Journal of Organometallic Chemistry, 386 (1990) 395–397
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands
JOM 20624

Synthesis of *trans*-*N,N,N*-triethyl-2-[4-(2-phenylethenyl)-phenoxy]-ethanammonium iodide

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(Received November 8th, 1989)

Abstract

The antispasmodic olefin *trans*-*N,N,N*-triethyl-2-[4-(2-phenylethenyl)phenoxy]-ethanammonium iodide has been made by hydroalumination of a trimethylsilylalkyne followed by palladium-catalyzed cross-coupling of the vinylalane with the appropriate aryl bromide.

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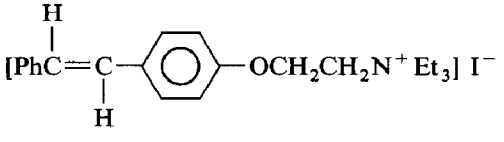
trans-*N,N,N*-Triethyl-2-[4-(2-phenylethenyl)phenoxy]-ethanammonium iodide (1) is a biologically active disubstituted olefin that is used as an antispasmodic ganglionic blocker [1,2].

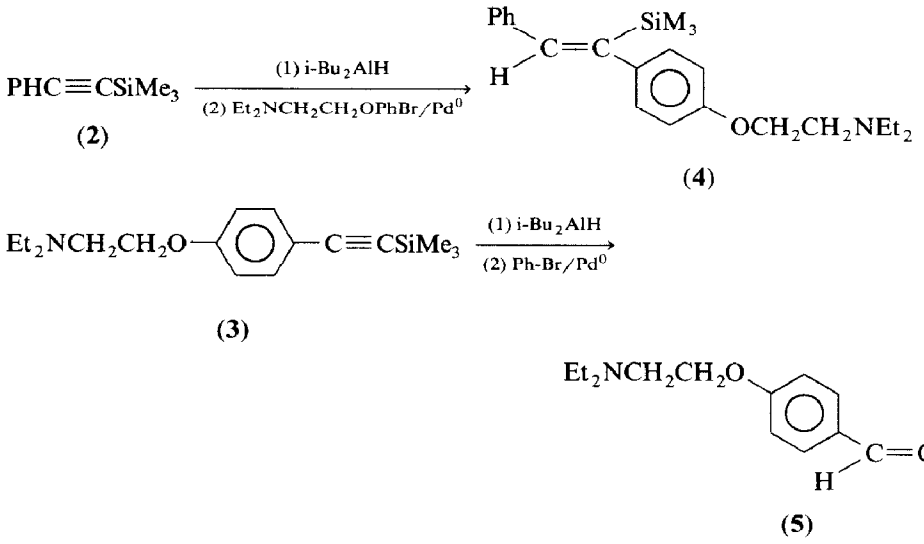
We describe here the synthesis of compound **1** by hydroalumination of a trimethylsilylalkyne followed by (i) palladium-catalyzed cross-coupling [3,4] of the vinylalane with the appropriate aryl bromide (ii) removal of the trimethylsilyl group and (iii) treatment of the desilylated olefin with ethyl iodide.



(1)

Thus trimethylsilylalkyne **2** or **3** was treated with diisobutylaluminium hydride in refluxing ether for 5 h to give the corresponding vinylalane [5] which was cross-coupled with bromobenzene [6] in the presence of catalytic amount (0.5 equiv.) of tetrakis-(triphenylphosphine)palladium [3,4] to give the vinylsilane **4** or **5**. The compounds **4** and **5** were transformed into the desired product (**1**) without further purification by protodesilylation with hydroiodic acid [7] followed by treatment of the desilylated species with ethyl iodide in refluxing tetrahydrofuran for 1 h. The desired product **1** was obtained in good overall yield after recrystallization from ethyl acetate/benzene.

0022-328X/90/$03.50



(62–67% overall yield)

Experimental

Melting points are uncorrected. GLC analysis was performed on PYE Unicam series 304 chromatograph with an OV1 on glass column.

Diisobutylaluminum hydride (Fluka) was used as a 1 *M* solution in hexane. Reaction mixtures were stirred magnetically under nitrogen. Oven-dried (160 °C) glassware was used. Tetrahydrofuran, diethyl ether, and benzene were distilled from sodium/benzophenone.

Preparation of trans-N,N,N-triethyl-2-[4-(2-phenylethenyl)-phenoxy]ethanammonium iodide (1). To 6 mmol of the trimethylsilylalkyne, **2** or **3**, in 12 ml of ether at room temperature were added dropwise 6.5 ml of 1 *M* diisobutylaluminium hydride in hexane (6.5 mmol) during 0.5 h. The mixture was refluxed for 5 h and the resulting vinylalane solution was then treated with a mixture of 6 mmol of the relevant aryl bromide and 0.3 mmol (0.05 equiv.) of tetrakis-(triphenylphosphine) palladium in 18 ml of dry tetrahydrofuran. The mixture was refluxed for 24 h and then treated with an excess of the water. The usual work-up and evaporation of solvents left the crude vinylsilane, **4** or **5**, which was refluxed with hydroiodic acid in 15 ml of benzene. Basic work-up and evaporation of solvents left a crude product which was refluxed with 6 mmol of ethyl iodide in 15 ml of tetrahydrofuran for 1 h. Work-up and evaporation of solvents left a residue, which was recrystallized from ethyl acetate/benzene to give 3.7 mmol (62% overall yield) from compound **2** or 4 mmol (67% overall yield) from compound **3** of the desired compound (**1**) as a white crystalline solid. GLC showed only one peak.

Acknowledgement

This research (Chem/1408/12) was supported by the Research Center, College of Science, King Saud University, Riyadh, Saudi Arabia.

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