Examiners' Report
June 2022

IAL Chemistry WCH16 01

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Introduction

This paper proved accessible to candidates of all ability levels. Many were able to demonstrate a sound knowledge and understanding of the chemistry and a familiarity with the practical techniques involved. A few candidates were not able to apply their knowledge to the specific context or gave answers which lacked precision, and these would benefit from more extensive direct experience of the common practical techniques to ensure that they know the basic facts and can express their ideas clearly and appropriately.

Candidates again proved their ability to answer calculation questions with many fully correct answers. Good presentation skills made it straightforward to award partial credit when errors had been made.

Question 1 (a)(i)

This question was a straightforward qualitative analysis on copper salts and most candidates were able to score both marks. Those that opted to give the name rather than the formula sometimes lost a mark for not including the oxidation state of the copper ion.

Question 1 (a)(ii)

Some candidates had not read the question carefully and thought that the test for chloride ions on the original solution was required, not realising that this test had already been done and a confirmatory test should be given. Many students were able to identify the correct reagent to test for the chloride ions, however, in some cases students did not specify that the ammonia had to be dilute or aqueous or that the sulphuric acid had to be concentrated. The concentrations of the reagents are important in this context as concentrated ammonia will dissolve both silver bromide and silver iodide and fumes of HCl are not evolved if the added acid is not concentrated.

Question 1 (b)(ii)

The ammonium ion was correctly identified by many but the formula of the dichromate ion, $Cr_2O_7^{2-}$ caused more difficulties. CrO_4, CrO_7^- and $Cr_2O_7^-$ were seen.

Question 2 (a)(i)

The tests for aldehydes and ketones were well known with many awarded at least 2 marks here. Candidates should appreciate that an inference may be drawn from the absence of a change in the reagent or mixture and therefore stating that there is no observation is incorrect.

2 A student is asked to identify three colourless liquids labelled **C**, **D** and **E**.
The compounds are all non-cyclic isomers with the formula $C_5H_{10}O$.

(a) (i) The student carries out three tests on separate samples of each compound.

Complete Table 1 to show the observations that the student makes.

(3)

| Sample | Observations on addition of reagents | | | Functional group |
	2,4-dinitro phenylhydrazine (Brady's reagent)	Fehling's or Benedict's solution and warm	$I_2(aq)$ and NaOH(aq) and warm	
C	orange precipitate	~~solution turns~~ ~~red~~ no reaction *(blue solution)*	pale yellow precipitate antiseptic smell	$R{-}COCH_3$
D	no reaction	no reaction *(blue solution)*	pale yellow precipitate antiseptic smell	$R{-}CH(OH)CH_3$
E	orange precipitate	solution turns red	no reaction	$R{-}CHO$

Table 1

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In this clip there are four observations where there is no observable change upon the addition of the reagent.

No reaction or no change is an appropriate response. In the reaction of liquid E with Fehling's or Benedict's, just the change in colour is not sufficient to gain the mark; the appearance of a precipitate must be stated.

2 A student is asked to identify three colourless liquids labelled **C**, **D** and **E**. The compounds are all non-cyclic isomers with the formula $C_5H_{10}O$.

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Complete Table 1 to show the observations that the student makes.

(3)

| Sample | Observations on addition of reagents | | | Functional group |
	2,4-dinitro phenylhydrazine (Brady's reagent)	Fehling's or Benedict's solution and warm	$I_2(aq)$ and $NaOH(aq)$ and warm	
C	orange precipitate	no observations no change	pale yellow precipitate antiseptic smell	R-COCH₃ methyl ketone
D	no precipitate (no observations) no change	no observations no change	pale yellow precipitate antiseptic smell	R-CH(OH)CH₃ 2° alcohol
E	orange precipitate	brick red precipitate forms	no observations /no change	R-CHO aldehyde

Table 1

ResultsPlus
Examiner Comments

In this example "no observation" would have been penalised, although once only.

The addition of "no change" allows all marks to be scored.

Question 2 (b)(i)

The best responses gave a concise and correctly ordered method for the recrystallisation process although some candidates misread the question and wrote details of measuring the melting temperature of a crystal.

The reason for recrystallisation prior to the determination of a melting temperature is to remove impurities. There were some responses where the order was not logical eg filtering before dissolving or washing before filtering the crystals. Some candidates did not refer to hot solvent being used in the first step and there were some responses that missed crucial details for marking points eg, cooling the solution after filtration and washing crystals with cold solvent after filtration. Most students referred to a correct drying process, with many referring to the use of a desiccator.

In general, the more concise responses reflected clarity of thought and tended to score well.

(b) (i) The orange precipitates produced on the addition of Brady's reagent to samples of **C** and **E** are called derivatives.
The melting temperatures of derivatives are used to confirm the identity of compounds.

Solids are recrystallised so that their melting temperatures may be determined accurately.

Describe in outline the procedure for the recrystallisation of a solid.

(4)

- dissolve the crystals in the minimum amount of hot solvent

- Cool the solution in an ice bath

- Wait for crystals to form

- Dry the crystals by heating in a warm oven
                          Placing

Question 2 (b)(ii)

Many candidates correctly identified pentan-2-one as the compound and gave a good explanation of their selection. Most either referred to the presence of a methyl ketone group or the presence of a ketone group in addition to the closeness of the measured melting temperature compared to the databook value.

Candidates who only considered the melting temperature and disregarded the information in Table 1 frequently identified **C** as 2-ethyl butanal, perhaps unaware that impure materials have a lower melting temperature than pure compounds. Some responses were seen where candidates suggested a compound that was not in the table.

(ii) The melting temperatures of the derivatives of the reaction of Brady's reagent with some aldehydes and ketones are shown.

Carbonyl compound	Melting temperature of the derivative / °C
3-methylbutanal	123
2-ethylbutanal	134
pentan-2-one	144
cyclopentanone	146
pentan-3-one	156

Table 2

The melting temperature of the recrystallised derivative of sample **C** is found to be 138–140 °C.

Suggest the identity of compound **C**.
Justify your choice, using the information at the start of the question and the data in Tables **1** and **2**.

(2)

C is penta-2-one because the melting temperature is close to 144°C and penta-2-one has a methyl ketone group ~ as shown by the iodoform test. C is non-cylic so cannot be cyclopentanone, Penta-3-one is not R-COCH₃ so C cannot be Penta-3-one. C is a ketone so cannot be an aldehyde. 3-methylbutanal or 2-ethyl butanal.

Question 2 (c)

This question once again required candidates to use the information previously given in earlier parts of the paper. Many were able to reason that, since the proton NMR signal from the protons which were not in the aldehyde group, formed a singlet then they must be in identical environments and the structure must therefore be tertiary. It was disappointing when the correct structure was given but the protons were not clearly labelled and failed to score M2.
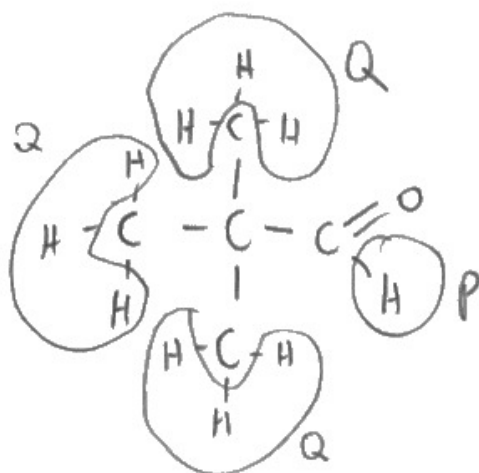
(c) The **high** resolution proton NMR spectrum of **E** is shown.
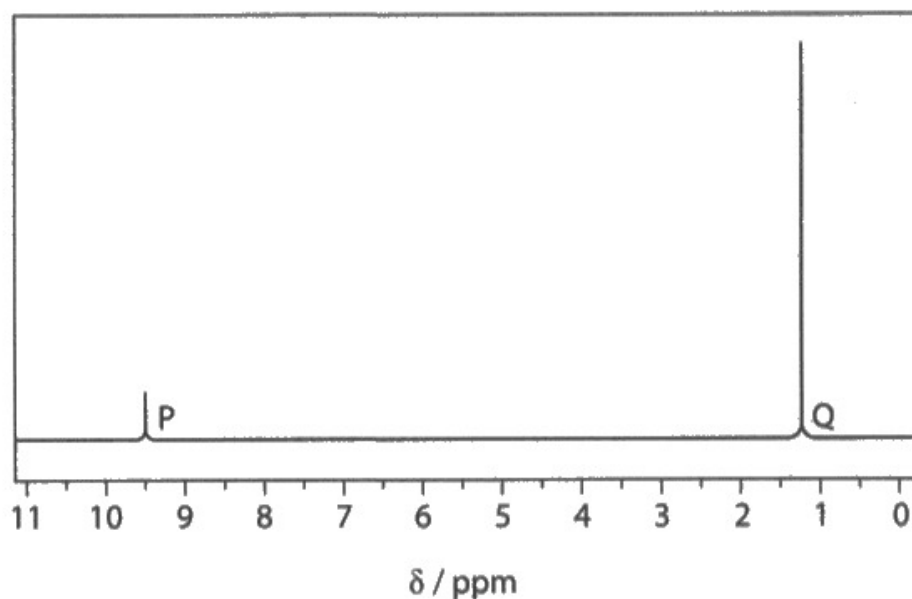
The relative numbers of protons responsible for the singlet peaks shown are P = 1 and Q = 9.

Use this information and the results of the tests in (a) to draw the displayed formula of **E**, labelling the proton environments.

(2)

(Total for Question 2 = 12 marks)

Question 3 (a)(i)

Many candidates were able to correctly identify both hazards and suggest the correct precautions. There were some incorrect hazards quoted from later stages in the determination such as iodine or sodium thiosulphate being toxic. Some students suggested the use of a mask as a precaution for NO_2 rather than a fume cupboard and occasionally there were references to NO_2 or HNO_3 being flammable.

Question 3 (a)(ii)

The most common mark scored was 1 for identifying the blue/green solution of copper ions. Marks were lost due to vague descriptions such as referring to a colour change to blue without stating that a solution was formed. There were also references to 'a gas is given off' or 'effervescence' or 'solid dissolves' which were too vague to be considered worthy of credit.

(ii) State what would be **seen** during the reaction.

(2)

Reddish brown copper dissolves. Colour of solution turns from colourless to blue. Brown NO_2 gas is seen.

ResultsPlus Examiner Comments

This response scores both marks. The production of a blue/green solution of copper (II) ions and the evolution of brown NO_2 gas are both clearly stated.

ResultsPlus Examiner Tip

The identification of gases produced during chemical reactions is frequently tested. Candidates would benefit from learning the properties of those named in the specification including ammonia, carbon dioxide and nitrogen dioxide.

Question 3 (a)(iii)

The addition of sodium carbonate or sodium hydrogen carbonate solution to neutralise acidic solutions is a common practical technique and appears as part of several Core Practicals. So it was surprising that relatively few candidates realised that the neutralisation would produce carbon dioxide very rapidly if the acid were not cooled. Many recognised that the neutralisation reaction was exothermic but without a link to effervescence or a vigorous reaction this failed to score. There were some responses that gained credit by stating that a reduction in temperature would slow the reaction.

(iii) Excess nitric acid must be neutralised before the iodide ions are added in Stage **2**.

Sodium carbonate solution is slowly added to the cooled solution.
The solution is neutralised when a faint precipitate appears.

Suggest why the solution is cooled before the addition of the sodium carbonate solution.

(1)

There is bubbling of the solution as carbon dioxide escapes and if solution was hot, it may spill vigorously.

ResultsPlus
Examiner Comments

This response correctly links the production of carbon dioxide gas during the neutralisation reaction to the danger of the mixture spilling over.

Question 3 (a)(iv)

Many candidates were able to score marks in this question. The best responses gave a concise and ordered method for the preparation of a standard solution using the correct apparatus. The requirement stated in the question for the solution to have a final volume of $250.0cm^3$ should have alerted candidates to the need for a volumetric flask, but there was a significant number of responses that instead used a beaker, conical flask, measuring cylinder or burette. Some did not refer to making up the solution to the mark or the line and instead just referred to adding $250cm^3$ of water. The most frequent omission was the addition of washings to the flask.

(iv) Ethanoic acid solution is added, drop by drop, to the neutralised mixture until the precipitate redissolves.

Describe how the solution in the beaker should then be made into $250.0\,cm^3$ of a homogeneous solution before Stage **2**.

(3)

Add the solution from the beaker to a graduted volumetric flask. Wash the beaker throughly with distilled water and add the washings to the volumetric flask until all the solution is transffered. Make the solution up to mark and shake and mix.

ResultsPlus Examiner Comments

This response uses the correct apparatus and describes a procedure to transfer all the solution to the volumetric flask so scores M1. Both M2 and M3 are scored in the final sentence.

Question 3 (b)

The most frequent score for this question was 2 for part(i) and 0 for part(ii).

Most candidates were able to correctly calculate the Ecell value and link the negative value to the lack of feasibility. Some calculated a negative value that was incorrect but earned M2 for making a similar link.

There were many valiant attempts at part (ii). There was evidence that candidates understood that a reaction with a small positive Ecell value, so thermodynamically feasible, might not take place due to kinetic factors such as a high activation energy. They reasoned therefore that, in this case, where a reaction with a negative Ecell value did occur, a reverse argument could be invoked and references to ' lower than expected activation energy' were often seen.

Credit was given to those candidates who recognised that the values for the standard electrode potentials were valid only under standard conditions of concentration. Responses that made general references to different conditions of temperature and/or pressure were not creditworthy. The best responses explained that CuI was precipitated and thus the equilibrium shifted right to produce more Cu+.

(b) In Stage **2**, 25.0 cm³ portions of the solution from Stage **1** are transferred into conical flasks.

10 cm³ of a solution of potassium iodide is added to each flask and the mixtures are swirled. Copper(I) iodide is precipitated.

The redox equation for the reaction between copper(II) ions and iodide ions is shown.

$$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$$
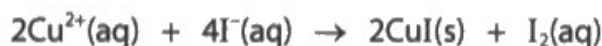
The standard electrode potentials for the half-equations are

Reduction $\quad Cu^{2+} + e^- \rightleftharpoons Cu^+ \qquad E^\ominus = +0.15\,V$

Oxidation $\quad\quad I_2 + 2e^- \rightleftharpoons 2I^- \qquad E^\ominus = +0.54\,V$

(i) Use the electrode potential data to explain why the redox reaction might not be expected to be feasible.

(2)

$E^\ominus_{cell} = -0.54 + 0.15 = -0.39\,V$

$E^\ominus_{cell}$ is negative, so reaction is not thermodynamically feasible.

(ii) Explain why the redox reaction does, in fact, take place.

(2)

CuI formed is solid, so there is always no Cu⁺ ions in the solution, so ~~equili~~ equilibrium of Cu²⁺ reduction is tend to the side of Cu⁺. This make the E⁰ of reduction of Cu²⁺ more positive

This response scored full marks in part(i).

The recognition that the precipitation of copper(I) iodide, removing Cu^+ ions from the reduction equilibrium would have an effect on the Cu^{2+}/Cu^+ cell potential was clearly expressed and scored both marks in (ii).

Question 3 (c)(i)

Many candidates scored a mark for correctly identifying that a solid complex would be formed at high concentrations of iodine. There were also references to the irreversible nature of the complex formation which would cause problems with the observation of the end point. Some responses focussed on the addition of a starch indicator in this titration "to make the end point easier to see" rather than the timing of the addition. These failed to answer the question adequately.

(i) Give a possible reason why the starch indicator is added when the iodine colour is pale yellow rather than at the start of the titration.

(1)

because iodine was clear colour (brown).
pale yellow is not clear enough to be seen.

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Examiner Comments

This response does not score the mark as it explains that the difficulty in detecting a colour change from pale yellow to colourless requires the addition of a starch indicator. This is not the question that was asked.

(i) Give a possible reason why the starch indicator is added when the iodine colour is pale yellow rather than at the start of the titration.

(1)

If addded at the begining a starch iodine insoluble complex would form.

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Examiner Comments

This response answers the question correctly.

Question 3 (c)(iii)-(iv)

Most candidates correctly calculated the mean titre using only concordant values. There were some responses that used all 3 titre values to calculate the mean but in these cases, there was the opportunity for TE into part (iv). Only a small proportion of responses showed incorrect stoichiometry, but a greater number neglected to scale the mass in the 25cm^3 aliquot up to the volume of the solution which was 250cm^3 . Many candidates had heeded advice to set out their work clearly.

(iii) The results of the titration are shown.

Titration number	1	2	3
Final burette reading / cm³	27.05	28.65	27.45
Initial burette reading / cm³	0.00	2.00	1.00
Titre / cm³	27.05	26.65	26.45

Complete the table and calculate the mean titre of the concordant results.

(1)

$$\text{mean titre} = \frac{27.05 + 26.65 + 26.45}{3} = 26.72 \ cm^3$$

(iv) The redox equation for the reaction between copper(II) ions and iodide ions is shown.

$$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$$
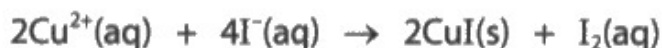
The reaction of sodium thiosulfate solution with iodine is shown.

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

Calculate the percentage by mass of copper in the 2.53 g of brass.

[A_r Cu = 63.5]

(3)

$$0.095 \ mol/dm^3 \rightarrow S_2O_3^{2-}$$

$$0.095 \times \frac{26.72}{1000} = 2.5384 \times 10^{-3} \ mol \ (S_2O_3^{2-})$$

$$mol \ of \ Cu^{2+} = 2.5384 \times 10^{-3} \ mol$$

$$mass \ of \ Cu^{2+} = 63.5 (2.5384 \times 10^{-3}) = 0.1611884 \ g$$

$$\frac{0.1611884}{2.53} \times 100 = 6.37 \ \%.$$

Only the two concordant titre values, titres 2 and 3, should have been included in the mean titre so part (iii) does not score the mark.

However, the incorrect value has been used to calculate the moles of thiosulfate in (iv) and scores 1 as transferred error.

The 1:1 stoichiometry of Cu^{2+}: thiosulfate is correct to find the mass of copper(II) and scores M2.

Unfortunately, this candidate has then failed to take into account the ratio of the volume of the copper(II) solution used for the titration (25.0cm^3) and the total volume of the solution (250.0cm^3) so the value is only one tenth of the correct answer.

Calculations that are set out clearly allow partial credit to be awarded.

Question 3 (c)(v)

Some candidates were able to score M1 for correctly identifying that more iodine would be produced as a result of not neutralising nitric acid. Most of those candidates also scored M2 for linking this to an increased titre value of thiosulphate and thus a higher % of copper. Some candidates however were not clear in their explanation of how the increase would affect the % copper calculated. There was evidence that a minority of candidates had not realised that the nitric acid was in excess and therefore needed to be neutralised. They then proposed that the nitric acid would react with copper and reduce the % in the brass sample.

(v) Nitric acid reacts with iodide ions to form iodine.

In another experiment the nitric acid was **not** neutralised before the start of Stage **2**.

Explain the effect on the value obtained for the percentage of copper in the sample.

(2)

The mean titre of $S_2O_3^{2-}$ would be greater since there is more iodine in solution to react. and be titrated. Therefore, moles of Cu^{2+} calculated is greater and hence percentage of copper value is greater.

ResultsPlus Examiner Comments

This response produces a well-reasoned answer that clearly links the increase in iodine leading to more thiosulfate being used in the titration. The statement at the end of the response indicating the increase in % copper answers the question.

ResultsPlus Examiner Tip

Explanations frequently need careful attention to the logical steps involved.

Question 4 (a)(ii)

Steam distillation appeared to be a well-known technique but the reason for its use in this case was not widely understood.

Some candidates offered explanations based on the difference in boiling temperature between water and limonene or the non-miscibility of the two liquids.

(ii) Give a reason for using steam distillation to extract limonene.

(1)

to extractlim to boil limonene before it reaches it boiling temperture at temperture lower than 176°C avoid decomposition of limonene.

Question 4 (a)(iii)

This was answered correctly by most candidates.

Question 4 (b)(i)

This was considered to be a straightforward question and a surprisingly large number of responses were blank.

A relatively large minority of candidates indicated that the chiral carbon was on the end of the C=C double bond.

Question 4 (b)(ii)

Many candidates were able to refer to the rotation of plane polarised light but fewer were able to correctly name the apparatus used to measure this and photometers and spectrometers were sometimes mentioned.

Some responses indicated some confusion about the type of light used, naming polarised or monochromatic light. There were also incorrect references to bending, reflecting or rotating light. A few referred to monochromatic light.

(ii) The limonene extracted from orange peel is D-limonene.

State how the presence of a single optical isomer could be confirmed, naming the apparatus used. No experimental details are required.

(2)

using polarimeter. It rotates the plane of plane polarised light.

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Examiner Comments

This is a concise, accurate response which uses the correct terminology to describe the apparatus and the effect of a single optical isomer on plane-polarised light.

Question 4 (b)(iii)

There was an error in this question. The density of D-limonene was given as 0.851 g cm^3. The value was correct, but the units were incorrect. Many candidates did not notice the typographical error and calculated the mass of limonene obtained by the student as expected and went on to correctly determine the moles of limonene. Sometimes the incorrect units given caused candidates to use the wrong formula to calculate the mass of limonene, but the incorrect value was accepted, and full credit was available.

The most frequent error was in calculating an incorrect Mr and so a failure to score M2 but most were able to score TE for M3.

(iii) A student obtained 1.20 cm^3 of limonene.

Calculate the amount, in moles, of limonene produced.

(3)

$$d = \frac{M}{V}$$

$$\downarrow$$

$$0.851$$

$$n \cdot \frac{M}{Mr}$$

$$m = 0.851 \times 1.20 = 1.0212 \, g$$

$$Mr \; limarere = C_{40}H_{16} = 136 \qquad (12 \times 10) + 16$$

$$n = \frac{M}{Mr} = \frac{1.0212}{136} = 7.51 \times 10^{-3} \, mol$$

ResultsPlus
Examiner Comments

The correct relationship between mass, volume and density is shown and the candidate goes on to calculate the moles of limonene using a correct Mr. Full marks scored.

(iii) A student obtained 1.20 cm³ of limonene.

Calculate the amount, in moles, of limonene produced.

(3)

$density = 0.851 \, g \, cm^3$

$density = mass \times volume$

$0.000851 = mass \times 0.0012$

$mass = \dfrac{0.000851}{0.0012}$ $mass = 0.709$

$number \, of \, moles = \dfrac{mass}{Mr} = \dfrac{0.709}{136} = 5.21 \times 10^{-3} \, mol.$
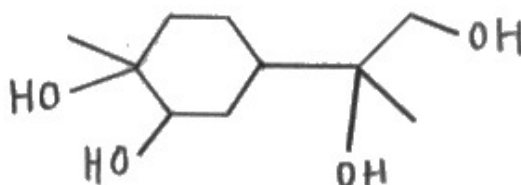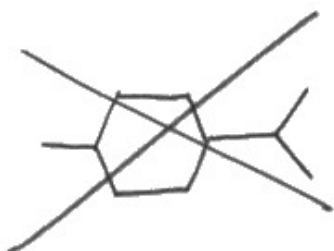
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Examiner Comments

This candidate has used the incorrect units given and consequently has made an error in the calculation of the mass of limonene produced. This has been ignored and full credit is given for the mass determined. The correct Mr is used to evaluate the moles limonene and full credit is given.

Question 4 (c)(ii)

Few candidates recognised that the use of excess potassium manganate(VII) would lead to the addition of two hydroxy groups and instead added a single hydroxy group to each double bond.

(ii) Draw the structure of the organic product which might be expected when **excess** acidified potassium manganate(VII) reacts with limonene.

(1)

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Examiner Comments

This candidate has correctly oxidised both C=C double bonds to produce diols. There is also a clear indication of an answer which is not to be marked, which has been crossed through.

Paper Summary

Based on their performance on this paper, candidates are advised to:

- read questions carefully and check that their answer addresses the question asked, including the required information
- use all the information given that might be necessary to produce answers
- revise the core practicals in the specification using all the resources available on the Pearson website and review the further suggested practicals
- practise naming apparatus and reviewing the use of common laboratory equipment
- show working for calculations clearly.

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