Benzyl 2-((E)-Tosyliminomethyl)phenylcarbamate

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Abstract: Benzyl 2-((E)-tosyliminomethyl)phenylcarbamate was prepared in good yield and characterized by the condensation reaction of benzyl 2-formylphenylcarbamate with p-toluenesulfonyl amine. The structure of the newly synthesized compound was determined using \(^1H\), \(^13C\)-NMR, IR and mass spectral data.

Keywords: Schiff base; Imine; p-toluenesulfonyl amine; condensation reaction

1. Introduction

The Schiff base, structurally known as imine or azomethine, is a nitrogen analog of aldehyde or ketone in which the C=O group is replaced by C=N-R group after water molecular elimination [1]. Schiff bases are some of the most widely used organic compounds which used as pigments and dyes, catalysts and intermediates in organic synthesis [2]. Schiff bases have also been shown to exhibit a broad range of biological activities, including antibacterial, antimalarial, anti-inflammatory, antiviral, and anticancer properties [3–5]. In continuation of our research interest in 2-aminobenzaldehyde for the synthesis of highly functionalized chiral heterocycles [6–9], we report here the preparation of a novel benzyl 2-((E)-tosyliminomethyl)phenylcarbamate.

The synthesis of the title compound 3 was achieved in one step, as presented in Scheme 1, which was performed by the condensation reaction of benzyl 2-formylphenylcarbamate (1) [10] with p-toluenesulfonyl amine (2). The reaction was carried out in toluene in the presence of 2 mol% of boron trifluoride diethyl etherate as a catalyst and provided the desired product in good yield. The structure of compound 3 was confirmed by \(^1H\)- and \(^13C\)-NMR, IR, mass spectral data, and all data are in accordance with the assumed structure.

Scheme 1. Synthesis of benzyl 2-((E)-tosyliminomethyl)phenylcarbamate (3).
2. Experimental Section

2.1. General Information

All reagents were used as received without further purification. Organic solutions were concentrated under reduced pressure using a Büchi rotary evaporator. Chromatographic purification of the title compound 3 was accomplished using forced-flow chromatography on ICN 60 32–64 mesh silica gel 63. Thin-layer chromatography (TLC) was performed on EM Reagents 0.25 mm silica gel 60-F plates. Developed chromatograms were visualized by fluorescence quenching and anisaldehyde stain. $^1$H and $^{13}$C-NMR spectra were recorded on a 400 MHz instrument as noted, and were internally referenced to residual protio solvent signals. Data for $^1$H-NMR were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), integration, coupling constant (Hz) and assignment. Data for $^{13}$C-NMR were reported in terms of chemical shift. IR spectra were recorded on Perkin-Elmer 1600 FT-IR spectrometer (Waltham, MA, USA), and reported in terms of frequency of absorption (cm$^{-1}$). High-resolution mass spectrometry data was recorded on a JEOL JMS-700 MStation mass spectrometer (JEOL, Tokyo, Japan).

2.2. Synthesis of Benzyl 2-((E)-Tosyliminomethyl)phenylcarbamate (3)

$p$-Toluenesulfonyl amine (2, 94 mg, 0.55 mmol) was added to a solution of BF$_3$·Et$_2$O (1 µL, 0.01 mmol) and benzyl 2-formylphenylcarbamate (1, 128 mg, 0.50 mmol) in toluene (2 mL) at room temperature. The resulting mixture was refluxed for 60 h until complete consumption of benzyl 2-formylphenylcarbamate 1 was observed as determined by TLC. After being cooled to room temperature, water (2 mL) was added and the products were extracted with dichloromethane (3 × 5 mL). The organic phase was washed with aqueous saturated NaCl solution (2 × 5 mL), dried with anhydrous MgSO$_4$, and concentrated in vacuo. The crude residue was purified by flash silica gel column chromatography using EtOAc/hexane (1/10) as eluent to afford the desired title compound 3 (64%, 154 mg).

White solid; m.p. 158–160 °C; $^1$H-NMR (400 MHz, CDCl$_3$) δ 10.83 (s, 1H), 9.02 (s, 1H), 8.46 (d, J = 8.4 Hz, 1H), 7.89–7.77 (m, 2H), 7.64–7.52 (m, 2H), 7.47–7.33 (m, 5H), 7.14 (ddd, J = 8.4, 6.9, 3.0 Hz, 3H), 5.20 (s, 2H), 2.39 (s, 3H); $^{13}$C-NMR (100 MHz, CDCl$_3$) δ 170.38, 153.17, 144.79, 142.28, 137.27, 136.43, 135.96, 135.33, 129.87, 128.61, 128.35, 128.21, 127.78, 118.76, 117.46, 67.11, 21.67; IR (film) 3248, 2923, 2855, 1735, 1597, 1562, 1537, 1447, 1376, 1317, 1222, 1162, 1088, 1067, 1067 cm$^{-1}$; HRMS (EI) m/z calcd for [M]$^+$ C$_{22}$H$_{20}$N$_2$O$_4$S: 408.1144 Found: 408.1158.

Supplementary Materials: $^1$H- and $^{13}$C-NMR spectra for compound 3 are available online at http://www.mdpi.com/1422-8599/2016/4/M912.

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Conflicts of Interest: The authors declare no conflict of interest.

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