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

CANDIDATE NAME	
CENTRE NUMBER	
CANDIDATE NUMBER	

4752941019

CHEMISTRY **9701/32**

Advanced Practical Skills 2 **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

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1	
2	
Total	

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1 Iodide ions are oxidised by iron(III) ions in the presence of acid.

$$2Fe^{3+}(aq) + 2I^{-}(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$$

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 reagents.

FB 1 is 0.0500 mol dm^{-3} aqueous acidified iron(III) chloride, $FeCl_3$.
FB 2 is 0.0500 mol dm^{-3} aqueous potassium iodide, KI.
FB 3 is 0.00500 mol dm^{-3} aqueous sodium thiosulfate, $Na_2S_2O_3$.
FB 4 is 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

- Using a 25 cm^3 measuring cylinder add the following to a 100 cm^3 beaker:
  - 10 cm^3 of **FB 2**
  - 20 cm^3 of **FB 3**
  - 10 cm^3 of **FB 4**
- Fill the burette labelled **FB 1** with aqueous acidified iron(III) chloride, **FB 1**.
- Run 20.00 cm^3 of **FB 1** into a second 100 cm^3 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

- Using a 25 cm^3 measuring cylinder add the following to a 100 cm^3 beaker:
  - 10 cm^3 of **FB 2**
  - 20 cm^3 of **FB 3**
  - 10 cm^3 of **FB 4**
- Fill a second burette with distilled water.
- Run 10.00 cm^3 of **FB 1** into a second 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into the beaker containing **FB 1**.
- 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 iron(III) chloride.
  Remember that the combined volume of **FB 1** and distilled water must always be 20.00 cm^3.
  Do not use a volume of **FB 1** that is less than 6.00 cm^3.

Record all your results in a single table. You should include the volume of iron(III) chloride, 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 change in the iron(III) ion concentration that occurs from the time when the mixtures are combined to the time when the solution turns blue-black. You must show your working.

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

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

(ii) Calculate how many moles of iodine, I_2, reacted with the number of moles of thiosulfate ions calculated in **(i)**.

moles of I_2 = ... mol

(iii) Calculate how many moles of iron(III) ions, Fe^{3+}, reacted to form the number of moles of iodine calculated in **(ii)**.

moles of Fe^{3+} = ... mol

(iv) Use your answer from **(iii)** to calculate the decrease in concentration of iron(III) ions in the total reaction mixture (60 cm^3) from the start of the experiment to the point when the solution turned blue-black.

decrease in Fe^{3+} concentration = ... mol dm^{-3}

[3]

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[Turn over

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

$$\text{'rate'} = \frac{\text{decrease in } Fe^{3+} \text{ concentration from } \textbf{(b)(iv)}}{\text{reaction time}} \times 10^6$$

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

If you were unable to answer **(b)(iv)**, you may assume that the decrease in Fe^{3+} concentration is 2.25×10^{-3} mol dm^{-3} (This is not the correct value).

[2]

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

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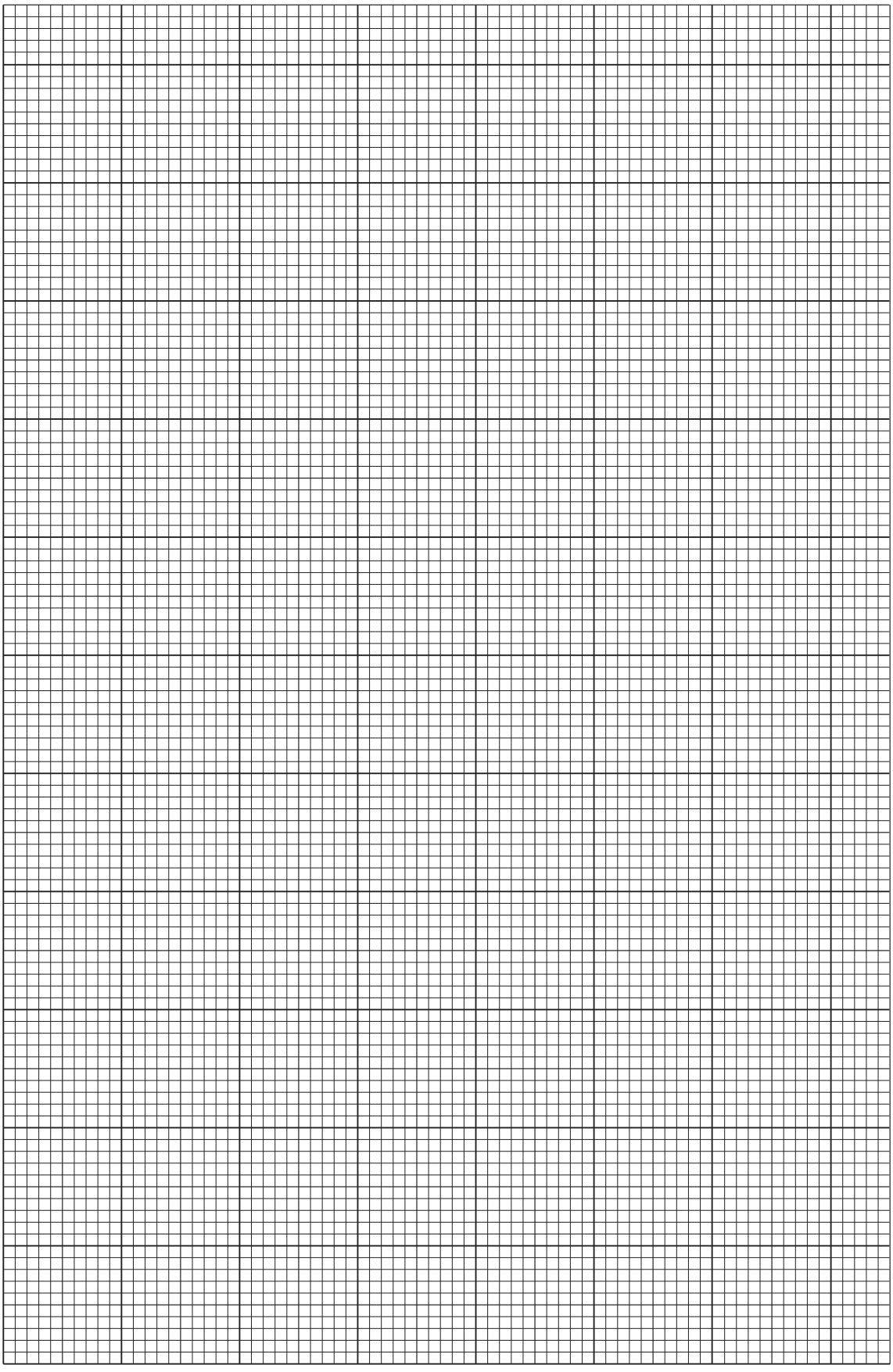

[4]

For Examiner's Use

I	
II	
III	
IV	

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

.....

.....

.....

..... [2]

(f) One source of error in this experiment arises from measuring the volumes of solutions.

(i) Calculate the maximum percentage error in the volume of **FB 1** used in **Experiment 1**.

maximum percentage error = ...%

(ii) Other than errors involving measurements of volumes, suggest an additional source of error in these experiments.

.....

.....

.....

(iii) In this experiment, thiosulfate ions reduce iodine while iron(III) ions oxidise iodide ions. What other reaction might take place that would affect your confidence in the conclusions you made in **(e)**?

.....

.....

[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 **FB 2**, **FB 3** and **FB 4** together and start the reaction by adding this mixture to **FB 1** and the distilled water.

- 10.00 cm^3 of **FB 1**
- 20.00 cm^3 of distilled water
- 10 cm^3 of **FB 2**
- 10 cm^3 of **FB 3**
- 10 cm^3 of **FB 4**

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]

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

Pb^{2+} Ba^{2+} H^{+} CrO_4^{2-} SO_4^{2-} Cl^{-}

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.

	FB 6	**FB 7**	**FB 8**
FB 5			
FB 6			
FB 7			

[5]

I	
II	
III	
IV	
V	

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

ion	Pb^{2+}	Ba^{2+}	H^{+}	CrO_4^{2-}	SO_4^{2-}	Cl^{-}
solution						

[3]

I	
II	
III	

For Examiner's Use

(c) The anion in **FB 9** is either the nitrite ion, NO_2^-, or the nitrate ion, NO_3^-.

(i) Describe a 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 **FB 9**?

.....

[6]

[Total: 14]

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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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