CANDIDATE NAME			
CENTRE NUMBER	CANDIE		
CHEMISTRY			9701/33
Paper 3 Advand	ced Practical Skills 1	October/No	vember 2015
Candidates ans Additional Mater	wer on the Question Paper. rials: As listed in the Confidential Instructions		2 hours
READ THESE I	NSTRUCTIONS FIRST		
Give details of t Write in dark blu You may use an Do not use stap	re number, candidate number and name on all the work you har he practical session and laboratory where appropriate, in the bo le or black pen. HB pencil for any diagrams or graphs. les, paper clips, glue or correction fluid. E IN ANY BARCODES.		
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	ysis Notes are printed on pages 10 and 11. is printed on page 12.	Sess	sion
	e examination, fasten all your work securely together. marks is given in brackets [] at the end of each question or	Labor	atory
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1 The formula of hydrated copper(II) sulfate is $CuSO_4$. xH_2O where x is the number of moles of water of crystallisation in one mole of salt. You will determine the value of x by titration.

When aqueous copper(II) ions react with aqueous iodide ions, I⁻, iodine is produced.

 $2Cu^{2+}(aq) + 4I^{-}(aq) \rightarrow I_2(aq) + 2CuI(s)$

The amount of iodine, I₂, produced can be found by titrating it with aqueous thiosulfate ions, S₂O₃²⁻.

 $2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^{-}(aq)$

FA 1 is aqueous $CuSO_4$. xH_2O containing 26.2 g dm⁻³. **FA 2** is 0.100 mol dm⁻³ sodium thiosulfate, $Na_2S_2O_3$. **FA 3** is aqueous potassium iodide, KI. starch indicator

(a) Method

- Pipette 25.0 cm³ of **FA 1** into a conical flask.
- Use the measuring cylinder to add 15 cm³ of **FA 3**, an excess of KI, to the conical flask. The solution will turn brown because iodine is formed.
- Fill the burette with **FA 2**.
- Add FA 2 from the burette until the colour of the mixture changes to pale brown.
- Add 10 drops of starch indicator. The mixture will turn blue-black.
- Continue adding **FA 2** from the burette until the dark colour suddenly disappears to leave an off-white solid. This is the end point of the titration.
- Carry out a rough titration and 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 certain 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.

Ι	
II	
III	
IV	
V	
VI	
VII	

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

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

(c) Calculations

Show your working and appropriate significant figures in **each** step of your calculations.

(i) Calculate the number of moles of thiosulfate ions present in the volume of FA 2 you have calculated in (b).

moles of $S_2O_3^{2-}$ = mol

(ii) Use your answer to (i), and the equations for the reactions involved, to deduce the number of moles of Cu²⁺ present in 25.0 cm³ of FA 1.

 $2Cu^{2+}(aq) + 4I^{-}(aq) \rightarrow I_{2}(aq) + 2CuI(s)$ $2S_{2}O_{3}^{2-}(aq) + I_{2}(aq) \rightarrow S_{4}O_{6}^{2-}(aq) + 2I^{-}(aq)$

moles of Cu^{2+} = mol

(iii) Use your answer to (ii) and the mass of $CuSO_4$.**x** H_2O present in the solution, to calculate the relative molecular mass, M_r , of $CuSO_4$.**x** H_2O .

 $M_{\rm r}$ of CuSO₄.**x**H₂O =

(iv) Determine the value of *x*. (Use data from the Periodic Table on page 12.)

X =

[6]

[Total: 14]

2 FA 4 is an impure sample of hydrated calcium chloride, $CaCl_2.2H_2O$. On heating, hydrated calcium chloride loses its water of crystallisation.

 $CaCl_2.2H_2O(s) \rightarrow CaCl_2(s) + 2H_2O(g)$

You will determine the purity of **FA 4** by measuring the loss in mass that occurs when it is heated. The impurity present in **FA 4** is not decomposed on heating.

(a) Method

You should read the instructions carefully before starting any practical work and draw a table for your results in the space below.

- Weigh a crucible and record its mass.
- Add between 1.80 g and 2.00 g of **FA 4** into the crucible.
- Reweigh the crucible and its contents and record the mass.
- Place the crucible on the pipe-clay triangle and heat gently for 1 minute and then strongly for a further 2 minutes.
- Allow the crucible and its contents to cool. Reweigh the crucible and contents and record the mass.
- Heat the crucible strongly for a further 2 minutes. Allow it to cool. Reweigh the crucible and contents and record the mass.
- Repeat the heating, cooling and weighing until you are satisfied that all the water of crystallisation has been removed.
- Calculate and record the mass of **FA 4** used and the total mass of water lost.

While you are waiting for the crucible to cool, you may wish to start work on Question 3.

Ι	
II	
III	
IV	
V	
VI	

5

(b) Calculations

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

(i) The percentage loss in mass on heating is defined as

 $\frac{\text{the loss in mass on heating}}{\text{the original mass}} \times 100.$

Calculate the percentage loss in mass of FA 4.

percentage loss in mass = %

(ii) Calculate the percentage loss in mass when **pure** hydrated calcium chloride, $CaCl_2.2H_2O$, is heated.

percentage loss in mass = %

(iii) Use your results to (i) and (ii) to calculate the percentage purity of FA 4, impure CaC l_2 .2H₂O.

percentage purity =%
[3]

(c) A student carried out this experiment using 2.60 g of FA 4.

Suggest whether this experiment would give a more accurate result for the percentage purity of **FA 4**. Explain your answer.

......[1]

(d) In your calculations you assumed that the impurity in FA 4 does not decompose on heating.

State how the percentage purity that you calculated in **(b)(iii)** would change if the impurity were to decompose on heating. Explain your answer.

......[1]

[Total: 11]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs. No additional tests for ions present should be attempted.

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

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

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

(a) (i) FA 5, FA 6 and FA 7 are aqueous solutions each containing one anion and one cation.

Carry out the experiments described below and record your observations for each solution in the table.

	FA 5	FA 6	FA 7
To a 1 cm depth in a test-tube, add a 1 cm depth of aqueous sodium carbonate.			
To a 1 cm depth in a test-tube, add a 1 cm depth of aqueous copper(II) sulfate.			
To a 1 cm depth in a test-tube, add a 1 cm depth of aqueous barium chloride or aqueous barium nitrate.			

- (ii) What ion is present in **both FA 6** and **FA 7**?
-
- (iii) The anion in **FA 5** is one of carbonate, chloride, nitrate or sulfate.

Which anion is present in FA 5?

.....

(iv) Write the ionic equation, including state symbols, for the reaction between **FA 5** and aqueous copper(II) sulfate.

.....

[7]

- (b) FA 8 contains two anions and two cations from the lists on pages 10 and 11.
 - To a 5 cm depth of distilled water in a boiling tube, add all the **FA 8**.
 - Shake the boiling tube thoroughly for one minute to make sure that no more of the solid will dissolve.
 - Filter the mixture into a clean boiling tube.
 - Place the filter funnel in a conical flask and wash the residue with a little distilled water.
 - Keep both filtrate and residue for tests (i) and (ii) below.

(i) Tests on the filtrate (the solution in the boiling tube)

Carry out the following tests and record your observations in the table below.

test	observations
To a 1 cm depth of the filtrate in a test-tube, add aqueous sodium hydroxide, then	
add aqueous hydrogen peroxide.	

(ii) Tests on the residue

Carry out the following tests and record your observations in the table below.

test	observations
Place the funnel containing the residue into a clean boiling tube. Pour approximately 5 cm ³ of dilute nitric acid onto the residue. Collect a 1 cm depth of solution in the boiling tube. Remove the funnel and return it to the conical flask.	
To this solution in the boiling tube, add aqueous sodium hydroxide.	

(iii)	Identify two cations present in FA 8 .
	cations present and
(iv)	Identify one anion present in FA 8 .
	anion present
(v)	Suggest what type of reaction is happening when hydrogen peroxide is added in test (b)(i) .

[8]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ian	reacti	ion with
ion	NaOH(aq)	NH ₃ (aq)
aluminium, A <i>l</i> ³+(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH₄⁺(aq)	no ppt. ammonia produced on heating	_
barium, Ba²+(aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca²+(aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr³+(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	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

ion	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 $NH_3(aq)$)
bromide, Br⁻(aq)	gives cream ppt. with Ag ⁺ (aq) (partially soluble in NH ₃ (aq))
iodide, I⁻(aq)	gives yellow ppt. with Ag ⁺ (aq) (insoluble in $NH_3(aq)$)
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; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO ₂ in air)
sulfate, SO ₄ ^{2–} (aq)	gives white ppt. with Ba ²⁺ (aq) (insoluble in excess dilute strong acids)
sulfite, SO ₃ ²-(aq)	SO ₂ liberated with dilute acids; gives white ppt. with Ba ²⁺ (aq) (soluble in excess dilute strong acids)

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 (ppt. dissolves with excess CO ₂)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H ₂	"pops" with a lighted splint
oxygen, O ₂	relights a glowing splint
sulfur dioxide, SO ₂	turns acidified aqueous potassium manganate(VII) from purple to colourless

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9701/33/O/N/15

4.0 Helium

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The Periodic Table of the Elements

Group

1.0 Hydrogen

=

2

PMT

6.9 Lithium	9.0 Ber yllium 4											10.8 Boron	12.0 Carbon	14.0 N Nitrogen	16.0 Oxygen 8	19.0 Fluorine	20.2 Neon 10
	24.3 Mg Magnesium 12											27.0 Al Aluminium 13	28.1 Si Silicon	31.0 Phosphorus 15	32.1 S Sultur 16	35.5 C1 Chlorine	39.9 Ar Argon
	40.1 Ca Calcium 20	45.0 Scandium 21	47.9 T ^{Titanium} 22	50.9 Vanadium 23	52.0 Cr Chromium 24	54.9 Manganese 25	55.8 Fe Iron	58.9 CO Cobalt 27	58.7 Ni Nickel	63.5 Cu Copper 29	65.4 Zn 2inc	69.7 Ga Gallium 31	72.6 Ge Germanium 32	74.9 AS Arsenic 33	79.0 Se Selenium 34	79.9 Br Bromine	83.8 Krypton 36
	87.6 Strontium 38	88.9 Yttrium 39	91.2 Zr Zirconium 40	92.9 Nb Niobium	95.9 Mo Molybdenum 42	Tc Technetium 43	101 Ru thenium 44	103 Rh Rhodium 45	106 Pd Palladium 46	108 Ag Silver	112 Cadmium 48	115 I n 149	119 Sn 50	122 Sb Antimony 51	128 Te ^{Tellunium} 52	127 	131 Xe 54
	137 Baa Barium 56	139 La Lanthanum 57 *	178 Hafnium 72	181 Ta ^{Tantalum} 73	184 V Tungsten 74	186 Re Rhenium 75	190 OS Osmium 76	192 r 1ridium 77	195 Pt Platinum 78	197 Au Gold 79		204 T 1 Thallium 81	207 Pb Lead	209 Bi Bismuth	Polonium 84	At Astatine 85	Rn Radon 86
1	Radium 88	AC Actinium 89	Rutherfordium 104	Dubnium 105	Sg ^{Seaborgium} 106	Bh Bohrium 107	Hssium 108	Mt Meitnerium 109	Ununnilium 110	Ununuium 111	Uub Ununbium 112		Ununquadium 114		Ununhexium 116		Ununoctium 118
1 1.2	*58-71 Lanthanides 190-103 Actinides	S S	*	140 Ce ^{Cerium}	141 Praseodymium 59	144 Neodymium 60	Promethium 61	150 Sa marium 62	152 Eu 63	157 Gd Gadolinium 64	159 Tb 65	163 Dysprosium 66	165 Holmium 67	167 Er 68	169 Tm 69	173 Yb 70	175 Lutetium 71
م	ت × ت ×	 a = relative atomic mass X = atomic symbol b = proton (atomic) number 		Thorium 90	Protactinium 91	Uranium 92	Neptunium 93	Pu Plutonium 94	Am Americium 95	Cm Curium 96	BK Berkelium 97	Cf Californium 98	ES Einsteinium 99	Fm Fermium 100	Md Mendelevium 101	Nobelium 102	Lr Lawrendium 103