

CANDIDATE
NAME

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CANDIDATE
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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

October/November 2015

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
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.
A copy of the Periodic Table is printed on page 12.

Qualitative Analysis Notes are printed on pages 10 and 11.

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.

- 1 In this experiment you will determine the ionic equation for the reaction of acidified potassium manganate(VII) with potassium iodide. Excess potassium iodide is used and the reaction produces iodine. The amount of iodine produced is measured by titration with sodium thiosulfate.

FA 1 is $0.0180 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 2 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4 .

FA 3 is $0.500 \text{ mol dm}^{-3}$ potassium iodide, KI.

FA 4 is $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

starch indicator

(a) Method

- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 2** to the conical flask.
- Use the measuring cylinder to add 20 cm^3 of **FA 3** to the conical flask.
- Fill the burette with **FA 4**.
- Carry out a rough titration. When the colour of the mixture becomes yellow/orange, add a few drops of starch indicator. Then titrate until the mixture goes colourless.
- Record all your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make sure 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 4** added in each accurate titration.

Keep FA 1 and FA 2 for use in Question 3 and FA 4 for use in Question 2.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b)** From your accurate titration results, obtain a suitable value for the volume of **FA 4** to be used in your calculations.
Show clearly how you have obtained this value.

Volume of **FA 4** required is cm^3 . [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 sodium thiosulfate in the volume of **FA 4** calculated in (b).

moles of $\text{Na}_2\text{S}_2\text{O}_3 = \dots\dots\dots$ mol

- (ii) Use the equation below to calculate the number of moles of iodine that reacted with the sodium thiosulfate in the titration.



moles of $\text{I}_2 = \dots\dots\dots$ mol

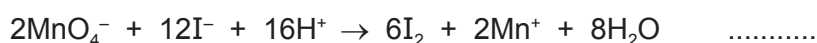
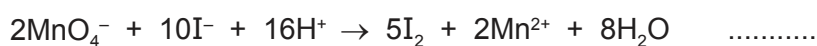
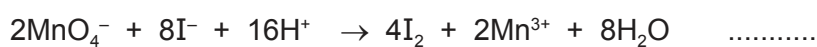
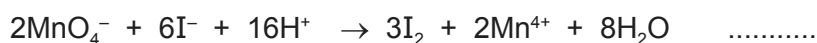
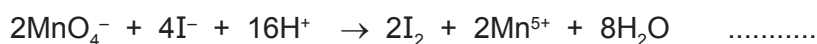
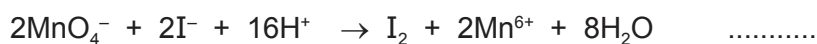
- (iii) Use information on page 2 to calculate the number of moles of potassium manganate(VII) in **FA 1** used in the titration.

moles of $\text{KMnO}_4 = \dots\dots\dots$ mol

- (iv) From your answers to (ii) and (iii), calculate the number of moles of iodine produced by the reaction of **2.00** moles of potassium manganate(VII) with excess potassium iodide.

moles $\text{I}_2 = \dots\dots\dots$ mol

- (v) Using your answer to (iv), put a tick next to the ionic equation that represents the reaction between **FA 1** and **FA 3**.



(vi) Prove that the iodide ion has been oxidised in the equation that you selected in (v).

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[5]

(d) (i) The error in calibration of the pipette you used is $\pm 0.06 \text{ cm}^3$.
Calculate the percentage error when measuring **FA 1**, using the pipette.

percentage error = %

(ii) A student suggested that the experiment would be more accurate if a pipette was used to measure solution **FA 3**.
State and explain whether you agree with the student.

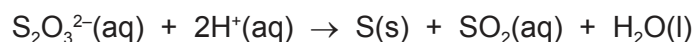
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[2]

[Total: 15]

- 2 In this experiment you will investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid is affected by the concentration of the acid.

When aqueous thiosulfate ions react with hydrogen ions, H^+ , in any acid, a pale yellow precipitate of sulfur is formed. The ionic equation for this reaction is given below.



The rate of the reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur.

FA 4 is 0.10 mol dm^{-3} sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 5 is 0.20 mol dm^{-3} hydrochloric acid, HCl .

(a) Method

Record **all** your measurements, in an appropriate form, in the space below.

Experiment 1

- Use the larger measuring cylinder to transfer 40 cm^3 of **FA 4** into the 100 cm^3 beaker.
- Rinse the larger measuring cylinder thoroughly with water, then add 30 cm^3 of **FA 5** to the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Look down through the solution in the beaker at the print on the insert.
- Stop timing as soon as the precipitate of sulfur makes the print on the insert invisible.
- Record the reaction time to the **nearest second**.
- Empty and rinse the 100 cm^3 beaker.
- Dry the outside of the beaker ready for Experiment 2.

Experiment 2

- Rinse the larger measuring cylinder, then use it to transfer 40 cm^3 of **FA 4** into the 100 cm^3 beaker.
- Use the smaller measuring cylinder to add 10 cm^3 of distilled water to the beaker.
- Use the same measuring cylinder to add 20 cm^3 of **FA 5** to the mixture in the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Stop timing as soon as the print on the insert becomes invisible.
- Record the reaction time to the **nearest second**.
- Empty and rinse the 100 cm^3 beaker.
- Dry the outside of the beaker ready for Experiment 3.

Experiment 3

- Carry out the reaction using a mixture of 40 cm^3 of **FA 4**, 20 cm^3 of distilled water and 10 cm^3 of **FA 5**.
- Measure and record the reaction time to the **nearest second**.

I	
II	
III	
IV	

[4]

- (b) (i) The 'rate of reaction' can be represented by the formula below.

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

Use this formula to calculate the 'rate of reaction' for Experiments 1 and 3.
Give the unit.

'rate of reaction' for Experiment 1 unit

'rate of reaction' for Experiment 3 unit

- (ii) Calculate the initial concentrations of hydrochloric acid in the reaction mixtures in Experiments 1 and 3.

initial concentration of HCl in Experiment 1 = mol dm⁻³

initial concentration of HCl in Experiment 3 = mol dm⁻³

- (iii) How is the 'rate of reaction' affected by the concentration of hydrochloric acid in the mixture?

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.....

- (iv) Predict how the reaction time measured in Experiment 1 would have been affected if the experiment had been carried out using 0.20 mol dm⁻³ sulfuric acid instead of 0.20 mol dm⁻³ hydrochloric acid.

Explain your answer.

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- (v) Predict how the reaction time measured in Experiment 3 would have been affected if the experiment had been carried out in a 250 cm³ beaker instead of a 100 cm³ beaker.

Explain your answer.

.....
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[5]

[Total: 9]

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 6** is a sodium compound containing one anion listed on page 11.

Dissolve the **FA 6** provided in about 15 cm³ of distilled water in a boiling tube. Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>
<p>(i) To a 1cm depth of the solution of FA 6 in a test-tube, add a few drops of aqueous barium chloride or aqueous barium nitrate, then</p> <hr style="border-top: 1px dashed black;"/> <p>add dilute hydrochloric acid.</p>	
<p>(ii) To a 1cm depth of the solution of FA 6 in a test-tube, add an equal volume of aqueous hydrogen peroxide, then</p> <hr style="border-top: 1px dashed black;"/> <p>add a few drops of aqueous barium chloride or aqueous barium nitrate, then</p> <hr style="border-top: 1px dashed black;"/> <p>add dilute hydrochloric acid.</p>	

<i>test</i>	<i>observations</i>
<p>(iii) To a 1 cm depth of the solution of FA 6 in a boiling tube, add an equal volume of FA 2, sulfuric acid, then</p> <p>heat the mixture gently and cautiously.</p>	
<p>(iv) To a 1 cm depth of the solution of FA 6 in a test-tube, add an equal volume of aqueous sodium hydroxide, then</p> <p>add a few drops of FA 1, aqueous potassium manganate(VII), then</p> <p>add FA 2, sulfuric acid.</p>	

(v) Identify the anion in **FA 6**, and state **one** piece of evidence for your identification.

anion

evidence

.....

.....

(vi) Give the chemical equation for the reaction between **FA 6** and hydrogen peroxide, H_2O_2 , in test (ii). State symbols are **not** required.

.....

[7]

- (b) **FA 7**, **FA 8**, **FA 9** and **FA 10** each contain one cation from the list on page 10. You will attempt to identify the cations by testing with aqueous sodium hydroxide and aqueous ammonia. In each case, use a 1 cm depth of the solution in a test-tube.

(i) Complete the table below.

test	observations			
	FA 7	FA 8	FA 9	FA 10
add sodium hydroxide				
add aqueous ammonia				

- (ii) Use your observations to identify, as far as possible, the cation present in each solution. If alternative identities are possible, state this clearly.

FA 7 cation

FA 8 cation

FA 9 cation

FA 10 cation

- (iii) Give the ionic equation for the reaction of **one** of your cations with a few drops of sodium hydroxide. State symbols are **not** required.

.....

- (iv) The precipitates obtained when alkalis are added to solutions of certain cations are sometimes difficult to see. Suggest how, using no additional apparatus, the experiment could be repeated in a way that would make these precipitates more visible.

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[9]

[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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
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 manganate(VII) from purple to colourless

The Periodic Table of the Elements

		Group																																																																																	
I	II	III	IV	V	VI	VII	0																																																																												
6.9 Li Lithium 3	9.0 Be Beryllium 4	1.0 H Hydrogen 1	10.8 B Boron 5	12.0 C Carbon 6	14.0 N Nitrogen 7	16.0 O Oxygen 8	19.0 F Fluorine 9	20.2 Ne Neon 10	23.0 Na Sodium 11	24.3 Mg Magnesium 12	27.0 Al Aluminium 13	28.1 Si Silicon 14	31.0 P Phosphorus 15	32.1 S Sulfur 16	35.5 Cl Chlorine 17	39.9 Ar Argon 18	39.1 K Potassium 19	40.1 Ca Calcium 20	45.0 Sc Scandium 21	47.9 Ti Titanium 22	50.9 V Vanadium 23	52.0 Cr Chromium 24	54.9 Mn Manganese 25	55.8 Fe Iron 26	58.9 Co Cobalt 27	58.7 Ni Nickel 28	63.5 Cu Copper 29	65.4 Zn Zinc 30	68.7 Ga Gallium 31	72.6 Ge Germanium 32	74.9 As Arsenic 33	79.0 Se Selenium 34	79.9 Br Bromine 35	83.8 Kr Krypton 36	85.5 Rb Rubidium 37	87.6 Sr Strontium 38	88.9 Y Yttrium 39	91.2 Zr Zirconium 40	92.9 Nb Niobium 41	95.9 Mo Molybdenum 42	98.9 Tc Technetium 43	101 Ru Ruthenium 44	103 Rh Rhodium 45	106 Pd Palladium 46	108 Ag Silver 47	112 Cd Cadmium 48	115 In Indium 49	119 Sn Tin 50	122 Sb Antimony 51	127 I Iodine 53	131 Xe Xenon 54	133 Cs Caesium 55	137 Ba Barium 56	139 La Lanthanum 57	178 Hf Hafnium 72	181 Ta Tantalum 73	186 Re Rhenium 75	188 W Tungsten 74	190 Os Osmium 76	192 Ir Iridium 77	195 Pt Platinum 78	197 Au Gold 79	201 Hg Mercury 80	204 Tl Thallium 81	207 Pb Lead 82	209 Bi Bismuth 83	210 Po Polonium 84	210 At Astatine 85	223 Fr Francium 87	226 Ra Radium 88	227 Ac Actinium 89	† 104 Rf Rutherfordium † 104	105 Db Dubnium 105	106 Sg Seaborgium 106	107 Bh Bohrium 107	108 Hs Hassium 108	109 Mt Meitnerium 109	110 Uun Ununnilium 110	111 Uuu Unununium 111	112 Uub Ununbium 112	114 Uuq Ununquadium 114	116 Uuh Ununhexium 116	118 Uuo Ununoctium 118

*58-71 Lanthanides
†90-103 Actinides

Key

a	X
b	

a = relative atomic mass †
X = atomic symbol
b = proton (atomic) number

140 Ce Cerium 58	141 Pr Praseodymium 59	144 Nd Neodymium 60	146 Pm Promethium 61	150 Sm Samarium 62	152 Eu Europium 63	157 Gd Gadolinium 64	159 Tb Terbium 65	163 Dy Dysprosium 66	165 Ho Holmium 67	167 Er Erbium 68	169 Tm Thulium 69	173 Yb Ytterbium 70	175 Lu Lutetium 71
90 Th Thorium 90	91 Pa Protactinium 91	92 U Uranium 92	93 Np Neptunium 93	94 Pu Plutonium 94	95 Am Americium 95	96 Cm Curium 96	97 Bk Berkelium 97	98 Cf Californium 98	99 Es Einsteinium 99	100 Fm Fermium 100	101 Md Mendelevium 101	102 No Nobelium 102	103 Lr Lawrencium 103