

Short Note

## 4-[(1,3-Benzothiazol-2-ylimino)methyl]phenyl Dodecanoate

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**Abstract:** A heterocycle, 4-[(1,3-benzothiazol-2-ylimino)methyl]phenyl dodecanoate, was synthesized and its IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, elemental analysis and MS spectroscopic data are presented. This new compound exhibited smectic A phase.

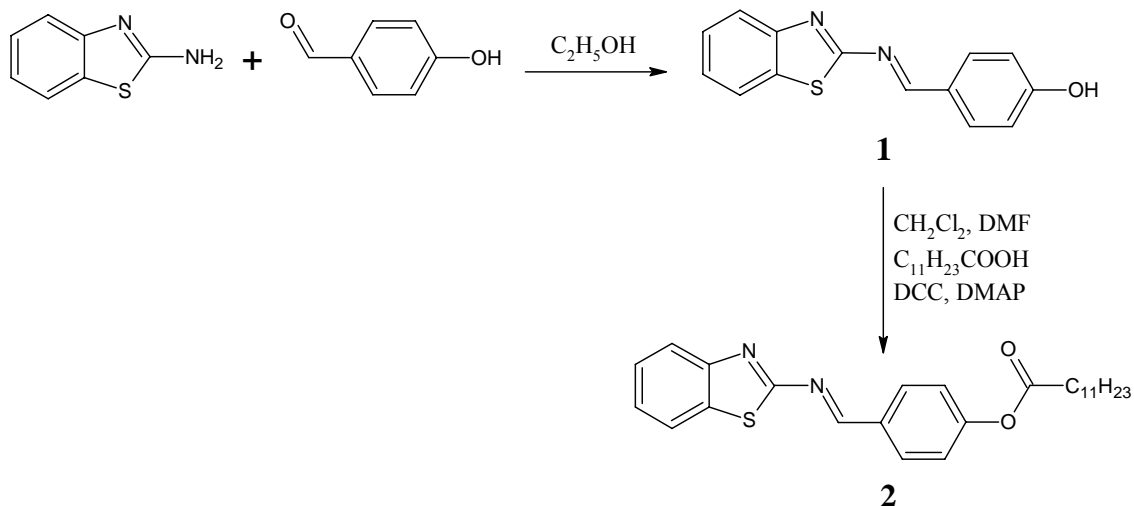
**Keywords:** 4-[(1,3-benzothiazol-2-ylimino)methyl]phenyl dodecanoate; heterocyclic liquid crystal; smectic A

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Schiff bases have attracted much attention ever since the discovery of the first room temperature liquid crystal, 4-methoxybenzylidene-4'-butylaniline [1]. Many kinds of heterocyclic structures, such as pyridine [2], furan [3], thiophene [4] and benzothiazole [5–7] have been introduced as core centre in liquid crystalline compounds. In this paper, we report the synthesis of a new Schiff base comprising the benzothiazole moiety: 4-[(1,3-benzothiazol-2-ylimino)methyl]phenyl dodecanoate. This new compound exhibits enantiotropic smectic A phase, as indicated by thermal (DSC) and polarizing optical microscopy studies.

In analogy to a recently published procedure [8], a solution of 2-aminobenzothiazole (6.01 g, 40 mmol) and 4-hydroxybenzaldehyde (4.88 g, 40 mmol) in absolute ethanol (60 mL) was heated under reflux for 3 h. The solvent was removed by slow evaporation and Schiff base **1** thus obtained was recrystallized from absolute ethanol. Then, Schiff base **1** (5.09 g, 20 mmol) in dimethylformamide (10 mL), was added to a solution of dodecanoic acid (4.01 g, 20 mmol) and 4-dimethylaminopyridine (1.22 g, 10 mmol) in dichloromethane (70 mL). The resulting mixture was stirred in an ice bath. To this solution, N,N'-dicyclohexylcarbodiimide (4.13 g, 20 mmol) in 10 mL of dichloromethane was

added dropwise while stirring in the ice bath for 1 h. The resulting mixture was subsequently stirred at room temperature for another 3 h. Then, the reaction mixture was filtered and the excess solvent was removed from the filtrate by evaporation. Recrystallization from absolute ethanol gave the Schiff base **1** as yellow solid (44%).

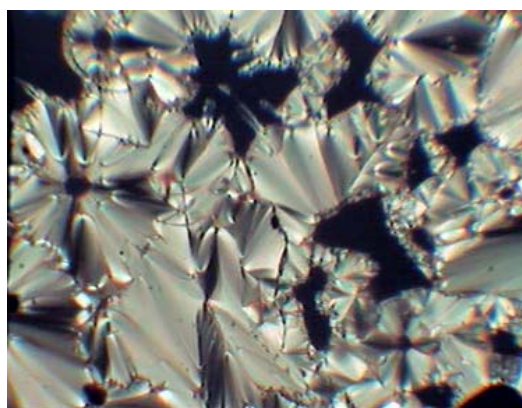


Thermal data obtained from DSC analysis (enthalpy changes,  $kJ\ mol^{-1}$  in bracket):

Heating: Crystal  $80.8\ ^\circ C$  (45.07) Smectic A  $85.6\ ^\circ C$  (7.44) Isotropic.

Cooling: Crystal  $52.7\ ^\circ C$  (37.81) Smectic A  $81.7\ ^\circ C$  (8.17) Isotropic.

Optical photomicrograph showing fan-shaped texture of smectic A phase observed under polarizing optical microscope:



MS (EI):  $m/z = 436$  ( $M^+$ , 9.2%), 254 (100), 225 (8.1), 57 (4.9), 43 (6.4).

IR (KBr,  $cm^{-1}$ ): 3064, 3033 (C-H aromatic), 2922, 2851 (C-H aliphatic), 1747 (C=O ester), 1618 (C=N, imine), 1600 (C=N, thiazole), 1509 (C=C aromatic).

$^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta/ppm$  0.88 (t, 3H,  $J = 7.0$  Hz,  $CH_3$ -), 1.27-1.43 (m, 16H,  $CH_3$ -( $CH_2$ ) $_8$ - $CH_2$ -), 1.70-1.80 (q, 2H,  $J = 7.3$  Hz,  $-CH_2-CH_2-COO-$ ), 2.59 (t, 2H,  $J = 7.6$  Hz,  $-CH_2-COO-$ ),

7.25 (d, 2H,  $J = 6.8$  Hz, Ar-H), 7.37 (t, 1H,  $J = 8.3$  Hz, Ar-H), 7.48 (t, 1H,  $J = 8.3$  Hz, Ar-H), 7.84 (d, 1H,  $J = 8.1$  Hz, Ar-H), 7.99 (d, 1H,  $J = 8.1$  Hz, Ar-H), 8.06 (d, 2H,  $J = 6.8$  Hz, Ar-H), 9.05 (s, 1H, -N=CH-).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta/\text{ppm}$  171.7 (-COO-), 164.8 (C=N), 154.6, 151.6, 134.63, 134.6, 132.2, 131.5, 126.4, 125.1, 123.0, 122.3, 121.6 for aromatic carbons, 34.4, 31.9, 29.5, 29.4, 29.3, 29.2, 29.0, 24.8, 22.6 for methylene carbons [-COO-(CH<sub>2</sub>)<sub>10</sub>-CH<sub>3</sub>], 14.1 (-CH<sub>3</sub>).

Elemental analysis: Calculated for C<sub>26</sub>H<sub>32</sub>N<sub>2</sub>O<sub>2</sub>S: C, 71.52%, H, 7.39%, N, 6.42%; Found: C, 71.65%, H, 7.50%, N, 6.53%.

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### References and Notes

1. Kelker, H.; Scheurle, B. A liquid-crystalline (nematic) phase with a particularly low solidification point. *Angew. Chem. Int. Ed.* **1969**, *8*, 884–885.
2. Naoum, M.M.; Fahmi, A.A.; Alaasar, M.A. Supramolecular hydrogen-bonded liquid crystals formed from 4-(4'-pyridylazophenyl)-4''-alkoxy benzoates and 4-substituted benzoic acids. *Mol. Cryst. Liq. Cryst.* **2008**, *487*, 74–91.
3. Kardas, D.; Mieczkowski, J.; Pocięcha, D.; Szydłowska, J.; Gorecka, E. Synthesis and properties of a new series of mesogenic compounds with pyridine, oxidopyridinium, thienyl and furyl moieties. *J. Mater. Chem.* **2001**, *11*, 741–748.
4. Wu, L.H.; Wang, Y.C.; Hsu, C.S. Synthesis and characterization of thiophene-containing liquid crystals. *Liq. Cryst.* **2000**, *27*, 1503–1513.
5. Belmar, J.; Parra, M.; Zuniga, C.; Perez, C.; Munoz, C. New liquid crystals containing the benzothiazol unit: Amides and azo compounds. *Liq. Cryst.* **1999**, *26*, 389–396.
6. Prajapati, A.K.; Bonde, N.L. Mesogenic benzothiazole derivatives with a polar nitro substituent. *Mol. Cryst. Liq. Cryst.* **2009**, *501*, 72–85.
7. Ha, S.T.; Koh, T.M.; Yeap, G.Y.; Lin, H.C.; Boey, P.L.; Yip, F.W.; Ong, S.T.; Ong, L.K. Synthesis and mesomorphic properties of 2-(4-alkyloxyphenyl)benzothiazoles. *Mol. Cryst. Liq. Cryst.* **2009**, *506*, 56–70.
8. Ha, S.T.; Koh, T.M.; Ong, S.T.; Ong, L.K. Synthesis of a new heterocycle with liquid crystal properties: 2-(3-Methoxy-4-hexadecanoyloxyphenyl)benzothiazole. *Molbank* **2009**, *2009*, M606.