

Short Note

4-[(2-Hydroxy-4-pentadecyl-benzylidene)-amino]-benzoic Acid Methyl Ester

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Abstract: A new Schiff base, 4-[(2-hydroxy-4-pentadecyl-benzylidene)-amino]-benzoic acid methyl ester was synthesized and its UV, IR, ¹H-NMR, ¹³C-NMR and ESI-MS spectroscopic data are presented.

Keywords: Schiff base; 4-[(2-hydroxy-4-pentadecyl-benzylidene)-amino]-benzoic acid methyl ester; cardanol

Introduction

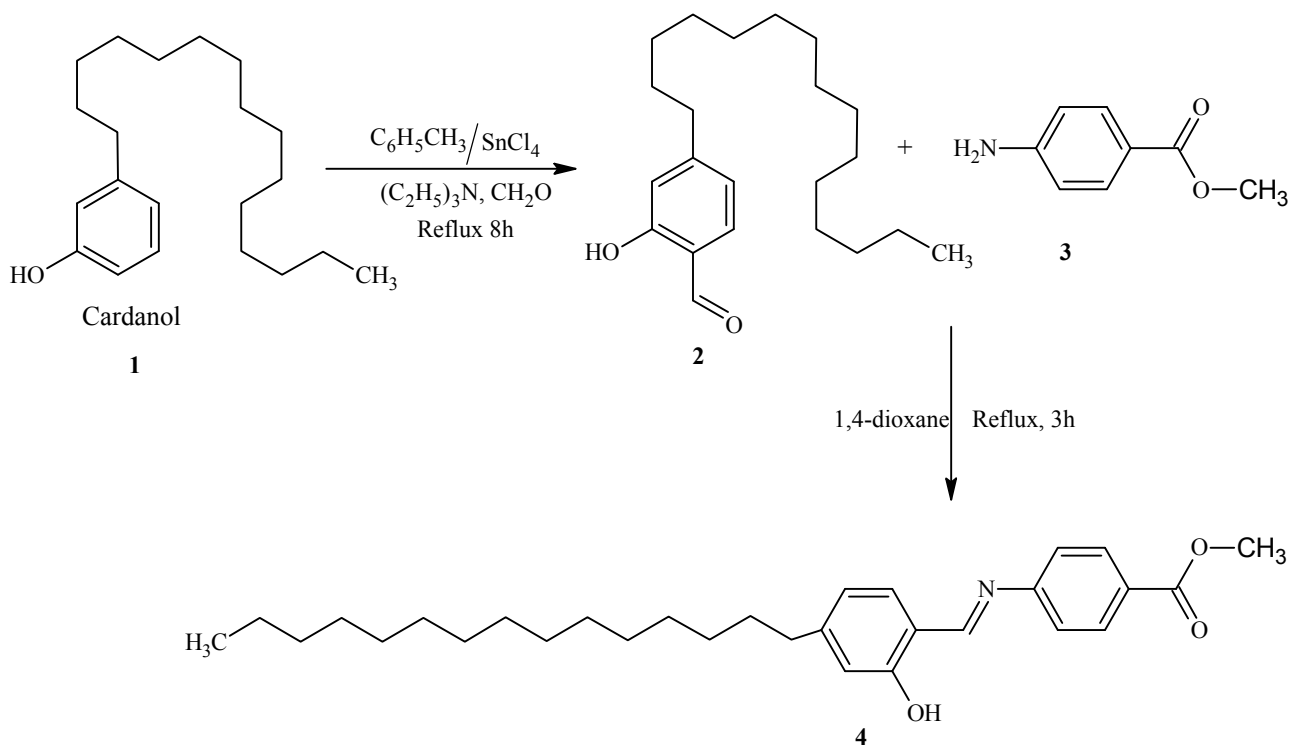
Schiff bases have received a considerable amount of attention from many researchers owing to their importance in exhibiting thermochromism and photochromism [1–4]. Aromatic Schiff bases possessing long alkyl chain have received overwhelming attention due to their possibility to show mesomorphic properties such as smectic and nematic phases [5,6]. As a continuation of our previous work, we report here another new Schiff base compound, 4-[(2-hydroxy-4-pentadecyl-benzylidene)-amino]-benzoic acid methyl ester.

Synthesis

2-Hydroxy-4-pentadecylbenzaldehyde was prepared from cardanol (3-pentadecylphenol, contained, e.g., in Cashew nut shell liquid (CNSL)) by formylation, using a standard procedure [7]. The title

compound was obtained by refluxing compound **2** with an equimolar amount of methyl-4-amino benzoate in 1,4-dioxane for 3 h. This new Schiff base was fully characterized by elemental analysis, UV, IR, MS, ^1H -NMR and ^{13}C -NMR data. As shown in Scheme 1.

Scheme 1. 4-[(2-hydroxy-4-pentadecyl-benzylidene)-amino]-benzoic acid methyl ester.



Experimental

All reagents were analytical grade. Purchased from Fluka, or Aldrich chemical Co., Ltd, and were as received without further purification. Melting point was determined in open capillary and is uncorrected. Absorption spectrum was recorded in CHCl_3 by a Hewlett Packard-8453 spectrophotometer. FT-IR spectrum was recorded on a Nicolet Fourier Transform Infrared Spectrophotometer: Impact 410 (Nicolet Instrument Technologies, Inc., Madison, WI, USA). ^1H -NMR and ^{13}C -NMR were obtained in $\text{DMSO}-d_6$ at 500 MHz for ^1H nuclei and 125 MHz for ^{13}C nuclei (Bruker Company, Rheinstetten, Germany). All chemical shifts were reported in parts per million (ppm) using residual proton or carbon signal in deuterated solvents as internal references. Coupling constants (J) are reported in Hertz (Hz). Mass spectra were recorded by electron spray ionization mass spectroscopy (ESI-MS) on a Bruker Daltonics microOTOF mass spectrometer. Elemental analysis (C, H and N) was performed on a Perkin Elmer 2400 analyzer. The purity of the compound was checked by TLC on silica gel and further purification was performed through column chromatography (silica gel, 60–120 mesh).

2-Hydroxy-4-pentadecylbenzaldehyde **2** (0.23 g, 0.71 mmole) in 1,4-dioxane (20 mL) and methyl-4-amino benzoate **3** (0.10 g, 0.71 mmol) was heated under reflux for 3 hours. The completion of the reaction was monitored by TLC. The reaction mixture was allowed to cool down to room temperature, and then poured onto ice cooled water with constant stirring. The precipitate was filtered, washed with water, dried and recrystallized from absolute ethanol. The resulting solid was further

purified by silica column, using a gradient mixture of hexane/ethyl acetate (90:10) as an eluent to obtain **4**.

Color: light yellow solid.

Yield: 0.21 g (65%).

Melting point: 212–214 °C.

$\lambda_{\text{max}} = 351 \text{ nm}$.

ESI-MS: $m/z = 465.817 \text{ (M}^+)$.

IR (KBr): $\nu_{\text{max}} \text{ (cm}^{-1}\text{)}$: 3441.68 (O-H str.), 2917.15 ((=C-H), w), 1723.44 (C=N str.).

$^1\text{H-NMR}$ (500 MHz, DMSO- d_6) δ ppm: 12.68 (s, 1H, OH), 8.94 (s, 1H, CH=N), 8.01 (d, $J = 7.7 \text{ Hz}$, 2H, Ar-H), 7.57 (d, $J = 7.8 \text{ Hz}$, 1H, Ar-H), 7.48 (d, $J = 7.8 \text{ Hz}$, 2H, Ar-H), 6.82 (d, $J = 7.7 \text{ Hz}$, 1H, Ar-H), 6.79 (s, 1H, Ar-H), 3.85 (s, 3H, OCH₃), 2.60 (t, $J = 7.6 \text{ Hz}$, 2H, Ar-CH₂), 1.56–1.13 (m, 26H, (CH₂)₁₃), 0.83 (t, $J = 6.8 \text{ Hz}$, 3H, CH₃).

$^{13}\text{C-NMR}$ (125 MHz, DMSO- d_6) δ ppm: 163.72, 161.46, 152.44, 143.56, 132.60, 131.07, 128.22, 121.10, 120.03, 117.12, (Aromatic carbons), 52.14 (OCH₃), 36.30, 31.92, 30.84, 30.63, 29.68, 29.67, 29.65, 29.55, 29.47, 29.35, 29.27, 22.68 (Methylene carbons), 14.09 (CH₃).

Elemental analysis: Calculated for C₃₀H₄₃NO₃: C, 77.38%; H, 9.31%; N, 3.01%; found: C 77.08%; H, 9.05%; N, 2.89%.

Acknowledgments

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Conflicts of Interest

The authors declare no conflict of interest.

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