

3-(2,5-Diethoxy-4-iodo-phenylethynyl)-[1,10]-phenanthroline

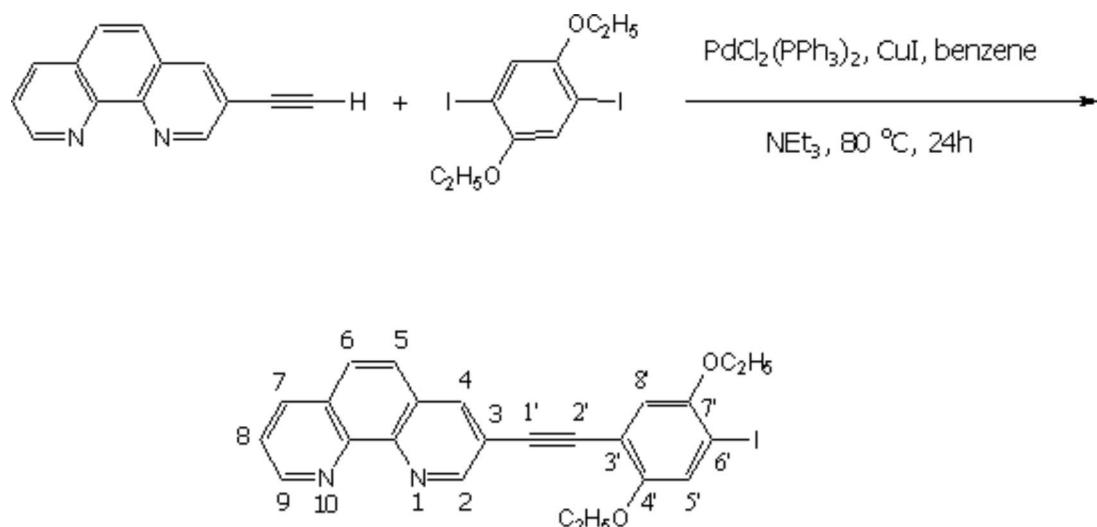
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The experimental procedure follows a protocol developed by Sonogashira [1]. All reactions were carried out under the atmosphere of dry argon by using standard Schlenk tube techniques.

To a mixture of 3-ethynyl-[1,10]-phenanthroline [2,3] (306 mg, 1.5 mmol) and 1,4-diethoxy-2,5-diodobenzene (3.135 g, 7.5 mmol) in dry benzene (25 mL), and triethyl amine (10 mL), were added CuI (28.6 mg, 0.15 mmol), and $[\text{PdCl}_2(\text{PPh}_3)_2]$ (52.6 mg, 0.075 mmol). The reaction mixture was kept at 80 °C for 24h while stirring vigorously and monitored with mass spectrometer to see the formation of the desired product. After removal of the solvent, the residue was washed with aqueous potassium cyanide (2%, 30 mL) and distilled water (100 mL), and purified by column chromatography (SiO_2 , CHCl_3) to collect 3-(2,5-diethoxy-4-iodo-phenylethynyl)-[1,10]-phenanthroline (222 mg, 0.45 mmol, 30%).

Melting Point: > 300°C.

IR (KBr, cm^{-1}): 3205, 2202, 1590, 1477, 1415, 1261, 1202, 1095, 1053, 940, 818, 729.

$^1\text{H-NMR}$ (200 MHz, d_6 -acetone): $\delta = 1.3$ (6H, 2 CH_3); 4 (4H, 2 CH_2); 6.6 (1H, 8'-H); 7.0 (1H, 5'-H); 7.25 (1H, 8-H); 7.4 (1H, 5-H); 7.7 (1H, 6-H); 8.0 (1H, 7-H); 8.2 (1H, 4-H); 8.8 (1H, 9-H); 9.0 (1-H, 2-H).

Elemental Analysis: Calculated for $\text{C}_{24}\text{H}_{19}\text{IN}_2\text{O}_2$: C, 58.31%; H, 3.87%; N, 5.67%. Found: C, 58.0%; H, 4.0%; N, 5.8%.

Acknowledgements

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