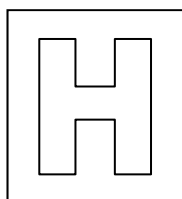


Candidate Name: _____

Class Adm No

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| Shift |
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| Laboratory |
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2018 Preliminary Exams Pre-University 3

H2 CHEMISTRY

9729/04

Paper 4 Practical

10th Sept 2018

2 hour 30 mins

Candidates answer on the Question paper.

READ THESE INSTRUCTIONS FIRST

Do not turn over this question paper until you are told to do so

Write your name, class and admission number on all the work you hand in.

Write in dark blue or black pen.

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

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

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

The use of an approved scientific calculator is expected, where appropriate.

You may lose marks if you do not show your working or if you do not use appropriate units.

Qualitative Analysis Notes are printed at the back of the Question Paper.

At the end of the examination, fasten all your work securely together.

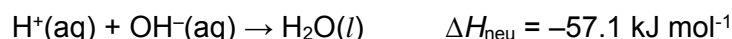
The number of marks is given in brackets [] at the end of each question or part question.

| Question | 1 | 2 | 3 | 4 | Total |
|----------|----|----|---|----|-------|
| Marks | 26 | 13 | 6 | 10 | 55 |

1 Investigation of acid-base titrations involving sodium hydrogen carbonate

According to the Arrhenius theory of acids and bases, an acid produces $\text{H}^+(\text{aq})$ ions and a base produces $\text{OH}^-(\text{aq})$ ions. The reaction of these two ions to form water molecules is known as acid-base neutralisation.

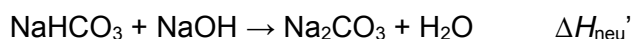
The equation for this neutralisation reaction is given below, and strong acid–strong base reactions are known to have an enthalpy change of neutralisation (ΔH_{neu}) of approximately $-57.1 \text{ kJ mol}^{-1}$.



However, the ΔH_{neu} for weak acid–strong base reactions are known to differ. Sodium hydrogen carbonate, NaHCO_3 , is an example of a weak acid.

FA 1 is 1.8 mol dm^{-3} sodium hydrogen carbonate, NaHCO_3 .

FA 2 is sodium hydroxide, NaOH , of concentration between $0.9 - 1.2 \text{ mol dm}^{-3}$.



As the precise concentration of **FA 2** is unknown, determination of $\Delta H_{\text{neu}}'$ can be done using a thermometric titration to simultaneously determine both the concentration of **FA 2** as well as $\Delta H_{\text{neu}}'$. Thermometric titration is a technique whereby equivalence points of a reaction can be located by observing temperature changes, hence eliminating the need for an indicator.

In **1(a)**, you will perform a weak acid–strong base thermometric titration. The data from this titration will be used to determine:

- the titration value at equivalence point, V_{eq} ,
- the precise concentration of **FA 2**, $[\text{NaOH}]$,
- the maximum temperature change, ΔT_{max} ,
- the enthalpy change of neutralisation, $\Delta H_{\text{neu}}'$.

(a) Determination of V_{eq} and ΔH_{neu} using thermometric titration

For this experiment, you will need to measure the **maximum temperature** of the reaction mixture when specified volumes of **FA 1** have been added.

In an appropriate format in the space provided on the **next page**, prepare a table to record your results. Record all values of temperature, T , to 0.1°C , and each total volume of **FA 1** added.

Note: You should aim to perform each subsequent addition of **FA 1** quickly.

1. Fill a burette with **FA 1**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 2** into a Styrofoam cup. Place this cup inside a second Styrofoam cup, which is placed in a 250 cm^3 glass beaker.
3. Stir and measure the temperature of this **FA 2**. Record this temperature.
4. Add 2.00 cm^3 of **FA 1** from the burette to the **FA 2** in the Styrofoam cup.
5. Using the thermometer, stir the mixture thoroughly and record the maximum temperature reached and the volume of **FA 1** added.
6. Repeat steps **4** and **5** until a total volume of 30.00 cm^3 of **FA 1** has been added.

Results

| Vol of FA 1 added / cm ³ | T / °C |
|--|--------|
| 0.00 | 29.4 |
| 2.00 | 30.4 |
| 4.00 | 31.5 |
| 6.00 | 32.2 |
| 8.00 | 32.9 |
| 10.00 | 33.4 |
| 12.00 | 34.0 |
| 14.00 | 33.9 |
| 16.00 | 33.6 |
| 18.00 | 33.3 |
| 20.00 | 33.0 |
| 22.00 | 32.8 |
| 24.00 | 32.6 |
| 26.00 | 32.4 |
| 28.00 | 32.3 |
| 30.00 | 32.2 |

M1
all 16
readings of
vol and T
tabulated
with correct
headers and
units

M2
T to ± 0.1 °C

- (i) Plot a graph of temperature, T , on the y -axis, against volume of **FA 1** added, on the x -axis on the grid in **Fig. 1.1**.

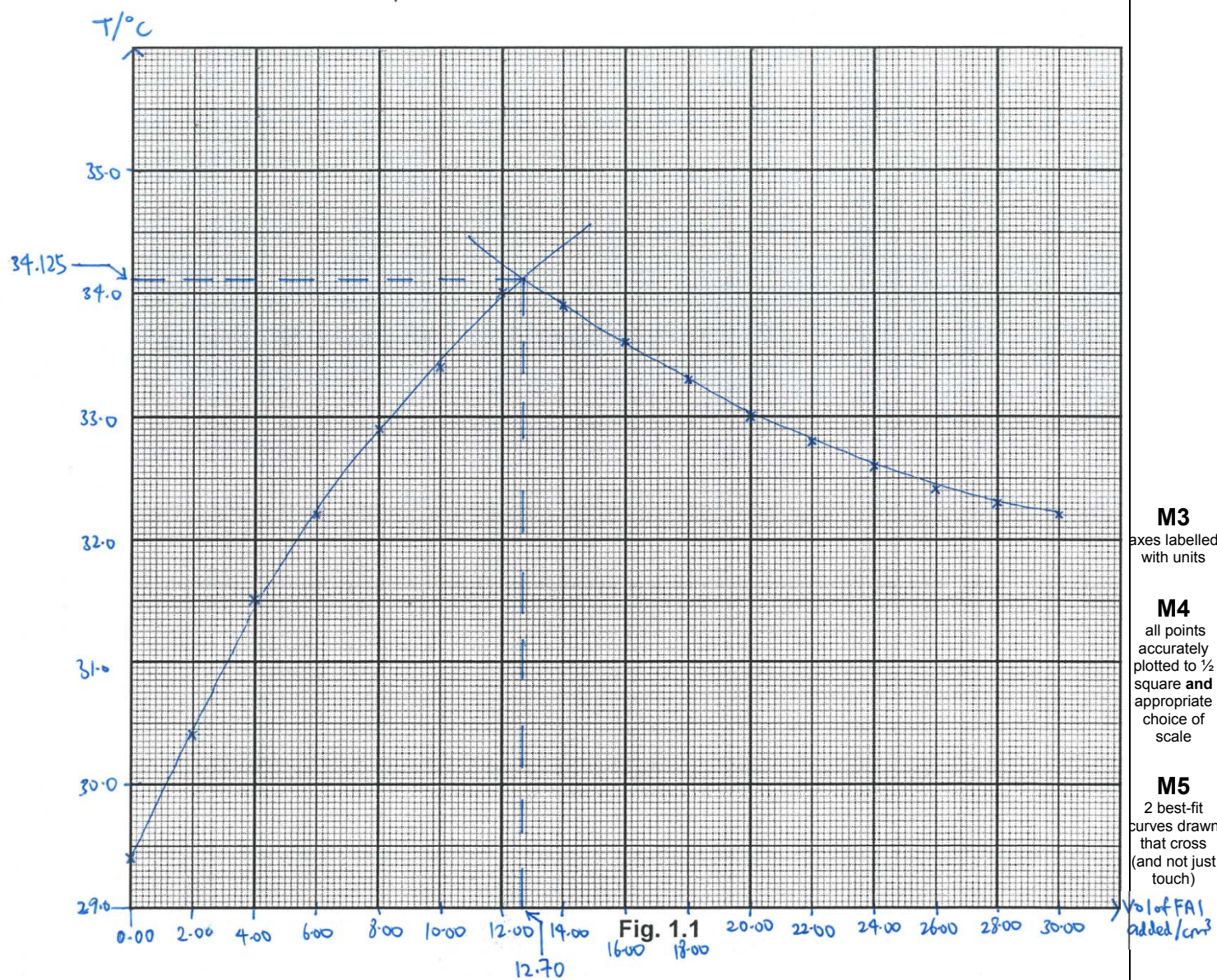


Fig. 1.1

Draw **two** most appropriate best-fit lines in **Fig. 1.1**, taking into account all of your plotted points.

Extrapolate (extend) these two best-fit lines until they cross each other.

[3]

(ii) From your graph in **Fig. 1.1**, determine:

- the titre at equivalence point, V_{eq} ,
- the maximum temperature reached, T_{max} ,
- the maximum temperature change, ΔT_{max} .

On your graph, show clearly how you obtained these values.

$$\Delta T_{\text{max}} = 34.125 - 29.4 = 4.7 \text{ }^{\circ}\text{C}$$

$$V_{\text{eq}} = 12.70 \text{ cm}^3$$

$$T_{\text{max}} = 34.1 \text{ }^{\circ}\text{C}$$

$$\Delta T_{\text{max}} = 4.7 \text{ }^{\circ}\text{C}$$

[3]

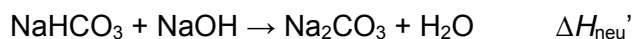
(iii) Determine the concentration of NaOH, [NaOH], in **FA 2**.

$$\text{Amount of NaHCO}_3 \text{ reacted} = 1.8 \times 12.70 \times 10^{-3} = 0.02286 \text{ mol}$$

$$[\text{NaOH}] = \frac{0.2286}{25.0 \times 10^{-3}} = 0.914 \text{ mol dm}^{-3}$$

$$[\text{NaOH}] \text{ in FA 2} = 0.914 \text{ mol dm}^{-3} \text{ [1]}$$

(iv) Determine the enthalpy change of neutralisation, $\Delta H_{\text{neu}}'$.



Assume that the reaction mixture has a density of 1.00 g cm^{-3} and a specific heat capacity, c , of $4.18 \text{ J g}^{-1} \text{ K}^{-1}$.

$$m = 25.0 + 12.70 = 37.7 \text{ g}$$

$$q = mc\Delta T = (37.7)(4.18)(4.7) = 740.6 \text{ J}$$

$$\Delta H = -\frac{q}{n_{LR}} = -\frac{740.6}{0.02286} = -32.4 \text{ kJ mol}^{-1} \text{ (3 sf)}$$

$$\Delta H_{\text{neu}}' = -32.4 \text{ kJ mol}^{-1} \text{ [3]}$$

(v) Comment on your value of $\Delta H_{\text{neu}}'$ obtained compared to $\Delta H_{\text{neu}} = -57.1 \text{ kJ mol}^{-1}$.

The value of $\Delta H_{\text{neu}}'$ obtained is less exothermic, as some of the heat energy released was used to completely dissociate the weak acid NaHCO₃.

[1]

For
Examiners'
Use

M6
both
construction
lines shown
on graph

M7
both readings
correct to $\frac{1}{2}$
square

M8
accuracy of
 ΔT_{max} ,
student value
 ≤ 0.6 from
supervisor's
value

M9

M10
correct mass
used

M11
correct
calculations,
do not mark
for sign

M12
all calc in (a)
to 3 or 4 sf
and have
correct units
and correct
sign for ΔH

M13
comparison
unambiguous
(do not
accept "value
s more/less")

- (vi) From your graph in **Fig. 1.1**, explain the shape of your best-fit line **before** equivalence point.

The curve is increasing with a decreasing gradient as the exothermic reaction releases the same amount of heat energy for each addition of FA 1, which causes a smaller temperature rise as the mass of the mixture increases. [1]

M14

(b) Determination of titration value at equivalence point, V_{eq}' , using 'regular' titration

FA 3 is hydrochloric acid, HCl , of unknown concentration.

Sodium hydrogen carbonate, NaHCO_3 , is also able to act as a weak base. In **1(a)**, NaHCO_3 acts as the acid while in **1(b)**, NaHCO_3 acts as the base.

For this experiment, you will titrate **FA 3** against **FA 1** to determine the titration value at equivalence point, V_{eq}' , using methyl orange as the indicator.

- (i) The use of thermometric titration in **1(a)** eliminated the need for an indicator as the equivalence point was located by observing temperature changes. 'Regular' acid-base titrations however, require the use of an indicator.

Explain why an indicator is required for 'regular' acid-base titrations (such as in **1(b)**).

To give a visible observation that corresponds to completion of reaction.

[1]

Titration of FA 3 against FA 1

1. Fill the burette with **FA 1**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 3** into a conical flask.
3. Add 2 – 3 drops of methyl orange indicator into the same conical flask.
4. Run **FA 1** from the burette into this flask until end-point is reached.
5. Record your titration results in the space provided. Make certain that your recorded results show the precision of your working.
6. Repeat points **1** to **5** as necessary until consistent results are obtained.

Results

| | 1 | 2 | 3 |
|---|-------|-------|---|
| Initial burette reading / cm^3 | 0.00 | 6.70 | |
| Final burette reading / cm^3 | 21.60 | 28.20 | |
| Volume of FA 1 added / cm^3 | 21.60 | 21.50 | |
| | ✓ | ✓ | |

[3]

- (ii) From your titrations, obtain a suitable volume of **FA 1** to be used in your calculations (titre at equivalence point, V_{eq}'). Show clearly how you obtained this value.

$$V_{eq}' = \frac{21.60 + 21.50}{2} = 21.55 \text{ cm}^3$$

$$V_{eq}' = 21.55 \text{ cm}^3 \text{ [1]}$$

For
Examiners'
Use

M15

M16
correct
headers and
units

M17
volumes to
 $\pm 0.05 \text{ cm}^3$
and correct
subtraction

M18
accuracy,
student value
 ≤ 0.6 from
supervisor's
value

M19

- (iii) Determine the concentration of HCl, [HCl], in **FA 3**.



$$\begin{aligned} \text{Amount of NaHCO}_3 &= 1.8 \times 21.55 \times 10^{-3} = 0.03879 \text{ mol} \\ &= \text{Amount of HCl} \end{aligned}$$

$$[\text{HCl}] = \frac{0.03879}{25.0 \times 10^{-3}} = 1.55 \text{ mol dm}^{-3} \text{ (3 sf)}$$

$$[\text{HCl}] \text{ in FA 3} = \underline{1.55 \text{ mol dm}^{-3}} \text{ [1]}$$

For
Examiners'
Use

M20

- (iv) Suggest if the use of more drops of methyl orange indicator is appropriate for this titration.

No, it is not appropriate as indicators are weak acids or bases and will affect the pH of the solution. [1]

M21

(c) Planning

The same 'regular' titration in **1(b)** can also be performed using an alternative method involving a pH meter, which is an instrument that can measure the pH of water-based solutions at any one instance of time.

Usage of a pH meter is simple as a digital pH reading will be displayed upon placing it into the solution of interest. However, it is known that most pH meters have to be calibrated (correlate to a standard) before each experiment to ensure the accuracy of measurements. For this titration, the standard chosen for calibration is usually a buffer solution of $\text{pH} \approx 4.0$.

Plotting pH against titration volume, V , will give a pH curve that enables the determination of titration value at equivalence point, V_{eq} .

Plan an experiment to determine the titration value at equivalence point, V_{eq} , for the titration of **FA 3** against **FA 1** using a pH meter.

You may assume that you are provided with

- a pH meter,
- **FA 1** and **FA 3**,
- 1.0 mol dm^{-3} of ethanoic acid, CH_3COOH ($K_{\text{a}} = 1.8 \times 10^{-5}$),
- 1.0 mol dm^{-3} of sodium ethanoate solution, $\text{CH}_3\text{COO}^-\text{Na}^+$,
- the glassware and equipment normally found in a school or college laboratory.

In your plan you should include brief details of

- the preparation of a suitable standard solution for calibration of the pH meter,
- the procedure that you would follow,
- the measurements you would take,
- an outline of how you would use your results to determine V_{eq} .

$$\text{pH} = \text{p}K_{\text{a}} + \lg \frac{[\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

$$4.0 = -\lg(1.8 \times 10^{-5}) + \lg \frac{n_{\text{CH}_3\text{COO}^-}/V}{n_{\text{CH}_3\text{COOH}}/V}$$

$$\frac{n_{\text{CH}_3\text{COO}^-}}{n_{\text{CH}_3\text{COOH}}} = 0.18$$

Since concentrations of ethanoic acid and sodium ethanoate are the same, $n \propto V$.
 \therefore for 18 cm^3 of ethanoic acid used, use 100 cm^3 of sodium ethanoate.

Preparation of standard solution and calibration of pH meter

1. Using two separate burettes, add 18.00 cm^3 of 1.0 mol dm^{-3} ethanoic acid and 100.00 cm^3 of 1.0 mol dm^{-3} sodium ethanoate to a 250 cm^3 volumetric flask.
2. Top up the volumetric flask to the mark with distilled water, stopper, invert and shake until homogeneous.
3. Pour out some of the standard solution into a 50 cm^3 beaker and place the pH meter probe into it. Calibrate the pH meter to read 4.0.
4. Wash the pH meter probe with distilled water before usage in experiment.

Procedures for titration and measurements taken

5. Carry out the same 'regular' titration as in **1(b)**, replacing the methyl orange indicator with the pH meter probe.
6. Record the pH measurements at 1 cm^3 intervals until 25.00 cm^3 of **FA 3** has been added.

For
Examiners'
Use

M22

correct choice of volumes to obtain pH between 3.9-4.1

M23

correct procedures for preparation of std sol

M24

calibration of pH meter, including washing of probe

M25

correct modification and measurements taken

Determining V_{eq}''

7. Plot a graph of pH against volume of **FA 3** added. The vertical region corresponds to the equivalence point.
8. Draw a vertical construction line from this vertical region to the x-axis to determine the value of V_{eq}'' .

[5]

[Total: 26]

M26correct usage
of plot /
sketch of plot
to determine
 V_{eq}''

2 Determination of the major component in a solid mixture

Sodium hydrogen carbonate, NaHCO_3 , also commonly known as *baking soda*, is a white crystalline solid primarily used in baking as a raising agent.

To increase the strength of *baking soda* as a raising agent, cream of tartar (a dry acid) is mixed with sodium hydrogen carbonate. This mixture is known as *baking powder*.

FA 4 is a sample of *baking powder*.

In this experiment, you will determine if sodium hydrogen carbonate is the major component, by mass, of the mixture **FA 4**.

(a) Thermal decomposition of *baking powder*

You may assume that the cream of tartar in **FA 4** is inert and does not decompose when heated.

1. Weigh and record the mass of an empty boiling-tube.
2. Transfer approximately 2 g of **FA 4** into the weighed boiling-tube. Reweigh and record the mass of the boiling-tube and **FA 4**.
3. Gently heat the **FA 4** in the boiling-tube for 2 minutes, then heat strongly for a further 2 minutes. **Take care not to lose any solid from the tube during heating.**
4. Warm the upper parts of the boiling-tube to evaporate any water that may have condensed while heating the solid.
5. Place the hot tube on the test-tube rack and leave to cool. **You are advised to continue with part 2(c) or to start another question while the tube cools.**
6. When cool, reweigh the boiling-tube and the residual solid.
7. Reheat, cool and reweigh the tube until decomposition is complete.

In an appropriate form in the space below, record all your balance readings, the mass of **FA 4** heated, the mass of residual solid, and the mass lost on heating.

Results

| | |
|---|-------|
| Mass of empty boiling-tube / g | 30.03 |
| Mass of boiling-tube + FA 4 / g | 32.14 |
| Mass of FA 4 heated / g | 2.11 |
| Mass of boiling-tube + residual solid (1) / g | 31.55 |
| Mass of boiling-tube + residual solid (2) / g | 31.53 |
| Mass of residual solid / g | 1.50 |
| Mass lost / g | 0.61 |

For
Examiners'
Use

M27
at least 4
balance
readings and
1 calculation
(accept tare)

M28
correct
headers (at
most 1
missing) and
units and all
mass
readings to
2dp

M29
correct
subtractions
(all three)

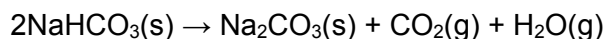
M30
evidence of
constant
mass (within
0.05g)

M31
accuracy of
ratio (mass
heated /
mass of
residue) ≤ 0.2
from
supervisor's
value

[5]

The thermal decomposition of NaHCO_3 produces Na_2CO_3 and two gases at the temperature of decomposition, CO_2 and H_2O .

For
Examiners'
Use



- (i) By finding the average M_r of the two gases produced, use your results in (a) calculate the **total** amount of gases lost upon complete decomposition of **FA 4**.

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

$$\text{average } M_r = \frac{[12.0 + 2(16.0)] + [2(1.0) + 16.0]}{2} = 31.0$$

$$\text{total amount of gases lost} = \frac{0.61}{31.0} = 0.0197 \text{ mol (3 sf)}$$

M32

$$\text{total amount of gases lost} = 0.0197 \text{ mol [1]}$$

- (ii) Taking into account the mole ratio of gases in the decomposition equation given, calculate the amount of $\text{CO}_2(\text{g})$ lost upon complete decomposition of **FA 4**.

$$\text{amount of } \text{CO}_2 \text{ lost} = 0.0197 / 2$$

$$= 0.00984 \text{ mol (3 sf)}$$

M33

$$\text{amount of } \text{CO}_2(\text{g}) \text{ lost} = 0.00984 \text{ mol [1]}$$

- (iii) Hence, calculate the mass of NaHCO_3 in the sample of **FA 4** heated.

$$\text{amount of NaHCO}_3 \text{ decomposed} = 2 \times 0.009838$$

$$= 0.01967 \text{ mol}$$

$$\text{mass of NaHCO}_3 = 0.01967 \times [23.0 + 1.0 + 12.0 + 3(16.0)]$$

$$= 1.65 \text{ g (3 sf)}$$

$$\text{mass of NaHCO}_3 = \underline{1.65} \text{ g [1]}$$

M34

- (iv) By means of calculation or otherwise, justify if NaHCO_3 is the major component, by mass, of **FA 4**.

$$\% \text{ by mass of NaHCO}_3 = \frac{1.652}{2.11} \times 100$$

$$= 78.3 \% \text{ (3 sf)}$$

NaHCO_3 is the major component as it makes up more than 50% of the sample.

M35

[1]

- (b) **Do not carry out your suggestions.**

*For
Examiners'
Use*

Suggest two ways in which you could show that cream of tartar does not decompose on heating.

- (i) There is no change in mass when cream of tartar is heated.

M36

- (ii) There is no change in the volume of a gas syringe connected to the boiling-tube when cream of tartar is heated.

M37

[2]

award 1m if
both mass
and volume
stated but no
mention of
absence of
change

- (c) A student is asked to weigh, with maximum precision, a solid.

Three balances are available.

- Balance A, reading to 1 decimal place,
- Balance B, reading to 2 decimal places,
- Balance C, reading to 3 decimal places.

Balance readings can be treated similarly to burette readings.

For example, the smallest division on a burette is 0.1 cm^3 .
The maximum error in a single burette reading is $\pm 0.05 \text{ cm}^3$.

Complete the following table.

| balance | maximum error for a single balance reading / g | maximum % error when weighing: |
|---------|--|---|
| A | ± 0.05 | 8.0 g of solid = $\frac{2 \times 0.05}{8.0} \times 100 = 1.25\%$ |
| B | ± 0.005 | 4.00 g of solid = $\frac{2 \times 0.005}{4.00} \times 100 = 0.250\%$ |
| C | ± 0.0005 | 0.400 g of solid = $\frac{2 \times 0.0005}{0.400} \times 100 = 0.250\%$ |

[2]

[Total: 13]

M38
correct max error

M39
ecf only if
(i) max error given is x2
(ii) max errors are incorrect by factor 10

3 Qualitative Analysis of an unknown double salt

Double salts are salts that contain more than one cation or anion, and are synthesised by crystallising a solution containing the different ions.

FA 5 is a double salt which contains **two cations** and **one anion**.

Empty out the **FA 5** provided into a 50 cm³ beaker. To this beaker, add 10 cm³ of water and stir to dissolve as much of the solid as possible.

This solution will be referred to as '**FA 5** solution'.

- (a) Perform the tests described in **Table 3.1**, and record your observations in the table. Test and identify any gases evolved.

Table 3.1

| tests | | observations |
|-------|--|---|
| 1. | Add about 1 cm depth of FA 5 solution to a boiling-tube, followed by NaOH(aq) dropwise, until excess (a further 2 cm depth). Warm the solution gently. | Red-brown ppt forms, insoluble in excess NaOH(aq). Gas produced turns red litmus paper blue. |
| 2. | Add about 2 cm depth of FA 5 solution to a test-tube. To this test-tube, add about half a spatula of zinc powder. Observe the mixture until no further changes are seen. | Yellow solution decolourises / turns pale green. <i>accept: gas produced extinguishes lighted splint with a 'pop' sound</i> |
| 3. | Add about 1 cm depth of FA 5 solution into a test-tube. To this test-tube, add barium nitrate dropwise. | White ppt forms, insoluble in excess HNO ₃ (aq). <i>allow omission of solubility in strong acid, but do not award M44 if sulfite is proposed as anion</i> |

[4]

- (b) Based on your observations in **3(a)**, deduce the identities of the ions present in **FA 5**.

cation 1: Fe³⁺ cation 2: NH₄⁺ anion: SO₄²⁻

[1]

- (c) Hence, suggest a possible chemical formula of the double salt **FA 5**.

chemical formula of **FA 5**: Fe(NH₄)(SO₄)₂ [1]

[Total: 6]

For
Examiners'
Use

M40

M41

M42

M43

M44

do not accept H⁺ as it was not in original salt

M45

ecf only if H⁺ instead of NH₄⁺ and others correct

4 Planning

The condenser is an apparatus frequently used in organic synthesis. It comprises of concentric glass tubes, an inner one through which hot gases can pass through, and an outer one through which a cool fluid can pass through.

Use of the condenser is required for the organic synthesis of aldehydes and carboxylic acids from alcohols. The product of the reaction between potassium dichromate, $K_2Cr_2O_7$, and primary alcohols can either be an aldehyde or a carboxylic acid depending on how the condenser is orientated.

- (a) Some organic synthesis procedures require *heating under reflux*.

Explain the role of the condenser in such procedures.

To prevent the loss of volatile / low boiling point organic reagents by condensing hot vapour back into the reaction flask.

[2]

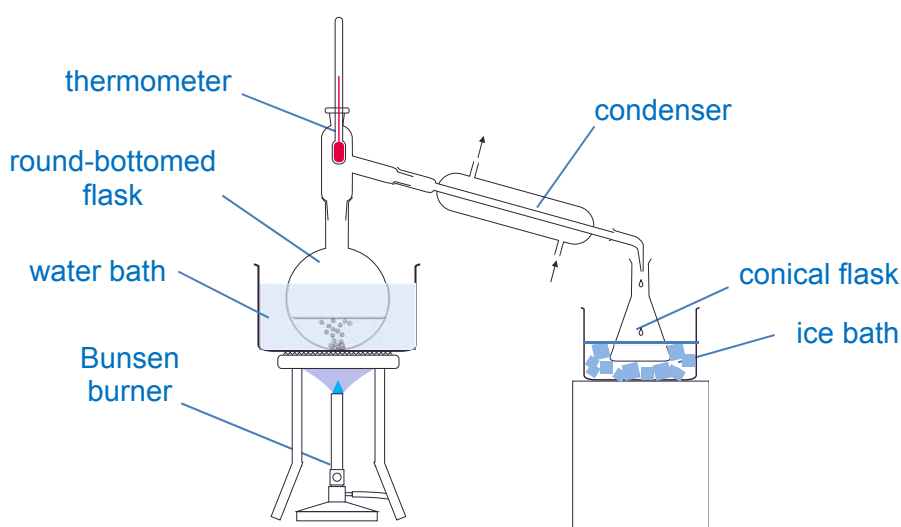
- (b) Plan an experiment to synthesise **propanal** (boiling point: $20\text{ }^{\circ}\text{C}$) from **propan-1-ol** (boiling point: $97\text{ }^{\circ}\text{C}$).

You may assume that you are provided with

- propan-1-ol, $\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$,
- potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$,
- commonly used organic chemicals
- the glassware and equipment normally found in a school or college laboratory.

In your plan you should include

- the reactants and conditions that you would use,
- a well-labelled diagram of the set-up that you would use,
- the procedure that you would follow and the safety precautions taken,
- how you would check the purity of your product.



Reactants: $\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$, $\text{K}_2\text{Cr}_2\text{O}_7$, dilute H_2SO_4
 Conditions: heat with distillation

For
Examiners'
Use

M46

M47

M48

correct
distillation
setup with
labels
(accept hot
plate, do **not**
mark for ice
bath)

M49

correct R&C
(must have
dilute acid
added), do
not mark for
precalc of
quantities

Procedure

1. To the 100 cm³ round-bottomed flask, add CH₃CH₂CH₂OH, K₂Cr₂O₇, and dilute H₂SO₄.
2. Set up the apparatus as shown in the diagram above in the fume cupboard / while wearing gloves / safety goggles.
3. Start the flow of water through the condenser and turn on the Bunsen burner to start the reaction. Maintain the water bath at approximately 40 °C. The temperature reading should remain constant at approximately 20 °C until all the propanal synthesised has boiled off.
4. When the temperature starts to increase further / no further distillate is collected in the conical flask, turn off the Bunsen burner to stop the reaction.
5. Test the purity of the propanal distillate by means of thin layer chromatography. A pure sample would only contain one spot after chromatography.

[4]

*For
Examiners'
Use*

M50

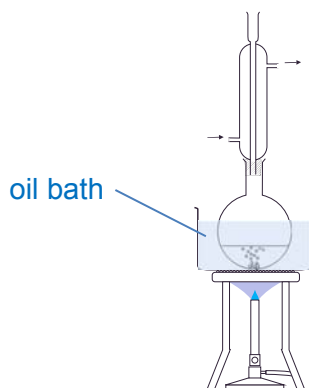
any 1 safety
procedure
written

M51

use of TLC to
test for purity
of product

- (c) Suggest appropriate modifications to your plan to synthesise **propanoic acid** (boiling point: 141 °C) from propan-1-ol instead of propanal.

You may wish to use diagrams to complement your answer.



Use a reflux setup instead of a distillation setup by orientating the condenser vertically.

Use a separatory funnel to extract out the propanoic acid product from the reaction mixture before testing its purity.

[2]

- (d) Give an explanation for any **two** modifications you have made in (c).

Reflux: To ensure that the volatile propanal does not escape the reaction flask and can continue to further be oxidised by $K_2Cr_2O_7$ into propanoic acid.

Separatory funnel: Unlike distillation, the propanoic acid product is mixed with residual reagents (such as propan-1-ol, $K_2Cr_2O_7$ and water) and has to be purified.

$KMnO_4$: Stronger oxidising agent used to ensure that all the propan-1-ol is fully oxidised into propanoic acid.

Heat longer: To ensure that all the propan-1-ol is fully oxidised into propanoic acid.

Oil bath: To allow for heating at higher temperatures close to boiling point of propan-1-ol as water will boil.

[2]

[Total: 10]

End of Paper 4

M52

diagram or mention of orientating condenser vertically. do not accept simply changing to $KMnO_4$

M53

mentions method to extract product from reaction mixture

M54

M55

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. insoluble in excess | green ppt. 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. insoluble in excess | off-white ppt. insoluble in excess |
| zinc, Zn ²⁺ (aq) | white ppt. soluble in excess | white ppt. soluble in excess |

(b) Reactions of anions

| ions | reaction |
|---|--|
| 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 with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acids) |

(c) Test for gases

| ions | reaction |
|-------------------------------|--|
| 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 |