

Molecules **2001**, *6*, M207

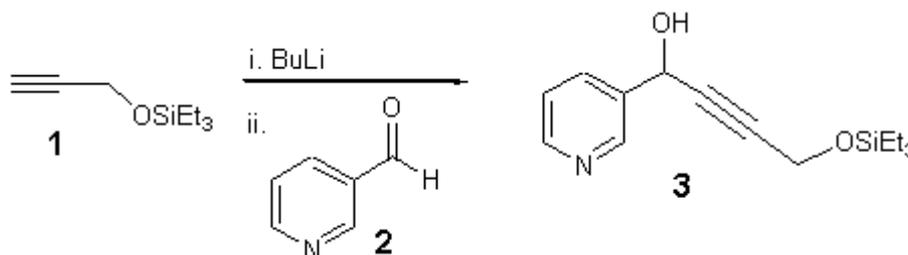
1-(Pyridin-3-yl)-4-(triethylsilyloxy)-2-butyne-1-ol

Vittorio E. Caprio, Michael W. Jones and Margaret A. Brimble*

Department of Chemistry, The University of Auckland, Private Bag 92019, Auckland, New Zealand, Fax: (+64 9) 3737599 ; E-mail: m.brimble@auckland.ac.nz

* Author to whom correspondence should be addressed.

Received: 8 December 2000 / Accepted: 15 December 2000 / Published: 25 March 2001



The general part of the experimental section [1] has been presented elsewhere. ⁿBuLi (1.6 M) (1.9 mL, 5.9 mmol) was added dropwise to a solution of silyl ether **1** (500 mg, 2.94 mmol) in THF (10 mL) at 78 °C under nitrogen and stirred for 45 min. Pyridine-3-carboxaldehyde **2** (315 mg, 2.94 mmol) was added dropwise and the resultant solution stirred for 1.5 h. The mixture was warmed to room temperature then water (10 mL) was added. The mixture was extracted with ethyl acetate (4 × 15 mL) and dried over magnesium sulfate. The solvent was removed under reduced pressure to give a yellow oil which was purified by flash chromatography using hexane-ethyl acetate (1:2) as eluent to afford the title compound **3** (773 mg, 95%) as a pale yellow oil.

IR (neat): 3308b, 2957s, 2913s, 2877s, 2235w, 1741s, 1591m, 1243s.

¹H NMR (200 MHz, CDCl₃): 0.63 [6H, q, *J* 8.0 Hz, OSi(CH₂CH₃)₃], 0.96 [9H, t, *J* 8.0 Hz, OSi(CH₂CH₃)₃], 4.40 (2H, d, *J* 1.8 Hz, H₄), 5.56 (1H, br. s, H₁), 7.31 (1H, m, H_{5'}), 7.88 (1H, ddd, *J*_{5',4'} 7.4, *J*_{4',6'} 1.7 and *J*_{2',4'} 1.7 Hz, H_{4'}), 8.56 (1H, dd, *J*_{6',4'} 1.7 and *J*_{6',5'} 4.8 Hz, H_{6'}), 8.75 (1H, d, *J*_{2',4'} 1.7 Hz, H_{2'}).

¹³C NMR (50 MHz, CDCl₃): 4.3 [CH₂, OSi(CH₂CH₃)₃], 6.6 [CH₃, OSi(CH₂CH₃)₃], 51.3 (CH₂, C₄), 62.0 (CH, C₁), 83.8 (quat., C₃), 85.4 (quat., C₂), 123.5 (CH, C_{5'}), 131.8 (CH, C_{4'}), 136.9 (CH, C_{6'}), 147.7 (CH, C_{2'}), 148.7 (quat., C_{3'}).

EI-MS: 277 (M⁺, 10%), 248 (M⁺-C₂H₅, 47%), 162 (M⁺-Si(CH₂CH₃)₃, 66%), 146 (70%), 117 (59%), 103 (84%), 75 (100%).

Anal. Calc. For C₁₅H₂₃NO₂Si, 277.14981; found M⁺, 277.14918.

Reference

1. Brimble, M. A.; Duncalf, L. J. *Molecules* **2000**, *5*, 162-166.

Sample availability: available from the authors and MDPI.

© 2001 MDPI. All rights reserved. *Molecules* website www.mdpi.org/molecules/