



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

CANDIDATE
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CHEMISTRY

9701/31

Advanced Practical Skills 1

October/November 2013

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 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.
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 11 and 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 |
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| For Examiner's Use | |
|--------------------|--|
| 1 | |
| 2 | |
| 3 | |
| Total | |

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



- 1 In this experiment you are to determine the relative formula mass of an iron(II) salt by titration with potassium manganate(VII).

FA 1 is the iron(II) salt.

FA 2 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 3 is dilute sulfuric acid, H_2SO_4 .

(a) Method

Preparing a solution of FA 1

- Weigh the 250 cm^3 beaker and record the mass in the space below.
- Add all the **FA 1** provided to the beaker. Weigh the beaker with **FA 1** and record the mass.
- Calculate the mass of **FA 1** used and record this in the space below.
- Use a measuring cylinder to add approximately 100 cm^3 of **FA 3** to the beaker. Stir until all the solid has dissolved.
- Transfer the solution into the 250 cm^3 volumetric (graduated) flask labelled **FA 4**.
- Wash out the beaker thoroughly using distilled water and add the washings to the volumetric flask. Make the solution up to the mark using distilled water.
- Shake the flask thoroughly to mix the solution before using it for your titrations.
- This solution of the iron(II) salt is **FA 4**.

Titration

- Pipette 25.0 cm^3 of **FA 4** into a conical flask.
- Use a measuring cylinder to add 20 cm^3 of **FA 3** to the flask.
- Fill the burette with **FA 2**.
- Titrate **FA 4** with **FA 2** until the solution changes to a permanent pink colour.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is 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 2** added in each accurate titration.

*For
Examiner's
Use*

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

[7]

- (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 cm³ of **FA 4** required cm³ of **FA 2** [1]

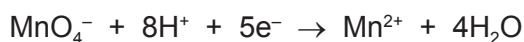
(c) Calculations

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

- (i) Calculate the number of moles of potassium manganate(VII) present in the volume of **FA 2** calculated in (b).

moles of $\text{KMnO}_4 = \dots\dots\dots \text{mol}$

- (ii) The half-equation for the reduction of a manganate(VII) ion is:



Give the half-equation for the oxidation of an iron(II) ion to an iron(III) ion.

.....

Therefore, 1 mole of manganate(VII) ions reacts with 5 moles of iron(II) ions.

- (iii) Calculate the number of moles of iron(II) ions present in 25.0 cm^3 of solution **FA 4**.

moles of Fe^{2+} in 25.0 cm^3 of **FA 4** = $\dots\dots\dots \text{mol}$

- (iv) Calculate the number of moles of iron(II) ions present in 250 cm^3 of solution **FA 4**.

moles of Fe^{2+} in 250 cm^3 of **FA 4** = $\dots\dots\dots \text{mol}$

- (v) In 1 mole of the iron(II) salt, **FA 1**, there is 1 mole of iron(II) ions.
Use the mass of **FA 1** you weighed out to calculate the relative formula mass of the iron(II) salt.

relative formula mass = $\dots\dots\dots$

[5]

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

- (d) (i) A 25 cm³ pipette is accurate to ± 0.06 cm³.
Calculate the maximum percentage error when the pipette was used to measure solution **FA 4**.

percentage error in measuring **FA 4** = %

- (ii) State the maximum error in the mass of the 250 cm³ beaker that you recorded in (a).

maximum error = g

- (iii) Calculate the maximum percentage error in the mass of **FA 1** used in (a).

maximum percentage error = %
[2]

[Total: 15]

- 2 In this experiment you are to determine the formula of hydrated barium chloride, **FA 5**, by heating to remove the water of crystallisation. You will heat **two** separate samples. The anhydrous barium chloride does not decompose when heated.

For
Examiner's
Use

FA 5 is hydrated barium chloride, $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$

(a) Method

Record **all** weighings, in an appropriate form, in the space below.

- Record the mass of the empty crucible **without** its lid.
- Add between 2.0 and 2.4 g of **FA 5** into the crucible. Record the mass of the crucible and its contents.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat the crucible and its contents gently for about **one** minute with the lid off. Then heat strongly for a further **four** minutes.
- Put the lid on the crucible and leave to cool for approximately 10 minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When the crucible is cool, **remove the lid**, and weigh the crucible with the residue.
- Record the mass of anhydrous barium chloride remaining in the crucible after heating and the mass of water lost.
- To prepare for the second experiment, use a spatula to remove the residue from the crucible into the beaker labelled **waste**.
- Reweigh the empty crucible **without** its lid.
- Carry out the experiment again. This time use between 1.5 and 1.9 g of **FA 5**.

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

[6]

(b) Calculation

Show your working in **each** step.

- (i) Calculate the **mean** number of moles of water removed from the hydrated salt in the experiments.
(A_r : H, 1.0; O, 16.0)

moles of H_2O = mol

- (ii) Calculate the **mean** number of moles of anhydrous barium chloride produced in the experiments.
(A_r : Ba, 137; Cl, 35.5)

moles of $BaCl_2$ = mol

- (iii) Calculate the value of **x** in the formula of hydrated barium chloride, $BaCl_2 \cdot xH_2O$.

x =
[3]

- (c) (i) Suggest how the experimental procedure could be modified to ensure that **all** of the water of crystallisation had been removed by heating hydrated **FA 5**.

.....
.....
.....

- (ii) Do you think that the results from your two experiments are consistent with each other?
Justify your answer by carrying out appropriate calculations.

[3]

[Total: 12]

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 5** is hydrated barium chloride.

FA 6 is the same iron(II) salt used in Question 1. It contains **one other** cation and **one** anion.

(i) Place a small spatula measure of **FA 6** into a test-tube. Dissolve the solid in about a 5 cm depth of distilled water. Use the solution for the following tests.

| <i>test</i> | <i>observations</i> |
|--|---------------------|
| To a 1 cm depth of aqueous FA 6 in a boiling tube, add aqueous sodium hydroxide until no further change occurs, then | |
| heat the mixture carefully. | |
| Dissolve a few crystals of FA 5 in a 1 cm depth of distilled water in a test-tube. Add a 1 cm depth of FA 6 , then | |
| add excess dilute hydrochloric acid to the mixture. | |

(ii) Identify the ions present in **FA 6**.

cations: Fe^{2+} and anion:

(iii) Give the ionic equation for the reaction of iron(II) ions with hydroxide ions.

.....

(iv) Place a **small** spatula measure of **FA 6** into a **hard-glass** test-tube.
Heat gently, then strongly, until no further change is observed.
Record your observations in the space below.

[7]

- (b) **FA 7**, **FA 8** and **FA 9** are aqueous solutions. Each contains **one** cation and **one** anion. **FA 3** is dilute sulfuric acid, H_2SO_4 .

For
Examiner's
Use

Mix pairs of solutions so that you can complete the table below.
For each test, use 1 cm depths of each solution in clean test-tubes.
Record your observations in the table.

| | FA 7 | FA 8 | FA 9 |
|-------------|-------------|-------------|-------------|
| FA 3 | | | |
| FA 7 | | | |
| FA 8 | | | |

From your observations, complete the following statements.

The anion in **FA 7** is

The cation in **FA 8** is

The anion in **FA 9** is

[6]

[Total: 13]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

| ion | reaction with | |
|--|--|--|
| | 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) | no ppt. (if 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 giving dark green solution | 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 |
| lead(II), Pb ²⁺ (aq) | white ppt. soluble in excess | white 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 |

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

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|--|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$ | yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| 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 $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\text{SO}_3^{2-}(\text{aq})$ | SO_2 liberated with dilute acids; 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 |
| sulfur dioxide, SO_2 | turns acidified aqueous potassium dichromate(VI) from orange to green |

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

CANDIDATE
NAME

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CHEMISTRY

9701/33

Advanced Practical Skills 1

October/November 2013

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

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Answer **all** questions.
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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 are to determine the relative formula mass of an iron(II) salt by titration with potassium manganate(VII).

FA 1 is the iron(II) salt.

FA 2 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 3 is dilute sulfuric acid, H_2SO_4 .

(a) Method

Preparing a solution of FA 1

- Weigh the 250 cm^3 beaker and record the mass in the space below.
- Add all the **FA 1** provided to the beaker. Weigh the beaker with **FA 1** and record the mass.
- Calculate the mass of **FA 1** used and record this in the space below.
- Use a measuring cylinder to add approximately 100 cm^3 of **FA 3** to the beaker. Stir until all the solid has dissolved.
- Transfer the solution into the 250 cm^3 volumetric (graduated) flask labelled **FA 4**.
- Wash out the beaker thoroughly using distilled water and add the washings to the volumetric flask. Make the solution up to the mark using distilled water.
- Shake the flask thoroughly to mix the solution before using it for your titrations.
- This solution of the iron(II) salt is **FA 4**.

Titration

- Pipette 25.0 cm^3 of **FA 4** into a conical flask.
- Use a measuring cylinder to add 20 cm^3 of **FA 3** to the flask.
- Fill the burette with **FA 2**.
- Titrate **FA 4** with **FA 2** until the solution changes to a permanent pink colour.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is 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 2** added in each accurate titration.

*For
Examiner's
Use*

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

[7]

- (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 cm³ of **FA 4** required cm³ of **FA 2** [1]

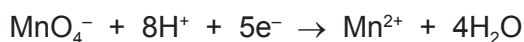
(c) Calculations

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

- (i) Calculate the number of moles of potassium manganate(VII) present in the volume of **FA 2** calculated in (b).

moles of $\text{KMnO}_4 = \dots\dots\dots$ mol

- (ii) The half-equation for the reduction of a manganate(VII) ion is:



Give the half-equation for the oxidation of an iron(II) ion to an iron(III) ion.

.....

Therefore, 1 mole of manganate(VII) ions reacts with 5 moles of iron(II) ions.

- (iii) Calculate the number of moles of iron(II) ions present in 25.0 cm^3 of solution **FA 4**.

moles of Fe^{2+} in 25.0 cm^3 of **FA 4** = mol

- (iv) Calculate the number of moles of iron(II) ions present in 250 cm^3 of solution **FA 4**.

moles of Fe^{2+} in 250 cm^3 of **FA 4** = mol

- (v) In 1 mole of the iron(II) salt, **FA 1**, there is 1 mole of iron(II) ions.
Use the mass of **FA 1** you weighed out to calculate the relative formula mass of the iron(II) salt.

relative formula mass =

[5]

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

- (d) (i) A 25 cm³ pipette is accurate to ± 0.06 cm³.
Calculate the maximum percentage error when the pipette was used to measure solution **FA 4**.

percentage error in measuring **FA 4** = %

- (ii) State the maximum error in the mass of the 250 cm³ beaker that you recorded in (a).

maximum error = g

- (iii) Calculate the maximum percentage error in the mass of **FA 1** used in (a).

maximum percentage error = %
[2]

[Total: 15]

- 2 In this experiment you are to determine the formula of hydrated barium chloride, **FA 5**, by heating to remove the water of crystallisation. You will heat **two** separate samples. The anhydrous barium chloride does not decompose when heated.

For
Examiner's
Use

FA 5 is hydrated barium chloride, $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$

(a) Method

Record **all** weighings, in an appropriate form, in the space below.

- Record the mass of the empty crucible **without** its lid.
- Add between 2.0 and 2.4 g of **FA 5** into the crucible. Record the mass of the crucible and its contents.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat the crucible and its contents gently for about **one** minute with the lid off. Then heat strongly for a further **four** minutes.
- Put the lid on the crucible and leave to cool for approximately 10 minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When the crucible is cool, **remove the lid**, and weigh the crucible with the residue.
- Record the mass of anhydrous barium chloride remaining in the crucible after heating and the mass of water lost.
- To prepare for the second experiment, use a spatula to remove the residue from the crucible into the beaker labelled **waste**.
- Reweigh the empty crucible **without** its lid.
- Carry out the experiment again. This time use between 1.5 and 1.9 g of **FA 5**.

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

[6]

(b) Calculation

Show your working in **each** step.

- (i) Calculate the **mean** number of moles of water removed from the hydrated salt in the experiments.
(A_r : H, 1.0; O, 16.0)

moles of H_2O = mol

- (ii) Calculate the **mean** number of moles of anhydrous barium chloride produced in the experiments.
(A_r : Ba, 137; Cl, 35.5)

moles of BaCl_2 = mol

- (iii) Calculate the value of **x** in the formula of hydrated barium chloride, $\text{BaCl}_2 \cdot x\text{H}_2\text{O}$.

x =
[3]

- (c) (i) Suggest how the experimental procedure could be modified to ensure that **all** of the water of crystallisation had been removed by heating hydrated **FA 5**.

.....
.....
.....

- (ii) Do you think that the results from your two experiments are consistent with each other?
Justify your answer by carrying out appropriate calculations.

[3]

[Total: 12]

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 5** is hydrated barium chloride.

FA 6 is the same iron(II) salt used in Question 1. It contains **one other** cation and **one** anion.

(i) Place a small spatula measure of **FA 6** into a test-tube. Dissolve the solid in about a 5 cm depth of distilled water. Use the solution for the following tests.

| <i>test</i> | <i>observations</i> |
|--|---------------------|
| To a 1 cm depth of aqueous FA 6 in a boiling tube, add aqueous sodium hydroxide until no further change occurs, then | |
| heat the mixture carefully. | |
| Dissolve a few crystals of FA 5 in a 1 cm depth of distilled water in a test-tube. Add a 1 cm depth of FA 6 , then | |
| add excess dilute hydrochloric acid to the mixture. | |

(ii) Identify the ions present in **FA 6**.

cations: Fe^{2+} and anion:

(iii) Give the ionic equation for the reaction of iron(II) ions with hydroxide ions.

.....

(iv) Place a **small** spatula measure of **FA 6** into a **hard-glass** test-tube.
Heat gently, then strongly, until no further change is observed.
Record your observations in the space below.

[7]

- (b) **FA 7**, **FA 8** and **FA 9** are aqueous solutions. Each contains **one** cation and **one** anion. **FA 3** is dilute sulfuric acid, H_2SO_4 .

For
Examiner's
Use

Mix pairs of solutions so that you can complete the table below.
For each test, use 1 cm depths of each solution in clean test-tubes.
Record your observations in the table.

| | FA 7 | FA 8 | FA 9 |
|-------------|-------------|-------------|-------------|
| FA 3 | | | |
| FA 7 | | | |
| FA 8 | | | |

From your observations, complete the following statements.

The anion in **FA 7** is

The cation in **FA 8** is

The anion in **FA 9** is

[6]

[Total: 13]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

| ion | reaction with | |
|--|--|--|
| | 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) | no ppt. (if 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 giving dark green solution | 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 |
| lead(II), Pb ²⁺ (aq) | white ppt. soluble in excess | white 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 |

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

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|--|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$ | yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| 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 $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\text{SO}_3^{2-}(\text{aq})$ | SO_2 liberated with dilute acids; 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 |
| sulfur dioxide, SO_2 | turns acidified aqueous potassium dichromate(VI) from orange to green |

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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
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CHEMISTRY

9701/34

Advanced Practical Skills 2

October/November 2013

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 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.
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.

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 **11** printed pages and **1** blank page.



- 1 **FB 1** is $0.125 \text{ mol dm}^{-3}$ hydrochloric acid, HCl .
FB 2 is an aqueous solution containing sodium hydroxide, NaOH , and sodium carbonate, Na_2CO_3 .
 bromophenol blue acid-base indicator

By carrying out titrations, you are to determine the percentage by mass of sodium carbonate in the mixture of sodium hydroxide and sodium carbonate in solution **FB 2**.

(a) Titration

- Fill a burette with **FB 1**.
- Pipette 25.0 cm^3 of **FB 2** into a conical flask.
- Add a few drops of bromophenol blue indicator.
- Titrate the mixture in the flask with **FB 1** until the blue-violet colour of the solution changes to yellow.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain 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 **FB 1** added in each accurate titration.

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

[7]

- (b)** From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

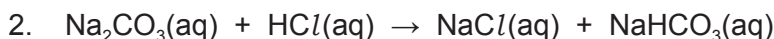
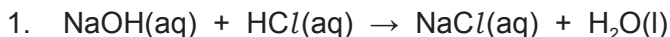
25.0 cm^3 of **FB 2** required cm^3 of **FB 1** [1]

(c) Calculations

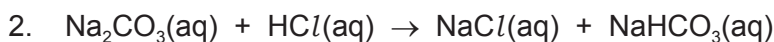
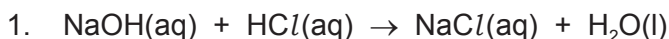
When the titrations were repeated using phenolphthalein as the indicator, 25.0 cm³ of **FB 2** required 23.25 cm³ of **FB 1**.

The following explains why different results are obtained using two different indicators.

- When **phenolphthalein** is used as the indicator, the following reactions have taken place at the end-point of the titration.



- When **bromophenol blue** is used as the indicator in **(a)**, the following reactions have taken place at the end-point of the titration.



Show your working and use appropriate significant figures in the final answer to all steps of your calculations.

- (i)** Calculate the number of moles of hydrochloric acid in the volume of **FB 1** calculated in **(b)**.

moles of HCl in volume in **(b)** = mol

- (ii)** Calculate the number of moles of hydrochloric acid in 23.25 cm³ of **FB 1**.

moles of HCl in 23.25 cm³ = mol

- (iii)** Use the following formula to calculate the number of moles of hydrochloric acid that react with the Na₂CO₃ in the titration using phenolphthalein indicator.

moles HCl = answer **(i)** – answer **(ii)** = mol

- (iv) Use your answer to (iii) to calculate the mass of sodium carbonate present in 25.0 cm³ of **FB 2**.
[A_r : C, 12.0; O, 16.0; Na, 23.0]

mass of Na₂CO₃ in 25.0 cm³ **FB 2** = g

- (v) The **overall** equation for the reaction of Na₂CO₃ with HCl when bromophenol blue is used as indicator is given below.



Calculate the number of moles of HCl that reacted with the Na₂CO₃ in the above equation in 25.0 cm³ of **FB 2**.

moles of HCl = mol

- (vi) Use your answers to (i) and (v) to calculate the mass of sodium hydroxide in 25.0 cm³ of **FB 2**.
[A_r : H, 1.0; O, 16.0; Na, 23.0]

mass of NaOH = g

- (vii) Calculate the percentage by mass of sodium carbonate in the mixture of sodium hydroxide and sodium carbonate in **FB 2**.

FB 2 contains % by mass Na₂CO₃
[6]

[Total: 14]

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

- 2 The percentage by mass of sodium carbonate in a mixture with sodium chloride can be estimated by adding a weighed sample of the mixture to a weighed **excess** of hydrochloric acid and measuring the mass of carbon dioxide evolved.
Sodium chloride does not react with hydrochloric acid.

FB 3 is 2.00 mol dm^{-3} hydrochloric acid, HCl .

FB 4 is a mixture of solid sodium carbonate, Na_2CO_3 , and solid sodium chloride, NaCl .

You are to determine the mass of carbon dioxide given off when the sodium carbonate in the mixture, **FB 4**, reacts with excess hydrochloric acid.

(a) Method

Record **all** weighings, in an appropriate form, in the space below.

- Use the measuring cylinder to transfer 75 cm^3 of **FB 3** into a 250 cm^3 conical flask.
- Weigh the flask and acid, and record the mass.
- Weigh the labelled tube containing **FB 4** and record the mass.
- Tip the **FB 4** into the acid in the flask, **a little at a time**.
- When the reaction slows down, swirl the flask for 2 to 3 minutes. Reweigh the flask and its contents, and record the mass.
- Reweigh the tube labelled **FB 4** with its stopper and any residual mixture not added to the acid, and record the mass.
- Calculate the mass of the mixture, **FB 4**, added to the acid.
- Record the mass of carbon dioxide given off in the reaction. This may be calculated using the following formula.

mass of $\text{CO}_2 = (\text{mass of flask} + \text{acid}) + (\text{mass } \text{FB 4} \text{ added}) - (\text{final mass of flask} + \text{contents})$

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

[6]

- (b) The reaction of sodium carbonate with hydrochloric acid is shown in the equation.



- (i) Calculate the mass of sodium carbonate that reacts with the hydrochloric acid to give the mass of carbon dioxide recorded in (a).
[A_r : C, 12.0; O, 16.0; Na, 23.0]

mass of Na_2CO_3 = g

- (ii) Calculate the percentage by mass of sodium carbonate in **FB 4**.

FB 4 contains % by mass Na_2CO_3
[2]

- (c) Mixtures of solids containing sodium carbonate can be analysed either by the procedure you used in **Question 1** or the procedure you used in **Question 2**.

The procedure used in **Question 2** is likely to give a less accurate value for the percentage of sodium carbonate.

- (i) Suggest a significant source of error in the experimental method used in **Question 2**.

.....
.....

- (ii) State whether the error identified above would increase or decrease the calculated percentage by mass of Na_2CO_3 in the mixture. Explain your answer.

.....
.....
.....

- (iii) Suggest an improvement to the experimental method or apparatus used in **Question 2** that would reduce the error given in (i).

.....
.....

[3]

[Total: 11]

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 name or correct formula of the element or compound must be given.

(a) You are provided with two solids in boiling tubes labelled **FB 5** and **FB 6**. Each solid contains one cation and one anion from those listed on pages 10 and 11.

- (i)** Add dilute nitric acid slowly to each boiling tube until the tube is approximately one third full. Record your observations in the space below. Keep these solutions for use in **(a)(ii)**.

(ii) Use the solutions from (i) in the following tests.

| test | observations | |
|---|---------------------------|---------------------------|
| | solution from FB 5 | solution from FB 6 |
| To a 1 cm depth of solution in a test-tube add aqueous sodium hydroxide, then | | |
| add excess aqueous sodium hydroxide. | | |
| To a 1 cm depth of solution in a test-tube add aqueous ammonia, then | | |
| add excess aqueous ammonia. | | |
| To a 1 cm depth of solution in a test-tube, add aqueous potassium iodide. | | |

(iii) From your observations, identify the cations present.

FB 5 contains **FB 6** contains

(iv) From your observations, what conclusions can be made about the anions present in **FB 5** and **FB 6**? Explain your reasoning.

.....

.....

.....

.....

[9]

- (b) (i) Dissolve half of the solid **FB 7** provided in a 4 cm depth of water in a boiling tube. Carry out the following tests and complete the table.

| <i>test</i> | <i>observations</i> | deductions about FB 7 |
|--|---------------------|------------------------------|
| To a 2 cm depth of the solution of FB 7 in a test-tube, add a 2 cm length of magnesium ribbon. | | |
| To a 2 cm depth of the solution of FB 7 in a boiling tube , add a 1 cm depth of dilute sulfuric acid. Warm the solution and add five drops of aqueous potassium manganate(VII). | | |

- (ii) Tip the remaining solid **FB 7** into a hard-glass test-tube. Heat the solid strongly and observe any changes. Do **not** test any gases given off. Record your observations in the space below.

- (iii) Suggest a further deduction you can make about **FB 7** from your observations in (ii).

.....

.....

[6]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

| ion | reaction with | |
|--|--|--|
| | 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) | no ppt. (if 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 giving dark green solution | 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 |
| lead(II), Pb ²⁺ (aq) | white ppt. soluble in excess | white 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 |

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

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|--|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$ | yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| 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 NO \rightarrow (pale) brown NO_2 in air) |
| sulfate, $\text{SO}_4^{2-}(\text{aq})$ | gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\text{SO}_3^{2-}(\text{aq})$ | SO_2 liberated with dilute acids; 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 |
| sulfur dioxide, SO_2 | turns acidified aqueous potassium dichromate(VI) from orange to green |

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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
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CHEMISTRY

9701/35

Advanced Practical Skills 1

October/November 2013

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 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.
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.

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 |
| |

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|--------------------|--|
| 1 | |
| 2 | |
| 3 | |
| Total | |

This document consists of **11** printed pages and **1** blank page.



- 1 You are provided with a solution of an organic acid which is known to be one of the following.

methanoic acid, HCOOH
 propanoic acid, $\text{C}_2\text{H}_5\text{COOH}$
 pent-2-enoic acid, $\text{CH}_3\text{CH}_2\text{CH}=\text{CHCOOH}$

The solution was made by dissolving 1.85 g of acid in 250 cm^3 of solution.

You are to suggest the identity of the acid by finding its relative molecular mass, M_r , using a titration method.

FA 1 is the solution of the unknown organic acid.

FA 2 is 0.100 mol dm^{-3} sodium hydroxide, NaOH .

phenolphthalein indicator

(a) Method

- Fill the burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Titrate **FA 1** with **FA 2** using phenolphthalein as indicator.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain 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 2** added in each accurate titration.

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

[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 1** required cm³ of **FA 2** [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 hydroxide in the volume of **FA 2** you have calculated in (b).

moles of NaOH = mol

- (ii) One mole of any of the organic acids reacts with one mole of sodium hydroxide. Calculate the concentration, in mol dm⁻³, of the acid in **FA 1**.

concentration of the acid in **FA 1** = mol dm⁻³

- (iii) Calculate the concentration, in g dm⁻³, of the acid used to make solution **FA 1**.

concentration of the acid in **FA 1** = g dm⁻³

- (iv) Using your answers to (ii) and (iii), calculate the relative molecular mass, M_r , of the acid in **FA 1**.

M_r of the acid =

- (v) Suggest which of the acids, methanoic, propanoic or pent-2-enoic acid, is present in **FA 1**.

Acid present is

- (vi) Suggest a test that could be carried out to distinguish pent-2-enoic acid from methanoic acid and propanoic acid.
Give the expected result of your test.

test

expected result

[5]

[Total: 13]

- 2 The formula of hydrated iron(II) sulfate is $\text{FeSO}_4 \cdot x\text{H}_2\text{O}$ where **x** shows the number of moles of water of crystallisation.

For
Examiner's
Use

The value of **x** can be found by heating solid hydrated iron(II) sulfate to remove the water of crystallisation.

FA 3 is hydrated iron(II) sulfate, $\text{FeSO}_4 \cdot x\text{H}_2\text{O}$.

(a) Method

Record **all** weighings, in an appropriate form, in the space below.

- Weigh and record the mass of the empty crucible.
- Tip the contents of the tube labelled **FA 3** into the weighed crucible. Reweigh and record the mass of the crucible and **FA 3**.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat **gently** for about three minutes.
- Leave the crucible to cool for approximately five minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When cool, reweigh the crucible with the residue.
- Reheat **gently** for three minutes, cool and reweigh the crucible until you are satisfied that all the water of crystallisation has been lost. It should not be necessary to reheat the crucible more than three times.

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

[6]

- (b) (i) Calculate the mass of water lost and the mass of anhydrous iron(II) sulfate that remains after the heating process.

mass of water lost = g

mass of anhydrous iron(II) sulfate = g

- (ii) Determine the value of x in the formula of hydrated iron(II) sulfate, $\text{FeSO}_4 \cdot x\text{H}_2\text{O}$.
(A_r : H, 1.0; O, 16.0; S, 32.1; Fe, 55.8)

value of x =

[3]

- (c) A group of students carried out this practical and made their measurements correctly. The students calculated a value of 9 for x . The textbook value of x is less than 9.

- (i) Suggest an error in the practical procedure of the experiment that could account for this result and explain why this gives a value of x that is too high.

.....
.....
.....
.....

- (ii) Suggest a modification that could be made to the experimental procedure to reduce this error. Explain why this modification should give an answer for x that is closer to the textbook value.

.....
.....
.....

[3]

[Total: 12]

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 name or correct formula of the element or compound must be given.

(a) You are provided with a solid **FA 4**. **FA 4** is a mixture that contains two cations and two anions.

- (i) Place a spatula measure of **FA 4** in a **hard-glass** test-tube. Heat the solid and identify the gas given off. Record all your observations.

.....

.....

.....

- (ii) To a spatula measure of **FA 4** in a test-tube, add a 1 cm depth of dilute nitric acid. Record your observations.

.....

.....

- (iii) To a spatula measure of **FA 4** in a test-tube, add approximately a 2 cm depth of distilled water to make a solution. Divide the solution into two portions.

To the first portion, add a 1 cm depth of aqueous sodium hydroxide. Record your observations.

.....

To the second portion, add a few drops of aqueous silver nitrate, then add a 1 cm depth of dilute nitric acid. Record your observations.

.....

- (iv) Use your results from (i) to (iii) to identify two anions and one cation that are present in **FA 4**.

anions present and

cation present

- (v) What further test could be carried out on **FA 4** to confirm the presence of the cation you suggested in (iv)? You should state the reagent to be used and the expected result.

Do not carry out this test.

.....

- (vi) To a spatula measure of **FA 4** in a test-tube, add a 1 cm depth of distilled water to make a solution. To this solution, add a few drops of aqueous barium chloride or barium nitrate.

Describe the appearance of the precipitate formed and state its identity.

appearance of precipitate

identity of precipitate

[9]

(b) **FA 5**, **FA 6**, **FA 7** and **FA 8** are aqueous solutions each containing one of the ions Al^{3+} , Ca^{2+} , Zn^{2+} and Pb^{2+} .

For
Examiner's
Use

(i) Carry out the following tests. Record your observations in the spaces provided in the table.

| test | observations | | | |
|---|--------------|------|------|------|
| | FA 5 | FA 6 | FA 7 | FA 8 |
| To a 1 cm depth of solution in a test-tube, add a few drops of aqueous sodium hydroxide, then | | | | |
| add excess aqueous sodium hydroxide. | | | | |
| To a 1 cm depth of solution in a test-tube, add a few drops of aqueous ammonia, then | | | | |
| add excess aqueous ammonia. | | | | |
| To a 1 cm depth of solution in a test-tube, add a 1 cm depth of aqueous potassium iodide. | | | | |

(ii) Use the Qualitative Analysis Notes on page **10** to identify the cation present in each of the solutions.

FA 5 is , **FA 6** is , **FA 7** is , **FA 8** is

[6]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

| ion | reaction with | |
|--|--|--|
| | 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) | no ppt. (if 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 giving dark green solution | 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 |
| lead(II), Pb ²⁺ (aq) | white ppt. soluble in excess | white 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 |

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

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|--|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$ | yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| 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 $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\text{SO}_3^{2-}(\text{aq})$ | SO_2 liberated with dilute acids; 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 |
| sulfur dioxide, SO_2 | turns acidified aqueous potassium dichromate(VI) from orange to green |

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General Certificate of Education
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CHEMISTRY

9701/36

Advanced Practical Skills 2

October/November 2013

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 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.
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 11 and 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 are to determine the concentration of aqueous potassium manganate(VII), **FB 3**, by titration.

In the titration potassium manganate(VII) is first reacted with acidified potassium iodide to produce iodine. The amount of iodine formed is then determined by titrating the mixture with sodium thiosulfate.

FB 1 is hydrated sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

FB 2 is dilute sulfuric acid, H_2SO_4 .

FB 3 is aqueous potassium manganate(VII), KMnO_4 .

FB 4 is aqueous potassium iodide, KI .
starch indicator

(a) Method

Preparing a solution of **FB 1**

- Weigh the 250 cm³ beaker and record the mass in the space below.
- Add all the **FB 1** to the beaker. Weigh the beaker with **FB 1** and record the mass.
- Calculate the mass of **FB 1** used and record this in the space below.
- Add approximately 100 cm³ of distilled water to the beaker. Stir until all the solid has dissolved.
- Transfer the solution into the 250 cm³ volumetric (graduated) flask labelled **FB 5**.
- Wash out the beaker thoroughly using distilled water and add the washings to the volumetric flask. Make the solution up to the mark using distilled water.
- Shake the flask thoroughly to mix the solution before using it for your titrations.
- This solution of sodium thiosulfate is **FB 5**.

Titration

- Use the measuring cylinder to add 20 cm³ of **FB 2** to a conical flask.
- Use the measuring cylinder to add 10 cm³ of **FB 4** to the same flask.
- Pipette 25.0 cm³ of **FB 3** into the same flask.
The colour of the mixture is caused by iodine.
- Fill the burette with **FB 5**.
- Begin each titration **without** adding the starch indicator.
Add 10 drops of starch indicator when the colour of the mixture becomes (pale) yellow.
The end-point is when the blue-black colour caused by the starch disappears.
- Perform a **rough** titration and record your burette readings 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 **FB 5** added in each accurate titration.

For
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Use

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

[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

suitable value = cm³ of **FB 5** [1]

(c) **Calculations**

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

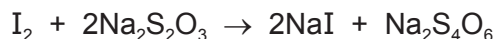
- (i) Calculate the number of moles of sodium thiosulfate, **FB 1**, that were weighed out. The relative formula mass of hydrated sodium thiosulfate is 248.2.

moles of sodium thiosulfate = mol

- (ii) Calculate the number of moles of sodium thiosulfate that were present in the volume of **FB 5** calculated in (b).

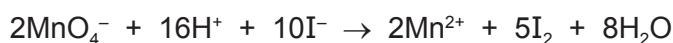
moles of sodium thiosulfate = mol

- (iii) Iodine produced by the reaction in the conical flask reacts with sodium thiosulfate. Use the equation below to calculate the number of moles of **iodine** that reacted with sodium thiosulfate in (ii).



moles of I_2 = mol

- (iv) The iodine is produced as a result of the oxidation of iodide ions in potassium iodide, **FB 4**, by potassium manganate(VII), **FB 3**. The ionic equation for this reaction is



Calculate the number of moles of potassium manganate(VII), KMnO_4 , that reacted to produce the iodine in (iii).

moles of KMnO_4 = mol

- (v) Calculate the concentration of potassium manganate(VII), in g dm^{-3} , in **FB 3**. (A_r : O, 16.0; K, 39.1; Mn, 54.9)

concentration of KMnO_4 = g dm^{-3}
[5]

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

- (d) (i) State the maximum error in any single reading of the burette.

maximum error = cm^3

- (ii) Calculate the maximum percentage error in volume of **FB 5** in your **first** accurate titre.

maximum percentage error = %
[1]

[Total: 14]

- 2 In this experiment you will heat two separate samples of a hydrated salt to drive off the water of crystallisation.
You will then calculate the relative atomic mass of the metal in the salt.

FB 6 is the hydrated salt.

The formula of **FB 6** is $\text{MSO}_4 \cdot 7\text{H}_2\text{O}$, where **M** is the metal.

(a) Method

Record **all** weighings, in an appropriate form, in the space below.

- Record the mass of the empty crucible **without** its lid.
- Add between 2.0 and 2.4 g of **FB 6** into the crucible. Record the mass of the crucible and its contents.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat the crucible and its contents gently and carefully for about **two** minutes, with the lid off. Then heat very strongly for a further **three** minutes.
- Put the lid on the crucible and leave it to cool for approximately 10 minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When the crucible is cool, **remove the lid**, and weigh the crucible with the residue.
- Record the mass of anhydrous MSO_4 remaining in the crucible after heating and therefore calculate the mass of water lost.
- To prepare for the second experiment, use a spatula to remove the solid residue from the crucible into the beaker labelled **waste**.
- Reweigh the empty crucible **without** its lid.
- Carry out the experiment again. This time use between 2.5 and 2.9 g of **FB 6**.

For
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Use

[6]

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

(b) Calculation

Show your working and give your answers to **three** significant figures.

- (i) State **and** explain which of your two experiments in **(a)** is likely to be the more accurate.

.....

.....

.....

- (ii) Calculate the number of moles of water removed from the hydrated salt in the **more accurate** experiment.
(A_r : H, 1.0; O, 16.0)

moles of H_2O = mol

- (iii) Complete the equation for the removal of water from hydrated **FB 6**.
Include state symbols.



- (iv) Using your answer to **(ii)**, calculate the number of moles of anhydrous MSO_4 produced in the **more accurate** experiment.

moles of MSO_4 = mol

- (v) Use the mass of anhydrous MSO_4 produced in the **more accurate** experiment to calculate the relative formula mass of MSO_4 .

relative formula mass of MSO_4 =

- (vi) Calculate the relative atomic mass of **M**.
(A_r : O, 16.0; S, 32.1)

(If you were unable to calculate the relative formula mass of anhydrous **MSO**₄ you may assume that it was 126.3. This is not the correct value.)

A_r of **M** =

- (vii) The relative atomic masses of some of the cations on page 11 are given below.
(A_r : Mg, 24.3; Ca, 40.0; Fe, 55.8; Cu, 63.5; Mn, 54.9; Zn, 65.4)

M is a cation of one of the elements listed above.
Suggest the identity of **M** and justify your answer.

.....
.....

- (viii) Suggest why it was **not** necessary to include the cations aluminium and chromium from page 11 in the list of relative atomic masses in (vii).

.....
.....
.....

[8]

- (c) The crucible was cooled with the lid on to prevent absorption of water vapour from the air. Suggest a better way of preventing water vapour being absorbed during cooling.

.....
.....

[1]

[Total: 15]

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) You will carry out further tests on the ions in **FB 6**.

- Put a spatula measure of **FB 6** into a test-tube.
- Half fill the test-tube with distilled water and stir until the solid dissolves.
- Use a 1 cm depth of the solution of **FB 6** in separate test-tubes for the tests you will carry out.

- (i) Add aqueous sodium hydroxide to **FB 6** solution.
Add aqueous ammonia to **FB 6** solution.

Record your observations below.

- (ii) Carry out a test of your choice to show that sulfate ions are present in **FB 6**.

reagent(s) used

observation(s)

.....

- (iii) Give the **ionic** equation for the reaction in test (ii).

.....

[4]

(b) **FB 7**, **FB 8** and **FB 9** are aqueous solutions, each containing one cation and one anion. None of the cations and none of the anions in **FB 7**, **FB 8** and **FB 9** are identical.

- (i) Add a 2 cm magnesium strip to a 2 cm depth of each solution in a clean test-tube. Mix pairs of solutions as shown so that you can complete the table shown below. Use 1 cm depths of solutions in clean test-tubes. Record your observations in the table.

| | FB 7 | FB 8 | FB 9 |
|--------------------------------------|-------------|-------------|---|
| add a 2 cm strip of magnesium ribbon | | | |
| FB 7 | X | | [Keep this mixture for use in test (iii)] |
| FB 8 | X | X | |

- (ii) The anion present in **FB 7** is the sulfate ion. Identify **FB 7**, giving evidence from your observations.

FB 7 is

evidence

.....

- (iii) Add a 1 cm depth of aqueous hydrogen peroxide, **FB 10**, to the mixture of **FB 7** and **FB 9** that you kept from (i). Then add three drops of starch.

Record your observation(s).

Identify the coloured chemical produced when hydrogen peroxide was added to the mixture of **FB 7** and **FB 9** and name the anion present in **FB 9**.

observations

.....

chemical produced

anion in **FB 9**

- (iv) Give the chemical formula of the substance you observed when solutions **FB 8** and **FB 9** were mixed.

.....

[7]

[Total: 11]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

| ion | reaction with | |
|--|--|--|
| | 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) | no ppt. (if 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 giving dark green solution | 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 |
| lead(II), Pb ²⁺ (aq) | white ppt. soluble in excess | white 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 |

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

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|---|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$ | yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$ |
| 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 $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids) |
| sulfite, $\text{SO}_3^{2-}(\text{aq})$ | SO_2 liberated with dilute acids; 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 |
| sulfur dioxide, SO_2 | turns acidified aqueous potassium dichromate(VI) from orange to green |

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