InkChemistry

ANSWER BOOK

Planning a synthetic route

Organic synthesis · Unit C22

AQA 3.3.14 · OCR A 4.2.3, 6.2.4, 6.2.5

21 questions · 76 marks

NAVY EDITION

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SECTION A · Short, 1 to 2 marks

A1 PSR-Q8

B1 propanal has a lower boiling point than propan-1-ol, so distillation removes it from the flask as it forms, before it can be oxidised further. **B1** under reflux the propanal condenses back into the flask and stays in contact with the oxidising agent, so it is oxidised on to propanoic acid. [2]

SECTION B · Standard, 3 to 5 marks

B1 PSR-Q15

B1 route A has one step, so it loses less product to repeated steps and takes less time. **B1** route A gives a mixture of primary, secondary and tertiary amines and a quaternary salt, which must be separated, lowering the yield of butylamine. **B1** route B has two steps, so its overall yield is the product of two yields. **B1** route B uses toxic potassium cyanide and flammable hydrogen or violently reactive $LiAlH_4$, but gives only the primary amine. **B1** a justified judgement: for example, route B is preferred where a pure primary amine is needed, route A where a quick product can be purified afterwards. [5]

B2 PSR-Q1

B1 step 1: potassium cyanide dissolved in ethanol and water, heated under reflux; reject HCN. **B1** step 1 is nucleophilic substitution, with cyanide as the nucleophile; ignore 'SN2'. **B1** step 2: hydrogen with a nickel catalyst, or $LiAlH_4$ in dry ether; reject [H] alone. **B1** step 2 is reduction of the C≡N group to CH_2NH_2. [4]

B3 PSR-Q4

B1 step 1 uses concentrated nitric acid with concentrated sulfuric acid as the catalyst. **B1** at about 50 °C, kept below 55 °C so that only one nitro group is substituted. **B1** the intermediate is nitrobenzene, $C_6H_5NO_2$. **B1** step 2 uses tin and concentrated hydrochloric acid heated under reflux, followed by excess sodium hydroxide solution to release phenylamine from its salt. [4]

B4 PSR-Q12

B1 step 1: benzene with ethanoyl chloride and an aluminium chloride catalyst, anhydrous, heated under reflux. **B1** the intermediate is phenylethanone, $C_6H_5COCH_3$. **B1** step 2: phenylethanone with $NaBH_4$ in water or methanol gives 1-phenylethanol. **B1** the hydride attacks either face of the planar C=O equally, so a racemic mixture forms and the rotations cancel. [4]

B5 PSR-Q13

B1 step 1: ethanol with acidified potassium dichromate(VI), warmed, distilling the ethanal off as it forms. **B1** the intermediate is ethanal, CH_3CHO. **B1** step 2: ethanal with potassium cyanide, followed by dilute acid, gives 2-hydroxypropanenitrile. **B1** potassium cyanide is acutely toxic and releases toxic hydrogen cyanide gas with acid, so it is used in a fume cupboard. [4]

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B6 PSR-Q16 [4]

B1 propanoic acid with methanol and a concentrated sulfuric acid catalyst, heated under reflux. **B1** propanoyl chloride with methanol at room temperature, with no catalyst needed. **B1** advantage: the acyl chloride reaction is fast and goes to completion, while esterification reaches an equilibrium. **B1** disadvantage: the acyl chloride is corrosive and releases hydrogen chloride fumes, so it needs a fume cupboard; accept a lower atom economy.

B7 PSR-Q18 [4]

B1 cutting the ester at its C(=O)–O link shows it is made from propanoic acid and propan-1-ol, each with three carbons. **B1** step 1: part of the propan-1-ol is heated under reflux with excess acidified potassium dichromate(VI) to give propanoic acid. **B1** reflux with excess oxidant is needed, because distilling the product off as it forms would give propanal. **B1** step 2: propanoic acid is heated under reflux with the rest of the propan-1-ol and a concentrated sulfuric acid catalyst to give propyl propanoate.

B8 PSR-Q20 [4]

B1 bromoethane is heated under reflux with magnesium in dry ether. **B1** this gives the Grignard reagent ethylmagnesium bromide, CH_3CH_2MgBr. **B1** the reagent reacts with solid carbon dioxide, followed by dilute acid, to give propanoic acid with one more carbon. **B1** water protonates the Grignard reagent, converting it into ethane, so it is lost before it can react with CO_2.

B9 PSR-Q21 [4]

B1 bromomethane is heated under reflux with magnesium in dry ether; accept chloromethane or iodomethane. **B1** $CH_3Br + Mg \rightarrow CH_3MgBr$, with the halogen matching the halogenoalkane named. **B1** the carbonyl compound is propanone, added in dry ether, because a ketone gives a tertiary alcohol. **B1** dilute acid is then added to convert the O–MgBr product into the alcohol, 2-methylpropan-2-ol.

B10 PSR-Q23 [4]

B1 the carbon chain is lengthened by nucleophilic addition of CN^- to a C=O, so a carbonyl compound must be made before the cyanide step; ethene and ethanol have no δ+ carbonyl carbon for the cyanide ion to attack. **B1** the C=O can only be made by oxidising an alcohol, so the hydration of ethene, with steam and a phosphoric acid catalyst, has to come first to put the OH group on the chain. **B1** the oxidation is run by distilling the ethanal off as it forms, because heating under reflux would oxidise it on to ethanoic acid, which does not undergo nucleophilic addition. **B1** the cyanide step comes last and gives the product directly, as a racemic mixture because CN^- attacks the planar C=O from either face; the dilute acid is added only after the cyanide, so that toxic hydrogen cyanide is not released from the potassium cyanide in excess acid.

B11 PSR-Q2 [3]

B1 propanal: acidified potassium dichromate(VI), warmed, with the aldehyde distilled off as it forms. **B1** propanoic acid: excess acidified potassium dichromate(VI), heated under reflux. **B1** propanone: acidified potassium dichromate(VI), heated under reflux; allow $K_2Cr_2O_7$ with H_2SO_4 for the reagent throughout.

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B12 PSR-Q3 **B1** $NaBH_4$ in water or ethanol at room temperature; accept methanol as the solvent. **B1** $LiAlH_4$ reacts violently with water, so it needs dry ether as the solvent and is a fire hazard. **B1** $NaBH_4$ is stable enough to use in water and still reduces aldehydes and ketones, so it gives the same product more safely. [3]

B13 PSR-Q6 **B1** butylamine has four carbons and 1-bromopropane three, so a new carbon–carbon bond must be made. **B1** substitution with cyanide is the reaction that adds one carbon to the chain, giving butanenitrile, which is reduced to butylamine. **B1** propylamine has the same three-carbon skeleton as 1-bromopropane, so substitution with ammonia is enough. [3]

B14 PSR-Q7 **B1** propylamine has a lone pair on nitrogen, so it is itself a nucleophile. **B1** it attacks unreacted 1-bromopropane to give secondary and then tertiary amines and a quaternary ammonium salt, so a mixture forms even with excess ammonia. **B1** reducing propanenitrile gives only the primary amine, as no halogenoalkane is present for the amine to attack. [3]

B15 PSR-Q11 **B1** step 1: propene with steam and a phosphoric acid catalyst at about 300 °C and 60 to 70 atm, or concentrated sulfuric acid followed by water. **B1** the intermediate is propan-2-ol, the major product because the more stable secondary carbocation forms. **B1** step 2: propan-2-ol heated under reflux with acidified potassium dichromate(VI) gives propanone. [3]

B16 PSR-Q14 **B1** in water, warmed, hydroxide acts as a nucleophile and substitutes the bromine to give propan-2-ol. **B1** in ethanol, heated under reflux, hydroxide acts as a base and eliminates HBr to give propene. **B1** both use the same reagent and both release bromide ions; the solvent and the temperature decide which product dominates. [3]

B17 PSR-Q17 **B1** a solvent does not appear in the equation or the product, yet it has to be bought, heated, separated from the product and recovered or disposed of. **B1** that costs energy and money and produces waste, which is not shown by the atom economy. **B1** most organic solvents are flammable and many are toxic, so removing the solvent also removes a hazard. [3]

B18 PSR-Q19 **B1** step 1: 1-bromopropane heated under reflux with potassium cyanide in ethanol and water. **B1** the intermediate is butanenitrile, $CH_3CH_2CH_2CN$, which carries the extra carbon. **B1** step 2: butanenitrile heated under reflux with dilute hydrochloric or sulfuric acid hydrolyses the C≡N to COOH, giving butanoic acid and ammonium ions. [3]

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B19 PSR-Q22 [3]

B1 magnesium is much less electronegative than carbon, so the carbon of the C–Mg bond is δ- and attacks a δ+ carbon. **B1** the ethyl group bonds to the carbonyl carbon, which keeps the groups it already had; methanal's carbon has two hydrogens, so the C–OH carbon carries one alkyl group: propan-1-ol. **B1** propanal's carbonyl carbon already carries an ethyl group, so the C–OH carbon ends with two alkyl groups: pentan-3-ol, a secondary alcohol.

SECTION C · Long, 6 marks or more

C1 PSR-Q5 [6]

Indicative content

B1 step 1: propene reacts with hydrogen bromide at room temperature by electrophilic addition, with no other reagent needed. **B1** the major product of step 1 is 2-bromopropane, $CH_3CHBrCH_3$, because the secondary carbocation is more stable than the primary one. **B1** step 2: 2-bromopropane is heated under reflux with potassium cyanide dissolved in ethanol and water, in nucleophilic substitution. **B1** the product of step 2 is 2-methylpropanenitrile, $(CH_3)_2CHCN$, in which the new carbon from cyanide is joined to the middle carbon. **B1** step 3: the nitrile is reduced with hydrogen over a nickel catalyst, or with $LiAlH_4$ in dry ether; reject $NaBH_4$, which does not reduce a nitrile. **B1** the product is 2-methylpropan-1-amine, accept 2-methylpropylamine; the branch arises because bromine, and so the cyanide carbon, went onto the middle carbon of propene.

Levels

Level 3 (5–6 marks) all three steps are given with reagents, conditions and the named product of each, the major product of step 1 is justified through carbocation stability, the reducing agent for the nitrile is one that works, and the branch is explained as following from where the bromine went.

Level 2 (3–4 marks) the three steps are given with mostly correct reagents and products; a condition is missing, the nitrile is 'reduced' with a reagent that does not work, or the branch is not explained.

Level 1 (1–2 marks) one or two steps appear, such as HBr or cyanide; 1-bromopropane is typically taken as the major product of step 1, so the route ends at an unbranched amine.

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