## Cambridge International AS \& A Level

## CANDIDATE NAME

CENTRE


## NUMBER

$\square$ CANDIDATE NUMBER $\square$

## CHEMISTRY

9701/33
Paper 3 Advanced Practical Skills 1
February/March 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 12 pages.

## 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 You will determine the concentration of sulfuric acid by reaction with a known concentration of sodium hydroxide using a thermometric method. The equation for the reaction is shown.

$$
2 \mathrm{NaOH}(\mathrm{aq})+\mathrm{H}_{2} \mathrm{SO}_{4}(\mathrm{aq}) \rightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}(\mathrm{aq})+2 \mathrm{H}_{2} \mathrm{O}(\mathrm{l})
$$

FA 1 is $1.90 \mathrm{moldm}^{-3}$ sodium hydroxide, NaOH .
FA 2 is dilute sulfuric acid, $\mathrm{H}_{2} \mathrm{SO}_{4}$.
(a) Method

- Place the cup in the $250 \mathrm{~cm}^{3}$ beaker.
- Use the $25 \mathrm{~cm}^{3}$ measuring cylinder to transfer $25.0 \mathrm{~cm}^{3}$ of FA 1 into the cup.
- Place the thermometer into the solution in the cup and record its temperature in the table of results.
- Fill a burette with FA 2.
- Run $5.00 \mathrm{~cm}^{3}$ of FA 2 into the solution in the cup.
- Stir the mixture and record the highest temperature reached.
- Repeat adding $5.00 \mathrm{~cm}^{3}$ volumes of FA 2 into the solution in the cup until $45.00 \mathrm{~cm}^{3}$ has been added. Record the highest temperature reached after each addition.


## Results

| volume of FA 2 added $/ \mathrm{cm}^{3}$ | 0.00 | 5.00 | 10.00 | 15.00 | 20.00 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| temperature of solution $/{ }^{\circ} \mathrm{C}$ |  |  |  |  |  |


| volume of FA 2 added $/ \mathrm{cm}^{3}$ | 25.00 | 30.00 | 35.00 | 40.00 | 45.00 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| temperature of solution $/{ }^{\circ} \mathrm{C}$ |  |  |  |  |  |

(b) (i) Plot a graph of temperature ( $y$-axis) against volume of acid added ( $x$-axis) on the grid provided. Select a scale on the $y$-axis to include a temperature $4.0^{\circ} \mathrm{C}$ above the highest temperature you recorded.
Label any points you consider to be anomalous.
Draw two lines of best fit, one for the rise in temperature and one for after the maximum temperature has been reached.
Extrapolate the two lines so they intersect.

(ii) Use your graph to determine the volume of sulfuric acid, FA 2, required to neutralise $25.0 \mathrm{~cm}^{3}$ of sodium hydroxide, FA 1.
(iii) Calculate the concentration of sulfuric acid in FA 2.

$$
\begin{equation*}
\text { concentration of } \mathrm{H}_{2} \mathrm{SO}_{4}=\ldots \ldots \ldots \ldots \ldots \ldots \ldots . . . . . . . . . . . . . . . . \mathrm{moldm}^{-3} \tag{1}
\end{equation*}
$$

(c) A student carrying out the same procedure used the results from their graph to determine the enthalpy of neutralisation for the reaction.

$$
\mathrm{H}^{+}(\mathrm{aq})+\mathrm{OH}^{-}(\mathrm{aq}) \rightarrow \mathrm{H}_{2} \mathrm{O}(\mathrm{l})
$$

(i) State how the student used their graph to determine the value of $\Delta T$ for use in the equation $q=m c \Delta T$.
$\qquad$
$\qquad$
(ii) The student correctly calculated the value of $\Delta H$ for the reaction as $\Delta H=-55.2 \mathrm{~kJ} \mathrm{~mol}^{-1}$. The theoretical value for $\Delta H_{\text {neut }}^{\ominus}$ given in the student's textbook is $-57.6 \mathrm{~kJ} \mathrm{~mol}^{-1}$.

Calculate the percentage error in the student's result compared with the theoretical value.
percentage error = \% [1]
(iii) Suggest why the student's result was less negative than the theoretical value. Explain your answer.
$\qquad$
$\qquad$

2 Solid sodium sulfite is often provided as the hydrated salt, $\mathrm{Na}_{2} \mathrm{SO}_{3} \bullet \mathrm{H}_{2} \mathrm{O}$, where $x$ is an integer. You will determine $x$ by using a solution of this sodium sulfite and reacting it with an excess of aqueous iodine.

$$
\mathrm{Na}_{2} \mathrm{SO}_{3}(\mathrm{aq})+\mathrm{I}_{2}(\mathrm{aq})+\mathrm{H}_{2} \mathrm{O}(\mathrm{I}) \rightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}(\mathrm{aq})+2 \mathrm{I}^{-}(\mathrm{aq})+2 \mathrm{H}^{+}(\mathrm{aq})
$$

The amount of iodine remaining will be determined by titration using a known concentration of sodium thiosulfate, $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$.

$$
\mathrm{I}_{2}(\mathrm{aq})+2 \mathrm{~S}_{2} \mathrm{O}_{3}^{2-}(\mathrm{aq}) \rightarrow 2 \mathrm{I}^{-}(\mathrm{aq})+\mathrm{S}_{4} \mathrm{O}_{6}^{2-}(\mathrm{aq})
$$

FA 3 is a solution containing $31.50 \mathrm{~g} \mathrm{dm}^{-3}$ of hydrated sodium sulfite, $\mathrm{Na}_{2} \mathrm{SO}_{3} \times \mathrm{H}_{2} \mathrm{O}$.
FA 4 is $0.100 \mathrm{moldm}^{-3}$ iodine, $\mathrm{I}_{2}$.
FA 5 is 0.100 moldm ${ }^{-3}$ sodium thiosulfate, $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$.
FA 6 is starch indicator.

## (a) Method

- Pipette $10.0 \mathrm{~cm}^{3}$ of FA 3 into a conical flask.
- Pipette $25.0 \mathrm{~cm}^{3}$ of FA 4 into the same flask.
- Swirl the flask to mix the contents.
- Fill the second burette with FA 5.
- Add FA 5 to the flask until the mixture is yellow.
- Add approximately 10 drops of FA 6.
- Complete the rough titration by adding FA 5 until the mixture is colourless.
- Record your burette readings in the space below.

The rough titre is $\qquad$ $\mathrm{cm}^{3}$.

- 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 5 added in each accurate titration.

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

(b) From your accurate titration results, calculate a suitable mean value to use in your calculations. Show clearly how you obtain the mean value.
$10.0 \mathrm{~cm}^{3}$ of FA 3 plus $25.0 \mathrm{~cm}^{3}$ of FA 4 required $\qquad$ $\mathrm{cm}^{3}$ of FA 5. [1]

## (c) Calculations

(i) Give your answers to (c)(ii), (iii) and (iv) to an appropriate number of significant figures.
(ii) Use your answer to (b) to calculate the amount, in mol, of sodium thiosulfate, FA 5, required to react with the excess iodine which remained in the conical flask.

$$
\text { amount of } \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}=
$$

$\qquad$ mol

Hence calculate the amount, in mol, of iodine, FA 4, remaining in the conical flask.

$$
\text { amount of } I_{2} \text { remaining }=
$$

$\qquad$ mol
(iii) Calculate the amount, in mol, of iodine, FA 4, added to the conical flask.
amount of $\mathrm{I}_{2}$ added $=$ $\qquad$ mol

Hence calculate the amount, in mol, of iodine that reacted with the $10.0 \mathrm{~cm}^{3}$ of sodium sulfite, FA 3.
amount of $\mathrm{I}_{2}$ that reacted with $\mathrm{Na}_{2} \mathrm{SO}_{3}=$
(iv) Use your final answer to (c)(iii) and the information on page 5 to calculate the amount, in mol, of sodium sulfite present in $1.00 \mathrm{dm}^{3}$ of FA 3.
(v) Use your answer to (c)(iv) to calculate the value of $x$ in $\mathrm{Na}_{2} \mathrm{SO}_{3} \cdot x \mathrm{H}_{2} \mathrm{O}$.

$$
x=
$$

(d) A student suggests that sodium carbonate should be added to each mixture of sodium sulfite and iodine in the conical flask before titrating with sodium thiosulfate.

State whether you agree with the student. Explain your answer.
$\qquad$
$\qquad$
[Total: 15]

## 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 FA 7 and FA 8 are solutions containing a total of three cations and two anions. Two of the cations and both of the anions are listed in the Qualitative analysis notes.
(a) (i) Carry out the following tests and record your observations. Use a fresh 1 cm depth of solution in a test-tube for each test.

| test | observations |  |
| :---: | :---: | :---: |
|  | FA 7 | FA 8 |
| Test 1 <br> Add a 1 cm depth of dilute nitric or hydrochloric acid and allow to stand for 2 minutes, then |  |  |
| add a few drops of aqueous barium nitrate or aqueous barium chloride. |  |  |
| Test 2 <br> Add a few drops of acidified aqueous potassium manganate(VII). |  |  |
| Test 3 <br> Add a few drops of aqueous iron(III) chloride and allow to stand for 1 minute. |  |  |

(ii) From your test results, give the formulae of the anions present in FA 7 and FA 8. If the tests do not allow you to positively identify an anion, write 'unknown'.
anion in FA 7 = $\qquad$
anion in FA $8=$
(b) (i) Select reagents for tests to identify as many of the cations as possible in FA 7 and FA 8. Carry out your tests and record your reagents, conditions and observations.
(ii) From your test results, give the formulae of as many cations as possible in FA 7 and FA 8. If the tests do not allow you to positively identify a cation, write 'unknown'.

FA 7 contains $\qquad$ .

FA 8 contains $\qquad$
(iii) Write an ionic equation for one reaction you observed in (b)(i). Include state symbols.

## 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 $\left[\mathrm{Ba}^{2+}(\mathrm{aq})\right]$ is very low | no ppt. |
| calcium, $\mathrm{Ca}^{2+}(\mathrm{aq})$ | white ppt. unless $\left[\mathrm{Ca}^{2+}(\mathrm{aq})\right]$ is very low | no ppt. |
| chromium(III), $\mathrm{Cr}^{3+}(\mathrm{aq})$ | grey-green ppt. soluble in excess 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 giving dark blue solution |
| iron(II), $\mathrm{Fe}^{2+}(\mathrm{aq})$ | green ppt. turning brown on contact with air insoluble in excess | green ppt. turning brown on contact with air insoluble in excess |
| iron(III), $\mathrm{Fe}^{3+}(\mathrm{aq})$ | 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), Mn ${ }^{2+}(\mathrm{aq})$ | 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, $\mathrm{Zn}^{2+}(\mathrm{aq})$ | 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, $I_{2}$ | 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


|  |  |
| :---: | :---: |
|  |  |
|  | ${ }^{\text {a }}$ |
| §㐫髧管 |  |
|  | ®® |
|  | \％ |
|  | 的莎喜， |
|  | ® E |
| 8 \％З | \％ |
|  | する |
| ธ ¢ 竞 | \％${ }^{2}$ |
|  | ～ว |
|  |  |
|  | ®「 「 |
|  | ® ¢ |

To avoid the issue of disclosure of answer－related information to candidates，all copyright acknowledgements are reproduced online in the Cambridge Assessment International Education Copyright Acknowledgements Booklet．This is produced for each series of examinations and is freely available to download at www．cambridgeinternational．org after the live examination series．

