UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

## CANDIDATE NAME



CENTRE


CANDIDATE NUMBER


## CHEMISTRY

Advanced Practical Skills
May/June 2011
2 hours
Candidates answer on the Question Paper.
Additional Materials: As listed in the Instructions to Supervisors

## 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 a soft pencil for any diagrams, graphs or rough working.
Do not use staples, paper clips, highlighters, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.
Answer all questions.
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 14 and 15.
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 $\mathbf{1 5}$ printed pages and $\mathbf{1}$ blank page.

You are to determine the percentage by mass of water in the borax crystals.

Borax reacts with hydrochloric acid according to the equation.

$$
\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7}(\mathrm{aq})+2 \mathrm{HCl}(\mathrm{aq})+5 \mathrm{H}_{2} \mathrm{O}(\mathrm{l}) \rightarrow 2 \mathrm{NaCl}(\mathrm{aq})+4 \mathrm{H}_{3} \mathrm{BO}_{3}(\mathrm{aq})
$$

1 FA 1 is an aqueous solution containing $38.10 \mathrm{gdm}^{-3}$ of borax crystals. Borax has the formula, $\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7} \cdot \mathbf{x H}_{2} \mathrm{O}$.
FA 2 is $1.00 \mathrm{~mol} \mathrm{dm}^{-3}$ hydrochloric acid, HCl .
You are also provided with an indicator suitable for the titration of a strong acid and a weak base.

The indicator provided is $\qquad$
(a) Method

## Dilution

- Fill the burette with FA 2.
- Run between $44.50 \mathrm{~cm}^{3}$ and $45.50 \mathrm{~cm}^{3}$ of FA 2 from the burette into the $250 \mathrm{~cm}^{3}$ graduated (volumetric) flask, labelled FA 3.
- Make the solution up to the mark with distilled water.
- $\quad$ Shake the flask to mix the solution of FA 3.

In the space below record your burette readings and the volume of FA 2 added to the graduated flask.

## Titration

- Fill a second burette with FA 3, the diluted hydrochloric acid.
- Pipette $25.0 \mathrm{~cm}^{3}$ of FA 1 into a conical flask.
- Add to the flask a few drops of the indicator provided.
- Titrate the borax in the flask with FA 3 until the appropriate colour change is observed for the end-point.

You should perform a rough titration.
In the space below record your burette readings for this rough titration.
$\mathrm{cm}^{3}$.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.

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(b) From your accurate titration results obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.
$25.0 \mathrm{~cm}^{3}$ of FA 1 required $\qquad$ $\mathrm{cm}^{3}$ of FA 3

## (c) Calculations

Show your working and appropriate significant figures in the final answer to each step of your calculations.
(i) Calculate the concentration of hydrochloric acid, in $\mathrm{mol} \mathrm{dm}^{-3}$, in the diluted solution, FA 3.
$\qquad$ $\mathrm{moldm}^{-3}$.
(ii) Calculate how many moles of HCl were present in the volume of FA 3 calculated in (b).
(iii) Calculate how many moles of $\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7}$ reacted with the HCl in (ii).

$$
\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7}(\mathrm{aq})+2 \mathrm{HCl}(\mathrm{aq})+5 \mathrm{H}_{2} \mathrm{O}(\mathrm{l}) \rightarrow 2 \mathrm{NaCl}(\mathrm{aq})+4 \mathrm{H}_{3} \mathrm{BO}_{3}(\mathrm{aq})
$$

The HCl run from the burette reacted with $\qquad$ mol of $\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7}$.
(iv) Calculate the concentration, in $\mathrm{gdm}^{-3}$, of $\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7}$ in FA 1 .
[ $A_{\mathrm{r}}$ : B, 10.8; O, 16.0; Na, 23.0]

$$
\text { The concentration of } \mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7} \text { in FA } 1 \text { is ...................... } \mathrm{gdm}^{-3} \text {. }
$$

(v) Use your answer to (iv) and the information at the start of question 1 to calculate the percentage by mass of water in the borax crystals, $\mathrm{Na}_{2} \mathrm{~B}_{4} \mathrm{O}_{7} \cdot \mathrm{xH}_{2} \mathrm{O}$.
[ $\left.A_{\mathrm{r}}: \mathrm{H}, 1.0 ; \mathrm{B}, 10.8 ; \mathrm{O}, 16.0 ; \mathrm{Na}, 23.0\right]$
$\qquad$ \% water.
(d) The maximum error for a $25 \mathrm{~cm}^{3}$ pipette commonly used in schools is $\pm 0.06 \mathrm{~cm}^{3}$. The maximum error in any single burette reading is $\pm 0.05 \mathrm{~cm}^{3}$.

Calculate the maximum percentage error in each of the following.
(i) The volume of FA $\mathbf{1}$ pipetted into the conical flask.
maximum percentage error in the pipetted volume $=$
(ii) The titre volume calculated in (b).
maximum percentage error in titre volume $=$

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2 Read through the question carefully before starting any practical work.

Sodium carbonate, $\mathrm{Na}_{2} \mathrm{CO}_{3}$,

- reacts exothermically with hydrochloric acid,
- does not decompose when heated,
- reacts with acids.

$$
\mathrm{CO}_{3}^{2-}(\mathrm{s})+2 \mathrm{H}^{+}(\mathrm{aq}) \rightarrow \mathrm{H}_{2} \mathrm{O}(\mathrm{I})+\mathrm{CO}_{2}(\mathrm{~g})
$$

Sodium hydrogencarbonate, $\mathrm{NaHCO}_{3}$,

- reacts endothermically with hydrochloric acid,
- decomposes when heated,

$$
2 \mathrm{HCO}_{3}^{-}(\mathrm{s}) \rightarrow \mathrm{H}_{2} \mathrm{O}(\mathrm{~g})+\mathrm{CO}_{2}(\mathrm{~g})+\mathrm{CO}_{3}^{2-}(\mathrm{s})
$$

- reacts with acids.

$$
\mathrm{HCO}_{3}^{-}(\mathrm{s})+\mathrm{H}^{+}(\mathrm{aq}) \rightarrow \mathrm{H}_{2} \mathrm{O}(\mathrm{I})+\mathrm{CO}_{2}(\mathrm{~g})
$$

You are to measure the temperature changes when samples of
(i) sodium carbonate,
(ii) sodium hydrogencarbonate,
(iii) a mixture of sodium carbonate and sodium hydrogencarbonate, react with an excess of hydrochloric acid.

FA 4 is sodium carbonate, $\mathrm{Na}_{2} \mathrm{CO}_{3}$.
FA 5 is sodium hydrogencarbonate, $\mathrm{NaHCO}_{3}$.
FA 6 is a mixture of sodium carbonate and sodium hydrogencarbonate.
FA 7 is $3.0 \mathrm{moldm}^{-3}$ hydrochloric acid, HCl .

## Method

## (a) sodium carbonate

- Support the plastic cup in a $250 \mathrm{~cm}^{3}$ beaker.
- Use a measuring cylinder to transfer $50 \mathrm{~cm}^{3}$ of FA 7 into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of the container labelled FA 4, containing $\mathrm{Na}_{2} \mathrm{CO}_{3}$.
- Carefully tip the sodium carbonate from the weighed container FA 4 into the hydrochloric acid in the plastic cup.
Note: There will be vigorous effervescence. Do not breathe the vapour. Add the solid in small portions with constant stirring using the thermometer.
- Record the highest temperature obtained.
- Reweigh the container FA 4 with any residual sodium carbonate. Record the mass.
- Empty and rinse the plastic cup and dry it using a paper towel.

In the space at the top of the next page, record, in an appropriate form,

- both balance readings and both temperature measurements,
- the mass of sodium carbonate, $\mathbf{m}_{1}$, used in the experiment,
- the temperature rise, $\Delta \mathbf{T}_{1}$.

Calculate the rise in temperature for each gram of sodium carbonate used in the experiment.

$$
\frac{\Delta \mathbf{T}_{1}}{\mathbf{m}_{1}}={\underset{\text { sign }}{+}}_{\begin{array}{c}
\text { value }
\end{array} \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots{ }^{\circ} \mathrm{Cg}^{-1}}
$$

(b) sodium hydrogencarbonate

- Support the plastic cup in a $250 \mathrm{~cm}^{3}$ beaker.
- Use a measuring cylinder to transfer $50 \mathrm{~cm}^{3}$ of FA 7 into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of the container labelled FA 5, containing $\mathrm{NaHCO}_{3}$.
- Carefully tip the sodium hydrogencarbonate from the weighed container FA 5 into the hydrochloric acid in the plastic cup.
Note: There will be vigorous effervescence. Add the solid in small portions with constant stirring using the thermometer.
- Record the lowest temperature obtained.
- Reweigh the container FA 5 with any residual sodium hydrogencarbonate.

Record the mass.

- Empty and rinse the plastic cup and dry it using a paper towel.

In the space below, record, in an appropriate form,

- both balance readings and both temperature measurements,
- the mass of sodium hydrogencarbonate, $\mathrm{m}_{2}$, used in the experiment,
- the temperature fall, $\Delta \mathbf{T}_{2}$.

Calculate the fall in temperature for each gram of sodium hydrogencarbonate used in the experiment.

$$
\frac{\Delta \mathbf{T}_{\mathbf{2}}}{\mathbf{m}_{\mathbf{2}}}=\underbrace{\begin{array}{l}
- \\
\text { value }
\end{array}}_{\text {sign }} .
$$

## (c) mixture of sodium carbonate and sodium hydrogencarbonate

- $\quad$ Support the plastic cup in a $250 \mathrm{~cm}^{3}$ beaker.
- Use a measuring cylinder to transfer $50 \mathrm{~cm}^{3}$ of FA 7 into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of a clean, dry, weighing-bottle or tube.
- Add to the tube between 8.5 g and 9.5 g of the mixture FA 6 .
- Record the mass of the weighing-bottle or tube + FA 6.
- Carefully tip the weighed mixture into the hydrochloric acid in the plastic cup.
- Note: There will be vigorous effervescence. Add the solid in small portions with constant stirring using the thermometer.
- Record the highest or lowest temperature obtained.
- Reweigh the weighing-bottle or tube with any residual mixture. Record the mass.

In the space below, record, in an appropriate form,

- all balance readings and temperature measurements,
- the mass of the mixture, $\mathbf{m}_{3}$, used in the experiment,
- the temperature change, $\Delta \mathbf{T}_{3}$.

Make certain that your recorded temperature change carries an appropriate sign.
(d) Transfer the following data from parts (a), (b) and (c).
(a) $\frac{\Delta \mathrm{T}_{1}}{\mathbf{m}_{1}}=+\ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots .{ }^{\circ} \mathrm{Cg}^{-1}$
(b) $\frac{\Delta \mathrm{T}_{\mathbf{2}}}{\mathrm{m}_{\mathbf{2}}}=-\quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots .{ }^{\circ} \mathrm{Cg}^{-1}$
(c) $m_{3}=\ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots . g \quad \Delta T_{3}=\square$
sign

The masses of sodium carbonate and sodium hydrogencarbonate in the weighed sample of the mixture used in experiment (c) can be represented as follows.

For Examiner's Use
mass of sodium carbonate $/ \mathrm{g}=\mathbf{W}$
mass of sodium hydrogencarbonate $/ \mathrm{g}=\left(\mathbf{m}_{3}-\mathbf{W}\right)$
Evaluate the following equation to determine a value for $\mathbf{W}$.

$$
\left[W \times \frac{\Delta T_{1}}{m_{1}}\right]+\left[\left(m_{3}-W\right) \times \frac{\Delta T_{2}}{m_{2}}\right]=\Delta T_{3}
$$

The mass of sodium carbonate was
(e) Use the information at the beginning of question 2 to outline an alternative method that could be used in a school laboratory to find the mass of sodium carbonate and the mass of sodium hydrogencarbonate in the mixture FA 6.
$\qquad$
$\qquad$
$\qquad$
$\qquad$

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. Marks are not given for chemical equations.
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 full name or correct formula of the reagents must be given.

FA 8, FA 9 and FA 10 are aqueous solutions each containing a sodium cation and a single anion which could be a nitrite, a nitrate or a halide.
(a) By reference to the Qualitative Analysis Notes on page 15, select a single reagent that would enable you to identify any solution containing the nitrite ion, $\mathrm{NO}_{2}{ }^{-}$.
reagent
Use this reagent to test each of the solutions. Record your observations in the table below. State clearly where no reaction has been observed.

| solution | observation |
| :---: | :---: |
| FA 8 |  |
| FA 9 |  |
| FA 10 |  |

(b) By reference to the Qualitative Analysis Notes on page 15, select one reagent that would show that a halide ion is present.
reagent $\qquad$

Use this reagent to test each of the solutions.
Record your observations in an appropriate form in the space below.

Select another reagent to identify or confirm which halide ions are present in the solutions.
reagent $\qquad$
Tick the appropriate statement about the use of this reagent.

| It is added to the tube already containing the first reagent. |  |
| :--- | :--- |
| It is added to a fresh sample of solution. |  |

Use this reagent to identify or confirm which halide ions are present in the solutions and record your observations in an appropriate form in the space below.
(c) From the results in (a) and (b) state which anions have been identified in the solutions.

Complete the following table.
Place a cross in any box if no anion has been identified.

| solution | FA 8 | FA 9 | FA 10 |
| :---: | :---: | :---: | :---: |
| anion present |  |  |  |
|  |  |  |  |

FA 11 and FA 12 are aqueous solutions each containing one cation from those listed in the Qualitative Analysis Notes printed on page 14.

For
Examiner's
Use
(d) Use aqueous sodium hydroxide and aqueous ammonia in separate tests to identify the cation present in each of the solutions. You will also require some of the solution, FA 11, for tests in (f).

Record the results of your experiments with sodium hydroxide and ammonia in an appropriate form in the space below.
(e) Identification of the cations in FA 11 and FA 12

Complete the table below.

| solution | FA 11 | FA 12 |
| :---: | :---: | :---: |
| cation present |  |  |
|  |  |  |

What is the evidence from your observations in (d) that enables you to identify the cation present in each of the solutions?

The evidence supporting the conclusion for the cation in FA 11 is
$\qquad$
$\qquad$
The evidence supporting the conclusion for the cation in FA 12 is
$\qquad$
$\qquad$
(f) Complete the following table.

|  | test | observations |
| :--- | :--- | :--- |
| (i)Pour 1 cm depth of FA 11 into a <br> test-tube. |  |  |
| Add 1 cm depth of aqueous <br> potassium iodide. |  |  |
| Divide this mixture into two parts <br> for use in (ii) and (iii) |  |  |
| (ii) | To the first part of the mixture <br> from (i) add a few drops of <br> starch solution. |  |
| (iii) | To the second part of the mixture <br> from (i) add aqueous sodium <br> thiosulfate, a drop at a time, until <br> no further change is observed. |  |

In part (i) and in part (iii) redox reactions have taken place.
Complete the table below to show the ion or molecule which has been oxidised and the ion or molecule which has been reduced in each of these reactions.

| reaction | the ion or molecule which has been |  |
| :---: | :---: | :---: |
|  | oxidised | reduced |
| (i) |  |  |
| (iii) |  |  |

## Qualitative Analysis Notes

Key: [ ppt. = precipitate ]

## 1 Reactions of aqueous cations

| ion | reaction with |  |
| :---: | :---: | :---: |
|  | $\mathrm{NaOH}(\mathrm{aq})$ | $\mathrm{NH}_{3}(\mathrm{aq})$ |
| aluminium, $\mathrm{Al} \mathrm{l}^{3+}(\mathrm{aq})$ | 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. |
| $\begin{aligned} & \text { chromium(III), } \\ & \mathrm{Cr}^{3+}(\mathrm{aq}) \end{aligned}$ | 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 |
| $\begin{aligned} & \text { iron(III), } \\ & \mathrm{Fe}^{3+}(\mathrm{aq}) \end{aligned}$ | red-brown ppt. insoluble in excess | red-brown ppt. insoluble in excess |
| $\begin{aligned} & \text { lead(II), } \\ & \mathrm{Pb}^{2+}(\mathrm{aq}) \end{aligned}$ | white ppt. <br> soluble in excess | white 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 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. <br> soluble in excess | white ppt. <br> soluble in excess |

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

## 2 Reactions of anions

| ion | reaction |
| :---: | :---: |
| carbonate, $\mathrm{CO}_{3}{ }^{2-}$ | $\mathrm{CO}_{2}$ liberated by dilute acids |
| chromate(VI), $\mathrm{CrO}_{4}{ }^{2-}(\mathrm{aq})$ | yellow solution turns orange with $\mathrm{H}^{+}(\mathrm{aq})$; gives yellow ppt. with $\mathrm{Ba}^{2+}(\mathrm{aq})$; gives bright yellow ppt. with $\mathrm{Pb}^{2+}(\mathrm{aq})$ |
| chloride, <br> $\mathrm{Cl}^{-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (soluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ); gives white ppt. with $\mathrm{Pb}^{2+}(\mathrm{aq})$ |
| bromide, <br> $\mathrm{Br}^{-}(\mathrm{aq})$ | gives cream ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (partially soluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ); gives white ppt. with $\mathrm{Pb}^{2+}(\mathrm{aq})$ |
| iodide, $\mathrm{I}^{-}(\mathrm{aq})$ | gives yellow ppt. with $\mathrm{Ag}^{+}(\mathrm{aq})$ (insoluble in $\mathrm{NH}_{3}(\mathrm{aq})$ ); gives yellow ppt. with $\mathrm{Pb}^{2+}(\mathrm{aq})$ |
| 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; <br> NO liberated by dilute acids <br> (colourless $\mathrm{NO} \rightarrow$ (pale) brown $\mathrm{NO}_{2}$ in air) |
| sulfate, $\mathrm{SO}_{4}{ }^{2-}(\mathrm{aq})$ | gives white ppt. with $\mathrm{Ba}^{2+}(\mathrm{aq})$ or with $\mathrm{Pb}^{2+}(\mathrm{aq})$ (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 dichromate(VI) from orange to green |

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