

Molecules **2001**, *6*, M206

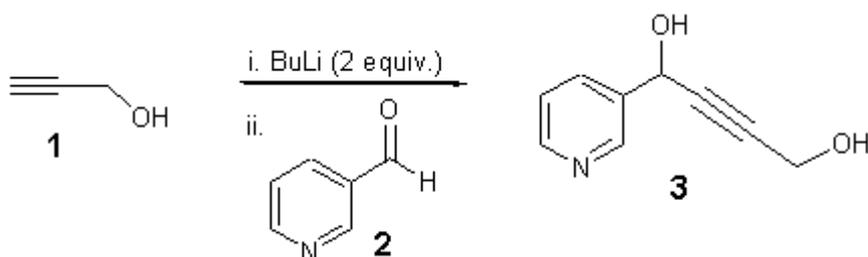
1-(Pyridin-3-yl)but-2-yn-1,4-diol

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) (6.8 mL, 10.8 mmol) was added to a solution of propargyl alcohol **1** (0.35 mL, 4.9 mmol) in THF (10 mL) under nitrogen at 20°C and the mixture stirred for 10 min. The reaction was then cooled to -78°C and a solution of pyridine-3-carboxaldehyde **2** (500 mg, 4.9 mmol) in THF (1 mL) was added dropwise. The resultant mixture was stirred for 1 h at -78°C, warmed to 0°C then stirred for 1 h. Saturated aqueous ammonium chloride (15 mL) was added and then the mixture was warmed to room temperature. The orange-yellow solution was extracted with diethyl ether (3 x 20 mL), dried over magnesium sulfate and concentrated under reduced pressure to afford a yellow oil. Further purification by flash chromatography using ethyl acetate as eluent gave the title compound **3** (195 mg, 27%) as a pale yellow oil.

IR (neat): 3368b, 2957s, 2932s, 2860s, 2235w, 1581w, 1466m, 1427m.

¹H NMR (200 MHz, CDCl₃): 4.34 (2H, d, *J* 1.6 Hz, H₄), 5.56 (1H, br. s, H₁), 7.25-7.33 (1H, m, H_{5'}), 7.88 (1H, d, *J*_{4',5'} 8.1 Hz, H_{4'}), 8.51 (1H, d, *J*_{5',6'} 4.0 Hz, H_{6'}), 8.78 (1H, s, H_{2'}).

¹³C NMR (50 MHz, CDCl₃): 50.5 (CH₂, C₄), 62.0 (CH, C₁), 84.3 (quat., C₃), 85.8 (quat., C₂), 123.6 (CH, C_{5'}), 135.0 (CH, C_{4'}), 136.9 (CH, C_{6'}), 147.8 (CH, C_{3'}), 148.7 (CH, C_{2'}).

EI-MS: 163 (M⁺, 96%), 146 (68%), 132 (51%), 117 (77%), 106 (42%), 80 (100%).

Anal. Calc. For C₁₅H₉NO₂, 163.06333; found M⁺, 163.06309.

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/