

Examiners' Report June 2023

International Advanced Level Chemistry WCH16 01



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Introduction

This paper proved accessible to well-prepared candidates who were able to demonstrate their knowledge and understanding of the chemistry tested and a familiarity with the practical techniques involved. WCH16 is the final paper covering the entire IAL syllabus and so it is important that all candidates have thoroughly revised the core practicals in preparation for this examination. Some candidates found parts of the paper quite challenging, especially where they were asked to apply their knowledge, or were asked to explain the practical procedures. It was clear that some candidates would benefit from a firmer grounding in the methods used in practical work and not just being able to follow practical procedures and collect results.

Question 1 (a)(i-ii)

In these questions the candidates were asked to give observations for chromium(III) ions. In Q01(a)(i) the candidates need to mention that a green precipitate was formed and in Q01(a)(ii) what would happen to the precipitate when more sodium hydroxide was added.

- A student investigated two aqueous solutions, labelled P and Q.
 Both solutions were green. Each solution contained one cation and one anion.
 - (a) Tests were carried out on solution P.

Complete the table.

	Test	Observation	Inference	
(i)	A few drops of aqueous sodium hydroxide were added to 5 cm ³ of P	green precipitate	Chromium(III) ions may be present in P	(1
(ii)	More sodium hydroxide solution was added to the mixture from (a)(i) until there was no further change	dissolved into a darker green sorthen	Chromium(III) ions are confirmed to be present in P	(1



A fully correct response for both Q01(a)(i) and Q01(a)(ii), a total of 2 marks awarded.



Make sure the transition metal cation colours have been learned.

- A student investigated two aqueous solutions, labelled P and Q.
 Both solutions were green. Each solution contained one cation and one anion.
 - (a) Tests were carried out on solution P.

Complete the table.

	Test	Observation	Inference	
(i)	A few drops of aqueous sodium hydroxide were added to 5 cm ³ of P	Green precipitate	Chromium(III) ions may be present in P	(1
(ii)	More sodium hydroxide solution was added to the mixture from (a)(i) until there was no further change	Purpee soution	Chromium(III) ions are confirmed to be present in P	(1



This was a popular incorrect answer for Q01(a)(ii). Only 1 mark awarded for Q01(a)(i).

Question 1 (a)(iii)

This question required the formula of the chloride ion to be given (Cl⁻).

A few drops of dilute nitric acid were added to 5 cm ³ of a fresh sample of P		
A few drops of aqueous silver nitrate were added to this acidified solution of P	A white precipitate formed	The formula of the anion likely to be responsible for the white precipitate is
	-	CL-



A few drops of dilute (iii) nitric acid were added to 5 cm³ of a fresh sample of P A few drops of aqueous The formula of the A white precipitate silver nitrate were formed anion likely to be responsible for the added to this acidified solution of P white precipitate is AgCI

The candidate has not read the question and has given the formula of the precipitate instead. 0 marks awarded.

(iii)	A few drops of dilute nitric acid were added to 5 cm ³ of a fresh sample of P		
	A few drops of aqueous silver nitrate were added to this acidified solution of P	A white precipitate formed	The formula of the anion likely to be responsible for the white precipitate is
			chlonde ion



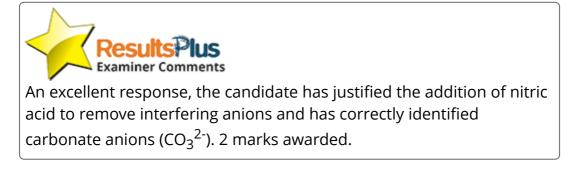
The formula of the ion has not been given. 0 marks awarded.

Question 1 (b)

This question was about a very easy anion spot test, but here the candidates were asked to justify the addition of nitric acid before the addition of silver nitrate.

(b) State why, in the silver nitrate test on **P**, the nitric acid was not needed in this case. Justify your answer by considering the role of nitric acid in the silver nitrate test.

The nitric acid is used to remove other
arishes that the silver nitrate might
auisn's that the silver nitrate might react with The this case there are
no other anions because theres only I anim
and 1 cation.



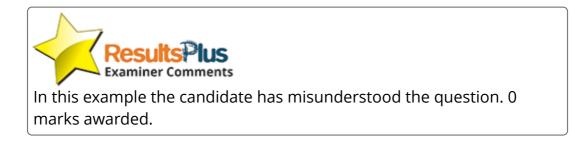
(b) State why, in the silver nitrate test on P, the nitric acid was not needed in this case. Justify your answer by considering the role of nitric acid in the silver nitrate test.

(2)

(2)

The role of nitric acid is to provide Ht so anions can reduce completely. The nitric acid

was not needed in this case because cit cannot further reduce.



(b) State why, in the silver nitrate test on P, the nitric acid was not needed in this case. Justify your answer by considering the role of nitric acid in the silver nitrate test.

because there are no other anions in the solution P who which could form a precipitate with ciliar nitrate, if there were then nitric acid would be needed to ensure a those anions do not form a precipitate and makeus think there is a silver halide being formed When there isn'+.

(2)



This response only scored 1 mark, as the candidate failed to name an interfering anion.

Question 1 (c)(i)

This question concerned another cation that also formed a green precipitate on the addition of dilute aqueous ammonia solution, the candidates were informed that solution Q contained iron(II) ions. Many candidates were awarded both marks, one for the colour change – this was GCSE knowledge, and the other mark for the change in oxidation state of the iron(II) to iron(III).

- (c) The student carried out tests on **Q** and inferred that it was a solution of iron(II) sulfate.
 - (i) The addition of dilute aqueous ammonia to a sample of solution **Q** produced a green precipitate which changed colour on standing.

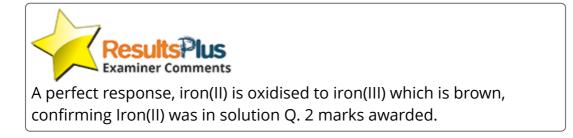
Explain why the colour change led the student to infer that **Q** contained iron(II) ions.

(2)

Fe²⁺ is oxidised by oxygen into Fe³⁺ so green precipitate turns brown on standing.

Therefore, a contains Fe²⁺ due to green precipitate turns brown, which means

Fe²⁺ has been oxidised into Fe³⁺.

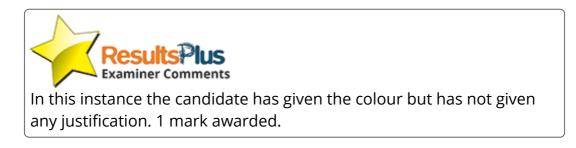


- (c) The student carried out tests on **Q** and inferred that it was a solution of iron(II) sulfate.
 - (i) The addition of dilute aqueous ammonia to a sample of solution **Q** produced a green precipitate which changed colour on standing.

Explain why the colour change led the student to infer that **Q** contained iron(II) ions.

The production of a green precipitate could mean
that either chromium was present or nickel or
iron (11). If it was chromium it wouldn't have
changed colour on standing, neither would the nickel.
But the sample changed to brown with time meaning
there was firon (11) ions.

(2)



- (c) The student carried out tests on **Q** and inferred that it was a solution of iron(II) sulfate.
 - (i) The addition of dilute aqueous <u>ammonia</u> to a sample of solution **Q** produced a green precipitate which changed colour on standing.

Explain why the colour change led the student to infer that **Q** contained iron(II) ions.

Fe NH3 does not change the colour of Fe²⁺ while MAN Cr or Cu react with NH2, the colour will change to pupple or dark blue.



In this example the candidate has not read the question correctly and seems to have focussed on the reaction with ammonia, rather than the sample being left to stand. 0 marks awarded.

- (c) The student carried out tests on **Q** and inferred that it was a solution of iron(II) sulfate.
 - (i) The addition of dilute aqueous ammonia to a sample of solution **Q** produced a green precipitate which changed colour on standing.

Explain why the colour change led the student to infer that **Q** contained iron(II) ions.

	-		
	~	- 14	
ε.	£.,		

(2)

Be coure	colour	was	chunged	on sta	dihe.		
			7-	1000			
Iron (12)	Airer	creen	colour	when	readt	with	
(+)	J	9		÷		-	

ammo nia



While the candidate has mentioned there is a colour change, they have not stated what the new colour is, nor have they commented on the oxidation of iron(II). 0 marks awarded.

Question 1 (c)(ii)

A further question on the analysis of anions. This one was about testing for the presence of sulfate ions. The majority of candidates were able to give a correct reagent and the correct observation.

(ii) Describe a test, and its positive result, that the student could have carried out to show the presence of <u>sulfate ions</u>.

(2)nitric acid and silver nitrate utile per precipitare forms the solution This candidate has copied the reagent required for testing for halide ions from an earlier part of question 1. Consequently they were awarded 0 marks.

(ii) Describe a test, and its positive result, that the student could have carried out to show the presence of sulfate ions.

Ba2+ ion solution such as BaCly , into the mple, the positive pesult will be white

(2)



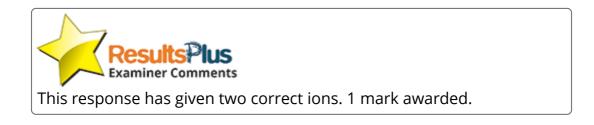
A correct response indicating that barium ions are required and that a white precipitate is produced if sulfate ions are present. 2 marks awarded.

Question 1 (d)

This question checked the candidates knowledge of the coloured ions of transition metals.

(d) Identify, by name or formula, a <u>metal cation</u>, other than chromium(III) and iron(II), which could give a **green** colour in an aqueous solution.

NIT 2+ VS+



(1)

Question 2 (a)(i)

....

This question was about an organic liquid and describes the reaction on the addition of water. About one third of all candidates were awarded all three marks. Many missed the link between the HCl produced in the first step and the NH₄Cl produced in the subsequent step.

2	Two organic compounds, X and Y, are colourless liquids.
	Each compound contains only one functional group.

(a) A few drops of deionised water are added to a beaker containing **X**. Misty fumes are formed.

A drop of concentrated ammonia on the tip of a glass rod is placed in the misty fumes. White smoke is formed.

(i) Deduce the functional group in X. Justify your answer by referring to the observations.

As reaction with water produced misty fumes of
HCLO, this shows it could be Acylchloride. When concentrated
Ammonia on glass rod comes in contact with misty
funes the white smoke of NH4CL forms so this contirms
that misty fumes Ewere HCLigs so the functional
group in x is Acyl chloride.

(3)



This clip shows a fully correct response. The acyl chloride has been identified, HCl fumes linked to misty fumes and NH₄Cl to the white smoke and so it was awarded 3 marks.

- 2 Two organic compounds, X and Y, are colourless liquids. Each compound contains only **one** functional group.
 - (a) A few drops of deionised water are added to a beaker containing **X**. Misty fumes are formed.

A drop of concentrated ammonia on the tip of a glass rod is placed in the misty fumes. White smoke is formed.

(i) Deduce the functional group in X.
 Justify your answer by referring to the observations.

concentrated ammonia forms white smoke with HClog.
This indicates that the misty fumes formed is HClego.
when few drops of deionised water is added to an acylchloride
, a carboxylic acid and HCI is produced. So the Functional
group in X is acyl chloride.



This candidate has correctly identified the functional group and the HCl as the misty fumes, however they have failed to mention NH_4Cl and so was only awarded 2 marks.

(3)

2 Two organic compounds, X and Y, are colourless Jquids. C-C. Each compound contains only one functional group: (a) A few drops of deionised water are added to a beaker containing X. 0 Misty fumes are formed. HCI. MHZ. A drop of concentrated ammonia on the tip of a glass rod is placed in the misty fumes. White smoke is formed. HCI + NH3 -> NHQ + C12. (i) Deduce the functional group in (\mathbf{X}) -Justify your answer by referring to the observations. (3)C-CI Become misty primes means howing MCI up produced, 40 the compound contains C-CI be functiona

This clip illustrates a common misconception, that X was a chloro alkane. The candidate has correctly spotted that the misty fumes are HCl and so was awarded 1 mark only.

Question 2 (a)(ii)

This question was about laboratory safety and how to reduce risk, with 90% of the candidates giving the correct response of using a fume cupboard.

 (ii) State the precaution that you would take to minimise the risk of carrying out this test on the misty fumes.
 Assume gloves, safety goggles and laboratory coat are worn.

The reaction should be done in a fume suppord.



 (ii) State the precaution that you would take to minimise the risk of carrying out this test on the misty fumes. Assume gloves, safety goggles and laboratory coat are worn.

(1)

(1)

Wear a face mask



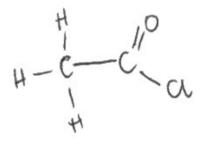
Question 2 (b)

In this part of the question, the candidates needed to use information from Q02(a) and a new piece of ¹³C NMR data.

(b) The ¹³C NMR spectrum of **X** has two peaks.

Draw the displayed formula of X.

(1)





A fully correct displayed formula of ethanoyl chloride, substance X, so 1 mark awarded.

(b) The ¹³C NMR spectrum of **X** has two peaks.

Draw the displayed formula of X.

$$\begin{array}{ccc} H & H & 0 \\ I & I & 0 \\ I & I & 0 \\ H & - C & - C & - C \\ H & H & H \end{array}$$

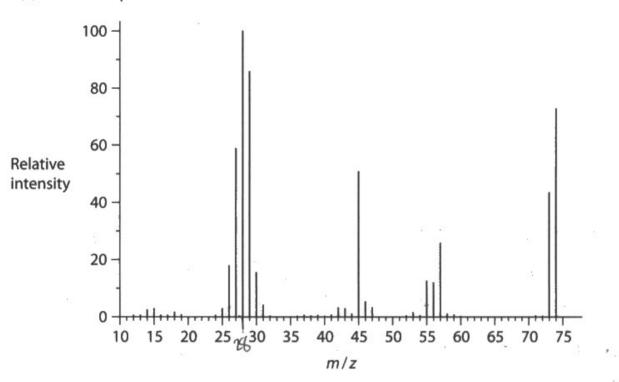


In this clip the candidate has correctly included the information from Q02(a) but has not read Q02(b). They have drawn propanoyl chloride and this compound would have three peaks in ¹³C NMR. 0 marks awarded.

Question 2 (c)(i)

Question 2 continues with the focus being substance Y for part (c). QO2(c)(i) concerned a mass spectrum of Y and a fragment at m/z = 45. Nearly half of the candidates missed this mark for a variety of reasons including:

- positive charge missing from an otherwise correct response.
- including a negative charge instead of a positive charge.
- including a "hanging bond" from an otherwise correct response.



(c) The mass spectrum of Y is shown.

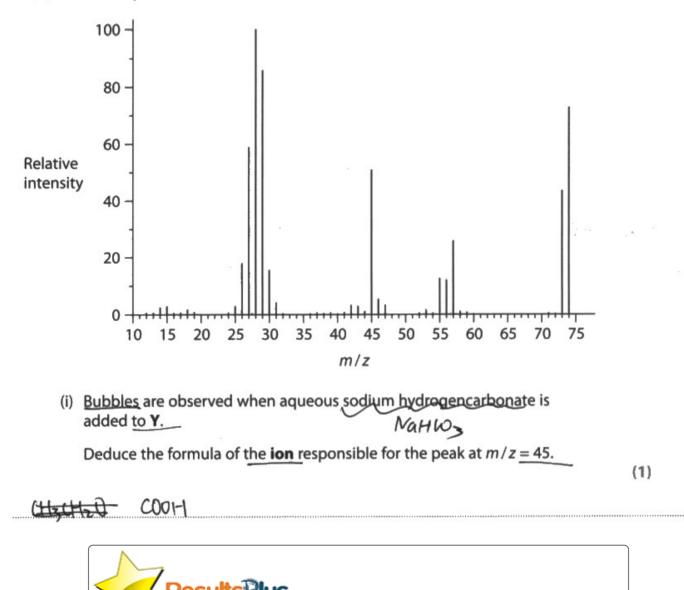
(i) Bubbles are observed when aqueous sodium hydrogencarbonate is added to **Y**.

Deduce the formula of the **ion** responsible for the peak at m/z = 45.

(1)

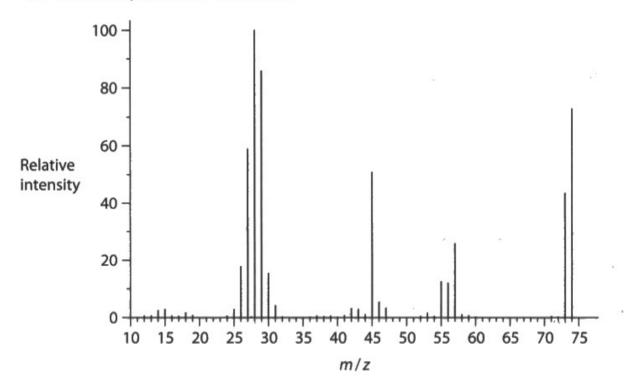


(c) The mass spectrum of Y is shown.



The positive charge was missing, so 0 marks awarded.

(c) The mass spectrum of Y is shown.

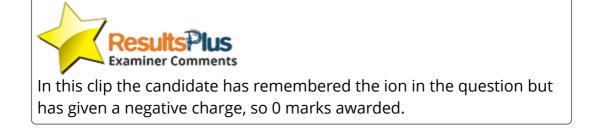


(i) Bubbles are observed when aqueous sodium hydrogencarbonate is added to Y. Corboxwica ad

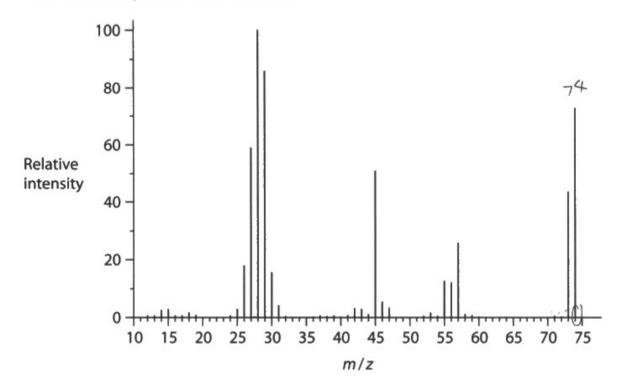
Deduce the formula of the **ion** responsible for the peak at m/z = 45.

(1)



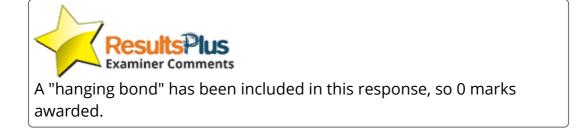


(c) The mass spectrum of Y is shown.



(i) Bubbles are observed when aqueous sodium hydrogencarbonate is added to **Y**.

Deduce the formula of the **ion** responsible for the peak at m/z = 45.

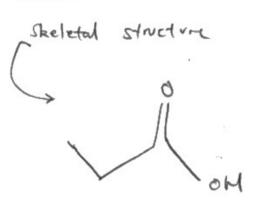


(1)

Question 2 (c)(ii)

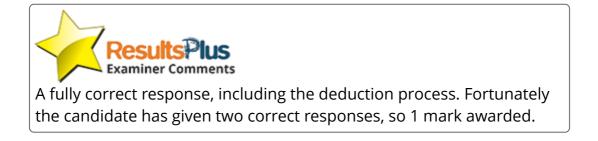
In this part of Q02(c) the candidates needed to use the m/z = 74, the molecular ion, and the mass of the COOH⁺ ion to deduce the structure of Y.

(ii) Draw the structural formula of Y.

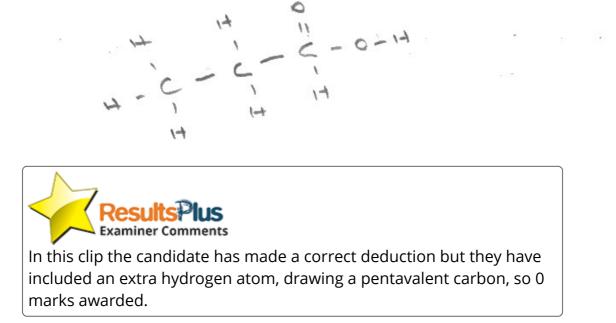


m/2 74 pmk (1) 74-405 = 27

CH3 CH2 COOH



(ii) Draw the structural formula of Y.

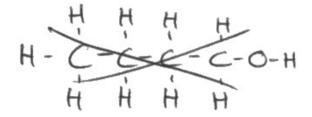


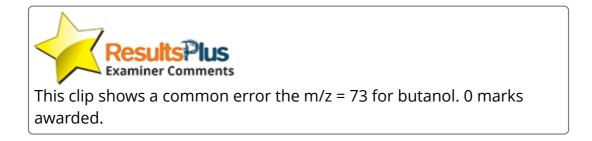
(ii) Draw the structural formula of Y.

CyHIDO

(1)

(1)





Question 2 (d)(i)

In this part of the question substances X and Y are directly compared. The candidates have been told that an ethyl ester is formed, they only need to give the name of an appropriate reagent, so a fairly straightforward question.

- (d) Both X and Y can be used to produce esters.
 - (i) Name the compound that would react with both X and Y to form ethyl esters.

(1)

ethanol



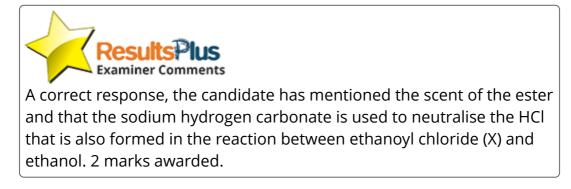
Question 2 (d)(ii)

This part of question 2 looks more closely at the esterification reaction, a further suggested practical. Many candidates failed to be awarded any marks on this question. This indicates that some practical procedures are overlooked.

(ii) A student prepared an ester using X and a suitable compound.

Explain why the student added aqueous sodium hydrogencarbonate to the reaction mixture to allow the presence of an ester to be detected.

The sodum hydrogen carbonate, neutralisis	r	HC	formed,
turebore no mushy himes are produced twelfore will			
smell to scent given off by the estor prope			
to a pare sumes of Hel.			



(ii) A student prepared an ester using **X** and a suitable compound.

Explain why the student added aqueous sodium hydrogencarbonate to the reaction mixture to allow the presence of an ester to be detected.

hydrogen carbonate reacts with cater Toubbes Soming Veing CO2 This clip shows a common misconception, that the sodium hydrogen carbonate reacts with the ester. 0 marks awarded.

(2)

(2)

Question 2 (e)

This final part of question 2 continues the comparison between substances X and Y. Here their reactions with concentrated ammonia are considered. Over half of the candidates were not awarded any marks for Q02(e).

(e) Both X and Y react with concentrated ammonia but form different products.

A fully correct response. A total of 2 marks awarded.

Identify these products, by name or formula. Product with X ethonamide Product with Y ammonium profancate (e) Both X and Y react with concentrated ammonia but form different products.

Identify these products, by name or formula.

(2)

Product with X CH2CH2COOTNH,

Product with Y

(H_C(NH_)4(H3



In this clip the product for X is an attempt to give the ammonium compound for Y, but the ammonium ion is incorrect.

The product for Y is incorrectly shown as an amine.

This shows common incorrect answers given in combination by other candidates. 0 marks awarded.

Question 3 (a)

Question 3 is based on the core practical 9a – following the rate of the iodine-propanone reaction by titrimetric method. Q03(a) concerned why part of the practical procedure is carried out. About two thirds of candidates failed to be awarded this mark, indicating that while candidates are able to follow practical methods, they are very unlikely to question the procedural steps.

3 This question is about an experiment to investigate the kinetics of the reaction between iodine and propanone with an acid catalyst. This Cu

The equation for the reaction is shown.

 $I_2(aq) + CH_3COCH_3(aq) + H^+(aq) \rightarrow CH_3COCH_2I(aq) + 2H^+(aq) + I^-(aq)$

To obtain the order of reaction with respect to iodine, the concentration of iodine in the reaction mixture was determined at various times.

Procedure

- Step 1 Mix 25 cm³ of 1.0 mol dm⁻³ sulfuric acid with 25 cm³ of 1.0 mol dm⁻³ propanone in a beaker.
- Step 2 Start a clock as 50 cm³ of 0.020 mol dm⁻³ iodine solution is added to the beaker. Mix the reactants thoroughly.
- Step 3 Tip a spatula measure of sodium hydrogencarbonate into a conical flask. After 3 minutes, pipette a 10.0 cm³ sample of the reaction mixture into the conical flask and mix thoroughly.
- Step **4** Titrate the iodine in the sample with 0.010 mol dm⁻³ sodium thiosulfate solution using a suitable indicator. Record the titre.
- Step 5 Repeat Steps 3 and 4 every 3 minutes to obtain four more titres.
- (a) State why the sulfuric acid and propanone concentrations are both much larger than the iodine concentration.

(1)

so that [theson] and tells (Octh) are in excess and remain constant so the only concentration changing is



3 This question is about an experiment to investigate the kinetics of the reaction between iodine and propanone with an acid catalyst.

The equation for the reaction is shown.

 $I_2(aq) + CH_3COCH_3(aq) + H^+(aq) \rightarrow CH_3COCH_2I(aq) + 2H^+(aq) + I^-(aq)$

To obtain the order of reaction with respect to iodine, the concentration of iodine in the reaction mixture was determined at various times.

Procedure

- Step 1 Mix 25 cm³ of 1.0 mol dm⁻³ sulfuric acid with 25 cm³ of 1.0 mol dm⁻³ propanone in a beaker.
- Step 2 Start a clock as 50 cm³ of 0.020 mol dm⁻³ iodine solution is added to the beaker. Mix the reactants thoroughly.
- Step 3 Tip a spatula measure of sodium hydrogencarbonate into a conical flask. After 3 minutes, pipette a 10.0 cm³ sample of the reaction mixture into the conical flask and mix thoroughly.
- Step 4 Titrate the iodine in the sample with 0.010 mol dm⁻³ sodium thiosulfate solution using a suitable indicator. Record the titre.
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- (a) State why the sulfuric acid and propanone concentrations are both much larger than the iodine concentration.

They 1	reed ho	be	<u>٥٨</u>	excess	<u></u> వల	icoline	can be	the
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Conitant reactant.
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A common response that missed the point of the question. The candidate has stated that the iodine is the limiting reactant. 0 marks awarded.

(1)

Question 3 (b)

Question 3 is based on the core practical 9a – following the rate of the iodine-propanone reaction by titrimetric method. For Q03(b) two thirds of candidates were awarded this mark and correctly stated that the addition of sodium hydrogen carbonate would neutralise the sulfuric acid and quench the reaction.

(b) State why sodium hydrogencarbonate is used in Step 3.

quenching to stop the reaction by neutralise sulfuric acid.

(1)



A perfect response citing both the acid neutralisation and the quenching of the iodine-propanone reaction. 1 mark awarded.

Question 3 (c)

Question 3 is based on the core practical 9a – following the rate of the iodine-propanone reaction by titrimetric method. This question was about the indicator employed in this practical. Nearly 60% of candidates correctly identified the indicator, starch, and the appropriate colour change at the end, blue-black to colourless. Some candidates named an acid-base indicator such as methyl orange and so missed both marks.

(c) Name the indicator that would be used for the titration in Step **4**, stating the colour **change** that would be seen at the end-point of the reaction.

(2)

starch would	be used as	an india	cator. The	colour	change.	Nould be
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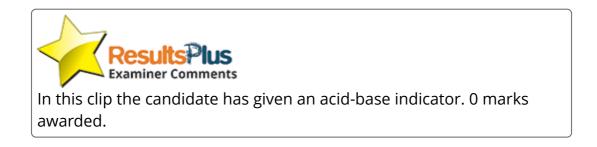
blue black to colourless.



(c) Name the indicator that would be used for the titration in Step **4**, stating the colour **change** that would be seen at the end-point of the reaction.

(2)

orange Uctrul rellow manae 10



Question 3 (d)(i)-(e)(ii)

Question 3 is based on the core practical 9a – following the rate of the iodine-propanone reaction by titrimetric method.

In Q03(d)(i) the candidates had to draw and label axes covering more than half the grid, plot the points and draw a straight line of best fit. It was apparent that many candidates were unable to find suitable scales for their graph.

In Q03(d)(ii) the candidates were asked why the volume of thiosulfate could be plotted instead of the concentration of iodine – many candidates just commented that it was easier.

In Q03(d)(iii) candidates had to give the order of react with respect to iodine and justify their answer by referring to the graph – many candidates lost the mark for stating an incorrect order.

In Q03(e)(i) candidates needed to interpolate half-lives from a given graph followed by some data manipulation. Some candidates lost a mark for giving half-lives as 7s and 15s.

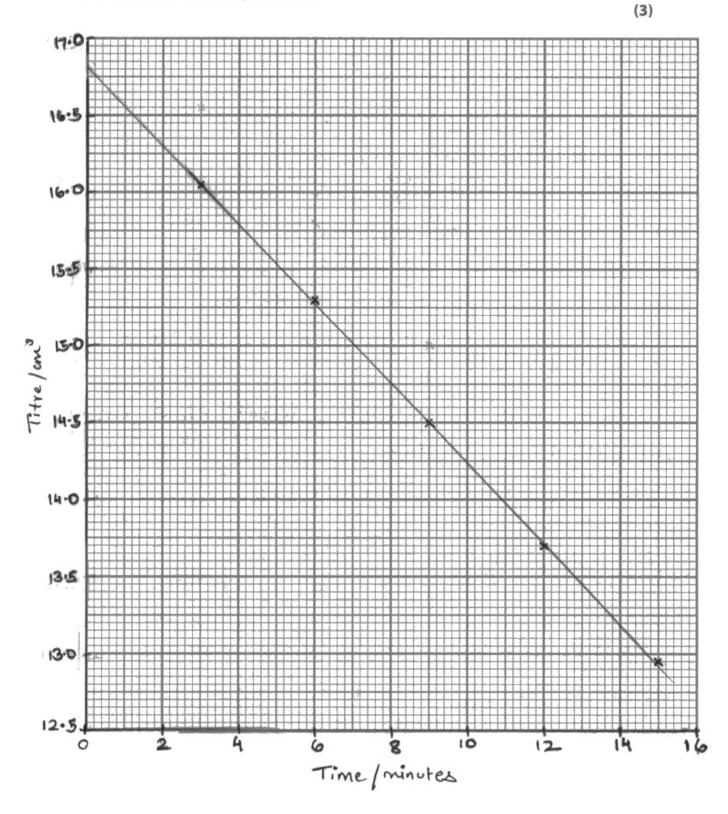
In Q03(e)(ii) the candidates needed to use information from the rest of Q03(d)(iii), Q03(e)(i) and Q03(e)(ii). This was mostly not awarded due to a missing rate constant (k) from the rate equation.

Very few candidates were awarded all 8 marks for these parts of question 3.

(d) Titration results from the experiment are shown.

Time/minutes	3	6	9	12	15
Titre/cm ³	16.05	15.30	14.50	13.70	12.95

(i) Plot a graph of titre against time.



(ii) State why the volume of thiosulfate may be used for plotting the graph rather than the concentration of iodine.

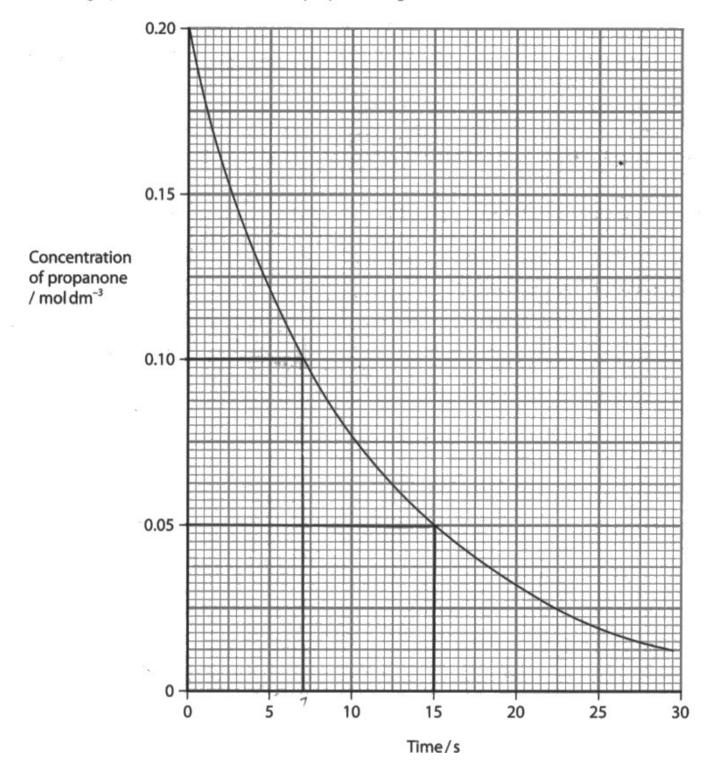
to the concentration of The volume io dehe 0 Proportiono 40

(iii) State the order of reaction with respect to iodine. Justify your answer by referring to your graph.

(1)

The rate Zero or des with respect fres it has a negative o cop line constant gradient. to Podel

- (e) Further experiments were carried out to determine the reaction orders with respect to propanone and sulfuric acid.
 - (i) A graph of the concentration of propanone against time is shown.



The reaction is first order with respect to propanone.

Determine two half-lives for this reaction. You **must** show your working on the graph.

(2)

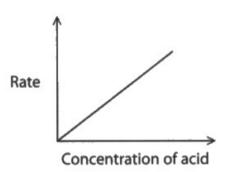
First half-life

73.

Second half-life

15-7 = 83.

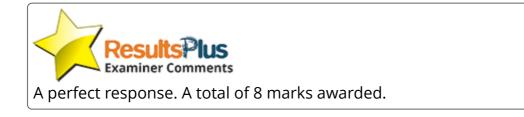
(ii) A graph of the reaction rate against the concentration of sulfuric acid is shown.



Deduce the rate equation for the overall reaction of iodine and propanone with an acid catalyst.

Use your answer from (d)(iii) and information from (e)(i) and the graph in (e)(ii).

Rate = K [CH3 COCH2] [H+]



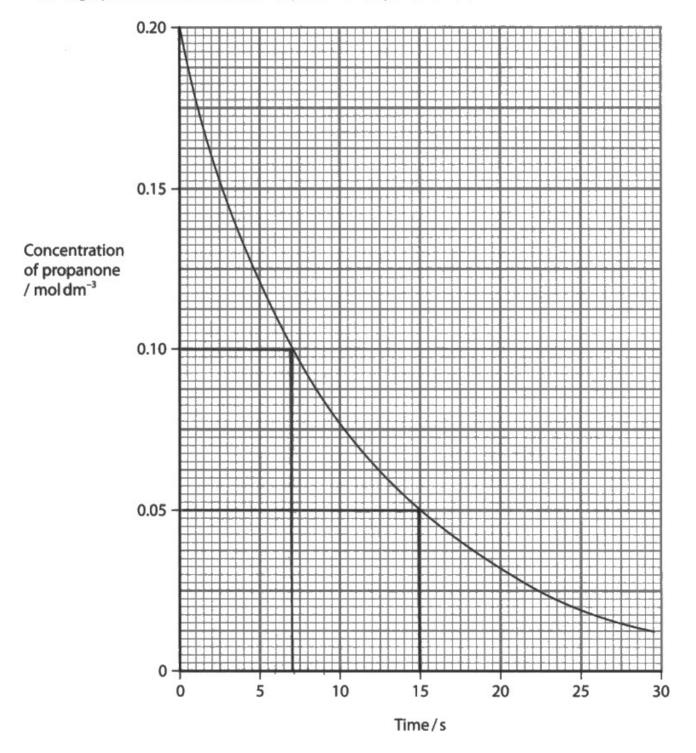
(d) Titration results from the experiment are shown.

Time/minutes	3	6	9	12	15
Titre/cm ³	16.05	15.30	14.50	13.70	12.95

(i) Plot a graph of titre against time. (3) ł 17 16 15 19 13 113 2 Ó 10 12 4 8 14 Timel minutes (ii) State why the volume of thiosulfate may be used for plotting the graph rather than the concentration of iodine.

beck	use velow		thiosul	fute	Pan	ís	more
acc	stare than	n (oncer	nitation	of	irdlag	as	th _z
conte	ntranten is	vnstable.	becal	-it-	may	ce 1110	<u>ــــــــــــــــــــــــــــــــــــ</u>
	the order of reactio y your answer by ref						(4)
		200	order				(1)

- (e) Further experiments were carried out to determine the reaction orders with respect to propanone and sulfuric acid.
 - (i) A graph of the concentration of propanone against time is shown.



The reaction is first order with respect to propanone.

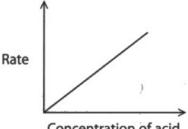
Determine two half-lives for this reaction. You **must** show your working on the graph. First half-life

7 s

Second half-life

155

(ii) A graph of the reaction rate against the concentration of sulfuric acid is shown.



Concentration of acid

Deduce the rate equation for the overall reaction of iodine and propanone with an acid catalyst.

Use your answer from (d)(iii) and information from (e)(i) and the graph in (e)(ii).



Here the candidate has lost a graph (Q03(d)(i)) mark due to an inappropriate scale.

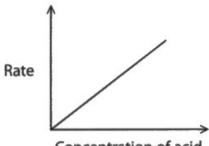
Q03(d)(ii) has not been awarded, as the candidate has not appreciated the connection between the volume of thiosulfate and iodine concentration.

Q03(d)(iii) has been lost as there is no justification.

For Q03(e)(i) the interpolation lines have been clearly drawn, but they have failed to complete the half-life determination for the second half-life.

A total of 4 marks awarded.

, (ii) A graph of the reaction rate against the concentration of sulfuric acid is shown.



Concentration of acid

Deduce the rate equation for the overall reaction of iodine and propanone with an acid catalyst.

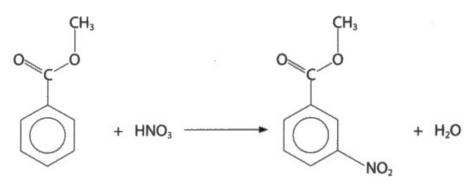
Use your answer from (d)(iii) and information from (e)(i) and the graph in (e)(ii).



Question 4 (a)

Question 4 was about a nitration of an arene compound. Here the candidates are given the practical procedure and are again asked to question various steps. In Q04(a) the candidates were asked about the use of an ice bath, rather than just using ice cubes to keep the temperature of the reaction below 15°C. Over 80% of the candidates failed to be awarded this mark as they failed to mention either more contact or that the cooling would be more effective.

4 A group of students prepared methyl 3-nitrobenzoate by the nitration of methyl benzoate.



Procedure

- Step 1 Measure 9 cm³ of concentrated sulfuric acid into a small, dry conical flask. Label the flask **A** and place it in an ice bath.
- Step 2 Add <u>4.0 cm</u>³ of <u>methyl benzoate</u> to flask **A**. Gently swirl the flask.
- Step 3 Mix 3 cm³ of concentrated nitric acid and 3 cm³ of concentrated sulfuric acid in a test tube to form the nitrating mixture.
 Place this test tube in the ice bath.
- Step 4 Place a thermometer in flask A. Add the nitrating mixture very slowly to flask A using a dropping pipette.
 Take care to ensure that the temperature of the flask contents does not rise above 15 °C.
- Step 5 Remove flask A from the ice bath and allow it to stand at room temperature for about 10 minutes.
 Pour the reaction mixture into a small beaker containing crushed ice.
 Stir the contents of the beaker with a glass rod.
- Step 6 Allow the ice to melt. Separate the solid methyl 3-nitrobenzoate by suction filtration. Wash the solid with a small amount of deionised water and then with a little ice-cold ethanol.
- Step 7 Recrystallise the methyl 3-nitrobenzoate using ethanol as the solvent.
- Step 8 Determine the melting temperature of the purified crystals of methyl 3-nitrobenzoate.
- (a) An ice bath is a mixture of ice and water in a beaker.

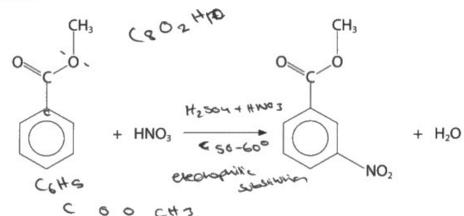
Suggest an advantage of using an ice bath in Steps 1 and 3 rather than a beaker containing only ice cubes. Justify your answer.

- To cool the beaker downfaster.	and lee - So you coal testra(1)	
ke bath contains very cold wat	tert which provide more contact	
with the beaker for quicker q		
cubes with less us surface area and	contact for cooling the vection	****



A perfect response, including a good comparison between just ice cubes and an ice bath. 1 mark awarded.

4 A group of students prepared methyl 3-nitrobenzoate by the nitration of methyl benzoate.



Procedure

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- (a) An ice bath is a mixture of ice and water in a beaker.

Suggest an advantage of using an ice bath in Steps **1** and **3** rather than a beaker containing only ice cubes. Justify your answer.

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rde d	readucy	7 helps	set ind	of wininum
source	imprises	Solution (coded	quierer



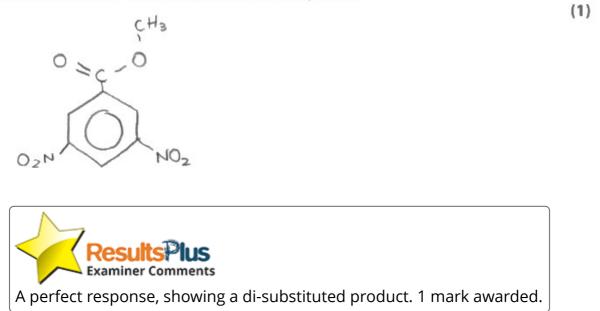
Here the candidate has given three answers. The comment on 'removing soluble impurities' is not correct. Finally a reference to cooling but no mention of surface area in contact is insufficient to score. 0 marks awarded.

Question 4 (b)

Question 4 was about a nitration of an arene compound. Here the candidates are asked to draw a possible structure of a nitrated arene that could be formed if the temperature is permitted to rise above 15°C. Just under half of the candidates were awarded this mark.

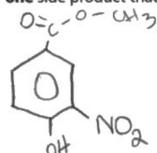
(b) Side products form if the temperature rises above 15 °C in Step 4.

Give the structure of one side product that may form.



(b) Side products form if the temperature rises above 15 °C in Step 4.

Give the structure of **one** side product that may form.



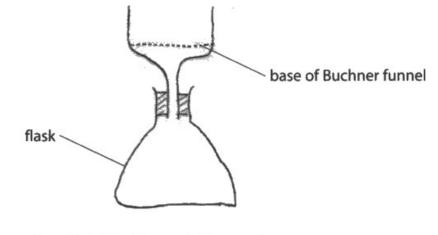


Question 4 (c)

Question 4 was about a nitration of an arene compound. Here the candidates are given the practical procedure and a "spot the errors" diagram of Buchner vacuum filtration apparatus.

The candidates had to mention:

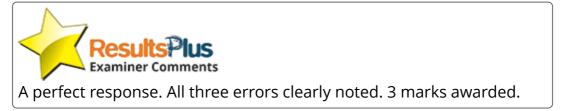
- a missing filter paper, not merely suggest that the dotted line labelled base of the Buchner funnel was incorrectly labelled, after all what would support the filter paper?
- the flask should be a Buchner flask instead of a conical flask.
- the presence of a vacuum pump, not merely an inference that "a vacuum needs to be connected".
 - (c) One student drew the suction filtration apparatus in Step 6 as shown.



Identify the **three** ways in which this diagram is incorrect. You may assume that the apparatus is suitably clamped.

(3)

•	no	side-c	urm	going	to	VQ	enn	- pi	mp	
•	no	vacu	um	pump)		*******			
				1,5,20			Buchn			
							ropht u			
							U I			



(c) One student drew the suction filtration apparatus in Step 6 as shown.

base of Buchner funnel flask Identify the three ways in which this diagram is incorrect. You may assume that the apparatus is suitably clamped. (3)an opening coming out of place so as to connect There must be vacuum pump in older for subba fittration to occur. offeel it is the lift Buchner Buc er pholes. mare flast. End 10 Mai ensure no lost products, an con hagram: er paper Buchnes 2 mare achum Examiner Comments This response was only awarded 2 marks, for the opening on the flask to connect to vacuum pump shown on the diagram below the text. The candidate was not awarded the filter paper mark as they have not included a solid line labelled filter paper in their own diagram.

Question 4 (d)

Question 4 was about a nitration of an arene compound. Here the candidates are given the practical procedure, step 7 mentions that the crude methyl 3-nitrobenzoate needs to be recrystallised. The question specifically asks for when each type of impurity (insoluble or soluble) are removed.

(d) Describe the stages in the recrystallisation of methyl 3-nitrobenzoate in Step 7, stating which stages are required to remove the insoluble and soluble impurities. Only outline details of the method are required.

(4)

Dissolve solid in minimum amount of hot solvent. This is to obtain a saturated

solution and minimise mass of solid left in solution. Filter the solution to

remove insoluble impurities. Cool filtrate in ice bath to maximise amount of

solid crystalline. Filter & by Using suction filtration to remove soluble

impurities. This allows solvent to be removed completely. Dry solid by

leave it in a warm place.



A perfect response. The candidate has written in a logical and concise manner, stating when each type of impurity has been removed. 4 marks awarded.

(d) Describe the stages in the recrystallisation of methyl 3-nitrobenzoate in Step 7, stating which stages are required to remove the insoluble and soluble impurities. Only outline details of the method are required.

perform a hot filtration to remove any insoluble
impurities, then perform a filtration with vacaun
to eliminate soluble impurities. Leave substance
in a dessicator to remove water which is an impurity
with a switchle drying agent such as call.
Add ethonol to remove impurities and dry with
a paper towel the crystals.



In this clip the candidate is relying on the examiner to assume that the crude product has been dissolved, so the dissolve in the minimum volume of hot ethanol was not awarded.

The candidate has correctly stated that the hot filtration is to remove insoluble impurities (lines 1 & 2).

The candidate has failed to mention that the filtrate needs to be cooled for crystals to form.

The candidate has stated that the vacuum filtration is to eliminate soluble impurities (lines 2 & 3).

2 marks awarded.



It might be helpful to write procedures as a logical sequence in bullet pointed steps.

(4)

Question 4 (e)

Question 4 was about a nitration of an arene compound. Here the candidates are given the practical procedure and are again asked to question various steps.

- (e) The crystals must be dried before the melting temperature can be determined. Methyl 3-nitrobenzoate cannot be dried by the addition of a solid drying agent such as anhydrous calcium chloride.
 - (i) Suggest why the addition of a solid drying agent is not suitable to dry methyl 3-nitrobenzoate.

(1)It is difficult to separate them

(ii) State how the crystals of methyl 3-nitrobenzoate could be dried.

(1)

By the devicator.

A brief but fully correct response. A total of 2 marks awarded.

- (e) The crystals must be dried before the melting temperature can be determined. Methyl 3-nitrobenzoate cannot be dried by the addition of a solid drying agent such as anhydrous calcium chloride.
 - (i) Suggest why the addition of a solid drying agent is not suitable to dry methyl 3-nitrobenzoate.

The product many is a solid and so it will not react with another solid drying agent, as both reactants are solids



Q04(e)(i) the drying agent reacting with the product, was a popular incorrect answer. 0 marks awarded for Q04(e)(i).

Question 4 (f)

Question 4 was about a nitration of an arene compound. In this part of the question the candidates were asked to determine the percentage yield, given an experimental mass of product formed. This question was generally well answered with over 60% of the candidates being awarded all three marks.

(f) The mass of dry methyl 3-nitrobenzoate crystals prepared by one of the students was 3.05 g.

Calculate the percentage yield by mass of methyl 3-nitrobenzoate using the data shown.

Compound Molar mass/gmol⁻¹ Density/gcm⁻³ methyl benzoate 136 1.08 methyl 3-nitrobenzoate 181 n(methyl benzoate 6944 mol. n(meth 3-nitrobenzoate) Percentage v %=53.3%



An excellent response, laid out in a logical manner. 3 marks awarded.

(f) The mass of dry methyl 3-nitrobenzoate crystals prepared by one of the students was 3.05 g.

Calculate the percentage yield by mass of methyl 3-nitrobenzoate using the data shown.

Compound	Molar mass/g mol ⁻¹	Density/g cm ^{−3}
methyl benzoate	136	1.08
methyl 3-nitrobenzoate	181	>

$$mol = \frac{3.05}{181}$$

Examiner Comments In this clip the candidate has evaluated the number of moles of methyl 3-nitrobenzoate only and this was not enough to gain a mark. There were many responses of this type scoring 0 marks.

(3)

Question 4 (g)

Question 4 was about a nitration of an arene compound. Here the candidates are given the practical procedure and are again asked to question various steps. In this part of the question the candidates were asked to suggest a melting point of the crude product and give a justification for their answer. About two thirds of candidates failed to be awarded any marks.

(g) The melting temperature range of methyl 3-nitrobenzoate is given in a data book as 78–80 °C.

Suggest a melting temperature **range** for a sample of the methyl 3-nitrobenzoate **before** recrystallisation. Justify your answer.

76°C as the impurity over the M lting Malt over a Mirease M the re

(2)



A fully correct response, mentioning both an appropriate range (between 70°C and 79°C) and with a justification that impurities would both depress the melting point and increase the range of temperatures over which the change of state occurs. 2 marks awarded. (g) The melting temperature range of methyl 3-nitrobenzoate is given in a data book as 78–80 °C.

Suggest a melting temperature **range** for a sample of the methyl 3-nitrobenzoate **before** recrystallisation. Justify your answer.

Meching Melting temperature range will be lower, about 70 ~ 75°C because lifzuid such as water and ethanal Present in methil 3-nitroben

zoate.



In this response the candidate has given an appropriate range but has not given any justification. 1 mark awarded.

(g) The melting temperature range of methyl 3-nitrobenzoate is given in a data book as 78-80 °C.

Suggest a melting temperature **range** for a sample of the methyl 3-nitrobenzoate **before** recrystallisation. Justify your answer.

before recrystablisation meeting temperature range will be maccurate as imporities will and there for me meeting temper and larger and there re unaccurate



In this response the candidate has attempted the justification and mentioned a larger range, but they have failed to mention that the melting point range has been depressed. They have not suggested a melting temperature range. 0 marks awarded. (2)

(2)

Paper Summary

Based on their performance on this paper, candidates should:

- Ensure that they read and re-read the question, paying particular attention to the command words and checking that their response answers the question.
- Practice experimental techniques and if unable to do so personally then watch demonstrations as this will help with recalling the techniques. While carrying out practical work, spend time thinking about why a particular procedure is being used rather than another.
- Practice drawing apparatus and being able to spot deliberate errors on diagrams.
- Show all working for calculations, in a logical manner, and remember to round the final answer only.
- Ensure that graphs are drawn with an appropriate scale that should cover half the grid in either direction.
- Make sure that when interpolating information from a graph, the lines are drawn with care and also remember to include the correct units when giving the answer.

Grade boundaries

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