

Examiners' Report
June 2012

GCE Chemistry 6CH08 01

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June 2012

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Introduction

The paper contained many questions which were accessible to all candidates, but also presented challenges by asking candidates for explanations as well as descriptions of some laboratory techniques. Knowledge of chromatography and understanding of recrystallisation was often poor. It was clear in several questions that even if candidates know what to do, they may not understand what reaction is occurring or the reasons behind the procedures.

Candidates were well prepared to do calculations on percentage yields and titrations. However, it is evident that many have little appreciation of significant figures. Volumes were sometimes quoted with three decimal places, which were mathematically correct but could never have been measured by any measuring cylinder. Numbers were often rounded at every stage, and in a multi-stage calculation this leads to an answer which may be several percentage points different from the true one.

Question 1(a)

Most candidates scored this mark. They were told that the colour of the two solutions was similar, and only a few gave two different colours.

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?

green (Cr³⁺ green, Ni²⁺ emerald green) (1)

(b) Tests were carried out on a dilute aqueous solution of chromium(III) chloride



ResultsPlus Examiner Comments

This candidate was clearly familiar with the appearance of the two solutions, and one answer was an expanded version of the other.



ResultsPlus Examiner Tip

If only one answer is needed don't give two, just in case one is wrong!

Question 1(b)

This question was high scoring. Most candidates knew that chromium(III) hydroxide dissolves in excess sodium hydroxide. The errors were mainly in realising that the precipitate which forms with aqueous ammonia is also chromium(III) hydroxide, and in writing the formulae needed.

(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 <i>Green colour.</i>	(1)
(ii)	Add dilute sodium hydroxide to the mixture made in (i), until the sodium hydroxide is present in excess.	<i>The precipitate dissolves in excess.</i>	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.	<i>A precipitate is formed which is soluble green.</i>	The substance containing chromium which is observed on adding the ammonia is $[\text{Cr}(\text{NH}_3)]^{2+}$ $[\text{Cr}(\text{NH}_3)_3]^{2+}$	(2)
(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 $[\text{Cr}(\text{H}_2\text{O})_2(\text{NH}_3)_4]^{2+}$	(1)
(v)	Warm another sample of the chromium(III) chloride solution with alkaline hydrogen peroxide solution, which acts as an oxidizing agent.	A yellow solution forms.	The formula of the ion causing the yellow colour is $[\text{Cr}(\text{O})(\text{H}_2\text{O})]^-$ $[\text{Cr}(\text{O})]^-$	(1)
	Add sulfuric acid to the resulting mixture.	The solution goes orange when sulfuric acid is added.	The ion causing the orange colour is dichromate(VI), $\text{Cr}_2\text{O}_7^{2-}$.	



ResultsPlus Examiner Comments

This candidate knew what would be observed in the experiment but not the species causing the changes.



ResultsPlus Examiner Tip

The inferences column is for deductions about the reactions which are occurring. Saying that the precipitate is green in the first test is an observation, and the name or formula of the precipitate was required.

(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 $\text{Cr}(\text{OH})_3$	(1)
(ii)	Add dilute sodium hydroxide to the mixture made in (i), until the sodium hydroxide is present in excess.	<i>A solid dissolves, green solution is formed</i>	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.	<i>green precipitate forms</i>	The substance containing chromium which is observed on adding the ammonia is $[\text{Cr}(\text{H}_2\text{O})_4(\text{OH})_2]$	(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 $[\text{Cr}(\text{NH}_3)_6]^{3+}$	(1)
(v)	Warm another sample of the chromium(III) chloride solution with alkaline hydrogen peroxide solution, which acts as an oxidizing agent.	A yellow solution forms.	The formula of the ion causing the yellow colour is CrO_3^- $\text{Cr}_2\text{O}_7^{2-}$	(1)
	Add sulfuric acid to the resulting mixture.	The solution goes orange when sulfuric acid is added.	The ion causing the orange colour is dichromate(VI), $\text{Cr}_2\text{O}_7^{2-}$.	



ResultsPlus Examiner Comments

Again this candidate knew what would be seen but could not explain the observations correctly.



ResultsPlus Examiner Tip

The precipitate formed in (i) is the same as the precipitate formed in (iii), so you need to be confident enough to put the same answer twice. This candidate wrote a formula containing chromium(II) ions in (iii).

To work out the charge on a complex ion like the one in (iv), the charges on the metal and the ligands have to be added together.

Question 1(c)

Only a few candidates missed the instruction to ignore any colour changes. Many realised that in the experiment with nickel chloride, the hydroxide precipitate would not dissolve in excess sodium hydroxide. However many answers were expressed badly. Some answers discussed whether chromium hydroxide and nickel hydroxide were amphoteric. Even if the comments made on them were correct, they could not score the mark unless they stated how the reaction with sodium hydroxide differed with the nickel salt.

(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)

The ~~precipitate~~ hydroxide precipitate of nickel(II) chloride is insoluble in excess sodium hydroxide solution and hence does not dissolve as it is not amphoteric.

(Total for Question 1 = 8 marks)



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Examiner Comments

This answer gives the required information about the different behaviour of the nickel salt in the first two tests.

(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)

Nickel (II) chloride would not dissolve in excess sodium hydroxide.



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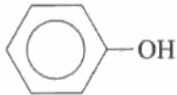
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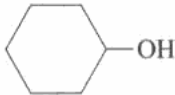
Nickel forms a precipitate of nickel(II) hydroxide in test (i) and it had to be clear that it is this hydroxide which does not dissolve in excess sodium hydroxide.

Question 2(a)

Most candidates knew that the bromine water would be decolorised. For the second observation there were several possibilities. An antiseptic smell was not allowed as the initial reactants are themselves strong smelling.

2 This question is about some reactions of phenol and cyclohexanol. $\text{CH}_2\text{OH} + \text{H}_2\text{SO}_4$

 phenol

 cyclohexanol H_2SO_4
 H^+ SO_4^{2-}

(a) Give **two** observations you would make when bromine water is added, drop by drop, to an aqueous solution of phenol. (2)

- A white precipitate is formed
- ~~effervescence~~ effervescence, Hydrogen produced.



ResultsPlus Examiner Comments

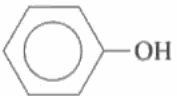
This answer scored one mark for the formation of the precipitate. Some hydrogen bromide would be produced as steamy fumes, but there would not be effervescence.

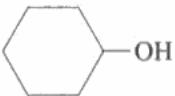


ResultsPlus Examiner Tip

Just because a gas is formed in a reaction does not mean that effervescence will be seen. Rapid bubbling may occur when an acid reacts with a solid, but not in a situation like this where two liquids are reacting to make a soluble gas.

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)

Br^- will be attacked by H^+ and substitution occurs. Thus, bromine water will decolourised from brown to colourless.



ResultsPlus Examiner Comments

This answer scores the mark for decolorisation of bromine. The question asked for observations, and the comments on how the decolorisation occurred were ignored.



ResultsPlus Examiner Tip

When you are asked for experimental observations it is not necessary to try to give a mechanism.

Question 2(b)

Most candidates knew the colour change which occurs when potassium dichromate(VI) oxidizes an alcohol. However, the formula of the product of the oxidation was often incorrect. As cyclohexanol is a secondary alcohol the oxidation product is a ketone. Many candidates thought the ring would break, and showed straight chain aldehydes or acids. Others added an extra carbon to the product, showing -CHO or -COOH attached to the ring. Sometimes the ring was shown with a 5-bonded carbon with an aldehyde or carboxylic acid group on it. It was disappointing at this level to see answers with potassium or chromium atoms attached to the ring. Those who realised that cyclohexanone formed usually gave a correct observation for the test with 2,4-dinitrophenylhydrazine, but the resulting negative test with Tollens' reagent was answered less well.

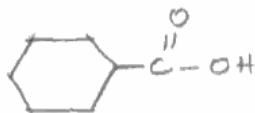
(b) (i) What is observed when cyclohexanol is warmed with a mixture of aqueous potassium dichromate(VI) and sulfuric acid?

(1)

There is a colour change; from pink to colourless

(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

A red/orange/yellow crystalline solid is formed

Tollens' reagent

No reaction



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Examiner Comments

This is one of the few examples with an incorrect colour change in (i). The formula with an extra carbon atom in (ii) was given frequently, and suggests that the candidate did not know the type of alcohol which was oxidized.

Carboxylic acids do not give a positive test with 2,4-dinitrophenylhydrazine, but lack of reaction with Tollens' reagent was allowed.



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Examiner Tip

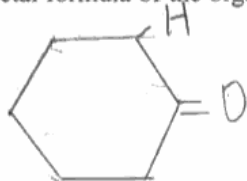
The number of carbon atoms in the product should be the same as at the start.

In cyclohexanol, the carbon atom carrying the -OH group is attached to two other carbons, so the alcohol is secondary. You need to decide on the type of alcohol before you can work out the oxidation products.

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

Water is formed colour change from orange to green (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

Orange precipitate formed

Tollens' reagent

Silver forms



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Examiner Comments

The formula given in (ii) is not correct. If hydrogen atoms are shown there should be two on each carbon. A ketone has been drawn, though the answer to (iii) suggests that the candidate thinks it is an aldehyde.



ResultsPlus

Examiner Tip

In a ring structure with no C=C bonds there are two hydrogen atoms attached to each carbon, except where other functional groups are present.

Question 2(c)(i)

Many candidates suggested using an indicator to show that the fumes were hydrogen chloride. This would only show whether the fumes are acidic, and is not a specific enough test for a hydrogen halide.

If the fumes are tested with ammonia, the method used cannot be bubbling through ammonia solution as this will not give a visible change. Testing hydrogen chloride with the fumes from concentrated ammonia gives a white smoke consisting of solid particles of ammonium chloride.

If the fumes are bubbled through silver nitrate solution there would be a visible change as the product, silver chloride, is insoluble, so this answer was allowed.

(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)

Handwritten chemical reaction showing phenol reacting with ethanoyl chloride to form phenyl ethanoate and HCl. The student has drawn the structures and labeled the HCl as 'steamy fumes'. There is a handwritten note 'steamy fumes is due to HCl' with an arrow pointing to the HCl product.



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Examiner Comments

This candidate used formulae to deduce that hydrogen chloride was formed, but a laboratory test was required.

(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)

Introduce NH_3
~~into~~ React the gas to the steamy fumes. Ring of white smoke forms.



ResultsPlus

Examiner Comments

This answer is correct and gained both marks.

Question 2(c)(ii)

This question was answered well. A few candidates did not read the question carefully and did not give both the bond and group causing the absorptions. Only a very small number thought that the C=O absorption was due to a ketone. There were more errors in the identifying the cause of peak Y, suggesting that candidates had not thought about the structure of the ester.

Identify the bond and group which cause each of the absorptions X and Y. (2)

X esters

Y ethanoates



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Examiner Comments

This candidate scored one mark for giving two correct groups, but the bonds should also have been specified.

Identify the bond and group which cause each of the absorptions X and Y. (2)

X C=O (bond) Esters (group)

Y C-O (bond) benzoates (group)



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Examiner Comments

This candidate scored the first mark only.



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Examiner Tip

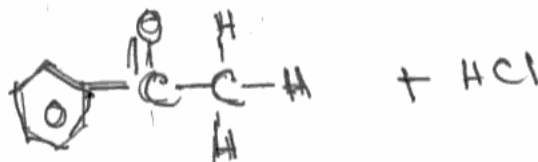
Benzoates are esters of benzoic acid. They form when benzoic acid or benzoyl chloride reacts with an alcohol. In this question phenol is acting like an alcohol.

Question 2(c)(iii)

There were many correct answers here. When marks were lost it was often due to lack of care rather than lack of knowledge of the formula of an ester.

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

(1)



ResultsPlus
Examiner Comments

This candidate has left out the second oxygen atom.

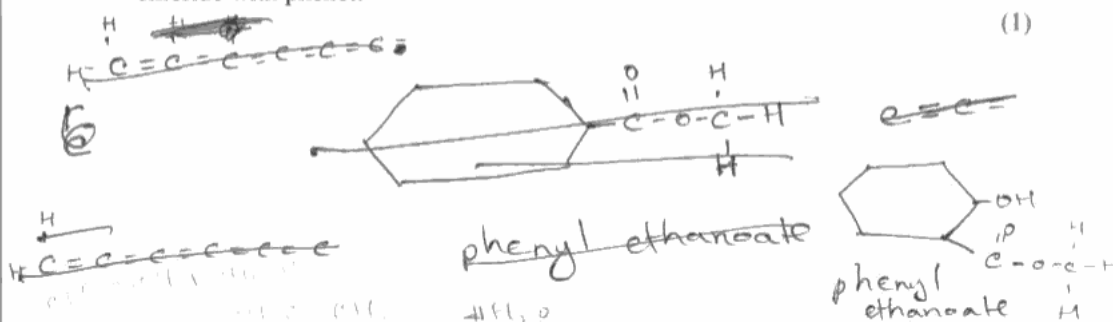


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Examiner Tip

The ester functional group contains two oxygen atoms. When you have to write the formula of an ester, start with a functional group and then decide what is on either side of it.

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

(1)



(Total for Question 2 = 11 marks)



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Examiner Comments

This candidate has not used the information that phenol is acting as an alcohol, which would lead to a reaction of the hydroxyl group with the ethanoyl chloride. The circle representing the delocalised ring is also missing.



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Examiner Tip

The question often contains useful information so read it carefully. Phenol is acting as an alcohol in this reaction so the hydroxyl group must be involved.

Question 3(a)

The question paper gave the equation for the reaction without state symbols, to test whether the candidates knew the appearance of the products. Many thought that there would be effervescence from the sulfur dioxide. Others gave secondary tests for sulfur dioxide, such as the effect on filter paper dipped in potassium dichromate(VI) solution. A direct observation rather than a test is expected in questions like this.

An alternative answer was to refer to the precipitate, or cloudiness, which appears when sulfur forms.

- (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)

Effervescence observed - A gas which turns acidified $\text{K}_2\text{Cr}_2\text{O}_7$ from orange to green is evolved.



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Examiner Comments

Sulfur dioxide would be detected by its choking smell. This observation, rather than a further test, is expected.

- (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)

A gas which has the rotten egg smell is liberated



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Examiner Comments

This candidate has confused sulfur dioxide with hydrogen sulfide.



ResultsPlus
Examiner Tip

Hydrogen sulfide is the gas which smells of rotten eggs. Sulfur dioxide has a choking smell.

Question 3(b)

Most candidates knew that copper(II) nitrate is blue.

Many correctly suggested in part (ii) that effervescence would be seen, but a variety of other answers appeared.

(b) (i) What colour is the diluted solution containing copper(II) nitrate? (1)

colourless

(ii) What would you observe in Step 2, before the formation of the copper(II) carbonate precipitate, when the sodium carbonate was added? (1)

~~A white~~ Quick effervescence would take place before the
~~form~~ The sodium carbonate dissolves, giving off a gas (CO₂)



ResultsPlus

Examiner Comments

The observation here should have been that bubbling or effervescence was seen. Naming the gas is not an observation.

(b) (i) What colour is the diluted solution containing copper(II) nitrate? (1)

Blue colour.

(ii) What would you observe in Step 2, before the formation of the copper(II) carbonate precipitate, when the sodium carbonate was added? (1)

~~A blue colour solution can be observed~~
Colourless gas which turns limewater milky can be seen.
$$\text{Cu}(\text{NO}_3)_2 + \text{Na}_2\text{CO}_3 \longrightarrow \text{CuCO}_3 + \text{Na}^+\text{NO}_3^-$$



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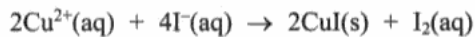
Examiner Comments

When the question asks for an observation, marks will not be given for a secondary test on identifying the product.

Question 3(c)(i)

The most common error was to give the wrong oxidation state of the copper or to omit it. Some candidates put the oxidation state at the end of the formula instead of after the copper.

(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)

~~Copper Iodide~~ ^(I) Copper(II) iodide



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Examiner Comments

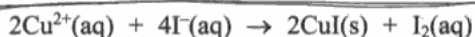
The correct number to include here is I.



ResultsPlus
Examiner Tip

Transition metals have more than one oxidation state, and the names of their compounds must show the oxidation state involved. Copper forms more than one compound with iodine so the number (I) must be included here.

(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)

Copper (I) iodide



ResultsPlus
Examiner Comments

In some answers the oxidation number looked more like a letter than a number. This suggests that candidates do not understand the meaning of the numbers included in the formula. This answer could be interpreted as a letter or a number and was allowed.



ResultsPlus
Examiner Tip

The number in the name shows the oxidation state of copper, and should be written as a Roman numeral. The letter "i" would be incorrect here.

Question 3(c)(ii)

CuI contains ions from two elements, and the question asked for the electronic configuration of the relevant ion to be considered. Despite this, there were many answers which did not make clear that they were discussing the copper(I) ion.

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

Because $3d^{10}$ orbital is completely filled, so there is no vacant electron to be excited.



ResultsPlus
Examiner Comments

This answer does not specify which ion is being discussed.



ResultsPlus
Examiner Tip

Read the question carefully and do what it asks. Correct information which does not answer the question will not score.

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

$Cu^{+} \rightarrow 1s^2 2s^2 2p^6 3s^2 3p^6 3d^{10}$

As it has a completely filled d-orbital, it is not a transition metal ion and is not coloured.



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Examiner Comments

This answer clearly refers to the copper(I) ion.

Question 3(d)

In this question many candidates showed that they had little understanding of how to round numbers appropriately or of significant figures.

The answer to part (i) was 0.00305. Some candidates rounded this to 0.0031. They then divided by 2 to find the number of moles of iodine reacting, getting 0.00155 which they rounded to 0.0016. This was doubled to find the number of moles of copper(II) ions, giving the answer 0.0032. The chemical principles were correct, but the early rounding led to the wrong answer. Candidates should quote intermediate numbers to a sensible number of significant figures, usually 3 or 4, but keep the full value in their calculator to use in later stages of the calculation.

An error in chemistry which was seen frequently was to use the ratio of moles of thiosulfate to iodide to calculate the number of moles of copper.

In part (iii) most candidates knew that they had to multiply the number of moles by the relative atomic mass in order to find the mass of copper, but many forgot to calculate the mass in the original solution, and only calculated the mass in the titrated sample. Again, candidates rounded their values inappropriately, leading to a wide range of answers. On this occasion, rounding which was early but not incorrect was not penalised as long as the chemistry was correct, but in real life situations this inappropriate rounding of numbers could lead to problems.

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



Results:

Mass of coins	2.10 g
Mean (average) volume of $0.125 \text{ mol dm}^{-3}$ sodium thiosulfate used in titration	24.40 cm^3

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

$$\begin{aligned} \text{no. of moles of sodium thiosulfate} &= 0.125 \text{ mol dm}^{-3} \times (24.40 \div 1000) \text{ dm}^3 \\ &= 3.05 \times 10^{-3} \text{ mol} // \end{aligned}$$

(ii) Calculate the number of moles of Cu^{2+} in the 25 cm^3 samples used for the titration.

$$\begin{aligned} \text{no. of moles of iodide ion} &= 1.0 \text{ mol dm}^{-3} \times (10 \div 1000) \text{ dm}^3 \quad (2) \\ &= 0.01 \text{ mol} \end{aligned}$$

so,

$$\begin{aligned} 4 \text{ mol of } \text{I}^- \text{ reacts with } 2 \text{ mol of } \text{Cu}^{2+} \\ \therefore 0.01 \text{ mol of } \text{I}^- \text{ reacts with } \frac{2}{4} \times 0.01 \text{ mol of } \text{Cu}^{2+} \\ = 5 \times 10^{-3} \text{ mol of } \text{Cu}^{2+} \end{aligned}$$

$$\begin{aligned} \therefore \text{In } 25 \text{ cm}^3 &= 5 \times 10^{-3} \times 2.5 \\ &= 0.0125 \text{ mol of } \text{Cu}^{2+} // \end{aligned}$$

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

$$\begin{aligned} \text{mass of copper} &= \frac{2.10}{0.0125} = 2.10 \times 0.0125 \\ &= 168 \text{ g} // \quad = 0.02625 \text{ g} // \end{aligned}$$

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

$$\begin{aligned} \% \text{ of copper} &= \frac{168}{2.10} \times 100\% \\ \% \text{ of copper} &= \frac{0.02625}{2.10} \times 100\% \\ &= 1.25\% // \end{aligned}$$



ResultsPlus Examiner Comments

This candidate did not find the number of moles of iodine reacting with the sodium thiosulfate and hence calculated the number of moles of copper wrongly. There was also an error in the method used to calculate the mass of copper.

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



Results:

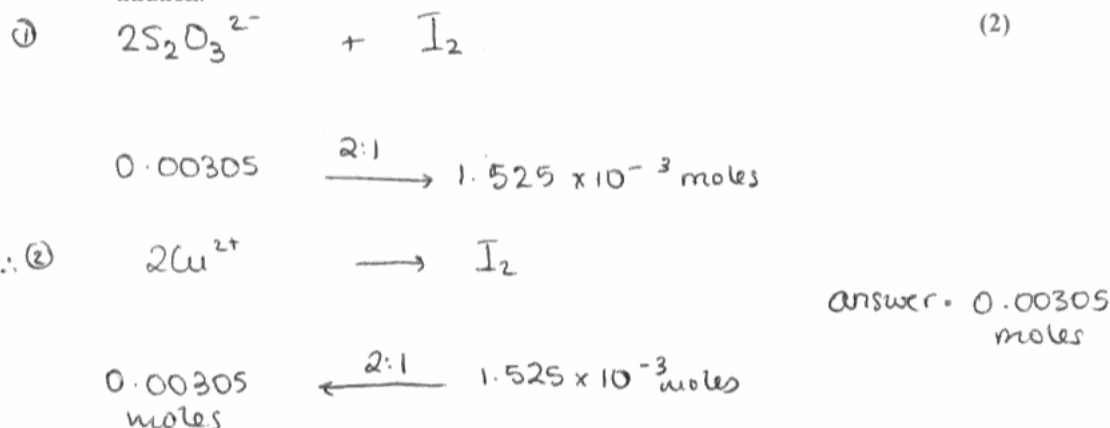
Mass of coins	2.10 g
Mean (average) volume of $0.125 \text{ mol dm}^{-3}$ sodium thiosulfate used in titration	24.40 cm^3

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

$$\begin{aligned} &0.125 \text{ mol dm}^{-3} \\ &\quad \downarrow \div 1000 \text{ cm}^3 \\ &\quad \times 24.40 \text{ cm}^3 \end{aligned} \quad \text{answer} = 0.00305 \text{ moles}$$

0.00305 moles

(ii) Calculate the number of moles of Cu^{2+} in the 25 cm^3 samples used for the titration. (2)



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

Cu^{2+} (2)

1.93675 g

$\uparrow \times 10$
 $\times 63.5 \text{ g mol}^{-1}$

0.00305 moles

Mass of $\text{Cu}^{2+} = 1.94 \text{ g}$

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

$$\frac{1.94}{2.10} \times 100 = 92.2\%$$



ResultsPlus
Examiner Comments

This is an example of an answer which is well set out.

Question 3(e)

The error in question (i) was stated to be the total error. However, many candidates thought it had to be doubled.

When measuring a volume of solution with a burette two readings are taken, so the total error is twice the error in each reading. In (ii) the error was given as the error in the mean titre, not in any one reading. This meant that again no doubling was needed.

- (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.

$$\begin{aligned}\text{Percentage error} &= \frac{0.01 \times 2}{2.10} \times 100\% \\ &= 0.952\%\end{aligned}\quad (1)$$

- (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.

$$\begin{aligned}\text{Percentage error in titration} &= \frac{0.10 \times 2}{24.40} \times 100\% \\ &= 0.82\%\end{aligned}\quad (1)$$

\therefore % error in titration < % error in balance reading \times shown



ResultsPlus

Examiner Comments

This candidate has doubled the errors each time.



ResultsPlus

Examiner Tip

A titre is calculated from the difference in two burette readings, so the error in the titre is double the error in each reading. If the error in a burette reading is $\pm 0.05 \text{ cm}^3$, then the error in the titre is 0.10 cm^3 .

- (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)

$$\begin{aligned} \text{percentage error} &= \frac{\pm 0.01}{2.10} \times 100 \% \\ &= \frac{\pm 0.01}{2.10} \times 100 \% = \pm 0.4762 \% \end{aligned}$$

- (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.

$$\begin{aligned} \text{percentage error of titration reading} &= \frac{2 \times \pm 0.10}{24.40} \times 100 \% && \text{percentage error in titration reading}^{(1)} \\ &= \pm 0.8197 \% && = \frac{\pm 0.10}{24.40} \times 100 \% \\ & && = \pm 0.4098 \% \end{aligned}$$

$\pm 0.8197 \%$ is less than $\pm 2.1 \%$ $\pm 0.4098 \% < \pm 0.4762 \%$,
percentage error in titration reading is less



ResultsPlus
Examiner Comments

This was correct and scored both marks.

Question 3(f)

Many candidates gave only one colour instead of giving the colour change. Others gave the colour change which would be seen if starch was used.

(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)

pale yellow colour.



ResultsPlus Examiner Comments

The change to colourless was needed as well as the original colour.



ResultsPlus Examiner Tip

When a colour change is asked for, the starting and final colours must both be given.

(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)

Blue black to colourless.



ResultsPlus Examiner Comments

This candidate missed the point that the titration was carried out without the use of starch.

Question 4(a)

Many answers suggested that safety goggles and laboratory coats were needed, even though the question stated that these were already in use.

A correct answer to part (i) was given more often than to part (ii). Concentrated acids were usually known to be corrosive or to burn skin, but then use of a fume cupboard was incorrectly suggested as a precaution. Many candidates thought that ethanol was toxic, and again use of a fume cupboard was one of the common suggested precautions. Other candidates knew that it was flammable, but then thought this meant that it could not be heated.

(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)

Both concentrated nitric and sulfuric acid are corrosive, gloves should be used.

(ii) Carrying out the recrystallization using ethanol. (1)

Ethanol is flammable, use ice-cold ethanol and keep the mixture away from flames or heat.



ResultsPlus Examiner Comments

The first part of the answer is correct, but the second was not allowed.

Many candidates seemed to think that flammable liquids cannot be heated, in which case ethanol could not be used for recrystallisation.



ResultsPlus Examiner Tip

Flammable liquids can be heated if suitable precautions are taken, such as the use of an electric heater. They should be kept away from naked flames.

(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)

1) A limited distance should be maintained as the acids are the most toxic ones.

(ii) Carrying out the recrystallization using ethanol. (1)

2) Ethanol should be poured slowly and hands should be in safe zone.



ResultsPlus Examiner Comments

These answers are too general. Specific hazards and precautions were required.

Question 4(b)

The purpose of adding sulfuric acid in nitration is to produce an electrophile. If the formula of the electrophile was given it had to be correct, and many candidates omitted the charge or gave it incorrectly. Saying that the sulfuric acid was a catalyst was not enough here.

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

(1)

To produce NO_2 ~~be the~~.



ResultsPlus

Examiner Comments

The charge on the nitronium ion should have been given here.



ResultsPlus

Examiner Tip

Benzene is attacked by electrophiles, so the sulfuric acid is needed in nitration to produce a positively charged ion.

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

(1)

Sulfuric acid act as an acid, nitric acid as a base.
To be able to neutralise the ~~acid~~ ^{nitric acid} to obtain NO_2^+



ResultsPlus

Examiner Comments

This answer is confused. Sulfuric acid does protonate nitric acid in the first stage of making the electrophile but it is incorrect to say that it neutralises it.

Question 4(c)

Most candidates knew how to carry out the calculations in this question, but in part (iii) a high proportion of candidates calculated the molar mass of methyl 3-nitrobenzoate incorrectly.

Many answers were rounded incorrectly, or quoted to an inappropriate number of significant figures. Credit is given for consequential correct working after a wrong answer, but this cannot be done if the candidate's working is not clear. This is why it is important to show the answer clearly at each stage.

(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)

$$\frac{0.5}{136} = 3.68 \times 10^{-3} \\ = 0.003 \text{ moles}$$

(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)

$$d = \frac{m}{V} \quad 5 : 1.09 = 4.6 \text{ cm}^3$$

(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.

$$\frac{3.4}{5.0} \times 100$$

$M_r(\text{methyl 3-nitrobenzoate}) = 136 + 32 + 14 = 182$ (3)

ratio: 1:1

$$0.03 \times 182 = 5.46 \\ = 5.5$$

$$\frac{3.4}{5.46} \times 100$$

$$\frac{3.4}{5.5} \times 100 = 61.82\% \\ = 61.8\% \\ \approx 62\%$$

ResultsPlus

Examiner Comments

In part (i) the answer should not have been rounded down, and even if rounded up to 0.004 it would not have been accepted, as one significant figure is not appropriate when the mass of methyl benzoate is given to two figures.

Rounding the volume in part (ii) was sensible.

The molar mass in part (iii) should have been 181.



ResultsPlus

Examiner Tip

The number of significant figures in an answer should be the same as the number in the data you are using. One significant figure is usually not enough.

When you calculate the molar mass of a compound containing a benzene ring don't forget that every time a group is substituted into the ring one hydrogen atom is removed from it.

(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} .

$$\text{moles} = \frac{5}{136} = 0.036 \text{ moles} \quad (1)$$

(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} .

$$\text{Volume} = \frac{5}{1.09} = 4.58 \text{ cm}^3 \quad (1)$$

(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.

$$\% \text{ age yield} = \frac{3.4}{5} \times 100 = 68\%$$

$$\text{moles} = \frac{3.4}{101} = 0.018 \text{ moles}$$

$$\% \text{ age yield} = \frac{0.018}{0.036} \times 100 = \boxed{52.18\%}$$



ResultsPlus

Examiner Comments

In part (i) the value of 0.0368 has been rounded wrongly, and so has the volume of 4.587 in part (ii).

A calculator display shows the number of moles in part (iii) as 0.00187845. This appears to have been used to calculate the percentage yield, as the value quoted of 0.018 would not produce the answer given by the candidate.



ResultsPlus

Examiner Tip

Don't round numbers in a calculation until the last stage, and when you do round numbers be careful to include an appropriate number of significant figures and round them correctly.

Question 4(d)

There were few good answers to this question, and it was clear that many candidates did not know how to make a chromatogram. Many techniques other than chromatography were described. These included fractional distillation, recrystallisation and elaborate filtration methods.

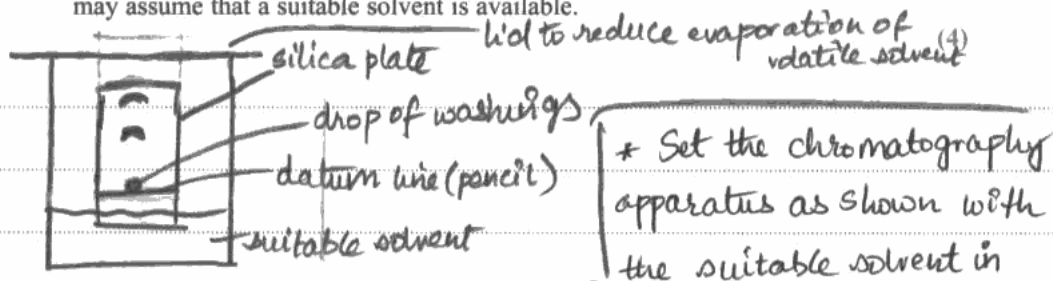
A chromatogram of the ethanol washings can be made on a silica plate, but answers describing paper chromatography were accepted. However, answers which just gave the theory of gas-liquid chromatography, HPLC or any other type of chromatography without practical detail did not fulfil the requirement to describe how to make a chromatogram.

Many candidates did not understand that in part (i) the aim was to separate methyl 2-nitrobenzoate and methyl 3-nitrobenzoate which are in the ethanol washings. A common suggestion was that methyl 2-nitrobenzoate and methyl 3-nitrobenzoate should be dissolved in a suitable solvent, and a silica plate placed in this solution. Answers which suggested spotting the sample on a plate often then said that the plate should be submerged in the solvent.

Candidates who did know the technique often suggested that the spots could be identified from their R_f values, and this was accepted as an alternative to putting spots of known compounds on the chromatogram for comparison. Running a two-way chromatogram using a different eluent is a technique used to improve separation, but it would not be useful here to confirm that the two methyl nitrobenzoates were present.

(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.

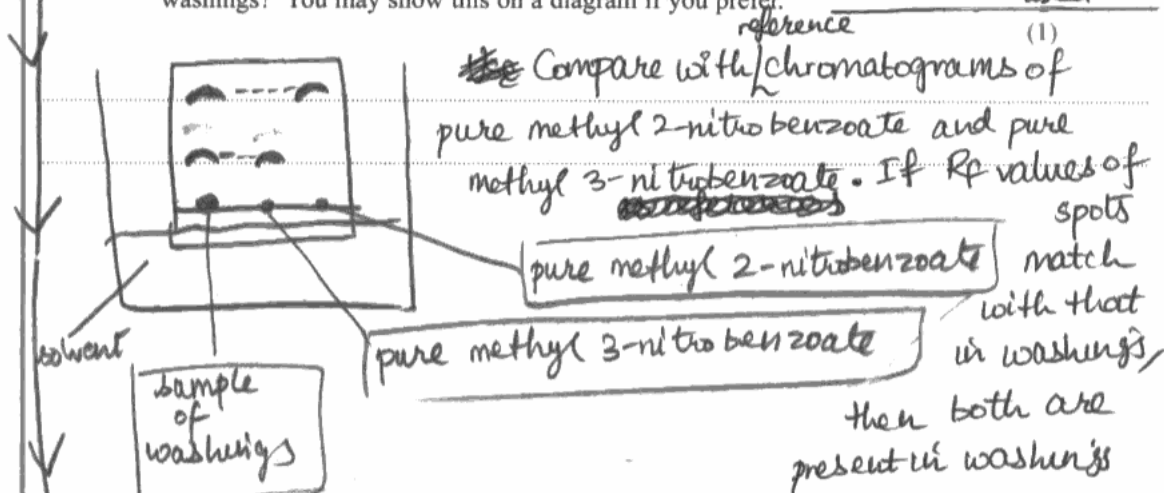
(i) Describe how to make a chromatogram with the ethanol washings from **step 3** in order to separate methyl 2-nitrobenzoate and methyl 3-nitrobenzoate. The chromatogram can be made on a plate covered with a layer of silica, and you may assume that a suitable solvent is available.



* a beaker, below the starting line on the silica plate (drawn in pencil)
 * Using a pipette, apply a few drops of the washings in step 3 on the starting line ABOVE the solvent level. Cover the beaker with a lid to reduce evaporation of the solvent. Leave the apparatus to stand for a few hours, not letting the solvent reach the top of the plate.

The drop should separate into two spots indicating presence of 2 chemicals (namely methyl 2-nitrobenzoate and methyl 3-nitrobenzoate)

(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.



Precaution: plate must not be touched with fingers
 - if ~~the~~ chromatogram is difficult to see, a LOCATING agent can be used to colour spots using oven if necessary



ResultsPlus

Examiner Comments

This is an example of a good answer, probably based on the candidate's knowledge of making a chromatogram to separate amino acids.

(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)

This can be done via paper chromatography. The paper is immersed in the solution containing the methyl 2-nitrobenzoate and methyl 3-nitrobenzoate. The molecules of the two compounds will have different R_f values and it can be compared to an existing data to determine which is the heavier or lighter compound. The paper is to be dipped into ninhydrin in order to be able to locate the spots.

(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)

The paper is then rotated 90° and ~~re~~ re-immersed into the solution of methyl 2-nitrobenzoate and methyl 3-nitrobenzoate.



ResultsPlus

Examiner Comments

The procedure used to make a chromatogram is not clear here. The original position of the sample is not mentioned, and submerging it in solvent would wash it off the paper. The candidate does not understand that the components of a solution are separated by using another solvent.



ResultsPlus

Examiner Tip

Chromatography separates mixtures because each component moves a different distance when a solvent runs through the mixture.

Two way chromatography is used if one solvent does not give good separation of the mixture.

Question 4(e)

Candidates may know how to carry out recrystallisation, but this question showed that many did not understand the theory behind it.

Many candidates thought that the table showed how much product was made, and did not understand the terms solvent and saturated. Often candidates said that Solvent 1 would produce a lower yield because methyl 3-nitrobenzoate was more soluble in it at both temperatures. The compound being recrystallised should be very soluble at higher temperatures, but should have a low solubility at lower temperatures so that it does not remain in solution. Some answers made little sense as they referred to the solubility of the solvent, rather than solubility in the solvent.

The numbers in the calculation were simple, but despite the information that 100g of solvent 2 could only dissolve 9.5g of methyl 3-nitrobenzoate there were answers saying that several hundred grams could be recrystallised from 50g solvent.

(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)

Solvent 1, even at 10°C has a solubility of 6 g per 100 g. This means that some of the product will be left in the organic solvent when it cools. Solvent 2 has only a solubility of 2 g per 100 g solvent at 10°C.

(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)

100g → 9.5g
50g → 4.75g



ResultsPlus Examiner Comments

This candidate is making a clear contrast between solubility of methyl 3-nitrobenzoate in the two solvents at the lower temperature.

The answer to part (ii) is correct and scored the mark even though the method is not shown fully.



ResultsPlus Examiner Tip

To find the mass of crystals which would form, you need to calculate the mass which dissolves at the higher temperature, and then subtract the mass which remains in solution when the mixture is cooled.

- (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.

A large amount of methyl 3-nitrobenzoate is required to dissolve in 100g of Solvent 1 at 70°C which should be minimised because it should dissolve in minimum volume of hot solvent. (1)

- (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.

$$\frac{9.5 \text{ g}}{100 \text{ g}} \times 50 \text{ g} = 4.75 \text{ g} \quad (1)$$

$$\frac{2.0 \text{ g}}{100 \text{ g}} \times 4.75 \text{ g} = 0.095 \text{ g}$$

$$50 - (4.75 + 0.095) = \underline{\underline{45.16 \text{ g}}}$$



ResultsPlus

Examiner Comments

This answer does not consider how much methyl 3-nitrobenzoate remains dissolved when the solution is cooled.

The mass of crystals calculated in (ii) is greater than the mass in 100g of saturated solution.



ResultsPlus

Examiner Tip

If a substance remains dissolved as the temperature drops, then it will not form crystals which can be separated.

Make sure you know the meanings of the words solvent, solute and saturated.

Question 4(f)

A method of assessing purity other than the use of spectroscopy was asked for. However, a significant number of candidates suggested infrared spectroscopy or use of nmr. Many did suggest measurement of melting or boiling temperatures. In a pure sample these temperatures will be sharp, and saying that they would melt over a range of a few degrees was not allowed.

Saying that the temperature should be compared with a data book value is not enough. The melting temperature should match the value quoted in data.

(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)

Find the melting ^{temperature} ~~point~~ of the methyl-3-nitrobenzoate and compare it to the value in the ~~formula~~ ^{data} booklet. If it is pure the melting ~~pt~~ temperature will be ~~at~~ concordant with

(Total for Question 4 = 17 marks)

the data booklet value and it will be accurate (not in a range of values)

TOTAL FOR PAPER = 50 MARKS



ResultsPlus
Examiner Comments

This is an example of a good answer.

(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)

Heating methyl 3-nitrobenzoate.
If the melting temperature is over a range of (1°-2°C)
then it proves that the methyl 3-nitrobenzoate is pure.

(Total for Question 4 = 17 marks)

TOTAL FOR PAPER = 50 MARKS



ResultsPlus
Examiner Comments

Heating is not the same as measuring the temperature at which a substance melts, so on its own this would not have scored. The melting temperature should be sharp, not over a range.

Paper Summary

There were many instances in this paper where candidates did not read the question carefully.

- in 1(c) colours did not have to be given;
- in 2(a) two observations were required;
- in 2(c)(iii) both bonds and groups were required;
- in 3(c)(ii) the ion under discussion was not identified;
- in 3(e)(i) a total error was given in the question;
- in 3(f) a colour change was required;
- in 4(d) a description of how to make a chromatogram was required, not the theory of chromatography.

Candidates could have improved their performance by careful reading of the question, and then setting out their answers clearly. If the language in an answer is unclear, or if multiple alternatives are given, then marks will not be awarded.

It is important to round numbers correctly in calculations, and not to round numbers too much in intermediate stages of calculations.

Candidates should always try to understand the reasons behind experimental procedures.

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