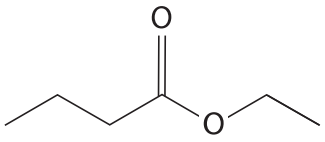
3 The ester ethyl butanoate is found in mangoes.



ethyl butanoate

Ethyl butanoate can be synthesised from butanoic acid.

Procedure

Step **1** Add 5 cm^3 of ethanol and 10 cm^3 of butanoic acid to a round-bottomed flask containing anti-bumping granules. Add 1 cm^3 of concentrated sulfuric acid drop by drop to the flask.

Step **2** Heat the mixture under reflux for 60 minutes. Allow the reaction mixture to cool before transferring it to a separating funnel.

Step **3** Add 15 cm^3 of cold water to the separating funnel and shake the mixture to wash the product. Remove the lower aqueous layer from the funnel.

Step **4** Wash the organic layer that remains in the separating funnel with 10 cm^3 of aqueous sodium hydrogencarbonate solution, $NaHCO_3(aq)$.

Step **5** Remove the aqueous layer from the funnel. Transfer the organic layer to a dry conical flask. Add a drying agent and leave for 10 minutes. Then add more drying agent if required. Remove the drying agent by filtration.

Step **6** Purify the dry organic liquid.

(a) Calculate which reactant is in excess in this synthesis.

Compound	Molar mass / g mol^{-1}	Density / g cm^{-3}
ethanol	46.0	0.79
butanoic acid	88.0	0.96

(2)

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(b) Explain why it is important from time to time in Step **4** either to invert the separating funnel and open the tap, or to remove its stopper.

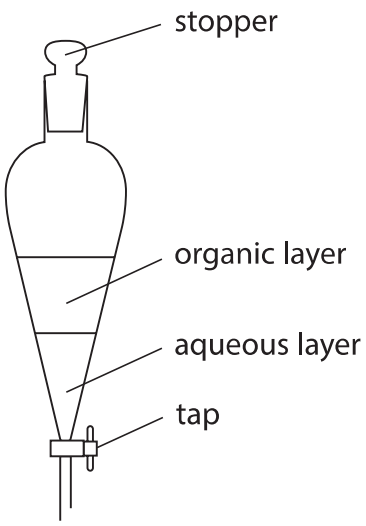
Include an equation to justify your answer. State symbols are not required.

(2)

(c) A student set up a separating funnel in Step **5** as shown and opened the tap to remove the lower layer.

Explain what happens.

Assume that the funnel is supported by suitable clamps.



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(d) Drying agents are used to remove traces of water from an organic liquid.

(i) Identify, by name or formula, a suitable drying agent for use in Step **5**.

(1)

.....

(ii) State how a student would decide whether or not more drying agent was needed in Step **5**.

(1)

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(iii) Name the technique used to obtain a pure sample of the dry organic liquid in Step **6**.

(1)

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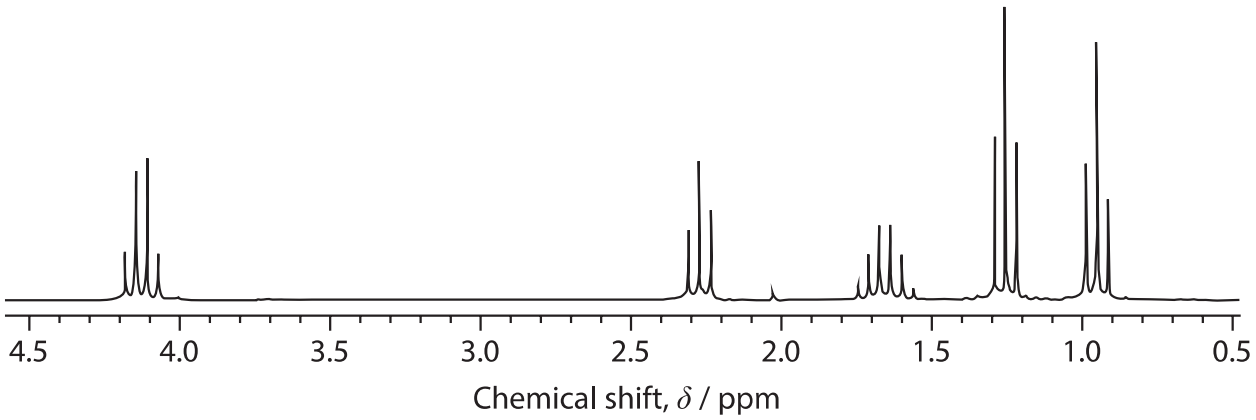
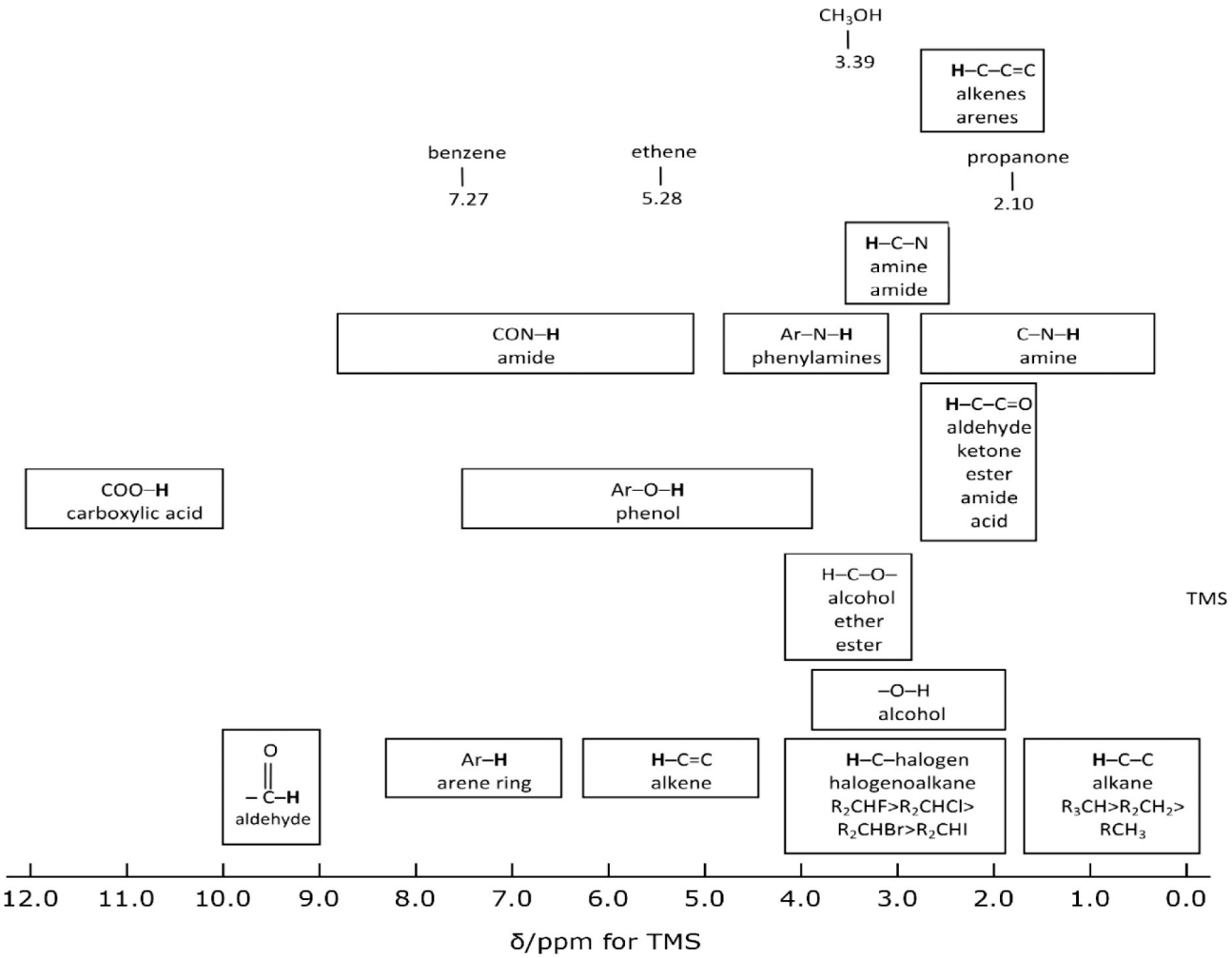
(e) The high resolution proton NMR spectrum of ethyl butanoate is shown.



^{1}H nuclear magnetic resonance chemical shifts relative to tetramethylsilane (TMS)



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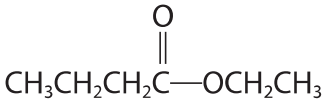
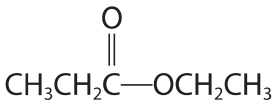
(i) Explain the peaks in the NMR spectrum between 2.2 and 2.4 ppm.



ethyl butanoate

(2)

(ii) Predict how the high resolution proton NMR spectrum of ethyl propanoate will differ from that of ethyl butanoate in terms of the number of peaks and their splitting patterns.



ethyl propanoate

(3)

(Total for Question 3 = 14 marks)

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