1-(Pyridin-3-yl)-4-(triethylsilyloxy)-2-butyn-1-ol

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The general part of the experimental section [1] has been presented elsewhere. \(^{9}\)BuLi (1.6 M) (1.9 ml, 5.9 mmol) was added dropwise to a solution of silyl ether 1 (500 mg, 2.94 mmol) in THF (10 mL) at 78 °C under nitrogen and stirred for 45 min. Pyridine-3-carboxaldehyde 2 (315 mg, 2.94 mmol) was added dropwise and the resultant solution stirred for 1.5 h. The mixture was warmed to room temperature then water (10 mL) was added. The mixture was extracted with ethyl acetate (4 x 15 mL) and dried over magnesium sulfate. The solvent was removed under reduced pressure to give a yellow oil which was purified by flash chromatography using hexane-ethyl acetate (1:2) as eluent to afford the title compound 3 (773 mg, 95%) as a pale yellow oil.

IR (neat): 3308b, 2957s, 2913s, 2877s, 2235w, 1741s, 1591m, 1243s.

\(^1\)H NMR (200 MHz, CDCl\(_3\)): 0.63 [6H, q, \(J = 8.0\) Hz, OSi(CH\(_2\)CH\(_3\))\(_3\)], 0.96 [9H, t, \(J = 8.0\) Hz, OSi(CH\(_2\)CH\(_3\))\(_3\)], 4.40 (2H, d, \(J = 1.8\) Hz, H4), 5.56 (1H, br. s, H1), 7.31 (1H, m, H5'), 7.88 (1H, ddd, \(J = 5',4',7.4\), \(J = 4',6'1.7\) and \(J = 2',4'1.7\) Hz, H4'), 8.56 (1H, dd, \(J = 6',4'1.7\) and \(J = 6',5'4.8\) Hz, H6'), 8.75 (1H, d, \(J = 2',4'1.7\) Hz, H2').

\(^1\)C NMR (50 MHz, CDCl\(_3\)): 4.3 [CH\(_2\), OSi(CH\(_2\)CH\(_3\))\(_3\)], 6.6 [CH\(_3\), OSi(CH\(_2\)CH\(_3\))\(_3\)], 51.3 (CH\(_2\), C4), 62.0 (CH, C1), 83.8 (quat., C3), 85.4 (quat., C2), 123.5 (CH, C5'), 131.8 (CH, C4'), 136.9 (CH, C6'), 147.7 (CH, C2'), 148.7 (quat., C3').

EI-MS: 277 (M\(^+\), 10%), 248 (M\(^+\)-C\(_2\)H\(_5\), 47%), 162 (M\(^+\)-Si(CH\(_2\)CH\(_3\))\(_3\), 66%), 146 (70%), 117 (59%), 103 (84%), 75 (100%).

Anal. Calc. For C\(_{15}\)H\(_{23}\)NO\(_2\)Si, 277.14981; found M\(^+\), 277.14918.

Reference

Sample availability: available from the authors and MDPI.

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