Examiners' Report/
Principal Examiner Feedback

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IAL Chemistry WCH03/01

Unit 3: Chemistry Laboratory Skills I

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Introduction

There were straightforward questions in this paper that all candidates could access, but there were also challenges for the most able students. They were given an opportunity to show the extent of their knowledge of the unit and experience of practical work. Many candidates did well on the titration calculation in question 3, but errors were seen more frequently in the calculation on percentage yield in question 4. Several questions required explanations of the theory behind experiments, and laboratory experience helps here. A significant proportion of candidates found it hard to apply their knowledge of organic reactions to the unfamiliar compounds in question 2.

Question 1

There were many good answers to this question. Most candidates successfully identified the two ions in the solid X, but in (d) there were fewer correct answers about what happened when the white precipitate of silver chloride was left to stand in sunlight. A wide variety of colours and products was suggested here.
In (e), the question was about a reaction of X with dilute sulfuric acid, and this was in contrast to the reaction with concentrated sulfuric acid in (f). Many candidates correctly predicted that a white precipitate would form when dilute sulfuric acid was added to the barium compound. In a question asking for an observation, the answer should include the colour, as well as the formation of the precipitate. However, when writing the equation for this reaction, the formula HCl was often labelled as a gas. Hydrogen chloride gas was the product in the reaction with concentrated sulfuric acid in (f), and here a chemical test was required to confirm its identity. Use of indicator paper just shows that the gas is acidic, and a test which is more specific was required. Testing with ammonia produces a white smoke; some candidates implied that the hydrogen chloride could be bubbled into ammonia solution, but this was not allowed as the test involves the formation of a white solid when the fumes of the two compounds react.

Question 2

Most candidates knew the colour change in (a) which occurs when an alcohol reacts with potassium dichromate(VI) and sulfuric acid, and many realised that only the primary alcohol, hex-5-en-1-ol would form a carboxylic acid. The question asked for a structural formula, but skeletal formulae were accepted since the point of the question was to show the identity of the acid correctly. The main error which occurred was the omission of the carbon-carbon double bond, both in skeletal formulae and in structural formulae. Details of the reaction of sodium with an alcohol have often been tested in previous papers, and marks are given for observations, so the answer to (b) should state what is seen eg bubbles, rather than simply identifying the gas. The sodium dissolves in this reaction, but many answers referred to a white solid dissolving; this was presumably the sodium salt, and was not allowed.

The colour changes in (c) and (d) which occur when hex-5-en-1-ol reacts with aqueous bromine and with acidified potassium manganate(VII) were well known, but candidates had difficulty in completing the skeletal formulae of the products correctly. They had to show the atoms or groups that added across the double bond in hex-5-en-1-ol. In the correct formula, the bonds from the carbon atoms to the added groups had to be shown. Simply writing “Br” at the left hand end of the outline on the paper implies a molecule with only five carbon atoms, unless the bond to the Br is drawn. Very few candidates knew that two OH groups add to the double bond in the reaction with potassium manganate(VII).

In (e) candidates had to look for evidence for the presence of a carbon-carbon double bond in the spectrum to identify the compound. Both compounds in the question are alcohols so both would have an absorption due to O-H stretch, and both contain C-H single bonds. The spectrum shown had an absorption corresponding to C=C stretch in alkenes at 1669-1600 cm^{-1} and C-H stretch in alkenes at 3100-3010 cm^{-1}, showing it was hex-5-en-1-ol. When a question asks for use of data, the numbers used to deduce the answer should be shown.

Question 3

Many candidates were not aware that Universal Indicator is not used in titrations as it does not give a sharp colour change. There were many misconceptions and vague answers. Candidates referred to the many colours it produced, or said that it was not suitable with a strong acid. Most candidates could suggest a suitable indicator, and marking was generous for the spelling of phenolphthalein as long as it was recognisable, though for this indicator the colours given were often the wrong way round.
The first two parts of the calculation in (b) were well done. Some candidates did not realise that, in order to find how many moles of acid reacted with the tablet, they had to subtract the number of moles of acid reacting with the sodium hydroxide from the number of moles at the start. In the final part of the calculation many did not use the 1:2 reacting ratio of magnesium hydroxide and hydrochloric acid. Most answers were to three significant figures as required.

Experiments should be repeated to make the results more reliable. In (c), if a single tablet is investigated, the acid-tablet mixture has to be divided. Then the separate portions can each be titrated and the results compared.

When a burette is used to deliver a volume of solution, a reading must be taken at the start and at the finish. This means the error in each reading is doubled when the percentage error in a titre is calculated. The factor of two was missing in many answers to (c)(ii).

Experiments to determine an enthalpy change should be designed so that the temperature change occurs quickly, and can be measured before cooling occurs. For the reaction of magnesium hydroxide with hydrochloric acid in (d), this could involve using finely powdered solid, using acid which is not very dilute or ensuring good mixing of reagents. Another possibility is to draw a cooling curve and use it to find the temperature change when corrected for cooling. Some candidates who chose this last possibility gave answers which were too brief to gain marks. For example “draw a graph and extrapolate” does not give sufficient indication of how to find the maximum temperature change.

Question 4

In (a)(i) candidates were advised to use the molecular formulae of cyclohexanol and cyclohexene; the formulae of these molecules differ by two hydrogen and one oxygen atom. Hence the expected answer was dehydration. Both oxidation and reduction were frequently suggested.

Concentrated phosphoric acid is very corrosive, and in (ii) the precaution taken when using it had to be appropriate to its damaging effect on skin. Candidates who said it was inflammable and then suggested wearing gloves were not allowed the mark. Some candidates suggested washing hands if in contact with acid, but this is remedial action and not a precaution.

The apparatus in the diagram in (b) was completely sealed, so an opening was required at some point on the right hand side of the condenser. The boiling temperature of the distillate could not be measured efficiently unless the bulb of the thermometer was opposite the opening of the condenser. Candidates need to answer precisely. Just saying that the thermometer needs to be higher is not enough. Many candidates marked the suggested modifications on a diagram which made their ideas clear.

A large proportion of candidates scored one mark in (c)(i) as they knew that cyclohexene could not form hydrogen bonds with water. However there was confusion about the type of intermolecular bonding in cyclohexene. Answers such as "non-polar substances do not dissolve in polar solvents" do not actually explain the lack of solubility, though if cyclohexene and water were correctly identified as respectively non-polar and polar, one mark was allowed. The diagrams given for the apparatus in (c)(ii) were nearly always of separating funnels, with the cyclohexene layer correctly labelled.

Most candidates could suggest a suitable drying agent in (d), with calcium chloride the most popular choice. However, very few answers stated that the liquid would then look clear. The word "clear" is not allowed for "colourless" when describing a solution, but is correct here as the liquid becomes less cloudy. The use of distillation for the final purification was usually given correctly.

The first part of the calculation in (f) was often carried out correctly, but a number of different errors occurred regularly in the second part. Some candidates incorrectly assumed that since one mole of cyclohexanol can produce one mole of cyclohexene, then $1 cm^3$ of cyclohexanol can produce $1 cm^3$ of cyclohexene. Some calculated the mass of 0.100 mol of cyclohexanol to be 10g, but then assumed this was the maximum yield of cyclohexene. Others deduced that the maximum yield was 0.100 mol of cyclohexene but calculated the molecular mass as 84, not 82.

Advice to candidates

Always read the question very carefully. It often contains hints.

When asked for a chemical test for a gas, try to give a test which is as specific as possible. Litmus shows whether a gas is acidic or alkaline, but using ammonia is a more specific test for a hydrogen halide.

If you are asked to use data to deduce something, your answer should show the data that you used.

Practice using skeletal formulae and check that they show the correct number of carbon atoms.

Learn the organic reactions on the specification and practice applying them to unfamiliar compounds.

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