Cambridge
International
AS \& A Level

## Cambridge International Examinations

Cambridge International Advanced Subsidiary and Advanced Level


## CENTRE

 NUMBER


## CHEMISTRY

9701/31
Paper 3 Advanced Practical Skills 1
October/November 2015
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.
A copy of the Periodic Table is printed on page 12.
Qualitative Analysis Notes are printed on pages 10 and 11.
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.


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

This document consists of $\mathbf{1 2}$ printed pages and 1 insert.

1 In this experiment you will determine the ionic equation for the reaction of acidified potassium manganate(VII) with potassium iodide. Excess potassium iodide is used and the reaction produces iodine. The amount of iodine produced is measured by titration with sodium thiosulfate.

FA 1 is 0.0180 mol dm ${ }^{-3}$ potassium manganate(VII), $\mathrm{KMnO}_{4}$.
FA 2 is $1.00 \mathrm{moldm}^{-3}$ sulfuric acid, $\mathrm{H}_{2} \mathrm{SO}_{4}$.
FA 3 is $0.500 \mathrm{moldm}^{-3}$ potassium iodide, KI.
FA 4 is 0.100 moldm ${ }^{-3}$ sodium thiosulfate, $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$. starch indicator

## (a) Method

- Pipette $25.0 \mathrm{~cm}^{3}$ of FA 1 into a conical flask.
- Use the measuring cylinder to add $25 \mathrm{~cm}^{3}$ of FA 2 to the conical flask.
- Use the measuring cylinder to add $20 \mathrm{~cm}^{3}$ of FA 3 to the conical flask.
- Fill the burette with FA 4.
- Carry out a rough titration. When the colour of the mixture becomes yellow/orange, add a few drops of starch indicator. Then titrate until the mixture goes colourless.
- Record all your burette readings in the space below.
$\qquad$ $\mathrm{cm}^{3}$.
- 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 4 added in each accurate titration.


## Keep FA 1 and FA 2 for use in Question 3 and FA 4 for use in Question 2.

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

(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 have obtained this value.
$\qquad$

## (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 in the volume of FA 4 calculated in (b).

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

$\qquad$ mol
(ii) Use the equation below to calculate the number of moles of iodine that reacted with the sodium thiosulfate in the titration.

$$
\mathrm{I}_{2}+2 \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3} \rightarrow \mathrm{Na}_{2} \mathrm{~S}_{4} \mathrm{O}_{6}+2 \mathrm{NaI}
$$

$$
\text { moles of } I_{2}=
$$

$\qquad$ mol
(iii) Use information on page 2 to calculate the number of moles of potassium manganate(VII) in FA 1 used in the titration.
moles of $\mathrm{KMnO}_{4}=$ $\qquad$ mol
(iv) From your answers to (ii) and (iii), calculate the number of moles of iodine produced by the reaction of $\mathbf{2 . 0 0}$ moles of potassium manganate(VII) with excess potassium iodide.
moles $I_{2}=$ $\qquad$ mol
(v) Using your answer to (iv), put a tick next to the ionic equation that represents the reaction between FA 1 and FA 3.

$$
\begin{aligned}
& 2 \mathrm{MnO}_{4}^{-}+2 \mathrm{I}^{-}+16 \mathrm{H}^{+} \rightarrow \mathrm{I}_{2}+2 \mathrm{Mn}^{6+}+8 \mathrm{H}_{2} \mathrm{O} \\
& 2 \mathrm{MnO}_{4}^{-}+4 \mathrm{I}^{-}+16 \mathrm{H}^{+} \rightarrow 2 \mathrm{I}_{2}+2 \mathrm{Mn}^{5+}+8 \mathrm{H}_{2} \mathrm{O} \\
& 2 \mathrm{MnO}_{4}^{-}+6 \mathrm{I}^{-}+16 \mathrm{H}^{+} \rightarrow 3 \mathrm{I}_{2}+2 \mathrm{Mn}^{++}+8 \mathrm{H}_{2} \mathrm{O} \\
& 2 \mathrm{MnO}_{4}^{-}+8 \mathrm{I}^{-}+16 \mathrm{H}^{+} \rightarrow 4 \mathrm{I}_{2}+2 \mathrm{Mn}^{3+}+8 \mathrm{H}_{2} \mathrm{O} \\
& 2 \mathrm{MnO}_{4}^{-}+10 \mathrm{I}^{-}+16 \mathrm{H}^{+} \rightarrow 5 \mathrm{I}_{2}+2 \mathrm{Mn}^{2+}+8 \mathrm{H}_{2} \mathrm{O} \\
& 2 \mathrm{MnO}_{4}^{-}+12 \mathrm{I}^{-}+16 \mathrm{H}^{+} \rightarrow 6 \mathrm{I}_{2}+2 \mathrm{Mn}^{+}+8 \mathrm{H}_{2} \mathrm{O}
\end{aligned}
$$

$\qquad$
$\qquad$
$\qquad$
$\qquad$
$\qquad$
$\qquad$
(vi) Prove that the iodide ion has been oxidised in the equation that you selected in (v).
$\qquad$
$\qquad$
$\qquad$
(d) (i) The error in calibration of the pipette you used is $\pm 0.06 \mathrm{~cm}^{3}$. Calculate the percentage error when measuring FA 1, using the pipette.
percentage error =
(ii) A student suggested that the experiment would be more accurate if a pipette was used to measure solution FA 3.
State and explain whether you agree with the student.
$\qquad$
$\qquad$
$\qquad$

2 In this experiment you will investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid is affected by the concentration of the acid.

When aqueous thiosulfate ions react with hydrogen ions, $\mathrm{H}^{+}$, in any acid, a pale yellow precipitate of sulfur is formed. The ionic equation for this reaction is given below.

$$
\mathrm{S}_{2} \mathrm{O}_{3}^{2-}(\mathrm{aq})+2 \mathrm{H}^{+}(\mathrm{aq}) \rightarrow \mathrm{S}(\mathrm{~s})+\mathrm{SO}_{2}(\mathrm{aq})+\mathrm{H}_{2} \mathrm{O}(\mathrm{l})
$$

The rate of the reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur.

FA 4 is $0.10 \mathrm{moldm}^{-3}$ sodium thiosulfate, $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$. FA 5 is $0.20 \mathrm{~mol} \mathrm{dm}^{-3}$ hydrochloric acid, HCl .

## (a) Method

Record all your measurements, in an appropriate form, in the space below.

## Experiment 1

- Use the larger measuring cylinder to transfer $40 \mathrm{~cm}^{3}$ of FA 4 into the $100 \mathrm{~cm}^{3}$ beaker.
- Rinse the larger measuring cylinder thoroughly with water, then add $30 \mathrm{~cm}^{3}$ of FA 5 to the beaker and start timing immediately.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Look down through the solution in the beaker at the print on the insert.
- Stop timing as soon as the precipitate of sulfur makes the print on the insert invisible.
- Record the reaction time to the nearest second.
- Empty and rinse the $100 \mathrm{~cm}^{3}$ beaker.
- Dry the outside of the beaker ready for Experiment 2.


## Experiment 2

- Rinse the larger measuring cylinder, then use it to transfer $40 \mathrm{~cm}^{3}$ of FA 4 into the $100 \mathrm{~cm}^{3}$ beaker.
- Use the smaller measuring cylinder to add $10 \mathrm{~cm}^{3}$ of distilled water to the beaker.
- Use the same measuring cylinder to add $20 \mathrm{~cm}^{3}$ of FA 5 to the mixture in the beaker and start timing immediately.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Stop timing as soon as the print on the insert becomes invisible.
- Record the reaction time to the nearest second.
- Empty and rinse the $100 \mathrm{~cm}^{3}$ beaker.
- Dry the outside of the beaker ready for Experiment 3.


## Experiment 3

- Carry out the reaction using a mixture of $40 \mathrm{~cm}^{3}$ of FA $4,20 \mathrm{~cm}^{3}$ of distilled water and $10 \mathrm{~cm}^{3}$ of FA 5.
- Measure and record the reaction time to the nearest second.

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

(b) (i) The 'rate of reaction' can be represented by the formula below.

$$
\text { 'rate of reaction' }=\frac{1000}{\text { reaction time }}
$$

Use this formula to calculate the 'rate of reaction' for Experiments 1 and 3. Give the unit.

‘rate of reaction’ for Experiment 1 unit<br>'rate of reaction' for Experiment 3 unit

(ii) Calculate the initial concentrations of hydrochloric acid in the reaction mixtures in Experiments 1 and 3.

(iii) How is the 'rate of reaction' affected by the concentration of hydrochloric acid in the mixture?
$\qquad$
$\qquad$
(iv) Predict how the reaction time measured in Experiment 1 would have been affected if the experiment had been carried out using $0.20 \mathrm{~mol} \mathrm{dm}^{-3}$ sulfuric acid instead of $0.20 \mathrm{moldm}^{-3}$ hydrochloric acid.
Explain your answer.
$\qquad$
$\qquad$
$\qquad$
(v) Predict how the reaction time measured in Experiment 3 would have been affected if the experiment had been carried out in a $250 \mathrm{~cm}^{3}$ beaker instead of a $100 \mathrm{~cm}^{3}$ beaker.
Explain your answer.
$\qquad$
$\qquad$

## 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 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.
Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.
(a) FA 6 is a sodium compound containing one anion listed on page 11.

Dissolve the FA 6 provided in about $15 \mathrm{~cm}^{3}$ of distilled water in a boiling tube.
Carry out the following tests and record your observations in the table below.

| test | observations |
| :--- | :--- | :--- |
| (i)To a 1cm depth of the solution <br> of FA 6 in a test-tube, add a few <br> drops of aqueous barium chloride <br> or aqueous barium nitrate, then |  |
| add dilute hydrochloric acid. |  |


| test | observations |
| :--- | :--- |
| (iii)To a 1 cm depth of the solution <br> of FA 6 in a boiling tube, add an <br> equal volume of FA 2, sulfuric <br> acid, then |  |
| heat the mixture gently and <br> cautiously. |  |
| (iv)To a 1 cm depth of the solution <br> of FA 6 in a test-tube, add an <br> equal volume of aqueous sodium <br> hydroxide, then |  |
| add a few drops of FA 1, aqueous <br> potassium manganate(VII), then |  |

(v) Identify the anion in FA 6, and state one piece of evidence for your identification.
anion
evidence $\qquad$
$\qquad$
$\qquad$
(vi) Give the chemical equation for the reaction between FA 6 and hydrogen peroxide, $\mathrm{H}_{2} \mathrm{O}_{2}$, in test (ii). State symbols are not required.
$\qquad$
(b) FA 7, FA 8, FA 9 and FA 10 each contain one cation from the list on page 10. You will attempt to identify the cations by testing with aqueous sodium hydroxide and aqueous ammonia.
In each case, use a 1 cm depth of the solution in a test-tube.
(i) Complete the table below.

| test | observations |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
|  | FA 7 | FA 8 | FA 9 | FA 10 |
| add sodium <br> hydroxide |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |
| add aqueous <br> ammonia |  |  |  |  |

(ii) Use your observations to identify, as far as possible, the cation present in each solution. If alternative identities are possible, state this clearly.

FA 7 cation $\qquad$
FA 8 cation $\qquad$
FA 9 cation $\qquad$
FA 10 cation $\qquad$
(iii) Give the ionic equation for the reaction of one of your cations with a few drops of sodium hydroxide. State symbols are not required.
$\qquad$
(iv) The precipitates obtained when alkalis are added to solutions of certain cations are sometimes difficult to see. Suggest how, using no additional apparatus, the experiment could be repeated in a way that would make these precipitates more visible.
$\qquad$
$\qquad$

## Qualitative Analysis Notes

Key: [ppt. = precipitate]

## 1 Reactions of aqueous cations

| ion | reaction with |  |
| :---: | :---: | :---: |
|  | $\mathrm{NaOH}(\mathrm{aq})$ | $\mathrm{NH}_{3}(\mathrm{aq})$ |
| aluminium, $\mathrm{Al} l^{3+}(\mathrm{aq})$ | white ppt. soluble in excess | white ppt. insoluble in excess |
| ammonium, $\mathrm{NH}_{4}{ }^{+}(\mathrm{aq})$ | no ppt. ammonia produced on heating | - |
| barium, $\mathrm{Ba}^{2+}(\mathrm{aq})$ | no ppt. (if reagents are pure) | no ppt. |
| calcium, $\mathrm{Ca}^{2+}(\mathrm{aq})$ | white ppt. with high [ $\mathrm{Ca}^{2+}(\mathrm{aq})$ ] | no ppt. |
| $\begin{aligned} & \text { chromium(III), } \\ & \mathrm{Cr}^{r^{+}(\mathrm{aq})} \end{aligned}$ | grey-green ppt. soluble in excess giving dark green solution | grey-green ppt. insoluble in excess |
| $\begin{aligned} & \text { copper(II), } \\ & \mathrm{Cu}^{2+}(\mathrm{aq}) \end{aligned}$ | pale blue ppt. insoluble in excess | blue ppt. soluble in excess giving dark blue solution |
| iron(II), <br> $\mathrm{Fe}^{2+}(\mathrm{aq})$ | green ppt. turning brown on contact with air insoluble in excess | green ppt. turning brown on contact with air insoluble in excess |
| iron(III), <br> $\mathrm{Fe}^{3+}(\mathrm{aq})$ | 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 |
| $\begin{aligned} & \text { manganese(II), } \\ & \mathrm{Mn}^{2+}(\mathrm{aq}) \end{aligned}$ | 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

| ion | reaction |
| :---: | :---: |
| carbonate, $\mathrm{CO}_{3}{ }^{2-}$ | $\mathrm{CO}_{2}$ liberated by dilute acids |
| chloride, <br> $\mathrm{Cl}^{-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (soluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ) |
| bromide, $\mathrm{Br}^{-}(\mathrm{aq})$ | gives cream ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (partially soluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ) |
| iodide, $\mathrm{I}^{-}(\mathrm{aq})$ | gives 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; NO liberated by dilute acids (colourless $\mathrm{NO} \rightarrow$ (pale) brown $\mathrm{NO}_{2}$ in air) |
| sulfate, $\mathrm{SO}_{4}{ }^{2-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ba}^{2+}(\mathrm{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\mathrm{SO}_{3}{ }^{2-}(\mathrm{aq})$ | $\mathrm{SO}_{2}$ liberated with dilute acids; gives white ppt. with $\mathrm{Ba}^{2+}(\mathrm{aq})$ (soluble in excess dilute strong acids) |

## 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 <br> (ppt. dissolves with excess $\mathrm{CO}_{2}$ ) |
| chlorine, $\mathrm{Cl}_{2}$ | bleaches damp litmus paper |
| hydrogen, $\mathrm{H}_{2}$ | "pops" with a lighted splint |
| oxygen, $\mathrm{O}_{2}$ | relights a glowing splint |
| sulfur dioxide, $\mathrm{SO}_{2}$ | turns acidified aqueous potassium manganate(VII) from purple to <br> colourless |

The Periodic Table of the Elements



