

5.5.2 Combine analytical evidence and evaluate purification

Name: _____ Class: _____ Date: _____ **Total: 28 marks**

KEY WORDS evidence • conditions

Objective

Combine analytical evidence and evaluate purification. All questions and marking guidance are original.

1 Key knowledge and worked examples

Use formula and multiple independent observations. Combustion gives empirical proportions; molecular mass gives the multiplier. IR and chemical tests constrain functional groups; carbon-13 and proton NMR constrain distinct environments and neighbours. A single matching signal does not prove identity.

■ Worked method

C₃H₆O with aldehyde H, C=O and positive Tollens' test fits propanal, not propanone. Propanal has three carbon environments. For a solid synthesis, minimum hot solvent supports recovery during recrystallisation. Dry the crystals before mass and melting-range measurements; trapped solvent can inflate yield and lower/broaden the melting range.

2 Practice

2.1 A compound contains only carbon, hydrogen and oxygen. Burning 0.740 g gives 1.760 g CO₂ and 0.900 g H₂O. Find empirical formula. Use C=12, H=1, O=16. [5]

2.2 The compound in 2.1 has molar mass 74 g mol⁻¹. Find molecular formula and explain why combustion alone cannot distinguish its structural isomers. [2]

2.3 An unknown C₃H₆O has an aldehyde proton, a C=O absorption and positive Tollens' test. Identify it and predict carbon-13 signal count. [2]

3 Exam-style questions

3.1 A student uses a large volume of hot solvent in recrystallisation, then washes the crystals with much warm solvent. Predict two effects on recovery and explain each. [2]

3.2 Explain when reflux, simple distillation and steam distillation are useful. Include one essential heated-apparatus safety control. [4]

3.3 A solid has a broad melting range below its expected value but a yield above 100%. Evaluate identity and purity, and propose the next preparation step. [3]

3.4 An unfamiliar molecule contains a primary amine and a primary alcohol. Predict its separate reactions with dilute HCl and controlled oxidation with aldehyde distillation. A supplied procedure uses a flammable solvent and a corrosive acid. Give a control for each hazard. [4]

3.5 An unknown ester contains 58.82% C and 9.80% H, the rest O. Its molecular ion is m/z 102. Use C=12, H=1, O=16. It has five carbon-13 environments and proton sets: 3H singlet near 3.7 ppm, 2H triplet near 2.3, 2H multiplet near 1.6, 3H triplet near 0.9. Find the molecular formula and a consistent structure. Explain why ethyl propanoate is rejected and what a fragment at m/z 31 can add to the evidence. [6]

Solutions

Original marking guidance: M1 method, A1 accuracy, B1 explanation.

2.1 Carbon moles = $1.760/44 = 0.0400$; carbon mass = 0.480 g. Hydrogen atoms = $2(0.900/18) = 0.100$ mol; mass = 0.100 g. Oxygen mass = $0.740 - 0.480 - 0.100 = 0.160$ g, oxygen = 0.0100 mol. Ratio 4:10:1, $C_4H_{10}O$.

2.2 Empirical mass 74, multiplier one, $C_4H_{10}O$. Different atom connectivities can share that formula.

2.3 Propanal, three distinct carbon environments/signals.

3.1 More product remains dissolved in the larger mother-liquor volume; warm wash dissolves more product than a little cold solvent, both lowering recovery.

3.2 Reflux heats without losing volatile reactants. Simple distillation collects a sufficiently separable volatile component. Steam distillation carries a suitable water-insoluble volatile compound at reduced operating temperature. Keep a heated reflux/distillation system open to pressure relief, not sealed.

3.3 The data suggest impurity/retained solvent, not proof of pure intended product. Recrystallise as appropriate and dry to constant mass, then remeasure melting range/mass and combine with independent identification evidence.

3.4 The amine accepts H^+ to form an ammonium salt with HCl. Controlled primary-alcohol oxidation gives an aldehyde when distilled before further oxidation. Use an appropriate non-flame heat source/keep ignition sources away from the flammable solvent; wear eye protection/use suitable handling controls for corrosive acid. The task treats separate functional-group reactions; actual selectivity must follow the supplied procedure.

3.5 In 100 g, amounts are C $58.82/12 = 4.902$, H 9.80, O $31.38/16 = 1.961$ mol. Ratio approximately 2.5:5:1, doubled to $C_5H_{10}O_2$; mass 102 matches the molecular ion. Methyl butanoate, $CH_3CH_2CH_2COOCH_3$, fits the five carbon environments and four proton sets. Ethyl propanoate needs an OCH_2 quartet, not an OCH_3 singlet. m/z 31 is consistent with CH_3O^+ and supports the methoxy assignment but is not unique structure proof.