

Nat Rate Lab

by Natalie Coplin

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University of Wales Trinity Saint David

Ordnance, Munitions and Explosives



Advanced Chemistry of OME - ACDA6020

Laboratory Report – Rate of Reaction – Iodine Clock

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Date – 11/2025

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1. Aim

Using an experiment known as the iodine clock, the purpose of this experiment is to determine the rate law for the formation of iodine in the reaction between hydrogen peroxide (H_2O_2) and iodine ions in aqueous solution. Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) will be used as the reducing agent, and starch as the colour change indicator which turns blue when it reacts with iodine.

aim could be clearer in explaining better where the blue colour originates

1.1. Hypothesis

The rate of iodine formation in the reaction between hydrogen peroxide and iodide ions is dependent upon the concentration of both reactants. It is hypothesised that increasing the concentration of either of these reactants will increase the rate of the reaction, therefore decreasing the time taken for the ascorbic acid to be fully consumed and a blue colour change to be seen from the reaction of starch with accumulated iodine.*correct*

1.2. Predicted Outcome

As the concentration of 3% hydrogen peroxide increases in the reaction solution, it is expected that the rate of iodine formation will also increase. The time taken for the blue colour to appear will decrease due to the higher availability of the oxidising agent driving the reaction forward.

this is concentration before dilution

As the concentration of 0.2M KI increases in the reaction solution, the rate of iodine formation is expected to also increase, leading to faster consumption of ascorbic acid. Once fully consumed, iodine accumulates and reacts with starch to produce a visible colour change. Therefore, increasing iodide concentration should reduce the time taken to reach the end point of the reaction.*excellent prediction*

1.3. Key Variables

For this experiment the variables are:

Independent – series 1 – H_2O_2 concentration, series 2 - KI concentration

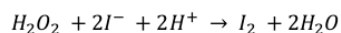
Dependent – time for solution to change colour.

Control – ascorbic acid and starch concentration, reaction solution volume.

1.4. Theory

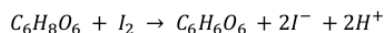
In this kinetics experiment hydrogen peroxide oxidises iodide ions to form iodine, while ascorbic acid constantly reduces iodine back to iodide - delaying the accumulation of iodine and the colour change. The end point can be observed when the blue colour forms, allowing the rate of the reaction to be monitored as a function of time.

The reaction above involves two stages. The first is the reaction between hydrogen peroxide and iodide ions. Hydrogen peroxide oxidises iodide ions to iodine, shown in Equation 1.



Equation 1 Hydrogen peroxide oxidises iodide ions

The second half of the reaction is the reduction of iodine back to iodide ions by the ascorbic acid, shown in Equation 2.



Equation 2 Ascorbic acid reduces iodine back to iodide ions

1.5. Rate Law and Order of Reaction

The order of a reaction describes how the rate of reaction is affected by the concentration of each reactant [1] [2]. It can only be determined experimentally by measuring how the reaction rate changes when the concentration of each reactants is varied. It is expressed using the rate law which illustrates the effect of varying concentration on the rate of the reaction [3].

Common orders of reaction include *important, but not necessary here*

Zero – rate is independent of the reactant concentration; changes in concentration do not affect the rate.

First – rate is directionally proportional to the reactant concentration; doubling the concentration doubles the rate of reaction.

Second - rate is proportional to the square of the reactant concentration; doubling the concentration increases the rate by a factor of four. [4]

The rate law for the iodine clock can be determined by monitoring the rate at which iodine reacts with starch to produce a blue colour change. The colour formation marks the point at which ascorbic acid is fully consumed, and therefore the end point of the reaction. The time taken for the colour change is inversely proportional to the rate of iodine formation – based on the same amount of iodine being produced in each experiment. The reciprocal of time (1/t) is an approximate measure of the rate of reaction:

$$\text{Rate} \propto \frac{1}{t}$$

Equation 3 Rate of reaction

Where time (t) is measured in seconds (s).

The general rate law for the reaction is:

$$rate = k[H_2O_2]^m[I^-]^n$$

Equation 4 Rate equation expressed in terms of reactant concentration

Given the rate is inversely proportional to time as shown in Equation 3, it can be expressed as:

$$1/t = k[H_2O_2]^m[I^-]^n$$

Where:

k = rate constant

[H₂O₂] = concentration of hydrogen peroxide

m = order of reaction with respect to hydrogen peroxide

[I⁻] = concentration of iodide ions

n = order of reaction with respect to iodide ions

Plotting log (1/time) against the log of the initial concentration of the reagents - [H₂O₂]₀ and [I⁻]₀, the order of each reaction can be determined.

This allows k to be calculated for each experiment using:

$$k = \frac{(1/time)}{([H_2O_2]^m[I^-]^n)}$$

Equation 5 Rate law equation

The average of k can then be determined.

1.6. Importance of kinetics in ordnance, munitions and explosives (OME)

Kinetic studies are invaluable in the field of OME for hazard and risk assessments and through-life surveillance. The rate of a reaction directly influences the performance, reliability and safety of explosives through the entire Manufacture to Target or Disposal Sequence (MTDS) [5]. Kinetic

studies into areas such as activation energy, rate of reaction and thermal decomposition all play a key role in understanding how energetic materials will behave across their lifecycle.

For example, techniques such as differential thermal analysis (DTA), differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) are used to assess the thermal stability of explosives and energetic polymers during development of new compounds [6], [7] .

By combining experimental data with kinetic models, critical factors, including decomposition pathways, stabiliser aging, manufacture operating conditions and burn rates can be accurately determined informing the decision-making process for all aspects of the explosive life cycle [8],

2. Results

2.1. Observations

Upon mixing the colourless reagents, a blue colour developed in each reaction within minutes. The time taken for the colour change appearance decreased in each test series as the concentration of either reactant increased, indicating the rate of reaction in each series was increasing.

2.2. Calculations

2.2.1. Hydrogen Peroxide

To calculate the initial concentration of hydrogen peroxide in the reaction mixture, the number of moles of hydrogen peroxide in the stock solution must first be determined. A 3% w/v solution of hydrogen peroxide contains 3 g per 100 ml, equivalent to 30g in 1000ml. Using the periodic table, the molar mass of H_2O_2 can be found:

Hydrogen: 1.008 g/mol

Oxygen: 15.999 g/mol

For $H_2O_2 = (2 \times 1.008) + (2 \times 15.999) = \mathbf{34.014 \text{ g/mol}}$.

Moles in 1 L:

$$\text{Moles} = \frac{\text{Mass (g)}}{\text{Molar mass (g/mol)}}$$

Equation 6 Molar concentration

$$\text{Moles of } H_2O_2 = \frac{30}{34.014}$$

$$\text{Moles of } H_2O_2 = \mathbf{0.882 \text{ mol}}$$

There are 0.882 mol of H_2O_2 in the stock solution, as this is calculated for 1 L the molarity is **0.882 mol/L**. The molarity is required to calculate the number of moles of H_2O_2

Next find the number of moles added to reaction solution. Convert the volume of H_2O_2 added to each reaction from millilitres to litres:

For experiment 1.1 this is 1 ml = 0.001 L

$$\text{Moles added (mol)} = \text{Molarity (mol/L)} \times \text{Volume added(L)}$$

Equation 7 Molarity Equation

$$\text{Moles} = 0.882 \times 0.001$$

$$\text{Moles} = \mathbf{0.000882 \text{ mol}}$$

Finally, rearranging Equation 7, calculate the initial concentration of H_2O_2 in the reaction solution.

$$\text{Initial } [\text{H}_2\text{O}_2] = \frac{\text{Moles added (mol)}}{\text{Volume of reaction solution (L)}}$$

$$\text{Initial } [\text{H}_2\text{O}_2] = \frac{0.000882}{0.025}$$

$$\text{Initial } [\text{H}_2\text{O}_2] = \mathbf{0.03528 \text{ mol/L}}$$

why didn't you use $C_1V_1 = C_2V_2$?

Table 1 shows the results from performing the above steps for each experiment in series 1.

Table 1 Moles of H_2O_2 added and initial concentration of H_2O_2 in series 1 experiments

Experiment	Volume of H_2O_2 added (L)	Moles added to reaction (mol)	$[\text{H}_2\text{O}_2]_0$ mol/L
1.1	0.001	0.000882	0.0353
1.2	0.002	0.001754	0.0706
1.3	0.003	0.002646	0.1058
1.4	0.004	0.003528	0.1411

2.2.2. Potassium Iodide

The molarity of the stock solution of potassium iodide is already known, therefore, only the initial concentration in the reaction solution needs to be calculated. This is done using:

$$C_1 \times V_1 = C_2 \times V_2$$

Equation 8 Dilution equation

Where:

C_1 = concentration of stock solution (0.2 M)

V_1 = volume of stock solution added (0.001 L)

C_2 = concentration of final solution (to be calculated)

V_2 = volume of final solution (0.025 L)

For experiment 2.1:

$$C_2 = \frac{C_1 \times V_1}{V_2}$$

$$C2 = \frac{0.2 \times 0.001}{0.025}$$

$$C2 = 0.008 \text{ M}$$

Table 2 shows the results from performing the above steps for each experiment in series 2.

Table 2 Moles of KI added and initial concentration in series 2 experiments

Experiment	Volume of KI added (L)	Moles added to reaction (mol)	[KI] ₀ mol/L
2.1	0.001	0.0002	0.008
2.2	0.002	0.0004	0.016
2.3	0.003	0.0006	0.024
2.4	0.004	0.0008	0.032

Table 3 shows the complete results from the experiments with the log of the reactant concentration and log of 1/t, which was plotted in Figure 1 and Figure 2.

Table 3 Complete experimental results

Experiment	Reactant	[Reactant] ₀ mol/L	Time (s)	1/t (s ⁻¹)	log[Reactant]	log(1/t)
1.1	H ₂ O ₂	0.0353	110	0.00909	-1.45223	-2.04139
1.2		0.0706	60	0.01667	-1.15120	-1.77815
1.3		0.1058	53	0.01887	-0.97551	-1.72428
1.4		0.1411	47	0.02128	-0.85047	-1.6721
2.1	KI	0.008	65	0.01538	-2.09691	-1.81291
2.2		0.016	74	0.01351	-1.79588	-1.86923
2.3		0.024	57	0.01754	-1.61979	-1.75587
2.4		0.032	49	0.02041	-1.49485	-1.6902

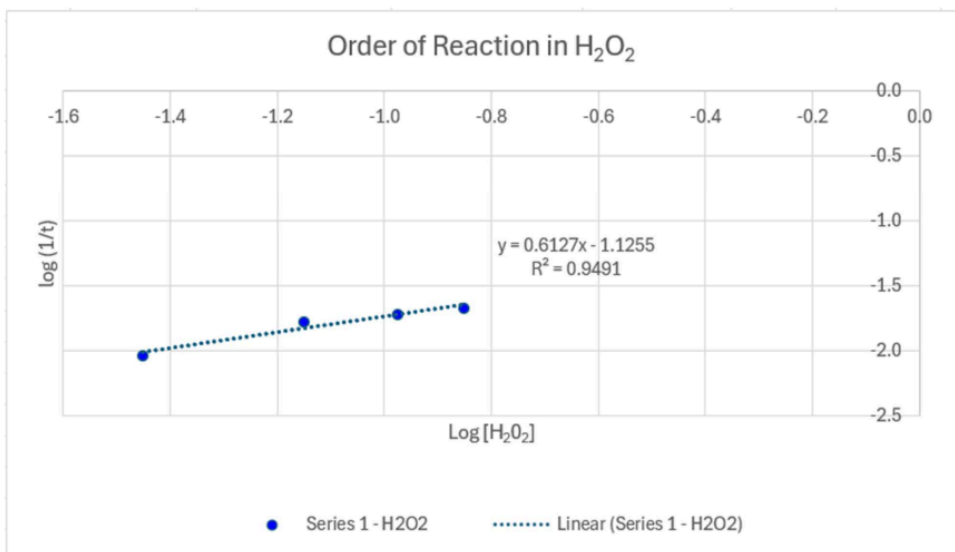


Figure 1 Series 1 result - H₂O₂

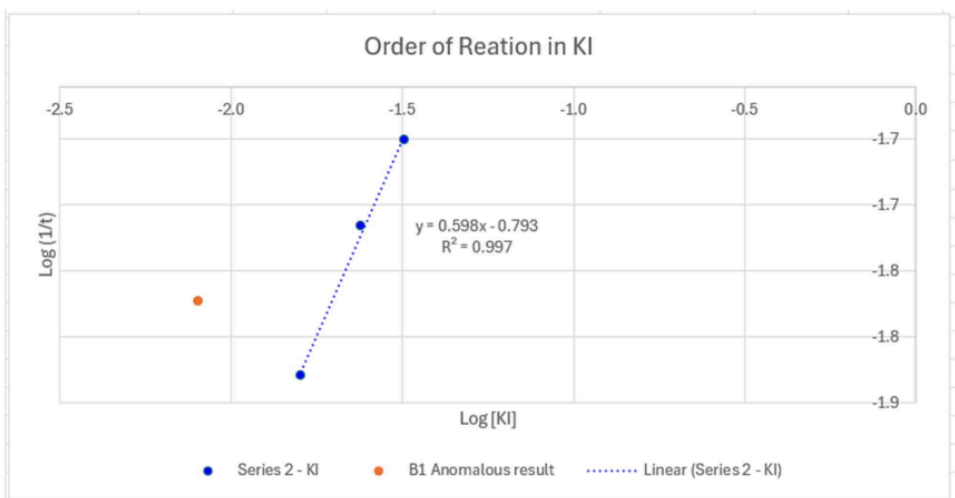


Figure 2 Series 2 Result - KI

2.2.3. Rate Constant - k

The rate constant k can be calculated using Equation 5, shown below for clarity.

$$k = \frac{(1/time)}{([H_2O_2]^m [I^-]^n)}$$

For experiment 1.1, this is as follows:

$$k = \frac{(0.00909)}{([0.0353]^{0.6127}[0.024]^{0.598})}$$

$$k = 0.6561746$$

This is repeated for each experiment. The complete results are shown in Table 4 along with the average k.

Table 4 Average of k (rate constant)

Experiment	k
1.1	0.65617
1.2	0.78672
1.3	0.69511
1.4	0.65708
2.1	1.09329
2.2	0.63446
2.3	0.64633
2.4	0.63303
Average (excluding 2.1)	0.67270

sig figs

Averaged k for the two series of experiments is **0.67270**, this is specific for this reaction. Experiment 2.1 is highlighted as it was identified as an anomalous results. This is discussed further in Appendix 3 – Anomalous Result *it is important part of the report, should not be discussed in appendix.*

2.2.4. Order of Reaction

The slopes of the log-log plots in Figure 1 and Figure 2 give the order of reaction with respect to H₂O₂ (m) and KI (n) respectively. These values correspond to the exponents of the concentration terms in the rate law. The overall order of reaction is the sum of these exponents.

$$rate = k[A]^m + [B]^n$$

Equation 9 Rate law equation

Where:

$$m = 0.6127 \text{ (H}_2\text{O}_2\text{)}$$

$$n = 0.598 \text{ (KI)}$$

Therefore:

$$\text{Overall order reaction} = m + n$$

Series 1 order of reaction = 0.6127

Series 2 order of reaction = 0.598

$$\text{Overall order} = 0.6127 + 0.598 = \mathbf{1.21}$$

Equation 10 Overall order of reaction

This indicates the order of reaction is neither first nor second with respect to either reactant. It exhibits non-linear (fractional) kinetics.

The overall order of reaction allows the units to be determined. The units for k depend on the order of the reaction [9].

$$\text{Rate units} = M \cdot L^{-1} \cdot s^{-1}$$

And as the order is fractional, the following is used:

$$\text{Units of } k = M^{x-1} \cdot L^{1-x} \cdot s^{-1}$$

Where:

$$x = 1.21$$

$$\text{Units of } k = M^{-0.21} \cdot L^{0.21} \cdot s^{-1}$$

2.2.5. Rate Law

The rate law for this experiment has been determined.

Given:

Order with respect to H_2O_2 – m = 0.6123

Order with respect to KI – n = 0.598

Rate constant – k = 0.673

Therefore:

$$\text{Rate} = \mathbf{0.673} * [H_2O_2]^{0.6123} * [KI]^{0.598}$$

3. Discussion

3.1. Experimental Error

The iodine clock experiment is inherently prone to experimental error. It involves measuring multiple reactants, visual observation of the colour change, and the need for accurate timing. Errors may also be introduced through environmental factors such as temperature changes. Table 5 lists sources of error in this experiment and makes suggestions for improvements. An in-depth discussion of these points can be found in Appendix 1.

Table 5 Experimental Error

Source of error	Problem	Solution	Example
Temperature fluctuations	Affects the rate of the reaction which may lead to increased/decreased rate of reaction	Temperature controlled environment	Ice bath
Experimental set-up	Variations in concentration leading to inaccurate rates of reaction	Purchasing reagents	Pre-made solutions
Equipment limitations	Inconsistent volume measurements leading to inaccurate measurement of rate of reaction	Accurate volume measuring equipment	Pipettes rather than pipettes fillers
Experimental process	Variable speed of pouring may affect rate of reaction between experiments	Automate pouring of reagents	Syringe pump
	Inconsistent mixing speed may affect rate of reaction	Mixing of reagents	Magnetic stirring
Interpretation of colour change	Different stage of colour change may be detected leading to inaccurate recording of endpoint	Automate detection of colour change	Colourimeter
Solution age	Old reagents may delay colour change leading to delayed end point	Freshly prepare solutions	
Equipment contamination	Contaminants may affect rate of reaction leading to inaccurate rate of reaction	Separate equipment for each reagent	

3.2. Experiment discussion

The iodine clock experiment successfully demonstrated the rate of iodine formation is dependent on the concentration of hydrogen peroxide and iodide ions. Reaction end point was visually monitored, and volume measurement method was not accurate. As discussed in 3.1 this can lead to experimental error and may have contributed to the anomalous result in series 2.

Log-log plots were used to determine the reaction orders, which were found to be neither first nor second order. The fractional orders of reaction were not expected and therefore further work is required. Repeat experiments using the same equipment would be an acceptable method, however, as discussed in Appendix 1, using more accurate equipment for the timing and volume measurements would provide greater confidence in the results.

Finally, performing the reaction at varying temperatures while keeping reactant concentration constant, would enable determination of activation energy (E_a) and the collision factor (A). Activation energy is the minimum energy necessary for a chemical reaction to occur [10], while collision factor represents the probability that molecular collisions in a reaction will occur in the correct orientation for a reaction to take place. Both are components in the Arrhenius equation which models the temperature dependence of the rate of reaction [11].

4. Conclusion

The results of this experiment support the initial hypothesis that the rate of the iodine formation is dependent on the concentration of hydrogen peroxide and iodide ions. As predicted, increase the concentration of either reactant resulted in an increase in the rate of reaction. This is seen with the decrease in time taken for the blue colour to appear.

Analysis of the results established the rate law to be:

$$Rate = 0.673 * [H_2O_2]^{0.6123} * [KI]^{0.598}$$

This confirms the prediction that higher concentrations of H_2O_2 or KI will lead to faster consumption of ascorbic acid, resulting in the accumulation of iodine which then reacts with starch more rapidly to produce the characteristic blue colour of the iodine clock reaction.

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6. Appendix 1 – Experimental Error

A detailed discussion of sources of error for the Iodine clock experiment is shown below as a follow on from Table 5.

- **Temperature changes** – the rate of reaction is highly dependent on the ambient temperature and that of the solutions [10]. Higher temperatures will increase the rate of reaction, while lower temperatures will slow it down. Conducting the experiment in a temperature-controlled environment will help reduce this effect. This could be done simply, by using an ice bath.
- **Experimental set-up** – preparation of solutions of specific concentration and accurate measuring of small volumes are both prone to human error. These could be reduced by purchasing reagents at the required concentrations and by using more accurate equipment such as pipettes.
- **Equipment limitations** – the pipette fillers provided for this experiment are incredibly inaccurate and prone to human error [11]. Consistent and good technique is essential however, small variations in volume measurements will still be common. It is recommended that pipette fillers are not used for experiments requiring accurate volume measurements. Alternatively, pipettes can easily measure the smaller volumes required in this experiment – brands such as Gilson, VWR and Eppendorf all produce suitable manual and electronic options – noting that any equipment used should be regularly calibrated. Water could be accurately measured by weighing.
- **Experimental process** – combining the reagents manually introduced a potential variability in the speed at which the student poured. A syringe pump could help improve control but for a simple experiment, this may be excessive. Stirring the mixed reagents was also performed manually. In this case by the same person, for a measured amount of time in an attempt to reduce error. This could be further improved using a magnetic plate and stirrer, which should remain at the same speed for each experiment.
- **Interpretation of the colour change** – observation of the colour change is performed manually and is highly subjective. Using a colourimeter would eliminate this error [12]. This monitors the absorbance of the solution over time, producing an absorbance peak on a spectrum the moment the solution turns blue. Data analysis is slightly more complicated; however, the final data will be more consistent and accurate.
- **Solution age** - it is not clear if the solutions were freshly prepared or made in advance of the laboratory session. Older samples may weaken the colour change, resulting in slower observation of the colour.

- **Equipment contamination** - equipment was reused with only simple washing between experiments and rinsing with deionised water. If this was not performed thoroughly residual contaminants may be introduced to the subsequent experiment which could affect the rate of reaction. Ideally, separate equipment should be used for each reagent, to reduce the risk of contamination.

For all of the above, a balance must be struck between the level of accuracy required and the availability and cost of more sophisticated equipment.

7. Appendix 2 – Methodology

7.1. Chemicals and equipment

- 0.20 M KI solution
- 0.01 M ascorbic acid solution
- 1% starch indicator
- Distilled water
- 3% H_2O_2 solution
- 4 x 50 ml conical flasks
- 2 x glass pipettes
- Small beakers
- Test tube
- Stopwatch
- Glass stirring rod
- Personal protective equipment – laboratory coat, safety glasses, gloves

7.2. Procedure

Figure 3 is a basic schematic of the experimental set up showing H_2O_2 in one beaker and the remaining reagents in another beaker, with the colour change appearing after a time.

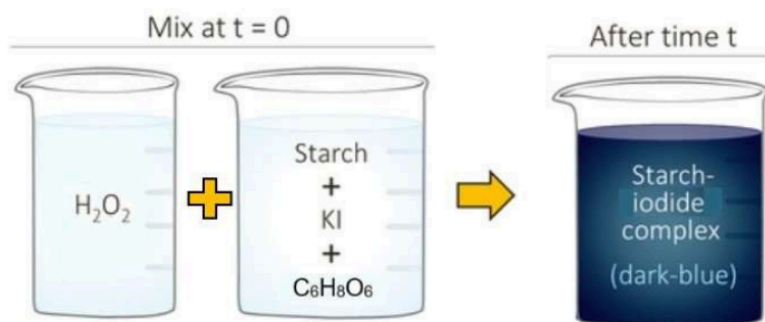


Figure 3 Diagrammatic representation of experimental set-up - adapted from [13]

Note for both series – the timer should be started as soon as both solutions are mixed and the solutions stirred for a set amount of time (i.e. 25 seconds).

Experimental set up:

The process below is repeated for each series – note the different volumes of each reactant between series, shown Table 6 and Table 7. Table 8 is provided for the recording of results.

1. Take four conical flasks, label them according to the experiment numbers (1.1 – 1.4), label the beaker and test tube with 'H₂O₂'.
2. Prepare solution A - using a glass pipette measure the required volume of KI and ascorbic acid into the flask. Using the sales, measure the required volume of water.
3. Add 1 ml of the starch solution to the same flask.
4. Prepare solution B - using the second pipette, measure the required volume of H₂O₂ into the test tube.
5. Ensure the stop clock is set to zero.
6. Pour solution B from the test tube into solution A in the flask and start the stop clock. Using the glass stirring rod stir the mixture for the decided time.
7. Observe the mixture until the blue colour appears. Promptly stop the stop clock.
8. Dispose of the solution down the sink, wash all equipment before rinsing with deionised water.

Table 6 Required volumes for each series 1 experiment

Experiment	A: KI 0.2 m (ml)	A: Ascorbic acid 0.01 M (ml)	A: Starch 1% (ml)	A: Water (ml)	B: H ₂ O ₂ 3% (ml)
1.1	3.0	1.5	1.0	18.5	1.0
1.2	3.0	1.5	1.0	17.5	2.0
1.3	3.0	1.5	1.0	16.5	3.0
1.4	3.0	1.5	1.0	15.5	4.0

Table 7 Required volumes for each series 2 experiment

Experiment	A: KI 0.2 m (ml)	A: Ascorbic acid 0.01 M (ml)	A: Starch 1% (ml)	A: Water (ml)	B: H ₂ O ₂ 3% (ml)
2.1	1.0	1.5	1.0	18.5	3.0
2.2	2.0	1.5	1.0	17.5	3.0
2.3	3.0	1.5	1.0	16.5	3.0
2.4	4.0	1.5	1.0	15.5	3.0

Table 8 Blank results table

Experiment	Reactant	[Reactant] ₀ mol/L	Time (s)	1/t (s ⁻¹)	log[Reactant]	log(1/t)
1.1	H ₂ O ₂					
1.2						
1.3						
1.4						
2.1	KI					
2.2						
2.3						
2.4						

8. Appendix 3 – Anomalous Result

It was clear from looking at series 2 results that there was a deviation from the expected straight line, indicating an anomalous results. The rate-concentration relationship should produce a straight line when $\log[\text{reactants}]$ is plotted against $\log(1/t)$. By calculating the coefficient of determination (R^2) after omitting each result, the anomalous result was determined to be 2.1 as this produced an R^2 value closer to 1 than by omitting 2.2 [14] [15]. This is shown in Figure 4 and Figure 5 where:

2.1 omitted $R^2 = 0.997$

2.2 omitted $R^2 = 0.8717$

Consequently, 2.1 was omitted from further calculations.

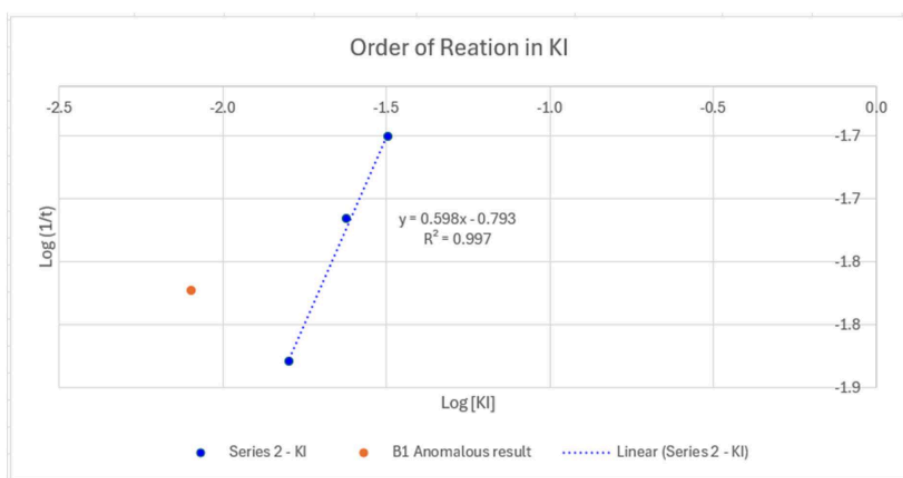


Figure 4 Series 2 results - 2.1 omitted

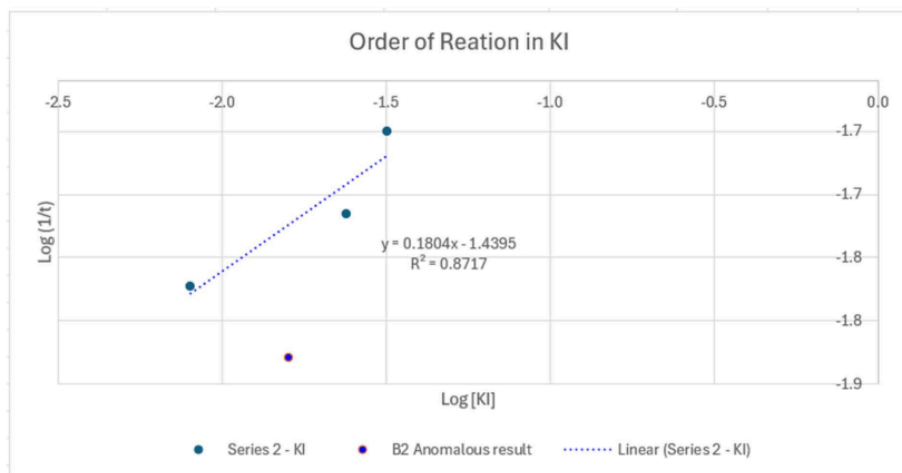


Figure 5 Series 2 results - 2.2 omitted

FINAL GRADE

74/100

GENERAL COMMENTS

Engagement with Literature 12/15

A strong and varied list of references was provided, including both academic and high-quality non-academic sources. These were used effectively to support theoretical explanations and contextual discussion, although the hypothesis itself was not supported by literature. Some references were included but not integrated into the narrative, reducing their impact. IEEE formatting was inconsistent (missing access dates, incorrect ordering). OME-related literature was strong, while the kinetics-specific and clock-reaction literature was less fully developed.

Knowledge and Understanding 15/20

The hypothesis and variables were mostly correct. Redox reactions involving H_2O_2 , I^- , I_2 , and vitamin C were accurately described and used appropriately in context. Reaction order determination was explained, though definitions of rate law and rate constant were not provided.

A clear explanation was given for why $\text{rate} \propto 1/t$ in the clock method. Calculations were well explained, and the relevance of kinetics to the OME lifecycle was articulated. Reaction orders were not rounded to expected integer values, leaving some interpretation incomplete.

Cognitive and Intellectual Skills 15/20

An anomalous result was identified and discussed, and the justification for excluding it was clearly reasoned. Potential sources of error were recognised, with appropriate mitigation strategies suggested. Observed data were compared with the hypothesis in a logical manner. Analysis would be strengthened by statistical evaluation (e.g. standard deviation of k). Interpretation focused mainly on trends and could be extended by addressing the mechanistic origin of fractional orders.

Practical Application Skills 22/30

Concentration and dilution calculations were performed correctly, although $C_1V_1 = C_2V_2$ was not applied for H_2O_2 . Significant-figure use was inconsistent. The unit of the rate constant was correctly derived.

Log-log plots were properly constructed, and best-fit lines were drawn and used to determine reaction orders. The rate constant k was calculated according to the laboratory handout. Overall, practical execution was strong, with minor issues in precision and method consistency.

Transferable Skills 10/15

The report was professionally structured, written in a clear and formal tone, and used precise scientific terminology. Tables and sections were well organised. However, portions of the text were repetitive and did not add new information. A few sections would benefit from tighter expression and clearer transitions.

PAGE 1

PAGE 2

PAGE 3

PAGE 4

Text Comment. aim could be clearer in explaining better where the blue colour originates

Text Comment. correct

Text Comment. this is concentration before dilution

Strikethrough.

Text Comment. excellent prediction

Text Comment. temperature, mixing.

Text Comment. variables mostly complete

Text Comment. repeated! theory overlaps with previous sections

Text Comment. important, but not necessary here

Text Comment. why didn't you use $C_1V_1 = C_2V_2$?

Text Comment. sig figs

Text Comment. it is important part of the report, should not be discussed in appendix.

Text Comment. these should be rounded off to 1