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Short Note

4-[3-(Pyridin-3-yl)prop-2-enoyl]phenyl 4-dodecyloxybenzoate

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Abstract: Steglich esterification between 1-(4-hydroxyphenyl)-3-(pyridin-3-yl)prop-2-en-1-one and 4-dodecyloxybenzoic acid has produced a new compound, 4-[3-(pyridin-3-yl) prop-2-enoyl]phenyl 4-dodecyloxybenzoate. The title compound was characterized by IR and NMR analysis.

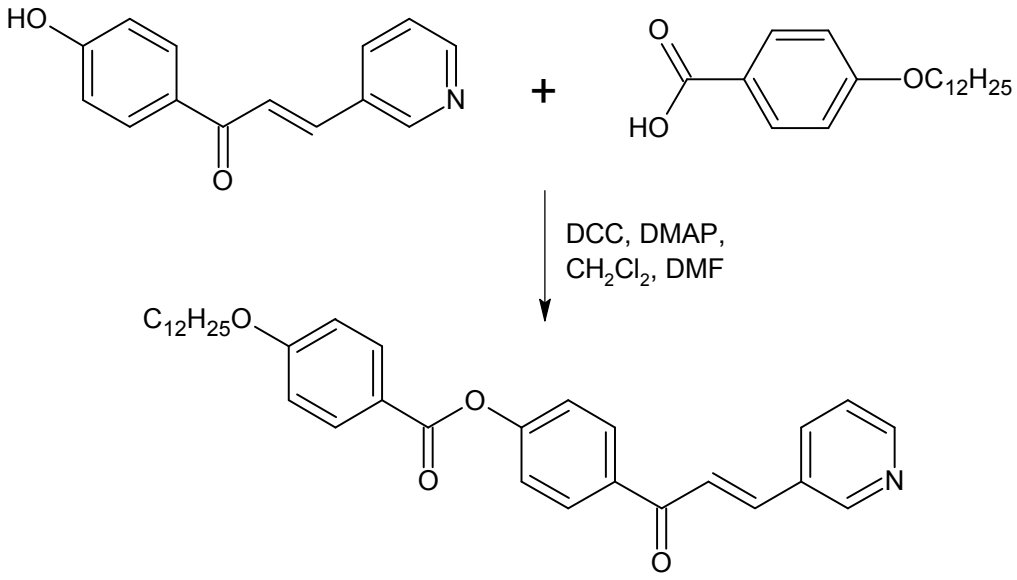
Keywords: pyridine; ester; liquid crystals

During the past decade, heterocyclic liquid crystals have received overwhelming attention due to their unique properties including a reduced packing ability (generally giving rise to lower melting points than their phenyl counterparts), a medium to strong lateral dipole, high anisotropy of the polarizability, low viscosity, *etc.* [1–2]. Our previous works paid attention on the synthesis of smectic liquid crystals containing benzothiazole moiety [3–5]. Heterocyclic pyridine is another type of core system that liquid crystal researchers have been focused on [6,7]. In this paper, we report a new pyridine derivative, 4-[3-(pyridin-3-yl)prop-2-enoyl]phenyl 4-dodecyloxybenzoate. The title compound was prepared according to the reaction steps outlined in Scheme 1.

As described in a recently published procedure [7,8], 1-(4-hydroxyphenyl)-3-(pyridin-3-yl)prop-2-en-1-one (0.45 g, 2 mmol) in dimethylformamide (DMF) (10 mL), was added to a solution of 4-dodecyloxybenzoic acid (0.61 g, 2 mmol) and 4-dimethylaminopyridine (DMAP) (0.12 g, 1 mmol) in dichloromethane (7 mL). The resulting mixture was stirred in an ice bath at 0 °C and this is followed by the addition of *N*,*N*′-dicyclohexylcarbodiimide (DCC) (0.41 g, 2 mmol) which previously dissolved in dichloromethane (10 mL). DCC was added in a dropwise manner into the mixture over a period of one hour under the cooling bath system. The resulting mixture was subsequently stirred at room

temperature for another 12 hours. Then, the reaction mixture was filtered and the excess solvent was removed from the filtrate by evaporation. Recrystallization from chloroform gave the product a white solid consistency (0.28 g, 27%). Main byproducts such as *O*-acylisourea and dicyclohexylurea were formed during the Steglich esterification.

Infrared spectrum, ^{1}H- and ^{13}C-NMR spectra and elemental data were collected using the following models, Perkin-Elmer 2000 FTIR spectrophotometer (Perkin Elmer Sdn Bhd, Malaysia), JEOL LA-400 MHz NMR spectrometer (JEOL Sdn Bhd, Malaysia) and Perkin Elmer 2400 LS Series CHNS/O analyzer (Perkin Elmer Sdn Bhd, Malaysia).



Scheme 1. Synthesis of 4-[3-(pyridin-3-yl)prop-2-enoyl]phenyl 4-dodecyloxybenzoate.

Melting point: 129.8 °C

IR (KBr, cm^{-1}): 3057 (C–H aromatic); 2921, 2852 (C–H aliphatic); 1729 (C=O ester); 1658 (C=O keto), 1277 (C–O, aromatic ether).

^{1}H-NMR (400 MHz, $CDCl_3$): δ/ppm 0.9 (t, 3H, *J* = 6.6 Hz, CH_3-), 1.3-1.5 (m, 18H, CH_3-(CH_2)$_9$-(CH_2)$_2$-O-), 1.8 (m, 2H, *J* = 7.8 Hz, -CH_2-CH_2-O-), 4.0 (t, 2H, *J* = 6.4 Hz, -CH_2-O-), 7.0 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.4 (m, 3H, Ar-H and pyridine-H), 7.6 (d, 1H, *J* = 16 Hz, olefinic-H), 7.8 (d, 1H, *J* = 16 Hz, olefinic), 7.9 (d, 1H, *J* = 8.7 Hz, pyridine-H), 8.1-8.2 (m, 4H, Ar-H), 8.6 (t, 1H, *J* = 5 Hz, pyridine-H), 8.9 (s, 1H, pyridine-H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ/ppm 14.23 (CH_3-), 22.79, 26.06, 29.44, 29.68, 29.73, 32.01 for methylene carbons (CH_3-(CH_2)$_{10}$-), 64.48 (-CH_2O-), 114.50, 120.97, 122.32, 123.70, 123.92 130.30, 130.72, 132.51, 134.73, 135.20, 141.17, 150.09, 151.23, 155.03, 163.92, 164.48 for olefinic, aromatic and ester carbons, 188.74 (C=O keto).

Anal. calculated for $C_{33}H_{39}NO_4$: C, 77.16; H, 7.65; N, 2.73. Found: C, 77.26; H, 7.84; N, 2.61.

IR, ^{1}H-NMR and ^{13}C-NMR spectra for the title compound are available in the Supplementary Information.

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Author Contributions

Sie-Tiong Ha and Yi-Jun Lau contributed equally to this work.

Conflicts of Interest

The authors declare no conflict of interest.

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