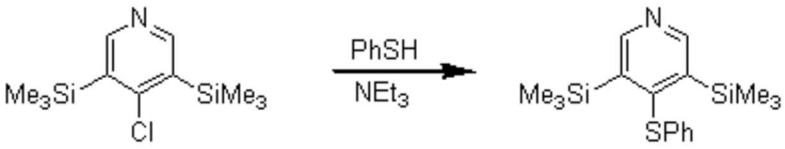
Molecules **2001**, *6*, M266

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

A.J. Kay and A.D. Woolhouse*

Industrial Research Ltd., P.O.Box 31 310, Lower Hutt, New Zealand
E-mail: t.woolhouse@irl.cri.nz

Received: 8 June 2001 / Accepted: 15 December 2001 / Published: 20 December 2001



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_2SO_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

1. Kay, A. J.; Woolhouse, A. D. *Molecules* **2001**, 6, M241.

Sample Availability: Available from the authors and from MDPI.