

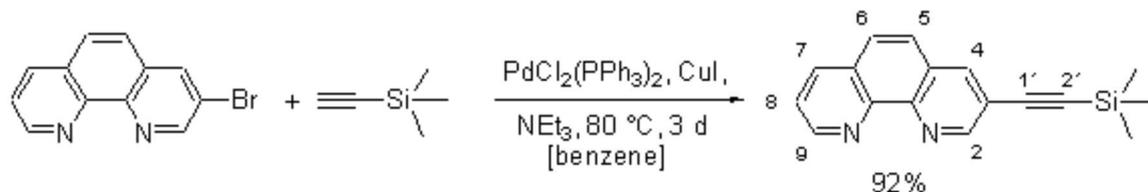
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3-Trimethylsilylanylethynyl-[1,10]phenanthroline

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The experimental procedure follows a protocol developed by Sonogashira [1] and recently used for the preparation of macrocyclic phenanthrolines.[2] All reactions were carried out under an atmosphere of dry argon by using standard Schlenk tube techniques. At first a solution of 3-bromo-[1,10]phenanthroline [3] (4.60 g, 18.0 mmol) and trimethylsilylanylethyne (5.30 g, 54.0 mmol) in benzene (30 mL) and dry triethylamine (15 mL) was prepared. After addition of PdCl₂(PPh₃)₂ (630 mg, 900 μmol) and CuI (523 mg, 1.79 mmol) the resulting mixture was kept at reflux for three days. After removal of the solvent, the black residue was dissolved in dichloromethane (150 mL), washed with aqueous potassium cyanide (2%, 100 mL) and with water (100 mL). The organic layer was dried over MgSO₄ and then purified by column chromatography (SiO₂, 1. CH₂Cl₂, 2. diethylether, R_f(Et₂O) = 0.10) to furnish 4.57 g (92%) of the title compound as colorless crystals.

Mp. 136 °C.

IR (KBr): n= 3056, 2956, 2154 (C° C), 1589, 1494, 1420, 1248, 1100, 863, 838, 731, 656 cm⁻¹.

¹H NMR (CDCl₃, 250 MHz): d = 0.29 (s, 9 H, Si(CH₃)₃), 7.58 (dd, J = 8.0 Hz, J = 4.3 Hz, 1 H, 8-H), 7.63 (d, J = 9.8 Hz, 1 H, 6-H), 7.74 (d, J = 9.8 Hz, 1 H, 5-H), 8.18 (dd, J = 8.0 Hz, J = 1.8 Hz, 1 H, 7-H), 8.28 (d, J = 2.1 Hz, 1 H, 4-H), 9.15 (dd, J = 4.3 Hz, J = 1.8 Hz, 1 H, 9-H), 9.16 (d, J = 2.1 Hz, 1 H, 2-H).

¹³C NMR (CDCl₃, 53 MHz): d = -0.2 (Si(CH₃)₃), 99.4 (C-1'), 101.7 (C-2'), 119.4 (C-3), 123.2 (C-8), 125.9 (C-4a), 127.2 (C-6a), 127.5 (C-5), 128.9 (C-6), 136.0 (C-7), 138.6 (C-4), 144.7 (C-10a), 145.8 (C-1a), 150.5 (C-9), 152.3 (C-2).

Anal. Calcd for C₁₇H₁₆N₂Si · 0.25 H₂O, C: 72.69, H: 5.92, N: 9.97. Found C: 72.63, H: 6.04, N: 10.06.

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3. Tzalis, D.; Tor, Y.; Failla, S.; Siegel, J.S. *Tetrahedron Lett.* **1995**, *36*, 3489.

Sample Availability: Available from the authors and from MDPI.

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