

Raffles Institution Year 6 H2 Chemistry 2025 Planning Experiments Lecture 2 Gravimetry, Gas Collection, Energetics and Kinetics

TAMP Framework

When approaching planning questions, it is important to

- 1. know the typical steps (processes) involved in different types of experiments
- 2. interpret and incorporate information provided in the question in your plan.

We are going to use the TAMP framework to help you organise information from the question to write a clear and coherent plan.

What does it stand for:	What do you have to do:
Topic & Aim	Identifying the Topic tested and the Aim of the experiment narrows the scope of your thinking quickly. The P rocess involved for the experiment should come to mind.
	Identify and ${\bf A}$ nalyse the clues / information given in the question which can be found in
	1. description of experiment
Analyse clues	2. apparatus provided
Analyse clues	3. chemicals provided
	4. bulleted points in question
	Analyse the clues / information allows you to appropriately adapt the typical
	Process for that particular experiment.
	Decide and write down the quantities of the chemicals to be used and measured.
Measurements	Some of these quantities may be
Measurements	 assumed to take particular values, or
	 calculated based on Clues or your assumptions.
	TAM involves you responding the unique question presented.
Process	Each type of experiment has a typical series of steps and considerations i.e. P rocess, which you can / should remember.
	How much of the typical series of steps and considerations is used in your answer depends on what you have A nalysed previously.

In these lecture, we will be

- 1. going into the considerations in planning the different topics.
- 2. highlight the typical steps and considerations i.e. Process in the different topics.
- 3. solve worked examples using the **TAMP** framework to adapt the **P**rocess to situation presented in the question.

GRAVIMETRIC ANALYSIS

Gravimetry involves the accurate measurement of mass as a means of quantifying a sample. The substance to be weighed may be formed from thermal decomposition or by precipitation.

THERMAL DECOMPOSITION

This method involves heating a compound until it decomposes to give a solid residue and a gaseous product.

General procedure:

- Record the mass of an empty boiling tube/crucible.
- Weigh accurately *m* g of the compound in the boiling tube. Record total mass.
- Heat the sample gently in the boiling tube using a Bunsen burner.
- Then heat strongly for at least 10 min.
- Cool and weigh the boiling tube and its contents.
- The process of heating, cooling and weighing is repeated until constant mass is achieved.

Points to take note:

- Apparatus used for heating: dry and clean boiling tube or crucible
- Heating should be gentle at first to prevent spattering of the sample. Strong heating is then carried out for about 10 min.
- The crucible and its contents should be cooled to room temperature. If the crucible and its contents are weighed when they are hot, the measured mass will be less than their true mass. Why? As hot air rises above the crucible, cold air is drawn in and as it heats up and rises, it lifts the crucible very slightly from the balance pan.
- Ideally, the crucible and its contents should be cooled in a desiccator (see below). The desiccator provides a dry atmosphere and allows the crucible and its contents to cool without absorbing moisture.
- The procedure of reheating, cooling and weighing of the solid until constant mass is important in gravimetric analysis because this ensures that the sample is <u>completely</u> decomposed.
- Possible source of error: Due to uneven heating, part of the sample may not have decomposed.



Basic safety considerations:

- Allow the crucible to cool sufficiently before handling and handle a hot crucible with crucible tongs. This is to ensure that your hands do not get burnt.
- While heating a sample using a boiling tube, move the boiling tube up and down, and do not heat it at one spot. This is to prevent the contents of the boiling tube from spurting due to intensive heating at one spot.
- Handle the boiling tube with a test tube holder at all times. This ensures that your hands do not get burnt.
- Point the boiling tube away from yourself and others. This ensures that no one gets hurt in case of spurting.

Common apparatus for gravimetric analysis using thermal decomposition

- electronic mass balance
- crucible / boiling tube
- desiccator



The table below shows some substances that decompose on heating.

Substance	Effect on heating	Examples
Carbonate	 Many carbonates decompose to produce oxide and carbon dioxide on heating. Example: MgCO₃(s) → MgO(s) + CO₂(g) Na₂CO₃ and K₂CO₃ are stable to heat and do not decompose on heating. 	 Determine the identity of M in MCO₃ Determine the % composition of MgCO₃ contaminated with K₂CO₃ (Na₂CO₃ and K₂CO₃ are stable to heat and do not decompose on heating.) Determine the % by mass of NaHCO₃ in a mixture of NaHCO₃ and Na₂CO₃. [see worked example 2]
Hydroxide	 Group 2 hydroxides decompose on heating to give their respective oxides and water Example: Mg(OH)₂(s) → MgO(s) + H₂O(g) 	 Determine the identity of M in M(OH)₂ Determine the % composition of Mg(OH)₂ in a mixture of Mg(OH)₂ and MgO.
Salts with water of crystallisation	 The water of crystallisation can be driven off, leaving the anhydrous salt. Example: CuSO₄•5H₂O(s) → CuSO₄(s) + 5H₂O(g) Difference in the mass before and after heating allows us to find the number of water of crystallisation present. 	 Determine the value of <i>n</i> in CuSO₄•<i>n</i>H₂O <i>[see worked example 1]</i> Determine the relative molecular mass of X in X•5H₂O

Gravimetry (Thermal decomposition)			
T opic & Aim			
Analyse clues			
Measurements	Mass (see belo	pw)	
		Mass of empty container / g	
	 Weighing 	Mass of container + solid / g	
Process		Mass of container + contents after heating / g	
	Heat gently	then strongly	
	 Heat-cool-w 	eigh until constant mass	

♦ ♦ Worked Example 1 ♦ ♦

	A sample of hydrated copper(II) sulfate has a formula of CuSO₄•nH₂O.
Topic ← (Gravimetry – thermal	You are to design an experiment in which the value of n can be determined accurately using a method involving weighing (i.e. gravimetric analysis) but not titration.
decomposition)	You may assume that the following are available:
Process Mass of empty container / g	♦ 5 g of CuSO ₄ •nH ₂ O(s) → Analyse clues
Weighing Mass of container + solid / g	✤ crucible
Mass of container + contents after heating / g • Heat gently, then strongly • Heat-cool-weigh until constant mass	
	(a) Outline a step-by-step procedure, giving full experimental details.
	(b) Show how the experimental data may be tabulated.
	(c) Show how the results may be used to find the value of n in the sample of CuSO ₄ .nH ₂ O.

(a) Procedure

- 1. Using an analytical balance, weigh and record the mass of a clean, empty and dry crucible.
- 2. Weigh out accurately about 5.00 g of solid CuSO₄•nH₂O into the crucible. Record the total mass of the crucible and the solid CuSO₄•nH₂O.
- 3. Using a Bunsen burner, heat the crucible and its contents gently at first, and then heat strongly for 10 minutes.
- 4. Cool and weigh the crucible and its contents.
- 5. Repeat the heating-cooling-weighing process until constant mass is achieved.



♦ ♦ Worked Example 2 ♦ ♦

	Sodium carbonate, Na ₂ CO ₃ , does not decompose on heating v burner. Sodium hydrogen carbonate, NaHCO ₃ , decomposes on	with a Bunsen heating.
	$2NaHCO_3(s) \longrightarrow Na_2CO_3(s) + H_2O(q) + CO_2(q)$	
	You are provided with a mixture of NaHCO ₃ and Na ₂ CO ₃ .	·/
	(a) Plan an investigation to determine the percentage by mass this mixture of NaHCO ₃ and Na ₂ CO ₃ by heating and weighin	of NaHCO₃ in ng alone.
	You may assume that you are provided with	
	• 2 g of a mixture of NaHCO ₃ and Na ₂ CO ₃ ,	
	 a boiling tube and a holder, 	Process Mass of amply container / g
	weighing balance,	Weighing Mass of container + solid / g
	Bunsen burner, the equipment permally found in a school or college lab	Mass of container + contents after heating / g
	• the equipment normally found in a school of college lab	Heat-cool-weigh until constant mass
	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 <u>measurements</u> that you would take and	
	how you would tabulate the experimental data,	stage by mass
	• Now you would use the results to determine the percer of NaHCO ₃ in this mixture of NaHCO ₃ and Na ₂ CO ₃ ,	lage by mass
	(b) By considering the products of the decomposition suggest	
	a crucible, without a lid, might be more appropriate than a b	poiling tube for
	this experiment.	
ç	Solutions	
<u>)</u>	(a)	
Ē	Procedure	
	1 Weigh an empty, dry and clean boiling tube and record its ma	ISS.
2	2 Transfer about 2.00 g of the mixture of Na ₂ CO ₃ and NaHCO	D_3 provided into the boiling tube. Weigh the
	boiling tube and its contents and record the total mass.	
ć	3 Using a Bunsen burner, heat the boiling tube with its conte	ents gently at first, and then strongly for 10^{-1}
	during heating.	
Z	4 Allow the boiling tube and its contents to cool. Then weigh t	the cooled boiling tube and its contents and
	record the total mass.	
5	5 Repeat this heating-cooling-weighing process until constant n	nass is achieved.
-	Tabulation of results	:
-		
L	Mass of empty boiling tube / g A	:
ŀ	Mass of boiling tube and sample / g B	
	after first heating / g	:
	after second heating / g D	
L	after third heating / g D	:
	Mass of mixture of Na ₂ CO ₃ and NaHCO ₃ used = $(B - A)$ g Mass of H ₂ O and CO ₂ lost = $(B - D) \alpha$	

Analysis of results

Total mass of H₂O and CO₂ lost = (B – D) g Total mass of 1 mole of H₂O and 1 mole of CO₂ = (18.0 + 44.0) = 62.0 g

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- 2
- 3
- 4
- 5

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 $2\text{NaHCO}_{3}(\text{s}) \longrightarrow \text{Na}_{2}\text{CO}_{3}(\text{s}) + \text{H}_{2}\text{O}(\text{g}) + \text{CO}_{2}(\text{g})$ $n(\text{H}_{2}\text{O}) \text{ lost} = n(\text{CO}_{2}) \text{ lost} = \frac{B-D}{62.0} \text{ mol}$ $n(\text{NaHCO}_{3}) = \frac{2(B-D)}{62.0} = \frac{B-D}{31.0} \text{ mol}$ Molar mass of NaHCO_{3} = 84.0 g mol⁻¹ Hence mass of NaHCO_{3} = 84.0 g mol⁻¹ Hence mass of NaHCO_{3} in the mixture = $\frac{84.0(B-D)}{31.0}$ g Mass of sample (mixture of Na_{2}CO_{3} and NaHCO_{3}) used = (B - A) g Percentage by mass of NaHCO_{3} in the mixture = $\frac{84.0(B-D)}{31.0(B-A)} \times 100\% = \frac{8400(B-D)}{31.0(B-A)}\%$

(b)

Heating the sample using an open crucible will allow water vapour to be lost more easily. With a boiling tube, the water vapour evolved will condense on the upper inner walls of the boiling tube to form tiny water droplets. Further heating will be needed to remove these water droplets.

PRECIPITATION

This may be used to determine the amount of an insoluble compound formed when two solutions are mixed.

General Procedure:

- Mix two solutions.
- Filter the mixture through a filter funnel lined with a sheet of pre-weighed filter paper.
- Wash the precipitate with several portions of cold deionised water.
- Dry the precipitate with the pre-weighed filter paper in the oven.
- Cool the precipitate with the pre-weighed filter paper in a desiccator for about 10 min.
- Weigh accurately the precipitate and the pre-weighed filter paper.
- Repeat the last 3 steps until constant mass is achieved.

	Type of salt	Solubility in water	Examples
1	Na ⁺ , K ⁺ , NH ₄ ⁺	All sodium, potassium and ammonium salts are soluble.	—
2	NO ₃ -	All nitrates are soluble.	
3	NO ₂ -	All nitrites are soluble except AgNO ₂ .	
4	C/⁻, Br⁻, I⁻	All halides are soluble except AgC <i>l</i> , AgBr, AgI, PbC <i>l</i> ₂ , PbBr ₂ and PbI ₂ . PbC <i>l</i> ₂ , PbBr ₂ and PbI ₂ are soluble in hot water.	Determine the identity of the metal cation in M C <i>l</i> .
5	SO4 ²⁻	All sulfates are soluble except BaSO ₄ , CaSO ₄ and PbSO ₄ .	Determine the identity of a sulfate MSO_4 . Determine the value of <i>n</i> in CuSO ₄ • <i>n</i> H ₂ O. Determine the solubility of cerium(IV) sulfate in water. (<i>Refer to worked example in Planning Notes</i> 3 on Equilibria)
6	SO3 ²⁻	Only sulfites of sodium, potassium and ammonium are soluble.	
7	CO32-	Only carbonates of sodium, potassium and ammonium are soluble.	Determine the amount of Zn^{2+} in a sample by precipitating out $ZnCO_3$.
8	OH⁻	Only hydroxides of sodium, potassium and barium are soluble. Ca(OH)₂ has limited solubility in water.	Determine the solubility and K_{sp} of Ca(OH) ₂ in water.



Some common ions and their precipitating reagents are shown below:

lon	Precipitating Reagent	Remarks
sulfate, SO4 ²⁻	BaC/2(aq)	Question: If contaminants such as halide ions are present, $Pb(NO_3)_2(aq)$ should not be used. Why?
	01 F D(1103)2(aq)	Answer: Lead(II) halides are insoluble and they will precipitate out together with PbSO ₄ .
halide ions: C/⁻, Br⁻, I⁻	AgNO ₃ (aq) or Pb(NO ₃)₂(aq)	Question: If sulfate is also present, Pb(NO ₃) ₂ (aq) should not be used. Why?
		Answer: PbSO ₄ is insoluble and it will precipitate out together with the lead(II) halides.
lead(II) ion, Pb²+	H ₂ SO ₄ (aq) or Na ₂ SO ₄ (aq)/ HC/(aq) or NaC/(aq)/ KBr(aq)/ KI(aq)	Question: PbCO ₃ , BaCO ₃ and Ag ₂ CO ₃ are insoluble in water. Theoretically, Na ₂ CO ₃ (aq) may also be used to precipitate out Pb ²⁺ , Ba ²⁺ and Ag ⁺ , but it is usually the least preferred compared to the reagents shown. Why?
barium ion, Ba ²⁺	H₂SO₄(aq) or Na₂SO₄(aq)	Answer: Many carbonates are insoluble. If the impurities contain metal ions that form insoluble salts with carbonate ions, the impurities will be precipitated out as well.
silver ion, Ag⁺	HC/(aq) or NaC/(aq)/ KBr(aq)/ KI(aq)	 Question: Why is NaOH(aq) not used to precipitate out any of these three ions? Answer: Pb(OH)₂ is amphoteric and dissolves in excess NaOH(aq). Ba(OH)₂ has appreciable solubility. AgOH is unstable and decomposes to form Ag₂O.

Gravimetry (Precipitation	
T opic & Aim	
Analyse clues	
Measurements	Mass / volume of reactantsMass during dry-cool-weigh cycle.
Process	 Mix reactants Filter Wash Dry (in oven) – cool – weigh until constant mass

♦ ♦ Worked Example 3 ♦ ♦

A PbCO₃ solid sample has been contaminated with solid BaCO₃. The percentage purity of the sample can be found using the gravimetric method.

The following sequence of steps has been proposed.

- Step 1: Weigh accurately 0.5 g of the sample into a small beaker.
- Step 2: Add 20 cm³ of dilute nitric acid to the sample. Stir and mix well.
- Step 3: Add 20 cm³ of hot dilute hydrochloric acid to the resulting solution and stir vigorously.
- Step 4: Allow the mixture to cool and the precipitate to settle.
- Step 5: Filter the mixture and wash the precipitate using a suitable solvent.
- Step 6: Transfer the precipitate into a pre-weighed crucible.
- Step 7: Dry the precipitate and crucible in the oven for about 10 min.
- Step 8: Cool the precipitate and crucible in a desiccator.
- Step 9: Weigh the precipitate and crucible.

(a) Suggest a reason for adding dilute nitric acid to the sample in step 2. Dilute nitric acid has been added to dissolve the sample. (Note: The sample and the precipitating agent must be combined in solution form. If either the sample or the precipitating agent is in solid form, the precipitate formed may coat the solid, preventing further reaction.) (b) Suggest a suitable concentration for the hydrochloric acid used in step 3. Assume that the sample contains 100% PbCO₃. n(PbCO₃) present = $\frac{0.5}{267.2}$ = 1.873 x 10⁻³ mol n(C/⁻) needed to fully precipitate out all the Pb²⁺ ions = $1.873 \times 10^{-3} \times 2 = 3.746 \times 10^{-3}$ mol [HC/] needed = $\frac{3.746 \times 10^{-3}}{20 \times 10^{-3}}$ = 0.187 mol dm⁻³ Suitable concentration to use: 0.200 mol dm⁻³ (Any concentration above 0.187 mol dm⁻³ is fine. However, due to corrosive nature of the acid, the concentration should not be above 2 mol dm⁻³.) (c) A student proposed the use of dilute sulfuric acid in place of the dilute hydrochloric acid. Comment on whether this is feasible. -----This is not feasible since sulfate ions will precipitate out both the Pb^{2+} and Ba^{2+} ions. (d) After all the precipitate has settled in step 4, suggest what can be done to check that all the Pb²⁺ ions have been precipitated out. A small amount of the solution above the precipitate can be decanted and tested with a few drops of concentrated hydrochloric acid (or any solution containing chloride ions). If no precipitate is formed, it confirms that all the Pb²⁺ are in the precipitate and none are in the solution. Figure shows the decanting process (e) In an analysis, the mass of the precipitate obtained was found to be m g. Outline how you would use the result to determine the percentage purity of the sample. Molar mass of PbC l_2 = 278.2 g mol⁻¹; Molar mass of PbCO₃ = 267.2 g mol⁻¹ n(PbCl₂) formed = $\frac{m}{278.2}$ mol Mass of PbCO₃ present in the sample = $\frac{m}{278.2}$ x 267.2 g = 0.9604 m g Percentage purity = $\frac{0.9604m}{0.5}$ x 100% = 192 m % (f) Suggest a significant modification to the given procedure to improve the reliability of the results.

Repeat steps 7 to 9 until a constant mass is obtained to ensure that the precipitate is completely dry.

GAS COLLECTION

- Some reactions give out gas when a reagent is added in excess to another.
- For a reaction that evolves a gas, the gas can be collected using suitable apparatus and the experimental data (e.g. total volume of gas evolved, volumes of gas at regular time intervals, etc.) can be used to study selected aspects of the reaction.

	Reaction which produces a gas	Experiments based on gas collection
	Na2CO3(s) + 2HC/(aq)	 Determination of percentage purity of a sample of impure Na₂CO₃ [see worked example 5]
1	\longrightarrow 2NaCl (aq) + H ₂ O(l) + CO ₂ (g)	 Determination of relative formula mass of Na₂CO₃
		 Study on kinetics of the reaction based on the rate of evolution of CO₂
0	$Zn(s) + 2HCl(aq) \longrightarrow ZnCl_2(aq) + H_2(g)$	Determination of percentage of zinc by mass in a sample of zinc sheet
2		Determination of relative atomic mass of Zn
		 Determination of molar gas constant, R
	MnO₂(s)	 Determination of concentration of H₂O₂ in a given H₂O₂ sample [see worked example 4]
3	$H_2O_2(aq) \longrightarrow H_2O(l) + \frac{1}{2}O_2(g)$	- Determination of volume strength of a given sample of $H_2 O_2$
		 Study on kinetics of the reaction based on the rate of evolution of O₂

- A labelled diagram for the apparatus set-up should preferably be drawn.
 - Consider how the reactants will be mixed and how the gas should be collected.
- Common apparatus used to collect a gas:
 - o graduated gas syringe
 - o inverted burette filled with water
 - o inverted measuring cylinder filled with water

Some common set-ups used in experiments involving gas collection are shown below.



- Gas collection method is fast, but it is not so accurate because
 - o some gases, e.g. CO₂, may dissolve in the water if the water displacement method is used.
 - some gas may escape from the experimental set-up instead of being collected in the burette/measuring cylinder/gas syringe.
 - swirling of the conical flask is variable, which will vary the volume of gas collected in the measuring cylinder;
 - air and the gas evolved are both compressible, and heat of reaction may cause fluctuation in gas volumes due to thermal expansion;
 - volume of gas collected in the gas syringe or by water displacement method is dependent on temperature and pressure.
 - the ideal gas equation must be used to determine the amount of substance present from the volume of gas collected. However, this is not a good estimate, especially for gases which deviate greatly from the ideal gas behaviour.



General Guideline _

For planning gas collection experiments, we assume the <u>volume of gas collected</u> to be between <u>50% and</u> <u>90%</u> of the capacity of the collection apparatus and use it to determine the amounts of reagents required in the pre-calculations.

Gas collection

T opic & Aim	
Analyse clues	
M easurements	 Mass / volume of reactants Initial and final readings of gas collection apparatus
Process	 May assume V_{gas} ≈ 50 – 90% of gas collection apparatus capacity. Setup diagram as shown in diagram Vessel for reaction Gas collection apparatus

♦ Worked Example 4 ♦ ♦

Hydrogen peroxide decomposes slowly in accordance to the following equation:	
$2H_2O_2(aq) \longrightarrow 2H_2O(I) + O_2(g)$	
This reaction can be catalysed by MnO ₂ .	
(a) Plan an investigation to determine the <u>actual concentration</u> of a sample of aqueous H_2O_2 .	\rightarrow
 You may assume that you are provided with: 100 cm³ sample of aqueous H₂O₂ with an approximate concentration of 0.140 to 0.160 mol dm⁻³, 2.0 g of powdered MnO₂, 100 cm³ graduated gas syringe, the equipment normally found in a school or college 	
 laboratory. In your plan you should include brief details of: the apparatus you would use, the quantities you would use. 	>
 a fully-labelled diagram of the set-up you would use, the procedure you would follow and the measurements that you would take, 	\rightarrow
 now you would use the results to determine the actual concentration of H₂O₂, how you would ensure that an accurate and reliable value of the actual concentration of aqueous H₂O₂ is obtained. 	
You may assume that 1 mole of gas occupies a volume of 24 dm ³ under labor atory conditions.	\rightarrow
(b) The actual concentration of H_2O_2 in the sample can also be determined via titration.	
Suggest whether a more accurate value would be obtained compared to the gas collection method in (a) .	
Gas collection Topic & Aim Analyse clues	

Topic & Aim	
Analyse clues	
Measurements	Mass / volume of reactants Initial and final readings of gas collection apparatus
Process	May assume V _{pas} = 50 – 90% of gas collection apparatus capacity. Setup diagram as shown in diagram Vessel for reaction Gas collection apparatus

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Solutions

(a) Pre-calculations Since a 100 cm³ gas syringe is used, let the volume of O₂(g) collected be <u>72.0 cm³</u> As 1 mole of gas occupies a volume of 24 dm³ under laboratory conditions, Amount of O_2 evolved = 72.0 × 10⁻³ ÷ 24 = 3.00 × 10⁻³ mol $2H_2O_2(aq) \longrightarrow 2H_2O(l) + O_2(q)$ Amount of H_2O_2 used = 2 × 3.00 × 10⁻³ $= 6.00 \times 10^{-3} \text{ mol}$ Assume that the concentration of $H_2O_2(aq)$ used is 0.150 mol dm⁻³. Thus, volume of H₂O₂ to be used = $6.00 \times 10^{-3} \div 0.150 \times 1000 = 40.0 \text{ cm}^3$ Experiment set-up graduated das svringe deliverv stoppe tube and rubber small tube tubing containing MnO₂ and tied to string conical flask retort stand with side arm H₂O₂(ag) Procedure 1. Using a burette, add 40.00 cm³ of H₂O₂(aq) into a clean and dry 250 cm³ conical flask with a side arm. 2. Weigh accurately about 1.00 g of $MnO_2(s)$ and place it in a small tube tied to a string. 3. Set up the apparatus as shown above. 4. Lower the filled tube into the conical flask taking care that the reagents do not mix. Stopper the conical flask. 5. Check that the initial reading of the 100.0 cm³ graduated gas syringe is set at the zero mark. 6. At a suitable time, loosen the stopper slightly to release the string to allow mixing of MnO₂ and $H_2O_2(aq)$. Stopper the conical flask immediately. 7. Swirl the conical flask to ensure that the reagents are well mixed. 8. Allow the reaction to progress until it has ceased as indicated by a constant reading of the syringe. 9. Record the final reading, $V \text{ cm}^3$, on the graduated gas syringe. 10. Repeat the experiment again to get consistent results of volume of $O_2(g)$ collected. Note: Both steps 8 and 10 help to ensure accuracy and reliability. Step 8 ensures that after the reaction has ceased, time is allowed for the pressure and/or temperature to stabilise before measurement of volume. Step 10 ensures that the experiment is repeated to obtain consistent values of O₂(g) collected. Calculations Assume average volume of $O_2(g)$ evolved = V cm³ As 1 mole of gas occupies a volume of 24 dm³ under laboratory conditions, Amount of O₂ evolved = $\frac{v}{24000}$ mol $2H_2O_2(aq) \longrightarrow 2H_2O(I) + O_2(g)$ Amount of H₂O₂ = $\frac{2V}{24000} = \frac{V}{12000}$ mol Since volume of H₂O₂ used = 40.00 cm³, concentration of H₂O₂ = $\frac{V}{12000} \times \frac{1000}{40.00}$ $\frac{V}{480}$ mol dm⁻³ (b) The actual concentration of H_2O_2 obtained via titration is more accurate than gas collection. In gas collection, the volume of the gas collected fluctuates with temperature and pressure, which affects the accuracy of the amount and concentration of H_2O_2 obtained. Also, some $O_2(g)$ may have escaped when the flask is being stoppered.

♦♦ Worked Example 5 ♦♦

Sodium carbonate reacts with hydroch carbon dioxide gas:	nloric acid to pro	oduce	
$Na_2CO_3(s) + 2HCl(aq) \longrightarrow 2NaCl(aq)$	aq) + CO ₂ (g) + H	H ₂ O(I)	
FA 1 is a solid mixture containing ab sodium carbonate and 20% by mass o	oout <u>80% by ma</u> f sodium chlorid	e. Clue	
Plan an investigation to determine the of sodium carbonate in FA 1 .	e percentage by	mass	Aim
 You may assume that you are provided 1.00 g of FA 1 (a mixture of so sodium chloride), 100 cm³ of 0.200 mol dm⁻³ a acid, 100 cm³ graduated gas syringe, 	d with: odium carbonate aqueous hydroc	e and hloric	
the equipment normally found in laboratory.	n a school or co	ollege	Topic: Gas collection
 In your plan you should include brief de the apparatus you would use, the quantities you would use, excess of HC/ a fully-labelled diagram of the se the procedure you would measurements that you would take the result percentage by mass of sodium of how you would ensure that an value of the percentage by 	etails of: so that there <u>et-up you would u</u> follow and ake, lts to determine carbonate in FA accurate and re u mass of so	is an use. the e the 1 . eliable odium	Reaction flask : mix Na ₂ CO ₃ (s) + HCl <u>Collect CO₂ gas</u> : 100 cm ³ gas syringe Excess HCl - Use calculated mass of solid to check volume of HCl Limiting solid mixture - Assume 50 cm ³ of CO ₂ to determine mass of so mixture to use.
carbonate is obtained. You may assume that 1 mole of gas 24 dm ³ under laboratory conditions.	occupies a volu	me of	 Volume of HCl(aq) Initial reading of syringe Final reading of syringe
	Topic & Aim Analyse clues Measurements	 Mass / v Initial an May ass Setup di 	/ volume of reactants and final readings of gas collection apparatus issume V _{pas} ≈ 50 – 90% of gas collection apparatus capacity. diagram as shown in diagram
		∘ Vess ∘ Gas	issel for reaction as collection apparatus

 $(-m_{2})^{-m_{2}}$

Solutions

Pre-calculations

Mass of FA1 to be used

Assume that 50.0 cm³ of CO₂ gas is collected.

As 1 mole of gas occupies a volume of 24 dm³ under laboratory conditions,

Amount of $CO_2 = \frac{50.0}{24000} = 2.08 \times 10^{-3} \text{ mol}$ $Na_2CO_3(s) + 2HC/(aq) \rightarrow 2NaC/(aq) + CO_2(g) + H_2O(l)$ Amount of $Na_2CO_3 = 2.08 \times 10^{-3} \text{ mol}$ Molar mass of $Na_2CO_3 = 106.0 \text{ g mol}^{-1}$ Mass of $Na_2CO_3 = (2.08 \times 10^{-3})(106.0) = 0.220 \text{ g}$ Assume that **FA 1** contains 80% Na_2CO_3 . Hence mass of FA 1 to be used = 0.220 / 0.8 = 0.275 g

Volume of HC/(aq) to be used

Amount of HC/(aq) = $(2)(2.08 \times 10^{-3}) = 4.16 \times 10^{-3} \text{ mol}$ Volume of HC/ to be used = $(4.16 \times 10^{-3})/0.200 = 2.08 \times 10^{-2} \text{ dm}^3 = 20.8 \text{ cm}^3$

Since HC*l*(aq) has to be in excess such that the Na₂CO₃ used is completely reacted and for the experiment to be repeated, a suitable volume of HC*l*(aq) to be used is 30 cm³.

Experimental set-up



Procedure

- 1. Using a burette, add 30.00 cm³ of HC/(aq) into a clean and dry 250 cm³ conical flask with a side arm.
- 2. Weigh accurately about 0.275 g of **FA1** and place it in a small tube tied to a string.
- 3. Set up the apparatus as shown above.
- 4. Lower the filled tube into the conical flask taking care that the reagents do not mix. Stopper the conical flask.
- 5. Check that the initial reading of the 100.0 cm³ graduated gas syringe is set at the zero mark.
- 6. At a suitable time, loosen the stopper slightly to release the string to allow mixing of **FA 1** and HC*l*(aq). Stopper the conical flask immediately.
- 7. Swirl the conical flask to ensure that the reagents are well mixed.
- 8. Allow the reaction to progress until it has ceased as indicated by a constant reading of the syringe.
- 9. Record the final reading, $V \text{ cm}^3$, on the graduated gas syringe.
- 10. Repeat the experiment again to get consistent results of volume of CO₂(g) collected.

Calculations

Let the **average** volume of CO₂(g) collected be V cm³ and the mass of FA1 used = $m_1 - m_2$ g

Amount of CO₂ formed =
$$\frac{V}{24000}$$
 mol
Amount of Na₂CO₃ = Amount of CO₂ = $\frac{V}{24000}$ mol
Mass of Na₂CO₃ = $\frac{V}{24000}$ x106.0 g
Percentage composition by mass of Na₂CO₃ in **FA 1** = $\left(\frac{V}{24000} \times 106.0 / (m_1 - m_2)\right)$ x 100% = $\frac{53}{120}$ mol

CHEMICAL ENERGETICS

Thermochemistry is the study of heat effects accompanying chemical reactions, the formation of solutions and changes in physical states of substances. The measurement of the heat of a reaction requires the careful determination of a measurable change in temperature, often with the use of a calorimeter.

Refer to your lecture notes on Chemical Energetics for the definitions of important terms. You must be able to construct energy cycles and calculate enthalpy changes of reactions using the experimental data obtained.

Symbol	Definition
Standard enthalpy change of reaction, ΔH_r^{\ominus}	The energy absorbed or released in a chemical reaction when the molar quantities of reactants stated in the chemical equation react at 298 K and 1 bar.
Standard enthalpy change of neutralisation, $\Delta H_{neut}^{\ominus}$	The energy change when an acid and a base react to form one mole of water at 298 K and 1 bar.
Standard enthalpy change of solution, ΔH_{soln}^{Θ}	The energy change when one mole of the substance is completely dissolved in a solvent to form an infinitely dilute solution at 298 K and 1 bar.
Standard enthalpy change of combustion, ΔH_c^{\ominus}	The energy released when one mole of the substance is completely burnt in excess oxygen at 298 K and 1 bar.

Common Experimental Setups used for



General Guideline

For planning energetics experiments where the <u>approximate value of the unknown enthalpy change of</u> <u>reaction is given</u>, we will assume that the reaction causes a <u>5–10 °C change in temperature</u> and perform <u>pre-calculation</u> to determine the appropriate qualities of reagents to use.

Common Graphs plotted in Chemical Energetics Experiments



General Procedure:

- 1. Monitor the temperature of a solution at regular time intervals, e.g. 0.5 min intervals. There should be a <u>minimum of 3</u> (including initial temperature) temperature readings before the addition of the other reagent.
- 2. At a suitable time (e.g. t = 3min), add the other reagent to the solution and take temperature readings at 0.5 min intervals.
- 3. Sufficient temperature readings (<u>minimum of 5</u> readings) must be taken <u>after highest or lowest temperature</u> is reached for reliable plotting of data points to be done.
- 4. Plot a graph of temperature against time. Draw two straight lines of best fit, one for the temperature of solution before addition of reagent at t = 3 min and the other for the cooling/warming of the solution once the reaction is completed. Extrapolate the two lines to t = 3 min and determine the change in temperature.

[Note: A stopwatch is required for this experimental method.]



Energetics	
Topic & Aim	
Analyse clues	
Measurements	Mass / volume of reactantsInitial and highest/lowest temperature
Process	 Reaction setup Reaction involving solution(s) – Styrofoam cup in beaker Reaction involving combustion – Spirit lamp Experiment type / data collected Continuous (i.e. with time) Mix different mass/volume of 2 reagents Initial T, mix, highest/lowest T ΔH = - mc\DeltaT/n May assume 5 – 10 °C temperature change

♦ ♦ Worked Example 6 ♦ ♦



Precalculation

Let the volume of water used = 50.0 cm³ Since solubility of NH₄C*l* in water is 383 g dm⁻³,

Max mass of NH₄C*l* that would dissolve in 50.0cm³ of water = $\frac{383}{1000} \times 50 = 19.15$ g

Let the temperature change be -7 °C.

Heat change = $50 \times 4.18 \times -7 = -1463 \text{ J}$

Since ΔH_{soln} (NH₄C*l*) \approx +15 kJ mol⁻¹, n(NH₄C*l*) = $-\frac{-1463}{15 \times 10^3}$ = 0.09753 mol mass of NH₄C*l* = 0.09753 x (14.0+4.0+35.5) = 5.218 g

Procedure

- Using a 50.0 cm³ burette (or measuring cylinder), transfer 50.0 cm³ of deionised water into a clean and dry polystyrene cup supported in a 250 cm³ beaker. Take the initial temperature of the water using the thermometer provided.
- 2. Using an analytical balance, weigh accurately about 5.22 g of NH₄C*l* in a clean and dry weighing bottle. Record the total mass of the weighing bottle and solid NH₄C*l*.
- 3. Empty the solid NH₄C*l* into the polystyrene cup, cover the cup with a lid and stir gently using the thermometer to dissolve all the NH₄C*l*. Record the lowest temperature reached.
- 4. Weigh the emptied weighing bottle and record its mass.
- 5. Repeat steps 1 to 4. Use the data obtained from each experiment to calculate a value for ΔH . Take the average of these two values.

Table of results

Mass of weighing bottle and solid NH ₄ Cl / g		
Mass of emptied weighing bottle / g		
Mass of solid NH ₄ C/ used / g		r
	_	The change in temperature (ΛT)
Lowest temperature reached / °C		will be negative as temperature
Initial temperature / °C		decreases when NH ₄ Cl dissolves
Change in temperature / °C		in water. $\Delta T = T_{min} - T_i$

Calculations to determine ΔH_{soln}

Assume specific heat capacity of solution to be 4.18 J cm⁻¹ K⁻¹. Assume no heat absorbed from surroundings.

Energy change for the reaction, q = (50.0)(4.18)(-T) = -209T J

Let mass of NH₄C*l* used be $\mathbf{m}_{\mathbf{A}}$ g. Molar mass of NH₄C*l* = 53.5 g mol⁻¹

Amount of NH₄C*l* used =
$$\frac{m_A}{53.5}$$
 mol

Enthalpy change of reaction

$$= - \frac{q}{amount of NH_4Cl}$$

= $- \frac{(-209T)(53.5)}{m_A} = + \frac{11181.5T}{m_A} \text{ J mol}^{-1}$

Since the reaction is endothermic and temperature decreases, enthalpy change of reaction must be positive.

Raffles Institution

♦ ♦ Worked Example 7 ♦ ♦

	The r	neutralisation of a	n acid by a bas	e is <u>exothermic.</u>
	A stu can l soluti volun	udent suggests the be determined b ion as the acid is ne of sodium hyd	tration of sulfuric acid ne temperature of the I amounts to a known ofoam cup.	
	The s	student proposes	the following hy	/pothesis.
	As th until t there in the lower	the acid is added the end-point is re will be a reducti e cup as the aci temperature tha	to the alkali, the eached. Upon fi on in the tempo d added is not n the solution in	e temperature will rise urther addition of acid, erature of the solution t reacting and is at a the cup.
	2	2NaOH(aq) + H ₂ S	$O_4(aq) \rightarrow Na_2$	SO₄(aq) + 2H₂O(I)
	(a)	Use the equati volume of app needed to neu NaOH.	on for the rea proximately 0.7 tralise 25.0 cn	ction to estimate the 75 mol dm ⁻³ H ₂ SO ₄ n ³ of 2.00 mol dm ⁻³
	(b) Using the information given, you are required to write a plan to <u>determine the precise concentration of</u> sulfuric acid.			u are required to write cise concentration of
	You may assume that you are provided with:			provided with:
		 FA 1 2.00 m FA 2 approximate burette a Styrofoam a thermomet graph paper other apparate college labor 	nol dm ⁻³ NaOH kimately 0.75 m cup er atus normally † ratory	found in a school or
		 Your plan should details of the the proceed measureme how you we been passed 	d include: e apparatus you lure you wou nts that you wou ould recognise t	u would use; uld follow and the uld take; that the end-point had
		 an outline of and recorde 	f how the resu d:	Its would be obtained
	(c)	Sketch the grap acid is added. La	h you would ex abel the end-po	xpect to obtain as the int.
	(d)	Show how the d used to detern sulfuric acid	ata obtained fro nine the prec	om the graph would be sise concentration of
L			Topic & Aim	
			Analyse clues	
			M easurements	Mass / volume of reactants
			Process	Initial and highest/lowest temperature Reaction setup a. Reaction involving solution(s) – Styrofoam cup in beaker b. Reaction involving combustion – Spirit lamp Experiment type / data collected a. Continuous (i.e. with time) b. Mix different mesk/lowume of 2 records
				D. WIN UNCERTEINASSIVULITE OF 2 TEAUETIS

20

3. Initial T, mix, highest/lowest T 4. $\Delta H = -\frac{mc\Delta T}{n}$ 5. May assume 5 – 10 °C temperature change (a) Use the equation for the reaction to estimate the volume of approximately 0.75 mol dm⁻³ H₂SO₄ needed to neutralise 25.0 cm³ of 2.00 mol dm⁻³ NaOH.

n(NaOH) reacted = 2 x n(H₂SO₄) reacted Amount of NaOH in 25.0 cm³ = (25.0 x 10⁻³) x 2.00 = 5.00 x 10⁻² mol Amount of H₂SO₄ needed = $\frac{1}{2}$ x 5.00 x 10⁻² = 2.50 x 10⁻² mol Volume of H₂SO₄ needed = $\frac{2.50 \times 10^{-2}}{0.75}$ x 1000 = 33.3 cm³

(b) Using the information given, you are required to write a plan to determine the precise concentration of sulfuric acid.

Procedure

- 1. Fill a 50.00 cm^3 burette with FA 2.
- 2. Pipette 25.0 cm³ of FA 1 into the clean and dry styrofoam cup supported in a 250 cm³ beaker.
- 3. Use a thermometer to measure the steady initial temperature of FA 1. Record this temperature, Ti.
- 4. Run 3.00 cm³ of FA 2 into the cup. Stir and record the highest temperature observed.
- 5. Immediately add another 3.00 cm³ of FA 2 from the burette and repeat the temperature measurement.
- 6. Repeat step 5 until a total of 48.00 cm³ of FA 2 has been added.

Results

Volume of FA2 added / cm ³	Highest temperature / °C	ΔT / °C	Volume of FA2 added / cm ³	Highest temperature / °C	ΔT / °C
0.00		0.0	24.00		
3.00			27.00		
6.00			30.00		
9.00			33.00		
12.00			36.00	The end-point is calculate	ed to be
15.00			39.00	33.3 cm ³ . Hence, 5 more readin are taken after the end-point reached for a reliable plotting data points. A total of 48.00 ci	readings
18.00			42.00		-point is
21.00			45.00		B_{00} cm ³
			48.00	FA2 is added.	0.00 011

(c) Sketch the graph you would expect to obtain as the acid is added. Label the end-point.



(d) Show how the data obtained from the graph would be used to determine the precise concentration of sulfuric acid. From (a), amount of H_2SO_4 needed = $\frac{1}{2} \times 5.00 \times 10^{-2} = 2.50 \times 10^{-2}$ mol Concentration of H₂SO₄ = $\frac{2.50 \times 10^{-2}}{V_{eq} \times 10^{-3}} = \frac{25}{V_{eq}}$ mol dm⁻³ heat of neutralisation = heat gained by solution $n(H_2O) \times \Delta H_{neut}^{\ominus} = (m_{soln} \times c_{soln} \times \Delta T)$ $\Delta H_{\text{neut}} = -\frac{m_{\text{soln}} \, x \, c_{\text{soln}} \, x \, \Delta T}{n_{\text{H2O}}} = -\frac{V_{\text{soln}} \, x \, \rho_{\text{soln}} \, x \, c_{\text{soln}} \, x \, \Delta T}{n_{\text{H2O}}} = -\frac{(V_{\text{soln}} + V_{\text{soln}}) \, x \, \rho_{\text{soln}} \, x \, c_{\text{soln}} \, x \Delta T}{n_{\text{H2O}}}$

♦ ♦ Worked Example 8 ♦ ♦

The neutralisation of sulfuric acid by sodium hydroxide is an exothermic reaction.

$$2NaOH(aq) + H_2SO_4(aq) \longrightarrow Na_2SO_4(aq) + 2H_2O(I) \qquad \Delta H_{neut} < 0$$

The concentration of sulfuric acid and the enthalpy change of neutralisation, ΔH_{neut} , Topic: Energetics can be determined graphically by measuring the temperature of the solution when L. different volumes of sulfuric acid and sodium hydroxide are reacted and keeping the total volume of solution constant.

Mass / volume of reactants

2. Experiment type / data collected a. Continuous (i.e. with time)

1. Reaction setup

Initial and highest/lowest temperature

b. Mix different mass/volume of 2 reagents 3. Initial T, mix, highest/lowest T 4. $\Delta H = -\frac{mc\Delta T}{mc\Delta T}$

May assume 5 – 10 °C temperature change

a. Reaction involving solution(s) – Styrofoam cup in beak
 b. Reaction involving combustion – Spirit lamp

- mix 2 reagents, measure T after each portion total volume of solution constant
- Plan an investigation to determine the concentration of sulfuric acid and the (a) Aim enthalpy change of neutralisation, ΔH_{neut} .

You may assume that you are provided with:

- FA1 approximately 1.0 mol dm⁻³ H₂SO₄
- FA 2 2.0 mol dm⁻³ NaOH
- a Styrofoam cup
- a thermometer
- the equipment normally found in a school or college laboratory Process

In your plan you should include brief details of:

- the apparatus you would use, _____measuring cylinder, Styrofoam cup
- the quantities you would use, so that the total volume of solution is 110 cm³, Clue
- the procedure you would follow, > mix 2 reagents portion by portion, measure T after each portion
- the measurements that you would take and the tabulation of data.
- Sketch the graph you would expect to obtain. On your graph, label (b)
 - Veq for the volume of sulfuric acid added
 - ΔT_{eq} for the temperature change

when stoichiometric amounts of sulfuric acid and sodium hydroxide has reacted.

- Hence, show how the data obtained from the graph would be used to determine the concentration of (C) sulfuric acid and the enthalpy change of neutralisation, ΔH_{neut} .
- (d) Explain if the concentration of $Ba(OH)_2(aq)$ be determined using this method.

Energetics

Topic & Aim Analyse clues

Measurements

(a) Plan an investigation to determine the concentration of sulfuric acid and the enthalpy change of neutralisation, ΔH_{neut} .

Pre-calculations

Let V_{eq} cm³ be the volume of sulfuric acid and (110 - V_{eq}) cm³ be the volume of sodium hydroxide used at which stoichiometric amount of sulfuric acid and sodium hydroxide has reacted.

mole ratio of NaOH: $H_2SO_4 = 2 : 1$ $n(NaOH) = 2 \times n(H_2SO_4)$ $(110 - V_{eq}) / 1000 \times 2.00 = 2 \times V_{eq} / 1000 \times 1.0$ $V_{eq} = 55.0 \text{ cm}^3$; 110 - $V_{eq} = 55.0 \text{ cm}^3$ (Note: these are approx. values given approx. conc. of sulfuric acid)

Hence, there should be at least 5 temperature measurements before AND after Veg = 55.0 cm³

Procedure

- 1. Using a 100 cm³ measuring cylinder, transfer 10.0 cm³ of **FA 1** into a clean and dry Styrofoam cup supported in a 250 cm³ beaker.
- 2. Use a thermometer to measure and record the steady initial temperature of FA 1.

Experiment	Volume o addec	f solutions I / cm³	Initial	Highest	Temperature	
Lypenment	FA 1	FA 2	FA1/°C temperature / °C		temperature / °C change, ΔT / °C	
1	10.0	100.0				h
2	20.0	90.0				Min. 5
3	30.0	80.0				
4	40.0	70.0				$V_{cr} = 55.0 \text{ cm}^3$
5	50.0	60.0				Veq = 00.0 cm
6	60.0	50.0				ĥ
7	70.0	40.0				Min. 5
8	80.0	30.0				≻readings after
9	90.0	20.0				V _{eq} = 55.0 cm ³
10	100.0	10.0				V

Using another 100 cm³ measuring cylinder, measure 100.0 cm³ of FA 2. Ensure that both FA 1 and FA 2 have the same initial temperature.

4. Add **FA 2** from the measuring cylinder to **FA 1**. Use the thermometer to stir the mixture gently. Measure and record the highest temperature of the mixture.

- 5. Empty, wash and carefully dry the Styrofoam cup.
- 6. Repeat steps 1 to 5 using volumes of **FA 1** and **FA 2** in the table below so that the total volume of the mixture is 110 cm³.
- 7. Record all measurements of volume, temperature and temperature change, ΔT , in the table below.
- (b) Sketch the graph you would expect to obtain. On your graph, label
 - V_{eq} for the volume of sulfuric acid added
 - ΔT_{eq} for the temperature change

when stoichiometric amounts of sulfuric acid and sodium hydroxide has reacted.



Hence, show how the data obtained from the graph would be used to determine the concentration of (C) sulfuric acid and the enthalpy change of neutralisation, ΔH_{neut} .

At V_{eq}, n(NaOH) used = (110 - V_{eq}) / 1000 x 2.0 mol = n(H₂O)
n(H₂SO₄) reacted = ½ x n(NaOH) mol
Concentration of H₂SO₄ =
$$\frac{1/2 \text{ x n}(\text{NaOH})}{V_{eq} \text{ x10}^{-3}}$$
 mol dm⁻³
Assume no heat lost to the surroundings, q = mc Δ T_{eq} = + (110 x c x Δ T_{eq}) J
 $\Delta H_{neut} = -\frac{q}{n_{H2O}} = -\frac{110 \text{ x c x } \Delta T_{eq}}{n_{H2O}}$ J mol⁻¹ = $-\frac{110 \text{ x c x } \Delta T_{eq}}{n_{H2O} \text{ x 10}^{3}}$ kJ mol⁻¹

(d) Explain if the concentration of $Ba(OH)_2(aq)$ be determined using this method.

The concentration of Ba(OH)₂ cannot be determined using this method as $Ba^{2+}(aq)$ will react with $SO_4^{2-}(aq)$ to form BaSO₄ ppt. The enthalpy change of precipitation of BaSO₄ will interfere with the temperature measurements leading to inaccurate results. _____

CH₃(CH₂)₃ (CH₂)₃CH₃

cis-dec-5-ene

♦ ♦ Worked Example 9 ♦ ♦

Alkynes, which contain C \equiv C bonds, can undergo partial hydrogenation when added to hydrogen gas in the presence of a poisoned catalyst known as Lindlar catalyst. The partial hydrogenation of 5-decyne to form cis-dec-5-ene is exemplified by the following:

$$CH_{3}CH_{2}CH_{2}CH_{2}C \equiv CCH_{2}CH_{2}CH_{2}CH_{3} + H_{2} \longrightarrow 5$$
-decyne

However, it is difficult to conduct the experiment in the laboratory to measure the enthalpy change of reaction directly. You are required to plan an experiment to find the enthalpy change of hydrogenation of 5-decyne, ΔH_1 , via Hess' Law.

Given that the enthalpy change of combustion of (a) hydrogen –286 kJ mol⁻¹, state the enthalpy changes that are needed to calculate ΔH_1 . Clue



is

Topic & Aim

Energetics (H_{combustion})

answer to (a). You may assume that you are provided with:

5-decyne

(b)

- cis-dec-5-ene
- metal calorimeter
- spirit lamp
- a thermometer
- the equipment normally found in a school or college laboratory

In your plan you should include brief details of:

- the experimental set-up and apparatus you would use,
- the quantities you would use,
- · the procedure you would follow,
- the measurements that you would take and the tabulation of data.
- 0 Before combustion After combustion 0

Mass of spirit lamp + 5 decyne

- Blow out flame (stop combustion) when temperature rises by 7 °C.
 - Temperature of water in calorimeter
 - Before combustion After combustion

Measurements

- Describe how you would use the measurements to determine the enthalpy change (C) chosen in (b). You may assume that the density and specific heat capacity of water is 1.0 g cm⁻³ and 4.18 J g⁻¹ K⁻¹ respectively.
- List two sources of errors that could affect the accuracy of your results. Discuss (d) how the experimental set-up in your answer to (b) could be improved so as to address these sources of errors.

(a) Given that the standard enthalpy change of combustion of hydrogen is -286 kJ mol^{-1} , state the enthalpy changes that are needed to calculate ΔH_1 .

<u>Answer</u>

Enthalpy change of combustion of 5-decyne, ΔH_c (5-decyne), and enthalpy change of combustion of cis-dec-5-ene, ΔH_c (cis-dec-5-ene).

<u>Reason</u>

Since the question states that the enthalpy change of hydrogenation of 5-decyne, ΔH_1 , is to be determined via Hess' Law and the standard enthalpy change of combustion of hydrogen is given, the following energy cycle can be constructed to determine ΔH_1 .







Procedure

- 1. Using a 100 cm³ measuring cylinder, place 100 cm³ of water in the calorimeter provided.
- 2. Using a thermometer, measure and record the steady initial temperature of the water in the calorimeter.
- 3. Add 5-decyne (or cis-dec-5-ene) to the spirit lamp.
- 4. Weigh and record the total mass of the spirit lamp and the 5-decyne (or cis-dec-5-ene).
- 5. Place the spirit lamp under the calorimeter as shown in the set-up.
- 6. Light the wick of the lamp. When the temperature of the water rises by about 5 10 °C, extinguish the flame and record the final temperature of the water.
- 7. Allow the spirit lamp to cool sufficiently. Then, reweigh the spirit lamp and remaining 5-decyne (or cis-dec-5-ene). Record the new total mass.

Mass of spirit lamp and 5-decyne (or cis-dec-5-ene) before combustion / g		Α
Mass of spirit lamp and 5-decyne (or cis-dec-5-ene) after combustion / g		В
Temperature of water in calorimeter before combustion	/ °C	С
Temperature of water in calorimeter after combustion	/ °C	D

(c) Describe how you would use the measurements to determine the enthalpy change chosen in (b). You may assume that the density and specific heat capacity of water is 1.0 g cm⁻³ and 4.18 J g⁻¹ K⁻¹ respectively.

Mass of 5-decyne (or cis-dec-5-ene) combusted = $(\mathbf{A} - \mathbf{B})$ g Amount of 5-decyne (or cis-dec-5-ene) combusted = $\frac{(\mathbf{A} - \mathbf{B})}{M_r}$ of 5-decyne (or cis-dec-5-ene) = \mathbf{E} mol Change in temperature, $\Delta T = (\mathbf{D} - \mathbf{C}) \circ \mathbf{C} = (\mathbf{D} - \mathbf{C}) \mathsf{K}$ Volume of water used = $\mathbf{m} \operatorname{cm}^3$ (or volume of water used in procedure, i.e. 100 cm³) Mass of water used = \mathbf{m} g Assume no heat lost to the surroundings, $\mathbf{q} = \mathbf{m} c \Delta T = (\mathbf{m})(4.18)(\mathbf{D} - \mathbf{C}) \mathsf{J}$

Enthalpy change of combustion of 5-decyne (or cis-dec-5-ene)

$$= -\frac{4.18m(D-C)}{E} \text{ J mol}^{-1} = -\frac{4.18m(D-C)}{E \times 10^3} \text{ kJ mol}^{-1}$$

(d) List two sources of errors that could affect the accuracy of your results. Discuss how the experimental set-up in your answer to (b) could be improved so as to address the sources of errors.

Possible Errors	Suggested Improvements
Incomplete combustion of fuel.	Increase oxygen content of surroundings (e.g. place a beaker of H_2O_2 solution mixed with a small amount of MnO_2 beside the set-up).
Rapid loss of fuel due to evaporation, especially immediately after the flame is extinguished.	Cover the wick with a small cap when the spirit lamp is not in use.
Heat loss from flame due to draughts.	Use a windshield to exclude draughts around the apparatus.
The use of a thermometer with divisions of 1 °C to measure a small temperature change of 5 – 10 °C results in less accurately measured temperatures.	Use a thermometer of higher precision (e.g. measures temperature to 0.1 °C) to improve the accuracy of temperature readings. Or use a thermocouple/data logger with a temperature probe.
The thermometer may have a long response time, e.g. due to a large bulb, leading to lower readings taken (since real/actual readings take longer to be registered). As such, the maximum temperature reached will be lower, making the calculated values of enthalpy changes lower as well.	Use a thermometer with a shorter response time, e.g. smaller bulb, or a thermocouple/data logger with a temperature probe, which would allow fast and easy access to the actual temperature.
*The heat capacity of the calorimeter was not taken into consideration.	*Calibrate the calorimeter used by first conducting a combustion experiment with another fuel for which its ΔH_c is known. (see below - Calibrating a calorimeter)

Calibrating a calorimeter

In most energetics experiment, the heat capacity of the calorimeter is ignored. However, in actual fact, the calorimeter does absorb some of the heat given off by the chemical reaction. If the heat capacity of the calorimeter is to be taken into consideration, the heat capacity of the set-up is first found by using a fuel with a known ΔH_c . E.g. Standard enthalpy change of combustion of hexane, $\Delta H_c^{\ominus}[C_6H_{14}(I)] = -4163 \text{ kJ mol}^{-1}$ can be used to calibrate the calorimeter. The same set-up (i.e. calorimeter with a specific amount of water) is then used to determine ΔH_c of the sample.

To find the heat capacity of the calorimeter, set up the experiment as shown below, placing hexane inside the spirit lamp. The experiment is conducted as described in the procedure on page 25.



Measurements to be recorded:

Mass of spirit lamp and hexane before combustion / g	Р
Mass of spirit lamp and hexane after combustion / g	Q
Temperature of water in calorimeter before combustion /	°C Ti
Temperature of water in calorimeter after combustion /	°C T _f

Treatment of results:

Mass of hexane combusted = (P - Q) g

Amount of hexane combusted = $\frac{P}{M_r}$ of h

$$\frac{-Q}{hexane}$$
 = R mol

Standard enthalpy change of combustion of hexane, $\Delta H_c^{\ominus}[C_6H_{14}(I)] = -4163 \text{ kJ mol}^{-1}$ Heat from combustion of R moles of hexane = -(4163 x R) kJ

Change in temperature, $\Delta T = (T_f - T_i) K$

Assume no heat lost to the surroundings.

Heat of combustion = Heat absorbed by calorimeter set-up (i.e. metal calorimeter and water) $-(4163 \times 10^3 \times R) + C\Delta T = 0$ (where C is the overall heat capacity of the metal calorimeter and water) $(4163 \times 10^3 \times R) = C(T_f - T_i)$

Heat capacity of calorimeter, C = $\frac{4163 \times 10^3 \times R}{T_f - T_i} J K^{-1}$

Note: With this value, C, we can now conduct the experiment using the same set-up (same metal calorimeter and same mass of water) but replacing hexane with 5-decyne, and subsequently, with cis-dec-5-ene, to determine their respective ΔH_c :

For each compound, $q = C\Delta T$. $\Delta H_c = -\frac{q}{n} = -\frac{C\Delta T}{n}$, where n is the amount of fuel combusted.

KINETICS

Planning questions in Reaction Kinetics typically fall under two categories.

	"Clock" reaction	Continuous method
Features	Involve measuring the time taken for a stated change to occur. The species responsible for the stated change is the "clock".	 Monitor the concentration of a reactant or product species over time. Concentration can be monitored by different methods, including: 1. Sampling and titration 2. Colorimetry 3. Gas collection
Examples	 lodine clock reaction (2021 Y5 Expt 8) Time taken for first appearance of blue-black starch-iodine complex Sulfur clock reaction Time taken for "X" to be obscured by solid sulfur produced. 	 Monitor how concentration of iodine produced changes with time using colorimetry Monitor how [H₂O₂] changes with time using sampling and titration <i>(example 11)</i> Monitor rate of decomposition of H₂O₂ using gas collection.
Things to note	 Identify the "clock" If the clock is a reactant, then rate is proportional to V_{clock}/t; if the clock is a product, then rate is proportional to 1/t. Total volume of every experiment is the same. This allows V_{reactant} to be proportional to [reactant]. This can be done by varying the V_{reactant} while keeping the other volumes constant. Water is added to the reaction mixture to keep the total volume constant. 	 The procedure for a typical sampling and titration consists of the following: Set up reaction. Draw sample from reaction mixture. Quench sample at appropriate time. Use titration to determine concentration of species in quenched sample.

Occasionally, there are planning questions which are even more contextualised and will require the inclusion of more variables (2017 A Levels Planning Question). As such, it is critical to understand the chemistry of the above points and incorporate or modify the experiment according to the context of the question.

First identify whether the experiment involves a clock reaction or uses a continuous method.

Analyse clues • Volume of reactants and water Measurements • Time taken for stated change to occur 1. Total volume of every experiment is the same. • Vary volume of one reactant and keep other constant, add water to maintain total volume. Process 2. Vreactant ∝ [reactant] 3. Rate ∝ 1/t or V _{clock} / t 4. Mix water and reactants. Last reactant added will start reaction. Start stopwatch. 5. Stop stopwatch when stated change occurs.	T opic & Aim	
Measurements • Volume of reactants and water • Time taken for stated change to occur 1. Total volume of every experiment is the same. • Vary volume of one reactant and keep other constant, add water to maintain total volume. Process 2. Vreactant ∝ [reactant] 3. Rate ∝ 1/t or V _{clock} / t 4. Mix water and reactants. Last reactant added will start reaction. Start stopwatch. 5. Stop stopwatch when stated change occurs.	Analyse clues	
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 Process 1. Total volume of every experiment is the same. Vary volume of one reactant and keep other constant, add water to maintain total volume. Process 2. V_{reactant} ∝ [reactant] Rate ∝ 1/t or V_{clock} / t Mix water and reactants. Last reactant added will start reaction. Start stopwatch. Stop stopwatch when stated change occurs. 	Weasurements	Time taken for stated change to occur
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Process 2. Vreactant ∝ [reactant] 3. Rate ∝ 1/t or V _{clock} / t 4. Mix water and reactants. Last reactant added will start reaction. Start stopwatch. 5. Stop stopwatch when stated change occurs.		• Vary volume of one reactant and keep other constant, add water to maintain total volume.
 3. Rate ∝ 1/t or V_{clock} / t 4. Mix water and reactants. Last reactant added will start reaction. Start stopwatch. 5. Stop stopwatch when stated change occurs. 	Process	2. V _{reactant} ∝[reactant]
4. Mix water and reactants. Last reactant added will start reaction. Start stopwatch.5. Stop stopwatch when stated change occurs.		3. Rate $\propto 1/t$ or V _{clock} / t
5. Stop stopwatch when stated change occurs.		4. Mix water and reactants. Last reactant added will start reaction. Start stopwatch.
		5. Stop stopwatch when stated change occurs.
	Kinetics (Continu	ious method)
<u>Kinetics (Continuous method)</u>	T opic & Aim	
Kinetics (Continuous method) Topic & Aim	• · ·	

Analyse clues	
M easurements	TimeConcentration (or measure of conc. E.g. colour intensity) at different times.
Process	 Measure concentration (or measure of conc) at different times. If sampling, quenching, and titration Mix reactants, last reactant starts the reaction. Start stopwatch. Draw sample from reaction mixture Quench at pre-determined time. Use titration to determine concentration of species in quenched sample.

♦ Worked Example 10 ♦ ♦

The investing the real	nitial rate of gated by the ' actions are as	the reaction bet 'clock" method, u follow: Clue	ween $S_2O_8^{2-}$ sing $S_2O_3^{2-}$ io	⁻ and I⁻ ions can b ons. The equations fo	oe T o or	pic : Kinetics (Clock reaction)
	S-0-2- 1 21-		.2-	reaction		
	5208- + 21	\longrightarrow I ₂ + 250	1-	reaction I		
	$I_2 + 2S_2O_3^{2-}$	——→ 2l [–] + S₄O	6 ^{2–}	reaction II		
When mixture react in any iou appear You a reaction	a small but e, the iodine b n reaction II u dine which is rance of a dee re to plan a on with respe	constant amount being slowly prod until all the S ₂ O ₃ ² further produced p blue colour in t series of experi ect to S ₂ O ₈ ²⁻ by t	of S ₂ O ₃ ^{2–} is uced by read has been u in reaction he presence ments to de he "clock" m	added to a reaction of will immediate used up. At that poin I will cause a sudde of starch. Attermine the order of thethod.	on ly it, en of	Clue im Vary V _{peroxodisulfate} to vary [peroxodisulfate].
You pr	ovided with th	e following:				
 aqu aqu aqu aqu sto wh 100 	ueous KI ueous Na ₂ S ₂ C ueous Na ₂ S ₂ C pwatch ite file 0 cm ³ beaker	• c • c • s • s • s • c • c • s • c • s • s • c • s • s • s • s • s • s • s • s • s • s	listilled water tarch solutior common appa tvailable in a aboratory	n aratus school	•	Reactants in main reaction : I and $S_2O_8^{2-}$ $S_2O_3^{2-}$ is used to delay the observation of I_2 .
The vo	lumes of read	ents for the expe	riment 1 have	e been predetermined	1.	
		•		·		
Exnt	Volume of	Volume of	Volume of	Volume of		
No	KI / cm ³	Na ₂ S ₂ O ₃ / cm ³	H_2O/cm^3	Na ₂ S ₂ O ₈ V / cm ³		
1	5	5	40	10		
1	5	5	40	10		
2						
3						
4						• Beaker : mix F , $S_2O_3^{2-}$, water and
5						starch.
(a) Co (b) D de m	escribe an escriber and the construction of the construction	ble above with ap experimental pro order of reaction v	propriate volu cedure that vith respect to	umes of each reagen would allow you to $S_2O_8^{2-}$ by the "clock	t. to k"	 Add 3208 informed and start stopwatch. Stop stopwatch when solution turns blue-black.
(c) Ex us	plain briefly h ed to determir	ow the <u>results of</u> ne the order of rea	the experime action with re	ental procedure can b spect to S ₂ O ₈ ^{2–} .		 V_{peroxodisulfate} ∝ [peroxodisulfate] Rate ∝ 1/time
Note:						
) ²⁻ will react y	with the added I-	(from KI) to f	orm Io		
- 02C	the formed T	will react with S	Ω_{0}^{2-} in the ex			
		$\frac{1}{2}$ will react with $\frac{1}{2}$		onetent emt of la		
• A C	onstant amt o	$1 \Im_2 \bigcup_3^-$ is used, i	esulting in a 2^{2} h of 2^{2}			
beii	ng generated	to use up the S ₂ C	³²⁻ before rea	acting with starch to		
aive						
<u> </u>				eT		

(a) Complete the table above with appropriate volumes of each reagent.

Expt No.	Volume of KI / cm ³	Volume of Na ₂ S ₂ O ₃ / cm ³	Volume of H ₂ O/ cm ³	Volume of Na ₂ S ₂ O ₈ , V / cm ³
1	5	5	40	10
2	5	5	30	20
3	5	5	20	30
4	5	5	10	40
5	5	5	0	50

- (b) Describe an experimental procedure that would allow you to determine the order of reaction with respect to $S_2O_8^{2-}$ by the "clock" method.
- 1. Using a 10 cm³ measuring cylinder, add 5 cm³ of KI and 5 cm³ of Na₂S₂O₃ to a 100 cm³ beaker.
- 2. Using a 50 cm³ measuring cylinder, add 40 cm³ of distilled water and 5 drops of starch solution to the same beaker.
- 3. Place the beaker on a white tile.
- 4. At an appropriate time, transfer 10 cm³ of Na₂S₂O₈ from a 10 cm³ measuring cylinder and start the stopwatch at the same time.

N	lote.	
1 1	ole.	

- volume of KI, Na₂S₂O₃ and total volume of solution must be kept constant
- rate of reaction $\propto 1/t$ and the initial $[S_2O_8{}^{2-}] \propto$ volume of $S_2O_8{}^{2-}$ used
- for graph plotting, there should be at least <u>5</u> data points
- 5. Record the time taken, t, for the solution to turn blue black.
- 6. Repeat steps 1 to 5 for Experiments 2 to 5 with varying volumes of distilled water and Na₂S₂O₈ according to the table.
- (c) Explain briefly how the results of the experimental procedure can be used to determine the order of reaction with respect to $S_2O_8^{2-}$.
 - Since the same fixed concentration of iodine is formed for each experiment when the stopwatch is stopped, rate of reaction $\propto \frac{1}{t}$.
 - Since the total volume of the reaction mixture is kept constant for all the experiments by adding appropriate volumes of water, initial $[S_2O_8^{2-}]$ in reaction mixture $\propto V$

<u>Method 1 – using $\frac{1}{4}$ </u>

Expt No.	Volume of KI / cm ³	Volume of Na ₂ S ₂ O ₃ / cm ³	Volume of H ₂ O/ cm ³	Volume of Na ₂ S ₂ O ₈ , V / cm ³	Time, t / s	$\frac{1}{t}$ / s ⁻¹
1	5	5	40	10		
2	5	5	30	20		

Comparing experiments 1 and 2,

When V is doubled and $\frac{1}{t}$ is constant	When V is doubled, 1/t is doubled	When V is doubled, 1/t is increased 4 times
⇒ $\frac{1}{t}$ does not change with change in V ⇒ rate does not change with change in [S ₂ O ₈ ²⁻] ⇒ order of reaction with respect to S ₂ O ₈ ²⁻ = 0	$\Rightarrow \frac{1}{t} \propto V$ $\Rightarrow \text{ rate } \propto [S_2O_8^{2-}]$ $\Rightarrow \text{ order of reaction with}$ respect to $S_2O_8^{2-} = 1$	$\Rightarrow \frac{1}{t} \propto V^{2}$ $\Rightarrow \text{ rate } \propto [S_{2}O_{8}^{2-}]^{2}$ $\Rightarrow \text{ order of reaction with }$ respect to $S_{2}O_{8}^{2-} = 2$

Method 2 – using Vⁿt

Expt No.	Volume of KI / cm ³	Volume of Na ₂ S ₂ O ₃ / cm ³	Volume of H ₂ O/ cm ³	Volume of Na ₂ S ₂ O ₈ , V / cm ³	Time, t / s	Vt / cm ³ s	V²t / cm ⁶ s
1	5	5	40	10			
2	5	5	30	20			

For all the experiments,

if t = constant,	if Vt = constant,	if V²t = constant,
$\Rightarrow \frac{1}{4} = \text{constant}$	$\Rightarrow V = \frac{constant}{t}$	$\Rightarrow V^2 = \frac{constant}{t}$
$\Rightarrow \frac{1}{t}$ does not change when V	$\Rightarrow \frac{1}{t} \propto V$ $\Rightarrow rate \propto [S_2O_8^{2-}]$	$\Rightarrow \frac{1}{t} \propto V^2$ $\Rightarrow \text{ rate } \propto [S_2O_8^2]^2$
changes \Rightarrow rate does not change with change in [S ₂ O ₈ ²⁻] Hence order of reaction with respect to S ₂ O ₈ ²⁻ = 0	Hence order of reaction with respect to $S_2O_8^{2-} = 1$	Hence order of reaction with respect to $S_2O_8^{2-} = 2$

Method 3 – Graphical method using $\frac{1}{t}$

Expt No.	Volume of KI / cm ³	Volume of Na ₂ S ₂ O ₃ / cm ³	Volume of H ₂ O/ cm ³	Volume of Na ₂ S ₂ O ₈ , V / cm ³	Time, t / s	$\frac{1}{t}$ / s ⁻¹
1	5	5	40	10		
2	5	5	30	20		
3	5	5	20	30		
4	5	5	10	40		
5	5	5	0	50		

For graphical method, need to have 5 data points for straight line graphs.

Plot a graph of
$$\frac{1}{t}$$
 against V



NOTE: Alternatively, the graph of $\lg \frac{1000}{time}$ against $\lg V$ can also be plotted.

♦ Worked Example 11 ♦ ♦ You are to investigate the order of reaction with respect to H2O2 Aim using a sampling method. Topic: ↘ Hydrogen peroxide decomposes catalytically according to the Kinetics (continuous method) following equation: Sampling, quench, titration $2H_2O_2(aq) \longrightarrow 2H_2O(l) + O_2(q)$ You are provided with the following: Aqueous hydrogen peroxide . Aqueous iron(III) chloride • Dilute cold sulfuric acid 0.02 mol dm⁻³ aqueous potassium manganate(VII) . 50 cm³ measuring cylinders . 50 cm³ burette 25 cm³ pipettes 250 cm³ beakers 250 cm³ conical flasks • Stopwatch . Glass rod Clue Iron(III) chloride catalyses the decomposition of hydrogen peroxide The amount of hydrogen peroxide remaining in the mixture at any titration with KMnO4 (need to time during the reaction may be determined by titration with Clue acidify) aqueous acidified potassium manganate(VII). (a) Write a balanced equation for the reaction between hydrogen peroxide and potassium manganate(VII). (b) Suggest why cold dilute sulfuric acid needs to be used in this Quench using cold reagent Clue experiment. (significantly slow down rate of decomposition of H_2O_2) • Acid medium for titration (c) Plan an investigation to determine the order of reaction with respect to H_2O_2 using a sampling, quenching and titration method. You may assume that you are provided with: • aqueous hydrogen peroxide aqueous iron(III) chloride • cold dilute sulfuric acid • 0.02 mol dm⁻³ aqueous potassium manganate(VII) • the equipment normally found in a school or college • laboratory In your plan you should include brief details of: the apparatus you would use, the quantities you would use, • Time (of quenching) the procedure you would follow, Final burette reading the measurements that you would take and the tabulation Initial burette reading of data. • $V(KMnO_4) \propto [H_2O_2]$ an outline of how the results would be used to determine • Plot V(KMnO₄) against time is equivalent the order with respect to hydrogen peroxide. to plotting [H₂O₂] against time.

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(a) Write a balanced equation for the reaction between hydrogen peroxide and potassium manganate(VII).

 $[O]: H_2O_2 \longrightarrow O_2 + 2H^+ + 2e^ [R]: MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O$ $Overall: 2MnO_4^- + 6H^+ + 5H_2O_2 \longrightarrow 2Mn^{2+} + 8H_2O + 5O_2$

(b) Suggest one use of dilute cold sulfuric acid in your experiment.

The addition of the acid dilutes and cools the mixture, significantly reducing the rate of decomposition. H₂SO₄ also serves to provide the acidic medium for the titration with MnO₄⁻.

(c) Plan an investigation to determine the order of reaction with respect to H₂O₂ using a sampling, quenching and titration method.

Procedure:

- 1. Fill a burette with the KMnO₄ solution provided.
- 2. Using a 100 cm³ measuring cylinder, add 100.0 cm³ of the H₂O₂ solution provided to a 250 cm³ conical flask labelled reaction mixture.
- 3. Using a 10 cm³ measuring cylinder, add 2.0 cm³ of the FeC*l*₃ solution into the same conical flask. Start the stopwatch and swirl the mixture thoroughly to mix its contents.

(Note: Since FeCl₃ is a catalyst, only a small amount/volume is required.)

- 4. Using a 50 cm³ measuring cylinder, measure 50.0 cm³ of the cold, dilute H_2SO_4 solution.
- 5. Using a 10.0 cm³ pipette, transfer 10.0 cm³ aliquot of the reaction mixture to a second 250 cm³ conical flask.
- 6. At time t = 2 min, transfer the 50.0 cm³ cold, dilute H_2SO_4 solution into the second conical flask and swirl the mixture.
- Immediately titrate the H₂O₂ in the second conical flask against the KMnO₄ solution. The end-point is reached when the solution turns from yellow to orange/pale pink. Record the titration results in the table below.

(Note: End point colour change is yellow (due to Fe^{3+}) to orange (mixture of yellow Fe^{3+} and pink of very dilute MnO_4^- . Also, only one titration is possible for each aliquot)

- 8. Wash out the second conical flask with water.
- 9. Repeat steps 4 to 8 at 5-minute interval.

Time from initial mixing, t / min	2	7	12	17	22	27	32	37	42
Final burette reading / cm ³									
Initial burette reading / cm ³									
Volume of KMnO ₄ used, V / cm^3									

