Suggested Solutions for 9729 2019 Prelim Practical Examinations

1 An experiment to investigate the behaviour of acids and bases in aqueous solution

FA 1 is 1.00 mol dm^{-3} sodium hydrogencarbonate, $NaHCO_3$

FA 2 is sodium hydroxide, NaOH, between 1.5 to 2.5 mol dm^{-3}

FA 3 is 2.00 mol dm^{-3} sulfuric acid, H_2SO_4

According to the *Arrhenius* theory of acids and bases, an acid produces $H^+(aq)$ ions and a base produces $OH^-(aq)$ ions, in aqueous solution.
Using the *Arrhenius* theory, an acid-base neutralisation reaction involves reacting together these two ions to produce water molecules.

The equation for this neutralisation reaction is given below.
$H^+(aq) + OH^-(aq) \longrightarrow H_2O(l)$ $\Delta H_{neutralisation} = -57.1$ kJ mol^{-1}

The reaction between $H^+(aq)$ and $OH^-(aq)$ involves bond formation and so is exothermic. It is possible to make use of this fact to perform a thermometric titration. This task involves two different acid-base reactions.

The first reaction is between sodium hydrogencarbonate, **FA 1**, and sodium hydroxide, **FA 2**.

Reaction 1:
$NaHCO_3(aq) + NaOH(aq) \longrightarrow Na_2CO_3(aq) + H_2O(l)$ $\Delta H_{reaction\ 1}$

The molar enthalpy change for **Reaction 1**, $\Delta H_{reaction\ 1}$, is the enthalpy change when 1.00 mol of $NaHCO_3$ reacts completely with NaOH.

Instead of using an indicator to determine the endpoint, you will perform a thermometric titration to determine the equivalence point of the reaction. The equivalence point is that point where $H^+(aq)$ from the acid and $OH^-(aq)$ from the base are present in equal molar amounts.

The second reaction is between sodium hydrogencarbonate, **FA 1**, and sulfuric acid, **FA 3**.

Reaction 2:
$NaHCO_3(aq) + \frac{1}{2}H_2SO_4(aq) \longrightarrow \frac{1}{2}Na_2SO_4(aq) + H_2O(l) + CO_2(g)$
$\Delta H_{reaction\ 2}$

The molar enthalpy change for **Reaction 2**, $\Delta H_{reaction\ 2}$, is the enthalpy change when 1.00 mol of $NaHCO_3$ reacts completely with H_2SO_4.

In your first experiment, you will perform a thermometric titration by adding portions of **FA 2** progressively to a known volume of **FA 1**. You will continue adding **FA 2** until the equivalence point is reached and passed. Throughout the experiment you will note and record the temperature of the mixture after each addition.

You will then analyse your results graphically in order to determine the
• titration volume at the equivalence point, $V_{equivalence}$,
• maximum temperature change, $\Delta T_{maximum\ 1}$,
• molar enthalpy change, $\Delta H_{reaction\ 1}$, for **Reaction 1**.

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In your second experiment, you will mix together a given volume of **FA 1** with a suitable volume of **FA 3**.

You will then determine the
• maximum temperature change, $\Delta T_{maximum\ 2}$,
• molar enthalpy change, $\Delta H_{reaction\ 2}$, for **Reaction 2**.

Reaction between FA 1 and FA 2

(a) (i) In this task you will need to record the maximum temperature of the reaction mixture when specified volumes of **FA 2** have been added. It is important that the volume of **FA 2** recorded is the total volume you have added up to that point when the temperature reading was made.

Note:
If you overshoot on an addition, record the **actual** total volume of **FA 2** added up to that point.

In an appropriate format in the space provided, record all values of temperature, *T,* to 0.1 °C, and each total volume of **FA 2** added to 0.05 cm^3.

1. Fill a burette with **FA 2**.
2. Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
3. Using a pipette, transfer 25.0 cm^3 of **FA 1** to the first Styrofoam cup.
4. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature.
5. From the burette, add 2.00 cm^3 of **FA 2** to the cup and stir the mixture thoroughly.
6. Read and record the maximum temperature of the mixture, *T,* and the volume of **FA 2** added.
7. Repeat points 5 and 6 until a total of 30.00 cm^3 of **FA 2** has been added. After each addition of **FA 2**, record the maximum temperature of the mixture and the total volume of **FA 2** added up to that point.

Results

For Examiner's Use

Volume of FA2 added/cm^3	*Maximum temperature/°C*
0.00	*31.8*
2.00	*33.0*
4.00	*34.0*
6.00	*34.8*
8.00	*35.4*
10.00	*36.0*
12.00	*36.4*
14.00	*36.6*
16.00	*36.4*
18.00	*36.2*
20.00	*35.8*
22.00	*35.3*
24.00	*35.0*
26.00	*34.8*
28.00	*34.6*
30.00	*34.4*

1	
2	

Note:

- *Correct headers and units (Final Temp not accepted as header);*
- *Proper tabulation of volumes and temperatures*
- *Thermometer readings to 1dp*
- *Total volume of FA2 to 2dp*

(a) **(ii)** On the grid below, plot a graph of temperature, T against volume of **FA 2** added.

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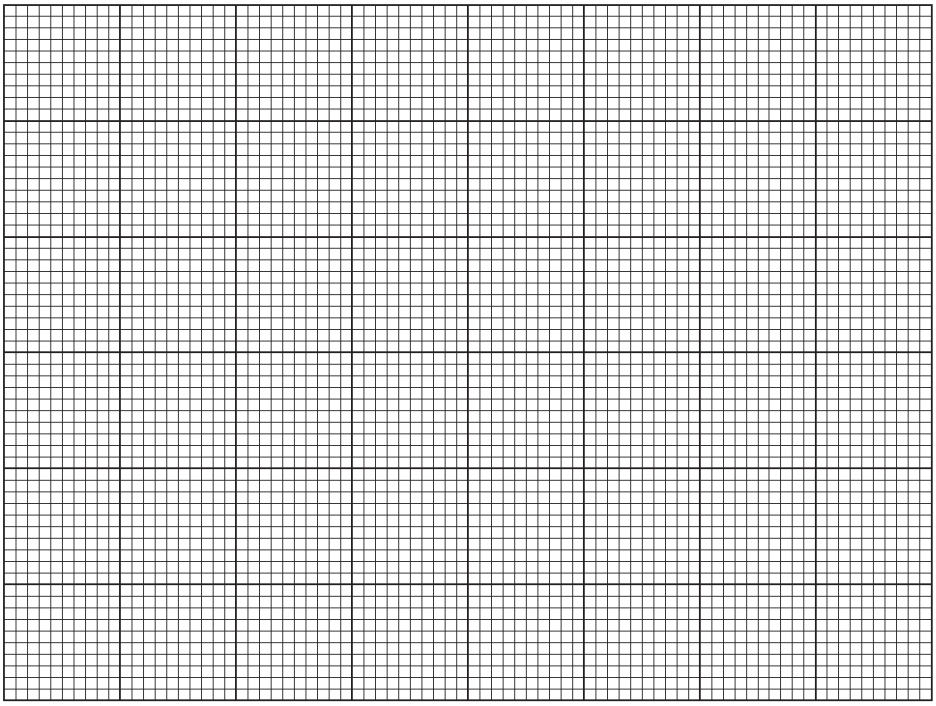

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(iii) Draw **two** smooth lines of best fit. Extrapolate these lines until they cross. Extrapolate these lines until they cross.

Note:

- *Axes correct way round + correct labels + units + scale.*
- *Awkward scales (e.g. 3:10) are not allowed.*
- *The plotted points must occupy at least half of the grid in both directions.*
- *All points must be correctly plotted within ±½ small square.*
- *Graph lines may be **curved/ straight** best-fit lines, drawn so as to best reflect the distribution of points **before** and **after** the equivalence point. These lines should be extrapolated until they **cross**.*

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Supervisor
Student
Difference

(iv) Determine from your graph,

• the maximum temperature reached, $T_{maximum}$,

• the maximum temperature change, $\Delta T_{maximum\ 1}$,

• the volume, $V_{equivalence}$, of **FA 2** needed to completely react with 25.0 cm^3 of **FA 1**.

Show on your graph how you obtained these values.

Record these values in the spaces provided below.

Maximum temperature reached, $T_{maximum}$ = ...

Volume of **FA 2** used, $V_{equivalence}$ = ...

Supervisor
Student
Difference

Hence, calculate the maximum temperature change, $\Delta T_{\text{maximum 1}}$

= .....

Note:

- *Correct reading, to ±½ small square, of Tmaximum; correct calculation of ΔTmaximum; correct reading, to ±½ small square, of Vequivalence.*
- *Accuracy based on difference between Supervisor's and Student's values*

Reaction between FA 1 and FA 3

For Examiner's Use

(b) The molar enthalpy change for **Reaction 2**, $\Delta H_{\text{reaction 2}}$, is the enthalpy change when 1.00 mol of $NaHCO_3$ reacts completely with H_2SO_4.

In this task you will calculate a value for the molar enthalpy change, $\Delta H_{\text{reaction 2}}$. To do this you will need to determine the maximum temperature change produced when measured volumes of **FA 1** and **FA 3** are mixed. After considering the concentrations of **FA 1** and **FA 3**, you will select a suitable volume of **FA 3** to add to 40 cm^3 of **FA 1**.

(i) Choose a suitable volume of **FA 3**, $V_{\textbf{FA 3}}$, to be added to 40 cm^3 of **FA 1**. Explain your answer.

Volume should be between $10cm^3 \leq V_{FA3} \leq 20cm^3$ to add sufficient **FA3** for complete reaction/provide an excess of acid for reaction. (Adding too much **FA3** will result in larger percentage uncertainty due to lower temperature rise)

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(ii) Identify the apparatus you intend to use to measure the volume of **FA 1**.

Explain your choice.

Burette. Since **FA1** is the limiting reagent, exact measurement is required. So precise/ accurate measurement of ±0.05 cm^3 is required.

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(iii) In an appropriate format in the space provided below, record all values of measured temperature for this experiment.

1. Label one styrofoam cup, **A**. Label a second Styrofoam cup, **B**.
2. Place cup **A** inside a styrofoam cup which is held in a glass beaker to prevent it tipping over.
3. Transfer 40 cm^3 of **FA 1** into cup **A**.
4. Stir the **FA 1** solution in cup with the thermometer. Read and record its temperature, $T_{\textbf{FA 1}}$. This is the initial temperature of **FA 1**.
5. Wash and dry the thermometer.

6. Transfer your chosen volume of **FA 3** into cup **B**. Stir the **FA 3** solution with the thermometer. Read and record its temperature, $T_{\mathbf{FA\ 3}}$. This is the initial temperature of **FA 3**.
7. Carefully add the contents of cup **B** to cup **A** in **small** portions to avoid too much frothing.
8. Place the lid on the cup and insert the thermometer through the lid. Stir the mixture.
9. Continue to stir the mixture. Measure and record the temperature, $T_{mixture}$ that shows the maximum change from the initial temperature.

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Results

T_{FA1}/°C	T_{FA3}/°C	$T_{mixture}$/°C
31.6	*31.0*	*31.8*

For Examiner's Use

Note:

- *Correct headers and units*
- *Proper tabulation of temperature of FA1, FA3 and maximum temperature change in a table.*
- *Recording of thermometer readings to 1dp*

Calculations

(c) For the purposes of these calculations, you should assume that the reaction mixture has a density of 1.00 g cm^{-3} and a specific heat capacity, c, of 4.18 J g^{-1} K^{-1}.

(i) Calculate the concentration of sodium hydroxide, [NaOH], in **FA 2**.

$$[NaOH] = \frac{25.0\ x\ 10^{-3}}{Vequivalence\ x\ 10^{-3}} = XXX$$

[NaOH] in **FA 2** = ...

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(ii) Calculate the heat change, q, for the reaction occurring in **(a)(i)**, and hence determine a value for the molar enthalpy change for **Reaction 1**, $\Delta H_{reaction\ 1}$.

m = 25.0 + $V_{equivalence}$

q = mc$\Delta T_{maximum}$

$nNaHCO_3 = 25.0 \times 10^{-3}$

ΔHreaction 1 = $-\frac{q}{25.0\ x\ 10^{-3}}$

Note: – sign must be present.

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(d) Use your results from **(b)(iii)** to calculate a value for the molar enthalpy change for **Reaction 2**, $\Delta H_{reaction\ 2}$. For the experiment in **(b)(iii)**, the weighted average initial temperature, $T_{average}$, of **FA 1** and **FA 3** may be calculated using the formula given below.

For Examiner's Use

$$T_{average} = \frac{(V_{FA1} \times T_{FA1}) + (V_{FA3} \times T_{FA3})}{(V_{FA1} + V_{FA3})}$$

Calculation of $T_{average}$ using formula given

$\Delta T_{maximum\ 2} = |T_{mixture} - T_{average}|$
$q = (V_{\mathbf{FA\ 1}} + V_{\mathbf{FA\ 3}}) \times 4.18 \times$ temperature change

$\Delta H_{reaction\ 2} = -\frac{q}{40.0\ x\ 10^{-3}}$

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$T_{average}$ = ...

q =.....

$\Delta H_{reaction\ 2}$ = ...

(e) Ionic equations for neutralisation, **Reaction 1**, and **Reaction 2** are shown below.

For Examiner's Use

$HCO_3^- + OH^- \longrightarrow CO_3^{2-} + H_2O$ $\quad \Delta H_{reaction\ 1}$

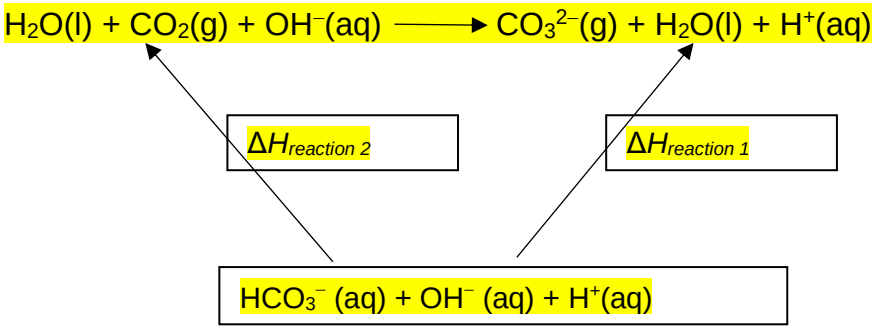
$HCO_3^- + H^+ \longrightarrow H_2O + CO_2$ $\quad \Delta H_{reaction\ 2}$

An equation for this reaction is given below.

Reaction 3:

$H_2O(l) + CO_2(g) + OH^-(aq) \longrightarrow CO_3^{2-}(g) + H_2O(l) + H^+(aq)$ $\Delta H_{reaction\ 3}$

Use your answers in **(c)(ii)** and **(d)**, draw an energy cycle to determine a value for the molar enthalpy change for this reaction, $\Delta H_{reaction\ 3}$.



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$\Delta H_{reaction\ 3} = \Delta H_{reaction\ 1} - \Delta H_{reaction2}$

(f) An alternative definition of acid-base behaviour was proposed by Brønsted and Lowry. **Reaction 1** and **Reaction 2** are both acid-base reactions involving hydrogencarbonate ions, HCO_3^-.

In terms of the *Brønsted-Lowry* theory of acids and bases, suggest the role of hydrogencarbonate ions, HCO_3^-, in **Reaction 1**.

Explain your answer in each case.

Reaction 1

Role : acid

Explanation : HCO_3^- loses an H^+ ion

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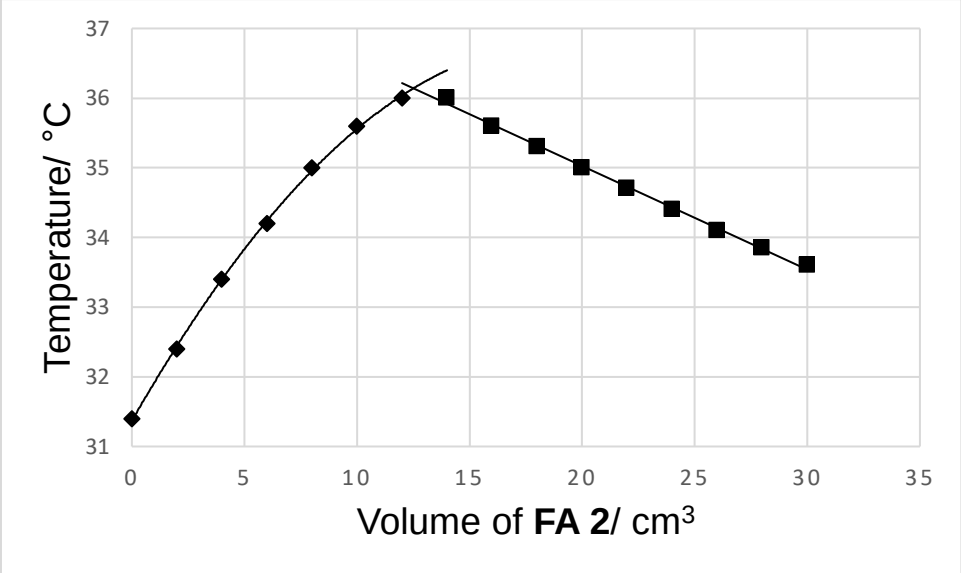
(g) A student conducted the same experiment using a digital thermometer and obtained the graph as shown below.



Explain the shape of the graph.

Before equivalence point, T rises/ gradient is positive with each addition of NaOH as reaction is exothermic.

Nearing equivalence point, T rise is smaller / gradient is less positive/ becoming gentler
as the same amount of heat is used to heat up a larger volume of reaction mixture / greater heat loss to surroudings due to steeper temperature gradient.

After equivalence point, T decreases OR gradient is negative with each addition of NaOH. No heat is evolved/ Reaction is completed and addition of **FA 2** cools the solution. (**FA 2** is at a lower temperature than the reaction mixture)

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(h) In the calculation of **(d)**, explain why the weighted average initial temperature was used.

The initial temperature and volume of sodium hydrogencarbonate and NaOH is different. Hence by considering the mass of the solution, the average initial temperature is calculated with respect to its mass and the average initial temperature will be more accurate.

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(h) With reference to the reaction between **FA 1** and **FA 2**, if the volume of NaOH is doubled, explain how it will affect the maximum temperature change?

No effect on the maximum temperature change. FA 1 is the limiting reagent OR the same amount of heat released at equivalence point is used to heat up the same total volume of solution.

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[Total: 27]

2 To determine the proticity of an unknown acid

For Examiner's Use

Acid base titration is commonly used to determine the concentration of an acid or base. In this experiment, the concentration of the acid is given and the aim of this investigation is to determine its proticity.

Acids are defined as substances that can donate hydrogen ions, H^+, to bases. Monoprotic acids contain one H^+ that can be donated per molecule. Diprotic acids contain two H^+ that can be donated per molecule.

You will determine using a titration method whether acid **Z** is monoprotic or diprotic.

- **FA 4** is a solution containing 11.55 g dm^{-3} of acid **Z**.
- **FA 5** is 0.105 mol dm^{-3} aqueous sodium hydroxide, NaOH.
- Thymol blue indicator

(a) Method

- Pipette 25.0 cm^3 of solution **FA 4** into a conical flask.
- Fill a burette with **FA 5**.
- Add 3 drops of thymol blue indicator to the conical flask.
- The colour change at the endpoint is yellow to blue.
- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 5** added in each accurate titration.

Results

Titration number	1	2	3	
Final burette reading /cm^3	24.50	49.00		
Initial burette reading /cm^3	0.00	24.50		
Volume of **FA 5**(used) /cm^3	24.50	24.50		

28	
29	
30	
31	

Note:

- *Correct headers and units*
- *Proper tabulation of burette readings*
- *Burette readings in the table recorded to the nearest 0.05 cm^3*
- *Check consistency of two uncorrected titres $\leq$0.10 cm^3*
- *Accuracy based on difference between Supervisor's and Student's values*

(b) From your titration results, obtain a suitable value for the volume of **FA 5** to be used in your calculations. Show clearly how you obtained this value.

Average of any two volume of **FA 1** to 2dp.

Volume of **FA 5** =..... | **32** |

(c) Calculations

(i) Calculate the amount of H^+ present in 25.0 cm^3 of **FA 4**.

Amt of H^+ = **(b)** × 0.105 / 1000

Amt of H^+

= (24.50/1000) × 0.105

= 2.573×10^{-3}

= 2.57×10^{-3} mol | **33** |

Amount of H^+ in 25.0 cm^3 of **FA 4** = ...

(ii) Calculate the amount of H^+ present in 1 dm^3 of **FA 4**.

Amt of H^+ = Amt of H^+ from **(c)(i)** × (1000 / 25)

Amt of H^+

= $2.573 \times 10^{-3} \times (1000 / 25)$

= 1.029×10^{-1}

= 1.03×10^{-1} mol | **34** |

Amount of H^+ in 1 dm^3 of **FA 4** = ...

(iii) **FA 4** contains 11.55 g dm^{-3} of acid **Z**. The relative molecular mass of **Z** is 126. Calculate amount of **Z** in 1 dm^3 of **FA 4**.

For Examiner's Use

Amt of Z = 11.15 / 126 = 9.17×10^{-2} mol

Amount of **Z** in 1 dm^3 of **FA 4** = ... | **35** |

(iv) Use your answers to **(ii)** and **(iii)** to determine whether **Z** is a monoprotic or a diprotic acid. Explain your answer.

Z is a monoprotic acid as the amount of H^+ in 1 dm^3 of **FA 4** is similar to the amount of **Z** in 1 dm^3 of **FA 4**.

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(v) Student **A** conducted this experiment on the day when the chemicals were freshly prepared. His average titre value was found to be *h* cm^3.

Three days later, he conducted the same experiment using the same chemicals but he found that the bottle of NaOH(aq) was left uncovered.

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Suggest how his new titre value would compare to *h* cm^3 and explain your answer.

New titre value is lower. Water evaporated from the NaOH (aq). Concentration of NaOH (aq) is higher.

OR

New titre value is higher. NaOH (aq) reacted with CO_2 in the air via acid-base reaction. Concentration of NaOH (aq) is lower.

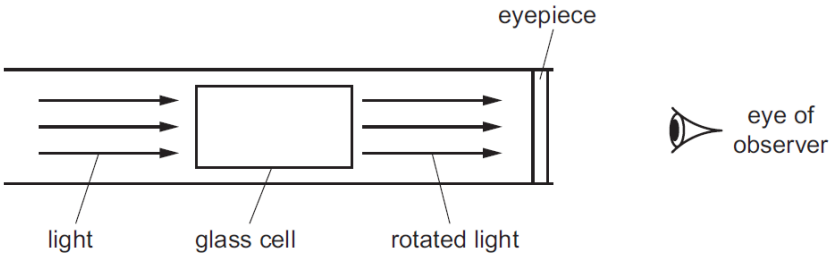
(d) Planning

For Examiner's Use

Sucrose, $C_{12}H_{22}O_{11}$, is a sugar. The concentration of a solution of sucrose can be measured by the optical rotation, α, of a sucrose solution instead of acid–base titration. The more concentrated the solution, the greater the optical rotation of the solution.

A polarimeter is used to measure optical rotation. Plane–polarised light is passed through a sample of the sucrose solution in a glass cell, and the observed angle of rotation, α_{obs}, is measured.

A simplified diagram of a polarimeter is shown.



If a glass cell of length 10 cm is filled with a solution of sucrose of concentration 1 g cm^{-3}, the measured angle of rotation is known as the specific rotation, [α].

The observed angle of rotation, α_{obs}, measured by the polarimeter is related mathematically to the concentration of the sucrose solution by the equation shown.

$$\alpha_{obs} = [\alpha]lc$$

α_{obs} is the observed angle of rotation.

[α] is a constant and is known as the specific rotation of sucrose solution.

l is path length which is 10 cm in the above description.

c is the concentration of sucrose, in g cm^{-3}

(i) Explain why a sucrose solution is able to rotate plane–polarised light.

Sucrose contains at least 1 chiral C/ Sucrose is optically active.

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(ii) Using the information given above, you are required to write a plan to determine the concentration of sucrose in a solution **X**.

For Examiner's Use

You may assume that you are provided with:

- solution **X**, of unknown sucrose concentration less than 0.0800 g cm^{-3};
- access to a polarimeter and instructions for use;
- graph paper;
- the apparatus and chemicals normally found in a school or college laboratory.

Your plans should include details of:

- the preparation of 250 cm^3 0.0800 g cm^{-3} sucrose solution using your answer in **(d)(ii);**
- the preparation of a suitable range of diluted solutions of accurate concentrations;
- an outline of how the results would be obtained;
- a sketch of the calibration curve you would expect to obtain;
- how the calibration line would be used to determine the concentration of sucrose in solution **X**.

Note:

- *Calculate mass of sucrose to use (20.0g)*
- *Proper weighing of sucrose (using electronic balance and reweighing vessel with residual solid)*
- *Procedure to prepare homogenous standard solution of 250 cm^3 0.0800 g cm^{-3} sucrose solution using volumetric flask.*
- *Description of procedure and apparatus (with stated volumes) to prepare diluted solutions.*
- *Concentrations chosen must be well spread out*
- *State the volumes of DI water and stock solution to be added to prepare diluted solutions (min. 3 sets)*
- *Note that total volume of stock solution used cannot exceed volume of stock solution prepared (i.e. 250cm^3)*
- *Scan/ Measure/ Record for each concentration.*
- *Sketch of α_{obs} against concentration. (straight line passing through origin)*
- *Measuring of α_{obs} of solution X and intrapolation to find accurate concentration.*

Example:

Weigh accurately a clean dry empty 100 cm^3 beaker using a weighing balance and record the mass.

Place about 20.00 g sucrose solid in the beaker and reweigh.

Calculate the accurate mass of the sucrose by subtracting the mass of the empty beaker from the mass of beaker and sucrose.

For Examiner's Use

Dissolve the sucrose in deionised water.

Transfer the sucrose solution and its rinsings to a 250.0 cm^3 volumetric flask.

Make-up to the mark with deionised water adding dropwise close to mark and shake the flask well to obtain a homogenous solution.

	100 cm^3 volumetric flask
Concentration of sucrose solution / g dm^{-3}	Volume of 0.0800 g dm^{-3} sucrose solution / cm^3
0.0800	NA
0.0400	50.00
0.0200	25.00
0.0100	12.50
0.0050	6.25

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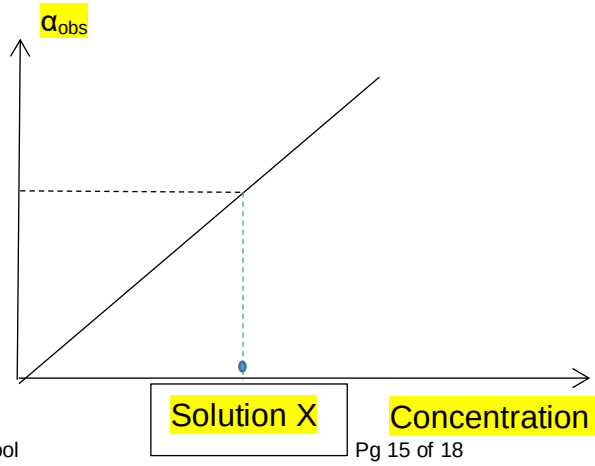
To prepare the diluted sucrose solutions, the appropriate volume of 0.0800 g dm^{-3} sucrose(aq) as shown in the table above is transferred using a burette to a 100 / 250 cm^3 volumetric flask and topped up to the mark with deionised water.

The flask is then well shaken to produce a homogeneous solution.

Measure the rotation values for each sucrose concentration.

A graph of rotation against concentration of the sucrose(aq) used is plotted. A straight line passing through the origin should be obtained.



The rotation value given by the sucrose solution prepared from solution X is measured and the corresponding concentration read off from the calibration graph.

(iv) The glass cell of 10 cm length is expensive, so one cell is used for all the solutions that are placed in the polarimeter.

Suggest how you would ensure that the concentration of solution in the cell is accurate each time the cell is used for the different sucrose solutions.

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Rinse/ wash out with small volume of solution of concentration to be used.

(v) Concentration of sucrose is the independent variable in this polarimeter experiment.

The glass cell of 10 cm length is replaced by a glass cell of 20 cm length. The 20 cm glass cell is filled with 0.0750 g cm^{-3} sucrose solution.

Given that α_{obs} of 0.0750 g cm^{-3} sucrose solution is +6.11°, predict the value for the observed angle of rotation, α_{obs}, for the sucrose solution of concentration 0.0750 g cm^{-3} when the 20 cm cell is used. Explain your answer.

Predicted angle = +12.22°

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[Total: 20]

3 Inorganic qualitative analysis

For Examiner's Use

(a) **FA 6** and **FA 7** each contain one anion and one cation.

Carry out the following tests and record your observations. For each test you should use a small spatula measure of **FA 6 or FA 7**

	observations	
test	**FA 6**	**FA 7**
Add a small spatula measure to a 1 cm depth of deionised water in a test-tube and shake. Add 2 drops of universal indicator.	**FA 6** is a white solid (ppt not accepted) **FA 6** is sparingly soluble/ insoluble Green to Blue/Purple	**FA 7** is a white solid (ppt not accepted) **FA 7** dissolves to give a colourless solution.

Record the pH of the mixture.	pH 10–14	Green to Orange/ Red pH 1–3
Heat a small spatula measure in a dry boiling tube until no further change.	Water vapour /condensation / steam observed on cooler part of boiling tube.	White solid forms on cold part or top of tube / white fumes/ smoke. Moist red litmus paper turned blue* On strong heating, moist blue litmus paper turned to red. Water vapour/ condensation/ steam observed on cooler part of boiling tube.
Add a small spatula measure in a 2 cm depth of dilute hydrochloric acid in a boiling tube. Decant 1 cm depth of this solution into two test-tubes.		

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To one test-tube, add aqueous sodium hydroxide.	White ppt formed; insoluble in excess NaOH	No (observable) reaction / no change / no ppt
To the second test-tube, add aqueous ammonia.	White ppt formed; insoluble in excess $NH_3(aq)$	No (observable) reaction / no change / no ppt

(b) Mix a spatula measure of **FA 6** with a spatula measure of **FA 7**. Heat the mixture in a boiling tube. Record your observations.

State the type of reaction that has occurred.

Observations:

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

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Type of reaction: Acid-base

(c) From your observations in **(a)** and **(b)**, identify the possible ions present in **FA 6** and **FA 7**.

If you are unable to identify an ion, write 'unknown'.

	cation	anion
FA 6	Mg^{2+}	OH^-/ strong conjugate base
FA 7	NH_4^+	Unknown

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55	

[Total: 8]

END OF PAPER