Mark scheme - Synthesis

<table>
<tr><th colspan="3">Question</th><th>Answer/Indicative content</th><th>Marks</th><th>Guidance</th></tr>
<tr><td>1</td><td>a</td><td>i</td><td>
Please refer to the marking instructions on this mark scheme for guidance on how to mark this question.

Level 3 (5–6 marks)
Calculates the correct mass of hexan-1-ol.
AND
Explains the purification steps, with most fine detail.

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)
Attempts a calculation of the mass of hexan-1-ol which is partly correct.
OR
Outlines the purification steps, with some fine detail.

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)
Attempts the calculation but makes little progress.
OR
Briefly outlines the purification steps, which may be incomplete.

There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant.

0 marks
No response or no response worthy of credit
</td><td>
6

(AO2.8×2)

(AO3.3×4)
</td><td>
Indicative scientific points may include:
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Calculation from moles
• n(hex-1-ene) = $\frac{4.20}{84.0}$ = 0.0500 (mol)
• n(hexan-1-ol) needed = 0.0500 × $\frac{100}{62.5}$ = 0.0800 (mol)
• mass needed = 0.0800 × 102 = 8.16 g
• OR volume $\frac{8.16}{0.82}$ = 9.95 cm^3

CHECK for extent of errors by ECF.
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Calculation from mass
• Theoretical mass hex-1-ene = 4.20 × $\frac{100}{62.5}$ = 6.72 g
• Theoretical n(hex-1-ene) = $\frac{6.72}{84}$ = 0.0800 (mol)
• Mass of hexan-1-ol = 102 × 0.0800 = 8.16 g

ALLOW small slip/rounding errors such as errors on M_r (e.g. use of 83 instead of 84 for hex-1-ene M_r)

Purification
• Use of a separating funnel to separate organic and aqueous layers
• Drying with an anhydrous salt
• Distillation

Fine detail
• Collection of upper layer (less dense from separating funnel)
• Example of drying agent, e.g. $MgSO_4$, $CaCl_2$
• Collection of fraction distilling at 63°C (boiling point of hex-1-ene)

Incorrect purification method NOT creditworthy
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Examples of partly correct calculations

Mass = 5.1 g from 0.0500 × 102 % yield omitted
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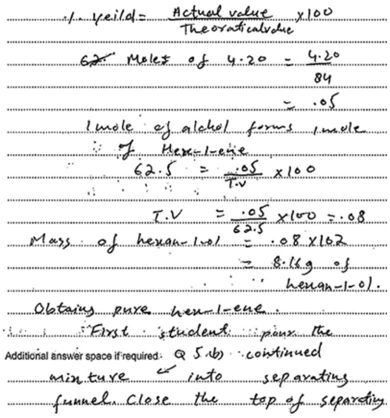
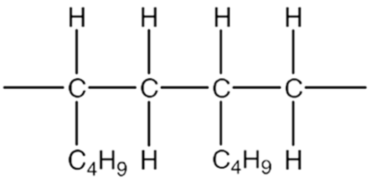
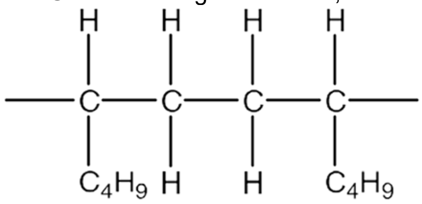
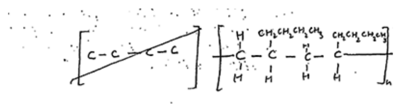
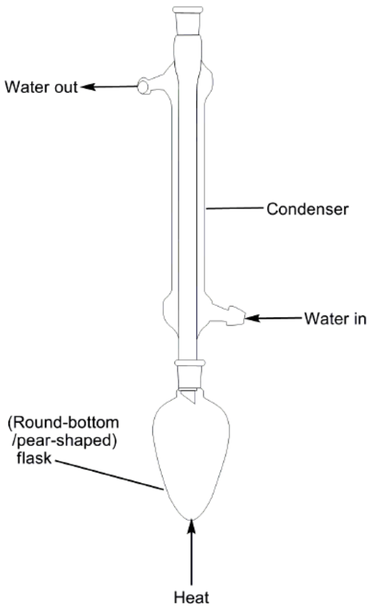
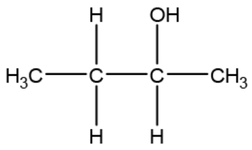
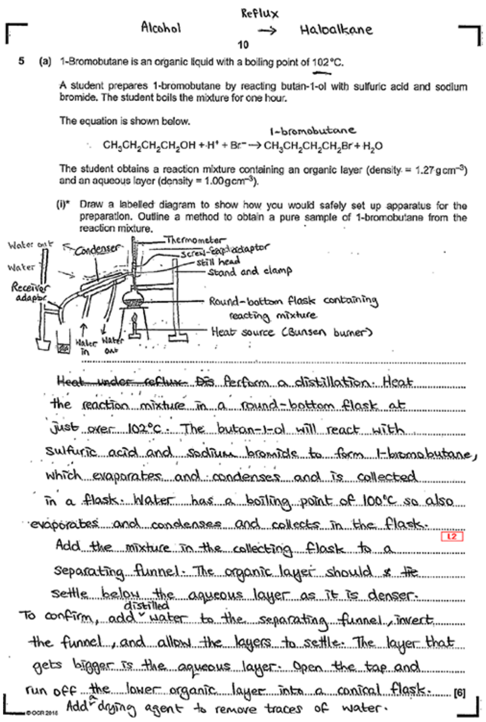
					Mass = 3.1875 g from $0.0500 \times \frac{62.5}{100} \times 102$ *% yield inverted* **<u>Examiner's Comments</u>** This question discriminated well and most were able to attempt to calculate a mass and explain the purification steps with some fine detail. It was evident that most candidates were aware of the apparatus required but the logic in the order was sometimes out of sequence. Drying agents were mentioned, some of the examples used were incorrect and errors were made with the boiling points if candidates mentioned distillation. **Exemplar 5**  % yeild = Actual value / Theoraticalvalue x100 Moles of 4.20 = 4.20 / 84 = .05 1 mole of alchol forms 1 mole of Hex-1-ene 62.5 = .05 / T.V x100 T.V = .05 / 62.5 x100 = .08 Mass of hexan-1-ol = .08 x102 = 8.16g of hexan-1-ol. Obtaing pure hex-1-ene. First student pour the Additional answer space if required: Q 5 (b) continued mixture into separating funnel. Close the top of separating This in a response of an excellent approach to this question, it was given 6 marks. The correct mass has been calculated and all of the fine detail, except distilling at 63°C
		ii	Yield of hex-1-ene is less ✓ A mixture of hex-1-ene and hex-2-ene forms ✓	2(AO3.2×2)	**ALLOW** hex-2-ene also forms **<u>Examiner's Comments</u>** Most candidates obtained the first mark, but very few stated that hex-2-ene forms which is why the yield is less. Many candidates stated this was due to secondary alcohols being more stable than primary ones and a significant number thought the yield would be the same as they had the same M_r.

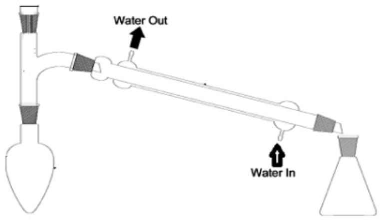
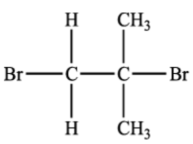
	b	i	 ✓ **NOTE**: C4H9– is allowed for $CH_3CH_2CH_2CH_2$–	1(AO2.5)	**ALLOW** correct structural **OR** displayed **OR** skeletal formula Must show two repeat units Polymer must have side links **IGNORE** brackets and use of ‘n’ **ALLOW** alternating side chains, i.e.  **<u>Examiner’s Comments</u>** Over half of the candidates answered this question incorrectly. Many drew the wrong repeat units or did not show two repeat units. The structural diagrams were very hard to decipher and not well drawn. **Exemplar 6**  Although the connectivity is not exact, this was allowed. Many students drew full displayed structures which were very difficult to decipher as they often encroached over the writing in the question.
		ii	Combustion for energy production ✓ for production of plastics **OR** other useful **organic** compounds ✓	2(AO1.1×2)	For energy production, **ALLOW** generate electricity/heating **ALLOW** as an (organic) feedstock **<u>Examiner’s Comments</u>** Most students did not gain many marks on this question, with a significant number stating that the plastics could be used to feed livestock or as fertilisers. Those that mentioned combustion, merely stated plastics could be used as a fuel. Some candidates made references to making them biodegradable or recycling, but these answers were not given.
			Total	**11**	

2		i	**Diagram** Diagram showing round bottom/pear shaped flask **AND** upright condenser ✓  **Labels** (Round-bottom/pear-shaped) flask **AND** condenser **AND** water in at bottom and out at top **AND** heat (source) ✓	**2**	**DO NOT ALLOW** conical flask, volumetric flask, beaker in place of round bottom/pear shaped flask **DO NOT ALLOW** distillation **DO NOT ALLOW** stopper/bung on top of condenser **IGNORE** a thermometer in condenser **IGNORE** a small gap between flask and condenser **ALLOW** diagram of heating apparatus as an alternative to heat label <u>**Examiner's Comments**</u> Most candidates were able to draw a suitable diagram to show the apparatus required for reflux but some included a stopper on top of the condenser. Many of the diagrams were labelled appropriately but common errors included incorrect direction of water flow or omission of the 'flask' label. A small but significant proportion of candidates drew a diagram showing distillation.
		ii	**Precipitate G** **1 mark** silver bromide/AgBr **AND** *M* = 1.88/0.01 = 188 (g mol^{-1}) 188 – 107.9 = 80.1 (so halide is Br^{-})✓ **Alcohol F and Haloalkane E** **2 marks** **E** and **F** clearly identified **F/alcohol:** butan-2-ol	**3**	**ALLOW** any combination of skeletal **OR** structural **OR** displayed formula as long as unambiguous **Note:** working is **required** for first mark **ALLOW** use of 108 as *A*r of Ag **Note: E** and **F** can be identified by correct name or structure **BUT IGNORE** incorrect names

			 **E/haloalkane:** E is haloalkane of C_4H_9X with • same halogen as **G** **AND** • same carbon chain as **F** ✓		**Examiner's Comments** This question, requiring candidates to analyse the information to identify compounds **E**, **F** and **G**, discriminated well. Many candidates deduced that G was a silver halide but not all provided working to back up their choice of AgBr. Some candidates appeared to guess and Ag*Cl* was commonly seen. Some candidates used the molar mass of **F** provided to deduce the molecular formula of $C_4H_{10}O$ but lower ability responses did not process this further. Higher ability candidates identified **F** as butan-2-ol, showing the chiral carbon clearly. Other alcohols were also seen including butan-1-ol and methylpropan-2-ol. The highest ability candidates linked all the information and provided a structure for **E** that was consistent with their suggestions for **F** and **G**.
			Total	**5**	
3		i	*Please refer to the marking instructions on page 5 of this mark scheme for guidance on how to mark this question.* **Level 3 (5–6 marks)** Correctly labelled diagram of reflux apparatus that works, with no safety problems **AND** An appreciation of most of the purification steps required to gain a pure sample *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)** Labelled diagram of apparatus (either reflux or distillation) but with safety/procedural problems **OR** clear diagram of reflux apparatus without labelling **AND** Some details of further purification steps *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)** Diagram of apparatus (**reflux OR separation OR distillation**) drawn with no labelling **OR** labelled diagram with significant safety/procedural	6	**Indicative scientific points may include:** **Apparatus set up for reflux:** • round-bottom/pear shaped flask • heat source • condenser *Detail: water flow in condenser bottom to top; open system.* **Purification** • Use of a **separating funnel** to separate organic and aqueous layers *Detail: Collect lower organic layer density greater* • **Drying** with an anhydrous salt, *Detail: e.g. $MgSO_4$, $CaCl_2$, etc.* • **Redistillation** *Detail: Collect fraction distilling at 102°C.* **Examiner's Comments** Candidates were not prepared to answer this type of question and the diagrams were hard to give credit to. Many had significant safety implications such as open beakers of butan-1-ol being heated by a Bunsen burner. Most mis-read the question and just outlined the method for purification and struggled to recall the practical details. Very few candidates mentioned the use of anhydrous salts,

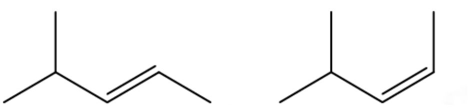
			AND / OR Few or imprecise details about further purification stages *There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant.* **0 marks** No response or no response worthy of credit.		referring instead to 'boiling off' the water. **Exemplar 4**  Reflux Alcohol → Haloalkane 10 5 (a) 1-Bromobutane is an organic liquid with a boiling point of 102 °C. A student prepares 1-bromobutane by reacting butan-1-ol with sulfuric acid and sodium bromide. The student boils the mixture for one hour. The equation is shown below. 1-bromobutane $CH_3CH_2CH_2CH_2OH + H^+ + Br^- \rightarrow CH_3CH_2CH_2CH_2Br + H_2O$ The student obtains a reaction mixture containing an organic layer (density = 1.27 g cm^{-3}) and an aqueous layer (density = 1.00 g cm^{-3}). (i)* Draw a labelled diagram to show how you would safely set up apparatus for the preparation. Outline a method to obtain a pure sample of 1-bromobutane from the reaction mixture. ~~Heat under reflux.~~ ~~Dis~~ Perform a distillation. Heat the reaction mixture in a round-bottom flask at just over 102°C. The butan-1-ol will react with sulfuric acid and sodium bromide to form 1-bromobutane, which evaporates and condenses and is collected in a flask. Water has a boiling point of 100°C so also evaporates and condenses and collects in the flask. L2 Add the mixture in the collecting flask to a separating funnel. The organic layer should ~~the~~ settle below the aqueous layer as it is denser. To confirm, add distilled water to the separating funnel, invert the funnel, and allow the layers to settle. The layer that gets bigger is the aqueous layer. Open the tap and run off the lower organic layer into a conical flask. [6] Add a drying agent to remove traces of water. ©OCR 2016 This candidate was credited 4 marks for this level 2 answer. Although they have drawn distillation apparatus instead of reflux, they have considered the boiling point of the product, detailed using a separating funnel, a drying agent and that the lower organic layer would be drawn off first.
		ii	**FIRST, CHECK THE ANSWER ON ANSWER LINE** **IF answer = 12.6 (g) award 2 marks** • n(1-bromobutane) = $0.150 \times \frac{61.4}{100}$ = 0.0921 (mol) ✓	2	**Common errors:** 33.4 (0.150 × 100/61.4 = 0.244 × 136.9) 1 mark **ALLOW ECF** for incorrect moles or incorrect M_r **of 1-bromobutane** (provided answer is to

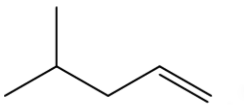
			• **Mass 1-bromobutane = 0.0921 × 136.9 = 12.6 (g) ✓** **3 SF required**		3 SF) **DO NOT ALLOW** 6.82 (using M_r of butan-1-ol) **ALLOW** calculation using masses, e.g. • **Theoretical = 0.150 × 136.9 = 20.535 (g) ✓** ***(ALLOW 20.535 rounded back to 20.5)*** • Actual mass = 20.535 × $\frac{61.4}{100}$ = 12.6 (g) ✓ *(20.5 also gives 12.6)* **Examiner's Comments** This question was well answered, but a significant number of candidates incorrectly used the Mr of butan-1-ol when calculating the mass of 1-bromobutane.
			Total	**8**	
4			*Please refer to the marking instructions on page 5 of the mark scheme for guidance on how to mark this question.* **Level 3 (5–6 marks)** A comprehensive explanation with all three scientific points covered thoroughly. *There is a well-developed description with a logical structure including correct chemical equations and an explanation with a clear line of reasoning including a fully labelled diagram.* **Level 2 (3–4 marks)** The candidate attempts all three scientific points but explanations are incomplete. **OR** Explains two scientific points thoroughly with no omissions. *The description has a line of reasoning presented with some structure and includes correct structural formulae and an accurate diagram of a distillation apparatus.* **Level 1 (1–2 marks)** A simple explanation based on at least two of the main scientific points **OR** The candidate explains one scientific point thoroughly with few omissions. *The description may be communicated in an unstructured way but it includes the correct*	**6**	**Indicative scientific points** **1. Oxidation reaction forming aldehyde** • acid / H+ **AND** dichromate / $Cr_2O_7^{2-}$ • heat **AND** distillation • organic product is butanal / $CH_3CH_2CH_2CHO$ • $CH_3CH_2CH_2CH_2OH + [O] \rightarrow CH_3CH_2CH_2CHO + H2O$ **2. Oxidation reaction forming carboxylic acid** • acid / H+ **AND** dichromate / $Cr_2O_7^{2-}$ • heat under reflux • organic product is butanoic acid / $CH_3CH_2CH_2COOH$ • $CH_3CH_2CH_2CH_2OH + 2[O] \rightarrow CH_3CH_2CH_2COOH + H_2O$ **3. Distillation** • diagram of apparatus with condenser • condenser has water flow • collection of organic product • product is separated to prevent further oxidation (to carboxylic acid)

			reagents and conditions for the formation of the aldehyde. *There is an attempt at a logical structure with a line of reasoning. The information is in the most part relevant.* **0 marks–***No response or no response worthy of credit.*		 **Examiner's Comment:** A very wide range of responses was seen in the second question marked using a level of response mark scheme and a greater proportion of candidates were able to access the highest level in this question. Diagrams of a distillation apparatus were often disappointing and many poor answers failed to identify the oxidation products. A Level 1 response usually named the oxidising agent and included a crude diagram of a distillation apparatus. Diagrams in Level two responses often included more detail with a condenser cooled by water flow and an indication of where butanal can be collected. A Level three response was expected to include balanced equations for the oxidation reactions.
			Total	**6**	
5		i	C_2H_5O ✓	1	**ALLOW** elements in any order **DO NOT ALLOW** any other answer <u>**Examiner's Comments**</u> This part was answered well by most candidates. Some candidates however wrote the molecular rather than the empirical formula, or attempted to show the empirical formula as C_2H_4OH instead of C_2H_5O.
		ii	Compound E:  ✓ **Stage 1**: Compound **E**: Bromine/Br_2 ✓ NaOH/KOH **OR** OH^- ✓ **Stage 2**: *Only award if intermediate contains at least* ***one*** *halogen atom*	3	**For structures:** **ALLOW** correct structural **OR** skeletal **OR** displayed formula **OR** mixture of the above **ALLOW** dichloro/diiodo compound **IGNORE** connectivity of bonds to CH_3 **ALLOW** chlorine/Cl_2 **OR** iodine/I_2 **IGNORE** conditions, e.g. u.v. **DO NOT ALLOW** H_2O **IGNORE** conditions

					NOTE: Max of **2 marks** available for **monobrominated** intermediate **1 mark** Reagent: HBr **AND** Intermediate: $CH_3C(CH_3)_2Br$ **OR** $BrCH_2CH(CH_3)_2$ **1 mark** Intermediate: $CH_3C(CH_3)_2Br$ **OR** $BrCH_2CH(CH_3)_2$ **AND** Reagent: NaOH **Examiner's Comments** This demanding part was answered poorly by weaker candidates and was good for differentiating higher ability candidates. The mark scheme allowed some credit for using a hydrogen halide to obtain a monosubstituted haloalkane for compound E. Surprisingly, reaction mechanism names were often given instead of reagents. Many candidates seemed to guess, sometimes showing the same reagents for both stages in the hope of getting a mark. Many showed an intermediate containing no halogen atom.
			Total	**4**	
6		i	**Equation** $CH_3CH(OH)CH_2CH_3 + [O] \rightarrow CH_3COCH_2CH_3 + H_2O$ ✓ **Structure of product could be allowed from equation** $CH_3COCH_2CH_3$ ✓	2	**ALLOW** molecular formulae: $C_4H_{10}O$ and C_4H_8O **ALLOW** C_4H_9OH **ALLOW** C_2H_5 for CH_3CH_2 **ALLOW** correct structural **OR** displayed **OR** skeletal formulae **OR** a combination of above as long as unambiguous **Examiner's Comments** The majority of candidates were able to identify the structure of the ketone formed in the oxidation of butan-2-ol but many were not able to construct a suitable equation. Water was often omitted from the equation on the right hand side whilst sometimes the equation was incorrectly balanced with a 2 being placed in front of the [O]. The most able candidates normally scored both marks.

		ii	Butan-2-ol/butanone is flammable **OR** Butan-2-ol / butanone is volatile / low boiling point **OR**		**IGNORE** vague answers about health and safety **ALLOW** alcohol for butan-2-ol **ALLOW** ketone for butanone
		ii	Butan-2-ol / butanone will evaporate / boil away ✓	1	**DO NOT ALLOW** the product or reactant. **DO NOT ALLOW** distillation
		ii	(Heat under) reflux **OR** a description of reflux with vertical condenser and a round bottomed or pear shaped flask with source of heat. ✓	1	**DO NOT ALLOW** any reference to closed system. **Examiner's Comments** Another question requiring candidates to evaluate a practical activity where responses were on the whole disappointing. Very few candidates were able to access both of the marks with the harder of the two marks being for suggesting why the apparatus was not suitable for the experiment. Clearly many candidates were able to suggest a better method of carrying out the experiment with reflux being often quoted.
			Total	**4**	
7	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	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.

			AND Few or imprecise details about further purification stages *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**	
8		i	$NaClO + 2HCl \rightarrow NaCl + Cl_2 + H_2O$ correct formulae of reactants, NaCl and chlorine (1) water and balancing (1)	2	**allow** $NaClO_3 + 6HCl \rightarrow NaCl + 3Cl_2 + 3H_2O$ for 1 mark
		ii	Test: add (a few drops of aqueous) silver nitrate (1) Result: white ppt (1)	2	**ignore** addition of dilute nitric acid before the $AgNO_3$ **ignore** redissolving in excess NH_3 or darkening of the ppt
		iii	separating funnel (1)	1	**allow** dropping pipette
			Total	**5**	
9		i	Elimination	1	**ALLOW** Dehydration
		ii	Same structural formula **AND** Different arrangement (of atoms) in **space OR** different **spatial** arrangement 	3	**ALLOW** have the same structure / displayed formula / skeletal formula **DO NOT ALLOW** same empirical formula **OR** same general formula Stereoisomers have the same formula or molecular formula is **not** sufficient Reference to *E/Z* isomerism or optical isomerism is **not** sufficient

					IGNORE names **IF** skeletal formula is not used **ALLOW** one mark if both stereoisomers of alkene **B** are shown clearly.
		iii		1	**ALLOW** correct structural **OR** skeletal **OR** displayed formula **OR** mixture of the above **IGNORE** names
		iv	** Please refer to the marking instruction point 10 for guidance on how to mark this question.* **Level 3 (5–6 marks)** Outlines full details of how a pure sample of **B** is obtained from the reaction mixture. **AND** Correctly calculates mass of **B** • *Purification steps are clear, in the correct order, using appropriate scientific terms.* • *Calculation shows all relevant steps and mass given to 3 significant figures.* **Level 2 (3–4 marks)** Some details of how a sample of **B** is obtained from the reaction mixture. **AND** Attempts a calculation which is mostly correct. • *Purification steps lack detail, e.g. no drying agent or no explanation of separation, or only some scientific terms used.* • *Calculation can be followed but unclear.* **Level 1 (1–2 marks)** Few or imprecise details of how a sample of **B** is obtained from the reaction mixture. **AND** Attempts to calculate the mass of B using mole approach but makes little progress with only 1 step correct. • *Purification step is unclear with few scientific terms and little detail, e.g. just 'separate the layers and dry'.* • *Calculation is difficult to follow and lacking clarity*	6	**Indicative scientific points, with bulleted elements, may include:** **1. Purification** • Use of a **separating funnel** to separate organic and aqueous layers • **Drying** with an anhydrous salt, e.g. $MgSO_4$, $CaCl_2$, etc. • **Redistillation** Incorrect purification method is **NOT** worthy of credit. **2. Mass of B obtained** • *n*(**A**) used = $\frac{9.26}{102}$ = 0.0908 (mol) = theoretical *n*(**B**) • **Actual** *n*(**B**) obtained = *n*(0.908) × $\frac{75}{100}$ = 0.0681 (mol) • mass **B** = 84 × 0.0681 = **5.72 g** **CHECK** for extent of errors by **ECF** Alternative correct calculation may calculate the mass of **B** as 0.0908 × 84 = 7.63 g, followed by 7.63 × $\frac{75}{100}$ = 5.72 g Calculation must attempt to calculate *n*(***A***) in mol. Simply finding 75% of the initial mass of alcohol A, 9.26, is **NOT** worthy of credit.

			0 marks No response or no response worthy of credit.		
			Total	**11**	