King Saud University
Faculty of Science
Department of Chemistry

CHEM 460

Green chemistry

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1443

Experiments (1-4): Vitamin C Clock Reaction.

Summary:
  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 difficultly of waste disposal is decreased.

Traditional Experiment: A Kinetic Study of an Iodine Clock Reaction [1]

Objectives:
 • Determine the order of the rate law (values of m and n) for an iodine clock reaction using the method of initial rates
 • Determine the rate constant (k) of the same rate law
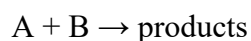• Determine the energy of activation of the reaction

Safety Notes
 • Eye protection must be worn at all times

Discussion
    In chemistry, we study kinetics to determine a possible reaction mechanism, or what might be happening at the molecular level.
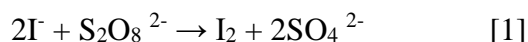For the general reaction:
$$A + B \rightarrow products$$
The rate law equation takes the form:
$$Rate = R = k[A]^m[B]^n$$
in which [A] and [B] are the concentrations in mol/L of species A and B, the exponents m and n are the orders of A and B, respectively, and k is the rate constant. The specific rate constant (k) is a proportionality constant that is a characteristic of every reaction at a specific temperature. The sum of the exponents m and n defines the order of the reaction.

In this experiment, we will be determining the rate of a reaction by measuring how long (Δt) it takes to produce a certain concentration (ΔC) of our product. By varying the concentrations of A and B (part I), we can then determine the values of m and n, using the Method of Initial Rates.
The reaction that you are going to study is that between the persulfate ion ($S_2O_8^{2-}$) and the iodide ion (I^-).
$$2I^- + S_2O_8^{2-} \rightarrow I_2 + 2SO_4^{2-} \qquad [1]$$
The generalized rate equation for this reaction is:
$$R = k\,[I^-]^m[S_2O_8^{2-}]^n$$

1. https://www.jmu.edu/chemistry/132%20Lab/Exp%202--Kinetic%20Study%20of%20and%20Iodine%20Clock%20Reaction.pdf

Reaction [1] proceeds at a rate that allows for convenient measurements of the rate equation. Rather than measuring the concentration of I_2 or SO_4^{2-} directly, we are going to add known amounts of $S_2O_3^{2-}$ ion to our reaction mixture, because it reacts instantaneously with I_2 according to the reaction equation:

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-} \qquad [2]$$

The iodine (I_2) produced in reaction [1] is absorbed immediately by reaction with thiosulfate ion as shown in reaction [2]. As long as any $S_2O_3^{2-}$ remains in solution, the concentration of I_2 is effectively zero. When all of the $S_2O_3^{2-}$ is used up, any additional I_2 that is made via reaction [1] will react with a starch indicator, which will then turn blue.

The combination of reactions [1] and [2] together with the starch indicator constitutes one type of Iodine Clock Reaction. The "clock" or color change indicated when enough iodine has been produced by reaction [1] to use up all of the $S_2O_3^{2-}$. A knowledge of the original $S_2O_3^{2-}$ concentration and the stoichiometric ratio between I_2 and $S_2O_8^{2-}$ leads to the quantity of $S_2O_8^{2-}$ that has reacted when the blue color appears. Because one mole of I_2 reacts with 2 moles of $S_2O_3^{2-}$ during reaction [2], the quantity of I_2 produced at the time the blue color first appears (= t) is equal to one-half the initial quantity of $S_2O_3^{2-}$ (which is known).

$$. t_1/t_2 = ([I^-]_2/[I^-]_1)^m \quad [3]$$

$$. t_3/t_2 = ([S_2O_8^{2-}]_2/[S_2O_8^{2-}]_3)^n \quad [4]$$

In Part II, the effect of temperature on the rate of reaction [1] will be studied. With the same set of concentrations, the reactions will be carried out at several temperatures in addition to room temperature. From these results and a graph based on equation [5], the activation energy (EA) for the reaction may be determined.

$$\ln k = -E_a/R + \ln A \qquad [5]$$

In equation [5], k is the rate constant at each of the different temperatures, E_a is the activation energy in J/mol, R is the gas law constant (8.314 J/molK), and T is the absolute temperature in Kelvin. The value of k can be calculated via the rate law equation [6], using the measured time t and the initial concentrations of the reactants: $[I^-]$, and $[S_2O_8^{2-}]$. Plotting ln k as a function of 1/T, the activation energy can be obtained from the slope (= -Ea/R) of the resulting straight line

$$1/t = k [I^-]^m [S_2O_8^{2-}]^n \quad [6]$$

Procedure
Part I: Dependence of Reaction Rate on Concentration
These three experiments are to be carried out at room temperature. The volumes of the KI, KCl, $(Na)_2S_2O_8$, $(NH_4)_2SO_4$ and $Na_2S_2O_3$ solutions should be measured with a pipet. The KCl and $(NH_4)_2SO_4$ solutions are used rather than water in diluting to a constant volume so that the total ionic strength of the reaction mixture, which has some effect on the reaction rate, can be kept approximately constant.

Table 1. Quantities of reactants used at room temperature

Reactant	Human health toxicity	Volume of reactant(ml)		
		Experiment 1	Experiment 2	Experiment 3
0.2 M KI	Moderate toxicity	1	2	2
0.2 M KCl		1	0	0
0.1 M $(Na)_2S_2O_8$		2	2	1
0.1 M $(NH_4)_2SO_4$		0	0	1
o.005 M $Na_2S_2O_3$	Low toxicity	1	1	1

1.For experiment 1, pipet the specified volumes of 0.200 M KI and 0.200 M KCl solutions into a 50 mL beaker, which will be used as the reaction vessel.

2. Pipet 1.00 mL of 0.005 M $Na_2S_2O_3$ into this beaker and add 2 drops of 1% starch solution.

3. Insert a thermometer into the reaction vessel.

4. Pipet the specified volumes of 0.100 M $(Na)_2S_2O_8$ and 0.100 M $(NH_4)_2SO_4$ into a separate 50 mL beaker.

5. Record the initial temperature of the solution.

6. Observe the time (or start a stopwatch) when pouring the persulfate solution from the beaker into the reaction vessel. (Pour the solution back and forth to mix thoroughly.)

7. Record the time required to turn the solution blue. (Constant observation is necessary because the blue color will appear suddenly.)

8. Rinsing the beakers thoroughly between experiments, repeat Experiment 1 until two elapsed times are within 5 seconds of each other.

9. Repeat steps 1 through 8 for Experiment 2, using the reactant quantities in Table 1 (which does not include KCl).

10. Repeat steps 1 through 8 for Experiment 3, using the reactant quantities in Table 1 (which does not include KCl).

Experiment	t(s)	Initial [I⁻]	Initial [$S_2O_8^{2-}$]	Rate
1				
2				
3				

Order of reaction with respect to I^- --------------

Order of reaction with respect to $S_2O_8^{2-}$ ----------

Overall order of reaction   ---------

Part II: Dependence of Reaction Rate on Temperature

You will be carrying out reaction [1] at the temperatures specified in Table 2. You will be using the same concentrations as in experiment 2 of Table 1.

Table 2. Iodine Clock Reaction and Temperature

Experiment	Temperature (^{o} C)
2	Room temperature
4	About 10° above room temperature
5	About 10° below room temperature
6	About 0 ^{o} C

1. Experiment 2 (room temperature) has already been carried out in Part 1.
2. For experiment 4, make the solutions as you did in experiment 2 in Part 1. Prior to mixing, heat the beakers in a warm water bath while keeping track of the temperature with a thermometer. When the temperature on the thermometer reaches about 10 ^{o} C above room temperature, mix the solutions and record the time it takes until color change
3. For experiments 5 and 6, use the same technique as in experiment 4 while using an ice bath rather than a warm water bath
4. Be sure to record the times of mixing and when the color change occurs, along with the temperature at the time of the color change.

Experiment	Temp(K)	1/T	Elapsed time (s)	.k	.lnk
2					
4					
5					
6					

Using Excel, plot ln k vs. 1/T and add a trendline for these four data points. Be sure to have Excel show the equation for the trendline and the R^2 value on the graph, and include the fully labeled graph with your report.

Slope of trendline (from graph) -----------
Calculated value of Ea (kJ/mol   --------------

Vitamin C Clock Reaction A Greener Approach[2]

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.

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 I3 - product and vitamin C (ascorbic acid) is used in the reaction in order to consume the I3 - product in this reaction.

Table 3. Chemicals used, human health toxicity data:

Chemical	Human health toxicity
2% lugol solution (2 g iodine ,2.1 g NaI , in 100ml water)	Low toxicity
1% Starch solution	Low toxicity
Vitamin C tablets (1000 mg vitamin C / 500 ml)	Low toxicity
Hydrogen peroxide (3%)	Low toxicity

Vitamin C Clock Reaction
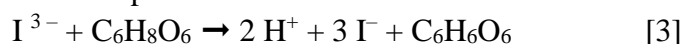
Introduction: Clock Reaction
In this experiment you will determine the order of reaction and the rate constant for the reaction of H_2O_2 with iodide. You will do this by varying the concentrations of the reagents, and measuring the reaction times. From this data you will construct a rate law for the reaction.

$$H_2O_2 + 3\ I^- + 2\ H^+ \longrightarrow I^{3-} + 2\ H_2O \qquad\qquad [1]$$
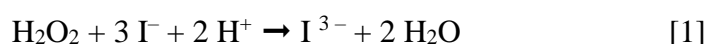
Because the rate of a reaction is a measure of change in the concentration of the reactant over time, the rate of reaction can be represented by the equation below, $\Delta[H_2O_2]$ is the change in the concentration of H_2O_2 and Δ t is reaction time.

$$\text{rate} = -\ \Delta[H_2O_2]\ /\ \Delta t \qquad\qquad [2]$$

Starch is present in the reaction mixture as an indicator for the product, I^{3-}. When I^{3-} 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^{3-}), you will add another reagent, vitamin C ($C_6H_8O_6$), to use up I^{3-} (see reaction below) as fast as it is formed by the reaction with H_2O_2. As soon as vitamin C is gone, however, the I^{3-} will persist and you will see the expected dark blue-black starch iodine color.

$$I^{3-} + C_6H_8O_6 \longrightarrow 2\ H^+ + 3\ I^- + C_6H_6O_6 \qquad\qquad [3]$$

2. https://www.beyondbenign.org/bbdocs/pdfs/GreenerClockReactionCaseStudy.pdf

From Reactions, we can see when one molecule of H_2O_2 is consumed, one molecule of $C_6H_8O_6$ will be consumed in reacting with I^{3-}. Consequently,

$$\Delta[H_2O_2] = \Delta[C6H8O6] \qquad\qquad [4]$$
$$\text{rate} = -\Delta[C_6H_8O_6] / \Delta t \qquad\qquad [5]$$

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. Δt 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:

$$\text{rate} = k[H_2O_2]^x [I^-]^y$$

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. This relationship is represented by the Arrhenius Equation.

- $\ln k = -E_a/R + \ln A$

- $t_2/t_1 = ([I^-]_1/[I^-]_2)^y$

- $t_3/t_1 = ([H_2O_2]_1/[H_2O_2]_3)^x$

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 difficultly of waste disposal is decreased.

Materials:

DI water   150mL beakers and 250mL beakers
Vitamin C tablets (1000mg per tablet)   digital thermometer

Lugol's iodine (2% I3 – by mass)   ice cubes and a bucket; for an ice bath
hydrogen peroxide (3% H2O2 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 pestl

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 120 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 500mL 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, Trail 2, and Trial 3)

2.1. There are three Trials, each with different concentrations (see the table below). Preform 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
Trial 1a	**25 ml**	**5 ml**	**20 ml**
Trial 2	**25 ml**	**2.5 ml**	**22.5 ml**
Trial 3	**25 ml**	**5 ml**	**20 ml**

Solution B

	3% hydrogen peroxide	Starch Solution	DI Water
Trial 1a	**10 ml**	**1 ml**	**39 ml**
Trial 2	**10 ml**	**1 ml**	**39 ml**
Trial 3	**5 ml**	**1 ml**	**44 ml**

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 1g to moles and then divide by the total volume of the stock solution (0.50L). Use $M_1V_1 = M_2V_2$ to find the concentration in the final solution. You used 0.50L 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).

Experiment	t(s)	Initial [I^-]	Initial [H_2O_2]	Rate
1				
2				
3				

Order of reaction with respect to I^- --------------

Order of reaction with respect to H_2O_2 ----------

Overall order of reaction

Temp(K)	1/T	Elapsed time (s)	.k	.lnk

Using Excel, plot ln k vs. 1/T and add a trendline for these four data points. Be sure to have Excel show the equation for the trendline and the R^2 value on the graph, and include the fully labeled graph with your report.

Slope of trendline (from graph) -----------

 Calculated value of Ea (kJ/mol   --------------

Additional Resources for Green Chemistry in General Chemistry :
- **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
 • 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
• 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
 • 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

-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

Experiments (5-6):Wood Ash Titration: A Greener Titration Experiment[1]

Traditional Experiment [2]:

In this traditional experiment, a solution of oxalic acid is prepared and titrated with sodium hydroxide solution to an end-point in order to calculate the concentration of sodium hydroxide.

Aim

Determination of the concentration (strength) of a given sodium hydroxide solution by titrating it against a standard solution of oxalic acid.

Theory

In the titration between oxalic acid (weak acid) and sodium hydroxide (strong base), following reaction takes place. In this titration phenolphthalein (HPh) is used as an indicator.

$(COOH)_2 + 2NaOH \rightarrow (COONa)_2 + 2H_2O$

Material Required

Burette (50 mL) : One
• Conical flask (100 mL) : One
 • White glazed tile : One
• Oxalic acid
• Phenolphthalein indicator

• Pipette (10 mL) : One
• Burette stand : One • Funnel : One
• Measuring flask (100 mL) : One
• Sodium hydroxide solution

Table 1. Chemicals used, human health toxicity data:

Chemical	Human health toxicity
Oxalic acid	High Toxicity
Sodium hydroxide	Causes severe skin burns and eye damage
Phenolphthal ein indicator solution (0.5% wt. in ethanol: water (1:1))	Suspected carcinogen - IARC Group 2B, Reproductive hazard

Procedure

1- Preparation of 0.1M Standard Solution of Oxalic Acid.
2- Pipette out 10 mL of oxalic acid solution in a washed and dried conical flask.
3- Add 1-2 drops of phenolphthalein indicator to the conical flask. Titrate the acid with sodium hydroxide solution till a very faint permanent pink colour is obtained. Add sodium hydroxide solution in small amounts initially and then dropwise.
4- Repeat the procedure until three concordant readings are obtained.

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1. https://www.beyondbenign.org/bbdocs/curriculum/higher-ed/CS_Wood_Ash_Titration.pdf
2. https://ncert.nic.in/pdf/publication/sciencelaboratorymanuals/classXI/chemistry/kelm206.pdf

3. **Calculations**

   Molarity of NaOH solution can be calculated by using the equation:

   Oxalic acid   Sodium hydroxide

   $$a_1 M_1 V_1 = a_2 M_2 V_2$$

   where, M_1 and V_1 are the molarity and volume of the oxalic acid solution.

   M_2 and V_2 are the molarity and volume of the sodium hydroxide solution.

   a_1 and a_2 are respectively the basicity of oxalic acid and acidity of sodium hydroxide.

   In this case $a_1 = 2$ and $a_2 = 1$.

   Also, Molar mass of oxalic acid, $(COOH)_2.2H_2O = 126$ g mol^{-1} and Molar mass of sodium hydroxide (NaOH) = 40 g mol^{-1}

   Calculate the concentration of sodium hydroxide solution in g/L by using the equation given below.

   Concentration (strength) in g/L = Molarity×Molar mass

   Result

   Concentration of NaOH solution is ————————— g/L

Wood Ash Titration

 A Greener Approach
   The greener approach for a titration lab involves extracting base from wood ash and titrating the base with a known standard of potassium hydrogen phthalate (KHP) to determine the concentration of base in the sample of wood ash. A greener indicator, thymolphthalein, is also used in place of phenolphthalein. The experiment can be used to discuss renewable feedstocks, and the procedure can be linked to other experiments, such as the preparation of biodiesel or soap. The wood ash used in this experiment can be disposed of in the trash, or can be composted.

Table 2. Chemicals used, human health toxicity data

Chemical	Amount used	Human health toxicity
Wood ash	50g	n/a
Water	250ml	n/a
Potassium hydrogen phthalate in water (10 mL)	0.08 g in 10 ml water	Low Toxicity
Thymolphthalein indicator (1 g in 100 mL ethanol/water (1:1))	0.1 ml	Low Toxicity

Wood Ash Titration: Renewable Resources for the Preparation of Biodiesel

SUMMARY:

   The 12 Principles of Green Chemistry guide us to use catalysts to improve the energy and atom efficiency of reactions. The principles also guide us to use renewable feedstocks. In this experiment, we can see how waste from one process can be used productively in another. Specifically, biodiesel can be made from waste vegetable oil, a renewable feedstock that was traditionally discarded by the food preparation industry. To prepare biodiesel from the vegetable oil, base is needed as a catalyst. In this lab we will focus on the catalyst and its source from another "waste material" – wood ash.

   Wood ash has been used as a valuable source of base throughout history. In this experiment we will extract the basic substances from a sample of wood ash and then determine their base potential compared to sodium hydroxide, the base often used in the production of biodiesel

Ash Water Titration: Renewable Resources for the Preparation of Biodiesel

Purpose:
   To reexamine the use of NaOH as a catalyst in the production of biodiesel, by using an alternative material (wood ash waste), to develop the technique of acid/base titration and to introduce the principles of green chemistry to students.

GREEN CHEMISTRY PRINCIPLES:
• Prevent waste: Waste wood ash is still useful for the extraction of base; left over wood ash can be recycled/composted, and base isolated can be used in further preparation of biodiesel

• Use renewable feedstocks: used wood ash as a base that can be used with another renewable feedstock (vegetable oil) to produce biodiesel
• Use safer solvents and reaction condition: no organic solvents are used
• Increases energy efficiency: experiment was run at ambient temperature and pressure

OBJECTIVES:
• To learn how to perform extractions
• To learn how to perform acid/base titrations
• To introduce students to the principles of green chemistry

MATERIALS
• wood ash   • potassium hydrogen phthalate (KHP)
• deionized water   • thymolphthalein indicator
• (2) 250mL beakers   • 250mL volumetric flask
• 50mL Erlenmeyer flask   • fluted filter paper (15cm diameter)
• 50mL burette   • balance   • funnel

ADVANCE PREPARATION
Finding wood ash: You can get wood ash from your fireplace, a pizza parlor that uses a wood oven, a fireplace store or a store that sell wood stoves. It is necessary to strain the wood ash with a colander to remove bits of charcoal, foreign matter, etc. It is recommended that the ash be strained twice. It is advisable to do this outdoors since a great deal of dust is usually produced. Use protective equipment to avoid exposure to dust (mask). The grey/white ash is what is needed – black charred wood should be kept to a minimum. If you choose to use wood ash from a fire pit make sure that it is dry and has never been rained on or wet.

Thymolphthalein indicator preparation: Dissolve 1g solid thymolphthalein in 50mL of 100% ethanol, then dilute with 50mL deionized water.

KHP is HOCOC6H4COOK; FW = 204.23 g/mole
NaOH; FW = 40.00 g/mole

Useful Formulas
MOLES KHP = MASS KHP ÷ FW KHP

Volume base (L) x Molarity base = Moles of base

At the titration endpoint:

Moles base = Moles acid

Moles base = Moles KHP

Procedure:

Preparation of ash water (base):
1. Put a small beaker on the balance, then place an opened fluted filter paper into the empty beaker, and zero the balance.

2. Scoop about 50g of wood ash into the filter. Accurately record the mass of the wood ash.

3. Transfer the wood ash from the filter paper to a 400mL beaker. Do this gently to avoid making a lot of dust. Don't worry if a little of the ash sticks to the filter paper.

4. Add 150mL deionized water to the ashes in the beaker. Do this slowly to avoid making a lot of dust. Stir the resulting slurry with a glass stirring rod for about 1 minute.

5. Suspend funnel over 300mL Erlenmeyer flask, placing the filter paper used in the earlier step inside the funnel.

6. Stir the ash water beaker well and pour as much as possible into the funnel without overflowing the filter paper. Don't worry if a little bit of ash gets into the filtered water. Wait until some of the liquid drains through the funnel then stir the slurry again and pour more into the funnel. Repeat until all of the slurry has been poured into the filter.

7. At this point it is likely that some of the ash is still in the beaker. If so, scrape as much as possible into the filter.

8. Obtain an additional 100mL deionized water and use it in several small portions to rinse the residue from the beaker into the filter.

9. After the dripping stops and no liquid is visible in the ashes in the filter, remove the filter paper and discard the used ashes as directed by your instructor.

10. Obtain a new fluted filter paper and filter the ash water from the Erlenmeyer flask into a 250mL volumetric flask. Add deionized water as needed to bring the 250mL volumetric flask to its mark. The ash water is now ready for titration.

Titration of ash water

1. Label two 50mL Erlenmeyer flasks A and B.

2. Zero weighing paper, and obtain approximately 70 to 80mg (0.070 to 0.080g) of potassium hydrogen phthalate (KHP) acid.

3. Record the exact mass of the KHP.

4. Transfer to Erlenmeyer A.

5. Repeat steps 2 and 3 and transfer to Erlenmeyer B.

6. Add about 10mL deionized water to the KHP in each flask and swirl to dissolve the solid.

7. Add 2 drops of thymolphthalein indicator to the acid solution in each flask, set aside.

8. Fill a 50mL buret with filtered ash water, taking care to remove the air bubble from the tip.

9. Place a white sheet of paper under the 50mL flask containing the acid so you can clearly see the color change.

10. Titrate the KHP solution with filter ash water quickly with flask A to estimate the endpoint of the reaction

11. Repeat the titration slowly with flask B to get an exact reading, to a light blue end point.

12. Calculate the molarity of the ash water base, using the fact that at the titration endpoint the moles of acid equals moles of base.

Data:

1. Mass ash = _____ g

2. Mass KHP = _____ g

3. Initial buret volume = _____ mL

4. Final buret volume = _____ mL

5. Volume base = _____ mL = _____ L

6. Moles KHP = Mass KHP / FW KHP = _____ moles

7. Titration results yield molarity of base in ash water

At titration endpoint:

Moles base = Moles acid, giving:

Moles base = Moles KHP, giving:

Volume base (L) x Molarity base = Moles KHP, giving:

(note: L, not mL)

Molarity base = Moles KHP / Volume base (L) = _____ molar

8. NaOH equivalence

Moles base = Molarity base x Volume ash water (L) = _____ moles

Recall that the volume of ash water is 250 mL, 0.25L

Mass base equivalent = Moles base x FW (NaOH) = _____ g NaOH equiv.

9. Ash to Base equivalence

Mass base equivalent / Mass ash = _____ g NaOH equivalent per g ash

Experiment (7):A Green Precipitation Reaction[1]

In this lab, students will determine the percent composition of zinc chloride by precipitation of zinc carbonate, in addition to other quantitative calculations.

$$ZnCl_2 + Na_2CO_3 \rightarrow ZnCO_3 + NaCl$$
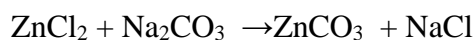
Safety information: Sodium carbonate is a skin irritant and should be handled with gloves. If any does come in contact with skin wash with soap and water.

Educational Goal: Students will understand…
 • How to quantitatively analyze a green precipitation reaction
 • Vocabulary and math concepts for percent composition, limiting reactant, and percent yield.

Student Objectives: Students will…
• Understand data quantitatively and qualitatively.
• Use data to determine limiting reactant and percent yield.
• Practice lab safety.

Materials:

• 1.0 g of sodium carbonate	• 15 mL of 1M zinc chloride
• 25-mL graduated cylinder	• 50-mL beaker
• 50-mL Erlenmeyer flask	• funnel
• balance • weigh paper	• filter paper

Green Chemistry Principles Addressed:
 Pollution Prevention
Less Hazardous Chemical Syntheses
Inherently Safer Chemicals for Accident Prevention.

Procedure:
1. Weigh out 1.0 g of sodium carbonate (Na2CO3) in to a 50 mL beaker. Record the actual weight.
   Mass of sodium carbonate (Na2CO3) : _____

   2. Add 15 mL of water to the 50 mL beaker to dissolve the solid to make an aqueous solution of sodium carbonate.

    3. Calculate:
   a. Given 1.0 g of sodium carbonate, how many grams of zinc chloride (ZnCl2) will be needed to complete the reaction?

   b. Now that you know how many grams you will need, what is the minimum volume of 1.0M zinc chloride that you will need to react completely?
   1. https://studylib.net/doc/7613807/doc---beyond-benign

4. Multiply the calculated volume in 3.b. by 1.5.
     Excess volume of 1.0 M zinc chloride ($ZnCl_2$) : _____
   5. Measure and add calculated amount of 1.0 M zinc chloride solution to sodium carbonate solution.
5. weigh the filter paper. Record weight on the Quantitative Data table.
6. Filter your products to obtain the precipitate. Allow to dry over night.
7. The following day, weigh the precipitate on the filter paper. Record weight on the Quantitative Data table.

Quantitative Data:

Object	Mass (g)
Sodium carbonate sample	
Filter paper	
Filter paper + zinc carbonate	
Zinc carbonate	

Analysis:
1. Based on your results, calculate the percent composition of carbonate in sodium carbonate . How do your results compare to the known formula?

2. Identify the limiting reactant for the reaction.

3. Determine the percent yield of zinc carbonate for the reaction .

 4. Calculate the atom economy of zinc carbonate for this reaction.

Experiment (8):Equilibrium/ Le Chatelier's Principle[1]

Objective of lesson: To give students an understanding of the concept of chemical equilibrium and to demonstrate Le Chatelier's Principle, i.e. if a stress is applied to a system at equilibrium, the system re-adjusts to relieve the stress applied. Part I is do be done as a demo and discussed in class as an introduction to Le Chatelier's Principle and the concept of equilibrium.

Chemical equilibrium is a state of dynamic balance where the rate of the forward reaction is the same as the rate of the backward reaction.

**Learning Outcomes:** At the end of this lesson, students will be able to:
 • Explain the concept of chemical equilibrium.
• Distinguish between static and dynamic equilibrium.
• State Le Chatelier's Principle.
• Describe how to set up an experiment that is at chemical equilibrium.
• Predict the effect of adding a stress to the system at equilibrium.

Materials required:
• Candle   • 1 balloon
 • Soda water   • 5 Erlenmeyer Flasks
   • Black tea   • Vinegar
• Ammonia – cleaning solution   • Tincture of iodine
 • Starch • Universal indicator   • Drinking straw.
• Hot plate   • Ice bath   • Glass stirring rods

ACTIVITY 1:
• Introduce to class idea of bottle of soda water. Does equilibrium exist inside the bottle?
• What gas is present in the bottle (dissolved and above the solution)?
• Remove gas from bottle by shaking and then trapping the gas in the balloon.
. Test for gas by pouring some of gas over lighted candle . What can we deduce?
 • Bubble gas into litmus indicator or cabbage juice (or better limewater). The gas is acidic (or turns limewater milky)– what is name of acid in the soda water?
• Discuss equilibrium inside bottle

$$CO_2 + H_2O \leftrightarrow H_2CO_3$$
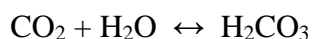
Problem: How can Le Chatelier's principle be used to predict the direction in which a system at equilibrium will shift when conditions are altered?

 Materials: Tincture of Iodine, starch solution, black tea solution, vinegar, ammonia cleaning solution water, test tube rack, 6 test tubes, 250 ml beakers (2), hot water bath and cold water bath.


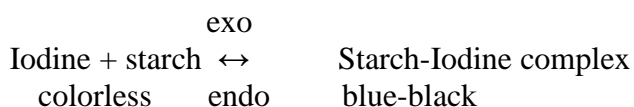1. https://studylib.net/doc/7332010/chapter-17-chemical-equilibrium

ACTIVITY 2:

1. Add a few drops of tincture of iodine to about 10mL of starch solution to each of the three test tubes. Make observations.
2. Heat one the test tube solutions to about 800 C and note the color in your chart.
3. Cool one the other test tubes by placing it in an ice-water bath.
4. Fill in the data table below with the observed color of the solution after each stress is added. The control group is the tube which is not stressed and the color of all stressed tubes can be compared to it.

Data:

Stress	Resulting Color
Control	
Raise temperature	
Lower temperature	

$$\text{Iodine + starch} \quad \leftrightarrow \quad \text{Starch-Iodine complex}$$
$$\text{Colorless} \qquad \text{blue-black}$$
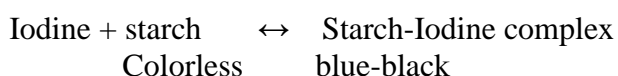
Which direction is exothermic and which is endothermic? How do we explain our results?

$$\text{exo}$$
$$\text{Iodine + starch} \quad \leftrightarrow \qquad \text{Starch-Iodine complex}$$
$$\text{colorless} \quad \text{endo} \qquad \text{blue-black}$$

ACTIVITY 3:

1. Place some black tea solution in three 50mL Erlenmeyer flasks. One of these will be used as a control.
2. To one of the flasks, add a few drops of vinegar. Note the change in color.
3. To another flask, add a few drops of ammonia cleaning solution. Note change in color.
4. Fill in the data table below with the observed color of the solution after each stress is added.

Data:

Stress	Resulting Color
Control	
Vinegar addition	
Ammonia addition	

$$\text{Tea} + H^+ \quad \leftrightarrow \text{TeaH}^+$$
$$\text{Dark} \qquad \text{light color}$$
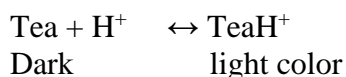
Conclusion Questions:

1. In Activity 2, what affect did heating the test tube have on the concentration of Starch-Iodine complex? Explain how you know this by using Le Chatelier's Principle.

2. What affect did cooling the test tube have on the concentration of Starch-Iodine complex? Explain how you know this by using Le Chatelier's Principle.

2. Which direction is exothermic _____.

Which is endothermic?_____ How do you explain your results?

4. In Activity 3 how could you determine whether or not a change occurred in equilibrium? Explain.

5. For each reaction in Activity 3, explain how each change can be explained by Le Chatelier's Principle. Be specific about what chemical was added that was part of your equilibrium system and discuss shifting of equilibrium.

6. Did these activities help you to understand Le Chatelier's Principle? Why or why not?

7. Which 12 Principles of Green Chemistry were used in this lab? What else could have been done to "green" up this lab

Experiment (9):Chemical or Physical Reaction[1]

Students will understand the difference in chemical reactions vs. physical reactions using all green materials.

Safety information:
Acetic acid, although very dilute in this lab, is a skin irritant and if it comes into contact with the skin should be washed with soap and water. Sodium bicarbonate solution, also very dilute, is a skin irritant, and if it comes into contact with the skin should be washed with soap and water. The sparkler is going to become hot and spray sparks, do NOT light it near anything flammable, hold at arm's-length from body.

Educational Goal: Students will understand …
• How to make observations
• That inferences then lead to conclusions
• The difference between an observation and the interpretation of the observation
• What makes a chemical reaction a chemical reaction
• What makes a physical reaction a physical reaction

Student Objectives: Students will …
• Use basic lab procedures
• Makes observations
• Make inferences from the observations
. Understand and be able to identify the difference between a chemical and physical change
• Practice lab safety

Materials
• 3% Hydrogen peroxide   • 0.1 M Na_2CO_3 (1mL)
• 0.1 M $CaCl_2$ (1mL)   • 1.0 M $NaHCO_3$ (1mL)
• $CaCl_2$ (s) (0.5g)   • Urea (s) (0.5g)
• Yeast (0.1g)   • Citric acid
• Vinegar   • Lemon juice (1mL)
• Cabbage Juice Indicator   • Steel wool
Ice
• 5 Test tubes   • 2 Well plates
• 2 50-mL beakers   • 1 100-mL Beaker
• 6 thermometers   • 4 spatulas
• 4 Stirring rods   • Plastic Pipettes
• 2 Hot plates   • 4 balances
• Bunsen Burner   • Striker

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1. https://www.beyondbenign.org/lessons/chemical-or-physical/

Green Chemistry Principles Addressed:
Pollution Prevention , Atom Economy , Less Hazardous Chemical Syntheses , Designing Safer Chemicals , Safer Solvents and Auxiliaries , Use of Renewable Feedstock , Design for Degradation and Inherently Safer Chemistry for Accident Prevention

Teacher Prep: Teacher should prepare …
1-   0.1 M solution of calcium chloride ($CaCl_2$ FW=110.98g/mol anhydrous)
Hint: To prepare 50 mL of 0.1 M solution of calcium chloride, dissolve 0.56 g $CaCl_2$ in 50 mL of water.

2-   0.1M solution of sodium carbonate (Na_2CO_3 FW=105.99g/mol anhydrous)
Hint: To prepare 50 mL of 0.1 M solution of sodium carbonate, dissolve 0.53 g Na_2CO_3 in 50 mL of water.

3-   1.0 M solution of sodium bicarbonate ($NaHCO_3$ FW= 84.01g/mol)
Hint: To prepare 50 mL of 1.0 M solution of sodium bicarbonate, dissolve 4.2 g $NaHCO_3$ in 50 mL of water.

4-   Cabbage Juice Indicator

• Chop the cabbage into small pieces until you have about 2 cups of chopped cabbage. Boil for at least ten minutes to allow time for the color to leach out of the cabbage. (Alternatively, you can place about 2 cups of cabbage in a blender, cover it with boiling water, and blend it.)

 • Filter out the plant material to obtain a red-purple-bluish colored liquid. This liquid is at about pH 7. (The exact color you get depends on the pH of the water.)

• Dilute about 100x until the juice is a light purple, it makes the changes in color easier to distinguish.
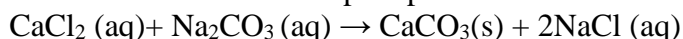
Procedure:

11- Reactions (Stations)

 1. Students will mix Hydrogen peroxide and yeast – reaction will produce a gas and the temperature should increase

$$2 H_2O_2 (aq) + yeast \rightarrow 2 H_2O (l) + O_2(g) + heat$$
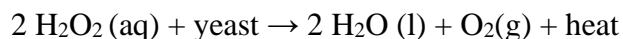
 2. Calcium Chloride and Sodium carbonate- Students will mix the two prepared solutions to see the formation of white precipitate
$$CaCl_2 (aq)+ Na_2CO_3 (aq) \rightarrow CaCO_3(s) + 2NaCl (aq)$$

 3. Lemon Juice and Sodium Bicarbonate and an acid base indicator/ cabbage juice- color change - Students will add lemon juice to cabbage juice and observe the color change and then will add sodium bicarbonate solution to observe a second color change

Lemon juice (aq) +cabbage juice indicator → purple to red color change

Acidic solution with cabbage juice indicator + 1.0 M $NaHCO_3$ (aq) → red to purple to blue color change

4. Citric acid solution and baking soda- Students will observe the formation of a gas and the temperature will decrease

   $C_6H_8O_7$ (aq) +3 $NaHCO_3$ (s) + heat → $3CO_2$ (g) + $3H_2O$ (l) + $Na_3C_6H_5O_7$ (aq)
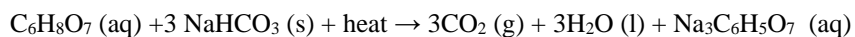
5. Boiling water- Students will boil water to observe the formation of a "gas" and an increase in temperature
   H_2O (l) + heat → H_2O (g)

6. Calcium Chloride and water- Students will observe an increase in temperature
   $CaCl_2$ (aq) + H_2O (l) → Ca^{2+} (aq) + $2Cl^-$ (aq) + H_2O (l) (exothermic)

7. Urea and water- Students will mix urea and water to observe a decrease in temperature
   $(NH_2)_2CO$(s) + H_2O (l)→ $(NH_2)_2CO$(aq) + H_2O(l) (endothermic)
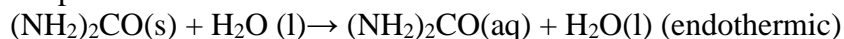
8. Steel wool and vinegar- Students will place a piece of steel wool in a beaker and saturate it with vinegar. Students will observe a change in color on the steel wool as well as a temperature increases. A gas will also most likely be produced.
   $3Fe$ (s) + $2CH_3COOH$ (aq) + O_2 (g) → $2FeO$ (s) + $Fe(CH_3COO)_2$ (aq)+H_2 (g) + heat

Procedure and Data:

Hydrogen Peroxide and Yeast

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. In a test tube add 40 drops of hydrogen peroxide. 2. Record the temperature. 3. Mass 0.1 grams of yeast and add to the test tube with the hydrogen peroxide. 4. Observe the reaction. Record the temperature. 5. Pour solution down the drain when finished and rinse test tube for the next group		

Sodium Carbonate and Calcium Chloride

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. In a well plate or test tube add 10 drops of sodium carbonate solution 2. Add 10 drops of calcium chloride solution 3. Swirl the solutions 4. Observe results 5. Rinse materials for the next group		

Lemon Juice and Sodium Bicarbonate -Indicator

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. Add 1-2 drops of cabbage juice into a well plate 2. Add 3-5 drops of lemon juice into the well plate with the cabbage juice 3. Observe 4. Add 3-5 drops of 1M sodium bicarbonate to the same well 5. Observe 6. Clear well plate for the next group		

Citric Acid and Baking Soda

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. Mass 0.3 grams of citric acid (or 0.5g of pixie stick) and place in a test tube 2. Add 20 drops of 1M $NaHCO_3$ 3. Observe the reaction. Record the temperature. 4. Pour solution down the drain when finished and rinse test tube for the next group		

Boiling Water

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. Add 20 ml of water to the empty beaker at the station. Record the temperature. 2. Place beaker on the hot plate CAUTION- Hot plate will be warm from previous group. 3. Increase the heat of the hot plate		

until the water in the beaker is boiling		
4. Record the temperature		
5. Observe		
6. Empty the beaker for the next group and remove it from the hot plate		

Calcium Chloride and Water

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. In a test tube add 0.5 grams of calcium chloride 2. Add 3 ml of water to the test tube 3. Record the temperature 4. Observe 5. Rinse the test tube for the next group		

Urea and Water

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. In a test tube add 0.5 grams of urea 2. Add 3 ml of water to the test tube 3. Record the temperature 4. Observe 5. Rinse the test tube for the next group		

Steel Wool and Vinegar

Steps	Observations	Questions/Conclusions Chemical or Physical?
1. Place a quarter size mound of steel wool into the beaker. 2. Submerge the steel wool in vinegar 3. Place a thermometer in the beaker and seal the beaker with parafilm 4. Record the temperature multiple times 5. Observe 6. Place steel wool in the garbage and rinse the beaker for the next group		

Question

1-The experiments you conducted produced either a chemical or physical change- place the experiments in the proper column

        Physical Change   Chemical Chang

Experiment (10):Catalysts and Oxygen[1]

Replacement lab: this lab replaces MnO_2 Manganese Dioxide catalytic reaction.

Goal: To demonstrate the effect of a catalyst on a chemical

Objectives: Students will...
• explain the concept of a catalyst and reaction rates
• understand how a catalyst can improve the efficiency of a process
• recognize that a chemical reaction involves reactants and products which may differ from each other
• recognize that the products of the reaction will be benign
• practice safe laboratory procedures

Materials:
• 1 x 250 ml beaker   • tap water
• thermometer (capable to reading 60˚C)   • hot plate
• 2 x test tube (25 x 100 mm size works best)
 • 10 ml graduated cylinder • Green food coloring
• Biodegradable liquid dish detergent (7th generation works well)
• 3% hydrogen peroxide (H_2O_2)
 • vitamin C tablets (equal to 3.40 grams of crushed vitamin C)
 • mortar and pestle

Lab Procedure:
1. Fill the 250 ml beaker halfway with tap water. Place thermometer inside beaker.
2. Place beaker on hot plate, and heat the water so that it maintains a temperature of 60˚C.
 3. Measure 10 ml of 3% H2O2 using the 10 ml graduated cylinder. Transfer the H2O2 to one test tube. Label test tube "Test tube A".
 4. Add 2 drops of food coloring to test tube A. Mix well.
5. Add 2 drops of biodegradeable liquid dish detergent to test tube A. Mix well.
6. Measure 10 ml of 3% H2O2 using the 10 ml graduated cylinder. Transfer the H2O2 to one test tube. Label test tube "Test tube B".
7. Add 2 drops of food coloring to test tube B. Mix well.
8. Add 2 drops of biodegradeable liquid dish detergent to test tube B. Mix well.
9. Using the mortar and pestle, crush enough vitamin C tablets to obtain 3.40 grams of it.
10. Add the 3.40 grams of crushed vitamin C into test tube B.
11. Place both test tubes into the water beaker (do not get any water into the test tubes).
12. Fill data in table in 2 minute intervals, starting at 0 minutes (before test tubes are placed into water bath).
13. Allow test tubes to sit in water bath for 10 minutes.
14. Remove test tubes and allow to cool in a test tube rack.
15. Turn off hot plate and clean area up

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1. http://www.uft.org/files/attachments/green-chemistry-handout.pdf

Student Data:

Time (min)	Water bath temperature (˚C)	Test Tube	Bubble formation in liquid visible Y/N	Foam formation Y/N	Color of liquid
0			A		
			B		
2			A		
			B		
4			A		
			B		
6			A		
			B		
8			A		
			B		
10			A		
			B		

Questions:

1. A chemical reaction is….

2. The chemical equation for the reactions in both test tubes is:
   What are the reactant(s)?
   What are the product(s)?
   Name the type of chemical reaction that occurs.
   Write a balanced equation for the reaction.

3. Which test tube had a catalyst? How do you know?

4. Name the catalyst used in this experiment.

5. What is the role of the catalyst?

6. How does using a catalyst improve the efficiency of a process?

7. Identify the hazards and the necessary safety procedures for this experiment.