Molecules 2001, 6, M202

Pyridine-3-carboxaldehyde O-methyloxime

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. Methoxylamine hydrochloride (858 mg, 10.3 mmol) and potassium carbonate (2.58 g, 18.7 mmol) were added to a solution of pyridine-3-carboxaldehyde (1 g, 9.34 mmol) in ethanol (15 mL) under nitrogen at room temperature and the mixture was stirred for 2 h [2]. Water (20 mL) and dichloromethane (20 mL) were then added and the organic and aqueous phases separated. The aqueous phase was extracted with dichloromethane (3 x 15 mL) and the organic extracts were combined. The organic extract was dried over magnesium sulfate and concentrated under reduced pressure. Purification by flash chromatography using ethyl acetate as eluent afforded the title compound (1.25 g, 98%) as a pale yellow oil.

IR (cm⁻¹, neat): 3394b, 2968m, 2938m, 1610m, 1591m, 1564m, 1421m, 1413m, 1056s, 1039s.

¹H NMR (200 MHz, CDCl₃): 4.01 (3H, s, OC*H*₃), 7.31 (1H, dd, $J_{5,6}$ 4.8 and $J_{5,4}$ 7.8 Hz, H5), 7.97 (1H, ddd, $J_{5,4}$ 7.8, $J_{4,6}$ 1.8 and $J_{4,2}$ 1.8 Hz, H4), 8.08 (1H, s, *H*C=N), 8.61 (1H, dd, $J_{6,4}$ 1.8 and $J_{6,5}$ 4.8 Hz, H6), 8.74 (1H, d, $J_{2,4}$ 1.8 Hz, H2).

¹³C NMR (50 MHz, CDCl₃): 62.0 (CH₃, O*C*H₃), 123.4 (CH, C5), 128.1 (quat., C3), 133.1 (CH, C4), 145.2 (CH, C6), 148.4 (CH, C2), 150.4 (CH, H*C*=N).

EI-MS: 136 (M⁺, 100%), 105 (M⁺-OCH₃, 16%), 78 (M⁺-C₂H₄NO, 82%), 51 (49%).

Anal. Calc. for C₇H₈N₂O, 136.0610; found M⁺, 136.0637.

Reference

- 1. Brimble, M. A.; Duncalf, L. J. Molecules 2000, 5, 162-166.
- 2. Goti, A.; Cicchi, S.; Fedi, V.; Nannelli, L.; Brandi, A. J. Org. Chem. 1997, 62, 3119-3125.

Sample availability: available from the authors.

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

1 von 1 01.04.2009 16:04