

Please check the examination details below before entering your candidate information

Candidate surname		Other names	
Pearson Edexcel International Advanced Level		Centre Number	Candidate Number
Friday 9 November 2018			
Morning (Time: 1 hour 15 minutes)		Paper Reference WCH06/01	
Chemistry Advanced Unit 6: Chemistry Laboratory Skills II			
You must have: Scientific calculator Ruler			Total Marks

Instructions

- Use **black** ink or **black** ball-point pen.
- **Fill in the boxes** at the top of this page with your name, centre number and candidate number.
- Answer **all** questions.
- Answer the questions in the spaces provided
– *there may be more space than you need.*

Information

- The total mark for this paper is 50.
- The marks for **each** question are shown in brackets
– *use this as a guide as to how much time to spend on each question.*
- You will be assessed on your ability to organise and present information, ideas, descriptions and arguments clearly and logically, including your use of grammar, punctuation and spelling.
- A Periodic Table is printed on the back cover of this paper.

Advice

- Read each question carefully before you start to answer it.
- Show all your working in calculations and include units where appropriate.
- Check your answers if you have time at the end.

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Answer ALL the questions. Write your answers in the spaces provided.

- 1 A blue solid was given to a student who suggested, from its **colour only**, that it was copper(II) sulfate. Another student suggested that it was copper(II) hydroxide, which is also blue. The students proposed some tests to distinguish between these two blue solids.

- (a) Complete the table to give the **expected** results for copper(II) sulfate and copper(II) hydroxide.

Test	Observation for copper(II) sulfate	Observation for copper(II) hydroxide	
(i) Add excess dilute hydrochloric acid			(1)
(ii) Add barium chloride solution to the products of test (i)			(1)

- (b) A flame test was carried out on the blue solid, but the flame colour observed was **not** that expected for a blue solid containing copper(II) ions.

Therefore another student suggested that the blue solid could be anhydrous cobalt(II) chloride, which can easily be distinguished from copper(II) compounds by the addition of deionised water.

- (i) Complete the table to give the **expected** result for the addition of deionised water to anhydrous cobalt(II) chloride.

Test	Observation	Inference	
Addition of deionised water	The colour of the solution formed is	The formula of the complex ion formed is	(2)

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- (ii) Cobalt(II) chloride can be distinguished from copper(II) sulfate and copper(II) hydroxide by testing for the anion. Describe a suitable test for the chloride ion, including the positive result.

(2)

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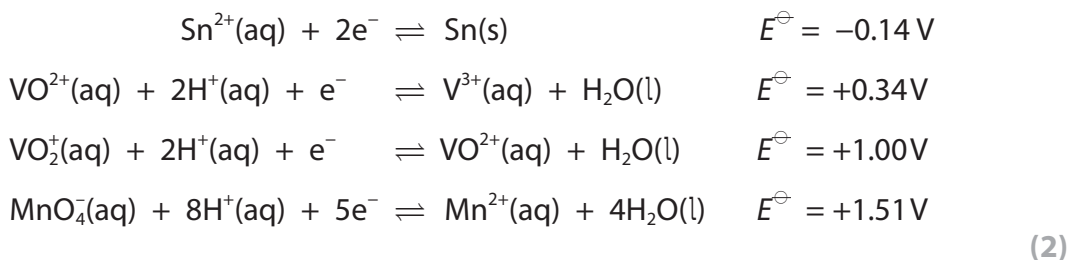


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- (c) The results from these tests made the students realise that the blue solid was not cobalt(II) chloride.

The teacher reminded the students that other transition metals form blue salts and told them that the blue solid in this experiment was hydrated oxovanadium(IV) sulfate, $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$.

Using the standard electrode potential data, select the reagent that would convert the blue vanadium(IV) compound into a yellow vanadium(V) compound. Justify your choice by calculating the appropriate $E_{\text{cell}}^\ominus$ value.



- (d) The blue hydrated oxovanadium(IV) sulfate can be converted into anhydrous oxovanadium(IV) sulfate by heating.

One student heated a sample of the salt in a crucible and saw a hard crust form on the surface.

A glass rod was used to break up the crust and the mass decreased on further heating.

- (i) Give a possible formula for the vanadium salt that was present in the sample beneath the crust.

(1)

- (ii) Give a reason why the mass decreased on further heating.

(1)



- (iii) Calculate the percentage loss in mass when all the hydrated oxovanadium(IV) sulfate, $\text{VOSO}_4 \cdot 5\text{H}_2\text{O}$, is converted into the anhydrous compound.

(2)

(Total for Question 1 = 12 marks)



- 2 The equation for the reaction between iodine and propanone in acidic solution is



The order of reaction with respect to iodine was investigated using a titration method.

The concentration of hydrogen ions and propanone were in large excess. 30 cm^3 of acidified aqueous propanone was added to a flask containing 30.0 cm^3 of 0.020 mol dm^{-3} aqueous iodine. At the same time, the contents were mixed thoroughly and a timer started.

A pipette was used to remove 10.0 cm^3 samples of the reaction mixture every 5 minutes. The samples were immediately run into flasks containing sodium hydrogencarbonate solution, which quenched the reaction.

The volume of sodium thiosulfate solution needed to react with the iodine in each quenched sample was then determined by titration.

- (a) Give an advantage of quenching the sample of the reaction mixture with a **solution** of sodium hydrogencarbonate rather than the solid.

(1)

- (b) The results were recorded in a table

Volume of sodium thiosulfate / cm^3	Time the sample was quenched / minutes
	0
18.50	5
16.10	10
13.50	15
10.90	20
8.50	25

- (i) Complete the table by estimating the volume of sodium thiosulfate that would be required for titration at time = 0.

(1)

- (ii) Plot a graph of volume of sodium thiosulfate on the vertical axis, against time on the horizontal axis.

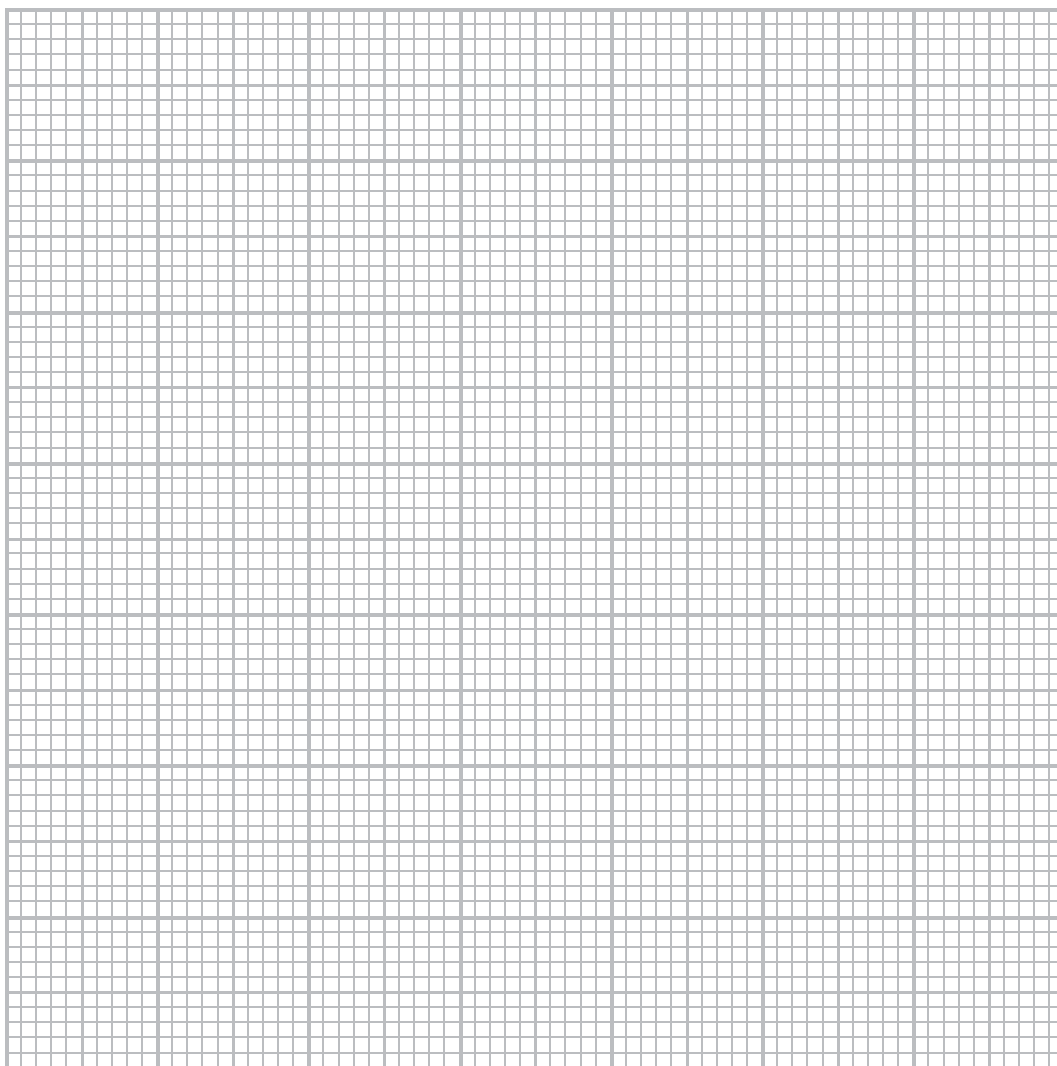
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- (iii) Calculate the gradient of the line drawn through the points.
Include units in your answer.

(2)



- (c) Assume that the volume of sodium thiosulfate required is proportional to the amount of iodine in the reaction mixture.

Deduce the order of reaction with respect to iodine. Justify your answer.

(1)

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- (d) Give a reason why it is acceptable for a sample of the reaction mixture to be quenched at times other than every 5 minutes, for example at 4 minutes 30 seconds.

(1)

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- (e) Explain why it is **not** possible to remove a sixth sample by pipette and state how it would still be possible to titrate a sixth sample.

(2)

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- (f) One factor, not controlled in this experiment, also affects the reaction rate. State this factor and describe how the investigation could be modified to control it.

(2)

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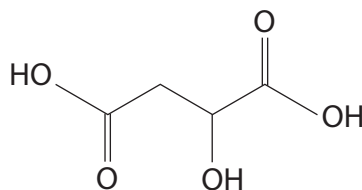
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(Total for Question 2 = 13 marks)



- 3 Malic acid is found in all fruit juices and is the main acid in some fruits such as apples. The structure of malic acid is



- (a) The concentration of acid in fruit juice can be determined by an acid-base titration. For this calculation, assume that **all** the acid present in apple juice is malic acid.

In an experiment to find the concentration of malic acid, 25.0 cm^3 samples of freshly-squeezed apple juice were pipetted into conical flasks and titrated against 0.100 mol dm^{-3} sodium hydroxide solution.

The results from one student are given in the table.

Titration number	1	2	3
Final burette reading / cm^3	17.10	36.10	26.80
Initial burette reading / cm^3	0.00	20.00	10.00
Volume of NaOH(aq) used / cm^3	17.10	16.10	16.80

- (i) State why it would **not** be good practice to use the results from this student to calculate a mean titre.

(1)

- (ii) The indicator phenolphthalein was used to determine the end-point for this titration.

Give the colour change at the end-point.

(1)

From to



- (iii) Suggest why the end-point may have been difficult to observe accurately when carrying out this titration.

(1)

- (iv) Another student obtained a mean titre of 16.80 cm^3 .

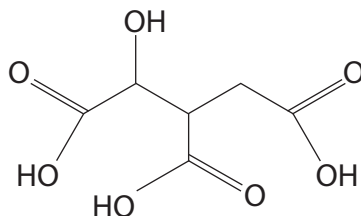
Use this result to calculate the concentration of malic acid, in mol dm^{-3} , in the apple juice.

Give your answer to **two** significant figures.

(3)

- (v) It has been assumed that all the acid in the apple juice was malic acid but other acids such as isocitric acid are also present.

The structure of isocitric acid is



Predict the mean titre if **all** of the acid in the apple juice is not malic acid, but an equal number of moles of isocitric acid.

Justify your answer.

(2)

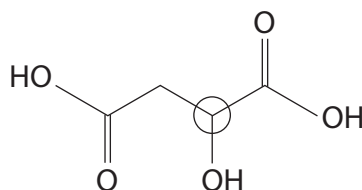
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- (b) The proton environment on the carbon atom circled on the structure of malic acid produces a peak in the proton nuclear magnetic resonance (nmr) spectrum.



- (i) State the splitting pattern that you would expect for this peak, in the **high** resolution proton nmr spectrum of the malic acid. Justify your answer.

(1)

- (ii) If the malic acid is dissolved in deuterium oxide, D_2O , the hydrogens of the carboxylic acid and alcohol groups are replaced by deuterium atoms. The deuterium atoms do **not** give rise to peaks in the proton nmr spectrum.

Deduce the number of peaks in the **low** resolution nmr spectrum of malic acid that would be seen if the malic acid was dissolved in D_2O .

(1)

- (iii) Identify, by name or formula, the substance that is added to a sample to provide the nmr standard reference peak with a chemical shift of zero.

(1)



(c) Malic acid has many peaks in its mass spectrum.

(i) Give the **structural** formula of the ion with $m/e = 45$.

(1)

(ii) Give the **molecular** formula of the ion with $m/e = 89$.

(1)

(Total for Question 3 = 13 marks)



- 4 The outline procedure for the preparation of methyl 3-nitrobenzoate by the nitration of methyl benzoate is shown.

Procedure

- Step 1 2.0 g of methyl benzoate is placed into a clean dry boiling tube.
- Step 2 4 cm³ of concentrated sulfuric acid is added to the methyl benzoate.
- Step 3 The boiling tube is then placed in an ice-water mixture and left to cool to below 5 °C.
- Step 4 1.5 cm³ of concentrated nitric acid is placed in a separate clean, dry boiling tube in the ice-water mixture. 1.5 cm³ of concentrated sulfuric acid is carefully added to the nitric acid, forming the nitrating mixture.
- Step 5 The nitrating mixture is added drop-by-drop to the acidified methyl benzoate, with stirring, and great care is taken to ensure that the temperature does not rise above 10 °C.
- Step 6 After all the nitrating mixture has been added, the boiling tube is left at room temperature for 15 minutes.
- Step 7 The reaction mixture in the boiling tube is poured into a beaker containing about 25 g of crushed ice. The mixture is stirred until solid methyl 3-nitrobenzoate forms.
- Step 8 When the ice has melted, the impure methyl 3-nitrobenzoate is filtered off under reduced pressure and washed with ice-cold deionised water.

- (a) Suggest why an ice-water mixture is used for cooling rather than only ice cubes.

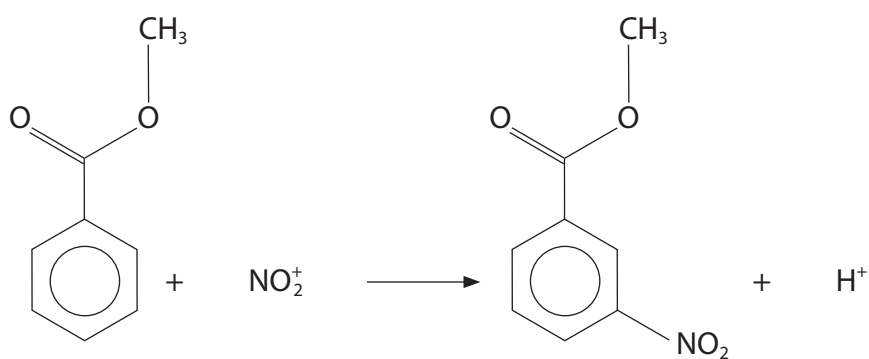
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(b) The equation for the nitration of methyl benzoate is



(i) Give the structure of a possible isomeric product of this reaction.

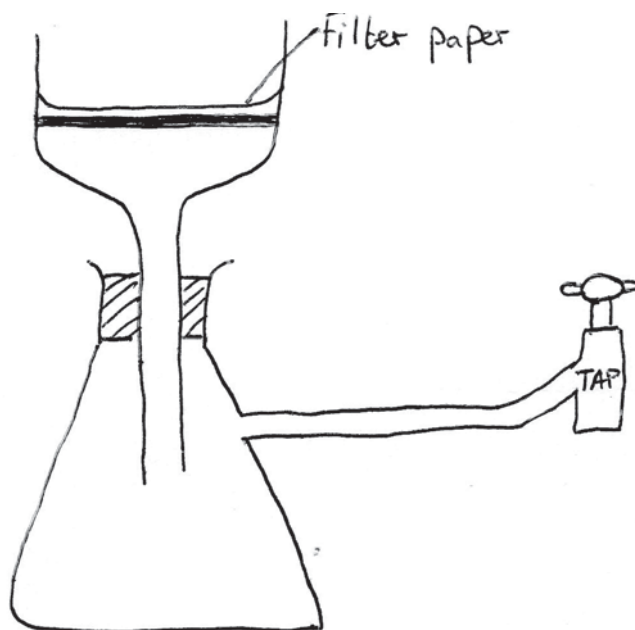
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(ii) Give the structure of a possible aromatic product of this reaction if the temperature of the reaction mixture rises above 10°C.

(1)



- (c) A student diagram of a reduced pressure filtration apparatus is shown. It has **two** mistakes.



State how the diagram should be modified to correct the **two** mistakes.

(2)

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(d) Ethanol is a suitable solvent for recrystallising methyl 3-nitrobenzoate.

Describe the steps you would use to carry out recrystallisation of the crude methyl 3-nitrobenzoate obtained at the end of Step 8 in order to remove any **soluble** impurities and to obtain a pure dry sample of methyl 3-nitrobenzoate.

(4)

(e) Calculate the mass of methyl 3-nitrobenzoate produced from 2.0 g of methyl benzoate if the percentage yield is 73%.

	methyl benzoate	methyl 3-nitrobenzoate
Molar mass / g mol ⁻¹	136	181

(2)



- (f) The purity of the methyl 3-nitrobenzoate can be tested by determining the melting temperature of the crystals. The literature value for the melting temperature of methyl 3-nitrobenzoate is 78–80°C.

State how this melting temperature range would change if impurities were present in the crystals.

(1)

(Total for Question 4 = 12 marks)

TOTAL FOR PAPER = 50 MARKS

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