CJC CJC CJC CJC CJC CJC	Catholic Junior College JC2 Preliminary Examinations Higher 2
CANDIDATE NAME	
CLASS	2T

## CHEMISTRY

Paper 4 Practical

9729/04 19 August 2019

2 hours 30 minutes

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

### READ THESE INSTRUCTIONS FIRST

Write your name and class in the boxes above.

Give details of the practical shift and laboratory, where appropriate, in the boxes provided.

Write in dark blue or black pen.

You may use an HB pencil for any diagrams or graphs.

Do not use staples, paper clips, glue or correction fluid.

Answer **all** questions in the spaces provided on the Question Paper.

The use of an approved scientific calculator is expected, where appropriate. You may lose marks if you do not show your working or if you do not use appropriate units.

Qualitative Analysis Notes are printed on pages 14 and 15.

At the end of the examination, fasten all your work securely together. The number of marks is given in brackets [ ] at the end of each question or part question.



For Examiner's Use		
1	16	
2	14	
3	13	
4	12	
Total	55	

This document consists of **15** printed pages and **1** blank page.

[Turn over

#### 1 Determination of the percentage by mass of iron(II) sulfate in an iron tablet

Iron tablets, taken for the prevention and treatment of iron deficiency, are health supplements readily available in pharmacies. These iron tablets contain iron(II) sulfate, which is a soluble form of iron. Assuming that all the iron in the tablets is in the form of Fe<sup>2+</sup>, it is possible to estimate the iron content by titration against potassium manganate(VII).

The equation for this reaction is

 $5Fe^{2+}(aq) + MnO_4^{-}(aq) + 8H^{+}(aq) \rightarrow 5Fe^{3+}(aq) + Mn^{2+}(aq) + 4H_2O(I)$ 

You are provided with the following.

FA 1 contains 5 iron tablets, crushed to a powder. FA 2 is 0.00500 mol dm<sup>-3</sup> potassium manganate(VII), KMnO<sub>4</sub>. 1.0 mol dm<sup>-3</sup> sulfuric acid, H<sub>2</sub>SO<sub>4</sub>. deionised water

In this experiment, you will determine the percentage of iron(II) sulfate heptahydrate,  $FeSO_{4.7}H_2O$ , present in the iron tablets **FA 1**.

#### (a) Preparation of FA 3, a solution of $FeSO_4.7H_2O$ .

- Use a measuring cylinder to transfer about 100 cm<sup>3</sup> of 1.0 mol dm<sup>-3</sup> sulfuric acid into a conical flask.
- Determine accurately the mass of the crushed iron tablets used, **FA1**, and record your readings in the space below.

mass of container + FA1 / g	m <sub>2</sub>
mass of container + residual FA1 / g	m <sub>1</sub>
mass of <b>FA1</b> used / g	<b>m</b> <sub>2</sub> – <b>m</b> <sub>1</sub>

mass of the crushed iron tablets used = ...... g

- Add all the crushed tablets, **FA 1**, to the sulfuric acid.
- Warm the mixture gently, do not overheat, and stir for about two minutes.
- Allow the flask to cool for around five minutes.
- Filter the mixture into a 250 cm<sup>3</sup> volumetric flask. Ensure that no solution is lost. <u>Note</u>: The filtration takes time. Proceed to Question 2 whilst the mixture is filtering.
- Wash out the conical flask with deionised water and add the washings through the filter into the volumetric flask.
- Make up the contents of the flask to the 250 cm<sup>3</sup> mark with deionised water. Stopper and mix the contents thoroughly to obtain a homogeneous solution.
- Label this solution **FA 3**.

#### (b) Titration of FA 3 with FA 2

- Fill the burette with FA 2.
- Pipette 25.0 cm<sup>3</sup> of **FA 3** into a conical flask.
- Use a measuring cylinder to add about 25.0 cm<sup>3</sup> of 1.0 mol dm<sup>-3</sup> sulfuric acid to the conical flask.
- Titrate with FA 2 until the appearance of a first permanent pale-pink colour.
- Record your titration results in the space below. Make certain that the recorded results show the precision of your practical work.
- Repeat the titration as many times as necessary to obtain reliable results.

#### **Titration Results**

	1	2
final burette reading / cm <sup>3</sup>		
initial burette reading / cm <sup>3</sup>		
volume of FA 2 used / cm <sup>3</sup>	<b>V</b> 1	<b>V</b> <sub>2</sub>
	<ul> <li>✓</li> </ul>	<ul> <li>✓</li> </ul>

[6]

From your titrations, obtain a suitable volume of **FA 2** to be used in your calculations. Show clearly how you obtained this volume.

average titre = 
$$\frac{v_1 + v_2}{2}$$
  
= ...... cm<sup>3</sup>

(c) (i) Calculate the amount of  $MnO_4^-$  ions contained in the mean titre.

mol of MnO<sub>4</sub><sup>-</sup> = 
$$cV = 0.00500 \times \frac{\text{average titre}}{1000}$$
  
= ..... mol

amount of  $MnO_4^-$  ions =..... [1]

(ii) Calculate the amount of  $Fe^{2+}$  ions that reacted with the  $MnO_4^-$  ions calculated in (c)(i).

```
mol of Fe<sup>2+</sup> reacted = 5 × mol of MnO<sub>4</sub><sup>-</sup>
= 5 × ans to (b)(i)
= ..... mol
```

(iii) Calculate the amount of  $Fe^{2+}$  ions in the five crushed tablets, **FA 1**.

```
mol of Fe<sup>2+</sup> in 5 crushed tablets = mol of Fe<sup>2+</sup> in 250 cm<sup>3</sup> solution
= \frac{250}{25.0} × ans to (b)(ii)
= ..... mol
```

(iv) Calculate the mass of FeSO<sub>4</sub>.7H<sub>2</sub>O in one tablet.
 [A<sub>r</sub>: Fe, 55.8; S, 32.1; O, 16.0; H, 1.0]

 $M_r$  of FeSO<sub>4</sub>.7H<sub>2</sub>O = 55.8 + 32.1 + 4(16.0) + 7[2(1.0) + 16.0] = 277.9 mass of FeSO<sub>4</sub>.7H<sub>2</sub>O in 5 tablets =  $nM_r$  = ans to (b)(iii) x 277.9 = ..... g ∴ mass of FeSO<sub>4</sub>.7H<sub>2</sub>O in 1 tablet =  $\frac{1}{5}$  x above ans = ..... g

- 5
- (v) Hence calculate the percentage by mass of FeSO<sub>4</sub>.7H<sub>2</sub>O in **one** tablet.

```
% by mass of FeSO<sub>4</sub>.7H<sub>2</sub>O in 1 tablet = \frac{5 \times \text{ans to (b)(iv)}}{\text{mass of FA1}} \times 100
= ......%
```

percentage by mass of FeSO<sub>4</sub>.7H<sub>2</sub>O in one tablet =.....[2]

- (d) Explain why in the procedure described in (a), the volume of sulfuric acid was measured using a measuring cylinder rather than a burette.
  - sulfuric acid is in <u>excess</u> and so, its volume need not be measured precisely.
    [1]
- (e) To confirm the formula of iron(II) sulfate heptahydrate in the tablet, a student dissolved one of the tablets in nitric acid and added a few drops of barium nitrate to the filtrate.
   State the observation expected from this chemical test. Give your reasoning.

white ppt of BaSO₄ formed confirmed the presence of SO₄<sup>2−</sup> ion.
 [1]
 [1]

#### 2 Determination of the enthalpy change for the reaction of citric acid with NaHCO<sub>3</sub>(aq)

Antacid, taken for quick relieve of occasional heartburn, is also readily available in pharmacies. Antacid contains mainly citric acid and sodium hydrogencarbonate, which react when in contact with water to give carbon dioxide.

In this experiment, you will determine the enthalpy change when citric acid reacts with sodium hydrogencarbonate.

You are provided with the following.

**FA 4** is  $0.8 \text{ mol } \text{dm}^{-3}$  citric acid.

FA 5 is solid sodium hydrogencarbonate, NaHCO<sub>3</sub>.

#### (a) Method

You will carry out the following experiment twice.

- Weigh between 6.5 g and 7.0 g of **FA 5** in a dry weighing bottle.
- Use a pipette to transfer 50.0 cm<sup>3</sup> of FA 4 into the plastic cup supported in a 250 cm<sup>3</sup> beaker.
- Place the thermometer in the acid in the plastic cup and record its initial temperature.
- Carefully add the weighed sample of **FA 5**, in *small portions*, into the acid in the plastic cup. Stir the mixture carefully with the thermometer.
- Record the lowest temperature reached.
- Reweigh the weighing bottle and any residual **FA 5**.

Record in a single table, in the space given on page 7,

- all measurements of mass and temperature, and
- the temperature fall,  $\Delta T$ .

Empty and rinse the plastic cup.

Repeat the experiment and calculate the mean value of

- $\Delta T$ , and
- mass of **FA 5** added.

#### Results

	Expt 1	Expt 2
mass of weighing bottle + FA 5 / g	<b>m</b> 1	m <sub>3</sub>
mass of weighing bottle + residual FA 5 / g	m <sub>2</sub>	m4
mass of <b>FA 5</b> used / g	m <sub>1</sub> – m <sub>2</sub>	m <sub>3</sub> – m <sub>4</sub>
initial temperature / °C	<b>T</b> 1	T <sub>3</sub>
lowest temperature / °C	T <sub>2</sub>	T <sub>4</sub>
temperature fall, $\Delta T / °C$	$T_1 - T_2$	$T_3 - T_4$

- mean value of  $\Delta T = \frac{(T_1 T_2) + (T_3 T_4)}{2} = \dots ^{\circ}C$
- mean mass of FA 5 added =  $\frac{(m_1 m_2) + (m_3 m_4)}{2}$  = ...... g

mean value of  $\Delta T$  =..... mean mass of FA 5 added =.....[4]

- (b) Citric acid is a tribasic acid; i.e. one mole of the acid reacts with three moles of sodium hydrogencarbonate.
  - (i) Write a balanced equation, with state symbols, to show how citric acid reacts with sodium hydrogencarbonate in the experiment. You may use H<sub>3</sub>A to represent citric acid.

 $H_{3}A(aq) + 3NaHCO_{3}(s) \rightarrow Na_{3}A(aq) + 3H_{2}O(l) + 3CO_{2}(g)$ [1]

(ii) Calculate the heat energy when FA 5 was added to the acid.

[Assume that 4.3 J are required to raise the temperature of 1.0 cm<sup>3</sup> of any solution by 1.0  $^{\circ}$ C.]

heat absorbed = mc  $\Delta T$ = 50 × 4.3 × mean  $\Delta T$ = ......J

heat energy =..... J [1]

(iii) In the experiment carried out in **2(a)**, excess citric acid was used. Using your answer to **2(b)(i)**, calculate the amount of citric acid that has reacted with the mean mass of **FA 5** added. [*A*<sub>r</sub>: Na, 23.0; O, 16.0; C, 12.0; H, 1.0]

```
M_{\rm r} \text{ of NaHCO}_3 = 23.0 + 1.0 + 12.0 + 3(16.0) = 84.0
mol of NaHCO<sub>3</sub> used = \frac{\text{mean mass of FA5}}{84.0}
= ..... mol
mol of citric acid reacted = \frac{1}{3} \times \text{mol of NaHCO}_3
= \frac{1}{3} \times \text{above answer}
= ..... mol
```

amount of citric acid = ..... mol [2]

(iv) Calculate the enthalpy change, in kJ mol<sup>-1</sup>, when 1 mol of citric acid reacts with sodium hydrogencarbonate. Your answer should include the appropriate sign.

 $\Delta H = + \frac{\text{ans to (b)(ii)}}{\text{ans to (b)(iii)}}$  $= + \dots \text{ kJ mol}^{-1}$ 

enthalpy change,  $\Delta H = \dots kJ \text{ mol}^{-1}$  [2]

- (v) Explain the significance of the sign you have given in **2(b)(iv)** and how it is related to your experimental results.
  - <u>+ sign because the reaction is endothermic (or heat is absorbed in the</u> reaction or <u>temperature falls</u> during the reaction)
    [1]
- (c) Explain why **FA 5** is added in *small portions* in the procedure described in **2(a)**.
  - to <u>prevent excessive effervescence / acid spray</u> which results in loss of solution.
    [1]

(d) A student repeated the experiment described in 2(a) on another day when the room temperature was much higher. Suggest how this higher room temperature would affect the value of  $\Delta T$ . Give your reasoning.

#### 3 Planning

The reaction between an acid and a metal hydroxide is exothermic. By using this fact, it is possible to determine the equivalence point of a neutralisation reaction without the use of an indicator. This process is known as *thermometric titration*.

In the experiment, the temperature is monitored as portions of acid are progressively added to a fixed volume of the alkali until the equivalence point is reached and passed. The data obtained is plotted and two best-fit graph lines are drawn. One line is drawn using data before the equivalence point and the second line using the remaining data. These lines are then extrapolated until they intersect.

- (a) In a thermometric titration where an acid is run into an alkali, state and explain how you would recognise that the equivalence point has been passed.
  - Since reaction is exothermic, temperature of the mixture <u>increases</u> until the equivalence point is reached.
  - Beyond the equivalence point; temperature of the mixture decreases
     as no further reaction takes place and the acid added is at a lower
     temperature than the mixture.

......[2]

(b) In this question, you are required to write a plan for a thermometric titration in which citric acid is added to 25.0 cm<sup>3</sup> of aqueous sodium hydroxide.

You are provided with the following materials.

- 2.00 mol dm<sup>-3</sup> sodium hydroxide, NaOH
- 0.8 mol dm<sup>-3</sup> citric acid
- equipment normally found in a school laboratory
- Using the information given above, show that the volume of citric acid required to reach the equivalence point is 20.90 cm<sup>3</sup>.
   You may use H<sub>3</sub>A to represent citric acid, which is a tribasic acid.

Since citric acid is tribasic,  $H_3A = 3NaOH$  [OR  $H_3A + 3NaOH \rightarrow Na_3A + 3H_2O$ ] mol of  $H_3A = \frac{1}{3} \times mol$  of NaOH

$$= \frac{1}{3} \times 2.00 \times \frac{25.0}{1000} = 0.0167 \text{ mol}$$

: approx. vol of H<sub>3</sub>A at equivalence point =  $\frac{0.0167}{0.8} \times 1000 = 20.90 \text{ cm}^3$ 

[1]

(ii) Outline how you would carry out the proposed thermometric titration using only the materials provided.

In your plan, you should include

- brief, but specific, details of the apparatus you would use, considering the levels of precision they offer;
- measurements you would make to allow for a suitable graph to be drawn in 3(b)(iii), in order to determine the value of ΔH<sub>n</sub><sup>-Φ</sup> for this reaction; and
- how you would recognise that the equivalence point had been passed.

Outline your plan as a series of numbered steps.

- 1. <u>Pipette 25.0 cm<sup>3</sup> of aq. NaOH into a plastic cup supported in a 250 cm<sup>3</sup></u> <u>beaker</u>. Record the initial temperature of aq. NaOH (using a thermometer calibrated to 0.2°C)
- 2. Fill a 50 cm<sup>3</sup> burette with the citric acid.
- 3. Add 5.00 cm<sup>3</sup> of citric acid from the burette to the plastic cup. Stir the mixture with the thermometer and record the highest temperature reached.
- 4. Add a further 5.00 cm<sup>3</sup> of citric acid to the plastic cup, and again record
  - the highest temperature reached.
- 5. Repeat step 4 until the <u>temperature recorded starts to decrease</u>, showing that equivalence point has been passed. Then add <u>three further</u> 5.00 cm<sup>3</sup>
  - portions of citric acid and record the steady temperature reached after each addition.

- (iii) Sketch, on the axes provided, the graph you would expect to obtain using information in 3(b)(i) and the measurements you planned to make in 3(b)(ii). Label clearly the axes. You should also indicate on your graph,
  - the equivalence point, and
  - the maximum temperature rise,  $\Delta T_1$ .



[3]

(iv) Write an ionic equation to represent the enthalpy change of neutralisation,  $\Delta H_n$ .

 $H^{+}(aq) + OH^{-}(aq) \rightarrow H_{2}O(I)$ [1]

(v) Given that  $\Delta T_1$  is 11.0 °C and using the information in **3(b)(i)**, determine the value of  $\Delta H_n^{-\Phi}$  for this reaction.

heat evolved = mc $\Delta T_1$  = (25.0 + 20.90) × 4.3 × 11.0 = 2171 J mol of H<sub>2</sub>O = mol of NaOH = 2.00 ×  $\frac{25.0}{1000}$  = 0.0500 mol  $\therefore \Delta H_n^{-6-} = -\frac{2171}{0.0500}$  = -4340 J mol<sup>-1</sup> = -43.4 kJ mol<sup>-1</sup>

 $\Delta H_{n^{-\Phi}} = \dots -43.4 \dots kJ mol^{-1}$  [2]

[Total: 13]

#### 4 Inorganic Analysis

You are provided with three solutions, **FA 6**, **FA 7** and **FA 8**, each containing one cation and one anion.

#### Identification of the anions in FA 6, FA 7 and FA 8.

One or more of the solutions contains a halide ion.

- (a) From the Qualitative Analysis Notes on page 15, you are to select and use
  - (i) one reagent to precipitate any halide ion that is present,
  - (ii) a second reagent to confirm the identity of any halide ion present.

# Since the solutions are coloured, you will need to remove traces of solution from the precipitate.

Record the tests performed, the practical procedures used and the observations made for each of the solutions.

Present this information as clearly as possible in a single table in the space below.

	Test	<b>FA 6</b>	FA 7	<b>FA 8</b>
1.	Add aq <u>AgNO<sub>3</sub></u> to each solution.	<u>white</u> ppt.	<u>yellow</u> ppt	no ppt
	Decant / filter to remove the coloured solution.			
2.	Add aq <u>NH<sub>3</sub> to the</u> <u>precipitate</u> obtained in the above test.	ppt <u>dissolves</u> to give a colourless solution	ppt <u>insoluble</u> in aq. NH₃	-

[4]

- (b) Use your observations to identify any halide ions present in the solutions, FA 6, FA 7 and FA 8, and state which ion is present in which solution.
  - $Cl^-$  present in FA 6, I<sup>-</sup> present in FA 7, and no halide ions present in FA 8

[1]

#### Identification of the cations in FA 6, FA 7 and FA 8.

(c) Using aqueous NaOH and aqueous NH<sub>3</sub>, it is possible to identify two of the cations present and to draw conclusions about the nature of the remaining cation.

Carry out tests with these reagents and record details of the observations made for each of the solutions.

Test	FA 6	FA 7	FA 8
<ol> <li>Add aqueous NaOH dropwise until no further change is seen.</li> </ol>	<u>red-brown</u> ppt. <u>insoluble</u> in excess NaOH	<u>grey-green</u> ppt <u>soluble</u> in excess NaOH giving a dark green solution	blue ppt turns brown with excess NaOH; insoluble in excess NaOH
<ol> <li>Add aqueous NH₃ dropwise until no further change is seen.</li> </ol>	<u>red-brown</u> ppt. <u>insoluble</u> in excess NH₃	<u>grey-green</u> ppt <u>insoluble</u> in excess NH <sub>3</sub>	<u>blue</u> ppt; <u>insoluble</u> in excess NH₃

[4]

(d) Use your observations in 4(c) to identify two of the cations present and state which of the solutions contain those cations.

The cation contained in **FA**...**6**..... is .....**Fe**<sup>3+</sup>.....

Evidence:

FA 6 gives red-brown ppt with NaOH and with NH<sub>3</sub>

The cation contained in **FA**...**7**..... is .....**C**r<sup>3+</sup>

Evidence:

FA 7 gives grey-green ppt with NaOH and with NH<sub>3</sub> OR grey-green ppt with NaOH, soluble in excess to give a green solution [2]

(e) From your observations in **4(c)**, what conclusion can you draw about the general nature of the third cation? Explain your answer.

•	FA 8 contains a transition metal cation deduced from the coloured
	solution and ppt.
••••	[1]
	[Total: 12]

## **Qualitative Analysis Notes**

[ppt. = precipitate]

## (a) Reactions of aqueous cations

action	reaction with		
cation	NaOH(aq)	NH₃(aq)	
aluminium, A <i>l</i> <sup>3+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH₄⁺(aq)	ammonia produced on heating	_	
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.	
calcium, Ca <sup>2+</sup> (aq)	white ppt. with high [Ca <sup>2+</sup> (aq)]	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), Cu²+(aq)	pale blue ppt. insoluble in excess	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	

## (b) Reactions of anions

anion	reaction	
carbonate, CO <sub>3</sub> <sup>2-</sup>	CO <sub>2</sub> liberated by dilute acids	
chloride, C <i>l⁻</i> (aq)	gives white ppt. with Ag⁺(aq) (soluble in NH₃(aq))	
bromide, Br⁻(aq)	gives pale cream ppt. with Ag <sup>+</sup> (aq) (partially soluble in NH <sub>3</sub> (aq))	
iodide, I⁻(aq)	gives yellow ppt. with Ag <sup>+</sup> (aq) (insoluble in $NH_3(aq)$ )	
nitrate, NO₃⁻(aq)	NH <sub>3</sub> liberated on heating with OH <sup>-</sup> (aq) and A <i>l</i> foil	
nitrite, NO₂⁻(aq)	NH <sub>3</sub> liberated on heating with OH <sup>-</sup> (aq) and A <i>l</i> foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO <sub>2</sub> in air)	
sulfate, SO₄²⁻(aq)	gives white ppt. with Ba <sup>2+</sup> (aq) (insoluble in excess dilute strong acids)	
sulfite, SO <sub>3</sub> ²⁻(aq)	SO <sub>2</sub> liberated with dilute acids; gives white ppt. with Ba <sup>2+</sup> (aq) (soluble in dilute strong acids)	

## (c) Tests for gases

gas	test and test result
ammonia, NH₃	turns damp red litmus paper blue
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater (ppt. dissolves with excess CO <sub>2</sub> )
chlorine, Cl <sub>2</sub>	bleaches damp litmus paper
hydrogen, H <sub>2</sub>	"pops" with a lighted splint
oxygen, O <sub>2</sub>	relights a glowing splint
sulfur dioxide, SO <sub>2</sub>	turns aqueous acidified potassium manganate(VII) from purple to colourless

## (d) Colour of halogens

halogen	colour of element	colour in aqueous solution	colour in hexane
chlorine, Cl <sub>2</sub>	greenish yellow gas	pale yellow	pale yellow
bromine, Br <sub>2</sub>	reddish brown gas / liquid	orange	orange-red
iodine, I2	black solid / purple gas	brown	purple

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