



Cambridge International AS & A Level

CANDIDATE
NAME

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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.

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.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

This document has **12** pages.

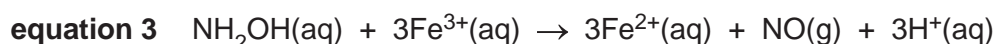
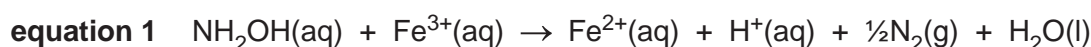
Quantitative Analysis

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

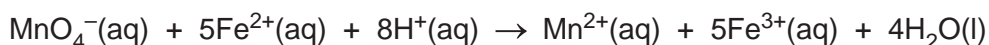
Show the precision of the apparatus you used in the data you record.

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.



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.



FA 1 is $0.0200\text{ mol dm}^{-3}$ 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, $\text{NH}_2\text{OH}\cdot\text{HCl}$, 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 cm³ of **FA 2** required cm³ 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 cm³ 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 $\text{NH}_2\text{OH}\cdot\text{HCl}$ = 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]

[Turn over

- 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 ₂ O ₃ ²⁻ (aq)].	
The rate is proportional to [S ₂ O ₃ ²⁻ (aq)] ² .	

[2]

6

- (c) The uncertainty in a 50 cm³ measuring cylinder is ± 0.5 cm³.

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

The Periodic Table of Elements

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

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
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 —

actinoids