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3,5-Bis(trimethylsilyl)-4-thiophenoxypyridine

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Reaction of 3,5-bis(trimethylsilyl)-4-chloropyridine [1] with thiophenol gave 3,5-bis(trimethylsilyl)-4-thiophenoxypyridine, a potential precursor to novel pyridinylidene derived nonlinear optical materials.

To an ice-cooled, stirred solution of 3,5-bis(trimethylsilyl)-4-chloropyridine (2.57 g, 10 mmol) in triethylamine (2 mL) was added portion-wise over 5 min neat thiophenol (1.38 g, 1.29 mL, 12.5 mmol). The mixture became quite viscous and a further 2-3 mL of triethylamine was added to ensure efficient mixing of the solution. The mixture was refluxed overnight, cooled, diluted with water (50 mL) and extracted with chloroform (3 x 50 mL). The combined organic extracts were washed with water (50 mL), dried (Na$_2$SO$_4$) and concentrated under vacuum to an orange oil. Flash column chromatography, eluting initially with hexanes to remove unchanged thiophenol and then with ethyl acetate:hexanes (1:4) gave, after concentration of the most mobile fraction under vacuum, a light yellow-orange oil. Prolonged pumping under vacuum gave a pale yellow solid (2.35 g) which was recrystallised (hexanes) to give the desired product as colourless prisms (1.61 g, 49%).

M.p. 67-69 °C.

IR (nujol): 1583, 1520, 1368, 1181, 1066, 1025, 857.

$^1$H NMR (300 MHz, CDCl$_3$): 0.00, s, 18H; 6.46-6.49, m, 2H; 6.79-6.84, m, 1H; 6.90-6.95, m, 2H; 8.55, s, 2H.

$^{13}$C NMR (75 MHz, CDCl$_3$): d 0.0 (CH$_3$), 124.8 (CH), 125.3 (CH), 128.7 (CH), 139.6 (C$_Q$), 142.3 (C$_Q$), 153.6 (C$_Q$), 156.6 (CH).

HRMS (FAB): Found: MH$^+$ m/z 332.13296 C$_{17}$H$_{25}$NSSi$_2$ requires 332.13191 D = 3.2 p.p.m.

Anal. Calc for C$_{17}$H$_{25}$NSSi$_2$ C 61.57, H 7.60, N 4.22. Found C 61.31, H 7.42, N 4.27.

Reference


Sample Availability: Available from the authors and from MDPI.

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