Experiment 8 : Iodine Clock Reaction – Determination of Reaction Order and Activation Energy

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I. Objective

Traditional heating and microwave heating methods were used to extract iron ions from sesame powder, and the heating extraction efficiency of the two was compared.

II. Technique

Learn the use of scale absorption tubes, reaction color judgment, reaction rate measurement and activation energy calculation.

III. Introduction

(I) Reaction Rate and Reactant Concentration

To measure the reaction rate between $S_2O_8^{2-}$ and I^- (Eq. S8-1), a limited amount of $S_2O_3^{2-}$ was added as a timing agent. It rapidly reacts with the I_2 produced (Eq. S8-2) immediately after mixing.

Once $S_2O_3^{2-}$ is completely consumed, the remaining I_2 reacts with I^- to form I_3^-, which then forms a blue-black complex with the starch indicator.

Based on the stoichiometric relationship between $S_2O_3^{2-}$ and $S_2O_8^{2-}$ (Eq. S8-3), the concentration change of $S_2O_3^{2-}$ is directly related to the color change time (Δt).

$$S_2O_8^{2-}(aq) + 2I^-(aq) \rightarrow 2SO_4^{2-}(aq) + I_2(aq) \quad \text{(S8-1)}$$

$$2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq) \quad \text{(S8-2)}$$

$$\Delta\left[S_2O_3^{2-}\right] = 2\Delta\left[S_2O_8^{2-}\right] \quad \text{(S8-3)}$$

(II) Integrated Rate Law and Graphing Method

The concentration of reactants decreases over time, and the relationship between concentration and time can be described using the integrated rate law.

For a single-reactant reaction, A → P, the common integrated rate laws are as follows:

1. Zero-order reaction

For a zero-order reaction of A (rate independent of [A]), the rate law and integrated rate law are given in Equations S8-4 and S8-5, respectively.

$$\text{rate} = \frac{-\text{d[A]}}{\text{dt}} = \text{k} \qquad \text{(S8-4)}$$

After the points $[A] = -kt + [A]$ (S8-5)

Here, $[A]_0$ represents the initial concentration of reactant A, and [A] is the concentration after time t.

Plotting [A] versus t gives a straight line with a slope of −k and a y-intercept of $[A]_0$.

2. First-order reaction

For a first-order reaction of reactant A, the rate law and integrated rate law are given in Equations S8-6 and S8-7, respectively.

$$\text{rate} = \frac{-\text{d[A]}}{\text{dt}} = \text{k} \qquad \text{(S8-6)}$$

After the points $\ln[A] = -kt + \ln[A]_0$ (S8-7)

Plotting ln[A] versus t gives a straight line with a slope of −k and a y-intercept of $\ln[A]_0$.

3. Second-order reaction

For a second-order reaction of reactant A, the rate law and integrated rate law are given in Equations 8 and 9, respectively.

$$\text{rate} = \frac{-\text{d[A]}}{\text{dt}} = \text{k[A]}^2 \qquad \text{(S8-8)}$$

$$\text{After the points } \frac{1}{[A]} = \text{kt} + \frac{1}{[A]_0} \qquad \text{(S8-9)}$$

Plotting 1/[A] versus t gives a straight line with a slope of k and a y-intercept of $1/[A]_0$.

The integrated rate law describes how reactant concentration changes with time. Conversely, if the concentration of reactant A is measured over time, plotting [A], ln[A], and 1/[A] versus t allows determination of the reaction order based on which plot gives a straight line. The rate constant can then be obtained from the slope. This graphing method determines reaction order using a single initial concentration, without varying starting concentrations.

In the first part of this experiment, the integrated rate law method was used to determine the reaction order and rate constant with respect to $S_2O_8^{2-}$. The initial concentrations of $S_2O_8^{2-}$ and I^- were kept constant in each trial, while different amounts of the timing agent $S_2O_3^{2-}$ were added (Table 2). When the timing agent was fully consumed, the solution changed color at different times (Δt). The concentration change of $S_2O_3^{2-}$ was calculated from the moles added, and graphing was used to determine the reaction order and rate constant. In this design, $[I^-]$ was maintained much greater than $[S_2O_8^{2-}]$, assuming $[I^-]$ remains effectively constant, so only the reaction order of $S_2O_8^{2-}$ was investigated.

(III) Determination of Activation Energy

Reaction rates generally increase with temperature because the rate constant increases as temperature rises. In 1888, Swedish chemist Svante Arrhenius proposed that reacting molecules must possess a minimum energy, called the activation energy. The relationship between activation energy and the rate constant is described by the Arrhenius equation (Equations S8-10 and S8-11).

$$k = Ae^{-E_a/RT} \quad \text{(S8-10)}$$

$$\ln k = \frac{-E_a}{R}\frac{1}{T} + \ln A \quad \text{(S8-11)}$$

Where:

- **k**: rate constant
- **A**: Arrhenius constant (frequency factor)
- **E_a**: activation energy
- **T**: absolute temperature (K)
- **e**: base of the natural logarithm
- **R**: gas constant (8.314 $J \cdot mol^{-1} \cdot K^{-1}$)

The rate constants at different temperatures are determined, and a plot of ln *k* versus 1/T gives a straight line with slope $-E_a/R$, from which the activation energy can be obtained.

In the second part of this experiment, the reaction rate was measured at different temperatures. Since the reaction rate is proportional to the rate constant and inversely proportional to the color change time Δ*t* (Eq. S8-12), Equation S8-13 can be derived, where k is the rate constant, Δt is the color change time, and c is a constant.

$$k = \frac{c}{\Delta t} \quad \text{(S8-12)}$$

$$\ln k = \ln c + \ln\left(\frac{1}{\Delta t}\right) \quad \text{(S8-13)}$$

Substituting Equation 13 into the Arrhenius equation gives Equations S8-14 and S-15. Therefore, plotting ln(1/Δt) versus 1/T yields a straight line with slope $-E_a/R$, allowing determination of the activation energy.

$$\ln c + \ln\left(\frac{1}{\Delta t}\right) = \frac{-E_a}{R}\frac{1}{T} + \ln A \qquad \text{(S8-14)}$$

$$\ln\left(\frac{1}{\Delta t}\right) = \frac{-E_a}{R}\frac{1}{T} + \ln A - \ln c \qquad \text{(S8-15)}$$

(IV) Catalyst

Catalysts are often added to chemical reactions to provide an alternative pathway with lower activation energy, thereby increasing the reaction rate. In the third part of this experiment, Copper(II) sulfate ($CuSO_4$) was added to the reaction mixture to observe its effect on the reaction rate.

Table 2. Concentrations and volumes of reagents used in the integrated rate law experiment.

number	2 % starch (mL)	1.0 M NaI (mL)	0.20 M $Na_2S_2O_3$ (mL)	DI water (mL)
1	1.00	5.00	5.50	1.50
2	1.00	5.00	4.50	2.50
3	1.00	5.00	3.50	3.50
4	1.00	5.00	2.50	4.50
*5	1.00	5.00	1.50	5.50
6	1.00	5.00	0.50	6.50

IV. Equipment

- Electromagnetic Heating Stirrer, Magnetic Stirrer, Safety Suction Ball.
- Graduated Piction Tube (10 mL), Erlenmeyer flask (50 mL, 10 pcs.), thermometer.
- Cork (6 pcs.), Beaker (100 mL, 4 pcs.), Stopwatch, Styrofoam soup cup (2 pcs.).

V. Chemicals

- 1.0M Sodium iodide (NaI)
- 0.15M Potassium persulfate ($K_2S_2O_8$)
- 0.20M Sodium thiosulfate ($Na_2S_2O_3$)
- 2% Starch solution
- 0.020M Copper(II) sulfate ($CuSO_4$)

VI. Procedure

(1.)Integral rate law formula

1. Wash and dry ten 50 mL Erlenmeyer flasks, then cool them to room temperature. Label each flask and accurately add 2% Starch solution, 1.0 M Sodium iodide (NaI), 0.20 M Sodium thiosulfate ($Na_2S_2O_3$), and deionized water according to the volumes listed in Table 2.

2 (i) Using a calibrated liquid dispenser, accurately transfer 5.0 mL of the final reactant, 0.15 M Potassium persulfate ($K_2S_2O_8$), into the first Erlenmeyer flask and start timing immediately. Seal the flask with a cork stopper and shake continuously to ensure the solution is well mixed.

(ii)Stop timing when the solution changes color, and record the color change time.

3 (i) Repeat Step 2 by adding 5.0 mL of 0.15 M Potassium persulfate ($K_2S_2O_8$) sequentially into the 2nd to 6th Erlenmeyer flasks and measure the color change time for each reaction.

(ii) Measure the solution temperature in the 5th flask before and after the reaction, and use the average value as the reaction temperature under room conditions.

Note: Add the $K_2S_2O_8$ solution to each flask at 1-minute or 30-second intervals, and start timing all reactions simultaneously.

(2.)Measurement of activation energy

4. Prepare two Erlenmeyer flasks and, according to the amounts listed for item 5 in Table 2, accurately add 2% Starch solution, 1.0 M Sodium iodide (NaI), 0.20 M Sodium thiosulfate ($Na_2S_2O_3$), and deionized water into the flasks.

5. (i) Place a magnetic stir bar into one of the flasks prepared in Step 4 and put it in a polystyrene cup ice bath.

   (ii) Use a stirrer to mix the solution until the temperature reaches equilibrium. Record the initial solution temperature before the reaction.

   (iii) Add 5.0 mL of 0.15 M Potassium persulfate ($K_2S_2O_8$), start timing immediately, and continue stirring. When the solution changes color, stop timing and record the solution temperature. Use the average of the pre- and post-reaction temperatures as the reaction temperature under ice-bath conditions.

   Note: At low ice-bath temperatures, the reaction time is long (approximately 10–20 minutes). The solution temperature should be monitored continuously, and ice should be added as needed to maintain sufficient ice in the system and keep a constant temperature.

6. (i) Place the other flask prepared in Step 4 in a 40 °C warm water bath (or a beaker containing warm water slightly above room temperature) and allow the solution inside the flask to reach thermal equilibrium.

   (ii) Add 5.0 mL of 0.15 M Potassium persulfate ($K_2S_2O_8$), start timing immediately, and record both the color change time and the solution temperature.

   Note: Use a 400 mL beaker filled with an appropriate amount of warm water. The temperature only needs to be higher than room temperature and maintained constant; it is not necessary to adjust strictly to 40°C.

(2.) Catalyst and reaction rate

7. (i) According to the reagent volumes listed for item 5 in Table 2, accurately measure each solution and place them into the flask.

   (ii) Add 2 drops of 0.020 M Copper(II) sulfate ($CuSO_4$) solution, then finally add 5.0 mL of 0.15 M Potassium persulfate ($K_2S_2O_8$). Start timing immediately and mix the solution thoroughly.

8. (i) Waste liquid from the experiment contains iodine and should be poured into the designated waste collection container for centralized treatment.

   (ii) Clean the glassware by brushing and remove all labels.

V. Precautions

1. Confirm the exact solution volumes to be added and carefully monitor the color change. Stop timing immediately when the solution color changes.

2. After adding the solution, cover the flask with a cork stopper and shake well to ensure complete mixing and reduce experimental error caused by incomplete reaction.

3. If the 2% Starch solution shows any precipitation, shake it thoroughly before sampling.

4. Multiple timers or group timing methods can be used to simultaneously monitor several flasks.

5. The reacted solution contains halogens and should be disposed of in the halogen waste container.

6. During the experiment, always wear a lab coat, safety goggles, gloves, and a mask. All chemicals used in this experiment are toxic to some extent, so follow laboratory safety regulations and instructor instructions carefully to ensure safety.

7. All chemicals and experimental equipment must not be taken out of the laboratory.

VII. Reference

1. Dept. of Chemistry, U. of Illinois at Urbana-Champaign *General Chemistry*

Experiments, Chemistry 102, **1991**, Stipes Publishing Co.

Experiment 8 : Iodine Clock Reaction – Determination of Reaction Order and Activation Energy

Name: _____ **Student ID:** _____

Department: _____ **Group:** _____ **Date:** _____

I. Experimental Data and Results

1. Response time determination

No.	0.20M NaI (mL)	0.20M NaCl (mL)	0.005M $Na_2S_2O_3$ (mL)	2% Starch (mL)	0.10M K_2SO_4 (mL)	0.10M $K_2S_2O_8$ (mL)	Reaction Time (Δt) (sec)	
1								
2								
3								

2. Calculate the initial concentrations of each reactant and the initial rates for tests 1, 2, and 3:

$$Rate = \frac{-\Delta[S_2O_3^{2-}]}{\Delta t} = \frac{-\frac{1}{2}\Delta[S_2O_3^{2-}]}{\Delta t}$$

No.	Initial concentration of reactants in the mixture (M)			Average time Δt (sec)	Initial rate rate (M/s)
	$[S_2O_3^{2-}]$	$[S_2O_8^{2-}]$	$[I^-]$		
1					
2					
3					

Detailed calculations:

3. Calculate the reaction orders m and n with respect to $S_2O_8^{2-}$ and I^-. (Express to two significant figures.

4. Calculate the rate constant of this reaction.

5. Write the correct rate law expression.

6. Design your own experimental volumes (fill in the shaded areas):

   Predicted color-change time: _____ ;

   Measured color-change time: _____

No.	0.20M NaI (mL)	0.20M NaCl (mL)	0.0050M $Na_2S_2O_3$ (mL)	2% Starch (mL)	0.10M K_2SO_4 (mL)	0.10M $K_2S_2O_8$ (mL)	Reaction Color-Change Time (Δt, s)
1	2.0	2.0	1.0	1.0	2.0	2.0	Δt_1
Longer time	2.0	2.0	1.0	1.0	4.0 − x	x	Δt_x
3	4.0	0	1.0	1.0	2.0	2.0	Δt_3
Shorter time	4.0	0	1.0	1.0	4.0 − y	y	Δt_y

Note: Reaction rate is inversely proportional to the color-change time (Δt). The relationship with reactant amounts is simplified as follows:

$$(1)\ \frac{rate\ X}{rate\ 1} = \frac{\Delta t_1}{\Delta t_2} = \frac{\left(\frac{x \times 0.10}{10.0}\right)^m}{\left(\frac{2.0 \times 0.10}{10.0}\right)^m} = \left(\frac{x}{2.0}\right)^m$$

$$(2)\frac{rate\ \mathrm{Y}}{rate\ 3}=\frac{\Delta t_3}{\Delta t_\gamma}=\frac{\left(\frac{y\times 0.10}{10.0}\right)^m}{\left(\frac{2.0\times 0.10}{10.0}\right)^m}=\left(\frac{y}{2.0}\right)^m$$

Detailed calculations: