BENCHMARK

VCE Chemistry Assessments

Chemistry

Solutions

Benchmark Open SAC 4.2: Identifying an unknown compound

- Writing time is **55 minutes**

Approved materials

- One scientific calculator

Materials supplied

- Data Book

Instructions

- Answer all questions in the spaces provided.
- Write using blue or black pen.

Students are **not** permitted to bring mobile phones or other electronic devices into the room.

Contents pages

Questions (3 questions, 40 marks) 2–11

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Questions

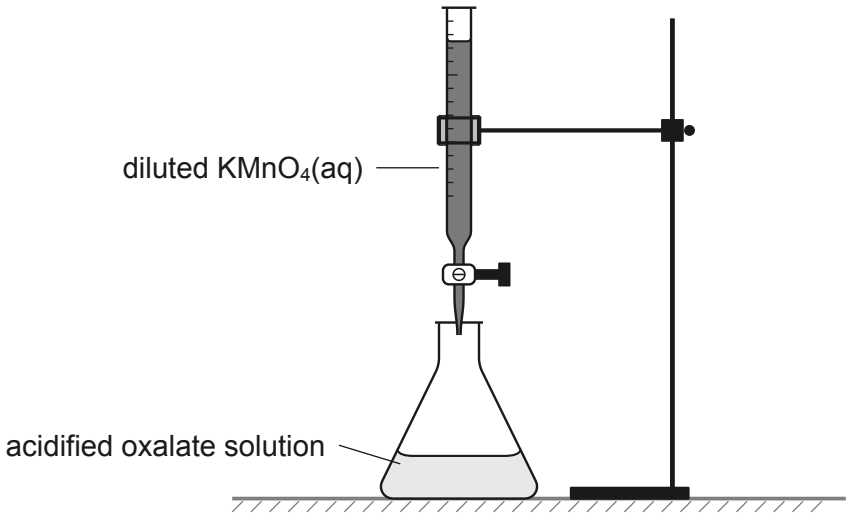
Instructions

- Answer **all** questions in the spaces provided.
- Write your responses in English.
- Give simplified answers to all numerical questions, with an appropriate number of significant figures; unsimplified answers will not be given full marks.
- Show all working in your answers to numerical questions; no marks will be given for an incorrect answer unless it is accompanied by details of the working.
- Ensure that chemical equations are balanced and that the formulas for individual substances include an indication of state, for example, $H_2(g)$, NaCl(s).
- Unless otherwise indicated, the diagrams in this book are **not** drawn to scale.

Question 1 (15 marks)

A researcher in a flavour laboratory plans to oxidise an unknown organic compound with acidified potassium permanganate. Solid potassium permanganate is not a primary standard, and the concentration of a stored permanganate solution slowly decreases. Before she uses her stock solution, the researcher therefore determines the concentration of permanganate ions in it by titration against sodium oxalate, which is a primary standard.

She dilutes 10.00 mL of the stock solution to 100.0 mL with water. She also dissolves 2.104 g of sodium oxalate, $Na_2C_2O_4$, in water and makes this solution up to 250.0 mL. Each 20.00 mL aliquot of the oxalate solution is acidified, warmed and titrated with the diluted permanganate solution, as shown.



The equation for the reaction is:

$$2MnO_4^-(aq) + 5C_2O_4^{2-}(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

The researcher records the burette readings for four trials, as shown in the table below.

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Initial reading (mL)	0.50	1.20	0.85	2.10
Final reading (mL)	21.15	21.55	21.24	22.46

a. Using the results in the table, calculate the mean concordant titre, in mL. 2 marks

① Titres: 20.65, 20.35, 20.39 and 20.36 mL; Trial 1 is not concordant and is excluded.

② mean titre = $\frac{20.35 + 20.39 + 20.36}{3}$ = 20.37 mL

b. Calculate the amount, in mol, of oxalate ions in each 20.00 mL aliquot. 2 marks

① $n(Na_2C_2O_4) = \frac{m}{M} = \frac{2.104}{134.0} = 0.015701\ldots$ mol

② $n(C_2O_4^{2-}$ in aliquot$) = 0.015701\ldots \times \frac{20.00}{250.0} = 1.256 \times 10^{-3}$ mol (to 4 s.f.)

c. Calculate the concentration, in mol L^{-1}, of permanganate ions in the stock solution. 3 marks

① $n(MnO_4^-) = \frac{2}{5} \times n(C_2O_4^{2-}) = \frac{2}{5} \times 1.2561\ldots \times 10^{-3} = 5.0244\ldots \times 10^{-4}$ mol

② $c(MnO_4^-$ diluted$) = \frac{n}{V} = \frac{5.0244\ldots \times 10^{-4}}{0.020366\ldots} = 0.024670\ldots$ mol L^{-1}

③ $c(MnO_4^-$ stock$) = 0.024670\ldots \times \frac{100.0}{10.00} = 0.2467$ mol L^{-1} (to 4 s.f.; accept 0.247 mol L^{-1})

The researcher has a bottle of a colourless liquid, Compound Y, which has the molecular formula $C_5H_{12}O$. Many different compounds have this molecular formula, so the label alone does not tell her which one is in the bottle.

First, to find out which functional groups are present in Compound Y, she carries out three tests. Each test uses a separate sample of the liquid, and her observations are recorded in the table below.

Test	Reagent added	Observation
1	sodium carbonate solution	no bubbles form
2	bromine water	the orange colour remains
3	acidified potassium permanganate solution, then warmed	the purple colour fades to colourless

d. Explain how the molecular formula of Compound Y shows that the results of Tests 1 and 2 could have been predicted without doing them. 2 marks

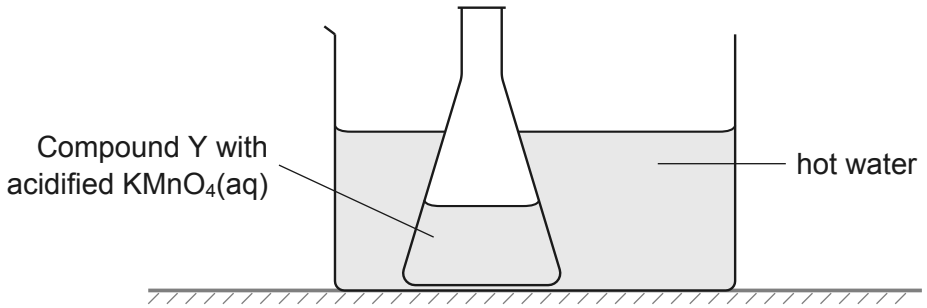
① A carboxyl group contains two oxygen atoms, but $C_5H_{12}O$ has only one, so Compound Y cannot have a carboxyl group (Test 1).

② $C_5H_{12}O$ has the maximum number of hydrogen atoms for five carbon atoms ($C_{n}H_{2n+2}O$), so it is saturated and has no carbon carbon double bond (Test 2).

e. State what the result of Test 3 indicates about Compound Y. 1 mark

① Compound Y has a hydroxyl group that is oxidised by permanganate, so it is a primary or secondary alcohol.

The researcher then adds 1.85 g of Compound Y to 30.00 mL of the stock permanganate solution, together with an excess of acid. She heats the mixture in a beaker of hot water, as shown in the diagram below, until no further change is seen, and Compound X forms.



The equation for the reaction is:

$$5C_5H_{12}O(l) + 2MnO_4^-(aq) + 6H^+(aq) \rightarrow 5C_5H_{10}O(l) + 2Mn^{2+}(aq) + 8H_2O(l)$$

f. Using your answer to part **c.**, show by calculation that the permanganate ions are the limiting reactant. 3 marks

① $n(Y) = \frac{m}{M} = \frac{1.85}{88.0} = 0.021022\ldots$ mol

② $n(MnO_4^-) = c \times V = 0.24670\ldots \times 0.03000 = 7.4010\ldots \times 10^{-3}$ mol

③ $n(MnO_4^-\text{ needed}) = \frac{2}{5} \times 0.021022\ldots$

$= 8.4090\ldots \times 10^{-3}$ mol, more than is present, so the permanganate ions are limiting (consequential on part c.).

g. Calculate the maximum mass, in g, of Compound X that can form. 2 marks

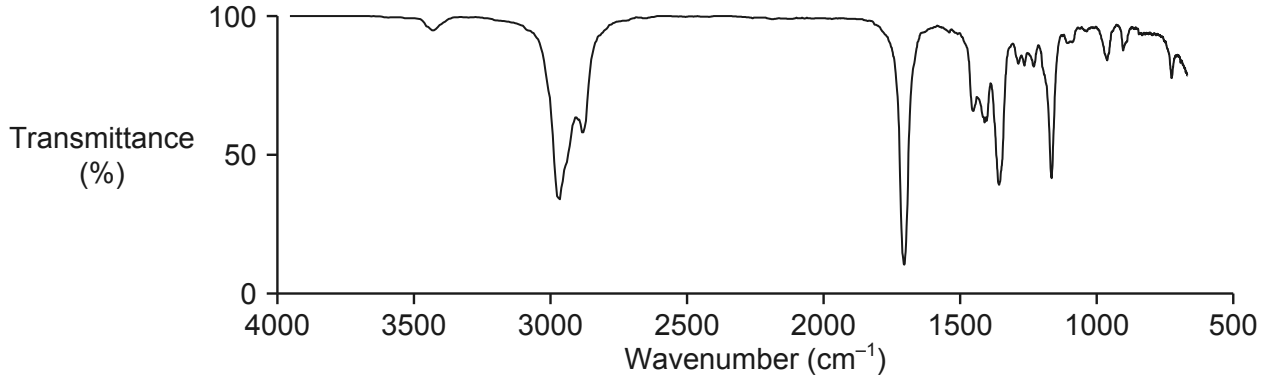
① $n(X) = \frac{5}{2} \times n(MnO_4^-) = \frac{5}{2} \times 7.4010\ldots \times 10^{-3} = 0.018502\ldots$ mol

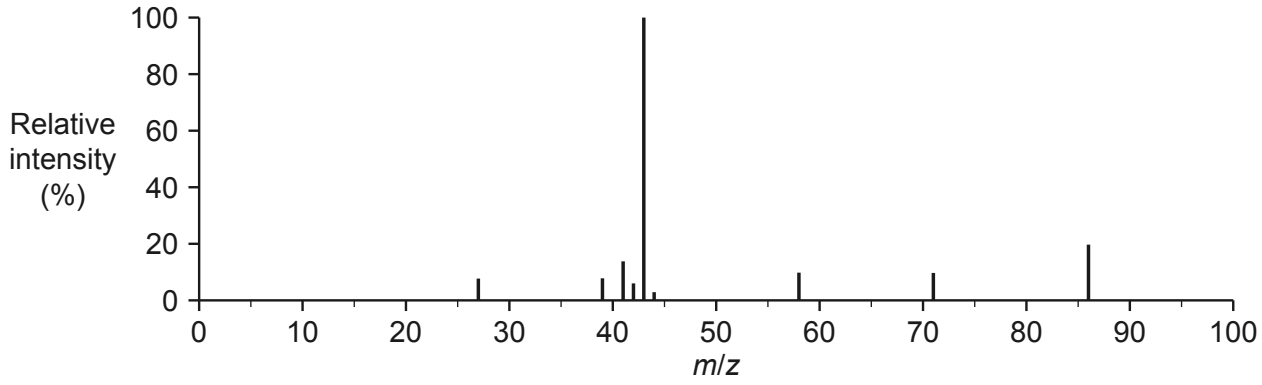
② $m(X) = n \times M = 0.018502\ldots \times 86.0 = 1.59$ g (to 3 s.f.)

Question 2 (15 marks)

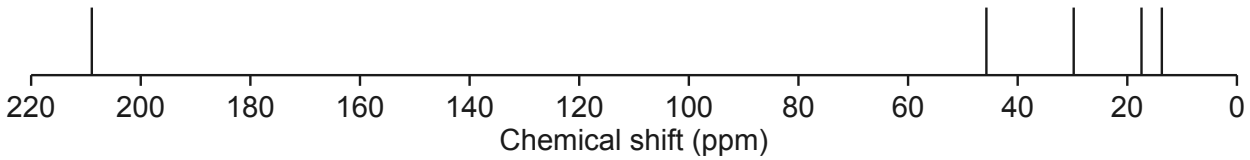
The researcher separates Compound X from the reaction mixture and purifies a sample of it. To identify Compound X, she records four spectra of the purified sample, because each kind of spectrum gives different information about the structure of a compound.

The infrared spectrum, mass spectrum, ^{13}C NMR spectrum and ^{1}H NMR spectrum of Compound X are shown below.

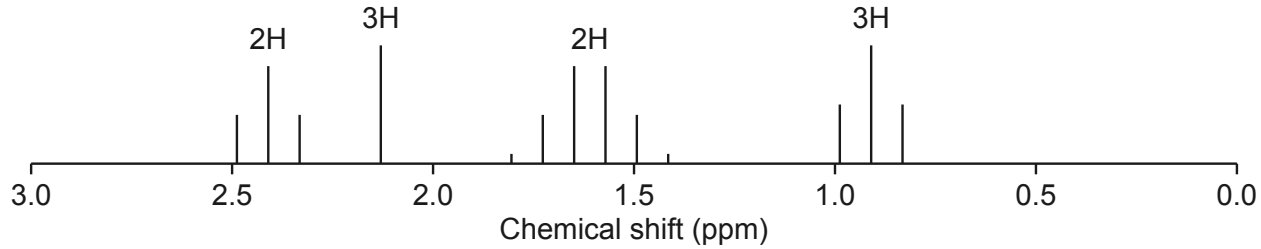




^{13}C NMR spectrum



^{1}H NMR spectrum



a. Refer to the infrared spectrum of Compound X shown at the start of this question.

i. Identify the bond responsible for the strong absorption near 1700 cm^{-1}. 1 mark

① C=O

ii. Explain how the infrared spectrum indicates that the purification has removed Compound Y from this sample. 2 marks

① Compound Y has a hydroxyl group, so it would give a broad O–H absorption between 3200 and 3600 cm^{-1}.

② The spectrum has no broad, strong absorption in this range, so no Compound Y is detected (accept an answer that notes the small, narrow dip near 3400 cm^{-1} but explains that it is not the broad O–H band of an alcohol).

b. Refer to the mass spectrum of Compound X.

i. State the *m/z* value of the molecular ion peak in the mass spectrum. 1 mark

① 86

ii. Write the formula of the ion responsible for the peak at *m/z* = 71. Include its charge. 1 mark

① $[C_4H_7O]^+$ (accept $[CH_3CH_2CH_2CO]^+$), formed by loss of a CH_3 group

c. Identify the type of carbon atom responsible for the signal at 209 ppm in the ^{13}C NMR spectrum. 1 mark

① the carbon atom of the C=O group of a ketone, $R_2C=O$ (accept carbonyl carbon of a ketone)

d. Refer to the ^1H NMR spectrum, in which the label above each signal gives the relative number of hydrogen atoms.

i. Explain the splitting pattern of the signal at 2.4 ppm in terms of the structure of Compound X. 2 marks

① The signal is split into three peaks (a triplet).

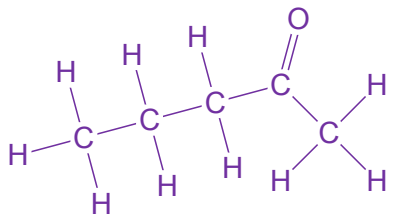
② By the n + 1 rule, these CH_2 protons have two neighbouring protons, on an adjacent CH_2 group.

ii. Deduce the group responsible for the signal at 2.1 ppm. Justify your answer. 2 marks

① a CH_3 group, because the integration is 3H

② The signal is a singlet, so the adjacent carbon atom has no hydrogen atoms: the CH_3 is bonded to the C=O carbon (2.1 to 2.7 ppm).

e. Draw the structural formula of Compound X. Show all atoms and bonds. 2 marks



① C=O group on the second carbon atom of the chain, drawn with every atom and bond.

② Unbranched chain of five carbon atoms with every C–H bond shown.

f. Give the IUPAC name of Compound X. 1 mark

① pentan-2-one

g. Pentanal has the same molecular formula as Compound X and also has five carbon environments. Explain how the ^{1}H NMR spectrum shows that Compound X is not pentanal. 2 marks

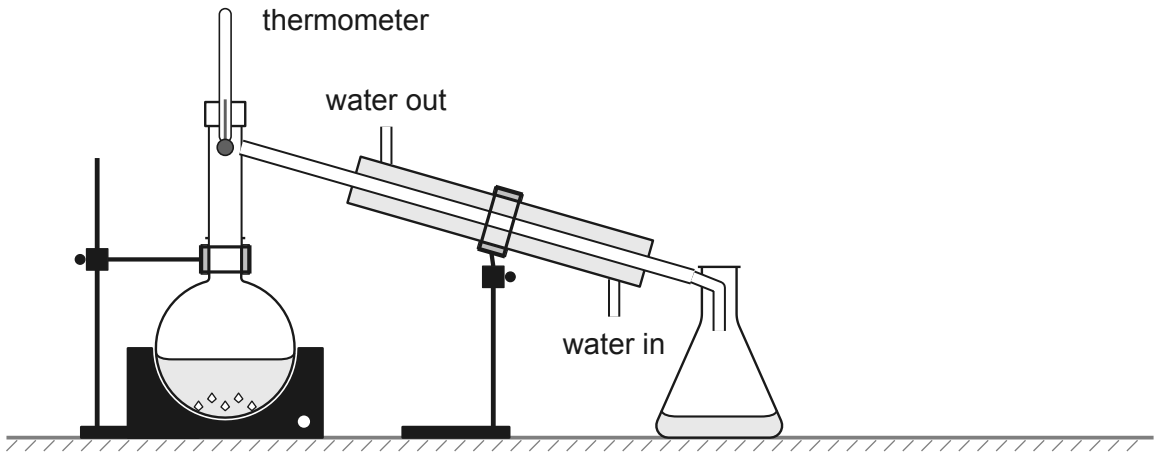
① Pentanal has an aldehyde proton, RCHO, which gives a signal at 9.4 to 10.0 ppm.

② The spectrum of Compound X has no signal in this range (it has a 3H singlet that pentanal would not have).

Question 3 (10 marks)

The researcher now repeats the reaction on a larger scale, so that she has enough of Compound X to use in further work. Before it can be used, she needs to know how pure the product is.

She extracts Compound X and the unreacted Compound Y from the reaction mixture with a solvent. She then removes the solvent and distils the remaining liquid using simple distillation, as shown in the diagram below.



At normal atmospheric pressure, Compound X boils at 102 °C and Compound Y boils at 119 °C.

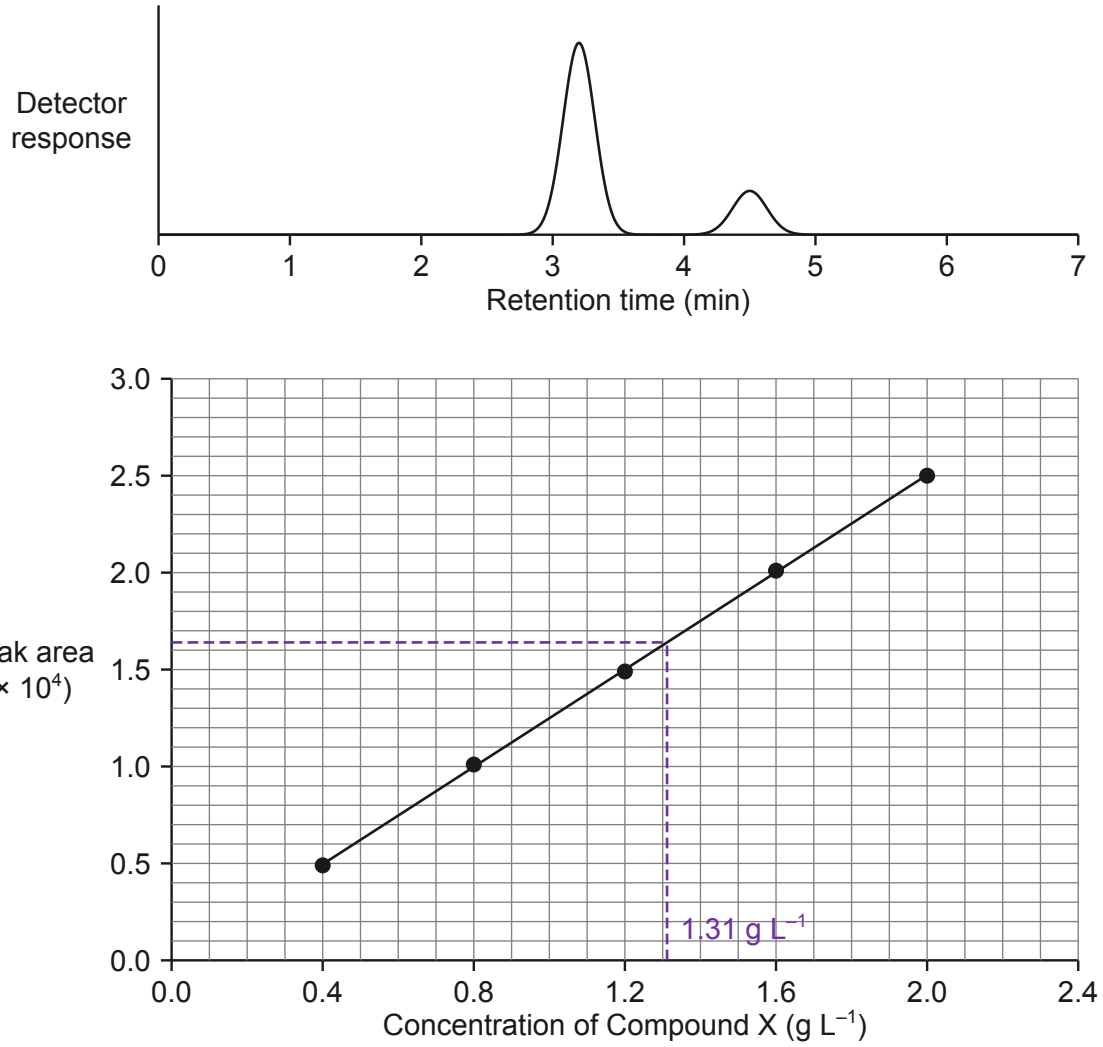
a. The distillate collected is mostly Compound X. Explain why the distillate still contains some Compound Y. 2 marks

① The boiling points differ by only 17 °C, so at the temperature at which X boils, some Y also vaporises.

② Simple distillation has only one vaporisation and condensation step, so the vapour that reaches the condenser, and the distillate, contains some Y as well as X.

The researcher dissolves 0.1527 g of the distillate in a solvent and makes the solution up to 100.0 mL. She analyses this solution by HPLC under the same conditions as standard solutions of Compound X.

Five standard solutions of Compound X, each of known concentration, were analysed first to construct a calibration curve. The column has a polar stationary phase. Under these conditions, Compound X has a retention time of 3.2 min and Compound Y has a retention time of 4.5 min. The peak area for Compound X in the sample is 1.64×10^4. The chromatogram of the sample and the calibration curve for Compound X are shown.



b. Refer to the chromatogram and the calibration curve.

i. State the evidence in the chromatogram that indicates the distillate still contains some Compound Y. 1 mark

① There is a second peak at 4.5 min, the retention time of Compound Y.

ii. Use the calibration curve to determine the concentration, in g L^{-1}, of Compound X in the sample solution. 1 mark

① 1.31 g L^{-1} (accept 1.28 to 1.34 g L^{-1})

iii. Calculate the percentage by mass of Compound X in the distillate. 2 marks

① m(X) = c × V = 1.31 × 0.1000 = 0.131 g

② percentage by mass = $\frac{m(X)}{m(distillate)} \times 100 = \frac{0.131}{0.1527} \times 100$

= 85.8% (to 3 s.f.; accept 84 to 88%, from part b. ii.)

c. A colleague claims: "A sample is pure Compound X if its chromatogram shows only one peak, at the retention time of Compound X." Evaluate this claim. In your answer, refer to the principles of chromatography and to the information provided by other analytical techniques. 4 marks

① A peak at the retention time of the X standard supports the presence of Compound X.

② Another compound could have the same retention time under these conditions, so a single peak does not prove that only X is present.

③ An impurity at a very low concentration may give a peak too small to detect.

④ The claim is not valid as stated: purity should be confirmed by a second method, such as the ^{1}H NMR spectrum or the boiling point.

End of questions

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