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Examiners' Report
January 2011

GCE Chemistry 6 (INT) 6CH08 01

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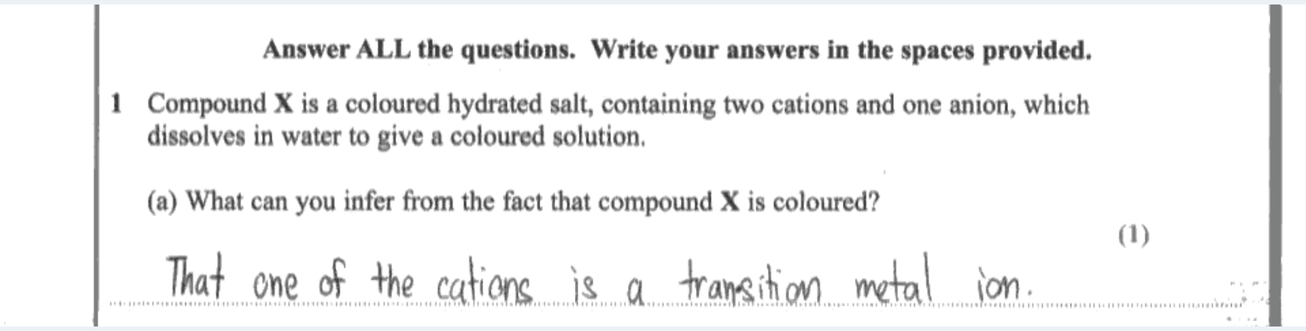
Introduction

This paper requires candidates to think as if they are in a laboratory environment. The emphasis is on what they would see during experiments and in producing logical deductions from this evidence.

Question 1(a)

Many candidates knew that a transition metal ion was present in X or that it contained a transition metal. The statement that ‘X **is** a transition metal’ was not credited since X is not the metal itself.



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

1 Compound **X** is a coloured hydrated salt, containing two cations and one anion, which dissolves in water to give a coloured solution.

(a) What can you infer from the fact that compound **X** is coloured?

(1)

That one of the cations is a transition metal ion.

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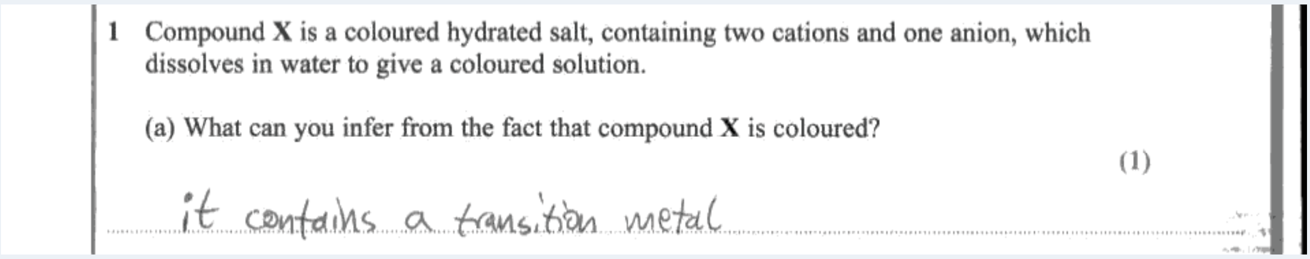
Examiner Comments

The candidate clearly understands that transition metal ions cause the colour - and perhaps has read the whole question and therefore knows that there might be other cations present as well.



1 Compound **X** is a coloured hydrated salt, containing two cations and one anion, which dissolves in water to give a coloured solution.

(a) What can you infer from the fact that compound **X** is coloured?

(1)

it contains a transition metal

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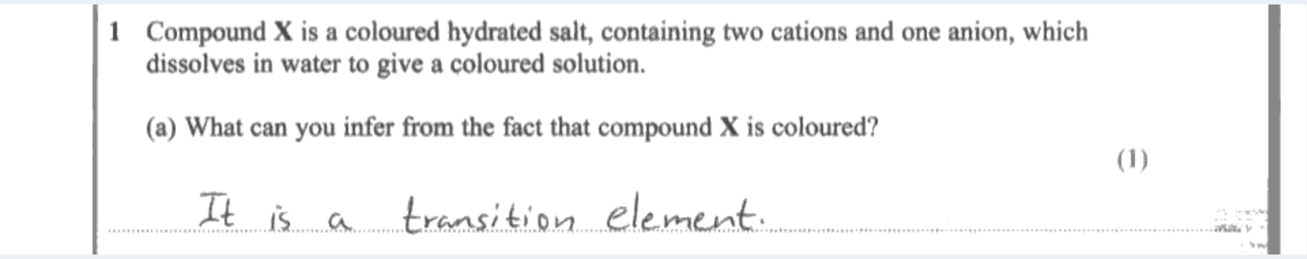
Examiner Comments

This just about scores - the candidate doesn't say that X is a transition metal.



1 Compound **X** is a coloured hydrated salt, containing two cations and one anion, which dissolves in water to give a coloured solution.

(a) What can you infer from the fact that compound **X** is coloured?

(1)

It is a transition element.

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Examiner Comments

X is not a transition element - it is a compound of a transition element.

Question 1(b)

Virtually every candidate knew the flame colour for potassium, even though many ignored the information later on in the question and abandoned it completely in their suggested formula for X in their answer to 1d.

Question 1(c)(d)

In 1(c)(i): The identity of $BaSO_4$ was widely known. For the weaker candidate it was often the only correct answer in question 1(c).

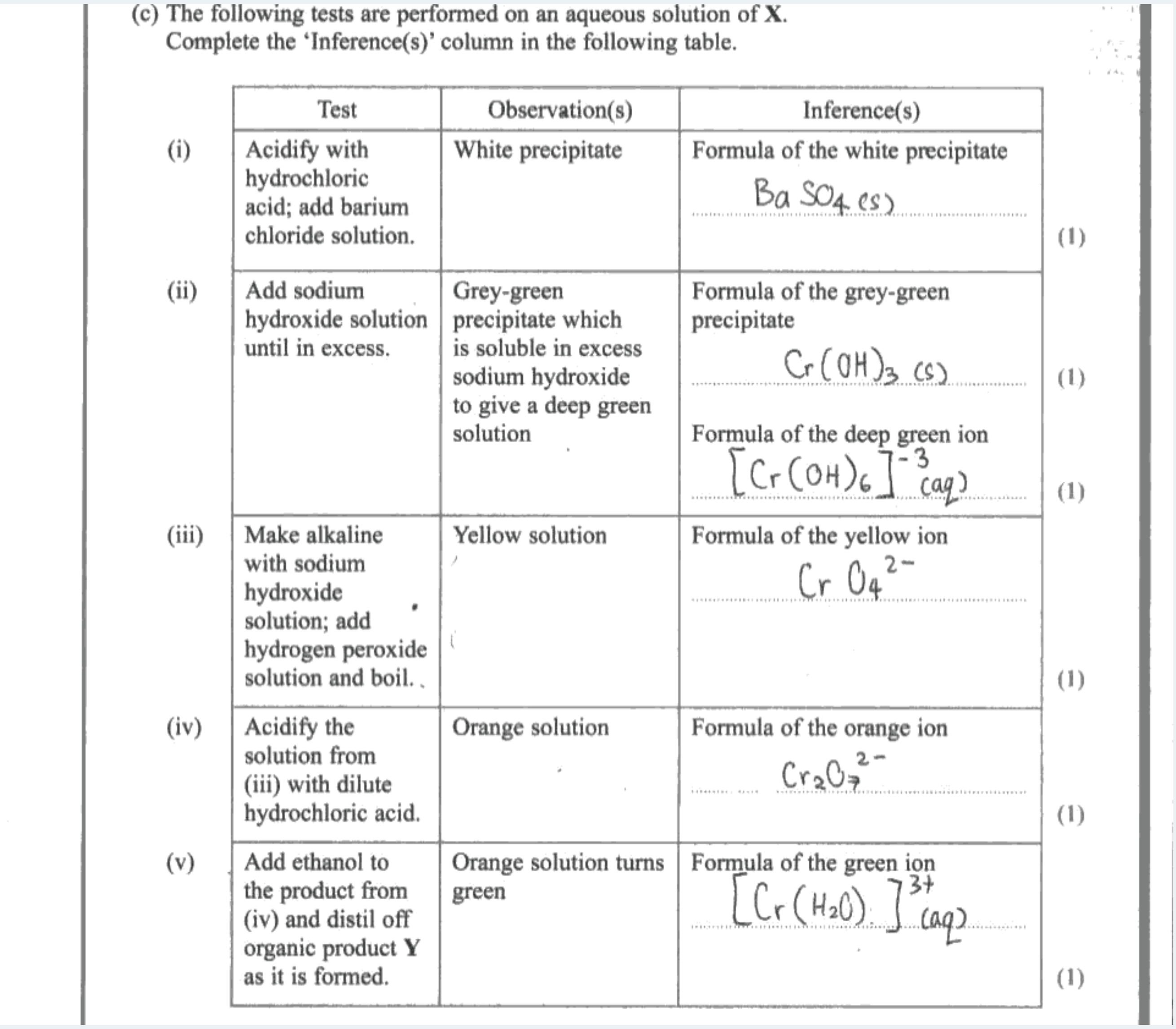
In 1(c)(ii): Large numbers of candidates gave $Cr(OH)_3$ correctly. Some included three molecules of water as well, which was allowed, though no other number of water molecules was. Candidates were somewhat less successful with $Cr(OH)_6^{3-}$, where positive charges were sometimes seen, or coordination numbers other than 6 which are not found in chromium chemistry.

In 1(c)(iii): Many candidates got this correct; near misses usually had a single negative charge.

In 1(c)(iv): The correct formula for the dichromate ion was common, even with answers which were otherwise very poor. For many the orange colour gave them the answer.

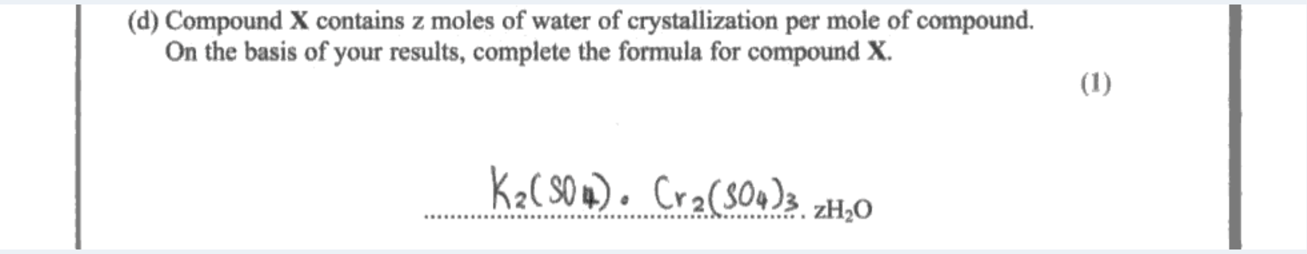
In 1(c)(v): Most candidates gave Cr^{3+}, with a few offering the hexaaqua ion.

In 1(d): There were few correct answers given for the identity of chromium potassium sulphate. A large number simply made no reference to the potassium ion, the most popular answer being $K_2Cr_2O_7$.



(c) The following tests are performed on an aqueous solution of **X**.
Complete the 'Inference(s)' column in the following table.

	Test	Observation(s)	Inference(s)	
(i)	Acidify with hydrochloric acid; add barium chloride solution.	White precipitate	Formula of the white precipitate $BaSO_4(s)$	(1)
(ii)	Add sodium hydroxide solution until in excess.	Grey-green precipitate which is soluble in excess sodium hydroxide to give a deep green solution	Formula of the grey-green precipitate $Cr(OH)_3(s)$	(1)
			Formula of the deep green ion $[Cr(OH)_6]^{-3}(aq)$	(1)
(iii)	Make alkaline with sodium hydroxide solution; add hydrogen peroxide solution and boil.	Yellow solution	Formula of the yellow ion CrO_4^{2-}	(1)
(iv)	Acidify the solution from (iii) with dilute hydrochloric acid.	Orange solution	Formula of the orange ion $Cr_2O_7^{2-}$	(1)
(v)	Add ethanol to the product from (iv) and distil off organic product **Y** as it is formed.	Orange solution turns green	Formula of the green ion $[Cr(H_2O)]^{3+}(aq)$	(1)



(d) Compound **X** contains z moles of water of crystallization per mole of compound.
On the basis of your results, complete the formula for compound **X**.

(1)

$K_2(SO_4) \cdot Cr_2(SO_4)_3 \cdot zH_2O$

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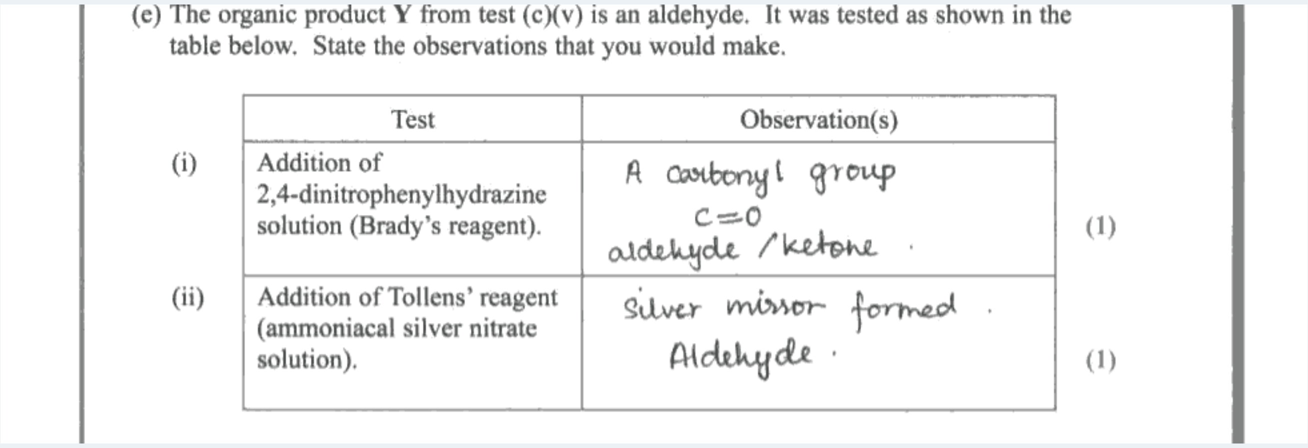
Examiner Comments

This is a very good answer, clear and well thought out.

Question 1(e)

In 1(e)(i): Large number of candidates got this correct. Those who didn't confused the test with the Benedict's or Fehling's tests or failed to mention a precipitate. Some thought, incorrectly, that the precipitate can be red.

In 1(e)(ii): An overwhelmingly large number of candidates gave the correct response to this question.



(e) The organic product **Y** from test (c)(v) is an aldehyde. It was tested as shown in the table below. State the observations that you would make.

	Test	Observation(s)	
(i)	Addition of 2,4-dinitrophenylhydrazine solution (Brady's reagent).	A carbonyl group $C=O$ aldehyde / ketone.	(1)
(ii)	Addition of Tollens' reagent (ammoniacal silver nitrate solution).	Silver mirror formed. Aldehyde.	(1)

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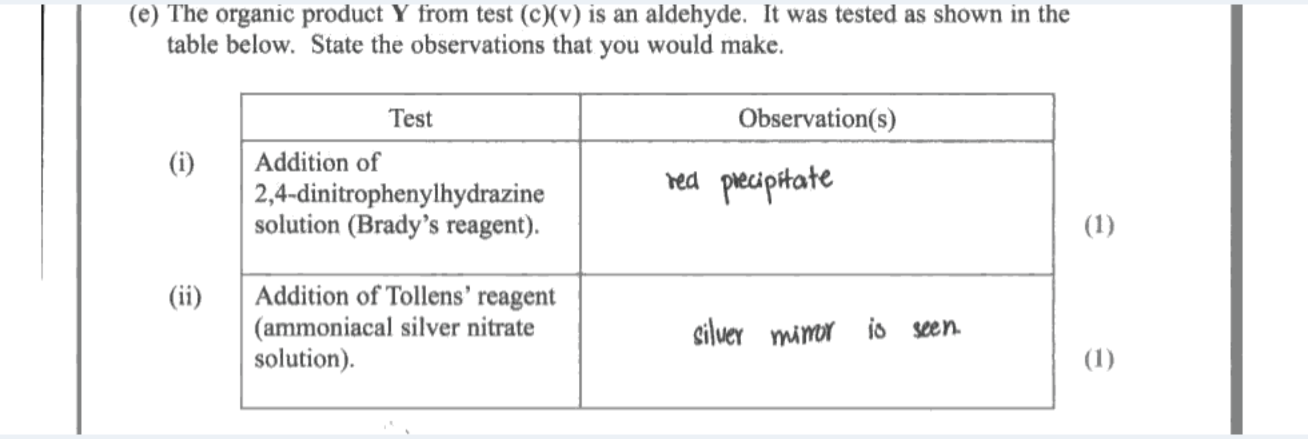
Examiner Comments

This is a good example of an answer for part (i) which is a true statement but not an answer to this question.



(e) The organic product **Y** from test (c)(v) is an aldehyde. It was tested as shown in the table below. State the observations that you would make.

	Test	Observation(s)	
(i)	Addition of 2,4-dinitrophenylhydrazine solution (Brady's reagent).	red precipitate	(1)
(ii)	Addition of Tollens' reagent (ammoniacal silver nitrate solution).	silver mirror is seen.	(1)

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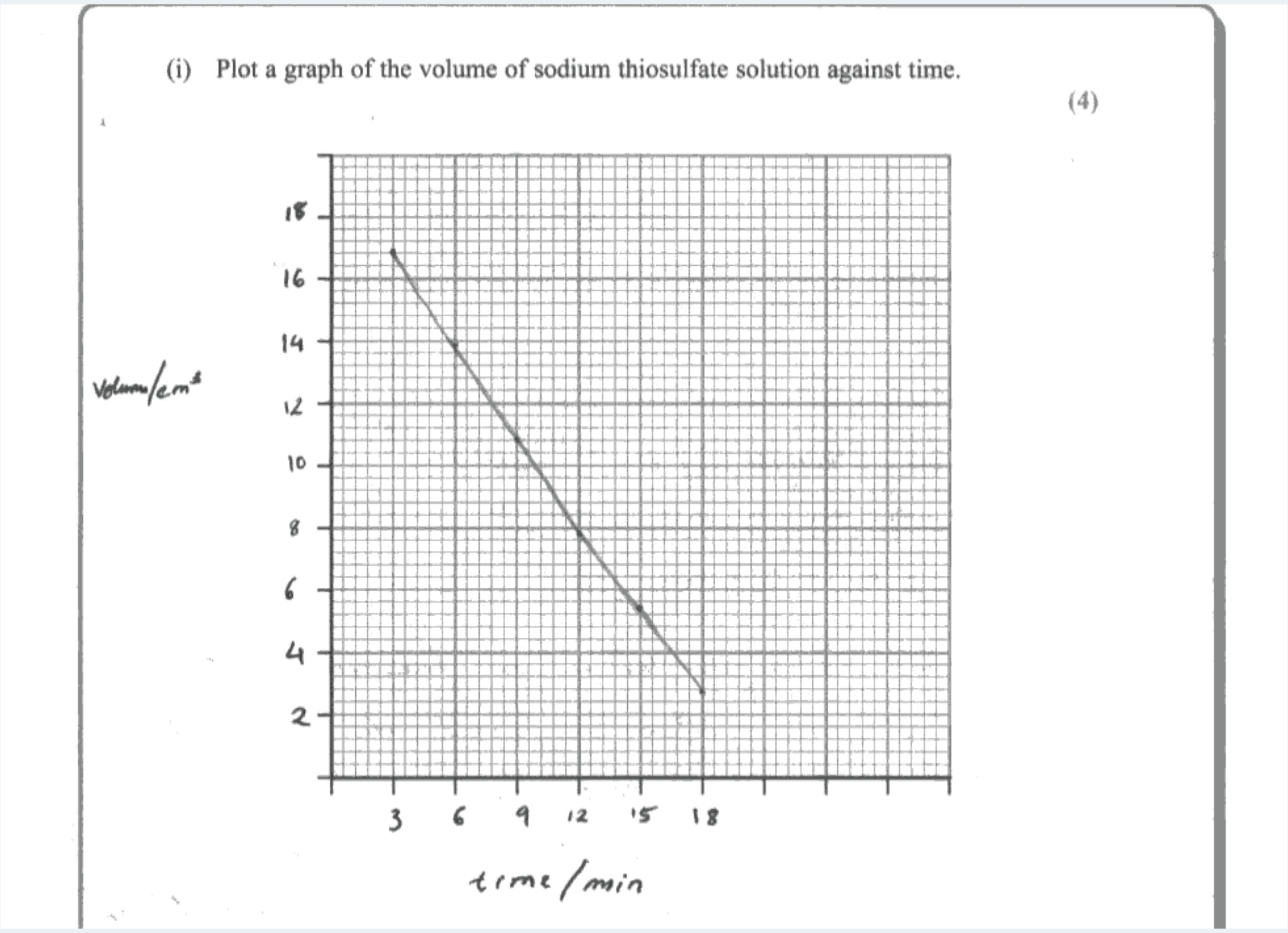
Examiner Comments

Part (i) does not score because the precipitate is not red.

Question 2(a)(i)

The graphs were largely very well drawn with sensible axes and the points clearly visible. Some answers incorrectly showed point-to-point lines. Any reasonable estimate of the best-fit straight line was credited. Very few scored poorly on this section.



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

(4)

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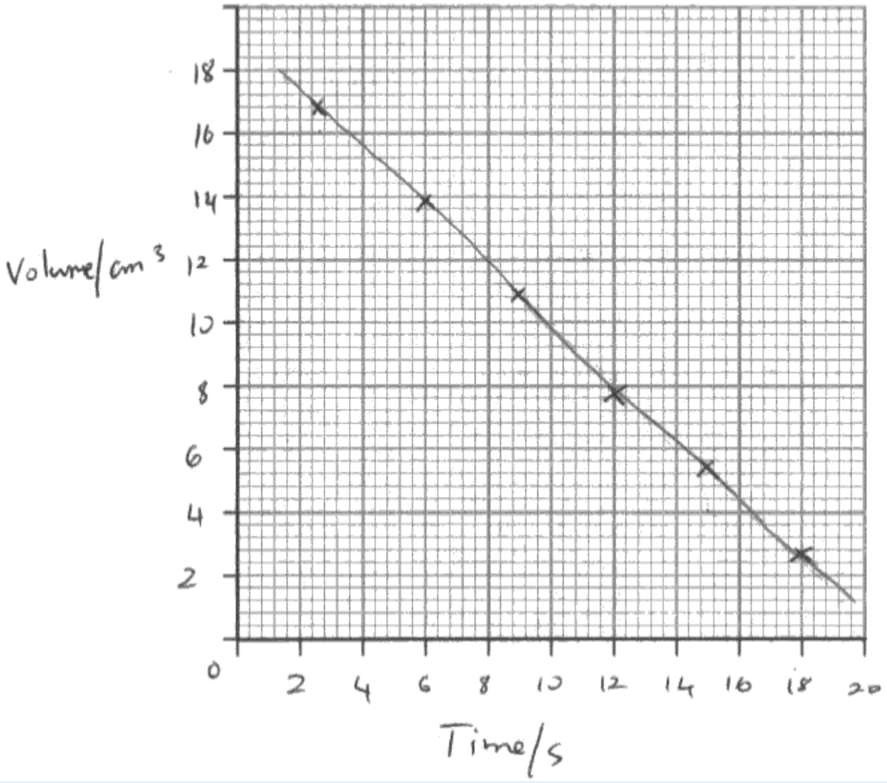
Examiner Comments

The points are not visible, so it is impossible to tell whether the line is a reasonable best fit.

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

(4)



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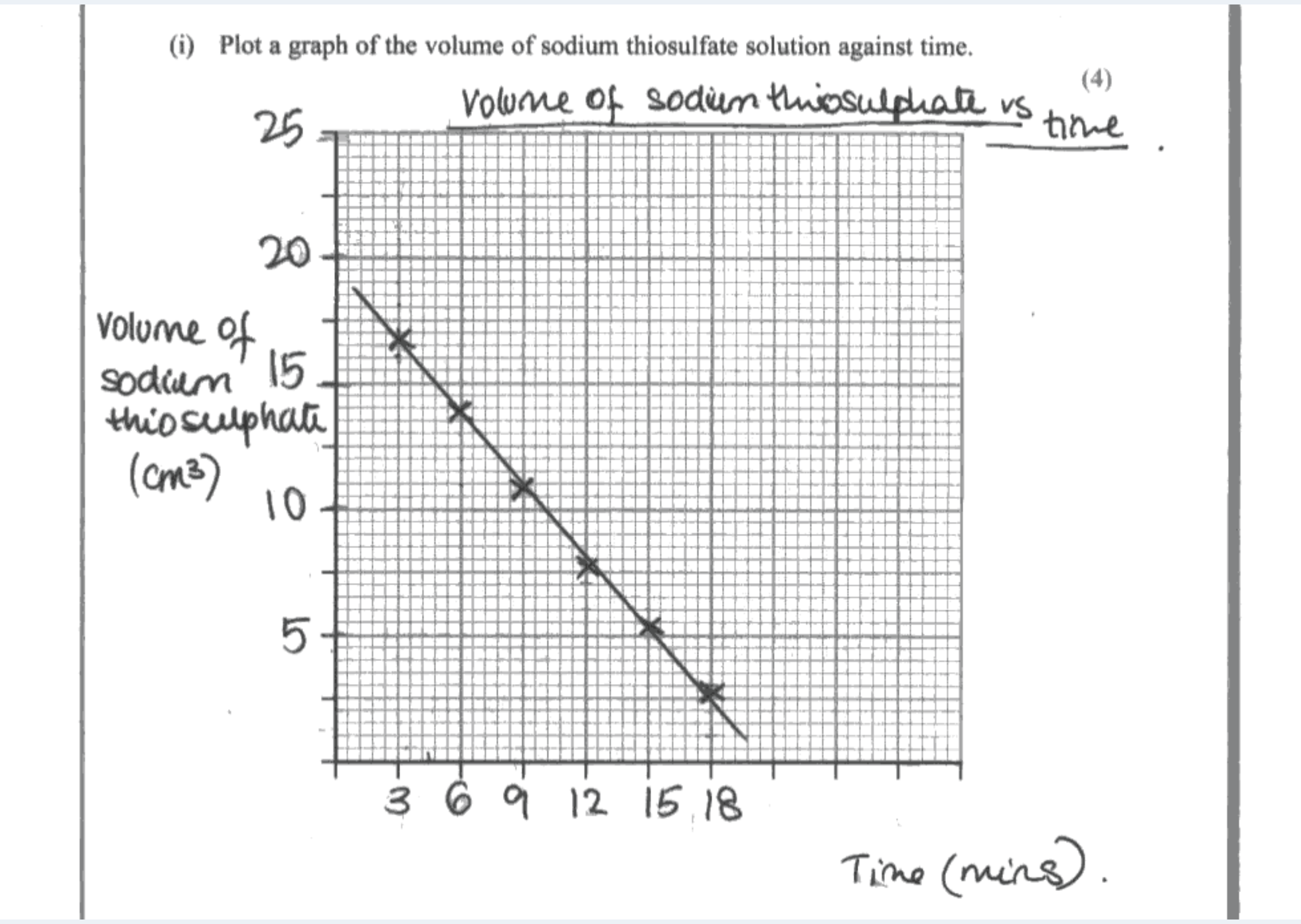
Examiner Comments

The line is not a best fit line but is joined point-to-point, so loses a mark because of this.



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

(4)

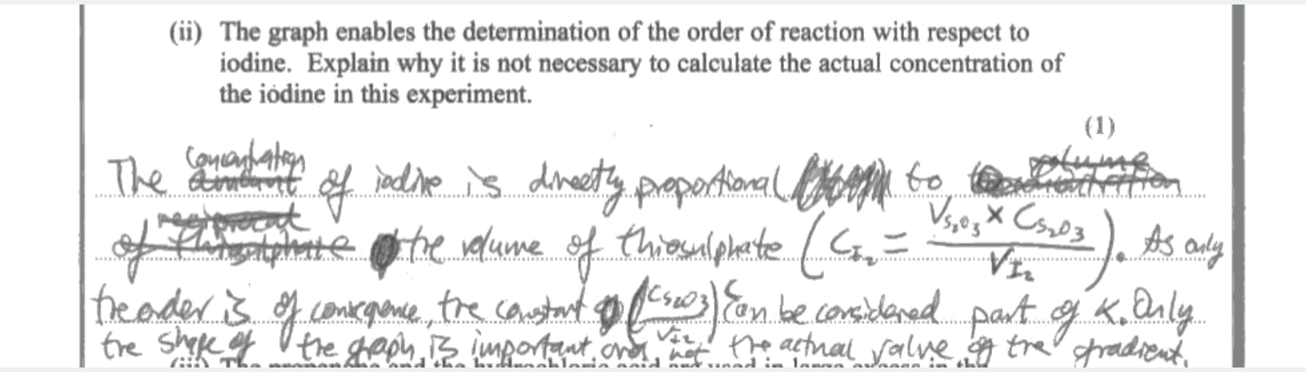
ResultsPlus

Examiner Comments

This is a good graph - clear scale, points, and best fit line. Well worth the 4 marks.

Question 2(a)(ii)

For full credit candidates must say that the **volume** of thiosulphate is proportional to the iodine concentration. Some gave answers based on stoichiometry but unrelated to the volume; others said that the volume **is related to** the iodine concentration, but did not say what this relationship was.



(ii) The graph enables the determination of the order of reaction with respect to iodine. Explain why it is not necessary to calculate the actual concentration of the iodine in this experiment.

(1)

The ~~amount~~ concentration of iodine is directly proportional to ~~concentration~~ ~~volume~~ ~~of reciprocal of thiosulphate~~ the volume of thiosulphate ($C_{I_2} = \frac{V_{S_2O_3} \times C_{S_2O_3}}{V_{I_2}}$). As only the order is of consequence, the constant $\left(\frac{C_{S_2O_3}}{V_{I_2}}\right)$ can be considered part of k. Only the shape of the graph is important ~~over~~ not the actual value of the gradient.

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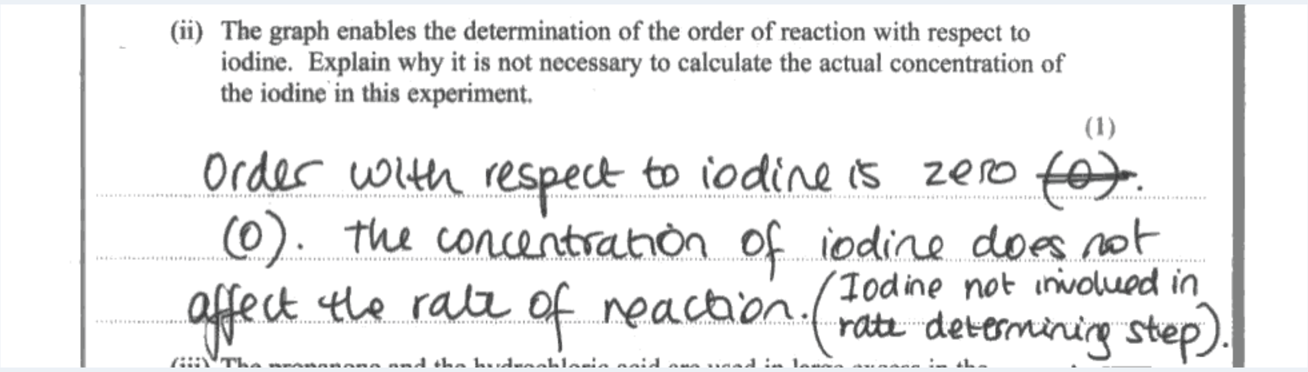
Examiner Comments

This scores the mark, because the statement as far as 'thiosulphate' covers the point. The rest is irrelevant and, fortunately, not contradictory.



(ii) The graph enables the determination of the order of reaction with respect to iodine. Explain why it is not necessary to calculate the actual concentration of the iodine in this experiment.

(1)

Order with respect to iodine is zero ~~(0)~~. (0). the concentration of iodine does not affect the rate of reaction. (Iodine not involved in rate determining step).

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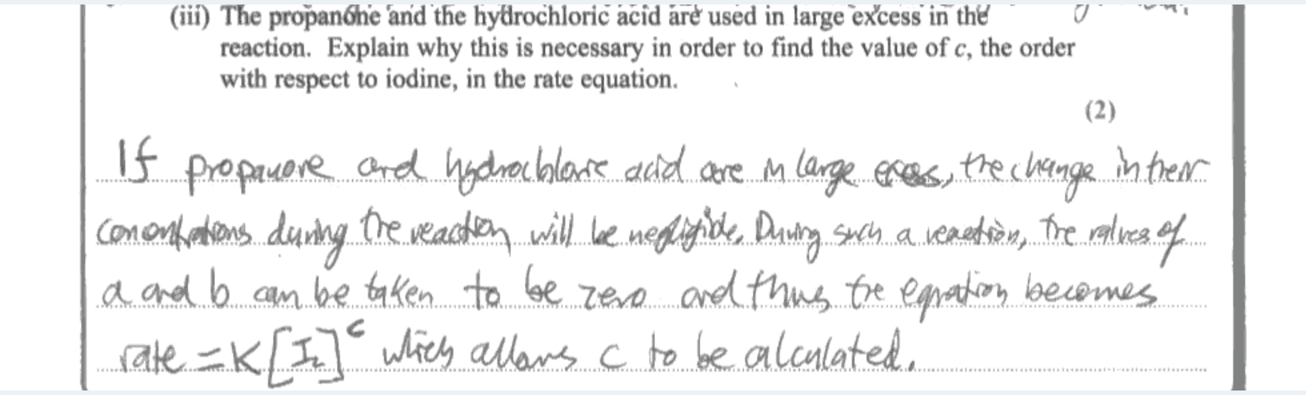
Examiner Comments

A true statement, but not the answer to this question.

Question 2(a)(iii)

Many candidates knew that the constancy of the propanone and acid concentrations meant that the kinetics observed was governed by iodine alone. Others were vague that excess also meant constancy but gave enough information to allow the second mark to be awarded. Weaker answers said that the propanone and acid concentrations did not affect the rate, clearly not true.



(iii) The propanone and the hydrochloric acid are used in large excess in the reaction. Explain why this is necessary in order to find the value of c, the order with respect to iodine, in the rate equation.

(2)

If propanone and hydrochloric acid are in large excess, the change in their concentrations during the reaction will be negligible. During such a reaction, the values of a and b can be taken to be zero and thus the equation becomes rate $= K[I_2]^c$ which allows c to be calculated.

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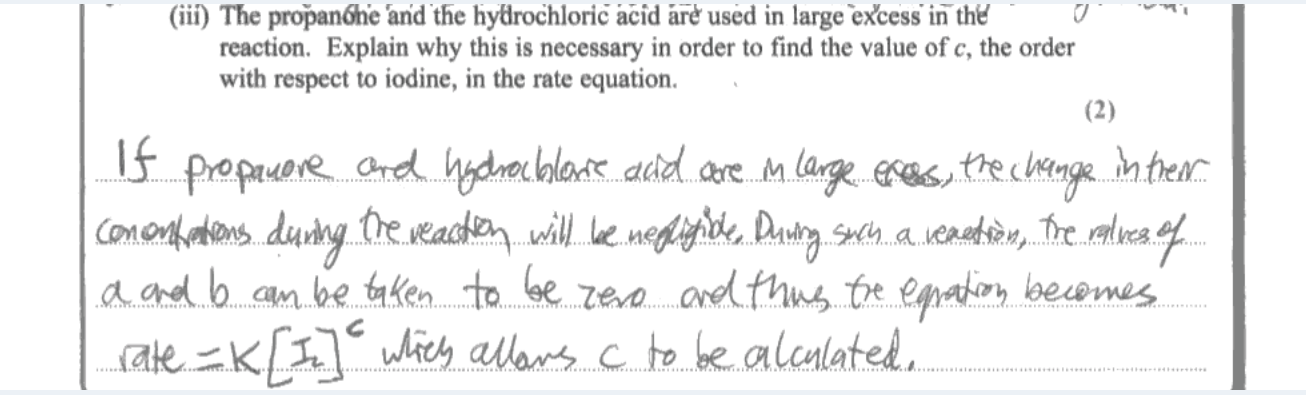
Examiner Comments

The inclusion of the equation for the rate in the presence of excess propanone and acid is a useful indicator that the candidate understands what is going on.

Question 2(a)(iv)



(iii) The propanone and the hydrochloric acid are used in large excess in the reaction. Explain why this is necessary in order to find the value of c, the order with respect to iodine, in the rate equation.

(2)

If propanone and hydrochloric acid are in large excess, the change in their concentrations during the reaction will be negligible. During such a reaction, the values of a and b can be taken to be zero and thus the equation becomes rate $= K[I_2]^c$ which allows c to be calculated.

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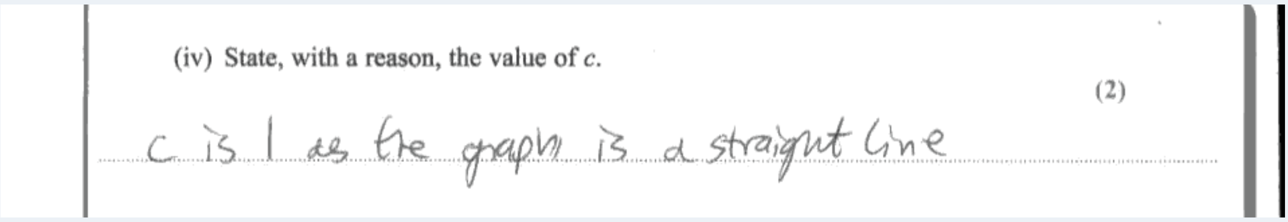
Examiner Comments

A variant on this theme 'To give complete reaction' was a commonly-seen error.

The best answers showed an understanding of what is going on in this experiment and could relate the gradient of the graph to the rate. Inevitably some seized on the straight-line graph as being indicative of a first-order reaction. Many said that the order with respect to iodine was zero, even though the rest of their answer suggested that they did not know why.



(iv) State, with a reason, the value of c.

(2)

c is 1 as the graph is a straight line

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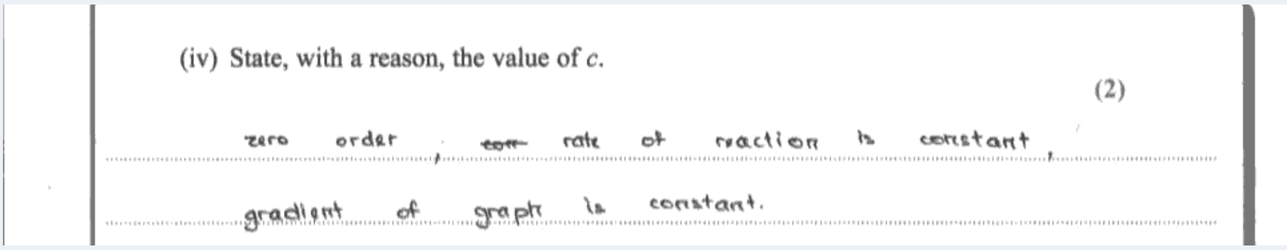
Examiner Comments

It was common to see first order as being derived from a straight-line graph. The candidate has not realized what the graph means.



(iv) State, with a reason, the value of c.

(2)

zero order, ~~con~~ rate of reaction is constant, gradient of graph is constant.

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Examiner Comments

In contrast this candidate does understand the meaning of the constant gradient.

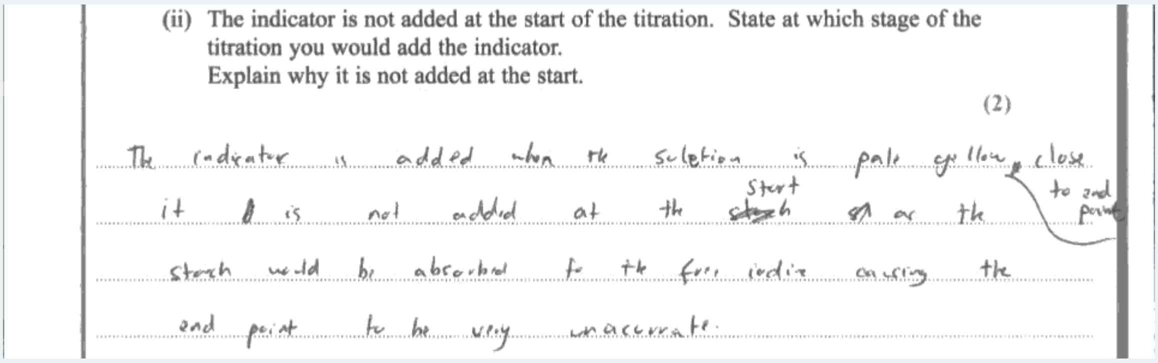
Question 2(b)(i)

Most candidates knew that starch is the indicator required for iodine titrations and gave the correct colour change. Some got this reversed, from colourless to blue-black. It is important always to give the colour **change**; so 'blue-black colour disappears' does not get the mark since the colour of the final solution is not stated. 'Blue-black solution decolourises' does score, however, since the implication is that the final solution is colourless.

Question 2(b)(ii)

Many candidates said that the starch should be added ‘near the endpoint’, without saying how they would know when this point had been reached. Many correctly said when the solution was ‘straw-coloured’ or ‘pale yellow’; others said that it was when the iodine colour had ‘faded’, the meaning of which was not clear. The significant number who added it ‘at the endpoint’ had missed the point of the indicator.

Candidates had to mention that early addition of starch gives an insoluble precipitate of the starch-iodine complex. Those who said that an irreversible complex, or a complex that prevented iodine from reacting is formed, also got credit. It was essential to make clear that the formation of the complex removes iodine irreversibly, since the soluble reversible complex is also between iodine and starch.



(ii) The indicator is not added at the start of the titration. State at which stage of the titration you would add the indicator.
Explain why it is not added at the start.

(2)

The indicator is added when the solution is pale yellow, close to end point it is not added at the ~~starch~~ start as the starch would be absorbed to the free iodine causing the end point to be very unaccurate.

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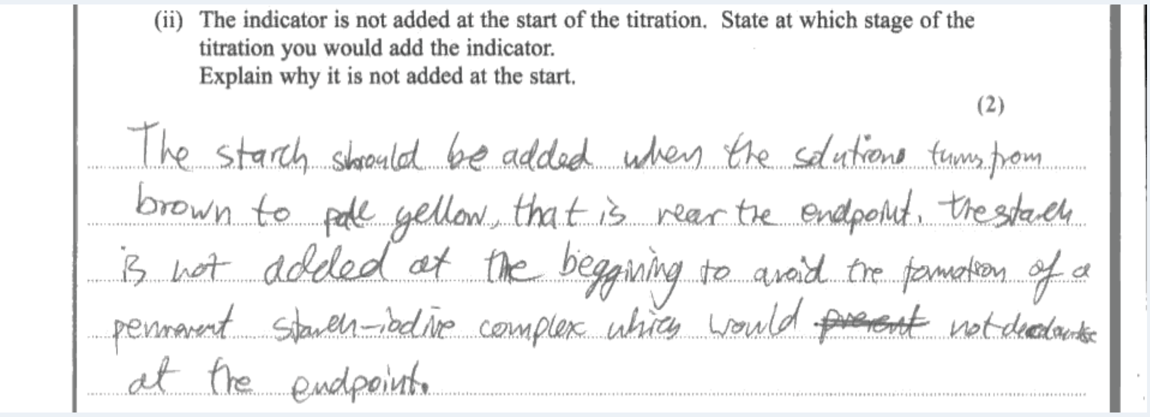
Examiner Comments

The point at which starch should be added is well understood. However the effect of earlier addition is not since 'absrobed' is not enough to suggest an irreversible reaction.



(ii) The indicator is not added at the start of the titration. State at which stage of the titration you would add the indicator.
Explain why it is not added at the start.

(2)

The starch should be added when the solutions turns from brown to pale yellow, that is near the endpoint. the starch is not added at the beggining to avoid the formation of a permanent starch-iodine complex which would ~~prevent~~ not decolourise at the endpoint.

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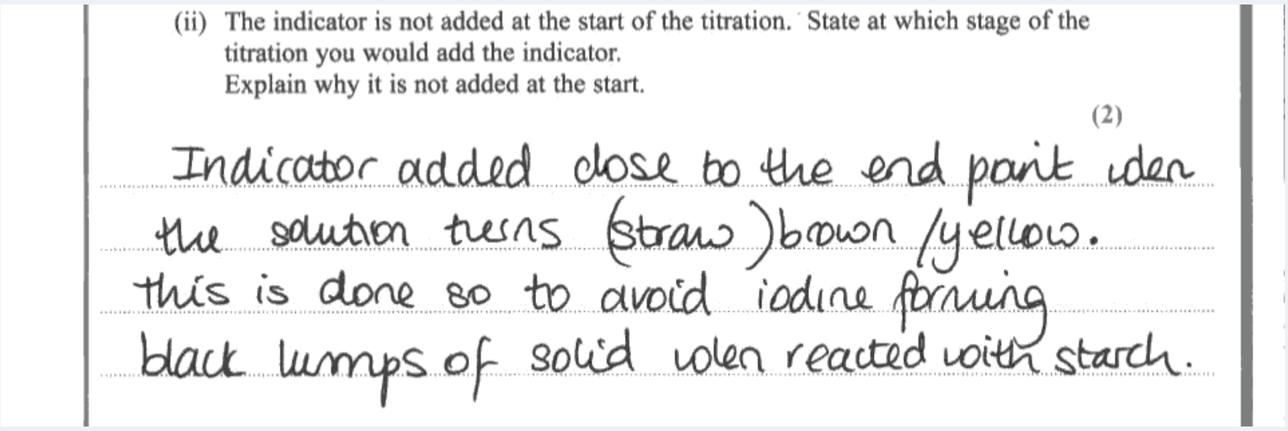
Examiner Comments

The 'permanent' comment enables the award of the second mark.



(ii) The indicator is not added at the start of the titration. State at which stage of the titration you would add the indicator.
Explain why it is not added at the start.

(2)

Indicator added close to the end point when the solution turns (straw) brown/yellow. this is done so to avoid iodine forming black lumps of solid when reacted with starch.

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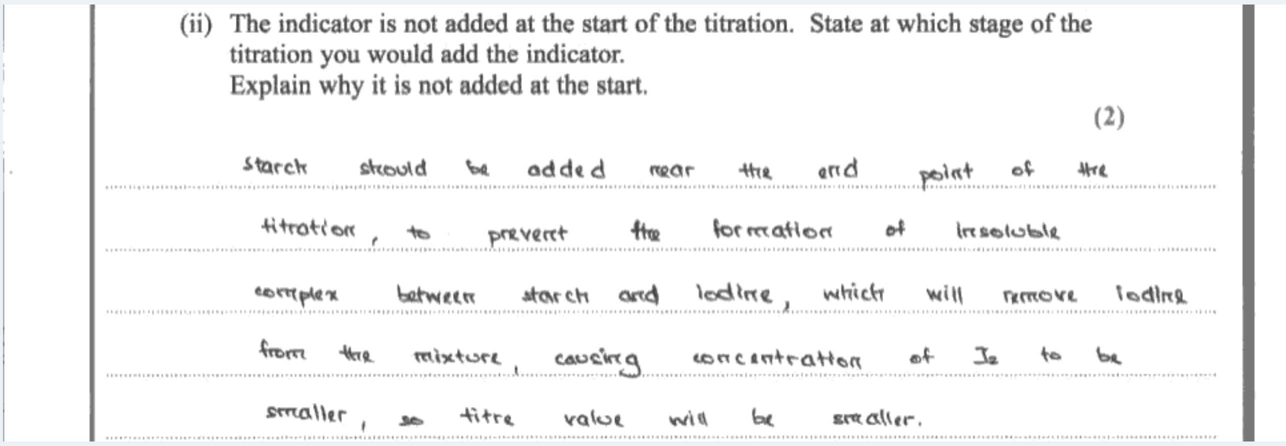
Examiner Comments

The 'black lumps of solid' are not said to be insoluble. Maybe the candidate means that, but has not said so.



(ii) The indicator is not added at the start of the titration. State at which stage of the titration you would add the indicator.
Explain why it is not added at the start.

(2)

Starch should be added near the end point of the titration, to prevent the formation of insoluble complex between starch and iodine, which will remove iodine from the mixture, causing concentration of I_2 to be smaller, so titre value will be smaller.

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Examiner Comments

Second mark awarded but not the first for this example. How do we know when we are near the endpoint?

Question 2(c)

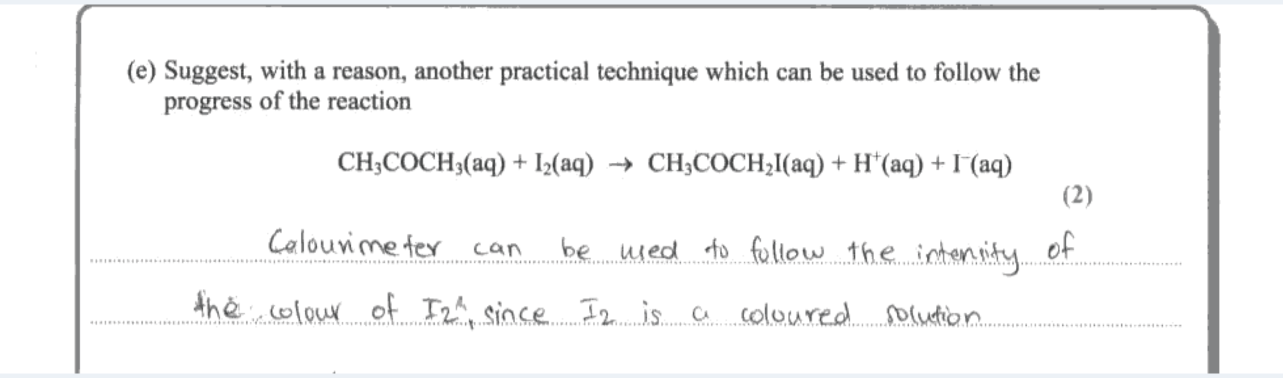
This was possibly the worst-answered question on the whole paper. The idea of increased relative error was very rarely seen. Many candidates believed that a small quantity could not be measured accurately simple by virtue of being small. Others, completely adrift from what the data mean, said that the results were not concordant with the previous titres. There was a significant group that said that the data was least accurate because the points did not fall on the graph, begging the question of where the graph had come from in the first place.

Question 2(d)

Many candidates correctly gave the order with respect to propanone, and mostly for the right reasons.

Question 2(e)

The most popular choices for alternative techniques for following the reaction were colorimetry, though candidates did not always mention that iodine is coloured; or conductivity. Some candidates suggested using the iodoform reaction or simply modified the existing experiment. Neither was able to make clear what the justification for these experiments could be.



(e) Suggest, with a reason, another practical technique which can be used to follow the progress of the reaction

$$CH_3COCH_3(aq) + I_2(aq) \rightarrow CH_3COCH_2I(aq) + H^+(aq) + I^-(aq)$$

(2)

Calourimeter can be used to follow the intensity of the colour of I_2, since I_2 is a coloured solution

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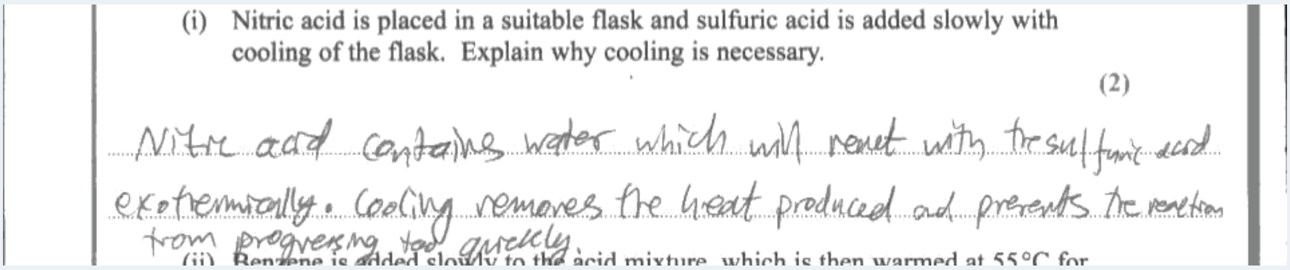
Examiner Comments

A clear understanding of a good technique and the basis for it.

Question 3(a)(i)

Everyone knew that the mixing of the acids is exothermic; only the better scripts made the point that this was due to water in the nitric acid reacting with sulphuric acid. Some related it to oxidising abilities, but many gave no reason.



(i) Nitric acid is placed in a suitable flask and sulfuric acid is added slowly with cooling of the flask. Explain why cooling is necessary.

(2)

Nitric acid contains water which will react with the sulfuric acid exothermically. Cooling removes the heat produced and prevents the reaction from progressing too quickly.

(ii) Benzene is added slowly to the acid mixture, which is then warmed at 55 °C for

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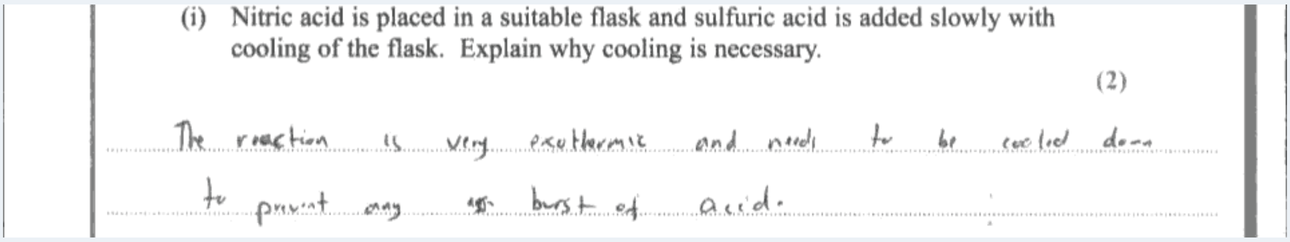
This is a good example of an answer which demonstartes an understanding of the reasons for the exothermic reaction.



(i) Nitric acid is placed in a suitable flask and sulfuric acid is added slowly with cooling of the flask. Explain why cooling is necessary.

(2)

The reaction is very exothermic and needs to be cooled down to prevent any burst of acid.

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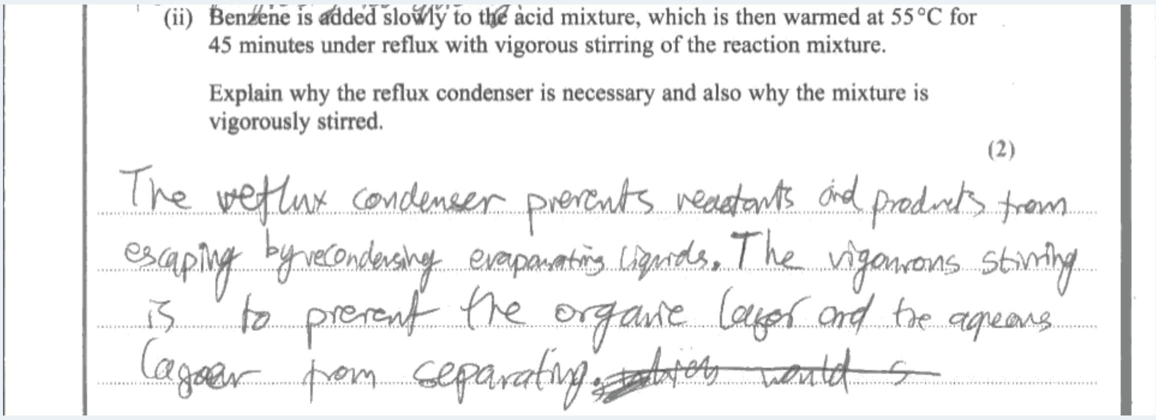
Examiner Comments

There is no understanding of why the reaction is exothermic - and the 'burst' comment is a minor example of an excessively vigorous view of what is happening.

Question 3(a)(ii)

Candidates need to be clear what can escape from the reaction mixture at 55°C and what cannot. Vague references to ‘escape of substances’ or ‘of products’ did not score the first mark. References to ‘volatile organic substances’ or to benzene itself were required. Many candidates know that benzene and the nitrating mixture are immiscible and gave this as the reason for stirring, although the weaker offerings said that this was to distribute the heat evenly.



(ii) Benzene is added slowly to the acid mixture, which is then warmed at 55 °C for 45 minutes under reflux with vigorous stirring of the reaction mixture.

Explain why the reflux condenser is necessary and also why the mixture is vigorously stirred.

(2)

The reflux condenser prevents reactants and products from escaping by recondensing evaporating liquids. The vigorous stirring is to prevent the organic layer and the aqueous layer from separating. ~~which would s~~

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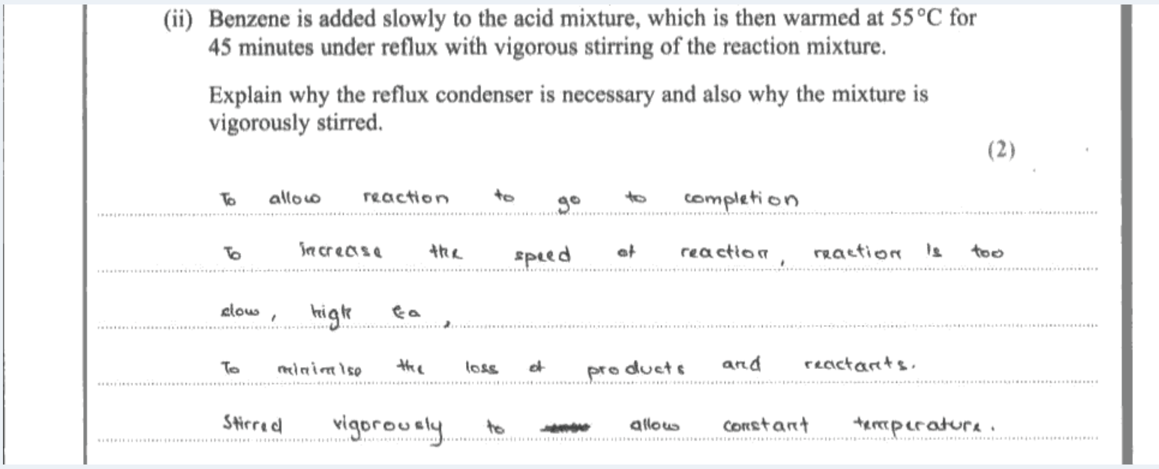
Examiner Comments

The first mark is marred somewhat by reference to the products. Nitrobenzene is not going to evaporate at 55C. The second point is not explicitly made about immiscibility. Taken as a whole the answer is worth 1 out of 2.



(ii) Benzene is added slowly to the acid mixture, which is then warmed at 55 °C for 45 minutes under reflux with vigorous stirring of the reaction mixture.

Explain why the reflux condenser is necessary and also why the mixture is vigorously stirred.

(2)

To allow reaction to go to completion

To increase the speed of reaction, reaction is too slow, high Ea,

To minimise the loss of products and reactants.

Stirred vigorously to allow constant temperature.

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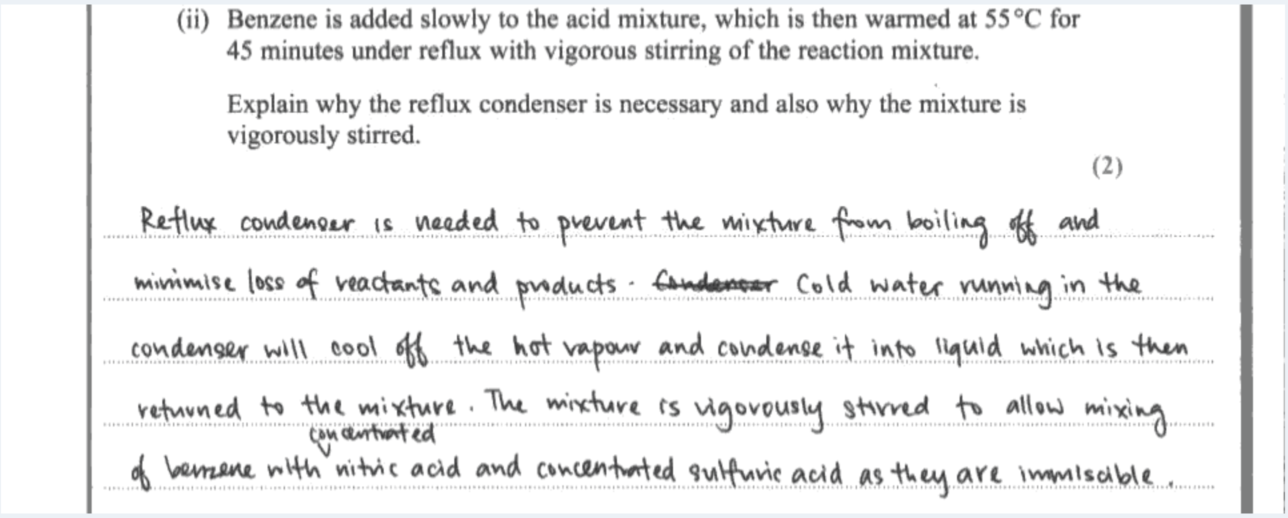
Examiner Comments

This is a typical example of a candidate who does not really know what is happening. The temperature point was made by many.



(ii) Benzene is added slowly to the acid mixture, which is then warmed at 55 °C for 45 minutes under reflux with vigorous stirring of the reaction mixture.

Explain why the reflux condenser is necessary and also why the mixture is vigorously stirred.

(2)

Reflux condenser is needed to prevent the mixture from boiling off and minimise loss of reactants and products. ~~Condenser~~ Cold water running in the condenser will cool off the hot vapour and condense it into liquid which is then returned to the mixture. The mixture is vigorously stirred to allow mixing of benzene with concentrated nitric acid and concentrated sulfuric acid as they are immiscible.

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Examiner Comments

Although 'products' are mentioned in this answer there is more which shows that the candidate understands that volatile compounds are condensed. The immiscibility point is made clearly.

Question 3(a)(iii)

Many candidates could give sensible precautions and say why they were used, although with some, the results of failing to do so were excessively dramatic. Candidates need to get a sense of perspective when considering hazard and risk.

Question 3(a)(iv)

Most knew that sodium carbonate removes residual acid in the organic layer.

Question 3(a)(v)

Calcium chloride was the most popular correct drying agent. The mark-scheme did not require this to be anhydrous but it was pleasing to see that many knew that it is. Many other substances that absorb or react with water, such as anhydrous copper(II) sulphate or cobalt chloride, or sulphuric acid, or phosphorus pentoxide, were also suggested.

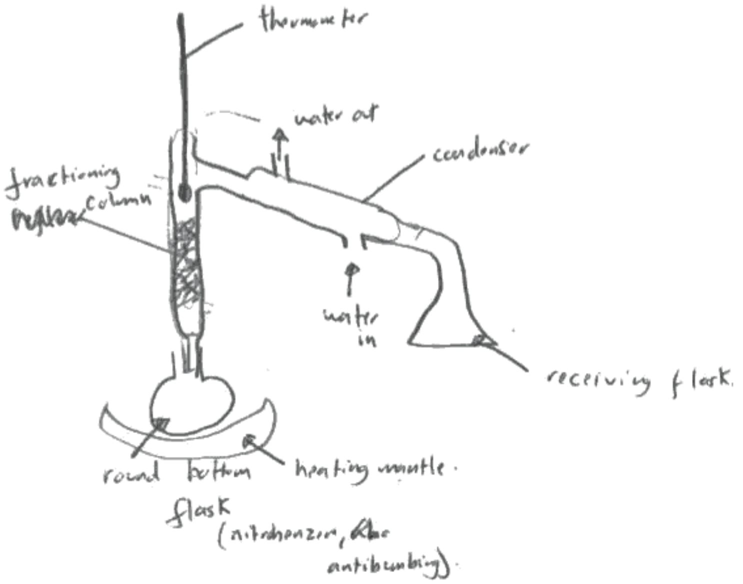
Candidates should realise that the drying agent needs to absorb significant amounts of water (not true for anhydrous copper(II) sulphate or cobalt chloride), and be a solid once it has done so and not contaminate the organic layer (not true for sulphuric acid or phosphorus pentoxide). Silica gel was allowed although it is not a very good drying agent compared to anhydrous calcium chloride.

Question 3(a)(vi)

A significant number of drawings of a distillation apparatus were reasonably good, drawn in section, and would work. There were few which failed because the molecules would have to know where to go once leaving the liquid; those that did usually were very confused about the structure of a Liebig condenser. It would be better if candidates routinely showed a heating mantle rather than just the word 'heat' or, worse, an arrow, under the distillation flask.

(vi) Draw the apparatus used to distil the nitrobenzene and collect the fraction boiling between 207 °C and 211 °C.

(4)



ResultsPlus
Examiner Comments

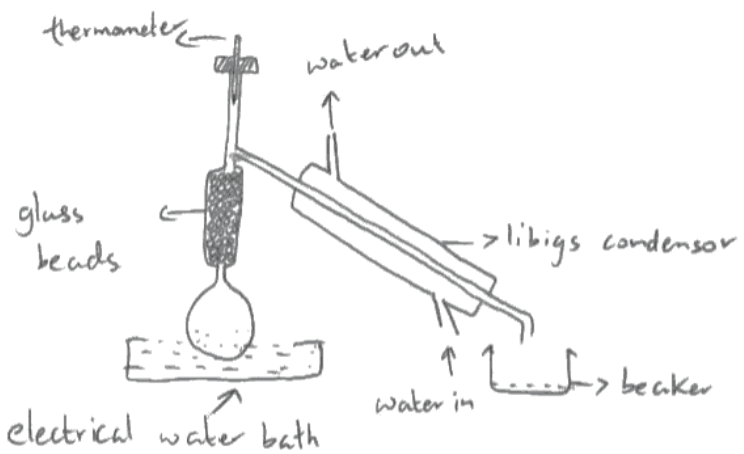
This is an example of an apparatus which cannot work. It is sealed, but much more serious is the complete lack of internal structure for the condenser. The candidate gets 1 mark for the correct heating but no other credit is possible.

ResultsPlus
Examiner Tip

Candidates should look at their diagram and ask if the molecules would have to know where to go. If the answer is 'yes', then the apparatus is not correctly drawn.

(vi) Draw the apparatus used to distil the nitrobenzene and collect the fraction boiling between 207 °C and 211 °C.

(4)



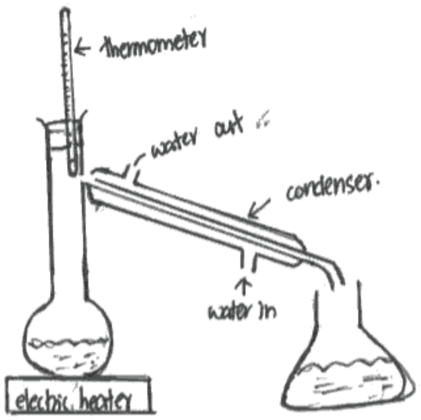
ResultsPlus

Examiner Comments

The thermometer is in the wrong place in this example, and a water bath will not distil any nitrobenzene.

(vi) Draw the apparatus used to distil the nitrobenzene and collect the fraction boiling between 207 °C and 211 °C.

(4)



ResultsPlus

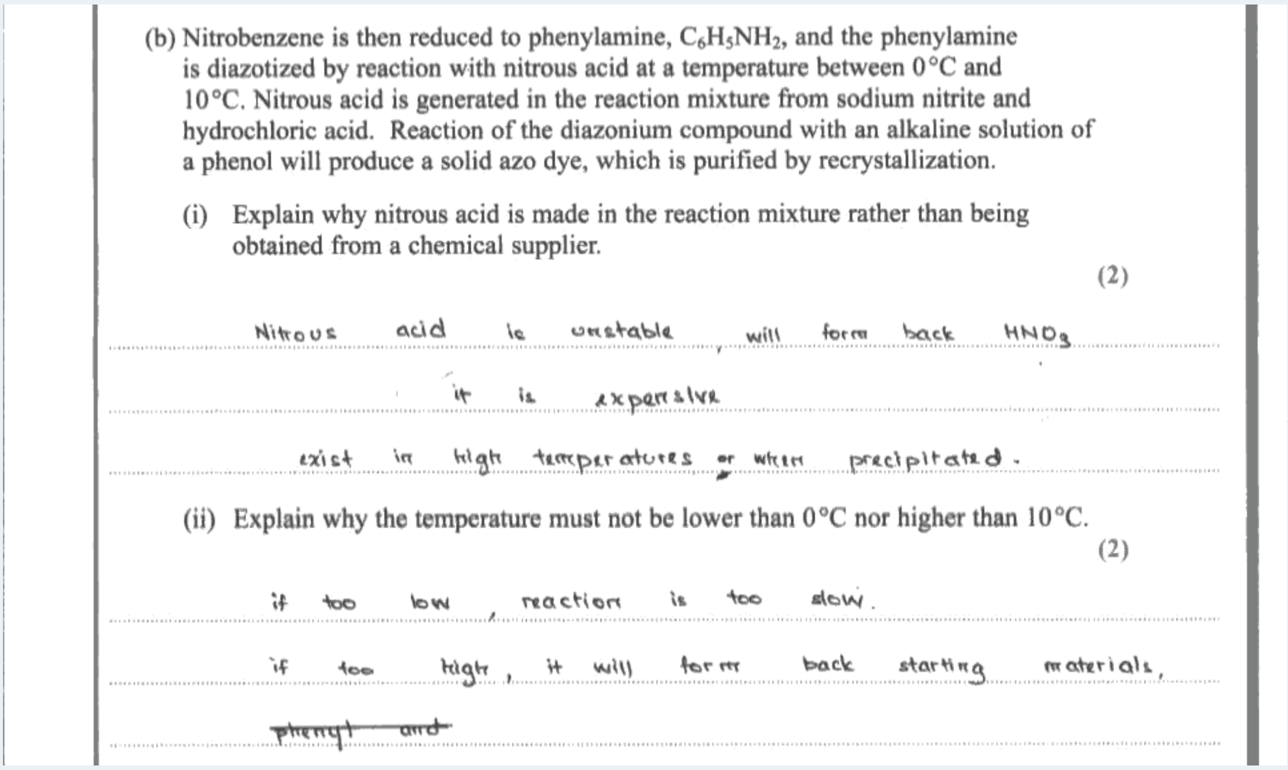
Examiner Comments

This is on the whole a very good diagram - the gap at the top of the condenser is regarded as a slip, given that the rest of the drawing is good.

Question 3(b)

In 3(b)(i): Many candidates did not know the properties of nitrous acid so scored poorly on this question. Even those who referred to its instability did not always make the point that it could not therefore be transported or stored.

In 3(b)(ii): Many candidates think that below 0°C diazotization does not occur at all, whereas it is simply too slow. Others think the mixture freezes, and although this was allowed, in reality the ionic strength of the mixture means that the freezing point is well below 0°C. Many knew that decomposition would occur above 10°C or more specifically that phenol would be produced; some however believe that phenylamine will be regenerated.



(b) Nitrobenzene is then reduced to phenylamine, $C_6H_5NH_2$, and the phenylamine is diazotized by reaction with nitrous acid at a temperature between 0 °C and 10 °C. Nitrous acid is generated in the reaction mixture from sodium nitrite and hydrochloric acid. Reaction of the diazonium compound with an alkaline solution of a phenol will produce a solid azo dye, which is purified by recrystallization.

(i) Explain why nitrous acid is made in the reaction mixture rather than being obtained from a chemical supplier.

(2)

Nitrous acid is unstable, will form back HNO_3

it is expensive

exist in high temperatures or when precipitated.

(ii) Explain why the temperature must not be lower than 0 °C nor higher than 10 °C.

(2)

if too low, reaction is too slow.

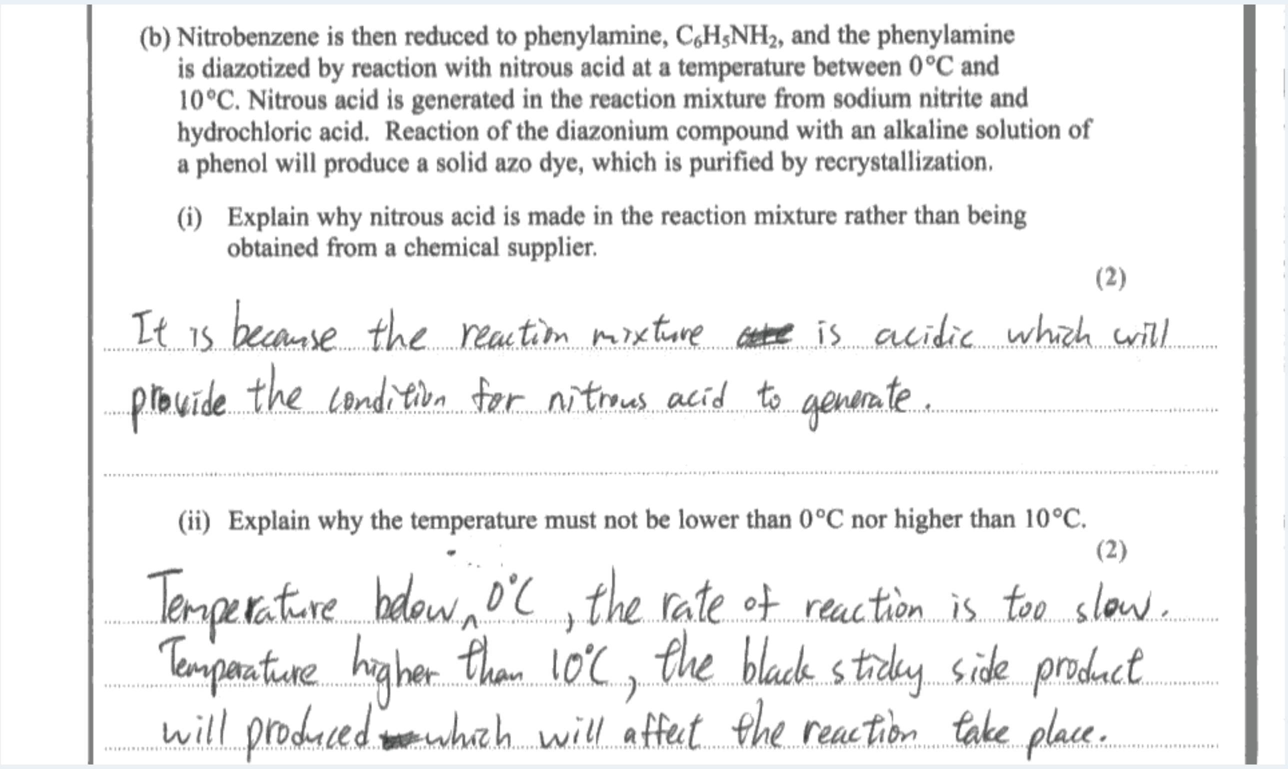
if too high, it will form back starting materials,

~~phenyl and~~

ResultsPlus

Examiner Comments

One mark in each section - the first in both. The starting materials are not reformed above 10C in (ii)



(b) Nitrobenzene is then reduced to phenylamine, $C_6H_5NH_2$, and the phenylamine is diazotized by reaction with nitrous acid at a temperature between 0 °C and 10 °C. Nitrous acid is generated in the reaction mixture from sodium nitrite and hydrochloric acid. Reaction of the diazonium compound with an alkaline solution of a phenol will produce a solid azo dye, which is purified by recrystallization.

(i) Explain why nitrous acid is made in the reaction mixture rather than being obtained from a chemical supplier.

(2)

It is because the reaction mixture ~~are~~ is acidic which will provide the condition for nitrous acid to generate.

(ii) Explain why the temperature must not be lower than 0 °C nor higher than 10 °C.

(2)

Temperature below 0°C, the rate of reaction is too slow. Temperature higher than 10°C, the black sticky side product will produced which will affect the reaction take place.

ResultsPlus

Examiner Comments

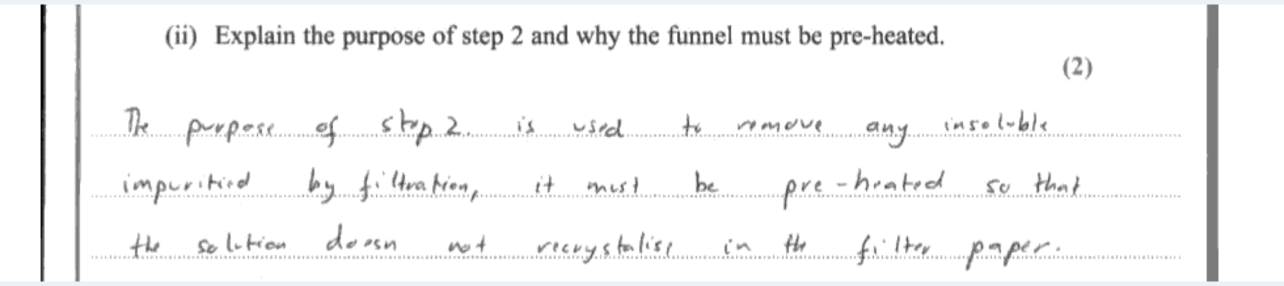
No credit for (i), but the answer for (ii) clearly gets both marks. There are various ways of saying that the mixture would decompose above 10C

Question 3(c)(i)

Many knew that a saturated solution is required for the azo-dye to recrystallize. A simple statement of ‘maximize yield’ was not allowed on its own – it needed some justification.

Question 3(c)(ii)

Many candidates knew that insoluble impurities are removed by filtration, but fewer recognised that cooling would cause the dye to recrystallize in the funnel.



(ii) Explain the purpose of step 2 and why the funnel must be pre-heated.

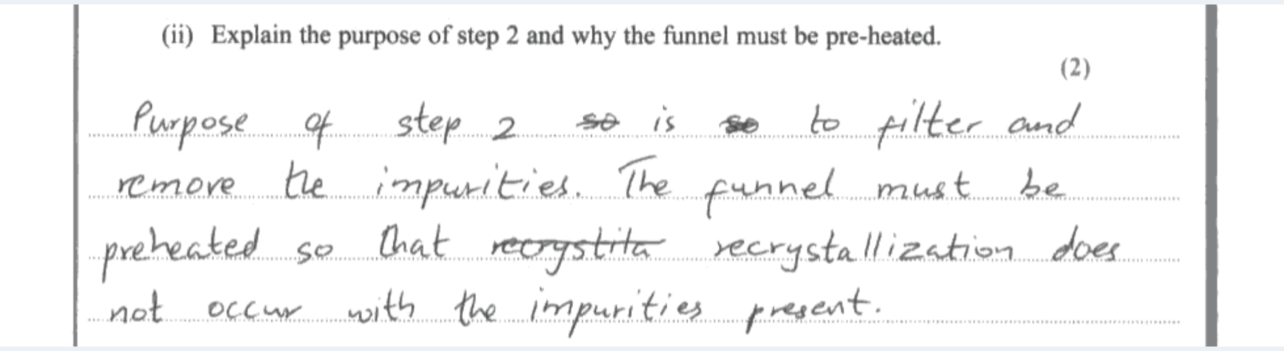
(2)

The purpose of step 2 is used to remove any insoluble impurities by filtration, it must be pre-heated so that the solution doesn not recrystalise in the filter paper.

ResultsPlus

Examiner Comments

A very good, clear and crisp answer.



(ii) Explain the purpose of step 2 and why the funnel must be pre-heated.

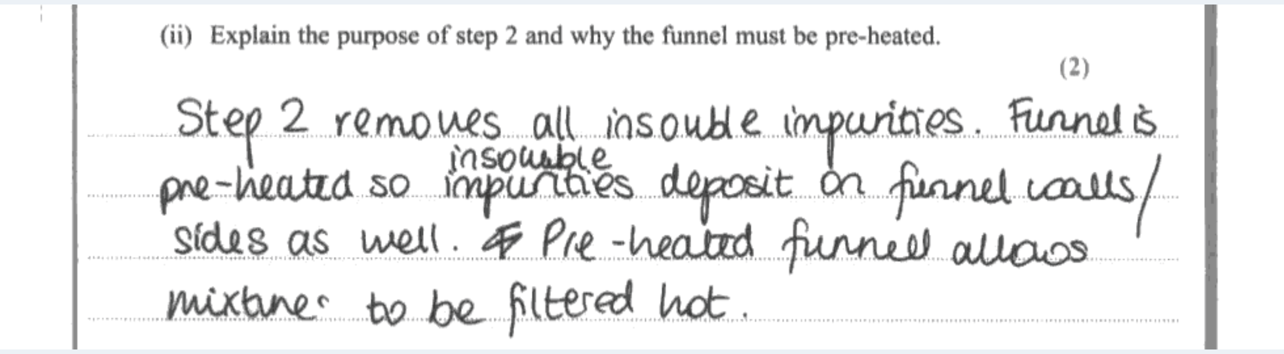
(2)

Purpose of step 2 ~~so~~ is ~~so~~ to filter and remove the impurities. The funnel must be preheated so that ~~recrystita~~ recrystallization does not occur with the impurities present.

ResultsPlus

Examiner Comments

This gets the second mark but not the first. There is no mention of insoluble impurities.



(ii) Explain the purpose of step 2 and why the funnel must be pre-heated.

(2)

Step 2 removes all insouble impurities. Funnel is pre-heated so $\overset{\text{insoluble}}{\text{impurities}}$ deposit on funnel walls/ sides as well. ~~F~~ Pre-heated funnell allows mixture to be filtered hot.

ResultsPlus

Examiner Comments

This gets the first mark but not the second.

Question 3(c)(iii)

This was often answered correctly; weaker candidates simply said that ‘impurities’ were removed, making no reference to their solubility.

Question 3(c)(iv)

Answers relating to melting or decomposition in an oven were often seen. It was clear that some candidates have no idea what a desiccator is.

Candidates must, as the introduction states, be encouraged to think in a practical manner when answering this paper. They need to imagine doing the experiment rather than memorising rote answers from other papers or giving reflex answers - the 'inconsistent titre' argument in 2c being but one example of this.

Candidates also need a sense of perspective regarding hazards. Some of their views on the mixing of sulphuric and nitric acids, or on the toxicity of some materials, verge on the apocalyptic. Very few things in A level Chemistry are likely to make flasks 'burst' or 'explode'. The proper understanding of hazard and then the evaluation of risk is part of the proper understanding of practical chemistry.

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