

Short Note

3,3'-(1,4-Phenylenebis(phenylazanediyl))bis(7-ethoxy-4-methyl-2H-chromen-2-one)

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Abstract: 3,3'-(1,4-Phenylenebis(phenylazanediyl))bis(7-ethoxy-4-methyl-2H-chromen-2-one) was synthesized from *N,N'*-diphenylbenzene-1,4-diamine and bromo-7-ethoxy-4-methylcoumarin based on Ullmann coupling reaction. The synthesized compound was characterized by UV-Visible, NMR, FT-IR, MS, elemental analysis and Fluorescence Spectrum.

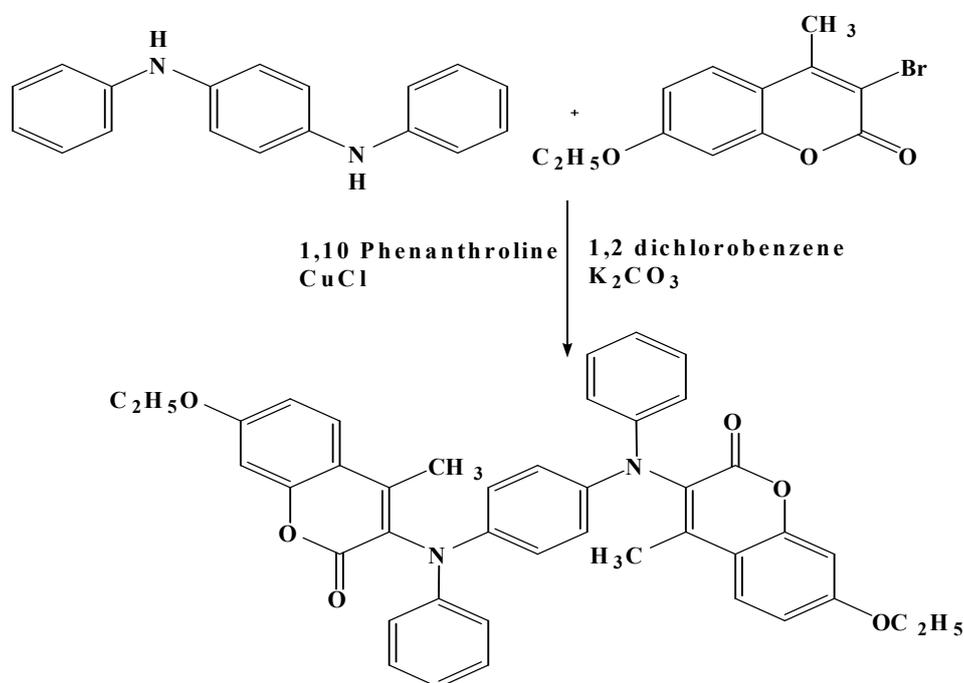
Keywords: coumarin; phenylenediamine; Ullmann coupling reaction; hole transporting material

The title compound 3,3'-(1,4-phenylenebis(phenylazanediyl))bis(7-ethoxy-4-methyl-2H-chromen-2-one) is used as an efficient hole and electron transporting materials in optoelectronic devices. Coumarin and its derivatives occur widely in nature, and have been extensively exploited in biological, chemical and physical fields [1]. Coumarins have outstanding optical properties, including an extended spectral range, high quantum yields, superior photostability and good solubility in common solvents. Many natural and synthetic coumarin derivatives are widely used as electroluminescent (EL) materials [2]. Coumarin derivatives have attracted much interest owing to their potential applications in organic light-emitting diodes (OLEDs) [3]. The arylamine moiety fulfills the requirement of easy and reversible oxidation and therefore constitutes the building block of many of the hole-transporting materials [4].

N,N'-diphenylbenzene-1,4-diamine was synthesized from p-phenylenediamine and bromobenzene by known procedure [5]. A yellow solid with melting point 135 °C was obtained. 3-bromo-7-ethoxy-4-methyl coumarin was synthesized from 3-bromo-7-hydroxy-4-methylcoumarin and ethyl bromide [6]. A brown solid was obtained which has a melting point of 72 °C. *N,N'*-diphenylbenzene-1,4-diamine

(0.05 mol), 3-bromo-7-ethoxy-4-methylcoumarin (0.1 mol), CuCl (0.02 g, 0.0002 mol), 1 g of 1,10 phenanthroline, 5.1 g of potassium carbonate (dried at 110 °C) were refluxed together with 25 mL dichlorobenzene for 18 h at 170 °C in oil bath under nitrogen atmosphere. Completion of the reaction was checked by TLC. After removal of the solvent in vacuo, ammonia solution (50 mL) was added and the mixture was left to stand for 2.0 h. Ethyl acetate (150 mL) and water (100 mL) were added. The organic phase was separated, washed with water (100 mL × 2) and brine solution (100 mL), dried over anhydrous sodium sulphate, filtered and the solvent was removed in vacuum. The product was purified with column chromatography using ethyl acetate–hexane as eluent to obtain brown solid which was recrystallized from hexane [7].

Scheme1. Synthesis of 3,3'-(1,4-phenylenebis(phenylazanediyl))bis(7-ethoxy-4-methyl-2H-chromen-2-one).



Yield: 40.15%.

M.p.: 109 °C.

IR (KBr) cm⁻¹: 3074, 2995, 2891, 2880, 1720, 1578, 1433, 1390, 1265, 1217, 1145, 821.

¹H-NMR (500 MHz, CDCl₃) δ ppm: 1.33 (6H, methyl protons, t, *J* = 7 Hz), 1.71 (6H, methyl protons, s), 3.98 (4H, methylene protons, q, *J* = 7.2 Hz), 6.21–7.52 (18H, Ar, m), 6.71 (2H, Ar, s).

¹³C-NMR (75 MHz, DMSO-*d*₆): 14.3, 16.1, 65.1, 107.0, 110.9, 117.9, 118.0, 118.8, 119.4, 125.0, 125.9, 127.2, 129.4, 132.7, 143.2, 151.4, 158.4, 162.0.

UV-Vis. (ethanol, nm): 400, 359, 324, 259. λ_{emission}: 485 nm. (λ_{excitation} = 405 nm).

MS: *m/z* (ESI-MS), 665.2 [(M+1)⁺].

Elemental analysis calculated for C₄₂H₃₆N₂O₆ (664.26): C, 75.89%; H, 5.46%; N, 4.21%; O, 14.44%. Found: C, 75.25%; H, 5.67%; N, 4.32%; O, 14.76%.

Acknowledgements

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