

Write your name here

Surname

Other names

Centre Number

Candidate Number

Edexcel GCE

Chemistry

Advanced

Unit 6B: Chemistry Laboratory Skills II Alternative

Tuesday 22 May 2012 – Morning

Time: 1 hour 15 minutes

Paper Reference

6CH08/01

Candidates may use a calculator.

Total Marks

Instructions

- Use **black** ink or 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.
- Keep an eye on the time.
- Try to answer every question.
- Check your answers if you have time at the end.

Turn over ►

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PEARSON

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

- 1 (a) The colours of aqueous solutions containing chromium(III) chloride and nickel(II) chloride are similar.

What colour are these solutions?

(1)

- (b) Tests were carried out on a dilute aqueous solution of chromium(III) chloride. Complete the table below.

You may use **either** names **or** formulae unless only **one** of these is specified.

	Test	Observations	Inferences	
(i)	Add a few drops of dilute sodium hydroxide solution to the chromium(III) chloride solution.	A precipitate forms.	The precipitate is	(1)
(ii)	Add dilute sodium hydroxide to the mixture made in (i), until the sodium hydroxide is present in excess.	The complex ion $[\text{Cr}(\text{OH})_6]^{3-}$ forms.	(1)
(iii)	Add a few drops of dilute ammonia to another sample of the chromium(III) chloride solution.	The substance containing chromium which is observed on adding the ammonia is	(2)



	Test	Observations	Inferences	
(iv)	Continue to add dilute ammonia to the mixture in (iii) until the ammonia is present in excess.	A solution forms.	The formula of the chromium containing ion in the solution is	(1)
(v)	Warm another sample of the chromium(III) chloride solution with alkaline hydrogen peroxide solution, which acts as an oxidizing agent. Add sulfuric acid to the resulting mixture.	A yellow solution forms. The solution goes orange when sulfuric acid is added.	The formula of the ion causing the yellow colour is The ion causing the orange colour is dichromate(VI), $\text{Cr}_2\text{O}_7^{2-}$.	(1)

(c) Tests (i) and (ii) above were repeated on an aqueous solution of nickel(II) chloride.

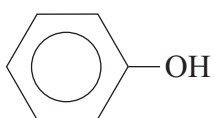
In what way, other than any difference in colour, does the reaction of dilute sodium hydroxide solution with nickel(II) chloride differ from its reactions with chromium(III) chloride?

(1)

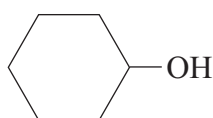
(Total for Question 1 = 8 marks)



2 This question is about some reactions of phenol and cyclohexanol.



phenol



cyclohexanol

(a) Give **two** observations you would make when bromine water is added, drop by drop, to an aqueous solution of phenol.

(2)

(b) (i) What is observed when cyclohexanol is warmed with a mixture of aqueous potassium dichromate(VI) and sulfuric acid?

(1)

(ii) Give the skeletal formula of the organic product of the reaction in (b)(i).

(1)

(iii) What change, if any, is observed when the organic product of the reaction in (b)(i) is mixed with the following reagents?

(2)

2,4-dinitrophenylhydrazine solution

Tollens' reagent

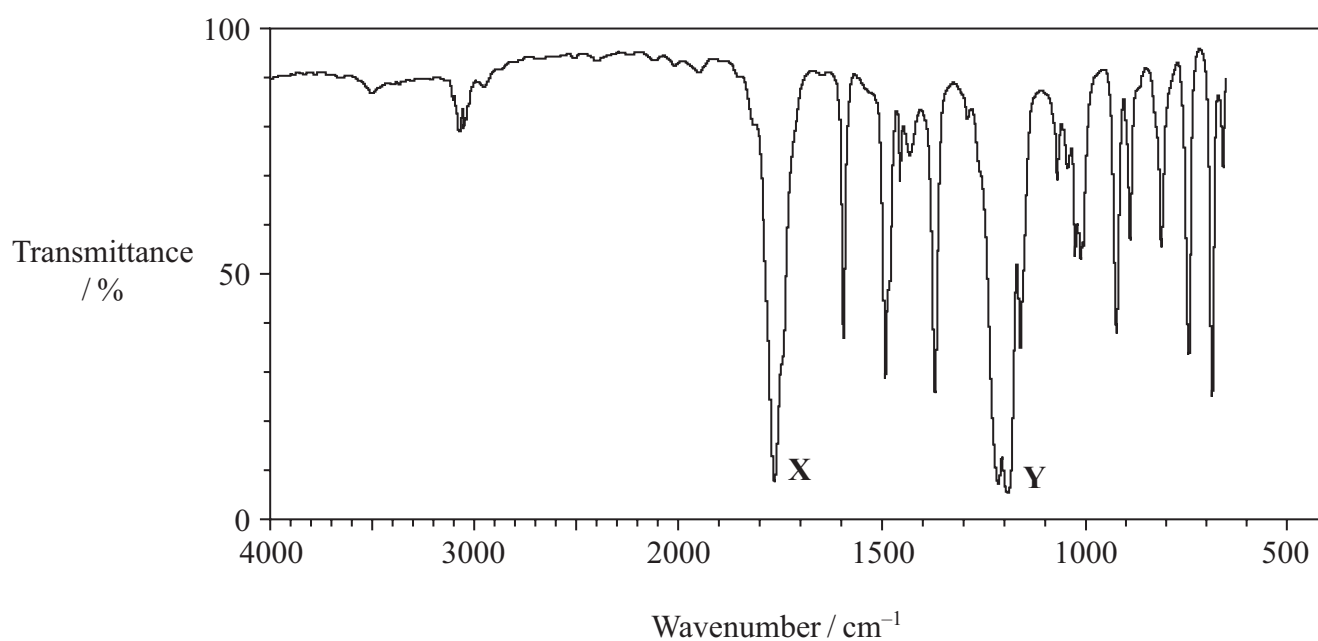
(c) Both phenol and cyclohexanol react with ethanoyl chloride to produce steamy fumes and an ester. Phenol behaves like an alcohol in this reaction.

(i) How could you show that the steamy fumes were due to the presence of a hydrogen halide, which in this case is hydrogen chloride?

(2)



(ii) The infrared spectrum below is for the ester produced in the reaction of ethanoyl chloride with phenol.



Bond	Group	Wavenumber range / cm ⁻¹
C—H	alkanes	2962 – 2853
	arenes	3030
O—H	alcohols and phenols	3750 – 3200
C—O	ethanoates	1250 – 1190
	benzoates	1310 – 1250 and 1150 – 1100
C=C	arenes	1600, 1580, 1500, 1450
C=O	ketones	1700 – 1680
	esters	1770 – 1715

Identify the bond and group which cause each of the absorptions X and Y.

(2)

X

Y



(iii) Draw the structural formula of the ester produced in the reaction of ethanoyl chloride with phenol.

(1)

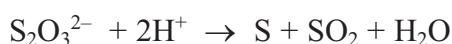
(Total for Question 2 = 11 marks)



3 Some old coins with a high copper content were analysed as follows.

Procedure

1. The coins were weighed and dissolved in concentrated nitric acid, producing a solution which contained copper(II) nitrate.
 2. The solution containing copper(II) nitrate was neutralized by adding sodium carbonate solution until a precipitate of copper(II) carbonate just appeared. Dilute ethanoic acid was then added, drop by drop, until the copper(II) carbonate precipitate just dissolved.
 3. The solution containing copper(II) nitrate was transferred to a volumetric flask and made up to 250 cm³ with distilled water.
 4. 25 cm³ portions of this solution were transferred to separate conical flasks. Then 10 cm³ of 1.0 mol dm⁻³ potassium iodide (an excess) was added to each flask.
 5. The liberated iodine was titrated with 0.125 mol dm⁻³ sodium thiosulfate solution.
- (a) One reason why the solution for titration must be neutralized is because sodium thiosulfate reacts with acid as shown below.



State **one** observation you would make when an acid reacts with sodium thiosulfate solution.

(1)

(b) (i) What colour is the diluted solution containing copper(II) nitrate?

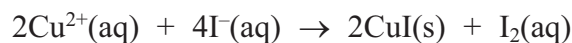
(1)

(ii) What would you observe in Step 2, before the formation of the copper(II) carbonate precipitate, when the sodium carbonate was added?

(1)



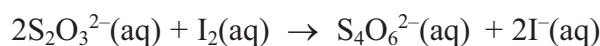
(c) The equation for the reaction producing iodine in Step 4 is shown below.



(i) Give the name of the precipitate formed in this reaction. (1)

(ii) Suggest, by considering the electronic configuration of the relevant ion, why the precipitate is white. (1)

(d) The equation for the reaction of thiosulfate ions in the titration is



Results:

Mass of coins	2.10 g
Mean (average) volume of 0.125 mol dm ⁻³ sodium thiosulfate used in titration	24.40 cm ³

(i) Calculate the number of moles of sodium thiosulfate used in the titration. (1)

(ii) Calculate the number of moles of Cu²⁺ in the 25 cm³ samples used for the titration. (2)



(iii) Hence calculate the mass of copper present in the original mass of coins. (2)

(iv) What is the percentage of copper in the coins? (1)

(e) (i) The balance used to weigh the coins produced a **total** error in the weighing of ± 0.01 g. Calculate the percentage error in the weighing. (1)

(ii) The error in the mean titre of 24.40 cm^3 was $\pm 0.10 \text{ cm}^3$. Show, by calculation, that the percentage error in the titration reading is less than the percentage error in the balance reading. (1)

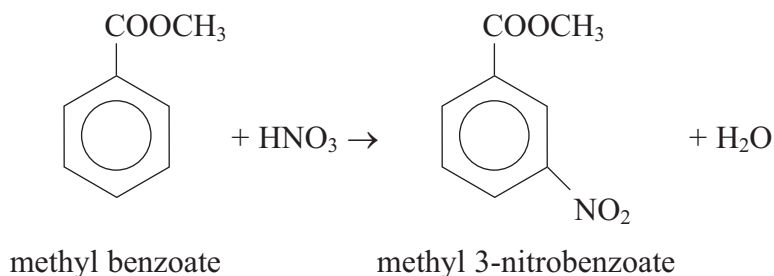
(f) Starch solution can be used to show the end point for this titration, or the titration can be self-indicating.

What colour change would be observed at the end point if starch was **not** used? (1)

(Total for Question 3 = 14 marks)



- 4 A student attempted to make a sample of methyl 3-nitrobenzoate using the following reaction.



Procedure

1. Transfer 9 cm³ of concentrated sulfuric acid into a 100 cm³ conical flask and cool it to below 10°C in an ice bath. Add 5.0 g of methyl benzoate, swirling the flask. Mix 3 cm³ of concentrated nitric acid with 3 cm³ of concentrated sulfuric acid in another small flask and cool it in ice.
 2. Add the mixture of nitric and sulfuric acids, drop by drop, to the methyl benzoate solution, making sure that the temperature stays below 15°C.
 3. Take the mixture out of the ice bath and leave it to stand for 10 minutes at room temperature. Pour the mixture over 40 g of crushed ice and collect the solid product by filtering the mixture under suction. Wash the precipitate, first with cold water, then with ice-cold ethanol. Keep the washings obtained with the ethanol for a further experiment.
 4. Purify the impure methyl 3-nitrobenzoate by recrystallization, using ethanol as the solvent, cooling the solution in an ice bath to assist recrystallization.
 5. Dry the recrystallized product and determine the yield.
- (a) The student wore goggles and a laboratory coat. For each of the processes below, state the hazard and give one further safety precaution which should be taken.

(i) Working with concentrated nitric and sulfuric acids.

(1)

(ii) Carrying out the recrystallization using ethanol.

(1)



(b) What is the purpose of adding sulfuric acid to the nitric acid in this reaction?

(1)

(c) (i) Calculate the number of moles in 5.0 g of methyl benzoate.

Assume the molar mass of methyl benzoate is 136 g mol^{-1} .

(1)

(ii) Methyl benzoate is a liquid at room temperature. What is the volume of 5.0 g of methyl benzoate?

The density of methyl benzoate is 1.09 g cm^{-3} .

(1)

(iii) After recrystallization and drying, 3.4 g of methyl 3-nitrobenzoate was obtained.

Calculate the percentage yield of methyl 3-nitrobenzoate, assuming that an excess of nitric and sulfuric acids was present.

(3)



(d) One reason for the low yield in this experiment is that methyl 2-nitrobenzoate is also formed. This compound dissolves in ethanol and would be present in the ethanol washings from **step 3**. Methyl 2-nitrobenzoate and methyl 3-nitrobenzoate are both pale yellow.

(i) Describe how to make a chromatogram with the ethanol washings from **step 3** in order to separate methyl 2-nitrobenzoate and methyl 3-nitrobenzoate. The chromatogram can be made on a plate covered with a layer of silica, and you may assume that a suitable solvent is available.

(4)

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(ii) How would you improve the chromatogram to confirm that both methyl 2-nitrobenzoate and methyl 3-nitrobenzoate are present in the washings? You may show this on a diagram if you prefer.

(1)

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(e) The table below gives data about the solubility of methyl 3-nitrobenzoate in two solvents. This data may be used to select the best solvent for recrystallization.

Temperature / °C	Solubility of methyl 3-nitrobenzoate / g per 100 g solvent	
	Solvent 1	Solvent 2
10	6.0	2.0
70	11.0	9.5

(i) Explain why using Solvent 1, rather than Solvent 2, would lead to a lower yield of recrystallized methyl 3-nitrobenzoate.

(1)

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(ii) 50 g of Solvent 2 was saturated with methyl 3-nitrobenzoate at 70 °C, and the solution was then cooled to 10 °C. Calculate the mass of methyl 3-nitrobenzoate crystals which would be obtained.

(1)

(f) What method, other than spectroscopy or chromatography, could be used to assess the purity of the methyl 3-nitrobenzoate? How would the result of the experiment indicate if it was pure?

(2)

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(Total for Question 4 = 17 marks)

TOTAL FOR PAPER = 50 MARKS



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