Commentary on candidate evidence

The candidate evidence has achieved the following marks for each section of the project.

Candidate 1

1 Abstract

The candidate was awarded **1 out of 1 mark** because the aim and findings that have been given are consistent with the conclusion.

The results are quoted as '% purity', which is not what the candidate determined, but as the conclusion given in the abstract is consistent with that given in the conclusion section, the mark was awarded.

2 Underlying chemistry

The candidate was awarded **2 out of 3 marks** because overall, the candidate has demonstrated a **reasonable** understanding of the underlying chemistry of the project.

Relevant underlying chemistry includes:

- A good description of back titration, with an example calculation and relevant balanced equations included.
- A description of making a standard solution and of primary standards is included. On page 6, the candidate correctly states that due to the insolubility of calcium carbonate a back titration must be carried out, however, in relation to standard solutions, they have also stated that it is a primary standard, that was made into a standard solution.
- Indicator chemistry however, this could have been further expanded to explain why phenolphthalein is a suitable indicator. How the colours of phenolphthalein arise could also have been included.
- A definition of Bronsted-Lowry is given, and it correctly describes the reaction involved in the project as an acid-base reaction, but the example given is for water.

The candidate included additional information that was not relevant. The following irrelevant information was not considered when awarding the marks for this section:

- resonance structures for carbonate ion and an attempt to explain these using sigma and pi bonding
- orbital box notation

- rules for electronic configuration
- quantum number information
- weak acid and pH of weak acid

% purity is an incorrect term and it is used throughout the project.

3 Data collection and handling

The candidate was awarded **5 out of 9 marks**. The marks were awarded as follows:

3(a) **1 out of 2 marks** was awarded because only one procedure has been given. The procedure does not contain the details of the titration end point colour changes which are required to follow the procedure, and so 2 marks could not be awarded. The titration colour changes are included in the results section.

There is no indication given as to what the 25 cm³ samples were pipetted into or whether all four samples were pipetted into the same flask. However, an Advanced Higher candidate could repeat the procedure and this information is clear when you look at the results section, and so 1 mark was awarded.

3(b) **1 out of 1 mark** was awarded because all the chemicals requiring additional safety measures have been included with justification.

1 M HCl and phenolphthalein have been included in the risk assessment, however, as these would not require additional safety measures, these did not need to be included.

3(c) **1 out of 1 mark** was awarded because a modification with evidence (titration results were too small, they were less than $5 \cdot 0 \text{ cm}^3$) was carried out and so 1 mark was awarded.

A control experiment was carried out. Although the control was discussed in the evaluation, it was not used to give an explanation as to whether the egg shell values would have had lower or higher values based on these results. The results of the control experiment could also have been used to calculate the final % values. The control experiment on its own would not have been awarded this mark.

3(d) **1 out of 1 mark** was awarded because all three egg types were duplicated. The control experiment has not been duplicated but this was not required.

3(e) **0 out of 1 mark** was awarded because although masses have been measured to an appropriate level of accuracy for this procedure, the candidate has not detailed what the 1.00 g of calcium carbonate or eggshell was weighed into and there is no mention of how it was transferred. The procedure does not mention how the 25 cm³ HCl was measured and so this mark was not awarded. From this information it was not possible to award the accuracy mark as there is no evidence that the methods used are accurate.

3(f) **1 out of 1 mark** was awarded because raw data has been recorded for titrations with initial and final burette readings. Although the colour change for the titration has not been given in the procedures section, this was dealt with in section 3(a). Colour changes for the titrations have been given on page 14, so the mark for raw data was awarded.

3(g) **0 out of 1 mark** was awarded because numerical data is appropriately presented; however, the table on page 20 has an incorrect heading. % purity is not the correct term (see conclusion section); however, the reason the mark was not awarded was because the heading '% purity' is an incomplete heading.

3(h) **0 out of 1 mark** was awarded because the cited references given on pages 4, 5, 6 and 8 are listed correctly at the end of the report, but the citations have been given as full URLs and this is not a relevant referencing system.

4 Data analysis

The candidate was awarded **4 out of 5 marks**. The marks were awarded as follows:

4(a) This part of the data analysis section is marked holistically with a maximum of 4 marks available.

The candidate was awarded **3 out of 4 marks** because overall, they have demonstrated a **reasonably good** analysis of the experimental data.

Only one type of chemical calculation is used, however, this is a back titration and uses two different mole ratios and concentrations, and these were carried out on three separate samples (x2 for duplicates) as well as a control. There is also a graph plotted. This is acceptable as an Advanced Higher level of analysis.

The average titre volumes have been rounded to one decimal place, which is an acceptable number of significant figures, and the rounding has been carried out correctly.

There are a number of small errors in the data analysis.

- There are two small errors in the calculations of titre volumes:
 - Free range 1 titre, 1 should be 10.5 and if this was disregarded the average titre would be 11.3 not 11.4. This would give a final % of 96.8 rather than the 96.6% quoted, however, this was regarded as being a small error in the final calculated value.
 - Free range 2 titre, 2 should be 11.5 although average titre would stay 11.4.
- The calculation for the control experiment uses 0.05 moll⁻¹ NaOH but in the table of raw data on page 11 the concentration is quoted as being 0.1 moll⁻¹. The procedure on page 9 also quotes 0.05 moll⁻¹. The calculation that has been carried out is correct for the values used (see bullet below), however, the titre volumes obtained as well as the calculated answer all suggest that

the concentration was 0.1 moll⁻¹ NaOH that was used. This would give a final answer of 98.6% calcium carbonate.

- The number of moles CaCO₃ for control should be 0.0112 (not 0.01112) but the correct value was used in the calculation.
- A bar chart has been presented which is an appropriate graph for this data and it has the minimum number of bars, so is acceptable. The grid lines are appropriate and although the axis label is % purity – this is consistent with the table of data used to generate the graph given on page 20. There is an issue with the labelling of the bars – incorrect rounding applied – for example, duck egg should be 99.45% to four significant figures and not the 99.50% that has been labelled (the plotting is correct for the calculated values).

4(b) **1 out of 1 mark** was awarded because the final percentages have been rounded to three significant figures and the lowest data measured was two significant figures, and so the final values are within the accepted range.

5 Conclusion

The candidate was awarded **0 out of 1 mark** because although the conclusion relates to the aim, the % purity term is not valid. These values are % by mass of calcium carbonate and do not relate to purity.

6 Analysis

The candidate was awarded **0 out of 1 mark** because they have not made a comparison to literature values, and these are obtainable from the internet.

7 Evaluation

The candidate was awarded **3 out of 4 marks**. The marks were awarded as follows:

The candidate has carried out a partial quantitative evaluation of uncertainties. Values associated with pipette, burette, standard flask, and balance have been given and % uncertainties calculated; however, they have not been combined. The candidate has not calculated the absolute error in the final values. 1 mark is awarded for the quantitative evaluation.

The candidate has made two valid evaluative statements with justification and so 2 marks are awarded.

- Statement: 'This could have altered the volume of acid required to react with the base.' Justification: '...it was not possible to remove all membrane from the eggshell before it was reacted with acid.'
- Statement: 'This could mean some Calcium Carbonate went unreacted and therefore is not included in calculations, leading to inaccurate results.'

Justification: 'It is possible that not all of the eggshell dissolved in the acid in the first part of the procedure.'

The following evaluative statements have been made but an appropriate justification has not been given and so 0 marks are awarded.

• Statement: 'I kept the rough titre to hand so that a direct comparison could be made.'

Justification: 'To ensure a similar end point was reached on each titration ...'

This statement has not been justified in terms of the candidate's results. The candidate could have justified this by saying that the results obtained were concordant and the end point colour was the same for each titration.

Statement: 'My results were valid in that they were close to this (control) value and did not go over 100%.
Justification: 'The control experiment I carried out gave a % purity value of more than 100%'.

The candidate has not shown an understanding that if the control experiment gave greater than 100%, then the actual values obtained for the eggshells are likely to be higher than the actual % calcium carbonate present in the eggshells.

Statement: 'A possible reason for gaining a value of more than 100% ...' (in the control).
Justification: '...more than one base was present in the eggshell.'

The justification given is incorrect as is the statement – the eggshell values were not more than 100%. This applies to the control only. The candidate has not appreciated that the presence of another base in the eggshell would lead to a higher than actual % calcium carbonate content.

In addition, the following justification has been given but the evaluative statement is not valid.

 Statement: '...less than 1.00g was actually reacted with the acid leading to inaccurate results.' Justification: 'It is possible that the eggshell was not completely dry before being weighed.'

A valid statement would need to be linked to the results obtained and the candidate's results do not support this statement. If a smaller mass than 1.00g was used in the calculations, the final values would be lower.

8 Structure

The candidate was awarded **1 out of 1 mark** because a title, contents page and page numbers are given.

Overall

The candidate was awarded a total of 16 out of 25 marks.

Candidate 2

1 Abstract

The candidate was awarded **1 out of 1 mark** because an aim and findings are given, and these findings are consistent with those given in the conclusion section. There is a conclusion for the aim to compare the two methods.

2 Underlying chemistry

The candidate was awarded **2 out of 3 marks** because overall, the candidate has demonstrated a **reasonable** understanding of the underlying chemistry of the project.

Relevant underlying chemistry includes:

- The structural equation of the condensation reaction has been given. This could have been expanded to include some theory about the reaction mechanism, the role of acetic anhydride and why it is used and instead of ethanoic acid, the role of nucleophiles and an explanation of why it is a condensation reaction.
- The structure of aspirin and the functional groups present in aspirin and salicylic acid are shown.
- A reasonable understanding is shown with the link between intermolecular forces and polarity and the distance a component travels on a thin layer chromatography (TLC) plate. There is a little confusion over what the mobile phase is in both the underlying chemistry and in the procedures. This could have been expanded further to relate to the actual project.
- The theory of melting point analysis has been described well with the link to intermolecular forces and the alteration in melting point temperature and the range included.
- The theory of infrared (IR) spectroscopy is given in reasonable detail and with a link to one functional group in aspirin.

The candidate included additional information that was not relevant. This irrelevant information was not considered when awarding the marks for this section:

- Structure and bonding in benzene as this part of the molecule is not involved in the synthesis, then this information is not relevant.
- Information about how ethyl acetate and cyclohexane are synthesised.
- Role of aspirin in reducing heart attacks.

3 Data collection and handling

The candidate was awarded **6 out of 9 marks**. The marks were awarded as follows:

3(a) **1 out of 2 marks** was awarded because the candidate has not clearly described **all procedures** in the past tense, using the impersonal passive voice.

The solvent used for the TLC is not stated within the procedures.

The incorrect tense has been used on a number of occasions:

- 'when the sample changes from a solid to liquid', page 11
- 'then a background is then taken', page 11
- 'which is used as a mobile phase', page 12

The procedure for the synthesis is clearly described in sufficient detail to be repeated and so 1 mark was awarded.

3(b) **0 out of 1 mark** was awarded because although the candidate has stated that a fume cupboard was used, no justification has been given for this in terms of chemicals requiring additional safety measures and no other safety measures with justifications have been given.

3(c) **1 out of 1 mark** was awarded because the candidate has carried out more than one procedure. The synthesis of aspirin melting point analysis, IR and TLC have all been described and the data recorded.

3(d) **1 out of 1 mark** was awarded because the aspirin synthesis by both methods has been duplicated.

Duplicates have not been carried out for the melting point and TLC analysis of each synthesised product but as the entire procedure was duplicated this was not necessary.

There is no requirement to duplicate IR analysis.

3(e) **1 out of 1 mark** was awarded because the apparatus and method used is appropriate to achieve the required level of accuracy. The candidate has used a two decimal place balance, and this is of sufficient accuracy for their procedure. They have used measuring cylinders for measuring acetic anhydride and as this reactant is in excess, this is of sufficient accuracy for their procedure.

3(f) **0 out of 1 mark** was awarded because although the raw data has been recorded for synthesis, melting point, IR and TLC analysis for both samples from method 1, IR spectra have not been included for the two method 2 samples and so this mark cannot be awarded.

3(g) **1 out of 1 mark** was awarded because the numerical data has been presented appropriately with the correct number of decimal places for the equipment being used. Appropriate units are used throughout. Tables have appropriate headings and units.

3(h) **1 out of 1 mark** was awarded because three citations are given in the body of the report and listed again at the end of the report.

4 Data analysis

The candidate was awarded **4 out of 5 marks**. The marks were awarded as follows:

4(a) This part of the data analysis section is marked holistically with a maximum of **4 marks** available.

3 out of 4 marks were awarded because overall, the candidate has demonstrated a **reasonably good** analysis of the experimental data.

The candidate has carried out % yield calculations, R_f calculations and interpreted IR spectra, and so the analysis is of an appropriate level of demand.

There is a slight error in the theoretical yield (this should be 6.52g not 6.51g - this is not a rounding error), while this is very slight (the final value should be 66.7% not 66.8%), this impacts on all four final % yield values, however this was regarded as being a small error in the final calculated value.

R_f values have been calculated for all of the samples.

IR spectra have been interpreted by assigning the major peaks on one of the spectra. A table of peaks and bonds identified has been given. The size of the spectra makes checking the wavenumbers difficult, and so full-size spectra should have been given. The spectra label %T is not appropriate, this should be % Transmission.

The candidate has also compared spectra of their samples with a library spectrum of an aspirin sample from Aldrich and has made comments on the similarity of the peaks. The candidate has confused the % match value on the spectra as a determination of the sample purity. For example, method 1 sample 1 has a 'purity' match of 89.41%. This in fact is only a statement that the two spectra match by 89.41% in terms of peak pattern. As the likely impurities would be unreacted salicylic acid, a large number of peaks would be common to both and would not show as a non-match when the two spectra are compared.

4(b) **1 out of 1 mark** was awarded because final % yield values are given to three significant figures, and this is correct from the raw data recorded. The R_f values are also given to the correct number of significant figures.

5 Conclusion

The candidate was awarded **1 out of 1 mark** because the stated aim was to synthesise aspirin by two methods and to compare the percentage yields and melting points of the two methods. The list of percentage yields and melting points of the two different methods is sufficient (in this case) to conclude the aim to compare the percentage yields and melting points.

6 Analysis

The candidate was awarded **1 out of 1 mark** because a comparison has been made between the synthesised aspirin IR spectra with Aldrich aspirin and the melting point data has been compared to the literature melting point value.

7 Evaluation

The candidate was awarded **3 out of 4 marks**. The marks were awarded as follows:

The following are valid evaluative statements with justification.

• Statement: '... it is anticipated that the 40.1% yield may not be accurate as may contain many impurities.'

Justification: '... it was found that the melting point was significantly lower than expected \ldots '

• Statement: '... the mixture was being heated for too long or at a too powerful setting.'

Justification: 'As a result of this when the mixture was added to a conical flask containing water, stage 8, an oil formed.'

• Statement: 'This may of lead to a decrease in the percentage yield as it will decrease the total final mass of product.'

Justification: 'The reaction may not have gone to completion and so not all of the salicylic acid reacted to become aspirin.' And

"... not completely all of the product was transferred from the water pump filter to the conical flask or watch glass ..."

In addition, the following justification has been given but the evaluative statement is not valid.

 Statement: 'The yield of the final product could have been increased by scratching the sides of the conical flask to encourage crystallisation.' Justification: 'This would have allowed the crystals to form quicker and therefore minimise the time that they were left in the lab where possible contamination may have occurred.'

This statement is not valid as scratching the side would not increase the yield of the final product obtained.

Statement: '... method 2 produced a purer aspirin product ...'

Justification: The results for method 2 duplicate sample had the highest infrared purity match of 91.34% ...'

This statement is not valid as the melting point data does not support this.

• Statement: '... signifying that the samples produced all have a strong match to aspirin.'

Justification: 'These peaks found in the synthesised aspirin are also found in the pure aspirin ...'

The peaks identified include peaks that would also be present in salicylic acid and so this statement is not valid.

8 Structure

The candidate was awarded **1 out of 1 mark** because a title, contents page and page numbers are given.

Overall

The candidate was awarded a total of 19 out of 25 marks.