Cambridge International AS & A Level	Cambridge International Examinations Cambridge International Advanced Subsidiary and Advar	nced Level				
CANDIDATE NAME						
CENTRE NUMBER	CANDIE					
CHEMISTRY			9701/33			
Paper 3 Advar	ced Practical Skills 1	October/No	ovember 2016			
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Additional Mate	swer on the Question Paper.					
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	e examination, fasten all your work securely together. marks is given in brackets [] at the end of each question or	Labo	ratory			
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	This document consists of 11 printed pages and 1 blank	page.				

1 Hydrogen peroxide, H₂O₂, can be oxidised to give oxygen, O₂. This reaction happens rapidly in the presence of acidified potassium manganate(VII), KMnO₄.

 $2MnO_4^{-}(aq) + 5H_2O_2(aq) + 6H^{+}(aq) \rightarrow 2Mn^{2+}(aq) + 5O_2(g) + 8H_2O(I)$

You will determine the concentration of a solution of hydrogen peroxide. You will first dilute the solution and then carry out a titration using acidified potassium manganate(VII), $KMnO_4$.

FA 1 is aqueous hydrogen peroxide, H_2O_2 . **FA 2** is 0.0200 mol dm⁻³ potassium manganate(VII), KMnO₄. **FA 3** is 1.0 mol dm⁻³ sulfuric acid, H_2SO_4 .

(a) Method

Dilution

- Pipette 25.0 cm³ of **FA 1** into the 250 cm³ volumetric flask.
- Add distilled water to make 250 cm³ of solution and shake the flask thoroughly.
- Label this diluted solution of hydrogen peroxide FA 4.

Titration

- Fill the burette with **FA 2**.
- Rinse the pipette thoroughly with distilled water and then with a little FA 4.
- Pipette 25.0 cm³ of **FA 4** into a conical flask.
- Use the measuring cylinder to add 25 cm³ of **FA 3** to the conical flask.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is cm³.

- 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 a suitable form below all of your burette readings and the volume of **FA 2** added in each accurate titration.

Keep FA 3 and FA 4 for use in Question 2.

[7]

(b) From your accurate titration results, obtain a suitable value for the volume of **FA 2** to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 4** required cm³ of **FA 2**. [1]

(c) Calculations

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

(i) Calculate the number of moles of potassium manganate(VII) present in the volume of **FA 2** calculated in (b).

moles of $KMnO_4$ = mol

(ii) Use your answer to (i) and the equation at the top of page 2 to calculate the number of moles of hydrogen peroxide present in 25.0 cm³ of FA 4.

moles of H_2O_2 = mol

(iii) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 4**.

concentration of H_2O_2 in **FA 4 =** mol dm⁻³

(iv) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 1**.

concentration of H_2O_2 in **FA 1** = mol dm⁻³ [4]

[Total: 12]

2 In **Question 1**, hydrogen peroxide was oxidised to form oxygen. Hydrogen peroxide can also be reduced to form water. This occurs, for example, when hydrogen peroxide, H₂O₂, reacts with iodide ions, I⁻(aq), to form iodine, I₂(aq).

 $H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2H_2O(I)$

The rate of this reaction can be measured by adding acidified hydrogen peroxide, H_2O_2 , to a mixture of iodide ions, I^- , thiosulfate ions, $S_2O_3^{2-}$, and starch indicator. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.

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

When all the thiosulfate has reacted, the iodine then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes the reaction mixture to turn blue-black.

In this experiment you will measure two reaction times and use one of these to calculate the concentration of the thiosulfate ions in **FA 6**.

FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 . **FA 4** is the diluted hydrogen peroxide, H_2O_2 , you prepared in **1(a)**. **FA 5** is 1.0 mol dm^{-3} potassium iodide, KI. **FA 6** is aqueous sodium thiosulfate, $Na_2S_2O_3$. starch indicator

Read through the instructions carefully before starting any practical work.

(a) Method

- Use the measuring cylinder to transfer 20 cm³ of **FA 3** into the 100 cm³ beaker.
- Use the measuring cylinder to add 10 cm³ of **FA 4** into the 100 cm³ beaker.
- Rinse the measuring cylinder with distilled water and drain.
- Use the measuring cylinder to transfer 20 cm³ of **FA 5** into the 250 cm³ beaker.
- Use the measuring cylinder to transfer 20 cm³ of **FA 6** into the 250 cm³ beaker.
- Use the measuring cylinder to transfer 10 cm³ of starch indicator into the 250 cm³ beaker.
- Add the contents of the 100 cm³ beaker to the 250 cm³ beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second.
- Rinse both beakers and drain.

Keep all FA solutions and starch indicator for use in (c).

reaction time =s [2]

(b) Calculations

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

(i) The expression for the rate of reaction is shown.

rate = <u>concentration of iodine at the reaction time</u> reaction time

5

The concentration of iodine at the reaction time is the concentration of iodine that would have been present at the time the blue-black colour appeared if no thiosulfate ions had been added.

In this experiment, you should assume the rate = 2.61×10^{-5} mol dm⁻³ s⁻¹.

Use this value for the rate to calculate the concentration of iodine that would have been present at the reaction time if no thiosulfate ions had been added.

concentration of $I_{\rm 2}$ = mol dm $^{\rm -3}$

(ii) Use your answer to (i) and the total reaction volume, to calculate the number of moles of iodine that would have been present at the reaction time if no thiosulfate ions had been added.

(If you were unable to calculate an answer to (i), you should use the value of 1.32×10^{-3} mol dm⁻³. This may not be the correct value.)

moles of I_2 = mol

(iii) Calculate the number of moles of thiosulfate ions that reacted with the moles of I₂, calculated in (ii).

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

moles of $S_2 O_3^{2-}$ = mol

(iv) Use your answer to (iii) to calculate the concentration of thiosulfate ions in FA 6.

concentration of $S_2O_3^{2-}$ in **FA 6** = mol dm⁻³ [4]

- (c) Repeat the experiment in (a) using the following quantities of each reagent.
 - add to the 100 cm³ beaker 20 cm³ of FA 3 10 cm³ of FA 4
 - add to the 250 cm³ beaker
 - 20 cm³ of **FA 5** 10 cm³ of **FA 6** 10 cm³ of starch indicator 10 cm³ of distilled water
 - Add the contents of the 100 cm³ beaker to the 250 cm³ beaker and start timing immediately.
 - Stir the mixture once and place the beaker on a white tile.
 - Stop timing as soon as the solution turns blue-black.
 - Record this reaction time to the nearest second.

reaction time =s [1]

(d) (i) Explain the relationship between the value of the reaction time in (a) and the value of the reaction time in (c).

(ii) A student states that the error in the **total volume** of the reaction mixture in (a) is the same as the error in the **total volume** of the reaction mixture in (c).

State whether or not you agree with the student and explain your answer.

[4]

[Total: 11]

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. 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.

(a) FA 7, FA 8, FA 9 and FA 10 are solutions that each contain a metal cation from those listed in the Qualitative Analysis Notes on page 10.
Carry out tests using reagents that will allow you to identify which metal ions are present.
Draw a single table to record your observations.
Complete the table below with your conclusions.

solution	FA 7	FA 8	FA 9	FA 10
metal ion present				

(b) Carry out the following test and record your observations.

test	observations
To a 1 cm depth of FA 10 in a boiling tube add a 0.5 cm depth of 20 'vol' hydrogen peroxide, then add one drop of aqueous sodium hydroxide.	

[2]

(c) FA 11 is a solid that contains one anion from those listed in the Qualitative Analysis Notes on page 11.

Place all of **FA 11** into a boiling tube and add a 3 cm depth of distilled water. Stir until all the solid has dissolved.

(i) Carry out the following test and record your observations.

test	observations
To a 1 cm depth of the solution of FA 11 in a test-tube add dilute sulfuric acid.	

(ii) Use your observations from (i) to identify the anion in FA 11.

The anion is

(iii) Use the remaining solution of FA 11 in the boiling tube to carry out a further test to support your identification of the anion. Record details of this test.

[Total: 17]

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Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	react	tion with
ion	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH₄⁺(aq)	no ppt. ammonia produced on heating	_
barium, Ba²⁺(aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca²⁺(aq)	white ppt. with high [Ca²+(aq)]	no ppt.
chromium(III), Cr³⁺(aq)	grey-green ppt. soluble in excess	grey-green ppt. insoluble in excess
copper(II), Cu²⁺(aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe²+(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), Fe³+(aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg²⁺(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn²⁺(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, Zn²+(aq)	white ppt. soluble in excess	white ppt. soluble in excess

2 Reactions of anions

ion	reaction
carbonate, CO ₃ ²⁻	CO ₂ liberated by dilute acids
chloride, C <i>l⁻</i> (aq)	gives white ppt. with Ag ⁺ (aq) (soluble in $NH_3(aq)$)
bromide, Br⁻(aq)	gives cream ppt. with Ag ⁺ (aq) (partially soluble in $NH_3(aq)$)
iodide, I⁻(aq)	gives yellow ppt. with Ag ⁺ (aq) (insoluble in NH ₃ (aq))
nitrate, NO₃⁻(aq)	NH_3 liberated on heating with $OH^-(aq)$ and Al foil
nitrite, NO₂⁻(aq)	NH_3 liberated on heating with OH ⁻ (aq) and A <i>l</i> 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 acids)
sulfite, SO ₃ ^{2–} (aq)	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

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		18	2	He	helium 4.0	10	Ne	neon 20.2	18	Ar	argon 39.9	36	Ϋ́	krypton 83.8	54	Xe	xenon 131.3	86	Rn	radon -										
		16 17				6	ш	fluorine 19.0	17	Cl	chlorine 35.5	35	Ъ	bromine 79.9	53	Ι	iodine 126.9	85	At	astatine -					71	Lu	Iutetium 175.0	103	Ļ	lawrencium
						∞	0	oxygen 16.0	16	S	sulfur 32.1	34	Se	selenium 79.0	52	Те	tellurium 127.6	84	Ро	polonium –	116	L<	livermorium -		70	γb	ytterbium 173.1	102	No	nobelium
		15				7	z	nitrogen 14.0	15	٩	phosphorus 31.0	33	As	arsenic 74.9	51	Sb	antimony 121.8	83	Bi	bismuth 209.0					69	Тп	thulium 168.9	101	Md	mendelevium
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							lo	ISS			9	24	ŗ	chromium 52.0	42	Mo	molybdenum 95.9	74	8	tungsten 183.8	106	Sg	seaborgium -		60	ΡŊ	neodymium 144.4	92		uranium
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