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Short Note

Synthesis of New Schiff Base: 4-[(Pyridin-3-ylmethylene)-amino]phenyldodecanoate

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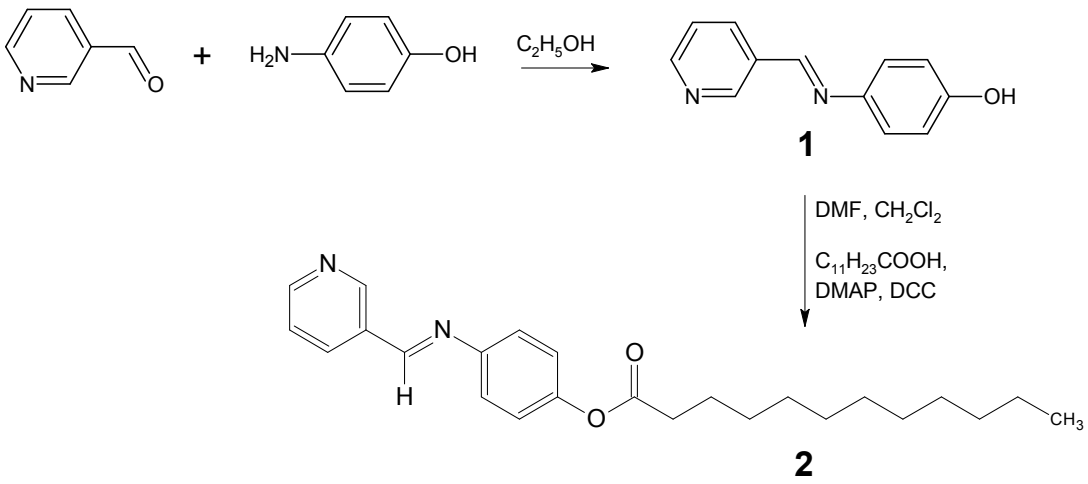
Abstract: A new Schiff base 4-[(pyridin-3-ylmethylene)amino]phenyldodecanoate was synthesized via Steglich esterification. IR, ^{1}H NMR, ^{13}C NMR and MS spectroscopic data are presented.

Keywords: 4-[(Pyridin-3-ylmethylene)amino]phenyldodecanoate; Schiff base, alkyl chain.

N-benzylideneaniline Schiff bases have received a considerable amount of attention from many researchers owing to their importance in exhibiting thermochromism and photochromism [1]. 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. The presence of a long alkyl chain at the *para* position of the aldehyde and aniline fragments of Schiff bases has been regarded as one of the important elements which favours the existence of liquid crystal phases [2-5].



A solution of 3-pyridinecarbaldehyde (4.28 g, 40 mmol) and 4-aminophenol (4.37g, 40 mmol) in absolute ethanol (70 mL) was heated under reflux for 3 hours. Compound **1** thus obtained was recrystallized from absolute ethanol. Then, Schiff base **1** (3.96 g, 20 mmol) in dimethylformamide (DMF) (4 mL), was added to a solution of lauric acid (4.01 g, 20 mmol) and 4-dimethylaminopyridine (DMAP) (1.22 g, 10 mmol) in dichloromethane (70 mL). The resulting mixture was stirred in an ice bath. To this solution, N,N'-dicyclohexylcarbodiimide (DCC) (4.12 g, 20 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 3 hours. 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 **2** as gray solid (2.21 g, 29%).

Melting Point: 80.0°C.

MS(EI): M^+ (m/z) = 380

IR (KBr, cm^{-1}): 2953, 2916, 2849 (C-H aliphatic); 1755 (C=O ester); 1626 (C=N); 1595, 1498 (C=C aromatic).

^{1}H NMR (400 MHz, $CDCl_3$): δ/ppm 0.89 (3H, t, *J* = 7.0 Hz, CH_3), 1.29-1.44 {m, 16H, $CH_3(C\underline{H}_2)_8$-}, 1.77 (qt, 2H, *J* = 7.3 Hz, -$C\underline{H}_2CH_2COO$-), 2.58 (t, 2H, *J* = 7.5 Hz, -$C\underline{H}_2COO$-), 7.14 (dd, 2H, *J* = 8.8 Hz, 2.2 Hz, Ar-H), 7.26 (dd, 2H, *J* = 8.8 Hz, 2.2 Hz, Ar-H), 7.43 (dd, 1H, *J* = 7.8 Hz, 4.8 Hz, Ar-H), 8.30 (dd, 1H, *J* = 7.9, 1.9 Hz, Ar-H), 8.51 (s, 1H, CH=N), 8.70 (dd, 1H, *J* = 4.7 Hz, 1.5 Hz, Ar-H), 9.02 (d, 1H, *J* = 1.9 Hz, Ar-H).

^{13}C NMR (100 MHz, $CDCl_3$): δ/ppm 172.8 (COO), 157.6 (CH=N), 152.5, 151.4, 149.8, 149.3, 135.3, 132.1, 124.2, 122.7 and 122.2 (aromatic carbons), 34.8 (-$\underline{C}H_2COO$-), 25.3 (-$\underline{C}H_2CH_2COO$-), 32.3, 30.0, 29.9, 29.7, 29.6, 29.5, 26.0 and 23.1 ($CH_3(C\underline{H}_2)_8$-), 14.5 (CH_3).

Elemental analysis: Calculated for $C_{24}H_{32}N_2O_2$: C, 75.75%, H, 8.48%, N, 7.36%; Found: C, 75.70%, H, 8.57%, N, 7.41%.

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