Reaction Kinetics:
Effects of Concentration & Temperature on an Oscillating Chemical Reaction

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Background Information:
This experiment is modeled after a well-studied reaction, known as the Cerium-catalyzed bromate-malonic acid oxidation, which is more classically called the Belousov-Zhabotinsky (B-Z) Reaction. This reaction is an oscillating reaction that will change colors in regular intervals. While the name sounds complicated, the chemistry principles governing the reaction are the same as many other reactions. The advantage of this reaction is that the rate of the reaction can be easily observed by clocking the amount of time it takes for the reaction to complete a cycle. This reaction will therefore be used to demonstrate the influences of the chemical concentrations and temperature on the rate of reaction. There are two parts to this laboratory. The first part will test the effects of concentration of the reactants on the reaction rate while the second part will test the effects of temperature on the reaction rate.

In the first part, students will observe how concentration affects the reaction rate or the speed at which a reaction occurs. Students will add twice the normal amount of one of the ingredients (extra KBrO₃) into the combined mixture of all four solutions. Students will observe that adding ingredients will increase the reaction rate. In the second part of the laboratory, students will experiment with how temperature affects the reaction rate. Specifically, students will heat their solutions to witness that an increase in temperature also increases or speeds up the oscillating cycle. Students will then compare their observed results to those predicted by the Arrhenius Equation.

Why is this lab important? Students will learn that the rate of a chemical reaction is influenced by the concentrations of the reactants and the temperature of the reaction. Furthermore, the changes in the rate of the reaction are predictable based on reaction kinetics theory.
Reaction Kinetics Theory

Influence of chemical concentrations:

The first part of the laboratory will demonstrate that the rate of reaction is dependent on the concentrations of the reactants. For reactions between two chemicals, the rate of the reaction is given by:

\[ \frac{d[A]}{dt} = k[A][B] \]

where

- \( d[A]/dt \) is the change in concentration during a time interval (in M/s)
- \( k \) is the second order reaction rate constant (in units of 1/(M×s))
- \( [A] \) is the concentration of reactant A (in units of M)
- \( [B] \) is the concentration of reactant B (in units of M)

A quick inspection of this equation will reveal that the rate of reaction will increase if the concentration of either A or B is increased. Therefore, the reaction will occur faster at higher concentrations of the reactants. This makes intuitive sense since more molecules present will increase the likelihood of two molecules colliding and reacting. The rate constant \( k \) is independent of the concentration of the reactant.

The reaction order is the sum of the exponents in the reaction rate equation. Often they are the same as the reaction stoichiometry coefficients, but sometimes they are not. You need to determine the reaction order for each of the reactants individually. This involves running a series of experiments and changing the concentration of one of the reactants and observing the reaction rate. For example, if we have the generic reaction of

\[ A + B = \text{products} \]

Then the reaction rate equation equals:

\[ \frac{d[A]}{dt} = k[A][B] \]

Once you have determined the reaction rate for the control (unmodified) conditions, then you double the concentration of reactant A and measure the reaction rate and compare it to the expected results:

- If the reaction rate does not change, then the reaction is zero order with respect to [A]; \( \text{rate} = k[2A][B] \)
- If the reaction rate doubles, then the reaction is first order with respect to [A]; \( \text{rate} = k[2A]^1[B] \)
- If the reaction rate quadruples, then the reaction is second order with respect to [A]; \( \text{rate} = k[2A]^2[B] \)

This gives you the reaction order with respect to [A]. You conduct a similar experiment to determine the reaction order with respect to [B]. The (total) reaction order is the sum of the reaction orders of the two reactants. For example, if a reaction is first order with respect to [A] and first order with respect to [B], then the reaction order is second order.

The BZ reaction conducted in this laboratory is not a simple second order reaction, but the reaction kinetics is similar to a second order reaction. For the calculations in this laboratory, you can treat the reaction constant of \( k \) as 1/(cycle time in seconds). In the first part of the laboratory, you will double the concentration of one of the reactants and observe the change in the rate of reaction. You will then use the observed reaction rate change to determine the reaction order with respect to that reactant.
Temperature dependence of reaction rates:

The second parameter that will be investigated is how temperature affects the rate of reaction. Fundamentally, molecules have greater kinetic energies at higher temperatures, thus there is more energy in each collision between molecules that can be used to conduct a reaction. The influence of the temperature on the rate of reaction is given by the Arrhenius Equation:

\[ k = A e^{(-E_a/RT)} \]

where
- \( k \) is the reaction rate constant (1/s)
- \( A \) is a collision frequency factor (1/s)
- \( E_a \) is the activation energy of the reaction (J/mol)
- \( R \) is the gas constant in units of 8.314 J/mol·K
- \( T \) is the temperature (in K), so don’t forget to convert \( T \) into Kelvin.

For the purposes of this experiment, the reaction constant will be treated as \( 1/(\text{cycle time}) \). This is not the normal formulation for the reaction rate constant but this reaction is so different from “normal” reactions. Therefore, use this formulation of \( k \) for this lab ONLY. The Energy of Activation for this reaction is 65,000 joules/mole. The collision frequency factor is a constant at all temperatures. Therefore, the new equation for this reaction is:

\[ 1/(\text{cycle time}) = A e^{(-65,000/8.314\cdot T)} \]

The most common approach to determine the rate of reaction at different temperatures is to measure the reaction rate (and hence \( k \)) at one temperature. The measured value of \( k \) combined with the activation energy, temperature, and gas constant are used to calculate the value of the collision frequency factor (\( A \)), which is constant at all temperatures for a particular reaction. The calculated value of \( A \) is then used with the activation energy, gas constant, and new temperature to calculate the reaction rate constant at the new temperature. For this laboratory experiment, the reaction rate will be measured at room temperature. From this measured reaction rate, you will calculate the collision frequency factor for this reaction. You will then predict the rate of the reaction at elevated temperatures. You will heat up the reaction and measure the increased rate of reaction. Lastly, you will compare your predicted rate of reaction to your observed rate of reaction at 40 °C.

An example of the calculation is given below using fictitious data. If the beginning reaction temperature was 28.6 °C (\( = 301.6 \) K) and the average cycle time of the first three cycles was 30 s, then our equation is

\[ 1/(30 \text{ s}) = A e^{(-65,000/(8.314\cdot 301.6))} \]

and we solve for \( A \):

\[ A = 6.04\times10^9 \text{ 1/s} \]

With the value of \( A \) known, the theoretical cycle time for the last cycle can be calculated for any temperature. We simply covert the new reaction into Kelvin and put it back into the equation.
with the value of A and solve for the new cycle time. Let’s calculate the cycle time of the reaction at 50 ºC (323 K), which is about the final temperature in your experiment. The equation is now:

\[
\frac{1}{(\text{cycle time})} = (6.04 \times 10^9) e^{(-65,000/(8.314 \times 323))}
\]

and we solve for cycle time to get:

\[
\text{cycle time} = 5.4 \text{ s}
\]

You will then compare this predicted value to your observed value. The above example used fictitious data, so do not expect to obtain the same value as above.

**Laboratory Hazards:**

Be careful when working with the hot plate, it will remain hot for a long period of time. Always wear your goggles and gloves while working with chemicals, especially for the following chemicals since some are corrosive and strong oxidizers that can cause burns. To help prevent inhalation of any of the following chemicals, your instructor will be turning on the exhaust vents for each station to draw away any fumes given off by the chemicals.

The chemical solutions in the experiment are as follows:

**Solution A** – 0.23M KBrO₃

Bromates (such as KBrO₃) are powerful oxidizing chemicals. (Remember, an oxidizer is a chemical that readily transfers oxygen atoms or will readily gain electrons in a chemical reaction.) In addition, potassium bromate (KBrO₃) ingestion may also cause gastrointestinal problems. KBrO₃ may also be a possible carcinogen (may cause cancer) and is a skin, eye, and respiratory irritant, so be careful!

**Solution B** – 0.31M malonic acid and 0.059M KBr

Malonic acid easily irritates mucous membranes, such as eyes and skin. The KBr in Solution B is not highly concentrated, yet high amounts of potassium bromide (KBr) are harmful if ingested, inhaled or repeatedly brought in contact with skin.

**Solution C** – 0.019M Ce(NH₄)₂(NO₃)₆ and **2.7M H₂SO₄**

Sulfuric acid is a strong acid and can cause burns at the concentration used in this experiment. Report any spills to your laboratory instructor. Cerium ammonium nitrate [Ce(NH₄)₂(NO₃)₆] is a strong oxidizer. It is also a skin, eye, and respiratory irritant.

**Solution D** (dark red) – ferroin or [Fe(o-phen)₃]SO₄

Ferroin is harmful if swallowed, inhaled, or absorbed through the skin. It is also a respiratory, eye, and skin irritant.

**Disposal**

Make sure to dispose of all chemicals in the HAZMAT (hazardous materials) container in the fume hood at the back of the lab room. No solutions may be disposed of down the drain. This is illegal and damages our rivers.
**Materials:**
You will need two sets of these previously prepared solutions:
- 15 mL Solution A (clear) – 0.23M KBrO₃
- 15 mL Solution B (clear) – 0.31M malonic acid and 0.059M KBr
- 15 mL Solution C (yellow) – 0.019M Ce(NH₄)₂(NO₃)₆ and 2.7M H₂SO₄
- 1.44 mL Solution D (dark red) – ferroin or [Fe(o-phen)₃]SO₄

1 hot plate (w/digital display)
1 150ml beaker
1 100ml beaker
1-inch magnetic stir bar
Additional KBrO₃ powder

Stop watch
Thermometer
Scale (on the side counter)
Tare boats

**Procedures:**

**Skip the grayed procedure for the online labs**

Read through all of these procedures as well as hazards before conducting the experiment.

Make sure that the two beakers and thermometer are clean. Rinse with DI water before beginning the experiment. DO NOT use TAP WATER to clean the glassware as this will contaminate the reaction.

Put on protective equipment.
- Goggles
- Gloves
- Lab coat (recommended)

Prepare experimental apparatus:
- Set up the hot plate – do NOT turn on the hot plate yet.
- Place the stir bar in the 150ml beaker on the hot plate and check that the stir bar works by turning the stir bar to a slow-medium speed.
- Test your thermometer, making sure it will measure in °C.
- Test your stop watch. Briefly practice using the START button and the LAP button. Some of the measured time intervals are rather fast, so it is best to be familiar with the stop watch before you start the experiment.

In each part of the laboratory, students will be adding together four solutions as follows: Solutions A and B will be added together first in a 150 mL beaker. Solution A is a 0.23M KBrO₃ mixture and Solution B is a 0.31M malonic acid and 0.059M KBr mixture. Solutions C and D will be added together in a 100 mL beaker and then immediately added into the large beaker. Solution C is a 0.019M Ce(NH₄)₂(NO₃)₆ and 2.7M H₂SO₄ mixture. Solution D is a ferroin solution, with the chemical formula [Fe(o-phen)₃]SO₄. In both parts of the experiment, the mixture of all four solutions will begin to change colors. These color changes will repeat (or oscillate) in a predictable pattern (or cycle). The amount of time the mixture of solutions takes to fully complete one of these cycles will be used as the reaction rate.
Part One – Effects of concentrations on the rate of reaction
1) Obtain 0.575 g of KBrO₃ powder before you start the reaction
   a) Place the tare boat on the scale (on the side counter).
   b) Press the TARE button to zero the scale
   c) Measure out 0.575 g of KBrO₃. Try to get within ± 0.01 g
      i) Be sure you do not pour KBrO₃ back into the container (this would contaminate the rest
         of the KBrO₃ in the container)
      ii) Place any excess KBrO₃ in the appropriate waste container provided.
   d) Keep the KBrO₃ in the weighing boat and return with it to your station.
2) Obtain the four reaction solutions in beakers. The “Procedural Diagram” on page 9 shows the
   amounts of each of the solutions you need.
   a) Take your 150 mL beaker to the fume hood in the back of the room and add 15 mL of
      solution A followed by 15 mL of solution B to the beaker.
   b) Return to your station and put the beaker on the stir plate. Add the stir bar to the beaker and
      turn the stir rate to “6” (if the yellow plates are used). If the plates have a dial and no values,
      use a slow-medium speed. Make sure the solution is mixing and the stir bar moves freely.
   c) Take the 100mL beaker to the fume hood containing Solutions C and D. Solution C is yellow
      and solution D is dark red. Add 15 mL of solution C (yellow) to the beaker.
   d) Add 1.44 mL of solution D to the beaker using the automatic pipet. You will need to
      dispense 1 mL using one pre-set pipet and 0.44ml using another pre-set pipet. It is very
      important to use the correct technique for measuring with the automatic pipet in order to
      dispense the proper amount of reagent.
      • Check to make sure the pipet is set to dispense the correct volume and the plastic tip is
        securely attached
      • Push the plunger down to the first stop position and insert the tip into solution D
      • Slowly release the plunger to draw the liquid into the tip. If you release the plunger too
        quickly, air bubbles will form and result in an erroneous volume.
      • Dispense the liquid into the beaker by pressing the plunger down to the first stop position.
        Pause for a second and then depress the plunger to the second stop position to expel the
        last drop of liquid. DO NOT let the pipet tip touch the solution in the beaker as this will
        contaminate the tip and subsequently the solution C. If this does happen, you will need to
        replace the tip with a clean one.
   e) The two solutions will mix in the small beaker and create a green color.
   f) Return to your station and add the contents of the small beaker to the large beaker. The best
      results are obtained when the green solution is added to the large beaker within 1 to 2
      minutes.
3) Upon mixing the solutions, the reaction will start and it will change colors. Pay
   particular attention to the sequence of the color changes.

Go to https://www.public.asu.edu/~jhwang43/
Click on “General Chemistry II Laboratory Exercises, By Thomas Cahill”
Click on “Lab2: Reaction Kinetics”
Click on “Effects of Concentrations in the Rate of Reaction”
Click on “Start the reaction and the Clock”
   a) Initially, the four solutions will resemble orange juice. This will fade to a cloudy green
      color, then to a clear green color, then to a blue color (MAKE SURE TO IGNORE THIS
      VERY FIRST BLUE COLOR!), to a purple color, then to a red color. Follow the color chart
      on the next page if you prefer.
b) After the red color, watch closely. The next color will be blue – but it will only be a quick flash of blue. Count this blue as the first blue. This is the color in the cycling pattern that will be used to mark the beginning and the ending of each cycle. You will record the time of every blue stage and use this to calculate the length of the cycle.

**Color Cycle Chart**

This figure displays the color pattern the reaction will progress through. The initial colors upon mixing all four solutions together are on the left. The repeating pattern will be purple, to red, to a flash of blue (clock this).

4) Clock the cycle times
   a) Once the mixture of solution changes to a red color, watch carefully for the blue. This blue flash is very rapid.
   b) At the first hint of this blue, hit START on your own stopwatch, not the one on the website.
   c) For every consecutive flash of blue, hit the LAP button on your stopwatch. Record the time on the data sheets.
   d) Do this for four complete cycles.
   e) At the end of the fourth cycle (namely at the 5th blue flash) record the time **AND add all the extra KBrO₃ that is in the weigh boat, by entering 0.575 in the entry field to the upper-right of the beaker on the hotplate.**
      i) This means, see the first blue (which is ignored); 1st flash of blue, (time 0:00.); 2nd flash of blue (clock time); 3rd flash of blue (clock time); 4th flash of blue (clock time) and lastly the 5th flash of blue clock the time AND add in extra KBrO₃)
      ii) The additional KBrO₃ will double the concentration of bromate in the reaction.
   f) Continue timing the cycles as above.
      i) Record the time of the 5th cycle, which was immediately after the addition of the bromate, but we will not use the data for the reaction kinetics since the bromate will not have
dissolved yet. You may not observe the red color while the additional bromate is dissolving, so watch closely for the blue flash after the purple color!

5) Once you have reached approximately 14 cycles, you can stop data collection for this reaction. Turn off the stir bar and dispose of the waste in the HAZMAT container in the fume hood in the back of the room. Clean out both of the beakers using DI water – DO NOT rinse with regular tap water.

6) Calculate the cycle times (in seconds) for each of the reaction cycles. The cycle time is the interval between two consecutive blue flashes.

7) Calculate the average cycle time for the first four cycles with the “normal” bromate concentration. Calculate the average cycle time for last 4 cycles (#11-14) after the addition of KBrO$_3$, to ensure that the time intervals are representative of when all the bromate has dissolved into the solution. Record these average cycle times on the data sheet.

8) Using the “normal” bromate concentrations as the baseline reaction rate, predict the cycle time of the reaction with double the bromate concentration assuming the reaction follows second order kinetics.
   a) Compare the predicted cycle time with the actual observed cycle time and describe whether the reaction is following first order or second order reaction kinetics.

Part Two – Effects of Temperature Changes
The second part of the laboratory is designed to investigate the influence of temperature on reaction kinetics. You will determine the rate of reaction at room temperature and then at an elevated temperature.

9) Repeat the process of preparing the solutions as was done in Part One - step 2 above. For Part Two, no extra KBrO$_3$ is needed; you simply mix the solutions together as explained above.

10) Place the thermometer in the beaker to record the temperature of the solution. Insert the thermometer through a rubber stopper and use a clamp stand to secure the stopper and position the thermometer in the solution without the tip touching the walls of the beaker or interfering with the stir bar.

   Go to https://www.public.asu.edu/~jhwang43/
   Click on “General Chemistry II Laboratory Exercises, By Thomas Cahill”
   Click on “Lab2: Reaction Kinetics”
   Click on “Effects of Temperature Changes”
   Click on “Start the reaction and the Clock”

   Or after you finish “Effects of Concentrations in the Rate of Reaction”,
   Click on “Back to Lab-2 Main Page”
   Click on “Effects of Temperature Changes”
   Click on “Start the reaction and the Clock”

11) Record the time of each of the blue flashes as was done in Part One. Also record the temperature of the solution at each blue flash. The temperature should be fairly constant for the first 4 cycles.
   a) At the end of the fourth cycle (namely at the 5th blue flash) turn on the hot plate to 185°C, by entering 185 in the entry field to the left of the beaker on the hotplate.
   b) This means see the first blue (which is ignored); 1st flash of blue, (time 0:00.); 2nd flash of blue (clock time); 3rd flash of blue (clock time); 4th flash of blue (clock time) and lastly the 5th flash of blue clock the time AND turn on the hot plate.

12) Continue to record the time and temperature at each blue flash.

13) Once the temperature reaches 50 °C, turn off the hot plate & stir bar and end the reaction.
   a) Dispose of the waste in the HAZMAT container in the back of the room.
b) Clean out both of your beakers and rinse with DI water (NOT regular tap water)
c) Put away all your materials and double check to make sure the hot plate is off.

14) Calculate the time interval for each of the cycles and record them on the data sheets.

Procedural Diagram:
# Reaction Kinetics Experiment Results

## Part One: Effects of Concentration Changes

<table>
<thead>
<tr>
<th>Rapid Blue Appearance #</th>
<th>Time (Min : Sec)</th>
<th>Time (Seconds)</th>
<th>Reaction Cycle #</th>
<th>Interval Cycle Time (Seconds)</th>
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</thead>
<tbody>
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**NOTES:**

If the first cycle is longer than 2 minutes, something has gone wrong and the experiment must be restarted!! This is most likely due to contaminated glassware, so wash the glassware well DI water. Your cycle times should be consistent durations between the first four samples and between samples #8 to 15. If they are erratic (e.g. 40 s, 18 s, 35 s, 17 s) then you may be missing some of the blue flashes and counting two cycles as one. Restart the experiment and watch closely; the red goes through blue to green quickly.

______ of 2 pts
Average cycle interval with the “initial” bromate concentrations: _______________ seconds  
(average cycles 1 to 4)

Average cycle interval with the “double” bromate concentrations: _______________ seconds  
(average # 11-14 cycles)

How much faster are the cycles after doubling the bromate concentration? Express this as a ratio, namely: (average “normal” cycle time)/(average “double bromate” cycle time)  
*Round to the nearest whole number  
____________________ -fold faster

Based on the expected reaction kinetics (see page 2); what order is the B-Z reaction with respect to bromate? Circle one of the answers below.

- **Zero order**
- **First order**
- **Second order**

______ of 5 pts
## Part Two: Effects of Temperature Changes

<table>
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<tr>
<th>Appearance #</th>
<th>Temperature (°C)</th>
<th>Time (Min : Sec)</th>
<th>Time (Seconds)</th>
<th>Reaction Cycle</th>
<th>Interval Cycle Time (Seconds)</th>
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Turn on hotplate to 185 °C

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<th>Appearance #</th>
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Stop data collection when temperature reaches 50 °C.

_____ of 2 pts
Average cycle interval at room temperature: ___________ seconds
(average cycles 1 to 4)

Initial reaction temperature ___________ Kelvin
(average cycles 1 to 4)

Using the Arrhenius Equation given earlier, and $E_a = 46,000 J/mol$, calculate the “A” term for this reaction at room temperature. (Show your calculations)

“A” (the collision frequency factor) = ___________ 1/seconds

The observed cycle interval at 50 °C: ___________ seconds
(Use the one interval where the temperature turned 50 °C)

Using the Arrhenius Equation and your calculated value of “A” above, calculate the expected cycle time for this reaction at the interval temperature 50 °C. (Show your calculations)

Expected cycle time at 50 °C = ___________ seconds

How well do the predicted and observed cycle times agree? Calculate the percent error: $\frac{[(\text{expected cycle time}) - (\text{observed cycle time})]}{\text{expected cycle time}} \times 100$

Percent error between expected and observed cycle times: ___________ %
Determine the Activation Energy of the reaction using your data from Part Two.

Taking the natural log of both sides of the Arrhenius equation, we have

\[
\ln(k) = -\frac{E_a}{R}T + \ln(A)
\]

\[
\ln(k) = (-\frac{E_a}{R})(1/T) + \ln(A)
\]

This is a linear equation and predicts that a graph of \( \ln(k) \) versus \( 1/T \) will have a slope equal to \(-E_a/R\) and a y-intercept equal to \( \ln(A) \). Thus, the Activation Energy \( (E_a) \) can be calculated from the slope of the line.

Construct a graph using Microsoft Excel and determine the Energy of Activation.

1) Plot \( \ln(k) \) (remember, \( k \) in this reaction = 1/cycle time) on the Y-axis and \( 1/T \) (in Kelvin) on the X-axis.
2) Plot all data entries as individual data points. This will result in four replicates of the room temperature cycle time being plotted.
3) Label the title and axes.
4) Add a linear trendline to the data and include both the linear equation and \( R^2 \) on the graph (see instructions below).
5) Graph your data and have your instructor check it BEFORE leaving the lab! It should look like the example graph below. If it looks different, then you have either conducted a computational error or the experiment needs to be re-run.

**Attach your graph to the lab report**

Be sure to label your axes and include the regression or trendline equation on the chart. Make sure your graph looks like the sample graph on the next page.

**Questions from Arrhenius plot:**

Slope of the graph

Calculate the Activation Energy for this B-Z reaction: \( E_a = \) \[ \] J/mol

(Show your work)

______ of 4 pts
Example:
Your graph should look like the example one presented below. However, the numbers will be different.

Excel Graphing Instructions:
1. Enter the data in an excel spreadsheet. It is easier to enter your raw data for cycle time (in seconds) and Temperature (in °C) and then enter formulas to convert to the desired axis units.
   For example, set up these columns for your data:
   - Cycle time (s)
   - k (1/cycle time)
   - ln k
   - Temp (°C)
   - Temp (Kelvin)
   - 1/T (Kelvin)
2. Select chart wizard.
3. Select XY scatter plot (without the data connection lines)
4. Select data series and select the data to be plotted – The x-axis is the independent variable data (the experimentally varied variable) – 1/T. The y-axis is the dependent variable data (responds to a change in the independent variable) – ln k.
5. Title the graph and label both axes with units.
6. Generate the graph as a new sheet.
7. Show the linear equation on the graph: Right click on any data point.
   Add trendline (select linear)
   Options – Display on chart
   Display R² value
Post Lab Questions:

1. If instead the final solution had been placed in an ice bath and the temperature had been decreased, how would the cycle times have been affected? Evaluate the effects of temperature change by manipulating the Arrenius equation \( k = A e^{(-E_a/R \cdot T)} \)
   Remember that in this experiment \( k = \frac{1}{\text{cycle time}} \)
   Would the cycle time decrease or increase? __________________________
   Why?

2. Suppose KBrO₃ [A] is really quite expensive, so only half the original amount is added into the final solution. How would this affect the cycle time? For the purposes of this experiment, the reaction constant will be treated as \( 1/(\text{cycle time}) \).
   Would the cycle time decrease or increase? __________________________
   Why?

3. Suppose that the reaction has a cycle time of 2 minutes at 15 °C. What would the expected cycle time be at 50 °C? Assume that the energy of activation for the reaction is 65,000 J/mol. Show your calculations.

   ______ of 4 pts

Total ______ of 25 pts