

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

$\text{C}_3\text{H}_6\text{O}$ with aldehyde H, $\text{C}=\text{O}$ 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 CO_2 and 0.900 g H_2O . Find empirical formula. Use $\text{C}=12$, $\text{H}=1$, $\text{O}=16$. [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 $\text{C}_3\text{H}_6\text{O}$ has an aldehyde proton, a $\text{C}=\text{O}$ 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]
