

# Cambridge International AS & A Level

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4	Paper 3 Advance	ed Pra	ctical	Skill	s 1			Oct	ober/ľ	Noven	nber	2023
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#### 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 You will determine the value of x in hydrated sodium carbonate,  $Na_2CO_3 \cdot xH_2O$ . x is **not** an integer.

You will carry out two methods to determine the value of  $\mathbf{x}$ . Each method involves sodium carbonate reacting with excess hydrochloric acid to release carbon dioxide.

 $Na_2CO_3 \cdot \mathbf{x}H_2O(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + CO_2(g) + \mathbf{x}H_2O(l)$ 

#### (a) Experiment 1

You will measure the volume of carbon dioxide released when hydrated sodium carbonate reacts with excess hydrochloric acid.

**FA 1** is  $0.500 \text{ mol dm}^{-3}$  hydrochloric acid, HC*l*. **FA 2** is hydrated sodium carbonate, Na<sub>2</sub>CO<sub>3</sub>•**x**H<sub>2</sub>O.

#### Method

- Weigh the container with **FA 2**. Record the mass.
- Fill the tub with water to a depth of approximately 5 cm.
- Fill the 250 cm<sup>3</sup> measuring cylinder completely with water. Holding a piece of paper towel firmly over the top, invert the measuring cylinder and place it in the water in the tub.
- Remove the paper towel and clamp the inverted measuring cylinder so the open end is in the water just above the base of the tub.
- Using the 50 cm<sup>3</sup> measuring cylinder, transfer 50.0 cm<sup>3</sup> of FA 1 into the flask labelled Z. Check that the bung fits tightly into the neck of flask Z, clamp flask Z and place the end of the delivery tube into the inverted 250 cm<sup>3</sup> measuring cylinder.
- Remove the bung from the neck of the flask. Tip all the **FA 2** from the container into the acid in the flask and replace the bung **immediately**. Remove the flask from the clamp and swirl it to mix the contents. You may need to shake the flask quite vigorously until the gas formed starts to collect in the measuring cylinder.
- Return the flask to the clamp. Leave for several minutes, shaking the flask occasionally.
- Weigh the container with any residual **FA 2**. Record the mass.
- Calculate the mass of **FA 2** added to the flask. Record the mass.
- When no more gas is collected, measure the final volume of gas in the measuring cylinder. Record the volume.

#### Results

Ι	
II	
III	
[3]	

#### (b) Calculations

(i) Calculate the amount, in mol, of carbon dioxide collected in the measuring cylinder at room conditions.

amount of CO<sub>2</sub> = ..... mol

Hence deduce the amount, in mol, of sodium carbonate present in the **FA 2** you added in your experiment.

amount of  $Na_2CO_3 = \dots mol$  [1]

(ii) Use your answer to (b)(i) and the mass of hydrated sodium carbonate,  $Na_2CO_3 \cdot xH_2O$ , you used in **Experiment 1** to calculate the relative formula mass,  $M_r$ , of the  $Na_2CO_3 \cdot xH_2O$ .

 $M_{\rm r} {\rm of Na}_2 {\rm CO}_3 {}^{\bullet} {\bf x} {\rm H}_2 {\rm O} = \dots$ [1]

(iii) Use your answer to (b)(ii) to calculate the value of **x** in the Na<sub>2</sub>CO<sub>3</sub>•**x**H<sub>2</sub>O. Show your working.

- (c) A student suggests that it would be better to use hot water in the tub.
  - (i) State whether using hot water would be an improvement. Explain your answer.

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- ......[1]
- (ii) State the effect, if any, that using hot water would have on the value of **x** calculated.

#### (d) Experiment 2

You will carry out a titration to measure the volume of hydrochloric acid that neutralises an aqueous solution of hydrated sodium carbonate,  $Na_2CO_3 \cdot \mathbf{x}H_2O$ .

 $Na_2CO_3 \cdot \mathbf{x}H_2O(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + CO_2(g) + \mathbf{x}H_2O(l)$ 

**FA 3** is  $0.100 \text{ mol dm}^{-3}$  hydrochloric acid, HC*l*.

FA 4 is an aqueous solution containing 14.30 g dm<sup>-3</sup> of hydrated sodium carbonate, Na<sub>2</sub>CO<sub>3</sub>•xH<sub>2</sub>O.

FA 5 is bromophenol blue indicator.

#### Method

- Fill the burette with **FA 3**.
- Pipette 25.0 cm<sup>3</sup> of **FA 4** into a conical flask.
- Add a few drops of **FA 5**.
- Carry out a rough titration and record your burette readings in the space below.

The rough titre is ..... cm<sup>3</sup>.

- Carry out as many titrations as you think necessary to obtain consistent results.
- Make sure your 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 3** added in each accurate titration.

Ι	
II	
III	
IV	
V	
VI	
VII	

[7]

(e) 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<sup>3</sup> of **FA 4** required ..... cm<sup>3</sup> of **FA 3**. [1]

#### (f) Calculations

- (i) Give your answers to (f)(ii), (f)(iii) and (f)(iv) to an appropriate number of significant figures.
- (ii) Calculate the amount, in mol, of hydrochloric acid present in the volume of FA 3 you calculated in (e).

amount of HCl = ..... mol [1]

(iii) Use the equation for the neutralisation to deduce the amount, in mol, of sodium carbonate present in 25.0 cm<sup>3</sup> of Na<sub>2</sub>CO<sub>3</sub>•**x**H<sub>2</sub>O.

amount of Na<sub>2</sub>CO<sub>3</sub> = ..... mol

Hence calculate the amount, in mol, of sodium carbonate in  $1.00 \,\mathrm{dm^3}$  of  $\mathrm{Na_2CO_3} \cdot \mathbf{xH_2O}$ .

amount of  $Na_2CO_3$  in 1.00 dm<sup>3</sup> = ..... mol [1]

(iv) Calculate the value of **x** in the sample of  $Na_2CO_3 \cdot xH_2O$ . Show your working.

(g) The aqueous solution of Na<sub>2</sub>CO<sub>3</sub>•**x**H<sub>2</sub>O, **FA 4**, was prepared by weighing and dissolving the solid to make 1.00 dm<sup>3</sup> of solution.

Mass of container + Na <sub>2</sub> CO <sub>3</sub> • <b>x</b> H <sub>2</sub> O	= 32.509 g
Mass of empty container	= 18.209 g
Mass of Na <sub>2</sub> CO <sub>3</sub> • <b>x</b> H <sub>2</sub> O	= 14.300 g

(i) State the maximum uncertainty in a single balance reading for the balance used.

maximum uncertainty = ± ...... g

Calculate the maximum percentage uncertainty in this mass of  $Na_2CO_3 \cdot \mathbf{x}H_2O$ . Show your working.

maximum percentage uncertainty = ± ...... %
[1]

(ii) Using the method in **Experiment 2** a student calculated the relative formula mass,  $M_r$ , of Na<sub>2</sub>CO<sub>3</sub>•**x**H<sub>2</sub>O to be 242.2. Assume that the uncertainty in the mass of **FA 4** is the only source of error in the experiment.

Calculate the maximum value for the relative formula mass of FA 4.

[Total: 23]

#### 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

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• 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.

- 2 (a) FA 6, FA 7 and FA 8 are salts each of which contains nitrogen. Each of the nitrogen-containing ions is different and all are shown in the Qualitative analysis notes.
  - (i) List the nitrogen-containing ions for which you will test.

Select reagents to positively identify the nitrogen-containing ions in each salt. Record your tests and the results with each salt in a suitable table in the space below.

(ii) Use your observations in (a)(i) to determine the formulae of the nitrogen-containing ions present in FA 6, FA 7 and FA 8.

FA 6 ..... FA 7 ..... FA 8 ..... [1]

- (b) FA 9 and FA 10 contain the same element. You will identify this element by carrying out tests.
  - (i) Test 1

Heat a small spatula measure of **FA 9** in a hard-glass test-tube. Heat until the reaction stops.

After heating, leave the tube to cool and keep it for Test 2. You may wish to start (b)(ii) while you wait.

Record your observations.

Name one product of the reaction.

product .....

#### Test 2

To the cooled solid product of **Test 1**, add a 2–3 cm depth of distilled water. Shake the test-tube and then leave the contents to settle.

Record your observations.

.....

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- [3]
- (ii) To a **very small** spatula measure of **FA 9** in a test-tube, add about a 2 cm depth of dilute sulfuric acid and about a 2 cm depth of distilled water. Shake to dissolve the **FA 9** and produce **FA 9**(aq).

You will use **FA 9**(aq) in **Test 3** and **Test 4**.

Test 3

To a 1 cm depth of aqueous iron(II) sulfate in a test-tube, add a few drops of FA 9(aq).

Record your observations.

.....

	Test 4	
	To a 1 cm depth of aqueous potassium iodide in a test-tube, add a few drops of FA 9(a	aq).
	Record your observations.	
		[2]
		[~]
(iii)	To a small spatula measure of <b>FA 10</b> in a test-tube, add distilled water to dissolve <b>FA 10</b> . This solution is <b>FA 10</b> (aq).	the
	To a 1 cm depth of <b>FA 10</b> (aq), add aqueous sodium hydroxide.	
	Record your observations.	
		[2]
(iv)	Identify the element that is present in FA 9 and FA 10.	
	element	[1]

[Total: 17]

# Qualitative analysis notes

# 1 Reactions of cations

cation	reaction	on with
	NaOH(aq)	NH <sub>3</sub> (aq)
aluminium, Al <sup>3+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH <sub>4</sub> +(aq)	no ppt. ammonia produced on warming	-
barium, Ba <sup>2+</sup> (aq)	faint white ppt. is observed unless [Ba <sup>2+</sup> (aq)] is very low	no ppt.
calcium, Ca <sup>2+</sup> (aq)	white ppt. unless [Ca <sup>2+</sup> (aq)] is very low	no ppt.
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu <sup>2+</sup> (aq)	pale blue ppt. insoluble in excess	pale blue ppt. soluble in excess giving dark blue solution
iron(II), Fe <sup>2+</sup> (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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg <sup>2+</sup> (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn <sup>2+</sup> (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 <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. soluble in excess

# 2 Reactions of anions

anion	reaction
carbonate, CO <sub>3</sub> <sup>2-</sup>	CO <sub>2</sub> liberated by dilute acids
chloride, C1 <sup>-</sup> (aq)	gives white ppt. with Ag <sup>+</sup> (aq) (soluble in NH <sub>3</sub> (aq))
bromide, Br <sup>–</sup> (aq)	gives cream/off-white ppt. with Ag <sup>+</sup> (aq) (partially soluble in NH <sub>3</sub> (aq))
iodide, I <sup>_</sup> (aq)	gives pale yellow ppt. with $Ag^+(aq)$ (insoluble in $NH_3(aq)$ )
nitrate, NO <sub>3</sub> <sup>-</sup> (aq)	$NH_3$ liberated on heating with OH <sup>-</sup> (aq) and Al foil
nitrite, NO <sub>2</sub> <sup>-</sup> (aq)	$NH_3$ liberated on heating with OH <sup>-</sup> (aq) and A <i>l</i> foil; decolourises acidified aqueous KMnO <sub>4</sub>
sulfate, SO <sub>4</sub> <sup>2–</sup> (aq)	gives white ppt. with Ba <sup>2+</sup> (aq) (insoluble in excess dilute strong acids); gives white ppt. with high [Ca <sup>2+</sup> (aq)]
sulfite, SO <sub>3</sub> <sup>2–</sup> (aq)	gives white ppt. with Ba <sup>2+</sup> (aq) (soluble in excess dilute strong acids); decolourises acidified aqueous KMnO <sub>4</sub>
thiosulfate, S <sub>2</sub> O <sub>3</sub> <sup>2–</sup> (aq)	gives off-white/pale yellow ppt. slowly with H <sup>+</sup>

# 3 Tests for gases

gas	test and test result
ammonia, NH <sub>3</sub>	turns damp red litmus paper blue
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater
hydrogen, H <sub>2</sub>	'pops' with a lighted splint
oxygen, O <sub>2</sub>	relights a glowing splint

### 4 Tests for elements

element	test and test result
iodine, I <sub>2</sub>	gives blue-black colour on addition of starch solution

# Important values, constants and standards

$R = 8.31 \mathrm{J}\mathrm{K}^{-1}\mathrm{mol}^{-1}$
$F = 9.65 \times 10^4 \mathrm{C  mol^{-1}}$
$L = 6.022 \times 10^{23} \text{ mol}^{-1}$
$e = -1.60 \times 10^{-19} \mathrm{C}$
$V_{\rm m}$ = 22.4 dm <sup>3</sup> mol <sup>-1</sup> at s.t.p. (101 kPa and 273 K) $V_{\rm m}$ = 24.0 dm <sup>3</sup> mol <sup>-1</sup> at room conditions
$K_{\rm w}$ = 1.00 × 10 <sup>-14</sup> mol <sup>2</sup> dm <sup>-6</sup> (at 298K (25 °C))
$c = 4.18 \mathrm{kJ  kg^{-1}  K^{-1}} (4.18 \mathrm{J  g^{-1}  K^{-1}})$

							The Pe	riodic Tal	The Periodic Table of Elements	ments							
								Group	dno								
1	5											13	14	15	16	17	18
-							-										2
							т										He
				Key			hydrogen 1.0										helium 4.0
e	4			atomic number								5	9	7	8	6	10
	Be		atc	atomic symbol	bol							В	U	z	0	ш	Ne
6.9	beryllium 9.0		rela	name relative atomic mass	ISS							boron 10.8	carbon 12.0	nitrogen 14.0	oxygen 16.0	fluorine 19.0	neon 20.2
	12	-										13	14	15	16	17	18
	Mg											Al	<u>S</u>	٩	S	Cl	Ar
sodium m 23.0	magnesium 24.3	ო	4	5	9	7	Ø	6	10	11	12	aluminium 27.0	silicon 28.1	Чd	sulfur 32.1	chlorine 35.5	argon 39.9
	20		22	23	24	25	26	27	28	29	30	31	32		34	35	36
	Ca	Sc	F	>	ŗ	Mn	Fe	ပိ	ïZ	Cu	Zn	Ga	Ge		Se	Br	Кr
potassium 39.1	calcium 40.1	scandium 45.0	titanium 47.9	vanadium 50.9	chromium 52.0	manganese 54.9	iron 55.8	cobalt 58.9	nickel 58.7	copper 63.5	zinc 65.4	gallium 69.7	germanium 72.6	arsenic 74.9	selenium 79.0	bromine 79.9	krypton 83.8
-	38	39	40	41	42	43	4	45	46	47	48	49	50		52	53	5
	ي ا	≻	Zr	qN	Mo	Ч	Ru	RЪ	Pd	Ag	Cq	In	Sn	Sb	Те	Ι	Xe
rubidium 85.5	strontium 87.6	yttrium 88.9	zirconium 91.2	niobium 92.9	molybdenum 95.9	technetium -	ruthenium 101 1	102 9	palladium 106.4	silver 107 9	cadmium 112 4	indium 114.8	tin 118.7	antimony 121 R	tellurium 127 6	iodine 126.9	xenon 131.3
+	56	57-71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86
Cs	Ba	lanthanoids	Ηf	Та	8	Re	Os	Ir	Ŧ	Au	Ha	11	Pb	Ē	Ро	At	Rn
caesium 132.9	barium 137.3		hafnium 178.5	tantalum 180.9	tungsten 183.8	rhenium 186.2	osmium 190.2	iridium 192.2	platinum 195.1	gold 197.0	mercury 200.6	thallium 204.4	lead 207.2	bismuth 209.0	polonium –	astatine -	radon -
87	88	89-103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118
Fr	Ra	actinoids	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	ü	ЧN	Fl	Mc	۲<	Ч S	Őġ
francium -	radium -		rutherfordium —	dubnium –	seaborgium -	bohrium –	hassium -	meitnerium -	darmstadtium -	roentgenium -	copernicium -	nihonium –	flerovium -	moscovium	livermorium -	tennessine -	ogan esson -
		57	58	59		61	62	63	64	65	66	67	68	69	20	71	
lanthanoids	S	La	Ce	ŗ	ΡN	Pm	Sm	Еu	рд	Tb	Ŋ	Но	ц	Tm	γb	Lu	
		lanthanum 138.9	cerium 140.1	praseodymium 140.9	neodymium 144.4	promethium -	samarium 150.4	europium 152.0	gadolinium 157.3	terbium 158.9	dysprosium 162.5	holmium 164.9	erbium 167.3	thulium 168.9	ytterbium 173.1	lutetium 175.0	
		89	06	91		93	94	95	96	97	98	66	100	101	102	103	
actinoids		Ac	Th	Ра		dN	Pu	Am	СB	В	Ç	Es	ШШ	РМ	No	Ļ	
		actinium -	thorium 232.0	protactinium 231.0	uranium 238.0	neptunium -	pluton ium –	americium -	curium	berkelium -	californium -	einsteinium –	fermium -	mendelevium -	nobelium -	lawrencium -	

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