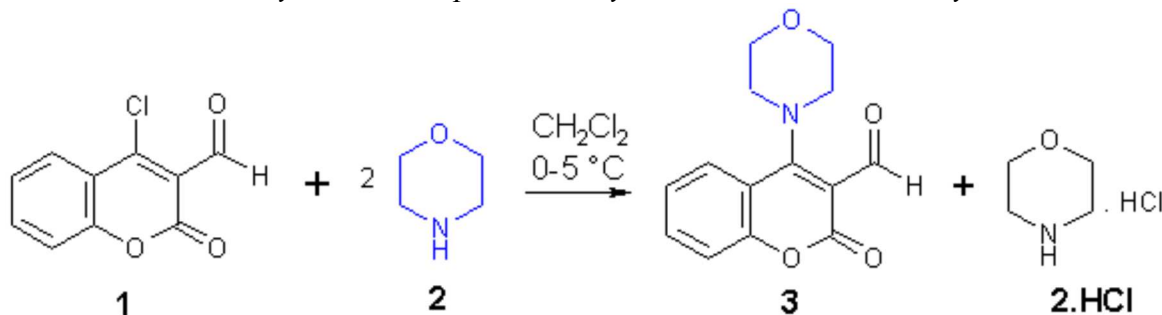


4-Morpholino-2-oxo-2H-chromene-3-carbaldehyde

Boyan I. Iliev and Ivo C. Ivanov*

Faculty of Pharmacy, Medical University of Sofia, Dunav 2, BG-1000 Sofia, Bulgaria
 Tel.: (+359 2) 981 7025; Fax: (+359 2) 987 9874; E-mail: i.ivanov@mbox.pharmfac.acad.bg

Received: 23 February 2001 / Accepted: 15 May 2001 / Published: 25 May 2001



The title compound **3** has been allegedly prepared earlier [1] by refluxing the same starting materials **1** and **2** in ethanol for 10 min. but no characteristic data of the product has been reported till now except its melting point. Our attempts to reproduce the procedure given in [1] failed: a complex mixture of unidentified products resulted (TLC-monitoring). The aldehyde **1** was prepared according to [2]).

A solution of morpholine (**2**; 1.75 g, 20 mmol) in 10 ml of dichloromethane was gradually added under stirring to an ice-cooled mixture of 4-chloro-2-oxo-2H-chromene-3-carbaldehyde (**1**; 2.09 g, 10 mmol) in 25 ml of dichloromethane. After stirring for 30 min. at 0-5 °C the mixture was washed with 3x10 ml of water in order to remove unreacted morpholine and its salt. The organic phase was dried over MgSO₄ and the solvent was evaporated under reduced pressure. The dry, flake-like residue was recrystallized from 1,4-dioxane. Yield: 1.44 g (56%) of **3** as yellow crystals, m.p. 162-164 °C, TLC homogeneous (TLC control: silica gel pre-coated Al-sheets Merck GF254, eluted by chloroform-acetone 3:2). Mp. 162-164 °C. After twofold recrystallization from dioxane: yield 1.00 g (39%), yellow crystals.

Mp. 166-168 °C (Lit. [1] m. p. 165 °C).

¹H NMR (100 MHz; CDCl₃): 3.4-3.8 [m, 4H, -N(CH₂)₂], 3.8-4.1 [m, 4H, O(CH₂)₂], 7.1-8.0 (m, 4H_{arom.}), 10.2 (s, CHO).

FT IR (cm⁻¹; nujol): 1699 (C=O, aldehyde), 1674 (C=O, lactone), 1607, 1590, 1526, 1302, 1286, 1111, 961, 916, 777, 758; (fluorolube): 2855-2950, 1701 (C=O, aldehyde), 1674 (C=O, lactone), 1607, 1590, 1526, 1428.

EI-MS [70 eV; *m/z* (%): 259 (M⁺; 82), 242 (100), 230 (27), 212 (45), 202 (34), 186 (13), 174 (43), 161 (14), 146 (93), 118 (27), 89 (36), 63 (20), 28 (75).

Anal. calcd. for C₁₄H₁₃NO₄ (259.26): C 64.86, H 5.05, N 5.40; Found C 64.83, H 5.10, N 5.34.

References

1. Moorty S.R.; Sundaramurthy V.; Subba Rao N. V. *Indian J. Chem.* **1973**, *11*, 854-856.
2. Heber D.; Ivanov I. C.; Karagiosov S. K. *J. Heterocycl. Chem.* **1995**, *32*, 505-509.

Sample Availability: Available from the authors and from MDPI.

© 2001 MDPI, Basel, Switzerland. All rights reserved.