InkChemistry

ANSWER BOOK

Preparing, purifying and testing an organic solid and liquid

Organic synthesis · Unit C22

AQA 3.3.9.2; RP10 · OCR A 4.2.3, 6.2.5; PAG 5, PAG 6

18 questions · 58 marks

NAVY EDITION

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SECTION A · Short, 1 to 2 marks

A1 PP-Q17

B1 ethanoic anhydride is corrosive and its vapour irritates the eyes and lungs, so it is handled in a fume cupboard with gloves and goggles. **B1** concentrated phosphoric or sulfuric acid is corrosive, so it is added a drop at a time wearing goggles and gloves; ignore 'wear a lab coat' on its own. [2]

A2 PP-Q21

B1 atmospheric pressure in the laboratory was below standard pressure, which lowers the boiling temperature. **B1** the thermometer reads low, because it is inaccurately calibrated or its bulb sits above the side arm rather than level with it. [2]

SECTION B · Standard, 3 to 5 marks

B1 PP-Q1

B1 the reaction is slow, so the mixture must be heated for a long time, and the reactants and ester are volatile. **B1** the vertical condenser condenses the vapour and returns it to the flask, so nothing is lost and the mixture cannot boil dry. **B1** anti-bumping granules let bubbles form smoothly, so the liquid boils evenly rather than in sudden violent bursts. **B1** a closed system would build up pressure as it is heated and could burst or blow apart; reject 'to let air in'. [4]

B2 PP-Q4

B1 the solution is saturated while hot and the product is much less soluble in cooler solvent. **B1** a cold funnel would cool the solution, so the product would crystallise in the funnel and paper and be lost with the insoluble impurities. **B1** the wash removes the solution of soluble impurities left on the surface of the crystals. **B1** the solvent is ice-cold and the volume small so that as little of the product as possible dissolves; accept 'to keep the yield high'. [4]

B3 PP-Q8

B1 the crude ester and sodium carbonate solution are put in the funnel and the stopper is inserted. **B1** the funnel is inverted and the tap opened to release the carbon dioxide, before and between gentle shakes. **B1** this is repeated until no more gas forms, and the funnel is left until two distinct layers separate. **B1** the stopper is removed and the lower aqueous layer is run off through the tap and discarded, leaving the ester. [4]

B4 PP-Q2

B1 water hydrolyses the excess ethanoic anhydride to ethanoic acid, which stays dissolved in the water. **B1** aspirin is only slightly soluble in cold water, so it crystallises out of the solution. **B1** cooling in ice lowers its solubility further, so more of the aspirin crystallises and the yield of crude solid is higher. [3]

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B5 PP-Q3 **B1** the product is much more soluble in the hot solvent than in the cold. **B1** with the minimum volume the hot solution is saturated, so as much product as possible crystallises on cooling. **B1** extra solvent keeps more product dissolved in the cold filtrate, which lowers the yield; ignore references to purity. [3]

B6 PP-Q5 **B1** a pure sample melts sharply, over a range of about 1 °C. **B1** at a temperature that matches the data-book value, 135 °C for aspirin. **B1** impurities disrupt the lattice, so an impure sample melts lower and over a wider range. [3]

B7 PP-Q6 **M1** mass of product = 26.51 - 24.36 = 2.15 g. **M1** each reading is ±0.01 g, so the mass by difference carries ±0.02 g. **A1** percentage uncertainty = 0.02 ÷ 2.15 × 100 = 0.93%, to two significant figures; allow 0.47% from ±0.005 g a reading. [3]

B8 PP-Q9 **B1** sodium carbonate neutralises the unreacted ethanoic acid and the sulfuric acid catalyst. **B1** the acids become ionic salts that dissolve in the aqueous layer and are removed with it. **B1** the neutralisation releases carbon dioxide, which builds up pressure that could force out the stopper unless it is released through the tap. [3]

B9 PP-Q10 **B1** the ester is run into a dry conical flask and anhydrous calcium chloride is added and swirled. **B1** more is added until fresh solid no longer clumps and the cloudy liquid turns clear, showing the water has been absorbed. **B1** the dry liquid is decanted or filtered away from the solid before it is redistilled. [3]

B10 PP-Q13 **B1** 1-bromobutane is denser than water, so it forms the lower layer. **B1** adding a few cm³ of water to the funnel: the layer that increases in volume is the aqueous layer. **B1** the lower organic layer is run off through the tap into a clean flask and kept; the upper aqueous layer is discarded. [3]

B11 PP-Q14 **B1** the range is wide and below 135 °C, so the sample is impure. **B1** it may still contain unreacted 2-hydroxybenzoic acid or ethanoic acid, or it may not be fully dry. **B1** the student should dry it to constant mass and recrystallise again, then repeat the melting point. [3]

B12 PP-Q15 **B1** esterification is reversible, so the mixture reaches equilibrium with acid and alcohol still unreacted. **B1** ester is lost on each transfer, dissolved in the aqueous wash and absorbed by the drying agent. **B1** the ester is volatile and evaporates whenever the liquid is exposed, and fractions outside the collection range are discarded. [3]

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B13 PP-Q18

B1 the range matches the boiling point of ethyl ethanoate, 77 °C, and shows that high-boiling impurities have been left behind. **B1** ethanol boils at 78 °C, inside the same range, so the fraction could still contain ethanol. **B1** the claim is not justified: a boiling range is a weak test here, and an infrared spectrum with no broad O–H, or a gas chromatogram with one peak, would be needed. [3]

B14 PP-Q19

B1 phenylamine and water are immiscible, so each exerts its own vapour pressure independently of the other. **B1** the mixture boils when the sum of the two vapour pressures equals atmospheric pressure. **B1** the two together reach atmospheric pressure below 100 °C, so phenylamine distils with the steam without being heated to 184 °C, where it could decompose. [3]

B15 PP-Q20

B1 the thermometer bulb is placed level with the side arm, so it reads the temperature of the vapour leaving. **B1** the temperature is read once it is steady and distillate is dripping from the condenser. **B1** a pure liquid distils at a steady temperature matching the data-book value; a mixture distils over a rising range. [3]

SECTION C · Long, 6 marks or more

C1 PP-Q7

Indicative content [6]

B1 dissolve the crude solid in the minimum volume of hot solvent, such as an ethanol and water mixture, so the solution is saturated. **B1** filter the hot solution through a warmed funnel if any insoluble impurities remain, so the product does not crystallise in the funnel. **B1** cool slowly to room temperature and then in ice, so the aspirin crystallises while soluble impurities stay in solution. **B1** collect the crystals by filtration under reduced pressure in a Büchner funnel and wash with a little ice-cold solvent. **B1** dry the crystals in a desiccator or a low oven, then pack a little of the powder into a capillary tube sealed at one end. **B1** heat slowly near the expected value in a melting point apparatus; a sharp melting point at 135 °C shows the sample is pure.

Levels

Level 3 (5–6 marks) every stage of the recrystallisation is present with its reason: minimum hot solvent, hot filtration, slow cooling then ice, suction filtration, washing with ice-cold solvent and drying, followed by the melting point test with a sharp melting point at the expected value as the sign of purity.

Level 2 (3–4 marks) dissolving in minimum hot solvent, cooling and filtering are described and the melting point named; why the solvent is minimal, why the wash is ice-cold, or what a sharp melting point shows is left unsaid.

Level 1 (1–2 marks) the solid is dissolved and recrystallised, often in a large volume of solvent or washed with warm solvent, both of which lose product.

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