

CANDIDATE
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CENTRE
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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

May/June 2017

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 10 and 11.
A copy of the Periodic Table is printed on page 12.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

| | |
|-------------------|--|
| Session | |
| | |
| Laboratory | |
| | |

| | |
|---------------------------|--|
| For Examiner's Use | |
| 1 | |
| 2 | |
| 3 | |
| Total | |

This document consists of **12** printed pages.

- 1 In this experiment you will determine the relative formula mass of a copper salt by titration.

A solution of the copper salt reacts with excess acidified potassium iodide, producing iodine. This iodine is then titrated with aqueous sodium thiosulfate, using starch indicator.

FA 1 is an aqueous solution of the copper salt prepared by dissolving 26.0g of the salt to make 1.00 dm³ of solution.

FA 2 is dilute sulfuric acid, H₂SO₄.

FA 3 is aqueous potassium iodide, KI.

FA 4 is 0.110 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃.
starch indicator

(a) Method

- Fill the burette with **FA 4**.
- Pipette 25.0 cm³ of **FA 1** into a conical flask.
- Use the measuring cylinder to add approximately 10 cm³ of **FA 2** to the same conical flask.
- Use the measuring cylinder to add approximately 20 cm³ of **FA 3** to the mixture in the conical flask. The mixture will now be a brown colour, due to iodine produced in the reaction.
- Begin your rough titration by adding **FA 4** from the burette until the mixture becomes light brown.
- Add 10 drops of starch indicator. The mixture will become darker.
- Continue titrating until the mixture becomes an off-white colour. This is the end-point.
- Add **one** drop of starch indicator to check that no traces of dark colour are produced. If the mixture stays off-white, the titration is finished. If some dark colour is produced, because iodine is still present, continue the titration.
- Record your burette readings and the rough titre in the space below.

The rough titre is cm³.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make sure any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 4** added in each accurate titration.

| | |
|-----|--|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |
| VII | |

Keep FA 3 and starch indicator for use in Question 3.

[7]

- (b)** From your accurate titration results, obtain a suitable value for the volume of **FA 4** to be used in your calculations.
Show clearly how you obtained this value.

The iodine produced required cm³ of **FA 4**. [1]

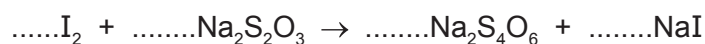
(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, in the volume of **FA 4** calculated in (b).

moles of $\text{Na}_2\text{S}_2\text{O}_3$ = mol

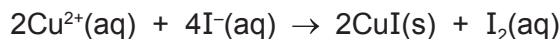
- (ii) Balance the equation for the reaction of iodine with sodium thiosulfate. State symbols are not required.



- (iii) Using your answer to (ii), calculate the number of moles of iodine that reacted with the number of moles of $\text{Na}_2\text{S}_2\text{O}_3$ calculated in (i).

moles of I_2 = mol

- (iv) Iodine, I_2 , is produced in the reaction between **FA 1** and **FA 3**. **FA 3** is in excess.



Using your answer to (iii), calculate the number of moles of copper(II) ions in 25.0 cm^3 of **FA 1**.

moles of Cu^{2+} ions = mol

- (v) Using your answer to (iv) and the information on page 2, calculate the relative formula mass of the copper compound in **FA 1**.

M_r of copper compound =
[4]

[Total: 12]

- 2 Malachite is a basic form of copper carbonate in which copper hydroxide is also present. The accepted chemical formula of malachite is $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$.

When malachite is heated, it decomposes as shown.



In this experiment, you will heat malachite to decompose it and use your results to obtain evidence about the accepted formula of malachite.

FA 5 is malachite, $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$.

(a) Method

Read through the method before starting any practical work.

In the space below prepare a **single** table for your results of **Experiments 1** and **2**.

Experiment 1

- Weigh a crucible with its lid and record the mass.
- Add between 2.5g and 3.0g of **FA 5** to the crucible. Weigh the crucible with **FA 5** and lid and record the mass.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible and contents gently for about two minutes, with the lid on.
- Remove the lid and continue heating gently for about three minutes.
- Replace the lid and leave the crucible and residue to cool for at least five minutes. Then reweigh the crucible and contents with the lid on. Record the mass.
- **While the crucible is cooling, you may wish to begin work on Question 3.**
- Calculate and record the mass of **FA 5** used and the mass of residue obtained.
- State the observation(s) you made while the reaction was taking place.

.....

Experiment 2

Repeat the method used in **Experiment 1**, using between 1.5g and 2.0g of **FA 5** in the second crucible.

Results

| | |
|-----|--|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |

[6]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use your results from **Experiment 1** to calculate the number of moles of copper oxide, CuO, obtained as residue.
Use the Periodic Table on page 12 for any data you may require.

moles of CuO obtained in **Experiment 1** = mol

- (ii) Use your answer to (i), the equation on page 4 and the mass of **FA 5** you used in **Experiment 1**, to calculate the relative formula mass, M_r , of malachite.

M_r of malachite (from **Experiment 1**) =

- (iii) Use your results from **Experiment 2** to calculate another value for the relative formula mass, M_r , of malachite.

M_r of malachite (from **Experiment 2**) =

- (iv) Use data from the Periodic Table to calculate the relative formula mass, M_r , of malachite from its accepted formula, $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$.

M_r of malachite (from formula) =

- (v) If the relative formula mass of malachite obtained from **either** of your experiments is within 2.5% of the answer in (iv), this is good evidence that the accepted formula, $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$, is correct.

Show by calculation whether either of your experiments supports the accepted formula.

[5]

(c) (i) State **one** way of improving the accuracy of the experimental method, using the same masses of **FA 5**.

Explain the benefit of your improvement.

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(ii) Explain why you would expect **Experiment 1** to be more accurate than **Experiment 2**.

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[3]

[Total: 14]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where reagents are selected for use in a test, the **name** or **correct formula** of the element or compound must be given.

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

(a) **FA 6** is another salt of copper. The anion present is one of those listed in the Qualitative Analysis Notes.

- (i) Transfer a **small** spatula measure of **FA 6** into a hard-glass test-tube. Heat gently at first, then heat strongly, until no further change occurs.

Record **all** your observations below.

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- (ii) Suggest the chemical formula of **FA 6**.

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[3]

- (b) (i) Dissolve the remainder of **FA 6** in an approximately 10 cm depth of distilled water in a boiling tube.

FA 7 is a solution of a salt containing one anion from those listed in the Qualitative Analysis Notes.

Two cations are also present.

Carry out the tests described below using separate portions of solutions **FA 6** and **FA 7**. Record your observations in the table.

| <i>test</i> | <i>observations</i> | |
|---|---------------------|-------------|
| | FA 6 | FA 7 |
| To a 1 cm depth of solution in a test-tube, add an equal volume of FA 3 , aqueous potassium iodide, followed by a few drops of starch indicator. | X | |
| To a 1 cm depth of solution in a boiling tube, add aqueous sodium hydroxide, then | | |
| heat gently and carefully . | | X |
| To a 1 cm depth of solution in a test-tube, add a few drops of aqueous silver nitrate. | | |
| To a 1 cm depth of solution in a test-tube, add aqueous ammonia. | | |
| To a 1 cm depth of solution in a test-tube, add a folded 3 cm length of magnesium ribbon. | | |

- (ii) What can you deduce about solution **FA 7** from its reaction with magnesium? Explain your answer.

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- (iii) Give the ionic equation for the reaction of the metal cation in **FA 7** with aqueous sodium hydroxide. Include state symbols.

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- (iv) What **type** of reaction took place when aqueous potassium iodide was added to **FA 7**? Use your observations to help you explain your answer.

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- (v) The observation you made when aqueous silver nitrate was added to **FA 7** does not allow the anion in **FA 7** to be identified with certainty.

Explain why you cannot be certain about the identity of the anion.

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- (vi) A student suggested that the anion in **FA 7** could be identified with more certainty if excess ammonia solution was added after the aqueous silver nitrate.

Explain why this suggestion is **not** correct.

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[11]

[Total: 14]

Qualitative Analysis Notes

1 Reactions of aqueous cations

| <i>ion</i> | <i>reaction with</i> | |
|--|--|--|
| | NaOH(aq) | NH ₃ (aq) |
| aluminium, Al ³⁺ (aq) | white ppt. soluble in excess | white ppt. insoluble in excess |
| ammonium, NH ₄ ⁺ (aq) | no ppt. ammonia produced on heating | – |
| barium, Ba ²⁺ (aq) | faint white ppt. is nearly always observed unless reagents are pure | no ppt. |
| calcium, Ca ²⁺ (aq) | white ppt. with high [Ca ²⁺ (aq)] | no ppt. |
| chromium(III), Cr ³⁺ (aq) | grey-green ppt. soluble in excess | grey-green ppt. insoluble in excess |
| copper(II), Cu ²⁺ (aq) | pale blue ppt. insoluble in excess | blue ppt. soluble in excess giving dark blue solution |
| iron(II), Fe ²⁺ (aq) | green ppt. turning brown on contact with air insoluble in excess | green ppt. turning brown on contact with air insoluble in excess |
| iron(III), Fe ³⁺ (aq) | red-brown ppt. insoluble in excess | red-brown ppt. insoluble in excess |
| magnesium, Mg ²⁺ (aq) | white ppt. insoluble in excess | white ppt. insoluble in excess |
| manganese(II), Mn ²⁺ (aq) | off-white ppt. rapidly turning brown on contact with air insoluble in excess | off-white ppt. rapidly turning brown on contact with air insoluble in excess |
| zinc, Zn ²⁺ (aq) | white ppt. soluble in excess | white ppt. soluble in excess |

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|---|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$) |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$) |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$) |
| nitrate, $\text{NO}_3^-(\text{aq})$ | NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil |
| nitrite, $\text{NO}_2^-(\text{aq})$ | NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air) |
| sulfate, $\text{SO}_4^{2-}(\text{aq})$ | gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\text{SO}_3^{2-}(\text{aq})$ | gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids) |

3 Tests for gases

| <i>gas</i> | <i>test and test result</i> |
|-------------------------------|---|
| ammonia, NH_3 | turns damp red litmus paper blue |
| carbon dioxide, CO_2 | gives a white ppt. with limewater (ppt. dissolves with excess CO_2) |
| chlorine, Cl_2 | bleaches damp litmus paper |
| hydrogen, H_2 | 'pops' with a lighted splint |
| oxygen, O_2 | relights a glowing splint |

The Periodic Table of Elements

| Group | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|-------------------------|------------------------|--------------------------|-------------------------|--------------------------|-------------------------|--------------------------|------------------------|--------------------------|------------------------|------------------------|-------------------------|-------------------------|-------------------------|--------------------------|------------------------|-----------------------|--|--|--|--|--|--|--|--|--|---|
| 1 | 2 | | | | | | | | | | | 13 | 14 | 15 | 16 | 17 | 18 | | | | | | | | | | |
| <div style="border: 1px solid black; padding: 5px; display: inline-block;"> Key atomic number atomic symbol name relative atomic mass </div> | | | | | | | | | | | 1 | | | | | | | | | | | | | | | | 2 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 3 | 4 | | | | | | | | | | | 5 | 6 | 7 | 8 | 9 | 10 | | | | | | | | | | |
| Li lithium 6.9 | Be beryllium 9.0 | | | | | | | | | | | B boron 10.8 | C carbon 12.0 | N nitrogen 14.0 | O oxygen 16.0 | F fluorine 19.0 | Ne neon 20.2 | | | | | | | | | | |
| 11 | 12 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | | | | | | | | | | |
| Na sodium 23.0 | Mg magnesium 24.3 | | | | | | | | | | | Al aluminium 27.0 | Si silicon 28.1 | P phosphorus 31.0 | S sulfur 32.1 | Cl chlorine 35.5 | Ar argon 39.9 | | | | | | | | | | |
| 19 | 20 | 21 | 22 | 23 | 24 | 25 | 26 | 27 | 28 | 29 | 30 | 31 | 32 | 33 | 34 | 35 | 36 | | | | | | | | | | |
| K potassium 39.1 | Ca calcium 40.1 | Sc scandium 45.0 | Ti titanium 47.9 | V vanadium 50.9 | Cr chromium 52.0 | Mn manganese 54.9 | Fe iron 55.8 | Co cobalt 58.9 | Ni nickel 58.7 | Cu copper 63.5 | Zn zinc 65.4 | Ga gallium 69.7 | Ge germanium 72.6 | As arsenic 74.9 | Se selenium 79.0 | Br bromine 79.9 | Kr krypton 83.8 | | | | | | | | | | |
| 37 | 38 | 39 | 40 | 41 | 42 | 43 | 44 | 45 | 46 | 47 | 48 | 49 | 50 | 51 | 52 | 53 | 54 | | | | | | | | | | |
| Rb rubidium 85.5 | Sr strontium 87.6 | Y yttrium 88.9 | Zr zirconium 91.2 | Nb niobium 92.9 | Mo molybdenum 95.9 | Tc technetium – | Ru ruthenium 101.1 | Rh rhodium 102.9 | Pd palladium 106.4 | Ag silver 107.9 | Cd cadmium 112.4 | In indium 114.8 | Sn tin 118.7 | Sb antimony 121.8 | Te tellurium 127.6 | I iodine 126.9 | Xe xenon 131.3 | | | | | | | | | | |
| 55 | 56 | 57–71 | 72 | 73 | 74 | 75 | 76 | 77 | 78 | 79 | 80 | 81 | 82 | 83 | 84 | 85 | 86 | | | | | | | | | | |
| Cs caesium 132.9 | Ba barium 137.3 | lanthanoids | Hf hafnium 178.5 | Ta tantalum 180.9 | W tungsten 183.8 | Re rhenium 186.2 | Os osmium 190.2 | Ir iridium 192.2 | Pt platinum 195.1 | Au gold 197.0 | Hg mercury 200.6 | Tl thallium 204.4 | Pb lead 207.2 | Bi bismuth 209.0 | Po polonium – | At astatine – | Rn radon – | | | | | | | | | | |
| 87 | 88 | 89–103 | 104 | 105 | 106 | 107 | 108 | 109 | 110 | 111 | 112 | | 114 | | 116 | | | | | | | | | | | | |
| Fr francium – | Ra radium – | actinoids | Rf rutherfordium – | Db dubnium – | Sg seaborgium – | Bh bohrium – | Hs hassium – | Mt meitnerium – | Ds darmstadtium – | Rg roentgenium – | Cn copernicium – | | Fl flerovium – | | Lv livermorium – | | | | | | | | | | | | |

| | | | | | | | | | | | | | | | |
|-------------|--------------------------------|------------------------------|-----------------------------------|--------------------------------|-----------------------------|-------------------------------|-------------------------------|---------------------------------|------------------------------|---------------------------------|------------------------------|-----------------------------|-------------------------------|--------------------------------|-------------------------------|
| lanthanoids | 57 La lanthanum 138.9 | 58 Ce cerium 140.1 | 59 Pr praseodymium 140.9 | 60 Nd neodymium 144.4 | 61 Pm promethium – | 62 Sm samarium 150.4 | 63 Eu europium 152.0 | 64 Gd gadolinium 157.3 | 65 Tb terbium 158.9 | 66 Dy dysprosium 162.5 | 67 Ho holmium 164.9 | 68 Er erbium 167.3 | 69 Tm thulium 168.9 | 70 Yb ytterbium 173.1 | 71 Lu lutetium 175.0 |
| actinoids | 89 Ac actinium – | 90 Th thorium 232.0 | 91 Pa protactinium 231.0 | 92 U uranium 238.0 | 93 Np neptunium – | 94 Pu plutonium – | 95 Am americium – | 96 Cm curium – | 97 Bk berkelium – | 98 Cf californium – | 99 Es einsteinium – | 100 Fm fermium – | 101 Md mendelevium – | 102 No nobelium – | 103 Lr lawrencium – |

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