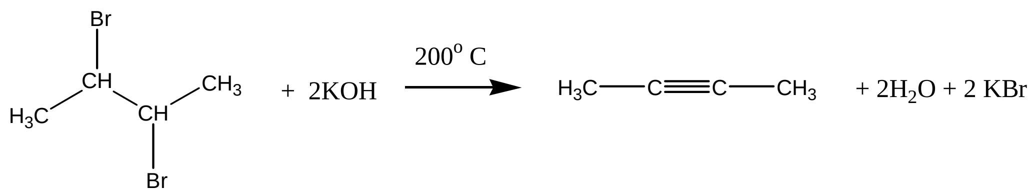
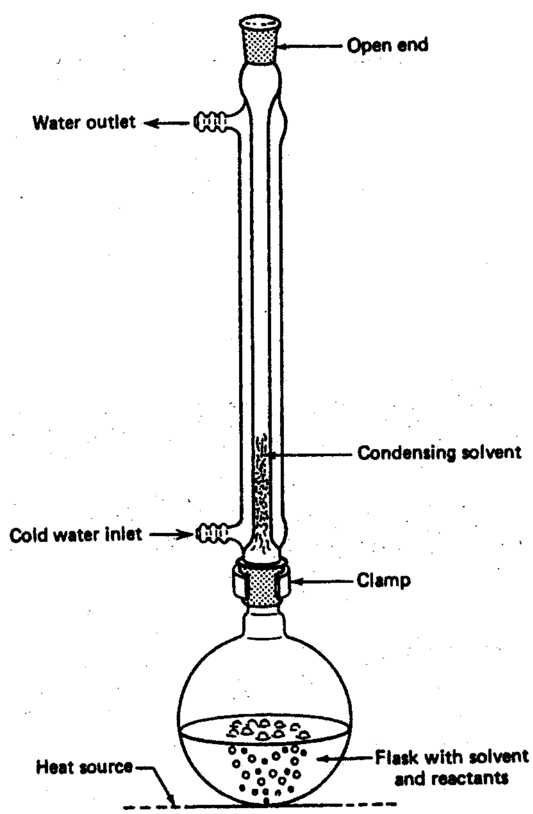
CHEM253 Experiment 10 **DEHYDROBROMINATION**

The dehydrobromination of dibromoalkanes is a general method to produce a double bond. By analogy, the sequential elimination of two molecules of hydrogen halide will lead to the formation of a triple bond as shown by the general reaction below.



The overall reaction equation represents a two-step reaction involving E2 mechanisms. The first elimination is rather facile since the optimal anti-periplanar transition state conformation can be easily attained. The second elimination is more energetic. This is why the reaction is done with concentrated strong base and elevated temperatures.

Many chemical reactions are done under reflux. By refluxing a reaction in a particular solvent, the reaction is kept at a constant temperature in a constant state of mixing. The boiling point of the solvent determines the reaction temperature. The solvent is boiled out of the reaction solution but quickly condenses and returns to the flask once the solvent vapors enter the reflux condenser. Chapter 22 of Zubrick addresses the topic of reflux.



A. Reaction and Isolation of Products

1. Add 3 grams of 1,2-dibromo-1,2-diphenylethane to a 100mL round bottom flask.

2. Add 1.5 g of solid potassium hydroxide (KOH) to the same round bottom. Solid KOH is caustic and should not be handled with bare hands.

3. Add 15 mL of 1,2-ethanediol (ethylene glycol) to the round bottom.

4. Add two boiling chips to the round bottom. Swirl and stir the reactants with a glass rod to mix. The solids will not totally dissolve at this time.

5. Attach the round bottom to a reflux condenser mounted on a ring stand and attach water hoses as shown in the preceding diagram

6. Use a thermwell mounted on an iron ring to heat the solution. Heat the mixture to boiling, and then adjust the heat input so that the boiling is maintained at a steady rate.

7. Reflux for 25 minutes. Note any changes in the reaction mixture.

Caution: Contents are hotter than a cup of McDonald's coffee!

8. Turn off the heat source. Transfer contents of round bottom to a small beaker while they are still hot. Rinse out the round bottom if some solid remains. Allow the solution to cool to room temperature.

9. Add 25 mL of water and chill in an ice bath. Make sure that you have a solid precipitate before you filter the solution.

10. Vacuum filter the solid (crude) product. You may wash it with a small amount of ice water.

B. Purification

1. Recrystalize the solid in a minimum amount of 95% ethanol. (Dissolve the solid in a minimum amount of hot ethanol and then cool in an ice bath.)

2. Vacuum filter the recrystalized product.

3. Determine the mass of your product.

C. Qualitative test:

Put a few crystals of your product in a small test tube.
Add five drops of 5% Br_2 in dichloromethane with shaking.
Heat the test tube in a hot water bath.
Note any color change. (It may take more than a few seconds.)

D. Verification of Identity and Purity

1. Do a comparative TLC of the starting material and product in 50% dichloromethane 50% hexane. The TLC should have three spots: a) starting material b) your product c) product standard

2. Dissolve a small sample in dichloromethane in a 2 mL vial for Gas Chromatography analysis.

3. Hand in a properly labeled sample of your product:

your name date lab title product identity

E. Clean up.

Dispose of filtrates in the proper container.

<u>Checklist for completing the "Prelab" section: </u>(refer to Laboratory Syllabus for complete directions)

____ *Title*.

____ *Purpose*.

Physical constants. Create a table of physical constants, solubility, and safety data for the chemical compounds referred to in the procedure:
http://domin.dom.edu/faculty/jbfriesen/chem253lab/soladvice.htm

Structures and equations.

1) Draw the structure of *meso*-1,2-dibromo-1,2-diphenylethane with proper stereochemistry.
2) Draw the structure of the reaction intermediate (alkene) with proper stereochemistry. (Section 9-8 in Wade)
3) Why is the second dehydrohalogenation is more energetic than the first?

http://www.usm.maine.edu/~newton/CHY252_254/252/text/U4/U4Intro.doc

___ *Flowchart.* Refer to "Procedure"

Calculations.

1) Calculate the number of moles in 3 grams of 1,2-dibromo-1,2-diphenylethane.
2) Calculate the number of moles of KOH in 1.5 grams.
3) Calculate the theoretical yield of 1,2-diphenylethyne.
4) Calculate the atom economy from the overall equation.
(see http://domin.dom.edu/faculty/jbfriesen/chem253lab/atom_economy.htm)

Safety Question: Solid KOH is considered to be caustic. What does “caustic” mean?

Experimental Observations and Data:

Hand in a copy of your experimental observations and data before you leave lab.

Experimental Observations: Refer to Laboratory Syllabus for guidelines.

___ Describe the appearance of each reagent you used.
___ Record masses or volumes of reagents used.
___ What were the noticeable signs that a reaction was taking place?
___ What was the reflux time? How did you time it?
___ What volume of ethanol was used for recrystallization?
___ What as the temperature of the ice bath?
___ How did product change after recrystallization?
___ Any interesting sites and smells?

Raw Data:

___ What data did you collect from parts C. and D.?

Source: "Experiments in Organic Chemistry" 3rd Edition
Richard K. Hill, emeritus University of Georgia
John Barbaro, Rocky Mountain College, formerly of University of Florida ISBN: 0-89892-311-5

Lab Report Checklist:
Results.

____ % yield of product → crude product mass x 100/theoretical yield. Show your work.

Discussion and Conclusion.

____ Give a specific example how the % yield could be increased.

____ What is going on in the Br_2 test? Include at least one chemical structure in your response.

____ Interpret the TLC and GC. Comment on the purity of your product.

____ Suggest one reason that diphenyl substituted substrate is preferable to an alkyl substituted substrate such as 3,4-dibromohexane? Include at least one chemical structure in your response.

____ Green Question: Ethylene glycol is a high volume chemical with production exceeding 1 million pounds annually in the U.S. According to its MSDS Airborne Exposure Limits, OSHA reports that the Permissible Exposure Limit (PEL) is 50 ppm. What exactly does the Permissible Exposure Limit mean in this context? Cite your source(s).