

Cambridge International AS & A Level

CANDIDATE NAME				
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CHEMISTRY				9701/31
Paper 3 Advanc	ed Practical Skills 1		October/N	ovember 2022
		listed in the confidential instructior	าร	
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INFORMATION			Ses	sion
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brackets [The Period]. lic Table is printed in the quest	ion paper.	Laboi	atory
 Important v question pation 	values, constants and standard	ds are printed in the		
	se in qualitative analysis are p	provided in the		
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This document has 16 pages. Any blank pages are indicated.

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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 Group 1 elements form salts with ethanedioic acid. These salts are ethanedioates and have the formula (COOM)₂•2H₂O, where **M** is the Group 1 metal.

Ethanedioate ions react with manganate(VII) ions as shown.

 $5(COO^{-})_{2}(aq) + 2MnO_{4}^{-}(aq) + 16H^{+}(aq) \rightarrow 10CO_{2}(g) + 2Mn^{2+}(aq) + 8H_{2}O(I)$

You will determine which metal is present in $(COOM)_2 \cdot 2H_2O$ by titrating a solution of this salt with manganate(VII) ions.

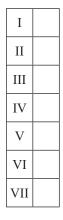
FA 1 is 10.14 g dm⁻³ aqueous hydrated ethanedioate of metal **M**, $(COOM)_2 \cdot 2H_2O$. **FA 2** is 0.0200 mol dm⁻³ potassium manganate(VII), KMnO₄. **FA 3** is 1 mol dm⁻³ sulfuric acid, H₂SO₄.

(a) Method

- Fill the burette with **FA 2**.
- Pipette 25.0 cm³ of **FA 1** into a conical flask.
- Use the measuring cylinder to add approximately 20 cm³ of **FA 3** into the conical flask.
- Place the conical flask on a tripod and gauze and heat carefully until the temperature of the solution is approximately 70°C.
- Remove the flame.
- Carefully lift the hot conical flask and place it on the white tile under the burette.
- During titrations, add **FA 2**, **slowly at first**, until a permanent pale pink colour is formed. The pink colour on initial addition may take several seconds to disappear.
- If the reaction mixture turns brown, reheat it to about 70 °C. If the brown colour disappears, continue with the titration. If the brown colour remains, discard the contents of the flask and begin a new titration.
- Perform a rough titration with **FA 2**. Record your burette readings in the space below.

The rough titre is cm³.

- 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.



[7]

(b) From your accurate titration results, calculate a suitable mean value to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 1** required cm³ of **FA 2**. [1]

(c) Calculations

- (i) Give your answers to (c)(ii), (c)(iii) and (c)(iv) to the appropriate number of significant figures.
- (ii) Calculate the amount, in mol, of manganate(VII) ions, MnO₄⁻, in the volume of **FA 2** calculated in (b).

amount of MnO_4^- = mol [1]

(iii) Calculate the amount, in mol, of ethanedioate ions that reacted with the manganate(VII) ions in (c)(ii).

amount of $(COO^{-})_2$ = mol

Hence calculate the concentration, in mol dm⁻³, of ethanedioate ions in **FA 1**.

concentration of $(COO^{-})_2$ = mol dm⁻³ [1]

(iv) Calculate the relative formula mass, M_r , of the hydrated ethanedioate, (COOM)₂•2H₂O.

 $M_{\rm r} = \dots$ [1]

(v) Identify M. Show your working.

M is [2]

(d) Explain why it is necessary to add FA 3 in each titration.

.....[1]

[Total: 15]

Question 2 starts on the next page.

5

2 Hydrated copper(II) sulfate, CuSO₄•5H₂O, can lose its water of crystallisation to form anhydrous copper(II) sulfate.

The enthalpy change for the dehydration of hydrated copper(II) sulfate is shown in the equation.

 $CuSO_4 \bullet 5H_2O(s) \rightarrow CuSO_4(s) + 5H_2O(l)$

You will carry out experiments to determine the enthalpy changes for the solution of hydrated and anhydrous copper(II) sulfate and then use Hess's law to determine the enthalpy change of dehydration.

FA 4 is hydrated copper(II) sulfate, $CuSO_4 \cdot 5H_2O$. **FA 5** is anhydrous copper(II) sulfate, $CuSO_4$.

(a) Determination of the enthalpy change of solution of hydrated copper(II) sulfate.

Method

- Weigh the container with **FA 4**. Record the mass.
- Support the cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25.0 cm³ of distilled water into the cup.
- Measure and record the temperature of the water.
- Tip all the FA 4 into the water and stir until the solid dissolves.
- Measure and record the lowest temperature reached.
- Rinse and dry the cup ready for the next experiment.
- Weigh the container with any residual **FA 4**. Record the mass.
- Calculate and record the change in temperature.
- Calculate and record the mass of **FA 4** used.

Results

(b) Calculations

(i) Calculate the energy change during this reaction.

energy change = J [1]

(ii) Calculate the amount, in mol, of hydrated copper(II) sulfate, **FA 4**, used in the experiment. Show your working.

amount of $CuSO_4 \cdot 5H_2O = \dots mol$ [1]

(iii) Calculate the enthalpy change, in kJ mol⁻¹, when 1.00 mol of hydrated copper(II) sulfate dissolves in water. This is the enthalpy of solution.

enthalpy change of solution = \dots kJ mol⁻¹ [1] sign value

(c) Determination of the enthalpy change of solution of anhydrous copper(II) sulfate.

Method

- Weigh the container with **FA 5**. Record the mass.
- Support the cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25.0 cm³ of distilled water into the cup.
- Measure and record the temperature of the water.
- Tip all the **FA 5** into the water and stir until the solid dissolves.
- Measure and record the highest temperature reached.
- Weigh the container with any residual **FA 5**. Record the mass.
- Calculate and record the change in temperature.
- Calculate and record the mass of **FA 5** used.

Results

[1]

(d) Calculations

(i) Calculate the enthalpy change, in kJ mol⁻¹, for the enthalpy change of solution of anhydrous copper(II) sulfate.

enthalpy change = kJ mol⁻¹ [2] sign value (ii) Use the enthalpy changes calculated in (b)(iii) and (d)(i) to calculate the enthalpy change of dehydration of hydrated copper(II) sulfate.

 $CuSO_4 \bullet 5H_2O(s) \rightarrow CuSO_4(s) + 5H_2O(l)$

Show clearly how you obtained your answer.

```
enthalpy change = ..... kJ mol<sup>-1</sup> [1]
sign value
```

(e) In the experiments in (a) and (c) you used the same method to determine the enthalpy change of solution of two solids.

Tick the box to indicate which statement is correct. Ignore the effect of differences in mass used.

The percentage error in (b)(iii) is less than the percentage error in (d)(i).	
The percentage errors in (b)(iii) and (d)(i) are equal.	
The percentage error in (b)(iii) is greater than the percentage error in (d)(i).	

Explain your choice.

[1] [Total: 10]

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) FA 6 is an aqueous solution that contains one cation and two anions. The three ions are listed in the Qualitative analysis notes.
 - (i) Carry out the following tests on **FA 6** and record your observations.

test	observations
Test 1 To a 1 cm depth of FA 6 in a boiling tube add aqueous sodium hydroxide, then	
heat gently.	
Test 2 To a 1 cm depth of FA 6 in a boiling tube add a 1 cm depth of aqueous sodium hydroxide and a piece of aluminium foil. Heat gently.	
Test 3 To a 1 cm depth of FA 6 in a test-tube add a few drops of hydrogen peroxide.	

(ii)	From your observations suggest two possible identities for the cation in FA 6 .	
	possible cations:	[1]
(iii)	Suggest a test that would allow you to determine which of the cations you suggested (a)(ii) is present in FA 6 .	in
	Carry out this test, record the result and hence identify the cation in FA 6 .	
	test	
	result	
	The cation present is	[2]
(iv)	From your observations in (a)(i) suggest two anions that could be present in FA 6 and gi their formulae.	ve
	possible anions: or or	[1]
(v)	Suggest an additional test that could be carried out to confirm the presence of one of t anions you suggested in (a)(iv) .	he
	Carry out this test, record the result and hence state the identity of the anion.	
	test	
	result	
	The anion present is	[2]

(b) Half fill the 250 cm³ beaker with water and heat to approximately 80 °C. Turn off the Bunsen burner. This will be your hot water bath.

FA 7 is an organic compound with an M_r between 40–57.

(i) Carry out **Test 2** and **Test 3** on **FA 7** and record your observations. The result for **Test 1** is shown in the table.

test	observations
Test 1 Add a small piece of sodium.	no change
Test 2 To a 0.5 cm depth of aqueous iodine in a test-tube add aqueous sodium hydroxide dropwise until the yellow colour just disappears. Then add a few drops of FA 7 and shake the test-tube. If no change is seen, warm the test-tube in your hot water bath.	
Test 3 To a 1 cm depth of FA 7 in a test-tube add a few drops of acidified potassium manganate(VII). Warm the test-tube in your hot water bath.	

[2]

(ii) Using the observations in (b)(i) suggest what can be deduced from each test about the functional groups present in **FA 7**.

	Test 1	
	Test 2	
	Test 3	[2]
(iii)	Use your deductions in (b)(ii) to suggest the identity of FA 7.	
	FA 7 is	[1]

[Total: 15]

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Qualitative analysis notes

1 Reactions of cations

cation	reaction	on with
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH₄⁺(aq)	no ppt. ammonia produced on warming	-
barium, Ba²⁺(aq)	faint white ppt. is observed unless [Ba²⁺(aq)] is very low	no ppt.
calcium, Ca²⁺(aq)	white ppt. unless [Ca²⁺(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), Cu²+(aq)	pale blue ppt. insoluble in excess	pale blue ppt. soluble in excess giving dark blue solution
iron(II), Fe²⁺(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), Fe³⁺(aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg²+(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn²⁺(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, Zn²⁺(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, C <i>l</i> ⁻(aq)	gives white ppt. with Ag⁺(aq) (soluble in NH₃(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 NH₃(aq))
nitrate, NO₃⁻(aq)	NH_3 liberated on heating with OH ⁻ (aq) and Al foil
nitrite, NO₂⁻(aq)	NH_3 liberated on heating with OH ⁻ (aq) and A <i>l</i> 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	$R = 8.31 \mathrm{J}\mathrm{K}^{-1}\mathrm{mol}^{-1}$
Faraday constant	$F = 9.65 \times 10^4 \mathrm{C}\mathrm{mol}^{-1}$
Avogadro constant	$L = 6.022 \times 10^{23} \mathrm{mol}^{-1}$
electronic charge	$e = -1.60 \times 10^{-19} C$
molar volume of gas	$V_{\rm m}$ = 22.4 dm ³ mol ⁻¹ at s.t.p. (101 kPa and 273 K) $V_{\rm m}$ = 24.0 dm ³ mol ⁻¹ at room conditions
ionic product of water	$K_{\rm w}$ = 1.00 × 10 ⁻¹⁴ mol ² dm ⁻⁶ (at 298 K (25 °C))
specific heat capacity of water	$c = 4.18 \mathrm{kJ} \mathrm{kg}^{-1} \mathrm{K}^{-1} $ (4.18 J g ⁻¹ K ⁻¹)

							The Pe	riodic Ta	The Periodic Table of Elements	ments							
								Grc	Group								
-	2											13	14	15	16	17	18
							-										5
							т										He
				Key			hydrogen 1.0										helium 4.0
n	4			atomic number								5	9	7	80	6	10
:	Be		ato	atomic symbol	lod							В	ပ	z	0	ш	Ne
lithium 6.9	beryllium 9.0		rels	name relative atomic mass	ISS							boron 10.8	carbon 12.0	nitrogen 14.0	oxygen 16.0	fluorine 19.0	neon 20.2
7	12											13	14	15	16	17	18
	Mg											Al	Si	٩	ი	Cl	Ar
sodium 23.0	magnesium 24.3	ю	4	5	9	7	8	0	10	11	12	aluminium 27.0	silicon 28.1	phosphorus 31.0	sulfur 32.1	chlorine 35.5	argon 39.9
	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
¥	Ca	Sc	F	>	ں د	Mn	Ъe	ပိ	Ī	Cu	Zn	Ga	Ge	As	Se	Ŗ	Ъ
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
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54
Rb	S	≻	Zr	qN	Mo	Ч	Ru	RЧ	Pd	Ag	ы	In	Sn	Sb	Чe	Ι	Xe
rubidium 85.5	strontium 87.6	yttrium 88.9	zirconium 91.2	niobium 92.9	molybdenum 95.9	technetium -	ruthenium 101.1	rhodium 102.9	palladium 106.4	silver 107.9	cadmium 112.4	indium 114.8	tin 118.7	antimony 121.8	tellurium 127.6	iodine 126.9	xenon 131.3
55	56	57-71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86
Cs	Ba	lanthanoids	Hf	Та	\geq	Re	Os	Ir	ħ	Au	Нg	11	Pb	Ξ	Ъо	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
ц	Ra	actinoids	Ъf	Db	Sg	Bh	Hs	Mt	Ds	Rg	ő	ЧN	F١	Mc	Ľ	Ts	Og
francium -	radium -		rutherfordium —	dubnium –	seaborgium -	bohrium –	hassium -	meitnerium -	darmstadtium -	roentgenium -	copernicium -	nihonium –	flerovium -	moscovium -	livermorium –	tennessine -	oganesson -
		57	58	59	60	61	62	63	64	65	66	67	68	69	20	71	
lanthanoids	sp	La	Ce	P	PN	Pm	Sm	Еu	Вd	Tb	Ď	Ч	ц	T	γb	Lu	
		lanthanum 138.9	cerium 140.1	praseodymium 140.9	ne	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	92	93	94	95	96	97	98	66	100	101	102	103	
actinoids		Ac	Th	Ра		ЧN	Pu	Am	CB	Ŗ	Ç	Es	Еm	Md	No	Ļ	
		actinium -	thorium 232.0	protactinium 231.0	uranium 238.0	neptunium -	plutonium –	americium -	curium –	berkelium -	californium -	einsteinium –	fermium -	mendelevium -	nobelium –	lawrencium –	

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