Supporting Information

Double Silylation of Olefins with HSiCl_3 in the Presence of Bu_4PCl . New Synthetic Method for α,β -Bis(trichlorosilyl)alkanes

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Experimental Section

General comment. All reactions were carried out in flame-dried glassware or stainless steel cylinder. All air-sensitive liquids were transferred by a standard syringe or double tipped-needle techniques. The trichlorosilane, tetrachlorosilane, Bu_4PCl , *n*-dodecane, styrene, 4-methylstyrene, 1-hexene, 1-decene, *trans*- β -methylstyrene, *trans*-stilbene, *trans*-3-hexene, and cyclopentene from Aldrich Chem. Co., Acros, Fluka, and TCI. Reaction mixtures were analyzed by GLC using a packed column (10% SE-30 or SE-54 on 80-100 mesh chromosorb W/AW, $\frac{1}{8}$ in. x 2.5 m) or a capillary column (SE-30, 30 m) with a Varian 3300 gas chromatograph, a thermal conductivity detector, and a Varian 4290 integrator. The progresses of reaction were also monitored by GLC in the time intervals of 1 h. Samples for characterization were purified by preparative GLC using a Donam system series DS 6200 gas

chromatograph with a thermal conductivity detector and a 4 m by $\frac{1}{8}$ in. stainless steel column packed with 15% SE-30 or SE-54. Product yields (on the basis of **1** used) were determined by GLC with use of internal standard methods or isolation. Proton and carbon NMR spectra were recorded on a Bruker Avance 300 spectrometer (300 MHz for ^1H ; 75 MHz for ^{13}C) using CDCl_3 as a solvent. The chemical shifts are given in ppm relative to the residual proton signal of the solvent: CDCl_3 7.25 ppm (^1H) and CDCl_3 77.0 ppm (^{13}C). GC/MS data were obtained with a HP6890/5973 system (70 eV, EI). Melting points (uncorrected) were measured with a Mel-Temp II melting point apparatus using sealed capillary tubes.

Reaction of Styrene (1a) with HSiCl_3 . Into a 25 mL dried stainless steel tube was charged with **1a** (1.00 g, 9.60 mmol), HSiCl_3 (5.20 g, 38.39 mmol), Bu_4PCl (0.28 g, 0.95 mmol), and *n*-dodecane (1.0 mL) under a dry nitrogen atmosphere. After the valve was closed, the reaction was carried out at 180 °C for 4 h and gave 1,2-bis(trichlorosilyl)ethylbenzene (**2a**),¹ a mixture of (2-dichlorosilyl-1-trichlorosilyl)ethylbenzene (**2a'-1**) and (1-dichlorosilyl-2-trichlorosilyl)ethylbenzene (**2a'-2**), and 2-phenylethyltrichlorosilane (**3a**)² in 77, 8, and 9% GLC yields, respectively, along with dihydrogen gas (ca. 198 mL at 20 °C). The reaction mixture was separated from Bu_4PCl by bulb to bulb vacuum distillation and fractionally vacuum-distilled to give 1,2-bis(trichlorosilyl)ethylbenzene (**2a**, 2.76 g) in 77% yield. In the analysis of compounds **2a'-1** and **2a'-2**, they could be not separated under our analytical GLC conditions but were just shown to be a isomeric mixture from analyzing the peak purities of GC/MS spectra and almost converted to **2a** during a vacuum distillation. Thus, their ^1H , ^{13}C , ^{29}Si NMR data could not be obtained due to difficulty in isolation. Mass spectra data of a mixture of

2a'-1 and **2a'-2**: MS (70 eV EI) m/z (%) 336 (9), 338 (14), 340 (10), 342 (3) (M^+), 237 (38), 239 (38), 240 (13) ($M - SiHCl_2$)⁺, 203 (31), 205 (19) ($M - SiCl_3$)⁺, 175 (100), 177 (69), 179 (13), 167 (24), 169 (8), 133 (16), 135 (16), 137 (5), 104 (25), 103 (26), 77 (24), 63 (18). Data for **2a**: bp 110-112 °C/0.3 torr. ¹H NMR (CDCl₃) δ 2.07 (dd, $J = 15.6, 3.0$ Hz, 1H, $CHH'SiCl_3$), 2.18 (dd, $J = 15.6, 12.6$ Hz, 1H, $CHH'SiCl_3$), 3.09 (dd, $J = 12.6, 3.0$ Hz, 1H, $PhCH$), 7.25-7.37 (m, 5H, $Ph-H$). ¹³C NMR (CDCl₃) δ 23.54 (CH_2SiCl_3), 36.72 ($CHSiCl_3$), 127.81, 128.92, 129.10, 143.08 ($Ph-carbons$). ²⁹Si NMR (60 MHz, CDCl₃) δ 7.37, 10.13. MS (70 eV, EI) m/z (relative intensity): 372 (11), 370 (5) (M^+), 241 (35), 239 (98), 237 (100), 201 (17), 165 (11), 135 (34), 133 (34), 104 (20), 103 (26), 77 (17). Anal. Calcd for C₈H₈Cl₆Si₂: C, 25.76; H, 2.16. Found: C; 25.60, H; 2.31.

Reaction of Styrene (1a) with a Mixture of HSiCl₃ and SiCl₄. Using the general procedure above, the reaction of **1a** (1.00 g, 9.60 mmol) with a mixture of HSiCl₃ (5.20 g, 38.39 mmol) and SiCl₄ (6.52 g, 38.38 mmol) in the presence of Bu₄PCl (0.28 g, 0.95 mmol) at 180 °C for 4 h gave **2a** and **3a** in 85 and 9% as GLC yields, respectively, along with dihydrogen gas (ca. 197 mL at 20 °C). Compound **2a** (2.78 g, 78%) was vacuum-distilled out from the reaction mixture

Reaction of 4-Methylstyrene (1b) with a Mixture of HSiCl₃ and SiCl₄. Using the general procedure above, the reaction of **1b** (1.00 g, 8.46 mmol) with a mixture of HSiCl₃ (4.59 g, 33.89 mmol) and SiCl₄ (5.76 g, 33.90 mmol) in the presence of Bu₄PCl (0.25 g, 0.85 mmol) at 180 °C for 4 h gave 1,2-bis(trichlorosilyl)-4-methylbenzene (**2b**) and 2-(4-methylphenyl)ethyltrichlorosilane (**3b**)³ in 83 and 13% GLC yields, respectively. The reaction mixture was fractionally vacuum-distilled to give **2b** (2.55 g) in 78% yield. Data for **2b**: bp 114-115 °C/0.3 torr. ¹H NMR (CDCl₃) δ 2.08 (dd, $J = 15.6, 3.0$

Hz, 1H, $\text{CHH}'\text{SiCl}_3$), 2.19 (dd, $J = 15.6, 12.6$ Hz, 1H, $\text{CHH}'\text{SiCl}_3$), 2.35 (s, 3H, CH_3), 3.08 (dd, $J = 12.6, 3.0$ Hz, 1H, PhCH), 7.16 (s, 4H, Ph-H). ^{13}C NMR (CDCl_3) δ 21.63 (CH_2SiCl_3), 23.97 ($\text{PhCH}(\text{SiCl}_3)$), 36.73 (Me-Ph), 129.41, 130.08, 131.22, 138.02 (Ph-carbons). ^{29}Si NMR (60 MHz, CDCl_3) δ 7.36, 10.21. MS (70 eV EI) m/z (relative intensity) 384 (5, M^+), 253 (99), 251 (100), 135 (21), 133 (21), 117 (47), 115 (22), 91 (18), 77 (5). Anal. Calcd for $\text{C}_9\text{H}_{10}\text{Cl}_6\text{Si}_2$: C, 27.93; H, 2.60. Found: C, 27.89; H, 2.54. Data for **3b**: ^1H NMR (CDCl_3) δ 1.71-1.77 (t, $J = 8.10$ Hz, 2H, CH_2SiCl_3), 2.34, (s, 3H, Me), 2.84-2.90 (t, $J = 8.7$ Hz, 2H, Me-PhCH_2), 7.13 (s, 4H, Ph-H), ^{13}C NMR (CDCl_3) δ 21.47, 26.70, 28.24, 128.20, 129.76, 136.44, 138.76.

Reaction of 1-Hexene (1c) with a Mixture of HSiCl_3 and SiCl_4 . Using the general procedure above, the reaction of **1c** (1.00 g, 10.88 mmol) with a mixture of HSiCl_3 (6.44 g, 47.55 mmol) and SiCl_4 (6.92 g, 40.73 mmol) in the presence of Bu_4PCl (0.32 g, 1.09 mmol) at 180 °C for 16 h gave 1,2-bis(trichlorosilyl)hexane (**2c**) and hexyltrichlorosilane (**3c**)² in 75 and 21% GLC yields, respectively. The reaction mixture was fractionally vacuum-distilled to give **2c** (2.94 g) in 70% yield. Data for **2c**: bp 101-103 °C/0.3 torr, ^1H NMR (CDCl_3) δ 0.92 (t, $J = 7.2$ Hz, 3H, CH_3), 1.28-1.39 (m, 2H, CH_2CH_3), 1.43-1.54 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 1.60 (dd, $J = 15.8, 10.6$ Hz, 2H, CH_2SiCl_3), 1.76-1.89 (m, 3H, $\text{CH}_2\text{CH}(\text{SiCl}_3)\text{CH}_2\text{Si}$). ^{13}C NMR (CDCl_3) δ 13.75 (CH_3), 22.77 (CH_2CH_3), 22.90 ($\text{CH}_2\text{CH}_2\text{CH}_3$), 28.13 (CH_2SiCl_3), 29.80 ($\text{CH}_2\text{CH}(\text{SiCl}_3)\text{CH}_2\text{Si}$), 29.86 (CHSiCl_3). ^{29}Si NMR (60 MHz, CDCl_3) δ 6.16, 7.30.

MS (70 eV EI) m/z (relative intensity) 219 (35, (M - SiCl₃)⁺), 217 (32), 179 (35), 177 (98), 175 (100), 135 (36), 133 (36), 55 (18). Anal. Calcd for C₆H₁₂Cl₆Si₂: C, 20.41; H, 3.43. Found: C, 20.52; H, 3.66.

Reaction of 1-Decene (1d) with a Mixture of HSiCl₃ and SiCl₄. Using the general procedure above, the reaction of **1d** (1.00 g, 7.13 mmol) with a mixture of HSiCl₃ (3.86 g, 28.50 mmol) and SiCl₄ (4.85 g, 28.55 mmol) in the presence of Bu₄PCl (0.21 g, 0.71 mmol) at 180 °C for 16 h gave 1,2-bis(trichlorosilyl)decane (**2d**) and decyltrichlorosilane (**3d**)⁴ in 72 and 12% using GLC yields, respectively. 86% of starting material **1d** was consumed. The reaction mixture was fractionally vacuum-distilled to give **2d** (1.90 g) in 65% yield. Data for **2d**: bp 120-121 °C/0.3 torr, ¹H NMR (CDCl₃) δ 0.88 (t, *J* = 0.63 Hz, 3H, CH₃), 1.27 (br s, 10H, CH₃(CH₂)₅), 1.44-1.51 (m, 2H, CH₂CH₂CH(SiCl₃)), 1.59 (dd, *J* = 15.8, 10.6 Hz, 2H, CH₂SiCl₃), 1.74-1.85 (m, 3H, CH₂CH(SiCl₃)CH₂Si). ¹³C NMR (CDCl₃) δ 14.09 (CH₃), 22.65, 22.94, 27.77, 29.16, 29.64, 30.12 ((CH₂)₇), 28.19 (CH₂SiCl₃), 31.80 (CHSiCl₃). ²⁹Si NMR (60 MHz, CDCl₃) δ 11.26, 12.49. MS (70 eV EI) m/z (relative intensity) 275 (24), 273 (25) ((M-SiCl₃)⁺), 219 (29), 217 (30), 205 (27), 203 (28), 191 (26), 189 (27), 177 (41), 175 (41), 135 (39), 133 (39), 99 (25), 71 (69), 57 (100), 55 (27). Anal. Calcd for C₁₀H₂₀Cl₆Si₂: C, 29.36; H, 4.93. Found: C; 29.46, H; 5.03.

Reaction of *trans*-1-Phenylprop-1-ene (1e) with a Mixture of HSiCl₃ and SiCl₄. Using the general procedure above, the reaction of **1e** (1.00 g, 8.46 mmol) with a mixture of HSiCl₃ (4.59 g, 33.89 mmol) and SiCl₄ (5.75 g, 33.84 mmol) in the presence of Bu₄PCl (0.25 g, 0.85 mmol) at 180 °C for 12 h gave

a 3: 2 mixture of two diastereoisomeric 1,2-bis(trichlorosilyl)propylbenzenes (**2e**), and 1-methyl-2-phenylethyltrichlorosilane (**3e**)⁵ in 83 and 14% GLC yields, respectively. The reaction mixture was fractionally vacuum-distilled to give **2e** (2.46 g) in 75% yield. Data for two diastereoisomers, D1 (D2)-**2e**: bp 112-115 °C/0.3 torr. ¹H NMR (CDCl₃) δ 1.57 (1.63) (d, *J* = 7.5 Hz, 3H, CH₃), 2.34-2.44 (m, 1H) (the overlapped CHCH₃ of D1 and D2), 3.38 (3.05) (d, *J* = 2.70 (11.7) Hz) (1H, PhCH), 7.30-7.35 (m, 5H) (the overlapped Ph-*H* of D1 and D2). ¹³C NMR (CDCl₃) δ 11.58 (15.80) (CH₃), 29.46 (31.81) (CHSiCl₃), 42.65 (44.99) (PhCH), 128.45, 128.49, 129.18, 129.57, 131.60, 132.67, 136.51 (the overlapped Ph-carbons of two diastereomers D1 and D2). ²⁹Si NMR (60 MHz, CDCl₃) δ 7.47 (9.21) 12.59 (13.21). The mass spectra (70 eV EI) for two isomers **2e** are very similar: *m/z* (relative intensity): 386 (11, (M+2)⁺), 255 (33), 253 (90), 251 (90), 225 (35), 223 (35), 215 (14), 135 (30), 133 (29), 117 (54), 115 (40), 91 (100), 63 (19). Anal. Calcd for C₉H₁₀Cl₆Si₂: C, 27.93; H, 2.60. Found: C, 27.98; H, 2.77. Data for **3e**: ¹H NMR (CDCl₃) δ 1.16-1.18 (d, 3H, CH₃), 1.77-1.85 (m, 1H, CHSiCl₃), 2.54 (dd, *J* = 13.8, 3.6 Hz, 1H, PhCHH'), 3.28 (dd, *J* = 13.8, 11.6 Hz, 1H, PhCHH'), 7.25-7.24 (m, 5H, Ph-*H*). ¹³C NMR (CDCl₃) δ 12.03, 29.85, 35.60, 126.48, 128.45, 128.91, 139.07. Anal. Calcd for C₉H₁₁Cl₃Si: C, 42.62; H, 4.37. Found for a mixture of two diastereomers **2e**: C, 42.51; H, 4.46.

Reaction of *trans*-Stilbene (1f) with a Mixture of HSiCl₃ and SiCl₄. Using the general procedure above, the reaction of **1f** (1.00 g, 5.55 mmol) with a mixture of HSiCl₃ (3.01 g, 22.22 mmol) and SiCl₄ (3.78 g, 22.25 mmol) in the presence of Bu₄PCl (0.16 g, 0.54 mmol) at 180 °C for 12 h gave a 3: 1

mixture of two diastereomeric 1,2-bis(trichlorosilyl)-1,2-diphenylethanes (**2f**) and 1,2-diphenylethyltrichlorosilane (**3f**)⁶ in 70 and 21% GLC yields, respectively, with 94% consumption of **1f**. The reaction mixture was vacuum-distilled to give a 3: 1 mixture of two diastereomers, 1*R*(*S*),2*S*(*R*)-**2f** and 1*R*(*S*),2*R*(*S*)-**2f**, (1.52 g, 123-127 °C/0.3 torr) in 61% isolated yield. Compound 1*R*(*S*),2*S*(*R*)-**2f** (mp: 96-97 °C) was isolated as a single crystal suitable for X-ray crystallographic determination by recrystallization from toluene solution at -23 °C. Data for 1*R*(*S*),2*S*(*R*) [1*R*(*S*),2*R*(*S*)]-**2f**: ¹H NMR (CDCl₃) δ 3.65 (3.60) (s, 2H, CHSiCl₃), 7.32-7.47 (7.09-7.29) (m, 10H, Ph-*H*). ¹³C NMR (CDCl₃) δ 45.18 (42.49) (CHSiCl₃), 128.84, 129.60, 130.87, 134.87 (127.99, 128.36, 131.69, 132.75) (Ph-*carbons*). ²⁹Si NMR (60 MHz, CDCl₃) δ 10.13 (5.81). Data of mass spectra for two diastereomers are very similar. MS (70 eV, EI) *m/z* (relative intensity) 454 (2), 452 (6), 450 (12), 448 (14), 446 (7) (*M*⁺), 317 (33), 316 (19), 315 (90), 314 (19), 313 (89), 241 (21), 227 (36), 226 (13), 225 (100), 224 (14), 223 (100), 180 (33), 179 (70), 178 (56), 177 (10), 176 (10), 165 (22), 152 (12), 135 (21), 133 (21), 125 (19), 89 (19), 77 (13), 63 (12). Anal. Calcd for C₁₄H₁₂Cl₆Si₂: C, 37.44; H, 2.69. Found for a mixture of two diastereomers **2f**: C, 37.68; H, 2.90. The ORTEP plot of 1*R*(*S*),2*S*(*R*)-**2f** is depicted in Fig 1.

Reaction of *trans*-3-Hexene (1g**) with a Mixture of HSiCl₃ and SiCl₄.** Using the general procedure above, the reaction of *trans*-**1g** (1.01 g, 12.00 mmol) with a mixture of HSiCl₃ (6.44 g, 47.55 mmol) and SiCl₄ (8.16 g, 48.03 mmol) in the presence of Bu₄PCl (0.35 g, 1.19 mmol) at 180 °C for 32 h gave

a 15: 1 mixture of two diastereomeric 3,4-bis(trichlorosilyl)hexanes (**2g**) and 3-(trichlorosilyl)hexane (**3g**) in 12 and 24% yields, respectively. 41% of starting material **1g** was consumed. Data for a mixture of two diastereomers D1 (D2)-**2g**: ^1H NMR (CDCl_3) δ 1.11-1.52 (t, $J = 7.5$ Hz, 6H, CH_2CH_3), 1.76-1.90 (m, 4H, CH_2CH_3), 2.01-2.06 (m, 2H, CHSiCl_3). ^{13}C NMR (CDCl_3) δ 14.54 (14.02) (CH_3), 20.67 (22.76) (CH_2), 37.72 (CHSiCl_3). ^{29}Si NMR (60 MHz, CDCl_3) δ 12.00 (12.16). The mass spectra (70 eV EI) for two isomers **2g** are very similar: m/z (relative intensity) 219 (31), 217 (32, $(\text{M} - \text{SiCl}_3)^+$), 179 (36), 177 (100), 174 (99), 139 (15), 137 (16), 135 (41), 133 (41), 83 (10), 55(31). Anal. Calcd for $\text{C}_6\text{H}_{12}\text{Cl}_6\text{Si}_2$: C, 20.41; H, 3.43. Found for a mixture of two diastereomers **2g**: C, 20.65; H, 3.75. Data for **3g**: ^1H NMR (CDCl_3) δ 0.91 (t, 3H, $J = 7.2$ Hz, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}(\text{SiCl}_3)$), 1.03 (t, 3H, $J = 7.2$ Hz, $\text{CH}(\text{SiCl}_3)\text{CH}_2\text{CH}_3$), 1.29-1.83 (m, 7H, $\text{CH}_2\text{CH}_2\text{CH}(\text{SiCl}_3)\text{CH}_2$). ^{13}C NMR (CDCl_3) δ 12.67, 14.10, 21.08, 21.34, 29.66, 34.95. MS (70 eV EI) m/z (relative intensity) 220 (3), 218 (3) (M^+), 191 (9), 153 (11), 135 (25), 133 (25), 85 (100), 57 (23), 55 (24). Anal. Calcd for $\text{C}_6\text{H}_{13}\text{Cl}_3\text{Si}$: C, 32.81; H, 5.97. Found: C, 32.58; H, 6.00.

Reaction of Cyclopentene (1h) with a Mixture of HSiCl_3 and SiCl_4 . Using the general procedure above, the reaction of **1h** (1.00 g, 14.68 mmol) with a mixture of HSiCl_3 (7.95 g, 58.69 mmol) and SiCl_4 (9.98 g, 58.74 mmol) in the presence of TBPC (0.43 g, 1.46 mmol) at 180 °C for 32 h gave a 1: 1 mixture of *cis*- and *trans*-1,2-bis(trichlorosilyl)cyclopentanes (**2h**) and trichlorosilylcyclopentane (**3h**)⁷ in 43 and 28% GLC yields, respectively. 85% of starting material **1h** was consumed. Data for *cis*-**2h**:

^1H NMR (CDCl_3) δ 1.81-1.85 (m, 1H), 1.99-2.04 (m, 1H), 2.11-2.14 (m, 6H) (CH_2 and CH). ^{13}C NMR (CDCl_3) δ 24.83, 28.61, 38.04. ^{29}Si NMR (60 MHz, CDCl_3) δ 9.62. Data for *trans*-**2h**: ^1H -NMR (CDCl_3) δ 0.97-0.93-1.03 (m, 1H), 1.80-1.84 (m, 1H), 1.95-2.08 (m, 6H) (CH_2 and CH). ^{13}C NMR (CDCl_3) δ 27.18, 29.02, 32.42. ^{29}Si NMR (60 MHz, CDCl_3) δ 11.04. The mass spectra (70 eV EI) for two isomers **2h** are very similar: m/z (relative intensity) 205 (30), 203 (100), 201 (98) ($\text{M} - \text{SiCl}_3$), 167 (37), 165 (54), 135 (38), 133 (38), 67 (93), 65 (13), 63 (16). Anal. Calcd for $\text{C}_5\text{H}_8\text{Cl}_6\text{Si}_2$: C, 17.82; H, 2.39. Found for a mixture of *cis*- and *trans*-**2h**: C, 17.77; H, 2.44.

X-ray Crystallography. A colorless crystal of compound 1*R*(*S*),2*S*(*R*)-**2f** was mounted on a glass fiber. Preliminary examination and data collection were performed with Mo K α radiation (λ = 0.71069

Å) on a Siemens SMART CCD equipped with a graphite crystal, incident-beam monochromator. Data were collected at 20 °C. Lp and absorption corrections were applied to the data. The structure was solved by direct methods and refined by full-matrix least-squares calculations with SHELX97.⁸ All non-hydrogen atoms were refined anisotropically; all hydrogen atoms were found on a difference Fourier map and refined with isotropic temperature factors 1.2 times those of the attached non-hydrogen atoms. Crystal and intensity data of 1*R*(*S*),2*S*(*R*)-**2f** are follows: Empirical formula C₇H₆Si, fw 224.56, crystal size 0.54 × 0.35 × 0.33 mm³; crystal system: monoclinic, space group: P1bar (No 2); a = 6.4332(15) Å, b = 8.6245(1) Å, c = 9.584(2) Å, α = 79.85(2)°, β = 71.86(2)°, γ = 68.32(2)°; V = 468.47(17) Å³, Z = 2, d_{calcd} = 1.592 Mg/m³, μ = 1.039 mm⁻¹, R [I > 2 σ (I)]: R1 = 0.0667, wR2 = 0.1794. Bond lengths [Å] and angles [deg] for 1*R*(*S*),2*S*(*R*)-**2f** are summarized in Table 1.

Figure 1. A Single Crystal X-ray Diffraction Structure of 1*R*(*S*),2*S*(*R*)-**2f**.

Table 1. Bond lengths [Å] and angles [deg] for 1*R*(*S*),2*S*(*R*)-**2f**

bond length (Å)		bond angles (°)	
Si(1)-C(1)	1.879(4)	C(1)-Si(1)-Cl(3)	112.88(12)
Si(1)-Cl(3)	2.0181(15)	C(1)-Si(1)-Cl(2)	111.56(12)
Si(1)-Cl(2)	2.0249(16)	Cl(3)-Si(1)-Cl(2)	108.74(7)
Si(1)-Cl(1)	2.0258(15)	C(1)-Si(1)-Cl(1)	110.16(12)
C(1)-C(2)	1.520(5)	Cl(3)-Si(1)-Cl(1)	105.82(7)
C(1)-C(1)#1	1.555(7)	Cl(2)-Si(1)-Cl(1)	107.39(7)
C(2)-C(3)	1.377(5)	C(2)-C(1)-C(1)#1	114.1(4)
C(2)-C(7)	1.394(6)	C(2)-C(1)-Si(1)	109.9(2)
C(3)-C(4)	1.384(6)	C(1)#1-C(1)-Si(1)	112.9(3)
C(4)-C(5)	1.380(8)	C(3)-C(2)-C(7)	118.5(4)
C(5)-C(6)	1.361(7)	C(3)-C(2)-C(1)	120.3(3)
C(6)-C(7)	1.376(6)	C(7)-C(2)-C(1)	121.2(3)
		C(2)-C(3)-C(4)	121.0(4)
		C(5)-C(4)-C(3)	119.8(4)
		C(6)-C(5)-C(4)	119.4(4)
		C(5)-C(6)-C(7)	121.4(4)
		C(6)-C(7)-C(2)	119.9(4)

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Figure 1. ¹H and ¹³C NMR spectra of 1,2-bis(trichlorosilyl)ethylbenzene (2a).

Chemical structure: CC1=CC=C(C(C1)C(Cl)Cl)C(Cl)Cl

¹H NMR spectrum (CDCl₃) showing peaks at the following chemical shifts (ppm): 7.163, 7.151, 7.126, 3.82, 3.107, 3.097, 3.065, 3.056, 2.347, 2.237, 2.180, 2.138, 2.109, 2.089, 2.058, and 2.046. Integration values are 3.82, 0.88, 3.00, 1.87, and 3.00.

Chemical structure of 4-(chloromethyl)benzyl chloride: ClCCc1ccc(C)cc1

¹³C NMR spectrum (ppm):

- 138.018
- 137.218
- 130.076
- 129.414
- 38.734
- 23.871
- 21.656

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Figure 3. ¹H and ¹³C NMR spectra of 1,2-bis(trichlorosilyl)hexane (**2c**).

Figure 4. ^1H and ^{13}C NMR spectra of 1,2-bis(trichlorosilyl)decane (**2d**).

Figure 5. ¹H and ¹³C NMR spectra of a mixture of two diastereomeric 1,2-bis(trichlorosilyl)propylbenzenes (**2e**).

Acquisition Time (sec)	2.6542	Comment	Imported from LUXNMR.	Date	27 Jul 2001 08:44:48
File Name	D:\Working\NMR Data\KSH010727-2\010727-2_001000fid	Frequency (MHz)	300.13	Nucleus	¹ H
Number of Transients	12	Original Points Count	32768	Pulse Sequence	zg30
Solvent	CHLOROFORM-D	Sweep Width (Hz)	6172.84	Temperature (degree C)	27.000

Acquisition Time (sec)	0.8700	Comment	Imported from LUXNMR.	Date	27 Jul 2001 09:05:04
File Name	D:\Working\NMR Data\KSH010727-2\010727-2_002000fid	Frequency (MHz)	75.48	Nucleus	¹³ C
Original Points Count	32768	Points Count	32768	Pulse Sequence	zgpg30
Sweep Width (Hz)	18832.39	Temperature (degree C)	27.000	Solvent	CHLOROFORM-D

Figure 6-1. ¹H and ¹³C NMR spectra of 1*R*(*S*),2*S*(*R*)-bis(trichlorosilyl)-1,2-diphenylethane (**2f**).

Acquisition Time (sec)	1.8880	Comment	H1-KSH-050609	Date	Jun 8 2005
File Name	D:\NMR Data\LWC\050609\H1-KSH-050609	Frequency (MHz)	599.44	Nucleus	¹ H
Original Points Count	18147	Points Count	32768	Pulse Sequence	s2pul
Sweep Width (Hz)	9611.92	Temperature (degree C)	20.000	Solvent	CHLOROFORM-D

Acquisition Time (sec)	1.3664	Comment	¹³ C experiment of cyclosporin	Date	28 May 2005 01:23:12
File Name	D:\NMR Data\LWC\2005\05052704\05052704_002000fid	Frequency (MHz)	100.62	Nucleus	¹³ C
Number of Transients	876	Original Points Count	32768	Pulse Sequence	zgpg30
Solvent	CHLOROFORM-D	Sweep Width (Hz)	23980.81	Temperature (degree C)	23.200

Figure 6-2. ¹H and ¹³C NMR spectra of 1R(S),2R(S)-bis(trichlorosilyl)-1,2-diphenylethane (2f).

Figure 7. ¹H and ¹³C NMR spectra of a mixture of two diastereomeric 3,4-bis(trichlorosilyl)hexanes (2g)

Acquisition Time (sec)	2.6542	Comment	Imported from UXNMR.	Date	02 Feb 2002 06:46:24
File Name	D:\Working\NMR Data\KSH02020201\02020201_001000fid	Frequency (MHz)	300.13	Nucleus	¹ H
Number of Transients	8	Original Points Count	32768	Points Count	32768
Solvent	CHLOROFORM-D	Sweep Width (Hz)	6172.84	Pulse Sequence	zgpg30
				Temperature (degree C)	27.000

Acquisition Time (sec)	0.8700	Comment	Imported from UXNMR.	Date	02 Feb 2002 06:49:36
File Name	D:\Working\NMR Data\KSH02020201\02020201_002000fid	Frequency (MHz)	75.48	Nucleus	¹³ C
Nucleus	¹³ C	Number of Transients	100	Original Points Count	32768
Pulse Sequence	zgpg30	Solvent	CHLOROFORM-D	Points Count	32768
Temperature (degree C)	27.000			Sweep Width (Hz)	18832.39

Figure 8-1. ¹H and ¹³C NMR spectra of *cis*-1,2-(bistrichlorosilyl)cyclopentane (**2h**).

Acquisition Time (sec)	5.3084	Comment	Imported from LUXNMR.	Date	02 Feb 2002 06:58:08
File Name	D:\NMR Data\KSH\02020202\02020202_001000f1d	Frequency (MHz)	300.13	Nucleus	¹ H
Original Points Count	32768	Points Count	32768	Pulse Sequence	zgpg30
Sweep Width (Hz)	6172.84	Temperature (degree C)	27.000	Solvent	CHLOROFORM-D

Acquisition Time (sec)	1.7400	Comment	Imported from LUXNMR.	Date	02 Feb 2002 07:21:36
File Name	D:\NMR Data\KSH\02020202\02020202_002000f1d	Frequency (MHz)	75.48	Nucleus	¹³ C
Original Points Count	32768	Points Count	32768	Pulse Sequence	zgpg30
Sweep Width (Hz)	18632.39	Temperature (degree C)	27.000	Solvent	CHLOROFORM-D

Figure 8-2. ¹H and ¹³C NMR spectra of *trans*-1,2-(bistrichlorosilyl)cyclopentane (**2h**).