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

CANDIDATE NAME									
CENTRE NUMBER						CANDIDATE NUMBER				

* 3 1 5 6 8 6 1 6 5 2 *

CHEMISTRY **9701/31**

Paper 31 Advanced Practical Skills **May/June 2009**

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 are advised to show all working in calculations.
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 **11** printed pages, **1** blank page and **1** Insert.

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1 You are provided with the following.
FA 1 is 0.15 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
FA 2 is aqueous copper(II) sulfate.
You are also provided with a 10% solution of potassium iodide, KI, and starch indicator.

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You are required to determine the concentration, in g dm^{-3}, of hydrated copper(II) sulfate, $CuSO_4.5H_2O$, in **FA 2**.

Dilution of FA 2

(a) By using a burette measure between 47.00 cm^3 and 47.50 cm^3 of **FA 2** into the 250 cm^3 graduated flask labelled **FA 3**.
Record your burette readings and the volume of **FA 2** added to the flask in the space below.

Make up the contents of the flask to the 250 cm^3 mark with distilled water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

Titration

Fill a second burette with **FA 1**.

Perform a rough (trial) titration as follows.

Pipette 25.0 cm^3 of **FA 3** into a conical flask.
Use the measuring cylinder provided to add 10 cm^3 of 10% potassium iodide to the flask.
The Cu^{2+} ions in **FA 3** oxidise the iodide ions to iodine, I_2, which can be titrated with **FA 1**.
The flask will also contain an off-white precipitate of copper(I) iodide, CuI.

Run **FA 1** from the burette, 1 cm^3 at a time, until the brown colour of the iodine solution has changed to pale brown.
Add approximately 10 drops of starch indicator. A blue-black colour should be seen as the starch reacts with the residual iodine.
Continue to add **FA 1** 1 cm^3 at a time until the blue-black colour of the starch-iodine complex disappears and there is no further colour change.

In this rough titration ..... cm^3 of **FA 1** were added.

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Perform sufficient further titrations to obtain reliable results.

Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

For Examiner's Use

i	
ii	
iii	
iv	
v	
vi	

[6]

(b) From your titration results obtain a volume of **FA 1** to be used in your calculations. Show clearly how you obtained this volume.

[1]

Calculations

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

(c) Use your answer to **(b)** to calculate how many moles of $Na_2S_2O_3$ were run from the burette into the conical flask.

..... mol of $Na_2S_2O_3$ were run from the burette into the conical flask.

Calculate how many moles of I_2 reacted with the $Na_2S_2O_3$ run from the burette.

$$2S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2e^-$$

$$I_2 + 2e^- \rightarrow 2I^-$$

..... mol of I_2 reacted with the $Na_2S_2O_3$ run from the burette.

Calculate how many moles of Cu^{2+} ions reacted with iodide ions to produce this amount of I_2.

$$2Cu^{2+} + 4I^- \rightarrow 2CuI + I_2$$

..... mol of Cu^{2+} reacted to form the I_2.

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Calculate the concentration, in mol dm^{-3}, of Cu^{2+} in **FA 3**.

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The concentration of Cu^{2+} in **FA 3** is ..... mol dm^{-3}.

Calculate the concentration, in mol dm^{-3}, of Cu^{2+} in **FA 2**.

i	
ii	
iii	
iv	
v	

The concentration of Cu^{2+} in **FA 2** is ..... mol dm^{-3}.

Calculate the concentration, in g dm^{-3}, of $CuSO_4.5H_2O$ in **FA 2**.
[A_r: Cu, 63.5; H, 1.0; O, 16.0; S, 32.1]

FA 2 contains ..... g dm^{-3} $CuSO_4.5H_2O$.
[5]

(d) The maximum error in any burette reading is ±0.05 cm^3.

Explain how the maximum error in a titration is therefore ±0.10 cm^3.

.....

.....

..... [1]

(e) Calculate the maximum percentage error in the average titre given in **(b)**.

The error is ..... %. [1]

[Total: 14]

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2 You are to investigate how the rate of formation of sulfur varies with the concentration of sodium thiosulfate, $Na_2S_2O_3$, in the reaction below.

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$$Na_2S_2O_3(aq) + 2HCl(aq) \rightarrow S(s) + 2NaCl(aq) + SO_2(g) + H_2O(l)$$

Care should be taken to avoid inhalation of $SO_2(g)$ that is given off during this reaction.

You are provided with the following.

FA 1, 0.15 mol dm^{-3} $Na_2S_2O_3$	a measuring cylinder to measure 50 cm^3
FA 4, 2.0 mol dm^{-3} HC*l*	a measuring cylinder or marked tube to measure 5 cm^3
a printed insert	a stop clock or clock with seconds hand

(a) Method – Read through the instructions before starting any practical work.

- Using the larger measuring cylinder transfer 50 cm^3 of **FA 1** into a 250 cm^3 beaker.
- Measure 5 cm^3 of **FA 4** in the smaller measuring cylinder (or marked tube).
- Tip the **FA 4** into the **FA 1** in the beaker and **immediately** start timing.
- Swirl the beaker to mix the solution and place it on top of the printed insert.
- View the printed insert from above so that it is seen through the solution.
- Note the time when the printing on the insert just disappears.

- Empty and rinse the beaker. Shake out as much of the rinse water as possible and dry the outside of the beaker.
- Repeat the experiment using 25 cm^3 of **FA 1** and 25 cm^3 of distilled water. Add 5 cm^3 of **FA 4** to start the reaction.

- Select suitable volumes of **FA 1** and distilled water for **one** further experiment to investigate the effect of sodium thiosulfate concentration on the rate of reaction. *Remember to use 5 cm^3 of* **FA 4** *and to keep the total volume of* **FA 1** *and distilled water constant.*

In an appropriate form record the following below:
- all measurements of volume and time (to the nearest second) for each experiment,
- calculated values of $^1/_{time}$ which are a measure of the rate of reaction.

Results

i	
ii	
iii	
iv	
v	
vi	
vii	
viii	
ix	

[9]

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(b) The total volume in each experiment is constant. Using volumes from the first two experiments, show by simple calculation that the volume of **FA 1** used is a measure of its concentration in the reaction mixture.

[1]

(c) What is the relationship between the rate of reaction and the time taken?

.....

.....

..... [1]

(d) For **each** experiment calculate the numerical value of (volume of **FA 1** $\times$ time).

experiment	(volume of **FA 1** $\times$ time) / (cm^3 s)
1	
2	
3	

Use your results in **(a)** and these calculated values to deduce the relationship between the concentration of $Na_2S_2O_3$ and the rate of formation of sulfur.

.....

.....

..... [2]

(e) Outline briefly how you would modify the experimental method to investigate the effect of temperature change on the reaction rate.

.....

.....

..... [1]

[Total: 14]

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[Turn over

3 **FA 5**, **FA 6**, **FA 7** and **FA 8** are aqueous solutions each containing one cation and one anion.

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You will carry out specified tests to deduce

- the cations present in two of the four solutions,
- the anions present in three of the four solutions.

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.

(a) Carry out the following tests. Record your observations in the spaces provided in the table.

	FA 5	**FA 6**	**FA 7**	**FA 8**
To 1 cm depth of solution in a test-tube add aqueous sodium hydroxide, a little at a time, until in excess.				
To 1 cm depth of solution in a test-tube add aqueous ammonia, a little at a time, until in excess.				

Using the qualitative analysis notes printed on page 11 and the observations above it is possible to identify the cation present in one of the solutions and also to identify possible cations in another of the solutions.

Solution ..... contains the single cation ..... .

Solution ..... contains one of the following cations, ...

[4]

i	
ii	
iii	
iv	

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Rinse and re-use test-tubes.

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(b) You are to select suitable reagents and carry out tests on the solutions to identify which solution or solutions contain either a nitrate or a nitrite ion.

Record in an appropriate form below the tests performed and the observations made.

i	
ii	

Nitrate or nitrite ions are contained in solution(s) ..... . [2]

(c) Carry out the following tests.

	FA 5	**FA 6**	**FA 7**	**FA 8**
To 1 cm depth of solution in a test-tube add 1 cm depth of dilute hydrochloric acid.				

Use these observations to identify the cation or anion present in each solution and complete the table below.

solution	anion/cation present	reason for selecting the ion

i	
ii	
iii	
iv	

[4]

(d) **FA 5** and **FA 7** can be mixed to confirm the identity of one ion in each of the two solutions.

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test	*observation*
To 1 cm depth of **FA 5** in a test-tube add 1 cm depth of **FA 7**.	

i	
ii	

This observation confirms the presence of …… in **FA 5** and …… in **FA 7**. [2]

[Total: 12]

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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 $\rightarrow$ (pale) brown NO_2 in air)
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acid) gives white ppt. with Pb^{2+}(aq)
sulfite, SO_3^{2-}(aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acid)

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) (aq) from orange to green

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