Benzene

Question Paper

Level	International A Level
Subject	Chemistry
Exam Board	Edexcel
Topic	Chemistry Lab Skills 2
Sub Topic	Benzene
Booklet	Question Paper

Time Allowed: **50 minutes**

Score: **/41**

Percentage: **/100**

Grade Boundaries:

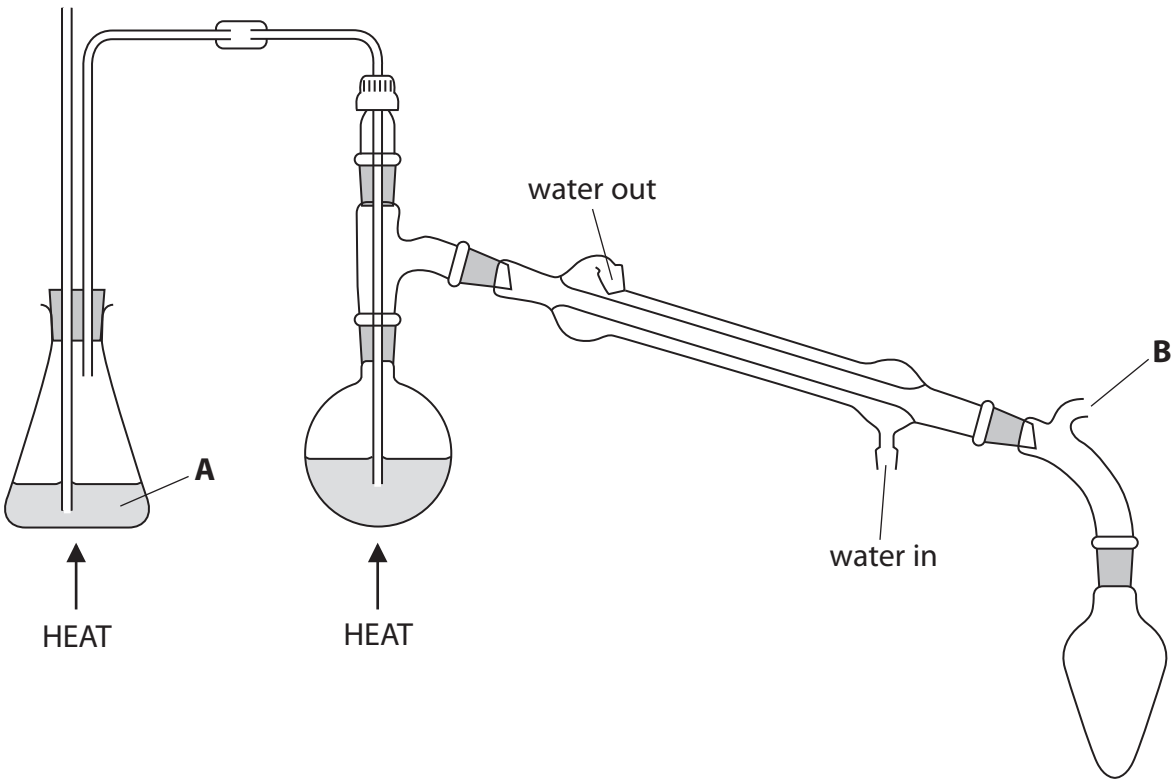
A*	A	B	C	D	E	U
>85%	'77.5%	70%	62.5%	57.5%	45%	<45%

1 Steam distillation is one method used to separate organic compounds from mixtures.

Some information about nitrobenzene is summarised in the table below.

Molecular formula	$C_6H_5NO_2$
Appearance	Oily yellow liquid
Density	1.20 g cm^{-3}
Boiling temperature	211°C
Solubility in water	0.19 g / 100 g of water at 20°C

(a) The diagram below shows a steam distillation apparatus used to extract nitrobenzene from a reaction mixture.



(i) Identify substance **A**.

(1)

(ii) Explain the purpose of the part of the apparatus labelled **B**.

(1)

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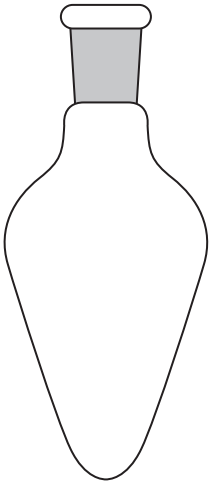
(iii) On the diagram below, draw and label the contents of the receiver at the end of the steam distillation.

(2)



(b) The nitrobenzene may be further purified by simple distillation.

Describe the steps needed **before** the product of steam distillation can be further distilled. Any apparatus or chemicals needed for these steps should be named but practical details are **not** required.

(3)

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(c) A bottle of nitrobenzene has the hazard labels shown below.

Symbol		
Meaning		

(i) Complete the table above with the meaning of each symbol.

(1)

(ii) Suggest **one** change or addition to the **apparatus** in part (a) that would reduce the risk from **both** these hazards.

(1)

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(Total for Question 1 = 9 marks)

2 Bromobenzene can be prepared from benzene by the following steps.

Step 1 Reflux 20.0 cm^3 of benzene with 6.0 cm^3 of bromine and about 10 g of iron filings, by heating on a water bath at 50 °C.

Step 2 After the reaction has finished, remove the water bath and heat to boiling until no bromine vapour can be seen.

Step 3 Cool the mixture and add 25 cm^3 of ethoxyethane (diethyl ether) to extract the bromobenzene.

Step 4 Wash the ethoxyethane layer with aqueous sodium hydroxide. Separate the ethoxyethane layer.

Step 5 Wash the ethoxyethane layer with water and repeat the separation.

Step 6 Dry the ethoxyethane layer with a suitable drying agent.

Step 7 Decant the dried solution.

Step 8 Distil the separated solution, collecting the fraction boiling around the boiling temperature of bromobenzene, 156 °C.

(a) Calculate the number of moles of bromine, Br_2, used in the experiment.

[Density of bromine 3.1 g cm^{-3}]

(2)

(b) Bromine reacts with iron to form iron(III) bromide, which reacts with bromine to produce the attacking electrophile in **Step 1**.

Write the chemical equations for these reactions.

(2)

(c) Why is the ethoxyethane layer washed with sodium hydroxide solution in **Step 4**?

(1)

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(d) Draw a diagram of the apparatus used to separate the ethoxyethane layer from the aqueous layer in **Step 5**. Clearly label the ethoxyethane layer.

[Densities: water 1.0 g cm^{-3}, ethoxyethane 0.7 g cm^{-3}]

(2)

(e) The bromobenzene formed in this reaction can be nitrated to make 2,4-dinitrobromobenzene.

Identify, by name or formula, the chemicals needed for this reaction.

(1)

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(f) 2,4-dinitrobromobenzene reacts with hydrazine hydrate to make 2,4-dinitrophenylhydrazine crystals.

The percentage yields for the reactions are:

75% for the formation of bromobenzene from benzene

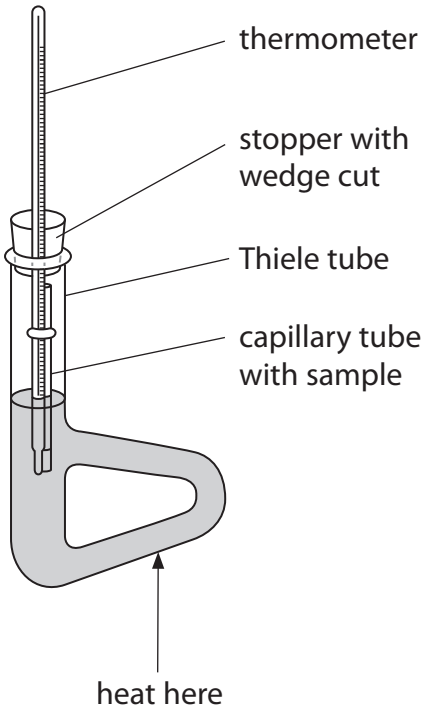
70% for the formation of 2,4-dinitrobromobenzene from bromobenzene

70% for the formation of 2,4-dinitrophenylhydrazine from 2,4-dinitrobromobenzene

Calculate the overall percentage yield of 2,4-dinitrophenylhydrazine from benzene, for this series of reactions.

(1)

(g) The purity of the 2,4-dinitrophenylhydrazine crystals can be checked by carrying out a melting temperature determination using the Thiele tube apparatus shown below.



(i) The capillary tube must be sealed at one end.
Describe how this is done.

(1)

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(ii) When crystals are placed in the capillary tube they often stick in the top.
Describe how to ensure the crystals reach the bottom of the capillary tube.

(1)

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(iii) Dibutyl phthalate is often used as the liquid in the Thiele tube.

Suggest **two** properties of dibutyl phthalate that make it a suitable liquid for this purpose.

(2)

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(iv) The melting temperature for crystals of 2,4-dinitrophenylhydrazine is 201°C.

Suggest the temperature **range** over which you would expect the crystals to melt before and after purification by recrystallization.

(2)

Before recrystallization.....

After recrystallization.....

(Total for Question 2 = 15 marks)

3 Cholesteryl benzoate was the first liquid crystal to be discovered. It can be prepared by the following procedure.

Step 1 Dissolve 1.0 g of cholesterol in 3 cm^3 of pyridine in a conical flask.

Step 2 Add 0.40 cm^3 of benzoyl chloride.

Step 3 Heat the mixture on a steam bath for about 10 minutes.

Step 4 Cool the mixture, and add 15 cm^3 of methanol.

Step 5 Collect the solid cholesteryl benzoate by suction filtration. Rinse the flask and the crude crystals with a little cold methanol.

Step 6 Recrystallize the cholesteryl benzoate using ethyl ethanoate as the solvent.

Some physical data for the chemicals involved are shown below.

	Molar mass / g mol^{-1}	Density / g cm^{-3}	Melting temperature / K	Boiling temperature / K
Cholesterol	386.7			633
Benzoyl chloride	140.6	1.21		470
Cholesteryl benzoate	490.8		423	
Pyridine	79.1			388
Ethyl ethanoate	88.1		190	350

(a) Suggest the apparatus you would use to measure the volume of benzoyl chloride.

(1)

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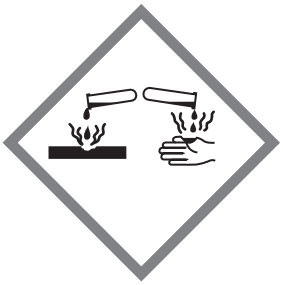
(b) The warning symbols on a bottle of benzoyl chloride are shown below. Write the meaning of each symbol in the space provided.

(2)



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(c) 1 mol of cholesterol reacts with 1 mol of benzoyl chloride to form 1 mol of cholesteryl benzoate.

(i) Determine which reactant is in excess by calculating how many moles of cholesterol and of benzoyl chloride are used in the preparation.

(3)

(ii) Calculate the percentage yield when 0.65 g of cholesteryl benzoate is obtained.

(2)

(d) Suggest how the mixture is cooled in **Step 4**.

(1)

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(e) Suggest why methanol is added to the cooled mixture in **Step 4**.

(1)

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(f) Describe how to carry out the recrystallization to obtain pure dry crystals of cholesteryl benzoate in **Step 6**.

(5)

(g) How would you show that the recrystallized cholesteryl benzoate crystals in **Step 6** are purer than the crude crystals obtained in **Step 5**?

(2)

(Total for Question 3 = 17 marks)