RIVER VALLEY HIGH SCHOOL
JC 2 PRELIM PRACTICAL EXAMINATION

H2 CHEMISTRY 9729

Paper 4

19 AUGUST 2021

2 HOURS 30 MINUTES

NAME ______________________

CLASS ______________________

INDEX NO. ______________________

INSTRUCTIONS TO CANDIDATES

DO NOT OPEN THIS BOOKLET UNTIL YOU ARE TOLD TO DO SO.

Read these notes carefully.

Write your name, class and index number in the spaces at the top of this page.
Give details of the practical shift and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use a 2B pencil for any diagrams or graph.
Do not use staples, paper clips, highlighters, 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.

Shift
Laboratory

For Examiner's Use	
total	/ 55

This Question Paper consists of **20** printed pages and **1** blank pages.

Answer **all** the questions in the spaces provided.

1 Determine the solubility product, K_{sp}, of calcium iodate(V), $Ca(IO_3)_2$

For Examiner's Use

Calcium iodate is used in the manufacture of disinfectants, antiseptics, and deodorants. Its solubility in water is low. When calcium iodate(V) is mixed with water, the following equilibrium is established.

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

The total amount of $IO_3^-(aq)$ in the saturated salt solution is determined using iodometry.

Excess potassium iodide, KI, is added to an acidified solution of the filtrate, liberating iodine.

Reaction 1 $IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$

The liberated iodine is then titrated with a standard solution of sodium thiosulfate.

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

(a) You are provided with

FA 1 a saturated solution of $Ca(IO_3)_2$ in $KIO_3(aq)$

FA 2 0.200 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$

FA 3 aqueous solution of potassium iodide, KI

FA 4 dilute sulfuric acid, H_2SO_4

Starch indicator

Titration of filtrate, FA 1

1. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
2. Using a measuring cylinder, add about 10 cm^3 of **FA 4** to the conical flask.
3. Using another measuring cylinder, add about 10 cm^3 of **FA 3** to the conical flask.
4. Add **FA 2** from the burette into the conical flask until a pale yellow solution is obtained.
5. Add about 5 drops of starch indicator and continue adding **FA 2** until the blue-black colour just disappears.
6. Record your titration results, to an appropriate level of precision, in the space on page 3.
7. Repeat the titration as many times as necessary until consistent results are obtained.

Results

Supervisor's mean
Student's mean
Difference

1	
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(b) **(i)** From your titrations, obtain a suitable volume of **FA 2** ($V_{FA\ 2}$) to be used in your calculations. Show clearly how you obtained this volume.

$V_{FA\ 2}$ = ...

5	

(ii) Use the volume of **FA 2** obtained in **(b)(i)** to calculate the amount of IO_3^-(aq) present in 25.0 cm^3 of **FA 1**.

6	

amount of IO_3^- present in 25.0 cm^3 of **FA 1** = ...

(iii) Calculate the concentration of $IO_3^-(aq)$ in **FA 1**.

7	

concentration of $IO_3^-(aq)$ in **FA 1** = ...

(iv) Calculate the concentration of $Ca^{2+}(aq)$ in **FA 1**.

8	

concentration of Ca^{2+} in **FA 1** = ...

(c) Write an expression for the solubility product, K_{sp}, of $Ca(IO_3)_2$ and using your answer in parts **(b)(iii)** and **(b)(iv)**, calculate a value for the K_{sp} of $Ca(IO_3)_2$, giving its units in your answer.

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K_{sp} = ...

(d) Another student prepared a solution of **FA 1** and performed the titration. He obtained a value of 7.96×10^{-6} for the K_{sp}. A literature value for this solubility product is 6.47×10^{-6} at 25 °C.

You should assume that apparatus of the same precision was used in both cases.

Suggest a possible reason for the higher value of K_{sp} obtained by the student.

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For Examiner's Use

Planning

The solubility of calcium iodate(V), at a particular temperature, can be defined as:

the mass of calcium iodate(V) that will dissolve in and just saturate 1000 cm^3 of solvent at that temperature.

A saturated solution is one in which no more solid can dissolve at a particular temperature. In a saturated solution with undissolved solid, the following equilibrium is established.

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^{-}(aq)$$

Like most salts, solubility of calcium iodate(V) increases when the temperature of the solution increases.

(e) Plan a procedure to determine the solubility of calcium iodate(V) in water.

You may assume that you are provided with:

- deionised water
- solid calcium iodate(V), $Ca(IO_3)_2$
- filter funnel and filter paper
- water bath
- thermometer
- the apparatus and equipment normally found in a school or college laboratory.

Your plan should include

calculation of the mass (approximate) of $Ca(IO_3)_2$ that will dissolve in 100 cm^3 of deionised water to give a saturated solution

[molar mass of $Ca(IO_3)_2$ = 390 g mol^{-1}; solubility of $Ca(IO_3)_2$ at 20 °C ≈ 6.15×10^{-3} mol dm^{-3}]

- practical details of how you would
  - prepare a saturated solution in 100 cm^3 of deionised water in a 250 cm^3 conical flask,
  - maintain the temperature of the mixture,
  - separate the saturated solution from the undissolved solid,
  - obtain the mass of **dry** solid,
  - ensure that an **accurate** and **reliable** value of solubility of calcium iodate(V) in water is obtained.

- details of how the results would then be used to obtain the solubility of $Ca(IO_3)_2$ in water at 20 °C.

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2 Determine the acid concentration and enthalpy change of neutralisation using calorimetry

For Examiner's Use

When an acid is added into an alkali, an exothermic reaction takes place and the temperature of the mixture increases. Maximum heat is given out when stoichiometric amounts of H^+ and OH^- are added together.

FA 5 is an aqueous solution prepared by mixing *equal volumes* of ***y*** mol dm^{-3} hydrochloric acid, HC*l*, and ***y*** mol dm^{-3} sulfuric acid, H_2SO_4.

FA 6 is 2.00 mol dm^{-3} sodium hydroxide, NaOH.

In this question, you are to follow the neutralisation of known volume of **FA 5**, ~~NaOH,~~ by measuring the highest temperature obtained as different volumes of **FA 6** are added.

By measuring the maximum temperature rise for different mixtures of the two reagents, you are to determine the following:

- the value of ***y***, concentration of the acids present in **FA 5**
- the enthalpy change of neutralisation, ΔH_{neut}, for the reaction

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

(a) Procedure:

1. Fill the burette to the 0.00 cm^3 mark with **FA 6**.
2. Place the polystyrene cup in a 250 cm^3 beaker and use a 50 cm^3 measuring cylinder to transfer 25.0 cm^3 of **FA 5** into the cup. Record the steady temperature of **FA 5** in Table 1 provided on page 10.
3. **Read through the following instructions before starting the experiment.**
4. Run 3.00 cm^3 of **FA 6** from the burette into the cup, stir the solution carefully with the thermometer and record the maximum temperature, T_x (where x is the total volume of **FA 6** added).
5. **Immediately** run a further 3.00 cm^3 of **FA 6** from the burette into the cup, stir and record the maximum temperature as before. Continue the addition of **FA 6**, in 3.00 cm^3 portions, until a total of 36.00 cm^3 of **FA 6** have been run from the burette.
6. Record all temperatures in Table 1.
7. Fill in the units for the final column of Table 1.
8. Complete the table by calculating ΔT and (total volume of mixture $\times$ ΔT) for each measurement.

Table 1

Volume of **FA 6** added / cm^3	Total volume of mixture in cup / cm^3	Temperature T_x / °C		ΔT $(T_x - T_0)$/ °C	Total volume of mixture × ΔT /__________
0.00	25.0	T_0			
3.00	28.0	T_3			
6.00	31.0	T_6			
9.00	34.0	T_9			
12.00	37.0	T_{12}			
15.00	40.0	T_{15}			
18.00	43.0	T_{18}			
21.00	46.0	T_{21}			
24.00	49.0	T_{24}			
27.00	52.0	T_{27}			
30.00	55.0	T_{30}			
33.00	58.0	T_{33}			
36.00	61.0	T_{36}			

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(b) On the grid provided on the next page, plot (total volume of mixture × ΔT) against the volume of **FA 6** added.

Draw two straight lines through the plotted points to find the equivalence point for the titration

(c) Using the graph in **(b)**, determine the volume of **FA 6** added at the equivalence point of the titration.

24	
25	
26	
27	

$V_{FA\ 6}$ = ...

(d) Using your results from **(c)**,

(i) calculate the concentration of the H^+ ions in **FA 5**.

28	

concentration of the H^+ ions in **FA 5** = ...

(ii) Hence, determine the value of **y**.

29	
30	
31	

value of **y** is ....

(e) **(i)** Using the graph in **(b)**, calculate ΔT_{max}.

| 32 | |

ΔT_{max} = ...

(ii) Hence, calculate the heat change for the reaction and the enthalpy change of neutralisation, ΔH_{neut}, for the reaction

$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$

[Assume that 4.18 J of energy is needed to raise the temperature of 1 cm^3 of the solution by 1 K]

| 33 | |
| 34 | |

Heat change = ...

ΔH_{neut} = ...

(f) Suggest one possible modification that would minimise the error or limitation in this thermometric titration. Explain how the suggested modification improves the accuracy of the results.

| 35 | |

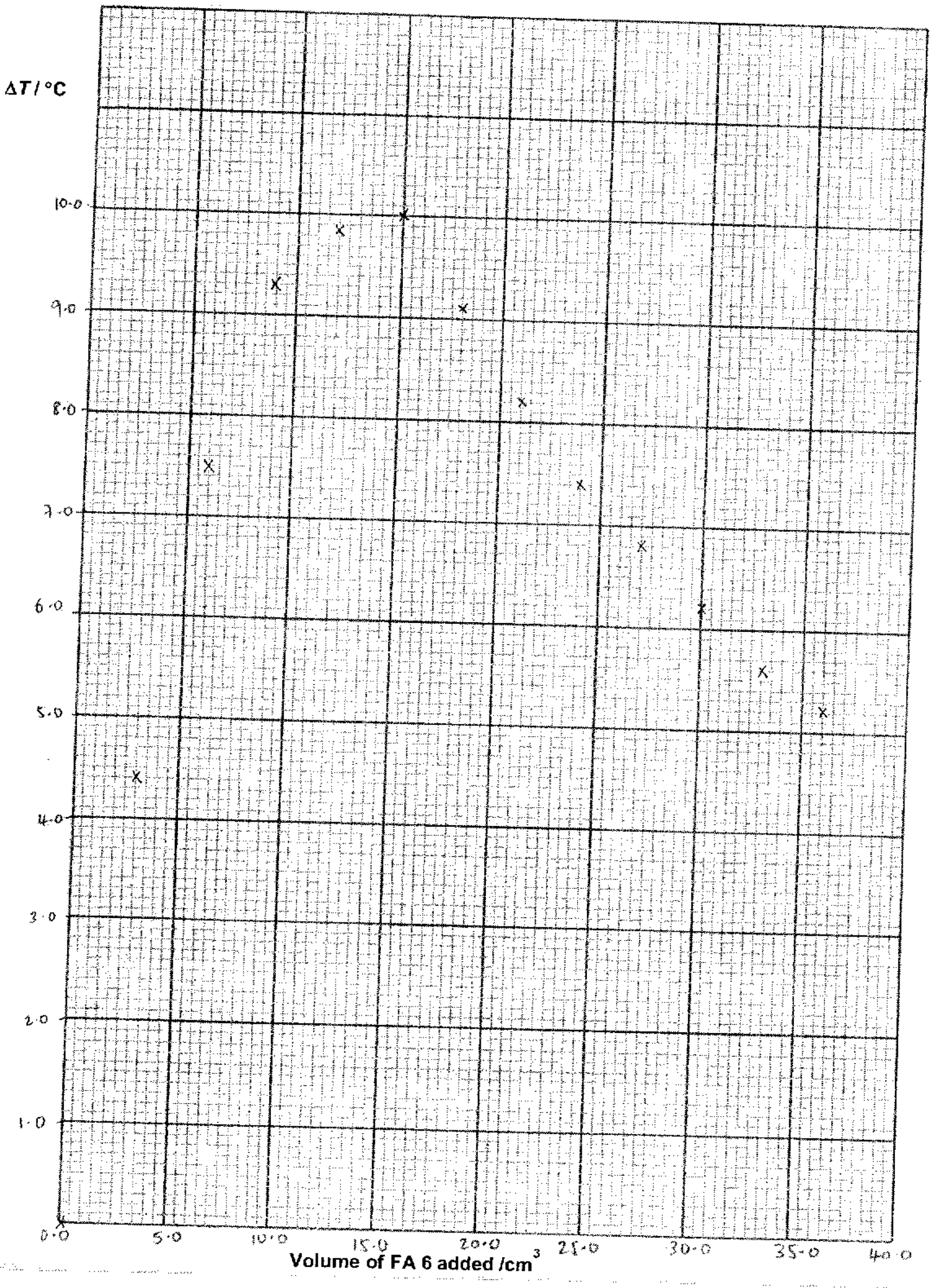
....

....

(g) On the grid provided below, plot a graph of ΔT against the volume of **FA 6** added.

Draw two best fit lines through the plotted points to find the equivalence point for the titration.

36	
37	
38	



Volume of **FA 6** added at equivalence point = ... cm^3

Briefly explain why the graph of ΔT against the volume of **FA 6** added before equivalence point is not drawn as a best fit straight line.

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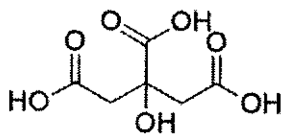
(h) The above experiment was repeated using citric acid instead of the acids in **FA 5**. The ΔT at the equivalence point was found to be a smaller value. Account for the difference.



citric acid

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For Examiner's Use

3 Qualitative Analysis

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

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

- colour changes seen;
- the formation of any precipitate and its solubility in an excess of the reagent added;
- the formation of any gas and its identification by a suitable test.

You should indicate clearly at what stage in a test a change occurs.

Rinse and reuse test-tubes where possible.

No additional tests for ions present should be attempted.

(a) Sandell's solution reacts in a similar way to Fehling's reagent.

You will need to heat Sandell's solution in a hot water bath when using it in tests.

FA 7, **FA 8** and **FA 9** are all solutions of carbohydrates.

- Sugars and starch are carbohydrates.
- Some sugars contain an aldehyde group which act as reducing agents.
- Other sugars do not contain an aldehyde group.

For each test, use 1 cm depth of the solution in a test-tube. Record all your observations in the table.

test	*observations*		
	FA 7	**FA 8**	**FA 9**
Add 2 or 3 drops of aqueous iodine.			
To prepare acidified potassium manganate(VII), add 2 drops of potassium manganate(VII) to a 1 cm depth of an appropriate acid. Add 2 or 3 drops of acidified potassium manganate(VII) and shake.			
Add a 3 cm depth of Sandell's solution and place the tube in the hot water bath for two minutes.			

41 | 42 | 43 | 44

(i) State the carbohydrate that could be starch.

Starch = ...

45

(ii) State the carbohydrate that contains an aldehyde

Carbohydrate that contains aldehyde = ...

(iii) Suggest a different test, other than using Fehling's reagent, that could be carried out to identify the presence of an aldehyde group.

State the reagent(s) you would use and the expected observation if the result were positive. **Do not carry out your test.**

reagent(s)	
observation	

46 | 47

(b) **FA 10** and **FA 11** are two of the components of Sandell's solution. Each contains one cation and one anion. For each test, use 1 cm depth of the solution in a test-tube

(i)

	observations	
test	**FA 10**	**FA 11**
Add a few drops of aqueous silver nitrate.		
Add a few drops of aqueous barium nitrate, then dilute nitric acid.		
Add a few drops of aqueous iodine.		
Add a 1 cm depth of aqueous iron(II) sulfate.		
Add a 1 cm depth of **FA 11**.		

48	
49	
50	
51	

(ii) Identify the ions in **FA 10** and **FA 11**. If you are unable to identify any of the ions, write 'unknown'.

	FA 10	**FA 11**
cation		
anion		

52	
53	

(iii) Suggest a simple chemical test that could be carried out to identify the presence of the anion in **FA 11**. **Do not carry out your test.**

test	
observation	

54	
55	

9 Qualitative Analysis Notes
[ppt. = precipitate]

9(a) Reactions of aqueous cations

cation	reaction with NaOH(aq)	reaction with 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

9(b) Reactions of anions

anion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br^-(aq)	gives pale 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, NO_3^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al 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 dilute strong acids)

9(c) 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 aqueous acidified potassium manganate(VII) from purple to colourless

9(d) Colour of halogens

halogen	*colour of element*	*colour in aqueous solution*	*colour in hexane*
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

RIVER VALLEY HIGH SCHOOL

JC 2 PRELIM PRACTICAL EXAMINATION

H2 CHEMISTRY 9729

Paper 4

19 AUGUST 2021

2 HOURS 30 MINUTES

NAME ______________________________

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Shift
Laboratory

For Examiner's Use	
total	55

This Question Paper consists of **20** printed pages and **1** blank page.

Answer **all** the questions in the spaces provided.

1 **Determine the solubility product, K_{sp}, of calcium iodate(V), $Ca(IO_3)_2$**

For Examiner's Use

Calcium iodate is used in the manufacture of disinfectants, antiseptics, and deodorants. Its solubility in water is low. When calcium iodate(V) is mixed with water, the following equilibrium is established.

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

The total amount of $IO_3^-(aq)$ in the saturated salt solution is determined using iodometry.

Excess potassium iodide, KI, is added to an acidified solution of the filtrate, liberating iodine.

Reaction 1 $IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$

The liberated iodine is then titrated with a standard solution of sodium thiosulfate.

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

(a) You are provided with

FA 1 a saturated solution of $Ca(IO_3)_2$ in $KIO_3(aq)$

FA 2 0.200 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$

FA 3 aqueous solution of potassium iodide, KI

FA 4 dilute sulfuric acid, H_2SO_4

Starch indicator

Titration of filtrate, FA 1

1. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
2. Using a measuring cylinder, add about 10 cm^3 of **FA 4** to the conical flask.
3. Using another measuring cylinder, add about 10 cm^3 of **FA 3** to the conical flask.
4. Add **FA 2** from the burette into the conical flask until a pale yellow solution is obtained.
5. Add about 5 drops of starch indicator and continue adding **FA 2** until the blue-black colour just disappears.
6. Record your titration results, to an appropriate level of precision, in the space on Page 3.
7. Repeat the titration as many times as necessary until consistent results are obtained.

Results

Titration	1	2
Final burette reading / cm^3	18.95	18.95
Initial burette reading/ cm^3	0.00	0.00
Volume of **FA 2** used / cm^3	18.95	18.95

Supervisor's mean
18.95
Student's mean
Difference

1	
2	
3	
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(b) **(i)** From your titrations, obtain a suitable volume of **FA 2** (***V*****FA 2**) to be used in your calculations. Show clearly how you obtained this volume.

Average volume of **FA 2** used = 18.95 cm^3

5	

(ii) Use the volume of **FA 2** obtained in **(b)(i)** to calculate the amount of $IO_3^-(aq)$ present in 25.0 cm^3 of **FA 1**.

Amount of $S_2O_3^{2-} = 0.200 \times \frac{18.95}{1000}$

$= 0.00374$ mol

$IO_3^- \equiv 3I_2 \equiv 6S_2O_3^{2-}$

amount of IO_3^- in 25.0 cm^3 of **FA 1**

$= 0.00374 \div 6$

$= 6.32 \times 10^{-4}$ mol

6	

(iii) Calculate the concentration of $IO_3^-(aq)$ in **FA 1**.

Concentration of dissolved IO_3^- in **FA 1**

$$= \frac{6.32 \times 10^{-4}}{25.0} \times 1000$$

$= 2.52 \times 10^{-2}$ mol dm^{-3}

7	

(iv) Calculate the concentration of $Ca^{2+}(aq)$ in **FA 1**.

Concentration of Ca^{2+} = $\frac{2.52 \times 10^{-2}}{2}$ = 0.0126 mol dm^{-3}

8	

(c) Write an expression for the solubility product, K_{sp}, of calcium iodate(V) and using your answer in parts **(b)(iii)** and **(b)(iv),** calculate a value for the solubility product, K_{sp}, of $Ca(IO_3)_2$, giving its units in your answer.

$K_{sp} = [Ca^{2+}][IO_3^-]^2$

$= (0.0126)(2.52 \times 10^{-2})^2 = 8.02 \times 10^{-6}$ mol^3 dm^{-9}

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(d) Another student prepared a solution of **FA 1** and performed the titration. He obtained a value for the solubility product, K_{sp}, of 7.96×10^{-6}. A literature value for this solubility product is 6.47×10^{-6} at 25 °C.

You should assume that apparatus of the same precision was used in both cases.

State a possible reason for the higher value of K_{sp} obtained by the student.

The student performed the experiment at a temperature higher than 25 °C. Hence, the position of equilibrium for $Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$ shifted to the right.

12	

Planning

For Examiner's Use

The solubility of calcium iodate(V), at a particular temperature, can be defined as:

the mass of calcium iodate(V) that will dissolve in and just saturate 1000 cm^3 of solvent at that temperature.

A saturated solution is one in which no more solid can dissolve at a particular temperature. In a saturated solution with undissolved solid, the following equilibrium is established.

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

Like most salts, solubility of calcium iodate(V) increases when the temperature of the solution increases.

(e) Plan a procedure to determine the solubility of calcium iodate(V) in water.

You may assume that you are provided with:

- deionised water
- solid calcium iodate(V), $Ca(IO_3)_2$
- filter funnel and filter paper
- water bath
- thermometer
- the apparatus and equipment normally found in a school or college laboratory.

Your plan should include

- calculation of the mass (approximate) of $Ca(IO_3)_2$ that will dissolve in 100 cm^3 of deionised water to give a saturated solution [molar mass of $Ca(IO_3)_2$ = 390 g mol^{-1}; solubility of $Ca(IO_3)_2$ at 20 °C ≈ 6.15×10^{-3} mol dm^{-3}]

- practical details of how you would
  - prepare a saturated solution in 100 cm^3 of deionised water in a 250 cm^3 conical flask,
  - maintain the temperature of the mixture,
  - separate the saturated solution from the undissolved solid,
  - obtain the mass of **dry** solid,
  - ensure that an **accurate** and **reliable** value of solubility of calcium iodate(V) in water is obtained.

- details of how the results would then be used to obtain the solubility of $Ca(IO_3)_2$ in water at 20 °C.

Approximate mass of $Ca(IO_3)_2$ that can dissolve in 100 cm^3 of deionised water
$= 6.15 \times 10^{-3} \times 390 \times \frac{100}{1000} = 0.240$ g

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1. Measure 100.0 cm^3 of deionised water using a 100.0 cm^3 measuring cylinder and add it to a 250 cm^3 conical flask. Place the conical flask in a thermostatically-controlled water bath at 20 °C. Place a thermometer into the flask to monitor the temperature of the water.

2. Weight <u>accurately</u> the mass of 1.0 g of $Ca(IO_3)_2$ in a weighing bottle. Using a spatula, add small amounts of pre–weighed $Ca(IO_3)_2$ to the deionised water in the conical flask and stir continuously the mixture with a glass rod. Continue adding $Ca(IO_3)_2$, with stirring, until some undissolved solid remains. Allow 10 min for the mixture to reach equilibrium.

3. Using an electronic balance, weigh <u>accurately</u> a dry filter paper. Record the mass as m_1. Using a dry filter funnel and the same dry filter paper, filter the mixture immediately.

4. Place the filter paper and its contents from (3) under an infra-red lamp for 30 mins.

5. Cool and weigh the filter paper and its contents.

6. Repeat the dry-cool-weigh process from (4) and (5) until the difference in mass is ≤ ±0.05g. Record the final mass of residue and filter paper as m_2.

7. Mass of $Ca(IO_3)_2$ residue = $m_2 - m_1$

8. Reweigh accurately the weighing bottle with residual $Ca(IO_3)_2$ to determine mass of $Ca(IO_3)_2$ transferred to conical flask.

   Mass of $Ca(IO_3)_2$ in filtrate = $(x - y) - (m_2 - m_1)$

Solubility of $Ca(IO_3)_2$ in water
$= \frac{(x-y)-(m_2-m_1)}{100} \times 1000$

Mass of $Ca(IO_3)_2$ + weighing bottle / g	x
Mass of residual $Ca(IO_3)_2$ + weighing bottle / g	y
Mass of $Ca(IO_3)_2$ added / g	$x - y$

[Total: 20]

2 Determination of acid concentration and enthalpy change of neutralisation using calorimetry

For Examiner's Use

When an acid is added into an alkali, an exothermic reaction takes place and the temperature of the mixture increases. Maximum heat is given out when stoichiometric amounts of H^+ and OH^- are added together.

FA 5 is an aqueous solution prepared by mixing *equal volumes* of ***y*** mol dm^{-3} hydrochloric acid, HC*l*, and ***y*** mol dm^{-3} sulfuric acid, H_2SO_4.

FA 6 is 2.00 mol dm^{-3} sodium hydroxide, NaOH.

In this question, you are to follow the neutralisation of known volume of **FA 5**, by measuring the highest temperature obtained as different volumes of **FA 6** are added.

By measuring the maximum temperature rise for different mixtures of the two reagents, you are to determine the following:

- the value of ***y***, concentration of the acids present in **FA 5**
- the enthalpy change of neutralisation, ΔH_{neut}, for the reaction

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

(a) Procedure:

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1. Fill the burette to the 0.00 cm^3 mark with **FA 6**.
2. Place the polystyrene cup in a 250 cm^3 beaker and use a 50 cm^3 measuring cylinder to transfer 25.0 cm^3 of **FA 5** into the cup. Record the steady temperature of **FA 5** in Table 1 provided on page 9.
3. **Read through the following instructions before starting the experiment.**
4. Run 3.00 cm^3 of **FA 6** from the burette into the cup, stir the solution carefully with the thermometer and record the maximum temperature, T_x (where x is the total volume of **FA 6** added).
5. **Immediately** run a further 3.00 cm^3 of **FA 6** from the burette into the cup, stir and record the maximum temperature as before. Continue the addition of **FA 6**, in 3.00 cm^3 portions, until a total of 36.00 cm^3 of **FA 6** have been run from the burette.
6. Record all temperatures in **Table 1**.
7. Fill in the units for the final column of **Table 1**.
8. Complete the table by calculating ΔT and (total volume of mixture × ΔT) for each measurement.

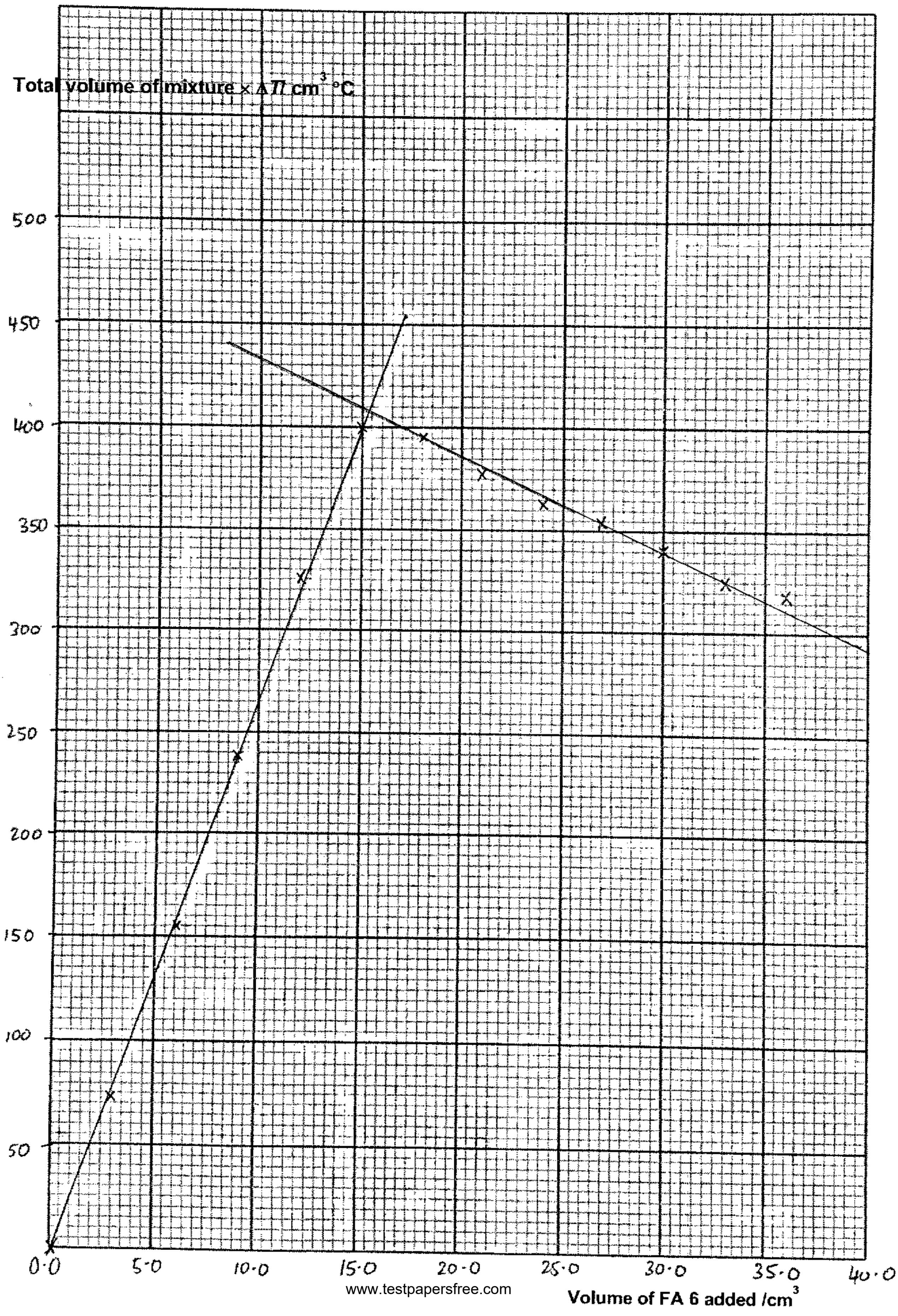
Table 1

Volume of **FA 6** added / cm^3	Total volume of mixture in cup / cm^3	Temperature T_x / °C		ΔT $(T_x - T_0)$/ °C	Total volume of mixture × ΔT / cm^3 °C
0.00	25.0	T_0	29.6	0.0	0.00
3.00	28.0	T_3	32.2	2.6	72.8
6.00*	31.0	T_6	34.6	5.0	155
9.00	34.0	T_9	36.6	7.0	238
12.00*	37.0	T_{12}	38.4	8.8	326
15.00*	40.0	T_{15}	39.6	10.0	400
18.00*	43.0	T_{18}	38.8	9.2	396
21.00	46.0	T_{21}	37.8	8.2	377
24.00	49.0	T_{24}	37.0	7.4	363
27.00	52.0	T_{27}	36.4	6.8	354
30.00*	55.0	T_{30}	35.8	6.2	341
33.00	58.0	T_{33}	35.2	5.6	325
36.00*	61.0	T_{36}	34.8	5.2	702

(b) On the grid provided on the next page, plot (total volume of mixture × ΔT) against the volume of **FA 6** added.

Draw two straight lines through the plotted points to find the equivalence point for the titration

Graph of (total volume of mixture × Δ*T*)/ cm³ °C against volume of **FA 6** added/ cm³



(c) Using the graph in **(b)**, determine the volume of **FA 6** added at the equivalence point of the titration.

Volume of **FA 6** added at equivalence point = 15.50 cm^3

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(d) Using your results from **(c)**,

(i) calculate the concentration of the H^+ ions in **FA 5**.

n_{NaOH} in 15.50 cm^3 of **FA 6** $= 2.00 \times \frac{15.50}{1000}$

$= 3.10 \times 10^{-2}$ mol

$OH^- \equiv H^+$

$[H^+]$ in FA 5 $= (3.10 \times 10^{-2}) \div \frac{25.0}{1000}$

$= 1.24$ mol dm^{-3}

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(ii) Hence, the value of *y*.

Since **FA 5** is an aqueous solution prepared by mixing *equal volumes* of *y* mol dm^{-3} hydrochloric acid, HCl, and *y* mol dm^{-3} sulfuric acid, H_2SO_4;

mole ratio of H^+ from H_2SO_4 : H^+ from HCl

2 : 1

concentration of HCl in **FA 5** $= \frac{1}{3} \times (1.24) = 0.413$ mol dm^{-3}

Since *equal volumes* of both acids were mixed to obtain FA 5

Concentration of HCl(aq) before dilution = $y = 2 \times 0.413$

= 0.827 mol dm^{-3}

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(e) **(i)** Using the graph in **(b)**, calculate ΔT_{max}.

Total volume of mixture $\times \Delta T_{max} = 420$ cm^3 °C

$\Delta T_{max} = \frac{420}{25.0+15.5} = 10.4$ °C

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(ii) Hence, calculate the heat change for the reaction and the enthalpy change of neutralisation, ΔH_{neut}, for the reaction

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

[Assume that 4.18 J of energy is needed to raise the temperature of 1 cm^3 of the solution by 1 K]

Heat released = $mc\Delta T_{max}$ = (25.0 + 15.5)(4.18)(10.4)

= 1760 J (ignore sign)

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$\Delta H_{neut} = -\frac{1760}{0.0310} = -56.8$ kJ mol^{-1}

(f) Suggest one possible modification that would minimise the error or limitation in this thermometric titration. Explain how the suggested modification improves the accuracy of the results.

Use a pipette instead of 50 cm^3 measuring cylinder to measure and transfer 25.0 cm^3 of FA 1 into the calorimeter. Pipette has a smaller absolute uncertainty, giving rise to lower percentage uncertainty in the volume of FA 1 measured.

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Add FA 2 in smaller portions in the region of the intersection to obtain more data point. This will reduce the uncertainty about where the best fit lines cross each other.

Use a (liquid-in-glass) thermometer with a smaller scale division than 0.2 °C so that the temperature measurement has lower percentage uncertainty, thus more accurate. (Reject: more accurate thermometer/ digital thermometer/ thermocouple thermometer)

(g) On the grid provided below, plot a graph of ΔT against the volume of **FA 6** added.

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Draw two best fit lines through the plotted points to find the equivalence point for the titration.

Volume of **FA 6** added at equivalence point = ...15.50......... cm^3

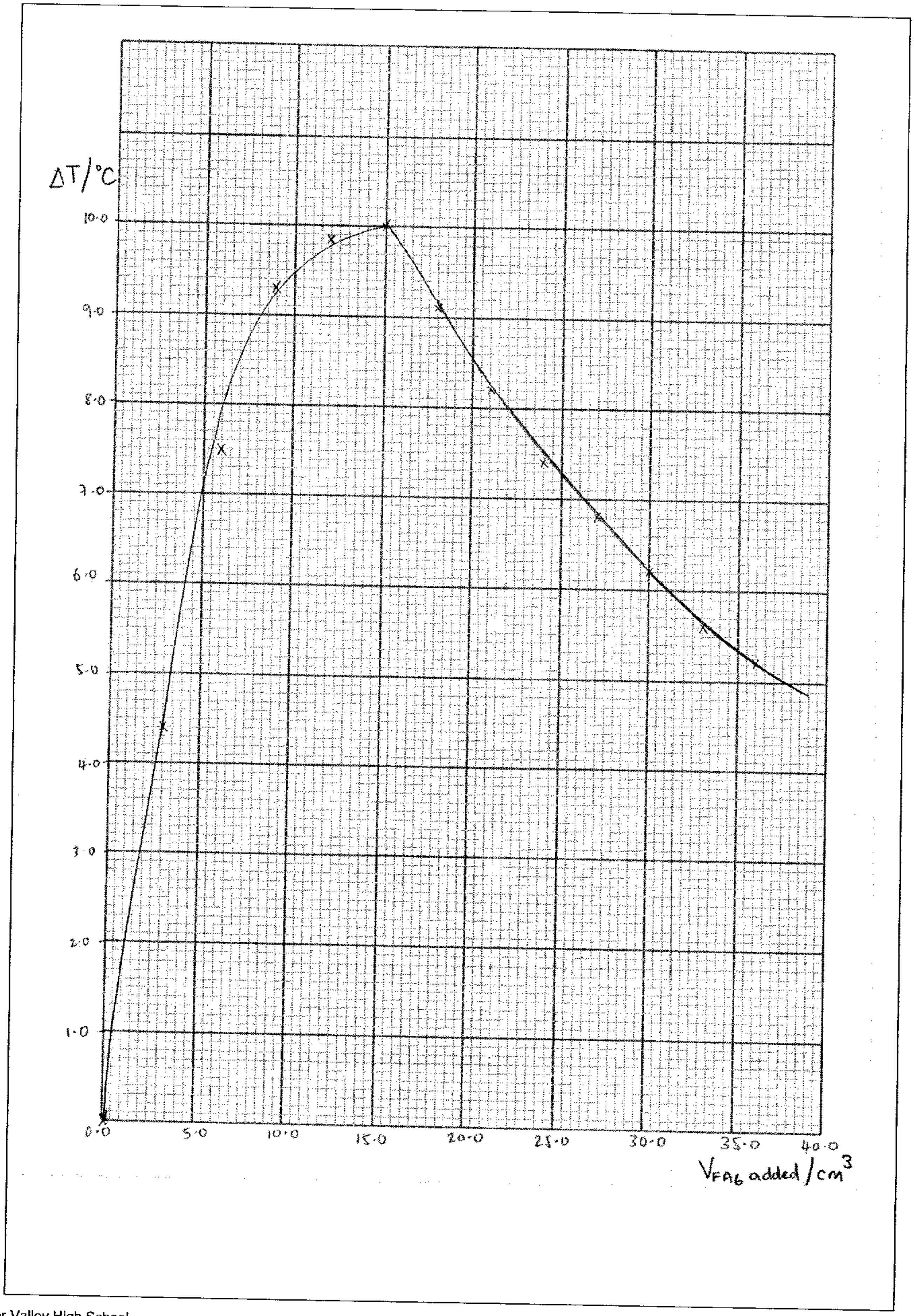
Briefly explain why the graph of ΔT against the volume of **FA 6** added before equivalence point is not drawn as a best fit straight line.

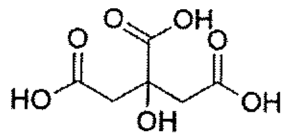
(Before equivalence point, V_{FA6} α nH_2O produced α heat evolved. NaOH is still limiting)

V_{total} increased as more FA 6 is added.

Since heat evolved = $mc\Delta T$ = (V_{total} × density) × $c\Delta T$ = (V_{total} × ΔT) × constant, **the heat evolved will produce a lower temperature rise in an increasingly larger volume of mixture**. Thus, graph of ΔT against V_{FA6} is non-linear.

However, (V_{total} × ΔT) is directly proportional to the energy evolved and thus give a linear relationship in the graph from **(b)**.


ΔT/°C
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
5.0
10.0
15.0
20.0
25.0
30.0
35.0
40.0
V_FA6 added/cm³

(h)	The above experiment was repeated using citric acid instead of the acids in **FA 5**. The ΔT at the equivalence point was found to be a smaller value. Account for the difference.  citric acid		
	Enthalpy change of neutralisation for weak acid (citric acid) and strong base (sodium hydroxide) is less exothermic compared to that of strong acid (hydrochloric acid) and strong base. Citric acid, a weak acid, is partially dissociated in water whereas the acids in **FA 1** are strong acids and fully dissociated. Some of the heat evolved from neutralisation is absorbed to dissociate the citric acid molecules completely, resulting in a smaller ΔT at the equivalence point.	**39**	
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3 Qualitative Analysis

For Examiner's Use

> Where reagents are selected for use in a test, the **name** or **correct formula** of the element or compound must be given.
>
> At each stage of any test you are to record details of the following:
>
> - colour changes seen;
> - the formation of any precipitate and its solubility in an excess of the reagent added;
> - the formation of any gas and its identification by a suitable test.
>
> You should indicate clearly at what stage in a test a change occurs.
>
> Rinse and reuse test-tubes where possible.
>
> **No additional tests for ions present should be attempted.**

(a) Sandell's solution reacts in a similar way to Fehling's reagent.

You will need to heat Sandell's solution in a hot water bath when using it in tests.

> **FA 7**, **FA 8** and **FA 9** are all solutions of carbohydrates.
>
> - Sugars and starch are carbohydrates.
> - Some sugars contain an aldehyde group so act as reducing agents.
> - Other sugars do not contain an aldehyde group.

For each test use **1** cm depth of the solution in a test-tube. Record all your observations in the table.

test	*observations*		
	FA 7	**FA 8**	**FA 9**
Add 2 or 3 drops of aqueous iodine.	blue-black/ dark blue colouration	yellow/ brown solution formed	yellow/ brown solution formed
To prepare acidified potassium manganate(VII), add 2 drops of potassium manganate(VII) to 1 cm depth of an appropriate acid. Add 2 or 3 drops of acidified potassium manganate(VII) and shake.	Solution remains purple / pink	Purple / pink MnO_4^- turns colourless	Solution remains purple / pink
Add a 3 cm depth of Sandell's solution and place the tube in the hot water bath for two minutes.	Solution remains blue/ No ppt formed.	brick red ppt	Solution remains blue/ No ppt formed.

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(i) State the carbohydrate that could be starch.

Starch = **FA 7**

(ii) State the carbohydrate that contains an aldehyde

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Carbohydrate that contains aldehyde = **FA 8**

(iii) Suggest a different test, other than using Fehling's reagent, that could be carried out to identify the presence of an aldehyde group.

State the reagent(s) you would use and the expected observation if the result were positive.

Do not carry out your test.

reagent(s) Tollens' reagent (warm) or acidified potassium dichromate or H^+ / $K_2Cr_2O_7$, (warm)

observation Silver mirror/ Black ppt or Orange solution turn green

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(b) **FA 10** and **FA 11** are two of the components of Sandell's solution. Each contains one cation and one anion. For all the tests, use 1 cm depth of each solution in a test tube.

(i)

	observations	
Test	**FA 10**	**FA 11**
Add a few drops of aqueous silver nitrate.	No ppt formed	Brown ppt formed
Add a few drops of aqueous barium nitrate, then dilute nitric acid.	White ppt formed. Ppt is insoluble in dilute nitric acid	White ppt formed Ppt is soluble in excess dilute nitric acid to form a colourless solution.
Add a few drops of aqueous iodine.	Cream ppt formed in brown solution.	yellow solution formed.
Add a 1 cm depth of aqueous iron(II) sulfate.	No ppt formed/ Solution remains blue.	Green ppt formed. Green ppt turns brown on standing.
Add a 1 cm depth of **FA 11**.	Blue ppt formed.	

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(ii) Identify the ions in **FA 10** and **FA 11**. If you are unable to identify any of the ions, write 'unknown'.

	FA 10	**FA 11**
cation	Cu^{2+}	Unknown
anion	SO_4^{2-}	OH^-

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(iii) Suggest a simple chemical test that could be carried out to identify the presence of the anion in **FA 11**. **Do not carry out your test.**

test Add NH_4^+(aq) (and warm)

observation **Colourless, pungent gas** evolved that turns **moist red litmus blue**. Gas is **NH_3**

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