1 Determination of solubility product, K_{sp}, of magnesium carbonate

Magnesium carbonate is basic and sparingly soluble in water, resulting in its use as drying agents in sports and antacids.

You are to carry out a titrimetric analysis to determine the solubility product of magnesium carbonate.

A saturated solution of magnesium carbonate can be prepared by dissolving excess solid magnesium carbonate into a measured volume of water. The mixture will be left to stand for some time before filtering. The filtrate can then be analysed through titration with a standard solution of hydrochloric acid.

The saturated solution of magnesium carbonate, FA 1, has been prepared for you, according to the procedure in **(a)**.

(a) Preparation of FA 1

1. Use a measuring cylinder to transfer 50 cm^3 of deionised water into a 100 cm^3 beaker.
2. Use a spatula to add solid magnesium carbonate into the same beaker, a little at a time with stirring until no more can dissolve.
3. Leave to stand for five minutes.
4. Filter the mixture to obtain the filtrate in a 250 cm^3 conical flask. Ensure that the filtrate is clear. Do not wash the residue.
5. The filtrate is **FA 1**.

You are provided with:
FA 1 saturated solution of magnesium carbonate
FA 2 0.0300 mol dm^{-3} hydrochloric acid solution
Methyl orange indicator

(b) (i) Dilution of FA 2

The concentration of **FA 2** is too high to be used in the titration. You will dilute **FA 2** before using it in **(c)**.

1. Pipette 25.0 cm^3 of **FA 2** into a 250 cm^3 volumetric flask.
2. Top up to the mark with deionised water.
3. Stopper and shake thoroughly.

(b) **(ii)** Calculate the concentration of hydrochloric acid in **diluted FA 2**.

[HC*l*] in **diluted FA 2** = ………………………………… [1]

(c) **(i)** **Titration of FA 1 against diluted FA 2**

1. Fill the burette with **diluted FA 2** from **(b)**.
2. Use a pipette to transfer 10.0 cm^3 of **FA 1** into a 100 cm^3 conical flask.
3. Add 1 – 2 drops of methyl orange indicator into the conical flask.
4. Run **diluted FA 2** from the burette into this flask until the appropriate colour change is observed.
5. Record your titration results, to an appropriate level of precision, in the space provided.
6. Repeat steps 1 to 5 until consistent results are obtained.

Results

[3]

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

Volume of **diluted FA 2** = ………………………………… [3]

(d) **(i)** Calculate the concentration of carbonate ions in **FA 1**, using your answers in **(b)(ii)** and **(c)(ii).**

$[CO_3^{2-}]$ in **FA 1 =** ………………………………… [2]

(ii) Hence, calculate the solubility product of magnesium carbonate. State its units.

K_{sp} of magnesium carbonate = ……………………………………

units ………………………………… [1]

(e) In **(a)**, it was instructed that the filtrate had to be clear and that the residue should not be washed.

(i) State and explain the effect on titre volume if the filtrate was **not** clear.

effect on titre volume ……………………………………………………………………

explanation ………………………………………………………………………………

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………………………………………………………………………………………… [1]

(ii) Explain why the residue should not be washed.

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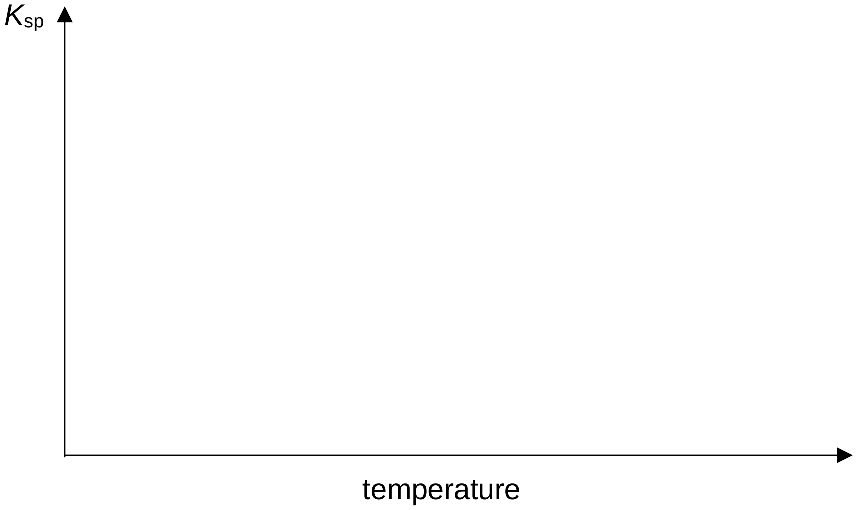
………………………………………………………………………………………… [1]

(f) The dissociation of magnesium carbonate in water is endothermic.

Sketch a graph to show how the solubility product of magnesium carbonate varies with temperature. Explain your answer.



explanation ………………………………………………………………………………………

……………………………………………………………………………………………………

…………………………………………………………………………………………… [2]

(g) **Planning**

The technique of gravimetry includes all analytical methods in which the analytical property is a measurement of mass or a change in mass. This technique can also be used in determining the solubility product of magnesium carbonate.

One of the possible approaches is to determine the mass of precipitate formed after mixing two solutions of known concentration. After preparing a mixture of saturated solution containing the precipitate, it can be passed through a pre-weighed filter paper that retains the precipitate. Residual moisture can be removed by drying the filter paper and the precipitate. The amount of precipitate can be determined, from which the concentration of ions in the saturated solution and hence solubility product can be determined.

(i) Plan an investigation to determine the solubility product of magnesium carbonate using the approach outlined above.

You are provided with the following:

- 0.400 mol dm^{-3} sodium carbonate solution
- 0.400 mol dm^{-3} magnesium nitrate solution
- apparatus and equipment normally found in a school laboratory

In your plan you should include brief details of:

- the apparatus you would use,
- the quantities you would use,
- the procedure you would follow,
- the measurements you would make to allow for gravimetric analysis.

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…………………………………………………………………………………………… [4]

(ii) Show how the measurements recorded in **(g)(i)** can be used to calculate the concentration of Mg^{2+} and CO_3^{2-}, in mol dm^{-3}, in the saturated solution.

[2]

[Total: 20]

2 Determination of the concentration of $NaHCO_3$ and the value for the enthalpy change of reaction of $NaHCO_3$ and NaOH

FA 3 is a solution of sodium hydrogencarbonate, $NaHCO_3$, of unknown concentration.
FA 4 is 1.50 mol dm^{-3} sodium hydroxide, NaOH.

In this experiment, you will perform a thermometric titration to determine the equivalence point for the reaction of **FA 3** and **FA 4**, where maximum heat is evolved, without the use of an indicator.

You will follow the reaction by measuring the temperature as volumes of **FA 4** are added in regular portions from a burette to a fixed volume of **FA 3** placed in a Styrofoam cup.

The data obtained will allow you to determine the temperature change and then analyse your results graphically in order to determine the equivalence point of the reaction.

In an appropriate format in the space provided on the next page, prepare a table in which you may record the following after each addition of **FA 4**:

- Total volume of **FA 4** added from the burette, starting from 0.00 cm^3, up to the point in time
- Total volume of solution in the cup, V_{total}
- Temperature measured, T

In the same table, you also need to calculate the corresponding values of:

- $\Delta T = T - T_0$, where T_0 is the initial temperature of **FA 3**
- ($V_{total} \times \Delta T$) to 3 significant figures

State clearly the units of each parameter on the header of the column.

Procedure

1. Fill the burette to the 0.00 cm^3 mark with **FA 4**.
2. Place the Styrofoam cup in a 250 cm^3 beaker to provide support for the cup.
3. Use a measuring cylinder to transfer 40.0 cm^3 of **FA 3** into the cup.
4. Measure and record the initial temperature of **FA 3**, T_0.
5. Run 3.00 cm^3 of **FA 4** from the burette into the cup, stir the solution with the thermometer and record the maximum temperature, T.
6. Immediately run a further 3.00 cm^3 of **FA 4** from the burette into the cup, stir and record the maximum temperature as before.
7. Continue the addition of **FA 4** in 3.00 cm^3 and record the maximum or minimum temperature reached after each addition. Do this until a total of 45.00 cm^3 of solution have been run from the burette.

(a) (i) Results

[3]

(a) **(ii)** Plot a graph of ($V_{total} \times \Delta T$) against total volume of **FA 4** added.

Draw two best-fit straight lines for the ascending and descending points respectively. Extrapolate both lines until they intersect to determine the:

- equivalence point of the reaction, V_{eq}
- maximum value of ($V_{total} \times \Delta T$)

V_{eq} = ……………………………………

Value of ($V_{total} \times \Delta T$) = …………………………….. [5]

(b) **(i)** Write down the chemical equation, with state symbols, for the reaction of $NaHCO_3$ and NaOH.

…………………………………………………………………………………………. [1]

(ii) Calculate the concentration of $NaHCO_3$ in **FA 3**.

[$NaHCO_3$] in **FA 3** = ………………………………… [2]

(iii) Given that the heat capacity of solution is 4.2 J K^{-1} cm^{-3}, calculate the enthalpy change of the reaction of $NaHCO_3$ and NaOH, ΔH_r.

ΔH_r = ………………………………… [2]

(c) You plotted a graph of (V_{total} × ΔT) against total volume of **FA 4** added in **(a)(ii)**. Suggest, with explanation, whether plotting a graph of ΔT against total volume of **FA 4** instead is likely to yield a more accurate result for the equivalence point.

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…………………………………………………………………………………………. [2]

(d) Performing the same experiment using a burette instead of a measuring cylinder to transfer **FA 3** into the Styrofoam cup is **not** expected to improve the accuracy of the results obtained. Suggest a reason why is this so.

…………………………………………………………………………………………………..

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………………………………………………………………………………………………… [1]

(e) You are provided with methyl orange indicator in **Question 1**.

Explain whether methyl orange indicator can be used to determine the end-point of the titration of $NaHCO_3$ against NaOH instead of performing thermometric titration.

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………………………………………………………………………………………………… [2]

[Total: 18]

3 Qualitative Analysis and Planning

You are provided with four different compounds labelled **FA 5**, **FA 6**, **FA 7** and **FA 8**. Each of these four bottles contains one of the following:

primary alcohol   aldehyde   monocarboxylic acid   metal halide

You will identify **FA 5**, **FA 6**, **FA 7** and **FA 8** by carrying out the prescribed experiments, as well as planning and conducting your own experiments.

For the prescribed experiments, record your observations in Table 3.1 below carefully. Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured.

You are advised to carry out the experiments in test (a) and (b) on each unknown compound one at a time. You are not required to carry out experiments in test (c) as the results are provided.

Table 3.1

Test		*Observations*			
		FA 5	**FA 6**	**FA 7**	**FA 8**
(a)	To 1 cm depth of the unknown in a test-tube, add 1 cm depth of sodium nitrite. **Test and determine if any gas is evolved.**				
(b)	To 1 cm depth of the unknown in a test-tube, add 1 cm depth of dilute sulfuric acid, followed by 5 drops of potassium manganate(VII) solution. Place the test-tube in the hot water bath. **Test and determine if any gas is evolved.**				

(c)	To 1 cm depth of aqueous iodine, add aqueous sodium hydroxide dropwise until a permanent yellow colour is obtained. Then add 3 drops of the unknown into the same test-tube. Place the test-tube in the hot water bath.	No yellow precipitate	No yellow precipitate	Yellow precipitate obtained	Yellow precipitate obtained

[4]

(d) **Identifying unknown metal halide**

(i) From your observations in Table 3.1, suggest which one of the unknown compounds is the metal halide.

FA …………

explanation ………………………………………………………………………………..

……………………………………………………………………………………………

…………………………………………………………………………………….. [1]

(ii) Suggest the reagent and carry out the test to confirm the identity of the halide in the metal halide.

reagent …………………………………………………………………………………

observations ……………………………………………………………………………

……………………………………………………………………………………………

FA ………… contains …………………….. anion. [2]

(d) **(iii)** In Table 3.2 below, describe two tests that will allow you to identify the cation in the metal halide. Perform the tests and record the observations in the same table. Use only bench reagents provided.

Table 3.2

Test	*Observations*
	FA …………

The cation in metal halide is ..………………………… [3]

(iv) The nature of the cation of the metal halide in aqueous solution accounts for its observations in test **(a)**. Describe and explain the nature of the cation by means of an equation.

equation ……………………………………………………………………………..

explanation …………………………………………………………………………..

………………………………………………………………………………………

…………………………………………………………………………………….. [2]

(e) **Identifying organic liquids**

(i) The three unknown organic compounds are primary alcohol, aldehyde and monocarboxylic acid.

Based on your observations in tests **(a)** and **(b)**, identify which is the monocarboxylic acid. Suggest the identity of the monocarboxylic acid and support your answer by giving the evidence.

FA …………

The identity of monocarboxylic acid is ……………………………………….

evidence ……………………………………..…………………………………….

……………………………………..…………………………………………….

……………………………………………….……………………………………… [2]

(ii) Describe how you would conduct a chemical test, using only the bench reagents provided, to distinguish between the remaining two unknown organic compounds.

Carry out the chemical test.

With aid of your observations and results provided in test **(c)**, identify which is the primary alcohol and the aldehyde.

chemical test …………………………………………………………………...

……………………………………………………………………………………….

observations ……………………………………………………………………

……………………………………………………………………………………….

The primary alcohol is **FA** …………

The identity of the primary alcohol is ……………………………………….

The aldehyde is **FA** …………

The identity of the aldehyde is ………………………………………. [3]

[Total: 17]

END OF PAPER

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

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 A*l* foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and A*l* foil; NO liberated by dilute acids (colourless NO → (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 on warming with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in dilute strong acids)

(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

(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