

### UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS General Certificate of Education

Advanced Subsidiary Level and Advanced Level

CHEMISTRY			9701/3
CENTRE NUMBER		CANDIDATE NUMBER	
CANDIDATE NAME			

Advanced Practical Skills 1

October/November 2011

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	

For Exam	iner's Use
1	
2	
Total	

This document consists of 10 printed pages and 2 blank pages.



1 You are required to investigate the effect of temperature on the rate of reaction of peroxydisulfate ions with iodide ions. Iodide ions are oxidised to iodine by peroxydisulfate ions.

For Examiner's Use

$$\mathrm{S_2O_8^{2-}(aq)}$$
 +  $\mathrm{2I^-(aq)}$   $\rightarrow$   $\mathrm{2SO_4^{2-}(aq)}$  +  $\mathrm{I_2(aq)}$ 

**FA 1** is aqueous potassium peroxydisulfate, K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>.

**FA 2** is an aqueous solution containing a mixture of potassium iodide, KI, sodium thiosulfate,  $Na_2S_2O_3$ , and starch.

When **FA 1** and **FA 2** are mixed together the potassium peroxydisulfate reacts with the potassium iodide to make iodine. As soon as this iodine is formed, it reacts with the sodium thiosulfate and is turned back into iodide ions. Only when all the sodium thiosulfate has reacted does iodine remain in the solution. The solution then turns blue-black because of the presence of the starch indicator. The rate of reaction can be determined by the time it takes for a blue-black colour to first appear in the colourless mixture.

### (a) Method

Read through the method and prepare a table on page 3 to record the initial and final temperatures and the reaction time for each experiment, before starting any practical work.

- Half-fill a 250 cm<sup>3</sup> beaker with water to act as a water bath.
- Place it on a tripod and gauze and heat it with a Bunsen burner to about 65 °C then remove the Bunsen. While your water is being heated continue with the following steps of the method.
- Fill the burette, labelled **FA 1**, with the aqueous potassium peroxydisulfate, **FA 1**.
- Fill the burette, labelled FA 2, with the mixture of solutions, FA 2.
- Measure 10.0 cm<sup>3</sup> of **FA 1** into a boiling tube.
- Measure 10.0 cm<sup>3</sup> of FA 2 into a second boiling tube.
- Place both boiling tubes in the water bath.
- Clamp one of the tubes and place a thermometer in this tube.
- When the temperature of this solution has reached about 60 °C, pour the contents of the second tube into the clamped tube. Start timing immediately, note the temperature and stir the mixture.
- Record this initial temperature.
- Stop timing as soon as the blue-black colour appears. Record this reaction time to the **nearest second** and record the final temperature.
- Repeat the experiment at decreasing temperatures as many times as necessary
  to generate data for plotting a graph. The experiment should not be performed at a
  temperature below about 30 °C. The temperature of the water bath may be adjusted
  by adding cold water or by reheating. (Boiling tubes may be rinsed and reused.)

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[5]

**(b)** The rate of reaction for each experiment can be represented by the following.

'rate' = 
$$\frac{1000}{\text{reaction time in seconds}}$$

Complete the following table for each of your experiments. The mean temperature is the average of the initial and final temperature for the experiment.

mean temperature / °C	'rate'

I II III IV V V VI

[6]

[Turn over

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(d)	the state of the s	For Examiner's Use
(e)	(i) Which of your experiments has the greatest percentage error in timing?	
(-)	(,) This is your oxportation as the greatest personning.	
	(ii) Calculate the percentage error in (i). You may assume that the error in measuring the time for a reaction is ±0.5 seconds.	
	error in <b>(i)</b> = % [2]	
(f)	A student had difficulty in drawing a line of best fit. Identify a source of error in the experimental <b>procedure</b> . Do not include any errors involving the precision of apparatus.	
	[1]	
(g)	Suggest a modification that could be used to reduce this error.	
	[1]	
(h)	Using <b>FA 1</b> , <b>FA 2</b> and distilled water, describe how you could investigate the effect of <b>concentration</b> of potassium peroxydisulfate on the rate of reaction.	
	[3]	
	[Total: 24]	

### 2 Qualitative analysis

At each stage of any test you are to record details of the following.

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- · 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.

**FA 3**, **FA 4** and **FA 5** are aqueous solutions containing one cation and one anion. One of these solutions is a dilute acid and this is the only acid present.

(a)	(1)	identify the dilute acid. You may not use indicator paper.
		reagent

(ii) Use this reagent to test all three solutions and record your results in an appropriate form in the space below.

I	
II	
III	
IV	
V	

(iii) From your observations in (ii), identify which solution is the dilute acid.

FA ..... is the dilute acid.

[5]

(b)	The acid you have identified in (a)(iii) is dilute sulfuric acid.
	Complete the following table.

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test	observations
To 1 cm depth of <b>FA 3</b> in a test-tube, add 1 cm depth of <b>FA 4</b> , then	
add excess hydrochloric acid.	
To 1 cm depth of <b>FA 4</b> in a test-tube, add 1 cm depth of <b>FA 5</b> , then	
add excess hydrochloric acid.	
To 1 cm depth of <b>FA 5</b> in a test-tube, add 1 cm depth of <b>FA 3</b> , then	
add excess hydrochloric acid.	

I	
II	
III	
IV	

(c) For the two unidentified solutions, complete the following table.

test	observations	
	FA	FA
To 1 cm depth of unknown in a boiling tube, add NaOH(aq)		
warm the tube carefully		

[2]

[4]

(d)	tested in (c	;), giving the relev	a), (b) and (c), identify the ions present in the two stant evidence for each. If you have not been able to lain why the evidence obtained was insufficient.		For Examiner's Use
	FA	cation	evidence		
					I
					II
		anion	evidence		III
					IV
	FA	cation	evidence		
		anion	evidence		
	••••••			[4]	
(e)		ne aqueous anion its identification?	ns was a bromide, what would be the minimum	evidence	
				[1]	
			ŗ	Total: 161	

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# **Qualitative Analysis Notes**

*Key:* [ppt. = precipitate]

# 1 Reactions of aqueous cations

i	reaction with		
ion	NaOH(aq)	NH <sub>3</sub> (aq)	
aluminium, Al <sup>3+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH <sub>4</sub> +(aq)	no ppt. ammonia produced on heating	_	
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.	
calcium, Ca <sup>2+</sup> (aq)	white ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.	
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess	
copper(II), Cu <sup>2+</sup> (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution	
iron(II), Fe <sup>2+</sup> (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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess	
lead(II), Pb <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
magnesium, Mg <sup>2+</sup> (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess	
manganese(II), Mn <sup>2+</sup> (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 <sup>2+</sup> (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 <sub>2</sub> liberated by dilute acids
CO <sub>3</sub> <sup>2-</sup>	
chromate(VI),	yellow solution turns orange with H+(aq);
$CrO_4^{2-}(aq)$	gives yellow ppt. with Ba <sup>2+</sup> (aq);
010 <sub>4</sub> (aq)	gives bright yellow ppt. with Pb <sup>2+</sup> (aq)
chloride,	gives white ppt. with Ag <sup>+</sup> (aq) (soluble in NH <sub>3</sub> (aq));
C <i>l</i> <sup>-</sup> (aq)	gives white ppt. with Pb <sup>2+</sup> (aq)
bromide,	gives cream ppt. with Ag+(aq) (partially soluble in NH3(aq));
Br <sup>-</sup> (aq)	gives white ppt. with Pb <sup>2+</sup> (aq)
iodide,	gives yellow ppt. with Ag+(aq) (insoluble in NH <sub>3</sub> (aq));
I <sup>-</sup> (aq)	gives yellow ppt. with Pb <sup>2+</sup> (aq)
nitrate,	$\mathrm{NH_{3}}$ liberated on heating with $\mathrm{OH^{-}}(\mathrm{aq})$ and $\mathrm{A}\mathit{l}$ foil
NO <sub>3</sub> <sup>-</sup> (aq)	
nitrite,	$\mathrm{NH_3}$ liberated on heating with $\mathrm{OH^-}(\mathrm{aq})$ and $\mathrm{A}\mathit{l}$ foil,
NO <sub>2</sub> <sup>-</sup> (aq)	NO liberated by dilute acids (colourless NO → (pale) brown NO <sub>2</sub> in air)
sulfate,	gives white ppt. with Ba <sup>2+</sup> (aq) or with Pb <sup>2+</sup> (aq) (insoluble in excess dilute
SO <sub>4</sub> <sup>2-</sup> (aq)	strong acids)
sulfite,	SO <sub>2</sub> liberated with dilute acids;
SO <sub>3</sub> <sup>2-</sup> (aq)	gives white ppt. with Ba <sup>2+</sup> (aq) (soluble in excess dilute strong acids)

# 3 Tests for gases

gas	test and test result
ammonia, NH <sub>3</sub>	turns damp red litmus paper blue
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater (ppt. dissolves with excess CO <sub>2</sub> )
chlorine, Cl <sub>2</sub>	bleaches damp litmus paper
hydrogen, H <sub>2</sub>	"pops" with a lighted splint
oxygen, O <sub>2</sub>	relights a glowing splint
sulfur dioxide, SO <sub>2</sub>	turns potassium dichromate(VI) (aq) from orange to green