# Kinetics of an Iodine Clock Reaction

Introduction:

In this experiment, you will determine the rate law for a reaction and the effect of concentration on the rate of the reaction by studying the initial reaction rate at several different reactant concentrations. You will also examine the effect of a catalyst on the reaction rate. You will investigate the effect of temperature on the rate of this reaction, which will allow you to calculate the changing rate constants and then the activation energy, E_a.

The primary reaction to be studied is the oxidation of I^- (iodide ion) by $S_2O_8^{2-}$ (persulfate ion) in aqueous solution:

$$2\,I^-(aq) + S_2O_8^{2-}(aq) \rightarrow I_2(aq) + 2\,SO_4^{2-}(aq) \qquad \textbf{Main reaction}$$
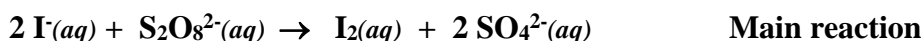
To find the rate of this reaction, you will need to monitor the change in the concentration of a reactant or product with time. We choose the iodine to monitor because it is a colored solution which can be seen. When this reaction is run by itself, a yellow/brown color will slowly appear as iodine is produced. You will do this by using a second reaction, referred to as a "clock" reaction. The clock reaction indicates when a specific amount of I_2 has been produced by the primary reaction.

The "clock" reaction you will use involves the reaction of a very small amount of $S_2O_3^{2-}$ (thiosulfate ion) with the I_2 produced in the primary reaction:

$$I_2(aq) + 2\,S_2O_3^{2-}(aq) \rightarrow 2\,I^-(aq) + S_4O_6^{2-}(aq) \qquad \textbf{Clock reaction}$$
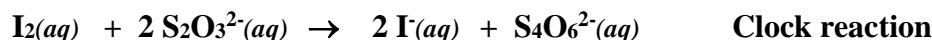
$S_2O_3^{2-}$ is essentially consuming the I_2 formed in the primary reaction. $S_2O_3^{2-}$ reacts very rapidly with I_2, so I_2 is consumed by $S_2O_3^{2-}$ as fast as it is formed. As soon as all of the $S_2O_3^{2-}$ ions have reacted and are used up, the I_2 still being formed in the first equation starts to accumulate. Once **excess** I_2 is present, it can react with the starch (an indicator in this reaction) to turn blue. Thus, "Δt" is calculated as the time elapsed between mixing the reagents (initial time, t_i) and the appearance of the blue color (final time, t_f). You can calculate the diluted concentration of the $S_2O_3^{2-}$ ions in the reaction mixture; that value can be used to calculate $\Delta[I_2]$ by using the stoichiometry of the clock reaction. Since the same amount of $S_2O_3^{2-}$ is added to each run, $\Delta[I_2]$ is also the same for each run. However, the amount of time for the appearance of the blue color varies, so Δt is not constant. Refer to Section 12.1 of Openstax Chemistry to calculate reaction rates.

Effect of Concentration on the Reaction Rate: Solving for orders of reactants

For Runs 1-3, you will vary the initial concentration of I^- while keeping the initial concentration of $S_2O_8^{2-}$ constant. By using the method of initial rates, you will be able to determine the order for I^- in the rate law.

For Runs 2, 4 and 5, you will vary the initial concentration of $S_2O_8^{2-}$ while keeping the initial concentration of I^- constant. By using the method of initial rates, you will also be able to find the order for $S_2O_8^{2-}$ in the rate law.

Once you determine the order of I^- and order of $S_2O_8^{2-}$, you can substitute these back into the rate law and calculate k (the rate constant) for each set of initial concentrations for runs 1 - 5. You will then determine the average value of k for the five room temperature runs.

Refer to Section 12.3 of Openstax Chemistry for calculating orders of reacants and rate laws.

Effect of a Catalyst on the Reaction Rate

A comparison of the reaction rate with and without a catalyst (run 6) will demonstrate catalytic action. Cu^{2+} ions, added in the form of a dilute $Cu(NO_3)_2$ solution, makes a suitable catalyst.

Effect of Temperature on the Reaction Rate: Calculation of E_a

The Arrhenius equation, $\ln k = -\dfrac{E_a}{R}\left(\dfrac{1}{T}\right) + \ln A$, describes the relationship between temperature and rate constant. You will run the reaction at four different temperatures, calculate the rate constant at each temperature, and use an Arrhenius graph of (1/T on the x-axis) versus (ln k on the y-axis) to determine the value of the activation energy, E_a.

Refer to Section 12.5 of Openstax Chemistry on using the Arrhenius equation to plot data and calculate Activation Energy (E_a).

Equations to use for the calculations:

Dilution: $M_1V_1 = M_2V_2$

Rate of reaction: $\dfrac{-D[I_2]}{Dt} = \dfrac{-D[S_2O_3^{2-}]}{2Dt}$

Rate law: rate $= k[I^-]^x [S_2O_8^{2-}]^y$ x = order of I^-, y = order of $S_2O_8^{2-}$, k = rate constant with units

Solving for orders of reactants using ratio of runs: $\dfrac{Rate_2}{Rate_1} = \dfrac{[I^-]_2^x[S_2O_8^{2-}]_2^y}{[I^-]_1^x[S_2O_8^{2-}]_1^y}$

- Runs 1, 2, and 3 will be used to solve for x (the order of I^-)
- Runs 2, 4, and 5 will be used to solve for y (the order of $S_2O_8^{2-}$)

Arrhenius equation to solve for Activation Energy: $\ln k = -\dfrac{E_a}{R}\left(\dfrac{1}{T}\right) + \ln A$, where R = 8.314 J/mol•K

Materials:

3 – 10 mL beakers	0.2% starch
250 mL beaker for waste	0.012 M $Na_2S_2O_3$
Clean, dry test tubes	0.20 M KI
Magnetic stir bar	0.20 M KNO_3
Forceps	0.20 M $(NH_4)_2S_2O_8$
Stir/hot plate	0.20 M $(NH_4)_2SO_4$
DI wash bottle	

Procedure:

Effect of Concentration on the Rate of Reaction

1. You will carry out five runs three times each to give a good average time per run. Be sure to collect three times that are close to each other. More than three trials might be needed. To keep the ionic strength and volume of all runs relatively constant, the non-reactive compounds KNO_3 and $(NH_4)_2SO_4$ are added to replace the reactants KI and $(NH_4)_2S_2O_8$ when less than 20 drops of either reactant is used.

Waste handling: Keep a large beaker at your bench to collect the reaction waste. When you have completed all trials, pour the contents of this beaker into the waste container in the fume hood.

Table I gives the composition in **drops** of the reaction mixtures that will be used to determine the order of reaction with respect to the iodide and persulfate ions.

Table I. Compostion of the Reaction Mixtures

Run No.	0.2% starch	0.012 M $Na_2S_2O_3$	0.20 M KI	0.20 M KNO_3	0.20 M $(NH_4)_2S_2O_8$	0.20 M $(NH_4)_2SO_4$
1	3	5	20	0	10	10
2	3	5	10	10	10	10
3	3	5	5	15	10	10
4	3	5	10	10	20	0
5	3	5	10	10	5	15
10 mL Beaker				**Test Tube**		

2. Use the dropper bottles containing starch, $Na_2S_2O_3$, KI, and KNO_3 to add the appropriate number of drops for Run 1 into one of the 10 mL beakers. Repeat this process with the other 10 mL beakers to prepare the other two trials for Run 1.

   Note: Make sure you use solutions from the same set of dropper bottles *and hold the bottles at the same angle* to obtain drops of consistent size!

3. Record the temperature of the mixture in one of these beakers. This will be room temperature and this temperature will remain relatively constant for Runs 1-5.

4. Obtain a stir plate from one of the benchs near the fumehoods. Add a magnetic stirring bar to one of the 10 mL beakers, place the beaker on the magnetic stirrer, and adjust the speed of the stirrer to obtain a slow but steady speed.

5. Use the dropper bottles containing $(NH_4)_2S_2O_8$ and $(NH_4)_2SO_4$ to combine the appropriate number of drops for Run 1 in a small clean, dry test tube. You can use the same test tube for all trials of runs 1-3 because the contents are the same. But you will need new clean dry test tubes for runs 4 and 5.

6. Pour the test tube solution into the 10 mL beaker as rapidly as possible, starting the timer as soon as the contents have been added. Stop the timer when the solution turns blue, and record the time, in seconds, in Data Table 2. Use forceps to remove the stir bar from the beaker to keep your fingers from getting blue. Rinse and dry the stir bar before adding it to the next beaker.

7. Repeat this process for the other two Run 1 trials, *so you will have three reaction times for Run 1*. Rinse the beakers and the test tubes with tap water and then DI water. Dry the beakers, but DO NOT attempt to dry the test tubes. Repeat steps 2 – 7 for runs 2-5.

Effect of a Catalyst

8. Repeat Run 3 (at room temperature) **one more time**, adding one drop of 0.020 M $Cu(NO_3)_2$ to the mixture in the **test tube**. Since you only need to calculate the rate of reaction (and not the rate constant), one trial should be sufficient. This will be Run 6.

Effect of Temperature and the Activation Energy

9. You have reaction times for Run 3 at room temperature, and need reaction times for Run 3 **at three other temperatures**, one at least 10-15°C colder (run 7) than room temperature, one about 10-15°C warmer (run 8) than room temperature, and another about 20-30°C warmer (run 9) than room temperature. **Make sure to do the cold temperature run first and only perform one trial at each temperature.**

The general procedure is to prepare the appropriate solutions in the beaker and the test tube, then place both the beaker and the test tube in a water bath (larger beaker) containing either ice-water or warm water, depending on the desired temperature. Put the thermometer in the test tube. Allow the solutions to remain in the water bath until the correct temperature has been reached, then combine the mixtures and time the reaction as before. Make sure to record the temperatures.

Waste: Dispose of the waste in the waste container in the fume hood.

Clean-Up: Rinse all glassware with tap water and then a final rinse with DI water. Wipe your entire bench with a damp paper towel. Put all equipment back where you found it.

Calculations: Show one example from **run 1** (unless otherwise indicated) of each calculation below in your lab report. Be sure to label each calculation and show 1) the formula or equation used, 2) the values used in the calculation, and 3) the final answer with units (if applicable).

1) Average time
2) Diluted concentration of I^-
3) Diluted concentration of $S_2O_8^{2-}$
4) Diluted concentration of $S_2O_3^{2-}$

5) Rate of reaction $= \dfrac{-\Delta[I_2]}{\Delta t} = \dfrac{-\Delta[S_2O_3^{2-}]}{2\Delta t}$

6) Order of I^- (use runs 1,2; 1,3; 2,3; show one sample calculation, report the unrounded value of each, average your three values, and then round your average to 0, 1, or 2)
7) Order of $S_2O_8^{2-}$ (use runs 2,4; 2,5; 4,5; show one sample calculation, show the unrounded value of each, average your three values, and then round your average to 0, 1, or 2)
8) Value of k from the rate law
9) Average of k values for runs 1-5 (room temperature runs)
10) **Graph 1**: Arrhenius Equation plot:
*   Use Results Table 4 to plot the Arrhenius graph needed to calculate the Activation Energy, E_a.
    *   1/T (x-axis) versus ln k (y-axis)
11) Calculate the activation energy from your graph of (1/T) vs (ln k).
    *   This can be shown on the page with the graph or immediately after the graph.
    *   Use the linear form of the Arrhenius equation to calculate E_a: $\ln k = -\dfrac{E_a}{R}\left(\dfrac{1}{T}\right) + \ln A$, where R = 8.314 J/mol•K

12) Report your final rate law: write the rate law for your reaction including the average k value (runs 1-5) with units and orders for I^- and $S_2O_8^{2-}$.

Kinetics of an Iodine Clock Reaction Pre-Lab Questions and Calculations

You will complete this quiz in Canvas 1 hour before your lab period. This page will not be turned in or graded. You may use this page to set up your calculations before you take the Canvas quiz.

1. Which substance(s) is/are used in the experiment to keep ion concentrations and volumes constant but do not appear in the equations?

2. What ion must be used up (limiting reactant) in the clock reaction to cause the solution to turn blue?

3. If 5 drops of 0.15 M KI are added to 40 drops of $Na_2S_2O_3$, what is the final concentration of KI?

4. Which chemical acts as a catalyst in this experiment?

5. What is the **minimum** recommended temperature for the cold temperature run (#7)?

6. What is the **maximum** recommended temperature for the hottest temperature run (#9)?

7. What variables/factors during the experiment will affect your results?

8. What are the safety concerns associated with this experiment?

9. What should be included in your clean-up protocol at the end of the experiment?

Name: _____   Partners: _____

Kinetics of an Iodine Clock Reaction Lab Report: pages 6 – 8, Calculations, Graph, Conclusion

Data and Results

Table 1: Drops Table of solutions for each run.

Run No.	0.2% starch	0.012 M $Na_2S_2O_3$	0.20 M KI	0.20 M KNO_3	0.20 M $(NH_4)_2S_2O_8$	0.20 M $(NH_4)_2SO_4$
1	3	5	20	0	10	10
2	3	5	10	10	10	10
3	3	5	5	15	10	10
4	3	5	10	10	20	0
5	3	5	10	10	5	15
10 mL Beaker				Test Tube		

Table 2 (3 pts): Times, temperatures for runs 1 – 9.

Run	Temperature (ºC)	Trial 1, s	Trial 2, s	Trial 3, s	Average time, s
1					
2					
3					
4					
5					
6					
7					
8					
9					

Table 3 (20 pts): Average Times, Reaction Rates, Diluted Concentrations, and rate constants. **Include the units for k.**

Run	Average time (s)	Rate of Reaction (M/s)	[I$^-$] (M)	[$S_2O_8^{2-}$] (M)	T (ºC)	k (_____)
1						
2						
3						
4						
5						
6						
7						
8						
9						

Table 4 (6 pts): Temperatures, rate constants, inverse temperature, natural log of k (ln k) – **Include the units for k and 1/T**

Run	T (K)	k, _____	1/T, _____	ln k
3				
7				
8				
9				

Calculations (25 pts): In the space below, show a sample of each calculation. For each one, show the equation used, values used, and final answer with units, if applicable.

1. Average time

2. Diluted concentration of I^-

3. Diluted concentration of $S_2O_8^{2-}$

4. Diluted concentration of $S_2O_3^{2-}$

5. Rate of reaction

6. Order of I^- (use runs 1,2; 1,3; 2,3; show one sample calculation, report the unrounded value of each, average your three values, and then round your average to 0, 1, or 2)

7. Order of $S_2O_8{}^{2-}$ (use runs 2,4; 2,5; 4,5; show one sample calculation, show the unrounded value of each, average your three values, and then round your average to 0, 1, or 2)

8. Rate constant, k

9. Average of k values for runs 1-5 (room temperature runs)

10. **Graph (5 pts):** Attach your Arrhenius Graph with title and labeled axes.

11. Activation Energy, E_a

12. Report your final rate law: write the rate law for your reaction including the average k value with units (runs 1-5) and orders for I^- and $S_2O_8{}^{2-}$.

Conclusion (5 pts): Write your conclusion on a separate piece of paper. You should state your final rate law (with values for orders x and y, and average k for runs 1 - 5) and activation energy, E_a, value. **Summarize** the effects of concentration, temperature, and catalyst on the rate of reaction.

Post-Lab Questions – These questions will not be graded as part of your lab report grade. You will be responsible for the information in these questions and able to answer these or similar questions on the post-lab quiz at the start of next week's lab period.

1. Explain how and why the reaction rate changes as concentrations of the reactants change.

2. Explain the general calculation procedure used to solve for reactant orders in the rate law. How did you determine which runs to use to solve for the order of each reactant?

3. Explain the general calculation steps used to calculate the activation energy. What data and results are used and how are they used?

4. Compare your average reaction time for Run 3 at room temperature to your reaction times at the colder and hotter temperatures. What effect does changing the temperature have on the rate of reaction? (What happens at cold temperatures? What happens at hot temperatures?) Explain WHY temperature has this affect for both cold and hot temperatures.

5. Compare your average reaction time for Run 3 without $Cu(NO_3)_2$ to your reaction time with $Cu(NO_3)_2$. How did the addition of $Cu(NO_3)_2$ affect the rate of reaction? WHY did $Cu(NO_3)_2$ have this affect? What did $Cu(NO_3)_2$ do that changed the reaction time?

6. Discuss at least 2 sources of error – how they affected your results and how you would correct them if you were to repeat the experiment.