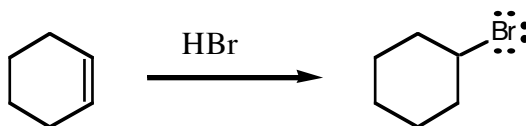


# Addition of HBr to Cyclohexene

## Introduction

In this reaction, we will be adding a concentrated HBr solution to an alkene to produce an alkyl halide. This reaction proceeds by an acid-base reaction between the alkene and HBr to produce a carbocation, followed by an association reaction between the carbocation and bromide ion.



Before coming to lab, please read all of the procedures given below. Write an introduction in your lab notebook. Fill out the following table and include it in your introduction.

Then review the following techniques: "Refluxing a Reaction," "Extraction and washing," "Drying and Organic Solvent," "Evaporating an Organic Solvent," and "Characterizing a Compound by IR."

reagent	volume	density	mass	MW	mmoles	eq
cyclohexene	0.75 ml					1.0
HBr 48%	2.5 ml	1.490 g/ml	soln: HBr:			
bromocyclohexane	x	x				

Concentrated hydrogen bromide is corrosive and toxic; please do not remove from the fume hood, and use care not to come in contact with the chemical.

## Procedure

Set-up and run the reaction:

- Add the cyclohexene to a conical vial using a graduated pipet.
- Add the HBr solution to the reaction using a graduated cylinder.
- Add a spin vane. (Note - if your spin vane has turned a dark color, it could color your reaction in this experiment. Don't panic - it still seems to work ok.)
- Add a reflux condenser, clamping the condenser in place, and set up the tubing.
- Turn on the water, then turn the stirring up until the two phases mix as much as possible without causing the spin vane to just vibrate (it will help if you are careful to center the conical vial on the stirrer-hot plate).

- Slowly heat the reaction until you can see it begin to reflux. It is not necessary to insulate the reaction with aluminum foil (that is only needed when doing a distillation or melting point determination).
- Heat at reflux for 1 hour (starting from the time you see it begin to reflux). If excessive foaming occurs, add a drop or two of Triton X-100. (You are ok as long as the bubbles don't get out of the top of the condenser.)

#### Isolation of the product:

- Allow the reaction to cool. When it nears room temperature, remove it from the reflux condenser, and remove the spin vane with forceps.
- Cool the vial for a minute in an ice bath (don't put a hot vial in the ice bath or it will crack!). You should be able to see a small layer of clear liquid on the top – this is the product, together with any unreacted cyclohexene.
- Add some diethyl ether to dissolve the organic compounds - mix it up by drawing it in and out of a plastic pipet a few times – two layers should form.
- With a pipet, carefully suck the aqueous layer (the bottom phase) out of the vial and into a separatory funnel (stopcock closed with beaker underneath!). Set the vial containing the top layer aside where it won't get spilled.
- Extract out any product that might remain in the water layer by adding several ml of fresh ether to the sep funnel and shaking carefully. Allow the layers to separate. Then drain off the water layer (bottom) into a beaker and set it aside for later disposal.
- Now add the ether layer from the reaction to the sep funnel, combining it with the ether layer already there. This should contain all of the product.
- Wash the ether layers to removing any remaining acid by adding a layer of sat'd  $\text{NaHCO}_3$  to the ether solution. Add it slowly, as there will be a lot of bubbling as the bicarb reacts with the acid. When the bubbling dies down, put in the stopper and shake it carefully, venting frequently. Drain off the aqueous (bottom) layer.
- Add fresh bicarb solution and repeat.
- Drain the ether layer into a 50 ml beaker and dry the solution over sodium sulfate. If the ether has evaporated so that you only have a small amount of solution, add more ether – if there is too little ether, it can be very hard to tell if it is free-flowing.
- Pour the dried ether solution into a clean, tared conical vial. Prepare a hot water bath in a small beaker at around  $60^\circ\text{C}$ . Evaporate the ether by setting the conical vial in the bath, with one end of your forceps inside to keep it from bumping. Don't fill it too full or it may boil over. If you have too much to fit in the conical vial, add some, boil it off, then add some more. When no more bubbles form and the level of liquid doesn't seem to be going down, all of the solvent should be gone.

Characterize the product:

- Note the appearance of your product; pure, authentic bromocyclohexane is a clear, colorless liquid.
- Once it is cool, obtain its mass and calculate the % yield.
- Take an IR of your product. Compare your spectrum to the IR of the starting material posted. Did the expected peaks appear (or disappear)? Mark the appropriate peaks on your IR and turn in your IR with the carbon copies of your lab.
- In your conclusion, discuss the appearance of the product, how much you obtained, the percent yield, and the IR that you obtained, as well as any comments you'd like to make.

Preparation for next week:

- Before you leave the lab, please label a clean conical vial and spin vane with your initials and put them in the drying oven - we will use these next week.

### Questions

- 1) Draw the mechanism for this reaction. Is the addition regioselective in this case? Why or why not?
- 2) Why was it necessary to reflux this reaction?
- 3) Why did the reaction have two layers?
- 4) What is the purpose of extracting the water layer with ether? (step 6)
- 5) What is the purpose of washing the ether layer with sodium bicarbonate (step 7)?
- 6) Write the reaction that takes place when sodium bicarbonate reacts with HBr. What causes the bubbling?
- 7) What bands on the IR tell you if the product was formed? How would you know if there was any starting material contaminating the product?