

Abstract

1-Phenylselenyl-1-alkynes easily prepared by deprotonation of the terminal alkynes with n-butyllithium at low temperatures readily undergoes hydroboration with dichloroborane-dimethly sulfide complex in *n*-pentane at 0°C for 2 hours in the presence of boron trichloride. The supernatant pentane layer was separated under a nitrogen atmosphere. It was then treated with 1,3-propandiol at 0°C and stirred for an hour. The resulting alpha-1-phenylselenyl-1-alkenylboronate esters are subjected to Suzuki coupling in the presence of potassium carbonate, dimethylformamide,

tetrakis(triphenylphosphine)palladium, and biphenyl magnesium bromide and the reaction mixture is refluxed for 3 hours. After worked up, the corresponding (Z)-trisubstituted alkenes containing phenylselenyl and biphenyl moieties are isolated in 72%-82% yields. The compounds are purified by column chromatography over alumina and the structures are confirmed by spectral data (PMR and CMR).

Introduction

(Z)-1-phenylselenyl-alkynes is prepared through the process of deprotonation of a terminal alkyne. Once the reaction goes through deprotonation it goes through hydroboration and then Suzuki coupling. In this report, the overall reaction is shown:



Highly Diastereoselective Synthesis of (Z)-Trisubstituted Alkenes Containing 1-phenylselenyl and biphenyl Moieties via Organoboranes Ariana Dela Garza and Narayan G. Bhat The University of Texas Rio Grande Valley, 1209 West University Drive, Edinburg, Texas 78539-2999 USA Email Address: ariana.delagarza01@utrgv.edu

Experimental Procedure

For the first step of this reaction the preparation of 1-Phenylselenyl-1-hexyzne. In an oven-dried 100 mL round bottom flask equipped with a magnetic stirring bar and septum inlet, 1hexyne (50 mmol, 4.11 mL) was reacted with n-butyllithium (20 mL, 2.5 M solution in n-hexane) at -78°C for 1 hour. To this reaction mixture, phenylselenyl chloride (50 mmol, 9.58 g) was added slowly via syringe while stirring and allowed to stir at -78°C for an additional hour. The reaction was then allowed to stir overnight at room temperature. The resulting product from the first step is then hydroborated with dichloroborane-dioxane complex (10 mmol, 1.15 mL) in the presence of boron trichloride (40 mL, 1 M solution in n-hexane) at 0°C for 2 hours. Water was added to the alkenyldichloroboroane at 0°C followed by the esterification with 1,3-propanediol (0.72 mL) producing (Z)-1-phenylselenyl-1-alkenyboronate ester. The resulting (Z)-1-phenylselenyl-1alkenylboronate ester is used in the final step. In a 100 mL round bottom flask with a side arm and a reflux condenser was added 5 mL of dimethyl formamide. (Z)-1-phenylselenyl-1hexenylboronate ester (5 mmol, 1.62g) 1 mmol of $Pd(PPh)_3$ (1.15g), potassium carbonate (5 mmol, 0.69g) and 4-bromobiohenyl (5 mmol). The reaction mixture is refluxed for 3 hours. After working it up, the corresponding (Z)-1-phenylselenyl-1-biphenyl-1-hexane is obtained in isolated yield.

Results & Discussion

This experiment is still in progress.



Figure 1: 1-hexyne reaction to produce 1-phenylselenyl-1-hexyne

Step 3:

Figure 3: (Z)-1-phenylselenyl-1-alkenylboronate ester subjected to Suzuki Coupling.







Summary & Conclusion

In summary, a simple was used to prepare (Z)-trisubstituted alkenes that hold 1-phenylselenyl and biphenyl. The experiment is currently in progress.

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