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

CANDIDATE NAME	
CENTRE NUMBER	
CANDIDATE NUMBER	

7351974356

CHEMISTRY **9701/31**

Advanced Practical Skills 1 **May/June 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 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	
Total	

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1 When iodide ions are mixed with peroxodisulfate ions, $S_2O_8^{2-}$, iodine is formed.

$$2I^-(aq) + S_2O_8^{2-}(aq) \rightarrow I_2(aq) + 2SO_4^{2-}(aq)$$

The rate of this reaction can be measured by adding thiosulfate ions, $S_2O_3^{2-}$, and some starch indicator to the mixture. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

When all the thiosulfate ions have reacted, the iodine which continues to be produced then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes for the reaction mixture to turn blue-black.

You are to investigate how the rate of reaction is affected by changing the concentration of the peroxodisulfate ions.

FA 1 is 0.0200 mol dm^{-3} aqueous potassium peroxodisulfate, $K_2S_2O_8$.
FA 2 is 1.00 mol dm^{-3} aqueous potassium iodide, KI.
FA 3 is 0.00500 mol dm^{-3} aqueous sodium thiosulfate, $Na_2S_2O_3$.
starch indicator

Read through the instructions carefully and prepare a table for your results on page 3 before starting any practical work.

(a) Method

Experiment 1

- Fill the burette labelled **FA 1** with aqueous potassium peroxodisulfate, **FA 1**.
- Run 20.00 cm^3 of **FA 1** into a 100 cm^3 beaker.
- Using a 25 cm^3 measuring cylinder add the following to a second 100 cm^3 beaker:
  - 20 cm^3 of **FA 2**
  - 10 cm^3 of **FA 3**
- Add 10 drops of starch indicator to the second beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

Experiment 2

- Fill a second burette with distilled water.
- Run 10.00 cm^3 of **FA 1** into the first 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into the beaker containing **FA 1**.
- Using a 25 cm^3 measuring cylinder add the following to the second 100 cm^3 beaker:
  - 20 cm^3 of **FA 2**
  - 10 cm^3 of **FA 3**
- Add 10 drops of starch indicator to the second beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

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Experiments 3 – 5

- Carry out three further experiments to investigate how the reaction time changes with different volumes of potassium peroxodisulfate.
  Remember that the combined volume of **FA 1** and distilled water must always be 20.00 cm^3.
  Do not use a volume of **FA 1** that is less than 6.00 cm^3.

Record all your results in a single table. You should include the volume of potassium peroxodisulfate, the volume of distilled water and the reaction time.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	

[9]

(b) In order to convert the times measured in the experiments into rates of reaction, it is necessary first to work out the concentration of $I_2(aq)$ that would have been produced in the reaction time if the thiosulfate had not been present. You must show your working.

(i) Calculate how many moles of thiosulfate ions, $S_2O_3^{2-}$, were added in each experiment.

moles of $S_2O_3^{2-}$ = ... mol

(ii) Calculate how many moles of iodine, I_2, must have been produced to react with this amount of thiosulfate ions.

moles of I_2 = ... mol

(iii) Calculate the concentration of iodine from **(ii)** in the total reaction volume.

concentration of I_2 = ... mol dm^{-3}

[3]

(c) The rate of the reaction can be represented by the following formula.

$$\text{'rate'} = \frac{\text{concentration of } I_2 \text{ from } \textbf{(b)(iii)}}{\text{reaction time}} \times 10^6$$

Use your experimental results to complete the following table. Include the volume of **FA 1**, the reaction time and 'rate' with their units.

If you were unable to answer **(b)(iii)**, you may assume that the concentration of iodine is 4.25×10^{-4} mol dm^{-3} (This is not the correct value).

[2]

(d) On the grid opposite, plot the 'rate' against the volume of **FA 1**. Draw a line of best fit.

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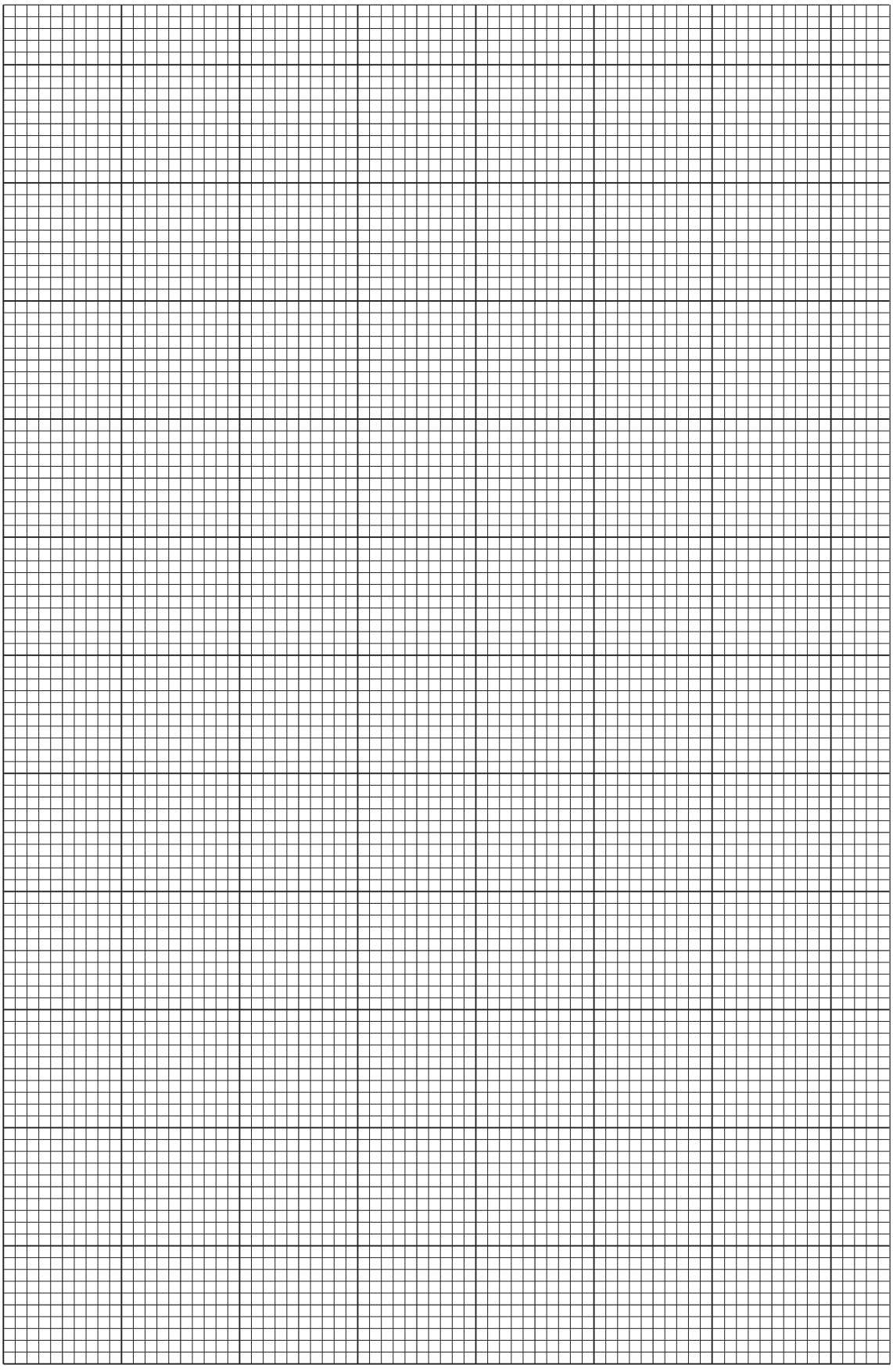

[4]

For Examiner's Use

I	
II	
III	
IV	

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(e) In your experiments, the volume of **FA 1** represents the concentration of potassium peroxodisulfate. From your results, what conclusion can you draw about the relationship between the rate of reaction and the concentration of potassium peroxodisulfate?

.....

.....

.....

..... [2]

(f) Assume that the error in the time measured for each reaction was ± 0.5 s.

(i) Calculate the maximum percentage error in the reaction time you recorded in **Experiment 1**.

maximum percentage error = ...%

(ii) Assuming this is the **only** source of error, calculate the minimum reaction rate for **Experiment 1**.

minimum reaction rate = ...

(iii) Suggest an additional source of error in these experiments and what improvement could be made to reduce this error.

.....

.....

.....

.....
[4]

I	
II	
III	
IV	

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(g) (i) Carry out one additional experiment using the following volumes of each reagent. Use the same method as in **(a)**, mix **FA 2**, **FA 3**, the distilled water and the starch together and start the reaction by adding **FA 1** to this mixture.

- 10.00 cm^3 of **FA 1**
- 20 cm^3 of **FA 2**
- 20 cm^3 of **FA 3**
- 10 drops of starch

Record the time for the reaction to go blue-black.

(ii) Explain the relationship between this time and the one you recorded in **Experiment 2**.

.....

.....

.....

[2]

[Total: 26]

For Examiner's Use

2 Qualitative Analysis

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

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs.
Marks are **not** given for chemical equations.
No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the full name or correct formula of the reagents must be given.

(a) **FA 5**, **FA 6**, **FA 7** and **FA 8** are aqueous solutions each of which contains a single cation and a single anion. Some of the ions present are listed below.

Cu^{2+} Pb^{2+} Cl^{-} OH^{-} CrO_4^{2-}

By observing the reactions that occur when pairs of the solutions are mixed together, you will be able to identify which solution contains which of these ions.

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Use 1 cm depth of each solution in a test-tube and record your observations in the following table. Test the solubility of any precipitate you observe in an excess of each reagent.

	FA 6	FA 7	FA 8
FA 5			
FA 6			
FA 7			

[5]

I	
II	
III	
IV	
V	

(b) From your observations, deduce which solution contains each of the following ions.

ion	Cu^{2+}	Pb^{2+}	OH^-	CrO_4^{2-}	Cl^-
solution					

[3]

I	
II	
III	

For Examiner's Use

(c) The anion in **FA 9** is either the sulfite ion, SO_3^{2-}, or the sulfate ion SO_4^{2-}.

(i) Describe a single test you could carry out that would give positive results for both of these ions.

.....

.....

(ii) Describe a test you could carry out that would distinguish between these two ions.

.....

.....

(iii) Carry out both of these tests and record your results in an appropriate form in the space below.

I	
II	
III	
IV	
V	
VI	

(iv) Which anion is present in **FA 9**?

.....

[6]

[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.]

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2 Reactions of anions

ion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-}(aq)	yellow solution turns orange with H^+(aq); gives yellow ppt. with Ba^{2+}(aq); gives bright yellow ppt. with Pb^{2+}(aq)
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
bromide, Br^-(aq)	gives cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
iodide, I^-(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq)); gives yellow ppt. with Pb^{2+}(aq)
nitrate, NO_3^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil; NO liberated by dilute acids (colourless NO → (pale) brown NO_2 in air)
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) or with Pb^{2+}(aq) (insoluble in excess dilute strong acids)
sulfite, SO_3^{2-}(aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acids)

3 Tests for gases

gas	*test and test result*
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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