Supporting Information

Direct Synthesis of Porous Organosilicas Containing Chiral Organic Groups within their Framework and a New Analytical Method for Enantiomeric Purity of Organosilicas

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Experimental section

Preparation of (*R*)-1-phenyl-1,2-bis(trimethoxysilyl)ethane

(*R*)-(+)-1-phenyl-1,2-bis(trimethoxysilyl)ethane was prepared from (*E*)-1-phenyl-2-trichlorosilylethene according to the reported procedures. To a mixture of $[PdCl(\pi\text{-}C_3H_5)]_2$ (65.9 mg, 0.18 mmol), (*R*)-(+)-2-bis[3,5-bis(trifluoromethyl)phenyl]phosphino-1,1'-binaphthyl (245 mg, 0.36 mmol), and (*E*)-1-phenyl-2-trichlorosilylethene (14.3g, 60 mmol) was added trichlorosilane(27.0 ml, 270 mmol) at 0 °C, and the mixture was stirred at 20 °C for 48 h. The excess trichlorosilane was removed under reduced pressure and the residue was dissolved in diethyl ether (180 mL). To the solution was added distilled methanol (36 mL), and triethylamine (27 mL) at 0 °C. The mixture was stirred at 50 °C for 14 h and then was filtered through Celite plug. The filtrate was concentrated in vacuo, and was distilled under reduced pressure (150℃/0.1 mmHg) to give 18.9 g (91% yield and 95% ee) of (*R*)-(+)-1-phenyl-1,2-bis(trimethoxysilyl)ethane.

(*R*)-(+)-1-phenyl-1,2-bis(trimethoxysilyl)ethane: $[\alpha]_D^{20}$ +17.1 (*c* 1.03, chloroform); ^1H NMR ($CDCl_3$) δ 1.16 (dd, *J* = 15.7, 12.1 Hz, 1H), 1.27 (dd, *J* = 15.7, 3.3 Hz, 1H), 3.32 (s, 9H), 3.47 (s, 9H), 7.08-7.13 (m, 1H), 7.24-7.26 (m, 4H); $^{13}C\{^1H\}$ NMR δ 10.2, 25.1, 50.2, 51.1, 125.1, 128.1, 128.5, 142.7; $^{29}Si\{^1H\}$ NMR δ -50.8, -43.7. Anal. Calcd for $C_{14}H_{26}O_6Si_2$: C, 48.53; H, 7.56. Found: C, 48.78; H, 7.83.

Determination of enantiomeric purity of constituent unit.

Acetonitrile (0.7 mL) and hydrofluoric acid 48 wt. % in water (0.1 mL) were added to porous organosilica (10 mg, 0.1mmol) in polypropylene tube and the mixture was stirred at room temperature for 15 h. To the solution were added the mixture of potassium fluoride (70mg, 1.2mmol), potassium hydrogen carbonate (601mg, 6.0mmol), and 30% aqueous hydrogen peroxide (0.6 mL) at 0 ^{o}C, and the mixture was stirred at 50 ^{o}C for 5 h. The mixture was filtered through Celite plug. The filtrate was concentrated in vacuo and the residue was purified by silica gel preparative thin-layer chromatography (hexane/ethyl acetate = 1/1) to give 3.1 mg of 1-phenyl-1,2-ethanediol. The enantiomeric purity of 1-phenyl-1,2-ethanediol was determined to be 95% by HPLC analysis with Daicel Chiralcel OB-H (hexane/2-propanol = 9/1).