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Short Note

4-({[4-(Methylthio)phenyl]methylene}amino)phenyl Dodecanoate

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Abstract: A new Schiff base ester, 4-({[4-(methylthio)phenyl]methylene}amino)phenyl dodecanoate was synthesized and its IR, ^{1}H NMR, ^{13}C NMR and EI-MS spectroscopic data are presented.

Keywords: Schiff base; mesogen; 4-({[4-(methylthio)phenyl]methylene}amino)phenyl dodecanoate

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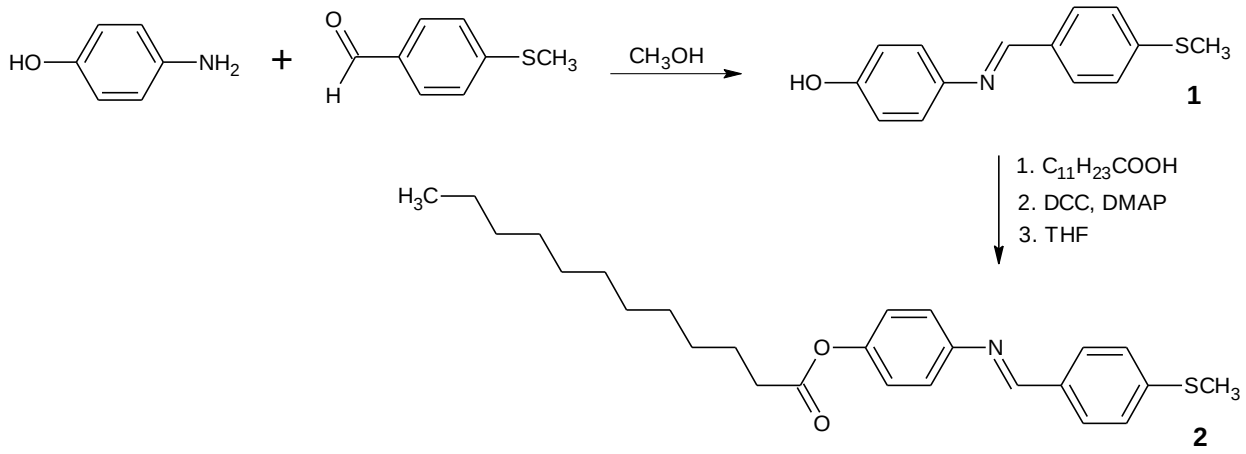
Mesogenic materials have many useful applications in scientific and technological areas, specifically as LCD devices, organic light emitting diodes, anisotropic networks, photoconductors and semiconductor materials [1-3]. Strong demand of new liquid crystals for applications has led to the synthesis of numerous mesogens in particular, thermotropic liquid crystals [4,5]. In the previous studies, we discovered that azomethine and ester are useful connecting units for generating mesomorphism in thermotropic liquid crystals with two and three aromatic rings. Aromatic azomethine ester comprising of different polarity of substituents has been known to either promote or suppress the mesomorphic properties [6-8]. In the present work, Schiff base ester and methylthio terminal moiety are incorporated to form a new compound, 4-({[4-(methylthio)phenyl]methylene} amino)phenyl dodecanoate.

Experimental

Analytical data were obtained on Perkin Elmer 2400 LS series CHNS/O analyzers. Electron impact mass spectra (EI-MS) were recorded by Hewlett Packard 5989A Mass Spectrometer operating at 70 eV ionizing energy. FT-IR data were recorded on a Perkin Elmer 2000-FTIR spectrophotometer. NMR spectra were recorded in $CDCl_3$ on a Bruker 400 MHz Ultrashield Spectrometer.

Scheme 1. Synthesis of 4-({[4-(methylthio)phenyl]methylene}amino)phenyl dodecanoate.



In analogy to a recently published procedure [9-12], a solution of 4-(methylthio)benzaldehyde (0.76 g, 5 mmol) and 4-aminophenol (0.55 g, 5 mmol) in methanol (40 mL) was heated under reflux for an hour. Schiff base **1** thus obtained was recrystallized from absolute ethanol. Then, Schiff base **1** (0.24 g, 1 mmol), was added into a solution of dodecanoic acid (0.26 g, 1.3 mmol) and 4-dimethylaminopyridine (DMAP) (0.02 g, 0.2 mmol) in THF (30 mL). The resulting mixture was stirred in an ice bath. To this solution, N,N'-dicyclohexylcarbodiimide (DCC) (0.21 g, 1 mmol) dissolved in THF (5 mL) was added dropwise while stirring in the ice bath for an hour. The resulting mixture was subsequently stirred at room temperature for another three hours. Then, the reaction mixture was filtered and the excess solvent was removed from the filtrate by evaporation. Recrystallization from hexane and methanol gave the pure Schiff base **2** (0.19 g, 45%).

Melting point: 105–106 ℃.

MS (EI): *m/z* (rel. int. %) = 425 (6) (M^+), 243 (100).

IR (KBr): ν_{max} (cm^{-1}), 2954, 2916, 2848 (C-H aliphatic), 1749 (C=O), 1621 (C=N), 1212, 1085 (C-O).

^{1}H NMR (300 MHz, $CDCl_3$): δ/ppm 0.90 (t, *J* = 6.6 Hz, 3H, C<u>H</u>$_3$-), 1.28–1.43 (m, 16H, CH_3-(C<u>H</u>$_2$)$_8$-), 1.78 (quint, 2H, *J* = 7.5 Hz, -C<u>H</u>$_2$-CH_2-COO-), 2.55 (s, 3H, C<u>H</u>$_3$S-), 2.58 (t, 2H, *J* = 7.5 Hz, -C<u>H</u>$_2$-COO-), 7.11 (d, 2H, *J* = 8.7 Hz, Ar-<u>H</u>), 7.23 (d, 2H, *J* = 8.7 Hz, Ar-<u>H</u>), 7.32 (d, 2H, *J* = 8.4 Hz, Ar-<u>H</u>), 7.81 (d, 2H, *J* = 8.7 Hz, Ar-<u>H</u>), 8.41 (s, 1H, -C<u>H</u>=N-).

^{13}C NMR (100 MHz, $CDCl_3$): δ/ppm 172.40 (-<u>C</u>OO-), 159.06 (-<u>C</u>H=N-), 149.55, 148.75, 143.30, 132.71, 129.10, 125.74, 122.12, 121.89 for aromatic carbons, 34.34 (-<u>C</u>H_2COO-), 31.85 (-<u>C</u>$H_2$$CH_2$COO-), 29.69, 29.65, 29.60, 29.46, 29.35, 29.29, 29.12 for methylene carbons [$CH_3CH_2CH_2$(<u>C</u>H_2)$_6$-), 24.90 (CH_3CH_2<u>C</u>H_2-), 22.63 (CH_3<u>C</u>H_2), 15.03 (-S<u>C</u>H_3), 14.15 (-<u>C</u>H_3).

Elemental analysis: Calculated for $C_{26}H_{35}NO_2S$, C, 73.37%, H, 8.29%, N, 3.29%; Found: C, 73.48%, H, 8.23%, N, 3.33%.

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