3-Dimethylaminomethylene-4-phenyl-1,3-dihydro-2H-1,5-benzodiazepin-2-one

Abdelouahhab El Rhazi, Abdesselam Baouid,* Said Elhazazi, Aicha Boudina and Mohamed Essaber

Laboratoire de Chimie Moléculaire, Equipe de Chimie des Hétérocycles et Valorisation des Extraits des Plantes, Département de Chimie, Faculté des Sciences-Semlalia, Université Cadi Ayyad, B.P 2390, 40001 Marrakech, Maroc. E-mail: elrhazi@yahoo.fr, baouid@yahoo.fr, s.elhazazi@ucam.ac.ma, aichaboudina@yahoo.fr, m.essaber@yahoo.fr

* Author to whom correspondence should be addressed.

Received: 23 July 2008 / Accepted: 27 August 2008 / Published: 4 September 2008

Keywords: 1,5-Benzodiazepin-2-one, dimethylformamide-dimethylacetal.

In this paper, we describe a facile and efficient method to prepare compound 2, which had been previously obtained by a Vilsmeier reaction using DMF/POCl₃ [1]. We found that employment of dimethylformamide-dimethylacetal (DMF-DMA) affords the title compound in better purity and higher yield.

A mixture of 0.47 g (1.98 mmol) of 4-phenyl-1,3-dihydro-2H-1,5-benzodiazepin-2-one 1 [2,3] in 4.5 ml of dimethylformamide-dimethylacetal (DMF-DMA) was stirred at 100 °C for 4 hours and then cooled to room temperature. Filtration and washing with a little cold diethyl ether gave 0.46 g (75%) of 3-dimethylaminomethylene-4-phenyl-1,3-dihydro-2H-1,5-benzodiazepin-2-one 2. The product obtained was recrystallized from diethyl ether.
Melting point: 224-225 °C (Diethyl ether).

MS (m/z, %): 292 ([M+H]^+, 100%).

$^1$H NMR (CDCl$_3$, 400 MHz), δ(ppm): 2.55 (s, 6H, (CH$_3$)$_2$N), 2.80 (s, 1H, NH), 7.40 (s, 1H, C=CH-N(CH$_3$)$_2$), 6.80-7.90 (m, 9H, H-Ar).

$^{13}$C NMR (CDCl$_3$, 100 MHz), δ(ppm): 43.50 (N(CH$_3$)$_2$), 98.02 (C-3), 121.47, 124.59, 127.34, 128.13, 128.67, 128.89, 130.65, 131.78, 141.86 (C-Ar), 151.18 (C=CH-N(CH$_3$)$_2$), 167.40 (C-4), 178.57 (C=O).

References:

© 2008 by the authors; licensee Molecular Diversity Preservation International, Basel, Switzerland. This article is an open-access article distributed under the terms and conditions of the Creative Commons Attribution license (http://creativecommons.org/licenses/by/3.0/).