

Short Note

Synthesis of a New Heterocycle with Liquid Crystal Properties: 2-(3-Methoxy-4-hexadecanoyloxyphenyl)benzothiazole

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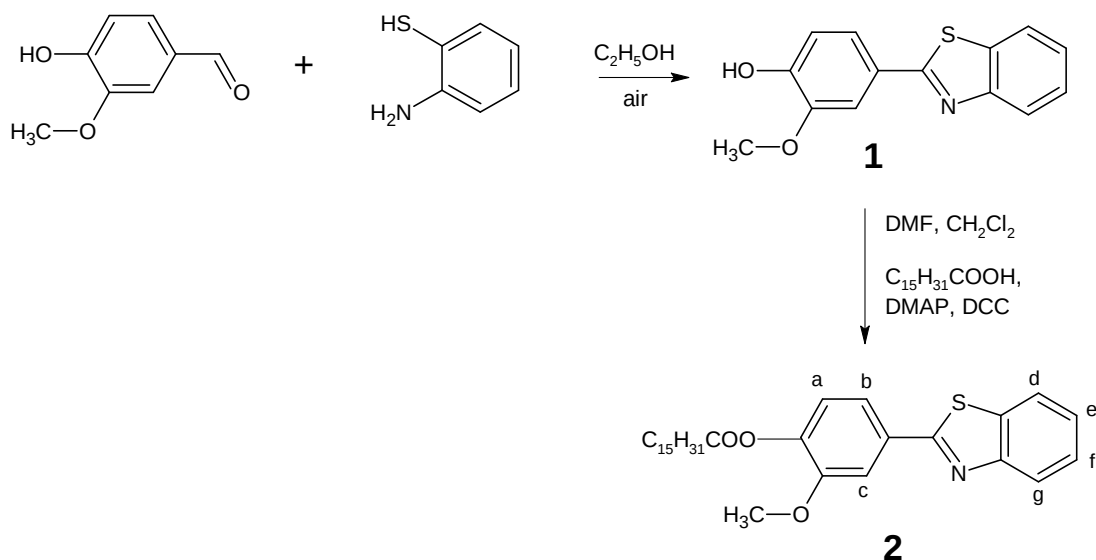
Abstract: A new heterocycle 2-(3-methoxy-4-hexadecanoyloxyphenyl)benzothiazole was synthesized and its IR, ¹H NMR, ¹³C NMR and MS spectroscopic data are presented.

Keywords: 2-(3-Methoxy-4-hexadecanoyloxyphenyl)benzothiazole; heterocycle; alkyl chain, liquid crystal

There has been considerable interest in heterocyclic molecules due to their diversified molecular design and remarkable optical [1-3], liquid crystalline [4] and electronic properties [5]. 2-Heterocyclic benzothiazole has emerged in its usage as a core unit in calamitic liquid crystals. It has been reported that liquid crystalline compounds incorporating a benzothiazole-fused ring exhibit good hole-transporting properties with a low ionization potential, making them of potential interest as hole-transporting materials in organic light-emitting devices (OLEDs) [6-8]. In view of the importance of these compounds, chemists are prompted to generate new derivatives by introducing different substituents into the existing skeleton of the molecule [9-11].

Preparation of Benzothiazole 1 [12,13]: 2-Aminothiophenol (2.50 g, 20 mmol) and vanillin (3.04 g, 20 mmol) in absolute ethanol (40 mL) was heated under reflux for 6 hours. 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 18°C and the solid formed was filtered and washed with cold ethanol/water (1:1.5) and dichloromethane.



Preparation of Benzothiazole 2: Benzothiazole **1** (2.57 g, 10 mmol) in dimethylformamide (DMF) (4 mL) was added to a solution of palmitic acid (2.56 g, 10 mmol) and 4-dimethylaminopyridine (DMAP) (0.61 g, 5 mmol) in dichloromethane (70 mL). The resulting mixture was stirred in an ice bath. To this solution, *N,N'*-dicyclohexylcarbodiimide (DCC) (2.06 g, 10 mmol) dissolved in dichloromethane (20 mL) was added dropwise while stirring in the ice bath for an hour. The resulting mixture was subsequently stirred at room temperature for another 24 hours. Then, the reaction mixture was filtered and the excess solvent was removed from the filtrate by evaporation. Recrystallization from absolute ethanol gave the product as a white solid (1.34 g, 27%).

Melting point: 56.1°C

MS (EI): M^+ (m/z) = 495 (1) $[M]^+$, 257 (100).

IR (KBr, cm^{-1}): 2949, 2915, 2850 (C-H aliphatic); 1764 (C=O ester); 1599 (C=N), 1483 (C=C aromatic), 755, 652 (C-S).

^1H NMR (300 MHz, CDCl_3): δ /ppm 0.88 (t, 3H, $J = 6.6$ Hz, CH_3), 1.22-1.48 {m, 24H, $\text{CH}_3(\text{CH}_2)_{12}$ -}, 1.76 (qt, 2H, $J = 7.3$ Hz, $-\text{CH}_2\text{CH}_2\text{COO}-$), 2.61 (t, 2H, $J = 7.4$ Hz, $-\text{CH}_2\text{COO}-$), 3.99 (s, 3H, OCH_3), 7.15 (d, H_a , $J = 8.2$ Hz, Ar-H), 7.38 (t, H_e , $J = 7.9$ Hz, Ar-H), 7.49 (t, H_f , $J = 8.0$ Hz, Ar-H), 7.60 (dd, H_b , $J = 8.2, 1.8$ Hz, Ar-H), 7.82 (d, H_c , $J = 1.9$ Hz, Ar-H), 7.90 (d, H_d , $J = 7.9$ Hz, Ar-H), 8.08 (d, H_g , $J = 8.1$ Hz, Ar-H).

^{13}C NMR (75 MHz, CDCl_3): δ /ppm 14.57 (CH_3), 23.13, 29.48, 29.72, 29.80, 29.95, 30.05, 30.10, 30.13, 32.36 for methylene carbons $\{\text{CH}_3(\text{CH}_2)_{12}-\}$, 25.42 ($-\text{CH}_2\text{CH}_2\text{COO}-$), 34.47 ($-\text{CH}_2\text{COO}-$), 56.54

(OCH₃), 111.31, 120.92, 122.03, 123.60, 123.77, 125.68, 126.79, 132.74, 135.54, 142.57, 152.06, 154.44, 167.64 for aromatic carbons, 172.02 (COO).

Elemental analysis: Calculated for C₃₀H₄₁NO₃S: C, 72.69%, H, 8.34%, N, 2.83%; Found: C, 72.80%, H, 8.42%, N, 2.72%.

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