Gold-Catalysed Alkenyl- and Arylsilylation Reactions Forming 1-Silaindenes

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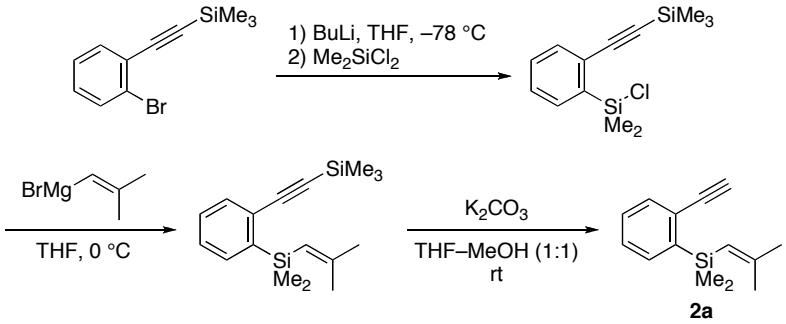
Supporting Information

General. All manipulations were carried out in a nitrogen-filled gloved box and with standard Schlenk techniques under an argon atmosphere. Column chromatography was performed with silica gel 60N (Kanto). Preparative thin-layer chromatography was performed with silica gel 60 PF_{254} (Merck). Gel permeation chromatography (GPC) was carried out on a JAI LC-908. Proton chemical shifts were referenced to the residual $CHCl_3$ signal at 7.26 ppm. Carbon chemical shifts were referenced to the central peak of $CDCl_3$ at 77.0 ppm.

Materials. [(2-bromophenyl)ethynyl]trimethylsilane,[1] 1-bromo-2-(2-methylprop-1-enyl)-benzene, [2] 1,4-dibromo-2,5-bis[(trimethylsilyl)ethynyl]benzene [3] and gold complexes ($R_3PAuNTf_2$)[4] were prepared according to the literature methods. 1-Bromocyclopentene was prepared by dehydrobromination of *trans*-1,2-dibromocyclopentene with 4 equiv of *t*-BuOK in refluxing THF. All other commercially available chemical resources were used as received without further purification.

(1) Z. U. Levi, and T. D. Tilley, *J. Am. Chem. Soc.*, 2009, **131**, 2796.
(2) M.-Y. Lin, A. Das and R.-S. Liu, *J. Am. Chem. Soc.*, 2006, **128**, 9340.
(3) K. Takamiya, Y. Konda, H. Ebata, N. Niihara and T. Otsubo, *J. Org. Chem.*, 2005, **70**, 10569.
(4) N. Mézailles, L. Ricard and F. Gagosz, *Org. Lett.*, 2005, **7**, 4133.

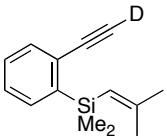
General Procedure for Preparation of Alkenyl- and Arylsilanes 2



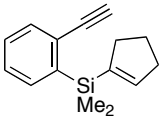
2a

(2-Ethynylphenyl)dimethyl(2-methylprop-1-enyl)silane (2a). To a solution of [(2-bromophenyl)ethynyl]trimethylsilane (5.32 g, 21.0 mmol) in THF (30.0 mL) was added dropwise *n*-butyllithium (1.55 M hexane solution, 15 mL) at –78 °C, and the mixture was stirred at –78 °C for 1 h. The resulting {2-[(trimethylsilyl)ethynyl]phenyl}lithium was added dropwise via cannula to a solution of dichlorodimethylsilane (7.6 mL, 63.6 mmol) in THF (30 mL) at –78 °C, and the reaction mixture was gradually warmed to room temperature over 1 h. The volatile materials were removed under reduced pressure, and the residue was filtered (Celite, hexane). The filtrate was evaporated to give crude chlorodimethyl{2-[(trimethylsilyl)ethynyl]phenyl}silane, which was used for next step without further purification.

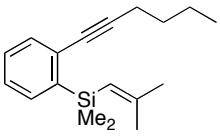
To a solution of isobutenylmagnesium bromomide in THF (0.5 M, 14 mL, 7 mmol) was added a solution of chlorodimethyl{2-[(trimethylsilyl)ethynyl]phenyl}silane (1.86 g, 6.97 mmol) in THF (5 mL) at 0 °C. The mixture was stirred at room temperature for 1 h, quenched by sat. NH_4Cl aq solution, and extracted with Et_2O. The extract was dried over $MgSO_4$, concentrated, and passed through silica gel (hexane). To a solution of the residue in THF–MeOH (1:1, 20 mL) was added potassium carbonate (108 mg, 0.78 mmol) at room temperature. The mixture was stirred at room temperature for 14 h, quenched by saturated NH_4Cl aqueous solution, and extracted with hexane–EtOAc (2:1). The extract was dried over $MgSO_4$ and concentrated. The residue was purified by column chromatography on silica gel (hexane). Further purification by GPC gave **2a** (763 mg, 3.56 mmol, 51%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.46 (s, 6H), 1.70 (s, 3H), 1.90 (s, 3H), 3.20 (s, 1H), 5.40 (s, 1H), 7.28-7.31 (m, 2H), 7.50-7.57 (m, 2H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –1.0, 23.5, 29.5, 80.2, 85.1, 122.1, 127.3, 127.8, 128.5, 133.3, 134.5, 142.6, 152.9; HRMS (EI) calcd for $C_{14}H_{18}Si$ $[M]^+$ 214.1178, found 214.1175.



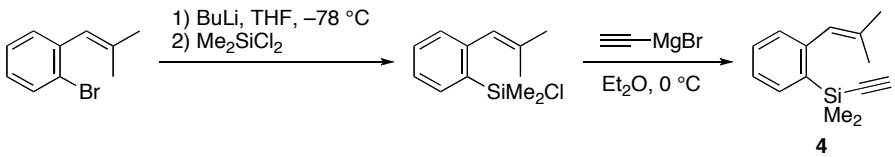
[2-(2-Deuterioethynyl)phenyl]dimethyl(2-methylprop-1-enyl)silane (2a-*d*). The title compound was prepared by deprotonation of **2a** with LDA followed by treatment with D_2O. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.46 (s, 6H), 1.70 (s, 3H), 1.90 (s, 3H), 3.20 (s, 1H, 83% D), 5.40 (s, 1H), 7.28-7.31 (m, 2H), 7.50-7.57 (m, 2H).



Cyclopentenyl(2-ethynylphenyl)dimethylsilane (2b). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.46 (s, 6H), 1.85 (quint, J = 8.1 Hz, 2H), 2.42 (t, J = 7.5 Hz, 4H), 3.17 (s, 1H), 6.09-6.13 (m, 1H), 7.27-7.33 (m, 2H), 7.40-7.45 (m, 1H), 7.48-7.54 (m, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –2.5, 24.2, 35.1, 36.4, 80.1, 85.1, 127.4, 127.8, 128.6, 133.3, 134.6, 141.5, 141.9, 143.0; HRMS (EI) calcd for $C_{15}H_{18}Si$ $[M]^+$ 226.1178, found 226.1174.



[2-(Hex-1-ynyl)phenyl]dimethyl(2-methylprop-1-enyl)silane (2c). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.44 (s, 6H), 0.95 (t, J = 7.5 Hz, 3H), 1.41-1.65 (m, 4H), 1.70 (s, 3H), 1.89 (d, J = 1.2 Hz, 3H), 2.41 (t, J = 7.2 Hz, 2H), 5.40 (s, 1H), 7.19-7.28 (m, 2H), 7.38-7.43 (m, 1H), 7.50-7.52 (m, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –1.0, 13.7, 19.4, 22.2, 23.4, 29.5, 30.7, 82.1, 93.4, 122.6, 126.5, 128.5, 129.4, 132.3, 134.3, 141.6, 152.2; HRMS (EI) calcd for $C_{18}H_{26}Si$ $[M]^+$ 270.1804, found 270.1801.



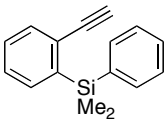
4

Ethynyldimethyl[2-(2-methylprop-1-enyl)phenyl]silane (4). [5] To a solution of 1-bromo-2-(2-methylprop-1-enyl)benzene (13.6 g, 64.5 mmol) in THF (60 mL) was added

(5) [1045858-14-7].

dropwise *n*-butyllithium (1.6 M hexane solution, 45 mL) at –78 °C, and the mixture was stirred at –78 °C for 1 h. The resulting [2-(2-methylprop-1-enyl)phenyl]lithium solution was added dropwise via cannula to a solution of dichlorodimethylsilane (21.7 mL, 178 mmol) in THF (30 mL) at –78 °C, and the reaction mixture was gradually warmed to room temperature. After evaporation of the volatile materials, the residue was distilled (90 °C/1 mmHg) to give chlorodimethyl[2-(2-methylprop-1-enyl)phenyl]silane.

To a solution of chlorodimethyl[2-(2-methylprop-1-enyl)phenyl]silane (3.71 g, 16.5 mmol) in Et_2O (17 mL) was added a solution of ethynylmagnesium bromide (0.5 M, 50 mL mmol) at 0 °C, and the mixture was stirred at room temperature for 5 h and quenched by sat. NH_4Cl aq solution, and extracted with hexane. The extract was dried over Na_2SO_4, concentrated, passed through silica gel (hexane), and concentrated. The residue was purified by column chromatography on silica gel (hexane:AcOEt = 100:1 then 50:1) to afford **4** (2.25 g, 64%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.42 (s, 6H), 1.65 (d, J = 1.2 Hz, 3H), 1.90 (d, J = 1.5 Hz, 3H), 2.52 (s, 1H), 6.52 (s, 1H), 7.12-7.18 (m, 1H), 7.18-7.26 (m, 1H), 7.32-7.39 (m, 1H), 7.74 (d, J = 1.2 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –0.5, 19.2, 25.8, 88.8, 94.6, 125.5, 126.5, 129.35, 129.43, 134.3, 134.6, 135.7 145.0; HRMS (EI) calcd for $C_{14}H_{18}Si$ $[M]^+$ 214.1178, found 214.1175.

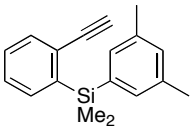


(2-Ethynylphenyl)dimethyl(phenyl)silane (2d).[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.67 (s, 6H), 3.12 (s, 1H), 7.24-7.40 (m, 6H), 7.50-7.58 (m, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –2.2, 80.7, 84.9, 127.6, 127.8, 129.0, 133.3, 134.3, 135.1, 137.8, 140.8; HRMS (EI) calcd for $C_{16}H_{16}Si$ $[M]^+$ 236.1021, found 236.1021.

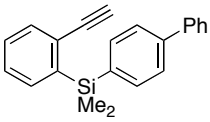
(2-Ethynylphenyl)dimethyl(2-methylphenyl)silane (2e). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.69 (s, 6H), 2.25 (s, 3H), 3.03 (s, 1H), 7.13-7.22 (m, 2H), 7.24-7.42 (m, 4H), 7.48-7.56 (m, 2H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –1.3, 23.2, 80.6, 84.6, 124.8, 127.4, 127.9, 128.8, 129.4,

(6) [142215-43-9]: T. Masuda, S. Katahira, K. Tsuchihara and T. Higashimura, *Polym. J.*, 1992, **24**, 491.

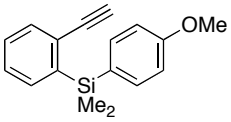
129.7, 133.3, 134.7, 135.3, 136.0, 141.3, 143.7; HRMS (EI) calcd for $C_{17}H_{18}Si$ $[M]^+$ 250.1178, found 250.1178.



(3,5-Dimethylphenyl)(2-ethynylphenyl)dimethylsilane (2f). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.65 (s, 6H), 2.32 (s, 6H), 3.15 (s, 1H), 7.03 (s, 1H), 7.17 (s, 2H), 7.23-7.35 (m, 3H), 7.50-7.54 (m, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –2.1, 21.4, 80.7, 85.1, 127.6, 127.8, 128.8, 130.8, 132.1, 133.3, 135.2, 136.8, 137.4, 141.1; HRMS (EI) calcd for $C_{18}H_{20}Si$ $[M]^+$ 264.1334, found 264.1336.



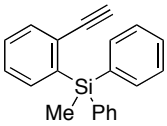
Biphenyl-4-yl(2-ethynylphenyl)dimethylsilane (2g). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.70 (s, 6H), 3.15 (s, 1H), 7.28-7.66 (m, 13H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –2.1, 80.8, 84.9, 126.3, 127.1, 127.3, 127.6, 127.9, 128.7, 129.0, 133.4, 134.8, 135.1, 136.5, 140.8, 141.0, 141.6; HRMS (EI) calcd for $C_{22}H_{20}Si$ $[M]^+$ 312.1334, found 312.1334.



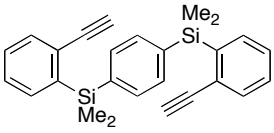
(2-Ethynylphenyl)(4-methoxyphenyl)dimethylsilane (2h). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.65 (s, 6H), 3.13 (s, 1H), 3.82 (s, 3H), 6.90-6.94 (m, 2H), 7.24-7.37 (m, 3H), 7.46-7.54 (m, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –2.1, 54.9, 80.6, 85.0, 113.4, 127.5, 127.8, 128.5, 128.8, 133.3, 135.1, 135.7, 141.3, 160.3; HRMS (EI) calcd for $C_{17}H_{18}OSi$ $[M]^+$ 266.1127, found 266.1124.

(2-Ethynylphenyl)dimethyl[4-(trifluoromethyl)phenyl]silane (2i). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.68 (s, 6H), 3.08 (s, 1H), 7.28-7.41 (m, 3H), 7.51-7.67 (m, 5H); ^{13}C NMR (75.5

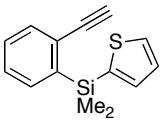
MHz, $CDCl_3$) δ –2.3, 81.0, 84.7, 124.1 (q, $^3J_{C-F}$ = 3.8 Hz), 124.3 (q, $^1J_{C-F}$ = 271.4 Hz), 127.8, 128.1, 129.4, 130.9 (q, $^2J_{C-F}$ = 31.3 Hz), 133.5, 134.6, 135.0, 139.7, 143.1; HRMS (EI) calcd for $C_{17}H_{15}F_3Si$ $[M]^+$ 304.0895, found 304.0896.



(2-Ethynylphenyl)(methyl)diphenylsilane (2j). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (s, 3H), 2.91 (s, 1H), 7.23-7.44 (m, 10H), 7.48-7.57 (m, 4H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.2, 81.2, 84.6, 127.7, 127.8, 128.3, 129.2, 129.3, 133.4, 135.3, 135.9, 136.7, 138.9; HRMS (EI) calcd for $C_{21}H_{18}Si$ $[M]^+$ 298.1178, found 298.1181.

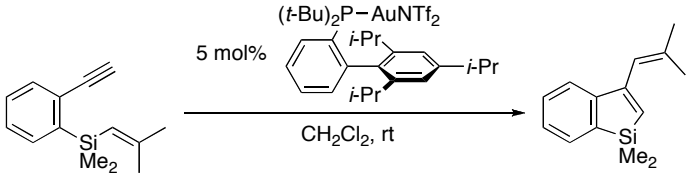


1,4-Bis[(2-ethynylphenyl)dimethylsilyl]benzene (2k). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.67 (s, 12H), 3.12 (s, 2H), 7.24-7.39 (m, 8H), 7.51-7.55 (m, 4H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –2.2, 80.7, 85.0, 127.6, 127.8, 128.9, 133.4, 133.5, 135.1, 138.5, 140.8; HRMS (EI) calcd for $C_{26}H_{26}Si_2$ $[M]^+$ 394.1573, found 394.1570.



(2-Ethynylphenyl)dimethyl(thiophen-2-yl)silane (2l). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.73 (s, 6H), 3.20 (s, 1H), 7.21-7.38 (m, 5H), 7.53 (d, J = 7.5 Hz, 1H), 7.66 (d, J = 4.5 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –1.0, 80.9, 84.8, 127.3, 127.9, 128.1, 129.1, 131.1, 133.3, 134.9, 135.6, 137.2, 140.6; HRMS (EI) calcd for $C_{14}H_{14}SSi$ $[M]^+$ 242.0585, found 242.0586.

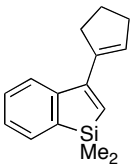
General Procedure for Intramolecular Alkenyl- and Arylsilylation.



2a **3a**

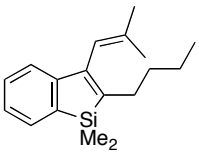
1,1-Dimethyl-3-(2-methylprop-1-enyl)-1*H*-benzo[*b*]silole (3a). To a Schlenk tube containing gold(I) complex **1** (4.8 mg, 5.3 μmol, 5 mol %) was added a solution of (2-ethynylphenyl)dimethyl(2-methylprop-1-enyl)silane (**2a**, 21.62 mg, 0.101 mmol) in dichloromethane (1 mL), and the mixture was stirred at room temperature for 2 h. The reaction mixture was passed through a column of Florisil® (hexane:AcOEt = 50:1). After removal of the volatile materials, the residue was subjected to preparative thin-layer chromatography on silica gel (hexane) to afford **3a** (15.77 mg, 73%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.35 (s, 6H), 1.85 (s, 3H), 1.96 (s, 3H), 5.99 (s, 1H), 6.20 (d, J = 1.0 Hz, 1H), 7.22-7.28 (m, 1H), 7.33-7.38 (m, 2H), 7.56 (d, J = 7.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ –3.9, 20.1, 26.5, 122.0, 122.2, 126.6, 129.1, 129.4, 131.5, 138.3, 139.4, 150.1, 156.0; HRMS (EI) calcd for $C_{14}H_{18}Si$ $[M]^+$ 214.1178, found 214.1180.

2-Deuterio-1,1-dimethyl-3-(2-methylprop-1-enyl)-1*H*-benzo[*b*]silole (3a-*d*). According to the general procedure, **3a**-*d* (15.61 mg, 72%) was obtained from **2a**-*d* (21.65 mg, 0.101 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.33 (s, 6H), 1.83 (d, J = 1.2 Hz, 3H), 1.94 (d, J = 1.2 Hz, 3H), 5.80 (d, 82% D), 6.18 (quint, J = 1.5 Hz, 1H), 7.20-7.27 (m, 1H), 7.32-7.35 (m, 2H), 7.54 (dt, J = 4.5, 1.2 Hz, 1H).



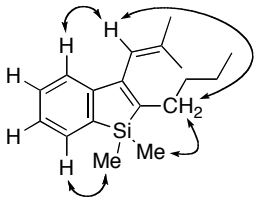
3-Cyclopentenyl-1,1-dimethyl-1*H*-benzo[*b*]silole (3b). According to the general procedure, **3c** (11.30 mg, 50%) was obtained from **2c** (22.75 mg, 0.100 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.25 (s, 6H), 1.82-1.91 (m, 2H), 2.40-2.47 (m, 2H), 2.52-2.58 (m, 2H), 5.73

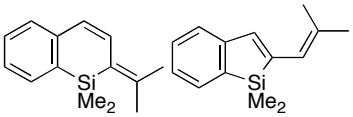
(s, 1H), 6.56 (dt, J = 1.5, 4.4 Hz, 1H), 7.22-7.29 (m, 1H), 7.31-7.38 (m, 1H), 7.51 (dd, J = 7.1, 2.0 Hz, 1H), 7.68 (d, J = 7.8 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –1.0, 23.7, 27.3, 38.2, 121.6, 124.2, 125.8, 128.8, 130.5, 133.0, 134.0, 135.7, 143.5, 153.4; HRMS (EI) calcd for $C_{15}H_{18}Si$ $[M]^+$ 226.1178, found 226.1176.



2-Butyl-1,1-dimethyl-3-(2-methylprop-1-enyl)-1*H*-benzo[*b*]silole (3c). According to the general procedure, **3c** (29.24 mg, 44%) was obtained from **2c** (66.65 mg, 0.246 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.33 (s, 6H), 0.91 (t, J = 7.2 Hz, 3H), 1.25-1.50 (m, 4H), 1.52 (s, 3H), 1.93 (d, J = 1.2 Hz, 3H), 2.31 (t, J = 7.8 Hz, 2H), 5.87 (s, 1H), 7.12-7.18 (m, 2H), 7.25-7.32 (m, 1H), 7.46-7.50 (m, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.3, 14.0, 20.0, 23.0, 25.2, 30.4, 32.2, 120.6, 122.2, 125.6, 129.5, 131.1, 136.5, 137.8, 143.9, 149.3, 150.7; HRMS (EI) calcd for $C_{18}H_{26}Si$ $[M]^+$ 270.1804, found 270.1805.

The stereochemical assignment of **3c** was established by ^{1}H NOE experiments as shown below.





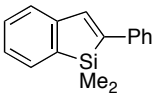
1,1-Dimethyl-2-isopropylidene-1,2-dihydrobenzo[*b*]siline (5)[7] and 1,1-dimethyl-2-(2-methylprop-1-enyl)-1*H*-benzo[*b*]silole (6).[8] Ethynyldimethyl[2-(2-methylprop-1-enyl)-phenyl]-silane (**4**, 62.60 mg, 0.292 mmol) was reacted in the presence of $(PPh_3)AuNTf_2$ (10.84 mg, 0.015 mmol, 5 mol%) in dichloromethane (1.5 mL) at room temperature for 4 h. The reaction mixture was passed through a column of Florisil® (hexane), and the volatile materials was evaporated. The residue was subjected to preparative thin-layer

(7) [1045858-16-9].
(8) [1045858-15-8].

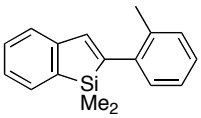
chromatography on silica gel (hexane) to afford a mixture of **5** and **6** (47.4 mg, 76%, 74:26). The two isomers were separated by GPC.

5: ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.42 (s, 6H), 1.96 (s, 3H), 2.06 (s, 3H), 6.20 (d, J = 10.8 Hz, 1H), 6.66 (d, J = 10.8 Hz, 1H), 7.10 (d, J = 7.5 Hz, 1H), 7.17 (t, J = 7.5 Hz, 1H), 7.23-7.31 (m, 1H), 7.48 (d, J = 6.9 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ 0.5, 20.5, 26.6, 126.2, 127.1, 127.6, 128.2, 128.7, 129.3, 133.6, 134.8, 141.4, 150.0; HRMS (EI) calcd for $C_{14}H_{18}Si$ $[M]^+$ 214.1178, found 214.1178.

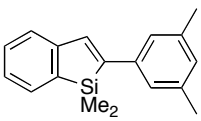
6: ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.44 (s, 6H), 1.87 (s, 3H), 1.92 (s, 3H), 6.20 (s, 1H), 7.01 (s, 1H), 7.14- 7.19 (m, 2H), 7.26-7.32 (m, 1H), 7.46 (d, J = 7.8 Hz, 1H); HRMS (EI) calcd for $C_{14}H_{18}Si$ $[M]^+$ 214.1178, found 214.1179.



1,1-Dimethyl-2-phenyl-1*H*-benzo[*b*]silole (7d).[9] According to the general procedure, **7d** (15.95 mg, 63%) was obtained from **2d** (25.22 mg, 0.107 mmol). The analytical data were identical to those reported in the literature.



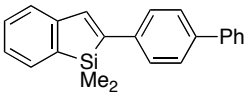
1,1-Dimethyl-2-(2-methylphenyl)-1*H*-benzo[*b*]silole (7e). According to the general procedure, **7e** (13.23 mg, 52%) was obtained from **2e** (25.33 mg, 0.101 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.40 (s, 6H), 2.35 (s, 3H), 7.07-7.31 (m, 7H), 7.33-7.40 (m, 1H), 7.57 (d, J = 7.2 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.4, 21.0, 124.1, 125.5, 126.1, 126.6, 127.9, 129.9, 130.3, 131.7, 134.9, 138.2, 140.7, 145.0, 147.9, 148.8; HRMS (EI) calcd for $C_{17}H_{18}Si$ $[M]^+$ 250.1178, found 250.1175.



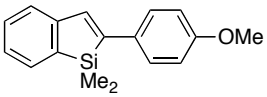
2-(3,5-Dimethylphenyl)-1,1-dimethyl-1*H*-benzo[*b*]silole (7f). According to the general

(9) [794512-47-3]: (*a*) C. Xu, A. Wakamiya and S. Yamaguchi, *Org. Lett.*, 2004, **6**, 3707; (*b*) L. Ilies, H. Tsuji, Y. Sato and E. Nakamura, *J. Am. Chem. Soc.*, 2008, **130**, 4240; (*c*) T. Matsuda, Y. Yamaguchi and M. Murakami, *Synlett*, 2008, 561.

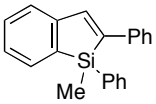
procedure, **7f** (17.13 mg, 64%) was obtained from **2f** (26.56 mg, 0.100 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.48 (s, 6H), 2.36 (s, 6H), 6.92 (s, 1H), 7.13 (s, 2H), 7.18-7.25 (m, 1H), 7.29-7.39 (m, 2H), 7.52-7.57 (m, 2H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.0, 21.4, 124.2, 124.4, 126.5, 128.9, 130.0, 131.6, 138.1, 138.4, 139.0, 141.0, 145.5, 149.0; HRMS (EI) calcd for $C_{18}H_{20}Si$ $[M]^+$ 264.1334, found 264.1337.



2-(Biphenyl-4-yl)-1,1-dimethyl-1*H*-benzo[*b*]silole (7g).[10] According to the general procedure, **7g** (13.23 mg, 42%) was obtained from **2g** (31.33 mg, 0.100 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.52 (s, 6H), 7.21-7.27 (m, 1H), 7.32-7.41 (m, 3H), 7.43-7.50 (m, 2H), 7.55-7.66 (m, 8H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.1, 124.3, 126.7, 126.86, 126.91, 127.2, 127.4, 128.8, 130.1, 131.7, 138.1, 138.3, 139.8, 140.7, 141.2, 144.8, 148.9; HRMS (EI) calcd for $C_{22}H_{20}Si$ $[M]^+$ 312.1334, found 312.1337.



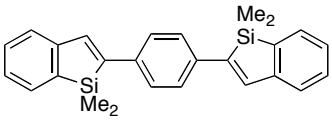
2-(4-Methoxyphenyl)-1,1-dimethyl-1*H*-benzo[*b*]silole (7h). According to the general procedure, **7h** (12.67 mg, 24%) was obtained from **2h** (53.59 mg, 0.201 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.47 (s, 6H), 3.84 (s, 3H), 6.90-6.96 (m, 2H), 7.16-7.23 (m, 1H), 7.26-7.38 (m, 2H), 7.43-7.49 (m, 3H), 7.53 (d, J = 6.6 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.1, 55.3, 114.2, 123.9, 126.2, 127.6, 130.0, 131.6, 131.8, 138.0, 139.1, 144.7, 149.2, 158.9; HRMS (EI) calcd for $C_{17}H_{18}OSi$ $[M]^+$ 266.1127, found 266.1129.



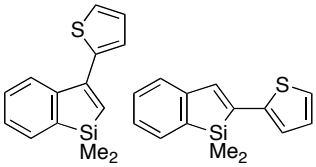
1-Methyl-1,2-diphenyl-1*H*-benzo[*b*]silole (7j).[11] According to the general procedure, **7j** (35.8 mg, 60%) was obtained from **2j** (59.6 mg, 0.20 mmol). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.79 (s, 3H), 7.18-7.63 (m, 14H), 7.69 (s, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –5.4, 124.4, 126.7, 126.9, 127.1, 128.1, 128.6, 129.9, 130.3, 132.3, 134.0, 134.5, 137.2, 138.7, 142.5,

(10) [1186331-75-8]: L. Ilies, H. Tsuji and E. Nakamura, *Org. Lett.* **2009**, *11*, 3966.
(11) [1027811-62-6]: T. Matsuda, Y. Yamaguchi and M. Murakami, *Synlett*, 2008, 561.

143.9, 149.3; HRMS (EI) calcd for $C_{21}H_{18}Si$ $[M]^+$ 298.1178, found 298.1180.



1,4-Bis(1,1-dimethyl-1*H*-benzo[*b*]silol-2-yl)benzene (7k).[12] The reaction of **2k** (78.9 mg, 0.20 mmol) was carried out in the presence of **1** (18.1 mg, 0.020 mmol, 10 mol %) in CH_2Cl_2 (2.0 mL) at 40 °C for 12 h. Preparative TLC (hexane:AcOEt = 50:1) followed by GPC afforded **7k** (17.4 mg, 22%). The analytical data were identical to those reported in the literature.



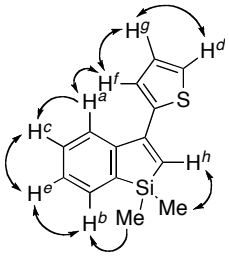
1,1-Dimethyl-3-(thiophen-2-yl)-1*H*-benzo[*b*]silole (3l) and 1,1-dimethyl-2-(thiophen-2-yl)- 1*H*-benzo[*b*]silole (7l). According to the general procedure, **3l** (24.29 mg, 50%) and **7l** (8.10 mg, 17%) were obtained from **2l** (48.30 mg, 0.199 mmol).

3l: ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.37 (s, 6H), 6.35 (s, 1H), 7.11 (dd, J = 5.4, 3.6 Hz, 1H), 7.26-7.33 (m, 3H), 7.37 (dt, J = 1.6, 7.4 Hz, 1H), 7.58-7.62 (m, 1H), 7.69 (d, J = 7.5 Hz, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –4.0, 123.2, 124.6, 125.4, 127.0, 127.1, 129.4, 131.9, 132.6, 140.1, 141.8, 147.8, 152.5; HRMS (EI) calcd for $C_{14}H_{14}SSi$ $[M]^+$ 242.0585, found 242.0585.

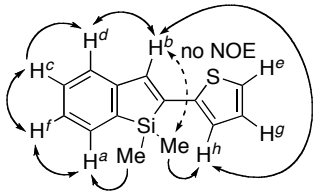
7l: ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.47 (s, 6H), 6.99-7.05 (m, 2H), 7.17-7.38 (m, 4H), 7.38 (s, 1H), 7.50-7.55 (m, 1H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ –3.3, 124.1, 124.3, 125.1, 126.5, 127.6, 130.1, 131.8, 137.5, 138.5, 139.8, 144.2, 148.8; HRMS (EI) calcd for $C_{14}H_{14}SSi$ $[M]^+$ 242.0585, found 242.0589.

(12) [794512-50-8]: (*a*) C. Xu, A. Wakamiya and S. Yamaguchi, *Org. Lett.*, 2004, **6**, 3707; (*b*) T. Matsuda, Y. Yamaguchi, N. Ishida and M. Murakami, *Synlett*, 2010, 2743.

The stereochemical assignment of **3l** and **7l** was established by ^{1}H NOE experiments as shown below.

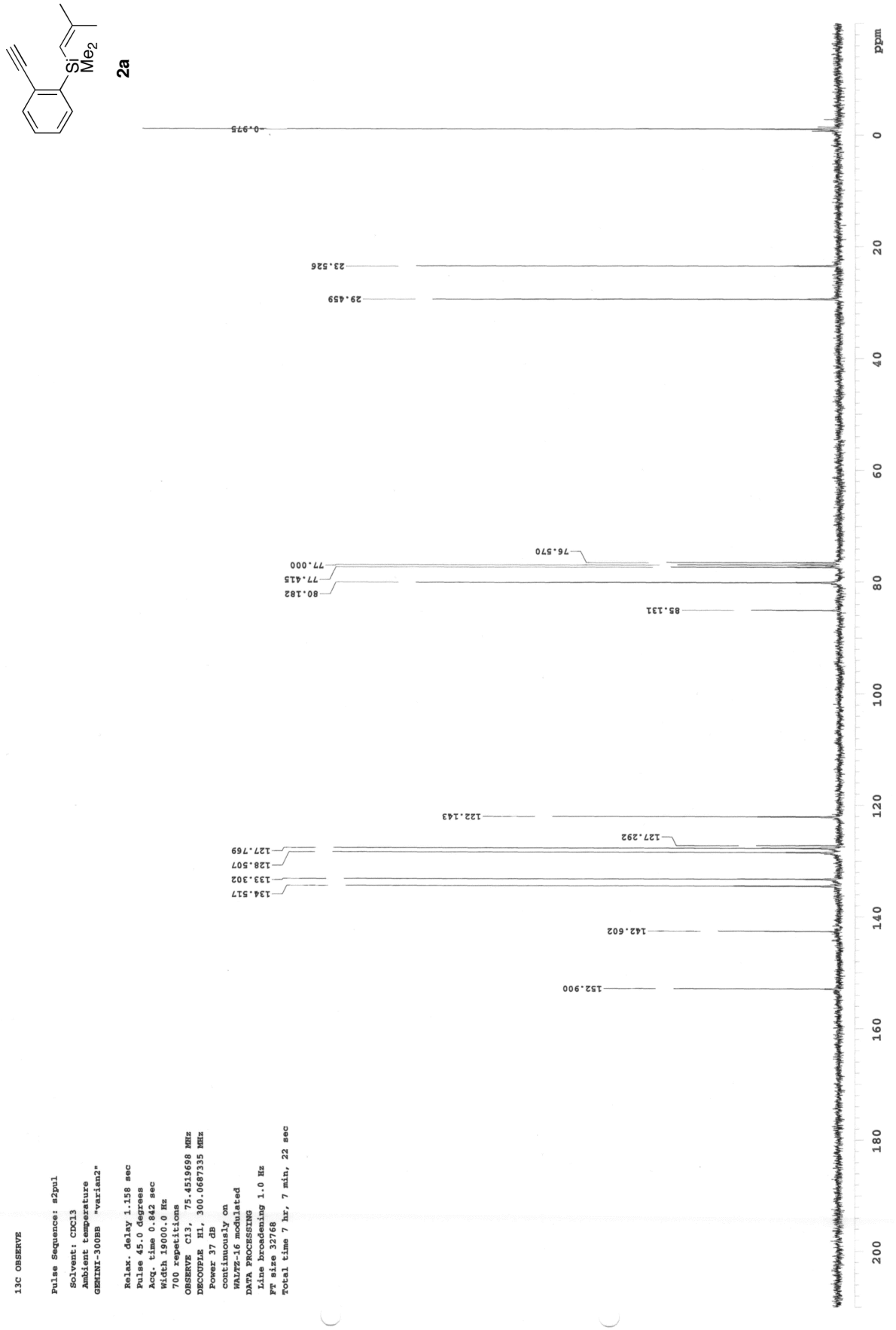


H^a (d): 7.68
H^b (d) : 7.59
H^c (dt): 7.36
H^d (dd): 7.31
H^e (d) : 7.29
H^f (dd): 7.27
H^g (dd): 7.10
H^h (s): 6.35

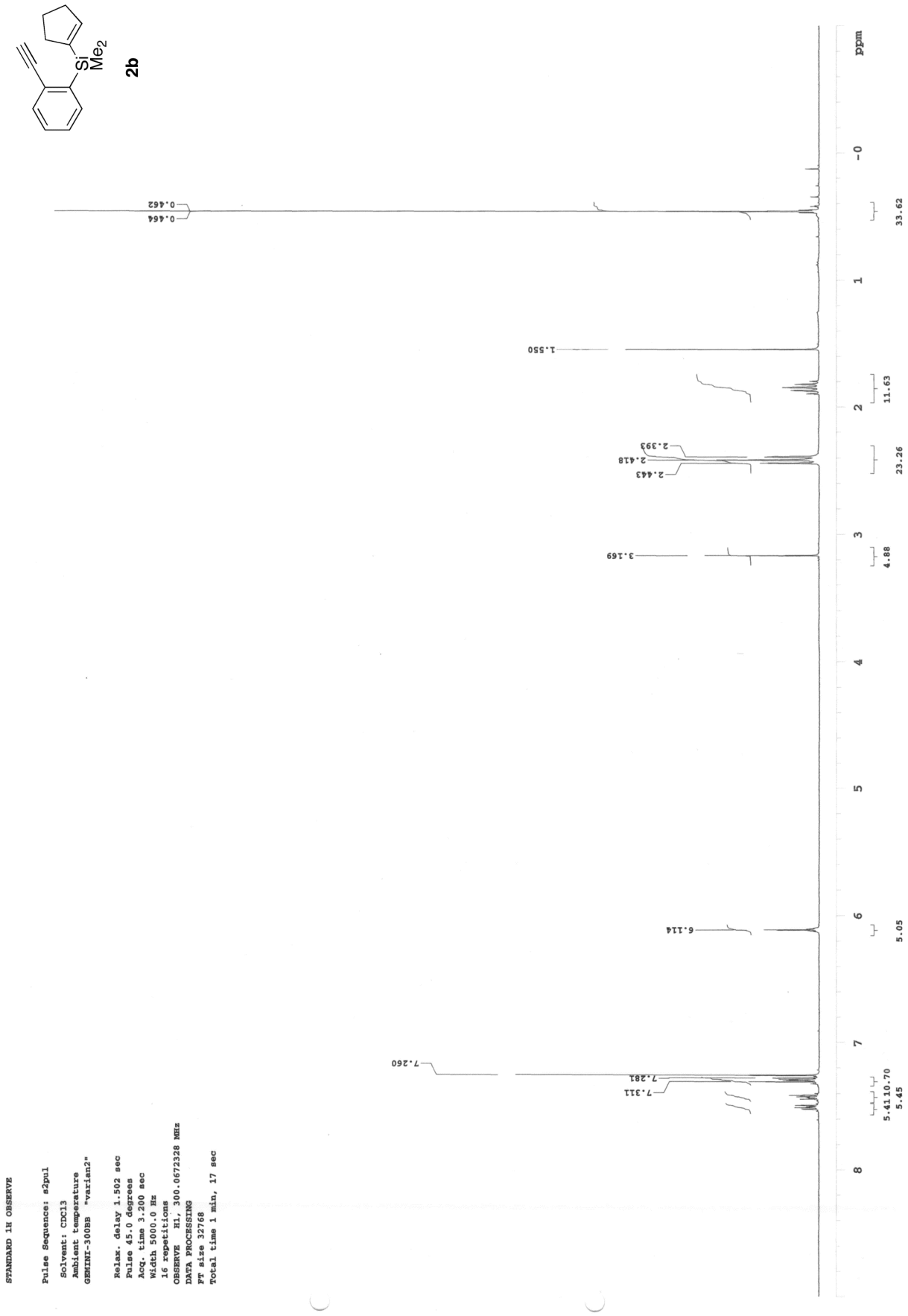


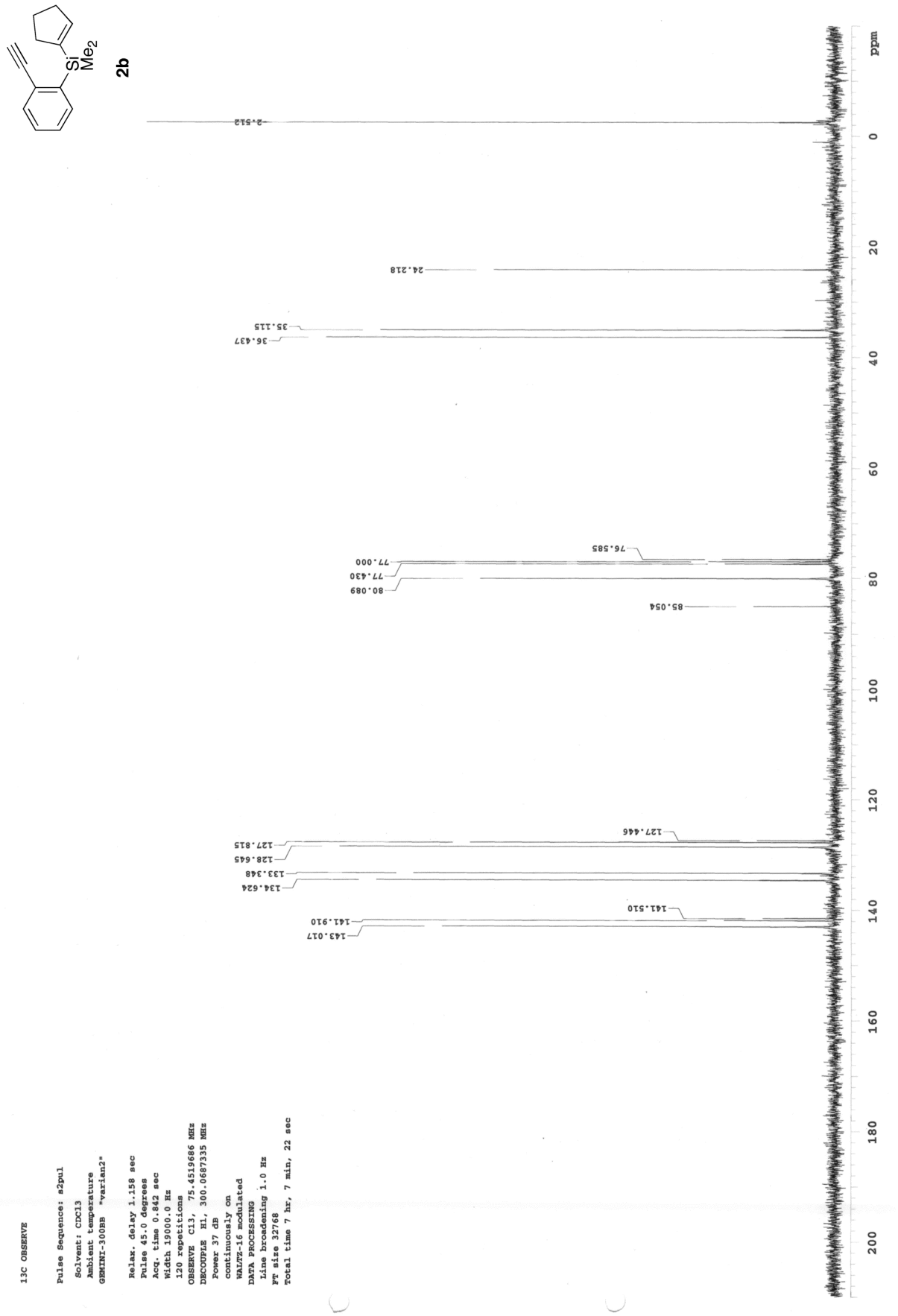
H^a (d): 7.53
H^b (s): 7.38
H^c (dt): 7.33
H^d (d): 7.27
H^e (dd): 7.22
H^f (dd): 7.19
H^g (dd): 7.03
H^h (dd): 7.00

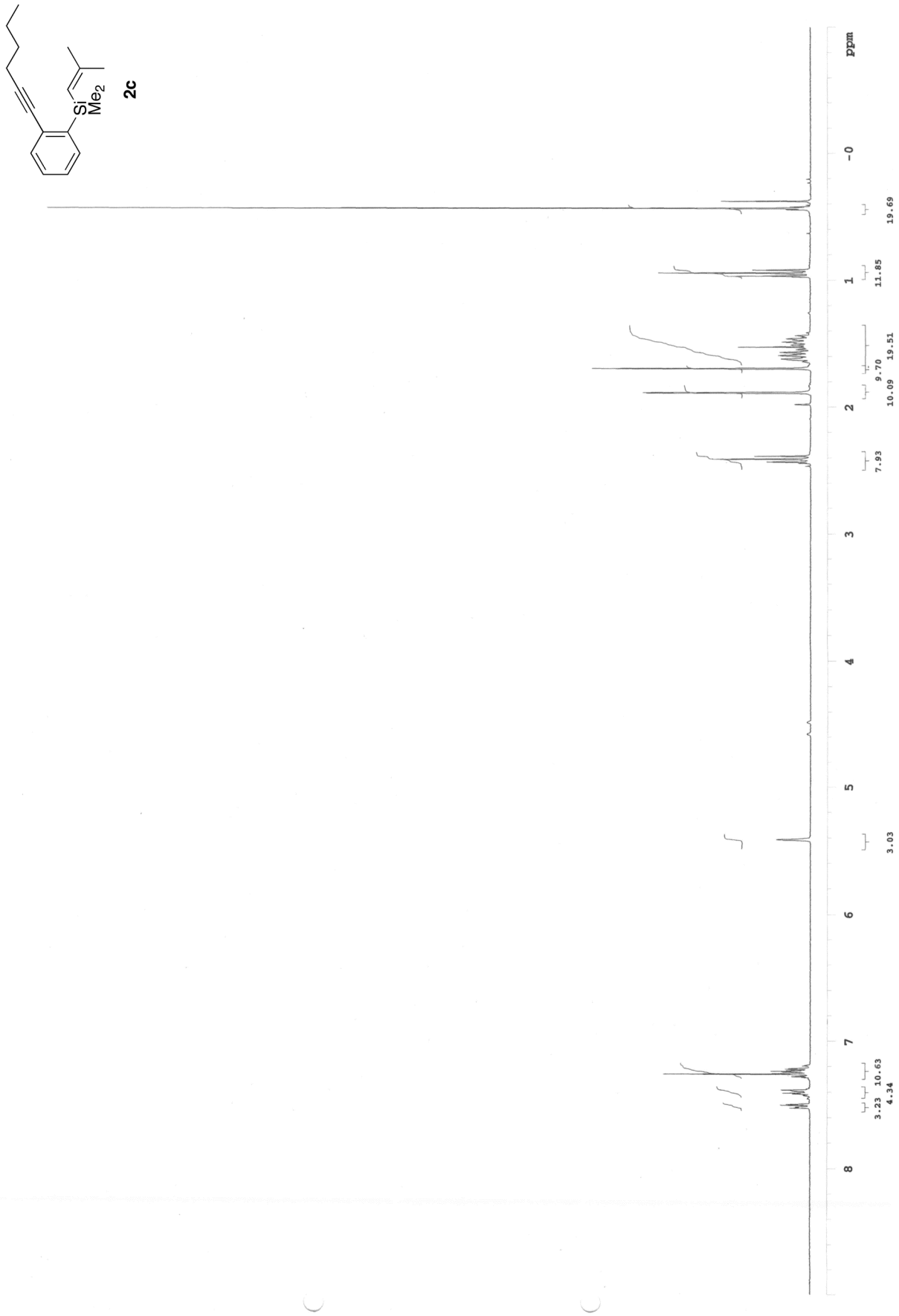

Si
Me2
2a
0.460
1.550
1.698
1.700
1.893
1.897
3.197
7.260
ppm
33.69
17.22
16.91
5.05
5.22
11.04
10.87
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


2a
Si
Me2
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
700 repetitions
OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec
-0.975
23.526
29.459
76.570
77.000
77.415
80.182
85.131
122.143
127.292
127.769
128.507
133.302
134.517
142.602
152.900
ppm
0
20
40
60
80
100
120
140
160
180
200


D
Si
Me2
2a-d
0.461
1.699
1.701
1.893
1.897
7.260
ppm
35.29
17.69
18.07
0.97
5.50
11.27
11.21
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


2b
Si
Me2
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.464
0.462
1.550
2.393
2.418
2.443
3.169
6.114
7.260
7.281
7.311
ppm
33.62
11.63
23.26
4.88
5.05
10.70
5.41
5.45


2b
Si
Me2
-2.512
24.218
35.115
36.437
76.585
77.000
77.430
80.089
85.054
127.446
127.815
128.645
133.348
134.624
141.510
141.910
143.017
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
120 repetitions
OBSERVE C13, 75.4519686 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec


Si
Me2
2c
ppm
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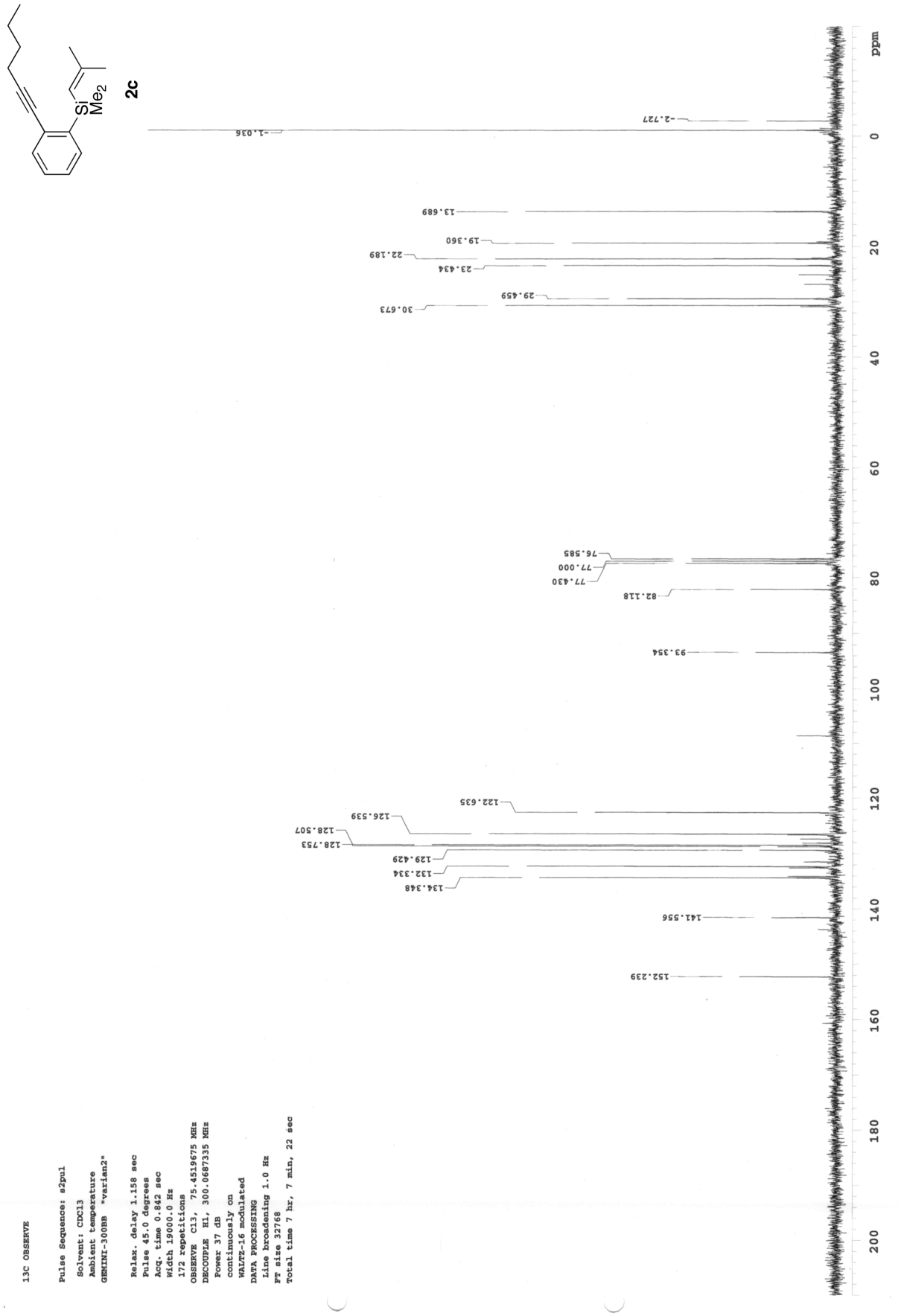
11.85
9.70
19.51
10.09
7.93
3.03
3.23
10.63
4.34


2c
Si
Me2
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13.689
19.360
22.189
23.434
29.459
30.673
76.585
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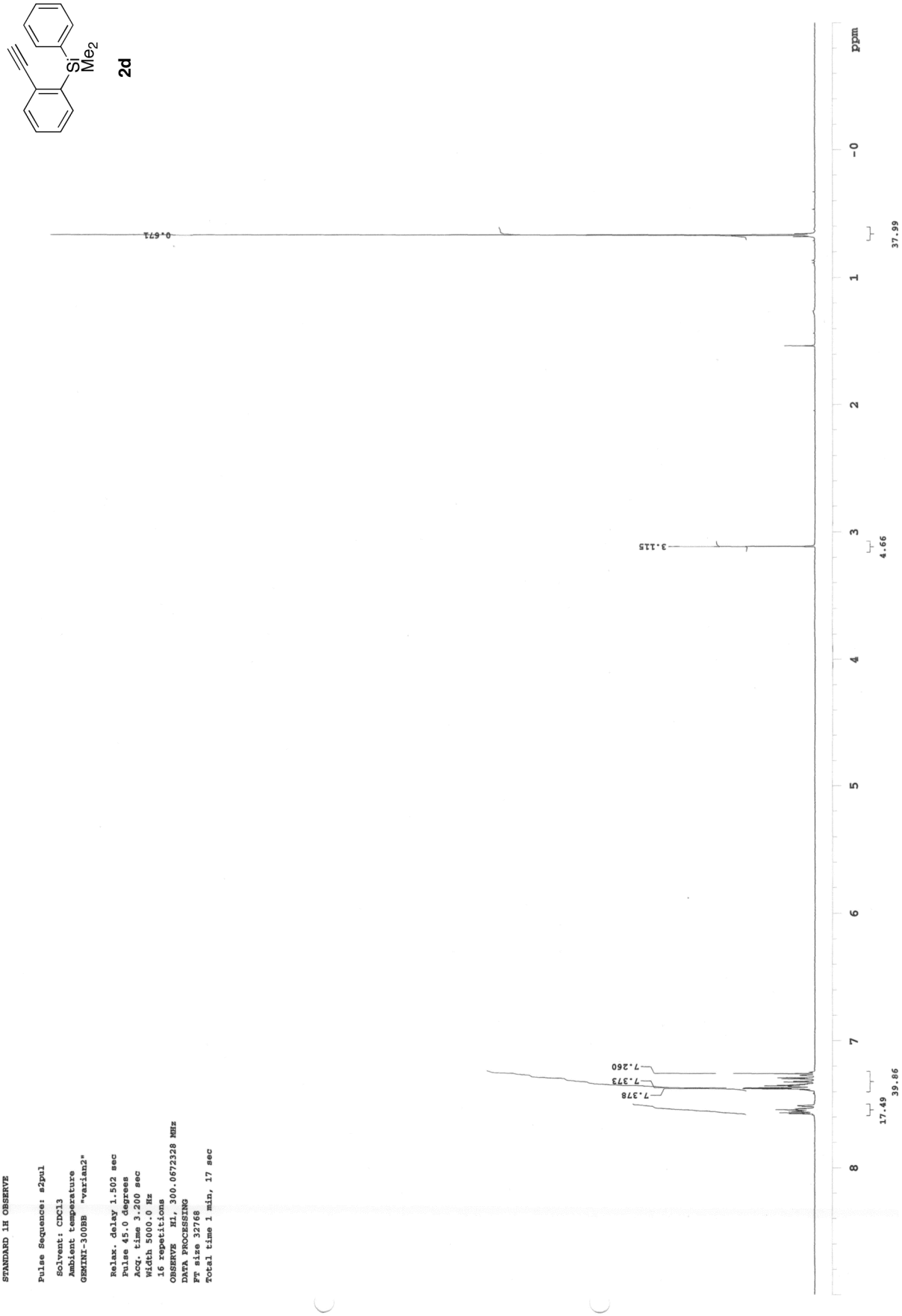
77.430
82.118
93.354
122.635
126.539
128.507
128.753
129.429
132.334
134.348
141.556
152.239
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
172 repetitions
OBSERVE C13, 75.4519675 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec


2d
Si
Me2
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
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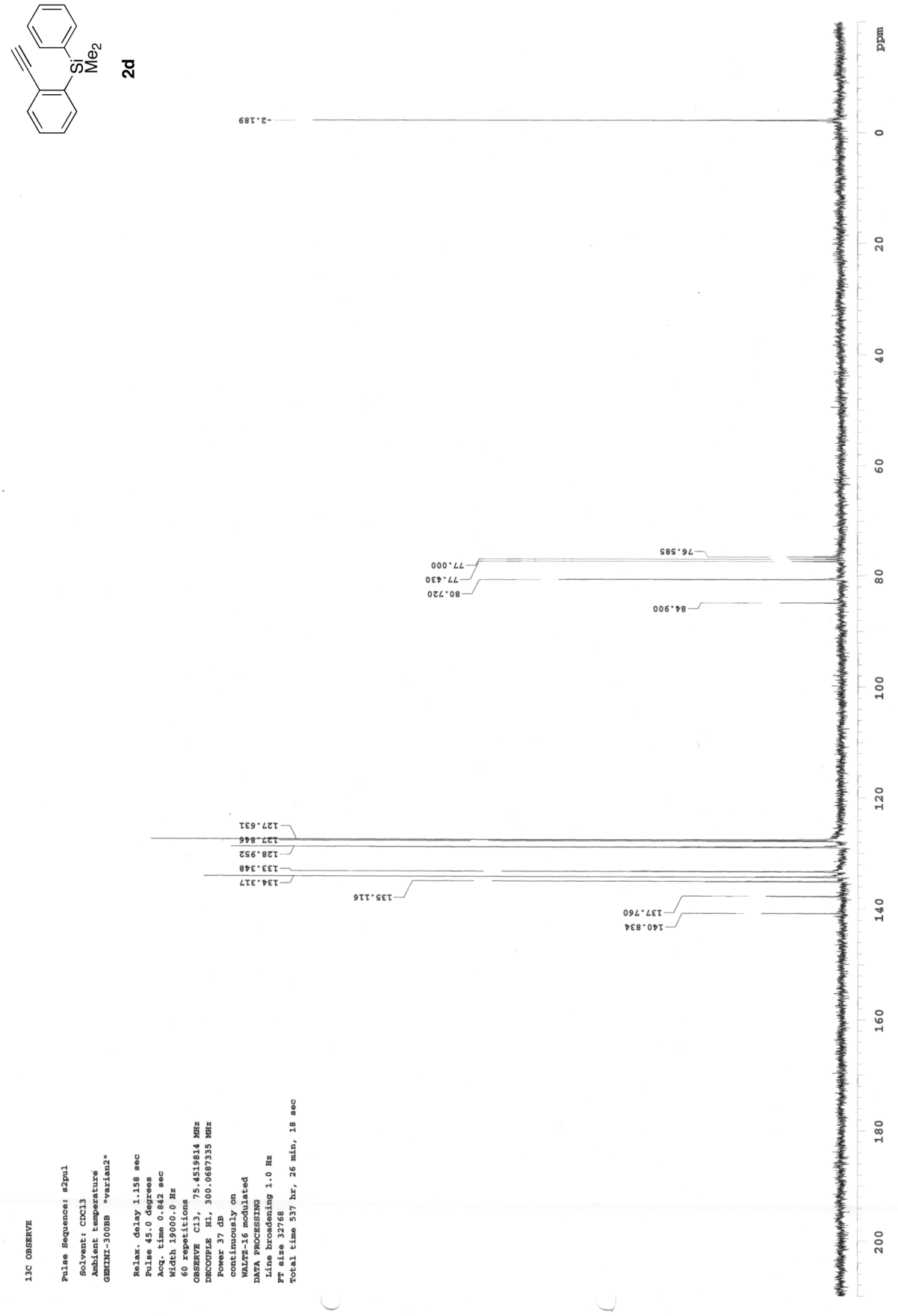
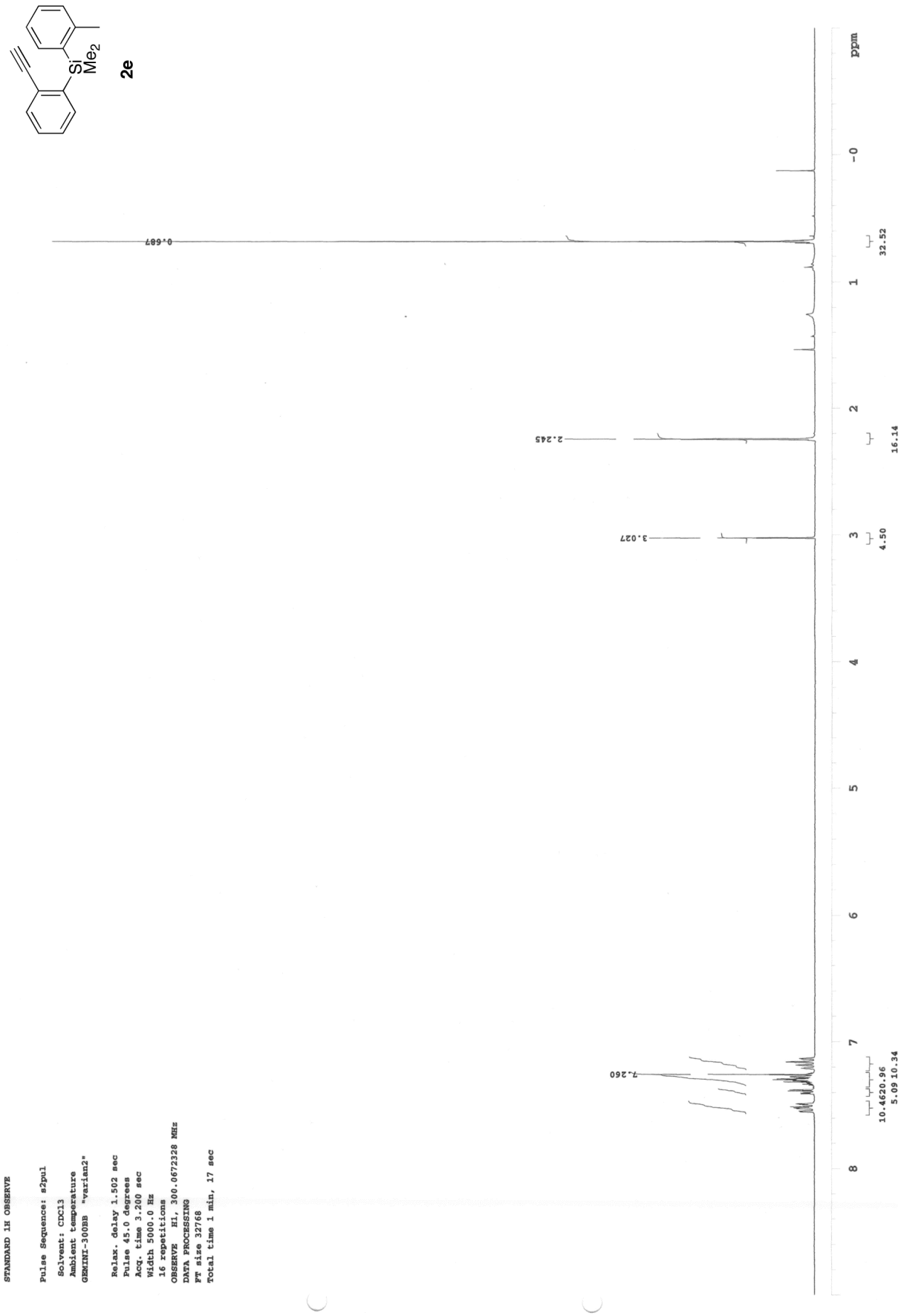
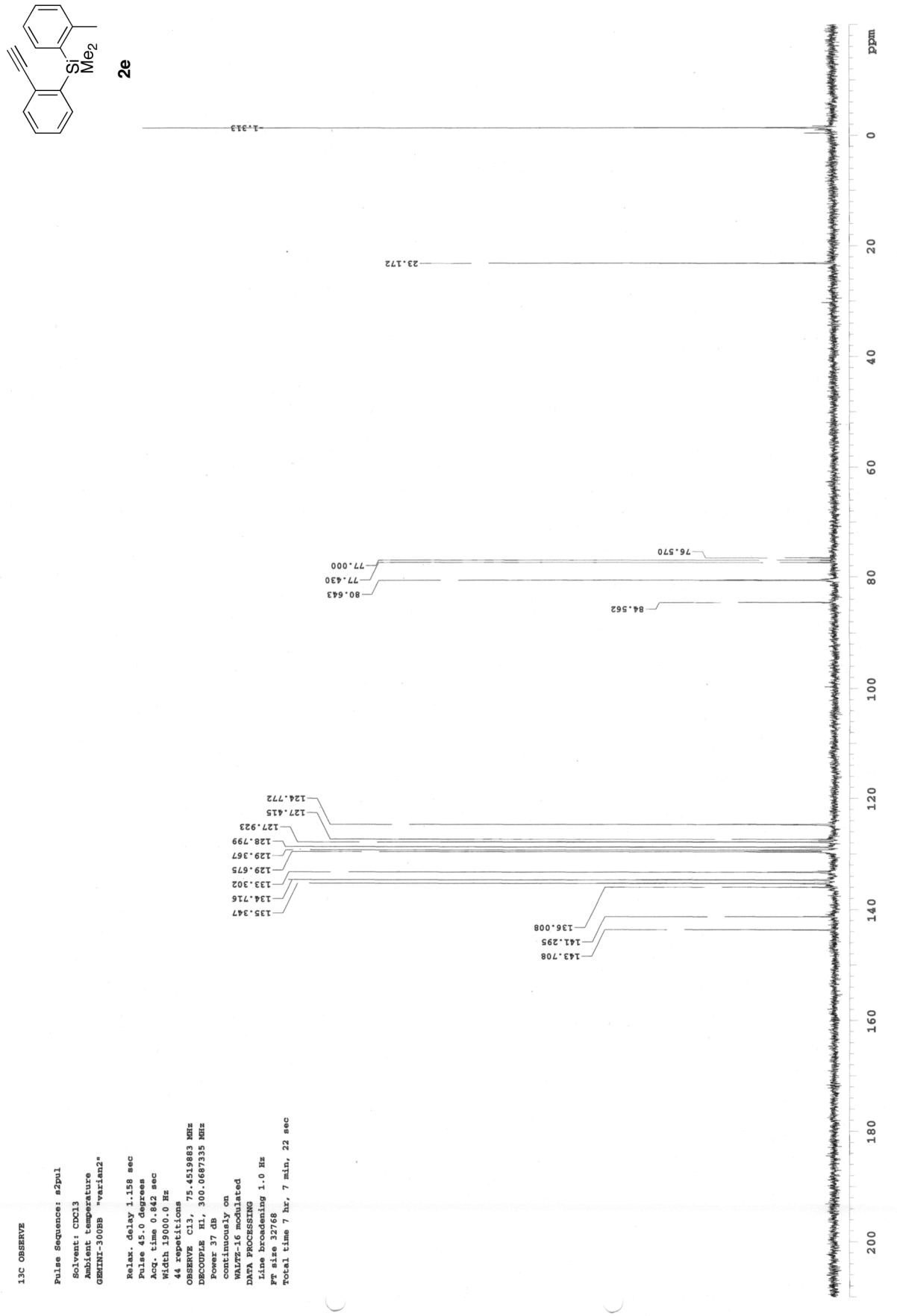
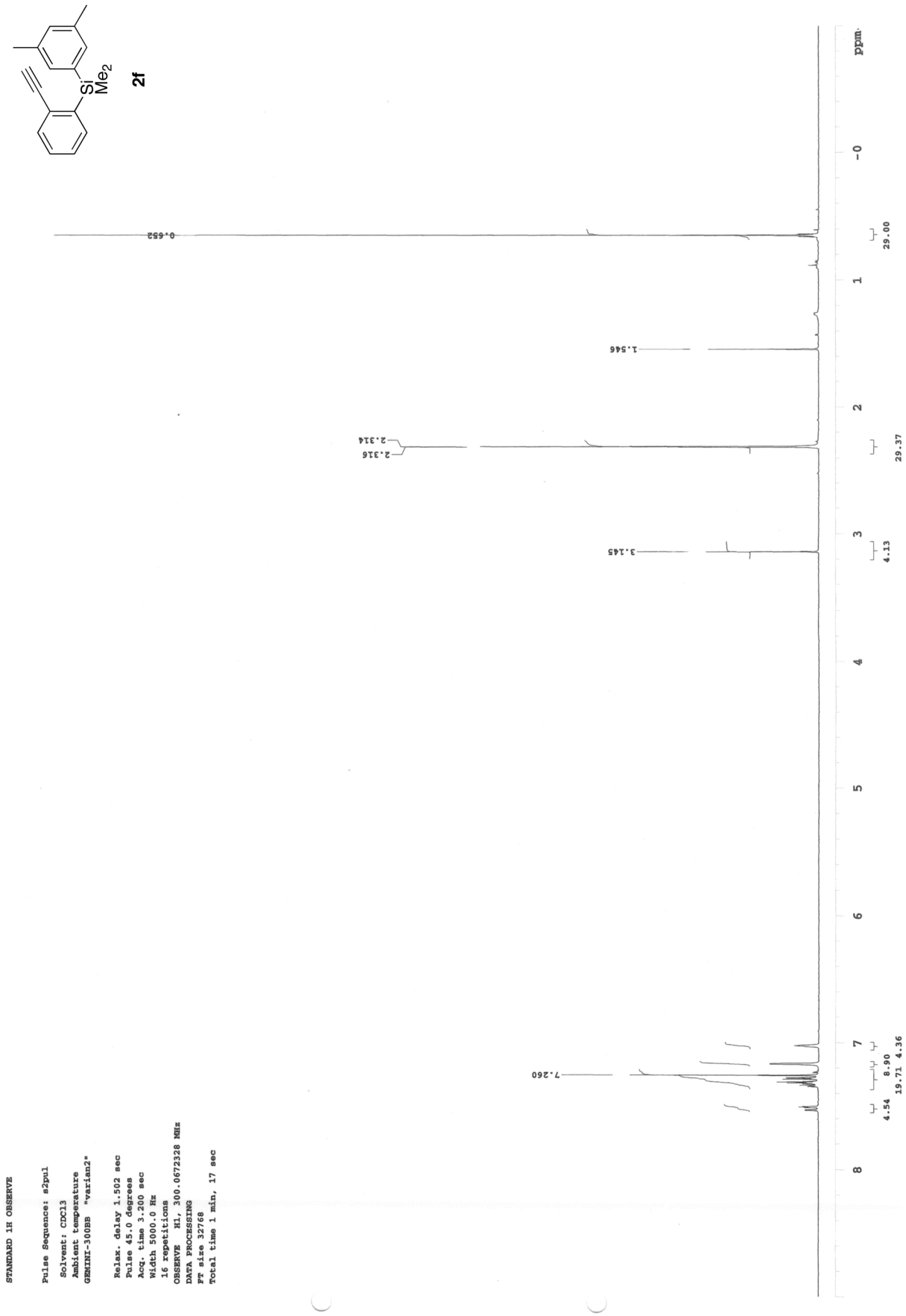
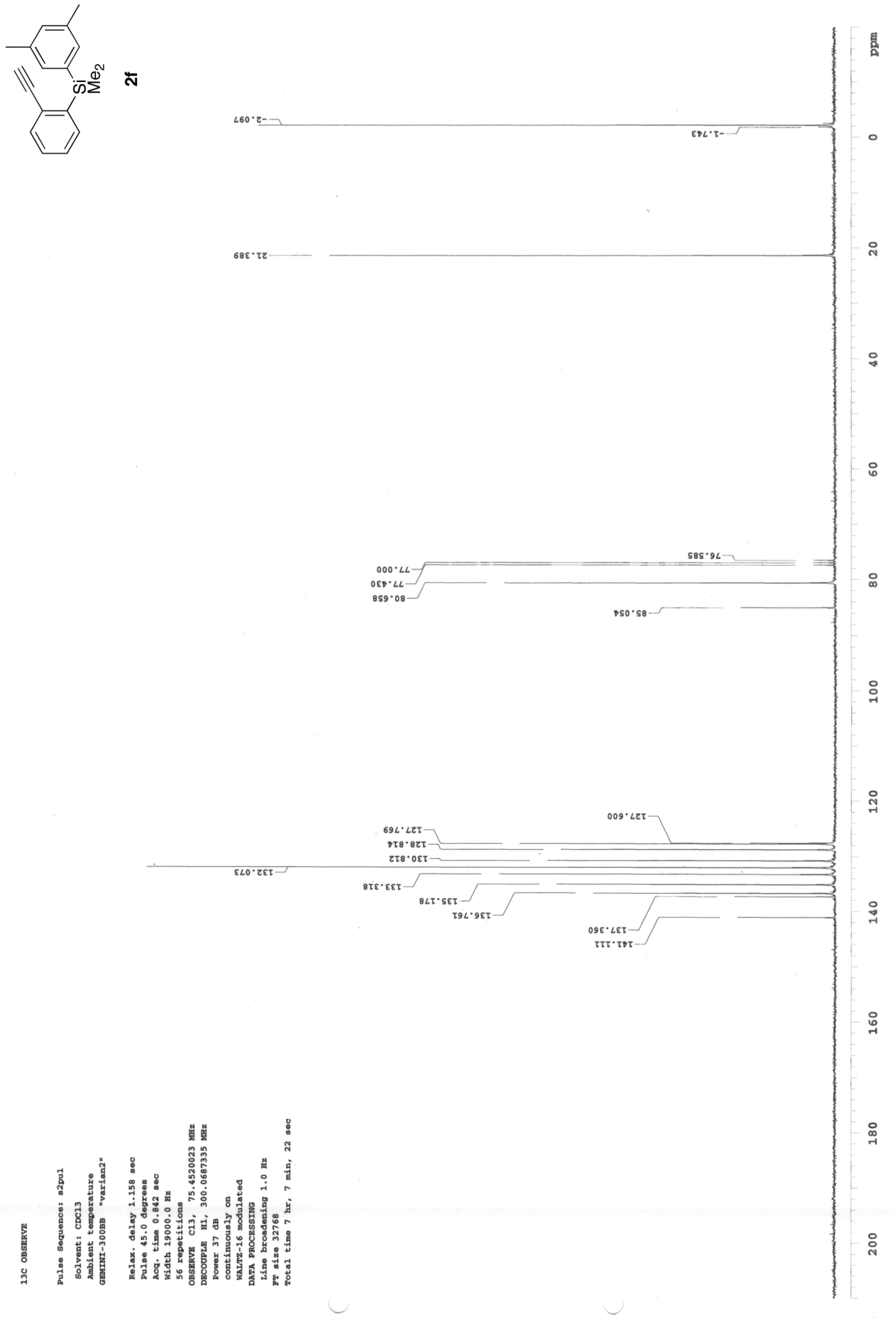
7.260
7.373
7.378
ppm
37.99
4.66
17.49
39.86

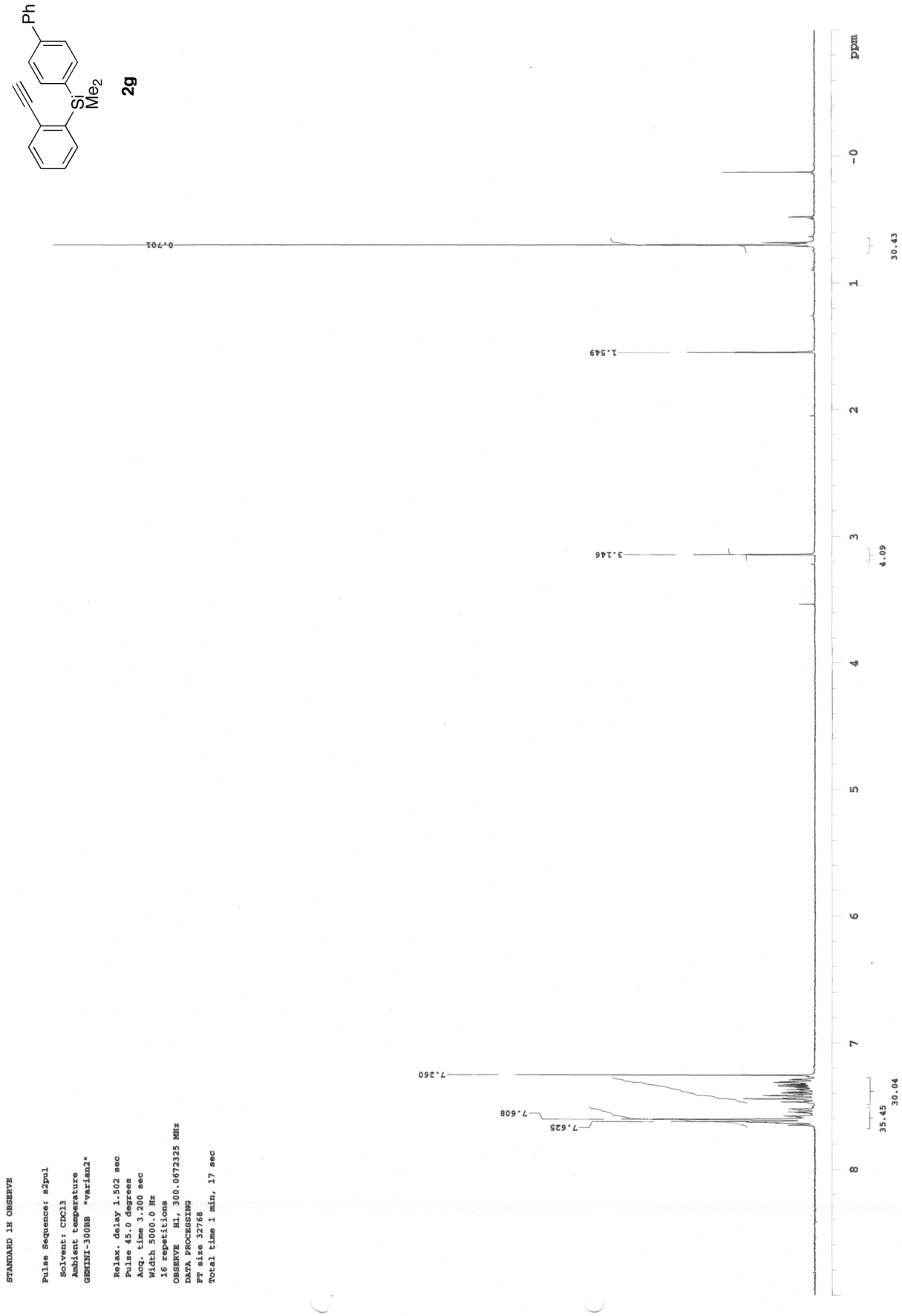

Si
Me2
2d
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
60 repetitions
OBSERVE C13, 75.4519814 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 537 hr, 26 min, 18 sec
-2.189
76.585
77.000
77.430
80.720
84.900
127.631
127.846
128.952
133.348
134.317
135.116
137.760
140.834
ppm
0
20
40
60
80
100
120
140
160
180
200

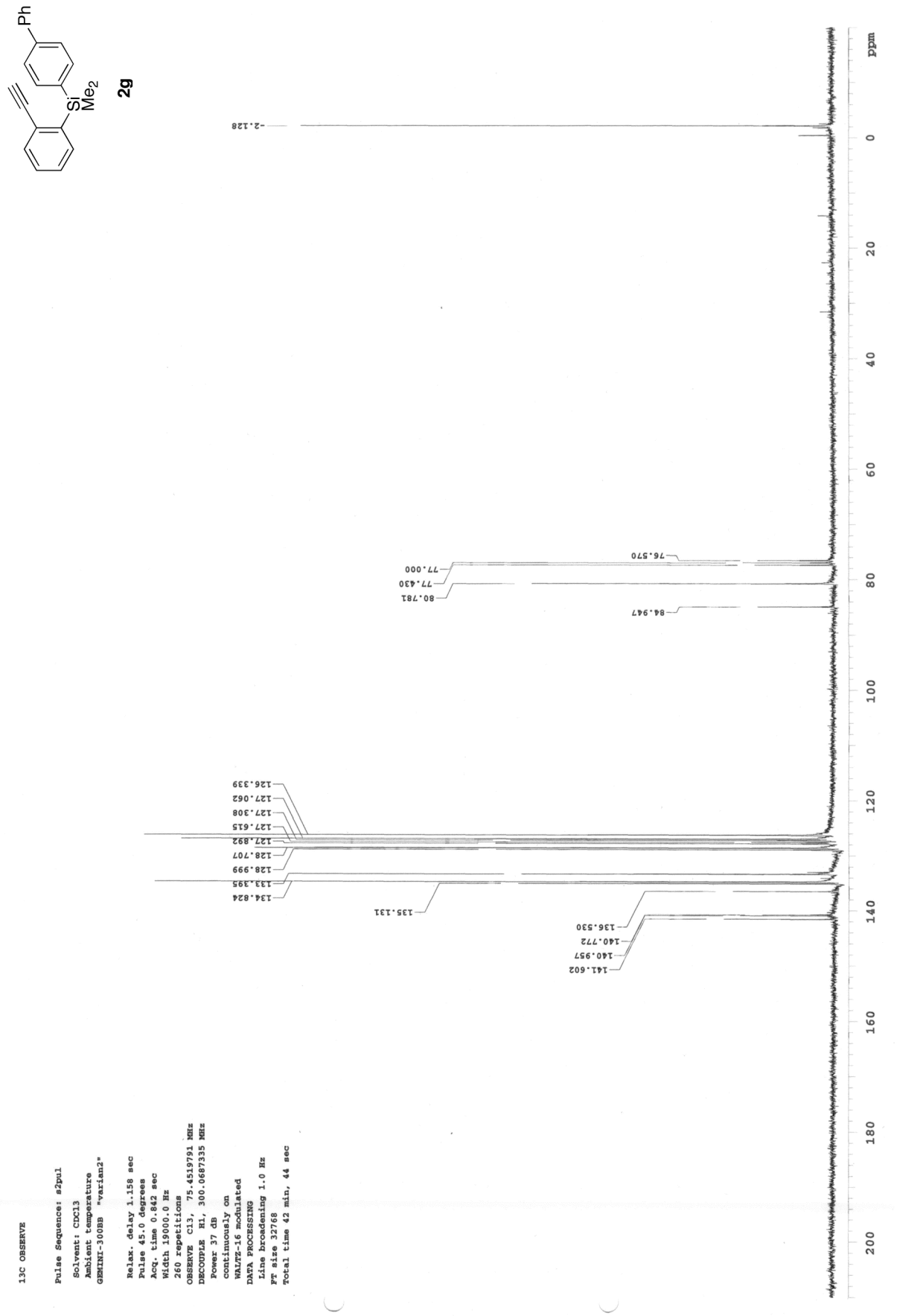

Si
Me2
2e
0.687
2.245
3.027
7.260
ppm
32.52
16.14
4.50
10.46 20.96
5.09 10.34
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec

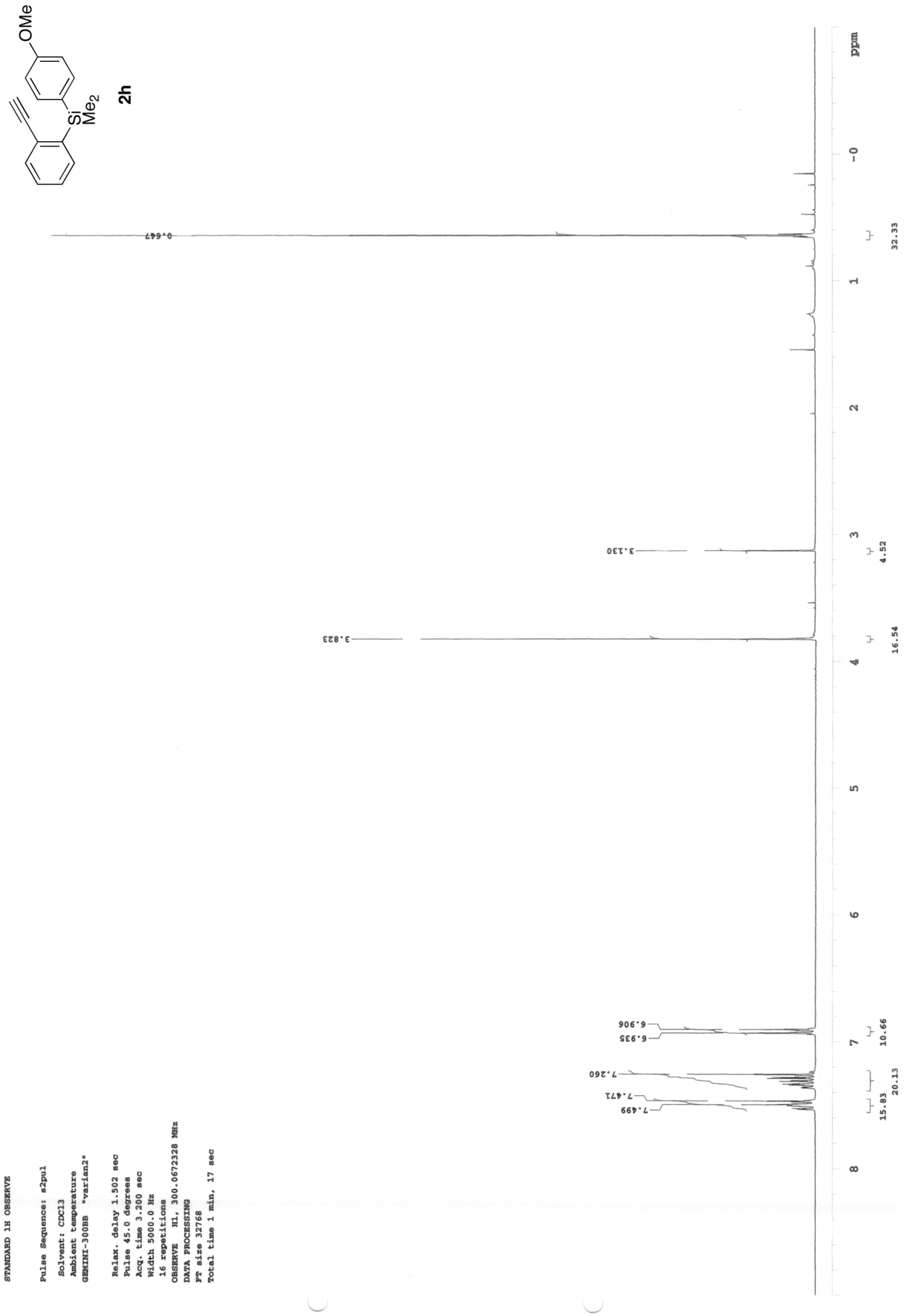

2e
Si
Me2
-1.313
23.172
76.570
77.000
77.430
80.643
84.562
124.772
127.415
127.923
128.799
129.367
129.675
133.302
134.716
135.347
136.008
141.295
143.708
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
44 repetitions
OBSERVE C13, 75.4519883 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

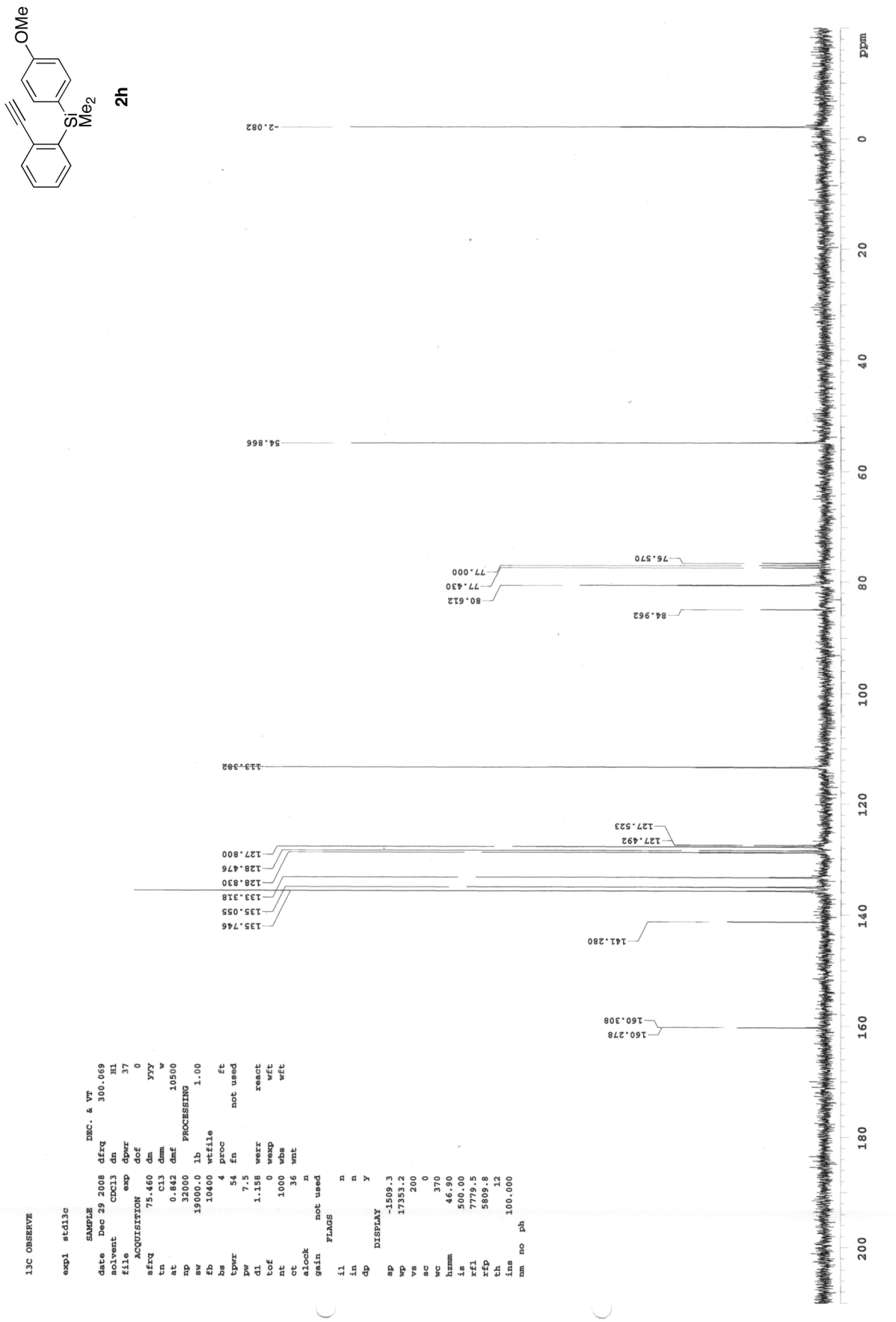

2f
Si
Me2
0.652
1.545
2.314
2.316
3.145
7.260
ppm
29.00
29.37
4.13
4.54
8.90
19.71
4.36
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


2f
Si
Me2
-2.097
-1.743
21.389
76.585
77.000
77.430
80.658
85.054
127.600
127.769
128.814
130.812
132.073
133.318
135.178
136.761
137.360
141.111
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
56 repetitions
OBSERVE C13, 75.4520023 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

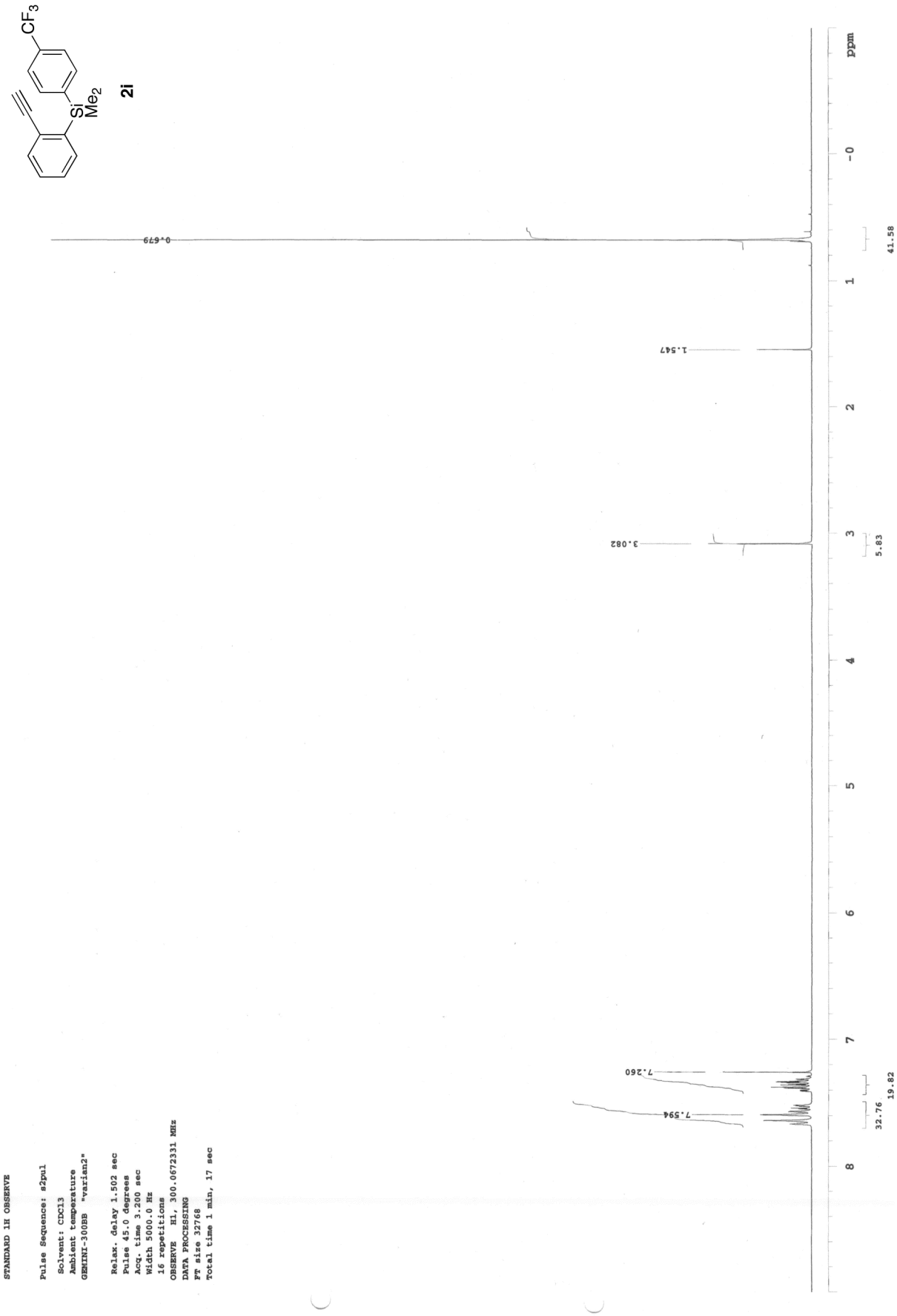

Ph
Si
Me2
2g
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.703
1.549
3.146
7.260
7.608
7.625
ppm
30.43
4.09
35.45
30.04

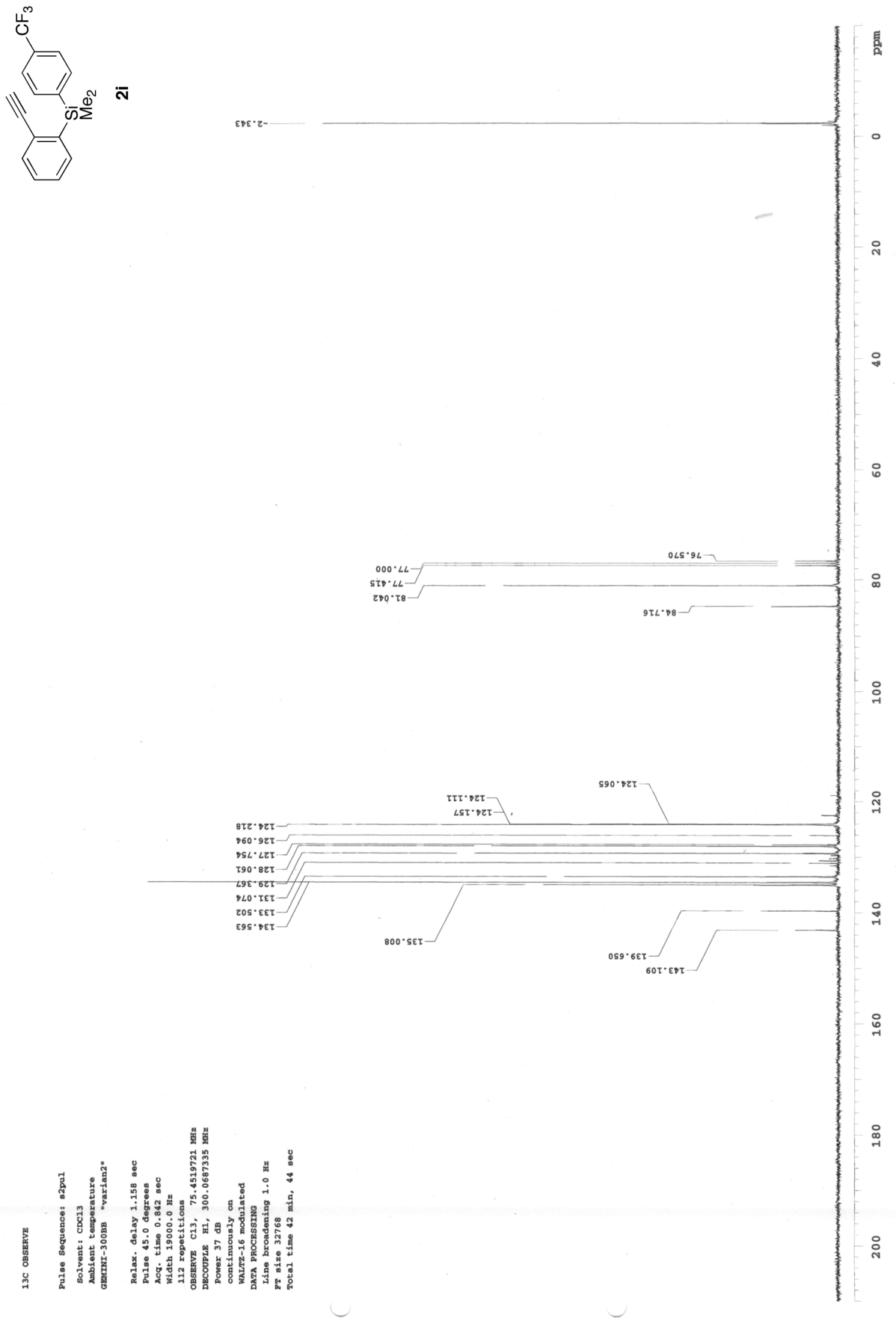

Ph
Si
Me2
2g
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
260 repetitions
OBSERVE C13, 75.4519791 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 42 min, 44 sec
-2.128
76.570
77.000
77.430
80.781
84.947
126.339
127.062
127.308
127.615
127.892
128.707
128.999
133.395
134.824
135.131
136.530
140.772
140.957
141.602
ppm
0
20
40
60
80
100
120
140
160
180
200

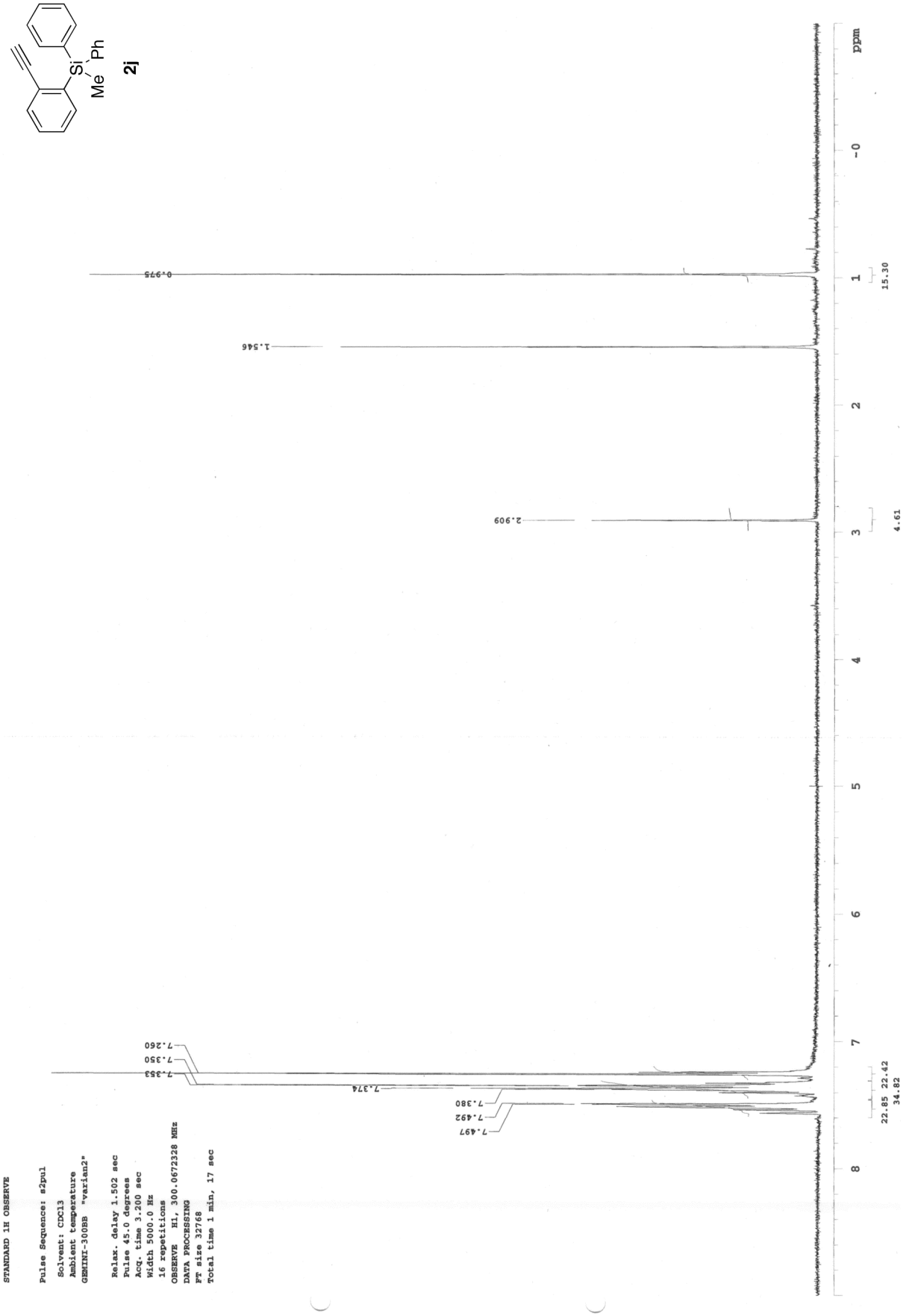

OMe
Si
Me2
2h
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.647
3.130
3.823
6.906
6.935
7.260
7.471
7.499
32.33
4.52
16.54
10.66
15.83
20.13
ppm

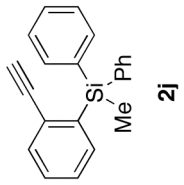



```
13C OBSERVE
exp1   stdi3c
    SAMPLE
    date   Dec 29 2008   dfrq    300.069
    solvent      CDCl3   dn          H1
    file           exp   dpwr        37
      ACQUISITION        dof          0
    sfrq          75.460 dm          yyy
    tn               C13  dmm           w
    at             0.842  dmf       10500
    np             32000  PROCESSING
    sw          19000.0   lb          1.00
    fb             10400  wtfile
    bs                 4  proc          ft
    tpwr              54  fn       not used
    pw               7.5
    d1             1.158  werr        react
    tof                0  wexp          wft
    nt              1000  wbs           wft
    ct                36  wnt
    alock              n
    gain       not used
      FLAGS
    il                 n
    in                 n
    dp                 y
      DISPLAY
    sp            -1509.3
    wp            17353.2
    vs                200
    sc                  0
    wc                370
    hzmm           46.90
    is            500.00
    rf1           7779.5
    rfp           5809.8
    th                 12
    ins           100.000
    nm   no  ph
```


CF3
Si
Me2
2i
0.679
1.547
3.082
7.260
7.594
ppm
41.58
5.83
32.76
19.82
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672331 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec

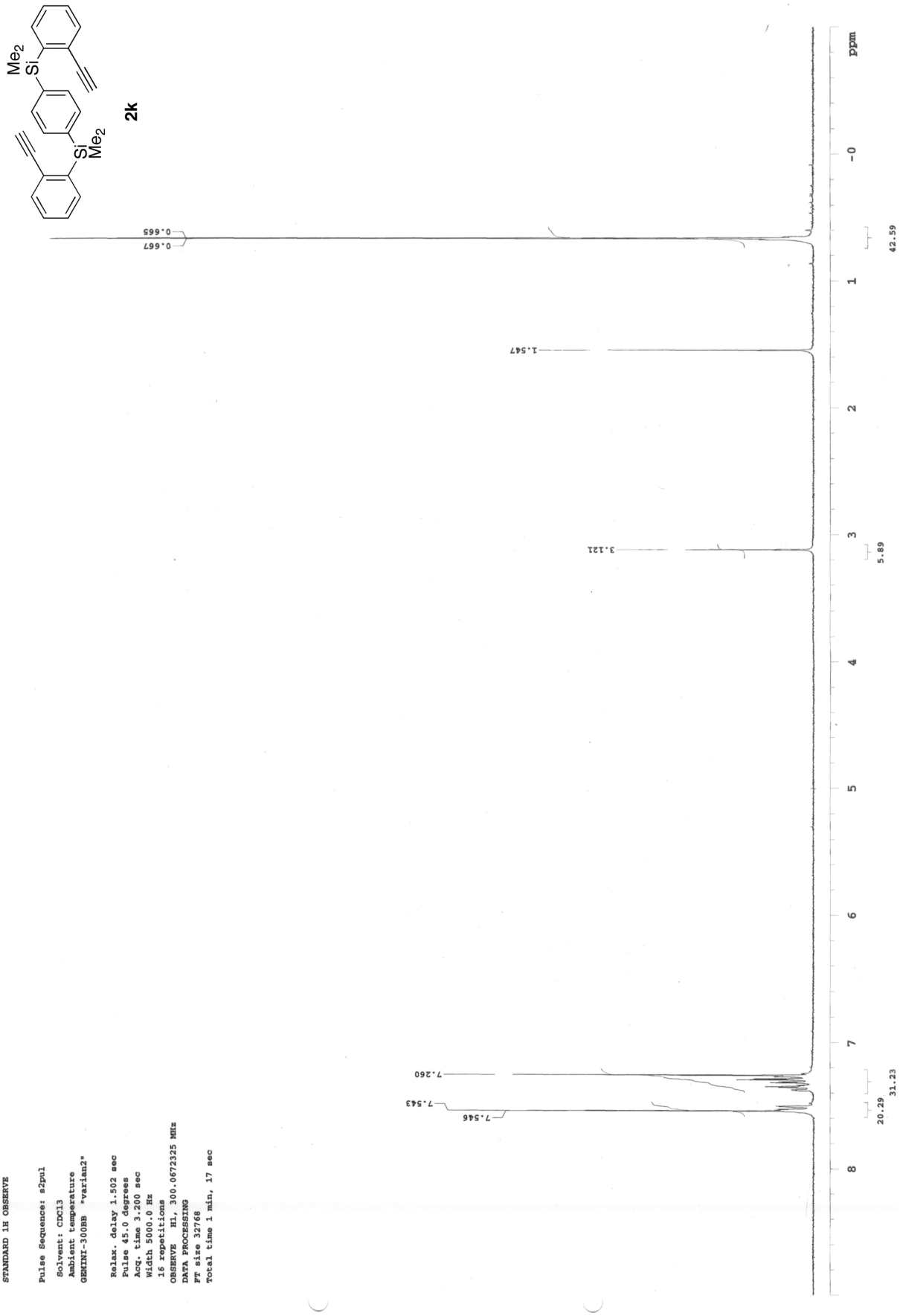

CF3
Si
Me2
2i
-2.343
76.570
77.000
77.415
81.042
84.716
124.065
124.111
124.157
124.218
126.094
127.754
128.061
129.367
131.074
133.502
134.563
135.008
139.650
143.109
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
112 repetitions
OBSERVE C13, 75.4519721 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 42 min, 44 sec

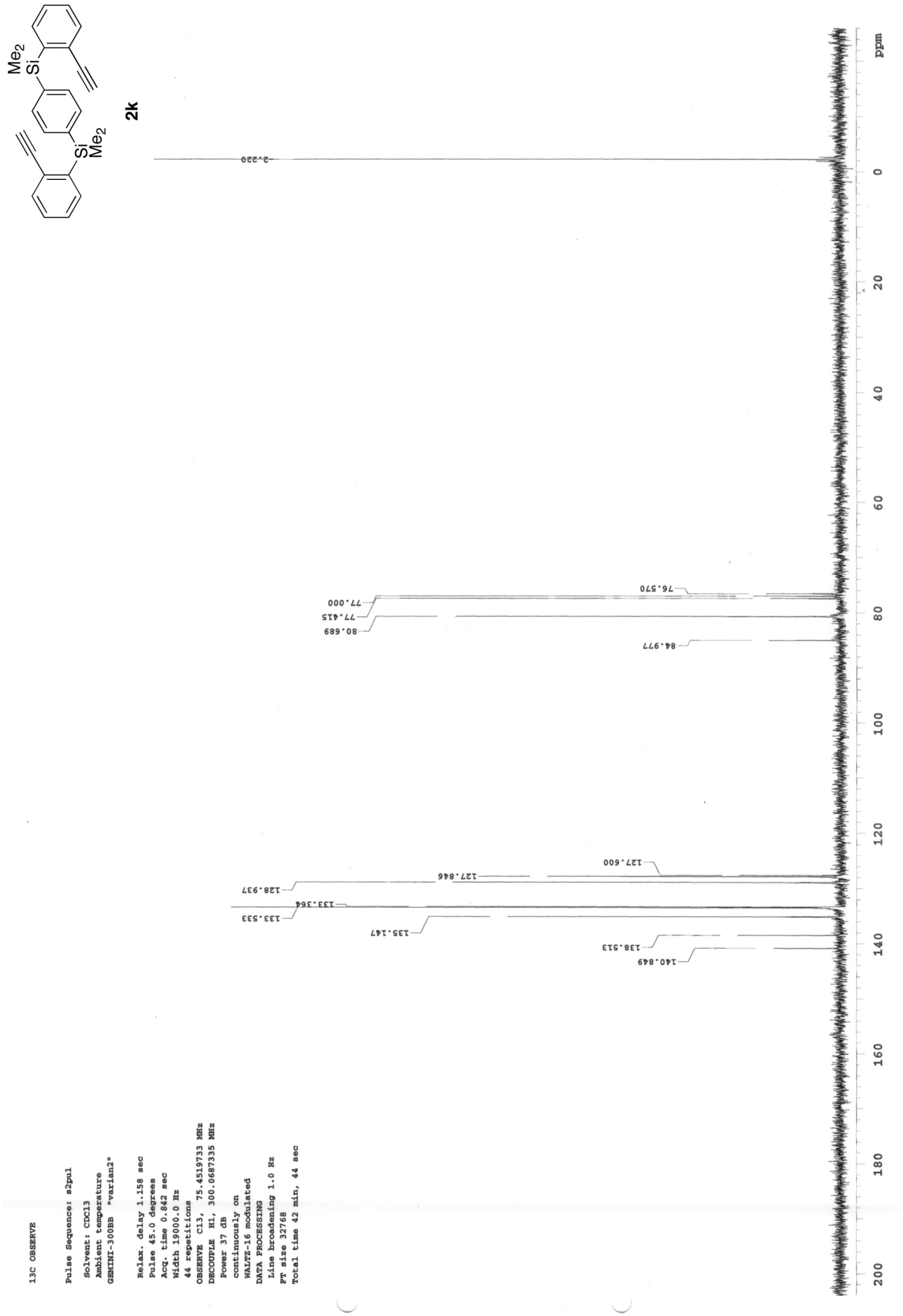

Si
Me
Ph
2j
0.975
1.546
2.909
7.260
7.350
7.353
7.374
7.380
7.492
7.497
ppm
15.30
4.61
22.85 22.42
34.82
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec

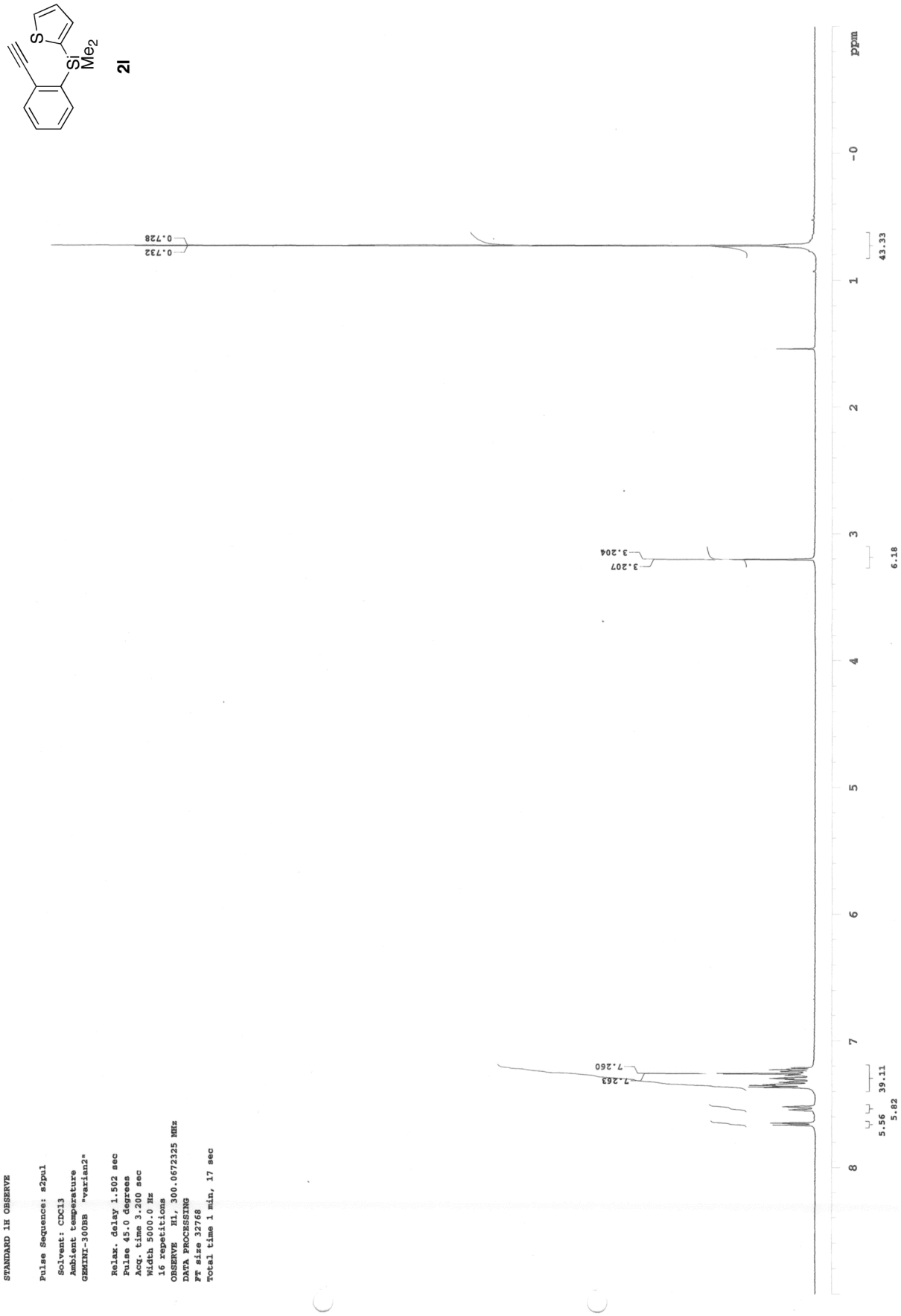

Si
Me
Ph

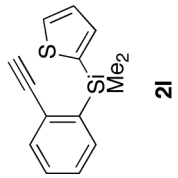
2j

-3.204
76.570
77.000
77.415
81.227
84.639
127.677
127.846
128.261
129.183
129.291
133.395
135.254
135.900
136.668
138.913
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
108 repetitions
OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 42 min, 44 sec


Me2
Si
Me2
Si
2k
0.667
0.665
1.547
3.121
7.260
7.543
7.546
ppm
42.59
5.89
20.29
31.23
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


Me2
Si
Si
Me2
2k
-2.220
76.570
77.000
77.415
80.689
84.977
127.600
127.846
128.937
133.364
133.533
135.147
138.513
140.849
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
44 repetitions
OBSERVE C13, 75.4519733 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 42 min, 44 sec


2l
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.732
0.728
3.207
3.204
7.263
7.260
ppm
43.33
6.18
39.11
5.56
5.82

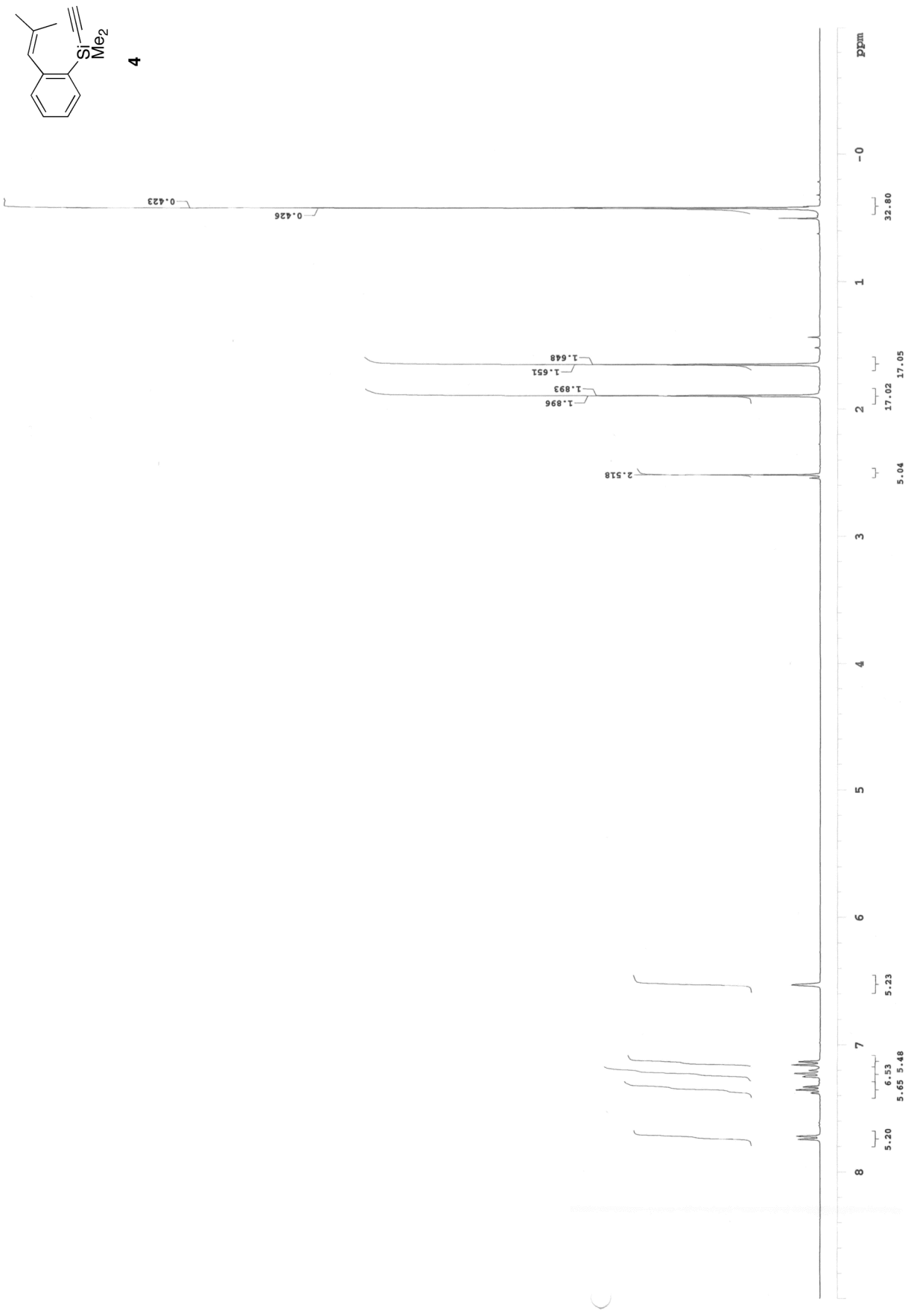


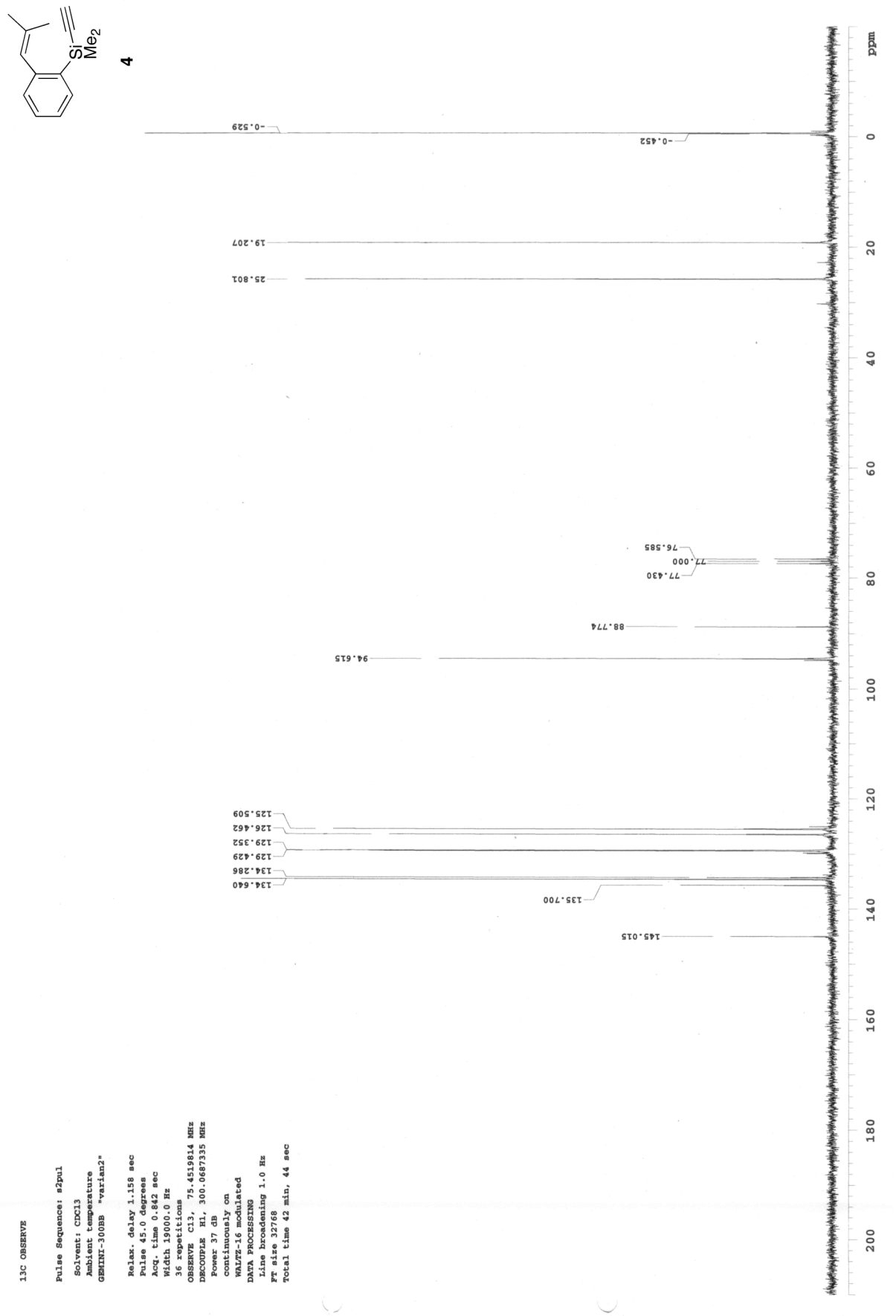
2l

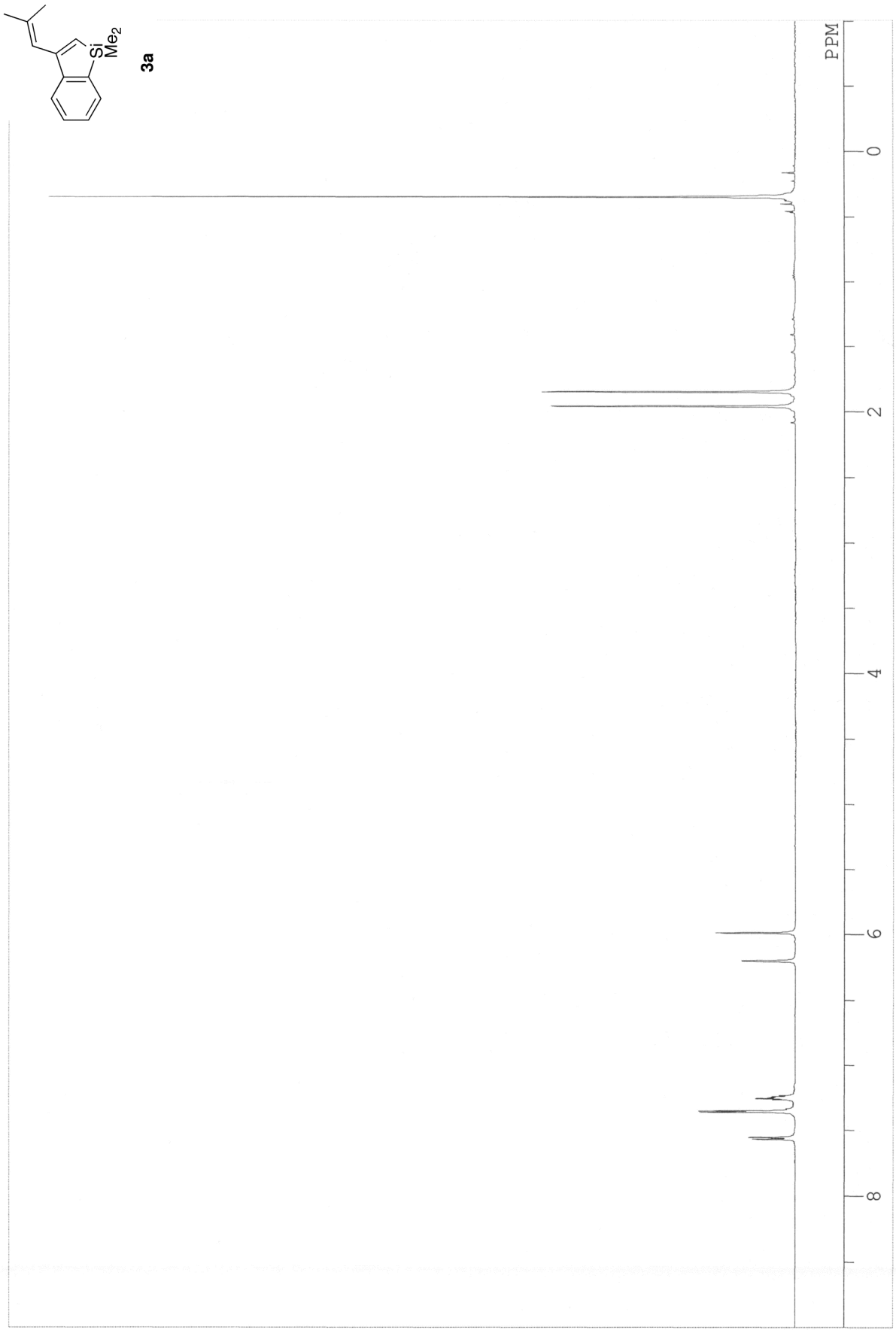
13C OBSERVE

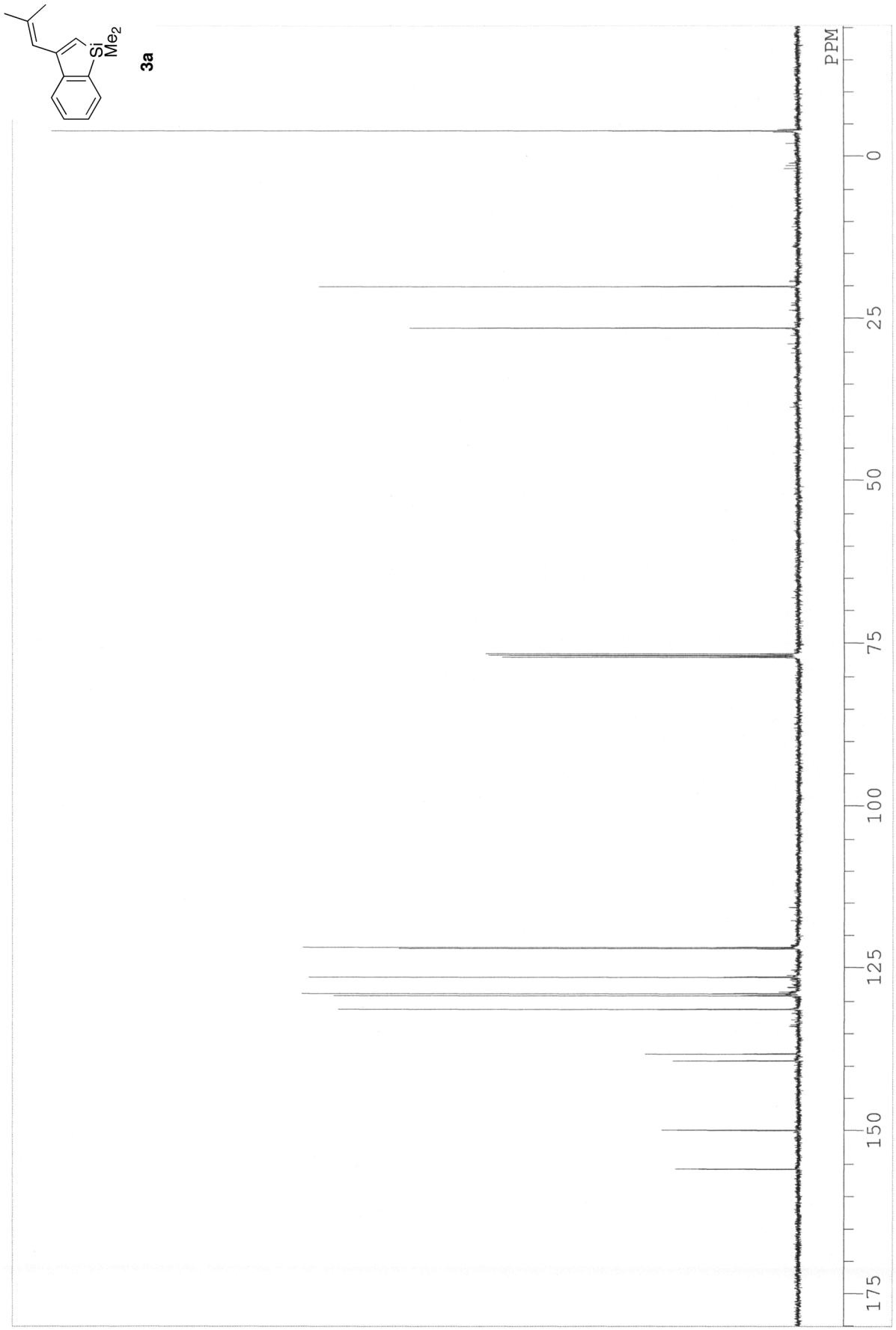
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

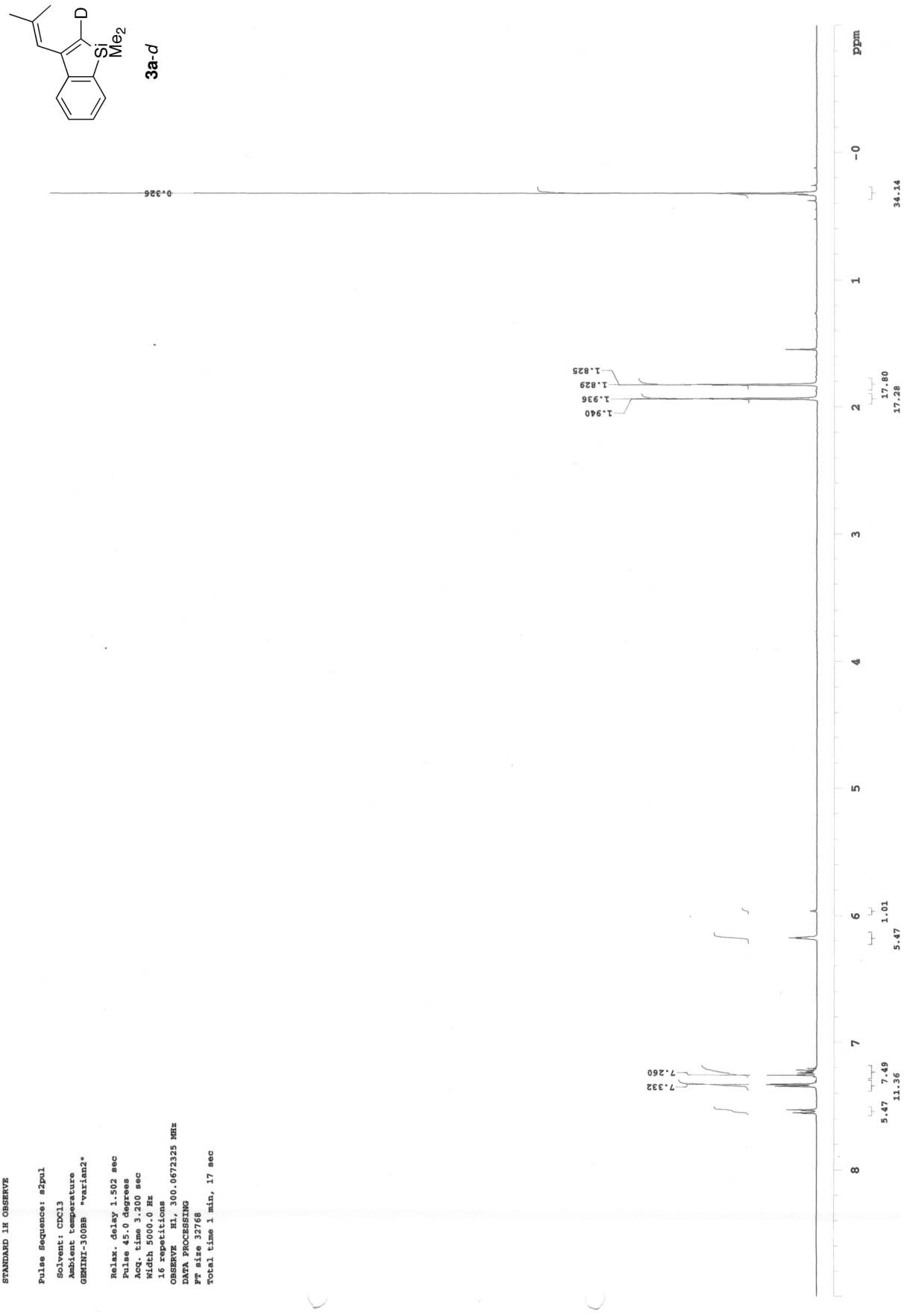
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
64 repetitions
OBSERVE C13, 75.4519756 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

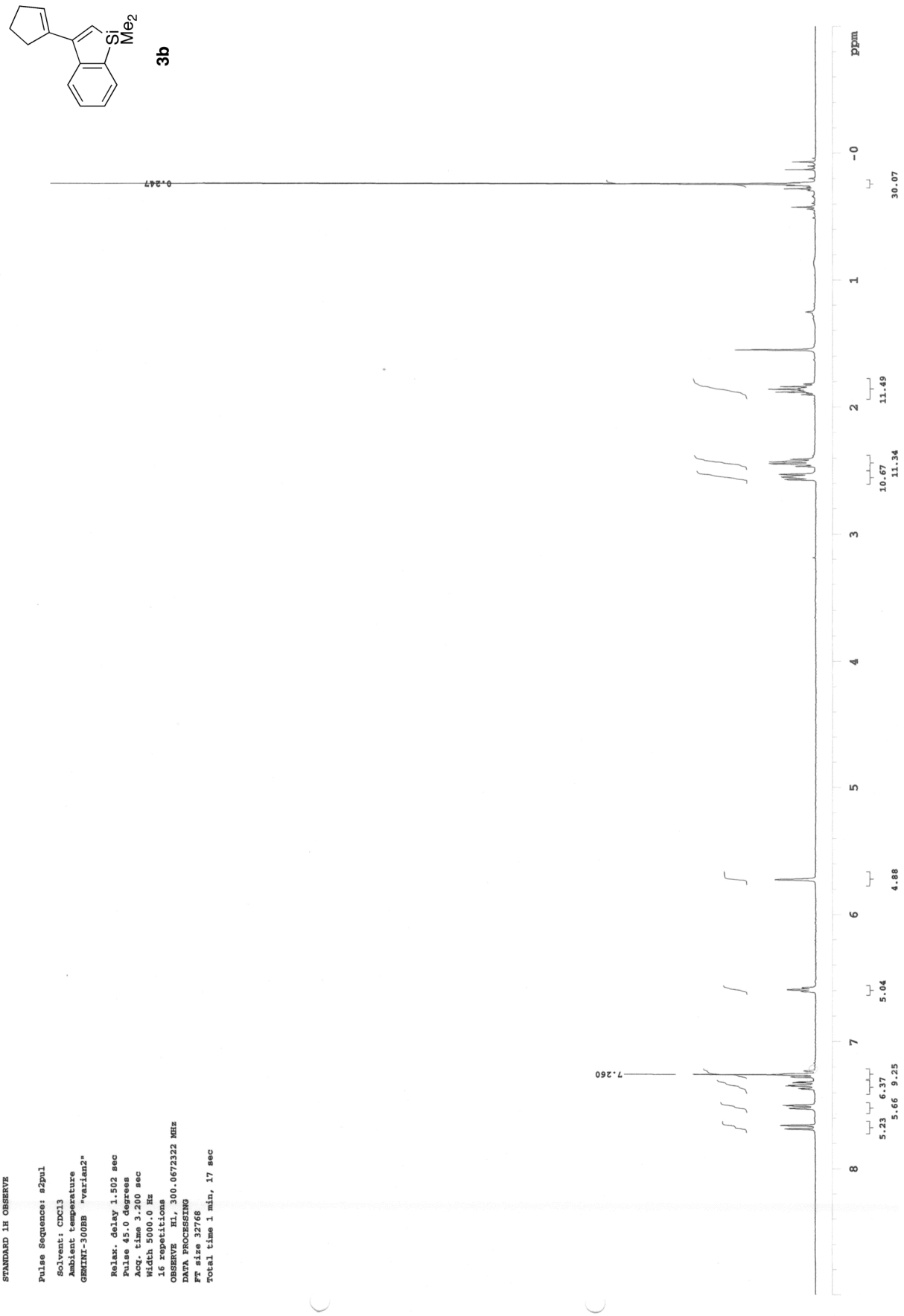

Si
Me2
4
0.423
0.426
1.648
1.651
1.893
1.896
2.518
ppm
32.80
17.05
17.02
5.04
5.23
6.53
5.65
5.48
5.20

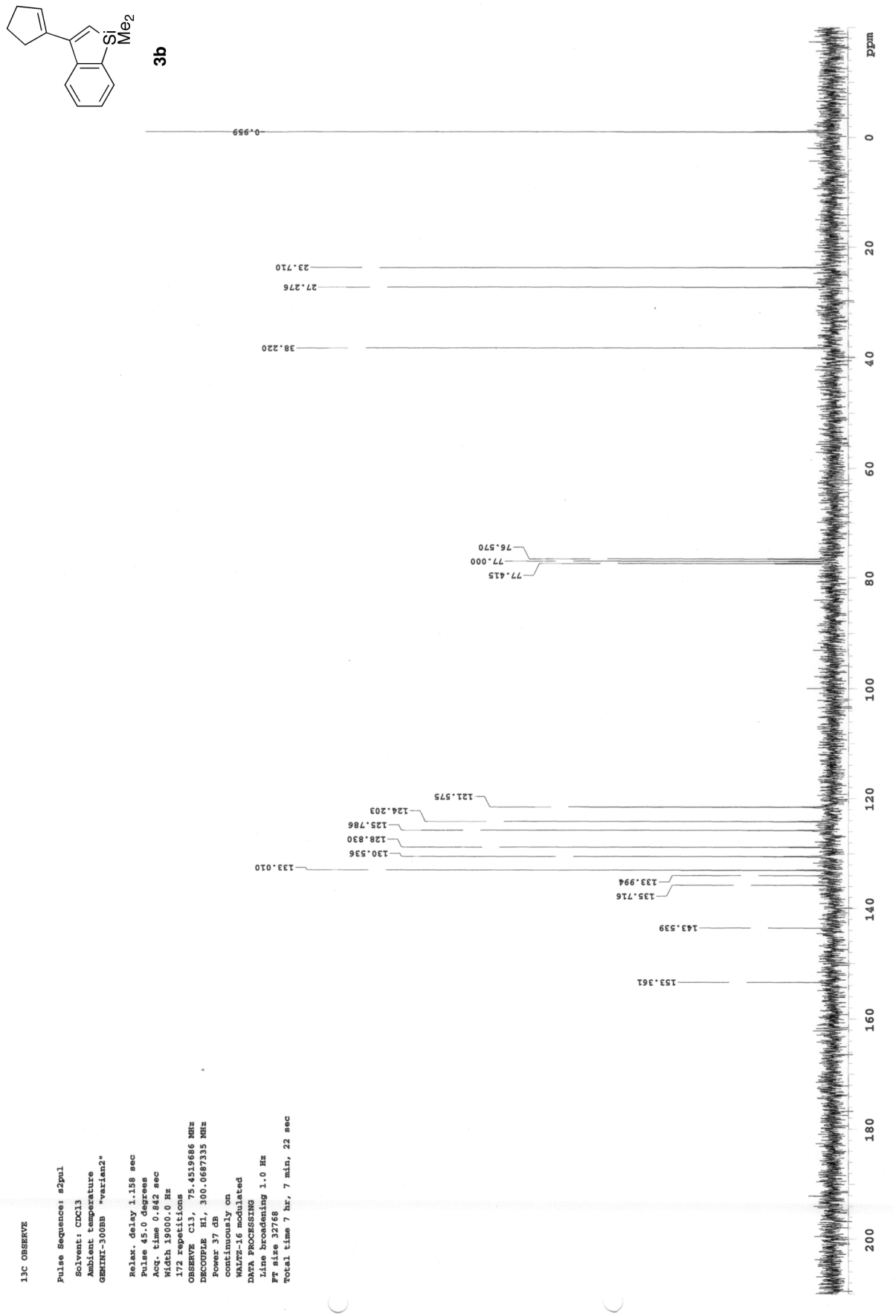

4
Si
Me2
-0.529
-0.452
19.207
25.801
76.585
77.000
77.430
88.774
94.615
125.509
126.462
129.352
129.429
134.286
134.640
135.700
145.015
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
36 repetitions
OBSERVE C13, 75.4519814 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 42 min, 44 sec

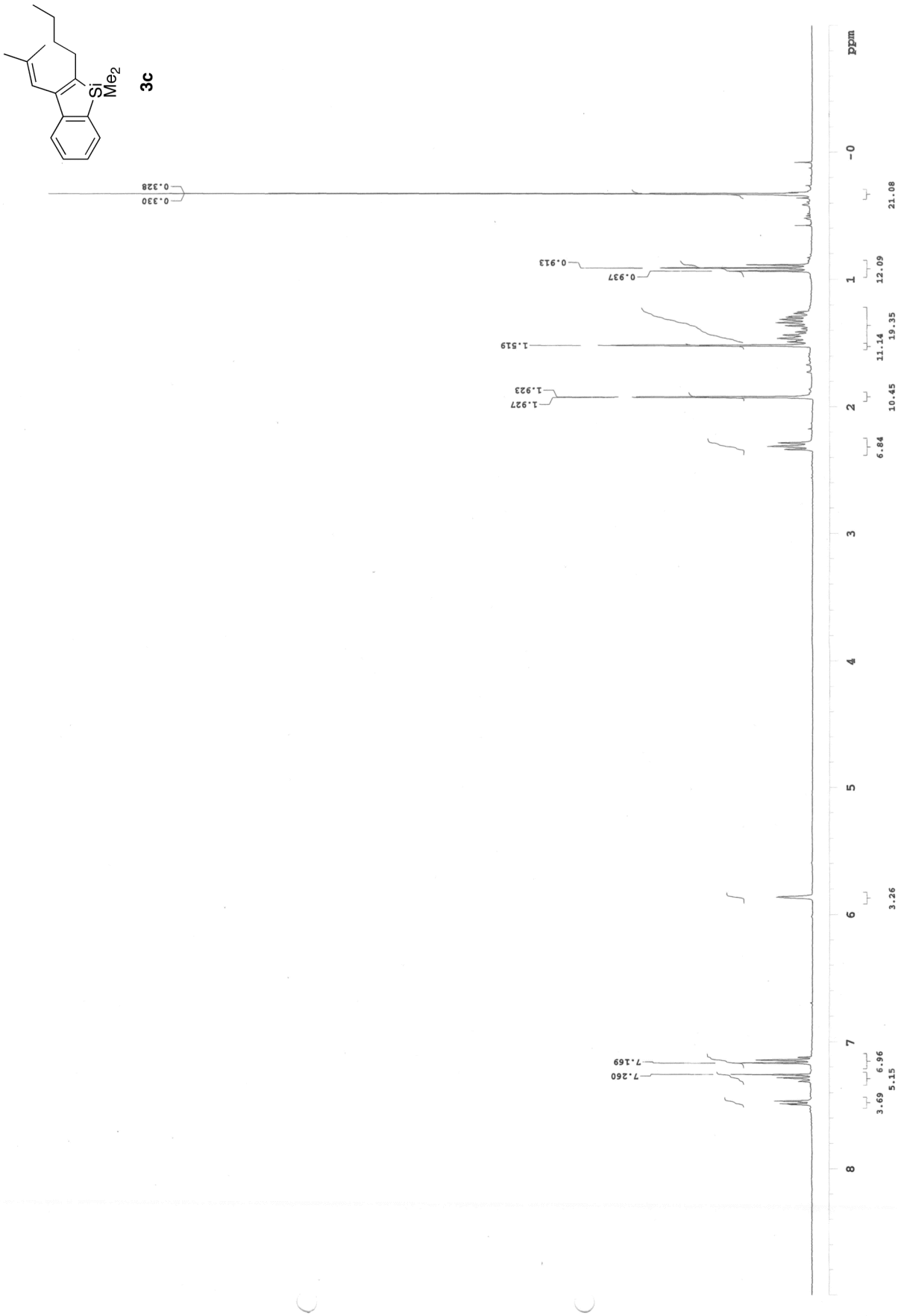

Si
Me2
3a
PPM
0
2
4
6
8

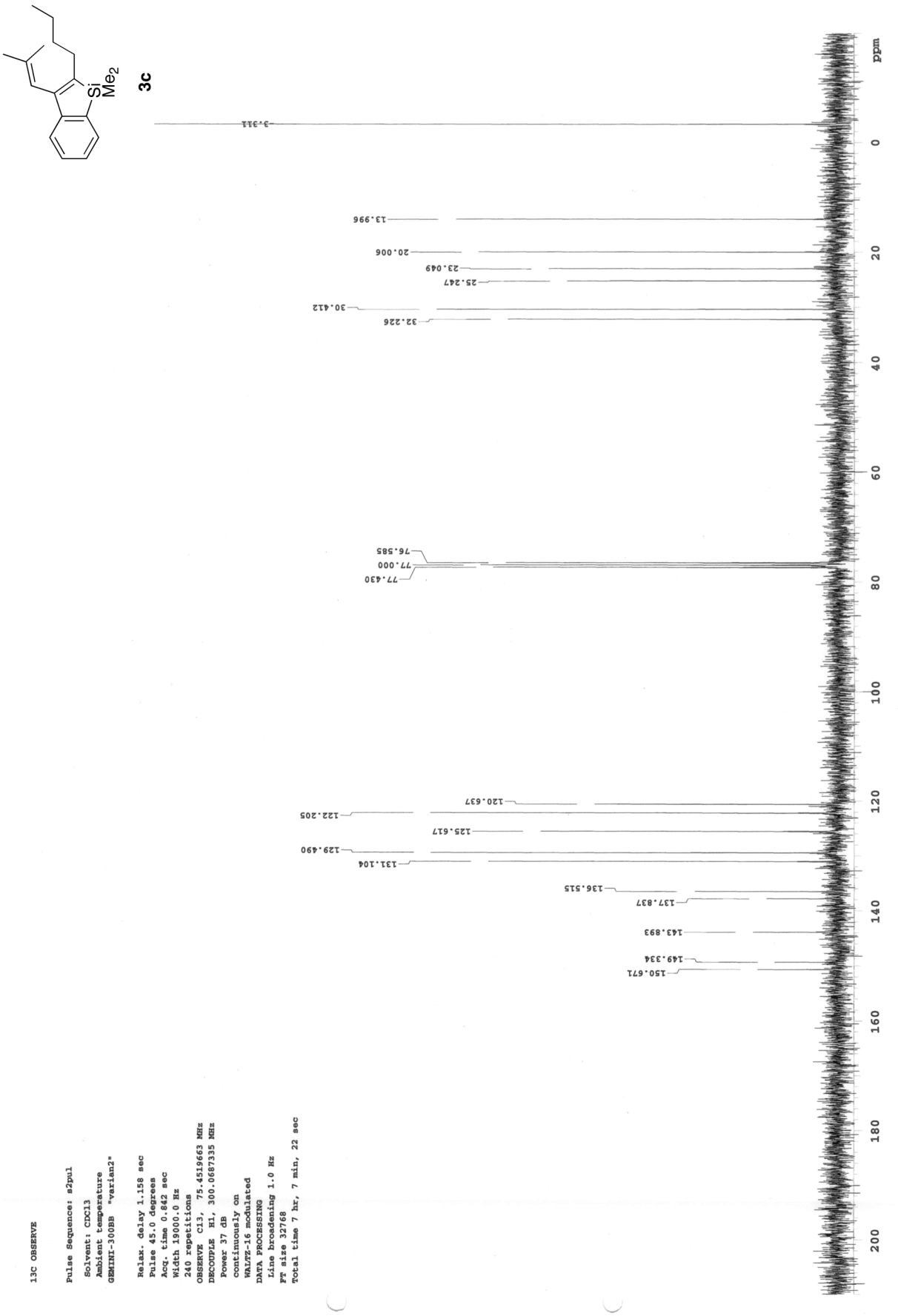

Si
Me2
3a
PPM
0
25
50
75
100
125
150
175

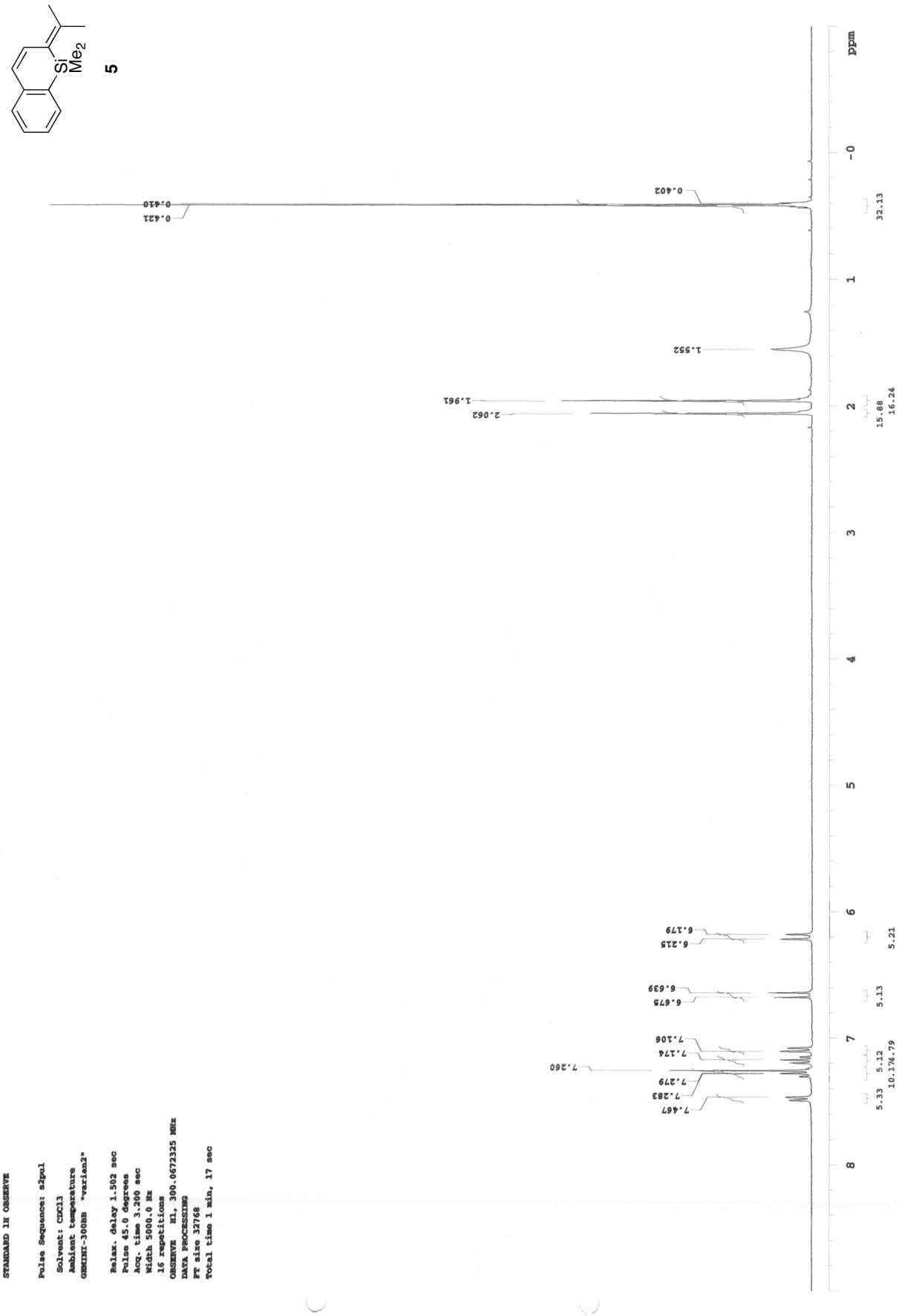

D
Si
Me2
3a-d
0.326
1.825
1.829
1.936
1.940
7.260
7.332
ppm
34.14
17.80
17.28
1.01
5.47
7.49
11.36
5.47
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec

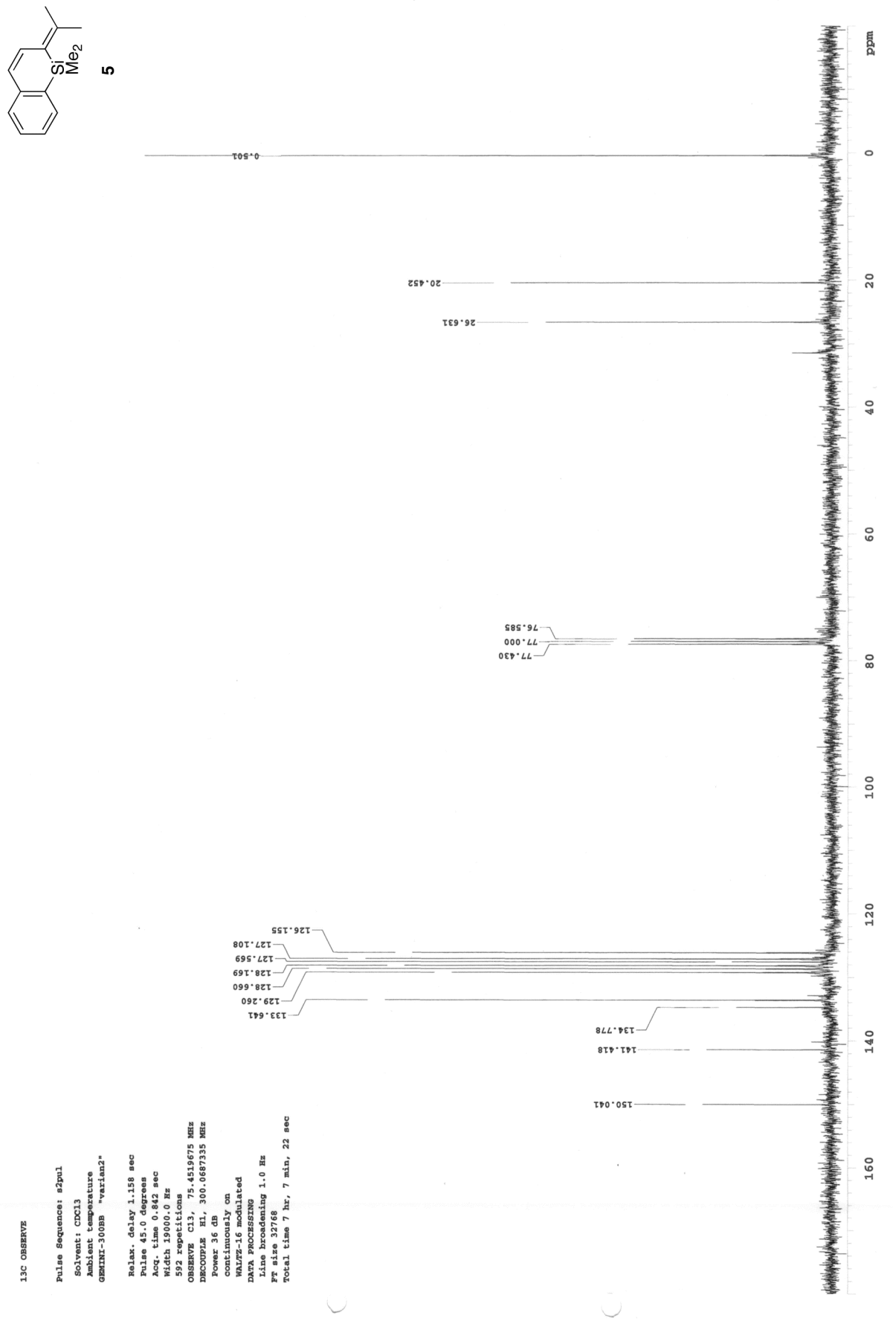

Si
Me2
3b
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672322 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.247
7.260
ppm
30.07
11.49
11.34
10.67
4.88
5.04
9.25
6.37
5.66
5.23

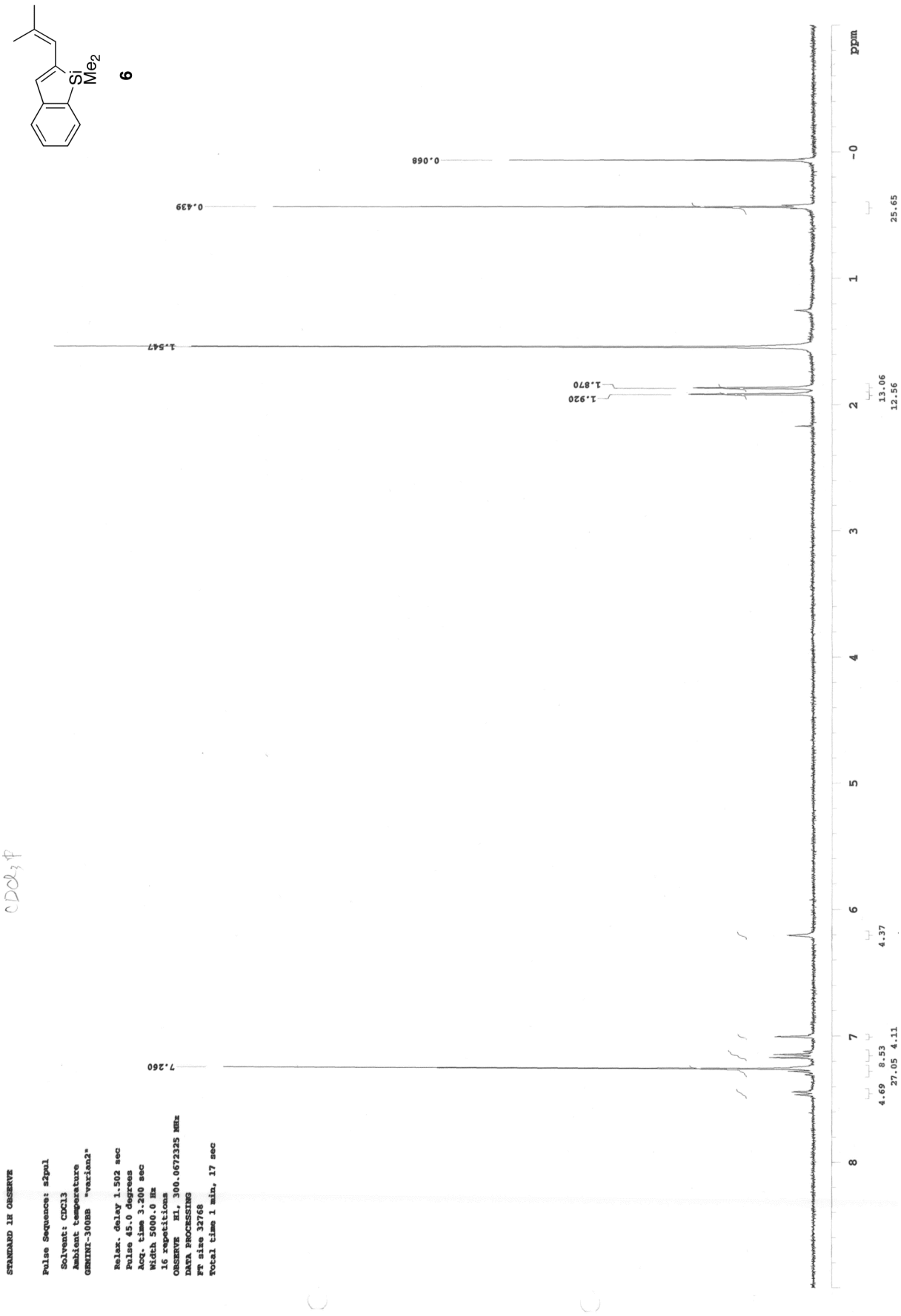

Si
Me2
3b
-0.959
23.710
27.276
38.220
76.570
77.000
77.415
121.575
124.203
125.786
128.830
130.536
133.010
133.994
135.716
143.539
153.361
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
172 repetitions
OBSERVE C13, 75.4519686 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

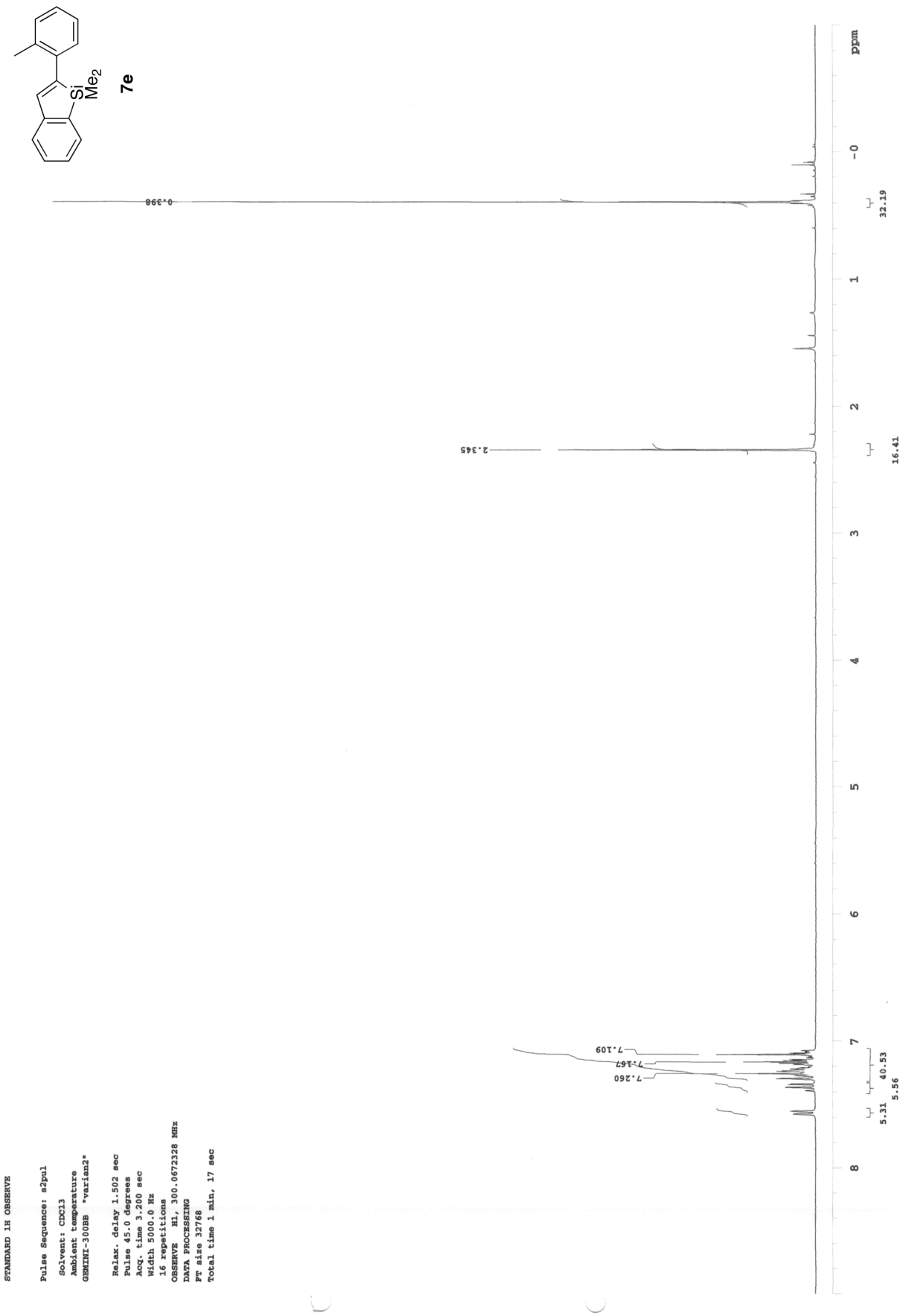

Si
Me2
3c
0.328
0.330
0.913
0.937
1.519
1.923
1.927
7.169
7.260
ppm
-0
1
2
3
4
5
6
7
8
21.08
12.09
19.35
11.14
10.45
6.84
3.26
6.96
5.15
3.69

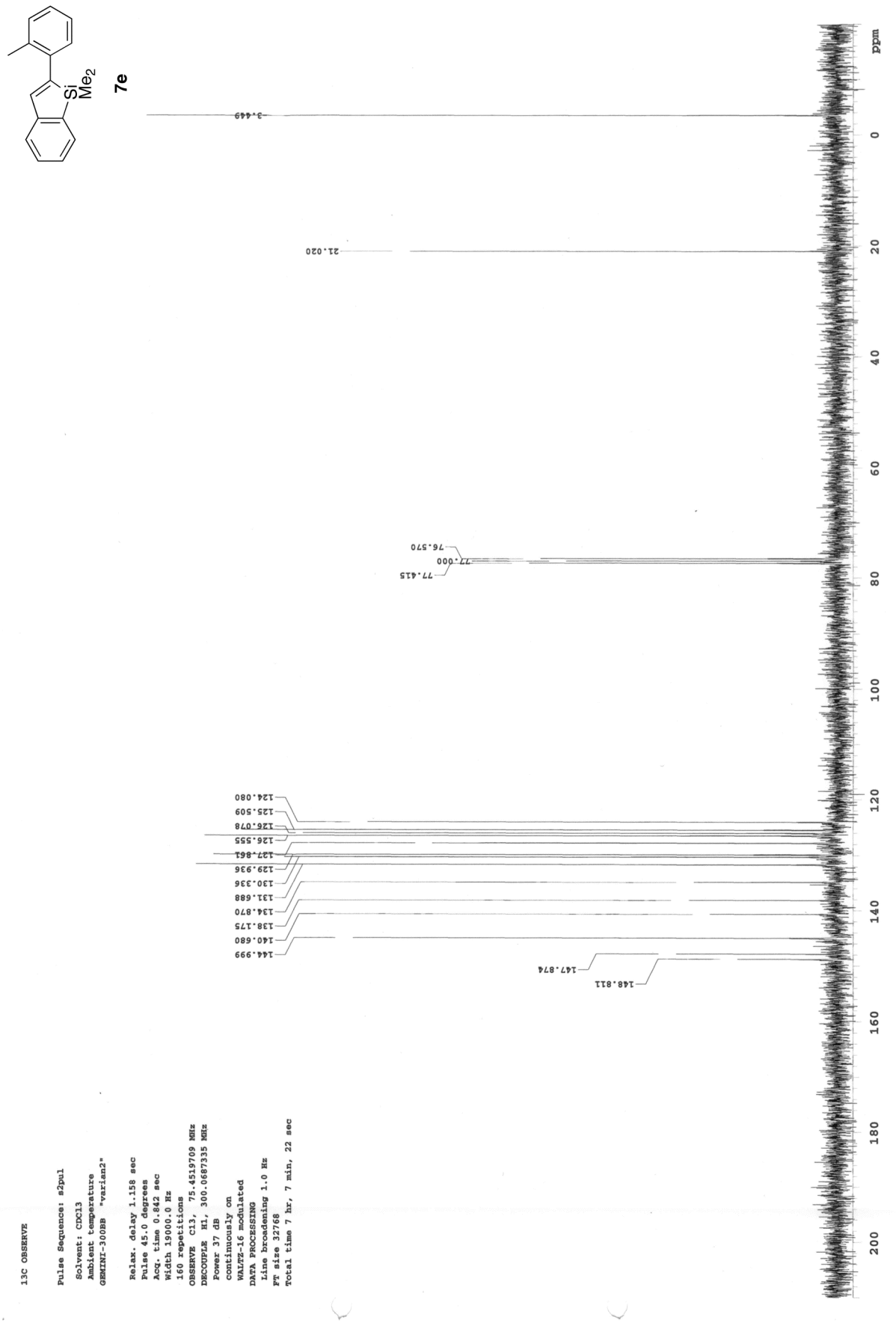

3c
Si
Me2
3.311
13.996
20.006
23.049
25.247
30.412
32.226
76.585
77.000
77.430
120.637
122.205
125.617
129.490
131.104
136.515
137.837
143.893
149.334
150.671
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
240 repetitions
OBSERVE C13, 75.4519663 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

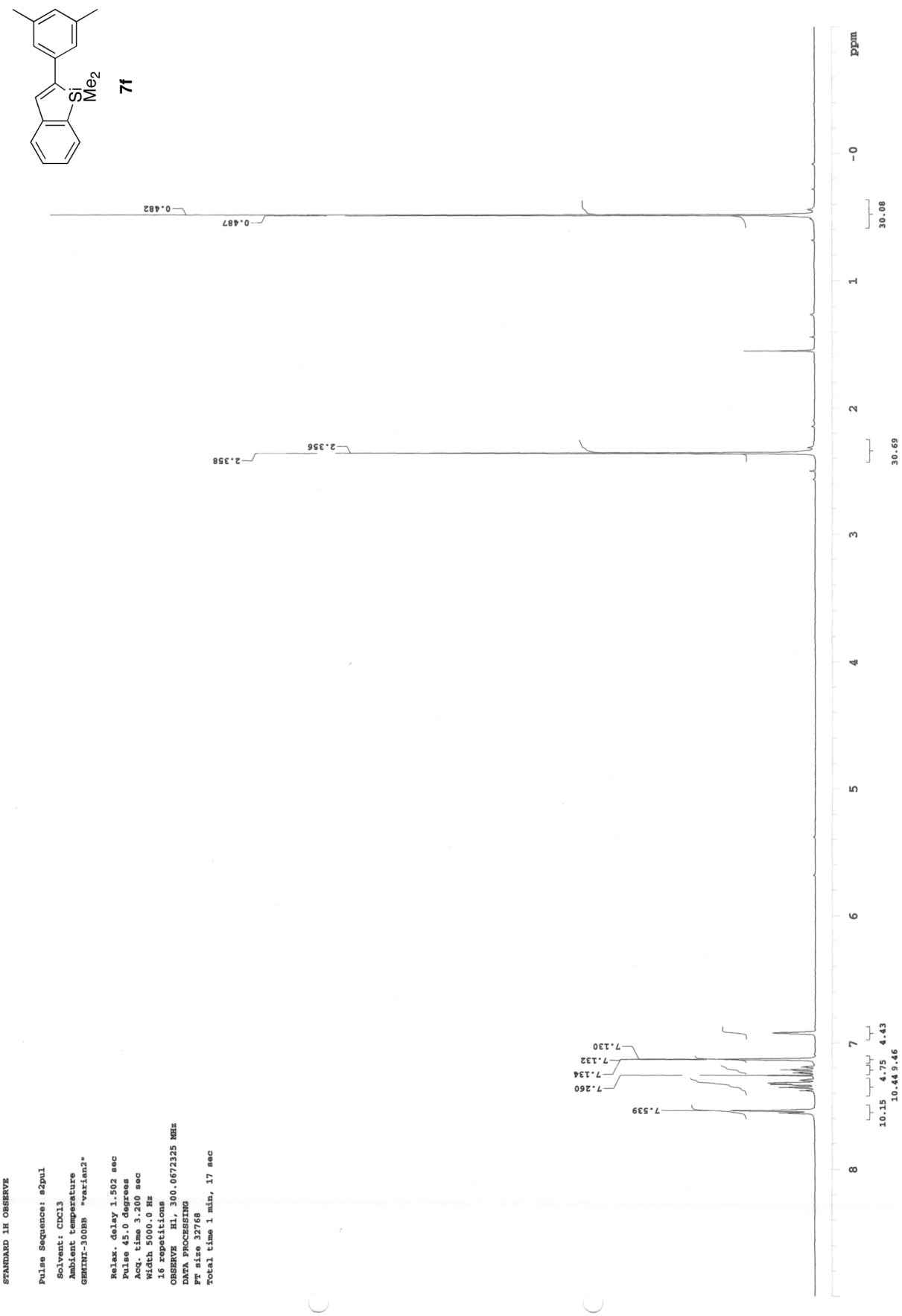

Si
Me2
5
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.402
0.421
0.410
1.552
1.961
2.062
6.179
6.215
6.639
6.675
7.106
7.174
7.260
7.279
7.283
7.467
ppm
32.13
15.88
16.24
5.21
5.13
5.12
5.33
10.174.79

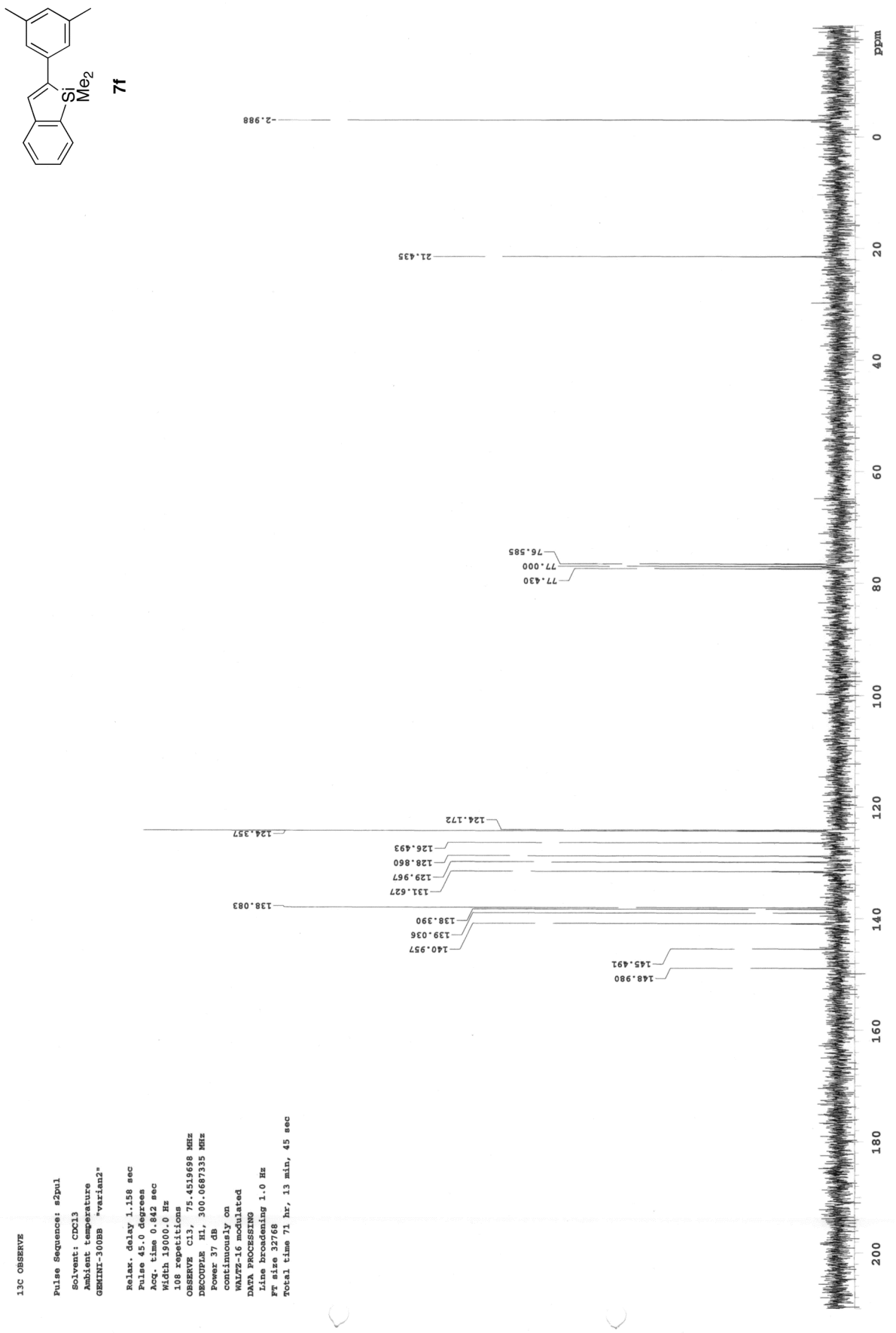

5
Si
Me2
0.501
20.452
26.631
76.585
77.000
77.430
126.155
127.108
127.569
128.169
128.660
129.260
133.641
134.778
141.418
150.041
ppm
0
20
40
60
80
100
120
140
160
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
592 repetitions
OBSERVE C13, 75.4519675 MHz
DECOUPLE H1, 300.0687335 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

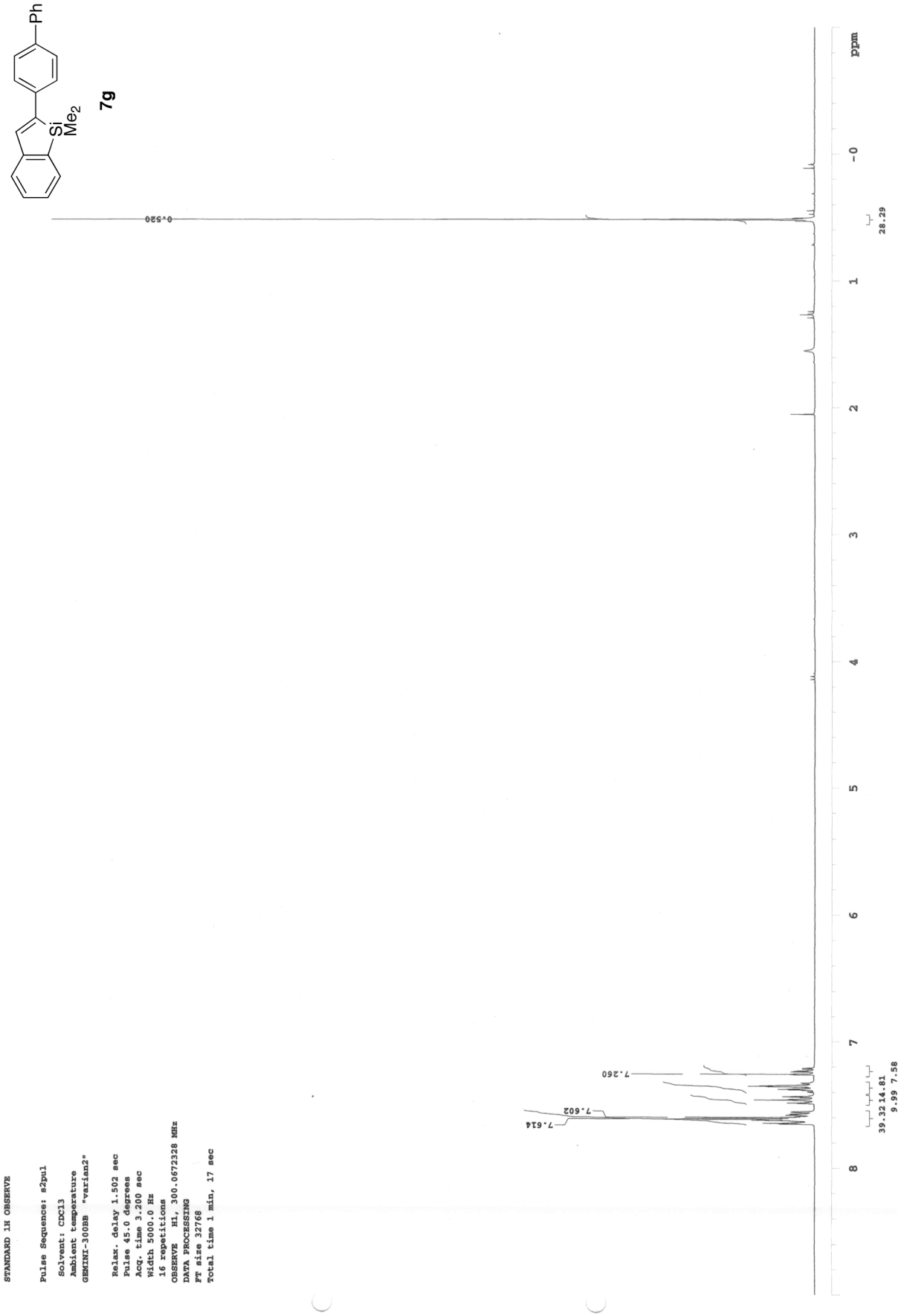

Si
Me2
6
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
7.260
1.547
1.920
1.870
0.439
0.068
ppm
4.69
8.53
27.05
4.11
4.37
13.06
12.56
25.65

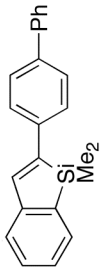

Si
Me2
7e
0.398
2.345
7.109
7.167
7.260
ppm
32.19
16.41
40.53
5.56
5.31
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


7e
Si
Me2
3.449
21.020
76.570
77.000
77.415
124.080
125.509
126.078
126.555
127.861
129.936
130.336
131.688
134.870
138.175
140.680
144.999
147.874
148.811
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
160 repetitions
OBSERVE C13, 75.4519709 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec


Si
Me2
7f
0.482
0.487
2.358
2.356
7.130
7.132
7.134
7.260
7.539
ppm
30.08
30.69
10.15
4.75
4.43
10.44
9.46
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec

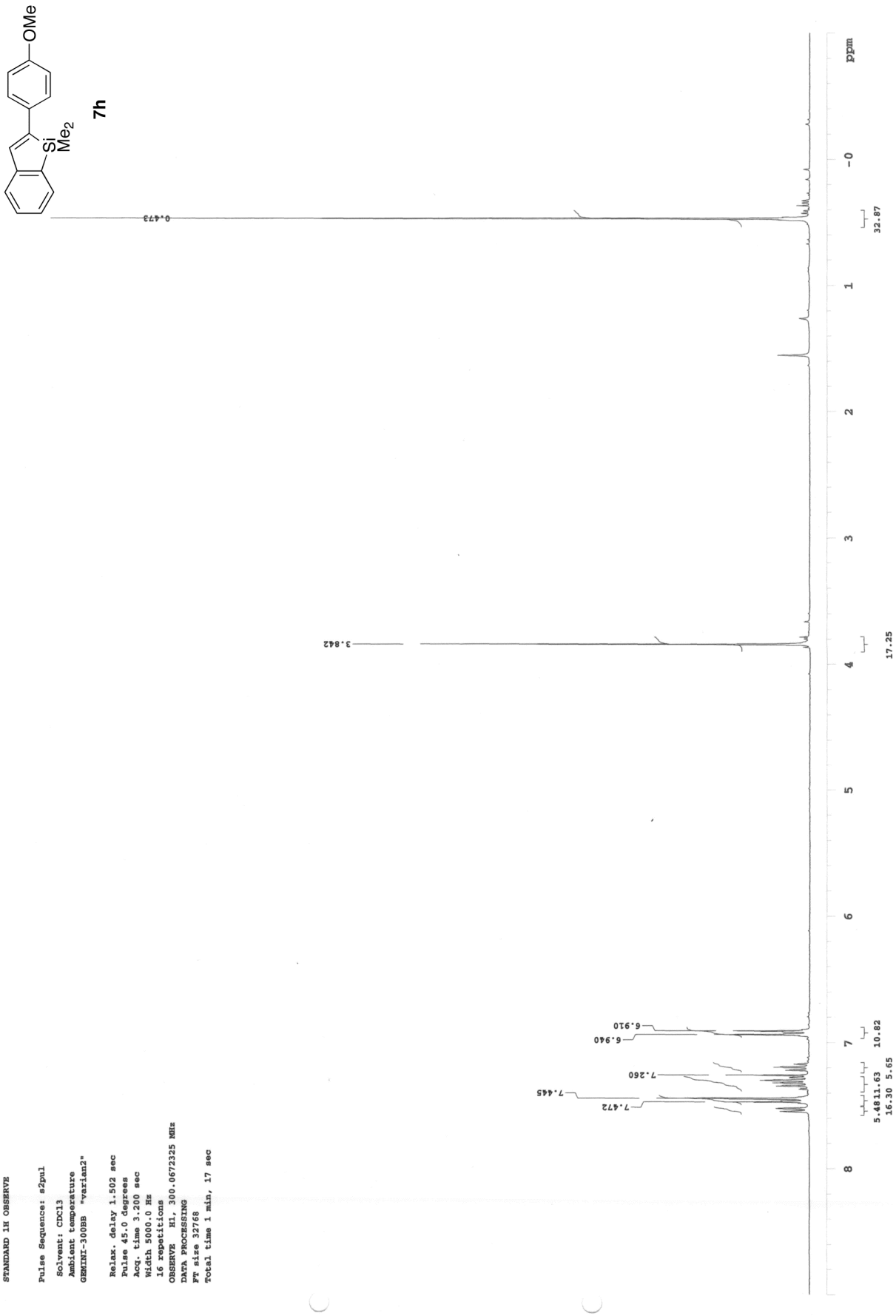

7f
Si
Me2
-2.988
21.435
76.585
77.000
77.430
124.172
124.357
126.493
128.860
129.967
131.627
138.083
138.390
139.036
140.957
145.491
148.980
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
108 repetitions
OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 71 hr, 13 min, 45 sec

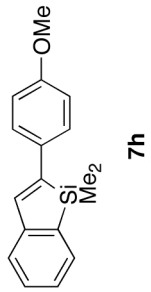

Ph
Si
Me2
7g
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.520
7.260
7.602
7.614
28.29
39.32 14.81
9.99 7.58
ppm


Ph
Si
Me2

7g

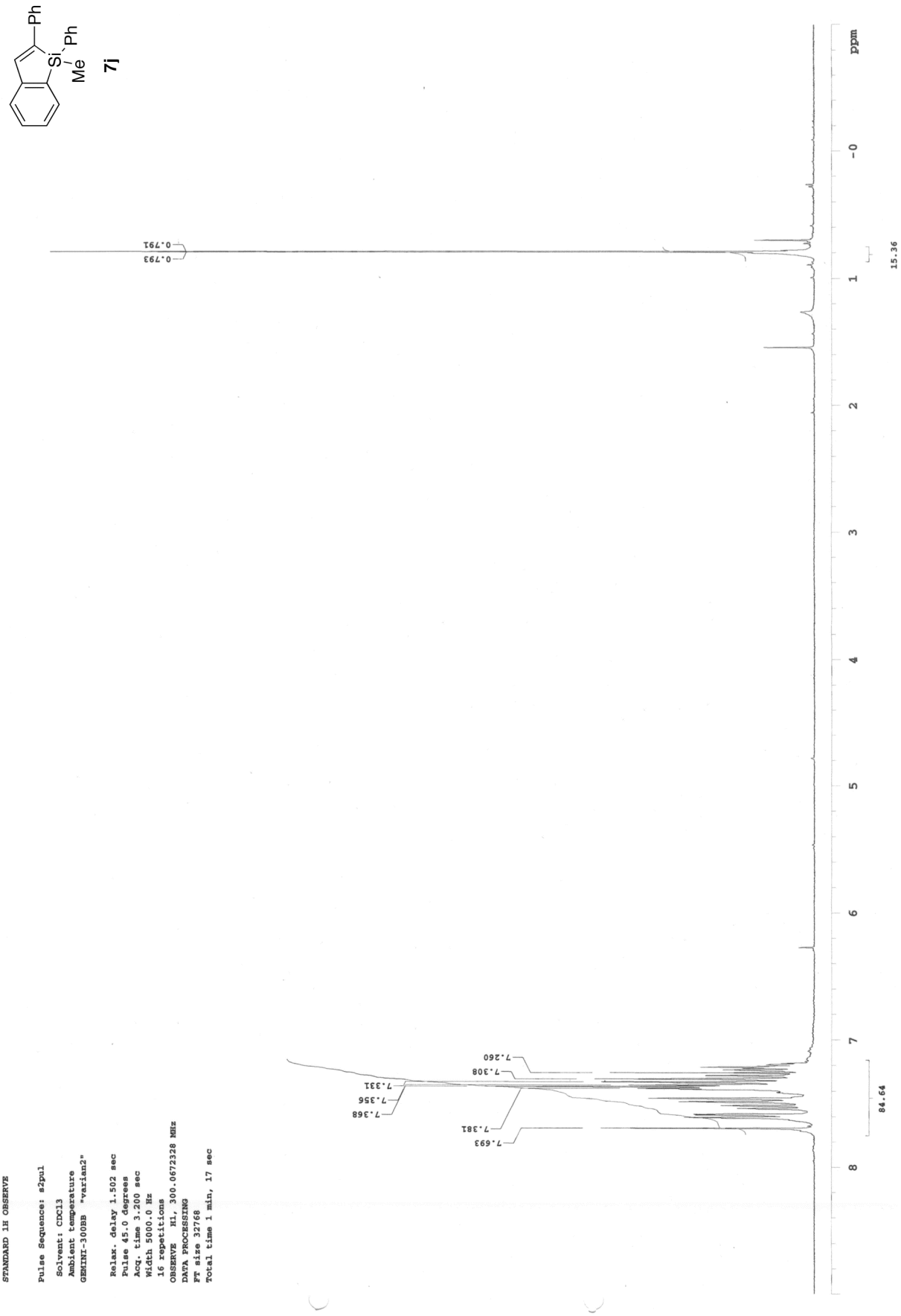
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
120 repetitions
OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec
-3.050
76.585
77.000
77.430
124.341
126.678
126.862
126.908
127.316
127.415
128.768
130.059
131.719
138.098
138.344
139.804
140.726
141.157
144.800
148.888
ppm
0
20
40
60
80
100
120
140
160
180
200

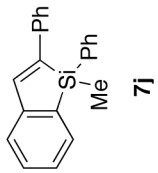

OMe
Si
Me2
7h
0.473
3.842
6.910
6.940
7.260
7.445
7.472
ppm
0
1
2
3
4
5
6
7
8
32.87
17.25
10.82
11.63
5.48
16.30
5.65
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


OMe
Si
Me2

7h

3.050
55.312
76.570
77.000
77.415
114.197
123.911
126.217
127.615
129.982
131.612
131.781
137.975
139.066
144.738
149.180
158.879
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
176 repetitions
OBSERVE C13, 75.4519686 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec


Ph
Si
Ph
Me
7j
0.793
0.791
7.260
7.308
7.331
7.356
7.368
7.381
7.693
ppm
15.36
84.64
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec

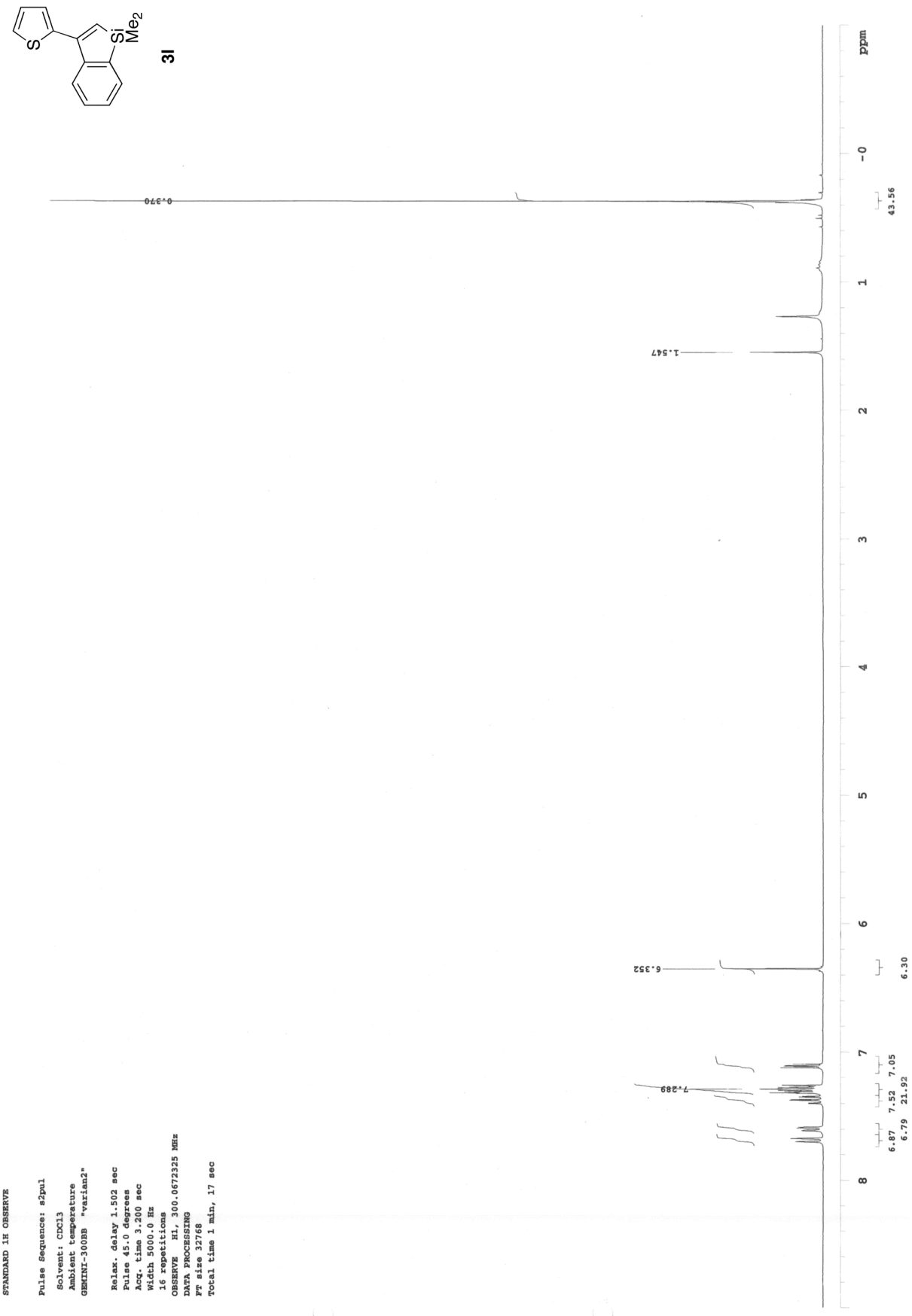


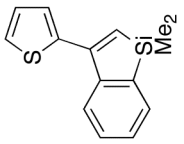
7j

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
84 repetitions
OBSERVE C13, 75.4519721 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

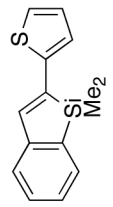

Si
Me2
S
3l
0.370
1.547
6.352
7.289
ppm
43.56
6.30
6.87
7.52
7.05
6.79
21.92
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec


S
Si
Me2

3l

-3.987
29.720
76.585
77.000
77.430
123.189
124.618
125.402
127.031
127.093
129.429
131.904
132.641
140.112
141.833
147.874
152.454
ppm
0
20
40
60
80
100
120
140
160
180
200
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
2084 repetitions
OBSERVE C13, 75.4519663 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 71 hr, 13 min, 45 sec


7l
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
0.475
0.473
1.541
7.258
7.260
7.376
ppm
40.05
5.66
33.20
6.90
14.19



7l

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
21940 repetitions
OBSERVE C13, 75.4519651 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 71 hr, 13 min, 45 sec