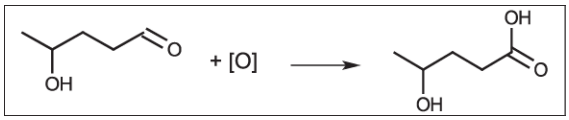
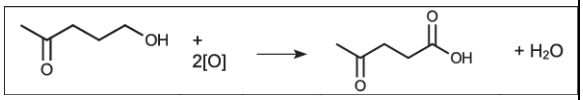
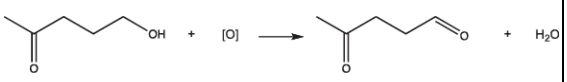
			Silver (mirror) ✓ **Equation** RCHO + [O] → RCOOH ✓		**ALLOW** ammoniacal silver nitrate **OR** Ag^+/NH_3 **ALLOW** $H^+/Cr_2O_7^{2-}$ **OR** acidified (potassium/sodium) dichromate **AND** Orange to green *(this would identify the aldehyde from the carboxylic acid, ketone and esters)*
	b		2,4−dinitrophenylhydrazine **AND** Orange/yellow/red precipitate ✓	1	**ALLOW** errors in spelling **ALLOW** 2,4(−)DNP **OR** 2,4(−)DNPH **ALLOW** Brady's reagent or Brady's Test **ALLOW** solid **OR** crystals **OR** ppt as alternatives for precipitate
	c	i	$CH_3COOC(CH_3)_3 + NaOH \rightarrow CH_3COONa + (CH_3)_3COH$ CH_3COONa ✓ Rest of equation correct ✓ **OR** $(CH_3)_3CCOOCH_3 + NaOH \rightarrow (CH_3)_3CCOONa + CH_3OH$ $(CH_3)_3CCOONa$ ✓ Rest of equation correct ✓	2	**Note:** the hydrolysis of either ester may be given **ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **DO NOT ALLOW** molecular formulae of products *(question requires structures of products to be shown)*
		ii	**Reagent and observation** $H^+/Cr_2O_7^{2-}$ **OR** acidified (potassium/sodium) dichromate **AND** Orange to green (with CH_3OH) ✓ **Equation** $CH_3OH + [O] \rightarrow HCHO + H_2O$ **OR** $CH_3OH + 2[O] \rightarrow HCOOH + H_2O$ ✓	2	**ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **DO NOT ALLOW** molecular formulae *(question requires structures of organic compounds to be shown)*
		ii i	**^{13}C NMR (1 mark)** (It is) not possible to identify (the esters) with ^{13}C NMR **AND** (both) spectra would contain four peaks (with similar chemical shifts) ✓ **^{1}H NMR (2 marks)**	3	**ALLOW** 'same number of peaks' in place of 'four peaks'

			(It is) possible to identify (the esters) with ^{1}H NMR (^{1}H NMR spectrum of) $CH_3COOC(CH_3)_3$ has a singlet/peak between 2.0−3.0 (ppm) (^{1}H NMR spectrum of) $(CH_3)_3CCOOCH_3$ has a singlet/peak between 3.0−4.3 (ppm) All **three** correct statements✓✓ Any **two** correct statements ✓		**ALLOW** any value or range of values within 2.0−3.0 **ALLOW** any value or range of values within 3.0−4.3
	d		**Possible structures for ketone (2 marks)**    All **three** correct ✓✓ Any **two** correct ✓ **Aldehyde (3 marks)** Peak at (δ) 1.2 shows HC–R **AND** No H on adjacent C atom as peak is singlet ✓ Peak at (δ) 9.6 shows H–C=O **AND** No H on adjacent C atom as peak is singlet ✓  **OR** (2,2−)dimethylpropanal ✓	5	**ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **IGNORE** names of ketones
			Total	**17**	
8		i	Leucine **AND** valine ✓	1	

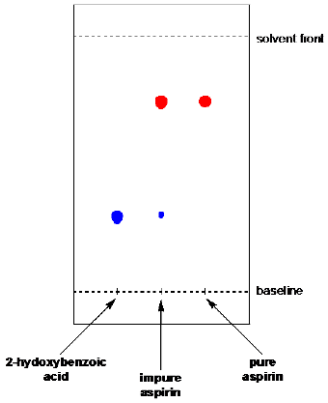
		ii	R_f values would be larger ✓ (amino acids) are more soluble (in more polar solvent so would travel further up the plate) ✓	2	
			Total	**3**	
9	a		 Curly arrow from OH^- to C(δ+) ✓ Dipole correct **AND** curly arrow from C=O bond to O(δ−) ✓	2	First curly arrow must come from either a lone pair on O or negative charge on O **Examiner's Comments** Some candidates lost a mark for the incorrect positioning of the curly arrow from the hydroxide ion. The mark scheme specifies that it should start at either the lone pair on the oxygen atom or the negative change on the oxygen atom.
	b		Measure distance moved by spot/distance moved by solvent ✓ Compare (R_f) value with data book values/known values ✓ Two amino acids have the same/similar R_f value **OR** similar adsorption **OR** move the same/similar distance ✓	2 1	**ALLOW** attempt at calculation of R_f value using distances measured on the chromatogram **IGNORE** explanation of how chromatography works **ALLOW** One spot contains two amino acids **ALLOW** Two amino acids have not separated **IGNORE** relative solubility **ALLOW** two of the amino acids have similar structures **Examiner's Comments** This question discriminated well with relatively few candidates able to score all three marks. Some candidates attempted to explain how the technique separates components between a mobile phase and a stationary phase which was not required by the question. There was some confusion with gas chromatography and retention times. Vague answers about all amino acids having similar structures did not score the final marking point to explain why only two spots appeared on the chromatogram.

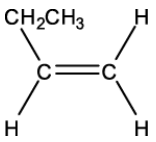
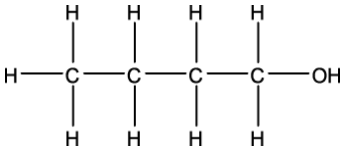
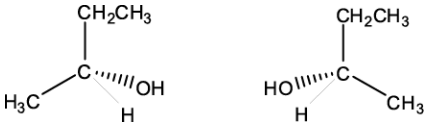
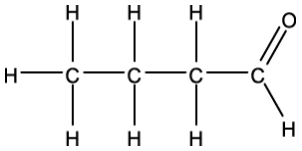
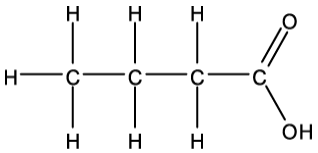
	c	i	The **pH** at which the amino acid exists as a zwitterion ✓ **QWC**: **zwitterion** spelled correctly in the correct context	1	**DO NOT ALLOW** PH/ph **ALLOW** zwitter ion **Examiner's Comments** This definition had been learned by the majority of candidates.
		ii		1	**ALLOW** correct structural **OR** displayed **OR** skeletal formulae **OR** combination of above as long as unambiguous **Two** COO^- groups are required in the structure **ALLOW** $–COO^-Na^+$ **OR** –COONa  **ALLOW** delocalised carboxylate **ALLOW**  **DO NOT ALLOW** -COO-Na **OR** -O-Na (covalent bond) **Examiner's Comments** Generally well answered but structures with only one carboxylate group were quite common and some candidates showed aspartic acid being protonated at high pH.
		ii i	**M1** structure  ✓ **M2** correct structure has three chiral centres  ✓	2	**ALLOW** correct structural **OR** displayed **OR** skeletal formulae **OR** a combination of above as long as unambiguous **ALLOW** tripeptide with the 3 amino acids in any order **ALLOW** cyclic tripeptide Isoleucine has two chiral centres, aspartic acid has one chiral centre and glycine has none. **ALL three** correct for one mark **ALLOW** chiral centres correctly identified if the three amino acids are part of a polypeptide chain **Examiner's Comments**

					A more challenging question with relatively few candidates able to show the position of all three chiral centres on a correct tripeptide structure. Most candidates identified the chiral centres in the amino acid backbone of aspartic acid and isoleucine and realised that glycine was not chiral, but many missed the second chiral centre in isoleucine.
			Total	**9**	
10	a		(Relative) solubility (in stationary phase) ✓	1	**ALLOW** how well the compound dissolves **IGNORE** retention time **AND** partition **DO NOT ALLOW** adsorption **OR** absorption **Examiner's Comments** The analysis of mixtures using gas chromatography was not well understood. The specification states that a liquid stationary phase separates by relative solubility and many incorrect answers focussed on the adsorption of molecules onto the solid beads rather the relative solubility of molecules in the liquid polymer that coated the beads. No credit was given to answers that stated that the separation produced different retention times.
	b	i	Compound **B** **AND** M^+ / molecular ion peak (at m/z) = 124 ✓	1	**ALLOW Mr** = 124 **IGNORE** compound B because *m*/*z* = 124 **ALLOW** $C_7H_8O_2^+$ = 124 **OR** $C_7H_8O_2$ = 124 **ALLOW** peak at (m/z =) 109 due to $HOC_6H_4O^+$ **ALLOW** peak at (m/z =) 109 due to loss of CH_3 **IGNORE** reference to other peaks in the spectrum **Examiner's Comments** This question was well answered. The majority of candidates focussed on the different molar masses of the compounds and many referred to the M^+ peak or molecular ion peak of compound **B**.
		ii	Compound (B) is less soluble in the stationary phase / liquid	1	**ORA** Answer refers to the first compound to emerge from the column **ALLOW** compound (B) is more soluble in mobile phase / gas **ALLOW** compound interacts less with stationary phase / liquid **OR** compound interacts more with mobile phase / gas **IGNORE** compound adsorbs less **IGNORE** compound is not very soluble (comparison needed) **IGNORE** volatility **OR** reactivity **Examiner's Comments**

					Relatively few creditworthy responses were seen here. The specification describes chromatography as an analytical technique that separates components in a mixture between a mobile phase and a stationary phase. Many candidates referred to differences in solubility or the strength of interactions without linking this to a particular phase in the column.
			Total	**3**	
11			**FIRST** react **all** with Tollens' reagent **AND** silver mirror / ppt / solid (formed) with compound **D** **OR** with Fehling's / Benedict's solutions **AND** (brick-red / orange) solid / precipitate (formed) with compound **D** ✓ ***NOTE**: eliminates **D***  ✓ **THEN** react **C** and **E** with H_2SO_4 / H^+ **AND** $K_2Cr_2O_7$ / $Cr_2O_7^{2-}$ / $Na_2Cr_2O_7$ **AND** colour change **OR** green colour with compound **C** **OR** no change **OR** no reaction **OR** no green colour with compound **E** ✓ 	4	**ALLOW** ammonia + silver nitrate for reagent **ALLOW** black solid / ppt **ALLOW** 'the aldehyde gives a silver mirror' **ALLOW** solid **OR** crystals **OR** ppt as alternatives for precipitate **ALLOW** correct structural **OR** displayed **OR** skeletal formulae **OR** combination of above as long as unambiguous **DO NOT ALLOW** molecular formulae for organic structures **IGNORE** all references to 2,4-dinitrophenylhydrazine / Brady's **ACCEPT** acidified dichromate **ALLOW** blue / green blue **IGNORE** equation for oxidation of **D** **ALLOW** equation for partial oxidation  **ALLOW** alternative sequences e.g. **FIRST** react **all** with H_2SO_4 **AND** $K_2Cr_2O_7$ colour change with **C** and **D** *eliminates **E*** At least one correct equation and structure of one product from either reaction required for the second mark. **NB** several possible products for the oxidation of **D**

					THEN react **C** and **D** with Tollens'…… *distinguishes between **C** and **D*** **Examiner's Comments** This question discriminated well. Most candidates were able to score at least one mark for the correct use of Tollens' reagent. Answers needed to refer to test tube reactions and any reference to the use of Brady's reagent was ignored since it would give a similar observation with all three compounds. Many excellent answers described a logical sequence of tests involving the use of ammoniacal silver nitrate and acidified potassium dichromate. Some neglected to acidify the dichromate and equations were quite often missing from the answer. The best candidates included full balanced equations which clearly showed the structures of the reactants and products.
			Total	**4**	
12	a		** Please refer to the marking instruction point 10 for guidance on how to mark this question.* **Level 3 (5–6 marks)** Correctly labelled diagram of apparatus that works, with no safety problems **AND** Full appreciation of further two steps required to gain pure sample *There is a well-developed diagram which is clear and structured. The information on further purification is detailed and relevant.* **Level 2 (3–4 marks)** Labelled diagram of apparatus but with safety / procedural problems **OR** clear diagram of functional apparatus without labelling **AND** Some details of further purification steps *The diagram presents apparatus that is in the most-part relevant with some correct labelling, and supported by some details of further purification steps.* **Level 1 (1–2 marks)** Diagram of apparatus drawn with no labelling **OR** labelled diagram with significant safety / procedural problems **AND** Few or imprecise details about further purification stages	6	**Indicative scientific points may include:** **Diagram** Includes following components: distillation flask heat source thermometer at outlet (bulb **level** with outlet) still-head water condenser (**correct direction** of water flow) receiving vessel **open** system. **Further purification** Shake and leave to settle in a separating funnel Separate layers by tapping off Add (a small amount of) anhydrous magnesium sulfate / anhydrous calcium chloride to organic layer (in a dry conical flask) (Re)distil the organic layer Collect fraction distilling at (between 150 °C and) 156 °C.

			The diagram is basic and unstructured. Any mention of purification steps is limited to generic term, e.g. 'drying', without relevant detail. **0 marks** No response or no response worthy of credit.		
	b		Lack of (further) effervescence	1	**ALLOW** fizzing / bubbling stops
	c		Take samples from reaction mixture at regular intervals Spot / run on a TLC plate, alongside cyclohexanol (and cyclohexanone) controls	2	**ALLOW** "frequent" for "regular" **ALLOW** measure / compare R_f value to cyclohexanol **IGNORE** reference to solvent or visualising chemicals / UV
	d		React (sample of distillate) with 2,4-dinitrophenylhydrazine recrystallise **AND** determine the melting point Compare melting point to known / library value for cyclohexanone (derivative)	3	**ALLOW** (2,4-)DNPH / Brady's reagent
			Total	**12**	
13		i	 Pure aspirin and 2-hydroxybenzoic acid correct Impure aspirin with 2 spots in line with aspirin and 2-hydroxybenzoic acid spots **AND** 2-hydroxybenzoic acid spot fainter than aspirin spot	2	Check measurements on diagram using online measuring tool. Distance from baseline to top of spot for aspirin = 70–80% of baseline → solvent front Distance from baseline to top of spot for aspirin = 25–35% of baseline → solvent front
		ii	Melting point range between 130–140°C **AND** Range ≥ 2°C	1	Range that starts <138 and finishes ≤140
			Total	**3**	
14	a		**F–K** clearly identified **Compound F:**	6	**ANNOTATE ANSWER WITH TICKS AND CROSSES** **ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous

					**IGNORE** names
			Compound G:		
					
			Compounds H and I:		**H** and **I** can be identified either way round
					
			Compound J:		
					
			Compound K:		
					
	b		(Add) 2,4-dinitrophenylhydrazine **AND** orange/yellow/red precipitate	3	**NOTE: (b)** is marked completely independently of **(a)** ALLOW errors in spelling **ALLOW** 2,4(−)DNP **OR** 2,4(−)DNPH **ALLOW** Brady's reagent or Brady's Test **ALLOW** solid **OR** crystals **OR** ppt as alternatives for precipitate
			Take melting point of crystals		**Mark second and third points independently of response for first marking point**
			Compare to known values		**DO NOT ALLOW** 2nd and 3rd marks for taking and comparing boiling points **OR** chromatograms
			Total	**9**	