

Short Note

2-[(4-Bromophenylimino)methyl]-5-pentadecylphenol

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Abstract: 2-[(4-Bromophenylimino)methyl]-5-pentadecylphenol has been synthesized by reaction of 2-hydroxy-4-pentadecylbenzaldehyde with 4-bromoaniline in 1,4-dioxane and its IR, ¹H-NMR, ¹³C-NMR and MS spectroscopic data are presented.

Keywords: 2-[(4-bromophenylimino)methyl]-5-pentadecylphenol; Schiff base; alkyl chain

Introduction

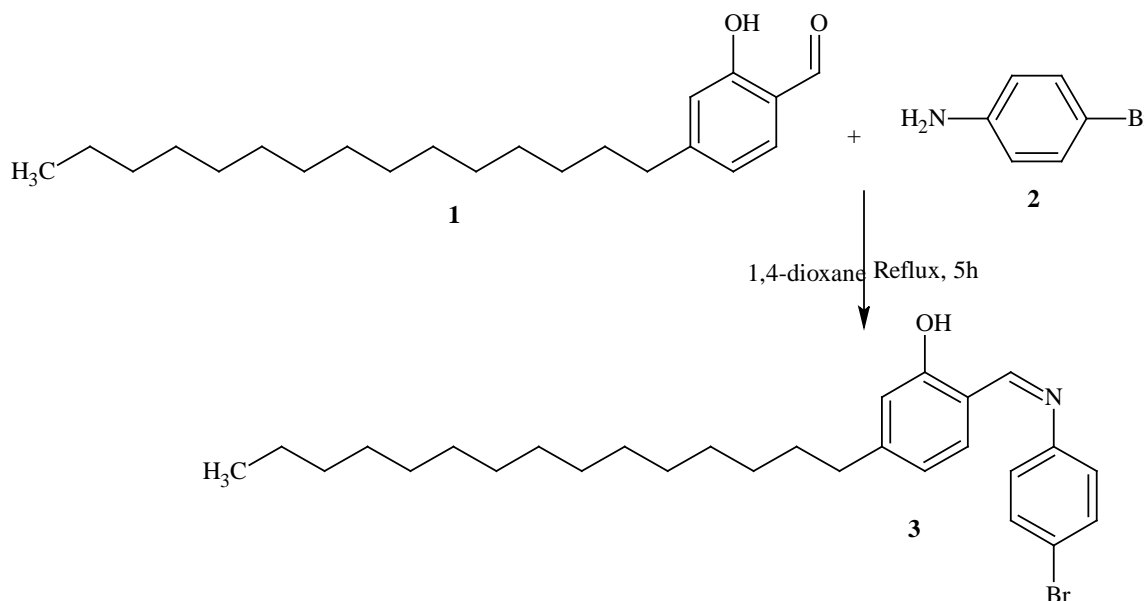
Compounds with the structure of -C=N- (azomethine group) are known as Schiff bases, which are usually synthesized by condensation of primary amines and active carbonyl groups. Schiff bases are an important class of compounds in the medicinal and pharmaceutical field, including antibacterial [1,2], antifungal [3,4] and antitumor activity [5,6]. 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 [7–11]. In this paper, we report the synthesis of a novel Schiff base from 4-bromoaniline and 2-hydroxy-4-pentadecylbenzaldehyde.

Result and Discussion

2-Hydroxy-4-pentadecylbenzaldehyde [12] was prepared from cardanol (3-pentadecylphenol, contained, e.g., in Cashew nut shell liquid) by formylation, using a standard procedure [12]. The title compound was obtained in good yield (81%) by refluxing compound **1** with an equimolar amount of 4-bromoaniline in 1,4-dioxane for 5 hours. This new Schiff base was fully characterized by elemental

analysis, UV, IR, MS, $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ data. The configuration at the imine unit was not investigated, compound **3** is arbitrarily shown in Z configuration in Scheme 1.

Scheme 1. Synthesis of 2-[(4-Bromophenylimino)methyl]-5-pentadecylphenol.



Experimental

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. WI, USA). $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ were obtained in $\text{DMSO-}d_6$ at 500 MHz for ^1H nuclei and 125 MHz for ^{13}C nuclei (Bruker, Germany). All chemical shifts were reported in parts per million (ppm) using residual proton or carbon signal in deuterated solvents as internal references. Mass spectrum was obtained using matrix-assisted laser desorption ionization mass spectrometry (MALDI-TOF) by using dithranol as a matrix. 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 **1** (1.10 g, 0.003 mol) in 1,4-dioxane (25 mL) and 4-bromoaniline **2** (0.57 g, 0.003 mol) was refluxed for 5 h. The completion of the reaction was monitored by TLC. The reaction mixture was allowed to cool down to room temperature, and then poured into ice cooled water with constant stirring. The precipitate was filtered, washed with water, dried and recrystallized from 1,4-dioxane. The resulting solid was further purified by silica column, using a gradient mixture of petroleum ether/ethyl acetate (80:20 to 60:40) as an eluent to obtain **3**.

Color: Yellow solid.

Yield: 0.80 g (81%).

Melting point: 189–191 °C.

$\lambda_{\max} = 346 \text{ nm}$.

MS: $m/z = 487.66 (M^+ + 1)$, 485.66.

IR (KBr): $\nu_{\max} (\text{cm}^{-1})$: 3435.31 (O-H str.), 1624.63 (C=N str.).

$^1\text{H-NMR}$ (500 MHz, DMSO- d_6) δ ppm: 12.80 (s, 1H, OH), 8.89 (s, 1H, CH=N), 7.61 (d, $J = 7.8 \text{ Hz}$, 2H, Ar-H), 7.53 (d, $J = 7.7 \text{ Hz}$, 1H, Ar-H), 7.34 (d, $J = 7.8 \text{ Hz}$, 2H, Ar-H), 6.80 (d, $J = 7.6 \text{ Hz}$, 1H, Ar-H), 6.77 (s, 1H, Ar-H), 2.60 (t, $J = 7.6 \text{ Hz}$, 2H, Ar-CH₂), 1.57–1.13 (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: 160.7, 157.2, 152.1, 143.6, 129.8, 128.9, 120.1, 119.9, 118.8, 116.5, 115.0, 114.7, 112.5 (aromatic carbons), 35.3, 35.1, 34.8, 31.2, 30.8, 30.1, 28.9, 28.8, 28.7, 28.6, 28.5, 22.0, 13.8 (methylene carbons), 13.5 (CH₃).

Elemental analysis: Calculated for C₂₈H₄₀BrNO: C, 69.12%; H, 8.29%; N, 2.88%; found: C, 68.94%; H, 8.36%; N, 2.73%.

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References

1. Panneerselvam, P.; Rather, B.A.; Reddy, D.R.S.; Kumar N.R. Synthesis and anti-microbial screening of some Schiff bases of 3-amino-6,8-dibromo-2-phenylquinazolin-4(3H)-ones. *Eur. J. Med. Chem.* **2009**, *44*, 2328–2333.
2. Jin, X.; Wang, J.; Bai, J. Synthesis and antimicrobial activity of the Schiff base from chitosan and citral. *Carbohyd. Res.* **2009**, *344*, 825–829.
3. Bharti, S.K.; Nath, G.; Tilak, R.; Singh, S.K. Synthesis, anti-bacterial and anti-fungal activities of some novel Schiff bases containing 2,4-disubstituted thiazole ring. *Eur. J. Med. Chem.* **2010**, *45*, 651–660.
4. Bagihalli, G.B.; Avaji, P.G.; Patil, S.A.; Badami, P.S. Synthesis, spectral characterization, in vitro antibacterial, antifungal and cytotoxic activities of Co(II), Ni(II) and Cu(II) complexes with 1,2,4-triazole Schiff bases. *Eur. J. Med. Chem.* **2008**, *43*, 2639–2649.
5. Liu, Y.; Yang, Z. Crystal structures, antioxidation and DNA binding properties of Eu(III) complexes with Schiff-base ligands derived from 8-hydroxyquinoline-2-carboxyaldehyde and three aroylhydrazines. *J. Inorg. Biochem.* **2009**, *103*, 1014–1022.
6. Nawaz, H.; Akhter, Z.; Yameen, S.; Siddiqi, H.M.; Mirza, B.; Rifat, A. Synthesis and biological evaluations of some Schiff-base esters of ferrocenyl aniline and simple aniline. *J. Organomet. Chem.* **2009**, *694*, 2198–2203.

7. Ha, S.T.; Ong, L.K.; Wong, J.P.W.; Yeap, G.Y.; Lin, H.C.; Ong, S.T.; Koh, T.M. Mesogenic Schiff's base ether with dimethylamino end group. *Phase Transit.* **2009**, *82*, 387–397.
8. Ha, S.T.; Ong L.K.; Ong, S.T.; Yeap, G.Y.; Wong, J.P.W.; Koh, T.M.; Lin, H.C. Synthesis and mesomorphic properties of new Schiff base esters with different alkyl chains. *Chin. Chem. Lett.* **2009**, *20*, 767–770.
9. Ha, S.T.; Koh, T.M.; Ong, S.T.; Beh, J.K.; Ong, L.K. Synthesis of a new liquid crystal, 3-hydroxy-4-[[6-methoxy-1,3-benzothiazol-2-yl]imino]methyl}phenyl palmitate. *Molbank* **2009**, *2009*, M608.
10. Ha, S.T.; Ong, L.K.; Win, Y.F.; Koh, T.M. 4-[(Pyridin-3-ylmethylene)amino]phenyltetradecanoate. *Molbank* **2009**, *2009*, M585.
11. Ha, S.T.; Ong, L.K.; Win, Y.F.; Koh, T.M. 4-[(Pyridin-3-ylmethylene)amino]phenylhexadecanoate. *Molbank* **2009**, *2009*, M584.
12. Payne, P.; Tyman, J.H.P.; Mehet, S.K.; Ninagawa, A. The synthesis of 2-hydroxymethyl derivatives of phenols. *J. Chem. Res.* **2006**, *37*, 402–405.

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