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

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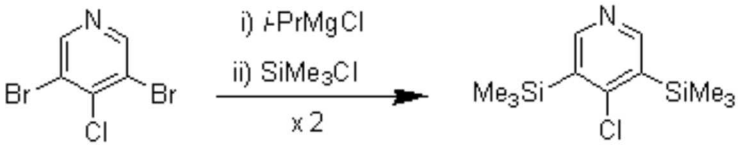

In connection with our studies of the nonlinear optical properties of pyridinylidene compounds we required some pyridine derivatives bearing bulky substituents at the 3- and 5-positions in addition to having an efficient leaving group at the 4-position. To this end we synthesised 3,5-bis(trimethylsilyl)-4-chloropyridine using a bromine-magnesium exchange method recently described [1].

To a stirred solution of 3,5-dibromo-4-chloropyridine [2] (5.27 g, 19.4 mmol) in anhydrous thf (20 mL) under a N_2 atmosphere and at room temperature was added dropwise a solution of isopropyl magnesium chloride in thf (10.2 mL, 1.91 M, 19.5 mmol). After 1 h, chlorotrimethylsilane (2.12 g, 2.47 mL, 19.5 mmol) was added and the mixture stirred overnight at room temperature. A further portion of isopropyl magnesium chloride (19.5 mmol) was then added, followed by a second addition of chlorotrimethylsilane (19.5 mmol) 1 h later. The mixture was stirred overnight, poured into water (50 mL) and extracted with dichloromethane (3 x 50 mL). The combined organic extracts were washed with water (50 mL), dried ($MgSO_4$) and concentrated under vacuum to a brown oil. This was subjected to flash chromatography (hexanes elution) to give 3,5-bis(trimethylsilyl)-4-chloropyridine as a white solid (3.47 g, 70 %). Recrystallisation (benzene) gave colourless crystals.

M.p. 68-70 ° C.

MS: Found: MH^+ m/z 258.08984 $C_{11}H_{20}ClNSi_2$ requires 258.08956 D = 0.8 p.p.m.

^{1}H NMR (300 MHz, $CDCl_3$): d 0.20, s, 18 H; 8.33, s, 2H.

^{13}C NMR (75 MHz, $CDCl_3$): d 0.0 (CH_3), 134.5 (C_Q), 156.9 (CH), 158.9 (C_Q).

IR (nujol): 1535, 1404, 1250, 1055.

References

1. Trecourt F.; Breton G.; Bonnet V.; Mongin F.; Marsais F.; Queguiner G. *Tetrahedron* **2000**, *56*, 1349.
2. den Hertog, H. J.; Hoogzand, C. *Recl. Trav. Chim Pays-Bas***1957**, *76*, 261.

Sample Availability: Available from the authors and from MDPI.

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