



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

CANDIDATE
NAME

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CENTRE
NUMBER

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CHEMISTRY

9701/36

Advanced Practical Skills 2

October/November 2012

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.
 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 15 and 16.

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 **13** printed pages and **3** blank pages.

- 1 You are to determine the concentration of aqueous copper(II) sulfate by titration. The concentration of Cu^{2+} ions in a solution can be found by reaction with an excess of aqueous iodide ions to produce iodine. The amount of iodine formed can be found by titration with thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$.

FB 1 is aqueous copper(II) sulfate, CuSO_4 .

FB 2 is $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FB 3 is aqueous potassium iodide, KI.

starch indicator

Read through the instructions carefully before starting any practical work.

(a) Method

- Fill the burette with **FB 2**.
- Pipette 25.0 cm^3 of **FB 1** into a conical flask.
- Use a measuring cylinder to add 10 cm^3 of **FB 3** into the conical flask.
- Titrate this mixture with **FB 2** until the colour of the mixture changes from brown to yellow-brown. An off-white precipitate will also be present in the flask throughout the titration.
- Add approximately 1 cm^3 of starch indicator.
- Continue the titration until the blue-black colour of the starch-iodine complex just disappears leaving the off-white precipitate.
- 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 2** added in each accurate titration.

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

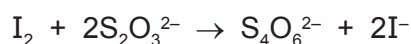
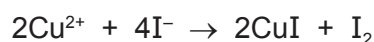
[6]

- (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 **FB 1** required cm³ of **FB 2** [1]

(c) Calculations

The equations for the formation of iodine and its reaction with thiosulfate ions are given below.



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

- (i) Calculate the number of moles of thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, present in the volume of **FB 2** in (b).

moles of $\text{S}_2\text{O}_3^{2-}$ = mol

- (ii) Using the equations above, deduce the number of moles of Cu^{2+} ions present in each 25.0 cm³.

moles of Cu^{2+} = mol

- (iii) Calculate the concentration, in mol dm⁻³, of copper(II) sulfate in **FB 1**.

concentration of CuSO_4 = mol dm⁻³
[3]

- (d) Three students repeated the experiment but each obtained different values for the concentration of CuSO_4 .

The students each suggested possible improvements.

Student 1 suggested that a larger quantity of starch should be added.

Student 2 suggested that a larger volume of potassium iodide, **FB 3**, should be added.

Student 3 suggested that the contents of the conical flask should be filtered before titration.

Comment on the effectiveness of **each** of these possible improvements. Explain your answers.

Student 1

.....
.....

Student 2

.....
.....

Student 3

.....
..... [2]

[Total: 12]

- 2 You are to determine the enthalpy change for the reaction between aqueous copper(II) sulfate and zinc. The enthalpy change of reaction can be found by measuring the temperature change when powdered zinc is added to aqueous copper(II) sulfate.

FB 4 is 1.10 mol dm⁻³ aqueous copper(II) sulfate, CuSO₄.
powdered zinc

(a) Method

- Weigh a 100 cm³ beaker.
- In the beaker weigh out between 2.1 g and 2.3 g of powdered zinc.
- Record the weighings and the mass of zinc in the space below.

mass of zinc used = g

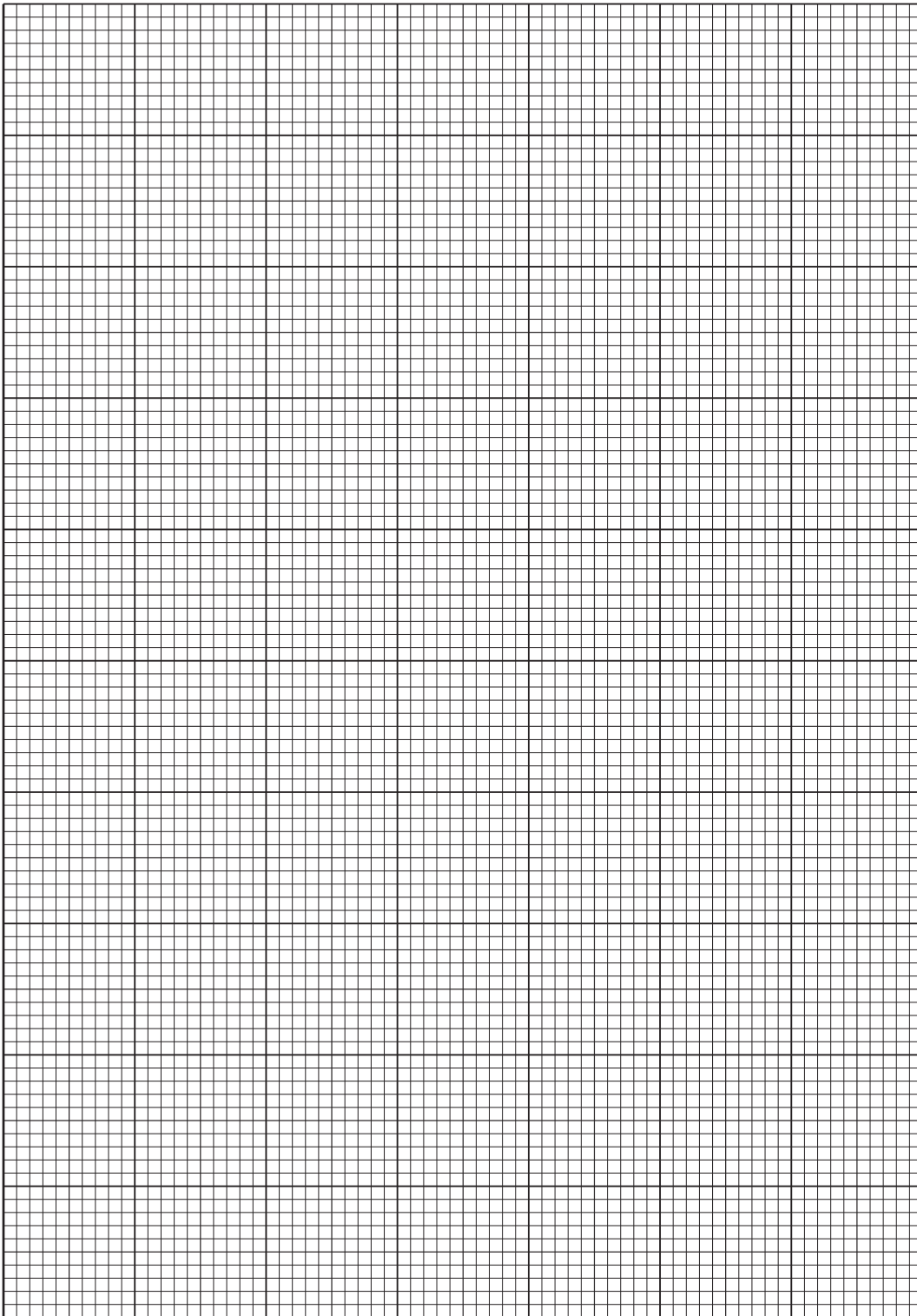
- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FB 4** into the plastic cup.
- Measure and record in the table below, the initial temperature of **FB 4** in the cup.
- Start the stop watch. Measure and record the temperature of **FB 4** in the cup after 1 minute, 2 minutes and 3 minutes.
- At time 3½ minutes, add the weighed zinc to **FB 4** in the cup and stir the mixture.
- From time 4 minutes, continue to stir the mixture and measure the temperature of the contents of the cup to complete the table.

Results

| | | | | | | | | | | | | | |
|------------------|---|---|---|---|---|---|---|---|---|---|----|----|----|
| time / min | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| temperature / °C | | | | | | | | | | | | | |

[2]

- (b) (i)** On the axes opposite, plot the temperature (*y*-axis) against time (*x*-axis). The temperature axis should allow you to include a point at least 5 °C greater than the maximum temperature recorded.



(ii) Complete the graph to show how the temperature of the contents of the cup varies with time.

- Draw one straight line through the points between time 0 minutes and 3 minutes.
- Draw one straight line through the points after the maximum was reached.
- Extrapolate these two lines and draw a vertical line at time $3\frac{1}{2}$ minutes.

[4]

(c) Calculation

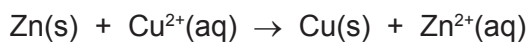
- (i) Use your graph to determine the change in temperature at 3½ minutes.

change in temperature = °C

- (ii) Calculate the heat energy produced in the reaction.
-
- (You may assume that 4.3 J are required to raise the temperature of 1.0 cm
- ³
- of any solution by 1.0 °C.)

heat energy produced = J

- (iii) The reaction between zinc and aqueous copper(II) sulfate is a displacement reaction shown in the equation below.



From the mass of zinc added and the concentration of **FB 4**, show that the copper(II) sulfate was in excess in your reaction.

[A_r: Zn, 65.4]

- (iv) Assuming that the copper(II) sulfate was in excess, use your answer to (ii) to calculate the enthalpy change of the reaction between Zn(s) and Cu
- ²⁺
- (aq).

Give your answer in kJ mol⁻¹ and include the relevant sign.

enthalpy change of reaction = kJ mol⁻¹
sign *value*

[6]

- (d) One source of error in this experiment is due to the accuracy to which the thermometer can be read.

What is the maximum error in a single temperature reading on a thermometer with graduations at 1 °C?

maximum error = °C

Calculate the maximum percentage error when measuring a temperature **rise** of 12.0 °C.

maximum percentage error = % [2]

[Total: 14]

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.

You are provided with solutions **FB 5**, **FB 6**, **FB 7** and **FB 8**.

FB 5 and **FB 6** each contain a compound of a transition element.

Half fill a 250 cm³ beaker with water. Heat to approximately 80 °C, then stop heating and switch off the Bunsen burner. You will need this as a hot water bath on **(b)(i)**. Continue work on **(a)** while the water heats.

(a) (i) Carry out the following tests on **FB 5**.

| <i>test</i> | <i>observations</i> |
|--|---------------------|
| To 1 cm depth of FB 5 in a test-tube, add aqueous ammonia. | |
| To 1 cm depth of FB 5 in a test-tube, add aqueous sodium hydroxide. | |
| To 1 cm depth of FB 5 in a test-tube, add aqueous barium chloride or aqueous barium nitrate then, | |
| add an excess of either hydrochloric acid or nitric acid. | |

- (ii) From these tests, what conclusions, if any, can you reach about the identity of **FB 5**?

..... [4]

- (b) (i) Carry out the following tests on **FB 6**.

| <i>test</i> | <i>observations</i> |
|---|---------------------|
| To 1 cm depth of FB 6 in a boiling tube, add 1 cm depth of FB 7 then, | |
| add 1 cm depth of ethanol. Place the boiling tube in the warm water bath and leave for a few minutes. | |
| To 1 cm depth of FB 6 in a test-tube, add 1 cm depth of FB 8 . | |

- (ii) From these tests suggest identities for the following.

The anion in **FB 6** is

The cation in **FB 7** is

The cation in **FB 8** could be **or**

- (iii) Suggest a test to determine which of the two possible cations is present in **FB 8**.
Do not carry out this test.

.....
..... [7]

- (c) Using your conclusions about the possible identities of **FB 5** and **FB 8**, predict the result of mixing solutions of each.
Do not carry out this test.

Prediction

..... [1]

- (d) Suggest what happened to the ethanol when it was warmed with the mixture of **FB 6** and **FB 7**.

..... [1]

- (e) You are to devise and carry out a test to confirm the identity of the cation in **FB 7**.

Record the test you use and the results of the test in the space below.

[1]

[Total: 14]

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 <i>Al</i> foil |
| nitrite, $\text{NO}_2^-(\text{aq})$ | NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and <i>Al</i> 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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