

Supporting Information

Nickel-Catalyzed Silaboration of Small-Ring Vinylcycloalkanes: Regio- and Stereoselective (*E*)-Allylsilane Formation via C-C Bond Cleavage

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Experimental Procedures

General. All reactions were performed under a nitrogen atmosphere using standard Schlenk techniques. Column chromatography was performed with ODS-endcapped silica gel (Cosmosil 140C₁₈-OPN, Nacalai). Preparative GPC (CHCl₃) was performed with a JAI LC-908 equipped with JAIGEL-1H and -2.5H columns. ¹H and ¹³C NMR spectra (CDCl₃) were recorded on a Varian Gemini 2000 (¹H at 300 MHz and ¹³C at 75.46 MHz) spectrometer. ¹¹B and ²⁹Si NMR spectra (CDCl₃) were recorded on a JEOL JNM-A400 (¹H at 400 MHz, ¹¹B at 128.15 MHz, and ²⁹Si at 79.30 MHz) spectrometer. Boron and silicon chemical shifts were referenced to external standards BF₃·OEt₂ and SiMe₄, respectively. High resolution mass spectra were recorded on JEOL JMS-HX110A (FAB) and JEOL JMS-SX100A spectrometers.

Nickel acetylacetone, Ni(acac)₂, was dried in vacuo at 110 °C prior to use. Tricyclohexylphosphine, diisobutylaluminum hydride was used as received.

Silylborane **1** was prepared according to the method we previously reported.⁵ Vinylcyclopropanes were prepared from commercially available cyclopropyl ketones or cyclopropanecarboxaldehyde via Wittig olefination. Vinylcyclobutanes were prepared from

commercially available cyclobutyl ketones via Wittig olefination.

General Procedure for the Reaction of Silylborane **1 and Vinylcyclopropanes (eq 1, 2).** To a mixture of Ni(acac)₂ (10.3 mg, 0.040 mmol), PCy₃ (0.33 M in toluene, 0.12 mL, 0.040 mmol), and vinylcyclopropane (1.2-2.4 mmol) was added DIBAH (1.0 M in toluene, 0.040 mL, 0.040 mmol) at 0 °C. After stirring at 0 °C for 30 min., **1** (210 mg, 0.80 mmol) was added to the mixture. The mixture was heated at 90 °C for 2-8 h. After cooled to room temperature, the mixture was passed through Florisil short column (ether). After evaporation of the volatile materials, the silaboration products **3** were isolated either by bulb-to-bulb distillation or column chromatography on ODS-endcapped silica gel. For the separation of **3h** from **4**, the crude reaction mixture was subjected to preparative GPC.

(E)-1-(Dimethylphenylsilyl)-2-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-pentene (3a**):** ¹H NMR (CDCl₃) δ 0.08 (s, 6H), 0.79 (t, *J* = 7.9 Hz, 2H), 1.23 (s, 12H), 2.08 (s, 2H), 2.08 (q, *J* = 7.6 Hz, 2H), 5.30 (t, *J* = 7.5 Hz, 1H), 7.08-7.49 (m, 10H); ¹³C NMR (CDCl₃) δ -3.1, 12.1 (br), 23.4, 24.7, 28.4, 82.9, 126.3, 127.6, 127.8, 128.3, 128.6, 128.7, 133.6, 136.1, 139.3, 142.3; ¹¹B NMR (CDCl₃) δ 33.7 (br); ²⁹Si NMR (CDCl₃) δ -4.3. HRMS (FAB) calcd for C₂₅H₃₅BO₂Si 406.2499, found 406.2495. Anal. Calcd for C₂₅H₃₅BO₂Si: C, 73.88; H, 8.68. Found: C, 73.65, H, 8.92.

(E)-1-(Dimethylphenylsilyl)-2-(4-fluorophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-pentene (3b**):** ¹H NMR (CDCl₃) δ 0.08 (s, 6H), 0.77 (t, *J* = 7.8 Hz, 2H), 1.21 (s, 12H), 2.02 (q, *J* = 7.6 Hz, 2H), 2.03 (s, 2H), 5.28 (t, *J* = 7.4 Hz, 1H), 6.86-6.96 (m, 2H), 7.00-7.08 (m, 2H), 7.24-7.32 (m, 3H), 7.35-7.41 (m, 2H); ¹³C NMR (CDCl₃) δ -3.0, 12.0 (br), 23.4, 24.7, 28.6, 82.9, 114.6 (d, ²J_{C-F} = 20.8 Hz), 127.6, 128.6, 128.8, 130.2 (d, ³J_{C-F} = 8.1 Hz), 133.6, 135.2, 138.1 (d, ⁴J_{C-F} = 3.5 Hz), 139.1, 161.5 (d, ¹J_{C-F} = 244.7 Hz). HRMS (FAB) calcd for C₂₅H₃₄BFO₂Si: 424.2405. Found 424.2406.

(E)-1-(Dimethylphenylsilyl)-2-methyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-pentene (3c**):** ¹H NMR (CDCl₃) δ 0.27 (s, 6H), 0.80 (t, *J* = 7.9 Hz, 2H), 1.24 (s, 12H), 1.50 (s,

3H), 1.68 (s, 2H), 2.07 (q, $J = 7.6$ Hz, 2H), 4.98 (tq, $J = 7.2, 1.1$ Hz, 1H), 7.31-7.38 (m, 3H), 7.47-7.55 (m, 2H); ^{13}C NMR (CDCl_3) δ -2.9, 11.8 (br), 18.6, 22.5, 24.8, 28.7, 82.9, 125.7, 127.7, 128.8, 131.1, 133.6, 139.7. HRMS (FAB) calcd for $\text{C}_{20}\text{H}_{33}\text{BO}_2\text{Si}$: 344.2343. Found: 344.2343.

(E)-1-(Dimethylphenylsilyl)-1-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-pentene (3d): ^1H NMR (CDCl_3) δ 0.23 (s, 3H), 0.25 (s, 3H), 0.85 (t, $J = 7.8$ Hz, 2H), 1.20 (s, 12H), 2.14 (q, $J = 7.3$ Hz, 2H), 3.06 (d, $J = 9.8$ Hz, 1H), 5.39 (ddt, $J = 15.0, 0.6, 6.5$ Hz, 1H), 5.73 (ddt, $J = 15.0, 9.7, 1.4$ Hz, 1H), 6.86-6.96 (m, 2H), 7.00-7.23 (m, 3H), 7.26-7.42 (m, 5H); ^{13}C NMR (CDCl_3) δ -4.8, -4.4, 11.4 (br) 24.7, 27.0, 42.3, 82.9, 124.5, 127.4, 127.5, 127.7, 127.8, 128.0, 129.0, 134.4, 137.2, 142.5; ^{29}Si NMR (CDCl_3) δ -3.2. HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{35}\text{BO}_2\text{Si}$: 406.2499. Found 406.2480. Anal. Calcd for $\text{C}_{25}\text{H}_{35}\text{BO}_2\text{Si}$: C, 73.88; H, 8.68. Found: C, 73.70, H, 8.78.

(E)-5-(Dimethylphenylsilyl)-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3-nonene (3e): Preparation of 3e was also carried out on a 4.0 mmol scale. Experimental procedure is as follows. To a mixture of $\text{Ni}(\text{acac})_2$ (51 mg, 0.20 mmol), PCy_3 (0.33 M in toluene, 0.60 mL, 0.20 mmol), and 2e (0.75 g, 6.0 mmol) was added DIBAH (1.0 M in toluene, 0.20 mL, 0.20 mmol) at 0 °C. After stirring at 0 °C for 30 min., 1 (1.04 mg, 4.0 mmol) was added to the mixture. The mixture was heated at 90 °C for 8 h. After cooled to room temperature, the mixture was passed through Florisil short column (ether). After evaporation of the volatile materials, 3e (1.33 g, 87%) was isolated by bulb-to-bulb distillation (130-150 °C/0.15 mmHg). ^1H NMR (CDCl_3) δ 0.22 (s, 3H), 0.23 (s, 3H), 0.75-0.95 (m, 5H), 1.00-1.50 (m, 6H), 1.24 (s, 12H), 1.53-1.66 (m, 1H), 2.10 (q, $J = 7.3$ Hz, 2H), 5.15 (dd, $J = 15.1, 9.0$ Hz, 1H), 5.27 (dt, $J = 15.1, 6.2$ Hz, 1H), 7.28-7.39 (m, 3H), 7.41-7.52 (m, 2H); ^{13}C NMR (CDCl_3) δ -5.2, -4.3, 11.6 (br), 13.9, 22.3, 24.7, 27.1, 28.5, 31.4, 32.1, 82.9, 127.5, 128.7, 129.8, 130.8, 134.1, 138.5. HRMS (FAB) calcd for $\text{C}_{23}\text{H}_{39}\text{BO}_2\text{Si}$: 386.2812. Found: 386.2823. Anal. Calcd for $\text{C}_{23}\text{H}_{39}\text{BO}_2\text{Si}$: C, 71.48; H, 10.17. Found: C, 71.32, H, 10.43.

(E)-1-(Dimethylphenylsilyl)-2-methyl-1-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-

yl)-2-pentene (3f): ^1H NMR (CDCl_3) δ 0.27 (s, 3H), 0.32 (s, 3H), 0.83 (t, $J = 7.8$ Hz, 2H), 1.23 (s, 12H), 1.54 (s, 3H), 2.12 (q, $J = 7.4$ Hz, 2H), 2.98 (s, 1H), 5.34 (t, $J = 6.9$ Hz, 1H), 6.98-7.42 (m, 10H); ^{13}C NMR (CDCl_3) δ -3.1, -2.9, 11.6 (br), 18.3, 22.7, 24.8, 47.8, 82.9, 124.9, 127.5, 127.8, 128.8, 129.0, 129.5, 134.3, 138.7, 142.0. HRMS (FAB) calcd for $\text{C}_{26}\text{H}_{37}\text{BO}_2\text{Si}$: 420.2656. Found: 420.2653.

(E)-1-(Dimethylphenylsilyl)-1,2-diphenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-pentene (3g): ^1H NMR (CDCl_3) δ 0.29 (s, 3H), 0.36 (s, 3H), 0.82 (t, $J = 7.8$ Hz, 2H), 1.23 (s, 12H), 2.06 (q, $J = 7.4$ Hz, 2H), 3.26 (s, 1H), 5.73 (t, $J = 7.2$ Hz, 1H), 6.87-6.95 (m, 1H), 7.03-7.24 (m, 5H), 7.25-7.41 (m, 4H); ^{13}C NMR (CDCl_3) δ -3.1, 12.0 (br), 23.9, 24.8, 47.3, 82.9, 124.8, 126.1, 127.4, 127.8, 127.89, 127.93, 128.5, 128.9, 132.8, 134.4, 138.2, 139.6, 141.6, 144.0. HRMS (FAB) calcd for $\text{C}_{31}\text{H}_{39}\text{BO}_2\text{Si}$: 482.2812. Found: 482.2801.

(E)-1-(Dimethylphenylsilyl)-2-cyclopropyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-pentene (3h): ^1H NMR (CDCl_3) δ 0.27-0.38 (m, 2H), 0.29 (s, 6H), 0.46-0.57 (m, 2H), 0.84 (t, $J = 7.9$ Hz, 2H), 1.25 (s, 12H), 1.38 (s, 2H), 1.40-1.51 (m, 1H), 2.24 (q, $J = 7.7$ Hz, 2H), 5.08 (t, $J = 7.1$ Hz, 1H), 7.27-7.38 (m, 3H), 7.45-7.55 (m, 2H); ^{13}C NMR (CDCl_3) δ -2.8, 4.8, 11.8 (br), 12.5, 21.5, 22.1, 24.8, 82.9, 127.6, 127.7, 128.8, 133.7, 134.8, 139.8. HRMS (FAB) calcd for $\text{C}_{22}\text{H}_{35}\text{BO}_2\text{Si}$: 370.2499. Found: 370.2501.

(E)-5-(Dimethylphenylsilyl)-4-[(dimethylphenylsilyl)methyl]-1,7-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3-heptene (4): ^1H NMR (CDCl_3) δ 0.22 (s, 3H), 0.25 (s, 3H), 0.26 (s, 3H), 0.29 (s, 3H), 0.44-0.55 (m, 1H), 0.75-0.92 (m, 3H), 1.13 (d, $J = 13.8$ Hz, 1H), 1.22 (s, 12H), 1.26 (s, 12H), 1.39 (t, $J = 15.2$ Hz, 1H), 1.58 (q, $J = 8.1$ Hz, 2H), 1.89 (d, $J = 13.8$ Hz, 1H), 1.91-2.00 (m, 1H), 2.04-2.15 (m, 1H), 4.85 (t, $J = 6.9$ Hz, 1H), 7.28-7.35 (m, 6H), 7.43-7.47 (m, 4H); ^{13}C NMR (CDCl_3) δ -4.5, -3.3, -2.2, -1.9, 11.2 (br), 11.9 (br), 23.0, 23.8, 24.2, 24.7, 24.8, 82.6, 82.8, 122.4, 127.5, 127.7, 128.6, 128.7, 133.6, 134.1, 135.6, 139.6, 140.2; HRMS (FAB) calcd for $\text{C}_{36}\text{H}_{58}\text{B}_2\text{O}_4\text{Si}_2$: 632.4060. Found: 632.4037. All ^1H NMR signals were assigned by a COSY experiment.

Examination on the Dependence of Product Distribution on the Nickel/Phosphine Ratio in the Silaboration of VCP 2a. Reactions of silylborane **1** with vinylcyclopropane **2a** were carried out in the presence of catalysts prepared with $\text{Ni}(\text{acac})_2/\text{PCy}_3$ ratios of 1:1, 1:2, and 1:4.

(a) Ni/P ratio of 1:1: To a mixture of $\text{Ni}(\text{acac})_2$ (12.8 mg, 0.050 mmol), PCy_3 (0.33 M in toluene, 0.15 mL, 0.050 mmol), **2a** (56 mg, 0.39 mmol), and toluene (0.15 mL) was added DIBAH (1.0 M in toluene, 0.050 mL, 0.050 mmol) at 0 °C. After stirring at 0 °C for 30 min., **1** (264 mg, 1.0 mmol) and **2a** (159 mg, 1.10 mmol) were added to the mixture. The reaction vessel was then immersed in a heating bath (90 °C). Portions of the reaction mixture (ca. 0.050 mL) were taken by a syringe 10, 20, 40, and 80 min. after the reaction was started. Each sample was subjected to

^1H NMR measurement. The product distribution was determined as follows: [time, **3a**/mol%, **5a**/mol%, unchanged **2a**/mol%]; [5 min, 16%, 12%, 72%], [10 min, 26%, 19%, 55%], [20 min, 32%, 25%, 43%], [40 min, 35%, 31%, 34%], [80 min, 37%, 34%, 29%].

(b) Ni/P ratio of 1:2: To a mixture of $\text{Ni}(\text{acac})_2$ (12.8 mg, 0.050 mmol), PCy_3 (0.33 M in toluene, 0.30 mL, 0.10 mmol), and **2a** (56 mg, 0.39 mmol) was added DIBAH (1.0 M in toluene, 0.050 mL, 0.050 mmol) at 0 °C. After stirring at 0 °C for 30 min., **1** (264 mg, 1.0 mmol) and **2a** (161 mg, 1.12 mmol) were added to the mixture. The reaction vessel was then immersed in a heating bath (90 °C). Portions of the reaction mixture (ca. 0.050 mL) were taken by a syringe 10, 20, 40, and 80 min. after the reaction was started. Each sample was subjected to ^1H NMR measurement. The product distribution was determined as follows: [time, **3a**/mol%, **5a**/mol%, unchanged **2a**/mol%]; [5 min, 1%, 6%, 93%], [10 min, 3%, 13%, 84%], [20 min, 12%, 33%, 55%], [40 min, 21%, 62%, 17%], [80 min, 30%, 69%, 1%].

(c) Ni/P ratio of 1:4: To a mixture of $\text{Ni}(\text{acac})_2$ (12.8 mg, 0.050 mmol), PCy_3 (56 mg, 0.20 mmol) **2a** (57 mg, 0.40 mmol), and toluene (0.30 mL) was added DIBAH (1.0 M in toluene, 0.050 mL, 0.050 mmol) at 0 °C. After stirring at 0 °C for 30 min., **1** (264 mg, 1.0 mmol) and **2a** (162 mg, 1.12 mmol) were added to the mixture. The reaction vessel was then immersed in a heating bath (90 °C). Portions of the reaction mixture (ca. 0.050 mL) were taken by a syringe 10,

20, 40, and 80 min. after the reaction was started. Each sample was subjected to ^1H NMR measurement. The product distribution was determined as follows: [time, **3a**/mol%, **5a**/mol%, unchanged **2a**/mol%]; [5 min, –, 4%, 96%], [10 min, –, 16%, 84%], [20 min, 5%, 43%, 52%], [40 min, 9%, 71%, 20%], [80 min, 10%, 87%, 3%].

Isomerization of **2a to 1-Phenylcyclopentene (eq 3).** To a mixture of $\text{Ni}(\text{acac})_2$ (7.7 mg, 0.030 mmol), PCy_3 (0.33 M in toluene, 0.090 mL, 0.030 mmol), and **2a** (87 mg, 0.60 mmol) was added DIBAH (1.0 M in toluene, 0.030 mL, 0.030 mmol) at 0 °C. The mixture was stirred at 0 °C for 30 min. and then heated at 90 °C for 8 h. After cooled to room temperature, the mixture was passed through Florisil short column (ether). ^1H NMR spectrum of the crude mixture showed >95% formation of 1-phenylcyclopentene.

General Procedure for the Reaction of Silylborane **1 and Vinylcyclobutanes (eq 4).** To a mixture of $\text{Ni}(\text{acac})_2$ (20.6 mg, 0.080 mmol) and **6** (1.2 mmol) was added DIBAH (1.0 M in toluene, 0.080 mL, 0.080 mmol) at 0 °C. After stirring at 0 °C for 30 min., **1** (107 mg, 0.41 mmol) was added to the mixture. The mixture was heated at 110 °C for 12 h. After cooled to room temperature, the mixture was passed through Florisil short column (ether). After evaporation of the volatile materials, the silaboration products **3** were isolated by column chromatography on ODS-endcapped silica gel (hexane only–hexane/ether 50/1).

(E)-1-(Dimethylphenylsilyl)-2-phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-hexene (7a): ^1H NMR (CDCl_3) δ 0.07 (s, 6H), 0.70 (t, J = 7.9 Hz, 2H), 1.19 (s, 12H), 1.41 (quint, J = 7.7 Hz, 2H), 1.94 (q, J = 7.4 Hz, 2H), 2.08 (s, 2H), 5.26 (t, J = 7.5 Hz, 1H), 7.05–7.15 (m, 2H), 7.15–7.35 (m, 6H), 7.36–7.45 (m, 2H); ^{13}C NMR (CDCl_3) δ –3.0, 10.7 (br), 24.7, 28.6, 31.6, 82.8, 126.2, 126.3, 127.6, 127.8, 128.69, 128.75, 133.6, 137.2, 139.4, 142.4; ^{11}B NMR (CDCl_3) δ 33.9 (br); ^{29}Si NMR (CDCl_3) δ –4.3. HRMS (FAB) calcd for $\text{C}_{26}\text{H}_{37}\text{BO}_2\text{Si}$: 420.2656. Found: 420.2656.

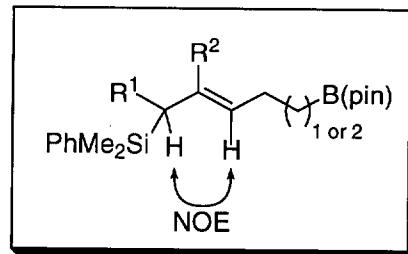
(E)-1-(Dimethylphenylsilyl)-2-methyl-1-phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-hexene (7b): ^1H NMR (CDCl_3) δ 0.26 (s, 3H), 0.31 (s, 3H), 0.75 (t, J = 7.8 Hz, 2H), 1.25

(s, 12H), 1.42 (quint, $J = 7.8$ Hz, 2H), 1.52 (s, 3H), 1.99 (q, $J = 7.4$ Hz, 2H), 3.00 (s, 1H), 5.29 (t, $J = 7.2$ Hz, 1H), 6.99-7.42 (m, 10H); ^{13}C NMR (CDCl_3) δ -3.1, -2.8, 11.0 (br), 18.3, 24.2, 24.8, 31.0, 48.0, 82.9, 124.9, 127.3, 127.5, 127.9, 128.8, 129.0, 134.3, 135.2, 138.7, 142.0. HRMS (FAB) calcd for $\text{C}_{27}\text{H}_{39}\text{BO}_2\text{Si}$: 434.2812. Found: 434.2812.

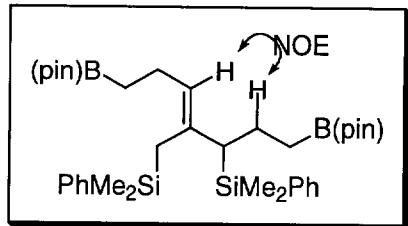
(E)-1-(Dimethylphenylsilyl)-1,2-diphenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-hexene (7c): ^1H NMR (CDCl_3) δ 0.32 (s, 3H), 0.36 (s, 3H), 0.73 (t, $J = 7.8$ Hz, 2H), 1.22 (s, 12H), 1.46 (quint, $J = 7.6$ Hz, 2H), 1.96 (q, $J = 7.5$ Hz, 2H), 3.30 (s, 1H), 5.66 (t, $J = 7.2$ Hz, 1H), 6.86-6.95 (m, 2H), 7.03-7.43 (m, 13H); ^{13}C NMR (CDCl_3) δ -3.2, -3.0, 10.8 (br), 24.4, 24.7, 32.2, 47.4, 82.8, 124.8, 126.0, 127.4, 127.8, 127.90, 127.93, 128.6, 128.9, 130.6, 134.3, 138.2, 140.6, 141.5, 144.0. HRMS (FAB) calcd for $\text{C}_{32}\text{H}_{41}\text{BO}_2\text{Si}$: 496.2969. Found: 496.2959.

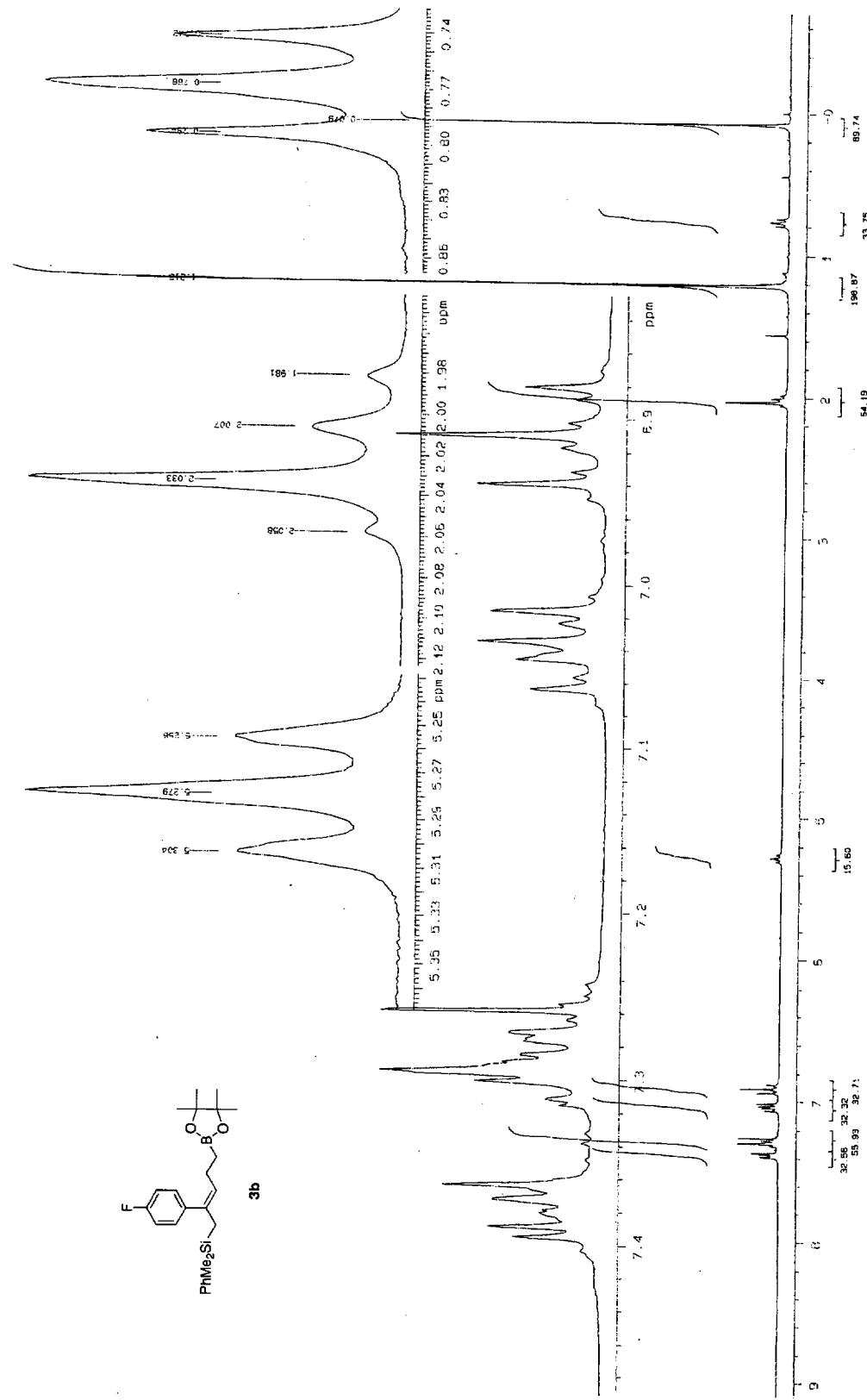
Isomerization of 7a to (α -Methylbenzylidene)cyclobutane (eq 5). To a mixture of $\text{Ni}(\text{acac})_2$ (51 mg, 0.20 mmol) and **7a** (158 mg, 1.0 mmol) was added DIBAH (1.0 M in toluene, 0.20 mL, 0.20 mmol) at 0 °C. The mixture was stirred at 0 °C for 30 min. and then heated at 110 °C for 8 h. After cooled to room temperature, the mixture was passed through Florisil short column (ether). ^1H NMR spectrum of the crude mixture showed >95% formation of (α -methylbenzylidene)cyclobutane. (α -methylbenzylidene)cyclobutane: ^1H NMR (CDCl_3) δ 1.91 (quint, $J = 1.7$ Hz, 3H), 2.01 (quint, $J = 7.9$ Hz, 2H), 2.80-2.97 (m, 4H), 7.15-7.36 (m, 5H); ^{13}C NMR (CDCl_3) δ 16.3, 16.8, 31.0, 32.3, 125.3, 125.8, 126.5, 128.0, 138.4, 141.1; HRMS (EI) Calcd for $\text{C}_{12}\text{H}_{14}$: 158.1096. Found: 158.1097.

Stereochemical Assignment of the Silaboration Products 3 and 7. The *E*-geometry of the silaboration products was determined for **3a-f** and **7a,b** by NOE experiments. Irradiation to the resonance of the vinyl proton γ to the silyl group resulted in NOE at the allylic proton α to the silyl group, and vice versa.

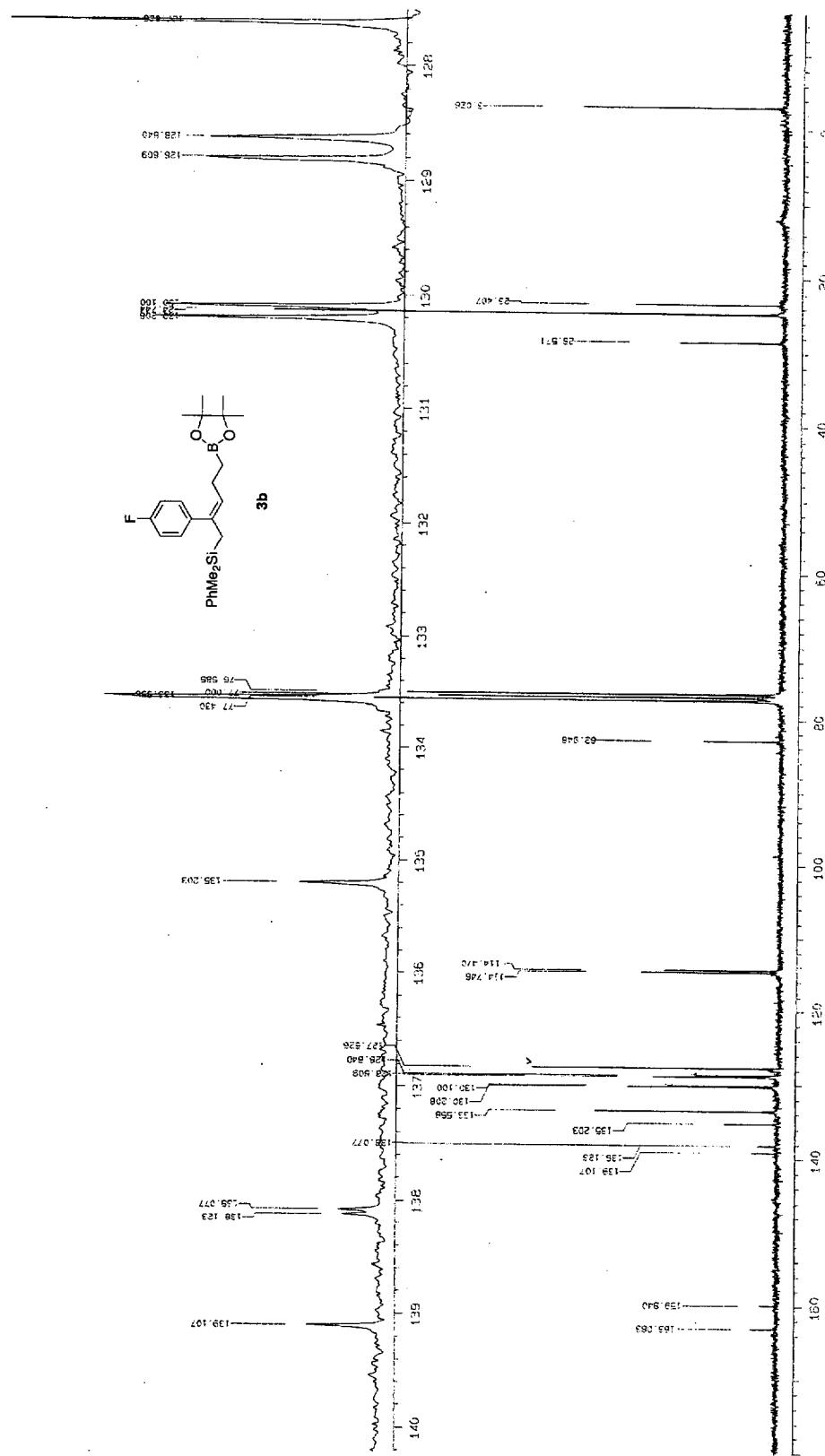


Stereochemical Assignment of the Silaboration Product 4. The *E*-geometry of the silaboration product **4** was determined by a NOESY experiment.

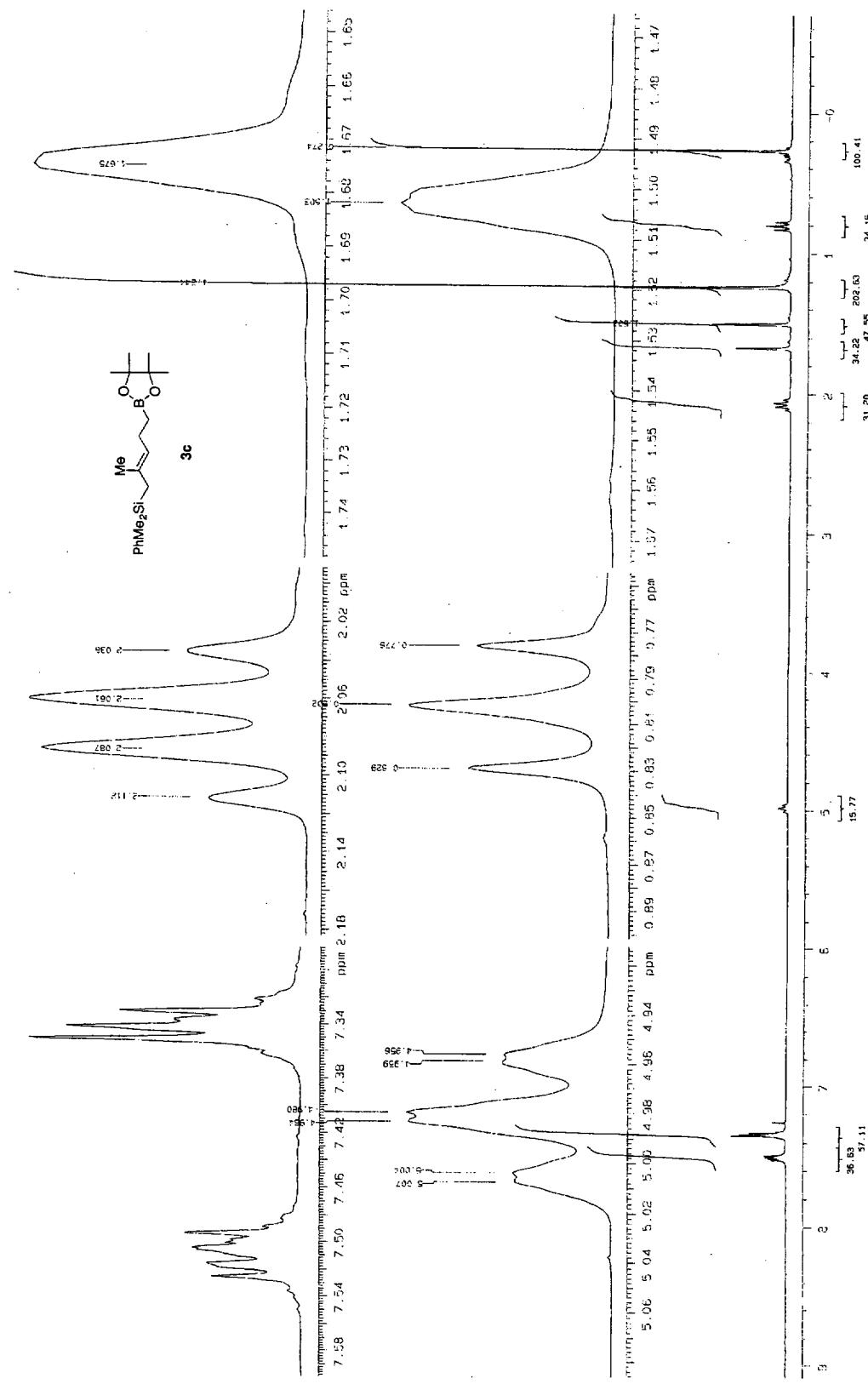




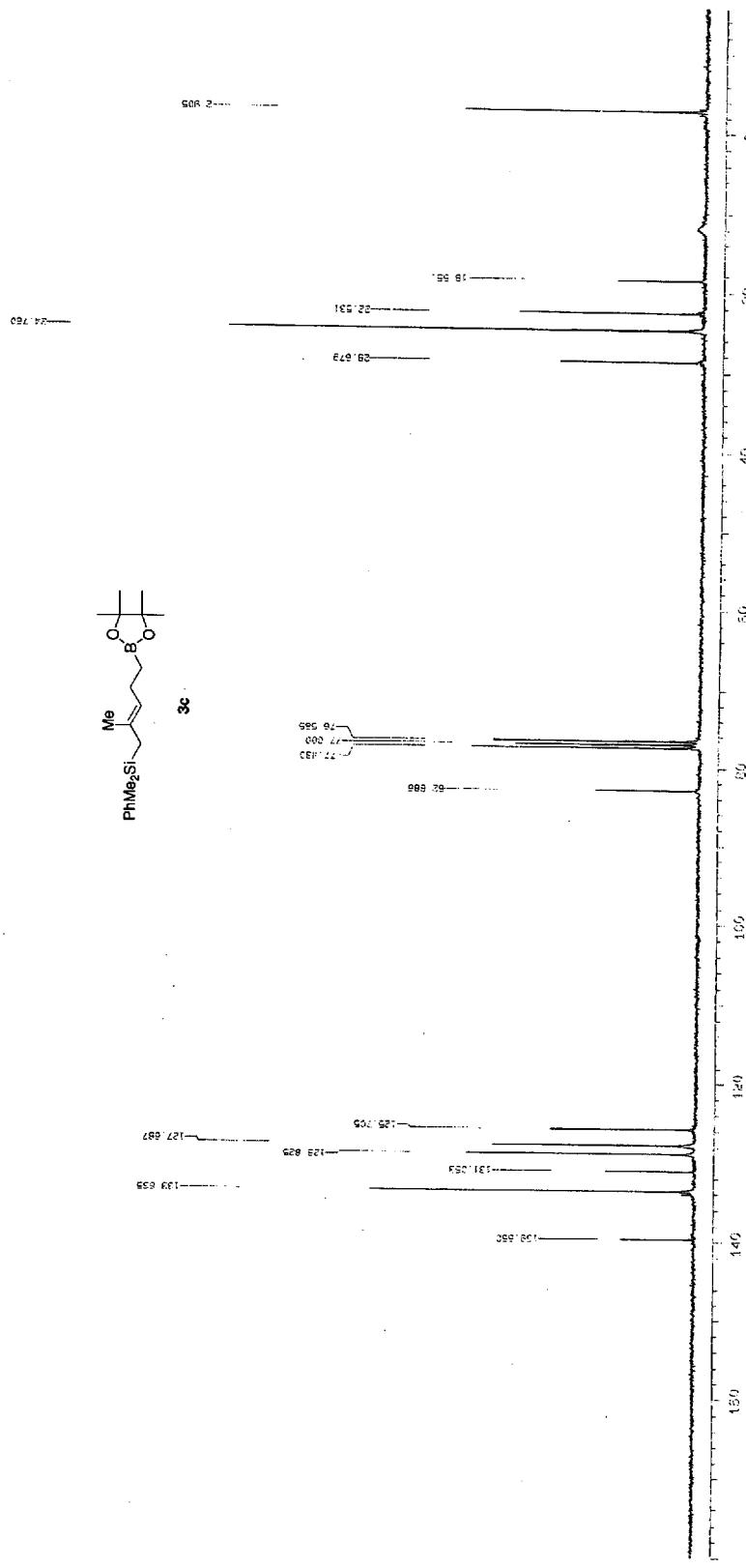
¹H NMR of **3b**.



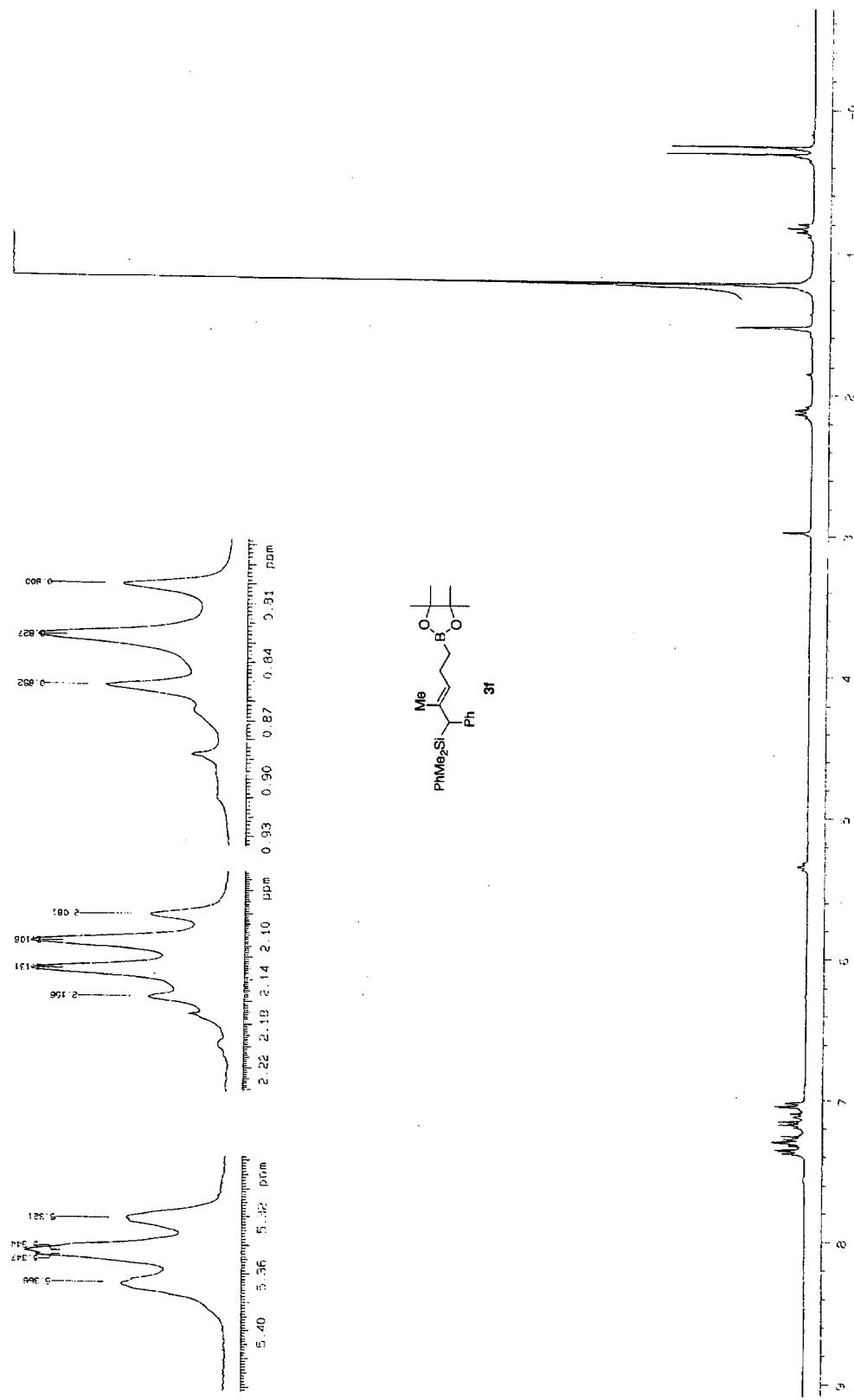
¹³C NMR of **3b**



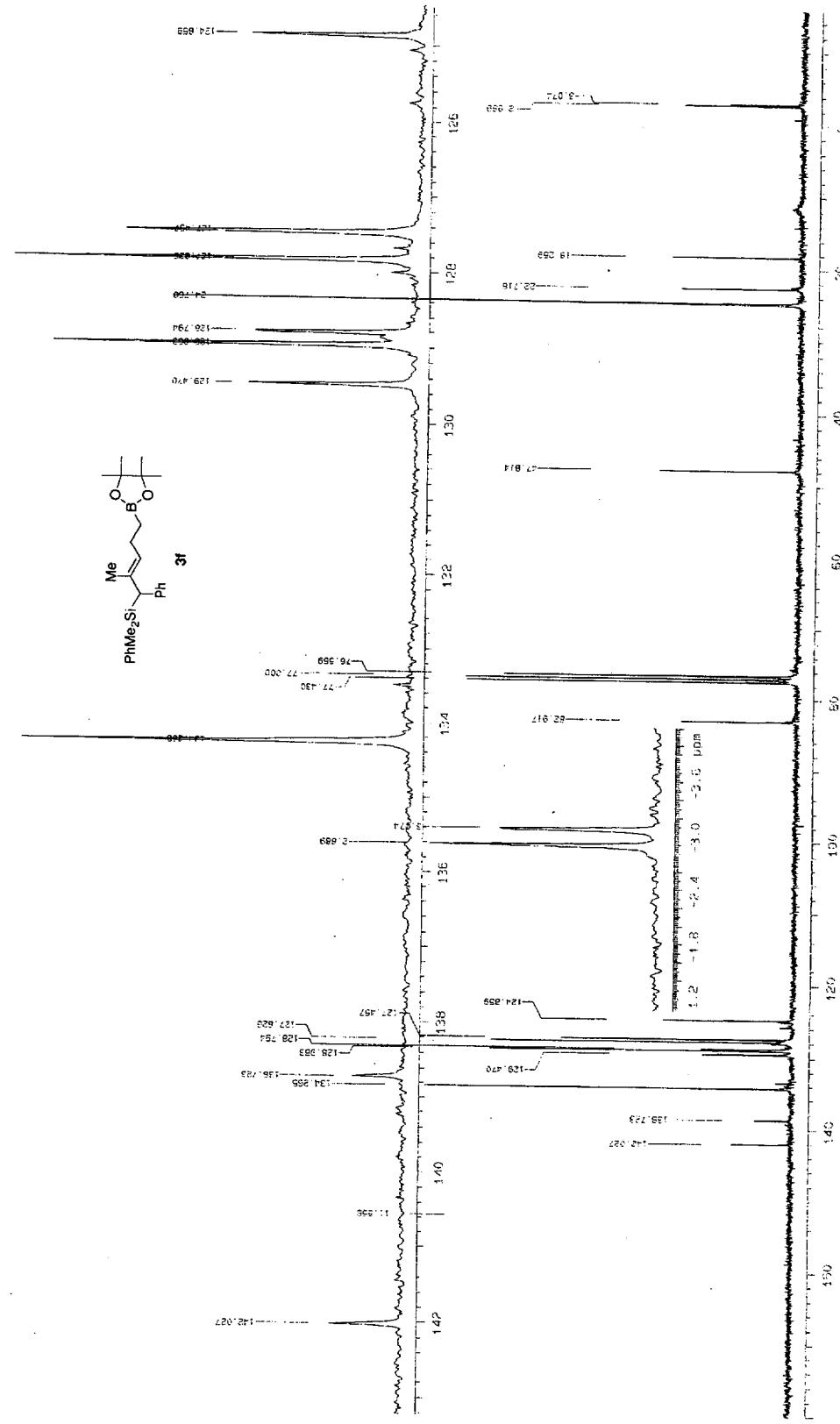
¹H NMR of 3c.



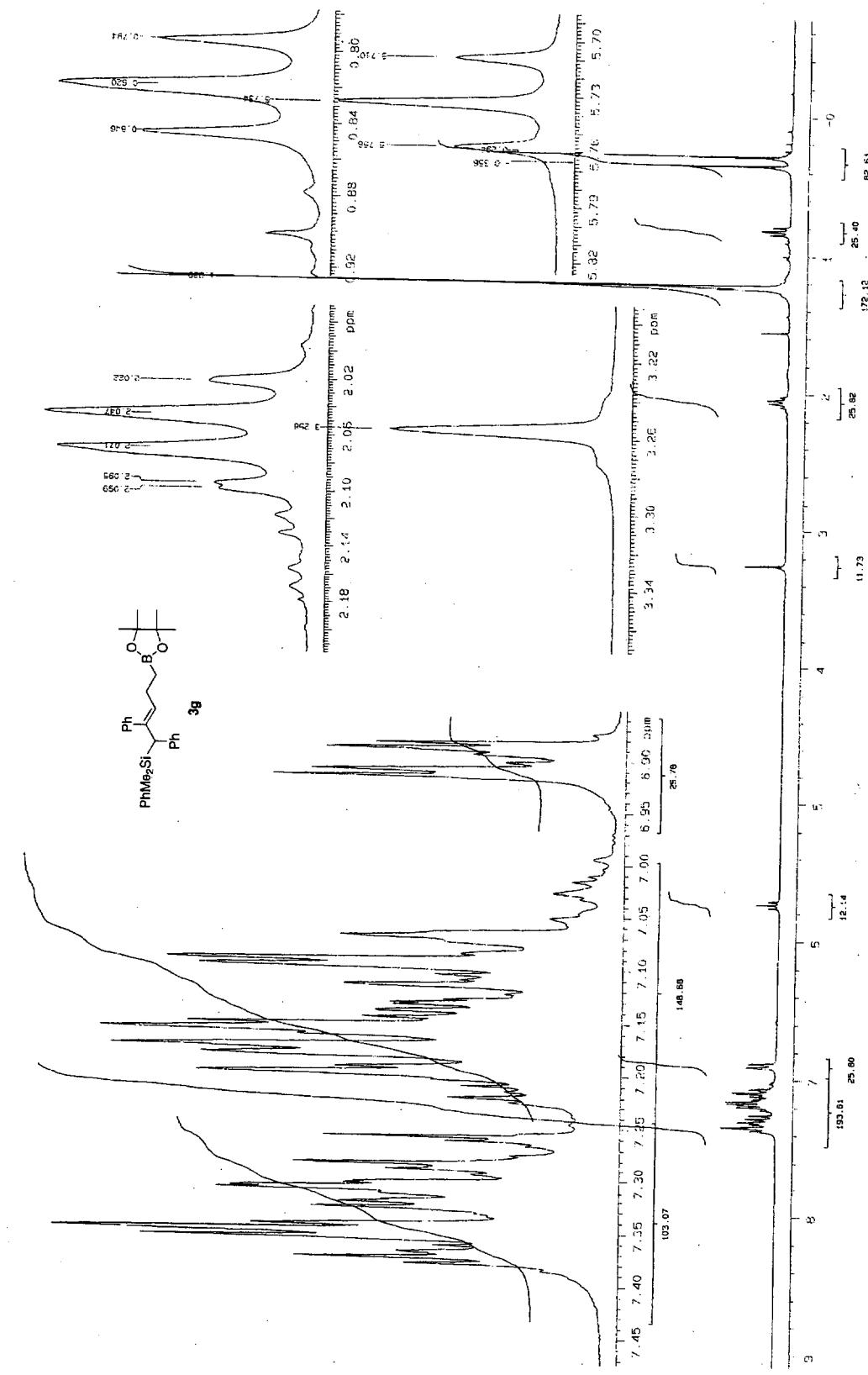
¹³C NMR of 3c.



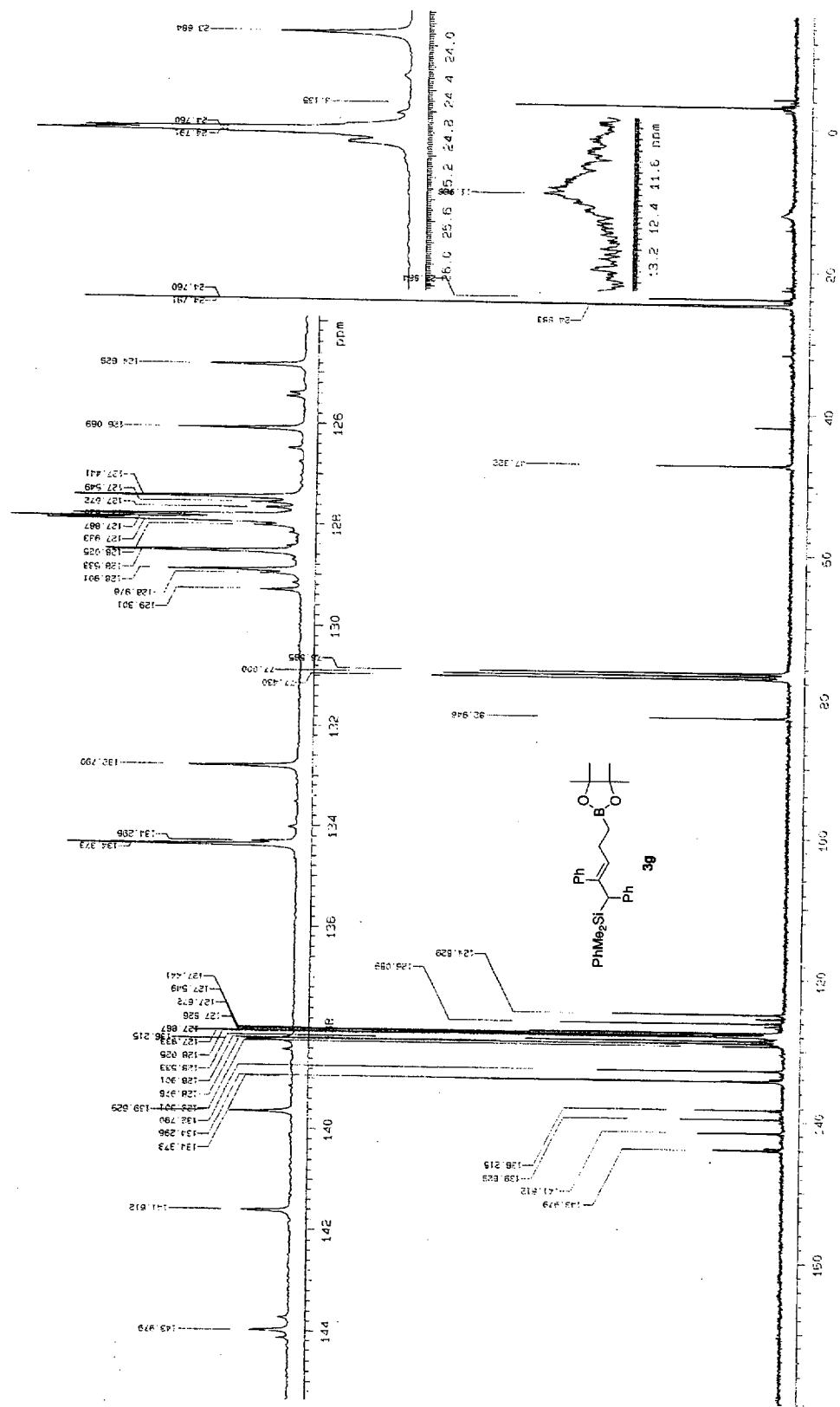
¹H NMR of **3f**.



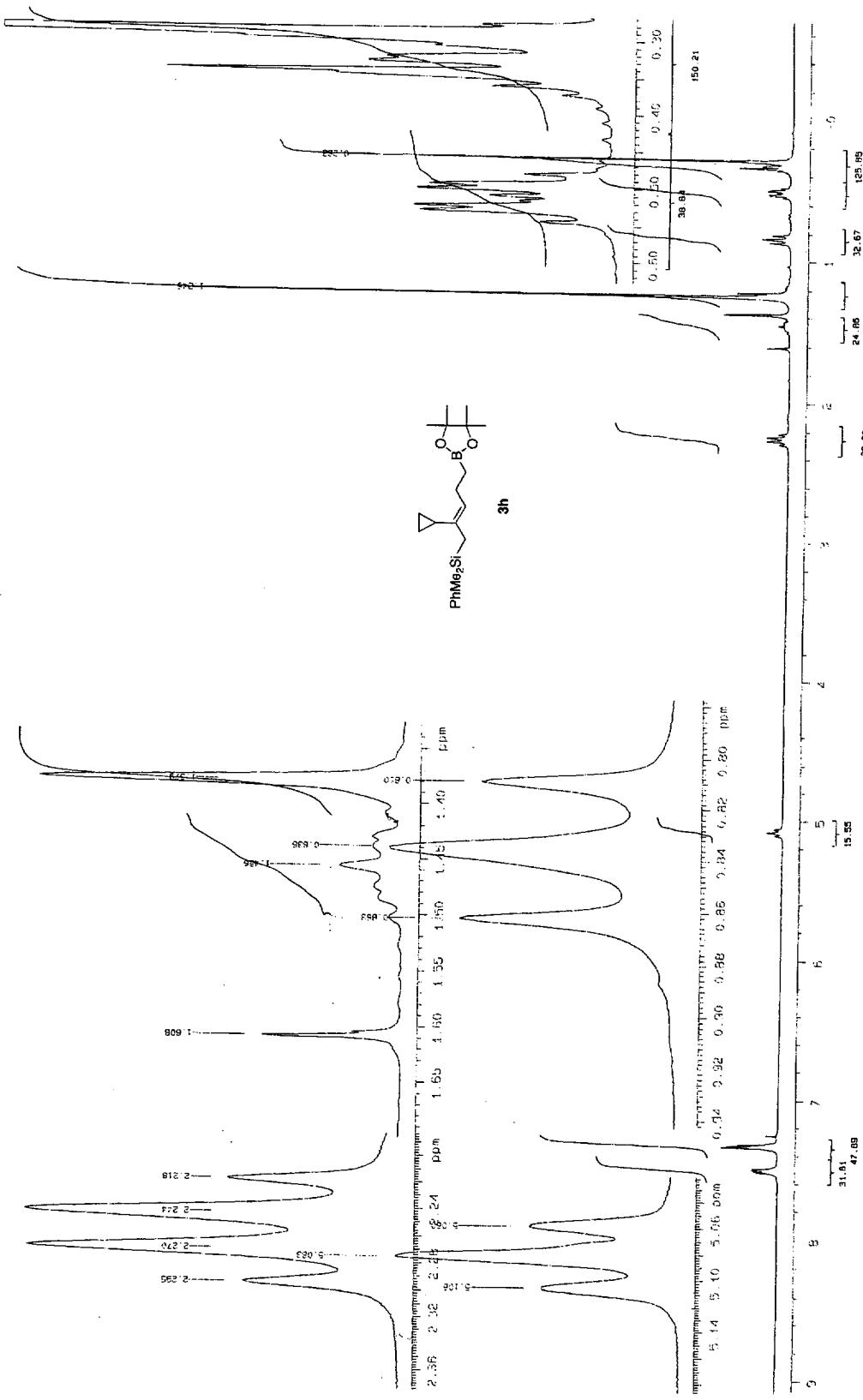
¹³C NMR of 3f.



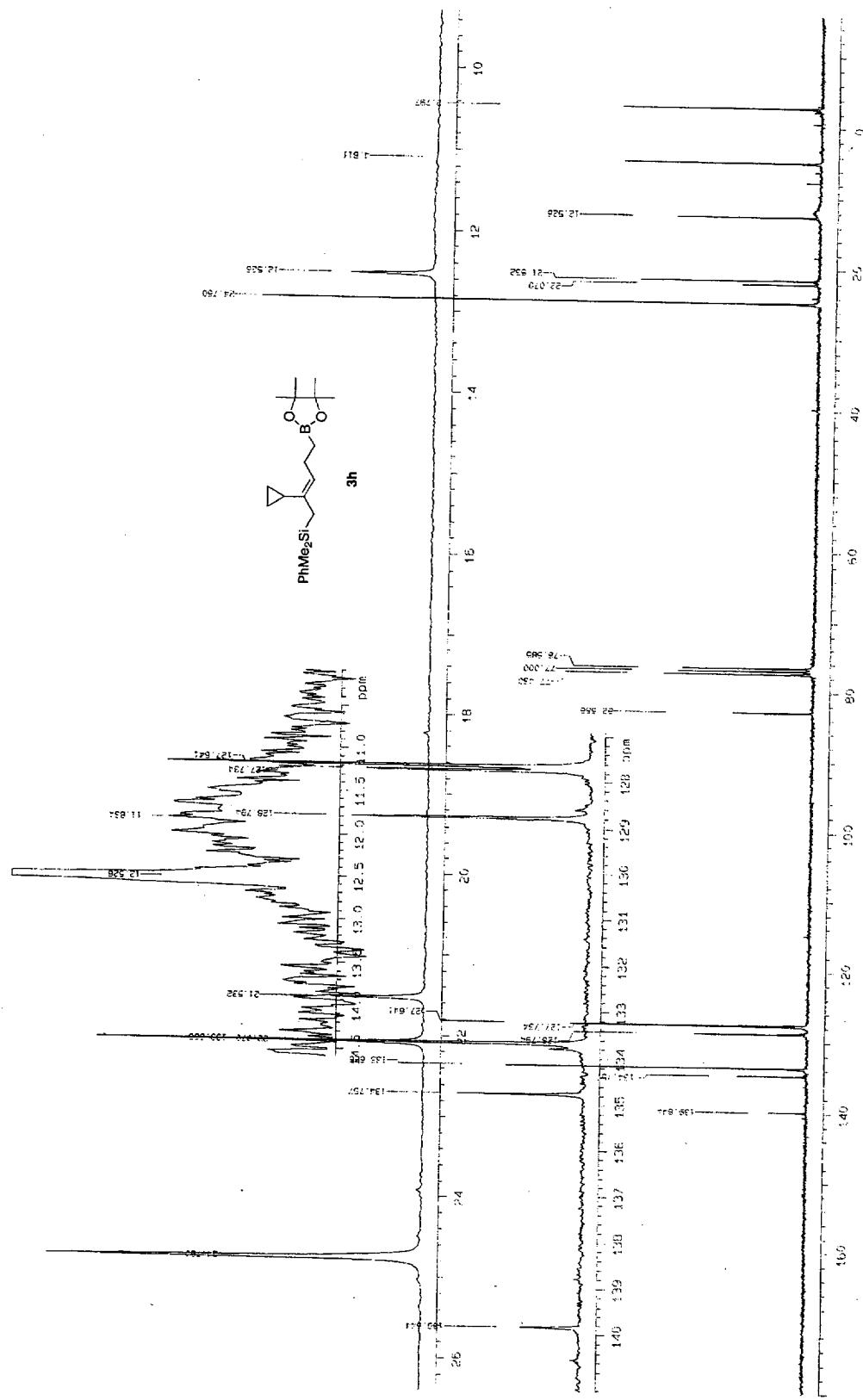
¹H NMR of 3g.



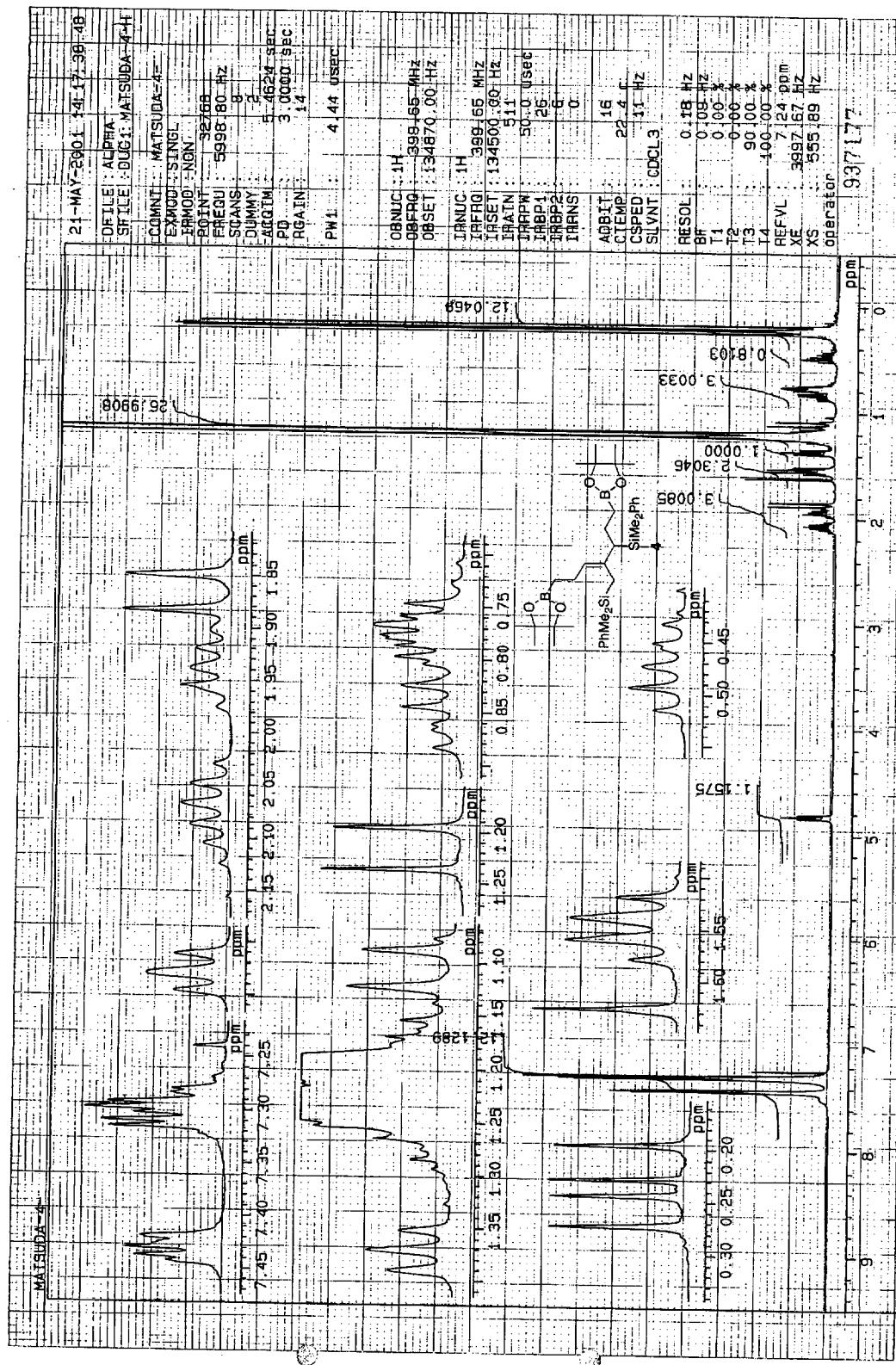
¹³C NMR of **3g**.

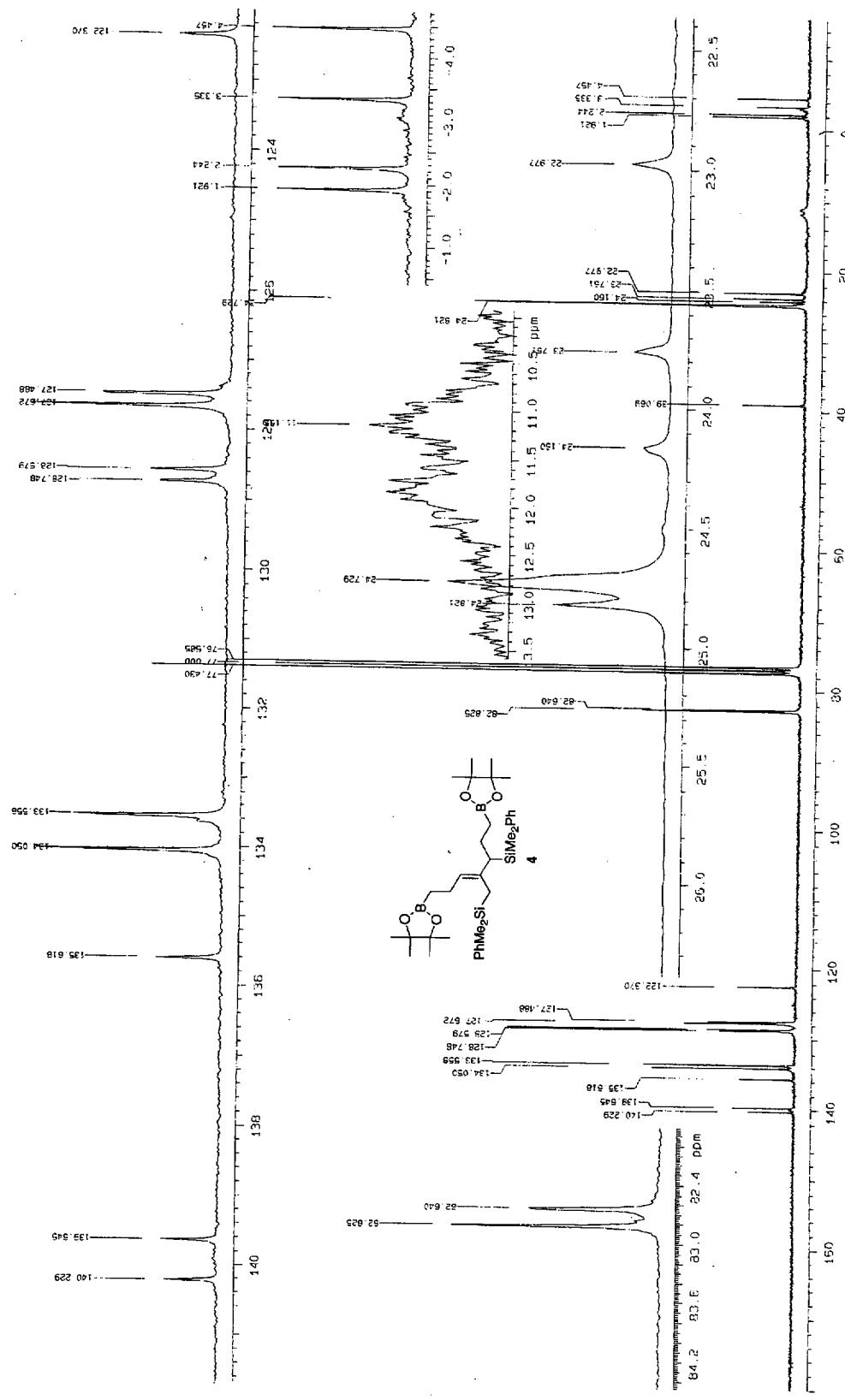


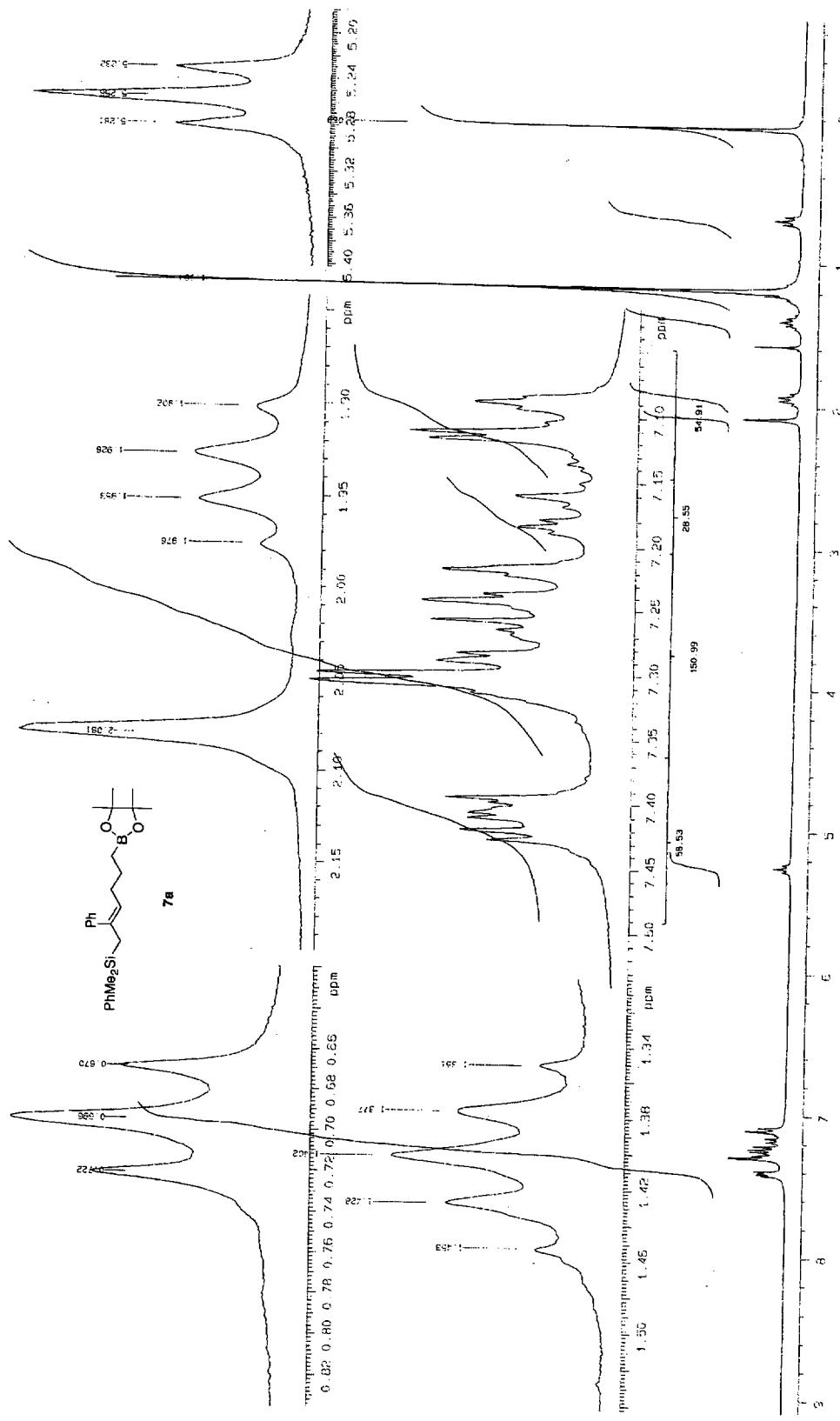
¹H NMR of 3h.

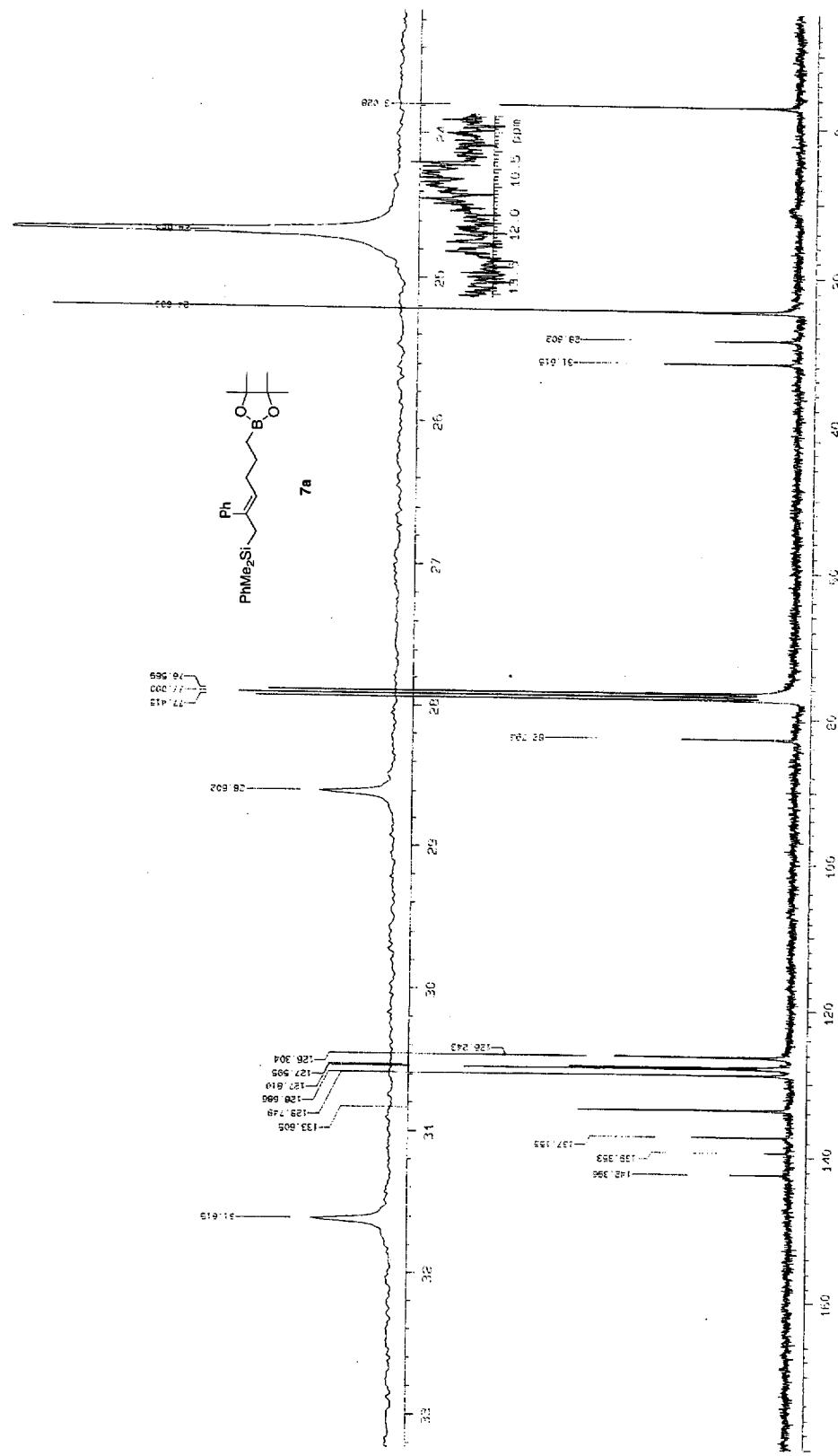


¹³C NMR of 3h.

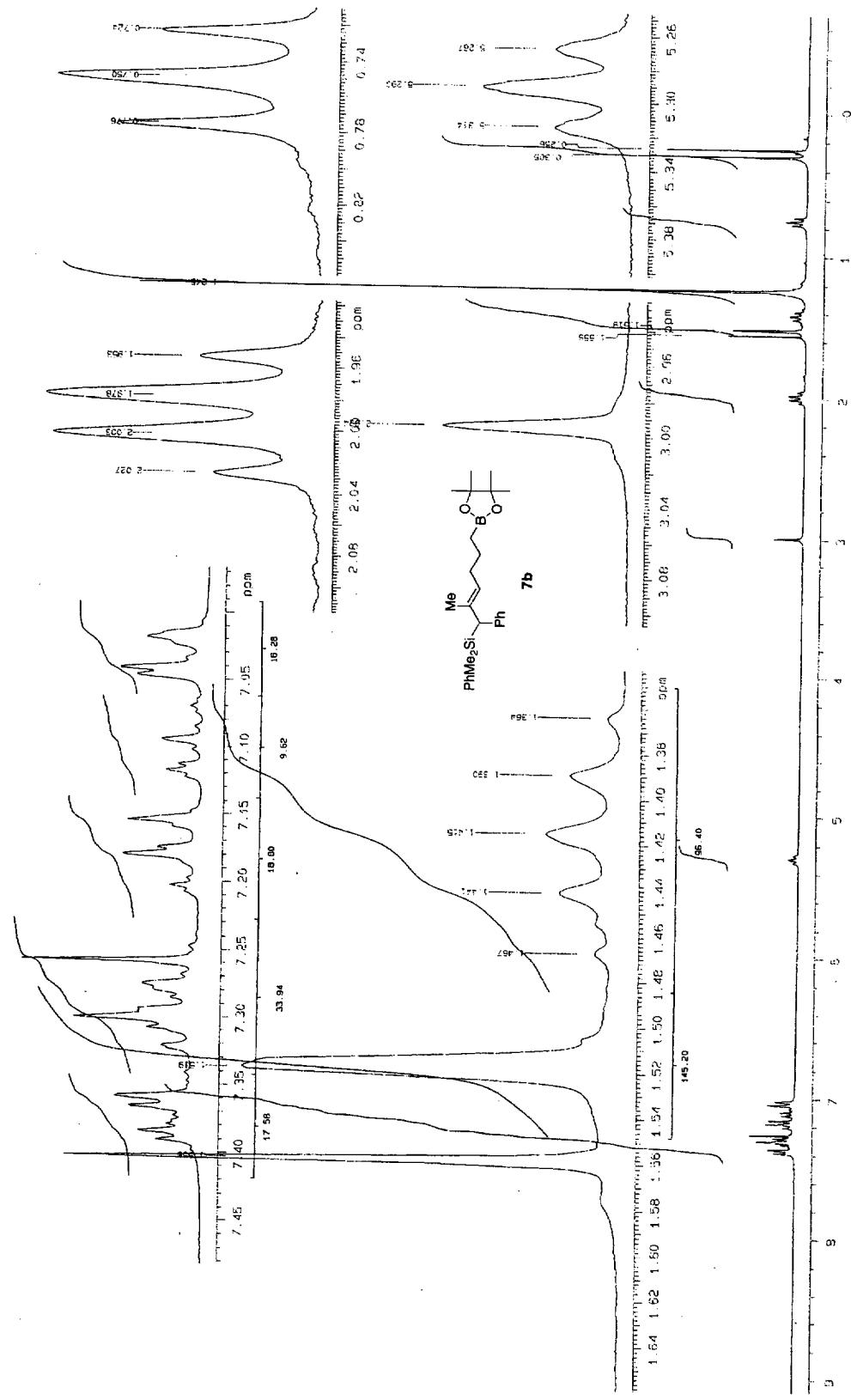
¹H NMR of 4.



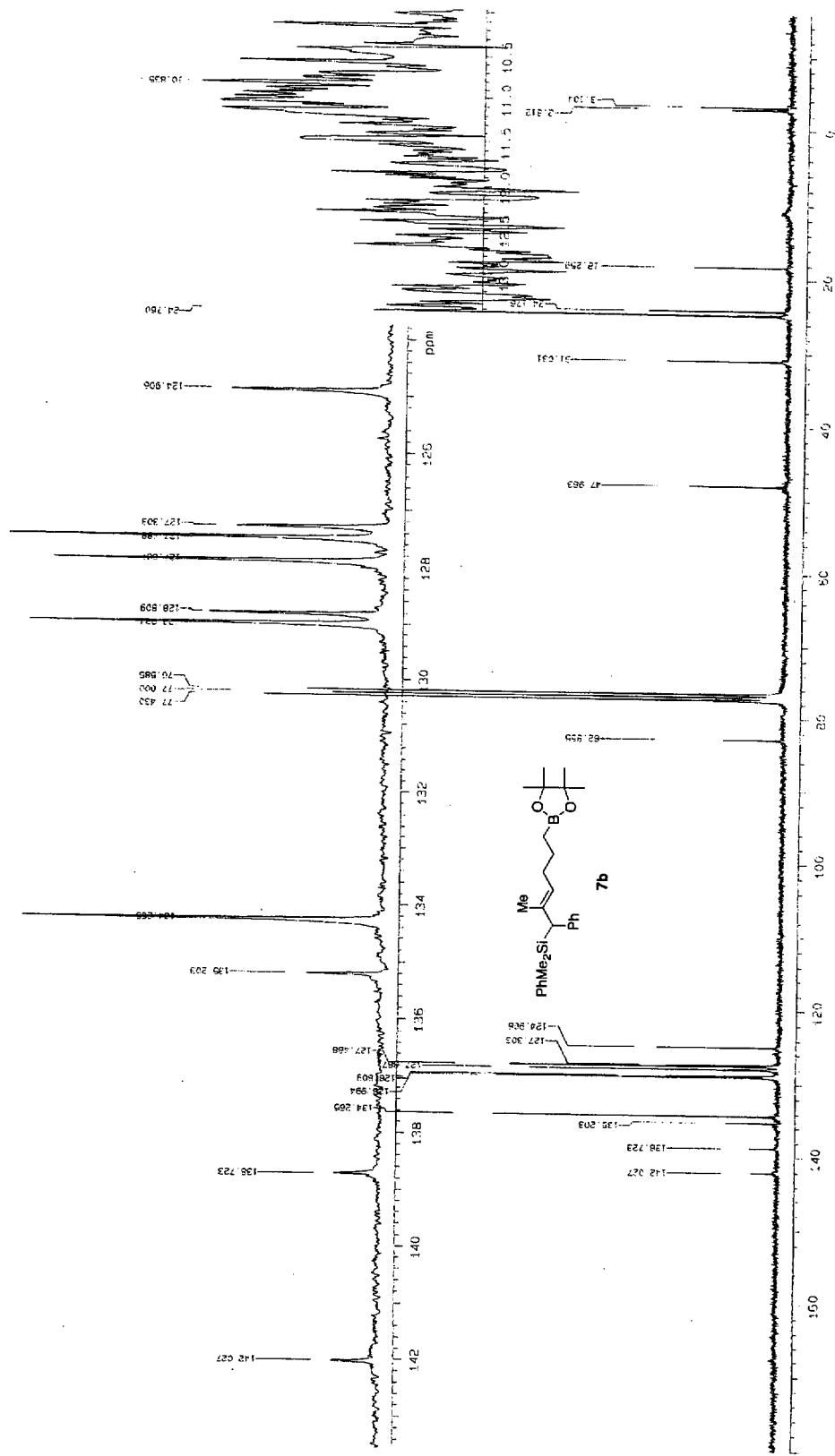




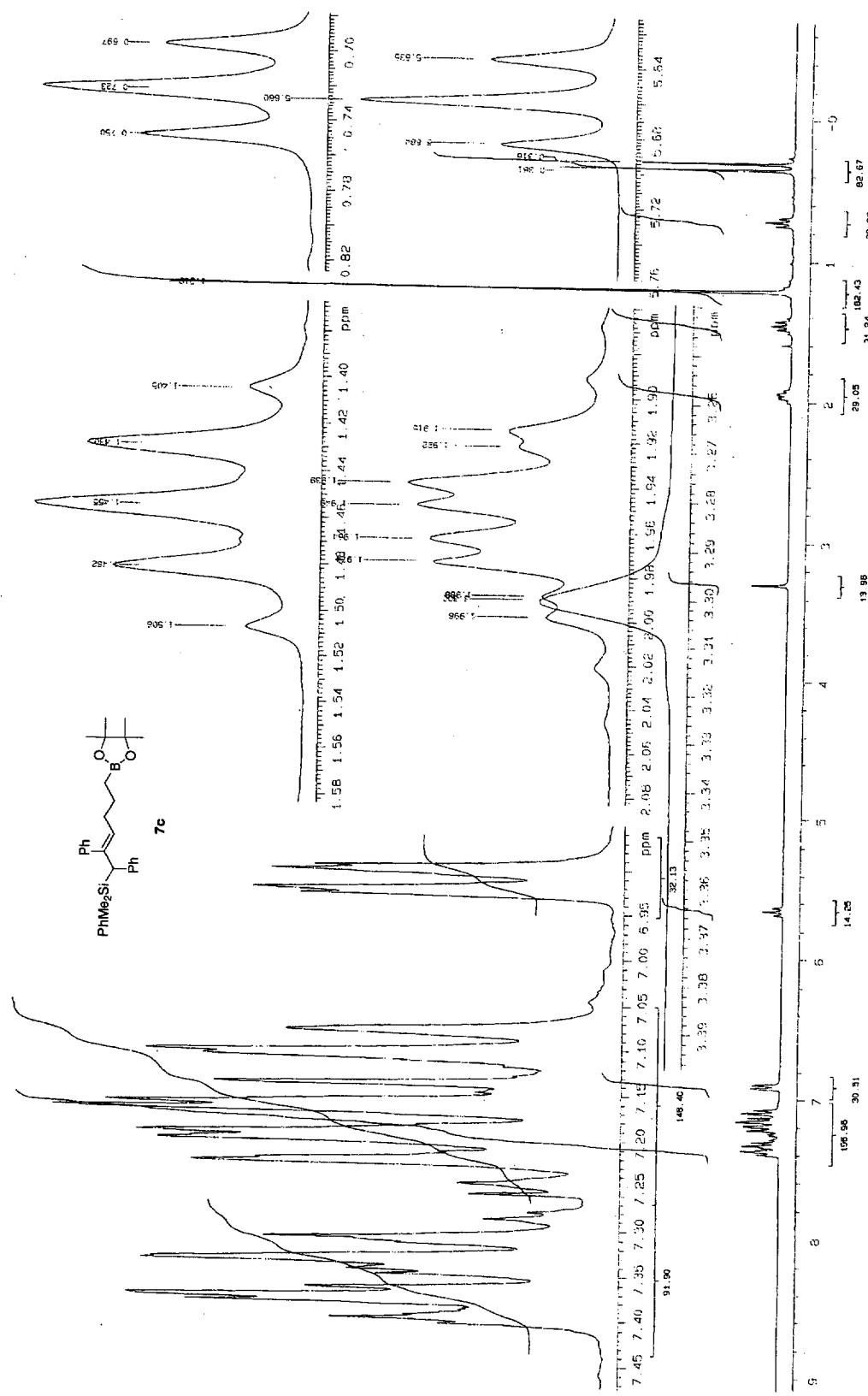
¹³C NMR of 7a.



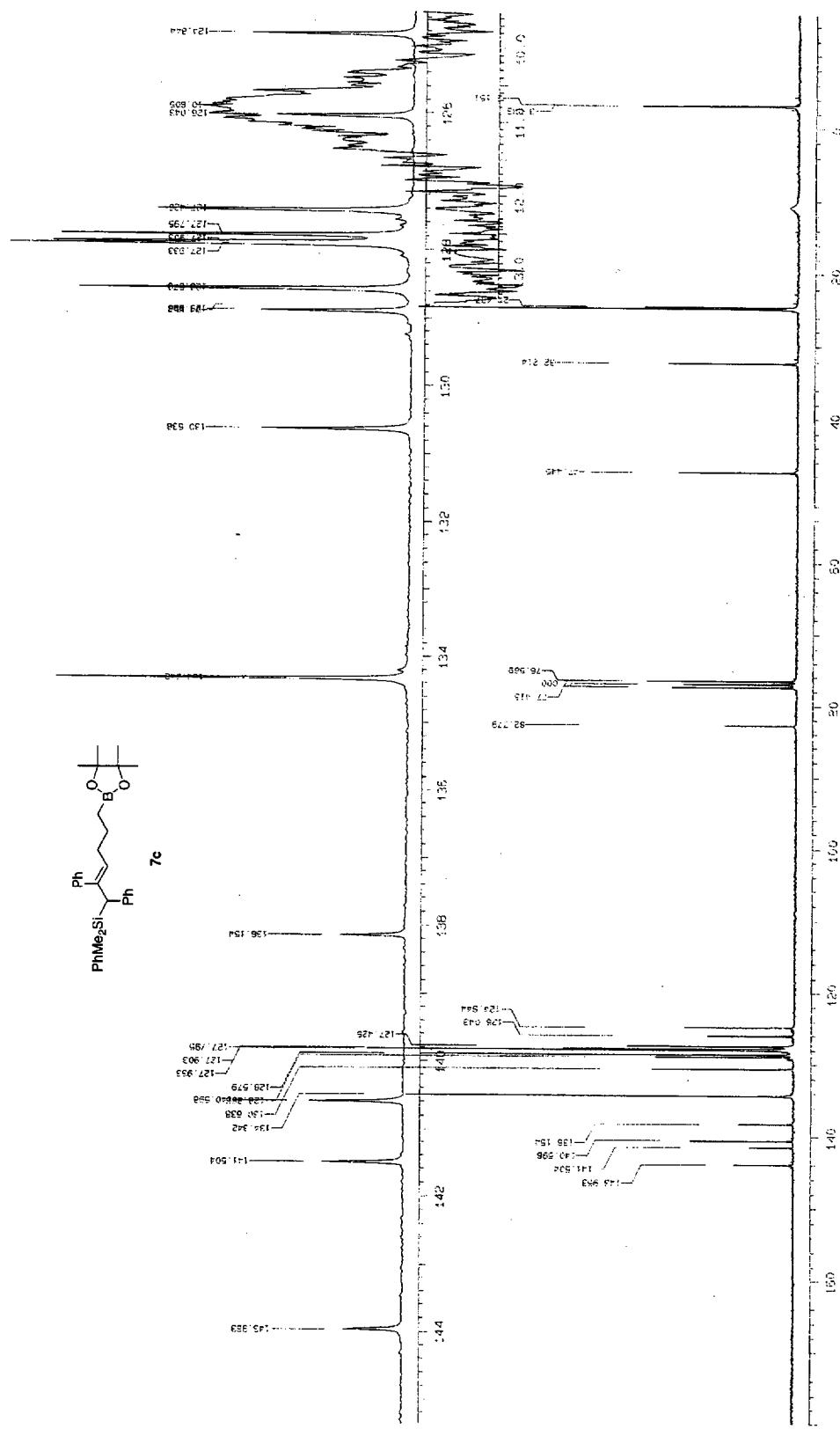
¹H NMR of **7b**.



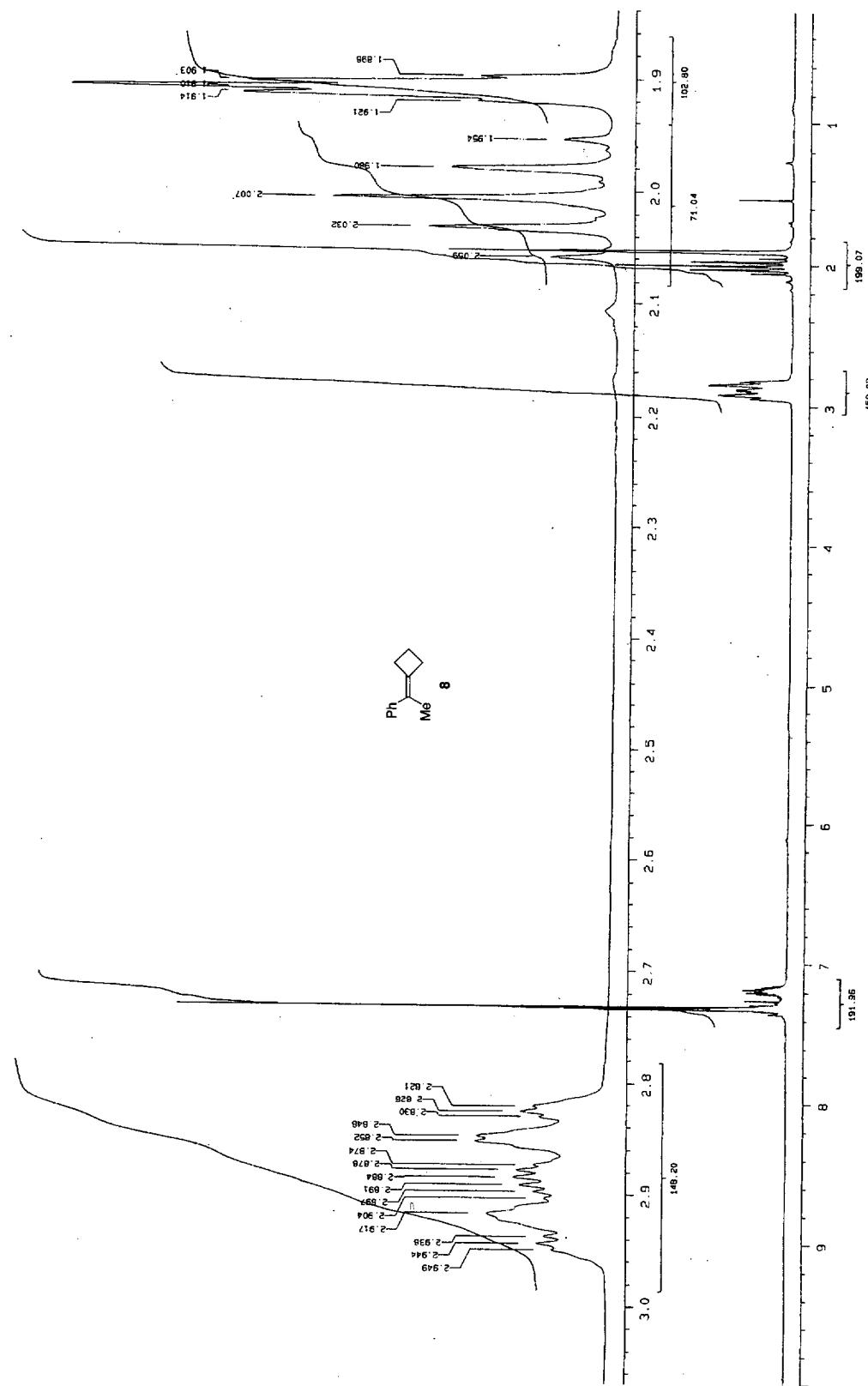
¹³C NMR of 7b.



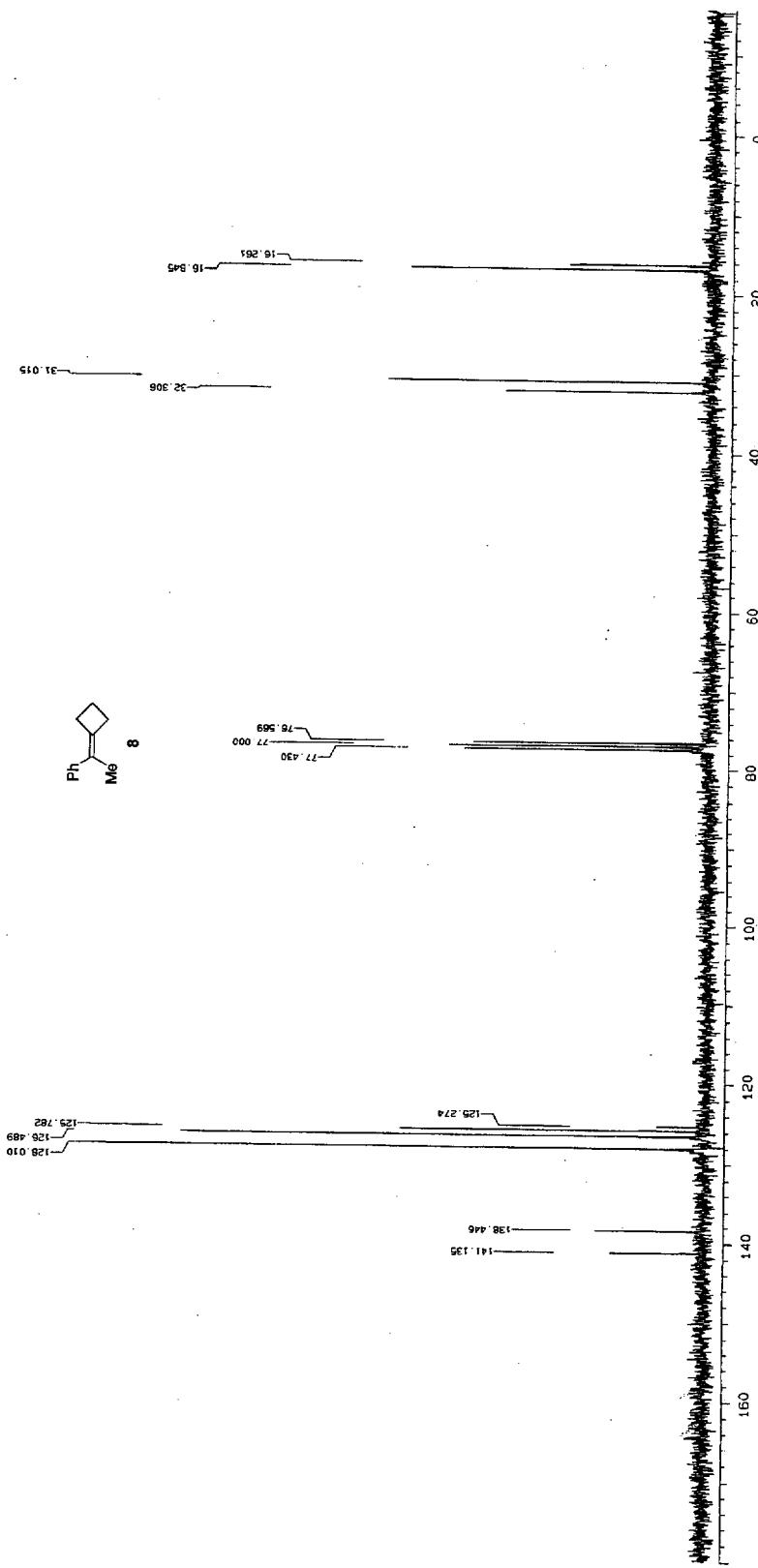
¹H NMR of 7c.



¹³C NMR of 7c.



¹H NMR of 8.



¹³C NMR of **8**.