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

CANDIDATE NAME									
CENTRE NUMBER						CANDIDATE NUMBER				

* 0 9 2 6 1 7 2 7 4 6 *

CHEMISTRY **9701/33**

Advanced Practical Skills 1 **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 12 and 13.

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	

This document consists of **13** printed pages and **3** blank pages and **1** insert.

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1 You are to investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid changes as the concentration of the sodium thiosulfate solution is varied.

When aqueous sodium thiosulfate reacts with aqueous hydrogen ions present in an acid, a fine suspension of solid sulfur is formed in the solution.

$$S_2O_3^{2-}(aq) + 2H^+(aq) \rightarrow S(s) + SO_2(g) + H_2O(l)$$

The rate of reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur. The beaker containing a constant volume of reaction mixture is placed on the printed insert supplied. The time is recorded when the print is no longer visible through the suspension of sulfur.

FA 1 is 0.150 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
FA 2 is 0.500 mol dm^{-3} hydrochloric acid, HC*l*.
distilled water

Read through the instructions carefully and prepare a table for your results on page 4 before starting any practical work.

In each of the following experiments you will use varying volumes of **FA 1** and distilled water and a fixed volume of **FA 2**. The **total** volume of solution used will be constant.

(a) Method

Experiment 1

- Use the measuring cylinder labelled **A** to transfer 50 cm^3 of **FA 1** into a 250 cm^3 beaker.
- Use the measuring cylinder labelled **B** to measure 20 cm^3 of **FA 2**.
- Tip the **FA 2** from measuring cylinder **B** into the beaker and **immediately** start timing.
- Stir the contents of the beaker once and place the beaker on the printed insert.
- View the print from directly above and through the solution in the beaker.
- Stop timing when the printed material on the insert is **just** no longer visible.
- Record the reaction time to the nearest second.
- Calculate and record the value of $\frac{(1000)}{(\text{reaction time})}$ to 3 significant figures.
- Empty, rinse and dry the beaker.

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Experiment 2

- Use the measuring cylinder labelled **A** to transfer 40 cm^3 of **FA 1** into the rinsed and dried beaker.
- Use the same measuring cylinder labelled **A** to transfer 10 cm^3 of distilled water into the same beaker.
- Use the measuring cylinder labelled **B** to measure 20 cm^3 of **FA 2**.
- Tip the **FA 2** from measuring cylinder **B** into the beaker and **immediately** start timing.
- Stir the contents of the beaker once and place the beaker on the printed insert.
- View the print from directly above and through the solution in the beaker.
- Stop timing when the printed material on the insert is **just** no longer visible.
- Record the reaction time to the nearest second.
- Calculate and record the value of $\frac{(1000)}{(\text{reaction time})}$ to 3 significant figures.
- Empty, rinse and dry the beaker.

Experiments 3 – 5

- Repeat the experiment using the following volumes of **FA 1** and distilled water.

Experiment 3	30 cm^3 of **FA 1**	+	20 cm^3 of distilled water
Experiment 4	20 cm^3 of **FA 1**	+	30 cm^3 of distilled water
Experiment 5	10 cm^3 of **FA 1**	+	40 cm^3 of distilled water

Experiment 6

When you have completed experiments 1 – 5, carry out one further experiment using a different volume of both **FA 1** and distilled water.

When you have completed all of your experiments, empty and rinse the beaker.

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[Turn over

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Record the results for all six experiments in the space below.
Your table should include columns for the following.

- experiment number
- volume of **FA 1**
- volume of distilled water
- reaction time
- $\frac{(1000)}{(\text{reaction time})}$

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	
X	
XI	

[11]

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

$$\text{'rate'} = \frac{(1000)}{(\text{reaction time})}$$

On the next page plot a graph of **'rate'** against the **volume of FA 1**.

Start each of the axes at zero.

Draw the line of best fit.

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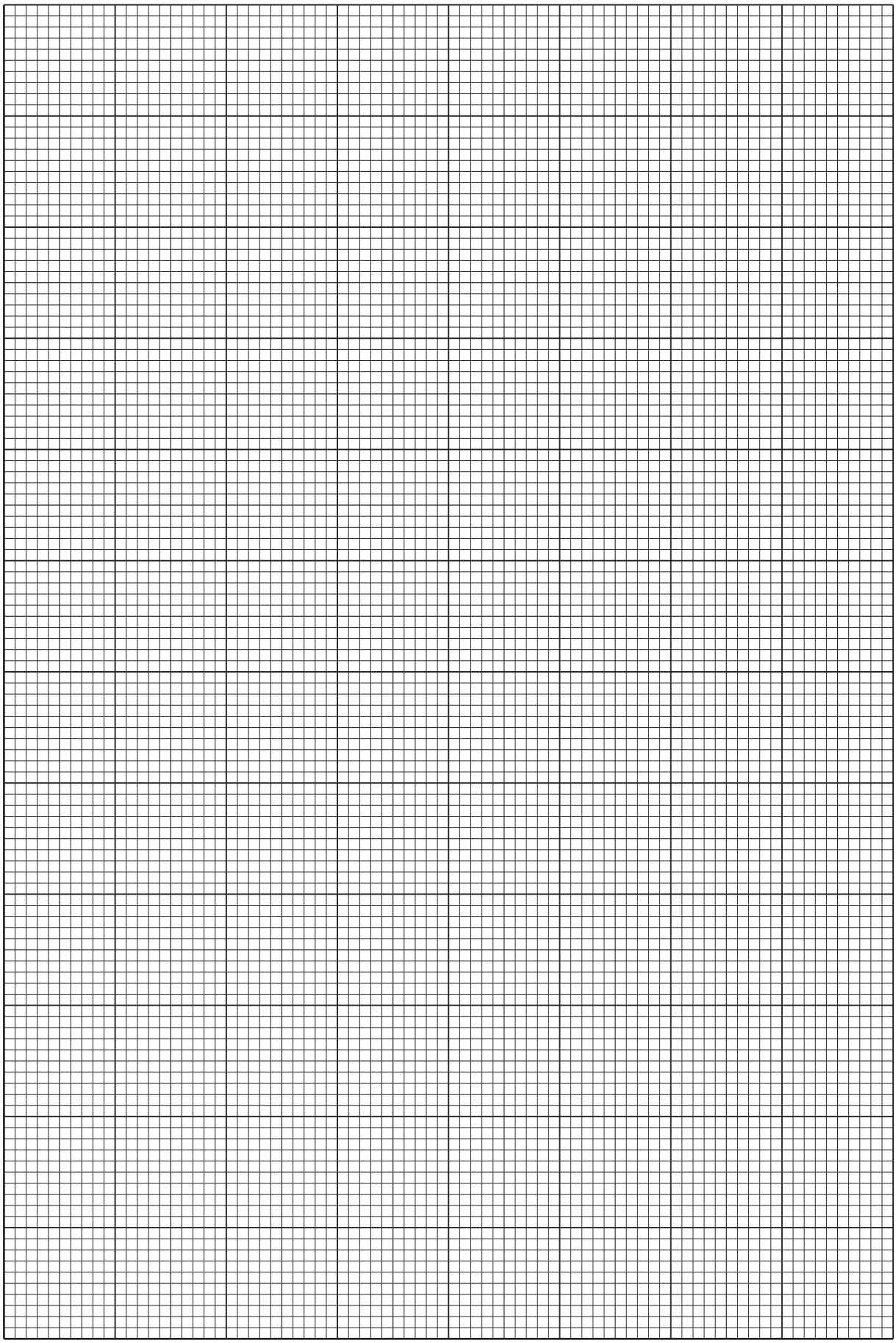

[5]

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I	
II	
III	
IV	
V	

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(c) A student carried out the experiments in a 100 cm^3 beaker instead of a 250 cm^3 beaker. State and explain what effect this would have on the times recorded.

.....

..... [2]

(d) **FA 1** is 0.150 mol dm^{-3} $Na_2S_2O_3$.
Calculate the initial concentration of $Na_2S_2O_3$ in the reaction mixture in **Experiment 5**. Show your working.

The initial concentration of $Na_2S_2O_3$ in **Experiment 5** = ... mol dm^{-3} [2]

(e) In your experiments, the volume of **FA 1** represents the initial concentration of sodium thiosulfate in the reaction mixture.
A text book states that the rate of reaction between aqueous sodium thiosulfate and hydrochloric acid is directly proportional to the concentration of sodium thiosulfate.
Use your graph to decide whether the statement in the text book is correct or not.
Explain your answer.

.....

.....

.....

..... [2]

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(f) When viewing the insert through the solution it is difficult to judge the exact moment when the printed material just disappears.

This uncertainty is different for each experiment and is greater for longer reaction times when the printed material disappears slowly.

Complete the table below, assuming the uncertainties given.

	Experiment 1	**Experiment 5**
recorded reaction time / s		
uncertainty / s	±2	±8
percentage uncertainty	%	%

[1]

(g) Complete the headings in the table below to record the volume of **FA 1** (aqueous sodium thiosulfate), the volume of distilled water and the volume of **FA 2** (hydrochloric acid).

In the second row copy the volumes used in **Experiment 3** from your table of results on page 4.

In the following two rows suggest volumes of each of the reagents that could be used in two further experiments, **Experiment 7** and **Experiment 8**, to investigate how the rate of reaction varies with a change in the concentration of the acid.

Do not carry out these experiments.

Experiment			
3			
7			
8			

[2]

[Total: 25]

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2 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.

Solutions **FA 3**, **FA 4**, **FA 5** and **FA 6** each contain one cation and one anion from those listed on pages 12 and 13.

(a) Some cations interfere with tests for anions and have to be removed from the solution before the tests for anions present can be performed. One way in which this can be carried out is to precipitate the cation in the form of its insoluble carbonate.

Carry out the following tests on both **FA 3** and **FA 5**.

test	*observation*	
	FA 3	**FA 5**
To 5 cm depth of solution in a boiling tube, add all of the sodium carbonate, Na_2CO_3, from one of the tubes provided. Stir the mixture.		

Retain the mixture from **FA 3** for use in **(b)**.

[4]

(b) Filter the mixture from **FA 3** from **(a)** into another boiling tube. Ignore any colour in the filtered solution.
Add 5 cm depth of dilute nitric acid. This removes any excess of carbonate ions.

Carry out the following tests on the acidified filtrate from **FA 3**.

test	*observation*
To 2 cm depth of the acidified filtrate from **FA 3** in a test-tube, add 1 cm depth of aqueous silver nitrate, then	
add an excess of aqueous ammonia.	

[1]

(c) Carry out the following test on **FA 4**.

test	*observation*
To 1 cm depth of **FA 4** in a test-tube, add 1 cm depth of **FA 3**, then	
add a few drops of starch solution.	

[2]

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(d) Carry out the following tests.

test	*observation*			
	FA 3	**FA 4**	**FA 5**	**FA 6**
To 1 cm depth of solution in a test-tube, use a dropping pipette to add, a little at a time, 0.5 cm depth of aqueous sodium hydroxide, then				
add a further 2 cm depth of aqueous sodium hydroxide.				
To 1 cm depth of solution in a test-tube, use a dropping pipette to add, a little at a time, 0.5 cm depth of aqueous ammonia, then				
add a further 2 cm depth of aqueous ammonia.				

[4]

(e) The results from tests in **(d)** should enable you to identify either a single cation in a solution, or a pair of cations which have identical reactions with the reagents used.
Identify any single ion that is present or suggest a pair of ions that may be present in each of the solutions.

solution	*cation(s)*
FA 3	
FA 4	
FA 5	
FA 6	

[2]

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(f) Where you were unable to identify a single cation in **(e)**, suggest a suitable reagent which would allow you to identify which cation is present in the solution.

Do not carry out this test.

The reagent to be used is .....

State the expected observations to identify the presence of each of the cations from the pair of ions you have given in **(e)**.

cation 1 .....

.....

cation 2 .....

..... [1]

(g) By considering the results of **all** your tests, enter one of the following responses in each of the boxes below.

- chloride
- bromide
- iodide
- no halide ion is present
- insufficient tests (have been performed to identify any halide ion)

FA 3	
FA 4	
FA 5	

[1]

[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.]

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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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