2.9 A-Level Chemistry: Required Practicals and Practical Skills: 1-hour Pack

Total Marks

AQA 7405 · Calculator allowed · about 60 minutes

Name: _____ Date: ____ / ____ / _____

Answer ALL questions. Show all your working.

1 A student prepared 1-bromobutane by refluxing butan-1-ol with a mixture of sodium bromide and concentrated sulfuric acid, which generates hydrogen bromide in situ:

$NaBr(s) + H_2SO_4(aq) \rightarrow NaHSO_4(aq) + HBr$

The hydrogen bromide then reacts with the butan-1-ol:

$C_4H_9OH + HBr \rightarrow C_4H_9Br + H_2O$

Mass of butan-1-ol used = 7.40 g (Mr = 74.0)
Mass of 1-bromobutane obtained after purification = 9.25 g (Mr of C_4H_9Br = 137.0)

(a) Explain the purpose of heating the reaction mixture under reflux, rather than simply heating it in an open flask. **(2)**

(b) After reflux, the crude 1-bromobutane was separated by distillation and then purified further. Describe how the distillate would be purified, and explain the purpose of each step. **(4)**

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(c) Calculate the theoretical yield of 1-bromobutane, in grams. **(2)**

(d) Calculate the percentage yield of 1-bromobutane obtained. **(2)**

(e) Suggest one reason, other than losses during purification/transfer, why the percentage yield is less than 100%. **(1)**

(Total for Question 1 is 11 marks)

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2 A student synthesised a solid organic product, Z, with a literature melting point of 122-124°C. The crude solid obtained directly from the reaction had a melting point range of 110-119°C. The student purified the crude product by recrystallisation, after which the melting point was found to be 121-123°C.

Theoretical yield of Z = 3.05 g
Mass of crude product obtained = 2.85 g
Mass of purified (recrystallised) product obtained = 2.10 g

(a) Describe how the student would purify the crude solid Z by recrystallisation. **(4)**

(b) Explain why the solvent used for recrystallisation must dissolve Z when hot but only sparingly when cold. **(2)**

(c) Explain what the melting point data indicate about the purity of the crude product and the purified product. **(3)**

(d) Calculate the percentage yield of the purified (recrystallised) product. **(2)**

(e) Suggest one reason why the percentage yield of the purified product is lower than the percentage yield of the crude product (2.85 g). **(1)**

(Total for Question 2 is 12 marks)

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3 The acid-catalysed reaction between propanone and iodine was studied:

$CH_3COCH_3(aq) + I_2(aq)$ -[H^+ catalyst]-> $CH_3COCH_2I(aq) + HI(aq)$

A colorimeter was used to follow the concentration of iodine during the reaction, since iodine is coloured orange-brown while the other species involved are colourless. Four experiments were carried out at the same temperature, each starting with different initial concentrations of propanone, hydrochloric acid (H^+) and iodine, and the initial rate was determined from the initial gradient of a concentration-time graph.

Experiment	$[CH_3COCH_3]$ / mol dm-3	$[H^+(aq)]$ / mol dm-3	$[I_2(aq)]$ / mol dm-3	Initial rate / mol dm-3 s-1
1	0.500	0.500	0.0100	1.15×10^{-5}
2	1.000	0.500	0.0100	2.30×10^{-5}
3	0.500	1.000	0.0100	2.30×10^{-5}
4	0.500	0.500	0.0200	1.15×10^{-5}

(a) Explain why a colorimeter can be used to follow the rate of this reaction. **(2)**

(b) In this experiment, a graph of $[I_2]$ against time is a straight line (constant negative gradient) rather than a curve. Explain what this observation shows about the order of reaction with respect to iodine. **(2)**

(c) Using the data in the table, deduce the order of reaction with respect to (i) propanone, (ii) $H^+(aq)$, and (iii) $I_2(aq)$, explaining your reasoning in each case by comparing pairs of experiments. **(6)**

(d) Write the overall rate equation for this reaction. **(1)**

(e) Calculate the rate constant, k, for this reaction using the data from Experiment 1, including its units. **(3)**

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(f) Predict and explain the effect on (i) the initial rate, and (ii) the total time taken for all the iodine to react, if the initial concentration of iodine in Experiment 1 were doubled to 0.0200 mol dm-3 (as in Experiment 4), while [propanone] and $[H^+]$ remain as in Experiment 1. **(2)**

(Total for Question 3 is 16 marks)

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Mark scheme · 2.9 A-Level Chemistry: Required Practicals and Practical Skills: 1-hour Pack

Question 1

(a) reflux allows the mixture to be heated strongly/continuously (to increase the rate of reaction) for an extended period **B1**

(a) the condenser continuously condenses and returns any volatile reactants/products (e.g. HBr, butan-1-ol) to the flask, preventing their loss by evaporation **B1**

(a) Answer: Reflux allows prolonged strong heating while the condenser returns volatile reactants/products, preventing losses.

(b) shake the distillate with sodium hydrogencarbonate solution in a separating funnel (releasing the tap to allow CO_2 gas to escape), to neutralise/remove any remaining acid (H_2SO_4/HBr) **B1**

(b) run off and discard the aqueous layer, keeping the organic layer of 1-bromobutane **B1**

(b) wash the organic layer with distilled water to remove remaining traces of inorganic/aqueous impurities **B1**

(b) dry the organic layer using an anhydrous drying agent (e.g. anhydrous calcium chloride or magnesium sulfate), then redistil, collecting the fraction boiling at the known boiling point of 1-bromobutane (approx. 101°C), to remove impurities with a different boiling point **B1**

(b) Answer: Wash with $NaHCO_3$(aq) to neutralise acid, then with water; dry with an anhydrous drying agent; redistil, collecting the correct boiling-point fraction.

(c) moles butan-1-ol = 7.40/74.0 = 0.100 mol (1:1 ratio, so moles product = 0.100 mol) **M1**

(c) theoretical mass = 0.100 x 137.0 = 13.70 g (cao) **A1**

(c) Answer: 13.70 g

(d) % yield = (9.25/13.70) x 100 (ecf from (c)) **M1**

(d) = 67.5% (awrt 67-68%) **A1**

(d) Answer: 67.5%

(e) a competing elimination reaction can occur, forming but-1-ene as a side product (oe: the reaction is an equilibrium/does not go to completion, so not all the alcohol is converted) **B1**

(e) Answer: Elimination side reaction forms but-1-ene (oe: reaction does not go to completion).

Question 2

(a) dissolve the crude solid in a minimum volume of a hot, appropriate solvent (one in which Z is soluble when hot but only sparingly soluble when cold) **B1**

(a) filter the hot solution (using a fluted/preheated filter funnel) to remove any insoluble impurities **B1**

(a) allow the filtrate to cool slowly (e.g. to room temperature, then in ice) so crystals of pure Z form, leaving soluble impurities in solution **B1**

(a) filter off the crystals under reduced pressure (Buchner funnel/flask), wash with a small volume of cold solvent, and allow to dry (e.g. in a desiccator or a warm oven below the melting point) **B1**

(a) Answer: Dissolve in minimum hot solvent, hot filter, cool slowly to crystallise, filter under reduced pressure, wash and dry.

(b) so a saturated (or near-saturated) solution can be made using a minimum volume of hot solvent, maximising the amount of Z that crystallises out (and hence the yield) on cooling **B1**

(b) impurities, present in small amounts, remain dissolved in the small volume of cold solvent even as Z crystallises out, so they are removed by filtration, purifying Z **B1**

(b) Answer: Maximises recovery on cooling while impurities stay dissolved and are removed by filtration.

(c) the crude product has a wide melting range (110-119°C), well below the literature value, showing it is impure - impurities disrupt the regular crystal lattice, lowering the melting point and broadening the melting range **B1**

(c) the purified product has a much narrower melting range (121-123°C), very close to the literature range (122-124°C) **B1**

(c) showing that the purified product is of much higher purity than the crude product **B1**

(c) Answer: Crude product is impure (wide, depressed range); purified product is close to the literature value and much narrower, so highly pure.

(d) % yield = (2.10/3.05) x 100 **M1**

(d) = 68.9% (awrt 68-69%) **A1**

(d) Answer: 68.9%

(e) some product remains dissolved in the cold solvent (the mother liquor) and/or is lost during transfers/filtration during recrystallisation, reducing the mass recovered **B1**

(e) Answer: Product lost in the mother liquor / during transfers and filtration.

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Question 3

(a) iodine is the only coloured species in the reaction mixture (the other reagents and products are colourless) **B1**

(a) so the absorbance measured is directly proportional to the concentration of iodine remaining, allowing $[I_2]$ to be monitored continuously over time **B1**

(a) Answer: Iodine is the only coloured species, so absorbance tracks $[I_2]$ directly.

(b) the reaction is zero order with respect to I_2 (rate is independent of $[I_2]$) **B1**

(b) the rate (gradient of the graph) stays constant even as $[I_2]$ decreases, whereas a reaction with a positive order in I_2 would give a curved (e.g. exponential) concentration-time graph **B1**

(b) Answer: Zero order with respect to iodine, since the rate (gradient) is constant regardless of $[I_2]$.

(c) (i) order with respect to propanone = 1 (first order) **B1**

(c) (i) comparing Exp 1 and Exp 2: doubling [propanone] (0.500 to 1.000), with $[H^+]$ and $[I_2]$ unchanged, doubles the initial rate (1.15 to 2.30 x 10^{-5}) - rate is directly proportional to [propanone] **B1**

(c) (ii) order with respect to H^+ = 1 (first order) **B1**

(c) (ii) comparing Exp 1 and Exp 3: doubling $[H^+]$ (0.500 to 1.000), with [propanone] and $[I_2]$ unchanged, doubles the initial rate - rate is directly proportional to $[H^+]$ **B1**

(c) (iii) order with respect to I_2 = 0 (zero order) **B1**

(c) (iii) comparing Exp 1 and Exp 4: doubling $[I_2]$ (0.0100 to 0.0200), with [propanone] and $[H^+]$ unchanged, has no effect on the initial rate (both 1.15 x 10^{-5}) - rate is independent of $[I_2]$ **B1**

(c) Answer: Order 1 with respect to propanone; order 1 with respect to H^+; order 0 with respect to I_2.

(d) rate = $k[CH_3COCH_3][H^+]$ (I_2 does not appear, as it is zero order; cao, ecf from (c)) **B1**

(d) Answer: rate = $k[CH_3COCH_3][H^+]$

(e) k = rate / ([propanone][H^+]) = 1.15 x 10^{-5} / (0.500 x 0.500) **M1**

(e) k = 4.60 x 10^{-5} (awrt) **A1**

(e) units: dm3 mol-1 s-1 (since rate = mol dm-3 s-1 and [propanone][H^+] = mol2 dm-6, so k = mol dm-3 s-1 / mol2 dm-6 = dm3 mol-1 s-1) **B1**

(e) Answer: k = 4.60 x 10^{-5} dm3 mol-1 s-1

(f) (i) no effect on the initial rate (remains 1.15 x 10^{-5} mol dm-3 s-1) because the reaction is zero order with respect to I_2, so rate does not depend on $[I_2]$ **B1**

(f) (ii) the total time taken would double, because there is twice the amount of I_2 to be used up but it is consumed at the same constant rate **B1**

(f) Answer: Initial rate unchanged; time taken for all iodine to react would double.

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