Carbonyls, Carboxylic Acids & Derivatives

Question Paper

Level	International A Level
Subject	Chemistry
Exam Board	Edexcel
Topic	Chemistry Lab Skills 2
Sub Topic	Carbonyls, Carboxylic Acids & Derivatives
Booklet	Question Paper

Time Allowed: **93 minutes**

Score: **/77**

Percentage: **/100**

Grade Boundaries:

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

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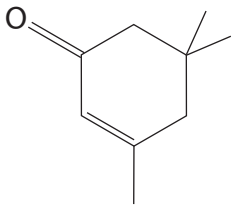
1 Isophorone is a colourless liquid with a peppermint smell, found in cranberries.
The structure of isophorone is shown below.



(a) There are two functional groups present in isophorone.

Name these functional groups and describe a **chemical** test and its result that could be used to identify each functional group.

(4)

Functional group	Test	Result

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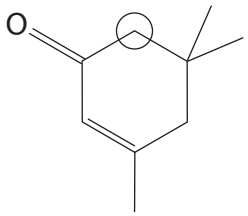
(b) Isophorone has several proton environments that would produce peaks in its proton nuclear magnetic resonance (nmr) spectrum. One of the environments is circled on the structure of isophorone shown below.



(i) The circled proton environment produces a peak in the low resolution nmr spectrum.

State and explain the splitting pattern that you would expect in this peak in the **high** resolution proton nmr spectrum of the molecule.

(1)

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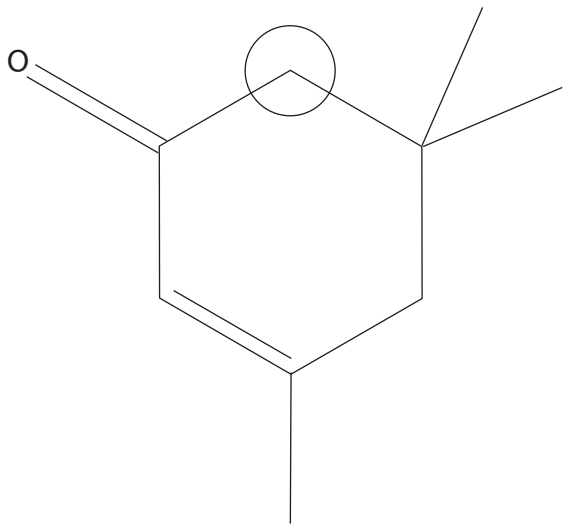
(ii) On the structure of isophorone shown below, circle each of the other proton environments that would produce a peak in the **low** resolution proton nmr spectrum of the molecule. Indicate clearly if any of the proton environments are identical.

(2)



(Total for Question 1 = 7 marks)

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2 Cupronickel is an alloy of copper and nickel. It is used to make 'silver' coins.

A coin is analysed by the following method.

Step 1 It is weighed on a balance which reads to two decimal places and found to have mass 4.00 g.

Step 2 Water is added to the coin in a beaker. Concentrated nitric and sulfuric acids are added and the coin dissolves.

Step 3 When the coin is completely dissolved, the solution is neutralized.

Step 4 The neutral solution is transferred, with the washings, to a 100 cm^3 volumetric flask, made up to the mark with water and mixed thoroughly.

Step 5 10 cm^3 samples of the solution are taken and an excess of potassium iodide is added, producing iodine.

Step 6 The iodine is titrated with 0.200 mol dm^{-3} sodium thiosulfate solution.

(a) Why, in **Step 2**, is water added before, rather than after, the acids?

(1)

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.....

(b) What is the colour of an aqueous iodine solution?

(1)

.....

(c) (i) To make the end point of the titration more obvious, an indicator is added just before the colour of the iodine disappears.

Name this indicator.

(1)

.....

(ii) Suggest why the indicator is not added to the iodine solution earlier in the titration.

(1)

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.....

(iii) e the colour change at the end point when the indicator is used in this titration.

(1)

.....

(d) The results for the titrations are shown below.

Titration number	1	2		
Burette reading (final) / cm^3	24.10	47.90	23.55	47.00
Burette reading (initial) / cm^3	0.00	24.10	0.00	23.55
Titre / cm^3				

(i) Complete the table.

(1)

(ii) Which titres should be used to calculate the mean? Explain your choice.

(1)

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.....

(iii) Calculate the mean titre.

(1)

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(iv) Calculate the percentage by mass of copper in the coin.

Use the equations below.

$$2Cu^{2+}(aq) + 4I^{-}(aq) \rightarrow 2CuI(s) + I_2(aq)$$

$$2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^{-}(aq)$$

(5)

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(v) The uncertainty in each burette reading is ±0.05 cm^3 and the uncertainty in each reading of the balance is ±0.005 g.

Calculate the percentage uncertainty in the third titre value and in the mass measurement. Use your results to decide whether using a balance that weighs to three decimal places would significantly improve the accuracy of the result.

(2)

(Total for Question 2 = 15 marks)

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3 Substance **G** is a colourless organic liquid with one functional group.

(a) A few drops of **G** are tested by the addition of 2,4-dinitrophenylhydrazine solution (Brady's reagent). A **positive** result is obtained.

(i) Describe what you would see when a positive result is obtained for this test.

(1)

.....

(ii) What can you deduce about **G** from this test?

(1)

.....

(b) Substance **G** is tested with Tollens' reagent. The test is **negative**.

(i) Identify the solutions used to make Tollens' reagent.

What condition is essential for this test to work?

What would you see when a **positive** result is obtained?

(4)

Solutions .....

.....

Condition .....

Positive result .....

(ii) Based on the results of the tests in (a)(i) and (b)(i), name the functional group present in **G**.

(1)

.....

(c) A few drops of substance **G** are tested using iodine in the presence of alkali (iodoform test). A positive result is obtained.

(i) What would be **seen** when a positive result is obtained?

(1)

.....

(ii) What information does a positive result give about substance **G**?

(1)

.....

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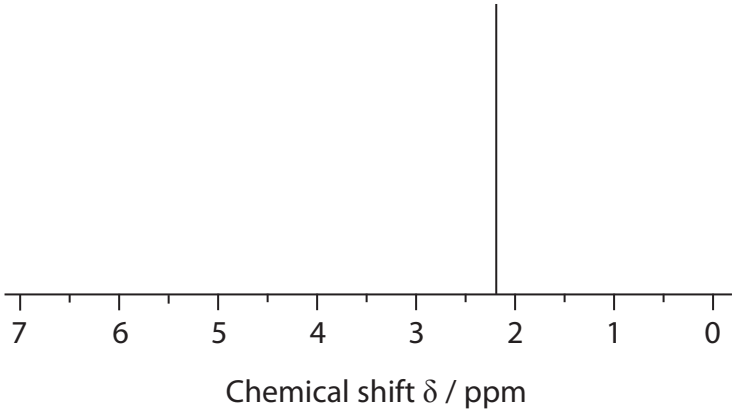
(d) The high resolution nmr spectrum of **G** is shown below.



Give **two** pieces of information about substance **G** that can be deduced from this spectrum. Use this information and your previous deductions to draw the displayed formula of **G**.

(3)

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.....

.....

Displayed formula of **G**:

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(e) The identity of substance **G** can be confirmed by making a larger quantity of the solid product from the reaction of **G** with 2,4-dinitrophenylhydrazine solution and then purifying the product by recrystallization from ethanol.

(i) The solid product is removed from the solution by filtration under reduced pressure. Give **two** advantages of the use of filtration under reduced pressure compared with normal filtration.

(2)

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.....

(ii) Draw a labelled diagram of the apparatus used for filtration under reduced pressure.

(3)

(iii) Outline all the steps of the **recrystallization** procedure. You should assume that the product contains impurities, some of which are very soluble in ethanol and others which are not soluble.

(4)

(iv) How would you use the purified product to confirm the identity of **G**? Practical details are not required.

(2)

(Total for Question 3 = 23 marks)

4 An ester is hydrolysed in the presence of an acid catalyst forming a carboxylic acid and an alcohol **C**. The alcohol contains four carbon atoms.

In order to investigate the kinetics of this reaction, two solutions, **X** and **Y**, were made up.

Solution **X**: 100 cm^3 of a 0.20 mol dm^{-3} solution of the ester
Solution **Y**: 100 cm^3 of a 0.20 mol dm^{-3} solution of hydrochloric acid

Flasks containing the two solutions were placed in a water bath at 50 °C and when both solutions had reached the temperature of the water bath, the solutions were mixed and a clock started. As soon as the clock was started, a 10 cm^3 sample was taken from the reaction mixture, transferred to a cooled conical flask and titrated with 0.050 mol dm^{-3} sodium hydroxide solution. Other samples were taken at two minute intervals and analysed in the same way.

Results:

Time/min	0	2				10	12	14	16
Titre/cm^3	20.0	23.4	26.2	28.5	30.5	32.1	33.4	34.5	35.5
V = (40 – titre)/cm^3	20.0	16.6	13.8	11.5	9.5	7.9	6.6	5.5	4.5

V is directly proportional to the concentration of the ester remaining in the solution.

(a) Why was each sample cooled before titration?

(1)

.....

(b) Two indicators are available for the titrations: phenolphthalein and methyl orange.
Which one should be used? Give a reason for your answer.

(1)

Indicator .....

Reason .....

.....

(c) (i) Explain why the titre at time zero is 20.0 cm^3 rather than 0.0 cm^3. No calculation is required.

(1)

.....

(ii) Explain why the titre increases as the reaction proceeds.

(1)

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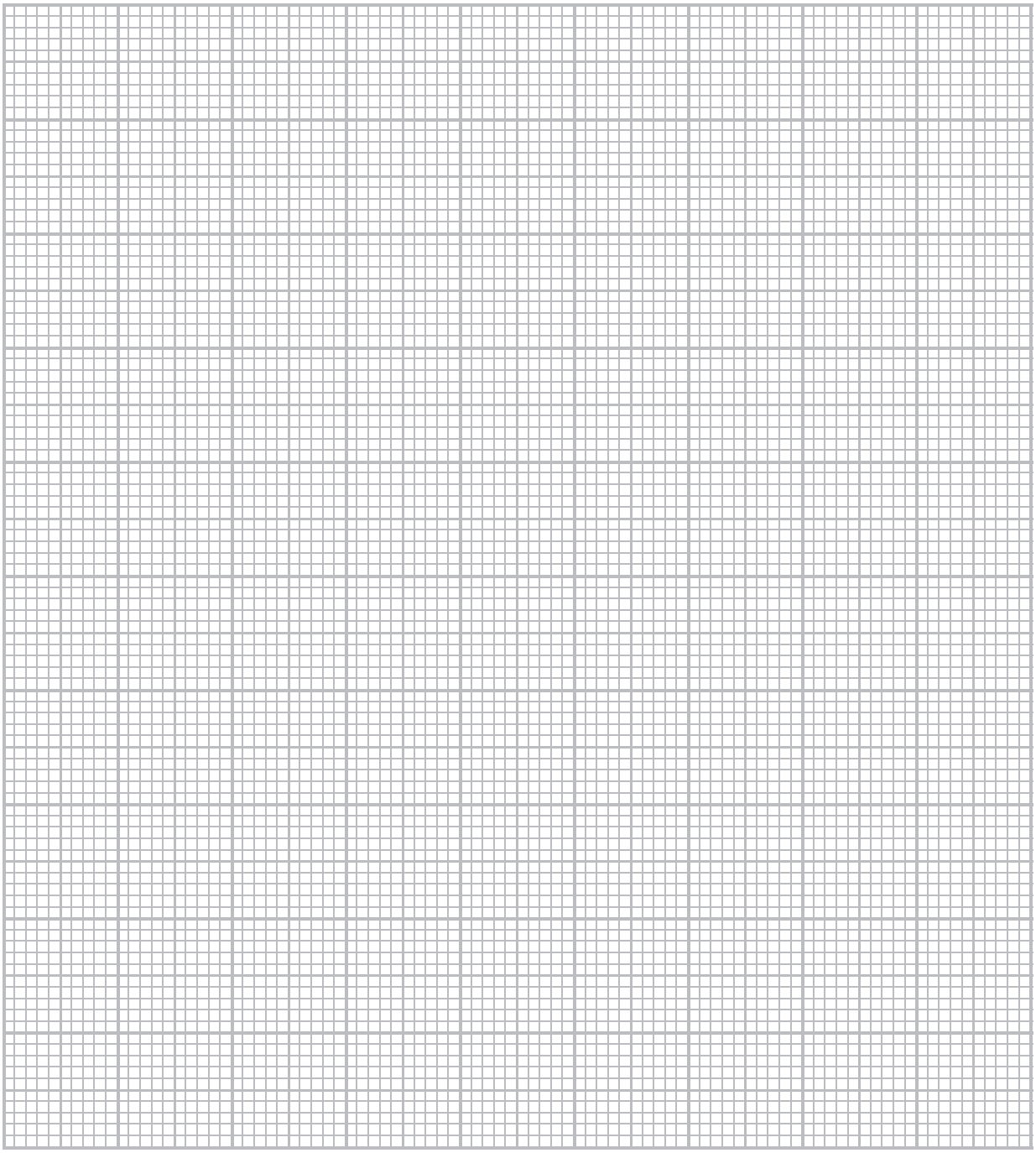
(d) Plot a graph of **V** on the vertical axis against time on the horizontal axis. Use your graph to determine the order of the reaction by measuring two successive half-lives.

(4)



First half-life .....

Second half-life .....

Order .....

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(e) The alcohol **C** is oxidized using potassium dichromate(VI) and dilute sulfuric acid. State the colour change observed.

(1)

From ..... To .....

(f) The oxidation results in the formation of either a carboxylic acid or a ketone.

(i) Suggest a chemical test that could be used to show that the purified product is a carboxylic acid.

Give the observation that you would make when this test is carried out.

(2)

Reagent .....

Observation .....

.....

(ii) Suggest a chemical test that could be used to show that the purified product is a ketone.

Give the observation that you would make when this test is carried out.

(2)

Reagent .....

Observation .....

.....

(g) Tests show that **C** is oxidized to a carboxylic acid. What type of alcohol is **C**?

(1)

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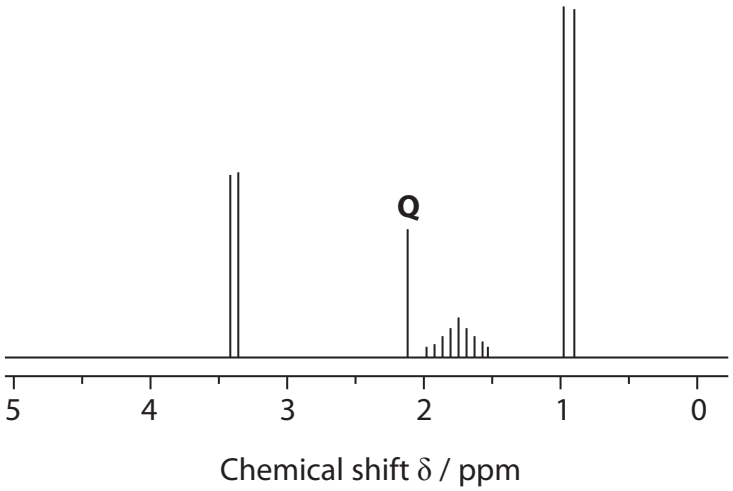
(h) A simplified nmr spectrum for alcohol **C** is shown below:



(i) What can you conclude from the fact that there are four sets of peaks?

(1)

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.....

(ii) Using your answers to (g) and (h)(i), and the fact that alcohol **C** contains four carbon atoms, draw the displayed formula of alcohol **C**.

(1)

(iii) On the displayed formula you have drawn in (h)(ii), circle the atom or group of atoms responsible for the peak labelled **Q**.

(1)

(Total for Question 4 = 17 marks)

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5 This question concerns the preparation of an ester, 3-methylbutyl ethanoate. The ester can be produced by the reaction of 3-methylbutan-1-ol and ethanoic anhydride:

$$C_5H_{11}OH + (CH_3CO)_2O \rightarrow CH_3COOC_5H_{11} + CH_3CO_2H$$

Reagents

- 3-methylbutan-1-ol [molar mass = 88.0 g mol^{-1}; density = 0.81 g cm^{-3}]
- ethanoic anhydride

Required product

- 3-methylbutyl ethanoate

  [molar mass = 130.0 g mol^{-1}; boiling temperature = 142 °C]

Safety information

- 3-methylbutan-1-ol is highly flammable
- 3-methylbutyl ethanoate is highly flammable
- ethanoic anhydride is corrosive, causing skin blistering and peeling

The steps of the experimental procedure are as follows:

Step 1 Place 10.0 cm^3 of 3-methylbutan-1-ol in a flask and add a few anti-bumping granules.

Step 2 Set up the apparatus for reflux. Pour 12.5 cm^3 of ethanoic anhydride (a slight excess) down the condenser. Warm the mixture until the reaction starts and then reflux gently for five minutes. Allow the mixture to cool.

Step 3 Transfer the cooled mixture to a separating funnel, leaving the anti-bumping granules in the flask. Add about 25 cm^3 of water and shake the mixture. Allow the two layers to separate and discard the lower aqueous layer. The addition of water converts any unreacted ethanoic anhydride into ethanoic acid.

Step 4 Add about 10 cm^3 of aqueous sodium hydrogencarbonate to the separating funnel and shake carefully. When the vigorous effervescence has finished, insert the stopper and shake the funnel, frequently releasing the pressure. Repeat the washing with further quantities of aqueous sodium hydrogencarbonate until no more gas is produced. Discard the lower aqueous layer each time.

Step 5 Transfer the ester to a conical flask and shake the flask for five minutes with a suitable drying agent.

Step 6 Filter the dried ester directly into a flask. Set up the apparatus for simple distillation, adding a few anti-bumping granules to the flask. Distil off the ester.

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(a) State **two** safety precautions, each related to a specific hazard of this experiment. You may assume that eye protection and laboratory coats are being worn and that the experiment was carried out in a fume cupboard.

(2)

Hazard 1 .....

Precaution 1 .....

Hazard 2 .....

Precaution 2 .....

(b) Draw a labelled diagram of the apparatus needed for heating under reflux in **Step 2**. You do not need to show stands or clamps.

(3)

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(c) Why are anti-bumping granules added to the flask in **Step 1**?

(1)

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.....

(d) What is the purpose of adding aqueous sodium hydrogencarbonate in **Step 4**?

(1)

.....

.....

(e) In the following list of substances, only one would be a suitable drying agent to use in **Step 5**. Identify this drying agent, giving a reason for your choice:

(2)

- concentrated phosphoric(V) acid
- sodium hydroxide solid
- anhydrous sodium sulfate
- concentrated sulfuric acid

Drying agent .....

Reason .....

.....

(f) In **Step 6**, the ester is distilled off. Suggest a suitable temperature range over which to collect the ester.

(1)

From .....°C to .....°C

(g) (i) Calculate the maximum mass of 3-methylbutyl ethanoate that could be obtained in this experiment from 10 cm^3 of 3-methylbutan-1-ol. Give your answer to **three** significant figures.

(3)

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(ii) A student carried out the synthesis and obtained 9.45 g of 3-methylbutyl ethanoate. Calculate the percentage yield.

(2)

(Total for Question 5 = 15 marks)