

# Protocol | Kinetics of the Iodine Clock Reaction

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## Actions

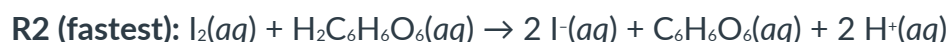
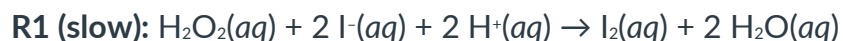
### Introduction

Watch the [conceptual introduction video Links to an external site.](#) for this experiment. Taking medication, driving a car, and washing laundry are three examples of chemistry in everyday life where we very much care about how—and more specifically, *how quickly*—chemical reactions occur. If we'd like to use these reactions on particular time scales, we need to study their speeds. The study of reaction speed broadly defines the field of [chemical kinetics Links to an external site.](#)

In studying kinetics, we are interested in the dependence of the [reaction rate Links to an external site.](#) on the concentrations of the reactants and on temperature. Each dependence can be concisely expressed in an equation: the [differential rate law Links to an external site.](#) expresses the dependence of the rate on reactant concentrations and the [Arrhenius equation Links to an external site.](#) shows how the rate constant  $k$  depends on temperature. Integration of the differential rate law with respect to time gives the [integrated rate law Links to an external site.](#), an equation that shows explicitly how the concentration of a reactant varies with time. For a reaction involving a single reactant A, the form of the integrated rate law depends on the kinetic order (or order of reaction) of A. The table below lists differential and integrated rate laws for zero-, first-, and second-order reactions of A. For each order, some function of  $[A]$  varies linearly with time; these are listed in the last column of the table.

Order	Differential Rate Law	Integrated Rate Law
0	$\text{rate} = k$	$[A] = [A]_0 - kt$
1	$\text{rate} = k[A]$	$[A] = [A]_0 e^{-kt}$
2	$\text{Rate} = k[A]^2$	$1/[A] = 1/[A]_0 + kt$

In this experiment, we will measure the kinetic orders of the reactants in a famous and fascinating reaction: the [iodine clock Links to an external site.](#). This reaction involves three related processes with different rates. A period of apparent inactivity is followed by a rapid and dramatic color change. Hydrogen peroxide and iodide ion are the rate-determining reactants in this reaction; ascorbic acid is used as a rapidly reacting “timer” and starch serves as a visual indicator.



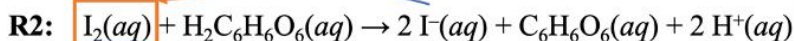
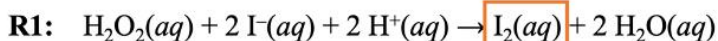
**Differential rate law for R1:**

$$\text{rate} = k[\text{I}^-]^x [\text{H}_2\text{O}_2]^y$$

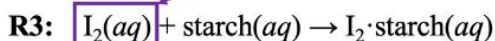
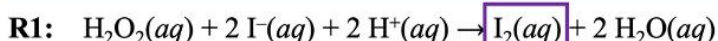
To perform the reaction, an aqueous solution of excess hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and starch is mixed with an aqueous solution of potassium iodide (KI) and ascorbic acid ( $\text{H}_2\text{C}_6\text{H}_6\text{O}_6$ ). Notice that iodine ( $\text{I}_2$ ) is *not* added into the initial reaction mixture. This means that in order for **R2** and **R3** to begin, they have to wait for **R1** to produce its products. Thus, at the very beginning of this reaction series, only **R1** will be producing products while ascorbic acid and starch wait patiently for their reaction partner (iodine,  $\text{I}_2$ ) to be made.

As **R1** produces iodine, iodine has the potential to react with ascorbic acid (**R2**) or react with starch to produce an iodine-starch complex that will turn the solution dark blue (**R3**). The rate of reaction **R2** is much higher than the rate of reaction **R3**. This means that when iodine is produced, ascorbic acid will immediately consume it before starch has a chance to complex with it. Starch will only have the opportunity to react with iodine (and turn the solution dark blue) once all of the ascorbic acid has been consumed by **R2**. Notice also that **R2** produces two iodide ions from iodine: this iodide can then be used as a reactant in **R1** to reform iodine (see the blue line below), as the reactions are happening simultaneously.

## While $\text{H}_2\text{C}_6\text{H}_6\text{O}_6$ Remains



## When $[\text{H}_2\text{C}_6\text{H}_6\text{O}_6] = 0$



Although **R2** is faster than **R3**, both reactions are very rapid in absolute terms. As a result, the reaction solution will darken at the moment all of the ascorbic acid has been consumed. In other words, darkening of the reaction solution serves as a signal that **R2** is complete and the concentration of ascorbic acid is equal to zero. By setting up the reaction with a known initial concentration of ascorbic acid and measuring the time it takes for the solution to darken, we can thus calculate an average rate for the consumption of ascorbic acid:

$$\text{rate} = \frac{[\text{H}_2\text{C}_6\text{H}_6\text{O}_6]_i - 0}{t}$$

Applying stoichiometry then enables us to calculate the rates of consumption or production of the other reactants and products, respectively. We will make use of different runs of the iodine clock reaction with different initial concentrations of iodide and hydrogen peroxide to determine the kinetic orders of these species (x and y in the differential rate law for **R1**). Additionally, using the temperature dependence of the average rates, we will measure the [activation energy](#) [Links to an external site.](#) of the reaction.

The table below lists the volumes of each reagent used in each trial of the reaction. Note that you will measure only one of trials 4 – 6 and obtain data for the other two high-temperature trials from other groups.

		Solution B		Solution A	
Trial	Volume H <sub>2</sub> O (mL)	Volume 0.0050 M H <sub>2</sub> C <sub>6</sub> H <sub>6</sub> O <sub>6</sub> (mL)	Volume 1.0 M KI (mL)	Volume 3% H <sub>2</sub> O <sub>2</sub> + starch (mL)	Temperature (°C)
1	—	30.0	5.0	10.0	~20
2	5.0	30.0	5.0	5.0	~20
3	2.5	30.0	2.5	10.0	~20
4	—	30.0	5.0	10.0	~40
5	—	30.0	5.0	10.0	~50
6	—	30.0	5.0	10.0	~60

For best results, measure volume and time as carefully as you can.

## Safety and Materials

The following reagents will be available:

- [Ascorbic acid \(H<sub>2</sub>C<sub>6</sub>H<sub>6</sub>O<sub>6</sub>\) solid](#)[Links to an external site.](#)
- [Potassium iodide \(KI\) solid](#)[Links to an external site.](#)
- [Hydrogen peroxide \(H<sub>2</sub>O<sub>2</sub>\), 3% w/w solution](#)[Links to an external site.](#)(0.882 M)
- [Starch indicator solution](#)[Links to an external site.](#)

Refer to the linked Safety Data Sheets above for important safety information on these materials.

## Relevant Experimental Techniques

- [Pipetting Liquids](#)
- [Preparing Solutions](#)

## Research Questions

1. What is the kinetic order of iodide in the oxidation step?
2. What is the kinetic order of hydrogen peroxide in the oxidation step?
3. What is the activation energy of the oxidation of iodide reaction?

## Procedures

### A. Preparation of Solutions

1. Obtain about 50 mL of **3% hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) solution** in a 100 mL beaker by pouring from the reagent bottle. Add about 8 drops of **starch solution** to the beaker and label it "Solution A."
2. Use volumetric technique to prepare 250.0 mL of a 0.00500 M ascorbic acid solution starting from **ascorbic acid ( $\text{H}_2\text{C}_6\text{H}_6\text{O}_6$ ) solid**. Label the holding flask "Ascorbic Acid."
3. Use volumetric technique to prepare 50.00 mL of a 1.00 M potassium iodide solution starting from **potassium iodide (KI) solid**. Label the holding flask "KI."
4. Label an empty 50 mL beaker "Solution B." This beaker will be used to mix the ascorbic acid and potassium iodide solutions prior to mixing with solution A.
5. Obtain about 50 mL of **deionized water** in a 100 mL beaker. Label the beaker "DI  $\text{H}_2\text{O}$ ."
6. Dedicate one pipet for each labeled beaker, including deionized water. Serological or volumetric pipers may be used; note that volumetric pipets can only dispense a single exact volume. Carefully keep track of your pipets throughout the experiment to avoid contamination: each pipet should contain only one solution.

### B. Trial 1: Baseline Concentrations of Iodide and Hydrogen Peroxide

1. Measure and record the room temperature to the nearest 0.1 °C. This will be used as the reaction temperature for trials 1 – 3.
2. Label an empty beaker with a capacity of at least 100 mL with "Reaction."
3. Pipet 10.0 mL of solution A into the Reaction beaker.
4. In the Solution B beaker, combine 30.0 mL of ascorbic acid solution and 5.00 mL of potassium iodide solution.
5. Prepare a timer using a mobile device or the timer provided. The reaction will begin in the next step and you should be ready to start the timer when solutions A and B are mixed!

6. Add the contents of the Solution B beaker to the Reaction beaker and start the timer. Stop the timer at the moment you observe a color change and record the reaction time to the nearest 0.1 sec. If you don't observe a color change after 1 min, complete the next step and then return to step 1.
7. In the sink, rinse and carefully dry the Solution B and Reaction beakers.

### C. Trial 2: Half Concentration of Hydrogen Peroxide

1. Pipet 5.0 mL of solution A into the now clean and dry Reaction beaker.
2. Pipet 5.0 mL of deionized water into the Reaction beaker.
3. In the Solution B beaker, combine 30.0 mL of ascorbic acid solution and 5.00 mL of potassium iodide solution.
4. Prepare a timer using a mobile device or the timer provided. The reaction will begin in the next step and you should be ready to start the timer when solutions A and B are mixed!
5. Add the contents of the Solution B beaker to the Reaction beaker and start the timer. Stop the timer at the moment you observe a color change and record the reaction time to the nearest 0.1 sec. If you don't observe a color change after 2 mins, complete the next step and then return to step 1.
6. In the sink, rinse and carefully dry the Solution B and Reaction beakers.

### D. Trial 3: Half Concentration of Iodide

1. Pipet 10.0 mL of solution A into the Reaction beaker.
2. In the Solution B beaker, combine 30.0 mL of ascorbic acid solution, 2.5 mL of potassium iodide solution, and 2.5 mL of deionized water.
3. Prepare a timer using a mobile device or the timer provided. The reaction will begin in the next step and you should be ready to start the timer when solutions A and B are mixed!
4. Add the contents of the Solution B beaker to the Reaction beaker and start the timer. Stop the timer at the moment you observe a color change and record the reaction time to the nearest 0.1 sec. If you don't observe a color change after 1 min, complete the next step and then return to step 1.
5. In the sink, rinse and carefully dry the Solution B and Reaction beakers.

**Concept Check.** In your lab notebook, prepare a graph that shows the concentration of ascorbic acid ( $\text{H}_2\text{C}_6\text{H}_6\text{O}_6$ ) as a function of time for trials 1 – 3. The graph should include three lines (one for each trial); label each line with its corresponding trial. Assume that  $[\text{H}_2\text{C}_6\text{H}_6\text{O}_6]$  decreases linearly with time in all three trials. Label the initial concentration of ascorbic acid (carefully consider dilution!) and the time points where the three lines

cross the x-axis. For one of the three trials, calculate the average rate of consumption of ascorbic acid.

### E. Baseline Runs at Elevated Temperatures

1. Your teaching assistant will assign you a reaction temperature between 40 and 60 °C. Record this temperature in your lab notebook.
2. Prepare the Reaction and Solution B beakers in the same manner as Trial 1 (see part B).
3. Set up a hot water bath using a hot plate and 400 or 600 mL beaker. After Partner II has set up the solution beakers, transfer their contents *separately* into test tubes and heat the contents to your assigned temperature. Use a thermometer to track the temperature of the contents of one of the test tubes. Clean and dry the Reaction beaker to prepare it to receive the heated solutions.
4. Prepare a timer and record the exact temperature of the water bath to the nearest 0.1 °C just before you mix the solutions.
5. Pour the contents of all of the test tubes simultaneously into the Reaction beaker and start the timer.
6. Record the time elapsed when the reaction changes color to the nearest 0.1 sec.
7. Add your exact temperature and reaction time to a table on the board. Record temperatures and times from the board for approximately 40, 50, and 60 °C (whichever temperatures you did not measure yourself). If multiple measurements at each temperature are available, just choose one for each approximate temperature.

### F. Cleanup

1. Dispose of any remaining solutions down the drain with copious water. Wash your glassware with soap and water, making sure to do a final water rinse, and return it to the appropriate locations in your lab drawers.

## Group Argumentation

At your bench, combine findings with the other students. Revisit the research questions for this experiment. Discuss how your data can be used to address the research questions and conceptually examine your results before calculating anything. Develop a claim for each research question. A claim answers the research question in the form of a conclusion you have drawn from your experiment. Use the white boards to generate a

full argument for one of the claims. An argument incorporates 1) the claim, 2) evidence drawn from your experimental results (generally actual values and/or plots are useful here), and 3) reasoning that explains your thinking associated with the evidence and how it supports the claim as well as any background information you used in drawing that conclusion (this is the most complex and lengthy portion). The template below can be used for developing your group argument. After you have developed your argument, discuss with another group to give each other feedback on the argument and make suggestions. Revise the argument based on the feedback and record it in your lab notebook before leaving.

Claim
Evidence
Reasoning

## Post-lab Calculations and Data Workup

Add the reaction times and temperatures from your lab notebook to Table 0 of the [data workup spreadsheet](#) [Download data workup spreadsheet](#). For Table 1, calculate the initial concentrations of iodide and hydrogen peroxide and the average rate of consumption of ascorbic acid (expressed as a positive value) in each trial of the reaction. In addition, list the temperature of each run in units of Kelvin. *Recommended:* develop Excel formulas to perform these calculations for trial 1 using cell references so that you can easily copy and paste them for the other trials. This is much more efficient than performing each calculation by hand!

Look for pairs of runs in Table 1 across which the value of only one initial concentration changes. Use one of these pairs to determine the order of iodide by examining how a change in  $[I^-]_i$  affects the average rate of consumption of ascorbic acid. Likewise, use



another one of these pairs to determine the order of hydrogen peroxide by examining how a change in  $[\text{H}_2\text{O}_2]_i$  affects the average rate of consumption of ascorbic acid. Write the differential rate law for the iodine clock reaction based on your results.

Find four trials of the reaction for which temperature is the only variable that changes. Applying the kinetic orders of iodide and hydrogen peroxide that you just determined, calculate  $k$  for each trial (use the average rate of consumption of ascorbic acid as the "rate" in the differential rate law). Use Table 2 to prepare a plot (Figure 1) with the inverse of the absolute temperature in Kelvin on the x-axis and the natural logarithm of  $k$  on the y-axis. Add a line of best fit with equation and  $R^2$  value and use the line of best fit to determine the activation energy of the reaction in kilojoules per mole. Consider the Arrhenius equation, which this graph models.