



# Vitamin C Clock Reaction

A case study prepared by Beyond Benign as part of the Green Chemistry in Higher Education program: A workshop for EPA Region 2 Colleges and Universities

# Vitamin C Clock Reaction

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## Vitamin C Clock Reaction

### Summary:

Rate laws are studied in general chemistry undergraduate courses. The laboratory experiments that demonstrate rate laws typically involve experiments that enable students to determine the order of a reaction and the rate constant. Clock reactions are popular experiments that demonstrate rate laws and the dependency on the rate law in relation to concentration and temperature. Clock reactions can be performed with a variety of reagents, including bisulfites, formaldehyde, mercuric ions, and thiosulfates. Most of these materials have safety concerns associated with them. By replacing these materials with safer household items that produce waste that is less hazardous, the safety risks are decreased and the difficulty of waste disposal is decreased.

### *Reduction in waste and purchasing costs:*

For every semester this reaction is implemented at Syracuse University with 500 students, there is an overall **cost savings of \$1,092.15** in purchasing and waste disposal costs and a **reduction in waste from 29.5 gallons to 7.5 gallons**. The greener version of the Clock Reaction also **eliminates the use of 1 kg potassium iodide (8 gal 0.2M solution), 1.8 kg ammonium persulfate (10.8 gal 0.2M solution) and 0.66 kg potassium nitrate (8.5 gal 0.2M solution)**, all of which have human and/or aquatic hazards.

### Background:

This case study is a result of an EPA Region 2 Source Reduction grant<sup>1</sup> titled *Green Chemistry in Higher Education: A Workshop for Region 2 Colleges and Universities*. The Green Chemistry in Higher Education workshop was carried out at Siena College on July 18-21, 2013. 29 faculty members participated from 20 different institutions in New York and New Jersey. The workshop consisted of three main focus areas: green chemistry case studies for lecture and course work, green chemistry laboratory exercises, and toxicology and environmental impact.

During the workshop participants were able to test a variety of greener laboratory exercises for introductory and organic chemistry courses. One of the labs was a Vitamin C Clock reaction,<sup>2</sup> an alternative laboratory exercise for traditional clock reactions. The experiment was very well received by many of the participants. Six participants indicated that they would be implementing the laboratory in their general chemistry courses in the 2013 - 2014 or 2014 - 2015 academic year: Gary Bonomo, Syracuse University (NY), Andy Eklund, Alfred University (NY), Jia Luo, Monmouth University (NJ), Abby O'Connor, The College of New Jersey (NJ), Elizabeth Sprague, RPI (NY), and Matthew Fountain, SUNY Fredonia (NY). Gary Bonomo, along with a colleague from Syracuse University was able to revise and implement the laboratory exercise in the General Chemistry II course at Syracuse University, which consists of about 500 students each semester. The reduction in chemicals used and cost savings for this new experiment are outlined in the following pages.

<sup>1</sup> Disclaimer: Although the information in this document has been funded wholly or in part by the United States Environmental Protection Agency under assistance agreement X9-96296312 to Beyond Benign, it has not gone through the Agency's publications review process and, therefore, may not necessarily reflect the views of the Agency and no official endorsement should be inferred.

<sup>2</sup> The Vitamin C Clock Reaction was adapted from the American Chemical Society's Green Chemistry Lab "Getting off to a safe start - Using safer starting materials for chemical reactions" in *Introduction to Green Chemistry: Instructional Activities for Introductory Chemistry*, American Chemical Society, 2002.

## ***Additional Resources for Green Chemistry in General Chemistry and Beyond:***

### **Greener Educational Materials (GEMs) Database (University of Oregon)**

- Website: <http://greenchem.uoregon.edu/gems.html>
- Description: Searchable database with Green Chemistry educational materials uploaded by faculty members and educators world-wide
- Most curriculum is available for download (free-of-charge) or with primary literature information
- Google map of Green Chemistry educators

### **American Chemical Society's Green Chemistry Institute**

- Website: [www.acs.org/greenchemistry](http://www.acs.org/greenchemistry)
- Description: Green Chemistry Resources for educators and students
- Experiments and Curriculum available for download
- List of ACS books on Green Chemistry

### **Green Chemistry Commitment**

- Website: [www.greenchemistrycommitment.org](http://www.greenchemistrycommitment.org)
- Description: A program of Beyond Benign to adopt Green Chemistry Learning Objectives in higher education.
- Case studies are available, university highlights, and curriculum resources

### **Beyond Benign**

- Website: [www.beyondbenign.org](http://www.beyondbenign.org)
- Description: Green Chemistry Curriculum available on-line (free-of-charge)
- Regional Outreach and Community Educational Events

### **GCEdNet - Green Chemistry Education Network**

- Website: <http://cmetim.ning.com/>
- Description: A place where Green Chemistry educators share resources
- Blogs, discussions and chat rooms

### **University of Scranton Greening Across The Chemistry Curriculum**

- Website: <http://www.scranton.edu/faculty/cannm/green-chemistry/english/drefusmodules.shtml>
- Description: Green Chemistry modules available for download
- Power point presentations, hand-outs available

### **Carnegie Mellon University Institute for Green Science**

- Website: <http://igs.chem.cmu.edu/>
- Description: Green Chemistry modules available for download
- Power point presentations, hand-outs available

## Traditional Experiment:

Clock reactions can be performed with a variety of reagents, including bisulfites, formaldehyde, mercuric ions, and thiosulfates. The traditional clock reaction involves the use of mercuric ions. However, the mercury compounds are typically not used in today's classrooms. The most common clock reaction involves the reaction of a peroxydisulfate ion with an iodide ion. In the experiments, students will measure the rate of a reaction and determine the rate law by measuring the amount of peroxydisulfate that reacts as a function of time. The rate of the following reaction is measured:



## Clock Reaction Traditional Experiment

Chemicals avoided per class of  
500 students:

**1 kg potassium iodide**  
(8 gal 0.2M solution)

**1.8 kg ammonium persulfate**  
(10.8 gal 0.2M solution)

**0.66 kg potassium nitrate**  
(8.5 gal 0.2M solution)

## Chemicals used and hazards:

The chemicals that are typically used in this experiment are listed below, along with a list of the hazards. The amounts are estimated based on a common procedure from one of the most widely used General Chemistry textbooks.<sup>3</sup>

Table 1. Chemicals used, human health and aquatic toxicity data for traditional clock reaction:

Chemical:	Amount used per group of 2 students:	Human health toxicity: <sup>4</sup>	Aquatic toxicity: <sup>4</sup>
potassium iodide, 0.2 M (MW = 166 g/mol)	122.5 mL (4.07 g/122.5 mL) (0.032 gal)	<i>Moderate toxicity</i> , LD50 (oral, mouse) 1,000 mg/kg	<i>Moderate toxicity</i> , LC50 (fish, 96 hr) 2,190 mg/l; EC50 (daphnia, 24 hr) 2.7 mg/l
starch solution, 1%	4.1 mL	<i>Low toxicity</i>	<i>Low toxicity</i>
sodium thiosulfate, 0.4 M (MW = 158.11)	32 mL (2.02 g/32 mL) (0.008 gal)	<i>Low toxicity</i> , Lethal dose (humans) 0.5-5 g/kg	<i>Low toxicity</i> , LC50 (fish, 96 hr) 24,000 mg/l
disodium Ethylenediaminetetraacetic acid, 0.1 M (MW = 372.24g (dihydrate))	0.5 mL (0.019g/0.5 mL)	<i>Low toxicity</i> , LD50 (oral, rat > 2,000 mg/kg)	<i>Low toxicity</i> , LC50 (fish, 96 hr) > 500 mg/l; EC50 (daphnia, 24 hr) > 100 mg/l
potassium nitrate, 0.2 M (MW = 101.1 g/mol)	129.5 mL (2.62 g/129.5 mL) (0.034 gal)	<i>High toxicity IARC Group 2A: Probably carcinogenic to humans, Reproductive &amp; developmental hazards</i> LD50 (oral, rat) 3,750 mg/kg	<i>Moderate toxicity</i> , LC50 (fish, 96 hr) 22.5 mg/l; EC50 (daphnia, 72 hr) 226 mg/l

<sup>3</sup> "Rates of Chemical Reactions I: A Clock Reaction", in Chemistry The Central Science Laboratory Experiments, 12<sup>th</sup> Edition, by Nelson, J.H., Kemp, K.C., and Stoltzfus, M., Pearson Education, 2012.

<sup>4</sup> Human health and aquatic toxicity data was gathered from Globally Harmonized Safety Data Sheets, which can be obtained from Sigma-Aldrich [<http://www.sigmaaldrich.com/united-states.html>].

### Traditional Experiment, continued:

The purchasing and waste disposal costs associated with this procedure are estimated in the following table. Purchasing costs were estimated based on prices available from Sigma-Aldrich:<sup>5</sup>

#### Total amounts of chemicals used and disposed of per class of 500 students:

- 1 kg potassium iodide (8 gal 0.2M solution)
- 1.8 kg ammonium persulfate (10.8 gal 0.2M solution)
- 0.66 kg potassium nitrate (8.5 gal 0.2M solution)
- 29.5 gallons liquid waste

## Clock Reaction Traditional Experiment

Volume of waste and purchasing and waste disposal costs per class of 500 students:  
29.5 gallons of liquid waste  
\$1,310.35 in purchasing and disposal costs

Table 2. Purchasing and waste disposal costs:

Chemical:	Amount per 100 students:	Waste disposal cost <sup>6</sup>	Purchasing cost: <sup>5</sup>	Purchasing cost per 100 students:	Waste disposal cost per 100 students:	Total cost (per 100 students)
potassium iodide, 0.2 M (MW = 166 g/mol)	1.6 gal (203.5 g/1.6 gal)	\$11.27/gal	\$200.50, 500 g	\$81.60	\$18.03	\$99.63
starch solution, 1%	0.054 gal	\$11.27/gal	\$63.60, 500 mL	\$26.08	\$0.61	\$26.68
ammonium persulfate, 0.2M (MW = 228.2 g/mol)	2.15 gal (367.4 g/2.15 gal)	\$11.27/gal	\$69.30, 500 g	\$50.92	\$24.23	\$75.15
sodium thiosulfate, 0.4 M (MW = 158.11)	0.4 gal (101 g/0.4 gal)	\$11.27/gal	\$45.50, 250 g	\$18.38	\$4.51	\$22.89
disodium Ethylenediaminetetraacetic acid, 0.1 M (MW = 372.24g (dihydrate))	0.0065 gal (0.95 g/0.0065 gal)	\$11.27/gal	\$34.30, 100g	\$0.33	\$0.07	\$0.40
potassium nitrate, 0.2 M (MW = 101.1 g/mol)	1.7 gal (130.9 g/1.7 gal)	\$11.27/gal	\$69.30, 500g	\$18.15	\$19.16	\$37.30
TOTAL (per 100 students):	5.9 gal			\$195.46	\$66.61	\$262.07

#### Total purchasing and waste disposal costs per class of 500 students:

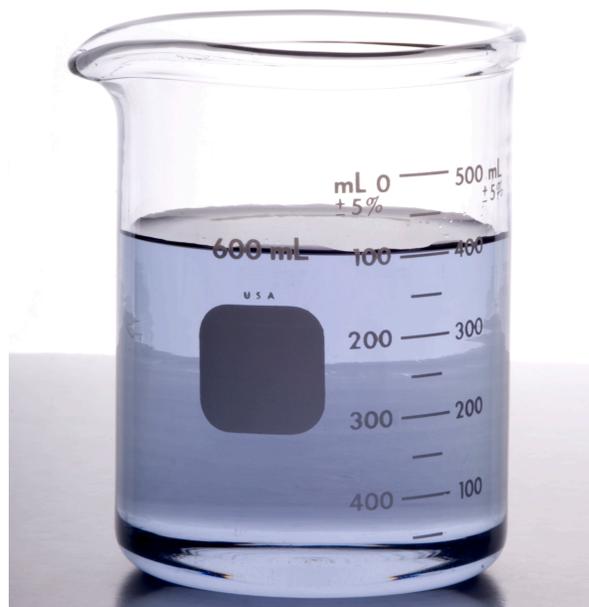
- \$977.30 in purchasing costs
- \$333.05 in waste disposal costs
- \$1,310.35 total cost

<sup>5</sup> Sigma-Aldrich [<http://www.sigmaaldrich.com/united-states.html>, Accessed July 18, 2014].

<sup>6</sup> Waste disposal costs are based on the EPA Cost Calculator Tool [<http://www.epa.gov/p2/pubs/resources/measurement.html#calc>, accessed December 2014].

## Vitamin C Clock Reaction A Greener Approach

Volume of waste and purchasing and waste disposal costs per class of 500 students:  
**7.5 gallons of liquid waste**  
**\$218.25 in purchasing and disposal costs**



### A Greener Approach:

The Vitamin C Clock Reaction is an ACS safer laboratory experiment (“Getting Off to a Safe Start: Using safer starting materials for chemical reactions” in Introduction to Green Chemistry, American Chemical Society, 2002, p. 5-11.) that replaces traditionally used chemicals described previously.

Gary Bonomo at Syracuse University worked with a colleague, Hewn Zheng, to adapt and implement a greener approach to the traditional Clock Reaction based on the ACS safer laboratory experiment that was performed during the Siena workshop in the summer of 2013. The lab has been implemented into the General Chemistry II lab in the spring of 2014 and will replace the traditional experiment in future courses.

In the greener approach to the Clock Reaction, iodine solution is reacted with hydrogen peroxide in order to measure the rate law for the reaction. Liquid starch is used as the indicator for the  $I_3^-$  product and vitamin C (ascorbic acid) is used in the reaction in order to consume the  $I_3^-$  product in this reaction.

Table 3. Chemicals used, human health and aquatic toxicity data:

Chemical:	Amount per group of 2 students:	Human health toxicity: <sup>4</sup>	Aquatic toxicity: <sup>4</sup>
2% Lugol solution (2 g iodine, 2.1 g NaI, in 100 mL water)	7.5 mL	<i>Low toxicity</i> , Iodine: (LD50 oral, rat) 14000 mg/kg; (LD50 oral, mouse) 22000 mg/kg; Sodium iodide: (LD50 oral, rat) 4340 mg/kg; (LD50 oral, mouse) 1000 mg/kg; Ethyl alcohol 200 Proof: (LD50 oral, rat): 7060 mg/kg; (LD50 oral, mouse) 3450 mg/kg; (LC50 inh, rat, 8 hr): 20000 ppm 8 hours; (LC50 inh, mouse, 4 hr): 39000 mg/m 4 hours	<i>High toxicity</i> Iodine: (LC50 fish) 1.7 mg/l, 96 hr; EC50 fish 0.2 mg/l, 48 hr; Sodium iodide: LC50 (fish, 96 hr) 860 mg/l; EC50 (daphnia, 48 hr) 0.17 mg/l
Starch solution (liquid laundry starch)	3 mL	<i>Low toxicity</i>	<i>Low toxicity</i>
Vitamin C tablets (500mg vitamin C/250 mL)	75 mL	<i>Low toxicity</i>	<i>Low toxicity</i>
Hydrogen peroxide (3%)	25 mL	<i>Low toxicity</i> , LD50 (oral, mouse) 2000 mg/kg; LD50 (dermal, rat) 4060 mg/kg; LC50 (inh, rat) 2000 mg/m	<i>Low toxicity</i>

## Vitamin C Clock Reaction A Greener Approach

Volume of waste and purchasing and waste disposal costs per class of 500 students:  
**7.5 gallons of liquid waste**  
**\$218.25 in purchasing and disposal costs**

### A Greener Approach:

The purchasing and waste disposal costs associated with this procedure are estimated in the following table. Purchasing costs were estimated based on prices available from Sigma-Aldrich:<sup>5</sup>

**Total amounts of chemicals used and disposed of per class of 500 students:**

- 0.5 gal Lugol solution (2g iodine, 2.1g NaI, 50mL ethanol, 50mL water)
- 5.2 gal aqueous solutions (starch solution and vitamin C table solution)
- 1.7 gal 3% hydrogen peroxide
- **7.5 gallons liquid waste**

Table 4. Purchasing and waste disposal costs:

Chemical:	Amount per 100 students:	Waste disposal cost <sup>6</sup>	Purchasing cost: <sup>5</sup>	Purchasing cost per 100 students:	Waste disposal cost per 100 students:	Total cost (per 100 students)
2% Lugol solution <sup>7</sup> (2 g iodine, 2.1 g NaI in 100 mL water)	375 mL (0.099 gal)	\$11.27/gal	\$54.10, 1L	\$20.29	\$1.12	\$1.12
Starch solution (liquid laundry starch)	150 mL (0.04 gal)	\$11.27/gal	\$2.97, 64 fl oz (0.5 gal)	\$0.24	\$0.45	\$0.69
Vitamin C tablets (500 mg tablets) (solution is 500mg vitamin C/250 mL)	3.750 mL (0.99 gal)	\$11.27/gal	\$4.37, 120 tablet bottle	\$2.00	\$11.16	\$13.16
Hydrogen peroxide (3%)	1,250 mL (0.33 gal)	\$11.27/gal	\$1.77, 16 oz bottle (0.125 gal)	\$4.67	\$3.72	\$8.39
<b>TOTAL (per 100 students):</b>	<b>1.5 gal</b>			<b>\$27.20</b>	<b>\$16.45</b>	<b>\$43.65</b>

**Total purchasing and waste disposal costs per class of 500 students:**

- **\$136.00 in purchasing costs**
- **\$82.25 in waste disposal costs**
- **\$218.25 total cost**

<sup>7</sup> Tincture of iodine can be used in place of Lugol solution (or Lugol's iodine). Tincture of iodine is a 2% solution containing 2 g iodine, 2.1 g NaI, 50 mL ethanol, 50 mL water. Potassium iodide can also be used in place of sodium iodide.

## Vitamin C Clock Reaction Summary

**Waste reduction:**  
*Reduction from 29.5 gal to  
7.5 gallons of liquid waste*

**Cost savings:**  
*Reduction from \$1,310.35 to  
\$218.25 in purchasing and  
disposal costs*



### Traditional Experiment Summary:

Total amounts of chemicals used and disposed of per class of 500 students:

- 1 kg potassium iodide (8 gal 0.2M solution)
- 1.8 kg ammonium persulfate (10.8 gal 0.2M solution)
- 0.66 kg potassium nitrate (8.5 gal 0.2M solution)
- **29.5 gallons total liquid waste**

Total purchasing and waste disposal costs per class of 500 students:

- \$977.30 in purchasing costs
- \$333.05 in waste disposal costs
- **\$1,310.35 total cost**

### A Greener Approach Summary:

Total amounts of chemicals used and disposed of per class of 500 students:

- 0.5 gal Lugol solution (tincture of iodine) (2g iodine, 2.1g NaI, 50mL ethanol, 50mL water)
- 5.2 gal aqueous solutions (starch solution and vitamin C table solution)
- 1.7 gal 3% hydrogen peroxide
- **7.5 gallons total liquid waste**

Total purchasing and waste disposal costs per class of 500 students:

- \$136.00 in purchasing costs
- \$82.25 in waste disposal costs
- **\$218.25 total cost**

### Conclusions:

In summary, by implementing a greener version of the traditional Clock Reaction, there are many benefits.

For every semester that this reaction is implemented at Syracuse University with 500 students, there is an overall cost savings of \$1,092.10 in purchasing and waste disposal costs and a reduction in the volume of waste of 22 gallons of waste. The greener version of the Clock Reaction also eliminates the use of potassium iodide, ammonium persulfate and potassium nitrate, all of which have human or aquatic hazards associated with them.

# Appendix A: Syracuse University Laboratory Exercise

## Vitamin C Clock Reaction

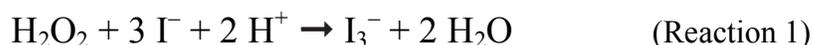
Modified by Hewen Zheng<sup>[1,2,3]</sup>

### Introduction: Clock Reaction

The Rate at which a chemical reaction proceeds is typically influenced by the amount of each reactant present and the temperature of the reaction vessel. And, typically, this relationship between the Reaction Rate and Reagent Concentration takes a simple form known as the *rate law*:

$$\text{rate} = k[\text{A}]^x[\text{B}]^y \quad (\text{Equation 1})$$

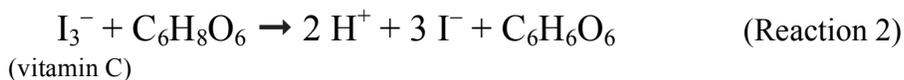
A and B are generic reacting species, k is a reaction specific proportionality constant known as the Rate Constant, and x and y are the reaction order. The rate law parameters k, x and y must be determined experimentally. In this experiment you will determine the *order of reaction* and the *rate constant* for the reaction of H<sub>2</sub>O<sub>2</sub> with iodide. You will do this by varying the concentrations of the reagents, and measuring the reaction times. From this data you will construct a *rate law* for the reaction.



Because the rate of a reaction is a measure of change in the concentration of the reactant over time, the rate of reaction can be represented by the equation below,  $\Delta[\text{H}_2\text{O}_2]$  is the change in the concentration of H<sub>2</sub>O<sub>2</sub> and  $\Delta t$  is reaction time.

$$\text{rate} = - \Delta[\text{H}_2\text{O}_2] / \Delta t \quad (\text{Equation 2})$$

Starch is present in the reaction mixture as an indicator for the product, I<sub>3</sub><sup>-</sup>. When I<sub>3</sub><sup>-</sup> binds to starch you see a dark blue-black color. The reaction in this experiment is called a clock reaction because instead of observing the gradual appearance of the product (I<sub>3</sub><sup>-</sup>), you will add another reagent, vitamin C, to use up I<sub>3</sub><sup>-</sup> (see reaction below) as fast as it is formed by the reaction with H<sub>2</sub>O<sub>2</sub>. As soon as vitamin C is gone, however, the I<sub>3</sub><sup>-</sup> will persist and you will see the expected dark blue-black starch iodine color.



From Reaction 1 and Reaction 2, we can see when one molecule of H<sub>2</sub>O<sub>2</sub> is consumed, one molecule of C<sub>6</sub>H<sub>8</sub>O<sub>6</sub> will be consumed in reacting with I<sub>3</sub><sup>-</sup>. Consequently,

$$\Delta[\text{H}_2\text{O}_2] = \Delta[\text{C}_6\text{H}_8\text{O}_6] \quad (\text{Equation 3})$$

$$\text{rate} = - \Delta[\text{C}_6\text{H}_8\text{O}_6] / \Delta t \quad (\text{Equation 4})$$

For a “Clock Reaction” to work, the process that uses up the product of the reaction of interest must be much faster than the reaction under study. Additionally, the reagent that reacts with the product must be present in limiting amount so that once the reagent is consumed an indicator (like starch) will change color.

Because we know the initial concentration of  $C_6H_8O_6$ , and that it is completely consumed as blue color appears. Using equation,  $\Delta [H_2O_2]$  can be calculated.  $\Delta t$  is measured by using a stopwatch to measure the time between mixing the reactants and appearance of the blue color. The rate law can be written as follows, where  $k$  is rate constant, and where  $x$  and  $y$  are the orders of the reactants:

$$\text{rate} = k[H_2O_2]^x[I^-]^y \quad (\text{Equation 5})$$

By varying the initial concentration of  $H_2O_2$  and  $I^-$ , the reaction orders ( $x$  and  $y$ ) and the reaction constant  $k$  can be experimentally determined.

The rate constant ( $k$ ) varies with temperature. As temperature increases, the number of effective collisions between molecules increases which increases the rate of reaction. Additionally, the minimum collision energy (activation energy;  $E_a$ ) must be met for a reaction to proceed. This relationship is represented by the Arrhenius Equation, which also contains a pre-exponential constant called the frequency factor ( $A$ ) and the gas constant ( $R$ ):

$$k = Ae^{\frac{-E_a}{RT}} \quad (\text{Equation 6})$$

$k$  = rate constant       $E_a$  = activation energy       $R$  = gas constant       $A$  = frequency factor

Taking the natural logarithm of Equation 6 yields the following equation:

$$\ln k = -\frac{E_a}{RT} + \ln A \quad (\text{Equation 7})$$

The equation can be rewritten, so that it matches the equation of a line ( $Y = mX + b$ ). Where each of the terms are:

$$Y = \ln k \quad X = \frac{1}{T} \quad m = \text{slope} = -\frac{E_a}{R}$$

$b$  = y-intercept =  $\ln A$

Thus the rewritten equation is as follows:

$$\ln k = \left(-\frac{E_a}{R}\right) \left(\frac{1}{T}\right) + \ln A \quad (\text{Equation 8})$$

By plotting  $\ln k$  vs.  $1/T$ , and finding the slope of the linear-fit of the graph the activation energy ( $E_a$ ) can be calculated. The slope is found by choosing two points on the line and dividing the change in  $Y$  by the change in  $X$ .

## Introduction: Green Chemistry

Green Chemistry is concerned with minimizing the use and generation of chemicals that are harmful to human health as well as the environment. Many starting materials (reactants) and solvents used in chemical processes can be highly toxic. Special care must be followed when dealing with these chemicals to insure the safety of workers. Also the disposal of the chemicals, especially solvents, can be problematic and costly. The principles of Green Chemistry require that a chemist investigate alternative reactants that are safer to use but produce the same product.

Clock Reactions can be done with a variety of reagents; such as bisulfites, formaldehyde, mercuric ions, and thiosulfates. These materials have safety concerns associated with them, but with proper precautions they can be handled safely to manage this risk. The waste produced from these Clock Reactions must also be processed. By replacing these materials with safer household items that produce waste that is less hazardous, the safety risks are decreased and the difficulty of waste disposal is decreased.

Identifying reactions that use nontoxic/nonhazardous starting materials to make a desired product minimizes danger to workers in manufacturing plants when they handle the chemicals and also prevents accidental release of harmful chemicals to the environment if leaks or explosions occur.

The above description focuses on the safety and waste concerns of Green Chemistry, however Green Chemistry is a much broader topic. One of the seminal works in the field of Green Chemistry is the book *Green Chemistry: Theory and Practice* by Paul T. Anastas and John C. Warner. It describes 12 principles for Green Chemistry, which are currently used by American Chemical Society (ACS) Green Chemistry Institute, <http://www.acs.org/content/acs/en/greenchemistry.html>

### **Materials:**

DI water	150mL beakers and 250mL beakers
Vitamin C tablets (500mg per tablet)	digital thermometer
Lugol's iodine (2% I <sub>3</sub> <sup>-</sup> by mass)	ice cubes and a bucket; for an ice bath
hydrogen peroxide (3% H <sub>2</sub> O <sub>2</sub> by mass)	Hotplates; for a warm water bath
liquid laundry starch	stopwatch
50mL graduated cylinders	10mL graduated cylinders
250mL volumetric flask	gravity filtration setup
mortar and pestle	

**Important Terms:** *you will define these as part of your Prelab Assignment*

- Activation Energy
- Frequency Factor
- Order of Reaction
- Rate Constant
- Rate Law

## Experimental Procedure

### Part 1: Preparation of vitamin C stock solution

- 1.1. Make a vitamin C solution by crushing a vitamin C tablet and dissolving it in 60 mL of distilled water using a 150ml beaker.
- 1.2. Place the beaker on the hotplate over low heat (50°C to 80°C). The solution will take about 10 minutes to dissolve.
- 1.3. Set up an apparatus for gravity filtration and filter the solution. Use a glass rod to decant the solution into the paper, leaving behind any substance that will not dissolve (these are materials that just give the tablet its shape and structure).
- 1.4. Add all the filtered solution into 250mL volumetric flask and fill to the calibration line with distilled water; using an eyedropper for the last few drops is advised. Cap the volumetric flask and invert several times to ensure uniformity of the solution.
- 1.5. Label the solution as “vitamin C stock solution”.

### Part 2: The effect of concentration on the clock reaction (*Trial 1a, Trial 2, and Trial 3*)

- 2.1. There are three Trials, each with different concentrations (see the table below). Perform Step 2.2 and Step 2.3 for Trial 1a, then for Trial 2, and then again for Trial 3.
- 2.2. Prepare “Solution A” and “Solution B” in two separate 150mL beaker. Label these beakers.
- 2.3. Pour solution A into a 250mL beaker. Then pour solution B and mix. Begin timing immediately and continue to mix until there is a color change. Record the time it takes for the color to change.

#### Solution A

	Vitamin C stock	2% Lugol's Iodine	DI Water
<b>Trial 1a</b>	25.0mL	5.0mL	20.0mL
<b>Trial 2</b>	25.0mL	2.5mL	22.5mL
<b>Trial 3</b>	25.0mL	5.0mL	20.0mL

#### Solution B

	3% Hydrogen Peroxide	Starch Solution	DI Water
<b>Trial 1a</b>	10.0mL	1.0mL	39.0mL
<b>Trial 2</b>	10.0mL	1.0mL	39.0mL
<b>Trial 3</b>	5.0mL	1.0mL	44.0mL

### Part 3: The effect of temperature on the clock reaction

- 3.1. **Trial 1b:** Prepare the same Solutions A and B that you used for Trial 1a, but cool the solutions to 0°C before mixing by placing the containers in an ice bath. Mix and timing the reaction as before.
- 3.2. **Trial 1c:** Prepare the same Solutions A and B that you used for Trial 1a, but this time using a warm water bath to heat the solutions to 40 °C. Mix and timing the reaction as before.

## How to Calculate the Concentrations:

### Concentration of Vitamin C:

Convert the 0.5g to moles and then divide by the total volume of the stock solution (0.250L).

Use  $M_1V_1 = M_2V_2$  to find the concentration in the final solution.

You used 0.025L of you stock solution and the final volume is 0.100L

### Concentration of Iodide:

The stock solution is a 2% solution of  $I_3^-$ . Multiple 0.02 by the mass of the iodine solution used (since Density is 1 g/mL, this is the same as the volume), for example 0.02 x 5 for Trial 1a.

Convert mass to moles by dividing by the molar mass of  $I_3^-$ .

Convert moles of  $I_3^-$  to moles of  $I^-$  (using Reaction 2, this means that you multiple by 3).

Divide moles of  $I^-$  by the total volume (0.100L).

### Concentration of Hydrogen Peroxide:

The stock solution is a 3% solution of  $H_2O_2$ . Multiple 0.03 by the mass of the hydrogen peroxide solution used (since Density is 1 g/mL, this is the same as the volume), for example 0.03 x 10 for Trial 1a.

Convert mass to moles by dividing by the molar mass of  $H_2O_2$ .

Divide moles of  $H_2O_2$  by the total volume (0.100L).

### Equations for PostLab – solving for x:

$$\frac{Rate_{1a}}{Rate_3} = \frac{k [H_2O_2]_{1a}^x [I^-]_{1a}^y}{k [H_2O_2]_3^x [I^-]_3^y}$$

(Equation 9a)

Since the  $[I^-]$  and  $k$  are the same for these two trials (1a and 3), the equation simplifies to:

$$\frac{Rate_{1a}}{Rate_3} = \left( \frac{[H_2O_2]_{1a}}{[H_2O_2]_3} \right)^x$$

(Equation 9b)

Take the natural log of both sides and rearrange to solve for x: *round to the nearest whole number*

$$x = \frac{\ln(Rate_{1a}/Rate_3)}{\ln([H_2O_2]_{1a}/[H_2O_2]_3)}$$

(Equation 9c)

### Equations for PostLab – solving for y:

$$\frac{Rate_{1a}}{Rate_2} = \frac{k [H_2O_2]_{1a}^x [I^-]_{1a}^y}{k [H_2O_2]_2^x [I^-]_2^y}$$

(Equation 10a)

Since the  $[H_2O_2]$  and  $k$  are the same for these two trials (1a and 2), the equation simplifies to:

$$\frac{Rate_{1a}}{Rate_2} = \left( \frac{[I^-]_{1a}}{[I^-]_2} \right)^y$$

(Equation 10b)

Take the natural log of both sides and rearrange to solve for y: *round to the nearest whole number*

$$y = \frac{\ln(Rate_{1a}/Rate_2)}{\ln([I^-]_{1a}/[I^-]_2)}$$

(Equation 10c)

### References

1. Getting off to a safe start - Using safer starting materials for chemical reactions, Green Chemistry Lab, 5-11
2. Luk, Yan-Yeung. Reaction Kinetics and Effect of Temperature. *General Chemistry Laboratory II CHEM 117 Lab Manual; Second Edition Revised Printing*. Kendal Hunt Publishing Company.
3. Wright, Stephen W. The Vitamin C Clock Reaction. *J. Chem. Educ.*, January **2002**, 79 (1), 41-43.

Name \_\_\_\_\_ Section \_\_\_\_\_

### Vitamin C Clock Reaction: *Post-Lab Questions*

Show your work for all calculations (attach pages if needed); include units and correct significant digits. Please note that the total volume of each solution is 100mL (Solution A and Solution B combine). Assume the densities of 2% Lugol's Iodine and 3% Hydrogen Peroxide are 1.00 g/mL. Please note that this table says asks for  $I^-$ , not  $I_3^-$  (refer to Reaction 2).

	Concentration of vitamin C	Concentration of iodide	Concentration of hydrogen peroxide	Reaction Time
<b><i>Trial 1a</i></b>				
<b><i>Trial 2</i></b>				
<b><i>Trial 3</i></b>				
<b><i>Trial 1b</i></b>				
<b><i>Trial 1c</i></b>				

Complete the above table. Show your calculations for the concentration of vitamin C, the concentration of iodine, and the concentration of hydrogen peroxide in the space below:  
[1.5 points per Concentration; 0.5 point per Reaction Time]

- 1. What is the relationship between the time it takes for a reaction to occur and the reaction rate? Please explain. [5 points]**
- 2. What is the relationship between the concentration of the reactants and the rate of this reaction? Please explain. [5 points]**
- 3. What is the relationship between the temperature and the rate of this reaction? Please explain. [5 points]**
- 4. Calculate the rate constant (k) at room temperature for this reaction. Please show your work here or on an attach piece of paper. [15 points]  
*Please note that you need to find a rate constant (k) for Trials 1a, 2, and 3; and then average them.***
- 5. There are many other types of Clock Reactions. One that is sometime used for educational purposes similar to this lab is the “Old Nassau Reaction”. Provide a list of the chemical reagents used in the Old Nassau Reaction and a list of the chemical reagents used in the Vitamin C Clock Reaction. Which one of these Clock Reactions would you regard as greener and why? Cite any sources that you used to obtain information for this question. [20 points]**
- 6. Using your data from Trial 1a, Trial 1b, and Trial 1c, calculate the  $1/T$  and  $\ln k$  for each Trial; show all calculations in the space below or on an attach piece of paper. Create a data table in *Microsoft Excel* (or a similar program) and then graph of  $\ln k$  vs.  $1/T$ . Add a linear trendline to this graph and show the equation of this line on the graph. Please print and attach the data table and graph as part of your report. [20 points]**
- 7. Using the equation of the line from Question 6, calculate the activation energy of this reaction. Please show your work. [5 points]**

Name \_\_\_\_\_ Section \_\_\_\_\_

## Vitamin C Clock Reaction: *Pre-Lab Assignment*

1. Define the following terms: [10 points each]

Activation Energy:

Frequency Factor:

Order of Reaction:

Rate Constant:

Rate Law:

2. Balance the equation: [10 points]



3. For a reaction that has positive activation energy, would you expect increasing the temperature to increase or decrease the reaction rate? Please explain your answer in using Equation 1 and Equation 6. [15 points]

4. In this experiment, we discuss the portions of Green Chemistry related to safety. However, green chemistry encompasses many other topics as well. Based upon the standards currently used by the American Chemical Society (the 12 Principles of Green Chemistry), please summarize the term "Green Chemistry" in about one or two paragraphs. [25 points]

*The 12 Principles of Green Chemistry are summarized at the following ACS website:*

*<http://www.acs.org/content/acs/en/greenchemistry/what-is-green-chemistry/principles/12-principles-of-green-chemistry.html>*



Vitamin C Clock Reaction: A case study prepared by Beyond Benign as part of the Green Chemistry in Higher Education program: A workshop for EPA Region 2 Colleges and Universities

Download this and other case studies at the following link:  
<http://www.greenchemistrycommitment.org/resources/case-studies/>