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3-Ethynyl-[1,10]phenanthroline

Christoph Michel, Davood Habibi and Michael Schmittel*

FB 8 - OC 1 (Chemie - Biologie), Universität GH Siegen, Adolf-Reichwein-Str., D-57068 Siegen, Germany, E-mail: schmittel@chemie.uni-siegen.de

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The experimental procedure follows a protocol developed by Eaborn [1]. To 3-trimethylsilanylethynyl-[1,10]phenanthroline (500 mg, 21.7 mmol), dissolved in THF (10 mL), was added 1 M KOH in methanol (10 mL). The resultant solution was stirred for 24 hours at room temperature. After addition of water (10 mL) the product was extracted with CH$_2$Cl$_2$ (3 x 10 mL). Removal of the solvent in vacuo afforded 426 mg of 3-ethynyl-[1,10]phenanthroline (96%) as a colorless solid.

Mp. 279 °C.

IR (KBr): $\tilde{\nu}$ = 3140 (s, C-H), 2086 (w, Cº C), 1588 (m, C=C), 1551 (m, C=C), 1499 (s, C=C), 1418 (s), 1264 (m), 1222 (s), 1096 (m), 904 (m), 838 (s, Ar-H), 729 (s, Ar-H) cm$^{-1}$.

$^1$H NMR (CDCl$_3$, 250 MHz): $\delta$ = 3.34 (s, 1 H, 2'-H), 7.58 (dd, $J_1 = 8.3$ Hz, $J_2 = 4.1$ Hz, 1 H, 8-H), 7.66 (d, $J = 8.8$ Hz, 1 H, 5-H), 7.73 (d, $J = 8.8$ Hz, 1 H, 6-H), 8.15 (dd, $J_1 = 8.3$ Hz, $J_2 = 1.5$ Hz, 1 H, 7-H), 8.27 (d, $J = 2.2$ Hz, 1 H, 4-H), 9.14 (dd, $J_1 = 4.1$ Hz, $J_2 = 1.5$ Hz, 1 H, 9-H), 9.17 (d, $J = 2.2$ Hz, 1 H, 2-H).

$^{13}$C NMR (CDCl$_3$, 53 MHz): $\delta$ = 80.5 (C-1’), 81.5 (C-2’), 118.3 (C-3), 123.2 (C-8), 125.8 (C-4a), 127.3 (C-6a), 127.4 (C-5), 128.9 (C-6), 136.0 (C-7), 138.9 (C-4), 144.9 (C-10a), 145.6 (C-1a), 150.4 (C-9), 152.2 (C-2).


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Reference


Sample Availability: Available from the authors and from MDPI.

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