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.


$^1$H NMR (100 MHz; CDCl3): 3.4-3.8 [m, 4H, -N(CH$_2$)$_2$], 3.8-4.1 [m, 4H, O(CH$_2$)$_2$], 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 C$_{14}$H$_{13}$NO$_4$ (259.26): C 64.86, H 5.05, N 5.40; Found C 64.83, H 5.10, N 5.34.

References

Sample Availability: Available from the authors and from MDPI.

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