

Suggested solution for 2017 SH 2 Prelim Practical

1 (a) (ii) Table 1: Weighings of FA1

Mass of weighing bottle and FA1 / g	3.696
Mass of weighing bottle and residual FA1 / g	3.497
Mass of FA1 transferred / g	0.199

Table 2: Titration of FA3 with FA4 using screened methyl orange as indicator

	1	2
Final burette reading / cm ³	30.00	36.90
Initial burette reading / cm ³	9.00	16.00
Volume of FA4 used / cm ³	21.00	20.90

(iii)
$$\text{average volume} = \frac{21.00 + 20.90}{2} = 20.95 \text{ cm}^3$$

(b) (i) amount of HNO₃ in 25.0 cm³ = amount of NaOH = 20.95 / 1000 x (9.10/40.0) = 4.77 x 10⁻³

amount of HNO₃ in 250.0 cm³ = 4.77 x 10⁻²

(ii) amount of HNO₃ in 25.0 cm³ = 25/1000 x 2 = 0.05 mol

(iii) amount of HNO₃ = 0.05 – 4.77 x 10⁻² = 2.34 x 10⁻³

(iv) amount of MgCO₃.xH₂O = 2.34 x 10⁻³ / 2 = 1.17 x 10⁻³

$M_r = 0.208 / 1.15 \times 10^{-3} = 170.2$

$x = [180.7 - (24.3 + 12.0 + 16.0 \times 3)] / 18 = 4.77 \approx 5$

(c) The acid will not be diluted and close to 2.00 mol dm⁻³, the titre value required to neutralise 25 cm³ of FA 3 will be about 250 cm³ / more than 10 times greater. Since this exceeds the capacity of burette, it is unsuitable.

2 (a) **Table 1: Weighings of FA1**

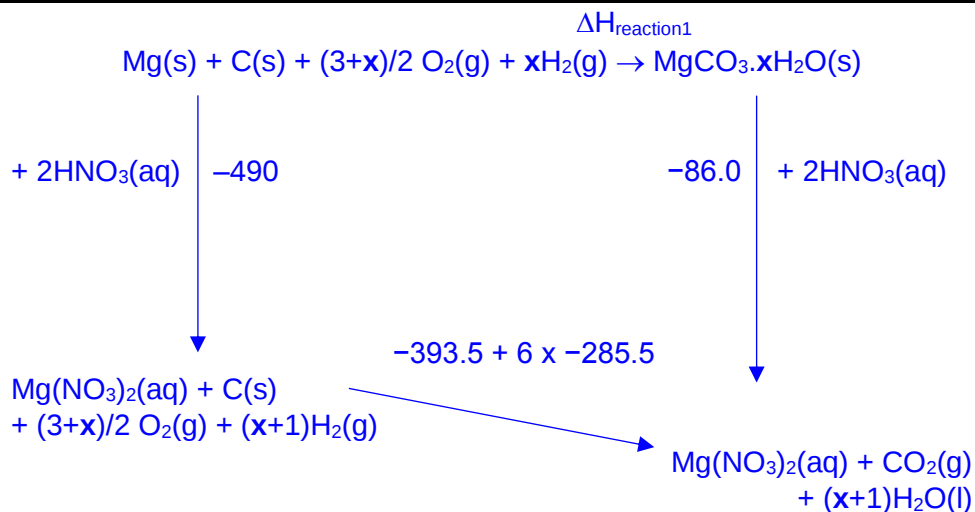
Mass of weighing bottle and FA1 / g	4.495
Mass of weighing bottle and residual FA1 / g	3.478
Mass of FA1 transferred / g	1.017

Table 2: Measurement of temperature

$T_i / ^\circ\text{C}$	30.3
$T_f / ^\circ\text{C}$	33.3
$\Delta T / ^\circ\text{C}$	3.0

(i) $\Delta q_{\text{reaction3}} = -mc\Delta T = -40 \times 4.18 \times 3.0 = -502 \text{ J}$

(iii)



By Hess's law,

$$\Delta H_{\text{reaction1}} = -490 - 393.5 + 6x - 285.5 - (-86.0) = -2510 \text{ kJ mol}^{-1}$$

OR

If they use $x = 3$

By Hess's law,

$$\begin{aligned}
 \Delta H_{\text{reaction1}} &= -490 - 393.5 + 4x - 285.5 - (-68.3) = -1957.2 \text{ kJ mol}^{-1} \\
 &= -1960 \text{ kJ mol}^{-1}
 \end{aligned}$$

(c) (i) $\text{percentage error} = (\pm 0.1 \times 2) / 3.0 \times 100 = \pm 6.67 \%$

(ii) $\Delta H_{\text{reaction3}} = -502 / (1.017 / 174.3) = -86.0 \text{ kJmol}^{-1}$
OR

If they use $x = 3$

$\Delta H_{\text{reaction3}} = -502 / (1.017 / 138.3) = -68.3 \text{ kJmol}^{-1}$

(ii) **Error:**
The experiment did not take into account heat exchange with surrounding / calorimeter / heat loss to surrounding / calorimeter.

Improvement 1:

Use cooling curve method where temperature changes after mixing are measured at 30s intervals for about 3 min, then max temperature is extrapolated to time of mixing. This accounts for heat exchange with surrounding, hence a more accurate ΔT is obtained.

Improvement 2:

Calibration of the calorimeter by making use of reaction with accurately known enthalpy change. The calibrated heat capacity, C of the calorimeter takes into account heat exchange with surrounding, hence a more accurate ΔT is obtained.

OR

Error:

The experiment did not repeat experiment to ensure consistent results.

Improvement:

Repeat experiment until $\Delta T/m$ between a pair of experiments is within 5%.

(d) **Suggested solution:**

Procedure:

1. Using three **10.0 cm³ measuring cylinders**, measure 5.0 cm³ of **FB 1**, **FB 2** and **FB 3**.
2. Mix separately, 5.0 cm³ of **FB 1** to 5.0 cm³ of **FB 2** and 5.0 cm³ of **FB 1** to 5 cm³ of **FB 3**, into 2 **test-tubes**
3. Measure for the rise in temperature using a **0.2 °C division thermometer**

Deduction:

- If both reaction mixture gives a temperature rise, **FB 1** is **HCl**.
- If one of the 2 reaction mixture does not give a temperature rise, then this pair of solutions must be **NH₃(aq)** and **KOH(aq)**. **HCl** can be identified.

Upon identifying **HCl**,

4. Using a 10.0 cm³ measuring cylinder, add 10.0 cm³ of **HCl** to a **Styrofoam cup** and measure the initial temperature, T_1 , using a **0.2 °C division thermometer**.

- Using another 10.0 cm³ measuring cylinder, transfer 10 cm³ of **one of the bases** into another Styrofoam cup.
- Wash and dry the thermometer
- Measure the initial temperature, T₂, of the **base** solution.
- Add **HCl** to **base** and stir with the thermometer and note the highest temperature reached, T_f and determine ΔT.
- Wash and dry the Styrofoam cup.
- Repeat steps 4 – 9 but replace base with the other base solution.

Assuming if **FB 1** is **HCl**

	FB 2	FB 3
T _i / °C		
T _f / °C		
ΔT / °C		

Deduction of results:

Since **HCl** is 2 mol dm⁻³, the limiting reagent is the base and the same amount of base is used / same amount of H₂O is produced in each reaction.

When total volume of reaction mixture is kept constant and the no of moles of water formed in the reaction is the same, ΔT depends only on the strength of the base used.

As weak acid dissociates partially, a portion of the energy released from neutralisation is used to complete the dissociation of the weak acid. Therefore less energy is released compared to a neutralisation between a strong acid and a strong base.

The reaction mixture that gives a lower ΔT must contain 1 mol dm⁻³ aqueous ammonia.

Alternative solution

Procedure:

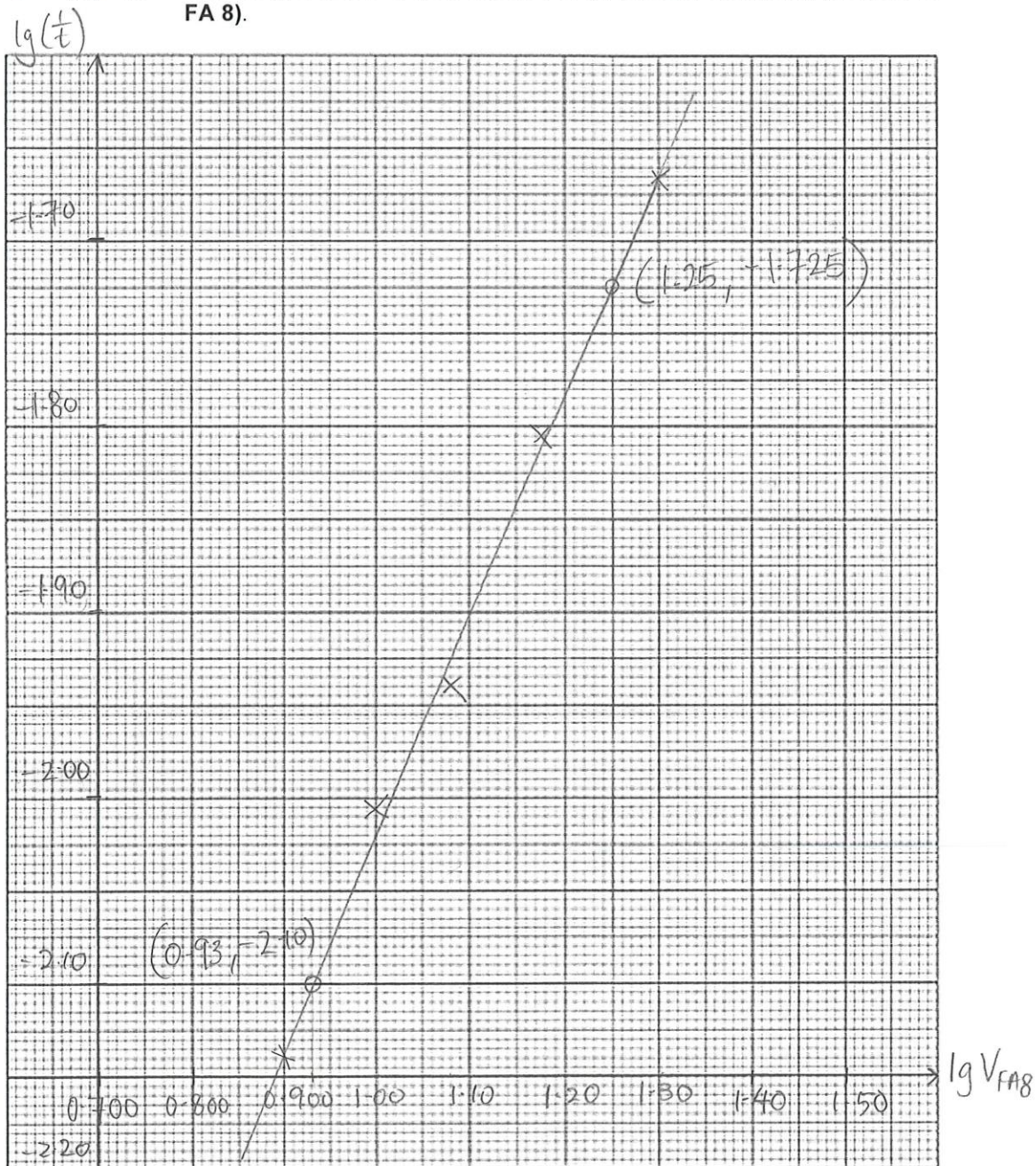
1. To 1 **test-tube**, add 5.0 cm³ of **FB 1** using a **10.0 cm³ measuring cylinder** and measure the initial temperature, T_1 , using a **0.2 °C division thermometer**.
2. Using another 10.0 cm³ measuring cylinder, measure 5.0 cm³ of **FB 2**.
3. Wash and dry the thermometer.
4. Measure the initial temperature, T_2 , of **FB 2**.
5. Transfer **FB 2** into the test-tube. Stir with the thermometer and note the highest temperature reached, T_f and determine ΔT .
6. Wash and dry the test-tube.
7. Repeat steps 1 – 6 but by mixing 5.0 cm³ of **FB 1** to 5 cm³ of **FB 3** and 5.0 cm³ of **FB 2** to 5 cm³ of **FB 3** respectively.

	FB 1	FB 2	FB 3
FB 1		$T_1 =$ $T_2 =$ $T_f =$ $\Delta T =$	$T_1 =$ $T_2 =$ $T_f =$ $\Delta T =$
FB 2			$T_1 =$ $T_2 =$ $T_f =$ $\Delta T =$

Results analysis: similar to first solution of using ΔT to identify the 3 solutions.

3	(a)	Expt	Vol of FA 8 / cm ³	Vol of DI / cm ³	time / sec	lg $V_{FA\ 8}$	lg $1/t$
		1	20.00	0.0	46	1.30	-1.66
		2	10.00	10.0	116	1.00	-2.06
		3	15.00	5.0	64	1.18	-1.81
		4	12.00	8.0	87	1.08	-1.94
		5	8.00	12.0	251	0.903	-2.40

- 3 (b) (i) Use the grid below to plot a graph of $\lg(1/\text{time})$ against $\lg(\text{volume of FA 8})$.



- (ii) Order of reaction wrt Fe^{3+} = gradient of graph
 $= (-1.725 - -2.10) / (1.25 - 0.93) = 1.17 \approx 1$

Test	Procedure	Observations
(a)	To 6 cm ³ of X , add barium nitrate solution until in excess.	White ppt. formed, insoluble in excess.
(b)	Filter the mixture from (a). Wash and retain the residue for test (c). Collect the filtrate for test (d) and (e).	
(c)	To separate portions of the residue (i) add 2 cm ³ of hydrochloric acid	A colourless and odourless gas evolved, which forms a white ppt with Ca(OH)₂
	(ii) add 2 cm ³ of organic compound Z [You are to test for any gas evolved]	A colourless and odourless gas evolved, which forms a white ppt with Ca(OH)₂
(d)	To 1 cm depth of the filtrate from (b) in a test-tube (i) add a few drops of organic compound Z and warm in a water bath for 5 minutes (ii) followed by 1 cm ³ of nitric acid and 5 drops of silver nitrate. Add excess ammonia solution.	Solution remains colourless. white ppt. formed, soluble in excess NH₃
(e)	To separate 1 cm depth of Y (i) add filtrate from (b) dropwise until in excess	Pale blue ppt. formed, insoluble in excess
	(ii) add NaOH(aq), followed by one spatula of zinc powder and warm. (CARE!)	Pale blue ppt. formed upon adding NaOH(aq). Vigorous effervescence, colourless and pungent gas evolved , which turns damp red litmus paper blue

(f)	(i)	carbonate / CO_3^{2-} in residue hydroxide / OH^- in filtrate															
	(ii)	<u>Carboxylic acid</u> is present. From test (c)(ii), <u>CO_2 is produced from the reaction of Z with carbonate</u> in the residue from (b), which suggests an acidic group is present. <u>Halogenoalkane</u> / <u>chlorine-containing</u> organic compound / <u>alkyl halides</u> / is present. From test (d)(i), a white ppt. is produced upon heating with OH^- and adding nitric acid and AgNO_3, which dissolves in excess NH_3, which suggests that the <u>white ppt. is AgCl</u>. Hence Cl is present in Z.															
(g)		cation: Cu^{2+} anion: NO_3^- / NO_2^-															
(h)		Possible solution:															
		<table><tr><th>Step</th><th>Procedure</th><th>Observations and conclusions</th></tr><tr><td>1</td><td>Add Cu^{2+} complex in NaOH(aq) to each sample in a test-tube, warm</td><td>For <u>propanal</u>, <u>brick-red ppt</u> is formed due to the presence of aliphatic aldehyde. For other compounds, <u>blue solution remains</u>.</td></tr><tr><td>2</td><td>Add 2,4-dinitrophenylhydrazine to a new sample of the remaining 3 unknowns, warm</td><td>For <u>benzaldehyde</u>, <u>orange ppt.</u> is formed due to the presence of carbonyl group. For remaining compounds, <u>no orange ppt. form</u>.</td></tr><tr><td>3</td><td>Add KMnO_4, dil H_2SO_4 to a new sample of the remaining 2 unknowns, heat</td><td>For <u>butanol</u>, <u>purple KMnO_4 decolourises</u> due to oxidation of primary alcohol. For the last compound, purple KMnO_4 remains.</td></tr><tr><td>4</td><td>Add $\text{PCl}_5(\text{s})$ to a new sample of the last unknown</td><td>For <u>2-methylpropan-2-ol</u>, <u>white fumes of HCl</u> observed due to presence of OH / alcohol group.</td></tr></table>	Step	Procedure	Observations and conclusions	1	Add Cu^{2+} complex in NaOH(aq) to each sample in a test-tube, warm	For <u>propanal</u> , <u>brick-red ppt</u> is formed due to the presence of aliphatic aldehyde. For other compounds, <u>blue solution remains</u> .	2	Add 2,4-dinitrophenylhydrazine to a new sample of the remaining 3 unknowns, warm	For <u>benzaldehyde</u> , <u>orange ppt.</u> is formed due to the presence of carbonyl group. For remaining compounds, <u>no orange ppt. form</u> .	3	Add KMnO_4 , dil H_2SO_4 to a new sample of the remaining 2 unknowns, heat	For <u>butanol</u> , <u>purple KMnO_4 decolourises</u> due to oxidation of primary alcohol. For the last compound, purple KMnO_4 remains.	4	Add $\text{PCl}_5(\text{s})$ to a new sample of the last unknown	For <u>2-methylpropan-2-ol</u> , <u>white fumes of HCl</u> observed due to presence of OH / alcohol group.
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