The experimental procedure follows a protocol developed by Sonogashira [1]. All reactions were carried out under an atmosphere of dry argon by using standard Schlenk tube techniques. To a mixture of 3-ethynyl-[1,10]phenanthroline (309 mg, 1.50 mmol), 1,4-diiodobenzene (2.47 g, 7.50 mmol), CuI (225 mg, 1.77 mmol) and [PdCl$_2$(PPh$_3$)$_2$] (75.0 mg, 99.7 m mol) in dry benzene (25 mL) and dry NEt$_3$ (10 mL), the reaction mixture was kept at 80 °C for 22 h while stirring vigorously. After removal of the solvent the residue was washed with aqueous KCN (2%, 30 mL) and purified by column chromatography (SiO$_2$, CHCl$_3$, $R_f$ = 0.21) to furnish 3-(4-iodo-phenylethynyl)-[1,10]phenanthroline as a yellow solid (122 mg, 20%).

Mp. > 300 °C.

IR (KBr): $\tilde{\nu} = 3205$ (s, C-H), 2202 (w, Cº C), 1590 (m, C=C), 1477 (s, C=C), 1415 (s), 1261 (m), 1095 (m), 1053 (m), 1202 (s), 940 (m), 818 (s, Ar-H), 729 (s, Ar-H) cm$^{-1}$.

$^1$H NMR (d$_6$-acetone, 250 MHz): $d = 7.47$ (d, $J = 8.6$ Hz, 2 H, 4'-H), 7.77 (dd, $J_1 = 8.3$ Hz, $J_2 = 4.0$ Hz, 1 H, 8-H), 7.88 (d, $J = 8.6$ Hz, 2 H, 5'-H), 7.98 (d, $J = 8.9$ Hz, 1 H, 5-H), 8.04 (d, $J = 8.9$ Hz, 1 H, 6-H), 8.47 (dd, $J_1 = 8.3$ Hz, $J_2 = 1.5$ Hz, 1 H, 7-H), 8.62 (d, $J = 2.2$ Hz, 1 H, 4-H), 9.14 (dd, $J_1 = 4.0$ Hz, $J_2 = 1.5$ Hz, 1 H, 9-H), 9.19 (d, $J = 2.2$ Hz, 1 H, 2-H).

$^{13}$C NMR (d$_6$-acetone, 53 MHz): $d = 88.6$ (C-2'), 93.0 (C-1'), 95.5 (C-6'), 119.7 (C-3), 123.0 (C-8), 124.3 (C-4a), 127.1 (C-6a), 128.6 (C-5), 128.8 (C-6), 130.2 (C-4'), 134.2 (C-5'), 136.9 (C-7), 138.9 (C-3'), 139.0 (C-4), 146.0 (C-10a), 146.9 (C-1a), 159.4 (C-9), 160.5 (C-2).


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Reference


Sample Availability: Available from the authors and from MDPI.

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