

CANDIDATE
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CHEMISTRY (US)

9185/35

Paper 3 Advanced Practical Skills 1

May/June 2014

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Center 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.

Qualitative Analysis Notes are printed on pages 11 and 12.

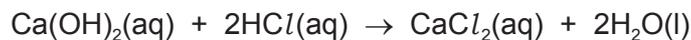
At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

| | |
|-------------------|--|
| Session | |
| | |
| Laboratory | |
| | |

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

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

- 1 Limewater is a saturated solution of calcium hydroxide. A sample of limewater was used to produce solution **FA 1**. You are to determine the concentration of calcium hydroxide, **FA 1** by titration with hydrochloric acid, HCl , which you will first dilute. The equation for the reaction between calcium hydroxide and hydrochloric acid is given below.



FA 1 is aqueous calcium hydroxide, Ca(OH)_2 .

FA 2 is $0.500 \text{ mol dm}^{-3}$ hydrochloric acid, HCl .

methyl orange indicator

(a) Method

Read through the method before starting any practical work.

(i) Dilution

- Fill the buret with **FA 2**.
- Run between 24.00 and 26.00 cm^3 of **FA 2** into the 250 cm^3 volumetric flask.
- Record your buret readings and the volume used in the space below.
- Make up the solution to 250 cm^3 with distilled water and shake the flask to ensure thorough mixing. This solution is **FA 3**.

volume of $0.500 \text{ mol dm}^{-3}$ HCl used = cm^3

(ii) Titration

- Empty and rinse the buret.
- Fill the buret with **FA 3**.
- Pipet 25.0 cm^3 of **FA 1** into an Erlenmeyer flask.
- Add a few drops of methyl orange indicator.
- Perform a **rough titration** and record your buret readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain accurate results.
- Make certain any recorded results show the precision of your practical work.
- Record, in a suitable form below, all of your buret readings and the volume of added in each accurate titration.

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

[8]

- (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 1** required cm³ of **FA 3**. [1]

(c) Calculations

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

- (i) Use your volume of **FA 2** from **(a)(i)** to calculate the concentration of **FA 3**.

concentration of **FA 3** = mol dm⁻³

- (ii) Use your answer to **(c)(i)** and the value calculated in **(b)** to calculate the number of moles of hydrochloric acid used to neutralize 25.0 cm³ of **FA 1**.

moles of HCl = mol

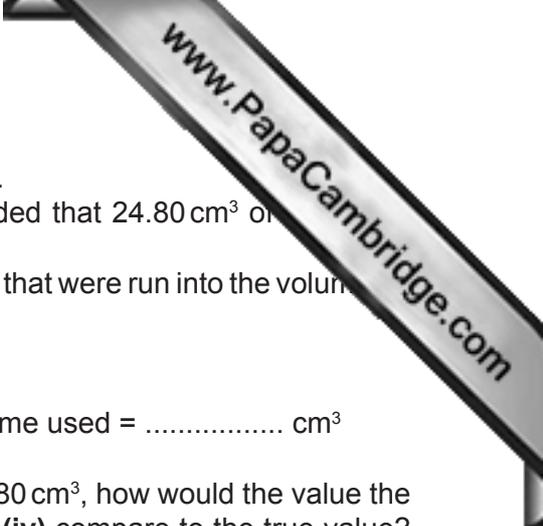
- (iii) Use your answer to **(c)(ii)** to calculate the number of moles of calcium hydroxide in 25.0 cm³ of **FA 1**.

moles of Ca(OH)₂ = mol

- (iv) Use your answer to **(c)(iii)** to calculate the concentration, in mol dm⁻³, of calcium hydroxide in **FA 1**.

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

concentration of Ca(OH)₂ = mol dm⁻³
[5]



- (d) (i) The maximum error in a single buret reading is $\pm 0.05 \text{ cm}^3$.
When making up the diluted acid, **FA 3**, a student recorded that 24.80 cm^3 of acid was used.
What are the smallest and largest possible volumes of acid that were run into the volumetric flask?

smallest volume used = cm^3 largest volume used = cm^3

- (ii) If the actual volume of acid added had been less than 24.80 cm^3 , how would the value the student calculated for the concentration of Ca(OH)_2 in **(c)(iv)** compare to the true value? Explain your answer.

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

[2]

- (e) If a sample of limewater is left open to the air, the concentration of calcium hydroxide decreases. Explain why.

.....
..... [1]

[Total: 17]

- 2 You are to determine the percentage by mass of zinc carbonate in a sample of pure zinc carbonate ore by means of thermal decomposition. You may assume that none of the components of the ore is affected by heating. The equation for the reaction occurring is given below.



FA 4 is zinc carbonate ore.

(a) Method

Read through the method **before** starting any practical work and prepare a table for your results in the space below.

- Weigh the empty crucible and record the mass in your table.
- Transfer **all** the **FA 4** into the crucible.
- Weigh the crucible with **FA 4** and record the mass.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible gently for about one minute and then strongly for four minutes.
- Remove the Bunsen burner and allow the crucible to cool.
- While the crucible is cooling start working on another question.
- Reweigh the cooled crucible with contents and record the mass.
- Record the mass of **FA 4** used and the mass of solid remaining after heating.
- Beneath your table, record any observations you have made while the solid was heated and cooled.

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

[4]

(b) Calculations

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

- (i) From your results in **(a)**, calculate the mass of carbon dioxide lost on heating **FA 4**.

mass of CO_2 lost = g

- (ii) Use your answer to **(i)** to calculate the mass of zinc carbonate present in the sample of **FA 4** that was heated.

[A_r : C, 12.0; O, 16.0; Zn, 65.4]

mass of ZnCO_3 = g

- (iii) Calculate the percentage by mass of zinc carbonate in the zinc carbonate ore.

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

percentage of ZnCO_3 = %
[4]

- (c) Suggest how a student, using all the same apparatus, could alter the method to be more confident that the percentage of zinc carbonate is correct. Explain your answer.

.....

.....

.....

..... [2]

[Total: 10]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- color 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 different sample of zinc carbonate ore, labelled **FA 5**. This sample contains zinc carbonate, ZnCO_3 , and one other salt. This additional salt contains a cation and a single anion from those listed on pages 11 and 12. By carrying out the tests you will be able to suggest the identities of the ions in the additional salt.

| <i>test</i> | <i>observations</i> |
|---|---------------------|
| <p>(i) Transfer the solid FA 5 into a boiling tube and add a 3 cm depth of dilute nitric acid to make solution FA 6.</p> <p>Use this solution in tests (ii) to (v).</p> | |
| <p>(ii) To a 1 cm depth of FA 6 in a test-tube, add aqueous sodium hydroxide.</p> | |
| <p>(iii) To a 1 cm depth of FA 6 in a test-tube, add aqueous ammonia.</p> | |
| <p>(iv) To a 1 cm depth of FA 6 in a test-tube, add a 1 cm depth of aqueous silver nitrate.</p> | |
| <p>(v) To a 1 cm depth of FA 6 in a test-tube, add a 1 cm depth of aqueous barium chloride or barium nitrate.</p> | |

- (vi) Suggest the identity of the cation and anion (apart from Zn^{2+} and CO_3^{2-}) present in **FA 5**. Explain your choice.

cation

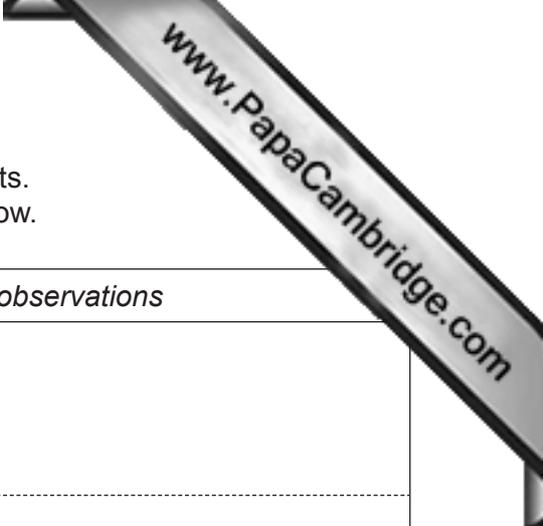
reason

anion

reason

[7]

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



- (b) You are provided with solid **FA 7**, which is a mixture of two salts. Use separate portions of **FA 7** to perform the experiments below.

| | <i>test</i> | <i>observations</i> | | | | | | | | | | | | |
|--|---|---------------------|----|--|-----|--|----|--|---|--|----|--|--|--|
| | (i) Heat a spatula measure of FA 7 in a hard-glass test-tube gently at first, then | | | | | | | | | | | | | |
| | heat more strongly and test the gas evolved with litmus paper. | | | | | | | | | | | | | |
| Dissolve a spatula measure of FA 7 in a test-tube with a 5 cm depth of distilled water for use in tests (ii) and (iii). | | | | | | | | | | | | | | |
| | (ii) To a 1 cm depth of FA 7 (aq) in a test-tube, add aqueous silver nitrate, | | | | | | | | | | | | | |
| | followed by aqueous ammonia. | | | | | | | | | | | | | |
| <table border="1" style="display: inline-table; vertical-align: top;"> <tr><td>I</td><td></td></tr> <tr><td>II</td><td></td></tr> <tr><td>III</td><td></td></tr> <tr><td>IV</td><td></td></tr> <tr><td>V</td><td></td></tr> <tr><td>VI</td><td></td></tr> </table> | I | | II | | III | | IV | | V | | VI | | (iii) To a 1 cm depth of FA 7 (aq) add barium chloride or barium nitrate, | |
| | I | | | | | | | | | | | | | |
| | II | | | | | | | | | | | | | |
| | III | | | | | | | | | | | | | |
| IV | | | | | | | | | | | | | | |
| V | | | | | | | | | | | | | | |
| VI | | | | | | | | | | | | | | |
| | followed by dilute nitric acid. | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |

- (iv) Use your observations and the Qualitative Analysis Notes from pages 11 and 12 to identify **three** of the ions present. Give evidence for your choice of ions.

ion evidence

.....

ion evidence

.....

ion evidence

.....

[6]

[Total: 13]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

| ion | reaction with | |
|--|--|--|
| | NaOH(aq) | NH ₃ (aq) |
| aluminum, 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) | gray-green ppt. soluble in excess giving dark green solution | gray-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 |
| 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 |

2 Reactions of anions

| <i>ion</i> | <i>reaction</i> |
|---|--|
| carbonate, CO_3^{2-} | CO_2 liberated by dilute acids |
| chloride, $\text{Cl}^-(\text{aq})$ | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); |
| bromide, $\text{Br}^-(\text{aq})$ | gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); |
| iodide, $\text{I}^-(\text{aq})$ | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); |
| 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 (colorless $\text{NO} \rightarrow$ (pale) brown NO_2 in air) |
| sulfate, $\text{SO}_4^{2-}(\text{aq})$ | gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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 manganate(VII) from purple to colorless |

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