Electronic Supplementary Information

Hydrotelluration of Aminoalkynes

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Materials and Methods

Proton nuclear magnetic resonance spectra (¹H NMR) were obtained at 200 MHz on a Bruker DPX-200 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant (J) in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained either at 50 MHz on the Bruker DPX-200 NMR. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Column chromatography was performed using Merck Silica Gel (230-400 mesh) following the methods described by Still¹. Thin layer chromatography (TLC) was performed using Merck Silica Gel GF₂₅₄, 0.25 mm thickness. For visualization, TLC plates were either placed under ultraviolet light, or stained with iodine vapor, or acidic vanillin. Most reactions were monitored by TLC for disappearance of starting material. The following solvents were dried and purified by distillation from the reagents indicated: tetrahydrofuran from sodium with a benzophenone ketyl indicator; dichloromethane from calcium hydride; TMEDA from potassium hydroxide. All other solvents were ACS or HPLC grade unless otherwise noted. Air- and moisture-sensitive reactions were conducted in flame-dried or oven dried glassware equipped with tightly fitted rubber septa and under a positive atmosphere of dry nitrogen or argon. Reagents and solvents were handled using standard syringe techniques. Bath temperatures for reactions were maintained by adding dry ice to acetone (-78°C), acetonitrile (-42°C), or aqueous calcium chloride. Temperatures above room temperature were maintained by use of a mineral oil bath with an electrically heated coil connected to a Variac controller.

Typical procedure for hydrodelluratrion of aminoalkynes by BuTeTeBu/NaBH₄: To a solution of the dibutyl diteluride (0.18 g; 0.5 mmol) in absolute ethanol (5 mL) under argon and magnetic stirring was added NaBH₄ (0.057 g; 1.5 mmol) in small portions until the color of the solution turned from dark red to pale yellow. After the appropriate aminoalkynes (1 mmol) in ethanol (10 mL) was added. The mixture was then heated at reflux for 24 h After this time, the mixture was cooled to room temperature and treated with water

¹ Still, W.C.; Kahn, M.; Mitra, A. J. Org. Chem. 1975, 43, 2923-2925

(10 mL) and ethyl acetate (100 mL). The organic phase was separated and dried over MgSO₄ and concentrated under vacuum. The residue was purified by flash chromatography and eluted with hexane/ethyl acetate (80:20).

Typical procedure for hydrodelluratrion of aminoalkynes by [BuTeLi]: To a suspension of the elemental tellurium (0.127 g; 1 mmol) in dry THF (5 mL) under argon and magnetic stirring was added *n*-butyllithium (0.4 mL of a 2.5 M solution in hexane; 1 mmol). A yellow solution was formed. To this solution was added the appropriate aminoalkynes (1 mmol) in ethanol (10 mL). The mixture was then heated at reflux for 24 h After this time, the mixture was cooled to room tempearture and treated with NH₄Cl (10 mL) solution and ethyl acetate (100 mL). The organic phase was separated and dried over MgSO₄ and concentrated under vacuum. The residue was purified by flash chromatography and eluted with hexane/ethyl acetate (80:20).



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