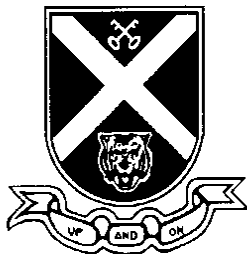


Name:		Shift:	
Class:		Laboratory:	

ST ANDREW'S JUNIOR COLLEGE



PRELIMINARY EXAMINATIONS

PAPER 4 PRACTICAL

Chemistry

28 August 2018

Higher 2

2 hours 30 minutes

READ THESE INSTRUCTIONS FIRST.

Write your name and class on all the work you hand in.
 Give details of the practical shift and laboratory in the boxes provided above.
 Write in dark blue or black pen.
 You may use a soft pencil for any diagrams or graphs.
 Do not use staples, paper clips, highlighters, glue or correction fluid.

Answer **all** the 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 16 and 17.

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

For Examiner's Use	
1	
2	
3	
Total	

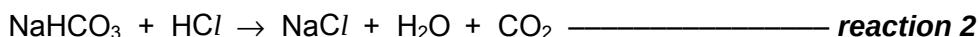
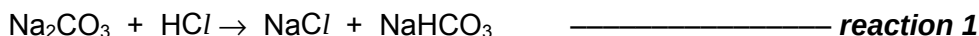
Answer all the questions in the spaces provided.

1 Determination of percentage by mass of sodium carbonate in a mixture of sodium carbonate and sodium hydrogen carbonate

The complete reaction between sodium carbonate and hydrochloric acid can be represented as shown:



However, when sodium carbonate is titrated against hydrochloric acid, it reacts with hydrochloric acid in two separate stages.



When a mixture of sodium carbonate and sodium hydrogen carbonate is titrated against hydrochloric acid using thymolphthalein indicator, the blue colour of the indicator is discharged when sodium carbonate is partially neutralised as represented by **reaction 1**.

Screened methyl orange is then added and a further quantity of hydrochloric acid is necessary to bring about a colour change when **reaction 2** is complete. This further amount of acid reacts with both the sodium hydrogen carbonate formed in **reaction 1** as well as the sodium hydrogen carbonate present initially in the mixture.

- (a) In this experiment, you will determine the amount of Na_2CO_3 and NaHCO_3 in **FA 1** by following the procedures below.

FA 1 is a solution containing sodium carbonate, Na_2CO_3 and sodium hydrogen carbonate, NaHCO_3 .

FA 2 is an aqueous solution containing 0.50 mol dm^{-3} of hydrochloric acid, HCl .

1. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
2. Add 5 drops of thymolphthalein indicator and titrate **FA 1** against **FA 2** in a burette.
3. When the blue colour is discharged, note and record the burette reading in the table provided on page 3. This end-point may not be easily seen. Therefore, you are advised to put a white tile under your conical flask to help you. If you are unsure if the blue colour has discharged or not, you may also add a few more drops of thymolphthalein to the solution.

Do not discard this mixture!

*(The volume of **FA 2** required to reach the first end-point need not be consistent but it should not exceed 15 cm^3 .)*

4. Add 3 drops of screened methyl orange to this mixture and **continue** the titration until the second end-point is reached. Record the final burette reading in the same table provided on page 3.
5. Perform sufficient titrations to obtain consistent results for the **second end-point**, which is the total volume of **FA 2** required.

Results

	1	2	3	
Final burette reading (with screened methyl orange) / cm ³				
Final burette reading (with thymolphthalein) / cm ³				
Initial burette reading / cm ³				
Total volume of FA 2 used to reach the second end-point / cm ³				
Place a tick for the consistent results				

[4]

(b) x is the average volume of **FA 2** required to reach the first end point.

y is the average **further** volume of **FA 2** required to reach the second end-point.

From your 2 consistent titration results, calculate x and y .

$x = \dots\dots\dots$

$y = \dots\dots\dots$ **[2]**

(c) **(i)** Using x , calculate the amount, in moles, of Na₂CO₃ in 25.0 cm³ of **FA 1**.

Number of moles of Na₂CO₃ = $\dots\dots\dots$

[1]

- (c) (ii) Using y , calculate the total amount, in moles, of NaHCO_3 in the reaction mixture.

Total number of moles of NaHCO_3 = [1]

- (d) From your answers in (c), calculate the amount, in moles, of NaHCO_3 in 25.0 cm^3 of **FA 1**.

Number of moles of NaHCO_3 = [1]

- (e) Calculate the percentage by mass of sodium carbonate in **FA 1**, which is a mixture of NaHCO_3 and Na_2CO_3 .

[A_r : Na, 23.0; H, 1.0; C, 12.0; O, 16.0]

[2]

- (f) A student decides to carry out the same experiment in (a) by using a pH meter to determine the percentage by mass of Na_2CO_3 in the mixture.

Given that $\text{p}K_{\text{b}}$ of $\text{CO}_3^{2-} = 3.68$ and $\text{p}K_{\text{b}}$ of $\text{HCO}_3^- = 7.62$, sketch a graph to show how the pH of the mixture changes as HCl is added. You may use your titre values in any set of experiments obtained in (a) for the volume of HCl at the two equivalence points.

[2]

Planning

- (g) Unlike Group 2 carbonates, solid sodium carbonate, Na_2CO_3 , does **not** decompose on heating with a Bunsen burner. However, for solid sodium hydrogencarbonate, NaHCO_3 , it will form sodium carbonate, steam and carbon dioxide when heated.

- (i) Write an equation, with state symbols, to represent the thermal decomposition of sodium hydrogen carbonate.

..... [1]

- (g) (ii) Given the above information, you are to devise a plan to determine the percentage by mass of NaHCO_3 in a solid mixture of NaHCO_3 and Na_2CO_3 .

You may assume you are provided with

- a boiling tube
- a pair of tongs
- 10 g of NaHCO_3 and Na_2CO_3 solid mixture
- a heat resistant mat
- equipment normally found in a school or college laboratory

In your plan, you should include details of:

- procedure you would follow, including the apparatus you would use and the safety precautions to take,
- measurements you would take,
- a brief outline of how the results can be used to determine the percentage by mass of NaHCO_3 in the mixture. You may make assumptions for the masses involved and use appropriate molar mass values to solve this.

[A_r : Na, 23.0; H, 1.0; C, 12.0; O, 16.0]

[6]

[6]

[Total: 20]

2 Determination of n in the complex ion formula $[\text{Cu}(\text{NH}_3)_n]^{2+}$

Many transition metal ions possess the ability to form complexes with ligands, which can either be organic or inorganic molecules/ions.

In this experiment, you will determine the value of n in the formula $[\text{Cu}(\text{NH}_3)_n]^{2+}(\text{aq})$, which is the complex ion formed when aqueous NH_3 is added to $\text{Cu}^{2+}(\text{aq})$.

Different volumes of CuSO_4 solution and aqueous ammonia are mixed and the corresponding temperature changes are measured.

FA 3 is 0.5 mol dm^{-3} CuSO_4 solution

FA 4 is 2.0 mol dm^{-3} aqueous ammonia

(a) Measure and record the temperatures of the given solutions **FA 3** and **FA 4**.

$T_{\text{FA 3}} = \dots\dots\dots^\circ\text{C}$

$T_{\text{FA 4}} = \dots\dots\dots^\circ\text{C}$ [1]

(b) Carry out the following procedures and prepare a table for your results on **page 8**.

Your table should include i) $V_{\text{FA 3}}$, ii) $V_{\text{FA 4}}$, iii) T_{initial} , iv) T_{max} , v) temperature rise, ΔT and vi) colour of solution and precipitate (if any).

Procedures:

Experiment 1

- Measure 9.00 cm^3 of **FA 3** from a burette into a boiling tube.
- Add into the boiling tube 1.00 cm^3 of **FA 4** from another burette.
- Note the temperature rise while stirring to ensure that you do not miss the highest temperature reached. Record this temperature as T_{max} .
- Note and record the colour of precipitate (if any) and the colour of solution observed.
- Discard the content in the boiling tube and wash the boiling tube thoroughly. Wipe the thermometer dry for use in **experiment 2**.

Experiment 2

- Measure 8.00 cm^3 of **FA 3** from a burette into the boiling tube.
- Add into the boiling tube 2.00 cm^3 of **FA 4** from another burette.
- Note the temperature rise while stirring to ensure that you do not miss the highest temperature reached. Record this temperature as T_{max} . Note and record the colour of precipitate (if any) and the colour of solution observed.
- Discard the content in the boiling tube and wash the boiling tube thoroughly. Wipe the thermometer dry for use in subsequent experiments.

Experiments 3 – 9

Perform 7 more experiments, each time reducing the volume of **FA 3** by 1.00 cm³ but increasing the volume of **FA 4** by 1.00 cm³ such that the total volume of solution is kept constant at 10.00 cm³.

The weighted average initial temperature, T_{initial} , of **FA 3** and **FA 4** can be calculated by using the formula:

$$T_{\text{initial}} = \frac{(V_{\text{FA 3}} \times T_{\text{FA 3}}) + (V_{\text{FA 4}} \times T_{\text{FA 4}})}{V_{\text{FA 3}} + V_{\text{FA 4}}}$$

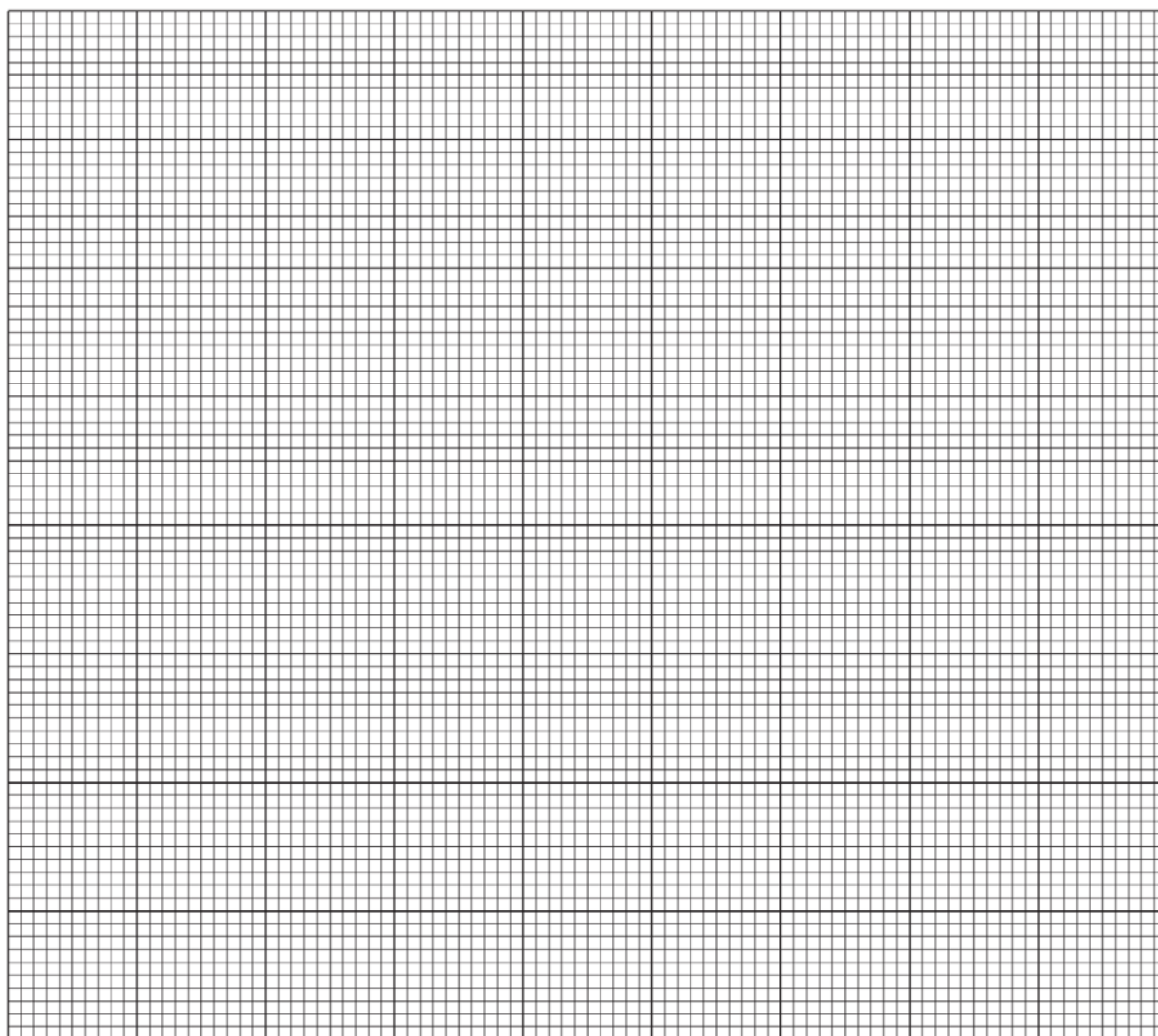
where $V_{\text{FA 3}}$ and $V_{\text{FA 4}}$ are the volumes of **FA 3** and **FA 4** in the mixture

$T_{\text{FA 3}}$ and $T_{\text{FA 4}}$ are the temperatures of **FA 3** and **FA 4** measured in **(a)**

[5]

Calculate T_{initial} for each experiment and include it in your table of results.

- (c) Plot a graph of temperature rise, ΔT , against volume of **FA 4**. Your plot should consist of **two** best-fit **straight lines**.



[3]

- (d) (i) Show on your graph how the volume of **FA 4** that corresponds to the highest temperature rise can be determined.

Volume of **FA 4** with highest temperature rise = cm^3

Hence, calculate the volume of **FA 3** that corresponds to this volume of **FA 4**.

Volume of **FA 3** = cm^3 [3]

- (d) (ii) Calculate the amount, in moles, of aqueous ammonia in the volume of **FA 4** with the highest temperature rise.

Amount of $\text{NH}_3(\text{aq}) = \dots\dots\dots$

[1]

- (iii) Calculate the amount, in moles, of Cu^{2+} ions in the volume of **FA 3** that you have determined in (i).

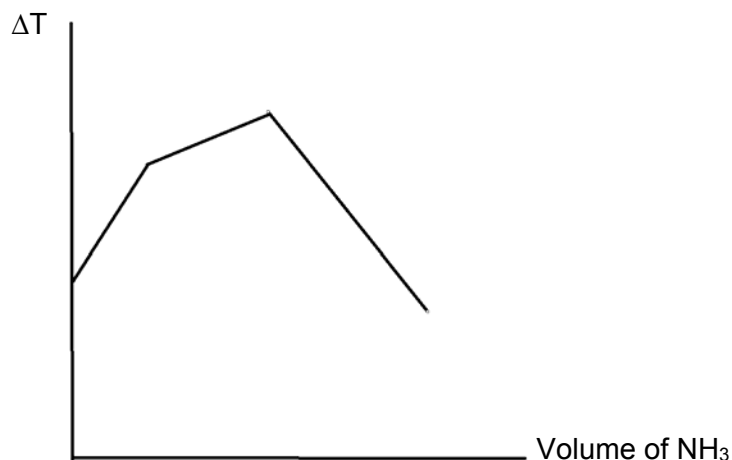
Amount of $\text{Cu}^{2+}(\text{aq}) = \dots\dots\dots$

[1]

- (iv) Calculate the number of moles of NH_3 bonded with one mole of Cu^{2+} ions in the solution and hence, deduce the value of n in the formula $[\text{Cu}(\text{NH}_3)_n]^{2+}$.

$n = \dots\dots\dots$ [1]

- (e) The graph of ΔT against volume of NH_3 should theoretically look like the one below, with a combination of two straight lines with different positive gradients before the change in temperature starts to decrease.



With reference to the observations that you have recorded in the table on page 8, explain, with the aid of two relevant equations, why the graph is a combination of two straight lines with different positive gradients.

.....

.....

.....

.....

..... [3]

- (f) Suggest an improvement that you will make to this experiment to improve the accuracy of the results obtained.

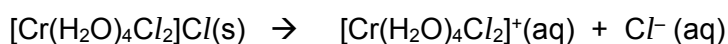
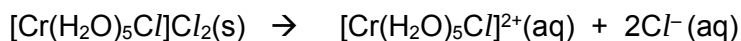
.....

..... [1]

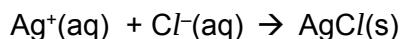
Planning

- (g) Chromium cations can also form complexes. A student was given a solid which has a formula of either $[\text{Cr}(\text{H}_2\text{O})_5\text{Cl}]\text{Cl}_2$ or $[\text{Cr}(\text{H}_2\text{O})_4\text{Cl}_2]\text{Cl}$. The relative formula mass of the compound is 248.5.

When dissolved in water, the chloride ions not involved in the complex ion would be liberated into the solution.



To determine the formula of the solid given, a student prepared a 250 cm³ of standard solution using 29.80 g of the given solid. He then determined the amount of free chloride ions by titrating 25.0 cm³ of the standard solution against 0.3 mol dm⁻³ silver(I) nitrate solution while using some potassium chromate(VI) as an indicator. When silver(I) nitrate solution was added, a white precipitate of AgCl was formed.



When all the free chloride ions had precipitated, a red–brown precipitate of silver chromate(VI) was formed to signify that the end–point had been reached.

- (i) The teacher commented that the student should have reduced the mass of solid used by half for the preparation of standard solution.
Explain, with the aid of suitable calculations, why 29.80 g is not a suitable mass to be used. [2]

- (g) (ii) You are to describe how the student should prepare the standard solution by using his teacher's proposal.

In your answer, include

- the procedures involved in the preparation of the standard solution,
- the apparatus and equipment used to prepare the standard solution.

[2]

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.....

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.....

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.....

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.....

.....

.....

[Total: 23]

3 Identifying cations in an unknown sample

- (a) You are given **FA 5**, which is an aqueous solution containing two cations and one anion. One of the cations is Cu^{2+} . The other cation can be found from the list in the **Qualitative Analysis Notes** provided on page 16.

Perform the tests described in Table 3.1. Record your observations in the table. In all the tests, the reagent should be added gradually until no further change is observed, with shaking after each addition. Test and identify any gases evolved. No additional or confirmatory tests for ions present should be attempted.

Table 3.1

Tests		Observations
1.	To 1 cm depth of FA 5 , add 2 cm depth of FA 1 (from question 1).	
2.	Add about 3 cm ³ of dilute aqueous ammonia to 1 cm ³ of FA 5 . Filter the mixture and note the appearance of both the filtrate and residue. Add dilute sulfuric acid dropwise to the filtrate till in excess.	
3.	Add a spatula of solid potassium iodide to 3 cm ³ of FA 5 . Filter the mixture and note the appearance of the filtrate. Add 1 cm ³ of hexane to the filtrate and shake well.	

(b) Consider your observations in Table 3.1.

(i) State the other cation which is present in **FA 5**.

..... [1]

(ii) **FA 5** turns Universal Indicator paper red. With reference to this and your observations in test 1, write an ionic equation to represent the reaction between **FA 1** and **FA 5**.

..... [1]

(iii) With reference to test 3, suggest the identity of the species which was responsible for the observations seen when hexane was added to the filtrate. Hence, suggest the property of **FA 5**.

..... [2]

Planning

(c) **FA 5** also contains an anion. To determine the identity, a student heated **FA 5** with Al and $NaOH(aq)$. A gas was evolved and it turned moist red litmus paper blue.

(i) Based on the observations given, state the possible identities of the anions.

..... [1]

(ii) You are provided with the reagents $NaOH(aq)$, $NH_3(aq)$, $HNO_3(aq)$ and $AgNO_3(aq)$.

Suggest a test to confirm the identity of the anion in **FA 5**. State the expected observations.

DO NOT carry out the test.

Test:

.....

Expected observation for each of the possible anions in (i).

.....

..... [2]

[Total: 12]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous 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)	ammonia produced on heating	–
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	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	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

(b) Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated on warming with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acids)

(c) Tests for gases

<i>gas</i>	<i>test and test result</i>
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 aqueous acidified potassium manganate(VII) from purple to colourless

(d) Colour of halogens

<i>Halogen</i>	<i>colour of element</i>	<i>colour in aqueous solution</i>	<i>colour in hexane</i>
chlorine, Cl_2	greenish yellow gas	pale yellow	pale yellow
bromine, Br_2	reddish brown gas / liquid	orange	orange-red
iodine, I_2	black solid / purple gas	brown	purple