Examiners' Report
June 2024

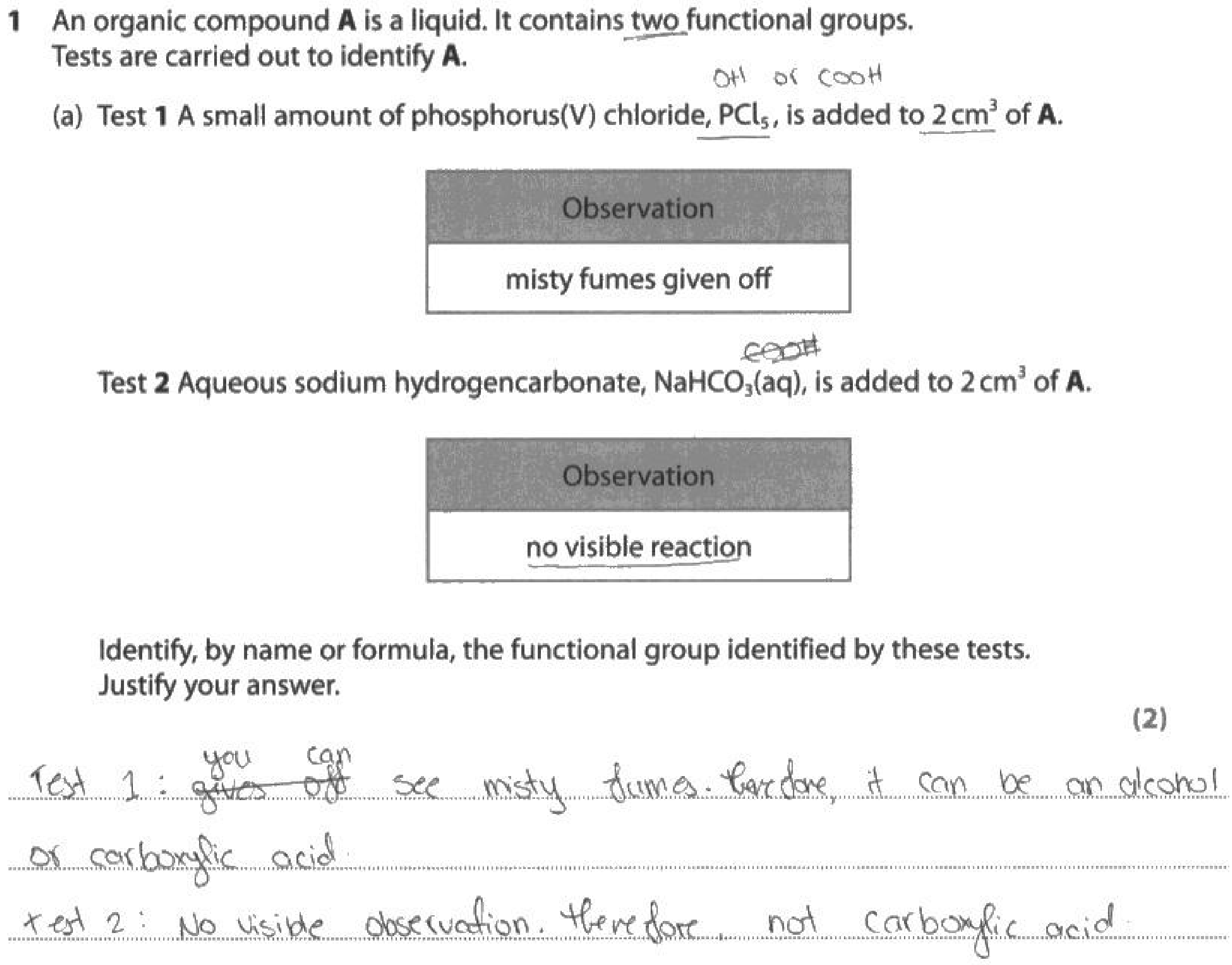
IAL Chemistry WCH16 01

Introduction

The paper had an emphasis on experimental techniques and there were good opportunities for all candidates to demonstrate their chemical understanding and the mean mark was 31.9. Many candidates had clearly prepared well for this paper and were able to apply their knowledge of practical work successfully to familiar and novel situations. However, it appeared that a significant number did not have a good understanding of how to use a separating funnel or reflux apparatus, nor a good grasp of how to minimise uncertainty in volumetric analysis. But the calculation and graph questions were done extremely well by the majority and there was no evidence of candidates running out of time.

Question 1 (a)

The two tests were clearly well known by the majority of candidates with nearly half getting full marks. However, a number did not link the presence of the OH group to the observation and so lost a mark and a few suggested other functional groups including aldehydes and alkenes.



1 An organic compound **A** is a liquid. It contains two functional groups.
Tests are carried out to identify **A**.

OH or COOH

(a) Test **1** A small amount of phosphorus(V) chloride, PCl_5, is added to 2 cm^3 of **A**.

Observation
misty fumes given off

~~COOH~~

Test **2** Aqueous sodium hydrogencarbonate, $NaHCO_3(aq)$, is added to 2 cm^3 of **A**.

Observation
no visible reaction

Identify, by name or formula, the functional group identified by these tests.
Justify your answer.

(2)

Test 1 : ~~gives off~~ you can see misty fumes. therefore, it can be an alcohol or carboxylic acid.

test 2 : No visible observation. therefore, not carboxylic acid.

ResultsPlus
Examiner Comments

This is a fully correct answer where the candidate has referred to the test results and identified the functional group present.

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Observation
misty fumes given off

Test **2** Aqueous sodium hydrogencarbonate, $NaHCO_3(aq)$, is added to 2 cm^3 of **A**.

Observation
no visible reaction

Identify, by name or formula, the functional group identified by these tests.
Justify your answer.

(2)

OH, because when solid PCl_5 is added to a ~~grou~~ compound containing OH, it releases misty fumes of HCl

ResultsPlus
Examiner Comments

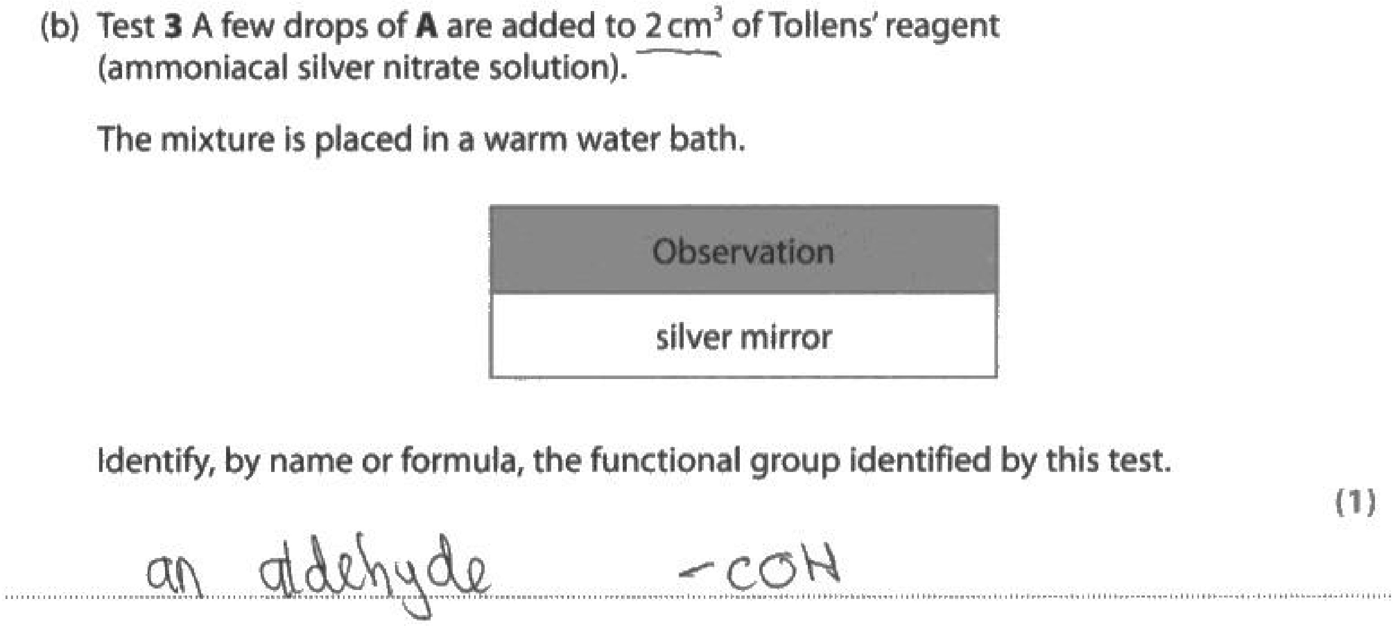
The candidate has correctly related the production of HCl to the presence of an OH group. However, they have not referred to the second test result that shows that the compound does not contain a COOH group, so only 1 mark is scored.

ResultsPlus
Examiner Tip

When you are asked to justify your answer, make sure you make use of all the information given to you.

Question 1 (b)

The Tollens' test used to identify an aldehyde was understood by almost most candidates. However, the most common error was where the candidate correctly stated the test identified an aldehyde but then gave the wrong formula of COH instead of CHO.



(b) Test **3** A few drops of **A** are added to 2 cm^3 of Tollens' reagent (ammoniacal silver nitrate solution).

The mixture is placed in a warm water bath.

Observation
silver mirror

Identify, by name or formula, the functional group identified by this test.

(1)

an aldehyde -COH

ResultsPlus
Examiner Comments

The question asks for the identity of the functional group by either name or formula. Here, the candidate has correctly identified the functional group by name, but they also given the wrong formula and so the mark is lost.

ResultsPlus
Examiner Tip

If you are asked to identify something by either name or formula, only give the one answer you are most confident about.

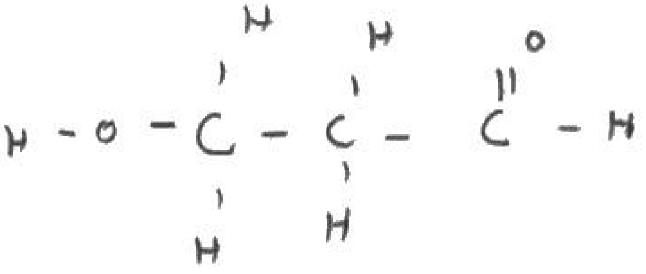
Question 1 (c)

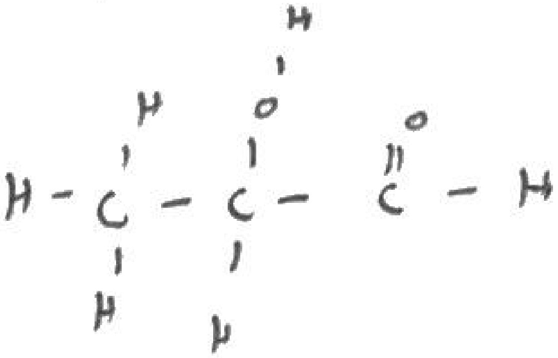
The majority of candidates were able to draw the correct displayed formula of the two structural isomers. However, common errors included the incorrect conductivity of the OH group, the same structure drawn twice and the wrong number of carbon atoms. Part (ii) was not as successfully done as many candidates did not respond to the question which asked for identification of the protons giving the described spectrum. Many circled just one proton attached to the carbon or just drew the structure, which was usually correctly identified but did not score.

(c) Compound **A** is non-cyclic and its mass spectrum has a molecular ion peak with *m/z* value of 74.

(i) Draw the **displayed** formula of the **two** structural isomers of **A**, using this information and your answers from (a) and (b).

(2)

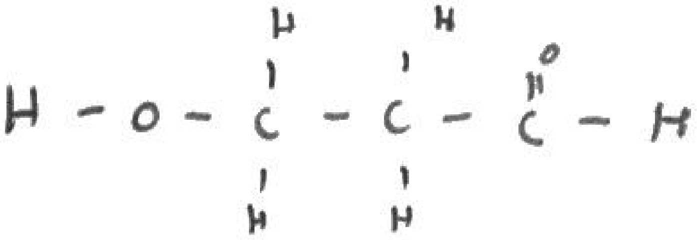




(ii) A low resolution proton NMR spectrum of **A** shows four peaks with relative areas of 1:2:2:1.

Deduce which of the structures in (c)(i) is correct, by identifying the relative peak areas on your formula.

(1)



ResultsPlus
Examiner Comments

The candidate has correctly identified the two structural isomers in part (i) and, in part (ii) has chosen the one that produces the described NMR spectrum. But they have not identified the hydrogen atoms that produce the spectrum, so no mark is awarded. This was a common mistake.

ResultsPlus
Examiner Tip

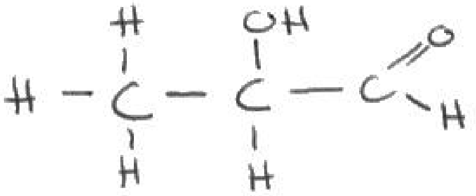
Read the question carefully and make sure all the points are covered in your answer.

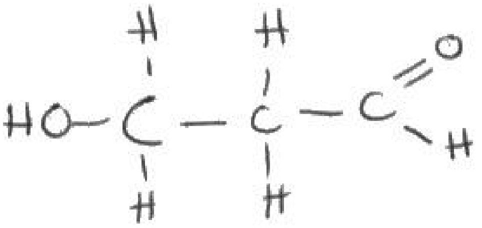
OH CHO

(c) Compound **A** is non-cyclic and its mass spectrum has a molecular ion peak with *m*/*z* value of 74. Mr = 74

(i) Draw the **displayed** formula of the **two** structural isomers of **A**, using this information and your answers from (a) and (b).

(2)

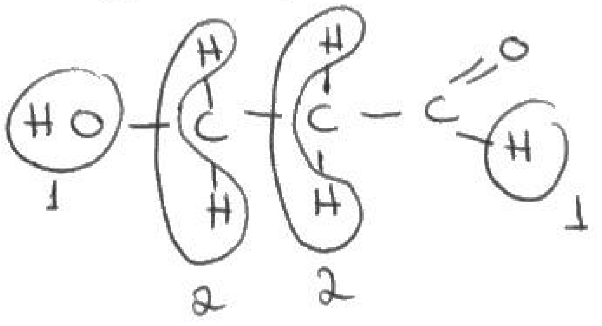




(ii) A low resolution proton NMR spectrum of **A** shows four peaks with relative areas of 1:2:2:1. 4 proton environments

Deduce which of the structures in (c)(i) is correct, by identifying the relative peak areas on your formula.

(1)

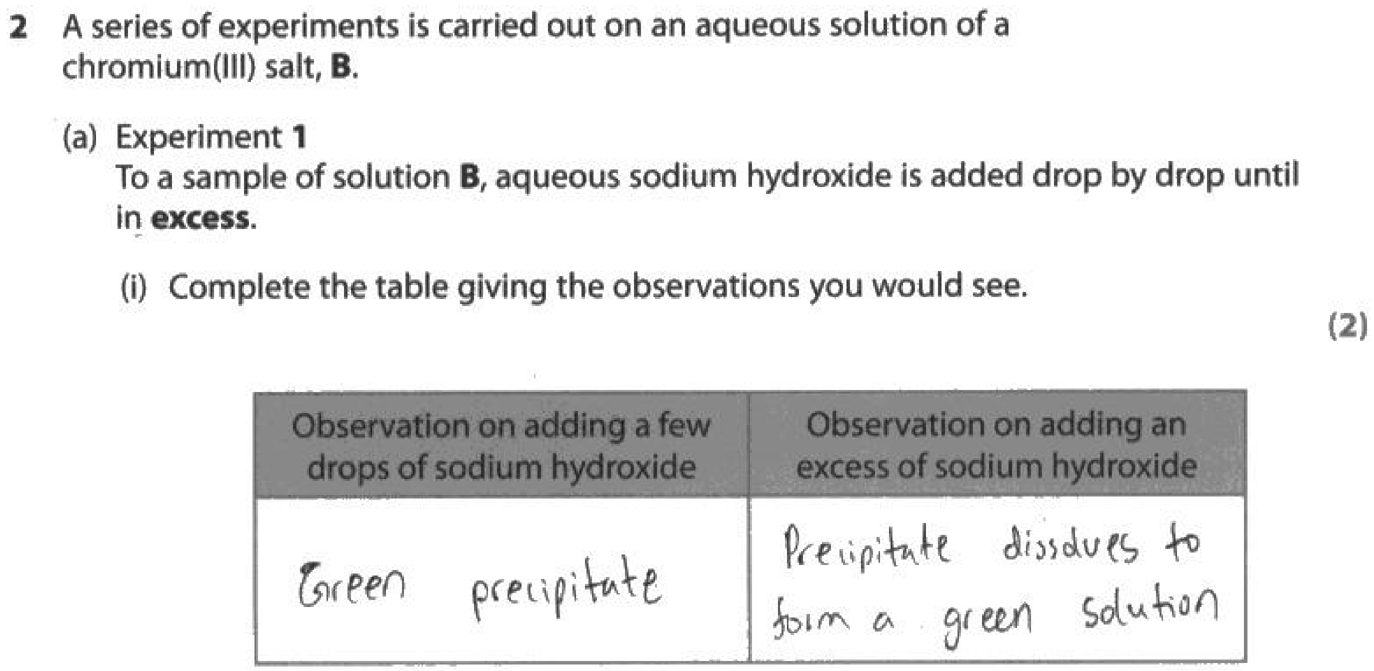


ResultsPlus
Examiner Comments

This is a fully correct answer. The candidate has correctly identified the two structural isomers in part (i). In part (ii), they have chosen the one that produces the described NMR spectrum and identified the hydrogen atoms that produce the spectrum.

Question 2 (a)(i)

Most candidates knew the reaction of chromium (III) ions with sodium hydroxide and there were many correct answers. But occasionally the second mark was lost as the candidate simply referred to the green precipitate dissolving but did not state its colour.



2 A series of experiments is carried out on an aqueous solution of a chromium(III) salt, **B**.

(a) Experiment **1**
To a sample of solution **B**, aqueous sodium hydroxide is added drop by drop until in **excess**.

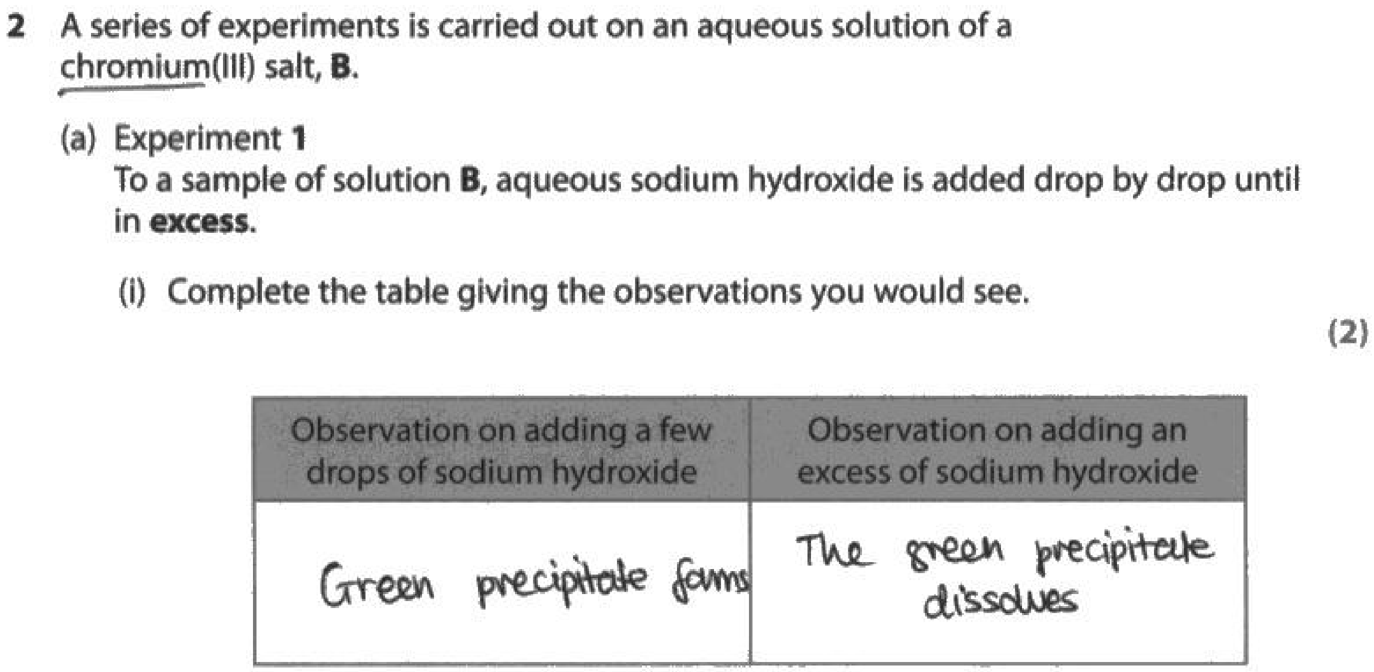
(i) Complete the table giving the observations you would see.

(2)

Observation on adding a few drops of sodium hydroxide	Observation on adding an excess of sodium hydroxide
Green precipitate	Precipitate dissolves to form a green solution

ResultsPlus
Examiner Comments

Here the candidate scores both marks for two correct observations.



2 A series of experiments is carried out on an aqueous solution of a chromium(III) salt, **B**.

(a) Experiment **1**
To a sample of solution **B**, aqueous sodium hydroxide is added drop by drop until in **excess**.

(i) Complete the table giving the observations you would see.

(2)

Observation on adding a few drops of sodium hydroxide	Observation on adding an excess of sodium hydroxide
Green precipitate forms	The green precipitate dissolves

ResultsPlus
Examiner Comments

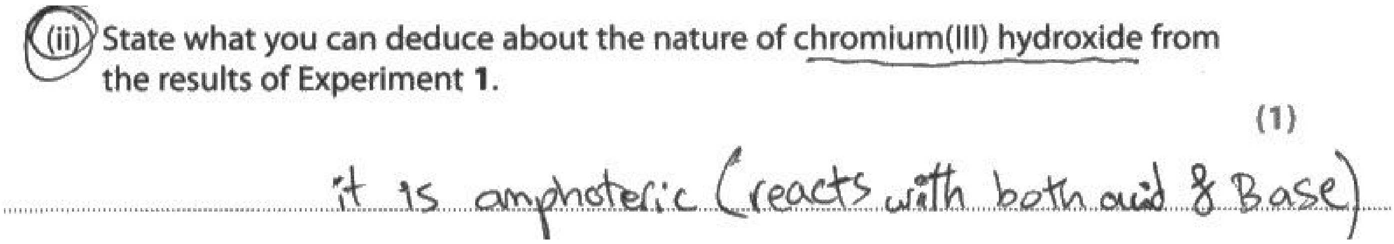
This was quite a common mistake where the candidate clearly knows the chemistry of chromium (III) compounds but has not stated the colour of the solution formed.

ResultsPlus
Examiner Tip

When asked to give observations, always mention the colour even if there has been no change.

Question 2 (a)(ii)

This question was poorly answered and only a minority of candidates scored the mark. Rather than deducing the answer from the results in part (i) candidates often discussed solubility, ligand exchange, redox and deprotonation.



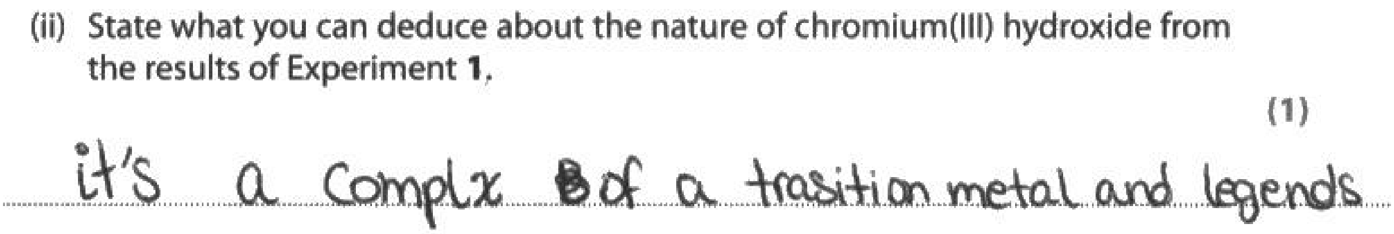
(ii) State what you can deduce about the nature of chromium(III) hydroxide from the results of Experiment **1**.

(1)

it is amphoteric (reacts with both acid & Base)

ResultsPlus
Examiner Comments

This candidate was in a minority with this correct answer. Either saying it is amphoteric or it reacts with an acid or base would score and here they have given both answers.



(ii) State what you can deduce about the nature of chromium(III) hydroxide from the results of Experiment **1**.

(1)

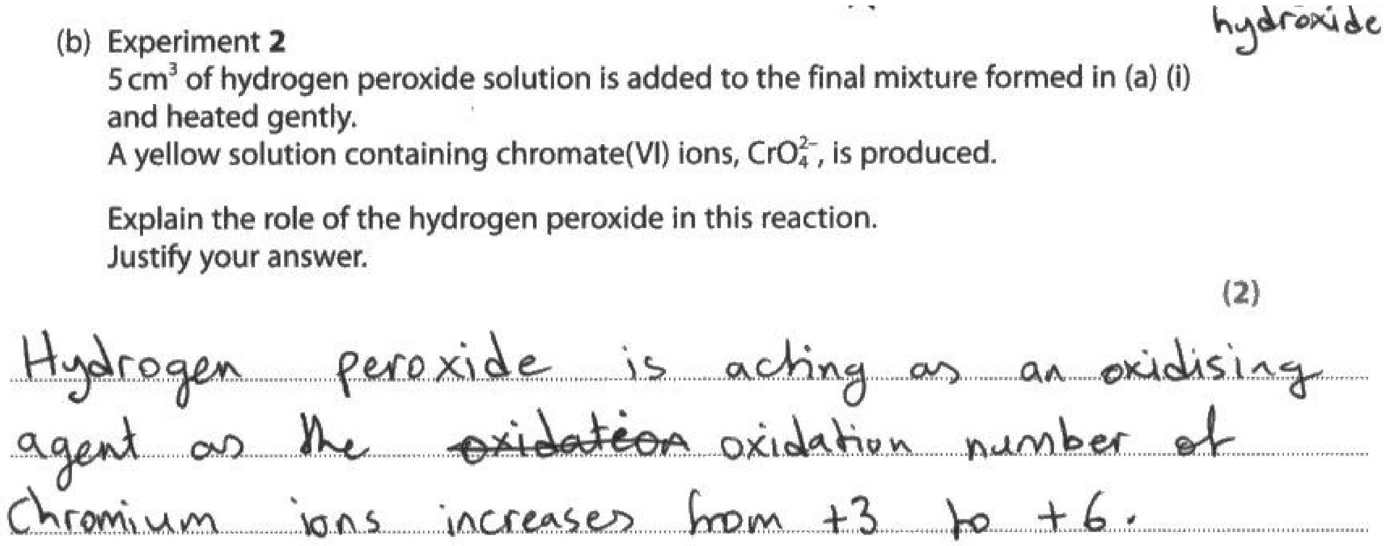
it's a Complx of a trasition metal and legends

ResultsPlus
Examiner Comments

This was a common wrong answer where the candidate has just mentioned some properties of transition metals.

Question 2 (b)

Candidates answered this question particularly well as most were able to identify hydrogen peroxide as an oxidising agent with very few getting confused between oxidation and reduction. The justification, by stating the correct oxidation numbers of chromium, also scored well.



hydroxide

(b) **Experiment 2**

5 cm^3 of hydrogen peroxide solution is added to the final mixture formed in (a) (i) and heated gently.

A yellow solution containing chromate(VI) ions, CrO_4^{2-}, is produced.

Explain the role of the hydrogen peroxide in this reaction.

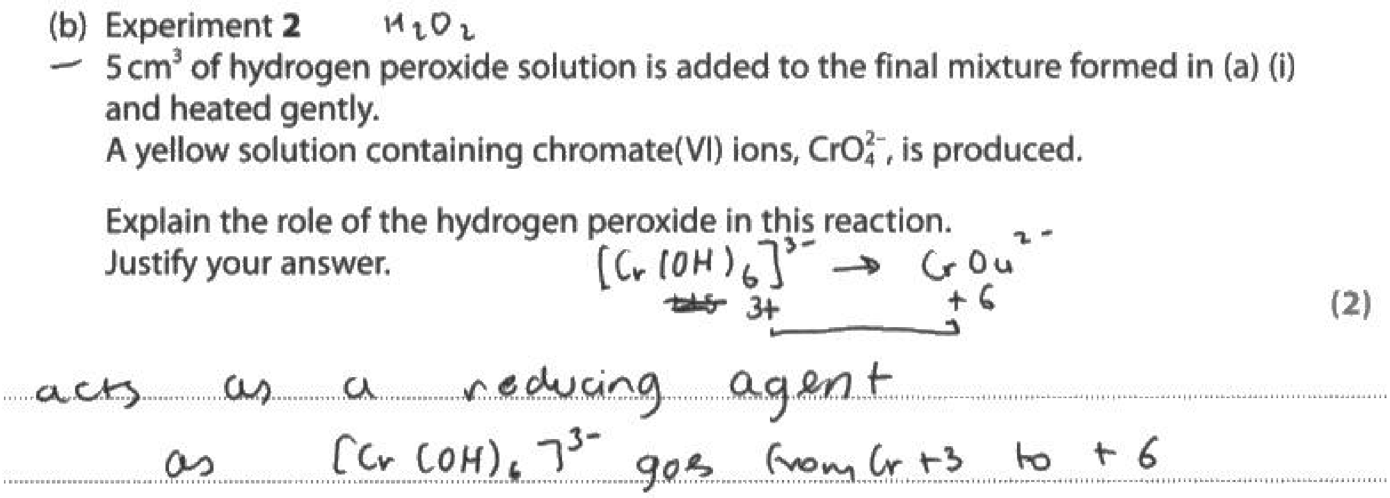
Justify your answer.

(2)

Hydrogen peroxide is acting as an oxidising agent as the ~~oxidateon~~ oxidation number of Chromium ions increases from +3 to +6.

ResultsPlus Examiner Comments

This is a fully correct answer where the candidate states that the hydrogen peroxide is an oxidising agent and justifies their answer with the correct oxidation numbers of chromium.



(b) Experiment **2** H_2O_2

5 cm^3 of hydrogen peroxide solution is added to the final mixture formed in (a) (i) and heated gently.
A yellow solution containing chromate(VI) ions, CrO_4^{2-}, is produced.

Explain the role of the hydrogen peroxide in this reaction.
Justify your answer.

$$\underset{3+}{[Cr(OH)_6]^{3-}} \rightarrow \underset{+6}{CrO_4^{2-}}$$

(2)

acts as a reducing agent

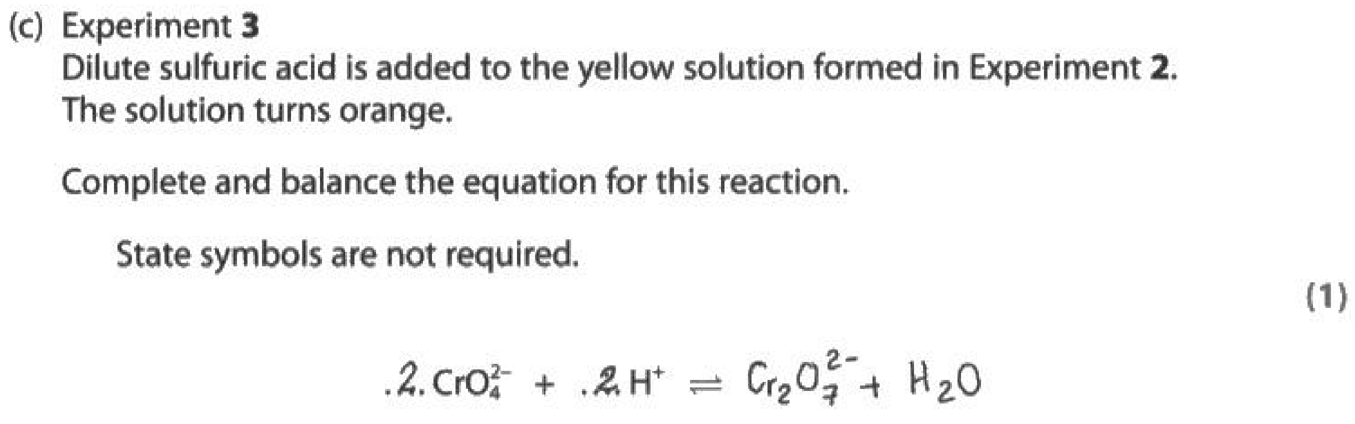
as $[Cr(OH)_6]^{3-}$ goes from Cr +3 to +6

ResultsPlus
Examiner Comments

The candidate has got confused between oxidation and reduction, but has scored the mark for the correct oxidation states of chromium.

Question 2 (c)

This equation proved to be more challenging than expected with many candidates putting OH^- ions as a product, rather than water.



(c) Experiment **3**

Dilute sulfuric acid is added to the yellow solution formed in Experiment **2**.
The solution turns orange.

Complete and balance the equation for this reaction.

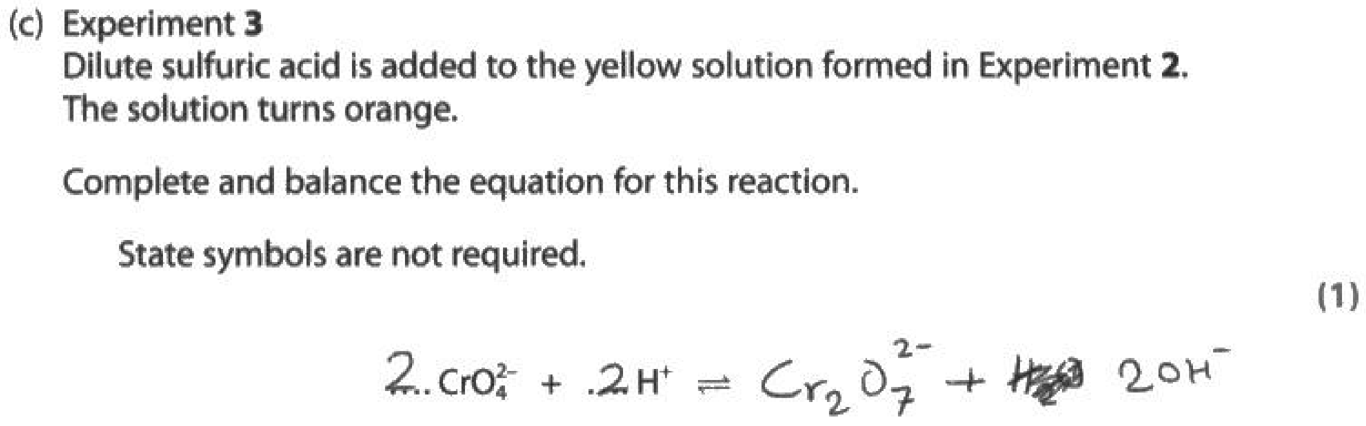
State symbols are not required.

(1)

$$2CrO_4^{2-} + 2H^+ \rightleftharpoons Cr_2O_7^{2-} + H_2O$$

ResultsPlus
Examiner Comments

An example of a fully correct equation.



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Complete and balance the equation for this reaction.

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(1)

$$2CrO_4^{2-} + 2H^+ \rightleftharpoons Cr_2O_7^{2-} + 2OH^-$$

ResultsPlus
Examiner Comments

This was a common wrong equation with OH^- instead of water on the right hand side.

Question 3 (a)(i)

Many candidates understood that manganate(VII) ions reacted with iodide ions and some gave detailed explanations of why this reaction took place based on entropy and the charges on the ions. However, few went on to explain that because the iodide was in excess all the manganate(VII) ions would be used up. A significant number simply talked about quenching without any reference to the reaction taking place and others got the reactions confused with some suggesting that the iodide ions reacted with the sulfuric acid.

3 A group of students carried out an experiment to investigate the reaction between potassium manganate(VII) and ethanedioate ions in acid conditions.

The equation for the reaction is shown.

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

Procedure

Step **1** Measure 10.0 cm^3 of potassium iodide solution into each of eight conical flasks.

Step **2** Measure 100.0 cm^3 of ethanedioic acid solution into a 250 cm^3 beaker. Add 25.0 cm^3 of potassium manganate(VII) solution and 5.0 cm^3 sulfuric acid to the beaker. Mix the contents of the beaker and start a timer.

Step **3** Immediately withdraw 10.0 cm^3 of reaction mixture and add it to the first conical flask containing (excess) potassium iodide solution.

Step **4** Continue removing 10.0 cm^3 of reaction mixture every minute for seven minutes. Each time, add the reaction mixture to a new conical flask containing the potassium iodide solution.

Step **5** Using starch as an indicator, titrate the iodine formed in the conical flasks with sodium thiosulfate solution.

The equation for the reaction in Step **3** is shown.

$$2MnO_4^-(aq) + 16H^+(aq) + 10I^-(aq) \rightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(l)$$

The equation for the titration in Step **5** is shown.

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

(a) (i) Explain why Step **3** effectively stops the reaction between potassium manganate(VII) and ethanedioate ions.

(2)

the reaction is quenched and potassium iodide reacts with ethanediote ions in the solution.

ResultsPlus
Examiner Comments

The reaction is stopped because the KI reacts with manganate(VII) ions. This candidate has chosen the wrong reaction, so no marks are awarded.

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(a) (i) Explain why Step **3** effectively stops the reaction between potassium manganate(VII) and ethanedioate ions.

(2)

Iodide ions react with the MnO_4^- ions more rapidly than $C_2O_4^{2-}$ ions and ~~due~~ due to I^- ions being in excess all the MnO_4^- ions are reduced to Mn^{2+} ions so $C_2O_4^{2-}$ ions can not react with MnO_4^-

ResultsPlus
Examiner Comments

This is an excellent answer. The candidate explains that the KI reacts with the manganate(VII) ions and as the KI is in excess, all the manganate(VII) ions are used up.

3 A group of students carried out an experiment to investigate the reaction between potassium manganate(VII) and ethanedioate ions in acid conditions.

The equation for the reaction is shown.

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

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(a) (i) Explain why Step **3** effectively stops the reaction between potassium manganate(VII) and ethanedioate ions.

(2)

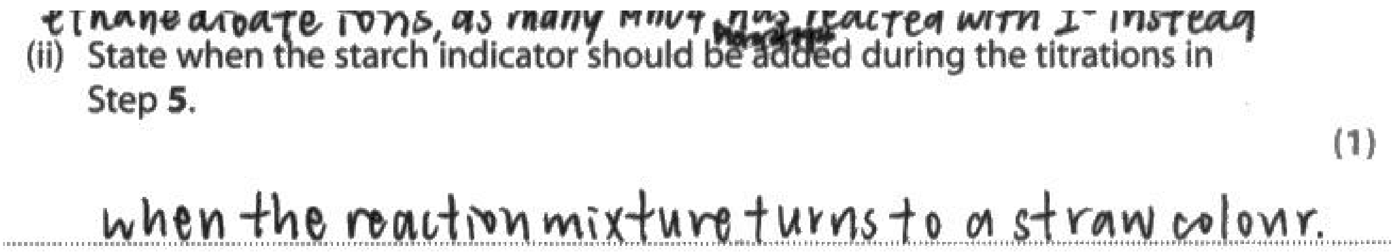
because potassium iodide react with potassium manganat (VII) so reaction stop. To quench the reaction.

ResultsPlus
Examiner Comments

The candidate scores the mark for saying the KI reacts with manganate(VII), but the second mark is not awarded as they do not say all the manganate(VII) is used up.

Question 3 (a)(ii)

Although most candidates knew that the starch should be added just before the end point, many failed to state how that point could be recognised. A number gave explanations for the delay in the addition of the indicator, which was not required and a few candidates clearly misunderstood the question and stated the colour change at the end of the titration.



ethanedioate ions, as many MnO4 has reacted with I⁻ instead

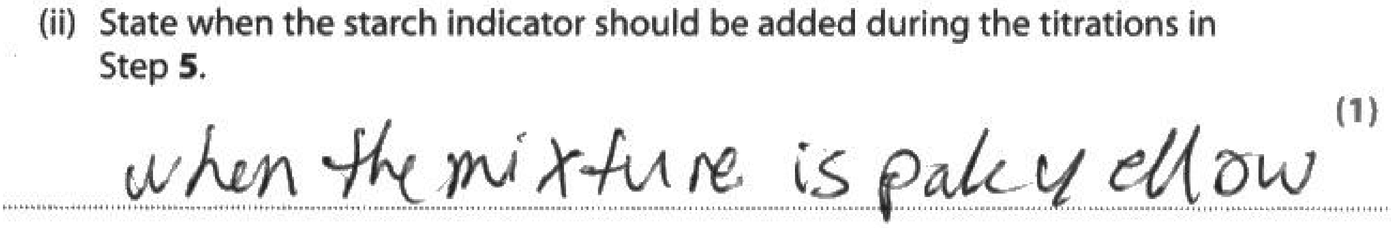
(ii) State when the starch indicator should be added during the titrations in Step **5**.

(1)

when the reaction mixture turns to a straw colour.

ResultsPlus Examiner Comments

Straw coloured is an alternative to pale yellow and both are acceptable answers.



(ii) State when the starch indicator should be added during the titrations in Step **5**.

(1)

when the mixture is pale yellow

ResultsPlus Examiner Comments

Pale yellow is an alternative to straw coloured and both are acceptable answers.

(ii) State when the starch indicator should be added during the titrations in Step **5**.

(1)

Starch indicator should be added just before the end-point.

ResultsPlus

Examiner Comments

Here the candidate knows that the starch should be added near the end point, but has not explained how that end point is detected.

Question 3 (b)(i)

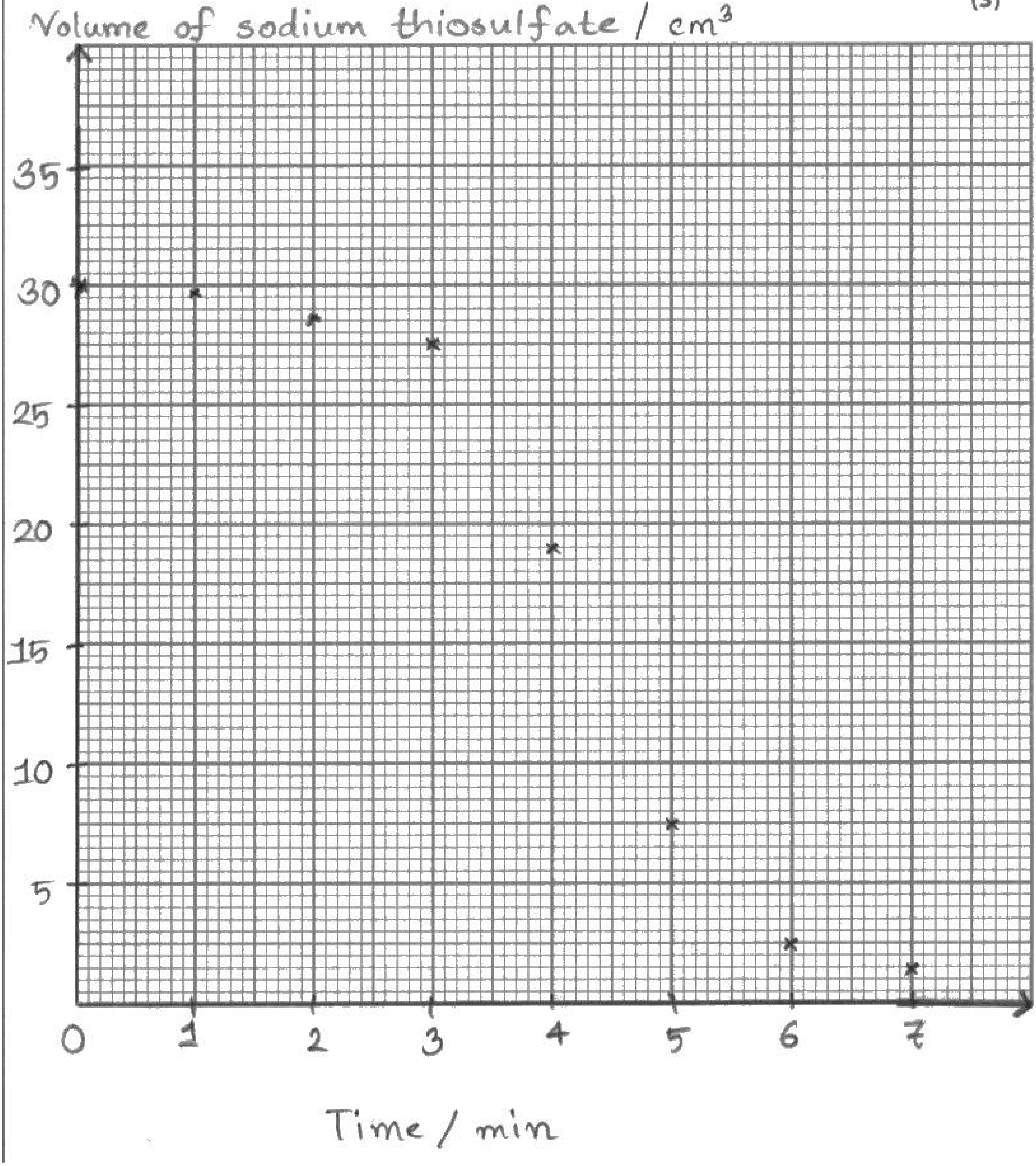
This graph question scored well with over half the candidates gaining full marks. Although the choice of scales was generally good, there were some awkward divisions seen that made plotting the points tricky and a number of non-linear scales were noted too. The lines of best-fit were well drawn, but a surprising number of candidates lost the mark because they used point-to-point plotting to join the points.

(b) A student's results are shown.

Time (t) / min	0	1	2	3	4	5	6	7
Volume of sodium thiosulfate / cm^3	30.00	29.80	28.60	27.50	19.00	7.50	2.50	1.50

(i) Plot a graph of volume of sodium thiosulfate against time.

(3)



ResultsPlus
Examiner Comments

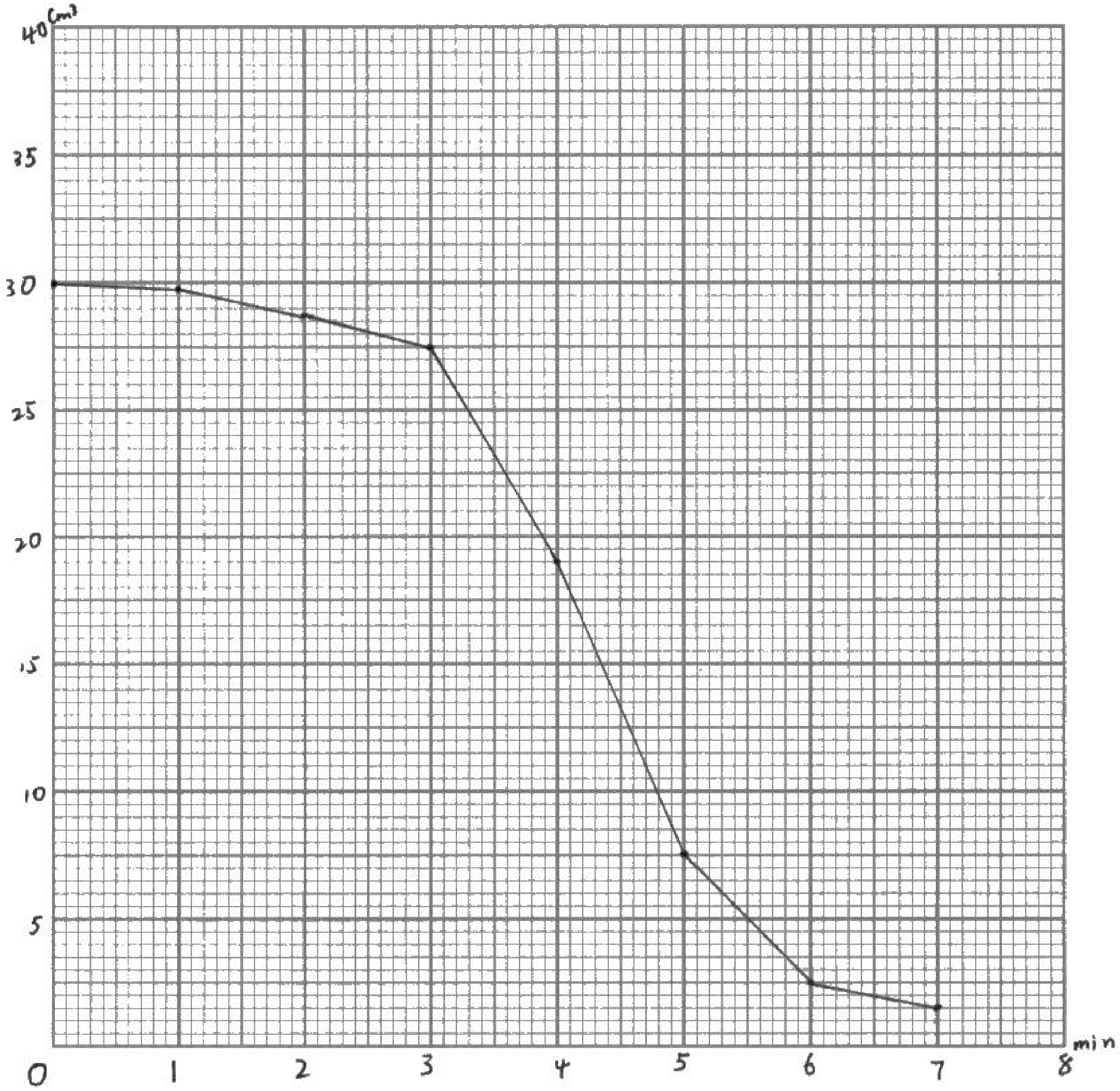
Here the candidate has chosen sensible scales and plotted the points correctly but has not drawn a curve of best-fit, so a mark is lost.

(b) A student's results are shown.

Time (t) / min	0	1	2	3	4	5	6	7
Volume of sodium thiosulfate / cm^3	30.00	29.80	28.60	27.50	19.00	7.50	2.50	1.50

(i) Plot a graph of volume of sodium thiosulfate against time.

(3)



ResultsPlus
Examiner Comments

Here the candidate has chosen sensible scales and plotted the points correctly. However, they have connected the points up dot-to-dot with a ruler instead of a curve of best-fit, so a mark is lost.

Question 3 (b)(ii)

The interpretation of the graph proved difficult for many candidates. A number missed out mentioning the slow start to the reaction and others talked about changes in gradient or volume instead of the rate.

(ii) Describe how the rate of reaction changes during the reaction.

(1)

at first, the rate is slow then it increases at 3 mins to 5 min then it starts slowing down again

ResultsPlus
Examiner Comments

Here the candidate correctly explains how the rate changes during the course of the reaction.

(ii) Describe how the rate of reaction changes during the reaction.

(1)

the rate of reaction is the gradient of the graph 'increases as the reaction proceeds as the gradient is getting more steep.

are Mn^{+2}

ResultsPlus
Examiner Comments

Here the candidate explains how the gradient is related to the rate of reaction but does not state how the rate changes with time, so no mark was scored.

This was quite a common error.

(ii) Describe how the rate of reaction changes during the reaction.

(1)

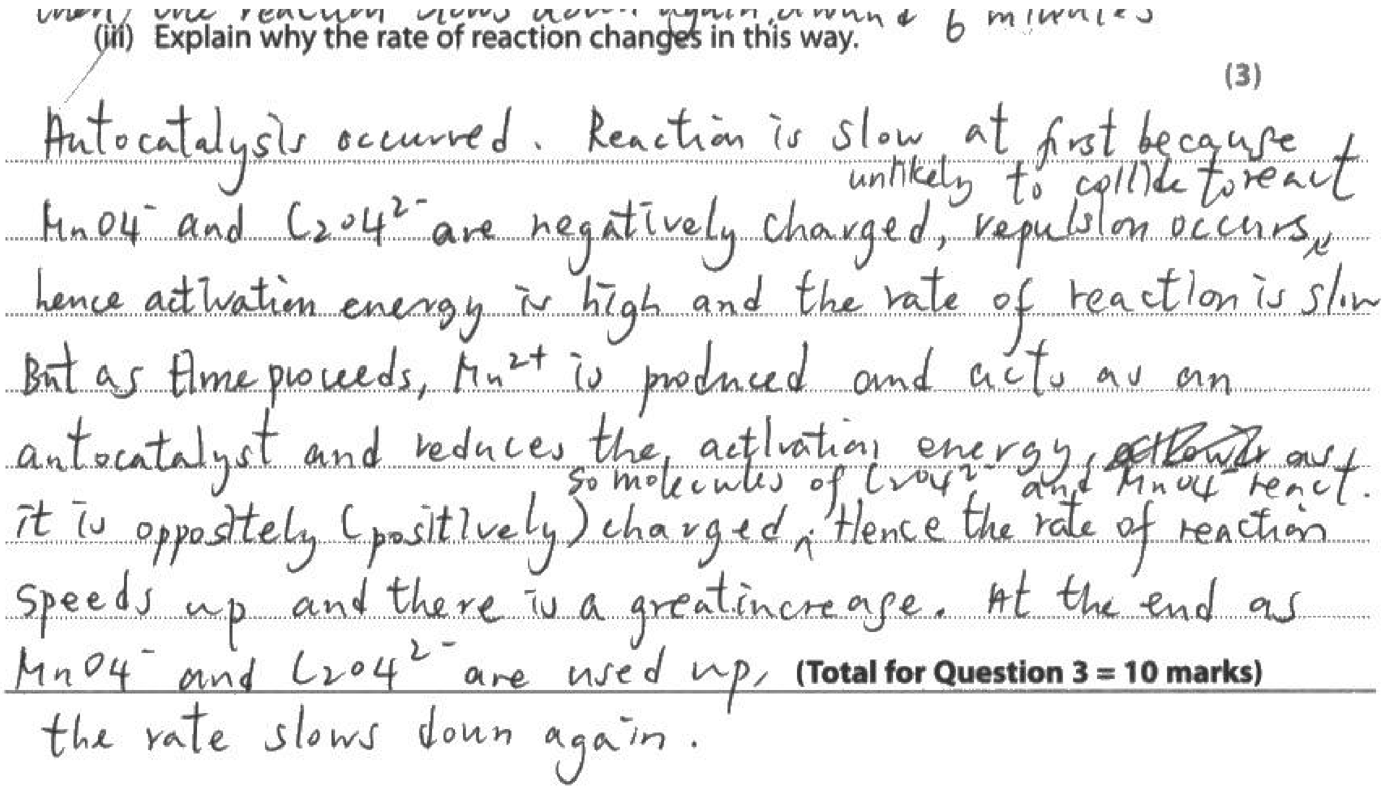
The rate of reaction is ~~[illegible]~~ fast at ~~[illegible]~~ the start of the reaction, then quickly slow down as the volume of sodium thiosulfate decreases.

ResultsPlus
Examiner Comments

Here the candidate has not mentioned the rate being slow at the start and so no mark is scored. This was a common error.

Question 3 (b)(iii)

Whilst there were many excellent answers to this question, it proved to be quite challenging to many candidates with over half of them scoring no marks. Those who were familiar with autocatalysis reactions usually identified the Mn^{2+} ions as the catalyst and then went on to explain why the reaction slowed at the end. But generally, there was a lot of confusion as many did not appreciate that the reaction being investigated was between manganate and ethanedioate ions and the thiosulfate titration was simply used to follow this reaction. This led to a number incorrectly stating it was the iodine or $S_4O_6^{2-}$ ions acting as a catalyst and the reaction slowed down because the thiosulphate was being used up.



6 minutes

(iii) Explain why the rate of reaction changes in this way.

(3)

Autocatalysis occurred. Reaction is slow at first because MnO_4^- and $C_2O_4^{2-}$ are negatively charged, repulsion occurs, unlikely to collide to react hence activation energy is high and the rate of reaction is slow. But as time proceeds, Mn^{2+} is produced and acts as an autocatalyst and reduces the activation energy, ~~allows~~ as it is oppositely (positively) charged, so molecules of $C_2O_4^{2-}$ and MnO_4^- react. Hence the rate of reaction speeds up and there is a great increase. At the end as MnO_4^- and $C_2O_4^{2-}$ are used up, the rate slows down again.

(Total for Question 3 = 10 marks)

ResultsPlus
Examiner Comments

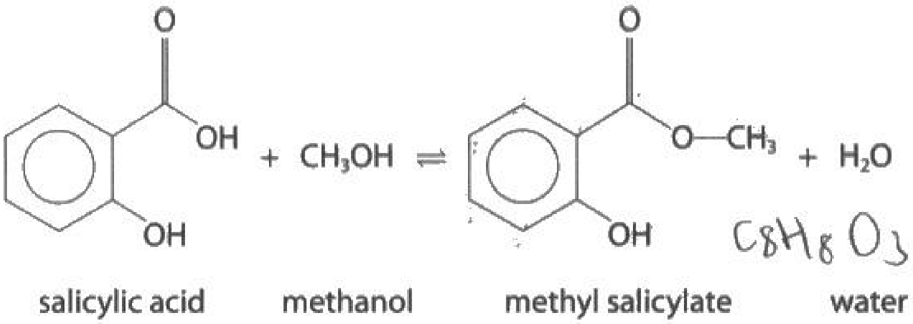
This is an excellent answer. The candidate has covered all three marking points and in addition, although not required, explained why the reaction between two negatively charged ions is slow at the start.

Question 4 (a)

Calculation errors were extremely rare in this question. Answers were usually logically laid out with a clear link to the molar ratio from the balanced equation. A small number of candidates worked out the excess volume of methanol used by using the density data and this novel solution also scored full marks.

4 This question is about the laboratory preparation of the ester methyl salicylate (oil of wintergreen).

The equation for this reaction is shown.



$C_8H_8O_3$

Procedure

Step **1** 20.0 g of salicylic acid and 100 cm^3 of methanol are placed into a round-bottomed flask.

Step **2** 15.0 cm^3 of concentrated sulfuric acid is added slowly, whilst swirling the flask.

Step **3** The mixture is heated gently under reflux for 45 minutes.

Step **4** After cooling, the mixture is poured into a separating funnel and about 50 cm^3 of iced water is added. The funnel is stoppered, shaken and allowed to settle. The aqueous layer is discarded.

Step **5** The organic layer is returned to the separating funnel and washed with 50 cm^3 of sodium carbonate solution.

Step **6** The aqueous layer is discarded leaving the crude methyl salicylate.

Step **7** The crude methyl salicylate is distilled and the fraction with the boiling temperature range 220–224 °C is collected.

Step **8** The pure methyl salicylate is transferred to a bottle and weighed.

(a) Show, by calculation, that the methanol is in excess in this preparation.

[M_r values: salicylic acid = 138.0 methanol = 32.0,
Density of methanol = 0.791 g cm^{-3}]

(3)

no. of moles of salicylic acid = $\frac{20.0}{138.0}$ = 0.145

no. of moles of methanol = $\frac{100 \times 0.791}{32.0}$ = 2.472

∵ 2.472 > 0.145

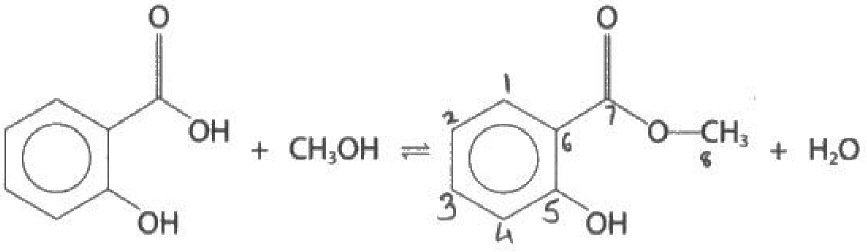
∴ methanol is in excess as the mole ratio of salicylic acid and methanol reacted should be in 1:1.

ResultsPlus
Examiner Comments

This is an excellent fully correct answer. The candidate has calculated the number of moles of methanol and salicylic acid and noted the 1:1 reacting ratio between them.

4 This question is about the laboratory preparation of the ester methyl salicylate (oil of wintergreen).

The equation for this reaction is shown.



salicylic acid   methanol   methyl salicylate   water

Procedure

Step 1 20.0 g of salicylic acid and 100 cm^3 of methanol are placed into a round-bottomed flask.

Step 2 15.0 cm^3 of concentrated sulfuric acid is added slowly, whilst swirling the flask.

Step 3 The mixture is heated gently under reflux for 45 minutes.

Step 4 After cooling, the mixture is poured into a separating funnel and about 50 cm^3 of iced water is added. The funnel is stoppered, shaken and allowed to settle. The aqueous layer is discarded.

Step 5 The organic layer is returned to the separating funnel and washed with 50 cm^3 of sodium carbonate solution.

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(a) Show, by calculation, that the methanol is in excess in this preparation.

[M_r values: salicylic acid = 138.0   methanol = 32.0,
Density of methanol = 0.791 g cm^{-3}]

(3)

mass of methanol = $100 cm^3 \times 0.791 g\ cm^{-3}$

no. of mole of methanol $= \frac{79.1 g}{32}$

$= 2.47 mol$

no. of mol of salicylic acid $= \frac{20.0}{138.0}$

$= 0.145 mol$

$\frac{2.47}{0.145} = 17$

methanol : salicylic acid

17 : 1

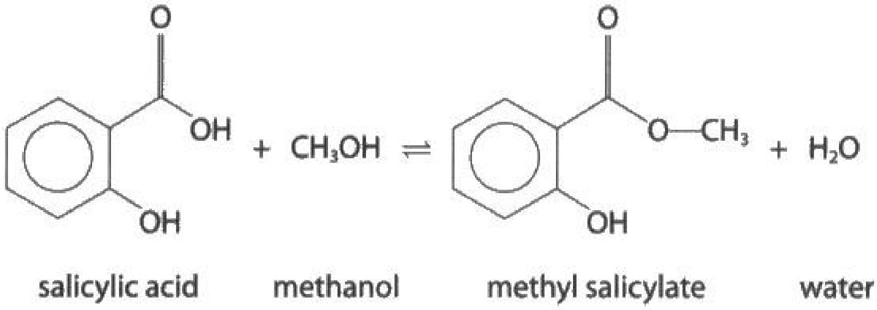
∴ methanol is in excess shown

ResultsPlus
Examiner Comments

The candidate has calculated the number of moles of methanol and salicylic acid and shown that the methanol is in excess by calculating the ratio of 17:1. This is a fully correct answer.

4 This question is about the laboratory preparation of the ester methyl salicylate (oil of wintergreen).

The equation for this reaction is shown.



Procedure

Step **1** 20.0 g of salicylic acid and 100 cm^3 of methanol are placed into a round-bottomed flask.

Step **2** 15.0 cm^3 of concentrated sulfuric acid is added slowly, whilst swirling the flask.

Step **3** The mixture is heated gently under reflux for 45 minutes.

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Step **8** The pure methyl salicylate is transferred to a bottle and weighed.

(a) Show, by calculation, that the methanol is in excess in this preparation.

[M_r values: salicylic acid = 138.0 methanol = 32.0,
Density of methanol = 0.791 g cm^{-3}]

(3)

$\frac{20}{138} = \frac{10}{69} = n(\text{salicylic acid}) = n(\text{methanol})$

1:1

$\text{mass} = \frac{10}{69} \times 32 = \frac{320}{69}$

$\rho = \frac{\text{mass}}{\text{volume}} = \frac{\left(\frac{320}{69}\right)}{x} = 0.7$

$\left(\frac{320}{69}\right) \div 0.791 = 5.86\text{ cm}$ needed

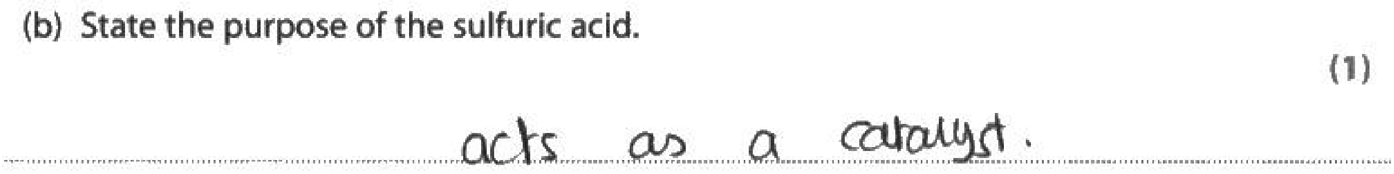
so exc

ResultsPlus
Examiner Comments

Here the candidate has come up with a very novel solution. They have calculated the mass of methanol required to react with the salicylic acid and then using the density of methanol calculated the volume required, 5.86 cm^3. The volume of methanol used is 100 cm^3, therefore the methanol has to be in excess and so all three marks are scored.

Question 4 (b)

The majority of candidates knew the sulfuric acid was a catalyst for this reaction, but a few did not score as they simply said it increased the rate.



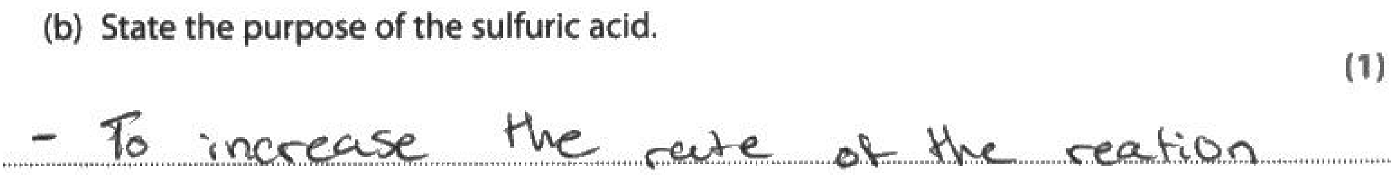
(b) State the purpose of the sulfuric acid.

(1)

acts as a catalyst.

ResultsPlus Examiner Comments

This correct answer was seen from the majority of the candidates.



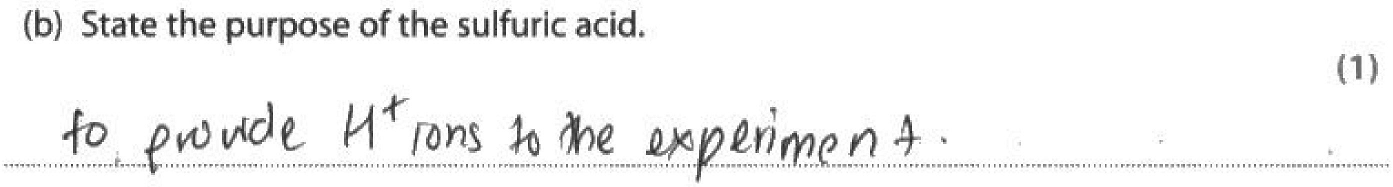
(b) State the purpose of the sulfuric acid.

(1)

- To increase the rate of the reation

ResultsPlus Examiner Comments

The sulfuric acid does increase the rate of reaction, but by not mentioning the role of the sulfuric acid as a catalyst the candidate has not scored the mark. This was a common error.



(b) State the purpose of the sulfuric acid.

(1)

to provide H^+ ions to the experiment.

ResultsPlus Examiner Comments

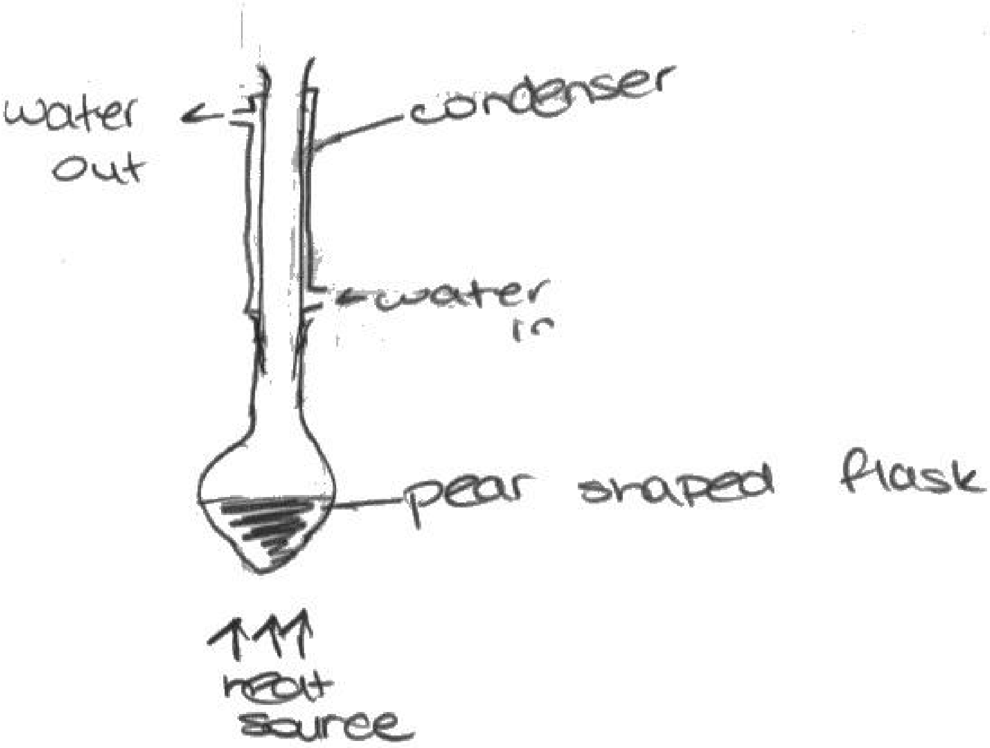
The sulfuric acid does provide H^+ ions, but this is insufficient for the mark to be scored and was another common wrong answer.

Question 4 (c)

The quality of diagrams was generally very poor as it was often hard to identify what apparatus the candidate had drawn. A few candidates just drew a flask with no condenser. Where the condenser was shown, the water flow was usually correct but often the top of the condenser was sealed. Many candidates drew a single piece of apparatus and a lack of heat or distillation apparatus were also noted.

(c) Draw a diagram of the apparatus used in Step **3** when the mixture is heated under reflux.

(3)

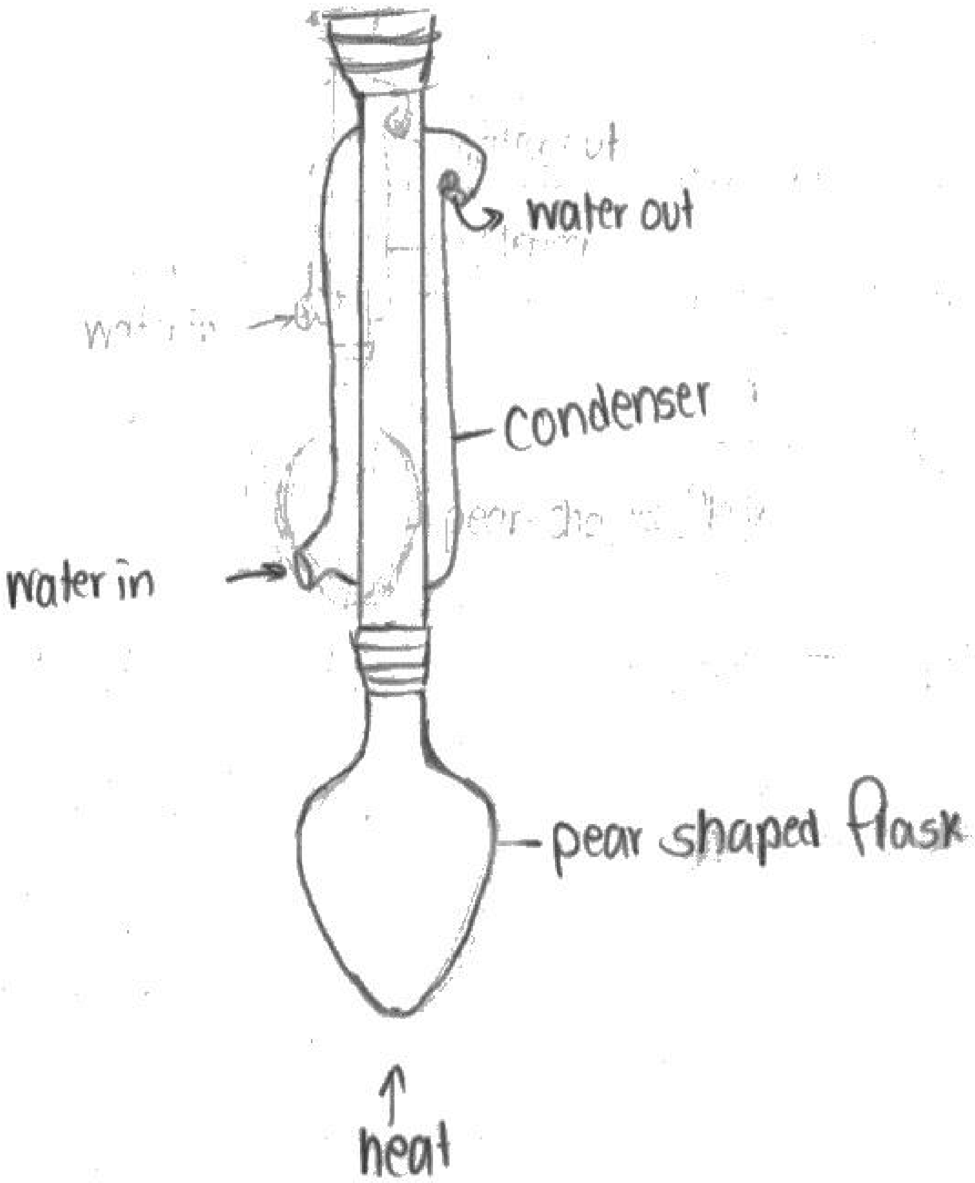


ResultsPlus
Examiner Comments

Here the candidate has scored all 3 marks. The thicker lines between the condenser and pear shaped flask make it clear that there are two pieces of apparatus.

(c) Draw a diagram of the apparatus used in Step 3 when the mixture is heated under reflux.

(3)



ResultsPlus
Examiner Comments

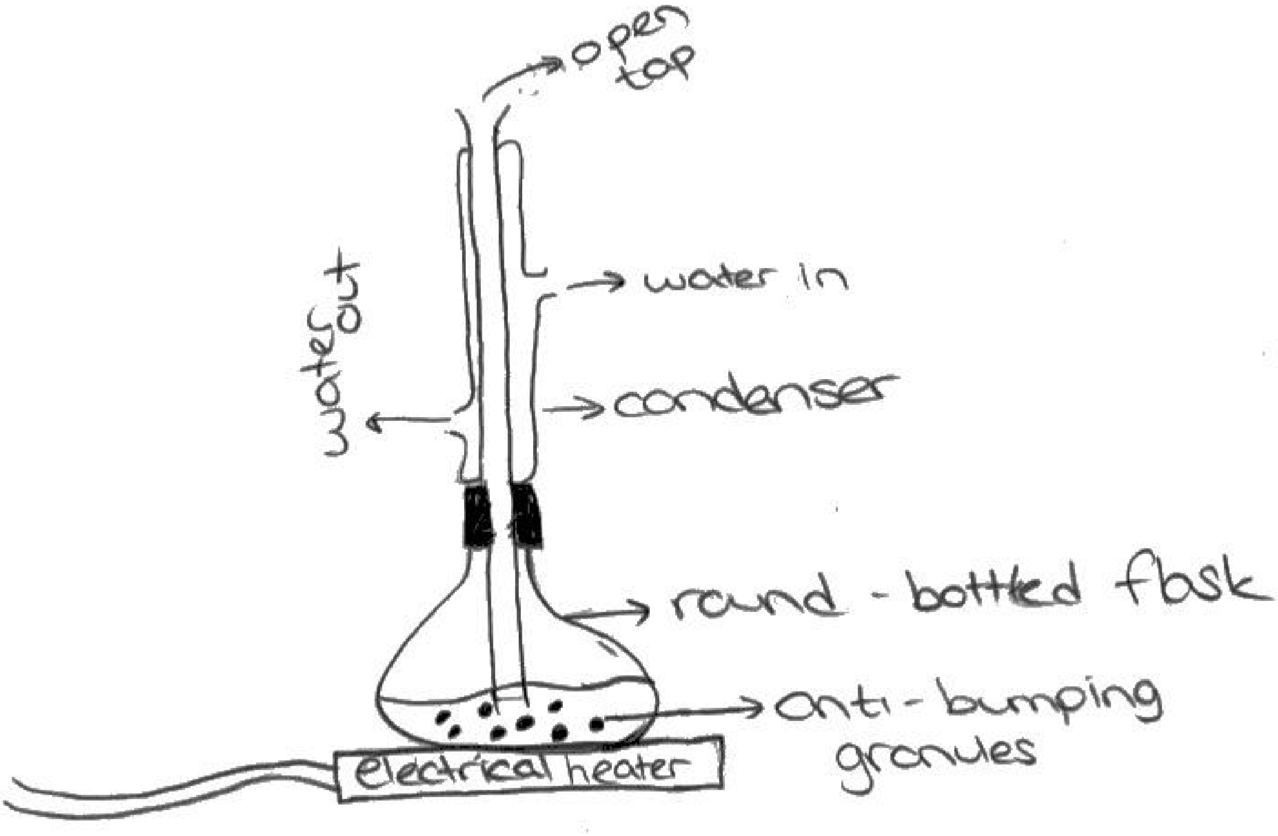
The quality of the diagram is good as it is clear what the apparatus is and it is fully labelled. However, there is no liquid in the flask, and it appears that the condenser is sealed at the top. So the candidate has only scored one mark for the condenser with the correct water flow.

ResultsPlus
Examiner Tip

Always put some liquid in the flask when drawing refluxing or distillation apparatus.

(c) Draw a diagram of the apparatus used in Step **3** when the mixture is heated under reflux.

(3)



ResultsPlus
Examiner Comments

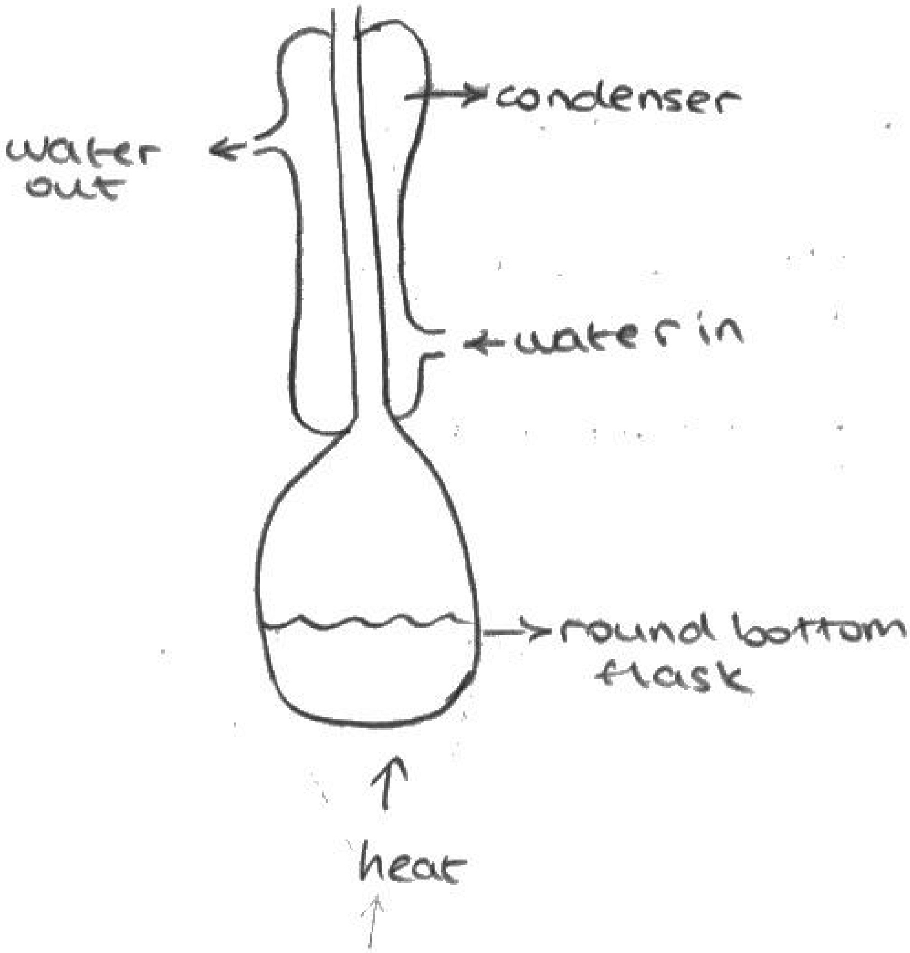
Here the candidate only scores 2 marks as the water flow in the condenser is the wrong way round.

ResultsPlus
Examiner Tip

Remember the water will always go in at the bottom of the condenser to prevent air bubbles forming.

(c) Draw a diagram of the apparatus used in Step **3** when the mixture is heated under reflux.

(3)



ResultsPlus
Examiner Comments

This diagram shows the flask and condenser as one piece of apparatus, which was a common error.

ResultsPlus
Examiner Tip

When drawing reflux apparatus make sure it is clear that the flask and condenser are two separate pieces of apparatus.

Question 4 (d)

Those candidates who had used a separating funnel for this purpose and were able to explain how the washing took place, were in a minority. The majority simply described how the sodium carbonate solution was added and the aqueous layer removed.

(d) Describe how to wash the organic layer with sodium carbonate solution in Step **5**.

(2)

· ADD sodium carbonate solution to the separating funnel and place a stopper at the top.

· Invert the separating funnel gently and invert again and remove the stopper to release the pressure. Repeat a few times until 2 layers form.

ResultsPlus
Examiner Comments

This is an excellent answer where the candidate explains how the separating funnel is used and how the pressure is released.

(d) Describe how to wash the organic layer with sodium carbonate solution in Step **5**.

(2)

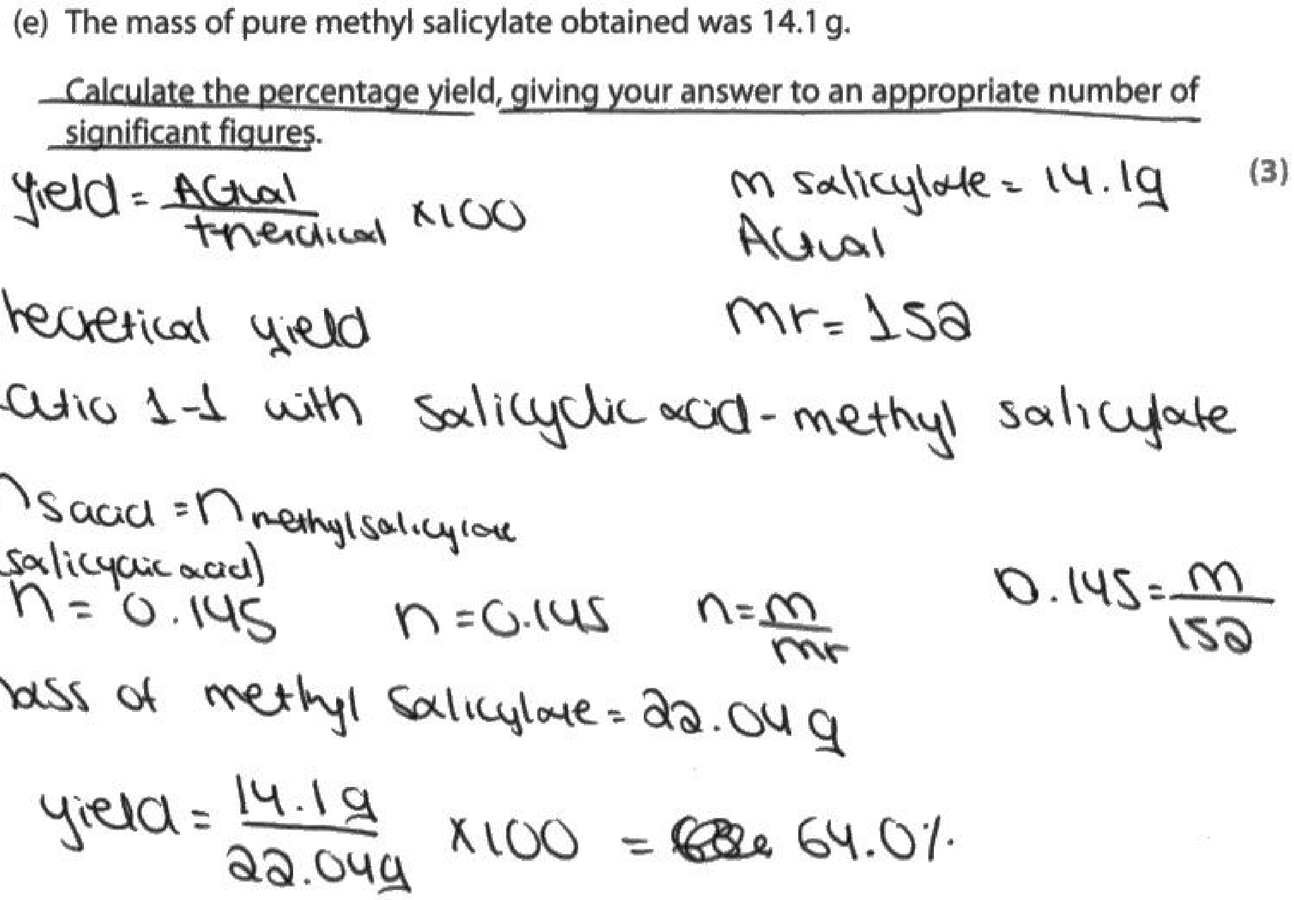
Add the solution to the seperating funnel & shake it ~~thro~~ thoroughly. Allow the aqueos and crude methyl salicylate layer to seperate.

ResultsPlus
Examiner Comments

The candidate has scored the first mark for adding the sodium carbonate solution to the separating funnel and shaking. However, they have made no reference to how the carbon dioxide or pressure is released.

Question 4 (e)

Although the majority of candidates scored full marks on this calculation, occasionally too many significant figures in the final answer meant the last mark was not awarded. For those who made a mistake with the formula mass of methyl salicylate, most still scored some marks through transfer error.



(e) The mass of pure methyl salicylate obtained was 14.1 g.

Calculate the percentage yield, giving your answer to an appropriate number of significant figures.

(3)

$\text{yield} = \frac{\text{Actual}}{\text{theoretical}} \times 100$

m salicylate = 14.1g
Actual

Theoretical yield

mr = 152

Ratio 1-1 with salicyclic acid - methyl salicylate

$n_{\text{sacid}} = n_{\text{methylsalicylate}}$
(salicyaic acid)
n = 0.145

n = 0.145

$n = \frac{m}{mr}$

$0.145 = \frac{m}{152}$

mass of methyl salicylate = 22.04 g

$\text{yield} = \frac{14.1\text{g}}{22.04\text{g}} \times 100 =$ 64.0%

ResultsPlus
Examiner Comments

The candidate has calculated the molar mass of methyl salicylate and used it correctly to calculate the percentage yield. Their final answer is to 3 significant figures, so full marks are scored.

(e) The mass of pure methyl salicylate obtained was 14.1 g.

Calculate the percentage yield, giving your answer to an appropriate number of significant figures.

(3)

$\frac{20}{138} = 0.1449$ mol

~~[illegible]~~

~~[illegible]~~

$\frac{14.1}{152} = 0.0927631$

64.02%

ResultsPlus
Examiner Comments

The candidate has calculated the molar mass of methyl salicylate and used it correctly to calculate the percentage yield. However, their final answer is to 4 significant figures so one mark is lost.

ResultsPlus
Examiner Tip

When doing calculations, think carefully about the final number of significant figures. Look for the minimum number of significant figures in the data given and use this number of significant figures in your final answer.

(e) The mass of pure methyl salicylate obtained was 14.1 g.

Calculate the percentage yield, giving your answer to an appropriate number of significant figures.

(3)

M_r methyl salicylate = 140

$n = \frac{m}{M}$ $\quad m = Mn$

$= 140 \times 0.1449 = 20.286$

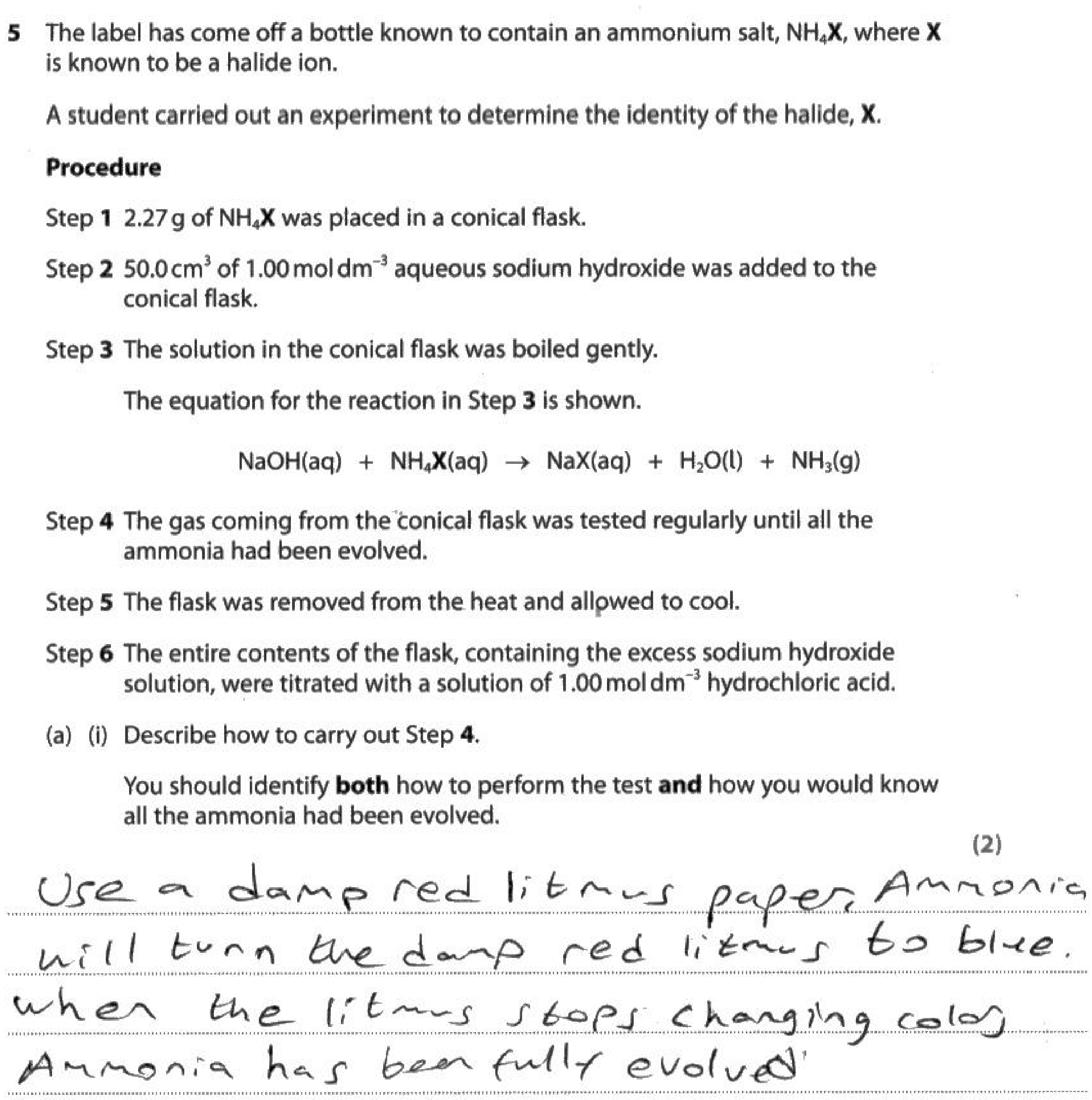
$$\frac{14.1}{20.286} \times 100 = 69.5\%$$

ResultsPlus
Examiner Comments

The candidate has made a mistake with molar mass of methyl salicylate, it should be 152 and they have calculated it as 140. However, they have used the mass correctly and their final answer is to 2 or 3 significant figures, so only one mark is lost.

Question 5 (a)(i)

The ammonia test had been learned by the majority of candidates and there were some excellent answers from candidates who explained how they would keep repeating the test until the red litmus papers stopped turning blue. A number described the alternative test using hydrochloric acid solution, but many omitted the fact that acid had to be concentrated for the test to work. Occasionally, candidates suggested heating until the pungent odour could no longer be detected or just heating to constant mass, neither of which gained any credit.



5 The label has come off a bottle known to contain an ammonium salt, NH_4**X**, where **X** is known to be a halide ion.

A student carried out an experiment to determine the identity of the halide, **X**.

Procedure

Step **1** 2.27 g of NH_4**X** was placed in a conical flask.

Step **2** 50.0 cm^3 of 1.00 mol dm^{-3} aqueous sodium hydroxide was added to the conical flask.

Step **3** The solution in the conical flask was boiled gently.

The equation for the reaction in Step **3** is shown.

$$NaOH(aq) + NH_4X(aq) \rightarrow NaX(aq) + H_2O(l) + NH_3(g)$$

Step **4** The gas coming from the conical flask was tested regularly until all the ammonia had been evolved.

Step **5** The flask was removed from the heat and allowed to cool.

Step **6** The entire contents of the flask, containing the excess sodium hydroxide solution, were titrated with a solution of 1.00 mol dm^{-3} hydrochloric acid.

(a) (i) Describe how to carry out Step **4**.

You should identify **both** how to perform the test **and** how you would know all the ammonia had been evolved.

(2)

Use a damp red litmus paper. Ammonia will turn the damp red litmus to blue. when the litmus stops changing colour Ammonia has been fully evolved

ResultsPlus
Examiner Comments

This is an excellent answer as both the test for ammonia and an explanation of how you know when all the ammonia has been evolved are clear.

5 The label has come off a bottle known to contain an ammonium salt, $NH_4\mathbf{X}$, where **X** is known to be a halide ion.

A student carried out an experiment to determine the identity of the halide, **X**.

Procedure

Step **1** 2.27 g of $NH_4\mathbf{X}$ was placed in a conical flask.

Step **2** 50.0 cm^3 of 1.00 mol dm^{-3} aqueous sodium hydroxide was added to the conical flask.

Step **3** The solution in the conical flask was boiled gently.

The equation for the reaction in Step **3** is shown.

$$NaOH(aq) + NH_4\mathbf{X}(aq) \rightarrow NaX(aq) + H_2O(l) + NH_3(g)$$

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(a) (i) Describe how to carry out Step **4**.

You should identify **both** how to perform the test **and** how you would know all the ammonia had been evolved.

(2)

Add sodium hydroxide solution to the salt and check for cold, damp smell of ammonia gas. Make sure to perform test in fume cupboard. When all the gas is evolved, after several hours, the smell will wear off.

ResultsPlus
Examiner Comments

Although the candidate knows ammonia has a pungent smell, this is not a safe way of testing and no marks are scored.

5 The label has come off a bottle known to contain an ammonium salt, $NH_4\mathbf{X}$, where **X** is known to be a halide ion.

A student carried out an experiment to determine the identity of the halide, **X**.

Procedure

Step 1 2.27 g of $NH_4\mathbf{X}$ was placed in a conical flask.

Step 2 50.0 cm^3 of 1.00 mol dm^{-3} aqueous sodium hydroxide was added to the conical flask.

Step 3 The solution in the conical flask was boiled gently.

The equation for the reaction in Step **3** is shown.

0.0134,

$$NaOH(aq) + NH_4\mathbf{X}(aq) \rightarrow NaX(aq) + H_2O(l) + NH_3(g)$$

0.05 ×7 0.05,

Step 4 The gas coming from the conical flask was tested regularly until all the ammonia had been evolved.

0.0134

Step 5 The flask was removed from the heat and allowed to cool.

Step 6 The entire contents of the flask, containing the excess sodium hydroxide solution, were titrated with a solution of 1.00 mol dm^{-3} hydrochloric acid.

(a) (i) Describe how to carry out Step **4**.

You should identify **both** how to perform the test **and** how you would know all the ammonia had been evolved.

(2)

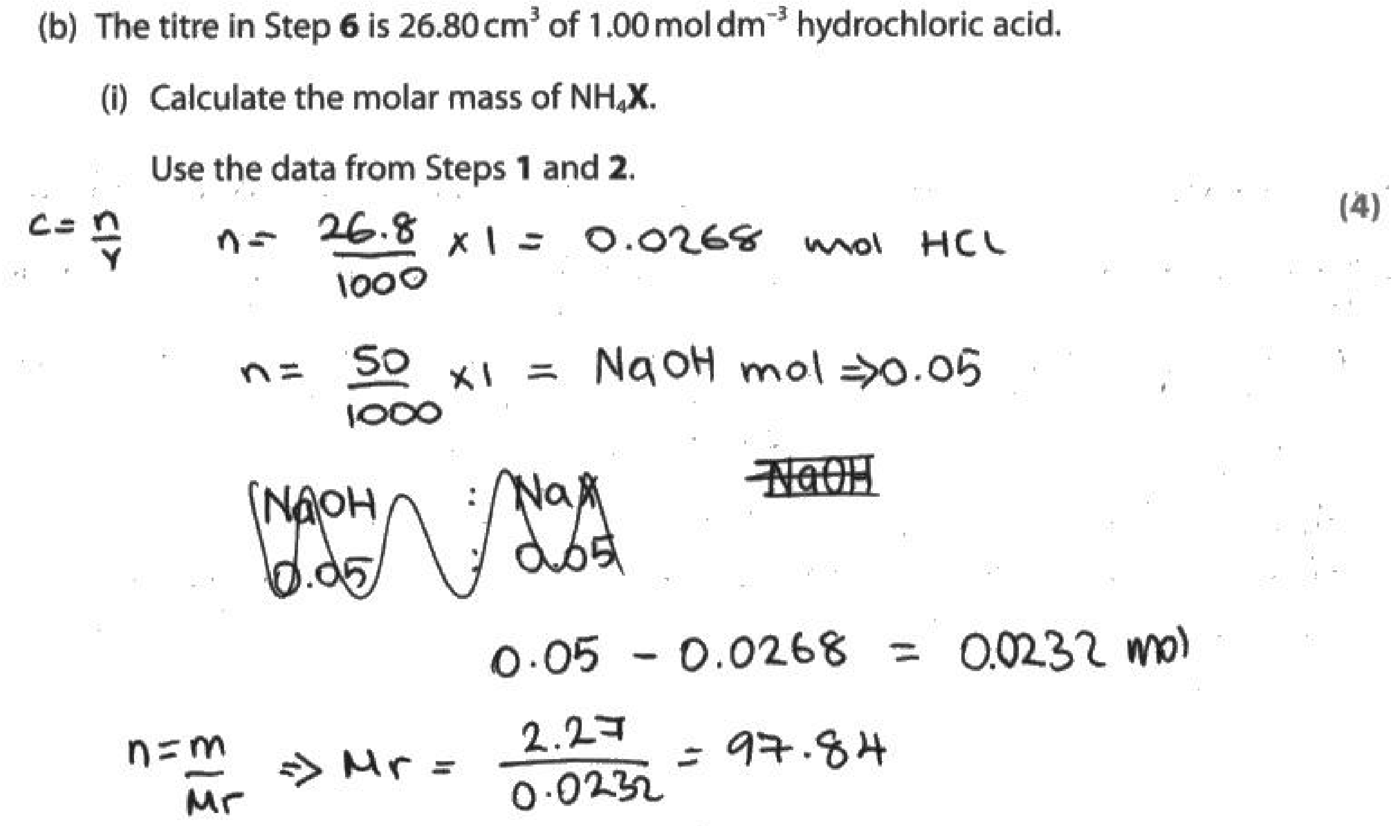
Dip a cotton wool ~~in to~~ into hydrochloric acid then hold the cotton wool towards the gas, a white smoke will be formed which is NH_4Cl. Colect the gas, repeat testing until no white smoke forms which means no more gas is evolving.

ResultsPlus
Examiner Comments

The candidate has used the alternative test for ammonia using hydrochloric acid. Unfortunately, they have not said that the acid has to be concentrated, so a mark is lost.

Question 5 (b)

The majority of candidates scored full marks for the calculation in part (i). Where mistakes were made, the most common was dividing by the wrong number of moles, usually 0.05 which gave the wrong molar mass, but allowed access to transfer error marks. With a correct calculation in (i), most candidate were able to identify Br as the halide in part (ii). The chemical test for the halide in part (iii) was also very well known, although occasionally a mark was lost as the candidates did not include nitric acid with the silver nitrate solution.



(b) The titre in Step **6** is 26.80 cm^3 of 1.00 mol dm^{-3} hydrochloric acid.

(i) Calculate the molar mass of NH_4**X**.

Use the data from Steps **1** and **2**.

(4)

$c = \frac{n}{V}$ $n = \frac{26.8}{1000} \times 1 = 0.0268$ mol HCl

$n = \frac{50}{1000} \times 1 =$ NaOH mol ⇒ 0.05

NaOH : NaX   ~~NaOH~~

0.05 : 0.05

0.05 − 0.0268 = 0.0232 mol

$n = \frac{m}{Mr} \Rightarrow Mr = \frac{2.27}{0.0232} = 97.84$

(ii) Identify the halide **X**, present in NH_4**X**, using your answer to (b)(i).

(2)

$97.84 - 18 = 79.84 \Rightarrow \boxed{Br}$

(iii) Give a chemical test, with the expected result, to confirm the identity of the halide ion in a sample of NH_4**X**.

(2)

Silver nitrate and nitric acid is added and a cream precipitate that dissolves in concentrated ammonia forms.

ResultsPlus
Examiner Comments

This is an excellent answer. The molar mass is correct in part (i), 18 has been subtracted and Br identified in part (ii) and the test for the bromide ion has the correct reagents in part (iii).

(b) The titre in Step **6** is 26.80 cm^3 of 1.00 $mol\,dm^{-3}$ hydrochloric acid.

(i) Calculate the molar mass of NH_4**X**.

Use the data from Steps **1** and **2**.

(4)

$n_{NaOH} = 1 \times \frac{50}{1000} = 0.05 mol$

$n_{titre} = 1 \times \frac{26.80}{1000} = 0.0268 mol$

$\frac{0.05}{0.0268} = 2$

$n_{titre} : n_{NaOH}$
1 : 2
0.0268 : ~~0.0268~~
0.05 mol

AFM = ~~0.05~~ $\frac{2.27}{0.05} = 45.4$

ResultsPlus
Examiner Comments

This was quite a common error where the candidate has used the wrong number of moles to calculate the molar mass. However, only one mark was lost in part (i).

(ii) Identify the halide **X**, present in NH_4**X**, using your answer to (b)(i).

(2)

Molar mass of X = 97.8 - 14 - 1×4 = 79.8 //

[illegible]

∴ Halide X is Br //

(iii) Give a chemical test, with the expected result, to confirm the identity of the halide ion in a sample of NH_4**X**.

(2)

Add $AgNO_3$ and dilute HCl into the sample of NH_4X. If the halide ion X is Br, there will have cream precipitate formed.

ResultsPlus
Examiner Comments

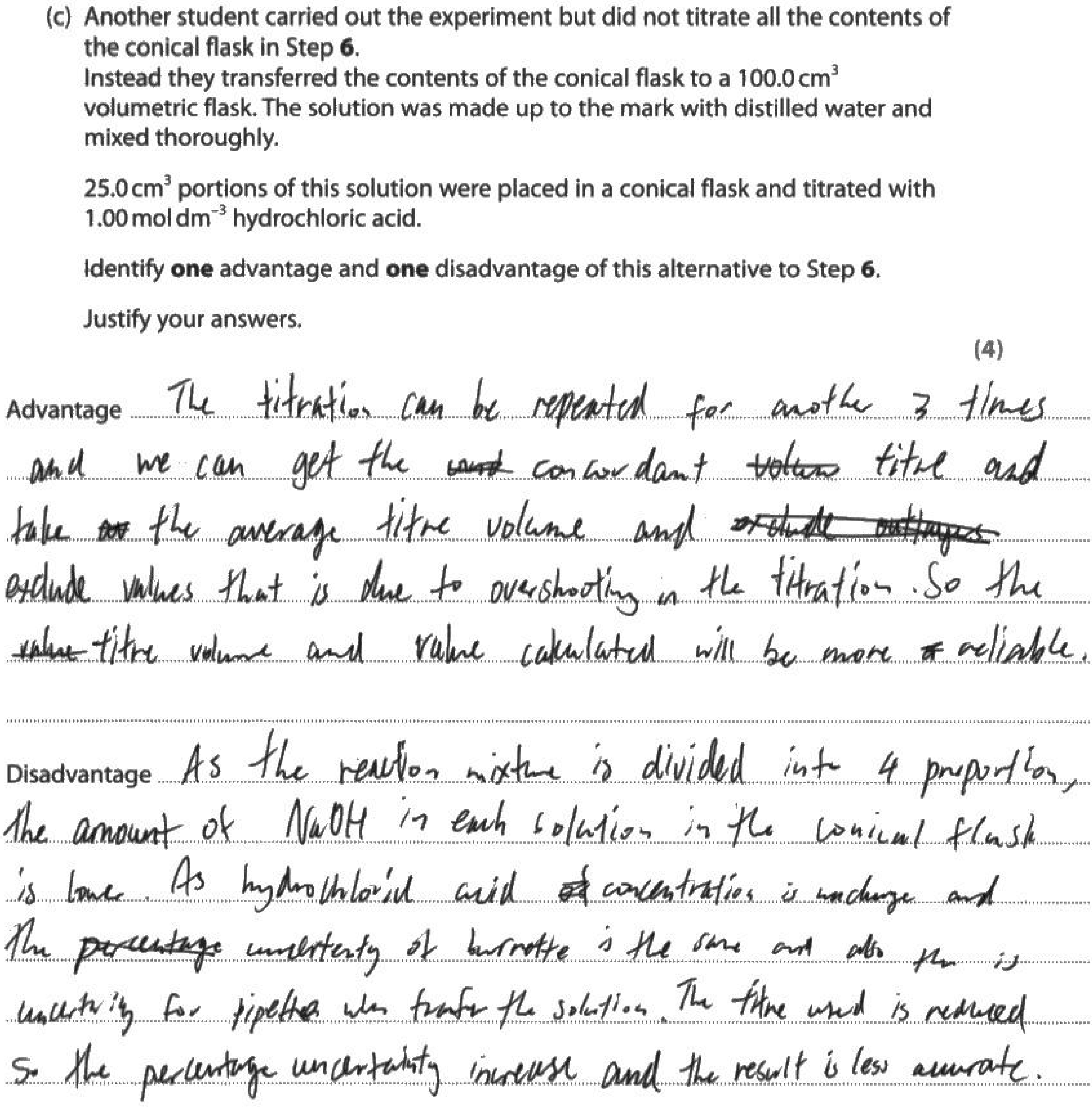
In part(iii), the candidate has used the wrong acid so only one mark is scored. Many candidates lost a mark here by using no acid or just saying acidified instead of nitric acid which is the only acid that will score.

ResultsPlus
Examiner Tip

When testing for halides with silver nitrate solution, you cannot use hydrochloric acid as it contains a halide so will always give a positive test.

Question 5 (c)

This last question was the most challenging on the paper and proved to be a very good discriminator. Some responses showed an excellent appreciation of the advantage of obtaining concordant results through repeated titrations and the disadvantage of a greater percentage uncertainty with a smaller titre. However, these were very much in the minority and there was a wide range of wrong answers. Incorrect advantages included the increased safety and lower cost of using a less hydrochloric acid solution and the shorter time for the titrations. Disadvantages included more time and many talked about transfer losses when making up the solution in a volumetric flask.



(c) Another student carried out the experiment but did not titrate all the contents of the conical flask in Step **6**.
Instead they transferred the contents of the conical flask to a 100.0 cm^3 volumetric flask. The solution was made up to the mark with distilled water and mixed thoroughly.

25.0 cm^3 portions of this solution were placed in a conical flask and titrated with 1.00 mol dm^{-3} hydrochloric acid.

Identify **one** advantage and **one** disadvantage of this alternative to Step **6**.

Justify your answers.

(4)

Advantage The titration can be repeated for another 3 times and we can get the ~~[illegible]~~ concordant ~~volume~~ titre and take ~~on~~ the average titre volume and ~~exclude outliers~~ exclude values that is due to overshooting in the titration. So the ~~value~~ titre volume and value calculated will be more ~~r~~ reliable.

Disadvantage As the reaction mixture is divided into 4 proportion, the amount of NaOH in each solution in the conical flask is lower. As hydrochloric acid ~~of~~ concentration is unchange and the ~~percentage~~ uncertainty of burrette is the same and also there is uncertainty for pipette when transfer the solution. The titre used is reduced so the percentage uncertainty increase and the result is less accurate.

ResultsPlus
Examiner Comments

This is an excellent answer with all 4 scoring points made.

(c) Another student carried out the experiment but did not titrate all the contents of the conical flask in Step **6**.
Instead they transferred the contents of the conical flask to a 100.0 cm^3 volumetric flask. The solution was made up to the mark with distilled water and mixed thoroughly.

25.0 cm^3 portions of this solution were placed in a conical flask and titrated with 1.00 mol dm^{-3} hydrochloric acid.

Identify **one** advantage and **one** disadvantage of this alternative to Step **6**.

Justify your answers.

(4)

Advantage Gives more accurate results, as the titre will be smaller and easier to manage and read, So more precise

Disadvantage - May not be accurate, as transfer errors could occur and some solution may remain in the transfer instrument (pipette) So titre decreases

ResultsPlus
Examiner Comments

Here the candidate has incorrectly suggested that having a smaller titre is an advantage. Their disadvantage of transfer loss is ignored, but they do manage to score a mark for the smaller titre.

Paper Summary

In order to improve their performance, candidates should:

- Always read the information in the question carefully, noting any instructions in bold type.
- Make sure they learn and understand the procedures in the core practicals.
- Understand how organic apparatuses work and practise drawing them.
- Learn the qualitative tests to identify organic groups and inorganic ions.
- Show working when carrying out calculations and think carefully about units, significant figures and rounding.
- Understand how to minimise the sources of measurement uncertainty when carrying out titrations.
- When drawing graphs ensure that half of the grid is used, the axes are labelled and the appropriate line of best-fit is drawn.