Cambridge Assessment International Education

Cambridge International AS & A Level

CANDIDATE NAME	

CENTRE NUMBER						CANDIDATE NUMBER					

0710657857

CHEMISTRY **9701/33**

Paper 3 Advanced Practical Skills 1 **May/June 2023**

2 hours

You must answer on the question paper.

You will need: The materials and apparatus listed in the confidential instructions
Insert (enclosed)

INSTRUCTIONS

- Answer **all** questions.
- Use a black or dark blue pen. You may use an HB pencil for any diagrams or graphs.
- Write your name, centre number and candidate number in the boxes at the top of the page.
- Write your answer to each question in the space provided.
- Do **not** use an erasable pen or correction fluid.
- Do **not** write on any bar codes.
- You may use a calculator.
- You should show all your working and use appropriate units.

Session
Laboratory

INFORMATION

- The total mark for this paper is 40.
- The number of marks for each question or part question is shown in brackets [ ].
- The Periodic Table is printed in the question paper.
- Important values, constants and standards are printed in the question paper.
- Notes for use in qualitative analysis are provided in the question paper.
- The insert contains additional resources referred to in the questions.

For Examiner's Use	
1	
2	
3	
Total	

This document has **12** pages.

Quantitative Analysis

Read through the whole method before starting any practical work. Where appropriate, prepare a table for your results in the space provided.

Show the precision of the apparatus you used in the data you record.

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

1 A redox reaction takes place between hydroxylamine, NH_2OH, and the iron(III) ion, Fe^{3+}, in acidic conditions. The iron(III) ion is reduced to an iron(II) ion, Fe^{2+}. The reaction is slow at room temperature but is complete in a few minutes at 100 °C. The reaction is shown by one of the following equations.

equation 1 $NH_2OH(aq) + Fe^{3+}(aq) \rightarrow Fe^{2+}(aq) + H^+(aq) + ½N_2(g) + H_2O(l)$

equation 2 $NH_2OH(aq) + 2Fe^{3+}(aq) \rightarrow 2Fe^{2+}(aq) + ½N_2O(g) + 2H^+(aq) + ½H_2O(l)$

equation 3 $NH_2OH(aq) + 3Fe^{3+}(aq) \rightarrow 3Fe^{2+}(aq) + NO(g) + 3H^+(aq)$

You will carry out a titration to determine which of equations 1, 2 or 3 best represents the reaction. The iron(II) ions formed in the reaction with the hydroxylamine are oxidised by manganate(VII) ions.

$$MnO_4^-(aq) + 5Fe^{2+}(aq) + 8H^+(aq) \rightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 4H_2O(l)$$

FA 1 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 2 is a solution prepared by boiling a 1.00 dm^3 aqueous mixture containing 3.30 g of hydroxylamine hydrochloride, $NH_2OH•HCl$, excess iron(III) chloride, $FeCl_3$, and excess sulfuric acid. Any water lost by evaporation was replaced after cooling.
FA 3 is dilute sulfuric acid.

Assume that one mole of hydroxylamine hydrochloride gives one mole of hydroxylamine in solution.

(a) Method

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 2** into a conical flask.
- Use the 25 cm^3 measuring cylinder to add 10 cm^3 of **FA 3** into the same conical flask.
- Perform a rough titration and record 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, all your burette readings and the volume of **FA 1** added in each accurate titration.

Keep FA 3 for use in Question 2(a).

Results

I	
II	
III	
IV	
V	
VI	
VII	

[7]

(b) From your accurate titration results, calculate a suitable mean value to use in your calculations. Show clearly how you obtain the mean value.

25.0 cm^3 of **FA 2** required .... cm^3 of **FA 1**. [1]

(c) **Calculations**

(i) Calculate the amount, in mol, of potassium manganate(VII) present in the volume of **FA 1** in **(b)**.

amount of $KMnO_4$ = ... mol [1]

(ii) Use your answer to **(c)(i)** to calculate the amount, in mol, of iron(II) ions in 25.0 cm^3 of solution **FA 2**.

amount of Fe^{2+} = ... mol [1]

(iii) Calculate the amount, in mol, of hydroxylamine hydrochloride that has reacted in the **FA 2** pipetted into the conical flask. Show your working.

amount of $NH_2OH{\bullet}HCl$ = ... mol [2]

(iv) Use your answer to **(c)(iii)** to deduce which of the three suggested equations corresponds to your results. Show your working.

The correct equation number is .... [1]

[Total: 13]

2 The reaction between thiosulfate ions and hydrogen ions produces a precipitate of sulfur. When the concentration of hydrogen ions, $[H^+(aq)]$, is kept constant, the rate is proportional to one of the following:

- the concentration of thiosulfate ions, $[S_2O_3^{2-}(aq)]$
- the square of the concentration of thiosulfate ions, $[S_2O_3^{2-}(aq)]^2$.

You will determine which relationship is correct by mixing solutions of sodium thiosulfate and sulfuric acid. You will measure the time taken for a fixed amount of sulfur to be precipitated.

Throughout these experiments care must be taken to avoid inhaling any SO_2 gas that is produced. It is very important that as soon as each experiment is complete, the contents of the beaker are emptied into the quenching bath and the beaker is rinsed thoroughly.

FA 3 is $1.00\,mol\,dm^{-3}$ sulfuric acid, H_2SO_4.
FA 4 is $0.100\,mol\,dm^{-3}$ sodium thiosulfate, $Na_2S_2O_3$.

(a) Method

Experiment 1

- Use the $50\,cm^3$ measuring cylinder to transfer $50.0\,cm^3$ of **FA 4** into the $100\,cm^3$ beaker.
- Use the $25\,cm^3$ measuring cylinder to measure $10.0\,cm^3$ of **FA 3**.
- Add **FA 3** to **FA 4** in the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on the printed insert.
- View the printing on the insert from above, through the solution.
- Stop timing when the print on the insert becomes obscured.
- Record this reaction time to the nearest second in Table 2.1.
- Empty the contents of the beaker into the quenching bath.
- Rinse and dry the beaker and glass rod so they are ready for use in Experiment 2.

Experiment 2

- Use the $50\,cm^3$ measuring cylinder to transfer $30.0\,cm^3$ of **FA 4** into the $100\,cm^3$ beaker.
- Use the same measuring cylinder to add $20.0\,cm^3$ of distilled water to the same beaker.
- Use the $25\,cm^3$ measuring cylinder to measure $10.0\,cm^3$ of **FA 3**.
- Add **FA 3** to the mixture of **FA 4** and distilled water in the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on the printed insert.
- View the printing on the insert from above, through the solution.
- Stop timing when the print on the insert becomes obscured.
- Record this reaction time to the nearest second in Table 2.1.
- Empty the contents of the beaker into the quenching bath.
- Rinse and dry the beaker and glass rod so they are ready for use in Experiment 3.

Experiment 3

- Carry out **one** further experiment to investigate how the reaction time changes with a different volume of **FA 4**.

  Do **not** use a volume of **FA 4** that is less than $20.0\,cm^3$.

  Record your results in Table 2.1.

Results

Table 2.1

experiment	volume / cm^3			reaction time / s
	FA 3	**FA 4**	distilled water	
1	10.0	50.0	0	
2	10.0	30.0	20.0	
3				

I	
II	
III	
IV	

[4]

(b) Calculations

(i) Complete Table 2.2. Use the following expression to calculate the rate.

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

Table 2.2

experiment	concentration of **FA 4** / $mol\,dm^{-3}$	rate / s^{-1}
1		
2		
3		

[2]

(ii) Use data from two of your experiments to deduce which relationship for the rate is correct. Place a tick next to the statement in Table 2.3 which most accurately fits your results.
Show your working.

Table 2.3

The rate is proportional to $[S_2O_3^{2-}(aq)]$.	
The rate is proportional to $[S_2O_3^{2-}(aq)]^2$.	

[2]

(c) The uncertainty in a 50 cm^3 measuring cylinder is ±0.5 cm^3.

Calculate the maximum percentage error in the volume of **FA 4** in Experiment 2.

maximum percentage error in volume of **FA 4** = ...% [1]

(d) Suggest improvements to your method and the processing of the results that would allow you to make a more reliable conclusion in **(b)(ii)**.

Do **not** suggest changes to the apparatus or to the concentrations of **FA 3** and **FA 4**.

Do **not** carry out any of your suggestions.

....

....

....

....

.... [3]

[Total: 12]

Qualitative Analysis

For each test you should record all your observations in the spaces provided.

Examples of observations include:
- colour changes seen
- the formation of any precipitate and its solubility (where appropriate) in an excess of the reagent added
- the formation of any gas and its identification (where appropriate) by a suitable test.

You should record clearly at what stage in a test an observation is made.

Where no change is observed you should write 'no change'.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

If any solution is warmed, a boiling tube must be used.

Rinse and reuse test-tubes and boiling tubes where possible.

No additional tests should be attempted.

3 **FA 5** is a solid mixture of two salts and contains two different anions. None of the ions present contains nitrogen.

(a) **(i)** Place a small spatula measure of **FA 5** in a hard-glass test-tube. Heat the test-tube, gently at first, then more strongly, for a total of approximately 2 minutes. Then allow the test-tube to cool.
Record your observations.

....

....

.... [2]

(ii) Place a large spatula measure of **FA 5** in a boiling tube. Add an approximately 3 cm depth of distilled water and shake the tube and contents. Record your observations.

Keep the contents of the test-tube for use in (a)(iii).

....

....

.... [2]

(iii) Put 1 cm depth of the solution from **(a)(ii)** in a test-tube. Add aqueous sodium hydroxide.
Record your observations.

.... [1]

(b) **(i)** Put 3 cm depth of dilute nitric acid in a test-tube. Carefully add a spatula measure of **FA 5**. Record your observations.

Keep the contents of the test-tube for use in (b)(iii).

....

....

.... [2]

(ii) Suggest what your observations in **(a)(ii)** tell you about the identity of one of the cations in **FA 5**.

.... [1]

(iii) Select reagents for further tests to identify any anion present in **FA 5**.
Carry out your tests and record your reagents, conditions and observations in the space below.

[2]

(iv) From your observations in **(b)(i)** and **(b)(iii)**, deduce the formulae of the two anions present in **FA 5**.

anions		

[1]

(v) Write an ionic equation for one reaction occurring in **(b)(i)** or **(b)(iii)**.
Include state symbols.

.... [1]

(c) **FA 6** is a solution of a salt. One of the two ions contains a nitrogen atom. Both ions are listed in the Qualitative analysis notes.

Select reagents to identify the ion containing the nitrogen atom. Use a 1 cm depth of **FA 6** in a boiling tube to carry out your tests. Record your tests, results and conclusion in the space below.

[3]

[Total: 15]

Qualitative analysis notes

1 Reactions of cations

cation	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 warming	–
barium, Ba^{2+}(aq)	faint white ppt. is observed unless [Ba^{2+}(aq)] is very low	no ppt.
calcium, Ca^{2+}(aq)	white ppt. unless [Ca^{2+}(aq)] is very low	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	pale 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

anion	reaction
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br^-(aq)	gives cream / off-white ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, I^-(aq)	gives pale yellow ppt. with Ag^+(aq) (insoluble in NH_3(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; decolourises acidified aqueous $KMnO_4$
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acids); gives white ppt. with high [Ca^{2+}(aq)]
sulfite, SO_3^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acids); decolourises acidified aqueous $KMnO_4$
thiosulfate, $S_2O_3^{2-}$(aq)	gives off-white / pale yellow ppt. slowly with H^+

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
hydrogen, H_2	‘pops’ with a lighted splint
oxygen, O_2	relights a glowing splint

4 Tests for elements

element	test and test result
iodine, I_2	gives blue-black colour on addition of starch solution

Important values, constants and standards

molar gas constant	$R = 8.31\,J\,K^{-1}\,mol^{-1}$
Faraday constant	$F = 9.65 \times 10^{4}\,C\,mol^{-1}$
Avogadro constant	$L = 6.022 \times 10^{23}\,mol^{-1}$
electronic charge	$e = -1.60 \times 10^{-19}\,C$
molar volume of gas	$V_m = 22.4\,dm^3\,mol^{-1}$ at s.t.p. (101 kPa and 273 K) $V_m = 24.0\,dm^3\,mol^{-1}$ at room conditions
ionic product of water	$K_w = 1.00 \times 10^{-14}\,mol^2\,dm^{-6}$ (at 298 K (25 °C))
specific heat capacity of water	$c = 4.18\,kJ\,kg^{-1}\,K^{-1}$ ($4.18\,J\,g^{-1}\,K^{-1}$)

The Periodic Table of Elements

Key

atomic number
atomic symbol
name
relative atomic mass

Group 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
							1 H hydrogen 1.0										2 He helium 4.0
3 Li lithium 6.9	4 Be beryllium 9.0											5 B boron 10.8	6 C carbon 12.0	7 N nitrogen 14.0	8 O oxygen 16.0	9 F fluorine 19.0	10 Ne neon 20.2
11 Na sodium 23.0	12 Mg magnesium 24.3											13 Al aluminium 27.0	14 Si silicon 28.1	15 P phosphorus 31.0	16 S sulfur 32.1	17 Cl chlorine 35.5	18 Ar argon 39.9
19 K potassium 39.1	20 Ca calcium 40.1	21 Sc scandium 45.0	22 Ti titanium 47.9	23 V vanadium 50.9	24 Cr chromium 52.0	25 Mn manganese 54.9	26 Fe iron 55.8	27 Co cobalt 58.9	28 Ni nickel 58.7	29 Cu copper 63.5	30 Zn zinc 65.4	31 Ga gallium 69.7	32 Ge germanium 72.6	33 As arsenic 74.9	34 Se selenium 79.0	35 Br bromine 79.9	36 Kr krypton 83.8
37 Rb rubidium 85.5	38 Sr strontium 87.6	39 Y yttrium 88.9	40 Zr zirconium 91.2	41 Nb niobium 92.9	42 Mo molybdenum 95.9	43 Tc technetium –	44 Ru ruthenium 101.1	45 Rh rhodium 102.9	46 Pd palladium 106.4	47 Ag silver 107.9	48 Cd cadmium 112.4	49 In indium 114.8	50 Sn tin 118.7	51 Sb antimony 121.8	52 Te tellurium 127.6	53 I iodine 126.9	54 Xe xenon 131.3
55 Cs caesium 132.9	56 Ba barium 137.3	57–71 lanthanoids	72 Hf hafnium 178.5	73 Ta tantalum 180.9	74 W tungsten 183.8	75 Re rhenium 186.2	76 Os osmium 190.2	77 Ir iridium 192.2	78 Pt platinum 195.1	79 Au gold 197.0	80 Hg mercury 200.6	81 Tl thallium 204.4	82 Pb lead 207.2	83 Bi bismuth 209.0	84 Po polonium –	85 At astatine –	86 Rn radon –
87 Fr francium –	88 Ra radium –	89–103 actinoids	104 Rf rutherfordium –	105 Db dubnium –	106 Sg seaborgium –	107 Bh bohrium –	108 Hs hassium –	109 Mt meitnerium –	110 Ds darmstadtium –	111 Rg roentgenium –	112 Cn copernicium –	113 Nh nihonium –	114 Fl flerovium –	115 Mc moscovium –	116 Lv livermorium –	117 Ts tennessine –	118 Og oganesson –

lanthanoids	57 La lanthanum 138.9	58 Ce cerium 140.1	59 Pr praseodymium 140.9	60 Nd neodymium 144.4	61 Pm promethium –	62 Sm samarium 150.4	63 Eu europium 152.0	64 Gd gadolinium 157.3	65 Tb terbium 158.9	66 Dy dysprosium 162.5	67 Ho holmium 164.9	68 Er erbium 167.3	69 Tm thulium 168.9	70 Yb ytterbium 173.1	71 Lu lutetium 175.0
actinoids	89 Ac actinium –	90 Th thorium 232.0	91 Pa protactinium 231.0	92 U uranium 238.0	93 Np neptunium –	94 Pu plutonium –	95 Am americium –	96 Cm curium –	97 Bk berkelium –	98 Cf californium –	99 Es einsteinium –	100 Fm fermium –	101 Md mendelevium –	102 No nobelium –	103 Lr lawrencium –