CHEMISTRY 344 - Organic Chemistry Laboratory II – Spring 2012
Lab #4: Green Chemistry Synthesis of Adipic Acid

In this lab, you will conduct a Green Chemistry synthesis of adipic acid, an important commodity chemical used in the synthesis of nylon. You will characterize your product by its melting point, then evaluate the process from a green chemistry standpoint.

Pre-lab Reading: You are expected to read the experimental procedure in this handout, and the following pages from your Solomons lecture text about Green Chemistry: 502, 595, 832, 835.

Pre-Lab Notebook Preparation: Complete the following information in your lab notebook before your pre-lab recitation. Use the numerals and headings as given.

Table of Contents: You will need to update the Table of Contents for each new lab with the date and brief title of each experiment, and the notebook page number on which the lab begins.

I. Title of the Experiment and Date.

II. Purpose of the experiment: One- or two-sentences describing what you are going to do and why. State your experimental goals.

III. Equations: Write the balanced equation for this reaction, ***including structures***.

IV. Table of Reagents: In your lab notebook create a table, like the one shown below, for the following reagents used in this lab: **cyclohexene, 30% hydrogen peroxide, sodium tungstate,** and **adipic acid.** Your table must include the name, structure, and all physical data. You can find this information in the Aldrich Chemical Catalog, the Merck Index, or on-line from the **ChemIndex** web site. The link to ChemIndex and instructions can be found on Dr. Brush's Lab web site: **http://webhost.bridgew.edu/ebrush/CH343%20Lab.htm**.

Chemical name and formula	structure	molecular mass (g/mole)	melting point (solids) (^{o}C)	boiling point (liquids) (^{o}C)	density (liquids) (g/mL)

V. Procedure: Summarize the lab procedure in your notebook. Do not copy the procedure word-for-word.

VI. Health, Environmental, and Waste disposal: Look up and summarize some of the health and environmental hazards for **cyclohexene** and **adipic acid** at the MSDS database: **http://hazard.com/msds/index.php**

***Laboratory Book*: The following should be completed before leaving lab, initialed and dated by you.**

VII. Data and Observations: Record a rough transcript of your experimental method in your lab notebook, indicating what you actually did and what you actually observed. Do not prepare this section in advance. This section should be written in a paragraph format and include: **experimental procedure, all reagent mass and volume measurements, observations, crude and pure product mass or volume, % yield calculations, and product analysis by melting point, chromatography analysis, or instrument analysis.** You will be judged on the depth of your observations and technical success (yields, purity, etc.).

VIII. Discussion and Conclusions: A brief, but critical evaluation of your results, and the success of your experiment. Address the experiments goals and purpose!

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Experimental Procedure

(1) Set your hotplate to a heating setting of 3.5. Place an Al block on top of your hotplate, insert a thermometer, and heat your Al block to a temperature of 100-110^{o}C.

(2) Obtain a clean, dry 25 mL, screw-capped vial, and add a special "large" magnetic stir bar. Take the vial (with stir bar) to the balances, and add the following reaction components IN THE ORDER GIVEN. You do not need to weigh the exact amounts, but you are expected to record the exact amount added:

- 0.25 g of sodium tungstate
- 0.25 g of Aliquat 336 (phase transfer catalyst). NOTE: this is a very viscous liquid, and you will need to be very patient with a small Pasteur pipette while adding this liquid to the vial
- 6.0 g (5.5 mL) of 30% hydrogen peroxide. CAUTION: strong oxidizer; will cause burns if contacts skin. Wear gloves when using this reagent.
- 0.19 g of potassium hydrogen sulfate ($KHSO_4$).
- Mix the components already added by swirling the vial for 10 seconds. Now add,
- 1.0 g of cyclohexene.

(3) Connect an air condenser to the vial, place in the Al block, and start stirring the reaction mixture. Slowly turn the stirring speed up to a setting of 8. Wait 10 seconds, then turn the stirring speed up to a setting of 10.

(4) Heat the light yellow mixture, vigorously stirring reaction mixture for 45 minutes at a temperature of 100-110^{o}C. **The temperature is very important and you must be in this range for the full 45 minutes.**

(5) After 45 minutes remove the vial from the Al block, and cool on your lab bench for 3 minutes. The yellow color should have dissipated, and the top cyclohexene layer should be gone. The reaction mixture should be a milky white color. Turn the heat setting on the hot plate to 3.

(6) Take a pipet and transfer the cloudy reaction mixture to a small beaker. The Aliquat 336 will appear as a liquid layer on top of the aqueous mixture, and stuck to the vial and stir bar. Avoid transferring the Aliquat 336 to the beaker.

(7) Cool the beaker in ice for 10 minutes. Your adipic acid product should precipitate as white crystals within 1 minute. If not, scratch the inside of the beaker for 30 seconds with a glass rod. If no adipic acid forms within 2 minutes of cooling and scratching, you will need to start over. Speak to your lab instructor.

(8) Isolate your crude adipic acid by filtration on a Buchner funnel. Rinse the inside of the beaker with 1-2 mL of ice-cold water. Leave the crude sample on the Buchner funnel for 5 minutes, and while you are waiting, clean and dry the 25 mL reaction vial, and determine its empty mass.

(9) After 5 minutes drying, scrape the crude, damp adipic acid into a pre-weighed 25 mL reaction vial and determine the mass of crude, wet product.

(10) **Recrystallization.** Add a stirrer to the vial, and an appropriate amount of deionized water (1 mL for every 0.9 g of crude product). Stir and heat the mixture at 80-100^{o}C. If the crude adipic acid does not dissolve in 5 minutes, add 5 drops of distilled water and heat for 2 minutes. Repeat until all adipic acid has dissolved.

(11) After all the adipic acid has dissolved, remove the vial from the Al block, and allow to cool slowly for 5-10 minutes on your lab bench. Once the recrystallized adipic acid begins to precipitate, place the vial in ice for 5 minutes, then isolate the product by filtration on a Buchner funnel, rinsing the vial with 1 mL of ice-cold water. Dry for 5 minutes, then scrape onto a clean watch glass and dry in an oven for 10-15 minutes (or longer if possible) at 115^{0}C. Cool to room temperature, and determine the mass of recrystallized product and its melting point.

Name:_____ **Lab Partner**_____

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Lab Report Grade _____(100 points)

The following questions are based on your pre-lab reading and the results of your experiment. You and your lab partner are encouraged to work together, but you must each turn in your own report! Answer each question in the space provided, and **show all calculation work below or on a separate sheet of paper**.

1) Mass of cyclohexene (limiting reagent)_____ (g)

2) moles of cyclohexene_____(moles)

3) Theoretical yield of adipic acid product (grams)_____(g)

4) Theoretical yield of adipic acid product (moles)_____(moles)

5) Experimental yield of crude adipic acid product (grams)_____(g)

6) Experimental yield of crude adipic acid product (moles)_____(moles)

7) Percent yield of crude product_____

8) Experimental yield of pure adipic acid product (grams)_____(g)

9) Experimental yield of pure adipic acid product (moles)_____(moles)

10) Percent yield of recrystallized product_____

11) Literature mp of adipic acid_____

12) Observed mp of recrystallized adipic acid product_____

(continued)

13) Based on the % yield of your crude and recrystallized product, and atom economy, discuss the efficiency of this process. A paragraph of several sentences is expected.

To answer the following questions you may need to refer to the assigned readings about green chemistry.

14) Green Chemistry Evaluation. What are some major goals behind Green Chemistry?

15) Atom Economy Calculation. Complete the following table by giving the chemical formula and molecular mass of each **reactant**. Reactants are **consumed** in the reaction and converted into the product.

Name of chemical substance	formula	molecular weight
Cyclohexene		
Hydrogen peroxide		

Atom Economy. The Atom Economy is the fraction of the atoms in the product to the fraction of all atoms in the reactants. You can calculate the Atom Economy for your reaction using this equation:

$$\textbf{Atom Efficiency} = \frac{\text{Molecular weight of the product}}{\text{Sum of the molecular weights of all reactants}} \times 100$$

Molecular weight of adipic acid_____

Sum of molecular weights of all reactants (from table)_____

Calculation_____

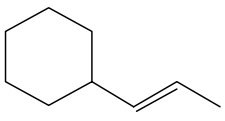
Atom Economy_____

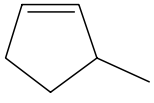
16) In your own words, define "Atom Economy". Do not just re-state the above equation. How does "atom economy" differ from "% yield"?

17) The traditional industrial method to produce adipic acid has an atom economy of about 30%. How does the atom economy of your reaction compare? Which process is more efficient and why?

18) The sodium tungstate serves as a catalyst in the synthesis of adipic acid. How does a catalyst contribute to making a chemical reaction or process "greener?"

19) Based on what you learned about this reaction, draw the structure of the organic product(s) for each of the following alkene oxidation reactions:

(a)  + H_2O_2 $\xrightarrow[\text{Aliquat 336}\\ 100^oC]{\text{tungstate catalyst}}$

(b)  + H_2O_2 $\xrightarrow[\text{Aliquat 336}\\ 100^oC]{\text{tungstate catalyst}}$