3,8-Bis-trimethylsilyl[1,10]-phenanthroline

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Received: 13 June 2005 / Accepted: 8 August 2005 / Published: 1 September 2005

Keywords: trimethylsilyl[1,10]-phenanthroline, Sonogashira procedure, phenanthroline

The experimental procedure follows a protocol developed by Sonogashira [1] and recently used for the preparation of macrocyclic phenanthrolines [2]. All reactions were carried out under the atmosphere of dry argon by using standard Schlenk tube techniques.

At first, a solution of 3,8-dibromo-[1,10]phenanthroline [3] (6.085 g, 18.0 mmol) and trimethylsilyl[1,10]-phenanthroline (7.055 g, 72.0 mmol) in benzene (30 mL) and dry triethylamine (15 mL) was prepared. After addition of PdCl$_2$(PPh$_3$)$_2$ (703 mg, 1.8 mmol) and Cul (686 mg, 3.6 mmol), the resulting mixture was kept at reflux for three days. After removal of the solvent, the black residue was dissolved in dichloromethane (150 mL), and washed with aqueous potassium cyanide (2%, 100 mL) and water (100 mL). The organic layer was dried over MgSO$_4$ and then purified by column chromatography (SiO$_2$, 1. CH$_2$Cl$_2$, 2. diethylether) to furnish 6.04 g (90%) of the title compound as colorless crystals.

Melting Point: ~150 °C.

IR (KBr, cm$^{-1}$): 3056, 2956, 2154, 1589, 1494, 1420, 1248, 1100, 863, 838, 731, 656.

$^1$H-NMR (200 MHz, CDCl$_3$): δ= 0.1 (18H, 6CH$_3$); 7.5 (2H, 5-H & 6-H); 8.2 (2H, 4-H & 7-H); 9.0 (2H, 2-H & 9-H).

Elemental Analysis: Calculated for C$_{22}$H$_{24}$N$_2$Si$_2$: C, 70.91%; H, 6.49%; N, 7.52%. Found: C, 70.7%; H, 6.4%; N, 7.8%.

Acknowledgements

The author gratefully acknowledges financial support from the Bu Ali Sina University, Hamedan, Iran.

References


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