



Cambridge International AS & A Level

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CHEMISTRY

9701/33

Paper 3 Advanced Practical Skills 1

May/June 2022

2 hours

You must answer on the question paper.

You will need: The materials and apparatus listed in the confidential instructions

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.

Session

Laboratory

For Examiner's Use

1

2

3

Total

This document has **12** pages. Any blank pages are indicated.



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 Acids donate protons, H^+ , in aqueous solution. The number of moles of H^+ donated per mole of acid is the **proticity** of the acid. In this experiment, you will carry out a titration to determine the proticity of phosphoric acid, H_3PO_4 , when it reacts with sodium hydroxide, NaOH .

FA 1 is aqueous phosphoric acid, containing $6.86 \text{ g dm}^{-3} \text{ H}_3\text{PO}_4$.

FA 2 is $0.150 \text{ mol dm}^{-3}$ sodium hydroxide, NaOH .

FA 3 is thymolphthalein indicator.

(a) Method

- Fill the burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Add a few drops of **FA 3**.
- 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 below all your burette readings and the volume of **FA 2** added in each accurate titration.

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 obtained the mean value.

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

(c) Calculations

- (i) Calculate the amount, in mol, of sodium hydroxide present in the volume of **FA 2** calculated in (b).

amount of NaOH = mol [1]

- (ii) Use the information on page 2 to calculate the amount, in mol, of phosphoric acid present in 25.0 cm³ of **FA 1**.

amount of H₃PO₄ = mol [1]

- (iii) Deduce whether phosphoric acid behaves as a monoprotic, diprotic or triprotic acid in this titration. Explain your reasoning.

H₃PO₄ is aprotic acid.

explanation

.....
 [1]

- (iv) Give the equation for this reaction of phosphoric acid, H₃PO₄, with sodium hydroxide.

..... [1]

- (d) (i) A student uses a pipette that is labelled 25.0 ± 0.06 cm³ to measure **FA 1**.

Calculate the maximum percentage error in the volume of **FA 1**. Show your working.

maximum percentage error =% [1]

- (ii) The student suggests it would be more accurate to measure the volume of **FA 1** with a burette instead of the pipette.

State whether you agree with the student. Explain your answer.

.....
 [1]

[Total: 14]

- 2 In this experiment you will identify the metal, **M**, in a metal carbonate, **MCO₃**, by thermal decomposition.



FA 4 is the metal carbonate, **MCO₃**.

(a) Method

- Weigh the empty crucible with its lid. Record the mass.
- Transfer all the **FA 4** from the container into the crucible.
- Weigh the crucible, lid and **FA 4**. Record the mass.
- Calculate and record the mass of **FA 4** used.
- Place the crucible and contents on a pipe-clay triangle.
- Heat the crucible gently, with the lid on, for approximately 1 minute.
- Heat strongly, with the lid off, for a further 4 minutes.
- Replace the lid and leave the crucible to cool for at least 5 minutes.

During the cooling period, you may wish to begin work on Question 3.

- When the crucible has cooled, weigh the crucible with its lid and contents. Record the mass.
- Heat strongly, with the lid off, for a further 2 minutes.
- Replace the lid and leave the crucible to cool for at least 5 minutes.
- When the crucible has cooled, reweigh the crucible with its lid and contents. Record the mass.
- Calculate and record the total loss of mass and the mass of residue obtained.
- This residue is **FA 5**.

Keep FA 5 for use in 2(d).

Results

I	
II	
III	
IV	
V	

[5]

(b) Calculations

- (i) Calculate the amount, in mol, of carbon dioxide given off in your experiment.

amount of CO₂ = mol [1]

- (ii) Calculate the relative formula mass, M_r , of MCO_3 .

M_r of MCO_3 = [1]

- (iii) From your results, deduce the identity of **M**.
Show your reasoning.

M is = [1]

- (c) A student carries out the same procedure, using the same mass of solid. However, the student uses the basic carbonate, $\text{MCO}_3 \cdot \text{M}(\text{OH})_2$, instead of the pure carbonate, MCO_3 .

When the metal hydroxide part of the basic carbonate decomposes, metal oxide and steam are produced. The metal carbonate part decomposes in the usual way.

State how the loss of mass from the student's solid compares with the loss of mass you obtained when you carried out your experiment. Explain your reasoning.

.....
..... [2]

- (d) Use a spatula to transfer a small quantity of your cold residue, **FA 5**, into a test-tube. Add about a 1 cm depth of dilute hydrochloric acid to the **FA 5** in the test-tube.

Record what you observe.

.....
.....

State whether or not the thermal decomposition of MCO_3 is complete.

Justify your answer based on your observations.

.....
.....
..... [2]

[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 (a) Solutions **FA 6** and **FA 7** each contain one cation and one anion. All the ions are listed in the Qualitative analysis notes.

- (i) Carry out the following tests, using a 1 cm depth of **FA 6** or **FA 7** in a test-tube for each test. Complete the table below.

Table 3.1

<i>test</i>	<i>observations</i>	
	FA 6	FA 7
Test 1 Add an equal volume of aqueous potassium iodide, then		
add excess aqueous sodium thiosulfate.		
Test 2 Add a small spatula measure of zinc powder. Leave the mixture to stand.		
Test 3 Add a few drops of aqueous silver nitrate.		
Test 4 Add aqueous sodium hydroxide.		

- (ii) Construct an ionic equation for **one** of the reactions taking place in **Test 2**. Include state symbols.

..... [1]

- (b) **FA 8** contains one anion and one cation. One of these ions contains nitrogen. Both ions are listed in the Qualitative analysis notes.

- (i) Transfer a small spatula measure of **FA 8** into a hard-glass test-tube. Heat the test-tube gently at the start, then strongly until no further change occurs. Leave the test-tube to cool.

Record all your observations.

.....
.....
.....
..... [2]

- (ii) Carry out further tests to identify **each** ion in **FA 8**.

Record, in a table in the space below, the reagents, conditions and observations for the tests that positively identify **each** ion.
You may wish to use the following page for rough working.

Deduce the chemical formula of **FA 8**.

You **must** use a boiling tube if any liquid is heated.

The formula of **FA 8** is [6]

[Total: 14]

Use this page for any rough working.

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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	$R = 8.31 \text{ J K}^{-1} \text{ mol}^{-1}$
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 $\text{J g}^{-1} \text{ K}^{-1}$)

The Periodic Table of Elements

[illegible]

57	La lanthanum	58	Ce cerium	59	Pr praseodymium	60	Nd neodymium	61	Pm promethium	62	Sm samarium	63	Eu europium	64	Gd gadolinium	65	Tb terbium	66	Dy dysprosium	67	Ho holmium	68	Er erbium	69	Tm thulium	70	Yb ytterbium	71	Lu lutetium
	138.9	140.1	140.9	144.4	—	150.4	152.0	157.3	158.9	162.5	164.9	167.3	168.9	173.1	175.0														
89	Ac actinium	90	Th thorium	91	Pa protactinium	92	U uranium	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
	227.0	232.0	231.0	238.0	237.0	239.0	238.0	237.0	237.0	239.0	241.0	243.0	247.0	251.0	252.0	257.1	259.1	267.1	268.1	269.1	277.1	288.1	289.1	293.1	294.1	294.1	261.1		

lanthanoids

actinoids