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114-2
Experiment 8 : Determination of the reaction series and activation energy of the iodine bell experiment

115.04.12~115.04.18 (time required: 2.5 hours)

1. Objective

1. The reaction rate between persulfate ($S_2O_8^{2-}$) and iodide (I^-) was studied using the redox reaction between thiosulfate ($S_2O_3^{2-}$) and iodine (I_2).
2. Reaction order and rate constants were determined using the initial rate method and the integrated rate method.
3. Activation energy was obtained by varying temperature, and the effect of catalysts on the reaction rate was also investigated.

2. Principle

1. Reaction rate and reaction stage

$$aA + bB \longrightarrow cC$$

$$\text{rate} = \frac{-d[A]}{a \times dt} = \frac{-d[B]}{b \times dt} = \frac{-d[C]}{c \times dt} \qquad \text{(eq. 8-1)}$$

Rate law formula $\Longrightarrow$ $\text{rate} = k[A]^m[B]^n$ (eq. 8-2)

Sum of total reaction series = m + n

(k = rate constant ; m、n = Experimental measurements are not determined by chemical equilibrium)

Reaction Between Persulfate and Iodide Ions

$S_2O_8^{2-} + 2I^- \longrightarrow 2SO_4^{2-} + I_2$ (eq. 8-3)

$\text{rate} = k[S_2O_8^{2-}]^m[I^-]^n$ (eq. 8-4)

$2S_2O_3^{2-} + I_2 \longrightarrow 2I^- + S_4O_6^{2-}$ (eq. 8-5)

$I^- + I_2 \longrightarrow I_3^-$

$I_3^- + \text{starch} \longrightarrow \text{blue complex}$

$\Delta [S_2O_3^{2-}] = 2\Delta [S_2O_8^{2-}]$ (eq. 8-6)

$$\text{rate} = \frac{-\Delta [S_2O_8^{2-}]}{\Delta t} = \frac{-1/2\ \Delta [S_2O_3^{2-}]}{\Delta t}$$ (eq. 8-7)

Initial reaction rate method

Vary the initial concentration of one reactant while keeping other conditions constant to determine its effect on the initial reaction rate.

Table 8-1. Concentrations and Volumes of Reagents Used in the Initial Rate Method.

No.	0.20 M NaI (mL)	0.20 M NaCl (mL)	0.005 M $Na_2S_2O_3$ (mL)	2% Starch (mL)	0.10 M K_2SO_4 (mL)	0.10 M $K_2S_2O_8$ (mL)
1	2.00	2.00	1.00	1.00	2.00	2.00
2	2.00	2.00	1.00	1.00	0	4.00
3	4.00	0	1.00	1.00	2.00	2.00

$$\frac{\text{(rate 2)}}{\text{(rate 1)}} = \frac{k(2[S_2O_8^{2-}]_1)^m([I^-]_1)^n}{k([S_2O_8^{2-}]_1)^m([I^-]_1)^n} = 2^m \quad \text{(eq. 8-9)}$$

$$\frac{\text{(rate 3)}}{\text{(rate 1)}} = \frac{k([S_2O_8^{2-}]_1)^m(2[I^-]_1)^n}{k([S_2O_8^{2-}]_1)^m([I^-]_1)^n} = 2^n \quad \text{(eq. 8-10)}$$

Integrated Rate Law Plots

1. zero order reaction

$$\text{rate} = \frac{-\,d[A]}{dt} = k$$

$$[A] = -\,kt + [A]_o$$

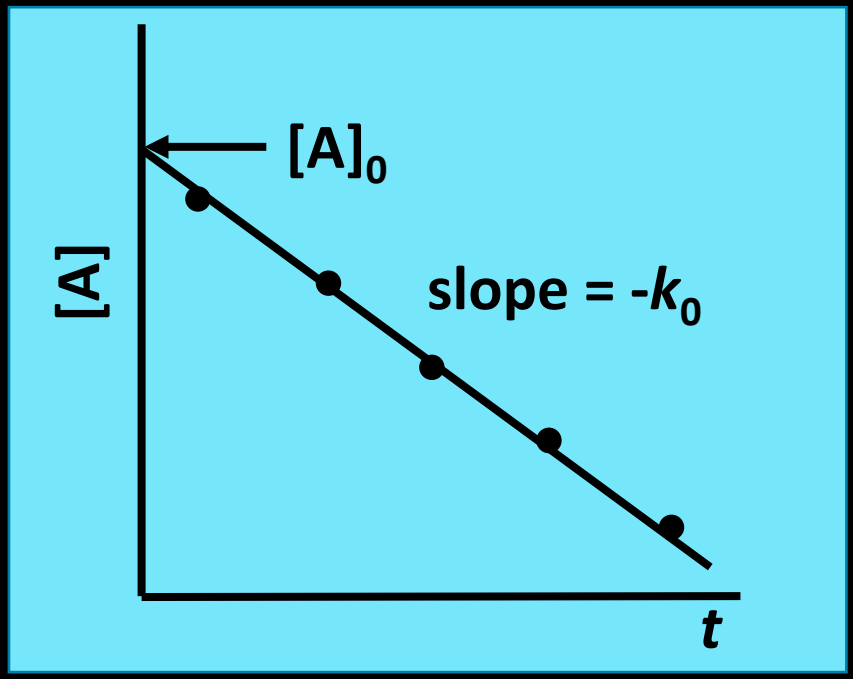

Figure 1. Zero-order reaction plot.

2. first order reaction

$$\text{rate} = \frac{-\,d[A]}{dt} = k[A]$$

$$\ln[A] = -\,kt + \ln[A]_o$$

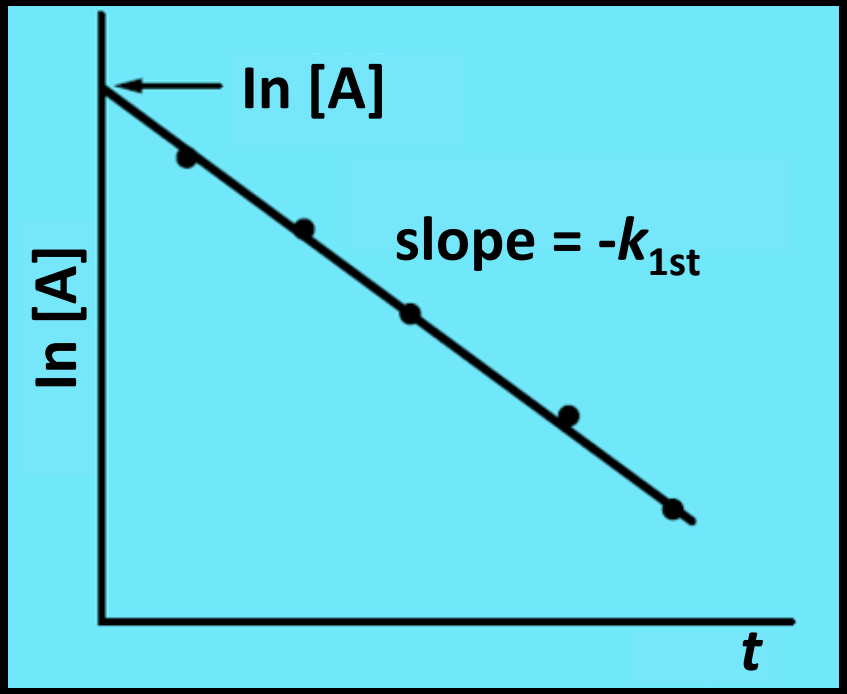

Figure 2. First-order reaction plot.

3. second order reaction

$$\text{rate} = \frac{-\,d[A]}{dt} = k[A]^2$$

$$\frac{1}{[A]} = kt + \frac{1}{[A]_o}$$

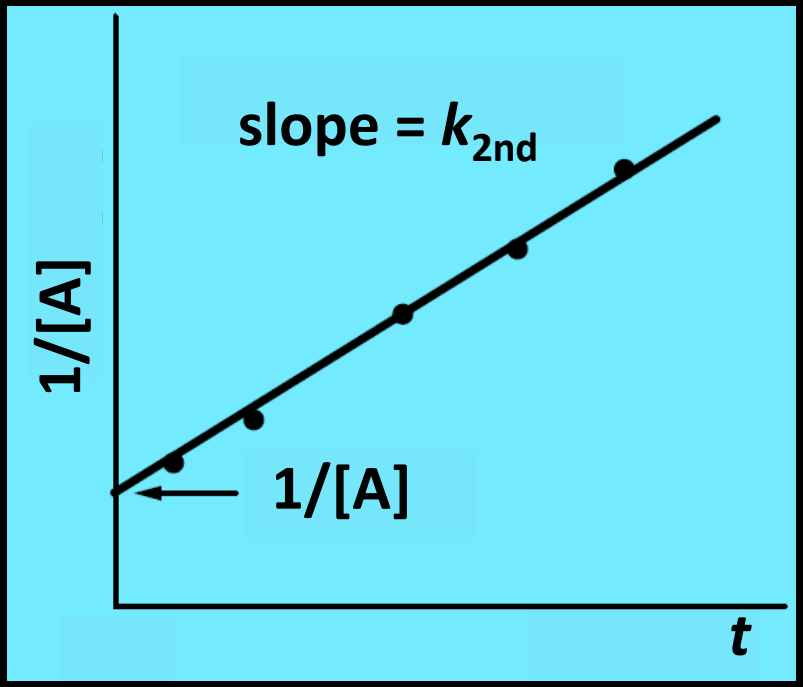

Figure 3. Second-order reaction plot.

Determination of Activation Energy

- **The minimum energy required for a reaction is called the activation energy (Ea).**

  Arrhenius equation:

$$k = Ae^{-E_a/RT}$$

$$\ln k = \frac{E_a}{R}\frac{1}{T} + \ln A$$

$$y = a\,X + b$$

Where:

k: rate constant

Ea: activation energy

R: gas constant, 8.314 J/mol·K

T: absolute temperature (K)

e: base of the natural logarithm

A: Arrhenius constant (frequency factor)

Catalyst

- In many chemical reactions, a suitable catalyst can be added to provide an alternative reaction pathway with lower activation energy, thereby increasing the reaction rate.

- In this experiment, $CuSO_{4(aq)}$ is added as a catalyst to the reaction solution to observe its effect on the reaction rate.

3. Equipment and Chemicals

Equipment

In cabinet	Provided by TA
Beaker (100 mL) x 2	Electronic thermometer
Safety suction ball x 1	Styrofoam soup cup x 2
Timer (self-bringing)	
Cork x 6	
Graduated pipette (10 mL) x 1	
Erlenmeyer flask (125 mL) x 6	

Chemicals

(I) Initial Rate Method	
0.20M Sodium Iodide (NaI)***	0.20M Sodium Chloride (NaCl)*
2% Starch Solution (starch)	0.10M Potassium Persulfate ($K_2S_2O_8$)****
0.10M Potassium Sulfate (K_2SO_4)**	0.005M Sodium Thiosulfate ($Na_2S_2O_3$)***

(II), (III), and (IV) Integrated Plot Method	
1.0M Sodium Iodide (NaI)***	0.15 M Potassium Persulfate ($K_2S_2O_8$)****
2% Starch Solution (starch)	0.20 M Sodium Thiosulfate ($Na_2S_2O_3$)***
Ice	0.02 M Copper(II) Sulfate ($CuSO_4$)***

* ：Corrosive、* ：Flammable、* ：Oxidizing、* ：Toxic

Reference：https://pubchem.ncbi.nlm.nih.gov/_Extract day: 2026.04.02

4. Experimental Procedures

A. Integral rate law formula

1. **Preparation:** Wash and dry six 50 mL Erlenmeyer flasks, then pipette the solutions into the flasks according to the ratios in Table 8-1.

Table 8-1. Reagent concentrations and volumes (initial rate method).

No.	0.20 M NaI (mL)	0.20 M NaCl (mL)	0.005 M $Na_2S_2O_3$ (mL)	2% Starch(mL)	0.10 M K_2SO_4 (mL)	0.10 M $K_2S_2O_8$ (mL)
1	2.00	2.00	1.00	1.00	2.00	2.00
2	2.00	2.00	1.00	1.00	0	4.00
3	4.00	0	1.00	1.00	2.00	2.00

The reaction begins immediately upon addition.

2. **Recording:** Add 0.10 M $K_2S_2O_8$ and start timing. Swirl for 20 s, then let stand until color change.
3. **Replication:** Stop timing at the color change and record the time. Repeat twice; redo if difference >5 s.

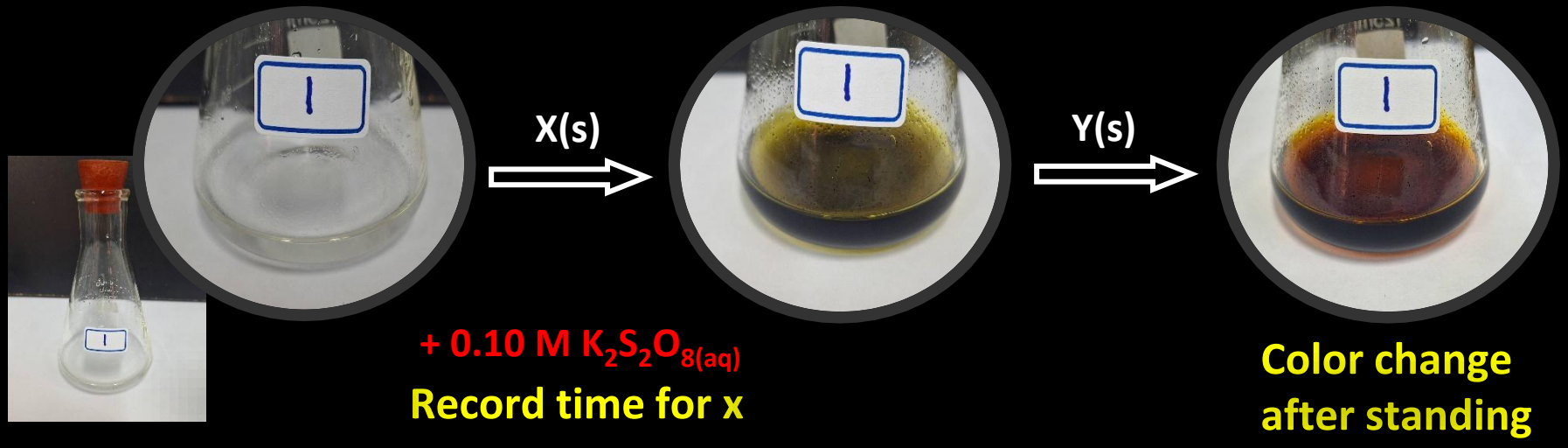

Figure 4. Reaction flow and color change

B. Integrated Rate Law Method

1. **Preparation:** Wash and dry five 125 mL Erlenmeyer flasks. Pipette solutions into each flask according to Table 8-2.

Table 8-2 Reagent concentrations and volumes for the integrated plot method.

No.	2% Starch (mL)	1.00 M NaI (mL)	0.20 M $Na_2S_2O_3$ (mL)	DI water (mL)	0.15 M $K_2S_2O_8$ (mL)
1	1.00	5.00	4.50	2.50	5.00
2	1.00	5.00	3.50	3.50	5.00
3	1.00	5.00	2.50	4.50	5.00
4	1.00	5.00	1.50	5.50	5.00
5	1.00	5.00	0.50	6.50	5.00

The reaction begins immediately upon addition.

2. **Reacting**：Add the final solution, 0.15 M $K_2S_2O_8$, start timing immediately, stopper with a cork, swirl 20 s, then let stand until color changes.
3. **Recording (I)**：Stop timing at color change and record the reaction time.
4. **Replication**：Repeat steps 2 and 3, adding 0.15 M $K_2S_2O_8$ to flasks 2–5 and measuring the reaction color-change time.
5. **Recording (II)**：Measure and record the solution temperature before and after the reaction at room temperature.

C. Measurement of activation energy

1. **Preparation:** Prepare two 125 mL Erlenmeyer flasks. Using a pipette, accurately measure the solutions into the flasks according to the volumes for sample No. 4 in Table 8-2.

Table 8-2. Reagent concentrations and volumes for sample No. 4 (Integrated Rate Law Method).

No.	2% Starch (mL)	1.00 M NaI (mL)	0.20 M $Na_2S_2O_3$ (mL)	DI water (mL)	0.15 M $K_2S_2O_8$ (mL)
4	1.00	5.00	1.50	5.50	5.00

The reaction begins immediately upon addition.

2. **Measure temperature:** Place flasks in an ice bath, equilibrate, measure temperature, add 5.00 mL 0.15 M $K_2S_2O_8$, start timing, and stir.
3. **Replication:** Repeat step 2 in 40°C warm water bath.

C. Catalyst and reaction rate

1. **Preparation:** Measure and add solutions to the flask according to Table 2 (item 5).
2. **Initiation:** Add 2 drops of 0.020 M $CuSO_4$, then 5.0 mL of 0.15 M $K_2S_2O_8$. Start timing and mix thoroughly.
3. **Disposal:** Dispose of iodine-containing waste in the designated container.
4. **Cleaning:** Brush and label glassware.

Table 8-2. Sample No. 4 reagent concentrations and volumes.

No.	2% Starch (mL)	1.00 M NaI (mL)	0.20 M $Na_2S_2O_3$ (mL)	DI water (mL)	0.15 M $K_2S_2O_8$ (mL)
4	1.00	5.00	1.50	5.50	5.00

The reaction begins immediately upon addition.

5. Safety and Precautions (1/2)

1. **Safety:** Standard PPE (lab coat, goggles, and gloves) is mandatory at all times.
2. **Chemical Handling:** Handle all chemicals with care, as they may be toxic or hazardous. Follow all laboratory safety regulations and the instructions of the TAs at all times.
3. **Prohibited Actions:** Removal of any chemicals or laboratory equipment from the lab is strictly prohibited.
4. **Color Judgment:** Confirm the exact solution volumes to be added and carefully monitor the color change. Stop timing immediately when the solution color changes.

5. Safety and Precautions (2/2)

6. **Uneven:** 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.
7. **Mixing:** If the 2% Starch solution shows any precipitation, shake it thoroughly before sampling.
8. **Timing:** Multiple timers or group timing methods can be used to simultaneously monitor several flasks.
9. **Waste:** The reacted solution contains halogens and should be disposed of in the halogen waste container.

6. Experimental Data

A. Integral rate law formula

1 . Reaction time measurement

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

2. Calculate the starting concentration of each reactant and the initial rate of tests 1, 2, and 3:

(Calculation process)

Initial rates of test 1, 2, and 3：rate (M/sec) = $\frac{-1/2\ \Delta[S_2O_3^{2-}]}{\Delta t}$

(Calculation process)

No.	Initial reactant concentration			Average time Δt (sec)	Reaction rate (M/sec)
	$[S_2O_3^{2-}]$	$[S_2O_8^{2-}]$	$[I^-]$		
1					
2					
3					

3．Calculate the reaction series m and n relative to $S_2O_8^{2-}$ and I^-. (expressed in two-digit significant figures)

(Calculation process)

4 . Calculate the rate constant for this experiment.

rate = $k([S_2O_8^{2-}])^m([I^-])^n$

(Calculation process)

k average is _____ ($1/M^2$)

5 . Write the correct rate law formula.

rate = (Calculation process)

B. Integrated Rate Law Method

1. Reaction time measurement

No.	2% Starch (mL)	1.00 M NaI (mL)	0.20 M $Na_2S_2O_3$ (mL)	DI water (mL)	0.15 M $K_2S_2O_8$ (mL)	average liquid temperature：_____℃ Reaction time (t, sec)
1						
2						
3						
*4						
5						

2. In five trials, the concentrations of reactants (NaI and $K_2S_2O_8$) remain constant, while only the amount of the timing agent $Na_2S_2O_3$ varies. Since $\Delta[S_2O_3^{2-}] = 2\Delta[S_2O_8^{2-}]$, when the solution changes color after time Δt (i.e., when $Na_2S_2O_3$ is consumed and the solution turns reddish-brown), the remaining $[S_2O_8^{2-}]$ can be calculated from the amount of $Na_2S_2O_3$ added.

(1) Calculate the moles of $S_2O_3^{2-}$ from the volume and concentration of $Na_2S_2O_3$ added to each flask.

$[S_2O_3^{2-}] \times \text{volume} = S_2O_3^{2-}\ \text{mole}$

(Calculation process)

(2) Calculate the number of moles of $S_2O_8^{2-}$ in solution.

$[S_2O_8^{2-}] \times$ volume = $S_2O_8^{2-}$ mole

(Calculation process)

(3) Using $\Delta[S_2O_3^{2-}] = 2\Delta[S_2O_8^{2-}]$, calculate the remaining moles of $S_2O_8^{2-}$ in each flask after time Δt, and determine its concentration (M, mol/L).

(Calculation process)

(4) Calculate $\ln[S_2O_8^{2-}]$ and $1/[S_2O_8^{2-}]$ for the five trials.

No.	1	2	3	4	5
$\ln [S_2O_8^{2-}]$					
$1 / [S_2O_8^{2-}]$					

3. Plot $[S_2O_8^{2-}]$, $\ln[S_2O_8^{2-}]$, and $1/[S_2O_8^{2-}]$ (y-axis) versus time Δt (x-axis).

4. Based on the results in (3), determine the reaction with respect to $K_2S_2O_8$:

Reaction order:

Rate constant:

C. Measurement of activation energy

1. Reaction time measurement

No.	1.00 M $NaI_{(aq)}$ (mL)	0.20 M $Na_2S_2O_{3(aq)}$ (mL)	0.15 M $K_2S_2O_{8(aq)}$ (mL)	Δt (sec)	T (^oC)	T (K)	ln (1/Δt)	1/T(K^{-1})
Ice bath	5.0	1.5	5.0					
RT	5.0	1.5	5.0					
Warm bath	5.0	1.5	5.0					

RT: room temperature

2. From the reaction times of Trial 4 at room temperature, in an ice bath, and in a warm water bath, plot ln(1/Δt) (y-axis) versus 1/T (x-axis). The activation energy is obtained from the slope of the least-squares fitted line.

3. Activation energy：_____ (kJ/mol)

D. Catalyst

1. Reaction time measurement

No.	1.00 M NaI	0.20 M $Na_2S_2O_3$	0.15 M $K_2S_2O_8$	0.02 M $CuSO_4$	T (^oC)	t (sec)
*4	5.0 mL	1.5 mL	5.0 mL	2 drops		
*4	5.0 mL	1.5 mL	5.0 mL	0 drop		

2. Based on the experimental results, explain the effect of a catalyst on the reaction rate.

7. Questions

1. If the rate law is determined to be rate = $k[S_2O_8^{2-}][I^-]$, use the table below to compare the initial reaction rates under different experimental conditions, and determine whether this experiment is qualitative or quantitative analysis.

No.	0.20 M NaI (mL)	0.20 M NaCl (mL)	0.005 M $Na_2S_2O_3$ (mL)	2% Starch (mL)	0.10 M K_2SO_4 (mL)	0.10 M $K_2S_2O_8$ (mL)
1	2.00	2.00	1.00	1.00	2.00	2.00
2	2.00	2.00	1.00	1.00	0	4.00
3	4.00	0	1.00	1.00	2.00	2.00

ANS:

2. Based on the amount of $Na_2S_2O_3$ used in Table, which flask will change color the fastest? Why?

No.	2% Starch (mL)	1.00 M NaI (mL)	0.20 M $Na_2S_2O_3$ (mL)	DI water (mL)	0.15 M $K_2S_2O_8$ (mL)
1	1.00	5.00	4.50	2.50	5.00
2	1.00	5.00	3.50	3.50	5.00
3	1.00	5.00	2.50	4.50	5.00
4	1.00	5.00	1.50	5.50	5.00
5	1.00	5.00	0.50	6.50	5.00

ANS:

3. After the iodine bell experiment, adding $Na_2S_2O_3$ solution will become colorless, why? If left for a period of time, will the solution turn reddish-brown again?

   ANS:

4. In iodine bell experiments, why does the rate of chemical reactions usually increase when the temperature rises? Please use Arrhenius equation to illustrate.

Ans：

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