Unusual Palladium-Catalyzed Silaboration of Allenes Using Organic Iodides as Initiators: Mechanism and Application

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Experimental Section

General Considerations. All reactions were run under a nitrogen atmosphere on a dual-manifold Schlenk line, unless otherwise mentioned, and in oven-dried glassware. All solvents were dried according to known methods and distilled prior to use.¹ $\text{PdCl}_2(\text{CH}_3\text{CN})_2$,² $\text{Pd}(\text{dba})_2$,³ allenes⁴ and borylsilane **5**⁵ were prepared by procedures previously reported. Reagents and chemicals were used as purchased without further purification. The purity of each product was checked by NMR analysis.

- (1) Perin, D. D.; Armarego, W. L. F.; In *Purification of Laboratory Chemicals*, 3rd ed.; Pergamon Press: New York, 1988.
- (2) Colquhoun, H. M.; Halton, J.; Thompson, D. J.; Twigg, T. V. *New Pathway for Organic Synthesis-Practical Applications of Transition Metals*; Plenum Press: New York, 1988, p 383.
- (3) Takahashi, Y.; Ito, T.; Sakai, S.; Ishii, Y. *J. Chem. Soc. Chem. Commun.* **1970**, 1065.
- (4) Brandsma, L.; Verkruijisse, H. D. *Synthesis of Acetylene, Allenes and Cumulenes*; Elsevier: New York, 1981.
- (5) For the synthesis of (dimethylphenylsilyl)pinacolborane, see: Suginome, M.; Matsuda, T.; Ito, Y. *Organometallics* **2000**, *19*, 4647.

Dimethyl(phenyl){(E)-2-phenyl-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}silane (7a): an oil; ^1H NMR δ 0.45 (s, 6 H), 1.16 (s, 12 H), 2.03 (s, 2 H), 6.81 (s, 1 H), 7.17-7.37 (m, 8 H), 7.59-7.61 (m, 2H); ^{13}C NMR δ -2.96, 15.96 (br, $\text{CH}_2\text{-B}$),

24.71, 83.25, 126.41, 127.62, 127.91, 128.81, 12.84, 134.30, 137.97, 138.30, 138.53, 139.29 ; HRMS calcd for $C_{23}H_{31}BO_2Si$ 378.2186, found 378.2195.

Dimethyl{(*E*)-2-(2-naphthyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}phenylsilane (7b): an oil; 1H NMR δ 0.53 (s, 6 H), 1.08 (s, 12 H), 1.88 (s, 2 H), 7.22 (s, 1 H), 7.35-7.53 (m, 7 H), 7.65-7.74 (m, 3 H), 7.79-7.84 (m, 1H), 7.92-7.97 (m, 1H); ^{13}C NMR δ -2.75, 15.98 (br, CH_2-B), 24.64, 83.09, 125.12, 125.13, 125.51, 125.54, 126.21, 126.94, 127.70, 128.19, 128.91, 131.79, 133.54, 134.31, 135.86, 136.78, 138.49, 141.05; HRMS calcd for $C_{27}H_{33}BO_2Si$ 428.2343, found 428.2349.

{(*E*)-2-(4-Methoxyphenyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7c): an oil; 1H NMR δ 0.43 (s, 6H), 1.16 (s, 12H), 2.01 (s, 2H), 3.79 (s, 3H), 6.73 (s, 1H), 6.83 (d, $J = 8.4$ Hz, 2H), 7.27-7.35 (m, 5H), 7.55-7.62 (m, 2H); ^{13}C NMR δ -2.94, 16.01 (br, CH_2-B), 24.70, 55.19, 83.22, 113.29, 127.59, 128.77, 130.11, 131.27, 134.29, 137.20, 137.41, 138.47, 158.11; HRMS calcd for $C_{24}H_{33}BO_3Si$ 408.2292, found 408.2300.

{(*E*)-2-(3-Methoxyphenyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7d): an oil; 1H NMR δ 0.44 (s, 6H), 1.15 (s, 12H), 2.03 (s, 2H), 3.79 (s, 3H), 6.73-6.77 (m, 2H), 6.92-6.96 (m, 2H), 7.20 (t, $J = 8.6$ Hz, 1H), 7.32-7.34 (m, 3H), 7.57-7.59 (m, 2H); ^{13}C NMR δ -2.92, 16.15 (br, CH_2-B), 24.72, 55.20, 83.25, 112.51, 113.97, 121.39, 127.62, 128.83 (2C), 134.29, 137.94, 138.27, 139.51, 139.85, 159.34; HRMS calcd for $C_{24}H_{33}BO_3Si$ 408.2292, found 408.2301.

{(*E*)-2-(2-Methoxyphenyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7e): an oil; 1H NMR δ 0.43 (s, 6H), 1.13 (s, 12H), 1.94

(s, 2H), 3.78 (s, 3H), 6.82-6.88 (m, 2H), 6.92 (s, 1H), 7.17 (td, $J = 8.8, 1.6$ Hz, 1H), 7.29-7.36 (m, 3H), 7.43 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.58-7.64 (m, 2H); ^{13}C NMR δ -2.75, 16.12 (br, $\text{CH}_2\text{-B}$), 24.72, 55.53, 83.16, 110.56, 119.74, 127.46, 127.54, 127.90, 128.69, 129.97, 134.02, 134.38, 138.40, 138.71, 157.11; HRMS calcd for $\text{C}_{24}\text{H}_{33}\text{BO}_3\text{Si}$ 408.2292, found 408.2300.

{(E)-2-(4-Chlorophenyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7f): an oil; ^1H NMR δ 0.44 (s, 6H), 1.16 (s, 12H), 1.99 (s, 2H), 6.72 (s, 1H), 7.24-7.38 (m, 7H), 7.55-7.60 (m, 2H); ^{13}C NMR δ -3.05, 16.09 (br, $\text{CH}_2\text{-B}$), 24.70, 83.35, 127.67, 128.02, 128.93, 130.11, 132.02, 134.26, 136.60, 136.94, 137.98, 140.42; HRMS calcd for $\text{C}_{23}\text{H}_{30}\text{BClO}_2\text{Si}$ 412.1797, found 412.1805.

{(E)-2-(4-Bromophenyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7g): an oil; ^1H NMR δ 0.43 (s, 6H), 1.15 (s, 12H), 1.97 (s, 2H), 6.69 (s, 1H), 7.22 (d, $J = 8.4$ Hz, 2H), 7.31-7.34 (m, 3H), 7.41 (d, $J = 8.4$ Hz, 2H), 7.55-7.57 (m, 2H); ^{13}C NMR δ -3.05, 16.06 (br, $\text{CH}_2\text{-B}$), 24.70, 83.37, 120.23, 127.68, 128.94, 130.46, 130.97, 134.26, 136.63, 137.39, 137.95, 140.60; HRMS calcd for $\text{C}_{23}\text{H}_{30}\text{BBrO}_2\text{Si}$ 456.1291, found 456.1295.

{(E)-2-(3-Bromophenyl)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7h): an oil; ^1H NMR δ 0.43 (s, 6H), 1.18 (s, 12H), 1.97 (s, 2H), 6.70 (s, 1H), 7.15 (t, $J = 6.8$ Hz, 1H), 7.21 (d, $J = 6.8$ Hz, 1H), 7.29-7.39 (m, 4H), 7.55-7.58 (m, 3H); ^{13}C NMR δ -3.08, 16.09 (br, $\text{CH}_2\text{-B}$), 24.79, 83.45, 122.16, 127.40, 127.70, 128.98, 129.31, 129.45, 131.54, 134.26, 136.38, 137.82, 140.59, 141.32; HRMS calcd for $\text{C}_{23}\text{H}_{30}\text{BBrO}_2\text{Si}$ 456.1291, found 456.1295.

1-{4-[(*E*)-2-(1,1-Dimethyl-1-phenylsilyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1-propenyl]phenyl}-1-ethanone (7i): an oil; ^1H NMR δ 0.46 (s, 6H), 1.17 (s, 12H), 2.03 (s, 2H), 2.59 (s, 3H), 6.80 (s, 1H), 7.32-7.39 (m, 3H), 7.45 (d, $J = 8.4$ Hz, 2H), 7.55-7.62 (m, 2H), 7.90 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR δ -3.09, 16.36, (br, $\text{CH}_2\text{-B}$), 24.69, 26.53, 83.40, 127.69, 128.08, 128.87, 128.98, 134.24, 134.90, 136.73, 137.73, 142.85, 143.39, 197.79; HRMS calcd for $\text{C}_{25}\text{H}_{33}\text{BO}_3\text{Si}$ 420.2292, found 420.2292.

Ethyl-4-[(*E*)-2-(1,1-dimethyl-1-phenylsilyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1-propenyl]benzoate (7j): an oil; ^1H NMR δ 0.44 (s, 6H), 1.15 (s, 12H), 1.37 (t, $J = 7.2$ Hz, 3H), 2.00 (s, 2H), 4.35 (q, $J = 7.2$ Hz, 2H), 6.78 (s, 1H), 7.30-7.38 (m, 3H), 7.40 (d, $J = 8.4$ Hz, 2H), 7.53-7.60 (m, 2H), 7.96 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR δ -3.07, 14.32, 16.27 (br, $\text{CH}_2\text{-B}$), 24.71, 60.80, 83.41, 127.70, 128.16, 128.67, 128.98, 129.21, 134.26, 136.90, 137.81, 142.39, 143.08, 166.61; HRMS calcd for $\text{C}_{26}\text{H}_{35}\text{BO}_4\text{Si}$ 450.2398, found 450.2397.

Dimethyl(phenyl){(*E*)-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-hexenyl}silane (7k): an oil; ^1H NMR δ 0.32 (s, 3H), 0.89 (t, $J = 7.2$ Hz, 3H), 1.15 (s, 12H), 1.28-1.40 (m, 4H), 1.73 (s, 2H), 2.05-2.11 (m, 2H), 5.74 (t, $J = 6.8$ Hz, 1H), 7.26-7.33 (m, 3H), 7.48-7.54 (m, 2H); ^{13}C NMR δ -3.01, 14.06, 14.52 (br, $\text{CH}_2\text{-B}$), 22.59, 24.69, 28.78, 31.42, 82.92, 127.47, 128.56, 134.19, 134.26, 139.09, 141.20; HRMS calcd for $\text{C}_{21}\text{H}_{35}\text{BO}_2\text{Si}$ 358.2499, found 358.2492.

{(*E*)-2-Cyclopentyl-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl}(dimethyl)phenylsilane (7l): an oil; ^1H NMR δ 0.31 (s, 6H), 1.15 (s, 12H), 1.45-1.85 (m, 8H), 1.73 (s, 2H), 2.68-2.78 (m, 1H), 5.67 (d, $J = 8.8$ Hz, 1H), 7.26-7.35 (m, 3H), 7.48-7.55

(m, 2H); ^{13}C NMR δ -2.94, 14.25 (br, $\text{CH}_2\text{-B}$), 24.66, 25.48, 33.21, 39.62, 82.87, 127.43, 128.49, 132.20, 134.17, 139.20, 146.39; HRMS calcd for $\text{C}_{22}\text{H}_{35}\text{BO}_2\text{Si}$ 370.2499, found 370.2509.

{(E)-2-Cyclooctyl-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-ethenyl} (dimethyl)phenylsilane (7m): an oil; ^1H NMR δ 0.32 (s, 6H), 1.16 (s, 12H), 1.30-1.75 (m, 14H), 1.73 (s, 2H), 2.57 (m, 1H), 5.70 (d, $J = 9.2$ Hz, 1H), 7.29-7.35 (m, 3H), 7.48-7.55 (m, 2H); ^{13}C NMR δ -2.92, 14.11 (br, $\text{CH}_2\text{-B}$), 24.73, 25.47, 26.38, 27.10, 32.34, 36.96, 82.88, 127.45, 128.50, 129.72, 134.19, 139.33, 148.11; HRMS calcd for $\text{C}_{25}\text{H}_{41}\text{BO}_2\text{Si}$ 412.2969, found 412.2962.

Dimethyl{2-methyl-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-propenyl}phenylsilane (7n): an oil; ^1H NMR δ 0.34 (s, 6H), 1.20 (s, 12H), 1.62 (s, 3H), 1.71 (s, 3H), 1.78 (s, 2H), 7.28-7.30 (m, 3H), 7.56-7.59 (m, 2H); ^{13}C NMR δ -0.48, 17.62 (br, $\text{CH}_2\text{-B}$), 21.57, 24.77, 25.94, 82.85, 125.75, 127.42, 128.23, 133.92, 141.07, 142.73; HRMS calcd for $\text{C}_{19}\text{H}_{31}\text{BO}_2\text{Si}$ 330.2186, found 330.2190.

1-(4-{2-Methyl-1-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-1-propenyl}phenyl)-1-ethanone (8b): an oil; ^1H NMR δ 1.35 (s, 12H), 1.56 (s, 3H), 1.78 (s, 3H), 1.96 (s, 2H), 2.56 (s, 3H), 7.25 (d, $J = 8.8$ Hz, 2H), 7.87 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR δ 20.70, 21.95, 24.50, 26.37, 83.01, 127.38, 127.88, 128.78, 129.81, 134.45, 150.69, 197.71; HRMS calcd for $\text{C}_{19}\text{H}_{31}\text{BO}_2\text{Si}$ 314.2053, found 314.2057.

NOE Experiments. The stereochemistry of borylsilylic compound **7a** was determined using the typical ^1H NMR NOE technique. The ^1H NMR signals for the olefin proton H_a and methylene protons H_b of product **7a** appear at 6.81 and 2.03 ppm, respectively. Irradiation at H_a signal gave an increase of H_c intensity by 1.66 %, and an increase of H_b intensity by 0.18 %. In addition, irradiation at H_b afforded an increase of H_c intensity by 1.86 %, but an increase of H_a intensity by only 0.31 %. These NOE results summarized below strongly suggest that **7a** has *E* stereochemistry. The NOE results of products **7k** and **7l** are also shown below. These data indicate that **7k** and **7l** have the *E* stereochemistry.

7a

Irrad.	Ha	Hb	Hc
Ha		0.18%	1.66%
Hb	0.31%		1.86%

7k

Irrad.	Ha	Hb	Hc
Ha		0%	1.65%
Hb	0%		1.61%

7l

Irrad.	Ha	Hb	Hc
Ha		0%	0.57%
Hb	0%		2.52%

(1S*,2S*)-3-(1,1-Dimethyl-1-phenylsilyl)-1,2-diphenyl-3-buten-1-ol (15a): ^1H NMR (400 MHz, CDCl_3): δ = 0.03 (s, 3H), 0.11 (s, 3H), 1.75 (br, 1H), 3.79 (d, J = 8 Hz, 1H), 5.14 (d, J = 8 Hz, 1H), 5.66 (d, J = 2 Hz, 1H), 6.13 (s, 1H), 7.20~7.42 (m, 15H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.24, -3.10, 56.77, 76.17, 126.94, 127.60, 127.36, 127.63, 128.00, 128.16, 128.28, 128.95, 129.58, 134.05, 137.35, 139.51, 142.64, 150.36; HRMS (FAB) calcd for $\text{C}_{24}\text{H}_{25}\text{Si}$ ($\text{M}^+ - \text{OH}$) 341.1726, found 341.1720.

(1S*,2S*)-3-(1,1-Dimethyl-1-phenylsilyl)-1-(4-methoxyphenyl)-2-phenyl-3-buten-1-ol (15b): ^1H NMR (400 MHz, CDCl_3): δ = 0.01 (s, 3H), 0.10 (s, 3H), 1.71 (d, J = 2.8 Hz, 1H), 3.74 (d, J = 8.4 Hz, 1H), 3.80 (s, 3H), 5.07 (dd, J = 8.4 Hz, J = 2.8 Hz, 1H), 5.63 (d, J = 2 Hz, 1H), 6.06 (d, J = 1.2 Hz, 1H), 6.79 (d, J = 3.2 Hz, 2H), 7.14 (d, J = 3.2 Hz, 2H), 7.20~7.41 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.23, -3.05, 55.21, 56.76, 75.79, 113.38, 126.92, 127.60, 128.01, 128.30, 128.33, 128.92, 129.54, 134.06, 134.82, 137.37, 139.80, 150.41, 158.84; HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{27}\text{OSi}$ ($\text{M}^+ - \text{OH}$) 371.1831, found 371.1825.

4-[(1S*,2S*)-3-(1,1-Dimethyl-1-phenylsilyl)-1-hydroxy-2-phenyl-3-butenyl]phenyl acetate (15c): ^1H NMR (400 MHz, CDCl_3): δ = 0.03 (s, 3H), 0.12 (s, 3H), 1.80 (br, 1H), 2.30 (s, 3H), 3.73 (d, J = 8 Hz, 1H), 5.12 (d, J = 8 Hz, 1H), 5.67 (d, J = 2 Hz, 1H), 6.11 (s, 1H), 6.98 (d, J = 6.4 Hz, 1H), 7.16~7.42 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.31, -3.19, 21.10, 56.75, 75.51, 120.97, 126.96, 127.64, 128.15, 128.27, 128.96, 129.52, 134.01, 137.17, 139.31, 140.15, 149.80, 150.32, 169.27; HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{27}\text{O}_2\text{Si}$ ($\text{M}^+ - \text{OH}$) 399.1781, found 399.1785.

(1S*,2S*)-1-(4-Chlorophenyl)-3-(1,1-dimethyl-1-phenylsilyl)-2-phenyl-3-buten-1-ol (15d): ^1H NMR (400 MHz, CDCl_3): δ = 0.02 (s, 3H), 0.12 (s, 3H), 1.76 (d, J = 2 Hz, 1H), 3.68 (d, J = 8 Hz, 1H), 5.07 (d, J = 8 Hz, 1H), 5.67 (d, J = 2 Hz, 1H), 6.08 (s, 1H), 7.02~7.41 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.39, -3.19, 56.78, 75.39, 127.12, 127.66, 128.05, 128.25, 128.38, 128.48, 129.00, 129.49, 132.91, 133.93, 137.02, 139.05, 141.03, 150.23; HRMS calcd for $\text{C}_{24}\text{H}_{25}\text{ClOSi}$ (M^+) 392.1363, found 392.1360.

(1*S,2*S**)-3-(1,1-Dimethyl-1-phenylsilyl)-1-(4-methylphenyl)-2-phenyl-3-buten-1-ol**

(15e): ^1H NMR (400 MHz, CDCl_3): δ = 0.03 (s, 3H), 0.13 (s, 3H), 1.73 (d, J = 3.2 Hz, 1H), 2.36 (s, 3H), 3.79 (d, J = 8 Hz, 1H), 5.11 (dd, J = 8 Hz, J = 3.2 Hz, 1H), 5.66 (d, J = 1.2 Hz, 1H), 6.10 (s, 1H), 7.10~7.38 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.22, 3.03, 21.10, 56.64, 76.00, 126.88, 127.09, 127.58, 128.04, 128.24, 128.68, 128.89, 129.60, 134.06, 136.90, 137.40, 139.67, 150.42; HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{27}\text{Si}$ ($\text{M}^+ - \text{OH}$) 355.1882, found 355.1878.

1-{4-[(1*S,2*S**)-3-(1,1-Dimethyl-1-phenylsilyl)-1-hydroxy-2-phenyl-3-butenyl]phenyl}**

-1-ethanone (15f): ^1H NMR (400 MHz, CDCl_3): δ = 0.02 (s, 3H), 0.12 (s, 3H), 1.86 (d, J = 2.8 Hz, 1H), 2.58 (s, 3H), 3.74 (d, J = 7.2 Hz, 1H), 5.15 (dd, J = 7.2 Hz, J = 2.4 Hz, 1H), 5.68 (d, J = 1.6 Hz, 1H), 6.11 (s, 1H), 7.12~7.42 (m, 12H), 7.80 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.34, -3.16, 26.59, 56.73, 75.54, 127.18, 127.25, 127.70, 128.07, 128.22, 128.39, 129.06, 133.96, 136.10, 137.05, 138.70, 148.04, 150.27, 197.85; HRMS (FAB) calcd for $\text{C}_{26}\text{H}_{27}\text{OSi}$ ($\text{M}^+ - \text{OH}$) 383.1832, found 383.1830.

4-[(1S*,2S*)-3-(1,1-Dimethyl-1-phenylsilyl)-1-hydroxy-2-phenyl-3-butenyl]benzonitrile (15g): ^1H NMR (400 MHz, CDCl_3): δ = 0.04 (s, 3H), 0.14 (s, 3H), 1.86 (br, 1H), 3.67 (d, J = 7.6 Hz, 1H), 5.11 (d, J = 7.6 Hz, 1H), 5.72 (d, J = 1.6 Hz, 1H), 6.10 (s, 1H), 7.10~7.48 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.18, -3.01, 57.05, 75.57, 111.19, 119.16, 127.62, 128.05, 128.40, 128.55, 128.76, 129.43, 129.77, 131.97, 134.14, 137.05, 138.66, 148.19, 150.37; HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{24}\text{NSi}$ ($\text{M}^+ - \text{OH}$) 366.1688, found 366.1685.

(1S*,2S*)-3-(1,1-Dimethyl-1-phenylsilyl)-2-phenyl-1-(2-thienyl)-3-buten-1-ol (15h): ^1H NMR (400 MHz, CDCl_3): δ = 0.07 (s, 3H), 0.14 (s, 3H), 1.88 (d, J = 3.6 Hz, 1H), 3.80 (d, J = 8.4 Hz, 1H), 5.39 (dd, J = 8.4 Hz, J = 3.2 Hz, 1H), 5.65 (d, J = 1.6 Hz, 1H), 6.07 (s, 1H), 6.85 (dd, J = 2.4 Hz, J = 0.8 Hz, 1H), 6.90 (dd, J = 5.2 Hz, J = 3.2 Hz, 1H), 7.19~7.38 (m, 11H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.11, -2.97, 57.68, 71.99, 124.34, 125.09, 126.24, 127.17, 127.64, 127.75, 128.39, 128.97, 134.07, 137.28, 139.17, 146.55, 150.26; HRMS (FAB) calcd for $\text{C}_{22}\text{H}_{23}\text{SSi}$ ($\text{M}^+ - \text{OH}$) 347.1300, found 347.1325.

(3*S,4*R**)-2-(1,1-Dimethyl-1-phenylsilyl)-3-phenyl-1-nonen-4-ol (15i):** ^1H NMR (400 MHz, CDCl_3): δ = 0.18(s, 3H), 0.26 (s, 3H), 0.87 (t, J = 7.2 Hz, 3H), 1.20~1.57 (m, 9H), 3.38 (d, J = 3.2 Hz, 1H), 3.99 (m, 1H), 5.67 (d, J = 2 Hz, 1H), 5.98 (s, 1H), 7.18~7.42(m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.03, -2.79, 14.04, 22.61, 25.60, 31.74, 34.96, 55.77, 73.06, 126.73, 127.14, 127.69, 128.27, 129.02, 129.58, 134.01, 137.49, 139.93, 151.51; HRMS (FAB) calcd for $\text{C}_{23}\text{H}_{22}\text{OSi}$ (M^+) 352.2222, found 352.2219.

(3*S,4*R**)-2-(1,1-Dimethyl-1-phenylsilyl)-3-phenyl-1-hepten-4-ol (15j):** ^1H NMR (400 MHz, CDCl_3): δ = 0.20(s, 3H), 0.27 (s, 3H), 0.87 (t, J = 7.6 Hz, 3H), 1.21~1.58 (m, 5H), 3.39 (d, J = 7.2 Hz, 1H), 4.02(m, 1H), 5.68 (d, J = 2 Hz, 1H), 5.99(d, J = 2 Hz, 1H), 7.19~7.44(m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.03, -2.81, 13.95, 19.10, 37.16, 55.82, 72.75, 126.70, 127.12, 127.66, 128.24, 129.00, 129.58, 133.98, 137.48, 139.90, 151.46; HRMS (FAB) calcd for $\text{C}_{21}\text{H}_{28}\text{OSi}$ (M^+) 324.1909, found 324.1905.

1-(4-((1S*)-2-(1,1-Dimethyl-1-phenylsilyl)-1-[(S*)-1-hydroxy-1-phenylmethyl]-2-propenyl)phenyl)-1-ethanone (15k): ^1H NMR (400 MHz, CDCl_3): δ = 0.05 (s, 3H), 0.11 (s, 3H), 2.01 (br, 1H), 2.56 (s, 3H), 3.81 (d, J = 8 Hz, 1H), 5.15 (d, J = 8 Hz, 1H), 5.50 (d, J = 1.2 Hz, 1H), 6.13 (s, 1H), 7.05~7.40 (m, 12H), 7.79 (d, J = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = -3.25, 26.44, 56.58, 75.87, 126.89, 127.43, 127.64, 127.99, 128.02, 128.85, 128.99, 129.75, 133.90, 135.44, 136.97, 142.53, 145.71, 149.95, 197.86; HRMS (FAB) calcd for $\text{C}_{26}\text{H}_{27}\text{OSi}$ ($\text{M}^+ - \text{OH}$) 383.1832, found 383.1830.

Ethyl-4-((1S*)-2-(1,1-dimethyl-1-phenylsilyl)-1-[(S*)-1-hydroxy-1-phenylmethyl]-2-propenyl)benzoate (15l): ^1H NMR (400 MHz, CDCl_3): δ = 0.05 (s, 3H), 0.12 (s, 3H), 1.39 (t, J = 6.8 Hz, 3H), 1.85 (br, 1H), 3.82 (d, J = 7.2 Hz, 1H), 4.36 (q, J = 6.8 Hz, 2H), 5.14 (d, J = 7.2 Hz, 1H), 5.71 (d, J = 1.6 Hz, 1H), 6.11 (s, 1H), 7.10~7.37 (m, 12H), 7.79 (d, J = 8 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = -2.91, -2.85, 14.58, 56.95, 61.09, 76.21, 127.22, 127.74, 128.00, 128.35, 129.11, 129.16, 129.54, 129.89, 134.26, 137.33, 142.80, 145.44, 150.34, 166.84; HRMS (FAB) calcd for $\text{C}_{27}\text{H}_{29}\text{O}_2\text{Si}$ ($\text{M}^+ - \text{OH}$) 413.1947, found 413.1940.

(1S*,2S*)-3-(1,1-Dimethyl-1-phenylsilyl)-2-(4-methoxyphenyl)-1-phenyl-3-buten-1-ol (15m): ^1H NMR (400 MHz, CDCl_3): δ = 0.03 (s, 3H), 0.11 (s, 3H), 1.77 (br, 1H), 3.72 (d, J = 8 Hz, 1H), 3.80 (s, 3H), 5.09 (d, J = 7.6 Hz, 1H), 5.64 (d, J = 2 Hz, 1H), 6.10 (s, 1H), 6.79 (d, J = 6.4 Hz, 2H), 7.07 (d, J = 6.4 Hz, 2H), 7.16~7.35 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -2.91, -2.80, 55.43, 56.19, 76.31, 113.94, 127.43, 127.56, 127.89, 127.97, 128.26, 129.18, 130.89, 131.40, 134.31, 137.73, 143.04, 150.90, 158.84; HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{27}\text{OSi}$ ($\text{M}^+ - \text{OH}$) 371.1842, found 371.1835.

(1S*,2S*)-2-Butyl-3-(1,1-dimethyl-1-phenylsilyl)-1-phenyl-3-buten-1-ol (15n): ^1H NMR (400 MHz, CDCl_3): δ = 0.36 (s, 3H), 0.39 (s, 3H), 0.73 (t, J = 7.2 Hz, 3H), 0.85~1.32 (m, 5H), 1.55 (m, 1H), 1.92 (s, 1H), 2.57 (m, 1H), 4.45 (d, J = 4 Hz, 1H), 5.72 (d, J = 2.4 Hz, 1H), 5.87 (d, J = 1.6 Hz, 1H), 7.10~7.53 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -2.66, 14.14, 23.03, 26.23, 29.82, 50.77, 74.91, 126.40, 127.02, 127.79, 128.09, 128.17, 129.42, 134.34, 137.92, 143.19, 152.11; HRMS (FAB) calcd for $\text{C}_{22}\text{H}_{30}\text{OSi}$ (M^+) 338.2066, found 338.2061.

(1S*,2S*)-2-Cyclohexyl-3-(1,1-dimethyl-1-phenylsilyl)-1-phenyl-3-buten-1-ol (15o):

^1H NMR (400 MHz, CDCl_3): δ = 0.10 (s, 3H), 0.17 (s, 3H), 0.98~1.32 (m, 5H), 1.53~1.87 (m, 7H), 4.79 (d, J = 8.8 Hz, 1H), 5.55 (s, 1H), 5.75 (s, 1H), 7.20~7.34 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -2.67, -2.62, 26.64, 26.88, 27.04, 29.83, 31.57, 39.40, 54.37, 75.01, 127.06, 127.35, 127.52, 127.92, 128.76, 129.49, 134.16, 138.16, 144.33, 149.84; HRMS (FAB) calcd for $\text{C}_{24}\text{H}_{32}\text{OSi}$ (M^+) 364.2222, found 364.2227.

(1S*,2S*)-2-Cyclopentyl-3-(1,1-dimethyl-1-phenylsilyl)-1-phenyl-3-buten-1-ol (15p):

^1H NMR (400 MHz, CDCl_3): δ = 0.30 (s, 3H), 0.34 (s, 3H), 0.90, (AB type, 2H), 1.31~1.72 (m, 6H), 1.84 (br, 1H), 2.10 (m, 1H), 2.66 (m, 1H), 4.65 (d, J = 4.8 Hz, 1H), 5.62 (m, 2H), 7.13~7.49 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = -2.64, 2.61, 24.01, 25.34, 31.88, 32.11, 40.63, 55.15, 75.56, 126.65, 126.74, 127.64, 127.73, 129.00, 129.12, 134.16, 138.07, 143.40, 151.57; HRMS (FAB) calcd for $\text{C}_{23}\text{H}_{30}\text{OSi}$ (M^+) 350.2066, found 350.2070.

STANDARD 1H OBSERVE

7.360
7.356
7.350

-2.035
-1.169
-0.457

7a

exp1 stdih
date Dec 3 2001 dfrq DEC. & VT
solvent CDCl3 dn H1 400.460
f1 test exp dpr 43
f1 ACQUISITION exp dpr 0
sfrq 400.461 dm nmh
tn H1 dm c
at 2.664 dm 11100
np 32016 15 PROCESSING 0.10
fb 63952 wffile
bs 3400 proc ft
tpwr 59 fn not used
dpr 6.0 werr
d1 1.000 werr
ntf 50100 werr
ct -68 wff aph
gain not used n
fl FLAG n
in n
dp n
sp -200.3
vp 4401.2
vs 115
sc 0
wc 250
hzmm 3.29
ft 30.17
rfl 2889.3
th 100.000
nm cdc ph

7.7 7.5 7.3 7.1 6.9 ppm

10 9 8 7 6 5 4 3 2 1 0 ppm

0.61 0.36
2.49 0.32

0.61 0.61
1.75 3.85

7.383
7.388
7.373
7.368
7.356

STANDARD 1H OBSERVE

expd sedps

SAMPLE	date	Nov 12 2001	strq	BEC.	480.460
solvent	CDCl3	exp	dn	480.460	
oil	exp	dpr	43		
ACQUISITION	480.461	nmr	nmr		
at	2.664	nmr	nmr		
np	32.016	nmr	nmr		
pw	693.076	nmr	nmr		
bs	3.0	nmr	nmr		
tpwr	59	nmr	nmr		
pw	6.0	nmr	nmr		
dr	50.000	nmr	nmr		
nt	100	nmr	nmr		
ct	0	nmr	nmr		
alloc	not used	nmr	nmr		
gain	1	nmr	nmr		
in	in	nmr	nmr		
in	in	nmr	nmr		
DISPLAY	-200.3	nmr	nmr		
sp	480.4	nmr	nmr		
vs	91	nmr	nmr		
sc	25.0	nmr	nmr		
sc	2.00	nmr	nmr		
hnmr	2.36	nmr	nmr		
ls	64.38	nmr	nmr		
rls	3417.7	nmr	nmr		
rff	2099.3	nmr	nmr		
rfp	2099.3	nmr	nmr		
line	1	nmr	nmr		
na	100.000	nmr	nmr		
cdc	ph	nmr	nmr		
ft	not used	nmr	nmr		
aph	wft	nmr	nmr

STANDARD 1H OBSERVE

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expl  stdlh
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date  Dec 3 2001  DEC. & VT
time  11:51:40  488.460
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solvent  CDCl3  dn
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ACQUISITION  exp  488.461  H1
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sfrq  488.461  da  non
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at  2.664  H1  11100  C
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sv  38816  lb  0.10  not used
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fb  3488  wdf  11100  not used
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bs  4  proc  4  ft
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tpr  59  yn  not used
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ct  64  wnt  64  not used
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in  n
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nae  cdc  ph

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STANDARD 1H OBSERVE

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 solvent CDCl3 dm
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 da 400.461 da
 tn 400.461 da
 at 2.884 dar
 nu 2.884 dar
 pw 600.00 lb
 fb 3400 wfile
 bs 4 proc
 tpr 4 not used
 dt 59 ft
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 tof 500.0 wexp
 nt 100 wds
 ct 172 wnt
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Chemical structure of 8b: CC(C)=C(c1ccc(OC)c1)C2OC(C)(C)O2

¹H NMR spectrum (CDCl₃) of compound 8b. The x-axis represents the chemical shift in ppm, ranging from 0 to 10. The spectrum shows several peaks with corresponding integration values below the baseline.

Chemical Shift (ppm)	Integration
7.260 - 7.850	7.260, 7.850
3.854	2.964
6.219	10.19
1.355 - 1.790	1.961, 1.780, 1.555, 1.179, 1.135

The chemical structure shows a central carbon atom bonded to a hydroxyl group (OH) with a wedge bond, a 4-methoxyphenyl group, a phenyl group, and a dimethyl(phenyl)silyl group. The dimethyl(phenyl)silyl group is represented as SiMe₂Ph.