## Cambridge International AS \& A Level

## CANDIDATE NAME

CENTRE $\square$

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## CHEMISTRY

9701/33
Paper 3 Advanced Practical Skills 1
October/November 2022
2 hours
You must answer on the question paper.
You will need: The materials and apparatus listed in the confidential instructions

## INSTRUCTIONS

- Answer all questions.
- Use a black or dark blue pen. You may use an HB pencil for any diagrams or graphs.
- Write your name, centre number and candidate number in the boxes at the top of the page
- Write your answer to each question in the space provided.
- Do not use an erasable pen or correction fluid.
- Do not write on any bar codes.
- You may use a calculator.
- You should show all your working and use appropriate units.


## INFORMATION

- The total mark for this paper is 40
- The number of marks for each question or part question is shown in brackets [ ]
- The Periodic Table is printed in the question paper.
- Important values, constants and standards are printed in the question paper.
- Notes for use in qualitative analysis are provided in the question paper.


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

This document has 16 pages. Any blank pages are indicated.

## Quantitative analysis

Read through the whole method before starting any practical work. Where appropriate, prepare a table for your results in the space provided.

Show the precision of the apparatus you used in the data you record.
Show your working and appropriate significant figures in the final answer to each step of your calculations.

1 An iron compound can contain $\mathrm{Fe}^{2+}$ ions, $\mathrm{Fe}^{3+}$ ions or both ions.
In this experiment, you will determine the percentage by mass of iron in FA 1, an unknown compound. You will first prepare a solution of the compound and then carry out a titration using acidified potassium manganate(VII), $\mathrm{KMnO}_{4}$.

$$
5 \mathrm{Fe}^{2+}(\mathrm{aq})+\mathrm{MnO}_{4}^{-}(\mathrm{aq})+8 \mathrm{H}^{+}(\mathrm{aq}) \rightarrow 5 \mathrm{Fe}^{3+}(\mathrm{aq})+\mathrm{Mn}^{2+}(\mathrm{aq})+4 \mathrm{H}_{2} \mathrm{O}(\mathrm{I})
$$

The end-point of the titration is when all the $\mathrm{Fe}^{2+}$ ions have been oxidised and so unreacted potassium manganate(VII) causes the colour of the solution to become a permanent pale pink.

FA 1 is a sample of the unknown compound.
FA 2 is $0.0100 \mathrm{~mol} \mathrm{dm}^{-3}$ potassium manganate(VII), $\mathrm{KMnO}_{4}$.
FA 3 is dilute sulfuric acid, $\mathrm{H}_{2} \mathrm{SO}_{4}$.
(a) Method

## Weighing the compound

- Weigh the sample of FA 1 and its container.
- Tip the FA 1 into the $250 \mathrm{~cm}^{3}$ beaker.
- Reweigh the container including any residual FA 1.
- Record both your readings clearly in the space below.
- Calculate the mass of FA 1 transferred into the beaker. Record the mass.


## Preparing the solution

- Add approximately $200 \mathrm{~cm}^{3}$ of distilled water to the beaker and stir until the FA 1 has dissolved.
- Pour the contents carefully into the $250 \mathrm{~cm}^{3}$ volumetric flask.
- Rinse the contents of the beaker with a little distilled water and add these washings to the flask.
- Fill the flask to the line with distilled water and shake thoroughly.
- Label this solution FA 4.


## Titration

- Fill the burette with FA 2.
- Pipette $25.0 \mathrm{~cm}^{3}$ of FA 4 into a conical flask.
- Use the $25 \mathrm{~cm}^{3}$ measuring cylinder to add $15 \mathrm{~cm}^{3}$ of FA 3 to the conical flask.
- Add FA 2 from the burette until the solution in the flask turns a permanent pink.
- Carry out a rough titration and record your burette readings in the space below.
rough titre $=$ $\qquad$ $\mathrm{cm}^{3}$
- Carry out as many accurate titrations as you think are necessary to obtain consistent results.
- Make sure that your recorded results show the precision of your practical work.
- Record in a suitable format, in the space below, all your burette readings and the volume of FA 2 added in each accurate titration.

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

(b) From your accurate titration results, calculate a suitable mean value to use in your calculations. Show clearly how you obtained this value.
(c) Calculations
(i) Give your answers to (c)(ii), (c)(iii) and (c)(iv) to the appropriate number of significant figures.
(ii) Calculate the amount, in mol, of manganate(VII) ions in the volume recorded in (b).

$$
\text { amount of } \mathrm{MnO}_{4}^{-}=
$$

$\qquad$ mol [1]
(iii) Calculate the amount, in mol, of iron(II) ions in the weighed sample of FA 1.

$$
\text { amount of } \mathrm{Fe}^{2+}=
$$

$\qquad$ mol [1]
(iv) Calculate the percentage by mass of iron in FA 1. Show your working.
(d) A student carries out the same experiment as in (a). The student receives a sample of FA 1 in a container with a lid. The student records the initial mass of the container with its lid and the sample of FA 1. Then the student records the mass of the container with the residue but forgets to replace the lid.

How would this error alter the student's answer to (c)(iv)?
Explain your answer.
$\qquad$
$\qquad$
(e) State two assumptions that have been made in calculating the percentage by mass of iron in FA 1 in (c)(iv).
$\qquad$
$\qquad$
$\qquad$

## Question 2 starts on the next page.

2 In this experiment you will determine the percentage by mass of magnesium in a hydrated salt, $\mathrm{MgX} \cdot 7 \mathrm{H}_{2} \mathrm{O}$, where X represents the anion.

You will measure the loss of mass when a sample of the hydrated salt is heated to form the anhydrous salt.

FA 5 is a pure sample of $\mathrm{MgX} \cdot 7 \mathrm{H}_{2} \mathrm{O}$
(a) Method

- Weigh the crucible with its lid. Record the mass.
- Add all of FA 5 to the crucible.
- Reweigh the crucible with its lid and FA 5. Record the mass.
- Support the crucible in the pipe-clay triangle on top of the tripod.
- Heat the crucible with the lid on gently for about 1 minute.
- Remove the lid and then heat strongly for a further 4 minutes.
- Replace the lid and allow the crucible to cool for at least 5 minutes.

While the crucible is cooling you may wish to begin work on Question 3.

- When the crucible is cool enough to handle, reweigh the crucible with its lid and its contents. Record the mass.
- Calculate and record the mass of FA 5 used.
- Calculate and record the mass of water lost.

| I |  |
| :---: | :--- |
| II |  |
| III |  |
| IV |  |
| V |  |

## (b) Calculations

(i) Calculate the amount, in mol, of water lost.
(ii) Calculate the percentage by mass of magnesium in FA 5 . Show your working.
percentage by mass of $\mathrm{Mg}=$ \% [2]
(c) Suggest two assumptions that must be made for this experiment to give an accurate value of the percentage of Mg .
$\qquad$
$\qquad$
$\qquad$
$\qquad$
[Total: 10]

## Qualitative analysis

For each test you should record all your observations in the spaces provided.
Examples of observations include:

- colour changes seen
- the formation of any precipitate and its solubility (where appropriate) in an excess of the reagent added
- the formation of any gas and its identification (where appropriate) by a suitable test.

You should record clearly at what stage in a test an observation is made.
Where no change is observed you should write 'no change'.
Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

If any solution is warmed, a boiling tube must be used.
Rinse and reuse test-tubes and boiling tubes where possible.
No additional tests should be attempted.

3 (a) FA 6 is an acidified aqueous solution of a salt which contains two cations and one anion, all of which are listed in the Qualitative analysis notes.
(i) Select a reagent or reagents for use in tests to identify the two cations. Record your observations.
(ii) The anion in FA 6 is either the sulfate ion, $\mathrm{SO}_{4}{ }^{2-}$, or the sulfite ion, $\mathrm{SO}_{3}{ }^{2-}$. Select a reagent or reagents for use in tests to identify the anion.
Record your observations.
(iii) Identify the ions present in FA 6 and give their formulae.

The cations present in FA 6 are $\qquad$ and $\qquad$ . .

The anion present in FA 6 is $\qquad$ .
(b) Carry out the tests and record your observations.

For each test use a 1 cm depth of FA 6 in a test-tube.

| test | observations |
| :---: | :---: |
| Test 1 <br> Add a 1 cm depth of aqueous sodium sulfite, then |  |
| add a 1 cm depth of dilute sulfuric acid. Rinse out this test-tube thoroughly. |  |
| Test 2 <br> Add a 1 cm depth of aqueous potassium iodide, then |  |
| add a few drops of starch solution. |  |

(c) FA 7, FA 8 and FA 9 are aqueous solutions of sodium salts. Each solution contains one of the following ions: $\mathrm{CO}_{3}{ }^{2-}, \mathrm{Cl}^{-}, \mathrm{Br}^{-}$.

You will identify which solution contains which ion.
(i) Carry out the tests and record your observations.

For each test use a 1 cm depth of FA 7, FA 8 or FA 9 in a test-tube.

| test | observations |  |  |
| :--- | :--- | :--- | :--- |
|  | FA 7 | FA 8 | FA 9 |
| Add a few drops <br> of aqueous <br> silver nitrate, then |  |  |  |
| add aqueous <br> ammonia. |  |  |  |

(ii) If your results are insufficient to identify which anion is present in each solution, carry out a further test. Record your test and observations.
(iii) Identify which solution contains which ion.

The $\mathrm{CO}_{3}{ }^{2-}$ ion is present in $\qquad$ . .

The $\mathrm{Cl}^{-}$ion is present in $\qquad$
The $\mathrm{Br}^{-}$ion is present in $\qquad$ .
[Total: 13]

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## Qualitative analysis notes

## 1 Reactions of cations

| cation | reaction with |  |
| :--- | :--- | :--- |
|  | $\mathrm{NaOH}(\mathrm{aq})$ | $\mathrm{NH}_{3}(\mathrm{aq})$ |
| aluminium, $\mathrm{Al}^{3+}(\mathrm{aq})$ | white ppt. soluble in excess | white ppt. insoluble in excess |
| ammonium, $\mathrm{NH}_{4}^{+}(\mathrm{aq})$ | no ppt. <br> ammonia produced on warming | - |
| barium, $\mathrm{Ba}^{2+}(\mathrm{aq})$ | faint white ppt. is observed unless <br> $\left[\mathrm{Ba}{ }^{2+}(\mathrm{aq})\right]$ is very low | no ppt. |
| calcium, $\mathrm{Ca}^{2+}(\mathrm{aq})$ | white ppt. unless $\left[\mathrm{Ca}{ }^{2+}(\mathrm{aq})\right]$ is very <br> low | no ppt. |
| chromium(III), $\mathrm{Cr}^{3+}(\mathrm{aq})$ | grey-green ppt. soluble in excess <br> giving dark green solution | grey-green ppt. insoluble in excess |
| copper(II), $\mathrm{Cu}^{2+}(\mathrm{aq})$ | pale blue ppt. insoluble in excess | pale blue ppt. soluble in excess <br> giving dark blue solution |
| iron(II), $\mathrm{Fe}^{2+}(\mathrm{aq})$ | green ppt. turning brown on <br> contact with air <br> insoluble in excess | green ppt. turning brown on <br> contact with air <br> insoluble in excess |
| iron(III), $\mathrm{Fe}^{3+}(\mathrm{aq})$ | red-brown ppt. insoluble in excess | red-brown ppt. insoluble in excess |
| magnesium, $\mathrm{Mg}^{2+}(\mathrm{aq})$ | white ppt. insoluble in excess | white ppt. insoluble in excess |
| manganese(II), $\mathrm{Mn}^{2+}(\mathrm{aq})$ | off-white ppt. rapidly turning brown <br> on contact with air <br> insoluble in excess | off-white ppt. rapidly turning brown <br> on contact with air <br> insoluble in excess |
| zinc, $\mathrm{Zn}^{2+}(\mathrm{aq})$ | white ppt. soluble in excess | white ppt. soluble in excess |

## 2 Reactions of anions

| anion | reaction |
| :---: | :---: |
| carbonate, $\mathrm{CO}_{3}{ }^{2-}$ | $\mathrm{CO}_{2}$ liberated by dilute acids |
| chloride, $\mathrm{Cl}^{-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (soluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ) |
| bromide, $\mathrm{Br}^{-}(\mathrm{aq})$ | gives cream/off-white ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (partially soluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ) |
| iodide, $\mathrm{I}^{-}(\mathrm{aq})$ | gives pale yellow ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ ( insoluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ) |
| nitrate, $\mathrm{NO}_{3}^{-}(\mathrm{aq})$ | $\mathrm{NH}_{3}$ liberated on heating with $\mathrm{OH}^{-}(\mathrm{aq})$ and Al foil |
| nitrite, $\mathrm{NO}_{2}{ }^{-}(\mathrm{aq})$ | $\mathrm{NH}_{3}$ liberated on heating with $\mathrm{OH}^{-}(\mathrm{aq})$ and Al foil; decolourises acidified aqueous $\mathrm{KMnO}_{4}$ |
| sulfate, $\mathrm{SO}_{4}{ }^{2-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ba}^{2+}(\mathrm{aq})$ (insoluble in excess dilute strong acids); gives white ppt. with high $\left[\mathrm{Ca}^{2+}(\mathrm{aq})\right]$ |
| sulfite, $\mathrm{SO}_{3}{ }^{2-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ba}^{2+}(\mathrm{aq})$ (soluble in excess dilute strong acids); decolourises acidified aqueous $\mathrm{KMnO}_{4}$ |
| thiosulfate, $\mathrm{S}_{2} \mathrm{O}_{3}{ }^{2-}(\mathrm{aq})$ | gives off-white/pale yellow ppt. slowly with $\mathrm{H}^{+}$ |

## 3 Tests for gases

| gas | test and test result |
| :--- | :--- |
| ammonia, $\mathrm{NH}_{3}$ | turns damp red litmus paper blue |
| carbon dioxide, $\mathrm{CO}_{2}$ | gives a white ppt. with limewater |
| hydrogen, $\mathrm{H}_{2}$ | 'pops' with a lighted splint |
| oxygen, $\mathrm{O}_{2}$ | relights a glowing splint |

## 4 Tests for elements

| element | test and test result |
| :--- | :--- |
| iodine, $\mathrm{I}_{2}$ | gives blue-black colour on addition of starch solution |

Important values, constants and standards

| molar gas constant | $R=8.31 \mathrm{JK}^{-1} \mathrm{~mol}^{-1}$ |
| :--- | :--- |
| Faraday constant | $F=9.65 \times 10^{4} \mathrm{C} \mathrm{mol}^{-1}$ |
| Avogadro constant | $L=6.022 \times 10^{23} \mathrm{~mol}^{-1}$ |
| electronic charge | $e=-1.60 \times 10^{-19} \mathrm{C}$ |
| molar volume of gas | $V_{\mathrm{m}}=22.4 \mathrm{dm}^{3} \mathrm{~mol}^{-1}$ at s.t.p. $(101 \mathrm{kPa}$ and 273 K$)$ <br> $V_{\mathrm{m}}=24.0 \mathrm{dm}^{3} \mathrm{~mol}^{-1}$ at room conditions |
| ionic product of water | $K_{\mathrm{w}}=1.00 \times 10^{-14} \mathrm{~mol}^{2} \mathrm{dm}^{-6}\left(\right.$ at $\left.298 \mathrm{~K}\left(25^{\circ} \mathrm{C}\right)\right)$ |
| specific heat capacity of water | $c=4.18 \mathrm{~kJ} \mathrm{~kg}^{-1} \mathrm{~K}^{-1}\left(4.18 \mathrm{Jg}^{-1} \mathrm{~K}^{-1}\right)$ |

The Periodic Table of Elements

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