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

CANDIDATE NAME	

CENTRE NUMBER						CANDIDATE NUMBER					

7700296171

CHEMISTRY **9701/34**

Advanced Practical Skills **May/June 2011**

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Instructions to Supervisors

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	
3	
Total	

This document consists of **12** printed pages and **1** insert.

DC (SJF) 33707/5

UNIVERSITY *of* CAMBRIDGE International Examinations

[Turn over

1 You are to determine the concentration, in mol dm^{-3}, of the aqueous sodium thiosulfate. To do this you will first produce iodine solution by reacting aqueous potassium iodide with aqueous potassium manganate(VII). In this reaction iodide ions are oxidised to iodine by manganate(VII) ions in acidic solution.

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$$2MnO_4^-(aq) + 10I^-(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(l)$$

You will then titrate the iodine with aqueous thiosulfate ions, in **FB 1**.

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

You are provided with the following.

FB 1 is a solution of sodium thiosulfate, $Na_2S_2O_3$, of unknown concentration.
FB 3 is 0.0050 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FB 4 is 0.10 mol dm^{-3} potassium iodide, KI.
FB 5 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4.
starch indicator

(a) Method

Dilution

- Fill the burette with **FB 1**.
- Run between 45.50 cm^3 and 46.50 cm^3 of **FB 1** from the burette into the 250 cm^3 graduated (volumetric) flask, labelled **FB 2**.
- Make the solution up to the mark with distilled water.
- Shake the flask to mix the solution of **FB 2**.

In the space below record your burette readings and the volume of **FB 1** added to the graduated flask.

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Titration

- Fill a second burette with **FB 2**, the diluted sodium thiosulfate.
- Pipette 25.0 cm^3 of **FB 3** into a conical flask.
- Using a 25 cm^3 measuring cylinder, add about 10 cm^3 of **FB 4**.
- Using the same measuring cylinder, add about 10 cm^3 of **FB 5**.
- Titrate the mixture in the flask with **FB 2** until the colour is pale yellow.
- Add about 10 drops of starch indicator. A blue-black colour should be seen as the starch reacts with the remaining iodine.
- Continue to add **FB 2** until the blue-black colour just disappears leaving a colourless solution.

You should perform a **rough titration**.
In the space below record your burette readings for this rough titration.

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 an appropriate form below all of your burette readings and the volume of **FB 2** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

(b) From your accurate titration results obtain a suitable value to be used in your calculations.
Show clearly how you have obtained this value.

The iodine produced by 25.0 cm^3 of **FB 3** required ……………… cm^3 of **FB 2**. [1]

(c) Calculations

For Examiner's Use

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

(i) Calculate how many moles of MnO_4^- were pipetted into the conical flask.

.... mol of MnO_4^-

(ii) Calculate how many moles of I_2 were produced from the number of moles of MnO_4^- calculated in **(i)**.

$$2MnO_4^-(aq) + 10I^-(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(l)$$

.... mol of I_2

(iii) Calculate how many moles of $S_2O_3^{2-}$ reacted with the I_2 in **(ii)**.

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

I	
II	
III	
IV	
V	
VI	

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

(iv) Calculate how many moles of $S_2O_3^{2-}$ were present in the 250 cm^3 graduated (volumetric) flask.

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

(v) Use your answer to **(iv)** and the volume of **FB 1** that was diluted to calculate the concentration, in mol dm^{-3}, of the original solution of sodium thiosulfate, **FB 1**.

The concentration of $Na_2S_2O_3$ in **FB 1** was .... mol dm^{-3}.

[6]

(d) The maximum error for a 25 cm^3 pipette commonly used in schools is ±0.06 cm^3.
The maximum individual error in any single burette reading is ±0.05 cm^3.

Calculate each of the following.

The maximum percentage error in the volume of **FB 3** pipetted into the conical flask

maximum percentage error in the pipetted volume of **FB 3** = ... %

The maximum percentage error in the titre calculated in **(b)**

maximum percentage error in the titre volume = ... %
[1]

[Total: 15]

2 You are to investigate how the rate of the following reaction varies with the concentration of sodium thiosulfate, $Na_2S_2O_3$.

For Examiner's Use

$$Na_2S_2O_3(aq) + H_2SO_4(aq) \rightarrow S(s) + Na_2SO_4(aq) + SO_2(g) + H_2O(l)$$

The rate can be found by measuring how long it takes for the solid sulfur formed to obscure the printing on the insert provided.

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

FB 5 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4.
FB 6 is 0.10 mol dm^{-3} sodium thiosulfate $Na_2S_2O_3$.

Read through the instructions carefully and prepare a table for your results on page 7 before starting any practical work.

(a) Method

- Using the 50 cm^3 measuring cylinder transfer 45 cm^3 of **FB 6** into a 100 cm^3 beaker.
- Using the 25 cm^3 measuring cylinder measure 10 cm^3 of **FB 5**.
- Tip the **FB 5** into the **FB 6** in the beaker and **immediately** start timing.
- Stir the mixture once with a glass rod and place it on top of the printed insert.
- View the printed insert from above so that it is seen through the mixture.
- Record the time, to the nearest second, when the printing on the insert **just** disappears.

- Empty and rinse the beaker. Shake out as much of the water as possible and dry the outside of the beaker.
- You will repeat the experiment to find out how the time for the printing on the insert to disappear changes when a different volume of **FB 6** is used.
- Using the 50 cm^3 measuring cylinder transfer 20 cm^3 of **FB 6** and 25 cm^3 of distilled water into the 100 cm^3 beaker.
- Using the 25 cm^3 measuring cylinder add 10 cm^3 of **FB 5** to the mixture and **immediately** start timing.
- Stir the mixture once with a glass rod and place it on top of the printed insert.
- View the printed insert from above so that it is seen through the mixture.
- Record the time, to the nearest second, when the printing on the insert **just** disappears.

- Select suitable volumes of **FB 6 and** distilled water for **two** further experiments to investigate the effect of volume of sodium thiosulfate on the time taken for the printing on the insert to **just** disappear.

Calculate the values of 1/time, where time is in seconds, to an appropriate number of significant figures.

In the space below, record, in an appropriate form, all measurements of volume, time, and your calculated values of 1/time.

For Examiner's Use

I	
II	
III	
IV	
V	

[5]

(b) Why was the **total** volume of solution kept constant in the experiments?

....

.... [1]

(c) It may be assumed that the rate of reaction is proportional to 1/time.
Draw a conclusion from your results about the relationship between the concentration of sodium thiosulfate used and the rate of reaction.
Explain your answer.

....

....

.... [1]

(d) In the four experiments, which value of the time measured had the greatest error?
Explain your answer.

....

....

.... [1]

(e) How could the procedure be adapted to find the effect of changing the concentration of acid on the rate of reaction?

....

....

.... [1]

[Total: 9]

For Examiner's Use

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 full name or correct formula of the reagents must be given.

There are two parts to this question. In the first part you will analyse three metal salts, **FB 7**, **FB 8** and **FB 9**, to identify the cation in each. In the second part you will carry out a series of tests on a different salt, **FB 10**, to identify the anion.

(a) **FB 7**, **FB 8** and **FB 9** each contain one of aluminium ions, lead ions or zinc ions.
By reference to the Qualitative Analysis Notes on pages 11 and 12, select **two** appropriate reagents to perform tests to identify the cations present.

reagent ....

Record the tests performed and the results of those tests in an appropriate form in the space below.

I	
II	
III	
IV	
V	

[5]

(b) From your observations, identify the cations present in **FB 7**, **FB 8** and **FB 9**. State the **minimum** evidence to support each of your choices.

FB 7 contains ……………………… cations.

evidence ....

....

FB 8 contains ………………………… cations.

evidence ....

....

FB 9 contains ………………………… cations.

evidence ....

....
[3]

For Examiner's Use

(c) You are provided with a solid sample of **FB 10** which is a metal salt. Use this sample to perform the experiments described below.
Record all your observations in the table.

test	*observations*
(i) Place a spatula measure of solid **FB 10** into a dry hard-glass tube. Hold the test-tube in a holder. Heat the test-tube gently at first	
and then **very strongly** for several minutes.	
Allow the test-tube to cool, and then half fill it with distilled water. Dissolve the solid residue. This is **FB 11**. Retain this for tests **(iv)** and **(v)**. **While you are waiting for the test-tube to cool, continue with experiment (c)(ii) and (iii).**	
(ii) Pour a 2 cm depth of aqueous copper(II) sulfate in a test-tube. Add a spatula measure of solid **FB 10**.	
(iii) Pour a 2 cm depth of aqueous aluminium sulfate into a test-tube. Add a spatula measure of solid **FB 10**.	

I	
II	
III	
IV	
V	

test	observations
(iv) Pour a 2 cm depth of solution **FB 11** into a test-tube. Add the same volume of aqueous barium chloride.	
Then, using a dropping pipette, add dilute hydrochloric acid until no further change is seen.	
(v) Pour 1 cm depth of **FB 11** into a test-tube and add an equal depth of aqueous copper(II) sulfate.	
Transfer the contents of the test-tube into an evaporating dish and place it over a Bunsen burner on a tripod and gauze. Heat strongly until all the water has been driven off and no further change is seen.	

[5]

For Examiner's Use

(d) Consider your observations in **(c)** and answer the following questions. In each case, provide evidence from your observations to support your conclusions.

(i) Identify the anion present in **FB 11** and state evidence to support your choice.

FB 11 contains the ………………………… anion.

evidence ....

....

(ii) Suggest what type of reaction occurs as **FB 10** is converted into **FB 11**.

....

....

(iii) Suggest an explanation for what you observed in **(c)(iii)**.

....

....

[3]

[Total: 16]

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

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