



Lab 3: Stoichiometry I

Stoichiometry of Chemical Reactions

Procedure Overview

- Fill a trough with water, immerse the graduated cylinder to fill it with water, invert the cylinder, and clamp it in place
- Obtain hydrogen peroxide in a clean, dry beaker
- Use the auto-dispenser in the hood to deliver bleach to Erlenmeyer flask
- Place the vial on the balance and press Tare to “zero” the mass reading
- Deliver desired mass of H_2O_2 to the vial (according to the table provided in the procedure) – record the mass exactly as it appears on the balance
- Using tweezers, carefully place the vial upright in the Erlenmeyer flask
- Complete the setup by firmly stoppering the flask and placing the other end of the rubber tubing into the mouth of the inverted graduated cylinder (someone should hold it in place so it doesn't fall out)
- Gently tilt the Erlenmeyer flask to the side, tipping the vial over, and gently swirl the contents of the flask to completely mix
- When bubble formation has stopped, record the volume of water displaced in the graduated cylinder
- Repeat these steps to fill out the table and sketch a plot of “Volume of O_2 ” vs. “Mass of H_2O_2 added” in your notebook and make sure you have enough good data values before moving on to part II
- Use the digital pipettor to measure the mass of 500 μL of bleach solution – repeat four times

- Wash the volumetric flask and rinse it well with DI water
- Use the auto-dispenser in the hood to deliver 1, 10-phenanthroline (or phen) solution to the flask – record the concentration of this stock solution
- Add DI water to the phen solution, diluting it to the mark on the neck of the flask
- Transfer the diluted phen solution to a clean, dry beaker
- Use the digital pipettor to deliver 100 μL of Fe^{2+} solution to the phen solution in the beaker
- Mix well and transfer a portion of the solution to the cuvette (note detailed instructions)



- Place the filled and capped cuvette in the colorimeter (note detailed instructions)
- Record the absorbance reading once it stabilizes
- Pour the cuvette contents back into the beaker, add a second portion of Fe^{2+} , and repeat the mixing and measuring steps.
- Repeat the Fe^{2+} additions, mixing, and measuring until you have completed the table provided in the procedure

Materials

To be Checked Out from the Stockroom:

Note: In order to check out these items, it is required to leave your UW Student ID card and also a photo ID (i.e., driver's license) if your UW ID does not have a photo on it.

LabPro unit with AC outlet adapter
TI-83 graphing calculator with connector cable
Colorimeter
Digital pipettor

From your Lab Drawer:

50 mL volumetric flask
150 mL beakers (2)
250 mL beaker
100 mL graduated cylinder
250 mL Wide-mouth Erlenmeyer flask
Tweezers
Small vial (1 - 2 dram vial)
Rubber tubing
Rubber stopper (one-hole #8) with glass tubing
Cuvette
Glass stirring rod
Grease pencil

Provided in the Lab:

6% Bleach (NaOCl) solution
3% hydrogen peroxide (H_2O_2) solution
Iron (Fe^{2+}) solution
1,10-phenanthroline solution with automatic dispenser
Ringstand
Arm clamp
Water trough
Kimwipes
Plastic wash bottle of deionized water



Disposable graduated plastic 1-mL pipets
Plastic pipet tips for digital pipettor

Waste Disposal:

All products of the bleach/peroxide reaction must be placed in the waste container located in the hood.

$\text{Fe}(\text{phen})_n^{2+}$ solutions and the Fe^{2+} solution used in this experiment are non-toxic and therefore can be disposed of in the sink.

After rinsing, disposable plastic pipets and pipet tips should be placed in the trash.



Procedure

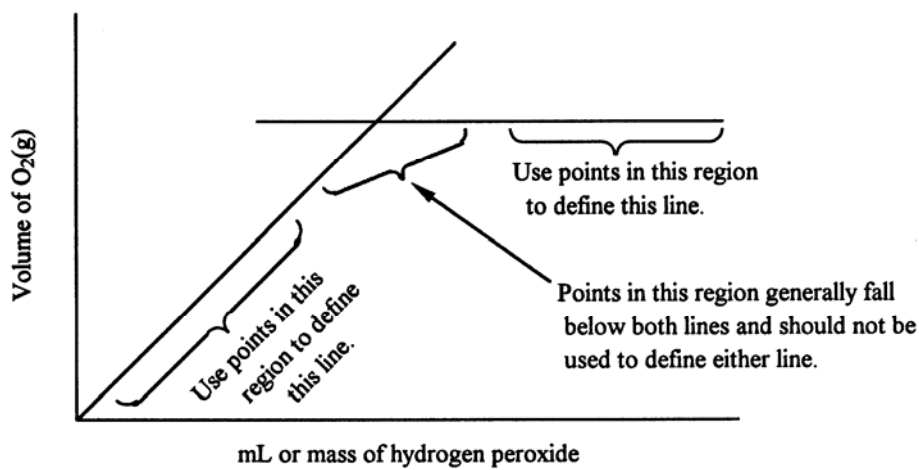
Part I. Hydrogen Peroxide and Bleach Stoichiometry

For this experiment you will prepare a set of reactions in which the volume of bleach remains constant (4 mL) while the mass of hydrogen peroxide is varied:

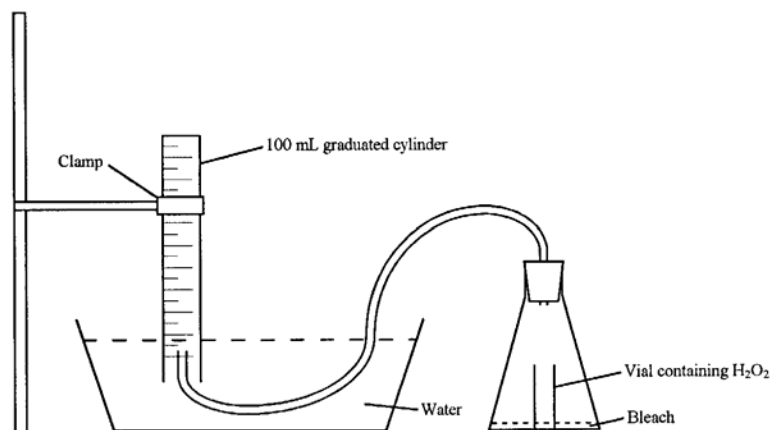
	Volume of Bleach	Mass of Hydrogen Peroxide
Run 1	4.0 mL	1.0 – 1.2 g
Run 2	4.0 mL	1.5 – 1.7 g
Run 3	4.0 mL	2.0 – 2.2 g
Run 4	4.0 mL	2.5 – 2.7g
Run 5	4.0 mL	3.0 – 3.2 g
Run 6	4.0 mL	3.5 – 3.7 g
Run 7	4.0 mL	4.0 – 4.2 g
Run 8	4.0 mL	4.5 – 4.7 g
Run 9	4.0 mL	See remark below
Run10	4.0 mL	See remark below
Run11	4.0 mL	See remark below

Remark:

Runs 1 to 8 may not adequately define the two lines required for the location of the equivalence point (see plot below). Use Runs 9, 10, 11,... to add more points in either of the two preferred regions. **Before proceeding with Runs 9, 10, 11, etc., plot your data and consult your TA.**



1. Fill a water trough with tap water. Completely immerse a 100 mL graduated cylinder (remove the plastic end from the cylinder) in the water trough, filling it with water. Turn the cylinder upside down, keeping the mouth below the surface of the water in the trough. Clamp the cylinder to a ringstand allowing room to slip a piece of rubber tubing into the mouth of the cylinder. The up-ended graduated cylinder should be full of water (NO AIR BUBBLES) and securely fastened to the ringstand. (See the figure below.) This is the oxygen-measuring vessel; gas formed by the reaction will bubble into the up-ended cylinder displacing some of the water. You can read the volume displaced directly from the graduations on the cylinder.



2. In a clean, dry 150 mL beaker obtain approximately 60 mL of hydrogen peroxide.
3. Your TA will demonstrate the use of the automatic dispenser that will be used to deliver 4.0 mL of bleach. Dispense 4.0 mL of bleach solution into the Erlenmeyer flask.



- Place the loose end of the rubber tubing into the mouth of the up-ended graduated cylinder. You may have to hold the tubing to prevent it from flopping around. Don't worry if a bubble or two escapes from the tubing into the cylinder.
- Place the vial on the balance and tare the balance (should read 0.000 g). Using a disposable pipet, add hydrogen peroxide to the vial until the mass is in the desired range. [If hydrogen peroxide is spilled onto the balance, immediately blot it up with a Kimwipe.] Record the exact mass weighed in your lab notebook. Use tweezers to lower the vial into the Erlenmeyer flask, taking care not to knock over the vial. Carefully stopper the flask with the rubber stopper; pushing it in firmly to form a good seal.
- Once the setup is complete, jiggle the reaction flask until the vial tips over and spills the hydrogen peroxide into the bleach. Swirl the flask to ensure complete mixing. **SWIRL GENTLY OR THE VIAL MAY BREAK!** Some reactions may finish rapidly, while others may take several minutes. Wait until the mixture in the flask stops fizzing and oxygen stops bubbling into the graduated cylinder, then record the amount of gas that was produced by reading the graduated cylinder.
- Empty the flask by pouring the reaction solution into your "*waste container*" (Use a 250 mL beaker to collect the waste from the various reactions. When your beaker is full, transfer the waste to the waste container in the hood). Rinse the flask and vial several times with water (the rinse water may be poured down the drain, it need not be put into the waste container); the flask and the vial do not have to be dry for subsequent runs.
- Repeat for all of the reactions listed above. **Note that it will be necessary to plot your data before performing reactions #9-11.** Any of the other runs for which the data points do not fall on either of the lines should be repeated. If in doubt, consult your TA.
- Use the digital pipettor (instructions below) to determine the density of the bleach solution. Dispense a 4.0 mL sample of bleach into a clean, dry beaker. (Note: Do not change the volume setting of the automatic dispensers.) Rinse a vial with deionized water and place it on the balance (the vial need not be dry). Press tare (the balance should read 0.000 g). Add 500 μL (0.500 mL) of bleach using the autopipettor. Record the mass of the bleach. Press tare. The balance should again read 0.000 g. Add a second 500 μL and record the mass. Repeat two more time so that you have four mass readings. Calculate the average density ($d = \text{mass(g)}/\text{volume(mL)}$). Dispose of all bleach used in this step in the waste container in the hood.

Instructions for the use of the digital pipettor:

- Attach a tip to the autopipet using a clockwise twisting motion.
- Holding the pipet vertically, press the operating button to the first stop. It MUST not be depressed all the way when filling the pipet.



- c. Immerse the tip below the surface of the bleach and slowly release the button to draw in the bleach. If any bleach adheres to the outside of the tip, touch the tip to the side of the beaker.
- d. Dispense the bleach by holding the pipet vertically and slowly pressing the button, making sure the tip is touching the wall of the vial. (This is most easily done by wrapping the vial in tissue and holding it; it should not be held with bare fingers.) After a brief pause, push the button to the last stop. Do not release the button until the pipet has been removed from the vial.
- e. The same tip may be used for the entire series of measurements. It should be removed after you have finished your density determinations. After rinsing with water, discard the used tip in the trashcan, not the waste container.

Part II: Iron (II) with 1,10-Phenanthroline Reaction Stoichiometry

1. Wash a 50 mL volumetric flask with soap and rinse several times with deionized water. Using the automatic dispenser in the hood, dispense 1.0 ml of the standard 1,10-phenanthroline (phen) solution into the 50.0 mL volumetric flask. **Record the concentration of this standard phen solution.** Add deionized water to the 50 mL mark.
2. Transfer this diluted phen solution to a clean dry 150 mL beaker.
3. The colorimeter/LabPro system is used to measure the absorbance of the $\text{Fe}(\text{phen})_n^{2+}$ that is formed as Fe(II) is added to the phen solution. Refer to **Appendix C** for instructions on the use of the LabPro system.
4. Pour deionized water into a clean, dry cuvette so that it is about 3/4 full. Cap the cuvette and place it in the colorimeter noting the proper orientation to the light path. Close the colorimeter lid and use the arrow keys to adjust the setting to 470nm. Push the **CAL** button and wait for the absorbance reading on the TI-83 screen to stabilize which should read 0. This is your first data point: 0 μL of Fe(II) added, absorbance 0.000.
5. Take the beaker containing the phenanthroline solution to the hood and dispense 100 μL (0.1 mL) of the Fe^{2+} solution into it following the instructions in Part I for the use of the digital pipettor. Mix the solution with a stirring rod.
6. Pour the solution into the cuvette so that it is about 3/4 full, then empty the cuvette back into the beaker. (This rinses out the previous solution.) Refill to about 3/4 full and cap the cuvette. Place it in the colorimeter noting the proper orientation to the light path and close the colorimeter lid.
7. When the reading has stabilized, record the absorbance displayed on the TI-83 screen and the total volume of Fe^{2+} solution added. The absorbance reading should be between 0 and 1.



8. Pour the solution in the cuvette back into the 150 mL beaker.
9. Repeat steps 5 through 8 until you have about the same number of points before and after the equivalence point. Make a rough plot of your data and discuss it with your TA before disassembling the LabPro unit.
10. Dispose of the titration products by washing them down the drain. To clean your cuvettes after use, rinse them with deionized water.

Before You Leave the Lab

1. Upon completing your work (includes all required calculations), show your notebook to your TA.
2. Clean your lab bench and have your TA check your equipment drawer, lab bench, and lab notebook.
3. Obtain your TA's signature in your lab notebook and turn in the duplicate pages of your lab notebook associated with this experiment.

Information to Enter in your Notebook during the Lab

- Actual masses of H_2O_2 for each trial
- Volume of O_2 generated for each trial
- Plot of volume of gas produced vs. mass of reagent added
- Estimation of g H_2O_2 at the equivalence point from the plot of data
- Mass of 500 μL bleach for each of 4 measurements
- Average mass, average density, and standard deviation of density for the bleach solution
- Calculation of moles of NaOCl in 4 mL bleach (6.0% (w/w) NaOCl)
- Calculation of moles of H_2O_2 at the equivalence point (H_2O_2 solution is 3% (w/w) H_2O_2)
- Calculation of mole ratio (moles H_2O_2 /moles NaOCl)

- Concentration of stock phen solution
- Plot of absorbance vs. volume (μL) of Fe(II) added
- Calculation of moles of phen in the 50 mL solution
- Estimation of μL of Fe(II) at the equivalence point from the plot of data
- Calculation of moles of Fe(II) at the equivalence point
- Calculate "n", the ratio of moles phen/moles Fe(II)

- Any other notes or observations that will help you remember what happened during the experiment (helpful when you work on your post-lab report or need to explain your results)