

Short Note

2-(4-Heptyloxyphenyl)benzothiazole

Sie-Tiong Ha^{1,2,*}, Teck-Ming Koh¹, Siew-Teng Ong^{1,2}, Teck-Leong Lee¹ and Yasodha Sivasothy³

¹ Department of Chemical Science, Faculty of Engineering & Science, Universiti Tunku Abdul Rahman, Jln Genting Kelang, Setapak 53300 Kuala Lumpur, Malaysia

² Department of Science, Faculty of Science, Engineering & Technology, Universiti Tunku Abdul Rahman, Jln Universiti Bandar Barat, 31900 Kampar, Perak, Malaysia

³ School of Chemical Sciences, Universiti Sains Malaysia, 11800 Minden, Pulau Pinang, Malaysia

* Author to whom correspondence should be addressed; E-mail: hast@utar.edu.my; hast_utar@yahoo.com

Received: 26 June 2009 / Accepted: 14 July 2009 / Published: 30 July 2009

Abstract: 2-(4-Heptyloxyphenyl)benzothiazole was synthesized and its IR, ¹H NMR and MS spectroscopic data are presented.

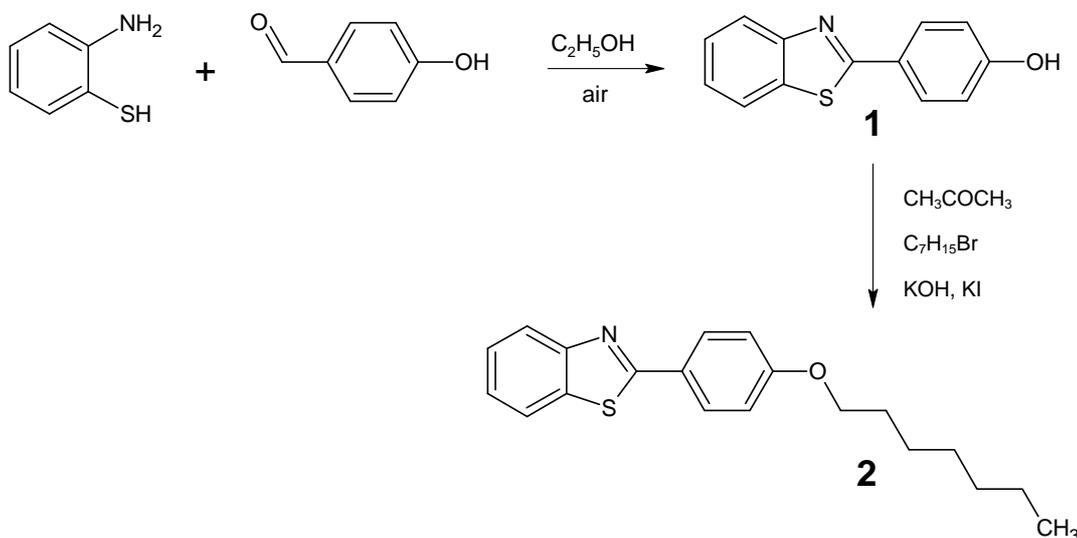
Keywords: 2-(4-heptyloxyphenyl)benzothiazole; heterocycle; ether chain

Benzothiazole-based liquid crystals are known to be good hole-transporting materials with a low ionization potential, making them of potential interest as hole-transporting materials in organic light emitting devices [1-3]. In view of the importance of these compounds, chemists are prompted to generate the derivatives by introducing different substituents into the existing skeleton of the molecule [4-6]. Although the smectogenic 2-(4-heptyloxyphenyl)benzothiazole was reported in a few patents [7-10], the use of dimethyl sulfoxide (DMSO) as a solvent for the formation of benzothiazole is less convenient as DMSO is difficult to be removed from the product (high b.p. of DMSO, 187°C). In this paper, we report the convenient preparation of 2-(4-heptyloxyphenyl)benzothiazole using ethyl alcohol (more volatile solvent) to ensure easy separation of the product. Furthermore, the title compound was obtained in two steps: cyclization to benzothiazole followed by Williamson etherification which is a

reverse method reported in the literature. Thus, we provide an alternative synthetic pathway of 2-(4-heptyloxyphenyl)benzothiazole.

Preparation of Benzothiazole 1

2-Aminothiophenol (5.01 g, 40 mmol) and 4-hydroxybenzaldehyde (4.88 g, 40 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. It was kept overnight at about 20 °C and the solid formed was filtered and washed with cold ethanol:water (1:1.5) and dichloromethane.



Preparation of Benzothiazole 2

In analogy to a recently published procedure [9], benzothiazole **1** (4.55 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 an hour with stirring. 1-Bromoheptane (4.48 g, 25 mmol) was then added to the flask and reflux was continued for 20 hours. The solid obtained was repeatedly recrystallized from absolute ethanol whereupon the pure compound was isolated as a white solid (3.58 g, 55%).

Melting point: 86.9 °C.

EI-MS m/z (rel. int. %): 325 (38) [M^+], 227 (100), 198 (10), 108 (5), 57 (7).

IR (KBr, cm^{-1}): 2921, 2852 (C-H aliphatic); 1603 (C=N); 1259, 1036 (C-O ether).

1H NMR (400 MHz, $CDCl_3$): δ/ppm 0.9 (t, $J = 6.8$ Hz, 3H, CH_3 -), 1.2-1.5 (m, 8H, CH_3 -(CH_2)₄- CH_2 - CH_2 -O-), 1.8 (qt, $J = 7.4$ Hz, 2H, - CH_2 - CH_2 -O-), 4.0 (t, $J = 6.6$ Hz, 2H, - CH_2 -O-), 7.0 (d, $J = 8.8$ Hz,

2H, Ar-H), 7.4 (t, $J = 8.1$ Hz, 1H, Ar-H), 7.5 (t, $J = 8.1$ Hz, 1H, Ar-H), 7.9 (d, $J = 8.0$ Hz, 1H, Ar-H), 8.0 (m, 3H, Ar-H).

Elemental analysis: Calculated for C₂₀H₂₃NOS: C, 73.81%, H, 7.12%, N, 4.30%; Found: C, 73.72%, H, 7.15%, N, 4.39%.

Acknowledgements

One of the authors, S.T. Ha would like to thank Universiti Tunku Abdul Rahman (UTAR) for the financial support through UTAR Research Fund (Vote No. 6200/H02), and the Malaysia Toray Science Foundation (UTAR Vote No. 4359/000) for funding this project. T.M. Koh would like to acknowledge UTAR for the award of the research and teaching assistantships.

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