Examiners' Report

June 2013

GCE Chemistry 6CH07 01

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# Introduction

The paper was accessible to all candidates, and there were very few occasions when a candidate did not attempt all the questions.

Candidates showed good knowledge of tests for inorganic compounds, but answers to questions on organic chemistry caused more difficulty. In general there was understanding of the principles behind calculations, but many candidates had very little understanding of how and when to round numbers, and of use of an appropriate number of significant figures.

The third question was the most challenging. The reasons behind practical procedures were often poorly understood.

## *Question 1 (a) (i)-(v)*

There were many high scores on this section of the paper.

Most candidates knew that the ion involved was potassium, but giving the formula as K without a charge was not allowed for the mark.

In (ii) many candidates saw the words "barium chloride" and "sulfate" and said that a white precipitate would be seen. They should have read the question more carefully as it said that sulfate ions were absent. Other candidates were perhaps thinking of reactions of sulfites, as they said that a white precipitate formed which dissolved on addition of acid.

In (iii), the addition of silver nitrate solution and formation of a yellow precipitate were well known as the test for an iodide ion, and many knew that this precipitate was insoluble in ammonia.

Those who thought that both iodide and sulfate ions were present produced some extraordinary suggestions for the formula of the salt.

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

1 Tests were carried out on compounds **X**, **Y** and **Z**. Complete the tables below.

(a) Compound **X** is a white, water-soluble solid.

| | Test | Observation | Inference (Name or formula) | |
|---|---|---|---|---|
| (i) | Flame test | Lilac flame | Potassium ion ($K^+$) | (1) |
| (ii) | To a solution of **X**, add barium chloride solution and acidify with hydrochloric acid | white ppt<br>Soluble | Sulfate ions absent | (1) |
| (iii) | To a solution of **X**, add dilute nitric acid followed by Silver Nitrate | yellow ppt | Iodide ions present | (2) |
| (iv) | Add concentrated aqueous ammonia solution to the mixture remaining from test (iii) | Insoluble | Confirms presence of iodide ions | (1) |

(v) The **formula** of **X** is: KI

(1)

**ResultsPlus**
**Examiner Comments**

No precipitate would form when barium chloride and hydrochloric acid are added to potassium iodide. This candidate may have been thinking about the reactions of sulfite ions.

**ResultsPlus**
**Examiner Tip**

Read the question carefully. If you see a white precipitate which dissolves, this suggests something different from no precipitate forming.

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

**1** Tests were carried out on compounds **X**, **Y** and **Z**. Complete the tables below.

(a) Compound **X** is a white, water-soluble solid.

| | Test | Observation | Inference (Name or formula) | |
|---|---|---|---|---|
| (i) | Flame test | Lilac flame | $K^+$ | (1) |
| (ii) | To a solution of **X**, add barium chloride solution and acidify with hydrochloric acid | A white ppt is formed | Sulfate ions absent | (1) |
| (iii) | To a solution of **X**, add dilute nitric acid followed by Silver nitrate | Forms yellow ppt | Iodide ions present | (2) |
| (iv) | Add concentrated aqueous ammonia solution to the mixture remaining from test (iii) | PPt doesn't dissolve | Confirms presence of iodide ions | (1) |

(v) The **formula** of **X** is: ~~K~~ $KISO_4$

(1)

**ResultsPlus**

**Examiner Comments**

Answers like this based on the presence, not the absence of sulfate ions, were quite common.

If the cation was identified wrongly, a mark could still be obtained for the formula of the salt which was consistent. However in this case the anion, sulfate, was said to be absent and an impossible formula was given.

**ResultsPlus**

**Examiner Tip**

Tests can be negative, and show which ions are not present.

## *Question 1 (b) (i)-(iii)*

This section also scored highly.

The majority of candidates correctly identified the positive ion as calcium, and used lime water to test the gas produced in (ii). The most common error was in (ii) where the observation was given as a white precipitate. Some candidates named calcium carbonate instead of giving the formula. The final mark was given if a correct formula was given, based on an earlier incorrect choice of cation.

(b) Compound **Y** is a white solid that is insoluble in water.

| | Test | Observation | Inference (Name or formula) | |
|---|---|---|---|---|
| (i) | Flame test | Yellow-red (brick red) flame | Calcium | (1) |
| (ii) | Add dilute hydrochloric acid to **Y** | The mixture fizzed and the solid Cacl2 | | |
| | Bubble the gas through Lime Water | It turned milky | $CO_2$ evolved | (2) |

(iii) The **formula** of **Y** is: Ca $CO_3$

(1)

**ResultsPlus**

**Examiner Comments**

The second column is for observations, so the answer $CaCl_2$ is not correct here.

**ResultsPlus**

**Examiner Tip**

Read through you answers to be sure they make sense. The first part of (ii) would only make sense if it said "the solid **formed** $CaCl_2$", and this would not be an observation.

(b) Compound **Y** is a white solid that is insoluble in water.

| | Test | Observation | Inference (Name or formula) | |
|---|---|---|---|---|
| (i) | Flame test | Yellow-red (brick red) flame | $Ca^{2+}$ | (1) |
| (ii) | Add dilute hydrochloric acid to **Y** | The mixture fizzed and the solid disappeared (melted) | | |
| | Bubble the gas through limewater | It turned milky | $CO_2$ evolved | (2) |

(iii) The **formula** of **Y** is: $CaCO_3$

(1)

**ResultsPlus**
**Examiner Comments**

The solid dissolves, and the word "disappear" was allowed as a description of what would be seen. However, as the candidate gave a second incorrect answer the mark was not given here.

**ResultsPlus**
**Examiner Tip**

Melting is not the same as dissolving, so choose your wording carefully.

## *Question 1 (c) (iii)*

The expected answers were a primary or secondary alcohol, as at this stage in the testing procedure the actual alcohol had not been identified. However candidates who returned to this question after doing (d) were credited for suggesting propan-1-ol or propan-2-ol.

| | | | | |
|---|---|---|---|---|
| (iii) | Warm **Z** with potassium dichromate(VI) solution and dilute sulfuric acid | Colour changes from orange to green | **Z** could be<br>alcohol<br>or<br>carboxylic acid | (2) |

**ResultsPlus**
**Examiner Comments**

The question said that Z forms a neutral solution, so carboxylic acid was not a possible answer for this reason, as well as the fact that carboxylic acids cannot be oxidised by acidified potassium dichromate(VI).

| | | | | |
|---|---|---|---|---|
| (iii) | Warm **Z** with potassium dichromate(VI) solution and dilute sulfuric acid | Colour changes from orange to green | **Z** could be<br>aldehyde<br>or<br>alcohol | (2) |

**ResultsPlus**
**Examiner Comments**

Aldehydes and primary and secondary alcohols can be oxidised by acidified potassium dichromate, so one mark was allowed for the combination given here, though the preceding test showed that an -OH group was present.

## *Question 1 (c) (i)-(ii)*

Careful reading of the question was required here. Some candidates missed the point that these were tests on an organic liquid, and suggested inorganic ions in their answers.

The test for bromine detects a carbon-carbon double bond, and simply referring to a double bond was not enough. The test shows the absence of a carbon-carbon double bond or alkene group, but does not prove what is present, so the inference that an alkane is present was not allowed.

As the solution in the test is neutral, the observation with phosphorus(V) chloride rules out a carboxylic acid and indicates an alcohol.

(c) **Z** is a colourless organic liquid with only one functional group. **Z** is completely miscible with water to form a neutral solution.

| | Test | Observation | Inference | |
|---|---|---|---|---|
| (i) | Add bromine water to **Z** | No colour change | Alkane/Not alkene | (1) |
| (ii) | Add solid phosphorus(V) chloride, $PCl_5$, to **Z** | Misty fumes (of hydrogen chloride) | Alcohol or alkanoic acid. | (1) |

**ResultsPlus**
**Examiner Comments**

The test shows that the liquid is not an alkene, but this does not mean it is an alkane, so this did not get the mark.

The solution is neutral, so alkanoic acid is not a possible choice.

**ResultsPlus**
**Examiner Tip**

Do not give two answers to a question because if one is wrong you will lose the mark for the correct one. This has happened here in both parts.

(c) **Z** is a colourless organic liquid with only one functional group. **Z** is completely miscible with water to form a neutral solution.

| | Test | Observation | Inference | |
|---|---|---|---|---|
| (i) | Add bromine water to **Z** | No colour change | Carbon double bond ~~alkene~~ absent. | (1) |
| (ii) | Add solid phosphorus(V) chloride, $PCl_5$, to **Z** | Misty fumes (of hydrogen chloride) | Presence of $OH^-$ ion | (1) |

**ResultsPlus**
**Examiner Comments**

A carbon-carbon double bond was not present, but as carbon forms double bonds to other atoms such as oxygen this was not allowed.

There are no hydroxide ions present.

**ResultsPlus**
**Examiner Tip**

Make sure you know the difference between a hydroxyl group and a hydroxide ion.

## *Question 1 (d) (i)*

Many candidates knew that, in principle, they had to find the number of moles of each atom in a sample of **Z**, and then find the simplest whole number ratio of these values to find the empirical formula. Unfortunately many candidates rounded their numbers too early. The 13.3 mol of hydrogen became 13. The 1.67 mol of oxygen became 2. This gave a formula of $C_5H_{13}O_2$. At this point the more thoughtful candidates might have realised that twelve hydrogen atoms saturate five carbon atoms, so this formula is impossible, but some then proceeded to draw structures of pentanol which did not even match the formula they had calculated.

The fact that early rounding of numbers leads to errors in calculations should be stressed to candidates.

However there were many correct calculations. Some lost the final mark by showing their answer as a structural formula rather than an empirical one, or as $(C_3H_8O)_n$.

(d) The composition by mass of **Z** is C 60.0%, H 13.3%, O 26.7%.

(i) Calculate the empirical formula of **Z**.

(2)

| | C | H | O |
|---|---|---|---|
| Mass | 60.0 | 13.3 | 26.7 |
| No of mol | 5 | 13.3 | 1.67 |
| simplest ratio | 5 | 12 | 2 |

Empirical formula of Z

$C_5H_{12}O_2$

**ResultsPlus**

**Examiner Comments**

This candidate has calculated the number of moles of carbon, hydrogen and oxygen correctly, but has then just rounded the numbers instead of finding the ratio.

**ResultsPlus**

**Examiner Tip**

Do not round numbers early in empirical formula calculations.

Compounds such as $C_6H_{12}$ and $C_6H_{14}$ have percentage compositions which are very similar, and early rounding would not show the difference.

(d) The composition by mass of **Z** is C 60.0%, H 13.3%, O 26.7%.

(i) Calculate the empirical formula of **Z**. **(2)**

| | C | H | O |
|---|---|---|---|
| $M_r$ | $\frac{0.6}{12}$ | $\frac{0.133}{1}$ | $\frac{0.267}{16}$ |
| | $\frac{0.05}{0.017}$ | $\frac{0.133}{0.017}$ | $\frac{0.017}{0.017}$ |
| ≈ | 3 | 8 | 1 |

$\Rightarrow$ Z : $C_3H_8O$

(ii) The molecular formula of Z is the same as its empirical formula. Give the

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**Examiner Comments**

This candidate is working in fractions rather than percentages. This, and other valid methods such as working out the factor needed to make the lowest number into a whole number, were allowed.

## *Question 1 (d) (ii)*

Many candidates realised at this point that they were dealing with propan-1-ol and propan-2-ol, and it was surprising that they did not return to (c)(iii) and amend their suggestions. It looked as if they did not realise that (c) and (d) were questions about the same compound.

Displayed formulae were usually given and only a few cases were seen where carbon was linked to the hydrogen in the OH group instead of the oxygen.

(d) The composition by mass of **Z** is C 60.0%, H 13.3%, O 26.7%.

(i) Calculate the empirical formula of **Z**.

(2)

C H O

$\frac{60}{12}$ $\frac{13.3}{1}$ $\frac{26.7}{16}$

5 : 13.3 1.67

2.9 : 7.9

3 : 8 : 1 $C_3H_8O$ //

(ii) The molecular formula of **Z** is the same as its empirical formula. Give the **displayed** formulae of the two possible isomers of **Z**.

(2)

$C_3H_7OH$

① $CH_3CH_2CH_2OH$ ② $(CH_3)_2CHOH$

**(Total for Question 1 = 18 marks)**

**ResultsPlus**
**Examiner Comments**

The question asked for displayed formulae, though two correct structural formulae were given one mark.

**ResultsPlus**
**Examiner Tip**

Learn the difference between empirical, molecular, displayed and structural formulae.

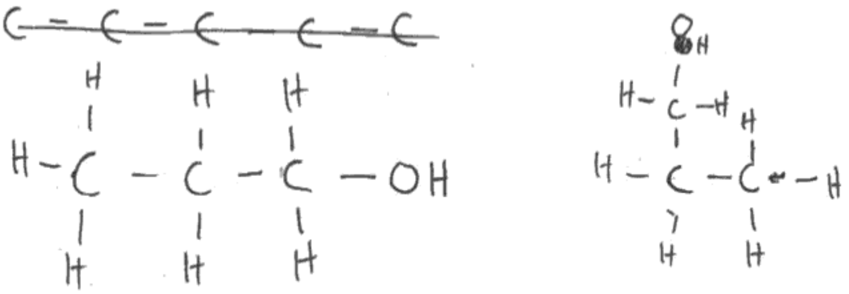
(d) The composition by mass of **Z** is C 60.0%, H 13.3%, O 26.7%.

(i) Calculate the empirical formula of **Z**.

(2)

(ii) The molecular formula of **Z** is the same as its empirical formula. Give the **displayed** formulae of the two possible isomers of **Z**.

(2)



**(Total for Question 1 = 18 marks)**

## ResultsPlus Examiner Comments

This shows two ways of drawing propan-1-ol, not two different isomers.

## ResultsPlus Examiner Tip

Remember that the OH is on a different position in the carbon chain in propan-1-ol and propan-2-ol. In this case the OH is on the end carbon in both formulae.

## *Question 2 (a) (i)*

Many candidates find ionic equations difficult. Some did not know the charge on a zinc ion. Others gave the wrong charge on the copper ion, despite the name copper(II) sufate indicating what it is. Many started from the complete equation and then deduced that sulfate ions were spectators, but for full marks the sulfate ions should not be shown in the final equation. State symbols were regularly missed out, again suggesting that candidates had not read the question carefully.

(a) (i) Write the **ionic** equation for the reaction between zinc and aqueous copper(II) ions, including state symbols.

**(2)**

~~$Cu^{2+} + SO_4^{2-}$ $Cu^{2+}_{(aq)}$~~

$Cu^{2+}SO_4^{2-}{}_{(aq)} + Zn_{(s)} \longrightarrow Zn^{2+}SO_4^{2-}{}_{(aq)} + Cu_{(s)}$

**ResultsPlus**
**Examiner Comments**

The sulfate ions should be cancelled out when they appear on both sides of the equation in the same state.

**ResultsPlus**
**Examiner Tip**

Don't show the spectator ions in your final ionic equation.

(a) (i) Write the **ionic** equation for the reaction between zinc and aqueous copper(II) ions, including state symbols.

**(2)**

$CuSO_{4(aq)} + Zn_{(s)} \rightarrow ZnSO_{4(aq)} + Cu_{(s)}$

$2e^- + Cu^{2+} \rightarrow Cu$

$Zn \rightarrow Zn^{2+} + 2e^-$

**ResultsPlus**

**Examiner Comments**

A number of candidates wrote two half equations, one for zinc turning into zinc ions, and one for copper ions turning into copper.

This candidate was allowed the state symbol mark only. Including the electrons shows that the difference between an ionic equation and a half equation is not clearly understood.

**ResultsPlus**

**Examiner Tip**

Only include electrons in equations if you are writing a half equation to illustrate redox.

## *Question 2 (a) (ii)-(iv)*

The calculation of energy transferred should have been easy as the expression was given. However candidates used a variety of incorrect numbers for the mass, including the mass of zinc powder, the sum of the mass of the powder and the solution, and a mass of one gram instead of the correct value of 50g.

Calculation of the number of moles of copper(II) sulfate in (iii) was nearly always correct.

In (iv) the two most common errors were forgetting the negative sign and giving the answer to more than three significant figures.

(ii) Calculate the quantity of heat energy produced in the experiment above, giving your answer in J. (Assume that the heat capacity of the mixture is 4.18 J $g^{-1}$ °$C^{-1}$ and its density is 1.00 g $cm^{-3}$.) Use the expression

energy transferred in joules = mass × specific heat capacity × temperature change

(2)

energy transferred in joules = 50 x 4.18 x (69.5 – 23)

= ~~97~~ 9718.5 J

(iii) Calculate the number of moles of copper(II) sulfate used in the experiment.

(1)

Moles = Concentration x Volume

Moles = 1.0 x $\frac{50}{1000}$

Moles = 0.05 mol

(iv) Use your answers from (a)(ii) and (a)(iii) to calculate the enthalpy change for the reaction in kJ $mol^{-1}$. Give your answer to **three** significant figures and include the appropriate sign.

(2)

1 kJ = 1000 J

x = 9718.5 J

x = 9.7185 kJ

enthalpy change = $\frac{9.7185 \text{ kJ}}{0.05 \text{ mol}}$

ΔH = -194.37 kJ $mol^{-1}$

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**Examiner Comments**

This candidate made the common mistake of giving the answer to (iv) to more than three significant figures.

**ResultsPlus**

**Examiner Tip**

Make sure you are clear about the difference between significant figures and decimal places.

(ii) Calculate the quantity of heat energy produced in the experiment above, giving your answer in J. (Assume that the heat capacity of the mixture is 4.18 J g$^{-1}$ °C$^{-1}$ and its density is 1.00 g cm$^{-3}$.) Use the expression

energy transferred in joules = mass × specific heat capacity × temperature change

(2)

Energy transferred = 50 × 4.18 × 46.5

= 9718.5 J

(iii) Calculate the number of moles of copper(II) sulfate used in the experiment.

(1)

moles = concentration × volume

$= 1.00 \times \frac{50}{10^3} = 0.05$ mol

(iv) Use your answers from (a)(ii) and (a)(iii) to calculate the enthalpy change for the reaction in kJ mol$^{-1}$. Give your answer to **three** significant figures and include the appropriate sign.

(2)

$\Delta H = \frac{9718.5}{0.5} \times 10^{-3}$ kJ mol$^{-1}$

= 19.44 kJ mol$^{-1}$

$\Delta H$ = −19.4 kJ mol$^{-1}$

(b) The thermometer used in this experiment gave an uncertainty in each

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**Examiner Comments**

This candidate has calculated the number of moles of copper(II) sulfate correctly, and then used the wrong value in (iv). However the method of calculation is clear, so one mark was allowed for including the negative sign and giving the answer to three significant figures.

## *Question 2 (b) (i)-(ii)*

A significant proportion of candidates failed to realise that the starting temperature of 23.0 °C could have been 0.5 °C too low and the finishing temperature of 69.5 °C could have been 0.5 °C too high, leading to a possible maximum temperature difference of 47.5 °C.

As two temperature readings are needed to calculate the difference, and there could be an error in each, the possible error is 2 x 0.5, and this must be used to find the percentage error.

(b) The thermometer used in this experiment gave an uncertainty in each temperature reading of ±0.5 °C.

(i) State the maximum temperature difference in this experiment that could have been obtained using this thermometer.

(1)

$0.5 - (-0.5) = 1\,°C$

(ii) What is the percentage error in the temperature change using this thermometer?

(1)

$\frac{1 \times 100}{(69.5 - 23)} = 2.15\,\%$

**ResultsPlus**

**Examiner Comments**

Using the value of ±0.5 to suggest a temperature difference of 1° was quite a common error This was not the maximum temperature difference in the experiment.

The answer to (ii) was correct.

(b) The thermometer used in this experiment gave an uncertainty in each temperature reading of ±0.5 °C.

(i) State the maximum temperature difference in this experiment that could have been obtained using this thermometer.

(1)

~~46.5 °C~~ ~~46.5 °C~~ ~~46.5°~~ ~~46.5 °C~~ 47.5 °C

(ii) What is the percentage error in the temperature change using this thermometer?

(1)

~~$= \frac{0.1}{47.5} \times 100$~~

~~$= 0.21\,\%$~~

$= \frac{0.1}{46.5} \times 100$

$= 0.22\,\%$

**ResultsPlus**

**Examiner Comments**

The answer to (i) is correct, but there is no reason to use the value of 0.1 in calculating the percentage error.

## *Question 2 (c)*

Candidates had difficulty in describing a suitable procedure clearly.

The first mark was given provided it was clear the temperature was recorded before adding the zinc, and candidates were not penalised if they did not describe leaving the solution to equilibrate with the surroundings. Some candidates wrote a description without ever referring to the addition of zinc. Others took measurements, but did not say of what, and many talked about measuring temperature with a stop clock.

The second mark was the most difficult to describe correctly. The mark was given for stating that the temperature should be measured after adding the zinc, at regular time intervals for several minutes. These measurements are used to draw a cooling curve, but many candidates said that temperature readings should be taken until they reached a maximum. Other candidates thought the zinc should be added in small portions, which would be of no use as cooling would occur between additions. Suggestions that the temperature should be read every 10 seconds were impracticable, and readings every five minutes would be so infrequent that cooling would occur. Readings at between 15 and 90 seconds were sensible, and enough readings should be taken to draw a cooling curve, which means a minimum of four points.

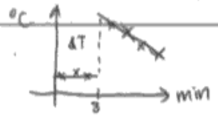
The third mark was not given for simply saying "plot a graph". There had to be a link with the results which had been collected, or stated to be a graph of temperature against time.

Finally the fourth mark had to refer to extrapolation of the cooling line to the time where the zinc was added, or attempt to explain the use of intersecting lines. Some candidates sketched a graph here which was a good way of explaining how to use the results.

(c) **Using the same equipment**, together with a stop clock, suggest a procedure that would improve the accuracy of this experiment by obtaining a more accurate temperature change. You must use the same mass of zinc powder and the same volume of 1.00 mol $dm^{-3}$ copper(II) sulfate solution.

(4)

Firstly, along with a stop clock, measure the temperature of the copper(II) sulfate solution with a 0–110°C thermometer for the first 3 minutes, stirring the 50 $cm^3$ and 1.00 mol $dm^{-3}$ solution continuously, and recording the temperature every minute. After 3 minutes, add the zinc powder with a mass of 5g little by little to ensure no frothing occurs. Stir the mixture steadily. Record the temperature of the mixture every ~~0.5 min~~ minute until the temperature reaches the highest point and starts to cool down. Draw a graph using the data, and extrapolate the lines to get an accurate temperature change at 3 minutes




**(Total for Question 2 = 13 marks)**

**ResultsPlus**

**Examiner Comments**

In this answer the zinc is added a little at a time, so there may be cooling between additions. The temperature is measured until it reaches its highest point, though the graph indicates that the temperatures were taken during the cooling period. The use of the data is shown clearly on the graph.

(c) **Using the same equipment**, together with a stop clock, suggest a procedure that would improve the accuracy of this experiment by obtaining a more accurate temperature change. You must use the same mass of zinc powder and the same volume of 1.00 mol $dm^{-3}$ copper(II) sulfate solution.

**(4)**

Add 50.0 $cm^3$ of copper (II) Sulfate Solution of concentration 1.00 mol $dm^{-3}$ ~~to~~ in a polystyrene cup, then ~~add~~ measure the initial temperature of the solution and avoid systematic error such as parallex error. Add 5 g of zinc powder to the polystyrene Cup and then stirr it for 10 seconds, use the stop watch to record the time, then cover the cup with a plastic lid with a therometer placed in it. Note down the final temperature after 5 minutes, then repeat the procedure.

**(Total for Question 2 = 13 marks)**

## ResultsPlus
**Examiner Comments**

Answers like this were seen frequently. If the mixture is left for five minutes there is no way of knowing what the maximum temperature is, as cooling may have started. Repeating the experiment and taking an average will be of no help.

## ResultsPlus
**Examiner Tip**

To find the maximum temperature you need to draw a cooling curve. This will show whether cooling occurred during the reaction, meaning that the highest temperature recorded should have been even higher.

## *Question 3 (a) (i)*

This experiment is one where many changes occur, so there were many possible answers.

Candidates were asked what they would see, so there was no credit given for describing what they would smell, or for identifying the products, or for giving tests for the gases evolved.

The amount of hydrogen iodide which forms is very small, and would appear as steamy fumes. The term "white fumes" was not allowed.

The hydrogen iodide would be oxidized to iodine, which might be seen as purple fumes, a black solid or a brown or black solution. Sulfur would be seen as a yellow solid.

The colour changes are very obvious to anyone who has seen the experiment, so simply saying that effervescence or a colour change occurred was not enough.

**3** Chloroalkanes and bromoalkanes can be made from alcohols by reaction of the alcohol with sodium chloride or bromide, in the presence of 50% aqueous sulfuric acid.

Iodoalkanes cannot be made from sodium iodide and sulfuric acid; red phosphorus and iodine can be used instead as the halogenating agent.

(a) (i) What would you **see** if concentrated sulfuric acid was added to solid sodium iodide? Give **two** observations.

(2)

1. effervescence

2. solid dissapears

**ResultsPlus**

**Examiner Comments**

This answer was too general to be given the marks. Effervescence usually refers to the sort of fizzing which occurs when an acid is added to a carbonate. In this reaction the most obvious observation is the production of coloured fumes and new solids and solutions being formed.

**ResultsPlus**

**Examiner Tip**

This reaction involves several redox reactions, as sulfuric acid can form three different products when it is reduced. It is a useful one to be familiar with.

3 Chloroalkanes and bromoalkanes can be made from alcohols by reaction of the alcohol with sodium chloride or bromide, in the presence of 50% aqueous sulfuric acid. NaCl

Iodoalkanes cannot be made from sodium iodide and sulfuric acid; red phosphorus and iodine can be used instead as the halogenating agent.

(a) (i) What would you **see** if concentrated sulfuric acid was added to solid sodium iodide? Give **two** observations. $H_2SO_4 + NaI$ $NaSO_4$

(2)

1. Purple vapours emmited due to Iodine.

2. Yellow flames due to Sodium.

**ResultsPlus**
**Examiner Comments**

The mark was given for the purple vapour, but a yellow solid, not yellow flames would be seen.

**ResultsPlus**
**Examiner Tip**

Sodium is a very reactive element, and would never be displaced in a reaction like this.

3 Chloroalkanes and bromoalkanes can be made from alcohols by reaction of the alcohol with sodium chloride or bromide, in the presence of 50% aqueous sulfuric acid.

Iodoalkanes cannot be made from sodium iodide and sulfuric acid; red phosphorus and iodine can be used instead as the halogenating agent.

(a) (i) What would you **see** if concentrated sulfuric acid was added to solid sodium iodide? Give **two** observations.

(2)

1. The solution will turn dark brown and perhaps a black get will form

2. We will see effervescence

**ResultsPlus**
**Examiner Comments**

The first mark was given for the description of a brown liquid or black solid, which is how the iodine might be seen.

No mark was given for saying that there would be effervescence.

## *Question 3 (a) (ii)*

Many candidates knew that sulfuric acid oxidizes hydrogen iodide or iodide ions, though oxidation and reduction were regularly confused.

Only a small number of answers were given the first mark. It had to be clear that hydrogen iodide was necessary for the reaction with the alcohol, or that when sulfuric acid reacted with sodium iodide there would be little hydrogen iodide available.

(ii) Explain why sodium iodide and sulfuric acid cannot be used to make iodoalkanes from alcohols.

(2)

Sulfuric acid is ~~no~~ very strong reducing agent and ~~it~~ ~~reduces~~ when hydrogen iodide forms it reduces it completely to Iodine

**ResultsPlus**
**Examiner Comments**

This candidate realises that hydrogen iodide is a necessary reagent but will not be around in the presence of concentrated sulfuric acid, so scored the first mark. However the sulfuric acid is an oxidizing agent, so the second mark was missed.

**ResultsPlus**
**Examiner Tip**

A quick way to work out whether oxidation or reduction is occurring is to use oxidation numbers. Iodine in HI is -1. In the element it is zero. Increase in oxidation numbers means oxidation is occurring.

(ii) Explain why sodium iodide and sulfuric acid cannot be used to make iodoalkanes from alcohols.

(2)

~~The~~ Instead of the alcohol and sodium Iodide reacting, $H_2SO_4$ and sodium Iodide will react.

**ResultsPlus**
**Examiner Comments**

This answer does not go far enough. It should say that the sulfuric acid oxidizes iodide ions. This means that little or no hydrogen iodide is present to react with the butanol.

**ResultsPlus**
**Examiner Tip**

Remember that halogenoalkanes are prepared using reactions that involve hydrogen halides. Sodium iodide does not react with butanol. Hydrogen iodide is needed.

## *Question 3 (b)*

The equation could be written showing phosphorus atoms or $P_4$ molecules, and could be shown with whole numbers or fractions.

Most candidates gave the correct formula for phosphorus(III) iodide, but it was surprising to see so many equations which were unbalanced. Equations showing I for iodine were not given the mark, nor were equations shown with ions.

(b) Give the equation for the reaction between phosphorus and iodine to form phosphorus(III) iodide. State symbols are not required.

(1)

$P_4 + 6I_2 \rightarrow 4PI_3$

**ResultsPlus**
**Examiner Comments**

This version of the equation is correct and scored the mark.

**ResultsPlus**
**Examiner Tip**

Learn the formulae of the molecules of non-metals. Halogens are always diatomic eg $Cl_2$. Phosphorus is $P_4$. Sulfur is $S_8$.

(b) Give the equation for the reaction between phosphorus and iodine to form phosphorus(III) iodide. State symbols are not required.

(1)

$P + I_2 \rightarrow PI_3$

**ResultsPlus**
**Examiner Comments**

Formulae are correct but the equation is not balanced.

**ResultsPlus**
**Examiner Tip**

When you are asked for an equation it must contain the correct species and be balanced.

## *Question 3 (c) (i)*

The slow addition of iodine suggests that the reaction is very exothermic or vigorous.

It would be dangerous to add the iodine too quickly, but simply saying that the reaction is dangerous without a reason did not score. It is true that the alcohol is volatile, but adding the iodine in portions through a reflux condenser reduces the hazard of a volatile liquid escaping, so answers simply saying "volatile" were not given credit.

(i) What does the manner in which the iodine is added in **step 3** suggest about the nature of the reaction?

(1)

The reaction is a slow one and needs to be made faster.

**ResultsPlus**
**Examiner Comments**

Iodine is added slowly because the reaction occurs very rapidly. This method of addition would not speed up a slow reaction.

(i) What does the manner in which the iodine is added in **step 3** suggest about the nature of the reaction?

(1)

Iodine is volatile so ~~explos~~ explosion may occur to stop this. we must do this precedure gently.

**ResultsPlus**
**Examiner Comments**

This shows lack of careful reading of the question. It asked what the manner of adding iodine showed about the nature of the reaction.

When considering the hazards in the preparation, it is the volatility of organic liquids, not the iodine, which is important.

## *Question 3 (c) (ii)*

The mark was given for suggesting a possible colour which would be seen if little iodine was left in the organic solvent, or for saying that the mixture would become colourless. Many answers said that solid iodine would no longer be visible, but this was not allowed, as it would have dissolved during the refluxing process. Some candidates suggested adding starch to test for iodine which did not answer the question of what to look for.

(ii) Completion of **step 4** requires that 'little or no iodine is visible'. State what you would look for in this step to ensure that this is true.

(1)

Complete dissolving of Iodine

**ResultsPlus**

**Examiner Comments**

If dissolved iodine is present it will make the solution coloured. This answer does not say what to look for to detect whether any iodine is present.

(ii) Completion of **step 4** requires that 'little or no iodine is visible'. State what you would look for in this step to ensure that this is true.

(1)

Violet solution turn colourless.

**ResultsPlus**

**Examiner Comments**

The term 'violet' was allowed here for the purple colour of iodine, which would get paler and disappear as it was used up.

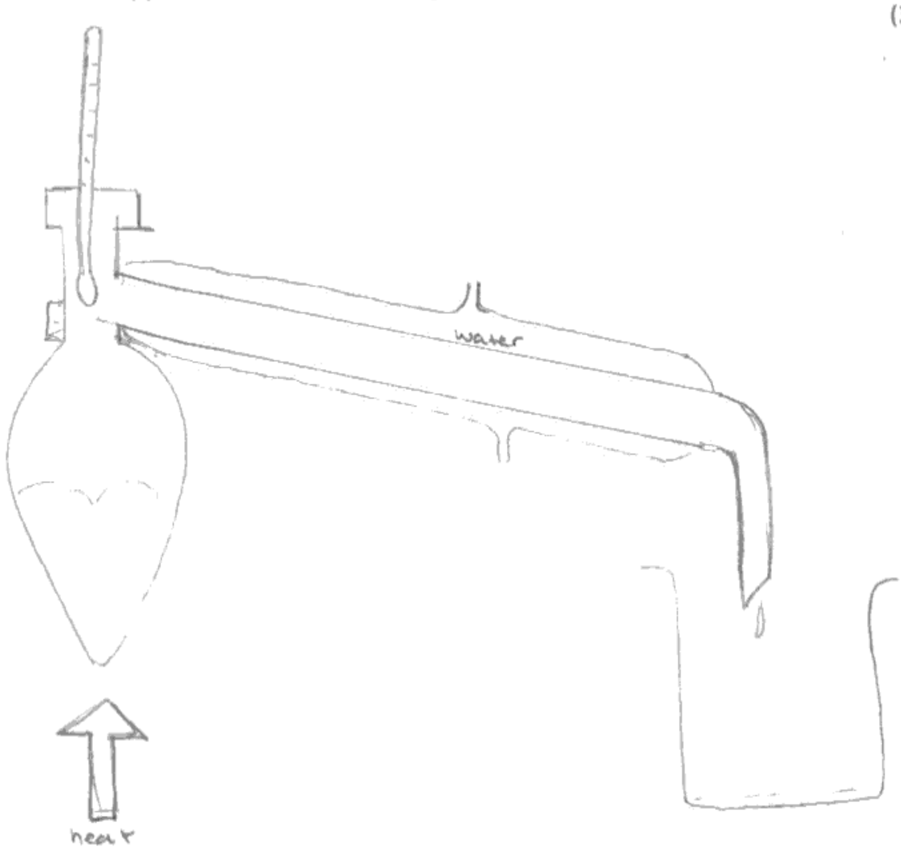
## *Question 3 (c) (iii)*

Many good diagrams were seen by examiners, and only a few candidates drew diagrams showing distillation under reflux. These candidates could still get one mark if the condenser was drawn correctly.

A mark was lost if the apparatus was completely sealed, though the receiver could be shown with a vent or leading to either an open flask. Some candidates lost marks by drawing a horizontal condenser or even one sloping back in to the distillation flask, which would therefore lead to reflux. The opening above the distillation flask could be sealed with a stopper, thermometer or tap funnel, but a mark was lost if it was left open or had a filter funnel in it.

(iii) Draw the apparatus that is used in **step 6** for distillation.

(3)



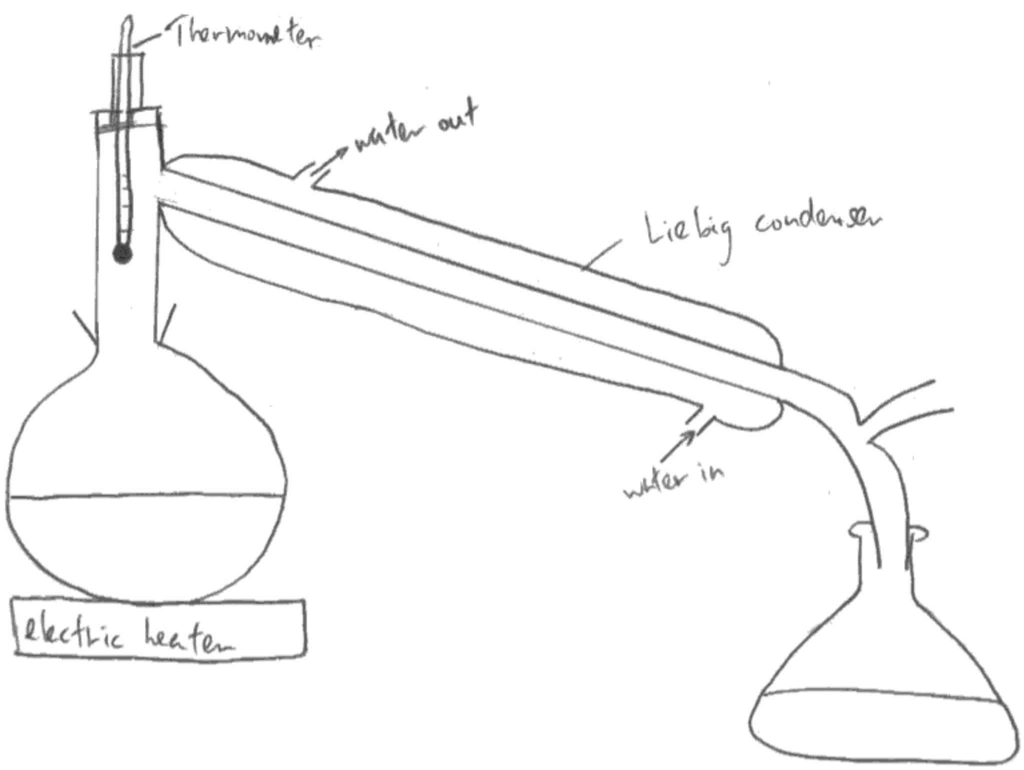

**ResultsPlus**

**Examiner Comments**

This answer lost a mark as it does not show the direction of water flow in the condenser. The tubes leading in and out of the condenser should be near the ends, though one like this would probably work as long as water entered at the bottom.

(iii) Draw the apparatus that is used in **step 6** for distillation.

(3)




**ResultsPlus**

**Examiner Comments**

This drawing scored full marks.

## *Question 3 (c) (iv)*

Sodium thiosulfate is added to remove residual iodine. Incorrect suggestions from candidates included neutralisation and the need for a dense solution so that the organic layer would separate from the aqueous layer.

(iv) Suggest why the first washing of the product in **step 7** is with dilute sodium thiosulfate solution rather than with water alone.

(1)

so that any acid present could be removed.

**ResultsPlus**
**Examiner Comments**

Thisosulfate ions react with acid forming sulfur, so it would certainly not be used to remove acid.

(iv) Suggest why the first washing of the product in **step 7** is with dilute sodium thiosulfate solution rather than with water alone.

(1)

It is used to ~~react away~~ remove some of the impurities by reacting with them and forming crystals.

**ResultsPlus**
**Examiner Comments**

This is not specific enough. There had to be reference to the iodine to get the mark.

## *Question 3 (c) (v)*

Calcium chloride only acts as a drying agent when it is anhydrous, and hydrated calcium chloride would not remove water. This distinction had to be clear for candidates to gain the mark, and answers simply saying "calcium chloride is a drying agent" were insufficient.

(v) State why calcium chloride used in **step 8** must be anhydrous.

(1)

Calcium chloride is a drying agent which will absorb all of the water left in the reaction, becoming hydrous at the end.

**ResultsPlus**
**Examiner Comments**

This answer gained the mark. It implies that the anhydrous calcium chloride, which is specified in the question, is changing when it absorbs water. The word "hydrous" was accepted for hydrated.

**ResultsPlus**
**Examiner Tip**

The question was about anhydrous calcium chloride, so your answer should not just refer to "calcium chloride" unless other comments are added.

(v) State why calcium chloride used in **step 8** must be anhydrous.

(1)

It is used as drying agent, so water should not present in it.

**ResultsPlus**
**Examiner Comments**

In this answer it is clear why the calcium chloride should be anhydrous.

## *Question 3 (c) (vi)*

From the description of the practical procedure, most candidates realised that 1-iodobutane is a liquid, and suggested some form of distillation. Only a few incorrectly suggested recrystallisation, or said "purification" which was too general for the mark to be given. Some answers said that the calcium chloride should be removed by filtration, indicating that the phrase "after decanting the mixture from the calcium chloride" had been missed or misunderstood.

(vi) To complete the preparation, after decanting the mixture from the calcium chloride, there should be a **step 9**. What is this step?

(1)

Filter the solution ~~to~~ to make sure that ~~total~~ calcium chloride is removed from the organic layer.

**ResultsPlus**
**Examiner Comments**

If decanting does not remove the calcium chloride the mixture could be filtered, but completion of the preparation requires final distillation.

(vi) To complete the preparation, after decanting the mixture from the calcium chloride, there should be a **step 9**. What is this step?

(1)

Crystallize the organic layer to form crystals of 1-iodobutane.

**ResultsPlus**
**Examiner Comments**

This candidate thinks that the product is a solid.

**ResultsPlus**
**Examiner Tip**

Look carefully at the information available to decide whether the product is a solid or a liquid.

The crude iodobutane in this experiment was distilled off in step 6, indicating it is a liquid.

## *Question 3 (d) (iii)*

In a question asking why actual yield is lower than maximum possible yield it is not enough to blame human error, or simply say "losses", and there was no suggestion in the question that reactants might be impure. It is also not a good idea to put a long list of suggestions, as if these include wrong answers the correct ones may not be given credit.

In this reaction other products, namely hydrogen chloride and $POCl_3$ form as well as 1-chlorobutane. However competing, or side reactions, are reactions giving products other than the desired one. In some reactions there might be multiple substitutions, or eliminations as well as substitutions. Some candidates were not clear about this distinction.

(iii) Give **one** reason why the actual yield is lower than the maximum possible yield.

(1)

• human errors - calculation errors • heat energy given off to environment

**ResultsPlus**
**Examiner Comments**

A significant number of candidates referred to heat loss as a reason for low yield.

**ResultsPlus**
**Examiner Tip**

Heat losses are a source of error in experiments where an enthalpy change is measured. They are not relevant to the yield in an organic preparation.

(iii) Give **one** reason why the actual yield is lower than the maximum possible yield.

(1)

Other products are also produce and there might be disubstitution or trisubstitution.

**ResultsPlus**
**Examiner Comments**

The reaction does produce other products, HCl and $POCl_3$, which is why it has a low atom economy. If two Cl atoms were somehow substituted, this would be a side reaction.

This answer does not clearly distinguish the two different reasons for a lower than maximum possible yield.

## *Question 3 (d) (i)-(ii)*

The method of calculating percentage yields is very familiar to candidates and was often carried out correctly. However it was clear that many candidates did not understand when to round values, and the appropriate number of significant figures to use.

In (i) the maximum mass of 1-chlorobutane is obtained by multiplying the number of moles of 1-chlorobutane by its molar mass. Candidates who rounded the number of moles of 1-chlorobutane, instead of working with the number stored in their calculator, got values for the percentage yield in (i) up to 2% from the correct value. Candidates should be advised to round numbers at the end of a calculation which has several steps.

(d) Chloroalkanes can be made from an alcohol and phosphorus(V) chloride, $PCl_5$.
The equation for the reaction of butan-1-ol with $PCl_5$ is

$$CH_3CH_2CH_2CH_2OH + PCl_5 \rightarrow CH_3CH_2CH_2CH_2Cl + HCl + POCl_3$$

This reaction is not suitable for the manufacture of 1-chlorobutane on a large scale.

(i) In a laboratory preparation of 1-chlorobutane, 95.0 g of butan-1-ol was used. Calculate the maximum mass of 1-chlorobutane that could be obtained.

(Assume the molar masses are, in g mol$^{-1}$, butan-1-ol = 74.0, 1-chlorobutane = 92.5)

**(2)**

$n = \frac{95.0}{74.0} = 1.28$ mol of butan-1-ol

∴ butan-1-ol : 1-chlorobutane

1 : 1

1.28 : $x$

$x = 1.28$ mol

$1.28 = \frac{m}{92.5}$

$1.28 \times 92.5 = m$

$118$ g $= m$

(ii) In practice, 95.3 g of 1-chlorobutane was obtained. Calculate the percentage yield.

**(1)**

% yield $= \frac{95.3}{118} \times 100 = 80.7\%$

**ResultsPlus**

**Examiner Comments**

This candidate lost one of the marks in (i) by rounding the number of moles too early.

In (ii) the calculation using 95.3÷118 would lead to a percentage of 80.76, which should not be rounded to 80.7.

**ResultsPlus**

**Examiner Tip**

Don't round numbers in calculations until you reach the end of the final step.

(d) Chloroalkanes can be made from an alcohol and phosphorus(V) chloride, $PCl_5$.
The equation for the reaction of butan-1-ol with $PCl_5$ is

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(i) In a laboratory preparation of 1-chlorobutane, 95.0 g of butan-1-ol was used. Calculate the maximum mass of 1-chlorobutane that could be obtained.

(Assume the molar masses are, in g mol$^{-1}$, butan-1-ol = 74.0, 1-chlorobutane = 92.5)

**(2)**

n of butanol = $\frac{95.0}{74.0}$ = 1.28

1.28 moles of 1-chlorobutane = $\frac{x}{92.5}$

$x$ = 118.4 g

(ii) In practice, 95.3 g of 1-chlorobutane was obtained. Calculate the percentage yield.

**(1)**

$\frac{95.3}{118.4} \times 100$

= 80.5% ≈ 81%

## ResultsPlus
**Examiner Comments**

This candidate has also rounded the number of moles too early in (i).

In (ii) the calculation shown comes to 80.48, which should not be rounded to 81.

## *Question 3 (d) (iv)*

This question asked for two reasons why the reaction would not be used industrially to produce 1-chlorobutane, but again long lists of suggestions were often given.

Some of the suggestions given were correct comments describing the reaction e.g. it is exothermic, but did not answer the question of why it is not used industrially. The most obvious answer is the low atom economy of the reaction. Saying that the atom economy is less than 100% was insufficient.

Many answers referred to the release of acidic fumes of hydrogen chloride, but it would be relatively easy to prevent this. Hydrogen chloride and hydrochloric acid are useful reagents, so the comment that "the other products" are not wanted is incorrect. If unwanted materials are produced then there is expense involved in separating and disposing of them, and reference to this gained credit.

(iv) Give **two** reasons why this reaction would not be used industrially to make 1-chlorobutane.

(2)

Because first $PCl_5$ is expensive to be used in an industy at a large scale, and second because ~~there are so many dangerous and~~ the economical yield percentage of this process or reaction ~~experiment~~ is low

**(Total for Question 3 = 19 marks)**

**TOTAL FOR PAPER = 50 MARKS**

**ResultsPlus**
**Examiner Comments**

This refers to the economical yield percentage, but should have used the term atom economy.

(iv) Give **two** reasons why this reaction would not be used industrially to make 1-chlorobutane.

(2)

• the percentage yield is not high enough

• expensive to produce

• HCl being produced which might be corrosive if in liquid state.

**(Total for Question 3 = 19 marks)**

**TOTAL FOR PAPER = 50 MARKS**

**ResultsPlus**

**Examiner Comments**

Answers which are more specific are needed.

The laboratory preparation gave a yield of over 80% but candidates could not know the yield in an industrial situation.

Corrosive liquids are often used in industry, with suitable precautions, so the corrosive nature of hydrogen chloride was not allowed for the mark.

# Paper Summary

This paper is about laboratory skills, and it is hoped that candidates will have first-hand practical experience of laboratory work. Where experiments are hazardous, using video recordings can be very useful.

Based on their performance on this paper candidates are offered the following advice:

- take time to read and understand the questions.
- consider how you can help examiners understand your reasoning.
- pay attention to your presentation, including detail in calculations (e.g. rounding and significant figures), and express explanations with care.
- be familiar with the changes which can be observed in chemical reactions.
- understand what is happening at a molecular level.
- understand why different procedures are used.

# Grade Boundaries

Grade boundaries for this, and all other papers, can be found on the website on this link:

http://www.edexcel.com/iwantto/Pages/grade-boundaries.aspx

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