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

CANDIDATE NAME

CENTRE NUMBER

CANDIDATE NUMBER

CHEMISTRY **9701/32**

Paper 32 Advanced Practical Skills **October/November 2008**

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 **12** printed pages.

SP (SC/KS) T56858/3

UNIVERSITY *of* CAMBRIDGE International Examinations

[Turn over

1 You are required to find the concentration in mol dm^{-3} of sodium thiosulphate, $Na_2S_2O_3$, in solution **FB 1**.

FB 1 contains sodium thiosulphate.
FB 2 is potassium manganate(VII) containing 28.44 g dm^{-3} $KMnO_4$.
FB 3 is 1.0 mol dm^{-3} sulphuric acid, H_2SO_4.
FB 4 is 10% potassium iodide containing 100 g dm^{-3} KI.
You are also provided with starch indicator.

Dilution of FB 2

(a) By using a burette measure between 41.00 cm^3 and 42.00 cm^3 of **FB 2** into the 250 cm^3 graduated (volumetric) flask labelled **FB 5**.
Record your burette readings and the volume of **FB 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 **FB 1**, the solution containing sodium thiosulphate.

Use a measuring cylinder to transfer 10 cm^3 of **FB 3** and 10 cm^3 of **FB 4** into a conical flask.
Pipette 25.0 cm^3 of **FB 5** into the conical flask containing the mixture of **FB 3** and **FB 4**.
The potassium manganate(VII) oxidises potassium iodide to iodine, I_2.

Titrate the liberated iodine with **FB 1** as follows. Run the solution from the burette into the conical flask until the initial red/brown colour of the iodine becomes pale yellow. Then add 1 cm^3 of the starch indicator and continue to add **FB 1** drop by drop until the blue/black colour of the starch/iodine complex disappears, leaving a colourless solution. This is the end-point of the titration.

Perform a rough (trial) titration and sufficient further titrations to obtain accurate results.

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

i	
ii	
iii	
iv	
v	
vi	

[6]

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

The volume of **FB 1** is …………….. cm^3. [1]

Calculations

Show your working and appropriate significant figures in all of your calculations.

(c) Calculate how many moles of $KMnO_4$ are contained in the **FB 2** run into the graduated flask. [A_r: K, 39.1; O, 16.0; Mn, 54.9]

………………….. mol of $KMnO_4$ are run into the graduated flask.

Calculate how many moles of $KMnO_4$ are then pipetted from the 250 cm^3 graduated flask into the titration flask.

………………….. mol of $KMnO_4$ are pipetted into the titration flask.

Use this answer to calculate how many moles of **iodine molecules**, $\mathbf{I_2}$, are formed when the manganate(VII) ions react with an excess of iodide ions in the titration flask.

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$
$$I^- \rightarrow \tfrac{1}{2}I_2 + e^-$$

………………….. mol of **iodine molecules**, I_2, are formed in the reaction.

Use this answer to calculate how many moles of sodium thiosulphate will react with the **iodine molecules** formed.

$$2S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2e^-$$
$$\tfrac{1}{2}I_2 + e^- \rightarrow I^-$$

………………….. mol of thiosulphate ions react with the **iodine molecules** formed in the reaction.

i	
ii	
iii	
iv	
v	

Calculate, to **3 significant figures**, the concentration in mol dm^{-3} of the sodium thiosulphate, $Na_2S_2O_3$, in **FB 1**.

The concentration of sodium thiosulphate in **FB 1** is ……………….. mol dm^{-3}.

[5]

[Total: 12]

2 **Read through the instructions before starting the experiment.**

The relative molecular mass, M_r, of a metal carbonate can be estimated by adding a weighed sample of the carbonate to a weighed excess of hydrochloric acid and measuring the mass of carbon dioxide evolved.

The tubes labelled **FB 6** and **FB 7** each contain the solid carbonate X_2CO_3.
FB 8 is 2.0 mol dm^{-3} hydrochloric acid.

Method

(a) Follow the instructions below to determine the mass of carbon dioxide given off when X_2CO_3 reacts with an excess of hydrochloric acid.

- Use a measuring cylinder to transfer 75 cm^3 of **FB 8** into a 250 cm^3 conical flask.
- Weigh the flask and acid **FB 8**.
- Weigh the tube labelled **FB 6** which contains the carbonate X_2CO_3.
- Tip the contents of the tube **FB 6** into the acid in the flask, a little at a time. This prevents loss of acid as spray from the vigorous reaction.
- When the reaction appears to be complete, swirl the flask and leave to stand for 2–3 minutes, then reweigh the flask and its contents.
- Reweigh the tube **FB 6** and any residual carbonate not added to the acid.
- Rinse out and drain the flask.
- Repeat the whole experiment using tube **FB 7**.

In an appropriate form below record the following.
- all measurements of mass made
- the mass of the carbonate, X_2CO_3, added
- the mass of carbon dioxide given off

[mass of CO_2 = (initial mass of flask + acid) + (mass of carbonate) – (final mass of flask + contents)]

Results

i	
ii	
iii	
iv	

[4]

Calculations

(b) From your results for each experiment calculate the mass of X_2CO_3 that would produce 1.0 g of CO_2.

With **FB 6** …………………….. g of CO_2 are given off from ………………… g X_2CO_3.

1.0 g of CO_2 is given off from ………………… g X_2CO_3.

With **FB 7** …………………….. g of CO_2 are given off from ………………… g X_2CO_3.

1.0 g of CO_2 is given off from ………………… g X_2CO_3.

[3]

(c) For each experiment calculate the relative molecular mass, M_r, of X_2CO_3.

$$X_2CO_3(s) + 2HCl(aq) \rightarrow 2XCl(aq) + CO_2(g) + H_2O(l)$$

[A_r: C, 12.0; O, 16.0]

M_r of X_2CO_3 from the experiment with **FB 6** is ………………………… .

M_r of X_2CO_3 from the experiment with **FB 7** is ………………………… .

[1]

(d) Carbon dioxide is soluble in aqueous solutions and this can lead to an error in the molecular mass calculated.

From your observations on carrying out the experiments suggest another significant source of error. Explain the effect this will have on the measurements made and the molecular mass calculated.

....

....

....

....[1]

(e) Some of the carbon dioxide given off in the reaction remains dissolved in the acid solution.

Suggest how you might modify the experimental method described to reduce or eliminate this error.

....

....

....[1]

(f) Carry out the following instructions.

- Half fill each of two test-tubes with distilled water and place the tubes in a test-tube rack.
- To one test-tube add 1 spatula measure of powdered barium carbonate, $BaCO_3$.
- To the second test-tube add 1 spatula measure of X_2CO_3.
- Stopper each test-tube and shake vigorously.
- Half fill each of two boiling-tubes with **FB 3**, dilute sulphuric acid.
- To one boiling-tube add 1 spatula measure of powdered barium carbonate, $BaCO_3$.
- To the second boiling-tube add 1 spatula measure of X_2CO_3.
- **Do not attempt to stopper or shake either of these boiling-tubes.**

Record your observations in the table below.

	$BaCO_3$	X_2CO_3
water		
FB 3 dilute sulphuric acid		

It is suggested that sulphuric acid could be used in place of hydrochloric acid in experiments to determine the M_r of metal carbonates.
Make use of your observations and your knowledge of the chemistry of barium, to explain why the use of sulphuric acid would not be appropriate if the carbonate is barium carbonate.

....

....

....

[2]

[Total: 12]

For Examiner's Use

3 **FB 9**, **FB 10** and **FB 11** are aqueous solutions, each containing one of the cations listed on page 11 of the qualitative analysis notes.

You will react **FB 9**, **FB 10** and **FB 11** with aqueous sodium hydroxide, NaOH, and aqueous ammonia, NH_3, to identify the cations present in each of these solutions. You will also perform tests to identify the anions present in **FB 9** and **FB 10**.

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

Note that three of the cations listed on page 11 may give **no** precipitate with aqueous NaOH.

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.

(a) Pour 1 cm depth of **FB 9**, **FB 10** and **FB 11** into separate test-tubes. Stand the tubes in a test-tube rack and add aqueous sodium hydroxide, NaOH, a little at a time until the reagent is in excess. Repeat the test with aqueous ammonia, NH_3, as the reagent.

Record your observations in an appropriate form below.

i	
ii	
iii	
iv	

[4]

(b) Using the observations above it is not possible to identify a single cation for any of the solutions. Use your observations and the qualitative analysis notes on page 11 to identify, for each solution, two **or** three cations which could be present.

FB 9 could contain the cations ....

FB 10 could contain the cations ....

FB 11 could contain the cations ....

[2]

(c) Use the qualitative analysis notes on page 11 to select further reagents or tests to identify precisely which cation is present in each of **FB 9**, **FB 10** and **FB 11**.

Record in an appropriate form below,

- details of the reagents to be used,
- the tests to be carried out,
- your observations when the additional tests are carried out.

A boiling-tube **must** be used if any solution is to be heated.

i	
ii	
iii	
iv	

Conclusion

FB 9 contains the cation ……………………………

FB 10 contains the cation ……………………………

FB 11 contains the cation ……………………………

[4]

For Examiner's Use

(d) **FB 9** and **FB 10** each contain one anion which is either a sulphate or a halide.

Use the qualitative analysis notes on page 12 to select appropriate reagents and tests to determine which anion is present in each solution.

Record in an appropriate form below,

- details of the reagents to be used,
- the tests to be carried out,
- your observations when the tests are carried out.

i	
ii	
iii	
iv	
v	
vi	

Conclusion

FB 9 contains the anion ……..........................……..

FB 10 contains the anion ……..........................……..

[6]

[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)	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. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. 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 pale 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 A*l* foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and A*l* foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulphate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+}(aq) or with Pb^{2+}(aq) (insoluble in excess dilute strong acids)
sulphite, 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
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green