THE REACTION OF RED FOOD COLOR WITH BLEACH

TECHNICAL AND THEORETICAL SKILLS
In this assignment you will
- monitor the rate of a chemical reaction
- prepare a volumetric solution
- determine the molar absorptivity of a compound
- use a spreadsheet to analyze data
- calculate the order and rate constant of a reaction

SAFETY
FD&C Red #3, or Erythrocin B sodium, the food coloring dye that you will use in this experiment, is intensely colored. Always wear safety glasses or goggles and a protective lab coat or apron. Handle the dye carefully avoiding spilling on the balance or bench top. Sodium hypochlorite, household bleach, is a bronchial irritant. Keep solutions covered or in the hood; avoid breathing the vapors. Immediately wipe any spills of either reagent and wash any residues with large quantities of water.

INTRODUCTION
In this assignment, you will study the rate of the reaction of FD&C Red #3 (Red #3) with sodium hypochlorite. Since this reaction is very visible, you will use a spectrophotometer to quantitatively follow the rate of disappearance of the colored reagent. Your rate data will allow you to determine the rate law and to propose a possible mechanism for the reaction.

Because of the extended conjugation of alternating double bonds within the molecule, the $\pi-\pi^*$ absorption occurs in the visible region of the spectrum at 530 nm. When the dye reacts with hypochlorite, the color disappears. Even though the product of this reaction has never been determined, we can carry out rate studies with the reagents to determine the mechanism that occurs. One possible explanation for this reaction is that the bleach oxidizes the central methylene carbon atom so that the molecule no longer has the extended conjugation system and the $\pi-\pi^*$ absorption of the less conjugated product occurs at a lower wavelength outside of the visible region of the spectrum. The product might be the alcohol (II) compound depicted in the reaction on the next page.

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A study of how the concentrations of the reactants affect the rate of the reaction gives insight into the mechanism and whether this simple explanation might be correct.

The Rate Law  The rate of a reaction can be represented either by the disappearance of reactants or the appearance of products. Since Red #3 is the only colored species in the reaction, we can monitor the rate of the reaction shown above by recording the decrease in the color of the solution with time. That is,

\[
\text{Rate} = \frac{-d[\text{Red } #3]}{dt} = k[\text{Red } #3]^a[\text{OCl}^-]^b
\]  

where the exponents \( a \) and \( b \) indicate the molecularity or order of the reaction with respect to each reagent, and \( k \) is the overall rate constant for the reaction at room temperature. The objective of this experiment is to determine the values of the exponents \( a \) and \( b \) and the value of \( k \) at room temperature.

If the concentration of the bleach is held constant throughout a reaction by having a large excess present, then the rate law simplifies to

\[
\text{Rate} = \frac{-d[\text{Red } #3]}{dt} = \kappa' [\text{Red } #3]^a
\]

where \([\text{OCl}^-]^b\) has been absorbed in the pseudo-rate constant \( \kappa' \). Rearranging this rate expression to

\[
\frac{-d[\text{Red } #3]}{[\text{Red } #3]^a} = \kappa' dt
\]

gives a form that can be integrated depending on the value of \( a \).

If \( a = 1 \), the integrated expression becomes

\[
-ln[\text{Red } #3] = \kappa' t
\]

and a plot of \( ln[\text{Red } #3] \) vs time will give a straight line with a slope of \( \kappa' \).
If, however, $a = 2$, the integrated expression becomes
\[
\frac{1}{[\text{Red #3}]} = \kappa' t \quad (5)
\]
and a straight line occurs with a plot of $1/[\text{Red #3}]$ vs time.

Thus, if one can experimentally determine the concentration of the dye at various times during a reaction, the relationship of that concentration with time that gives a linear fit allows an experimenter to establish the molecularity of the reaction with respect to the dye (i.e. a value for $a$).

A second set of rate data, collected for reactions where the concentration of the dye is held constant and the initial excess concentration of the bleach is changed in a simple ratio between trials, can then allow a determination of a value for $b$.

For example, since the pseudo rate constant $\kappa' = k [\text{OCl}^-]^b$, if $[\text{OCl}^-]$ is doubled between trials and the $\kappa'$ also doubles, then $b$ must = 1. On the other hand, if $[\text{OCl}^-]$ is doubled and the observed rate increases by a factor of 4 then $b = 2$. However, if $[\text{OCl}^-]$ is doubled between trials and there is no change in the observed rate of the reaction, then $b = 0$ and it can be concluded that the bleach is not involved in the rate determining steps of the mechanism.

**Pre-Lab Assignment**

1. Calculate the molarity of a 6.15% (w/v%) sodium hypochlorite (NaOCl) solution.

2. Calculate the molarity of a solution of Erythrocin B (MW 879.9 g/mol) if a 0.5028-g sample is diluted to 100 mL in a volumetric flask and then two serial dilutions are carried out in which 5-mL aliquots are diluted to 100 mL using volumetric equipment.

3. The half-life of a substance that is being consumed by a first-order rate reaction is the time required for the concentration to fall to one half of the initial value. Show that for $A_o = \frac{A_t}{2}$, the equation

   \[
   \ln \text{ becomes } t_{1/2} = \frac{0.693}{k}
   \]

where $A_o$ is the amount at time 0, $A_t$ is the amount at time $t$, and $t_{1/2}$ is the time required for the concentration to fall to one half the initial value.

4. In a few brief sentences, explain
   - why it is necessary to mix the solution very rapidly when the bleach is added.
   - the purpose of the blank solution.
   - how the shapes of the plots [dye] vs time, ln[dye] vs time, and 1/[dye] vs time are used to determine the order of the reaction.

**Procedure**
The TA will assign you into a group with 1 or 2 other students. If you are in a group of three, allocate the duties so that for each rate reaction trial, one person is the timer, one person is the
data recorder, and one person reads the absorbances as indicated by the timer. In groups of two
one person will be the timer and recorder. You should rotate roles between rate runs. Before
you begin work, agree upon who will be responsible for each part of the work and the report.
Record this in your notebooks. Sign each of the notebooks indicating the agreement. You will
turn in one report with all the group member names on it and the allocation of responsibility.

Solution Preparation: You will need to prepare several solutions to carry out the necessary
rate runs. It is most efficient to have all the solutions ready before you start any of the runs.

Solution 1. Weigh about 0.35 g of food coloring dye to the nearest tenth of a milligram using
weighing paper. Record the weight and quantitatively transfer the dye to a 100-mL volumetric
flask. Re-weigh the weighing paper to determine the actual weight of dye that you transferred to
the volumetric flask and make up the volumetric solution to the mark following standard
procedures for the preparation of a volumetric solution. Transfer the solution to a clean, dry
container. Label this container as “Solution 1.”

Solution 2. Thoroughly rinse and clean the inner walls of the volumetric flask three to four times
with distilled water. Using a 5-mL volumetric pipet, transfer 5 mL of Solution 1 to the 100-mL
volumetric flask. Again use standard procedures to complete the preparation of this volumetric
solution. Transfer this solution to a clean, dry flask and label this solution as “Solution 2”

Solution 3. Repeat the serial dilution procedure for a second time being sure to thoroughly rinse
and clean the inner walls of the volumetric flask with distilled water. Also, be sure to rinse the
5-mL volumetric pipet with Solution 2 before transferring 5 mL of Solution 2 to the 100-mL
volumetric flask. Dilute the solution to the mark and mix well. Transfer this solution to a clean,
dry flask. Label this solution as “Solution 3”

Sodium Hypochlorite. Obtain about 15 mL of concentrated bleach (record the concentration of
the bleach in your notebook) in a clean beaker. Label as “bleach”.

Syringe Preparation: Obtain three 1-mL plastic syringes from your TA. Label the three
syringes: one as H₂O, one as Red #3 (Solution 3), and one as bleach.

Check with the TA to see whether a 5-mL plastic syringe is available for dispensing the BLEACH
solution. If not, use the 1-mL plastic syringe as instructed.

Calibration of the spectrophotometer: After the colorimeter has been plugged in for five
minutes, set the wavelength of the spectrophotometer to 530 nm. Fill a disposable culture tube
about half-full with water. This is your blank solution. Set the absorbance of the blank solution
to zero in the spectrophotometer. Remove the culture tube containing the blank.

Using the 1mL syringe add 6 mL of Solution 3 to a NEW culture tube. Place the tube in the
spectrophotometer. Measure and record the absorbance of the solution. The absorbance of this
solution should fall between 0.3 to 0.6. (consult your TA if the value falls outside of this range.)

Solution Mixing Practice: Since the rate of the reaction depends on the concentration of the
reactants and the reaction begins as soon as the reactants come in contact with each other, it is
necessary to have complete mixing of the solutions as quickly as possible. To practice this
technique, place 5 mL of water in a NEW culture tube. Using the “Solution 3” syringe quickly
squir 1 mL of Solution 3 directly and forcefully into the tube trying to force the dye to the bottom of the tube. Holding the tube between the thumb and first finger shake the tube quickly be sharp snaps of the wrist. Repeat the procedure until you are comfortable with this technique and can mix a solution in under 10 seconds. Use the same culture tube for the practice as the concentrations do not matter.

**Trial preparation:** Label six disposable culture tubes with numbers 1 – 6. To each of the tubes add the appropriate amount of Solution 3 and distilled water as specified in the first two columns of the table below.

**NOTE:**

**DO NOT ADD THE BLEACH TO ANY OF THE SOLUTIONS AT THIS POINT.**

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<tbody>
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<td>4</td>
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<tr>
<td>6</td>
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<td>1 mL</td>
<td>3 mL</td>
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**Reaction-rate-data collection:** When you are ready to collect data, place the tube labeled #1 in the spectrophotometer. The recorder-timer will watch the clock and signal when the absorbance readings should be made and record the value read by the other observer. When indicated by the timer, the observer should use the plastic syringe to quickly squirt the specified amount of bleach for that trial into the solution and mix it well. **Record this time as time = 0 (do NOT record the absorbance at this time).** As soon as the solution is mixed place the tube in the colorimeter and immediately record the absorbance and the NEW time. Leaving the tube in the colorimeter continue to record the time, and absorbance of the solution every 10 seconds for 15 minutes or until the absorbance reaches around 0.01.

**IMPORTANT:** For **TRIALS 5 & 6 ONLY**, record the time, and absorbance of the solution every **5 seconds** for 15 minutes or until the absorbance reaches around 0.05.

When you are finished with trial #1, rotate the responsibility of each group member and continue with the rest of the trials by following the same data collection procedure until you have completed all 6 trials. If the specified amount of bleach is more than 1 mL, add the aliquots of bleach as quickly as possible to minimize the error in “time 0.”

**REPORT**

**PRE-LAB WORK (INDIVIDUAL)**

**Introduction**

In preparing for lab set up your notebook with the following information:

- The title of the experiment
- A reference identifying the procedure handout
• A short introduction summarizing the goals of the experiment and the techniques that you will use in the experiment
• A brief flow chart summary of the key procedures of the experiment
• MSDS information for sodium hypochlorite solutions and erythrocin B sodium
• A table to record the preliminary information on dye weight, bleach concentration and initial solution absorbances.
• Two BLANK data tables to record the measured absorbances and times for the various rate runs. Leave space for a heading that will identify the trial number and the experimental conditions for the trial.

Data:
• Record your partner’s name and the agreed upon allocation of responsibility for the experiment and the report. Indicate in whose notebooks the various data trials will be recorded. All notebooks need to have at least two trials of original data.
• Record the weight of food coloring used to prepare Solution 1.
• Record the concentration of the bleach solution.
• Record the absorbance of Solution 3.

In tabular format record the time and absorbance readings for each of the six rate trials. Be sure the headings for the tables reflect the experimental conditions for the trial.

Calculations:
• Calculate the molar concentrations of the three dye solutions. The molecular weight of FD&C Red #3 is 879.9 g/mol.
• Using Beers Law, \[ A = \varepsilon \cdot bc \], determine the molar absorptivity for FD&C Red #3. Use the concentration and absorbance of Solution 3 and assume the diameter of the culture tube (the path length) is 1 cm.
• Calculate the initial concentration of dye in each trial. This is the concentration at time \( t = 0 \).
• Set up a spreadsheet table for each trial. Label the columns, time, Absorbance, \([\text{dye}]\), \(\ln[\text{dye}]\), \(1/[\text{dye}]\). Note: [ ] means concentration in mol/L.
• Calculate the data for each cell. (Note: Use the measured absorbance of the solution during the trial, the calculated molar absorptivity for the dye, and a path length of 1 cm to determine the \([\text{dye}]\) during the rate runs.)

Graphs:
Prepare three graphs for each trial:
• \([\text{dye}]\) vs. time
• \(\ln[\text{dye}]\) vs. time
• \(1/[\text{dye}]\) vs time.
Be sure to title your graphs, and label the axes including units.

Analysis:
• From a visual inspection of these plots, select the linear relationships to establish \( a \), the order of the reaction with respect to the dye.
• Using the plots that give linear relationships, determine the rate constant for each trial.
• By comparing the slopes of the lines for trials 1, 5, and 6, determine \( b \), the order of the reaction with respect to hypochlorite.
Conclusions:
Summarize your results and the justifications for using the graphs you did to determine the order of the reaction. Write your experimental rate law based on the order of the dye and the order of the sodium hypochlorite that you obtained in your calculations. Kinetics studies can disprove a proposed mechanism, but can never prove a mechanism. Explain whether the results that you have obtained support a mechanism in which the rate determining step involves the reaction of one hypochlorite ion with one molecule of dye.

<table>
<thead>
<tr>
<th>MASTERY CHART</th>
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<tbody>
<tr>
<td>Performance self-assessment</td>
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<tr>
<td><strong>Manipulative skills</strong></td>
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<tr>
<td>make a volumetric solution</td>
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<tr>
<td>use a volumetric pipet</td>
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<tr>
<td>perform serial dilutions</td>
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<tr>
<td>take measurements on a colorimeter</td>
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<tr>
<td>use EXCEL for repetitive calculations</td>
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<tr>
<td>use EXCEL to plot relationships between variables</td>
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<tr>
<td><strong>Theoretical skills</strong></td>
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<tr>
<td>calculate serial dilution concentrations</td>
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<tr>
<td>apply calculus to chemical systems</td>
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<td>use experimental data to support hypotheses</td>
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Signature: _____________________________ Date: __________
CAS Numbers and Hazards Identification:

**Erythrocin B Sodium (FD&C Red #3) – 0016423-68-0**
Skin contact is not expected to create acute health effects. Solid particles on the eye (powder/dust) may cause pain and be accompanied by irritation. No chronic health effects known. No known medical conditions aggravated by prolonged or repeated contact. Not listed as a carcinogen.

Vendor: Noveon Hilton Davis, Inc.
2235 Langdon Farm Rd.
Cincinnati, Ohio 45237-4790
800-477-1022 X3752

**Sodium hypochlorite (household bleach) – 7681-52-9**
Corrosive: May cause severe irritation or damage to eyes and skin. Harmful if swallowed. Protect eyes when handling. For prolonged use, wear gloves. Wash after contact with product. Avoid breathing vapors and use only in a well ventilated area.

Vendor: Discount home retailer such as Wal-Mart, Home Depot, Target

**Instructor Notes:**

(1) In order to minimize waste, reduce spillage, and maintain cleanliness in the labs and balance areas, we prepackage approximately 0.3 g samples of the solid Red #3 dye in small “plastic” bags, which are then heat sealed. (Kapak sealpak pouches, VWR Scientific #11214-301)

(2) We provide each group with two 1-mL plastic disposable syringes without needles (one for water and one for dye) and one 5-mL plastic syringe (without needle) for the bleach. Students are reminded to mix the dye and water aliquots well before adding the bleach solution. (1-mL disposable plastic syringes VWR Scientific #66064-752; 5-mL disposable plastic syringes Fisher Scientific #03-377-22)

(3) Two groups of students (2-4/group) sharing a spectrophotometer, can easily acquire the data for six rate runs each in a three-hour lab period.

(4) Students are instructed to make sure they have at least seven data points for each trial. This is particularly important for the trials where the order of the reaction with respect to the bleach concentration is being determined.