InkChemistry

ANSWER BOOK

Test-tube reactions for functional groups, and high-resolution mass spectrometry

Organic analysis · Unit C19

AQA 3.3.6.1, 3.3.6.2 · OCR A 4.2.4, 6.1.2, 6.3.1

18 questions · 58 marks

NAVY EDITION

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SECTION A · Short, 1 to 2 marks

A1 TTR-Q1

B1 bromine water is added and the mixture is shaken. **B1** the bromine water changes from orange to colourless. [2]

A2 TTR-Q6

B1 the green colour shows only that dichromate(VI) has been reduced to Cr^{3+} by something that is oxidised. **B1** aldehydes are also oxidised by acidified dichromate, so the unknown could be an aldehyde as well as a primary or secondary alcohol. [2]

A3 TTR-Q12

B1 the test tube was not clean and grease-free, so the silver formed as a precipitate rather than a coating on the glass. **B1** silver has still been formed, so the result is positive for an aldehyde. [2]

A4 TTR-Q22

B1 both are acids and react with sodium hydroxide, phenol forming sodium phenoxide. **B1** only ethanoic acid gives effervescence with sodium hydrogencarbonate; phenol is too weak an acid to release carbon dioxide. [2]

SECTION B · Standard, 3 to 5 marks

B1 TTR-Q11

B1 the molecular ion breaks beside the C=O, giving a positive acylium ion and a neutral radical, and only the ion is detected. **B1** pentan-2-one gives CH_3CO^+ at m/z 43, by loss of a propyl radical. **B1** pentan-2-one also gives $CH_3CH_2CH_2CO^+$ at m/z 71, by loss of a methyl radical. **B1** pentan-3-one gives $CH_3CH_2CO^+$ at m/z 57, by loss of an ethyl radical, and no CH_3CO^+ at 43, since it has no CH_3CO group. [4]

B2 TTR-Q13

B1 warm each with acidified potassium dichromate(VI): 2-methylbutan-2-ol, a tertiary alcohol, leaves it orange. **B1** pentan-1-ol and pentan-2-ol both turn it from orange to green. **B1** oxidise each of these two in distillation apparatus and warm each distillate with Tollens' reagent; allow Fehling's solution. **B1** the distillate from pentan-1-ol is pentanal and gives a silver mirror; pentan-2-one from pentan-2-ol gives no change. [4]

B3 TTR-Q15

B1 m/z 58 fits propanone, C_3H_6O, but also other formulae with the same nominal mass, such as C_4H_{10} and $C_2H_6N_2$. **B1** a high-resolution spectrum would fix the formula, for example 58.0417 for C_3H_6O against 58.0780 for C_4H_{10}. **B1** even with the formula C_3H_6O fixed, propanal and prop-2-en-1-ol are isomers with the same precise mass. **B1** the conclusion is therefore not justified until tests show no silver mirror with Tollens' and no change with bromine water or dichromate. [4]

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B4 TTR-Q16 — [4]

B1 sodium hydrogencarbonate gives effervescence only with ethanoic acid. **B1** bromine water turns from orange to colourless only with cyclohexene. **B1** Tollens' reagent, warmed, gives a silver mirror only with propanal. **B1** the remaining liquid, propan-1-ol, turns acidified dichromate(VI) from orange to green on warming; this test comes last because propanal would also turn it green.

B5 TTR-Q21 — [4]

B1 chlorine is about 75% ^{35}Cl and 25% ^{37}Cl, two isotopes differing by two mass units. **B1** $C_3H_7{}^{35}Cl^+$ gives the peak at 78 and $C_3H_7{}^{37}Cl^+$ the peak at 80, in the ratio of the isotope abundances, 3 : 1. **B1** dichloromethane gives three peaks, at m/z 84, 86 and 88, from $^{35}Cl^{35}Cl$, $^{35}Cl^{37}Cl$ and $^{37}Cl^{37}Cl$. **B1** their heights are in the ratio 9 : 6 : 1, from 3 × 3, 2 × 3 × 1 and 1 × 1.

B6 TTR-Q2 — [3]

B1 effervescence, or bubbles, of a gas that turns limewater milky; reject 'a gas is produced' on its own. **B1** $CH_3COOH(aq) + NaHCO_3(s) \rightarrow CH_3COONa(aq) + H_2O(l) + CO_2(g)$, with all species correct. **B1** the equation is balanced and every species carries a correct state symbol.

B7 TTR-Q4 — [3]

B1 a drop of sodium hydroxide solution is added to silver nitrate solution, giving a brown precipitate. **B1** dilute ammonia solution is added drop by drop until the precipitate just dissolves. **B1** warmed with an aldehyde in a water bath, it gives a silver mirror on the inside of the tube.

B8 TTR-Q5 — [3]

B1 propanal is an aldehyde and is oxidised to propanoic acid by the mild oxidising agent. **B1** copper(II) is reduced to copper(I), giving a brick-red precipitate of Cu_2O from the blue solution. **B1** propanone is a ketone, has no hydrogen on the carbonyl carbon and would need a C–C bond broken to be oxidised, so the solution stays blue.

B9 TTR-Q7 — [3]

B1 a high-resolution spectrometer measures m/z to four decimal places. **B1** the precise masses of ^1H and ^{16}O are not whole numbers; only ^{12}C is exactly 12.0000. **B1** so the precise relative molecular masses differ: C_4H_8 56.0624 and C_3H_4O 56.0261; allow values within 0.0001.

B10 TTR-Q8 — [3]

M1 C_4H_8O: 4 × 12.0000 + 8 × 1.0078 + 15.9949 = 72.0573. **M1** C_5H_{12}: 5 × 12.0000 + 12 × 1.0078 = 72.0936. **A1** 72.0574 matches 72.0573 to four decimal places within 0.0001, so the formula is C_4H_8O.

B11 TTR-Q14 — [3]

B1 with Tollens' reagent propanal gives a silver mirror and propanone gives no change. **B1** with Fehling's solution propanal turns the blue solution to a brick-red precipitate and propanone leaves it blue. **B1** both reagents give a positive result only with the aldehyde, because only the aldehyde is oxidised.

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B12 TTR-Q19 [3]

B1 ethanol, ethanal and propan-2-ol give the precipitate of triiodomethane, CHI_3. **B1** ethanal is the one aldehyde with a CH_3CO group, and ethanol and propan-2-ol contain a $CH_3CH(OH)$ group that the alkaline iodine oxidises to CH_3CO. **B1** methanol has no methyl group on its C–OH carbon, and the carbonyl of pentan-3-one, $CH_3CH_2COCH_2CH_3$, sits between two ethyl groups, so neither gives a precipitate.

B13 TTR-Q20 [3]

M1 n = (100 × height of M+1 peak) ÷ (1.1 × height of M peak). **M1** n = (100 × 0.99) ÷ (1.1 × 18.0) = 99 ÷ 19.8. **A1** n = 5.0, so the molecule has 5 carbon atoms, a whole number with no unit.

SECTION C · Long, 6 marks or more

C1 TTR-Q17 [6]

Indicative content

B1 Tollens' reagent is made fresh: sodium hydroxide added to silver nitrate, then dilute ammonia until the brown precipitate just dissolves. **B1** a few drops of the unknown are added and the tube is warmed in a water bath, not over a flame, because the organic liquid is flammable. **B1** a silver mirror shows an aldehyde; no change rules an aldehyde out. **B1** for the second test the unknown is warmed in a water bath with potassium dichromate(VI) and dilute sulfuric acid. **B1** orange to green shows a primary or secondary alcohol only if the Tollens' test was negative, because aldehydes also turn it green. **B1** precautions: Tollens' residues are washed away at once because they can form explosive compounds, and gloves are worn because dichromate(VI) is toxic and carcinogenic.

Levels

Level 3 (5–6 marks) both tests are described with the preparation of the reagent, water-bath warming and the safety reasons, the observations and what each shows, including the point that a positive dichromate result identifies an alcohol only once an aldehyde has been ruled out; all the precautions named are matched to their reasons.

Level 2 (3–4 marks) both tests are described with observations and the water bath; preparing Tollens' reagent, disposing of its residues, or reading the two results together is not covered.

Level 1 (1–2 marks) one test is described with its positive result; a positive dichromate test is typically taken to prove an alcohol, and the tubes are heated over a flame.

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