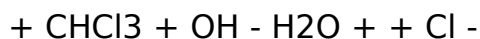


Carbene addition lab

[Environment](#), [Water](#)

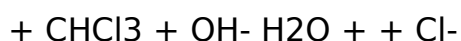


A solution of cyclohexene, aqueous sodium hydroxide, and benzyl triethylammonium were used to synthesize 7, 7 - dichloronorcarane by the following reaction:



After completion of the experiment, the percent recovery was calculated to be 46. 21% Introduction The purpose of this experiment was to form 7, 7-dichloronorcarane by means of carbene trapping.

Cyclohexene, aqueous sodium hydroxide, and benzyl triethyl ammonium chloride were used to synthesize 7, 7 - dichloronorcarane by the following reaction:



In order to carry out this reaction in a reasonable time, a two-phase reaction was used, avoiding the lengthy requirements to carry out the experiment as a single-phase reaction, as well as the risk of undesirable products due to the presence of water. The components of the two-stage reaction were as follows: An organic phase containing the alkene and CHCl_2 ; and an aqueous phase containing the base OH^- .

Since the reactants, CHCl_2 and OH^- would separate into different phases, benzyl triethylammonium chloride was added as a phase-transfer catalyst. Because the benzyl triethylammonium chloride has both hydrophilic and hydrophobic properties, it could cross the phase boundary and facilitated the transport of the hydroxide ion from the aqueous phase to the organic phase. Expected spectral changes in IR and ^{13}C -NMR Procedure To begin, 0. 324g of cyclohexene was mixed with 1 ml of 50% aqueous sodium hydroxide, 1ml

of chloroform and 0.040g of the phase transfer catalyst benzyl triethylammonium chloride.

The reaction was stirred and heated at 40 degrees Celsius for an hour before extracting the 7,7-dichloronorcaradiene through 2 methylene chloride extraction processes (1ml methylene chloride). The product was then air-dried until no more liquid would evaporate and weighed giving a percent recovery of 43%. The substance was then used to conduct an IR and ^{13}C -NMR spectra test giving the results.

Results and Calculations

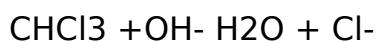
Calculating moles of cyclohexene
 $(81\text{g cyclohexene}/1\text{ml}) * (0.40\text{ml cyclohexene}) = 0.324\text{g cyclohexene}$
 $0.324\text{g cyclohexene} * (1\text{mol cyclohexene}/82.\text{g cyclohexene}) = 3.95 * 10^{-3}\text{ mol cyclohexene}$

Calculating moles of chloroform
 $1.49\text{g/ml chloroform} * 1\text{ml of chloroform} = 1.49\text{g of chloroform}$
 $1.49\text{g of chloroform} * (1\text{mol of chloroform}/119.4\text{g chloroform}) = 1.25 * 10^{-2}\text{ moles chloroform.}$

Calculating moles of benzyl triethyl ammonium chloride
 $(1.08\text{g benzyl triethylammonium chloride}/\text{ml}) * 0.040\text{ml of benzyl triethylammonium chloride} = 0.0432\text{ grams benzyl triethylammonium chloride} * 1\text{mol}/227.$

$8\text{g benzyl triethylammonium chloride} = 1.896 * 10^{-4}\text{ moles benzyl triethylammonium chloride}$

Finding the limiting reagent



(balanced as-is with a catalyst in rxn)

$3.95 * 10^{-3}\text{ mol cyclohexene}$
 $1.25 * 10^{-2}\text{ moles chloroform}$
 $0.058\text{ moles of sodium hydroxide}$

Cyclohexene has the lowest amount on the reactant side and is the limiting reagent.

Amount of 7,7-dichloronorcaradiene = $3.95 * 10^{-3}\text{ moles} * 165.06\text{g 7,7-dichloronorcaradiene}/\text{mol} = 0.652\text{g 7,7-dichloronorcaradiene}$

theoretical yield

Experimental yield of dichloronorcarane = test tube with – test tube without
(27.159 grams – 26.890 grams) = 0.269 grams dichloronorcarane. Percent recovery (Actual mass yield / theoretical mass yield) * 100% = percent recovery (0.69 grams / 0.652 grams) * 100% = 41.26% yield recovery
Formation of 7, 7 dichloronorcarane

+ CHCl_3 + OH^- H_2O + Cl^- .

Discussion and Conclusions Discussion Topics and Questions

- a. Compare IR and ^{13}C -NMR spectra of cyclohexene and 7, 7-dichloronorcarane and point out changes in IR vibrational frequencies and ^{13}C -NMR resonances that support the formation of 7, 7-dichloronorcarane. Be sure to identify the C-H stretch of the cyclopropane ring hydrogens.
- b. Why did you need to stir the mixture vigorously during the reaction? Since the reactants are in different phases, stirring vigorously allowed the catalyst to transfer a hydroxide ion from the aqueous phase to the organic phase, allowing the reaction to proceed as desired.
- c. Why did you wash the organic phase with saturated sodium chloride solution? Saturated sodium chloride was important for the removal of water. If the water was not removed from the solution, it could react and create undesirable reactions, such as those described in Pavia: (4) (5)
- d. Describe two chemical tests and the visual results that would indicate whether cyclohexene is present or absent. Bromine in methylene chloride – If the addition of bromine (red liquid) results in a colorless solution, cyclohexene is present. Potassium Permanganate

(Baeyer test) – This reaction depends on the color change from purple MnO_4^- ion into a brown precipitate of MnO_2 .

- e. Draw the structures of the products that you would expect from the reactions of cis and trans-2-butene. Be sure to comment on the stereochemistry of the products (enantiomers and meso forms).
- f. Provide a mechanism for the formation of the following products of a carbene addition reaction.