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Short Note

2-[4-(Hexadecyloxy)-3-methoxyphenyl]-1,3-benzothiazole

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Abstract: A new heterocycle, 2-[4-(hexadecyloxy)-3-methoxyphenyl]-1,3-benzothiazole, was prepared and its IR, ^{1}H NMR, ^{13}C NMR, elemental analysis and MS spectroscopic data are reported.

Keywords: 2-[4-(hexadecyloxy)-3-methoxyphenyl]-1,3-benzothiazole; heterocycle; terminal ether chain

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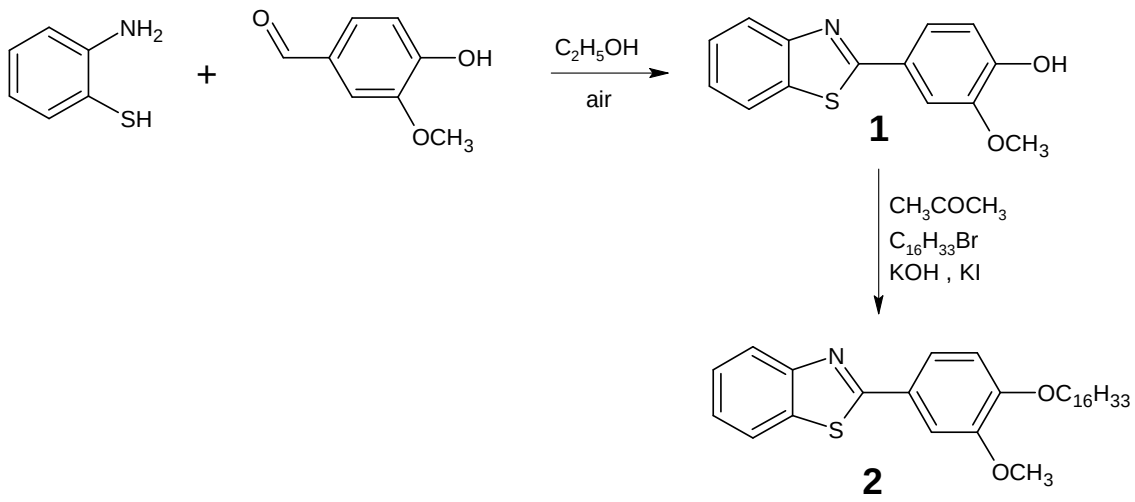
Interest in the study of mesomorphic heterocyclic compounds has dramatically increased in recent years due to their wider range of structure templates, as well as their optical and photochemical properties [1]. Heterocyclic compounds, if designed properly, offer an opportunity to produce metal-containing mesogenic materials (metallomesogens) [2]. By modifying the molecular template of the existing heterocyclic compounds [3–5] to generate calamitic liquid crystals, we synthesized the title compound via (i) cyclization of benzothiazole and followed by (ii) Williamson etherification. Unfortunately, the presence of lateral methoxy group tends to diminish the mesogenic properties by disrupting the molecular packing [6].



In analogy to a recently published procedure [6], 2-aminothiophenol (5.01 g, 40 mmol) and vanillin (6.09 g, 40 mmol) in ethyl alcohol (40 mL) was heated under reflux for 6 h. The reaction mixture was subsequently cooled to room temperature. Then, distilled water (60 mL) was added slowly until the mixture turned cloudy. The mixture was kept overnight at about 20 ^{o}C and benzothiazole **1** formed was filtered and washed with cold ethanol/water (1:1.5) and dichloromethane. Then, benzothiazole **1** (5.15 g, 20 mmol) in acetone (40 mL), was added to a solution of potassium hydroxide (1.12 g, 20 mmol) in distilled water (5 mL). This was followed by addition of a small amount of potassium iodide into the mixture. The reaction mixture was heated under reflux for 1 h with stirring. 1-Bromohexadecane (7.63 g, 25 mmol) was then added to the flask and reflux was continued for 20 h. The solid obtained was filtered and recrystallized from absolute ethanol whereupon the pure compound was isolated as a white solid (6.17 g, 64%).

Melting point: 82 ^{o}C.

MS (EI): M^{+} (m/z) = 481 (50), 257 (100), 228 (8), 57 (9), 43 (15).

IR (KBr, cm^{-1}): 3074, 3000 (C-H aromatic); 2922, 2849 (C-H aliphatic); 1603 (C=N thiazole); 1499 (C=C aromatic); 1267 (C-O ether).

^{1}H NMR (400 MHz, $CDCl_3$): δ/ppm 0.9 (t, 3H, *J* = 7.0 Hz, C<u>H</u>$_3$-), 1.4-1.6 (m, 26H, CH_3-(C<u>H</u>$_2$)$_{13}$-), 1.9 (quint, 2H, *J* = 7.0 Hz, -C<u>H</u>$_2$-CH_2-O-), 4.0 (s, 3H, C<u>H</u>$_3$O-), 4.1 (t, 2H, *J* = 6.9 Hz, -C<u>H</u>$_2$-O-), 7.0 (d, 1H, *J* = 8.4 Hz, Ar-H), 7.4 (t, 1H, *J* = 8.2 Hz, Ar-H), 7.5 (t, 1H, *J* = 8.4 Hz, Ar-H), 7.6 (dd, 1H, *J* = 8.4, 2.1 Hz, Ar-H), 7.7 (d, 1H, *J* = 2.1 Hz, Ar-H), 7.9 (d, 1H, *J* = 8.5 Hz, Ar-H), 8.0 (d, 1H, *J* = 8.1 Hz, Ar-H).

^{13}C NMR (100 MHz, $CDCl_3$): δ/ppm 126.6, 125.2, 123.2, 121.9, 121.5, 112.6, 110.5 for aromatic carbons, 69.5 (-O-<u>C</u>H_2-), 56.6 (<u>C</u>H_3O-), 32.3, 30.1, 30.0, 29.9, 29.8, 29.7, 29.5, 26.3, 23.1[-(<u>C</u>H_2)$_{14}$-CH_2-O-], 14.5 (-CH_3).

Elemental analysis: Calculated for $C_{30}H_{43}NO_2S$: C, 74.80%, H, 9.00%, N, 2.91%; Found: C, 74.93%, H, 8.95%, N, 2.84%.

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