METHYLLITHIUM PROMOTED WITTIG REARRANGEMENTS OF α-ALKOXYSILANES

Supporting Information

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Materials and Methods

Reactions were carried out in oven- or flame-dried glassware under nitrogen atmosphere, unless otherwise noted. All solvents were reagent grade. Diethyl ether and tetrahydrofuran (THF) were freshly distilled from sodium/benzophenone under nitrogen. Cyclohexane, methyllithium, *t*-butyllithium were purchased from Aldrich. Except as otherwise indicated, all reactions were magnetically stirred and monitored by thin layer chromatography with Whatman 0.25-mm precoated silica gel plates. Flash chromatography was performed with silica gel 60 Å (particle size 230-400 Mesh ASTM) supplied by Whatman Inc. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise stated. Infrared spectra were recorded on a Nicolet IR/42 spectrometer. Proton and carbon NMR spectra were recorded on a Varian Gemini-300 spectrometer. Chemical shifts are reported relative to the residue peaks of solvent chloroform (δ 7.24 for ^{1}H and δ 77.0 for ^{13}C). High-resolution mass spectra (HRMS) were measured by the Mass Spectrometry Laboratory of the Department of Chemistry and Biochemistry at the University of South Carolina.

α-(Trimethylsilyl)α-*d*-benzyl alcohol (6): A solution of α,α-d_2-benzyl alcohol (1.1 g, 10 mmol) in THF was cooled to 0 °C under nitrogen atmosphere. MeLi (7.9 mL, 1.4 M in diethyl ether, 11 mmol) was added dropwise via syringe. Upon complete addition the reaction was stirred for another 15 min before 1.52 mL (12 mmol) TMSCl was added dropwise. The solution was then stirred under nitrogen at 0 °C for an additional 15 min before being cooled to -76 °C. *t*-BuLi (10 mL, 1.7 M in hexanes, 14 mmol) was then added dropwise. After 5 min the dry ice-acetone bath was removed. The solution was stirred for an additional 1 hr before it was diluted with diethyl ether and quenched with saturated aqueous NH_4Cl. The organic phase was washed with water and brine. It was then dried over $MgSO_4$ and concentrated. Silica gel chromatography (3 to 10% diethyl ether in pentane gradient) afforded 0.38 g (21%) of **6** (along with 1.22 g (67%) of α,α-d_2-benzyltrimethylsilyl ether) as a colorless liquid. The spectroscopic data for **6** were consistent with those previously reported in the literature (Chuang, T.-H.; Fang, J.-M.; Jiaang, W.-T.; Tsai, Y.-M. *J. Org. Chem.* **1996**, *61*, 1794-1805).

Preparation of 1: To a solution of α-(trimethylsilyl)benzyl alcohol (0.85 g, 4.7 mmol) in 50 mL cyclohexane was added the trichloroacetimidate of crotyl alcohol (2.0 g, 9.2 mmol) followed by 0.1 mL TMSOTf in 1 mL cyclohexane via syringe. A white precipitate formed in several minutes. The reaction mixture was stirred at room temperature overnight before being filtered. The filtrate was diluted with petroleum ether, washed with saturated aqueous $NaHCO_3$, 1 N HCl, and brine. The organic phase was dried over $MgSO_4$ and concentrated. Silica gel chromatography (1% diethyl ether in pentane) furnished 740 mg (67%) of **1** and 277 mg (25%) of **7** as a 2:1 mixture of two diastereomers, all as colorless oils.

For **1**: IR (thin film) 3024, 2959, 1600, 1450, 1248, 1051 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.33-7.12 (m, 5 H), 5.60 (m, 2 H), 4.14 (s, 1 H), 4.09-4.01 (m, 1 H), 3.72-3.63 (m, 1 H), 1.73 (dd, *J* = 4.8, 1.0 Hz, 3 H), 0.0 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.7, 128.6, 128.3, 128.0, 125.9, 125.5, 76.9, 71.1, 17.8, -3.9; HRMS (EI) *m/z* 233.1361 [$(M-H)^+$; calcd for $C_{14}H_{21}OSi$ 233.1362].

For **7**: IR (thin film) 3024, 2928, 1450, 1248, 1020 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.18-7.13 and 7.33-7.26 (m, 5 H), 5.83 and 5.65 (ddd, *J* = 5.5, 10.4, 17.3 Hz and 7.7, 10.2, 17.3 Hz, 1 H), 5.23-4.98 (m, 2 H), 4.26 and 4.23 (s, 1 H), 3.88 and 3.74 (q, *J* = 6.32 and 6.32 Hz, 1 H), 1.20 and 1.23 (d, *J* = 6.30 and 6.30 Hz, 3 H), 0.00 and -0.02 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.3 and 142.2, 141.4 and 140.5, 127.9 and 127.8, 125.9 and 125.7, 125.4 and 125.4, 116.2 and 113.6, 75.5 and 75.0, 74.7 and 74.0, 22.4 and 22.2, -3.96 and -3.96; HRMS (EI) *m/z* 233.1356 [$(M-H)^+$; calcd for $C_{14}H_{22}OSi$ 233.1362].

Preparation of 1-d_1: Applying the representative procedure above to 0.53 g (2.9 mmol) of **6** and 1.6 g (7.3 mmol) of the trichloroacetimidate of crotyl alcohol afforded after silica gel chromatography (3% diethyl ether in pentane) 0.71 g (56%) of **1-d_1** as a colorless liquid. IR (thin film) 2963, 2070, 1601, 1493, 1440, 1248 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.33-7.12 (m, 5 H), 5.60 (m, 2 H), 4.09-4.01 (m, 1 H), 3.72-3.63 (m, 1 H), 1.73 (dd, *J* = 4.8, 1.0 Hz, 3 H), 0.0 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6, 128.5, 128.3, 128.0, 125.8, 125.5, 76.3 (t, J_{C-D} = 19.8 Hz), 71.0, 17.8, -3.9.

Preparation of 21: Applying the representative procedure above to 1.32 g (10 mmol) α-(trimethylsilyl)allyl alcohol and 4.3 g (18.6 mmol) of the trichloroacetimidate of *cis*-2-penten-1-ol afforded

after silica gel chromatography (3% diethyl ether in pentane) 0.61 g (31%) of **21** as a colorless liquid. IR (thin film) 2967, 1767, 1746, 1458, 1248, 841 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.77 (ddd, *J* = 18.0, 10.8, 7.2 Hz, 1 H), 5.59-5.39 (m, 2 H), 5.10-4.90 (m, 2 H), 4.13-4.06 (m, 1 H), 3.93-3.89 (m, 1 H), 3.57 (dt, *J* = 7.2, 3.0, 1.5 Hz, 1 H), 2.03 (quent, *J* = 7.2 Hz, 2 H), 0.94 (t, *J* = 7.2, 14.4 Hz, 3 H), 0.00 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.6, 135.0, 126.2, 112.1, 75.7, 65.7, 20.9, 14.3, -3.98; HRMS (EI) *m/z* 198.1438 [$(M)^+$; calcd for $C_{11}H_{22}OSi$ 198.1440].

For the detailed synthetic procedures and the spectroscopic data of compounds **10**, **13**, **15**, **18**, and **23**, see the preceding Letter this issue.

Wittig rearrangement reaction of 1 with MeLi. Preparation of 2a/b, 3a/b, and 4a/b: A solution of 176 mg (0.73 mmol) of silane **1** in 10 mL THF was cooled to 0 °C under nitrogen. MeLi (1.4 M in diethyl ether, 0.9 mL, 1.26 mmol) was added dropwise via syringe. The solution was stirred overnight at room temperature. It was then quenched with saturated aqueous NH_4Cl, diluted with diethyl ether, washed with 1 N HCl, water, and brine. The organic phase was dried over $MgSO_4$ and concentrated. Silica gel chromatography (1 to 5% diethyl ether in hexane gradient) afforded 35 mg (20%) of **2a**, 38 mg (22%) of **2b**, 24 mg (20%) of **3a/b** as an inseparable mixture (1.2:1) of diastereomers, and 16 mg (9%) of **4a/b** as an inseparable mixture (2:1) of diastereomers, all as colorless oils.

For **2a**: IR (thin film) 3567, 3065, 2961, 1444, 1248 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39-7.03 (m, 5 H), 5.51 (ddd, *J* = 17.1, 10.8, 5.4 Hz, 1 H), 5.12-4.98 (m, 2 H), 3.15 (m, 1 H), 1.59 (bs, 1 H), 1.25 (d, *J* = 6.9 Hz, 3 H), -0.02 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.2, 138.8, 128.0, 125.1, 124.1, 116.7, 72.8, 43.4, 14.5, -2.3; HRMS (EI) *m/z* 233.1359 [$(M-H)^+$; calcd for $C_{14}H_{21}OSi$ 233.1362].

For **2b**: IR (thin film) 3567, 3065, 2961, 1444, 1248 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.10 (m, 5 H), 6.06 (m, 1 H), 5.30-5.10 (m, 2 H), 2.98 (m, 1 H), 1.60 (bs, 1 H), 0.77 (d, *J* = 6.9 Hz, 3 H), -0.05 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.9, 141.4, 128.0, 125.0, 124.3, 115.6, 74.1, 45.3, 13.4, -2.44; HRMS (EI) *m/z* 233.1359 [$(M-H)^+$; calcd for $C_{14}H_{21}OSi$ 233.1362].

For **3a/b**: The spectroscopic data were consistent with those previously reported in the literature (For **3a** see: Kobayashi, S.; Nishio, K. *J. Org. Chem.* **1994**, *59*, 6620-6628. For **3b** see: Kang, S-K.; Kim, D-Y.; Hong, R-K.; Ho, P-S. *Synth. Commun.* **1996**, *26*, 1493-1498).

For **4a/b**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.10 (m, 5 H), 5.85 and 5.70 (m, 1 H), 5.05-4.86 (m, 2 H), 4.46 and 4.44 (d, *J* = 6.6 Hz, 1 H), 2.52-2.38 (m, 1 H), 1.00 and 0.88 (d, *J* = 6.9 Hz, 3 H), 0.00 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.8 and 143.7, 141.3 and 140.9, 127.7 and 127.6, 126.9 and 126.8, 127.7 and 126.6, 114.3 and 114.2, 79.0 and 78.7, 46.0 and 45.8, 16.2 and 14.6, 0.1 and 0.1. For a prior preparation see: Hollis, T. K.; Robinson, N. P.; Whelan, J.; Bosnich, B. *Tetrahedron Lett.* **1993**, *34*, 4309-4312.

Wittig rearrangement reaction of 1-*d*$_1$ with MeLi. Preparation of 2a/b, 3a/b-*d*$_1$, and 4a/b: Applying the representative procedure above to 90 mg (0.38 mmol) of **1-*d*$_1$** afforded after silica gel chromatography (3 to 10% diethyl ether in pentane gradient) 11 mg (12%) of **2a/b** as a mixture (1.4:1) of diastereomers, 66 mg (73 %) of **3a/b-*d*$_1$** as a mixture (1.1:1) of diastereomers, all as colorless oils. (Though not isolated, ^{1}H NMR spectrum of the crude reaction mixture revealed that a 1.2:1 mixture of **4a/b** was also formed in approximately 1.5% yield.)

For the spectroscopic data of **2a** and **2b** see above.

For **3a/b-*d*$_1$**: IR (thin film) 3412, 2961, 1640, 1449 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.20 (m, ArH), 5.87-5.70 (m, 1 H), 5.25-5.00 (m, 2 H), 2.59 and 2.48 (m, 1 H), 1.82 (bs, 1 H), 1.10 and 0.87 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.6 and 140.2, 128.2 and 128.1, 127.6 and 127.3, 126.8 and 126.5, 116.9 and 115.6, 46.2 and 44.5, 16.5 and 13.9; GC/MS (EI) *m/z* 162.0, 107.1, 77.0.

Wittig rearrangement reaction of 7 with MeLi. Preparation of 8 and 9: Applying the representative procedure above to 117 mg (0.50 mmol) of silane **7** afforded after silica gel chromatography (3 to 10% diethyl ether in pentane gradient) 61 mg (75%) of **8** and 24 mg (21%) of **9** as colorless oils.

For **8**: The spectroscopic data were consistent with those previously reported in the literature (Kang, S-K.; Kim, D-Y.; Hong, R-K.; Ho, P-S. *Synth. Commun*. **1996**, *26*, 1493-1498).

For **9**: IR (neat) 3341, 2961, 1696, 1450, 1248 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39-7.03 (m, 5 H), 5.54-5.50 (m, 1 H), 5.20-5.00 (m, 1 H), 2.80-3.00 (dm, 1 H), 2.54 (dd, J = 14.1, 9.9 Hz, 1 H), 1.89 (br s, 1 H), 1.60 (dt, *J* = 6.3, 1.5, 1.5 Hz, 3 H), 0.03 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.9, 131.3, 127.9, 125.0, 124.9,124.6, 70.5, 39.9, 18.0, -4.18; HRMS (EI) *m/z* 233.1359 [$(M-H)^+$; calcd for $C_{14}H_{21}OSi$ 233.1362].

Wittig rearrangement reaction of 10 with MeLi. Preparation of 11 and 12: Applying the representative procedure above to 118 mg (0.51 mmol) of silane **10** afforded after silica gel chromatography (3 to 10% diethyl ether in pentane gradient) 59 mg (50%) of **11** and 26 mg (32%) of **12** as colorless oils.

For **11**: IR (thin film) 3503, 2967, 1634, 1487, 1223, 1032 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.20 (m, 5 H), 4.88 (m, 1 H), 4.68 (m, 1 H), 2.76 (AB, Δ = 74.7 Hz, *J* = 13.8 Hz, 2 H), 1.58 (bs, 1 H), 1.26 (s, 3 H), -0.01 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.1, 141.3, 127.7, 125.0, 124.7, 115.8, 69.0, 44.3, 24.7, -4.1; GC/MS (EI) *m/z* 234.2, 233.2, 219.2, 179.2, 73.0, 55.0; HRMS (EI) *m/z* 233.1355 [$(M-H)^+$; calcd for $C_{14}H_{21}OSi$ 233.1362].

For **12**: The spectroscopic data were consistent with those previously reported in the literature (Kobayashi, S.; Nishio, K. *J. Org. Chem*. **1994**, *59*, 6620-6628).

Wittig rearrangement reaction of 13 with MeLi. Preparation of 14: Applying the representative procedure above to 74 mg (0.27 mmol) of silane **13** afforded after silica gel chromatography (10% diethyl ether in pentane) 5 mg (9%) of **14** as the sole rearrangement product. The spectroscopic data for **14** were consistent with those previously reported in the literature (Sidduri, A.; Rozema, M. J.; Knochel, P. *J. Org. Chem*. **1993**, *58*, 2694-2713).

Wittig rearrangement reaction of 15 with MeLi. Preparation of 16 and 17: Applying the representative procedure above to 400 mg (1.83 mmol) of silane **15** and 3.3 mL (4.6 mmol) MeLi afforded after silica gel chromatography (5 to 10% diethyl ether in pentane gradient) 89 mg (33%) of **16** and 60 mg (15%) of **17** as colorless oils.

For **16**: The spectroscopic data were consistent with those previously reported in the literature (Shinokubo, H.; Miki, H.; Yokoo, T.; Oshima, K.; Utimoto, K. *Tetrahedron* **1995**, *51*, 11681-11692).

For **17**: IR (thin film) 3509, 3305, 2959, 1248, 841 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.16 (m, 5 H), 2.92 (ABd, Δ = 39.3 Hz, J = 16.8, 2.7 Hz, 2 H), 2.22 (br s, 1 H), 1.89 (t, J = 2.4 Hz, 1 H), 0.00 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.9, 128.0, 125.7, 124.8, 79.8, 72.0, 70.3, 28.2, -4.1; HRMS (EI) *m/z* 217.1041 [$(M-H)^+$; calcd for $C_{13}H_{17}OSi$ 217.1049].

Wittig rearrangement reaction of 18 with MeLi. Preparation of 19 and 20: Applying the representative procedure above to 73 mg (0.30 mmol) of silane **18** afforded after silica gel chromatography (5 to 10% diethyl ether in pentane gradient) 43 mg (59%) of **19** and 10 mg (19%) of **20** as colorless oils.

For **19**: IR (thin film) 2953, 1716, 1240, cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 7.40-7.10 (m, 5 H), 6.47 (d, J = 16.2 Hz, 1 H), 6.31 (dt, J = 6.6, 16.2 Hz, 1 H), 3.34 (d, J = 6.6 Hz, 2 H), 2.41-2.48 (m, 2 H), 0.74-0.81 (m, 2 H), 0.00 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.7, 136.9, 133.4, 128.5, 127.5, 126.2, 122.3, 46.2, 37.1, 10.2, -1.8; HRMS (EI) *m/z* 246.1432 [calcd for $C_{15}H_{22}OSi$ 246.1440].

For **20**: The spectroscopic data were consistent with those previously reported in the literature (Enholm, E. J.; Satici, H.; Prasad, G. *J. Org. Chem.* **1990**, *55*, 324-329).

Wittig rearrangement reaction of 21 with MeLi. Preparation of 22: Applying the representative procedure above to 130 mg (0.66 mmol) of silane **21** afforded after silica gel chromatography (10% diethyl ether in pentane) 119 mg (92%) of **22** as a clear oil. IR (thin film) 2959, 1717, 1636, 1250, 839 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.76-5.64 (m, 1 H), 5.17-5.11 (m, 2 H), 3.03 (q, J = 7.8 Hz, 1 H), 2.50-2.24 (m, 2 H), 1.75 (sep, J = 7.5 Hz, 1 H), 1.48 (sep, J = 7.5 Hz, 1 H), 0.85 (t, J = 7.5

Hz, 3 H), 0.75-0.69 (m, 2 H), -0.02 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 211.7, 136.5, 117.5, 58.9, 36.1, 24.3, 11.7, 9.9, -1.8; HRMS (EI) *m/z* 198.1424 [calcd for $C_{11}H_{22}OSi$ 198.1440].

Wittig rearrangement reaction of 23 with MeLi. Preparation of 24 and 25: Applying the representative procedure above to 97 mg (0.44 mmol) of silane **23** afforded after silica gel chromatography (3 to 10% diethyl ether in pentane gradient) 58 mg (60%) of **24** and 20 mg (21%) of **25** as colorless oils.

For **24**: IR (thin film) 2942, 1716, 1640, 1497, 1252, 847 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.10 (m, 5 H), 2.62 (t, *J* = 7.4 Hz, 2 H), 2.58 (t, *J* = 7.5 Hz, 2 H), 1.85 (q, *J* = 7.5 Hz, 2 H), 0.18 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 247.9, 141.8, 128.4, 128.3, 125.9, 47.6, 35.2, 23.7, -3.2; HRMS (EI) *m/z* 219.1210 [$(M-H)^+$; calcd for $C_{13}H_{19}OSi$ 219.1210].

For **25**: IR (thin film) 2953, 1718, 1250, 841 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.10 (m, 5 H), 2.70 (s, 2 H), 2.39-2.45 (m, 2 H), 0.71-0.77 (m, 2 H), -0.05 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.2, 134.4, 129.3, 128.6, 126.9, 49.3, 36.6, 10.2, -1.9; HRMS (EI) *m/z* 233.1279 [calcd for $C_{13}H_{20}OSi$ 220.1283].

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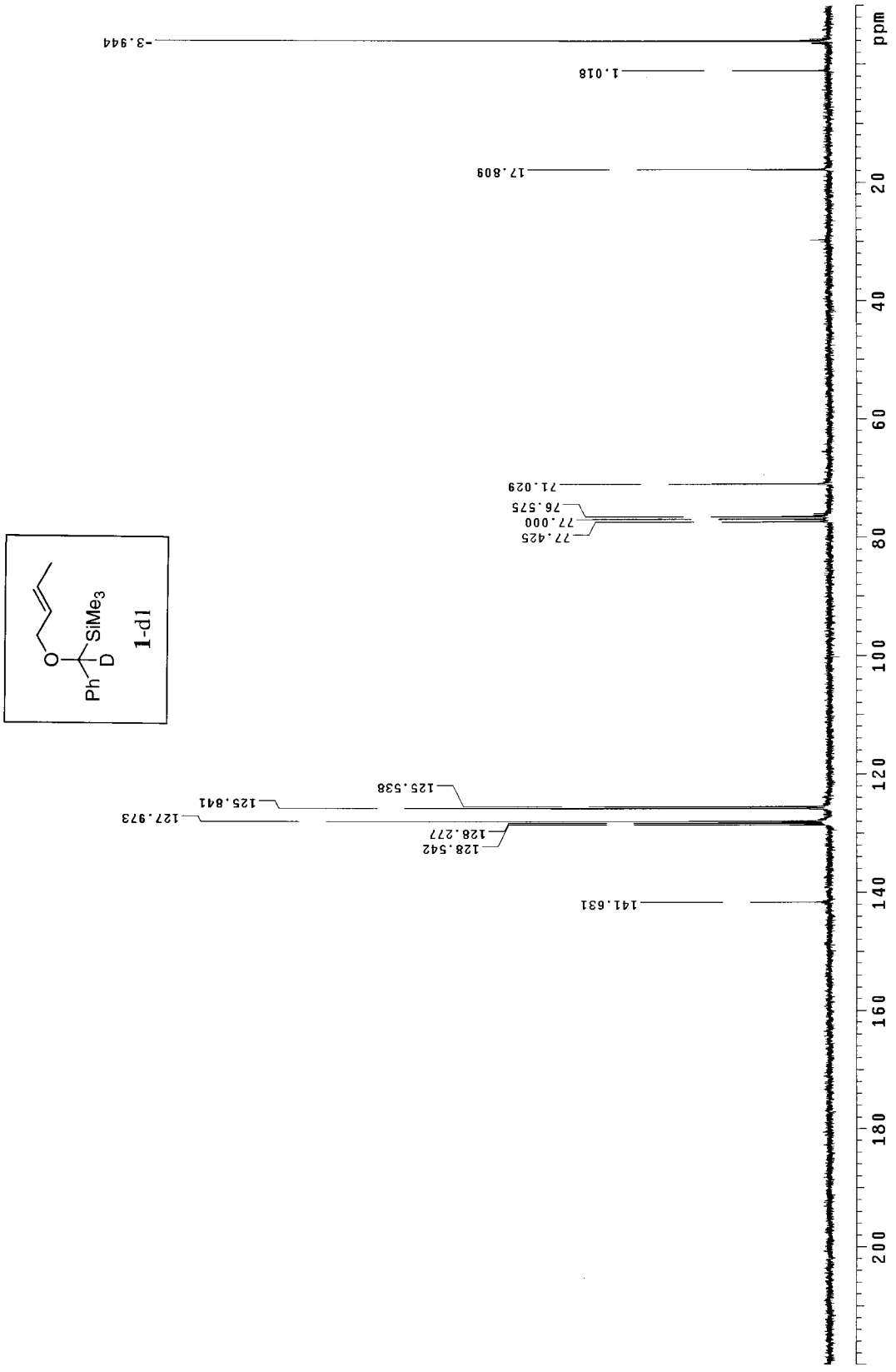
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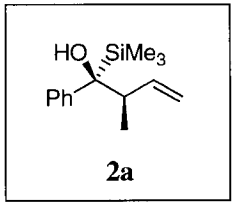

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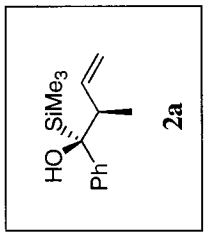
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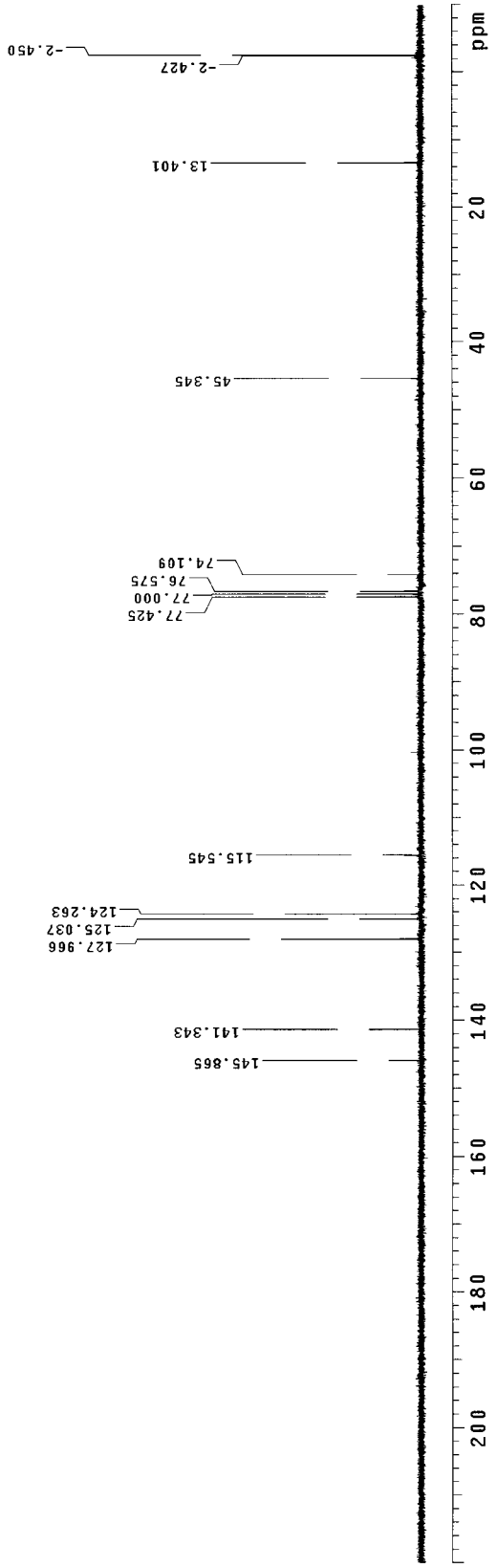
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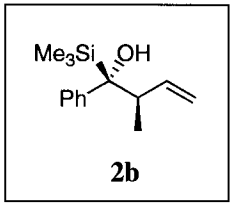

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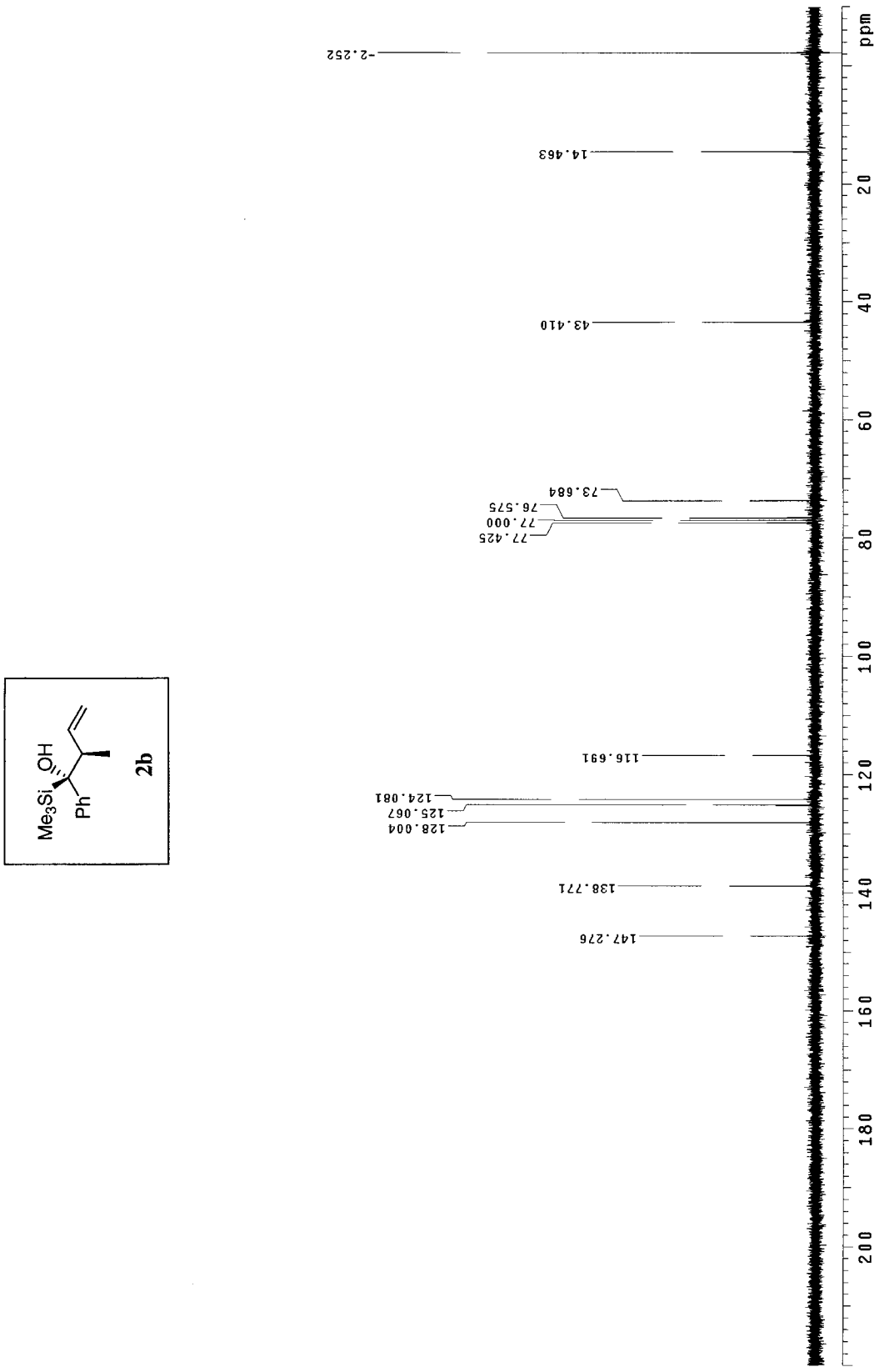
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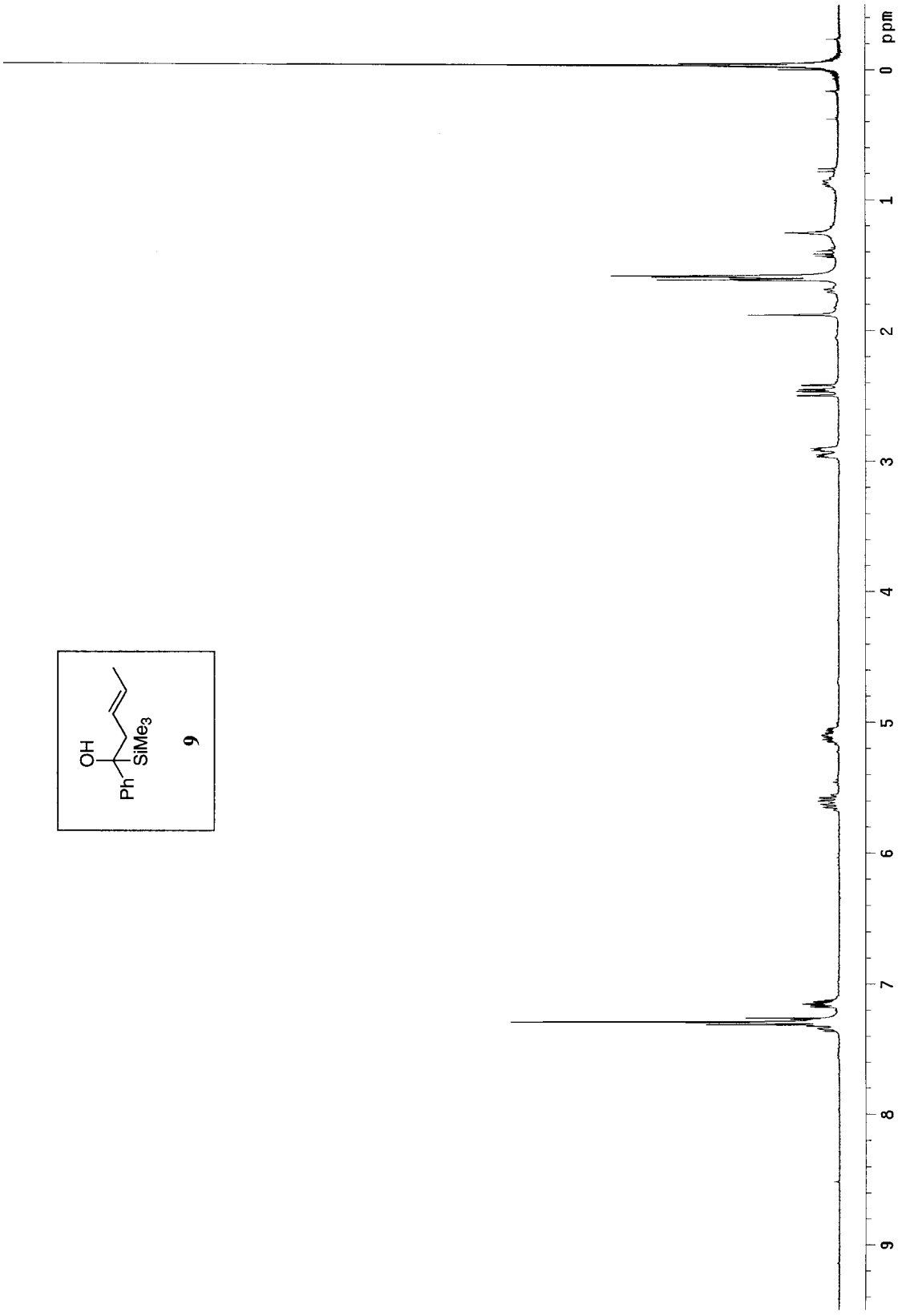
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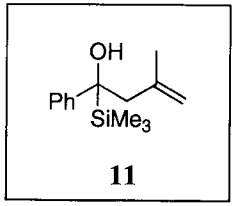

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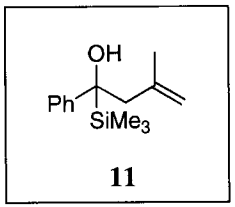
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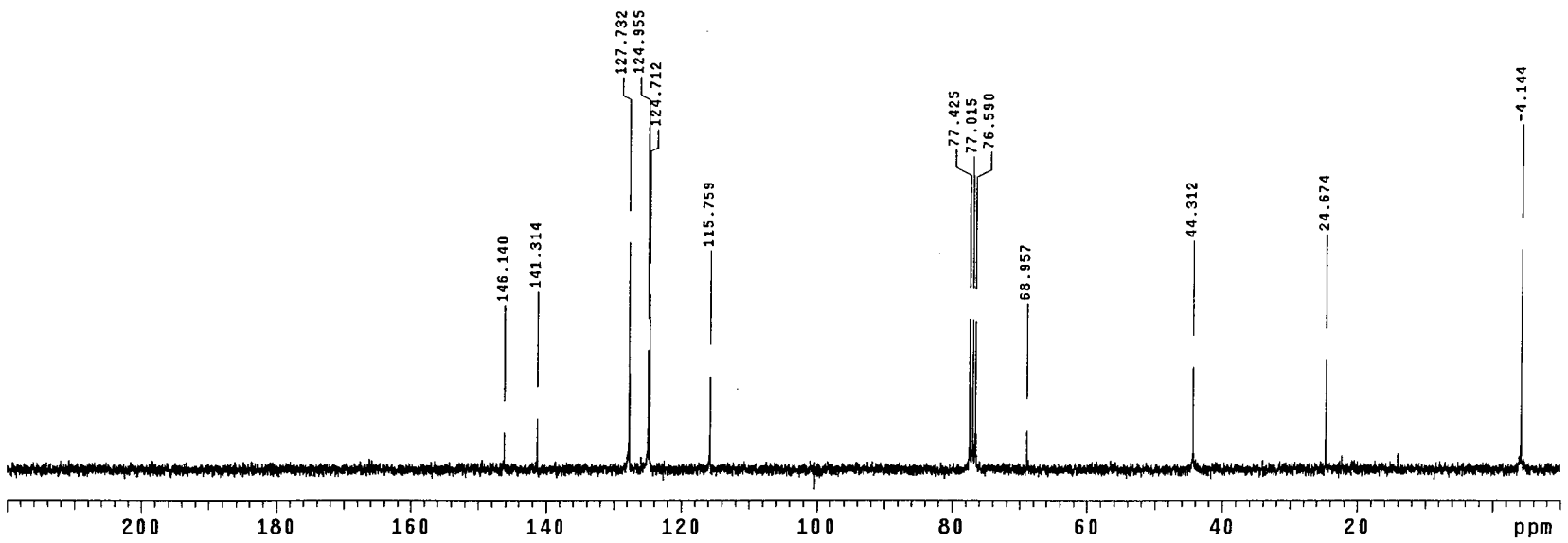
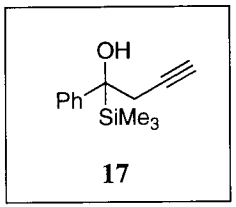
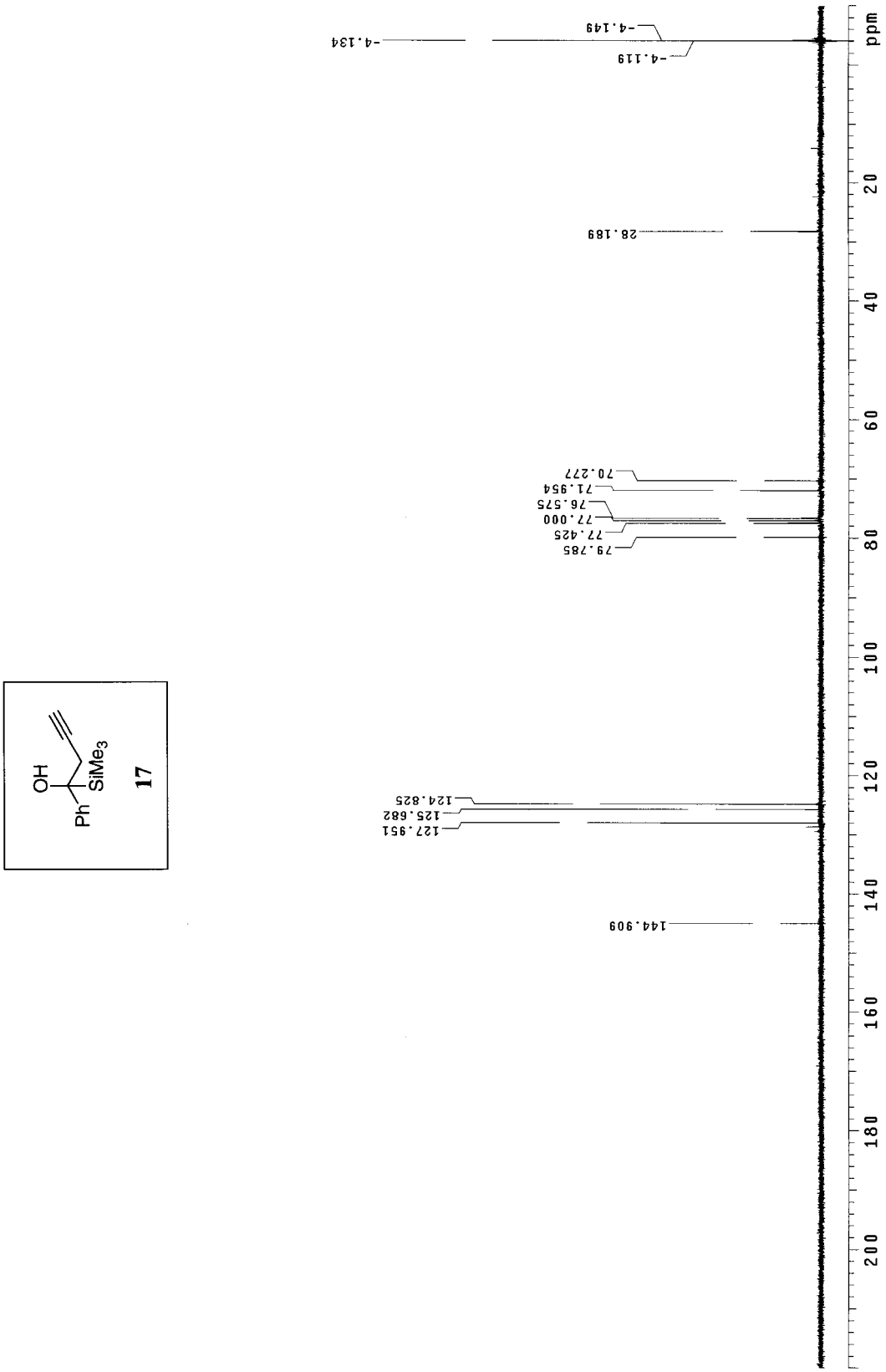
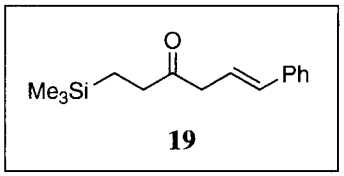
11


146.140
141.314
127.732
124.955
124.712
115.759
77.425
77.015
76.590
68.957
44.312
24.674
-4.144
200
180
160
140
120
100
80
60
40
20
ppm

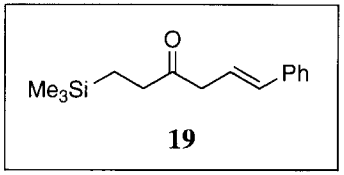


17

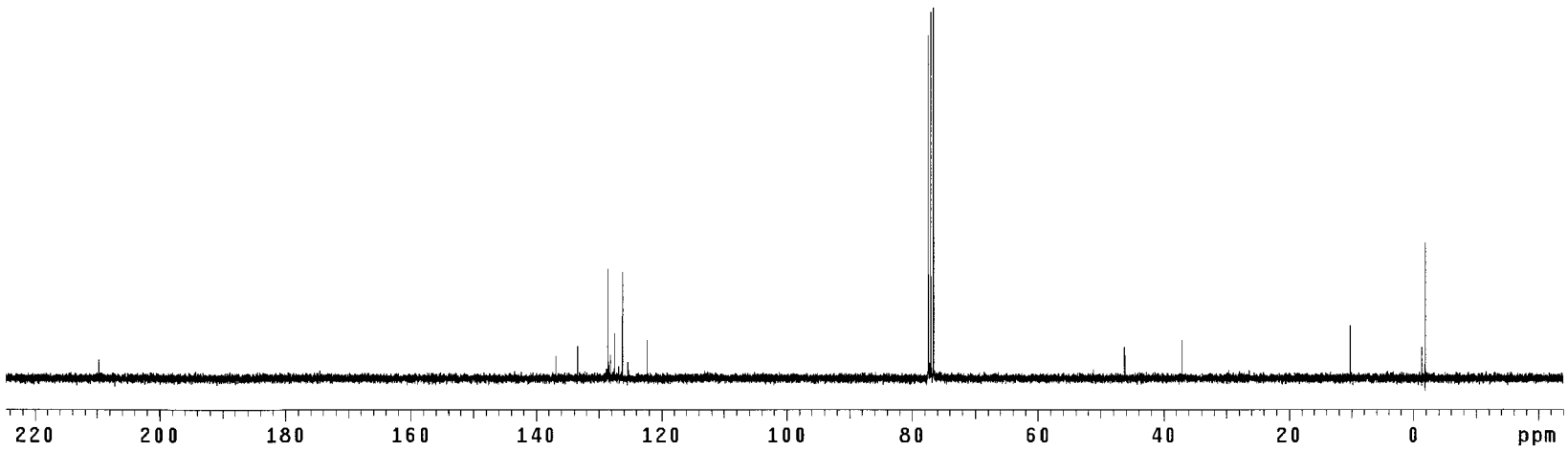

OH
Ph
SiMe3
17
-4.134
-4.149
-4.119
28.189
70.277
71.954
76.575
77.000
77.425
79.785
124.825
125.682
127.951
144.909
ppm
20
40
60
80
100
120
140
160
180
200

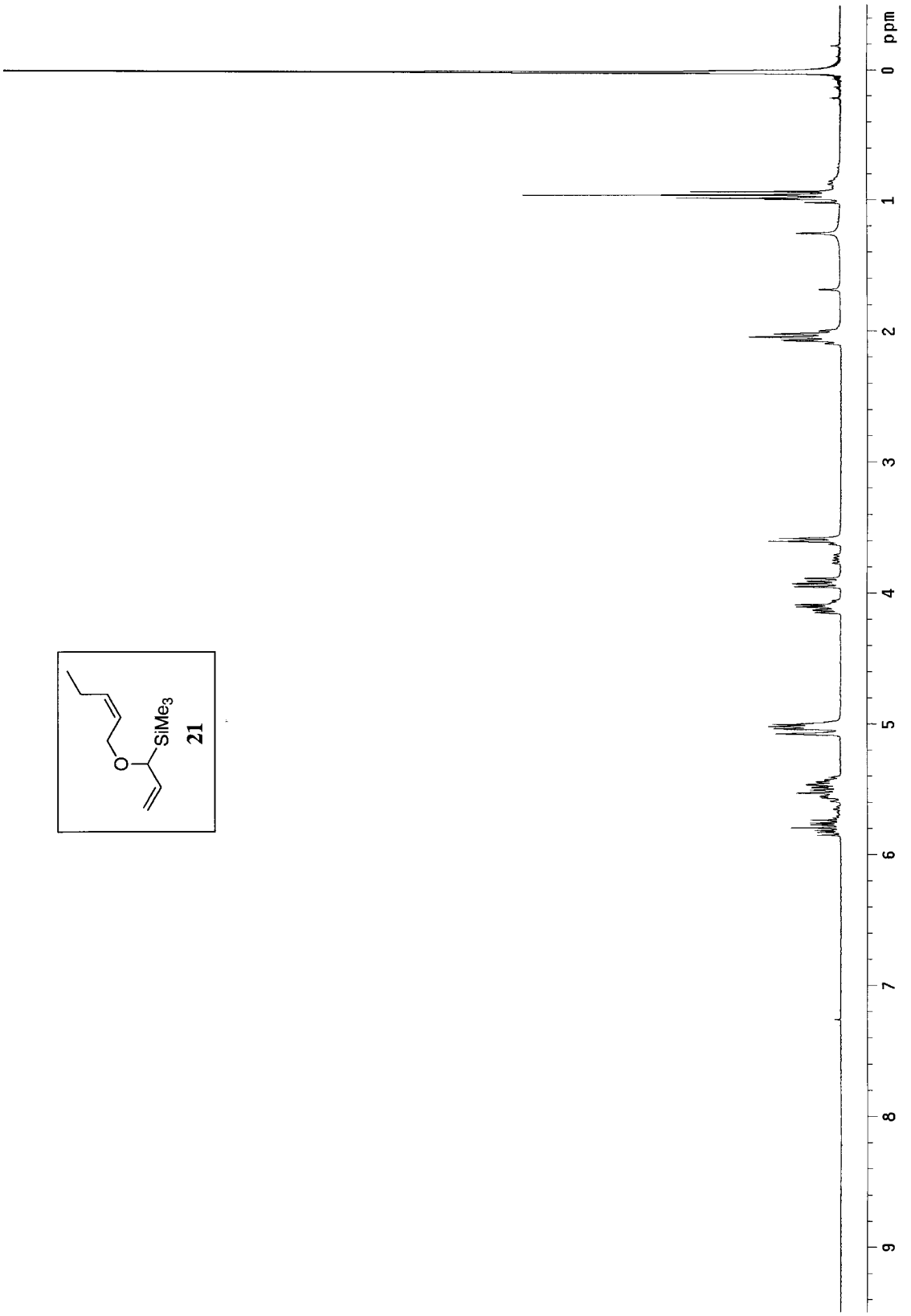


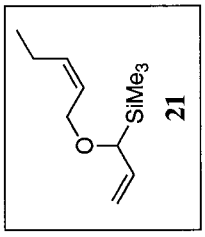
19

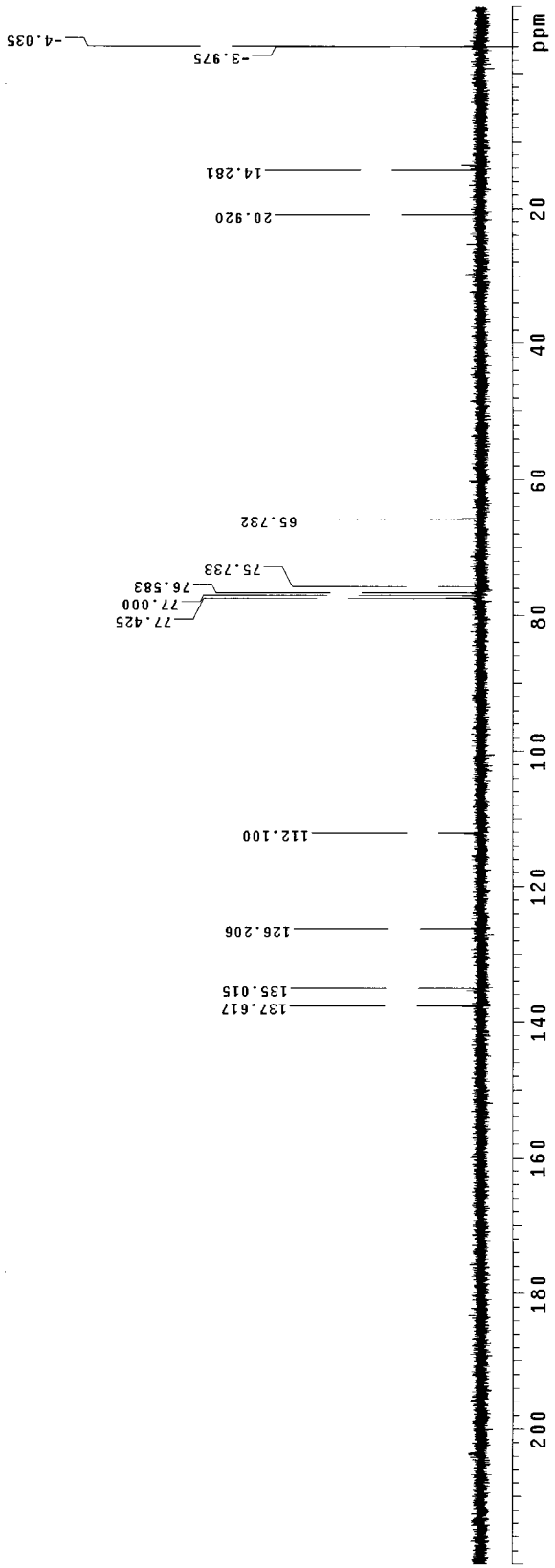


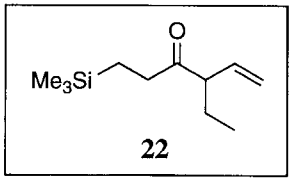
19




SiMe3
21
ppm
0
1
2
3
4
5
6
7
8
9

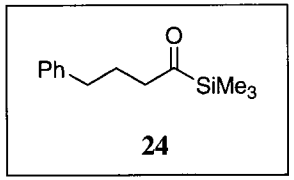

SiMe3
21


-4.035
-3.975
14.281
20.920
65.732
75.733
76.583
77.000
77.425
112.100
126.206
135.015
137.617
ppm
20
40
60
80
100
120
140
160
180
200

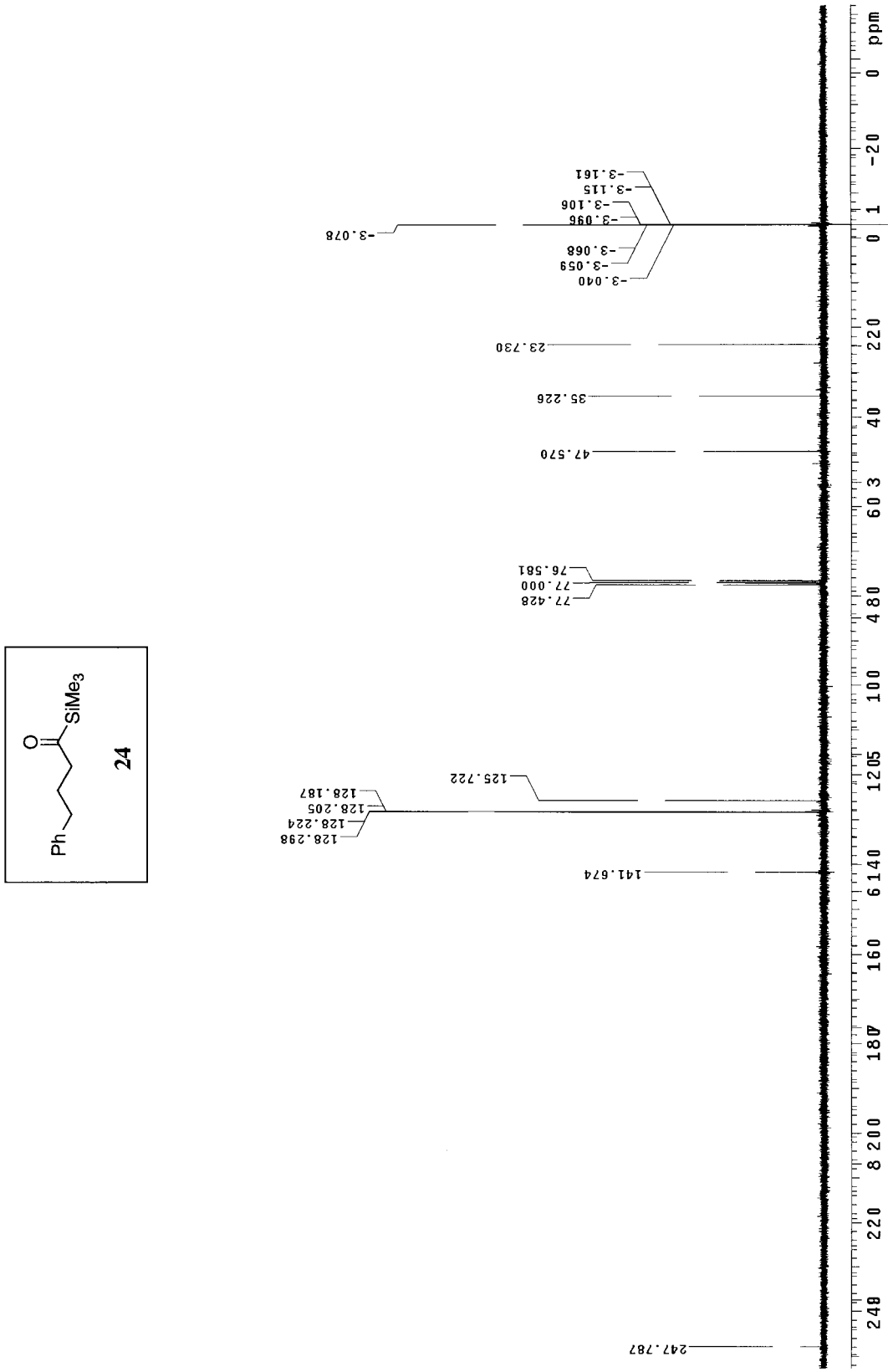


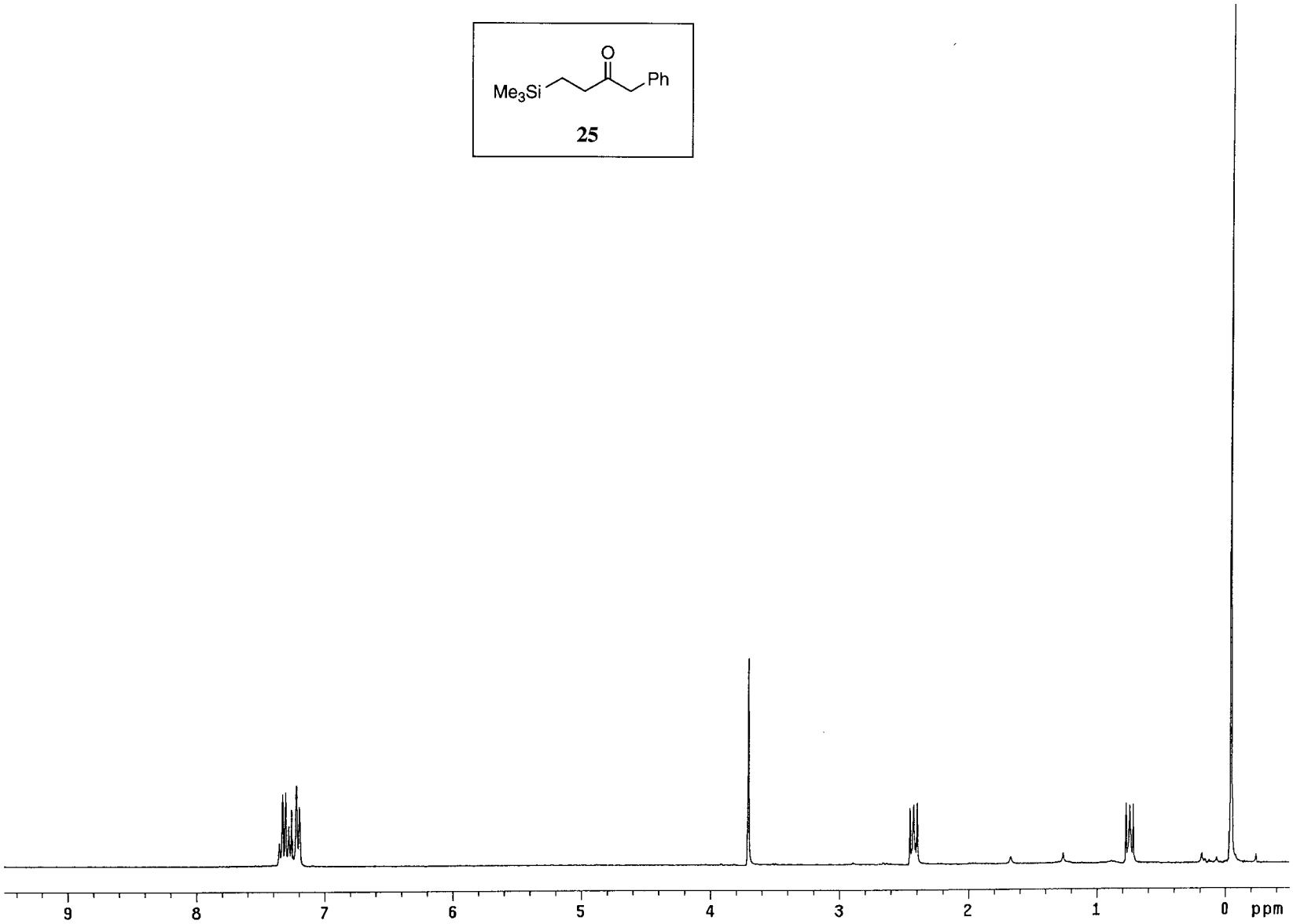
22

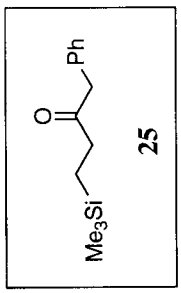
211.743
136.530
117.481
77.428
77.000
76.581
58.899
36.129
24.260
11.730
9.898
-1.785
ppm
200
180
160
140
120
100
80
60
40
20
O
Me3Si
22

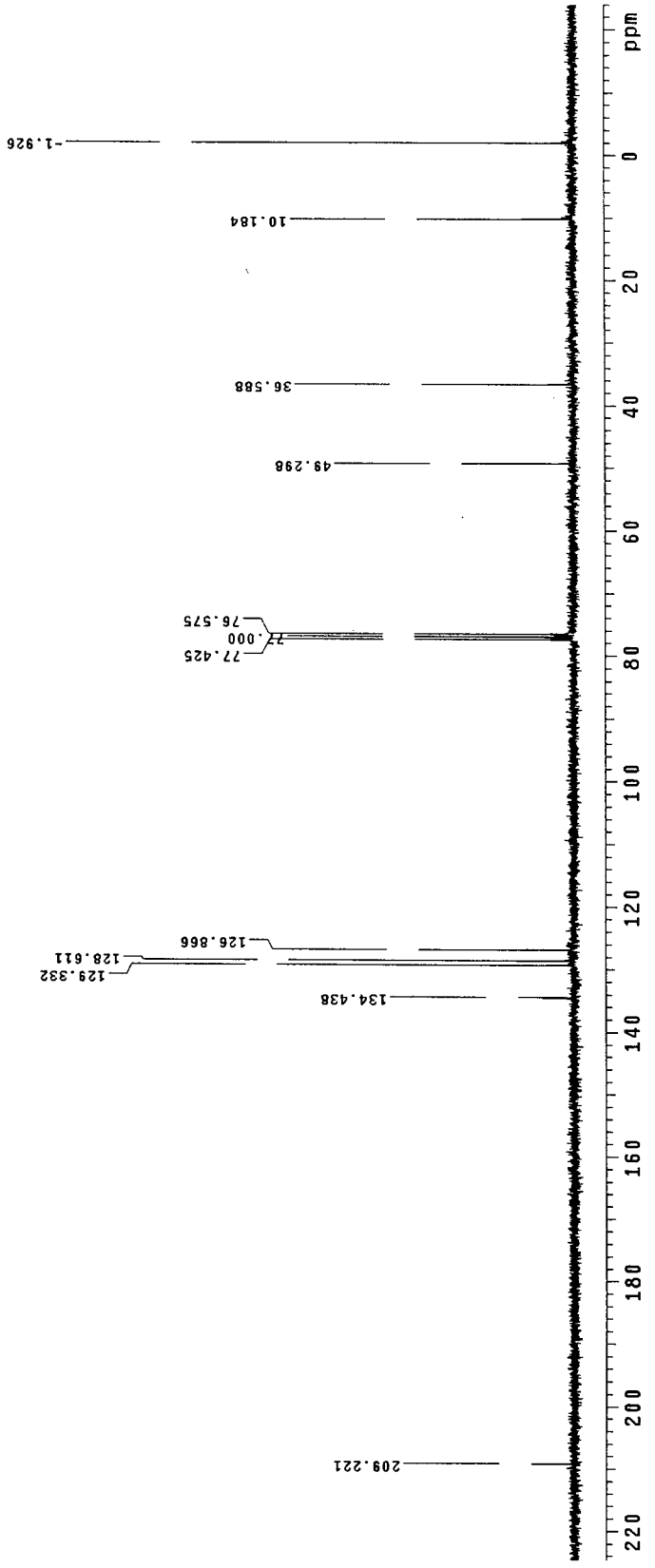


24


Ph
O
SiMe3
24
247.787
141.674
128.298
128.224
128.205
128.187
125.722
77.428
77.000
76.581
47.570
35.226
23.730
-3.040
-3.059
-3.068
-3.078
-3.096
-3.106
-3.115
-3.161
ppm


O
Me3Si
Ph
25
9
8
7
6
5
4
3
2
1
0
ppm


Ph
O
Me3Si
25


-1.926
10.184
36.588
49.298
76.575
77.000
77.425
126.866
128.611
129.332
134.438
209.221
ppm
0
20
40
60
80
100
120
140
160
180
200
220