Supporting Information for
Iron-catalyzed aryl- and alkenyllithiation of alkynes and its application to benzosilole synthesis

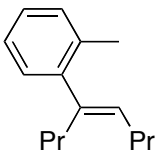
Eiji Shirakawa,* Seiji Masui, Rintaro Narui, Ryo Watabe, Daiji Ikeda, and Tamio Hayashi*

General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a nitrogen atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL JNM LA500 spectrometer (^{1}H, 500 MHz; ^{13}C, 125 MHz) using tetramethylsilane (^{1}H and ^{13}C) as an internal standard. GC spectra were taken on Hewlett-Packard HP6890. GC-MS spectra were taken on Shimazu GCMS-QP5050A. High-resolution mass spectra were obtained with a Bruker Daltonics microTOF-Q spectrometer (APCI and ESI). Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as an eluent. Unless otherwise noted, reagents were commercially available and used without further purification. Diethyl ether and tetrahydrofuran were purified by passing through an alumina/catalyst column system (GlassContour Co.). All the aryl- and alkenyllithiums were prepared from the corresponding halides by halogen–lithium exchange using *t*-BuLi (1.95 equiv) except otherwise noted.

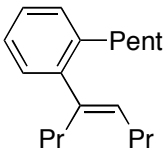
Preparation of 1-Cyclopentylidenepentyllithium (5c). A solution of 1-iodo-5-decyne[1] (1.12 g, 4.24 mmol) in diethyl ether (10 mL) was placed in a 20 mL Schlenk tube. To this solution was added dropwise over 3 min *t*-BuLi (1.59 M in pentane, 5.20 mL, 8.27 mmol) at –78 ˚C. The solution was stirred at –78 ˚C for 1 h, then at –20 ˚C for 0.5 h and at 30 ˚C for 3 h. 1-Cyclopentylidenepentyllithium was obtained in 92% yield (0.26 M, 15 mL).

Iron-Catalyzed Aryl- and Alkenyllithiation of Alkynes (Table 2): A General Procedure. A solution of $Fe(acac)_3$ (14.1 mg, 0.0400 mmol) in diethyl ether (4.0 mL) was placed in a 20 mL Schlenk tube and stirred for 10 min at the temperature specified in Table 2. To this solution was added successively an alkyne (0.80 mmol) and a solution (0.20–0.26 M in diethyl ether or tetrahydrofuran) of an organolithium (1.60 mmol). After the time specified in Table 2, methanol (0.50 mL) was added and stirring was continued for 5 min. A saturated NH_4Cl aqueous solution (2 mL) and H_2O (10 mL) were added and the resulting mixture was extracted with diethyl ether (10 mL x 3). The combined organic layer was washed with brine (6 mL) and dried over anhydrous magnesium sulfate. After evaporation of the solvent, the residue was subjected to SiO_2 chromatography (column or thin layer) to give the corresponding aryl- and alkenyllithiation products, whose isomer ratio was determined by GC, GC-MS, and/or ^{1}H NMR. Analytically pure sample was obtained by GPC purification.

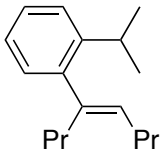
The spectral data of the aryl- and alkenyllithiation products in Tables 1 and 2 are as follows.



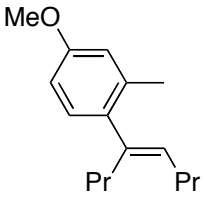
(*E*)-4-(2-Methylphenyl)-4-octene (4am).[2] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.5 Hz, 3 H), 0.96 (t, *J* = 7.5 Hz, 3 H), 1.29 (sext, *J* = 7.6 Hz, 2 H), 1.45 (sext, *J* = 7.4 Hz, 2 H), 2.16 (q, *J* = 7.3 Hz, 2 H), 2.26 (s, 3 H), 2.31 (t, *J* = 7.8 Hz, 2 H), 5.24 (t, *J* = 7.3 Hz, 1 H), 7.02–7.06 (m, 1 H), 7.08–7.17 (m, 3 H).



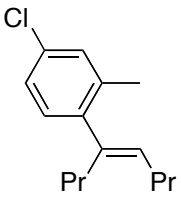
(*E*)-4-(2-Pentylphenyl)-4-octene (4cm). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.4 Hz, 3 H), 0.89 (t, *J* = 6.6 Hz, 3 H), 0.97 (t, *J* = 7.4 Hz, 3 H), 1.24–1.38 (m, 6 H), 1.45 (sext, *J* = 7.3 Hz, 2 H), 1.51–1.62 (m, 2 H), 2.16 (q, *J* = 7.4 Hz, 2 H), 2.30 (t, *J* = 7.8 Hz, 2 H), 2.56 (t, *J* = 8.1 Hz, 2 H), 5.25 (t, *J* = 7.4 Hz, 1 H), 7.02 (d, *J* = 7.7 Hz, 1 H), 7.10 (td, *J* = 7.1, 2.3 Hz, 1 H), 7.16 (td, *J* = 7.7, 1.5 Hz, 1 H), 7.18 (d, *J* = 7.7 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 14.2, 14.4, 21.6, 22.7, 23.2, 30.3, 31.6, 32.2, 33.1, 34.6, 125.1, 126.5, 128.9, 129.5, 130.0, 140.4, 140.6, 144.5. HRMS (APCI) Calcd for $C_{19}H_{30}$: M^+, 258.2342. Found: m/z 258.2340.



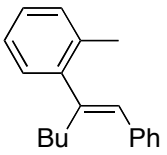
(*E*)-4-(2-Isopropylphenyl)-4-octene (4dm). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.3 Hz, 3 H), 0.96 (t, *J* = 7.3 Hz, 3 H), 1.19 (d, *J* = 7.0 Hz, 6 H), 1.30 (sext, *J* = 7.3 Hz, 2 H), 1.45 (sext, *J* = 7.3 Hz, 2 H), 2.16 (q, *J* = 7.3 Hz, 2 H), 2.30 (t, *J* = 7.8 Hz, 2 H), 3.13 (sept, *J* = 7.0 Hz, 1 H), 5.23 (t, *J* = 7.2 Hz, 1 H), 7.00 (d, *J* = 7.6 Hz, 1 H), 7.09 (t, *J* = 7.4 Hz, 1 H), 7.21 (t, *J* = 7.5 Hz, 1 H), 7.27 (d, *J* = 7.5 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 14.4, 21.5, 23.2, 24.6, 29.7, 30.3, 35.1, 125.0, 125.4, 126.8, 129.3, 129.8, 140.5, 143.7, 146.5. HRMS (APCI) Calcd for $C_{17}H_{26}$: M^+, 230.2029. Found: m/z 230.2034.



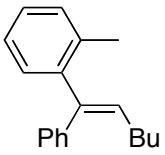
(*E*)-4-(4-Methoxy-2-methylphenyl)-4-octene (4em). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.3 Hz, 3 H), 0.97 (t, *J* = 7.3 Hz, 3 H), 1.29 (sext, *J* = 7.5 Hz, 2 H), 1.45 (sext, *J* = 7.3 Hz, 2 H), 2.15 (q, *J* = 7.3 Hz, 2 H), 2.26 (s, 3 H), 2.30 (t, *J* = 7.8 Hz, 2 H), 3.79 (s, 3 H), 5.23 (t, *J* = 7.3 Hz, 1 H), 6.67 (dd, *J* = 8.3, 2.6 Hz, 1 H), 6.72 (d, *J* = 2.6 Hz, 1 H), 6.97 (d, *J* = 8.3 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.3, 14.6, 20.6, 21.7, 23.5, 30.6, 34.5, 55.5, 110.8, 115.7, 130.31, 130.32, 137.1, 137.7, 140.5, 158.3. HRMS (APCI) Calcd for $C_{16}H_{24}O$: M^+, 232.1822. Found: m/z 232.1823.



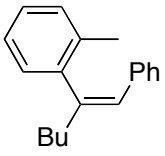
(*E*)-4-(4-Chloro-2-methylphenyl)-4-octene (4fm). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.4 Hz, 3 H), 0.96 (t, *J* = 7.4 Hz, 3 H), 1.26 (sext, *J* = 7.6 Hz, 2 H), 1.45 (sext, *J* = 7.3 Hz, 2 H), 2.15 (q, *J* = 7.3 Hz, 2 H), 2.23 (s, 3 H), 2.28 (t, *J* = 7.7 Hz, 2 H), 5.23 (t, *J* = 7.3 Hz, 1 H), 6.96 (d, *J* = 8.2 Hz, 1 H), 7.07 (dd, *J* = 8.2, 1.8 Hz, 1 H), 7.13 (d, *J* = 1.8 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 14.3, 21.0, 21.4, 23.1, 30.3, 33.9, 125.4, 129.9, 130.4, 130.6, 131.8, 137.4, 139.7, 143.2. HRMS (APCI) Calcd for $C_{15}H_{21}Cl$: M^+, 236.1326. Found: m/z 236.1333.



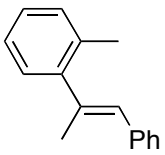
(*E*)-2-(2-Methylphenyl)-1-phenyl-1-hexene (4an). A colorless oil. ^{1}H NMR analysis was conducted for a 90:8:2 mixture of **4an**, a regioisomer (*E*) of **4an**, and the stereoisomer of **4an**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.85 (t, *J* = 7.2 Hz, 3 H), 1.31 (sext, *J* = 7.4 Hz, 2 H), 1.34–1.42 (m, 2 H), 2.38 (s, 3 H), 2.60 (t, *J* = 8.0 Hz, 2 H), 6.36 (s, 1 H), 7.17–7.30 (m, 5 H), 7.34–7.42 (m, 4 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 20.1, 23.1, 30.5, 32.6, 125.5, 126.6, 126.8, 128.4, 128.8, 128.9, 129.2, 130.3, 135.3, 138.2, 144.1, 144.3. HRMS (APCI) Calcd for $C_{19}H_{22}$: M^+, 250.1716. Found: m/z 250.1711. GC-MS (EI): m/s (%): 250 (80) [M^+], 207 (42), 193 (78), 179 (26), 129 (61), 115 (100), 105 (29), 91 (64).



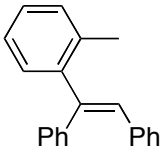
(*E*)-1-(2-Methylphenyl)-1-phenyl-1-hexene (Regioisomer of 4an). ^{1}H NMR analysis was conducted for a 90:8:2 mixture of **4an**, a regioisomer (*E*) of **4an**, and the stereoisomer of **4an**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.91 (t, *J* = 7.3 Hz, 3 H), 2.07 (s, 3 H), 2.34 (q, *J* = 7.4 Hz, 2 H), 5.70 (t, *J* = 7.5 Hz, 1 H). Other peaks were not distinguished due to overlap. GC-MS (EI): m/s (%): 250 (58) [M^+], 207 (95), 192 (58), 179 (22), 129 (43), 115 (80), 105 (29), 91 (60), 77 (30).



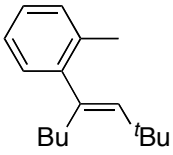
(*Z*)-2-(2-Methylphenyl)-1-phenyl-1-hexene (Stereoisomer of 4an). ^{1}H NMR analysis was conducted for a 90:8:2 mixture of **4an**, a regioisomer (*E*) of **4an**, and the stereoisomer of **4an**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.47 (s, 1 H). Other peaks were not distinguished due to overlap. GC-MS (EI): m/s (%): 251 (47) [MH^+], 207 (100), 193 (58), 179 (22), 129 (43), 115 (80), 105 (29), 91 (60), 77 (30).



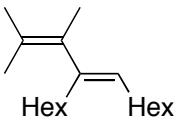
(*E*)-2-(2-Methylphenyl)-1-phenylpropene (4ao).[3] A yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.22 (s, 3 H), 2.39 (s, 3 H), 6.41 (s, 1 H), 7.20–7.31 (m, 5 H), 7.38–7.43 (m, 4 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.0, 20.1, 125.8, 126.6, 127.0, 128.2, 128.3, 129.1, 129.3, 130.4, 134.9, 138.2, 139.2, 146.0. HRMS (APCI) Calcd for $C_{16}H_{16}$: M^+, 208.1247. Found: m/z 208.1243.



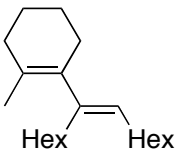
(*E*)-2-(2-Methylphenyl)-1,2-diphenylethene (4ap).[3] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.14 (s, 3 H), 6.63 (s, 1 H), 7.12–7.35 (m, 14 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.6, 125.8, 126.9, 127.2, 127.6, 128.2, 128.3, 129.6, 130.0, 130.26, 130.33, 130.6, 136.4, 137.6, 140.4, 143.1, 144.2. HRMS (APCI) Calcd for $C_{21}H_{18}$: M^+, 270.1403. Found: m/z 270.1402.



(*E*)-2,2-Dimethyl-4-(2-methylphenyl)-3-octene (4aq). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.84 (t, *J* = 6.8 Hz, 3 H), 1.20 (s, 9 H), 1.20–1.34 (m, 4 H), 2.26 (s, 3 H), 2.43 (t, *J* = 7.9 Hz, 2 H), 5.20 (s, 1 H), 7.01–7.04 (m, 1 H), 7.08–7.16 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 19.9, 23.3, 30.8, 31.6, 32.4, 33.0, 125.2, 126.2, 129.1, 130.0, 135.3, 139.7, 140.1, 145.7. HRMS (APCI) Calcd for $C_{17}H_{26}$: M^+, 230.2029. Found: m/z 230.2029.

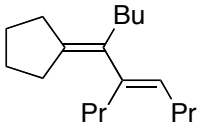


(*E*)-4-Hexyl-2,3-dimethyl-2,4-undecadiene (6ar). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.1 Hz, 3 H), 0.89 (t, *J* = 7.1 Hz, 3 H), 1.22–1.38 (m, 16 H), 1.65 (s, 3 H), 1.66 (s, 6 H), 2.02 (q, *J* = 7.3 Hz, 2 H), 2.07 (t, *J* = 7.6 Hz, 2 H), 5.00 (t, *J* = 7.2 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 14.3, 18.7, 20.2, 22.1, 22.92, 22.94, 27.9, 28.5, 29.3, 29.9, 30.0, 30.2, 32.08, 32.13, 125.2, 127.0, 132.5, 142.7. HRMS (APCI) Calcd for $C_{19}H_{36}$: M^+, 264.2812. Found: m/z 264.2810.

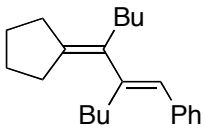


(*E*)-7-(2-Methyl-1-cyclohexenyl)-7-tetradecene (6br). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.882 (t, *J* = 7.1 Hz, 3 H), 0.884 (t, *J* = 7.1 Hz, 3 H), 1.20–1.38 (m, 16 H), 1.55–1.62 (m, 4 H), 1.58 (s, 3 H), 1.93–1.99 (m, 4 H), 2.03 (q, *J* = 7.2 Hz, 2 H), 2.07 (t, *J* = 7.2 Hz, 2 H), 5.02 (t, *J* = 7.3 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 14.3, 20.9, 22.8,

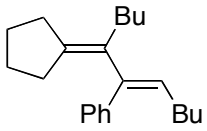
22.9, 23.5, 23.6, 27.9, 28.5, 29.2, 29.8, 29.9, 30.1, 30.3, 31.6, 31.99, 32.02, 126.7, 127.2, 134.9, 141.9. HRMS (APCI) Calcd for $C_{21}H_{38}$: M^+, 290.2968. Found: m/z 290.2976.



(*E*)-6-Cyclopentylidene-5-propyl-4-decene (6cm). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.0 Hz, 3 H), 0.88 (t, *J* = 7.4 Hz, 3 H), 0.91 (t, *J* = 7.4 Hz, 3 H), 1.21–1.33 (m, 6 H), 1.38 (sext, *J* = 7.3 Hz, 2 H), 1.52 (quint, *J* = 6.8 Hz, 2 H), 1.63 (quint, *J* = 6.9 Hz, 2 H), 1.97–2.06 (m, 6 H), 2.22 (t, *J* = 7.2 Hz, 4 H), 5.08 (t, *J* = 7.3 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 14.3, 14.6, 21.9, 22.8, 23.3, 26.6, 27.0, 30.0, 30.1, 30.5, 31.5, 31.7, 32.4, 128.2, 134.7, 137.7, 141.1. HRMS (APCI) Calcd for $C_{18}H_{32}$: M^+, 248.2499. Found: m/z 248.2505.

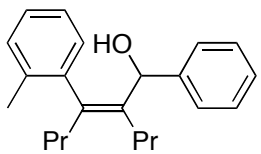


(*E*)-3-Cyclopentylidene-2-butyl-1-phenyl-1-heptene (6cn). A colorless oil. ^{1}H NMR analysis was conducted for a 94:6 mixture of **6cn** and a regioisomer (*E*) of **6cn**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.1 Hz, 3 H), 0.90 (t, *J* = 7.0 Hz, 3 H), 1.25–1.40 (m, 8 H), 1.57 (quint, *J* = 6.8 Hz, 2 H), 1.68 (quint, *J* = 7.0 Hz, 2 H), 2.11 (t, *J* = 6.8 Hz, 2 H), 2.25–2.35 (m, 6 H), 6.20 (s, 1 H), 7.19 (t, *J* = 7.3 Hz, 1 H), 7.25 (d, *J* = 7.9 Hz, 2 H), 7.32 (t, *J* = 7.7 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 14.3, 22.8, 23.2, 26.5, 27.1, 29.6, 30.2, 30.6, 30.7, 31.6, 32.5, 126.0, 127.7, 128.2, 128.8, 134.4, 138.8, 139.0, 145.0. HRMS (APCI) Calcd for $C_{22}H_{32}$: M^+, 296.2499. Found: m/z 296.2502. GC-MS (EI): m/z (%): 296 (17) [M^+], 267 (2), 239 (78), 225 (2), 183 (92), 169 (32), 155 (20), 141 (38), 129 (28), 115 (20), 91 (100), 79 (23).



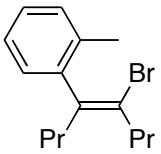
(*E*)-7-Cyclopentylidene-6-phenyl-5-undecene (Regioisomer of 6cn). ^{1}H NMR analysis was conducted for a 94:6 mixture of **6cn** and a regioisomer (*E*) of **6cn**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.836 (t, *J* = 7.1 Hz, 3 H), 0.843 (t, *J* = 7.2 Hz, 3 H), 5.44 (t, *J* = 7.3 Hz, 1 H). Other peaks were not distinguished due to overlap. GC-MS (EI): m/s (%): 296 (10) [M^+], 225 (48), 169 (100), 141 (24), 91 (40).

Aryllithiation Followed by Reaction with Electrophiles (Scheme 3): A General Procedure. A solution of $Fe(acac)_3$ (7.1 mg, 0.020 mmol) in diethyl ether (2.0 mL) was placed in a 10 mL Schlenk tube and stirred at 0 ˚C for 10 min. To this solution was added successively 4-octyne (44.1 mg, 0.400 mmol) and a solution of *o*-tolyllithium (0.23 M in diethyl ether, 3.5 mL, 0.80 mmol), and the mixture was stirred at 30 ˚C for 1.5 h. An electrophile (0.88 mmol) was added at 0 ˚C and the mixture was stirred at the time and temperature specified in Scheme 3. A saturated NH_4Cl aqueous solution (2 mL) and H_2O (10 mL) were added and the resulting mixture was extracted with diethyl ether (10 mL x 3). The combined organic layer was washed with brine (6 mL) and dried over anhydrous magnesium sulfate. After evaporation of the solvent, the residue was subjected to SiO_2 chromatography to give the corresponding products. Analytically pure sample was obtained by GPC purification.



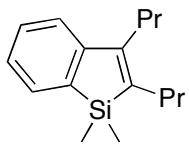
(*Z*)-3-(2-Methylphenyl)-1-phenyl-2-propyl-2-hexen-1-ol (9a). A yellow oil. Observed as two conformers in 58/42 ratio in ^{1}H NMR. The underlined peaks could not be characterized to each conformer. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87/0.79 (t, *J* = 7.4/7.2 Hz, 3 H), 0.90/0.89 (t, *J* = 7.4/7.4 Hz, 3 H), 1.13–1.46 (m, 4 H), 1.92–2.10

(m, 3 H), 2.33/2.26 (s, 3 H), 2.36–2.51 (m, 1 H), 5.17/5.16 (d, J = 3.8/3.8 Hz, 1 H), 7.02–7.05/7.06–7.08 (m, 1 H), 7.15–7.31 (m, 8 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.55, 14.64, 14.9, 15.0, 19.8, 19.9, 21.2, 21.3, 24.1, 24.5, 29.57, 29.64, 35.5, 36.4, 74.1, 74.4, 125.49, 125.54, 125.7, 126.0, 126.7, 126.8, 126.9, 127.0, 128.0, 128.1, 129.5, 129.7, 130.2, 130.5, 130.49, 135.54, 137.4, 138.1, 139.5, 141.0, 141.6, 141.8, 143.0, 143.5. HRMS (ESI) Calcd for $C_{22}H_{28}ONa$: $[M+Na]^+$, 331.2032. Found: m/z 331.2038.

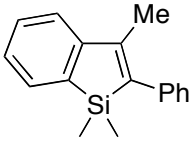


(*Z*)-4-Bromo-5-(2-methylphenyl)-4-octene (9b). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, J = 7.4 Hz, 3 H), 1.01 (t, J = 7.4 Hz, 3 H), 1.28–1.48 (m, 2 H), 1.70 (sext, J = 7.4 Hz, 2 H), 2.21 (ddd, J = 16.4, 11.8, 6.5 Hz, 1 H), 2.23 (s, 3 H), 2.44 (ddd, J = 13.6, 10.2, 6.2 Hz, 1 H), 2.58 (dt, J = 14.2, 7.1 Hz, 1 H), 2.69 (dt, J = 14.6, 7.3 Hz, 1 H), 7.02 (dd, J = 6.3, 1.7 Hz, 1 H), 7.15–7.21 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.4, 14.2, 19.4, 21.2, 22.0, 36.9, 39.0, 125.6, 125.7, 127.0, 128.4, 130.1, 135.0, 140.3, 143.5. HRMS (APCI) Calcd for $C_{15}H_{21}Br$: M^+, 280.0821, 282.0802. Found: m/z 280.0822, 282.0805.

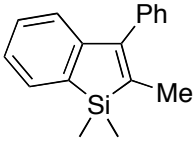
Iron-Catalyzed Benzosilole Synthesis through Aryllithiation-Cyclization (Table 3 and Scheme 4): A General Procedure. A solution of $Fe(acac)_3$ (7.1 mg, 0.020 mmol) in diethyl ether (2.0 mL) was placed in a 10 mL Schlenk tube and stirred for 10 min at the temperature specified in Table 3 and Scheme 4. To this solution was added successively an alkyne (0.40 mmol) and a solution (0.20–0.26 M in diethyl ether) of an *o*-(trimethylsilyl)phenyllithium (0.64 mmol). After the time specified in Table 3 and Scheme 4, methanol (0.50 mL) was added and stirring was continued for 5 min. A saturated NH_4Cl aqueous solution (2 mL) and H_2O (10 mL) were added and the resulting mixture was extracted with diethyl ether (10 mL x 3). The combined organic layer was washed with brine (6 mL) and dried over anhydrous magnesium sulfate. After evaporation of the solvent, the residue was subjected to SiO_2 chromatography to give the corresponding benzosiloles, whose isomer ratio was determined by GC, GC-MS, and/or ^{1}H NMR. Analytically pure sample was obtained by GPC purification.



1,1-Dimethyl-2,3-dipropyl-1*H*-benzo[*b*]silole (11am).[4] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.29 (s, 6 H), 0.96 (t, J = 7.3 Hz, 3 H), 0.99 (t, J = 7.4 Hz, 3 H), 1.52 (sext, J = 7.5, 2 H), 1.53 (sext, J = 7.4, 2 H), 2.38 (t, J = 8.0 Hz, 2 H), 2.51 (t, J = 7.8 Hz, 2 H), 7.15 (t, J = 7.1 Hz, 1 H), 7.27 (d, J = 8.8 Hz, 1 H), 7.32 (t, J = 7.9 Hz, 1 H), 7.48 (d, J = 7.0 Hz, 1 H).

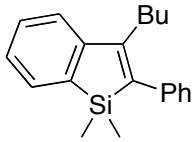


1,1,3-Trimethyl-2-phenyl-1*H*-benzo[*b*]silole (11ao).[4] A colorless oil. ^{1}H NMR analysis was conducted for a 97:3 mixture of **11ao** and its regioisomer (**11ao'**).^{1}H NMR (500 MHz, $CDCl_3$) δ 0.34 (s, 6 H), 2.13 (s, 3 H), 7.16 (dd, J = 8.4, 1.4 Hz, 2 H), 7.22 (tt, J = 7.5, 1.2 Hz, 1 H), 7.25 (td, J = 6.9, 1.8 Hz, 1 H), 7.36 (tt, J = 7.6, 1.7 Hz, 2 H), 7.40–7.42 (m, 2 H), 7.56 (dt, J = 6.9, 1.1 Hz, 1 H). GC-MS (EI): m/s (%): 250 (72) [M^+], 235 (100).

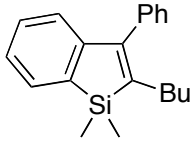


1,1,2-Trimethyl-3-phenyl-1*H*-benzo[*b*]silole (11ao').[4] A colorless oil. ^{1}H NMR analysis was conducted for a 97:3 mixture of **11ao** and **11ao'**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.35 (s, 6 H), 1.83 (s, 3 H). Other peaks were not distinguished due to overlap. GC-MS (EI): m/s

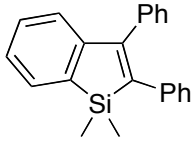
(%): 250 (100) [M$^+$], 236 (69), 135 (68).



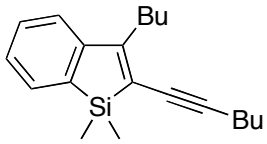
3-Butyl-1,1-dimethyl-2-phenyl-1*H*-benzo[*b*]silole (11an).[4] A colorless oil. ^{1}H NMR analysis was conducted for a 94:6 mixture of **11an** and its regioisomer (**11an'**). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.32 (s, 6 H), 0.83 (t, *J* = 7.4 Hz, 3 H), 1.31 (sext, *J* = 7.4, 2 H), 1.49–1.58 (m, 2 H), 2.51 (t, *J* = 8.0 Hz, 2 H), 7.11 (dd, *J* = 8.2, 1.4 Hz, 2 H), 7.22 (t, *J* = 7.6 Hz, 1 H), 7.23 (td, *J* = 6.9, 1.8 Hz, 1 H), 7.35 (t, *J* = 6.3 Hz, 2 H), 7.39 (td, *J* = 6.7, 1.3 Hz, 1 H), 7.41 (d, *J* = 7.6 Hz, 1 H), 7.56 (d, *J* = 7.1 Hz, 1 H). GC-MS (EI): m/s (%): 292 (90) [M$^+$], 277 (35), 250 (100), 233 (49).



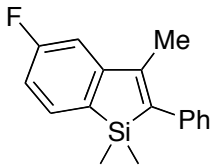
2-Butyl-1,1-dimethyl-3-phenyl-1*H*-benzo[*b*]silole (11an').[4] A colorless oil. ^{1}H NMR analysis was conducted for a 94:6 mixture of **11an** and **11an'**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.39 (s, 6 H), 0.81 (t, *J* = 7.3 Hz, 3 H), 2.23 (t, *J* = 7.5 Hz, 2 H). Other peaks were not distinguished due to overlap. GC-MS (EI): m/s (%): 292 (100) [M$^+$], 249 (68), 233 (69), 221 (38), 191 (24), 165 (21), 135 (65).



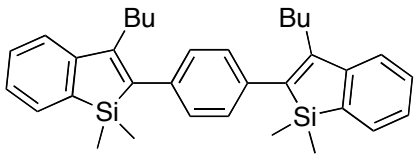
1,1-Dimethyl-2,3-diphenyl-1*H*-benzo[*b*]silole (11ap).[4] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.47 (s, 6 H), 6.97 (dd, *J* = 8.4, 1.3 Hz, 2 H), 7.06 (t, *J* = 6.9 Hz, 2 H), 7.13 (t, *J* = 7.4 Hz, 2 H), 7.19 (dt, *J* = 6.7, 1.5 Hz, 2 H), 7.22–7.36 (m, 5 H), 7.61 (d, *J* = 8.8 Hz, 1 H).



3-Butyl-2-(1-hexynyl)-1,1-dimethyl-1*H*-benzo[*b*]silole (11as). An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.33 (s, 6 H), 0.95 (t, *J* = 7.4 Hz, 3 H), 0.96 (t, *J* = 7.3 Hz, 3 H), 1.39–1.62 (m, 8 H), 2.48 (t, *J* = 6.9 Hz, 2 H), 2.75 (t, *J* = 7.8 Hz, 2 H), 7.22 (ddd, *J* = 6.9, 5.5, 3.0 Hz, 1 H), 7.32–7.38 (m, 2 H), 7.48 (dt, *J* = 6.8, 1.0 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ –4.3, 13.8, 14.2, 20.1, 22.1, 23.1, 29.8, 31.0, 31.5, 79.5, 100.8, 122.0, 123.2, 127.0, 129.9, 131.8, 138.1, 149.0, 162.6. HRMS (APCI) Calcd for $C_{20}H_{28}Si$: M$^+$, 296.1955. Found: m/z 296.1951.

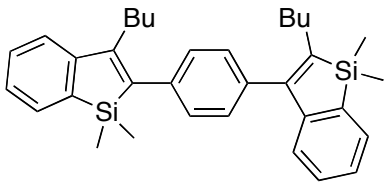


5-Fluoro-1,1,3-trimethyl-2-phenyl-1*H*-benzo[*b*]silole (11bo). A colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.33 (s, 6 H), 2.09 (s, 3 H), 6.93 (ddd, *J* = 9.7, 7.8, 2.3 Hz, 1 H), 7.09 (dd, *J* = 10.6, 2.3 Hz, 1 H), 7.15 (d, *J* = 6.9 Hz, 2 H), 7.24 (t, *J* = 7.4 Hz, 1 H), 7.37 (t, *J* = 7.8 Hz, 2 H), 7.48 (dd, *J* = 7.7, 6.4 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ –3.8, 14.4, 109.8 (d, $^2J_{C–F}$ = 21.6 Hz), 113.0 (d, $^2J_{C–F}$ = 20.6 Hz), 126.0, 128.0, 128.5, 132.6 (d, $^3J_{C–F}$ = 8.2 Hz), 133.2 (d, $^4J_{C–F}$ = 3.6 Hz), 140.8, 144.9, 147.4 (d, $^4J_{C–F}$ = 3.1 Hz), 153.8 (d, $^3J_{C–F}$ = 7.2 Hz), 165.3 (d, $^1J_{C–F}$ = 245.2 Hz). HRMS (APCI) Calcd for $C_{17}H_{17}FSi$: M$^+$, 268.1078. Found: m/z 268.1075.

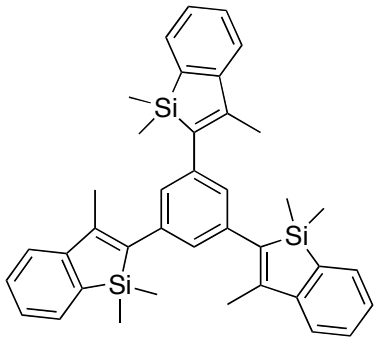


1,4-Bis[2-(1,1-dimethyl-3-butyl-1*H*-benzo[*b*]silolyl)]benzene (13am). A colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.37 (s, 12 H), 0.85 (t, *J* = 7.4 Hz, 6 H), 1.35 (sext, *J* = 7.3 Hz, 4 H), 1.58 (quint, *J* = 7.7 Hz, 4 H), 2.59 (t, *J* = 8.0 Hz, 4 H), 7.12 (s, 4 H), 7.24 (td, *J* = 7.1, 1.5 Hz, 2 H), 7.40 (t, *J* = 7.7 Hz, 2 H), 7.42 (d, *J* = 7.1 Hz, 2 H), 7.58 (d, *J* = 6.9 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ –3.7,

13.9, 23.0, 27.7, 31.6, 122.2, 126.4, 127.7, 129.9, 131.7, 138.6, 139.0, 142.7, 150.1, 153.1. HRMS (APCI) Calcd for $C_{34}H_{43}Si_2$: MH^+, 507.2898. Found: m/z 507.2883.



1-[2-(1,1-Dimethyl-3-butyl-1*H*-benzo[*b*]silolyl)]-4-[3-(1,1-dimethyl-2-butyl-1*H*-benzo[*b*]silolyl)]benzene (13am'). A colorless solid. ^{1}H NMR analysis was conducted for a 84:16 mixture of **13am'** and **13am**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.38 (s, 6 H), 0.41 (s, 6 H), 0.83 (t, *J* = 7.5 Hz, 3 H), 0.84 (t, *J* = 7.4 Hz, 3 H), 1.27 (sext, *J* = 7.3 Hz, 2 H), 1.34 (sext, *J* = 7.3 Hz, 2 H), 1.45 (quint, *J* = 7.6 Hz, 2 H), 1.58 (quint, *J* = 7.7 Hz, 2 H), 2.30 (t, *J* = 7.9 Hz, 2 H), 2.59 (t, *J* = 8.0 Hz, 2 H), 7.18 (s, 4 H), 7.22–7.45 (m, 6 H), 7.52–7.63 (m, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ –3.8, –3.3, 13.9, 14.0, 22.9, 23.0, 27.7, 30.1, 31.6, 32.8, 122.2, 123.2, 126.0, 126.4, 127.6, 127.8, 129.2, 129.7, 129.9, 131.5, 135.5, 138.0, 139.0, 140.0, 142.8, 144.6, 150.0, 151.4, 153.1, 153.3. HRMS (APCI) Calcd for $C_{34}H_{43}Si_2$: MH^+, 506.2898. Found: m/z 507.2990.



1,3,5-Tris[2-(1,1,3-trimethyl-1*H*-benzo[*b*]silolyl)]benzene (13an). A colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.38 (s, 18 H), 2.23 (s, 9 H), 6.85 (s, 3 H), 7.26 (td, *J* = 6.7, 2.1 Hz, 3 H), 7.41 (td, *J* = 7.7, 1.2 Hz, 3 H), 7.43 (d, *J* = 6.5 Hz, 3 H), 7.43 (d, *J* = 6.9 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ –3.6, 14.8, 121.9, 125.3, 126.6, 130.0, 131.5, 138.3, 140.8, 142.9, 148.2, 151.1. HRMS (APCI) Calcd for $C_{39}H_{42}Si_3$: M^+, 594.2667. Found: m/z 595.2660.

References

1 J. K. Crandall and W. J. Michaely, *J. Org. Chem.*, 1984, **49**, 4244–4248.
2 E. Genin, V. Michelet and J.-P. Genêt, *J. Organomet. Chem.*, 2004, **689**, 3820–3830.
3 X. Xu, J. Chen, W. Gao, H. Wu, J. Ding and W. Su, *Tetrahedron*, 2010, **66**, 2433–2438.
4 M. Tobisu, M. Onoe, Y. Kita and N. Chatani, *J. Am. Chem. Soc.*, 2009, **131**, 7506–7507.

NMR Spectra of the Products

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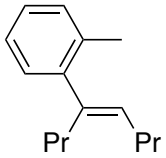
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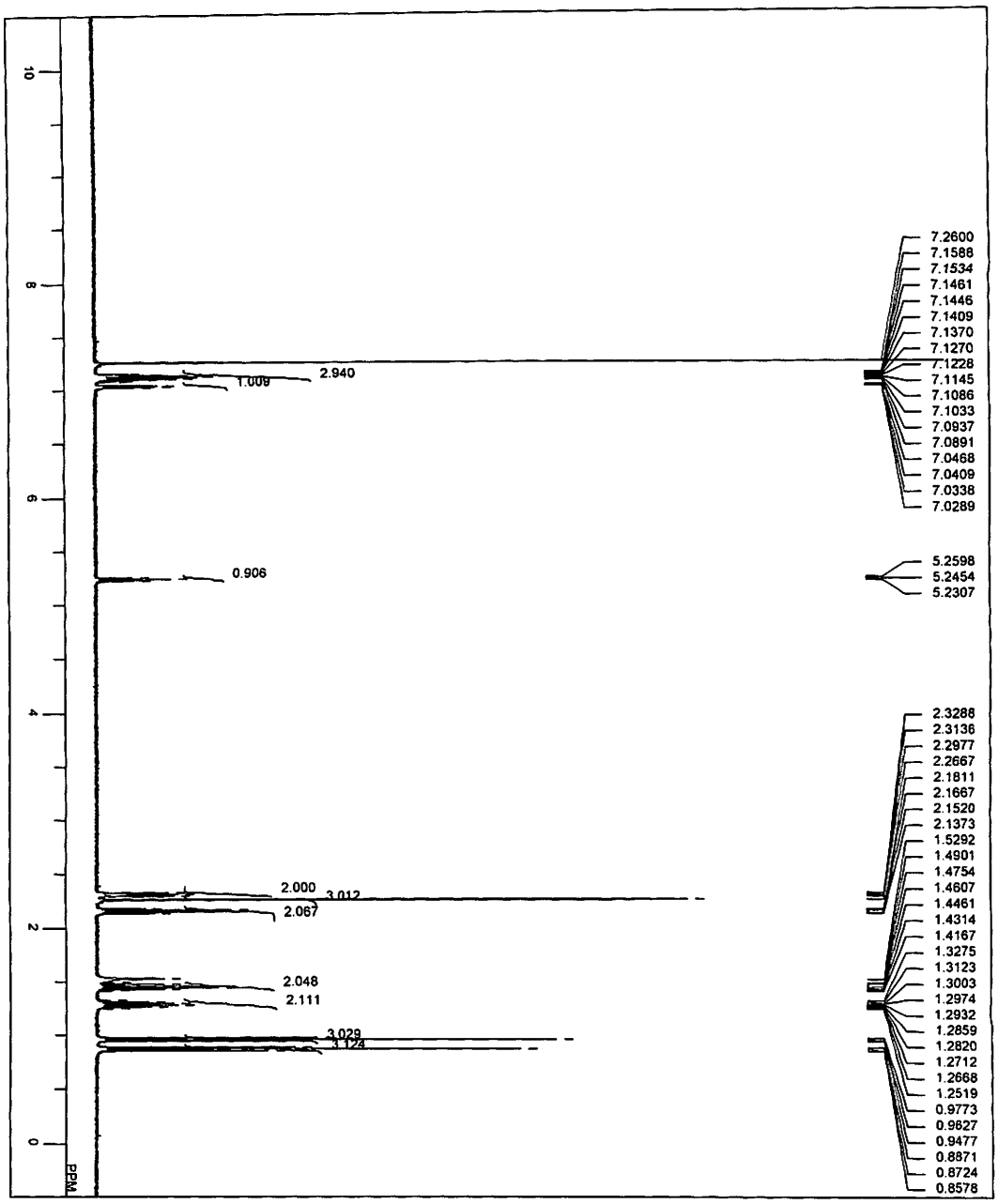
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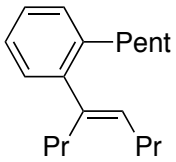
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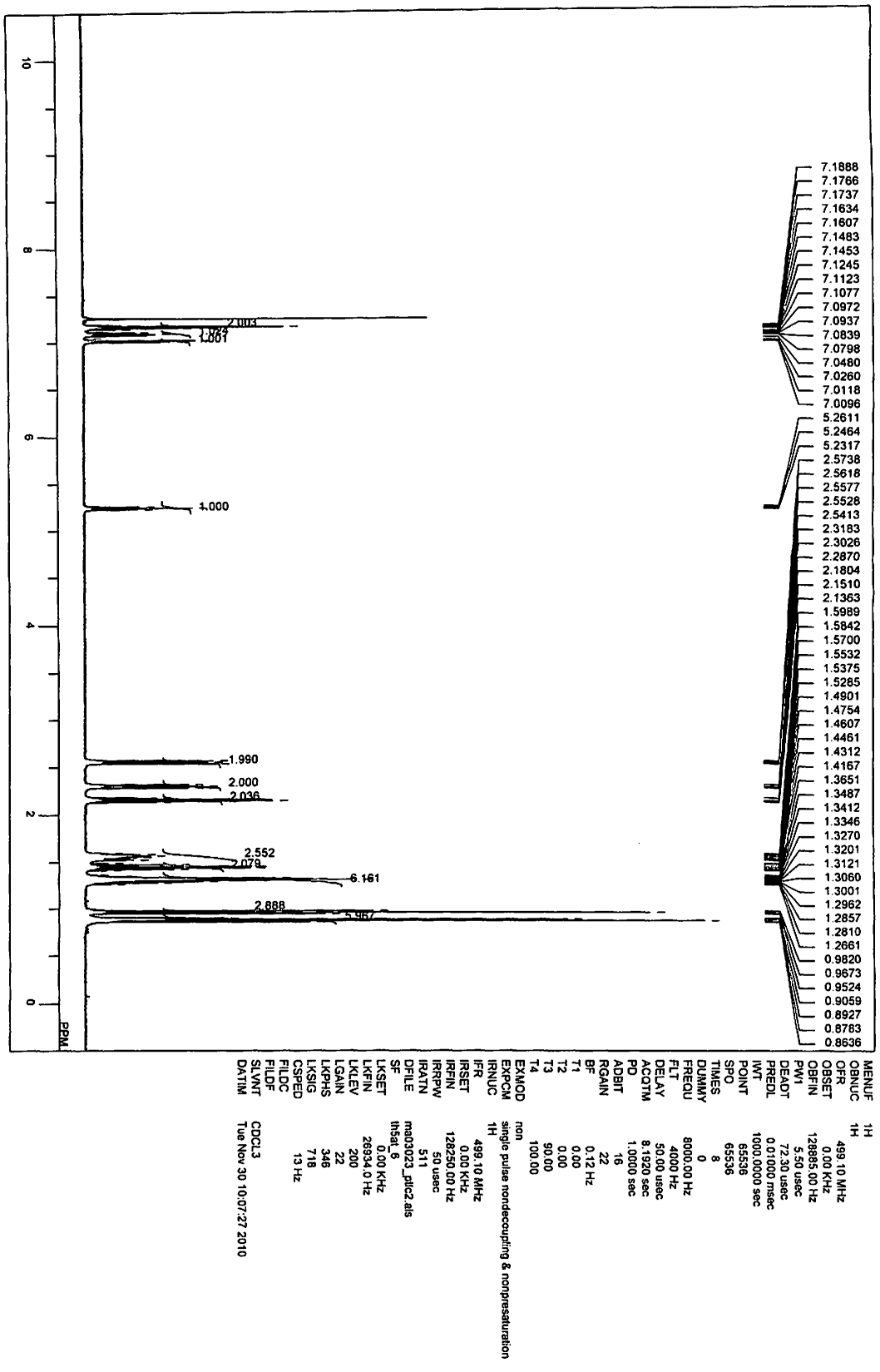
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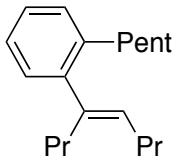
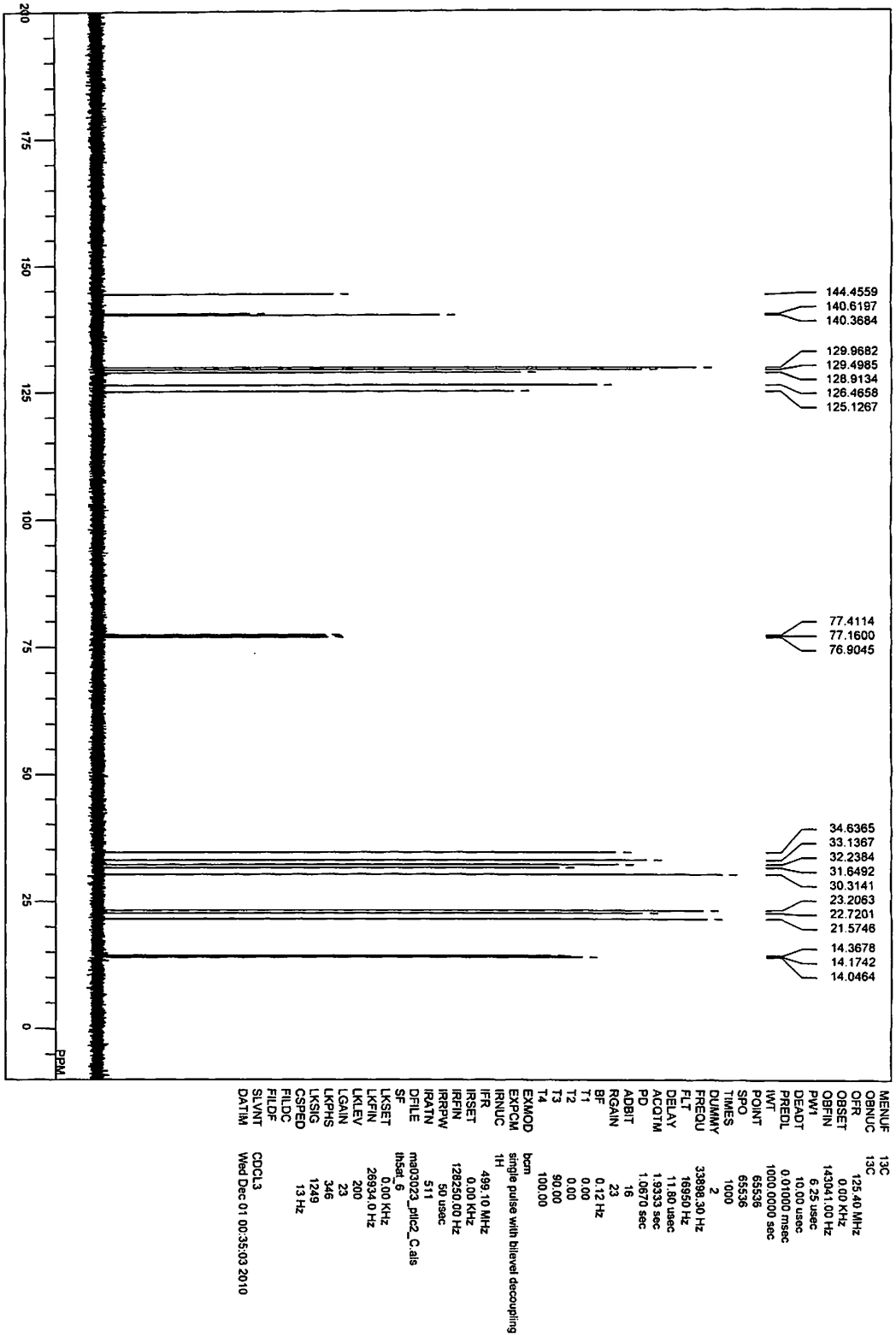
DATIM Tue Nov 30 10:07:27 2010

4cm


Pent
Pr
Pr

^{13}C NMR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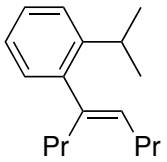
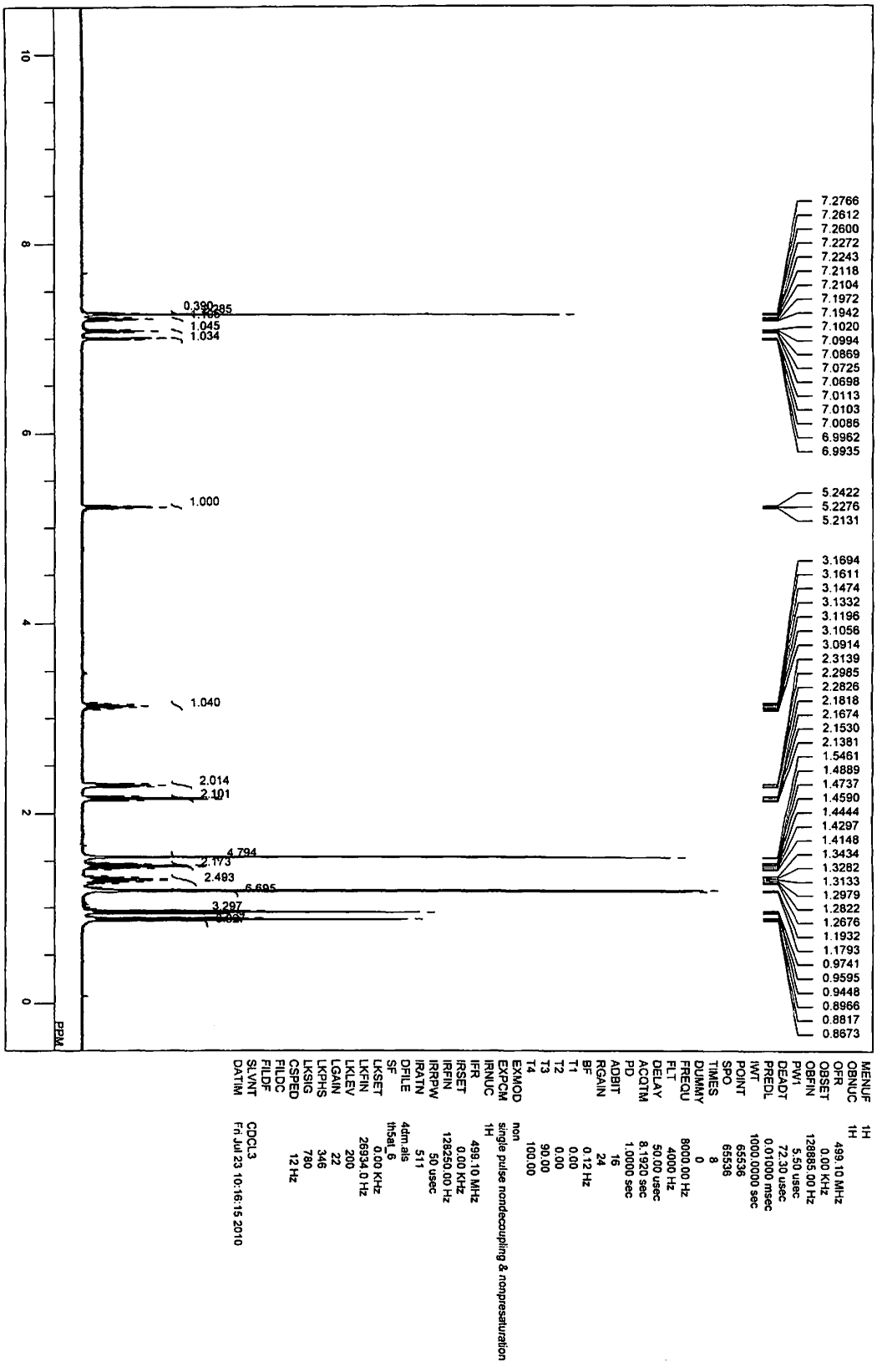
140.3684
129.9682
129.4985
128.9134
126.4658
125.1267
77.4114
77.1600
76.9045
34.6365
33.1367
32.2384
31.6492
30.3141
23.2063
22.7201
21.5746
14.3678
14.1742
14.0464
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 14304.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 1000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma03023_ptlc2_C.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 346
LKSIG 1249
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Wed Dec 01 00:35:03 2010

4dm


Pr
Pr

^{1}H NMR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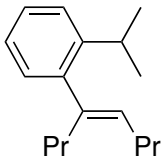
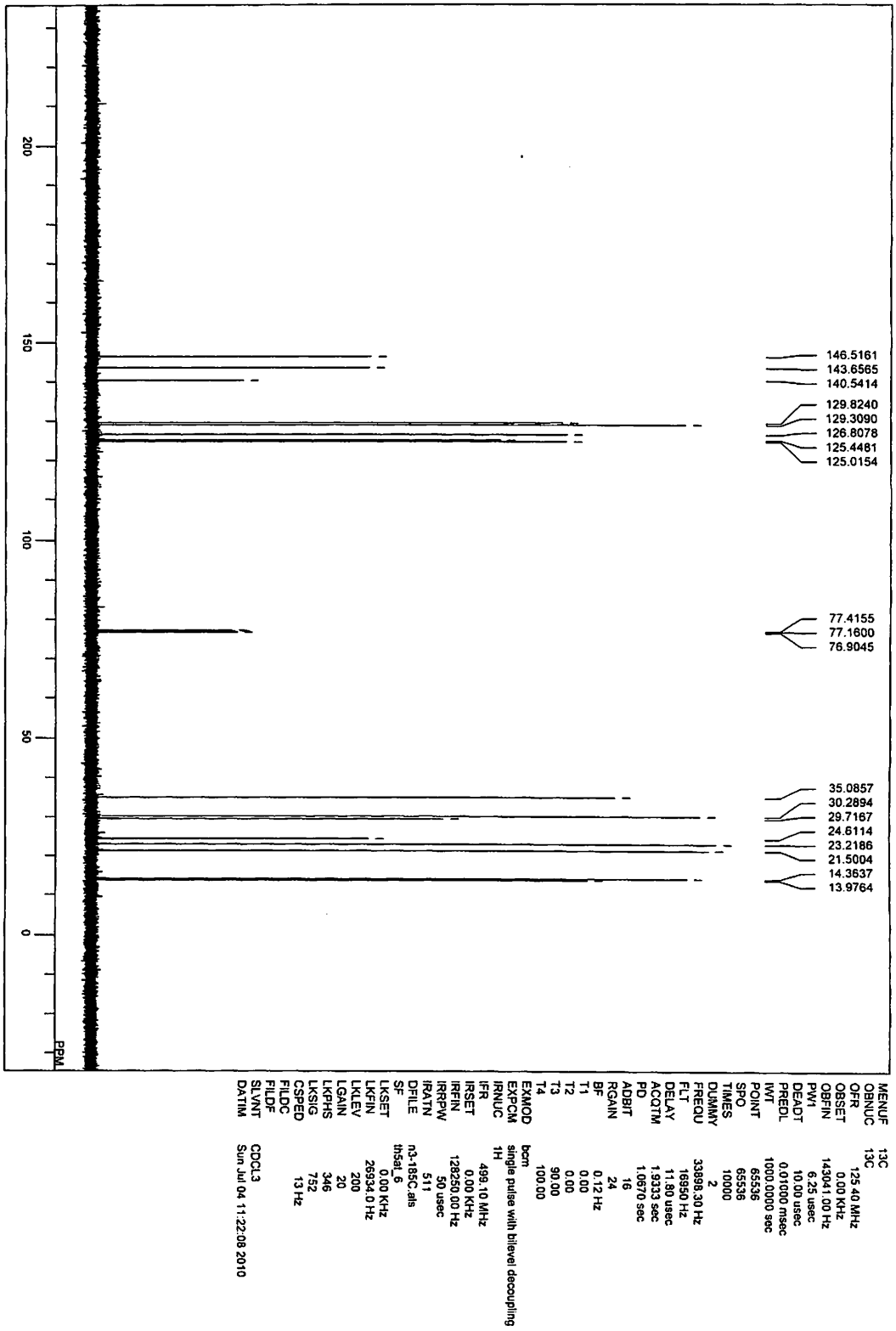
7.2600
7.2272
7.2243
7.2118
7.2104
7.1972
7.1942
7.1020
7.0994
7.0869
7.0725
7.0698
7.0113
7.0103
7.0086
6.9962
6.9935
5.2422
5.2276
5.2131
3.1694
3.1611
3.1474
3.1332
3.1196
3.1056
3.0914
2.3139
2.2985
2.2826
2.1818
2.1674
2.1530
2.1381
1.5461
1.4889
1.4737
1.4590
1.4444
1.4297
1.4148
1.3434
1.3282
1.3133
1.2979
1.2822
1.2676
1.1932
1.1793
0.9741
0.9595
0.9448
0.8966
0.8817
0.8673
0.390
1.045
1.034
1.000
1.040
2.014
2.101
4.794
2.173
2.493
6.695
3.297
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPPW 50 usec
IRATN 511
DFILE 4dm.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 780
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Fri Jul 23 10:16:15 2010

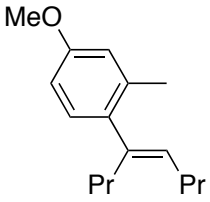
4dm


Pr
Pr

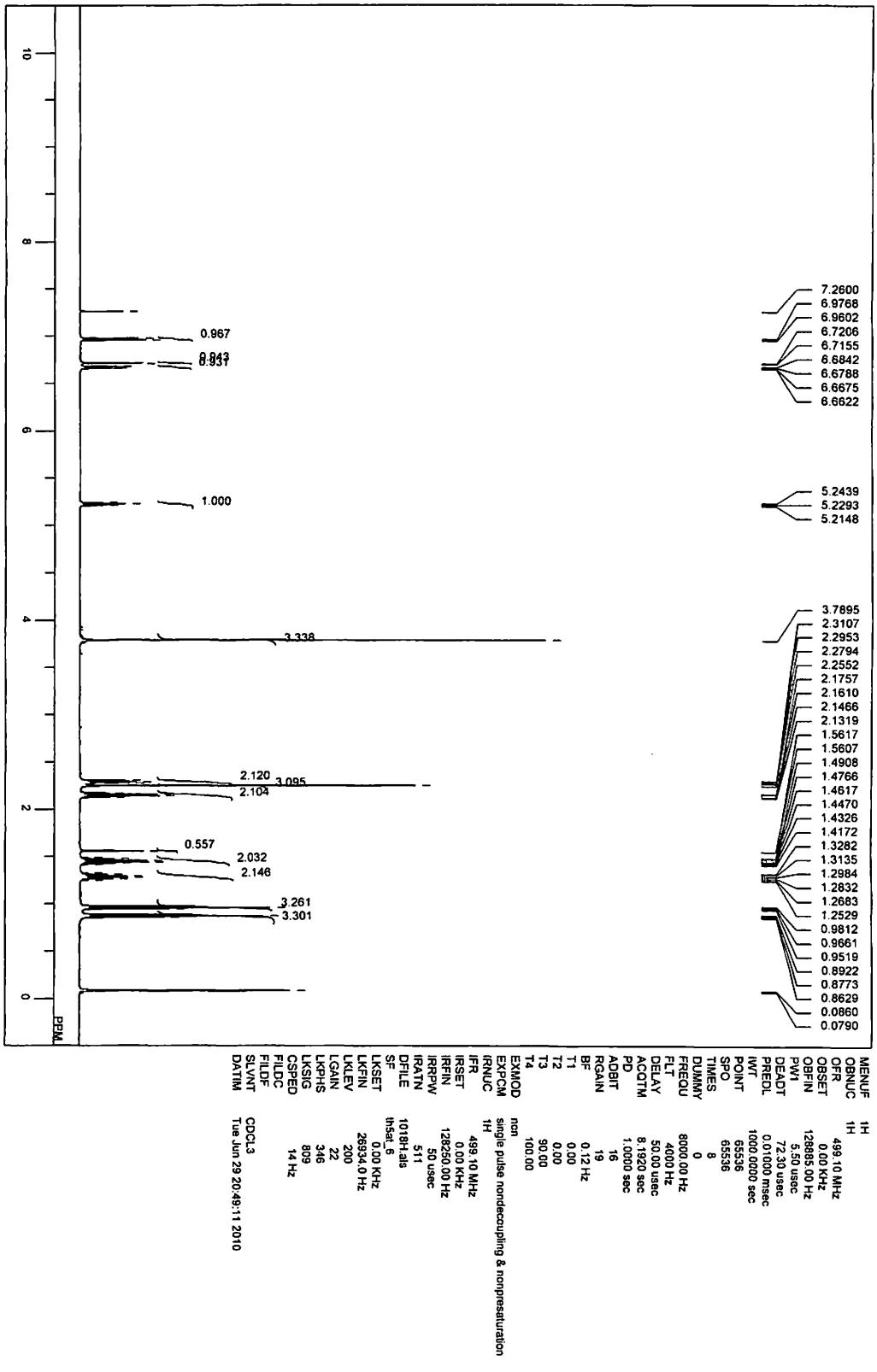
^{13}C NMR


146.5161
143.6565
140.5414
129.8240
129.3090
126.8078
125.4481
125.0154
77.4155
77.1600
76.9045
35.0857
30.2894
29.7167
24.6114
23.2186
21.5004
14.3637
13.9764
200
150
100
50
0
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33698.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE n3-185C.ais
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 752
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 04 11:22:08 2010

4em


MeO
Pr
Pr

^{1}H NMR


PPM
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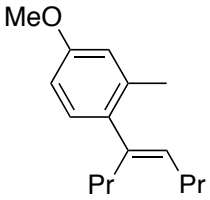
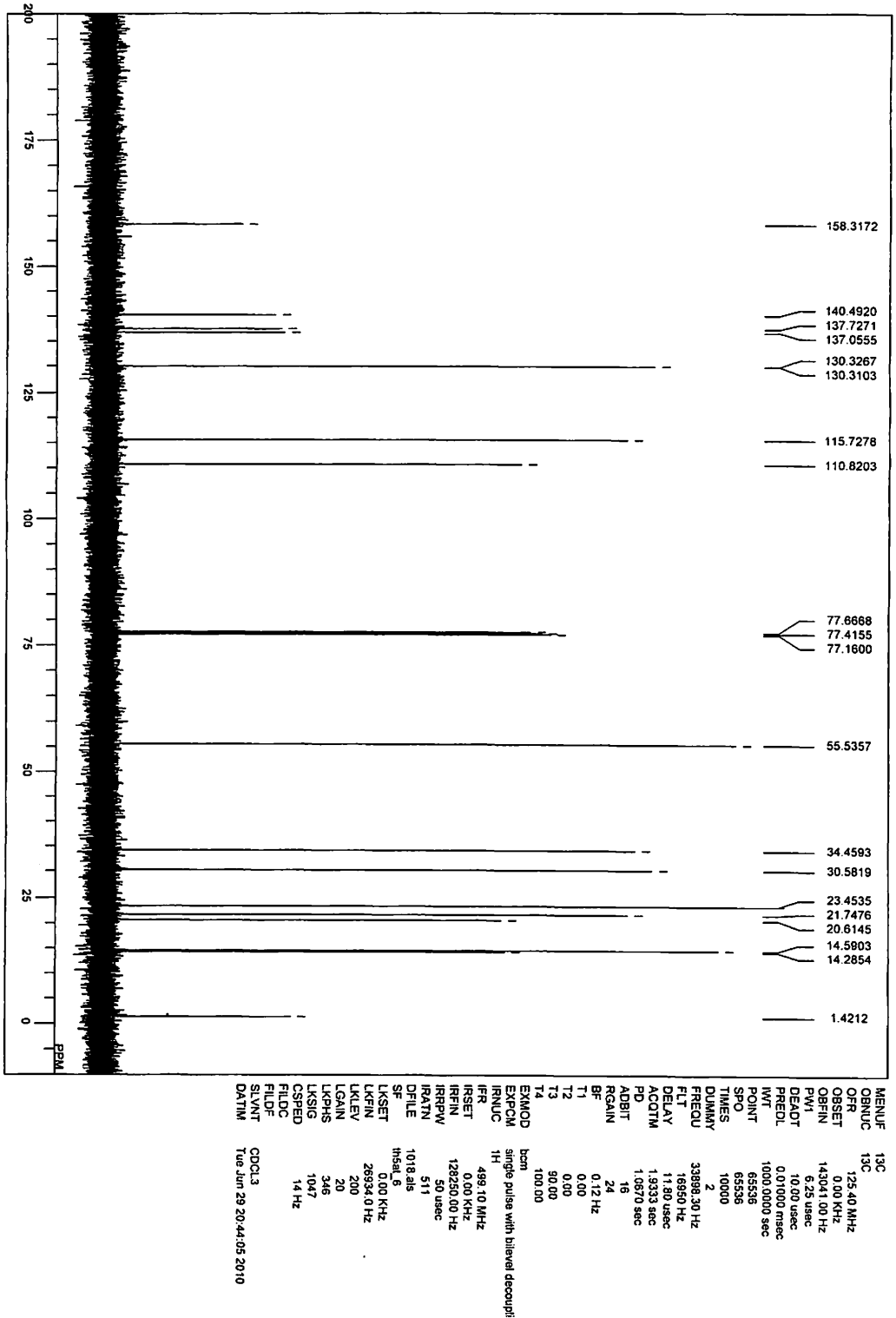
1.000
3.338
2.120
3.095
2.104
0.557
2.032
2.146
3.261
3.301
7.2600
6.9768
6.9602
6.7206
6.7155
6.6842
6.6788
6.6675
6.6622
5.2439
5.2293
5.2148
3.7895
2.3107
2.2953
2.2794
2.2552
2.1757
2.1610
2.1466
2.1319
1.5617
1.5607
1.4908
1.4766
1.4617
1.4470
1.4326
1.4172
1.3282
1.3135
1.2984
1.2832
1.2683
1.2529
0.9812
0.9661
0.9519
0.8922
0.8773
0.8629
0.0860
0.0790
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 19
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1018H.als
SF tHsat_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 809
CSPEED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Jun 29 20:49:11 2010

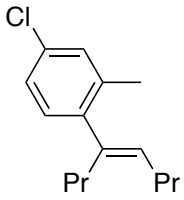
4em


MeO
Pr
Pr

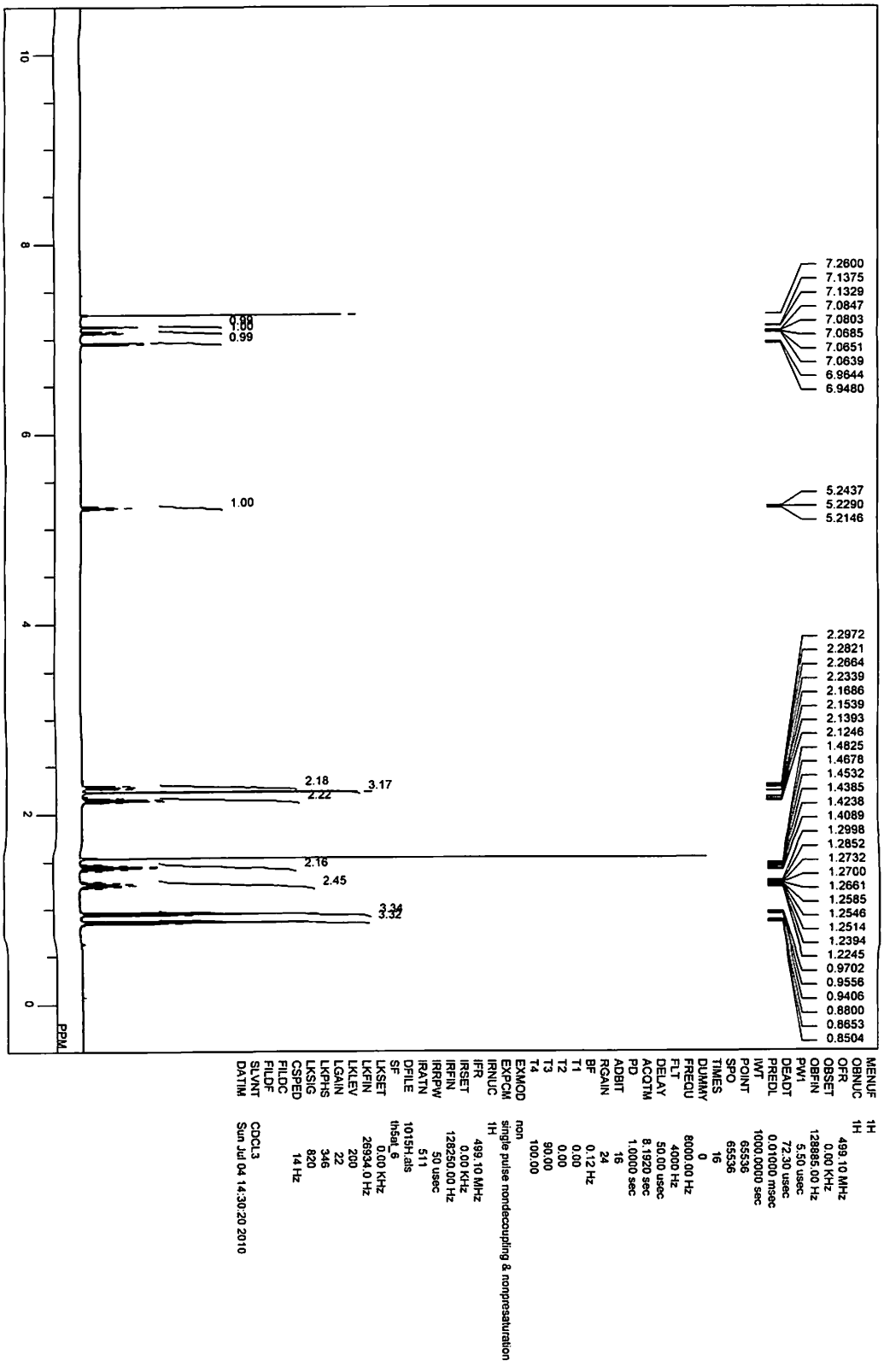
^{13}C NMR


158.3172
140.4920
137.7271
137.0555
130.3267
130.3103
115.7278
110.8203
77.6668
77.4155
77.1600
55.5357
34.4593
30.5819
23.4535
21.7476
20.6145
14.5903
14.2854
1.4212
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0870 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPW 50 usec
IRATN 511
DFILE 1018.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV
LGAIN 20
LKPHS 346
LKSIG 1047
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Jun 29 20:44:05 2010

4fm


Cl
Pr
Pr

^{1}H NMR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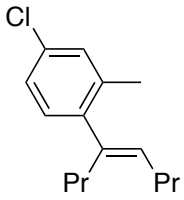
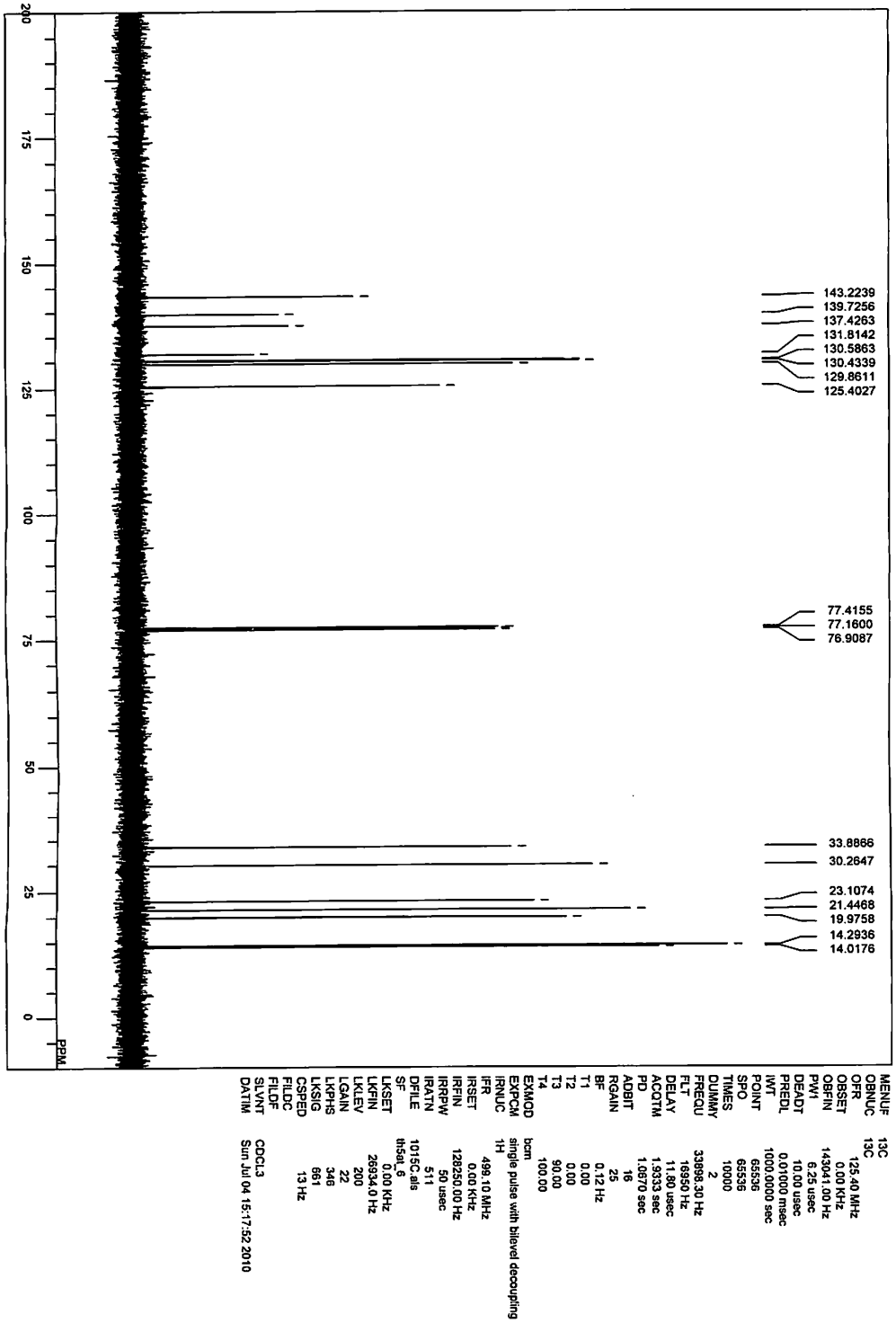
7.1375
7.1329
7.0847
7.0803
7.0685
7.0651
7.0639
6.9644
6.9480
5.2437
5.2290
5.2146
2.2972
2.2821
2.2664
2.2339
2.1686
2.1539
2.1393
2.1246
1.4825
1.4678
1.4532
1.4385
1.4238
1.4089
1.2998
1.2852
1.2732
1.2700
1.2661
1.2585
1.2546
1.2514
1.2394
1.2245
0.9702
0.9556
0.9406
0.8800
0.8653
0.8504
0.99
1.00
1.00
0.99
1.00
2.18
2.22
3.17
2.16
2.45
3.34
3.32
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBFIN 0.00 KHz
OBSET 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1015H.als
SF th5a1_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 820
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 04 14:30:20 2010

4fm


Cl
Pr
Pr

^{13}C NMR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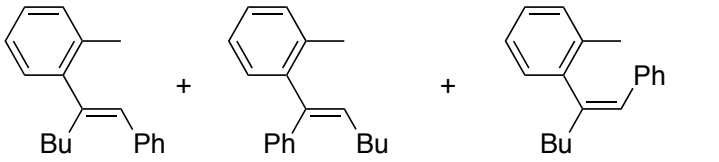
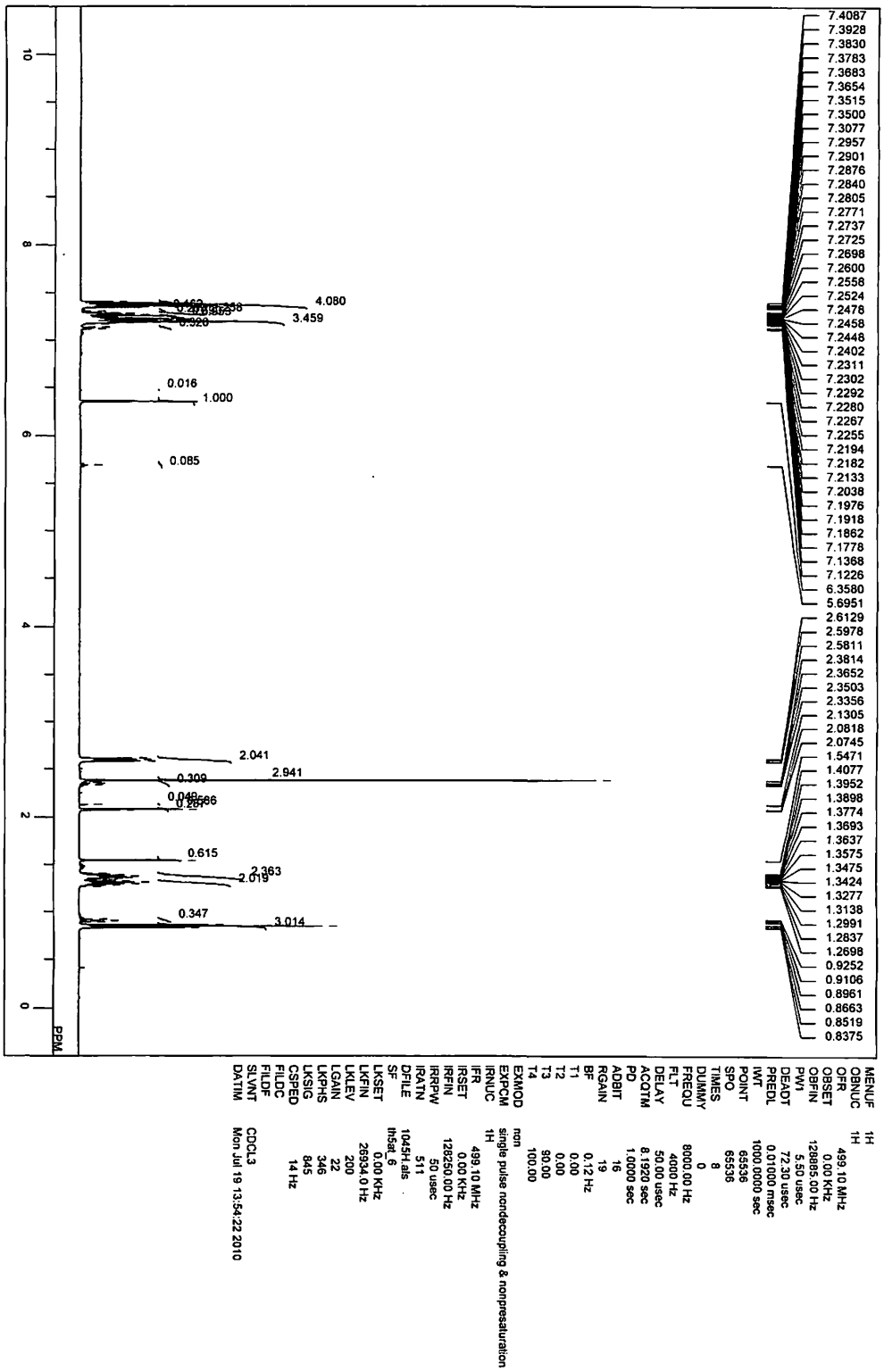
137.4263
131.8142
130.5863
130.4339
129.8611
125.4027
77.4155
77.1600
76.9087
33.8866
30.2647
23.1074
21.4468
19.9758
14.2936
14.0176
200
175
150
125
100
75
50
25
0
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 498.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1015C.als
SF this_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 661
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 04 15:17:52 2010

4an + regioisomer (*E*) of **4an** + stereoisomer of **4an** (90:8:2)

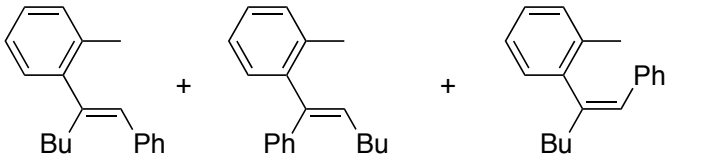


4an regioisomer (*E*) of **4an** Stereoisomer of **4an**

^{1}H NMR

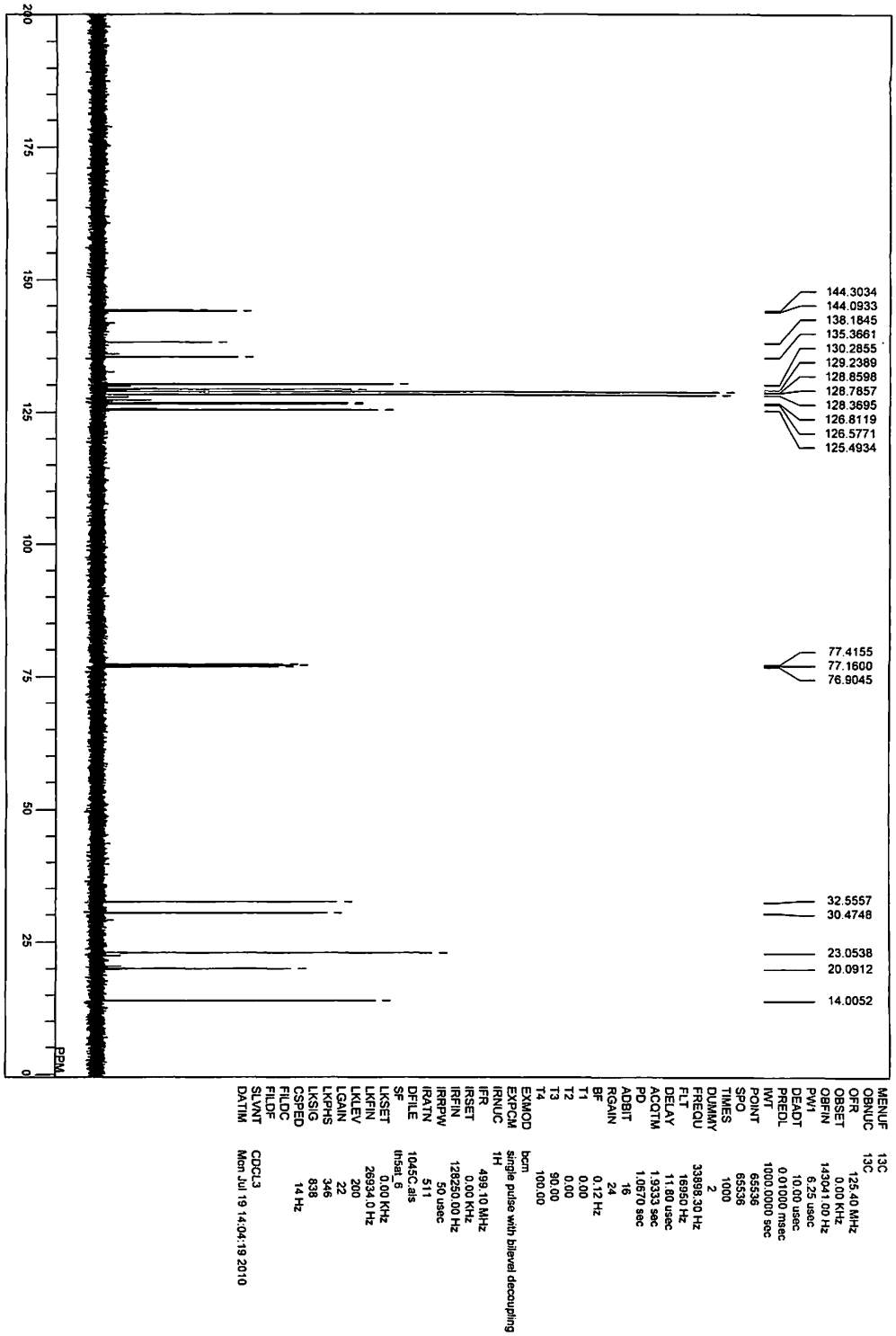


4an + regioisomer (*E*) of **4an** + stereoisomer of **4an** (90:8:2)

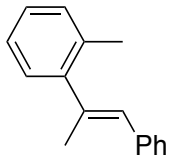


4an regioisomer (*E*) of **4an** Stereoisomer of **4an**

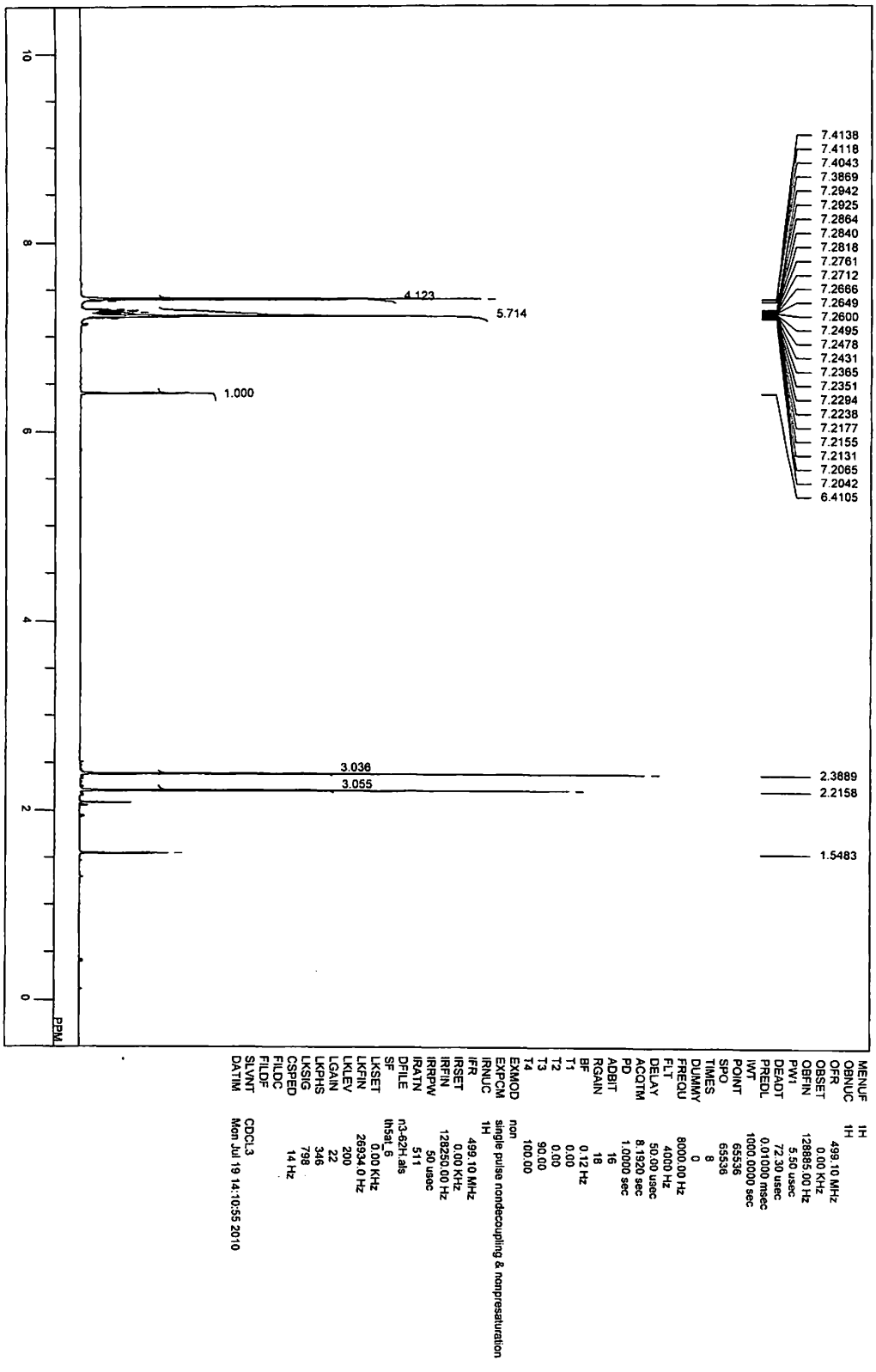
^{13}C NMR



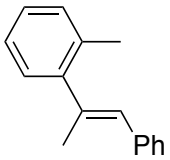
4ao


Ph

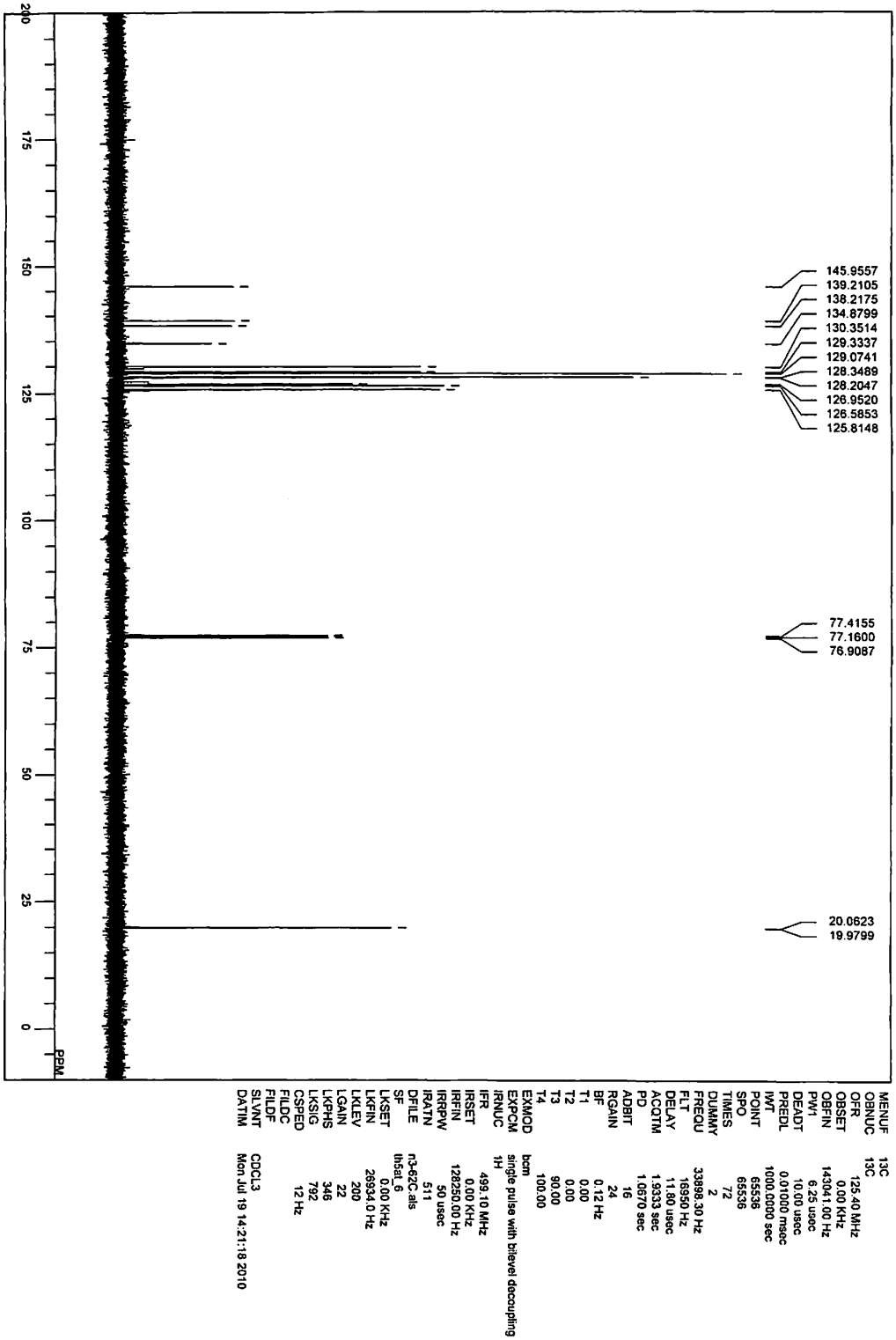
^{1}H NMR


7.4138
7.4118
7.4043
7.3869
7.2942
7.2925
7.2864
7.2840
7.2818
7.2761
7.2712
7.2666
7.2649
7.2600
7.2495
7.2478
7.2431
7.2365
7.2351
7.2294
7.2238
7.2177
7.2155
7.2131
7.2065
7.2042
6.4105
4.123
5.714
1.000
3.036
3.055
2.3889
2.2158
1.5483
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128685.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 18
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE n3-62H.als
SF 1h5al_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 798
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Mon Jul 19 14:10:55 2010

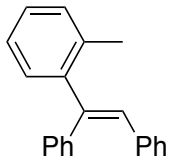
4ao


Ph

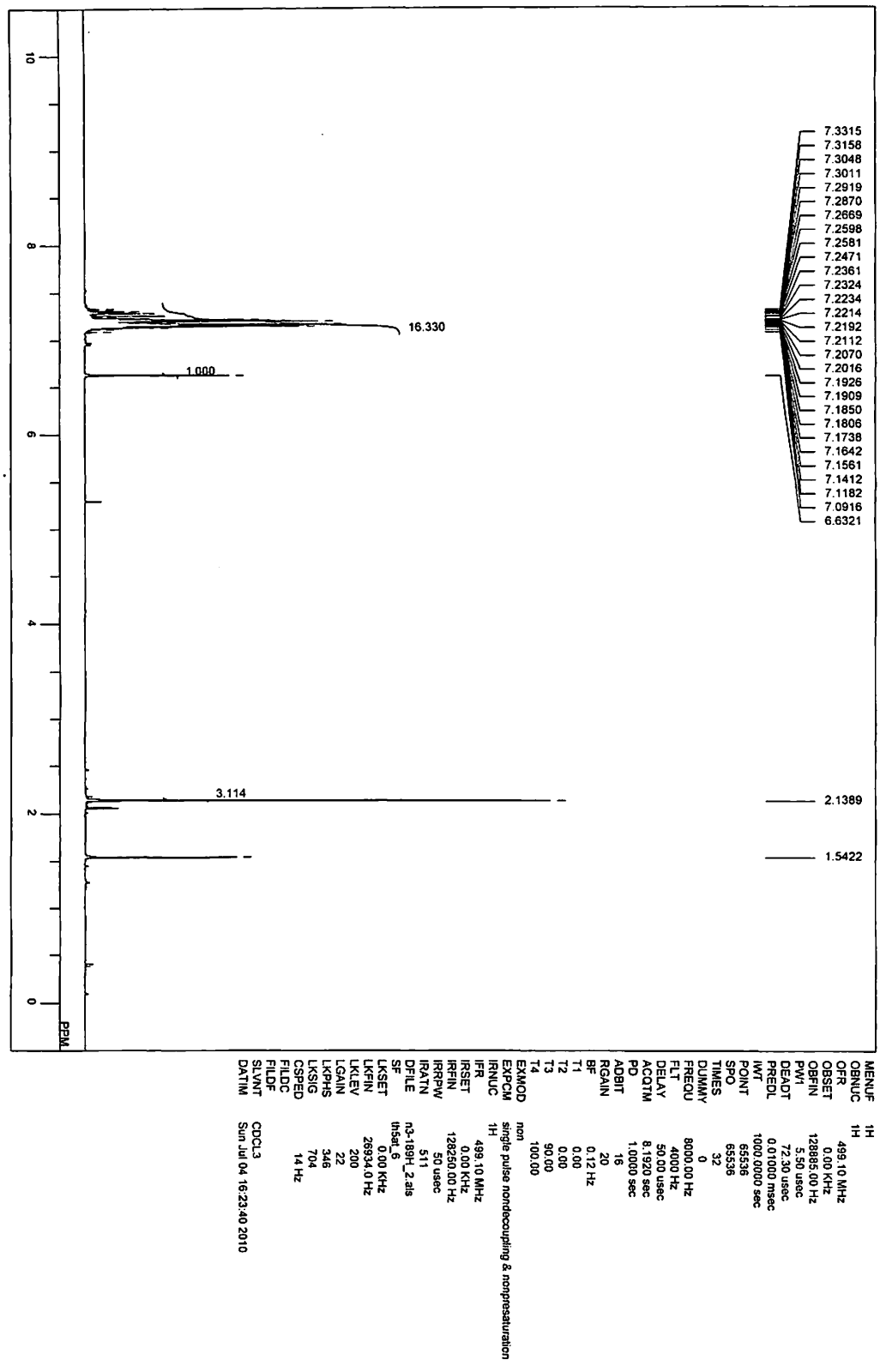
^{13}C NMR


145.9557
139.2105
138.2175
134.8799
130.3514
129.3337
129.0741
128.3489
128.2047
126.9520
126.5853
125.8148
77.4155
77.1600
76.9087
20.0623
19.9799
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 14304.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 72
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE n3-62C.ais
SF 0.00 KHz
LKSET thSat_6
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 792
CSPED 12 Hz
FILDF
FILDC
SLVNT CDCl3
DATIM Mon Jul 19 14:21:18 2010

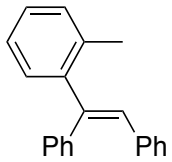
4ap


Ph
Ph

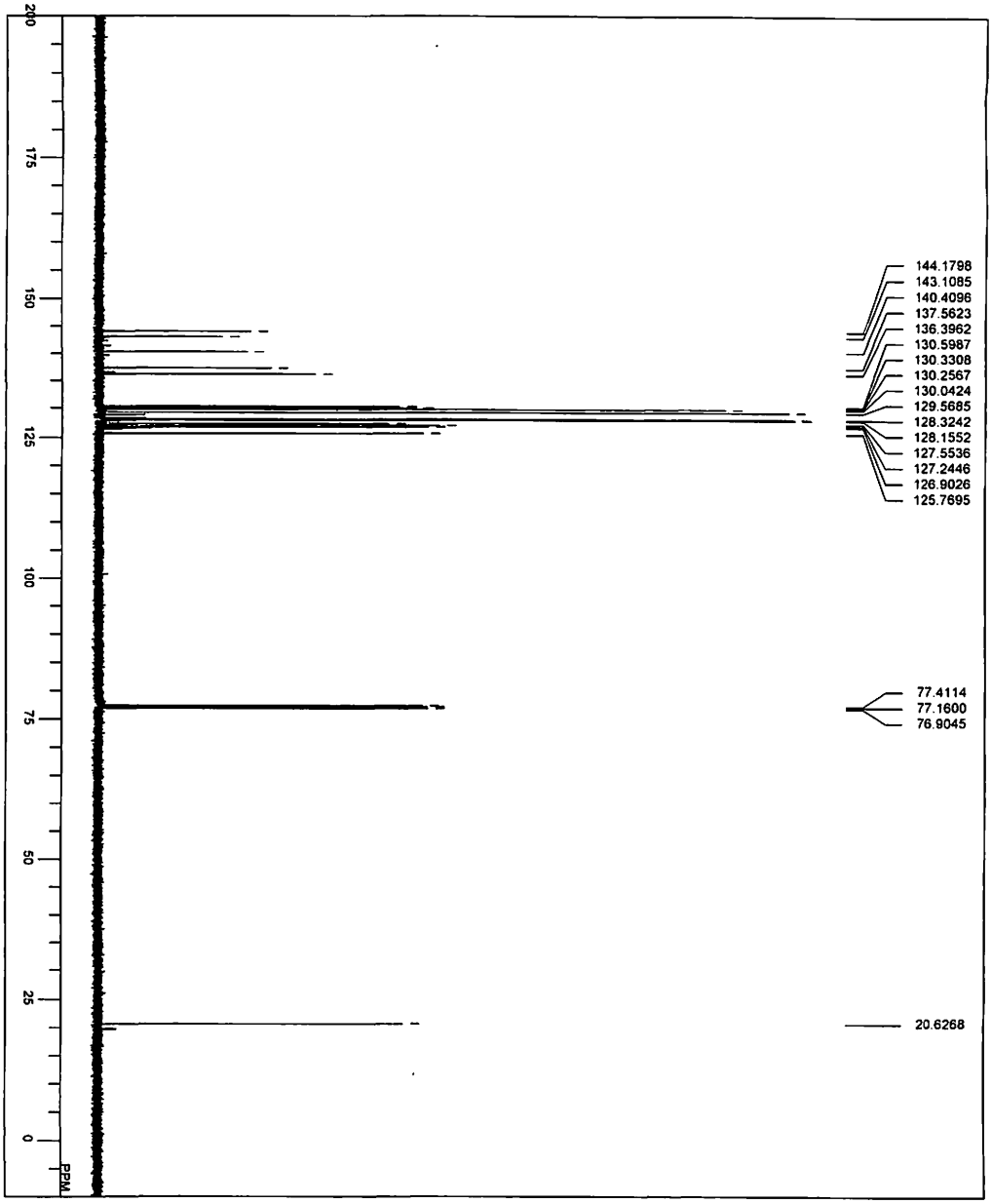
^{1}H NMR


7.3315
7.3158
7.3048
7.3011
7.2919
7.2870
7.2669
7.2598
7.2581
7.2471
7.2361
7.2324
7.2234
7.2214
7.2192
7.2112
7.2070
7.2016
7.1926
7.1909
7.1850
7.1806
7.1738
7.1642
7.1561
7.1412
7.1182
7.0916
6.6321
16.330
1.000
3.114
2.1389
1.5422
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 32
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 20
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE n3-189H_2.als
SF tmse_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 704
CSPED 14 Hz
FILDF
SLVNT CDCL3
DATIM Sun Jul 04 16:23:40 2010

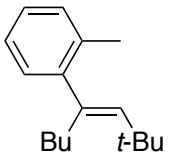
4ap


Ph
Ph

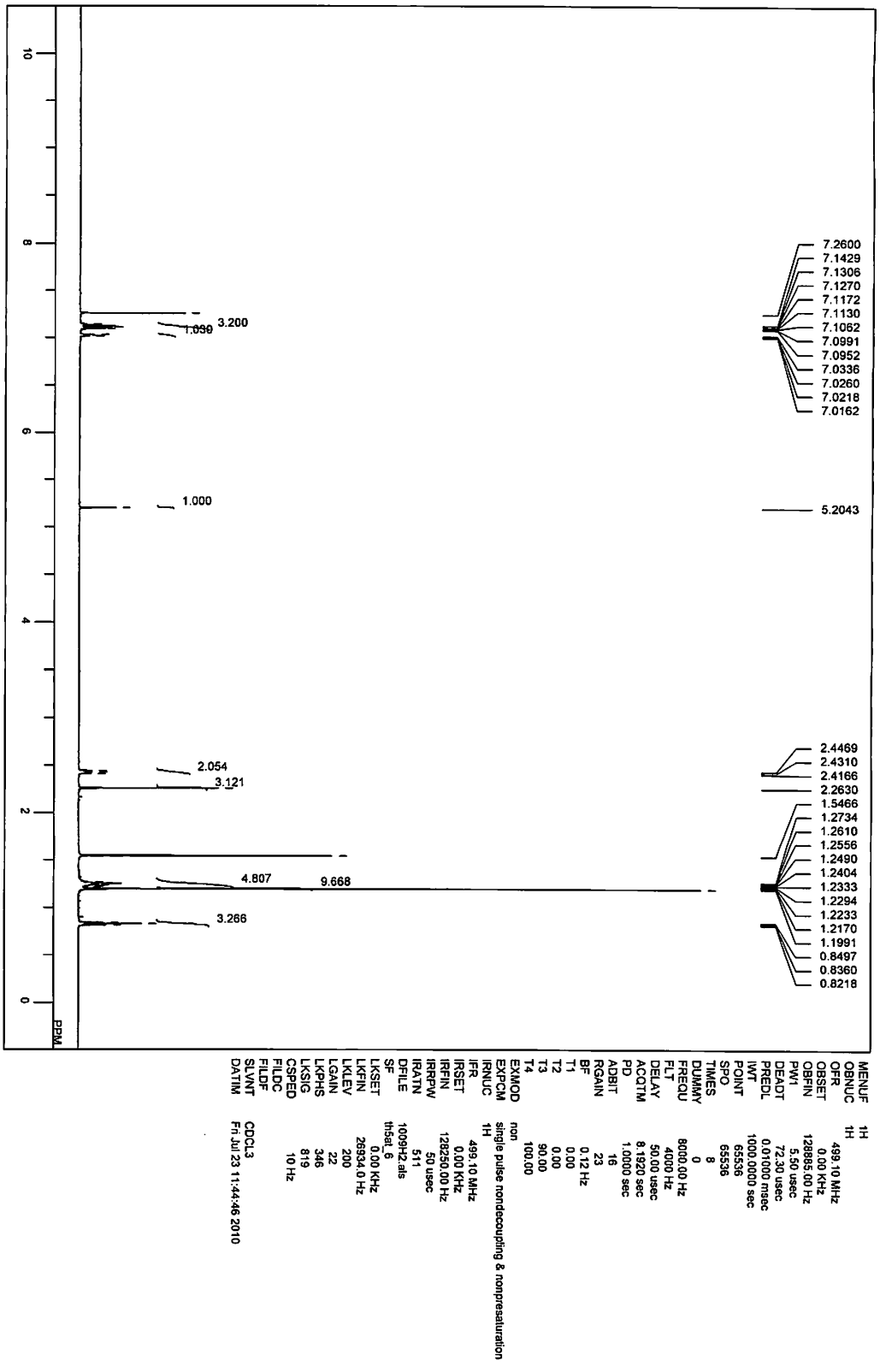
^{13}C NMR


144.1798
143.1085
140.4096
137.5623
136.3962
130.5987
130.3308
130.2567
130.0424
129.5685
128.3242
128.1552
127.5536
127.2446
126.9026
125.7695
77.4114
77.1600
76.9045
20.6268
200
175
150
125
100
75
50
25
0
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE h5at_6
SF 0.00 KHz
LKSET 26934.0 Hz
LKFIN 200
LKLEV 22
LGAIN 346
LKPHS 616
LKSIG 14 Hz
CSPEED
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 04 12:53:40 2010

4aq


Bu
t-Bu

^{1}H NMR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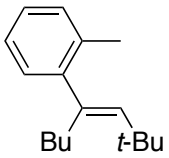
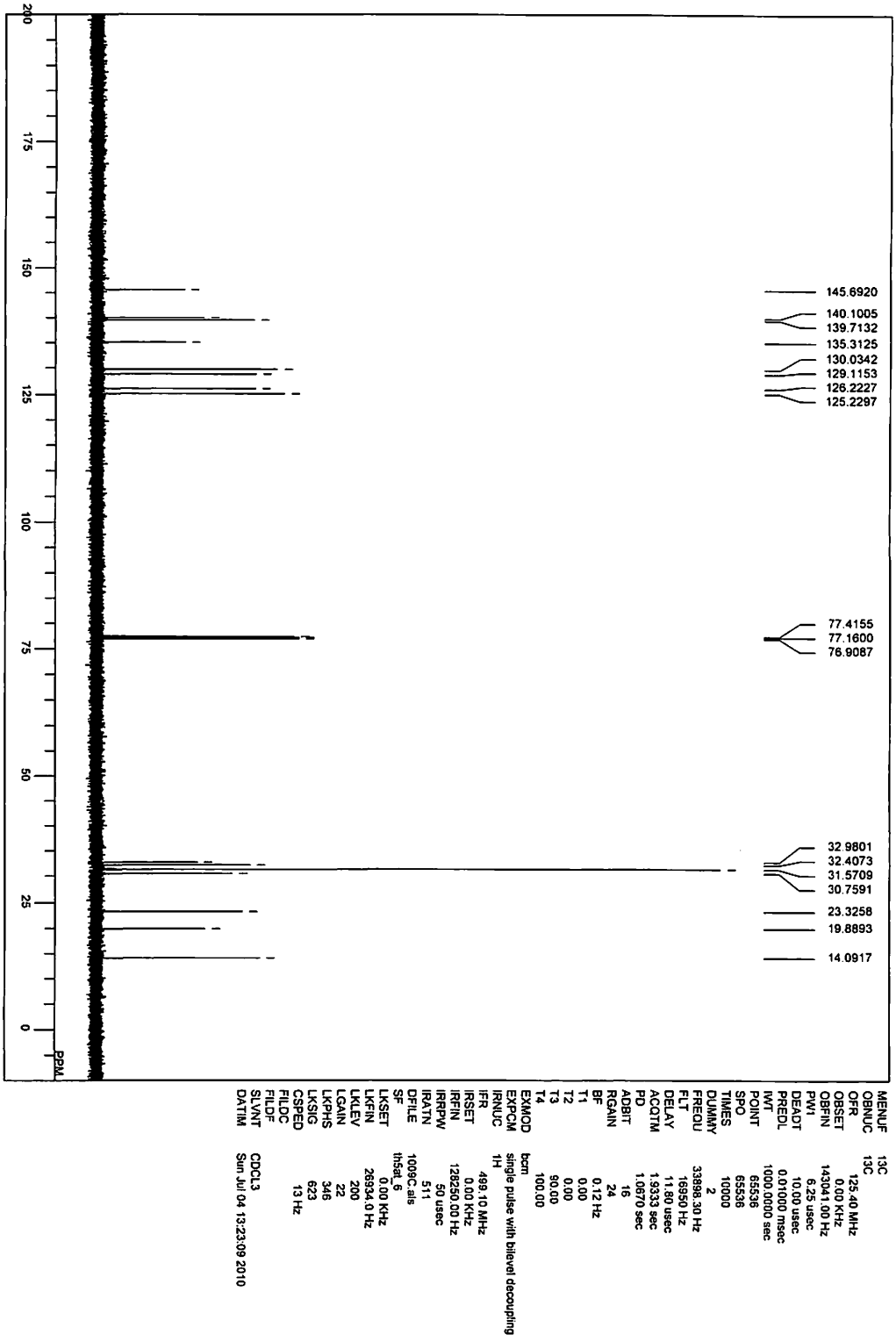
7.1429
7.1306
7.1270
7.1172
7.1130
7.1062
7.0991
7.0952
7.0336
7.0260
7.0218
7.0162
3.200
1.030
1.000
5.2043
2.054
3.121
2.4469
2.4310
2.4166
2.2630
1.5466
1.2734
1.2610
1.2556
1.2490
1.2404
1.2333
1.2294
1.2233
1.2170
1.1991
0.8497
0.8360
0.8218
4.807
9.668
3.266
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1009H2.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 819
CSPED 10 Hz
FILDF
FILDC
SLVNT CDCL3
DATIM Fri Jul 23 11:44:46 2010

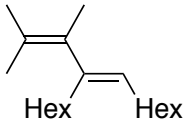
4aq


Bu
t-Bu

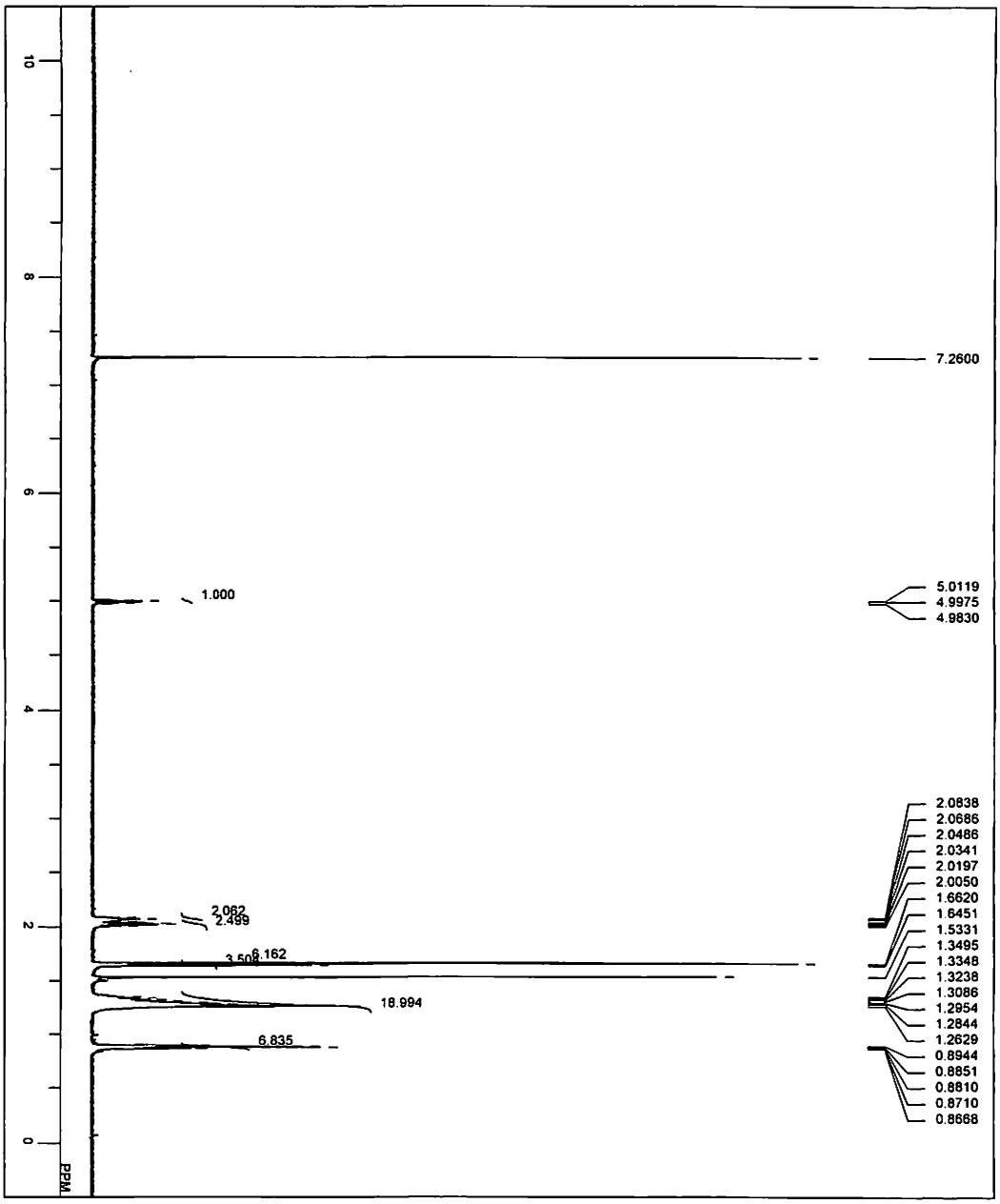
^{13}C NMR


145.6920
140.1005
139.7132
135.3125
130.0342
129.1153
126.2227
125.2297
77.4155
77.1600
76.9087
32.9801
32.4073
31.5709
30.7591
23.3258
19.8893
14.0917
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1009C.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 623
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 04 13:23:09 2010

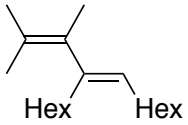
6ar


Hex
Hex

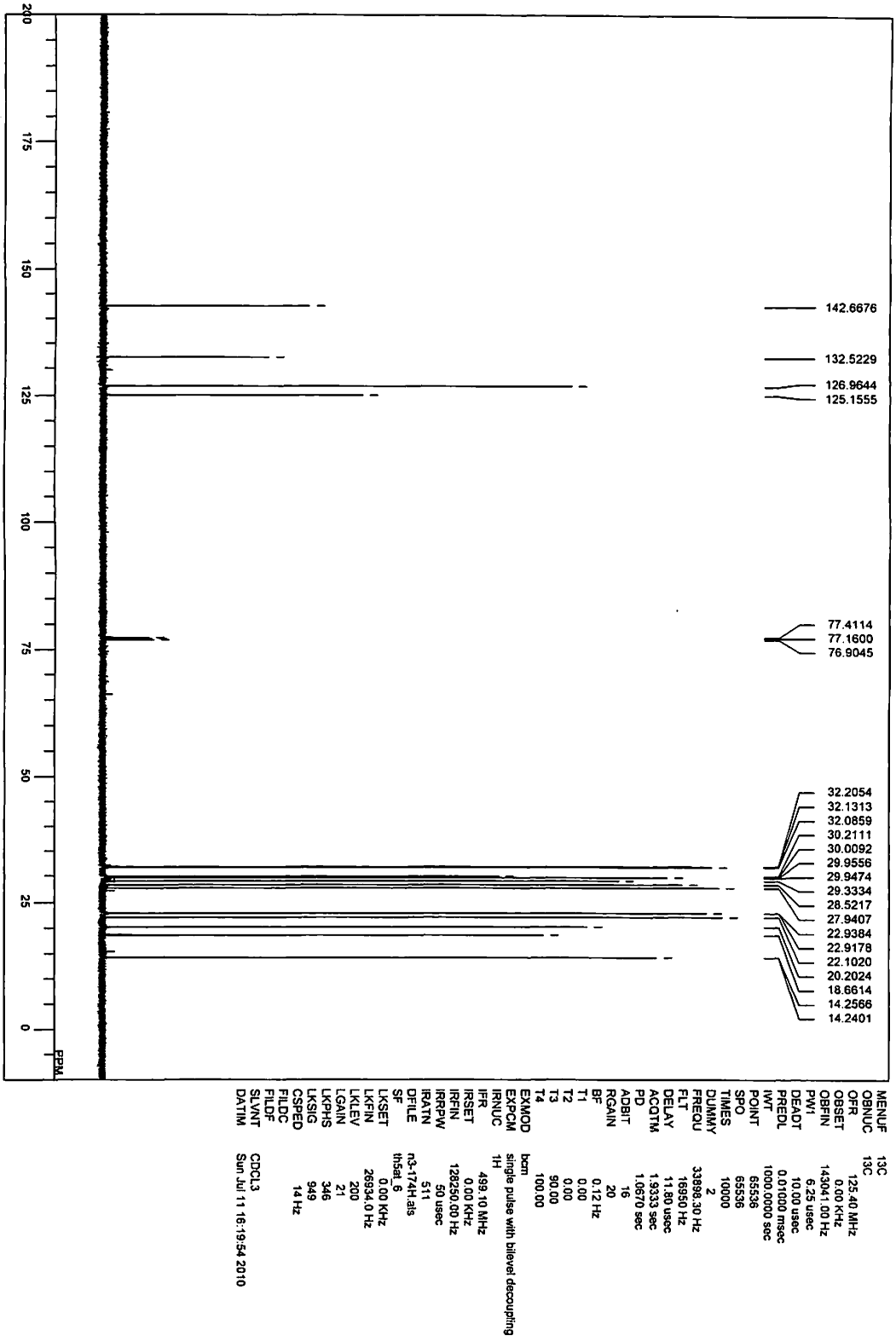
^{1}H NMR


7.2600
5.0119
4.9975
4.9830
2.0838
2.0686
2.0486
2.0341
2.0197
2.0050
1.6620
1.6451
1.5331
1.3495
1.3348
1.3238
1.3086
1.2954
1.2844
1.2629
0.8944
0.8851
0.8810
0.8710
0.8668
1.000
2.062
2.499
3.508
6.162
18.994
6.835
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE nanui_3-174_pttc_a.als
SF tf5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 850
CSPED 13 Hz
FILDF
FILDC
SLVNT CDCL3
DATIM Wed May 12 08:22:27 2010

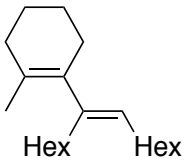
6ar


Hex
Hex

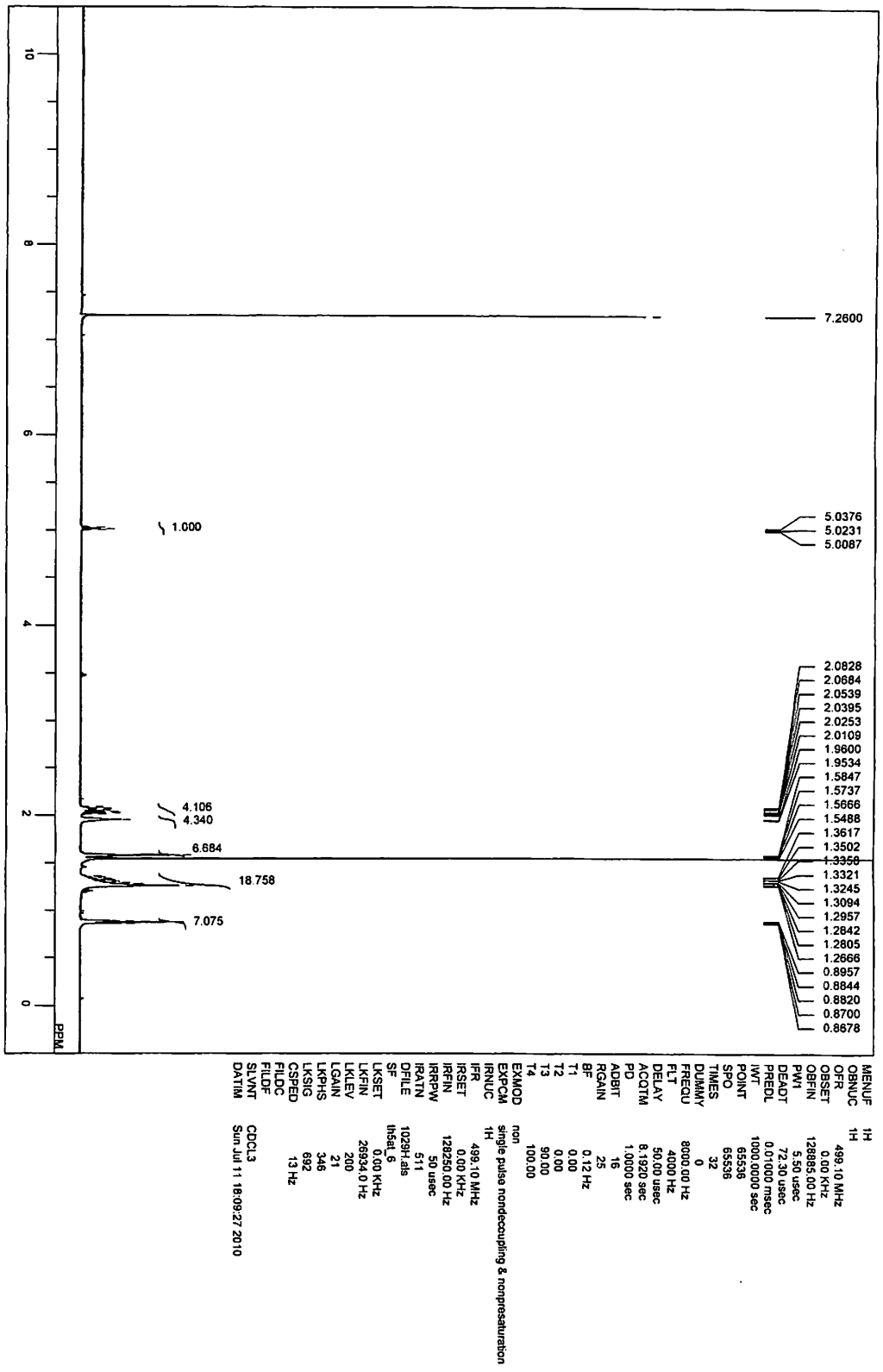
^{13}C NMR


142.6676
132.5229
126.9644
125.1555
77.4114
77.1600
76.9045
32.2054
32.1313
32.0859
30.2111
30.0092
29.9556
29.9474
29.3334
28.5217
27.9407
22.9384
22.9178
22.1020
20.2024
18.6614
14.2566
14.2401
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 14304.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 20
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPW 50 usec
IRATN 511
DFILE n3-174H.ats
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 21
LKPHS 346
LKSIG 949
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 11 16:19:54 2010

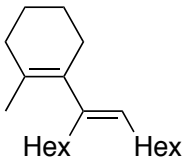
6br


Hex
Hex

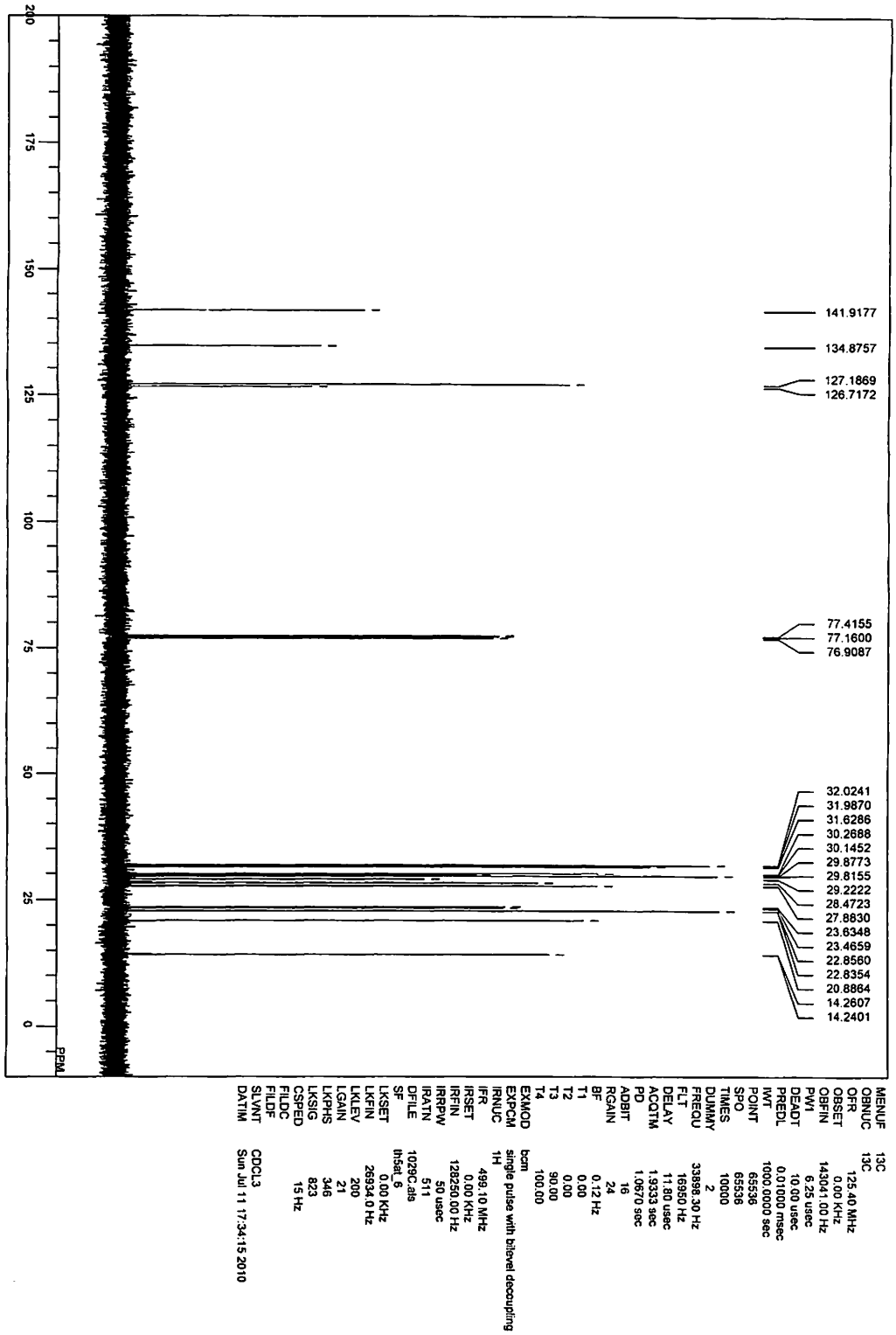
^{1}H NMR


7.2600
5.0376
5.0231
5.0087
2.0828
2.0684
2.0539
2.0395
2.0253
2.0109
1.9600
1.9534
1.5847
1.5737
1.5666
1.5488
1.3617
1.3502
1.3358
1.3321
1.3245
1.3094
1.2957
1.2842
1.2805
1.2666
0.8957
0.8844
0.8820
0.8700
0.8678
1.000
4.106
4.340
6.684
18.758
7.075
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 32
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1029H.als
SF thSat_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LKPHS 21
LGAIN 346
LKSIG 692
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 11 18:09:27 2010

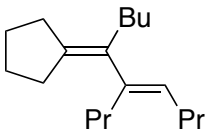
6br


Hex
Hex

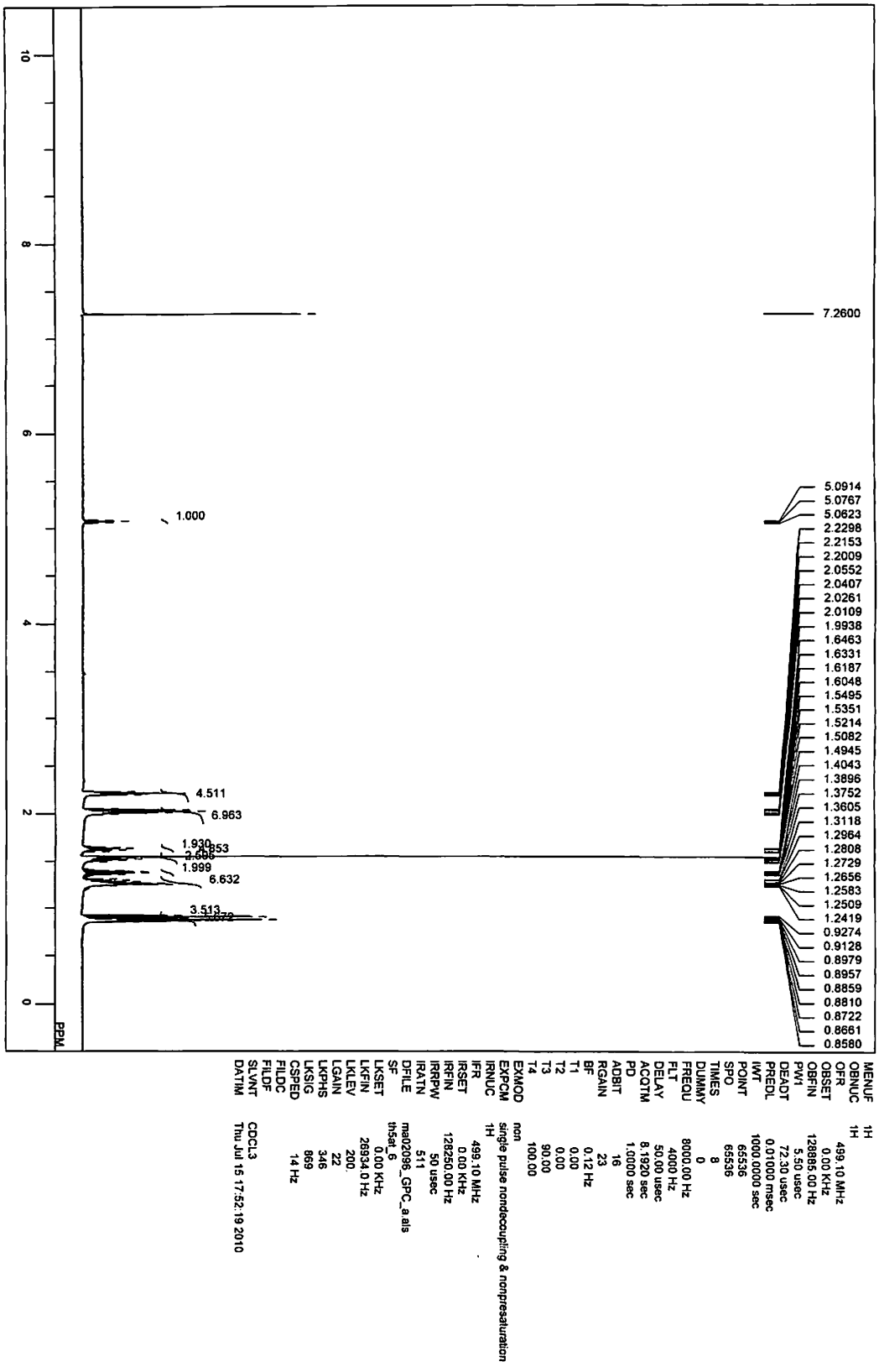
^{13}C NMR


141.9177
134.8757
127.1869
126.7172
77.4155
77.1600
76.9087
32.0241
31.9870
31.6286
30.2688
30.1452
29.8773
29.8155
29.2222
28.4723
27.8830
23.6348
23.4659
22.8560
22.8354
20.8864
14.2607
14.2401
200
175
150
125
100
75
50
25
0
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 1029C.dat
SF bhSat_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 21
LKPHS 346
LKSIG 823
CSPED 15 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Jul 11 17:34:15 2010

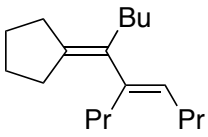
6cm


Bu
Pr
Pr

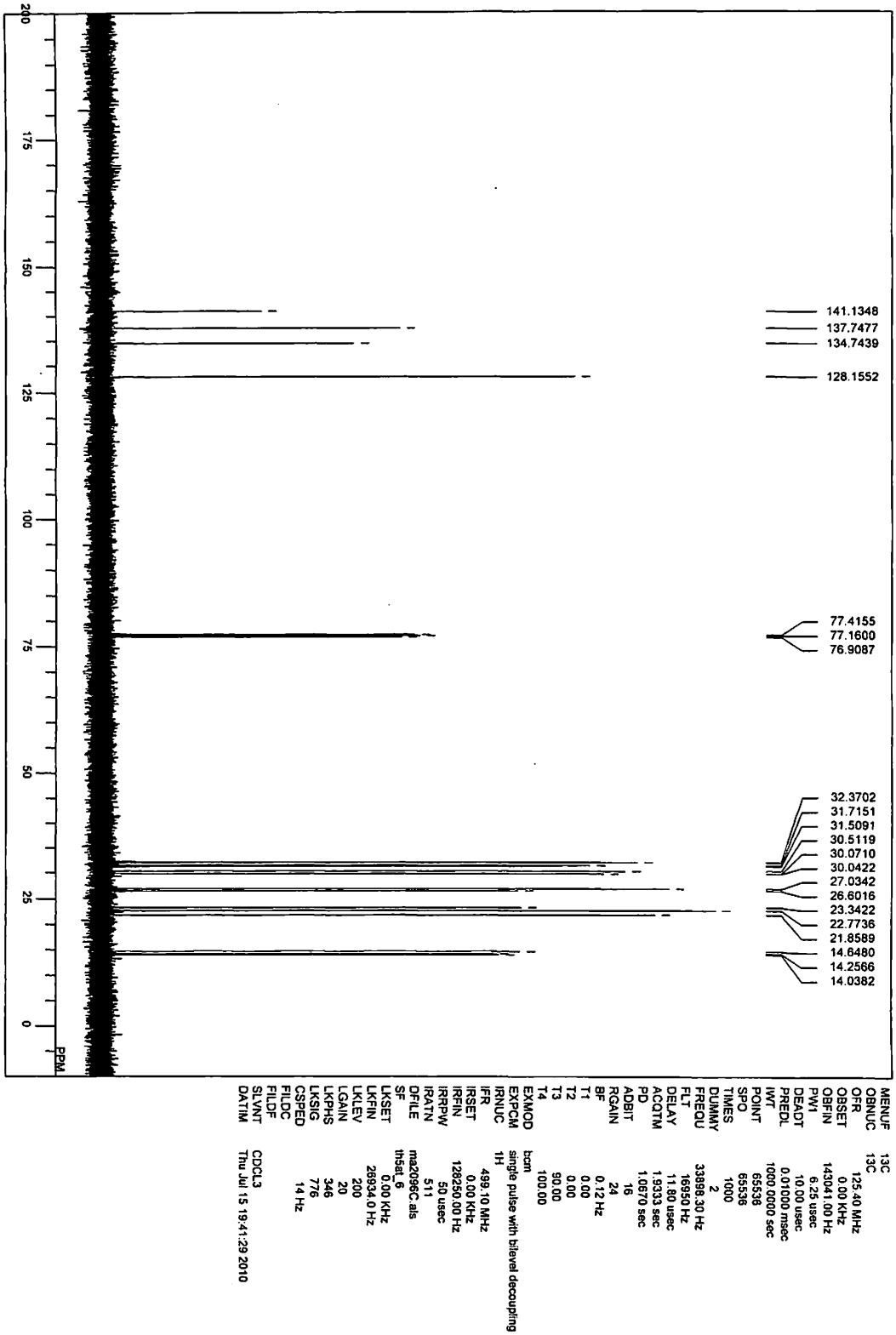
^{1}H NMR


7.2600
1.000
4.511
6.963
1.930
3.853
3.666
1.999
6.632
3.513
5.0914
5.0767
5.0623
2.2298
2.2153
2.2009
2.0552
2.0407
2.0261
2.0109
1.9938
1.6463
1.6331
1.6187
1.6048
1.5495
1.5351
1.5214
1.5082
1.4945
1.4043
1.3896
1.3752
1.3605
1.3118
1.2964
1.2808
1.2729
1.2656
1.2583
1.2509
1.2419
0.9274
0.9128
0.8979
0.8957
0.8859
0.8810
0.8722
0.8661
0.8580
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
WT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma02096_GPC_a.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200.
LGAIN 22
LKPHS 346
LKSIG 869
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu Jul 15 17:52:19 2010

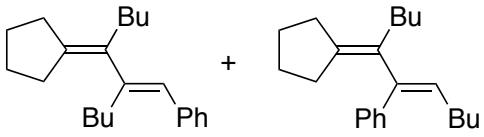
6cm


Bu
Pr
Pr

^{13}C NMR

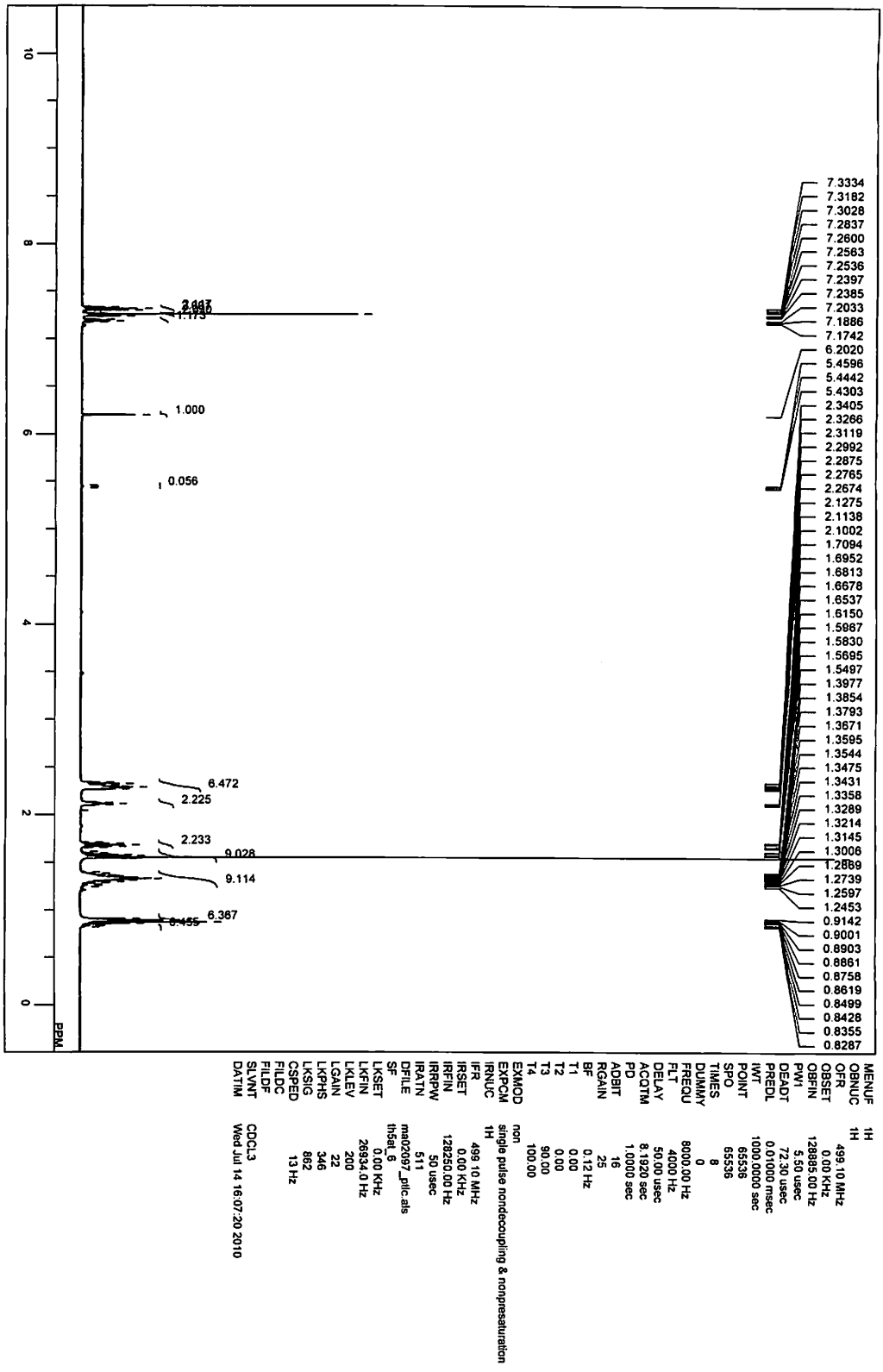

141.1348
137.7477
134.7439
128.1552
77.4155
77.1600
76.9087
32.3702
31.7151
31.5091
30.5119
30.0710
30.0422
27.0342
26.6016
23.3422
22.7736
21.8589
14.6480
14.2566
14.0382
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 1000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma2096C.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 776
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu Jul 15 19:41:29 2010

6cn + regioisomer (*E*) of **6cn** (94:6)

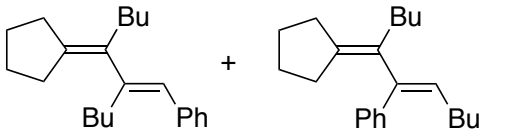


6cn regioisomer (*E*) of **6cn**

^{1}H NMR

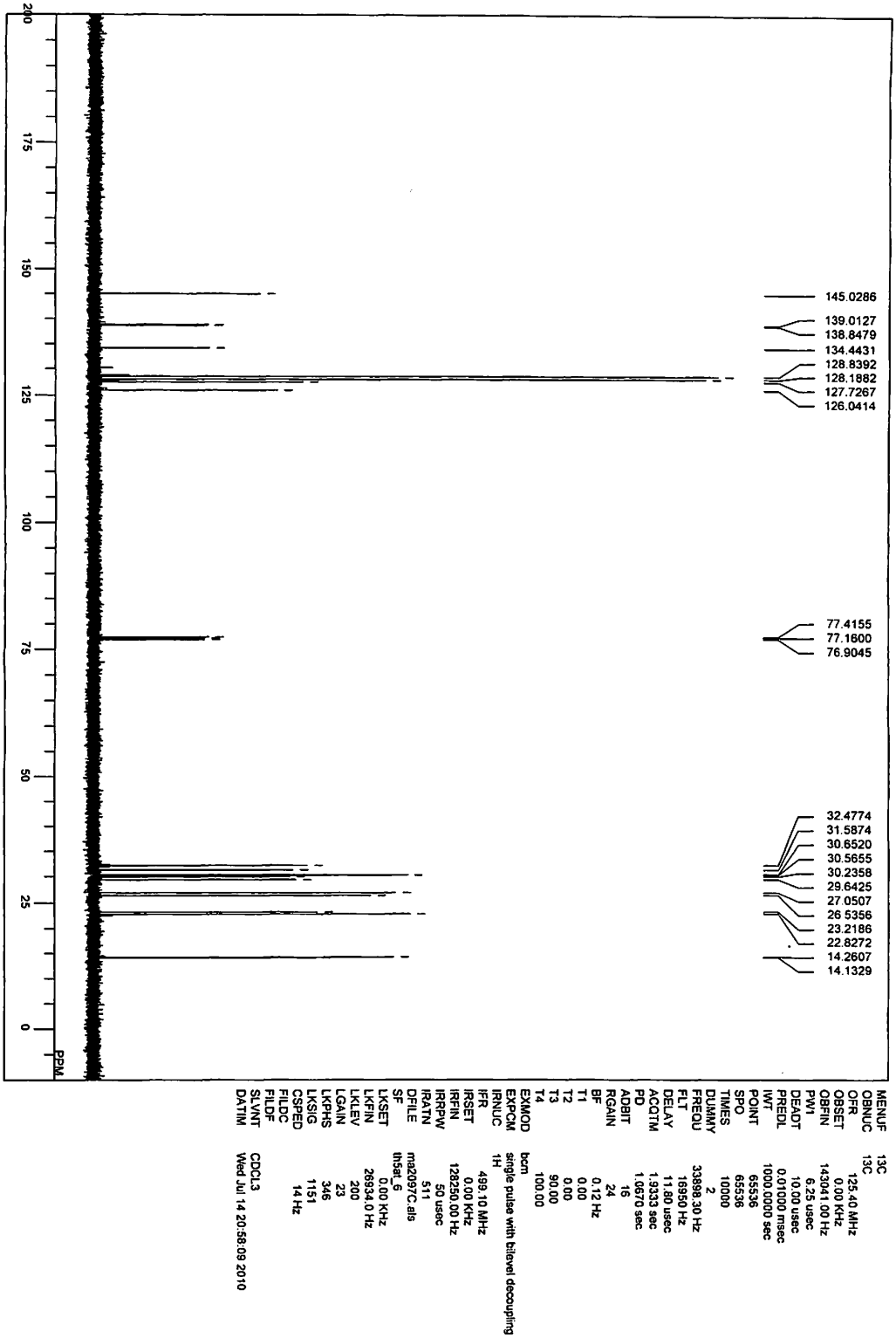


6cn + regioisomer (*E*) of **6cn** (94:6)

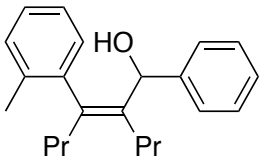


6cn regioisomer (*E*) of **6cn**

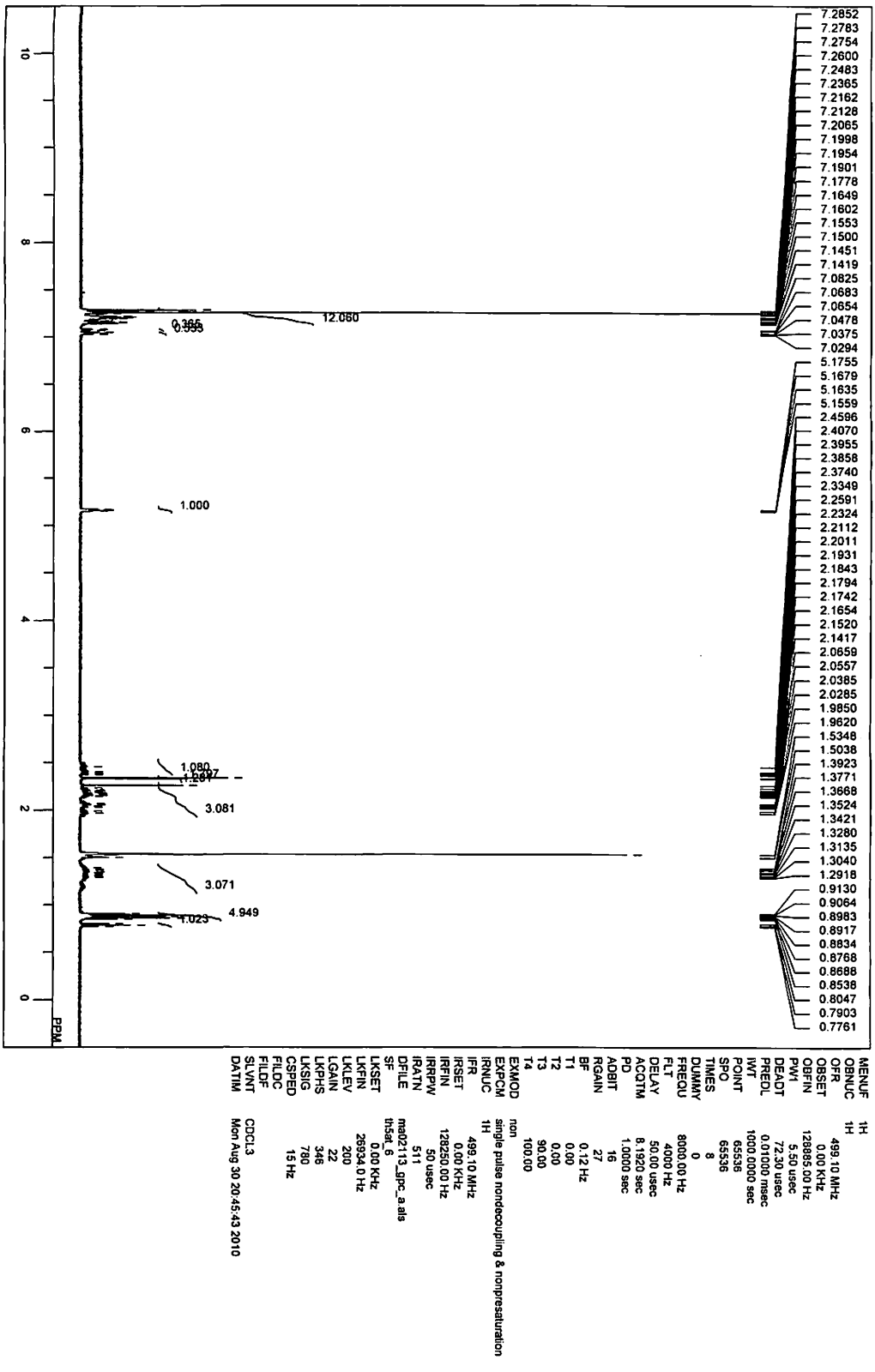
^{13}C NMR



9a


HO
Pr
Pr

^{1}H NMR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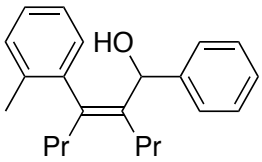
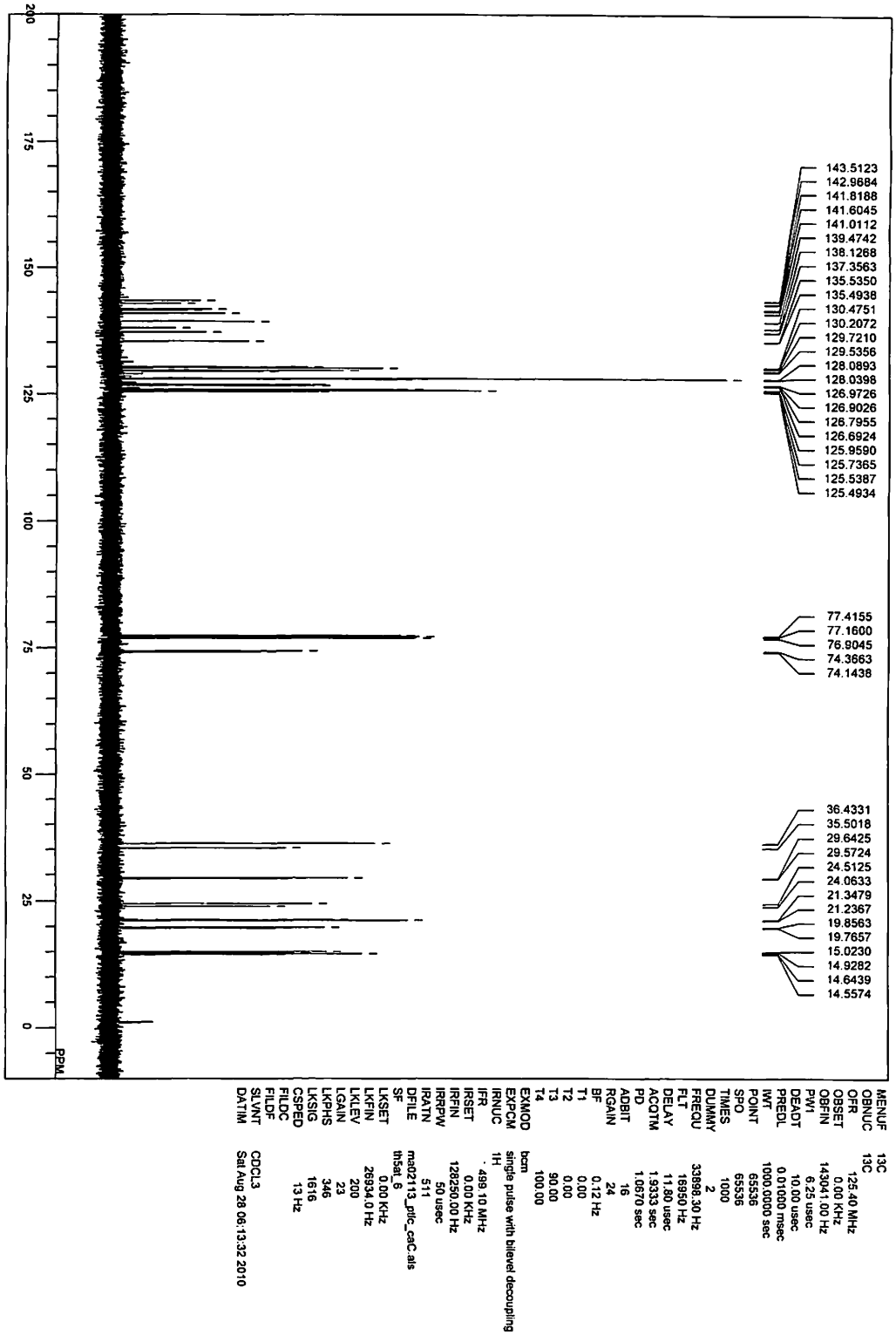
7.2754
7.2600
7.2483
7.2365
7.2162
7.2128
7.2065
7.1998
7.1954
7.1901
7.1778
7.1649
7.1602
7.1553
7.1500
7.1451
7.1419
7.0825
7.0683
7.0654
7.0478
7.0375
7.0294
5.1755
5.1679
5.1635
5.1559
2.4596
2.4070
2.3955
2.3858
2.3740
2.3349
2.2591
2.2324
2.2112
2.2011
2.1931
2.1843
2.1794
2.1742
2.1654
2.1520
2.1417
2.0659
2.0557
2.0385
2.0285
1.9850
1.9620
1.5348
1.5038
1.3923
1.3771
1.3668
1.3524
1.3421
1.3280
1.3135
1.3040
1.2918
0.9130
0.9064
0.8983
0.8917
0.8834
0.8768
0.8688
0.8538
0.8047
0.7903
0.7761
0.365
0.993
12.080
1.000
1.080
1.297
3.081
3.071
4.949
1.023
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IVT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ms02113_gpc_a.als
SF th5al_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 780
CSPED 15 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Mon Aug 30 20:45:43 2010

9a


HO
Pr
Pr

^{13}C NMR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141.8188
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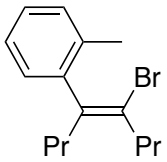
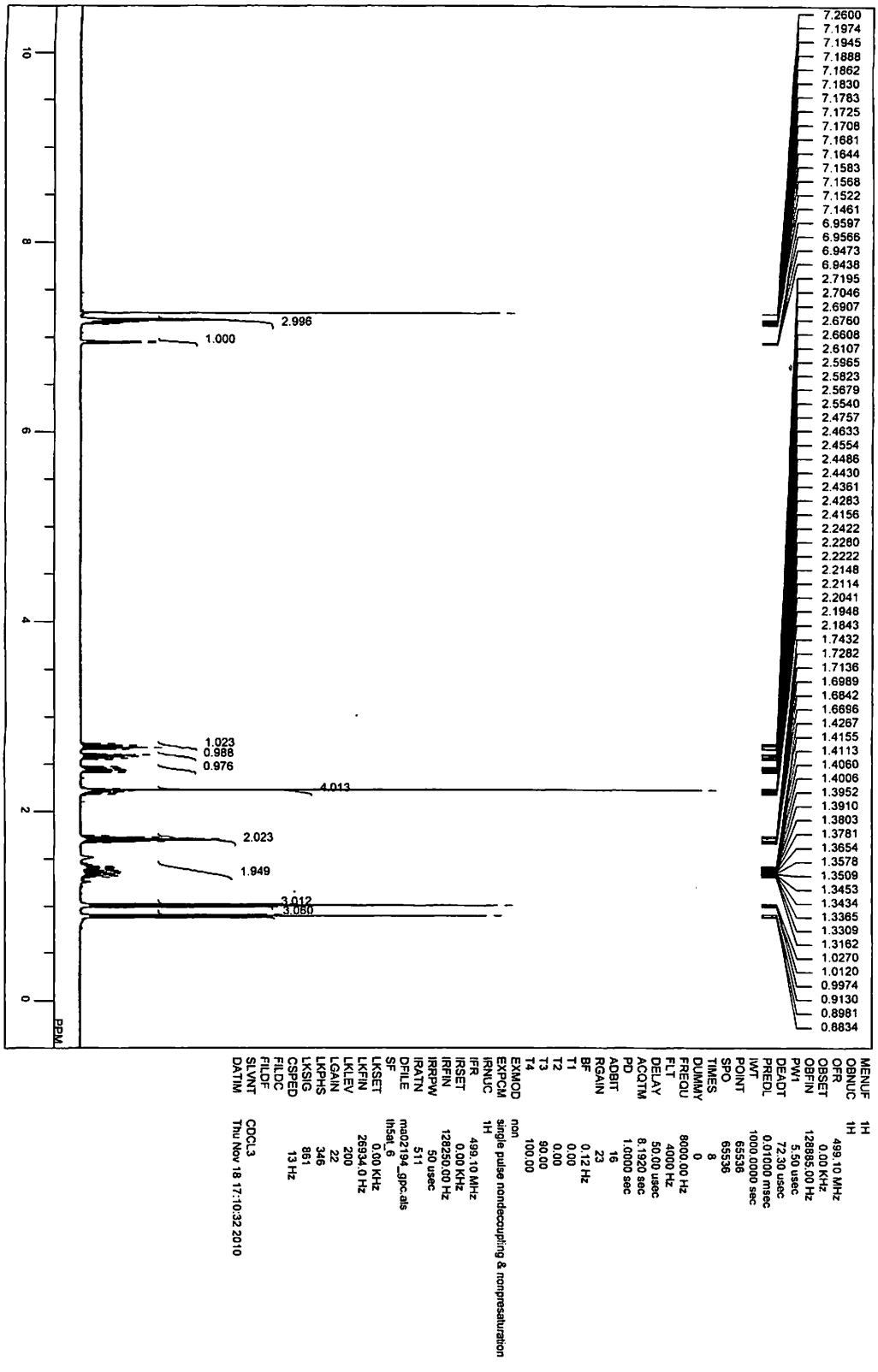
141.0112
139.4742
138.1268
137.3563
135.5350
135.4938
130.4751
130.2072
129.7210
129.5356
128.0893
128.0398
126.9726
126.9026
126.7955
126.6924
125.9590
125.7365
125.5387
125.4934
77.4155
77.1600
76.9045
74.3663
74.1438
36.4331
35.5018
29.6425
29.5724
24.5125
24.0633
21.3479
21.2367
19.8563
19.7657
15.0230
14.9282
14.6439
14.5574
200
175
150
125
100
75
50
25
0
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 1000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma02113_ptfc_ca6.als
SF thSat_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 346
LKSIG 1616
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat Aug 28 06:13:32 2010

9b


Br
Pr
Pr

^{1}H NMR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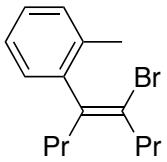
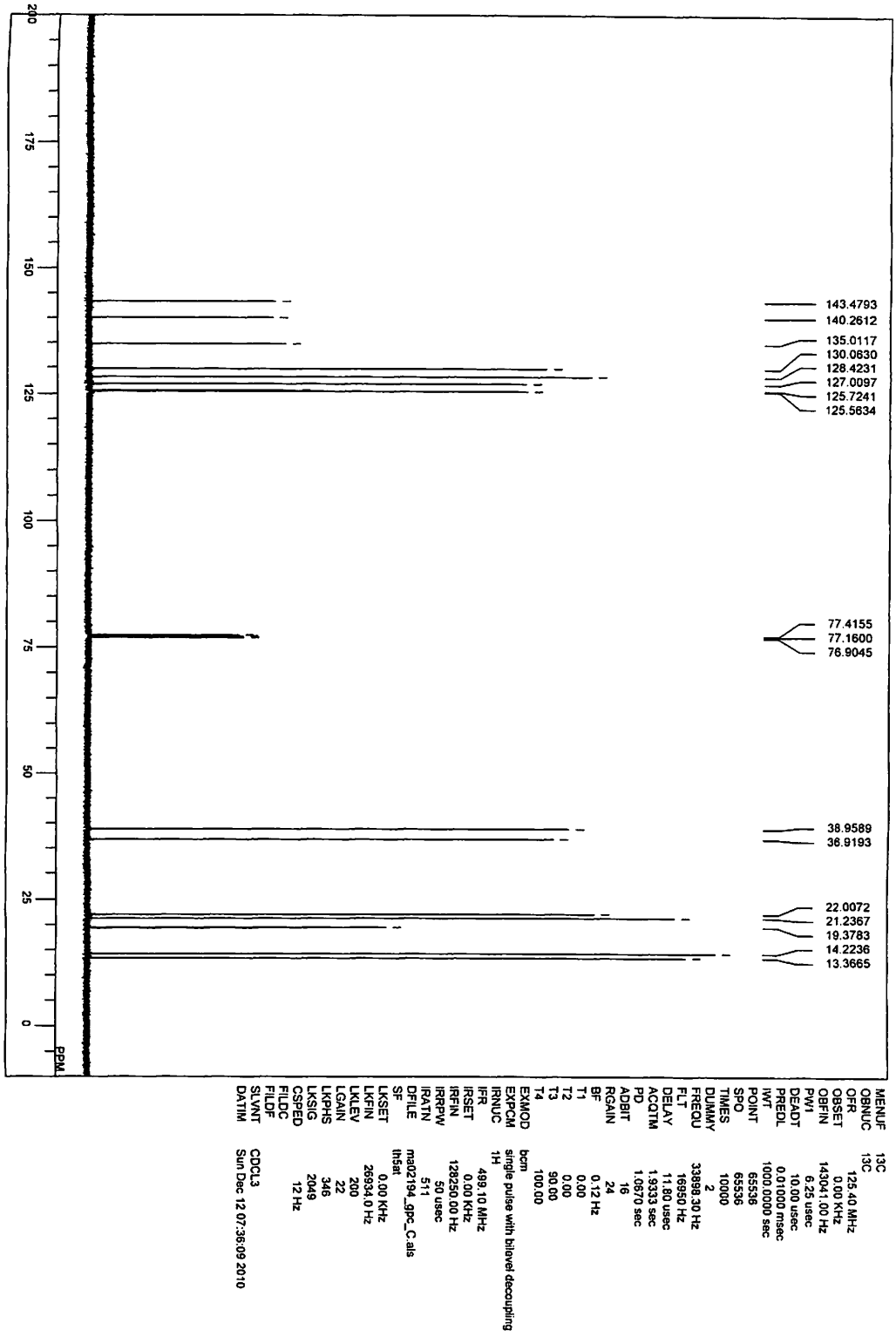
7.1974
7.1945
7.1888
7.1862
7.1830
7.1783
7.1725
7.1708
7.1681
7.1644
7.1583
7.1568
7.1522
7.1461
6.9597
6.9566
6.9473
6.9438
2.7195
2.7046
2.6907
2.6760
2.6608
2.6107
2.5965
2.5823
2.5679
2.5540
2.4757
2.4633
2.4554
2.4486
2.4430
2.4361
2.4283
2.4156
2.2422
2.2280
2.2222
2.2148
2.2114
2.2041
2.1948
2.1843
1.7432
1.7282
1.7136
1.6989
1.6842
1.6696
1.4267
1.4155
1.4113
1.4060
1.4006
1.3952
1.3910
1.3803
1.3781
1.3654
1.3578
1.3509
1.3453
1.3434
1.3365
1.3309
1.3162
1.0270
1.0120
0.9974
0.9130
0.8981
0.8834
2.996
1.000
1.023
0.988
0.976
4.013
2.023
1.949
3.012
3.060
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nompresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ms02194_gpc.als
SF lhsat_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 851
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu Nov 18 17:10:32 2010

9b


Br
Pr
Pr

^{13}C NMR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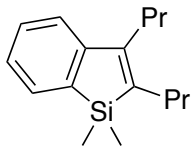
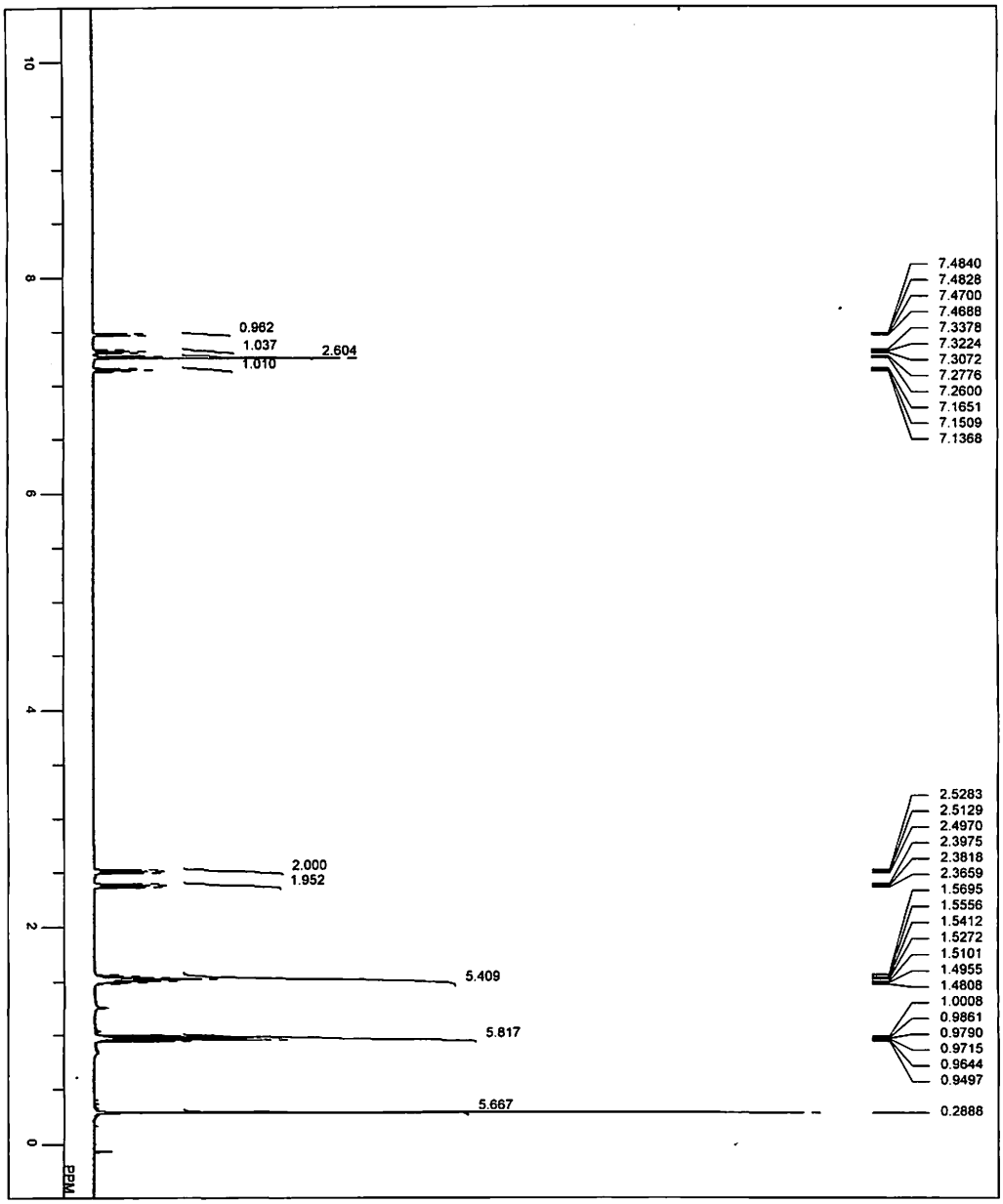
135.0117
130.0630
128.4231
127.0097
125.7241
125.5634
77.4155
77.1600
76.9045
38.9589
36.9193
22.0072
21.2367
19.3783
14.2236
13.3665
200
175
150
125
100
75
50
25
0
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 14304.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IVT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma02194_gpc_C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 2049
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Dec 12 07:36:09 2010

11am


Pr
Pr
Si

^{1}H NMR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7.4700
7.4688
7.3378
7.3224
7.3072
7.2776
7.2600
7.1651
7.1509
7.1368
0.962
1.037
1.010
2.604
2.5283
2.5129
2.4970
2.3975
2.3818
2.3659
1.5695
1.5556
1.5412
1.5272
1.5101
1.4955
1.4808
2.000
1.952
5.409
1.0008
0.9861
0.9790
0.9715
0.9644
0.9497
5.817
5.667
0.2888
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma021111_plic2.als
SF lh5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
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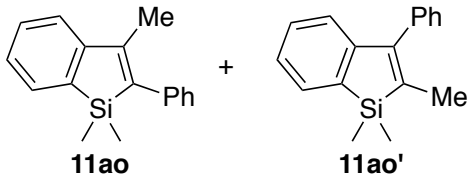
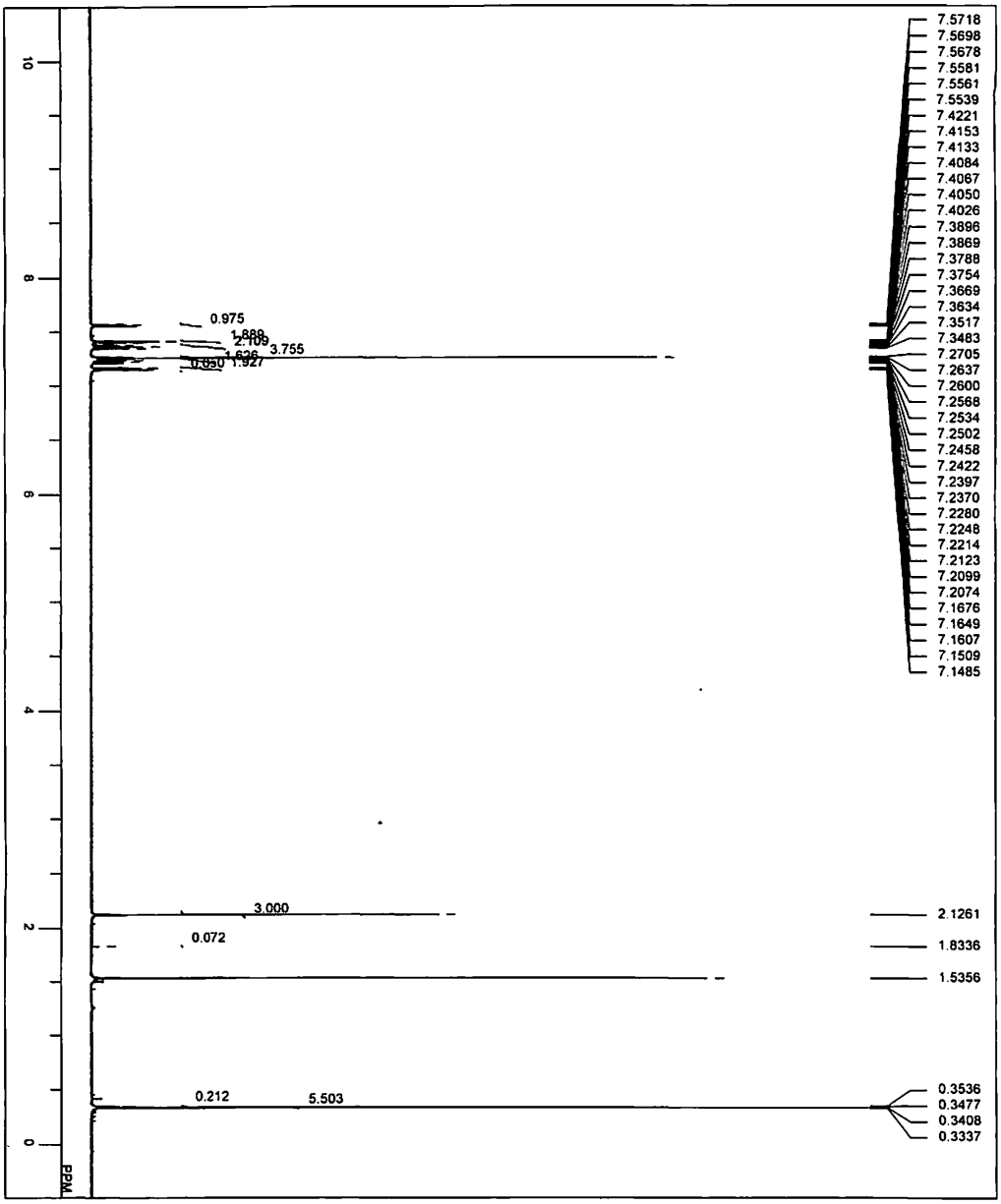
FILDC
FILDF
SLVNT CDCL3
DATIM Thu Aug 19 10:54:22 2010

11ao + **11ao'** (97:3)

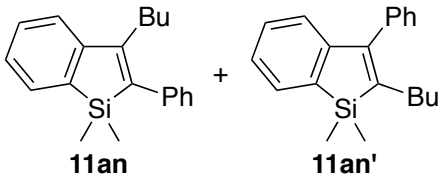


11ao **11ao'**

^{1}H NMR

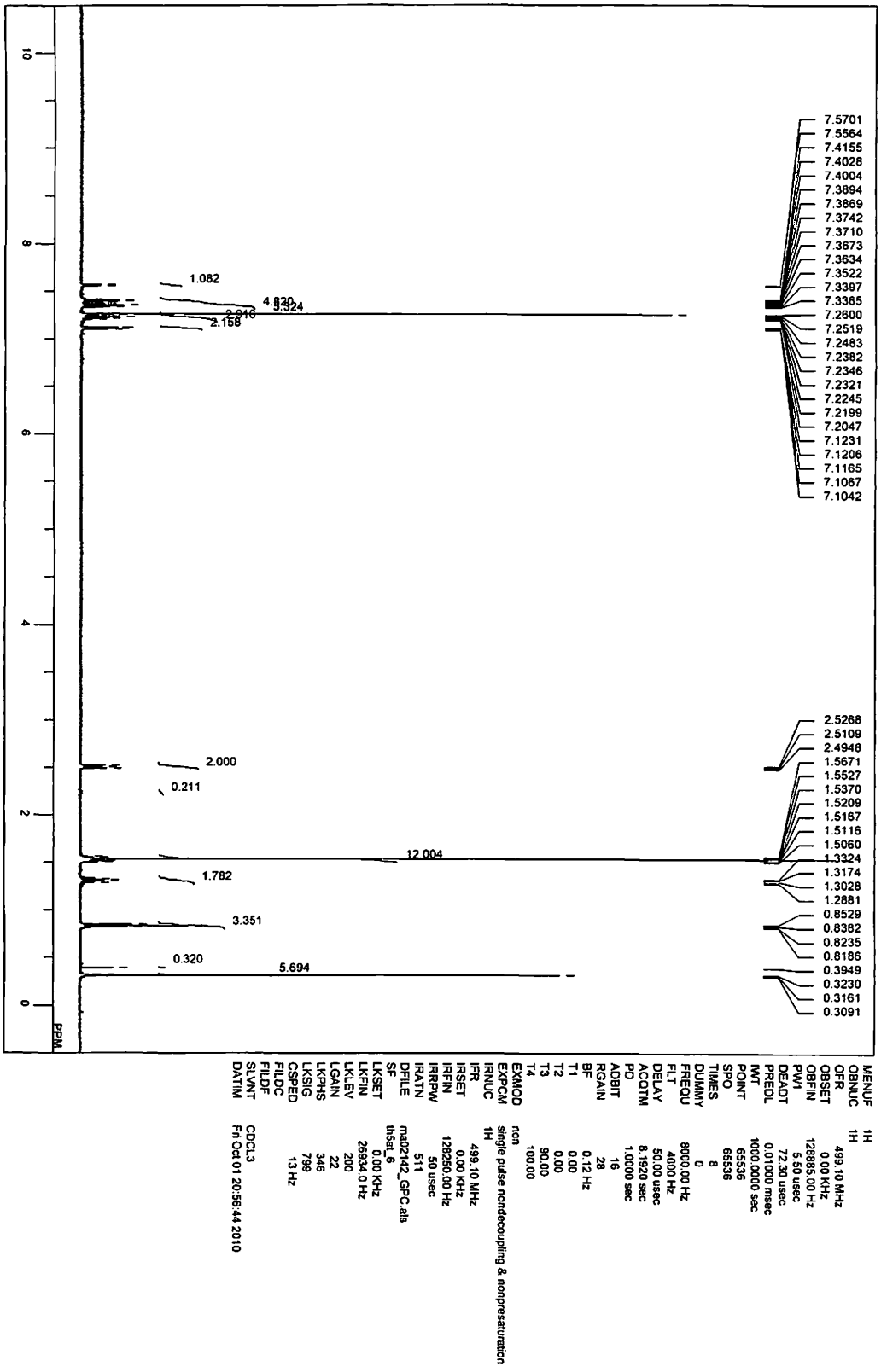


11an + **11an'** (94:6)

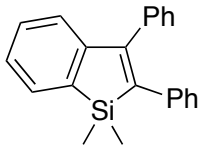


11an **11an'**

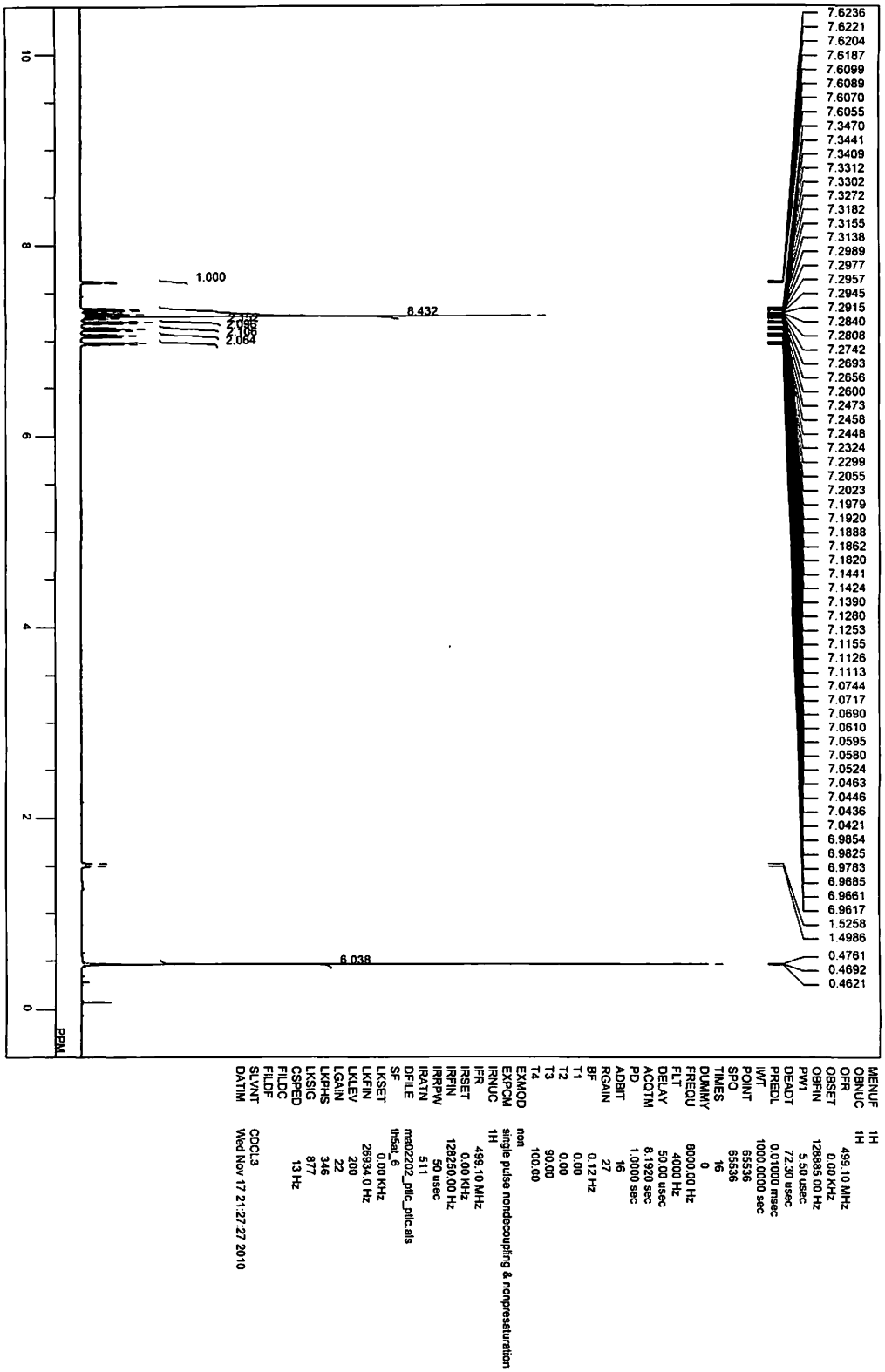
^{1}H NMR



11ap


Ph
Ph
Si

^{1}H NMR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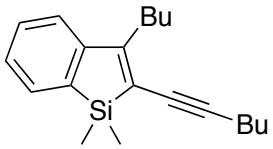
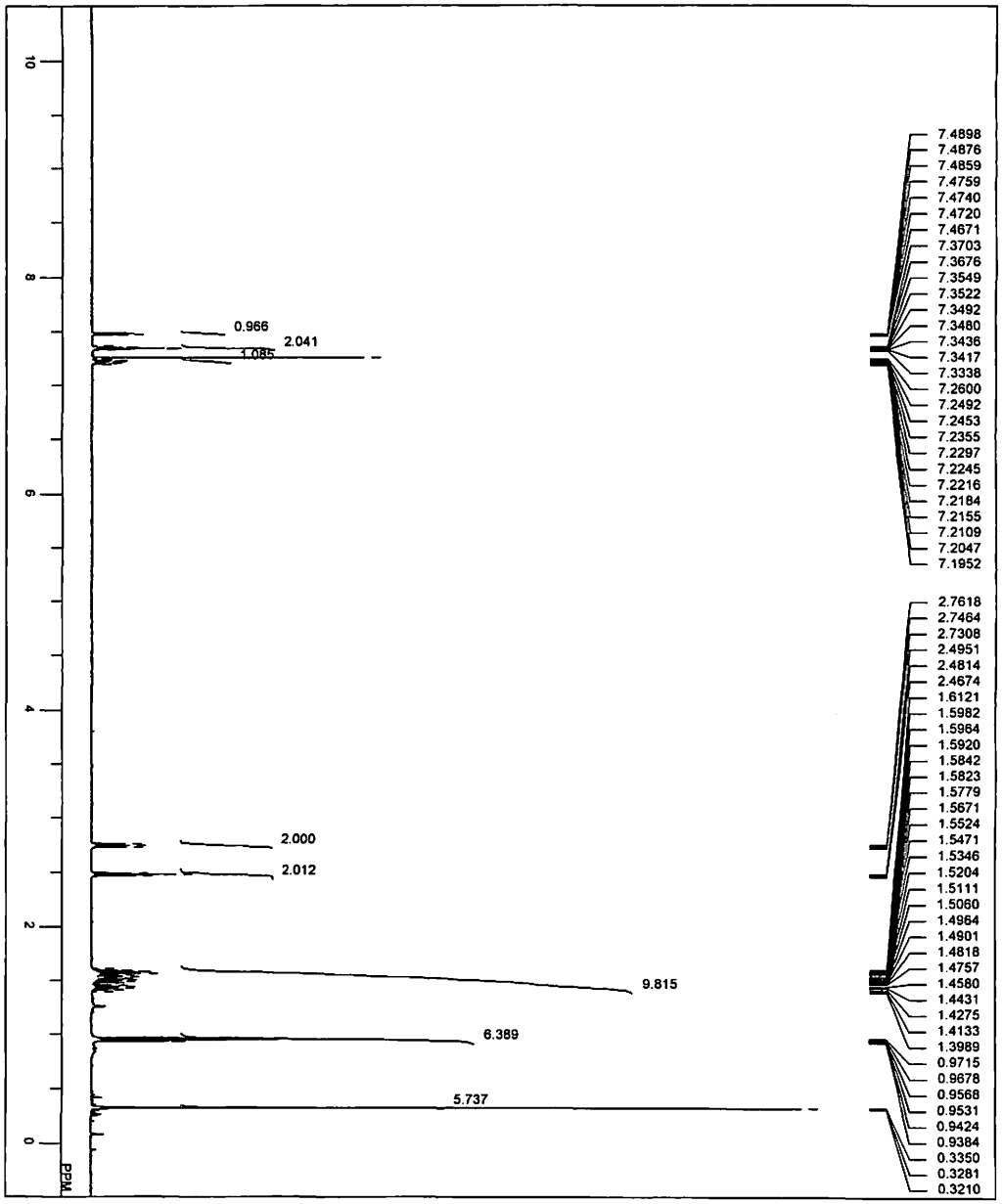
7.6204
7.6187
7.6099
7.6089
7.6070
7.6055
7.3470
7.3441
7.3409
7.3312
7.3302
7.3272
7.3182
7.3155
7.3138
7.2989
7.2977
7.2957
7.2945
7.2915
7.2840
7.2808
7.2742
7.2693
7.2656
7.2600
7.2473
7.2458
7.2448
7.2324
7.2299
7.2055
7.2023
7.1979
7.1920
7.1888
7.1862
7.1820
7.1441
7.1424
7.1390
7.1280
7.1253
7.1155
7.1126
7.1113
7.0744
7.0717
7.0690
7.0610
7.0595
7.0580
7.0524
7.0463
7.0446
7.0436
7.0421
6.9854
6.9825
6.9783
6.9685
6.9661
6.9617
1.5258
1.4986
0.4761
0.4692
0.4621
1.000
8.432
2.064
6.038
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma02202_ptic_ptic.als
SF tn5at_6
LKSET 0.00 KHz
LKFIN 28934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 877
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Wed Nov 17 21:27:27 2010

11as


Bu
Si
Bu

^{1}H NMR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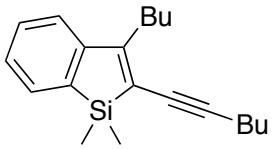
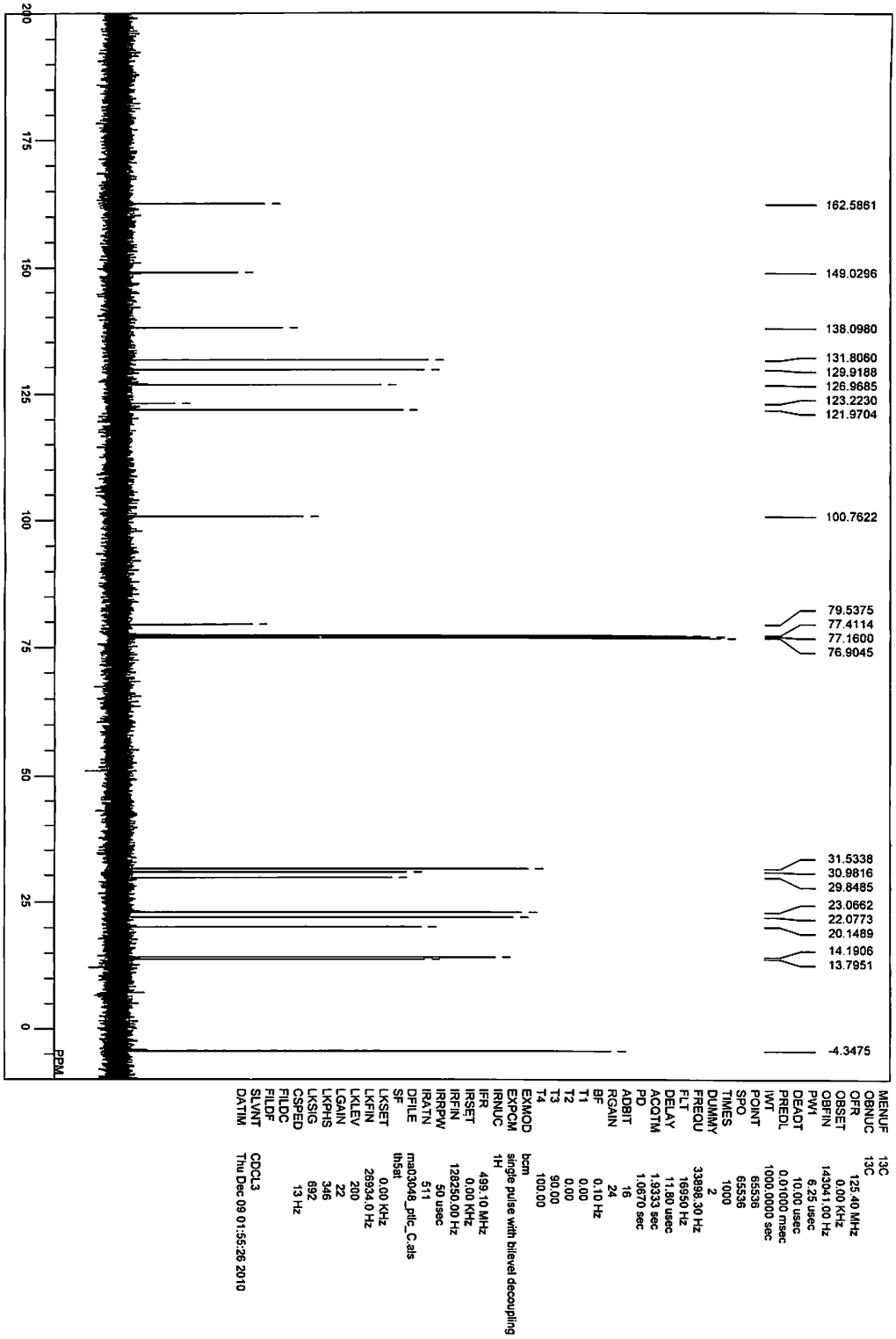
1.085
2.000
2.012
9.815
6.389
5.737
PPM
7.4898
7.4876
7.4859
7.4759
7.4740
7.4720
7.4671
7.3703
7.3676
7.3549
7.3522
7.3492
7.3480
7.3436
7.3417
7.3338
7.2600
7.2492
7.2453
7.2355
7.2297
7.2245
7.2216
7.2184
7.2155
7.2109
7.2047
7.1952
2.7618
2.7464
2.7308
2.4951
2.4814
2.4674
1.6121
1.5982
1.5964
1.5920
1.5842
1.5823
1.5779
1.5671
1.5524
1.5471
1.5346
1.5204
1.5111
1.5060
1.4964
1.4901
1.4818
1.4757
1.4580
1.4431
1.4275
1.4133
1.3989
0.9715
0.9678
0.9568
0.9531
0.9424
0.9384
0.3350
0.3281
0.3210
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128685.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 23
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma03048_ptc.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 934
CSPED 9 Hz
FILDF
FILDC
SLVNT CDCL3
DATIM Thu Dec 09 01:14:22 2010

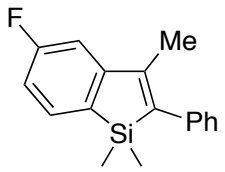
11as


Bu
Si
Bu

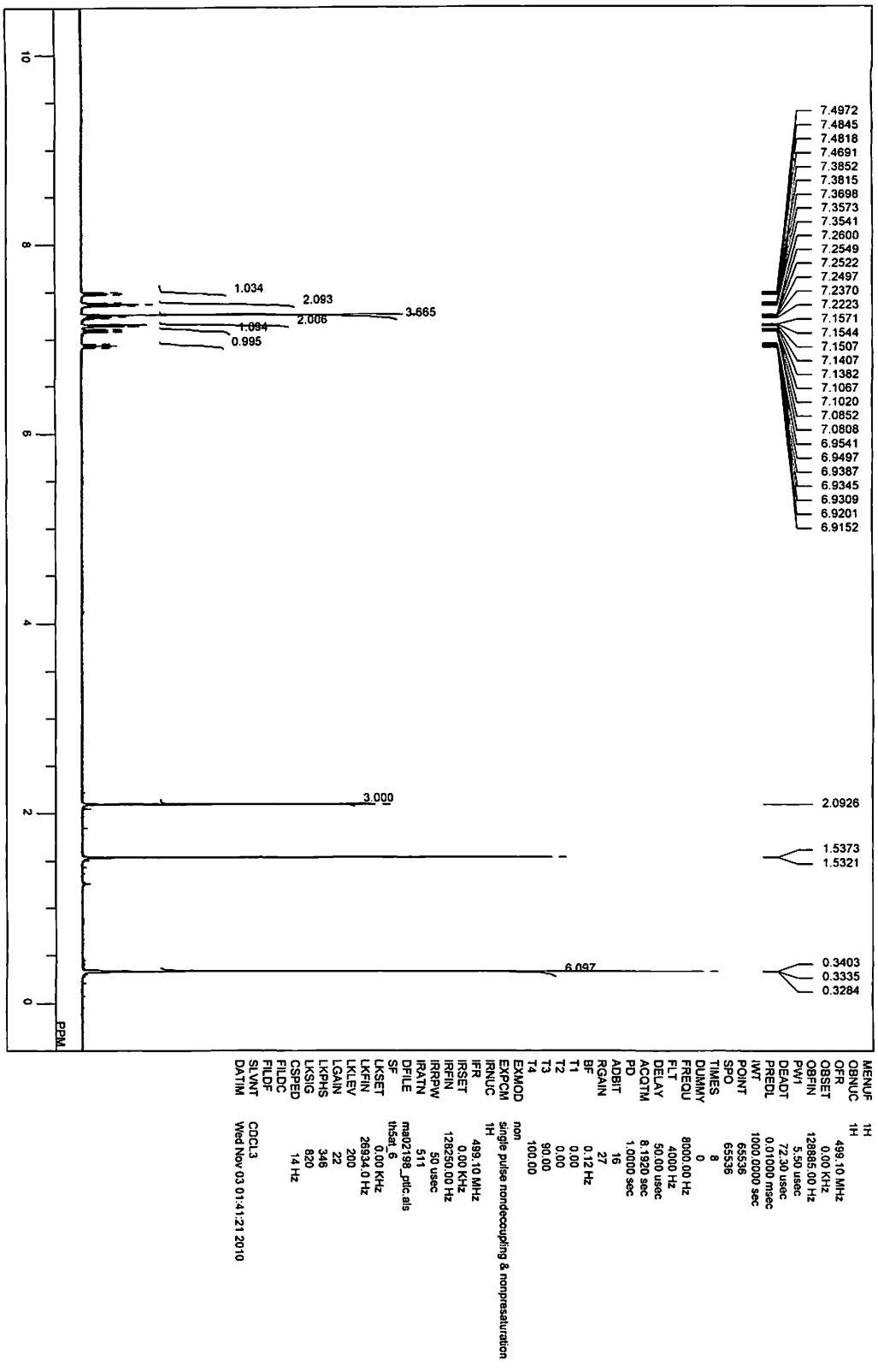
^{13}C NMR


200
175
150
125
100
75
50
25
0
PPM
162.5861
149.0296
138.0980
131.8060
129.9188
126.9685
123.2230
121.9704
100.7622
79.5375
77.4114
77.1600
76.9045
31.5338
30.9816
29.8485
23.0662
22.0773
20.1489
14.1906
13.7951
-4.3475
MENUF
OBNUC
OFR
OBSET
OBFIN
PW1
DEADT
PREDL
IWT
POINT
SPO
TIMES
DUMMY
FREQU
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC
FILDF
SLVNT
DATIM
13C
13C
125.40 MHz
0.00 KHz
14304.00 Hz
6.25 usec
10.00 usec
0.01000 msec
1000.0000 sec
65536
65536
1000
2
33898.30 Hz
16950 Hz
11.80 usec
1.9333 sec
1.0670 sec
24
16
0.10 Hz
0.00
0.00
90.00
100.00
bcm
single pulse with bilevel decoupling
1H
499.10 MHz
0.00 KHz
128250.00 Hz
50 usec
511
ma03048_ptic_C.als
tih5at
0.00 KHz
26934.0 Hz
200
22
346
692
13 Hz
CDCL3
Thu Dec 09 01:55:26 2010

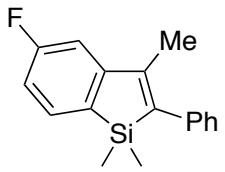
11bo


F
Me
Si
Ph

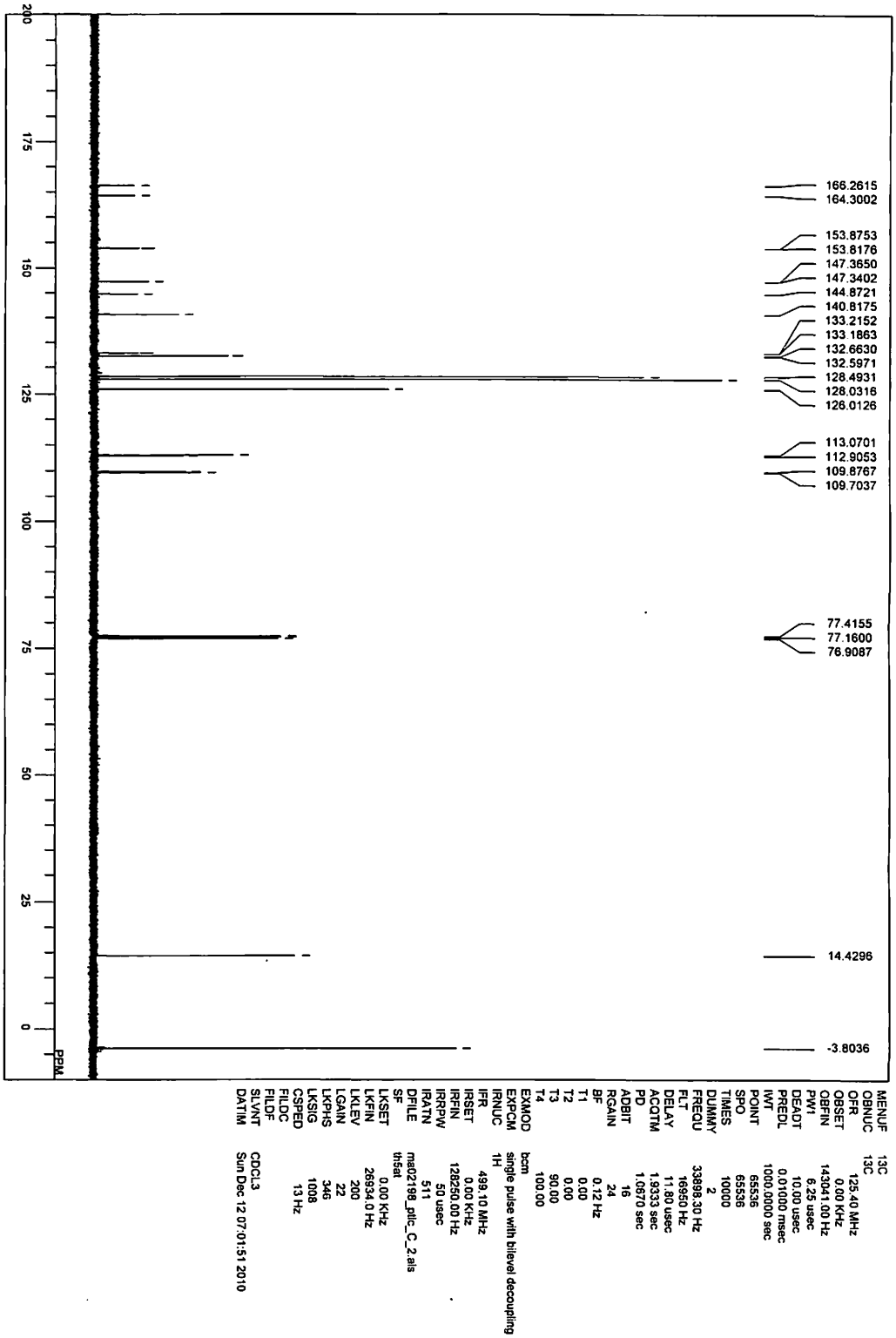
^{1}H NMR


7.4972
7.4845
7.4818
7.4691
7.3852
7.3815
7.3698
7.3573
7.3541
7.2600
7.2549
7.2522
7.2497
7.2370
7.2223
7.1571
7.1544
7.1507
7.1407
7.1382
7.1067
7.1020
7.0852
7.0808
6.9541
6.9497
6.9387
6.9345
6.9309
6.9201
6.9152
1.034
2.093
3.665
2.006
1.084
0.995
3.000
6.097
2.0926
1.5373
1.5321
0.3403
0.3335
0.3284
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.192D sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma02198_ptic.als
SF th5at_6
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 820
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Wed Nov 03 01:41:21 2010

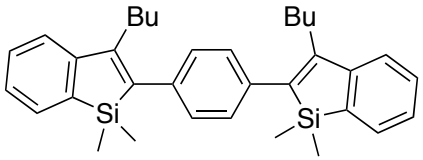
11bo


F
Me
Si
Ph

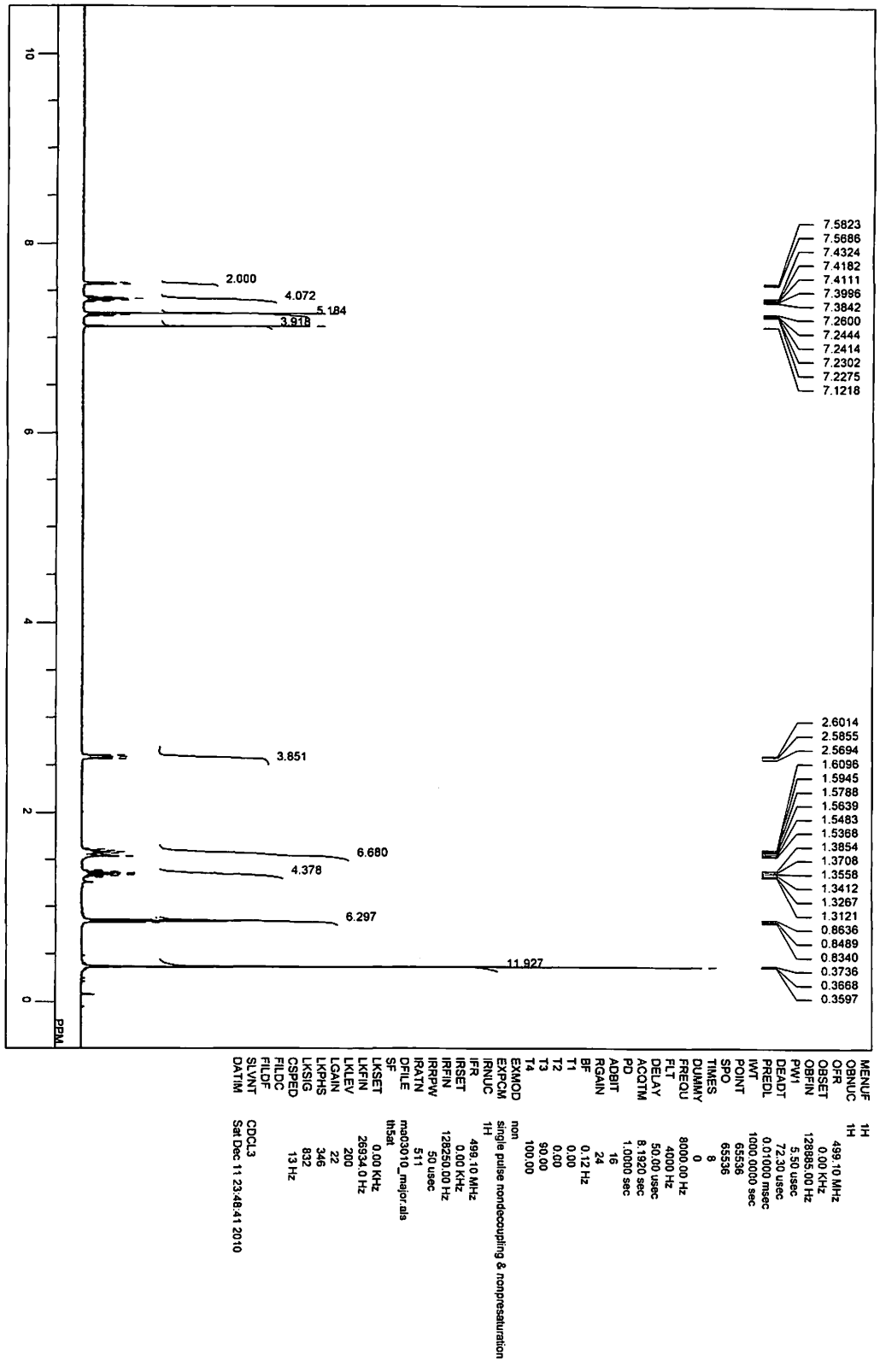
^{13}C NMR


166.2615
164.3002
153.8753
153.8176
147.3650
147.3402
144.8721
140.8175
133.2152
133.1863
132.6630
132.5971
128.4931
128.0316
126.0126
113.0701
112.9053
109.8767
109.7037
77.4155
77.1600
76.9087
14.4296
-3.8036
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 14304.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0570 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma02198_ptic_C_2.als
SF tfsat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 1008
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCl3
DATIM Sun Dec 12 07:01:51 2010

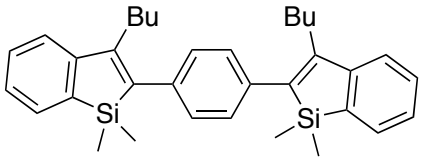
13am


Bu
Bu
Si
Si

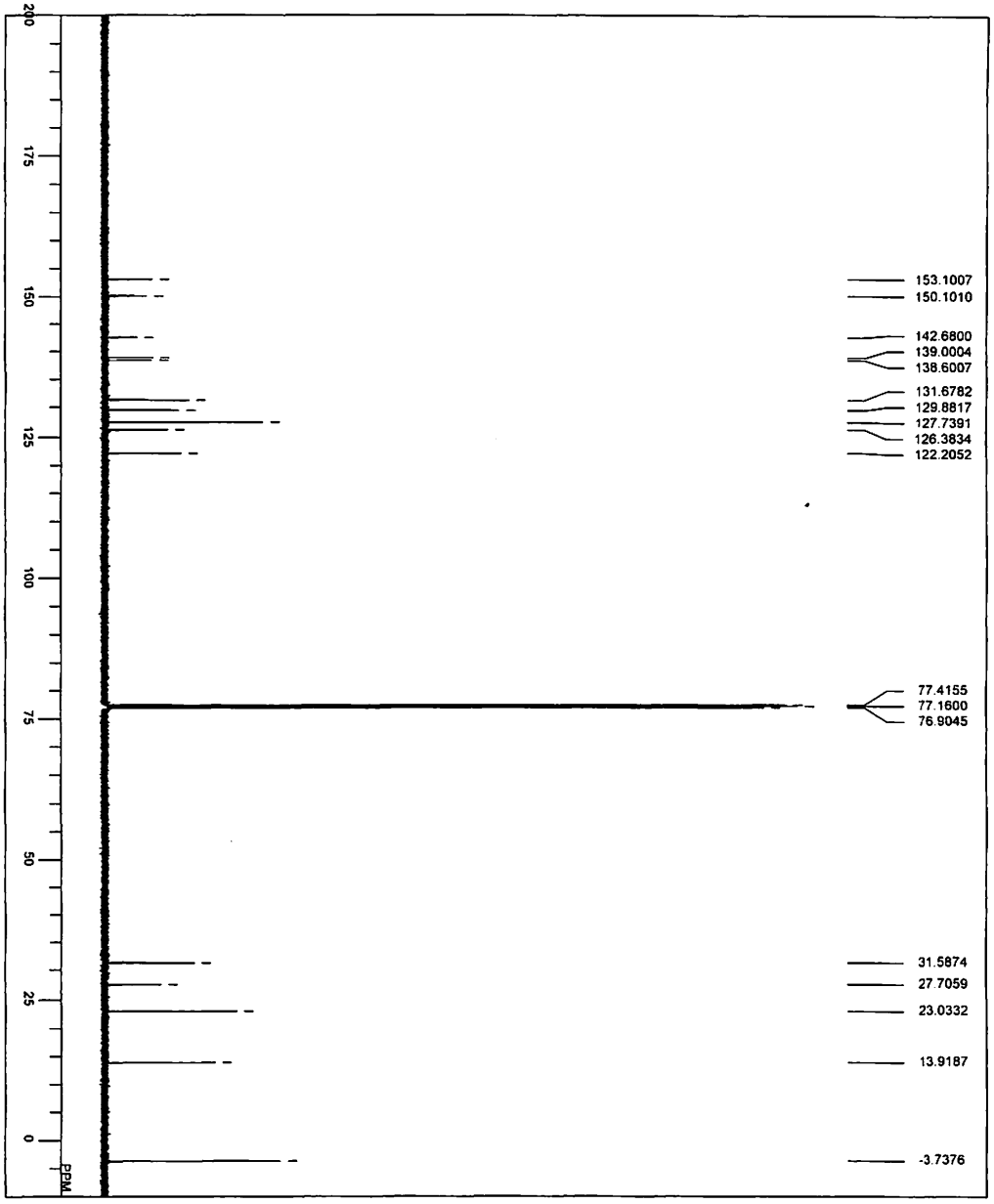
^{1}H NMR


7.5823
7.5686
7.4324
7.4182
7.4111
7.3996
7.3842
7.2600
7.2444
7.2414
7.2302
7.2275
7.1218
2.6014
2.5855
2.5694
1.6096
1.5945
1.5788
1.5639
1.5483
1.5368
1.3854
1.3708
1.3558
1.3412
1.3267
1.3121
0.8636
0.8489
0.8340
0.3736
0.3668
0.3597
2.000
4.072
5.184
3.918
3.851
6.680
4.378
6.297
11.927
PPM
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ms03010_major.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 832
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat Dec 11 23:48:41 2010

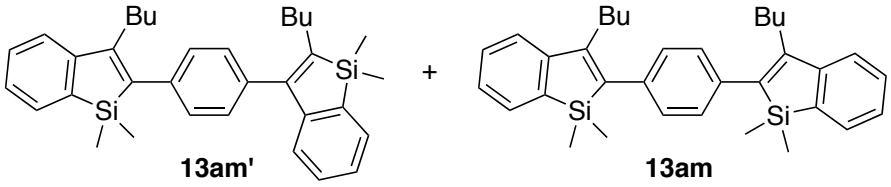
13am


Bu
Bu
Si
Si

^{13}C NMR

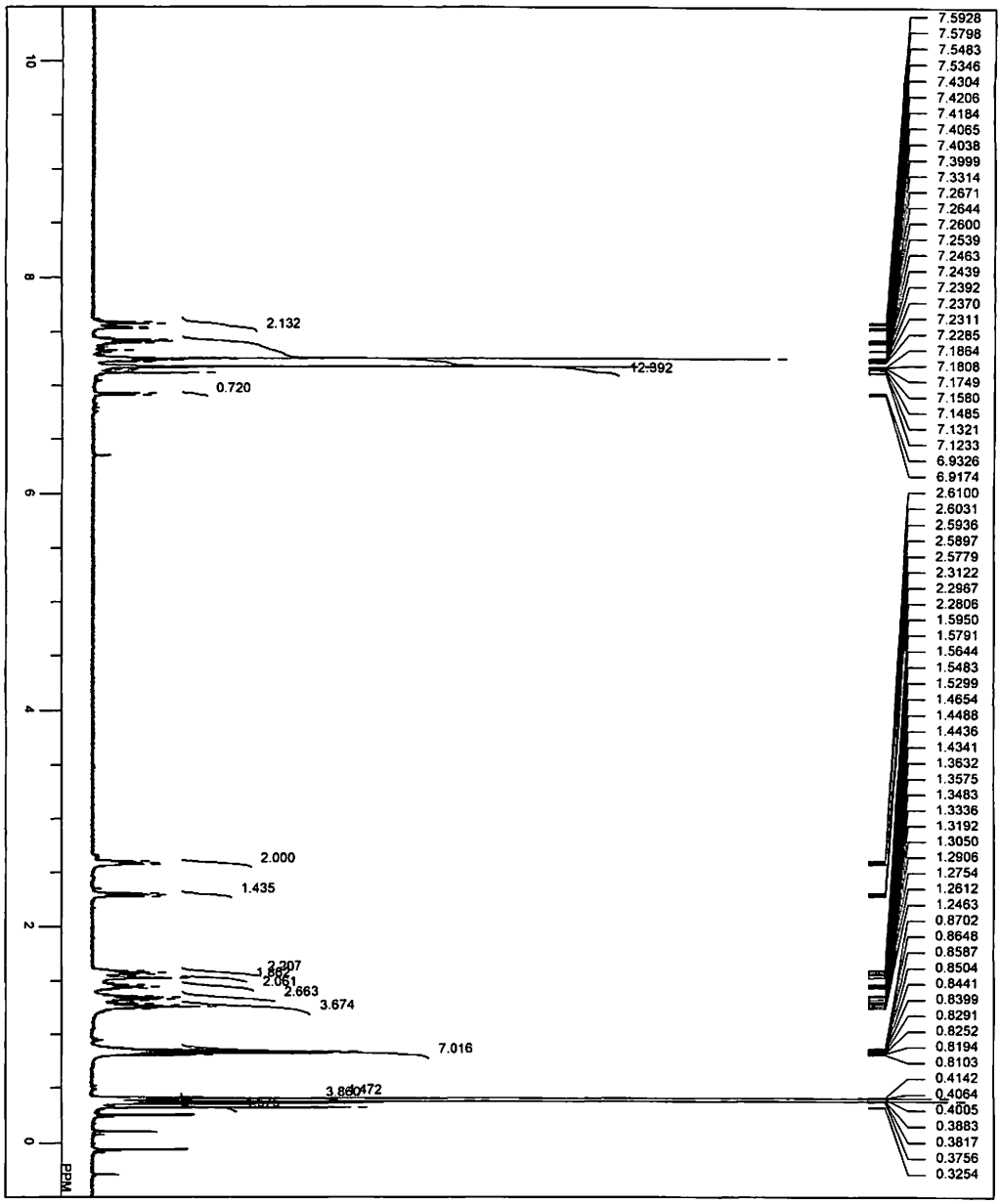

153.1007
150.1010
142.6800
139.0004
138.6007
131.6782
129.8817
127.7391
126.3834
122.2052
77.4155
77.1600
76.9045
31.5874
27.7059
23.0332
13.9187
-3.7376
PPM
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 14304.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IVT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 10000
DUMMY 2
FREQU 33998.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0570 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma030110_majorC.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 459
CSPED 10 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sun Dec 12 05:18:01 2010

13am' + **13am** (84:16)

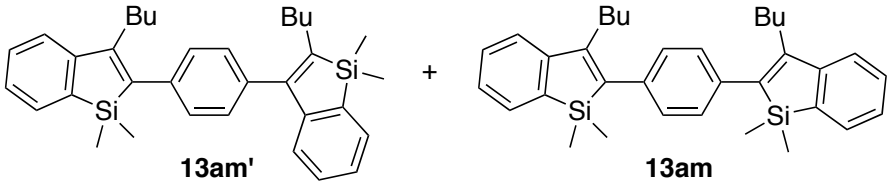


13am' **13am**

^{1}H NMR

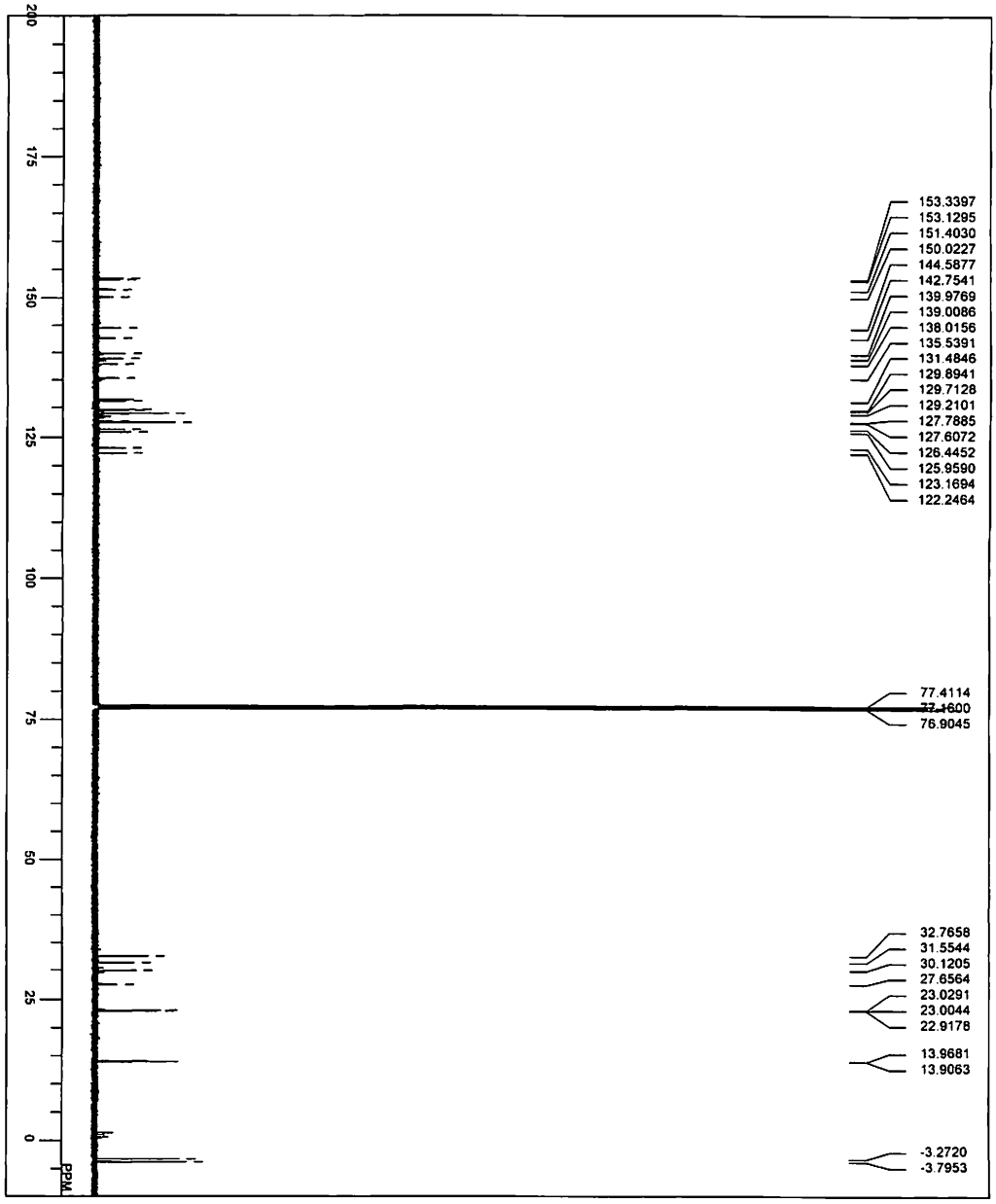


13am' + **13am** (84:16)

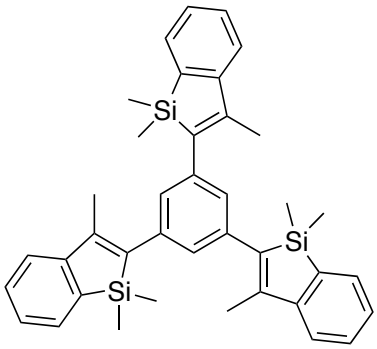


13am' **13am**

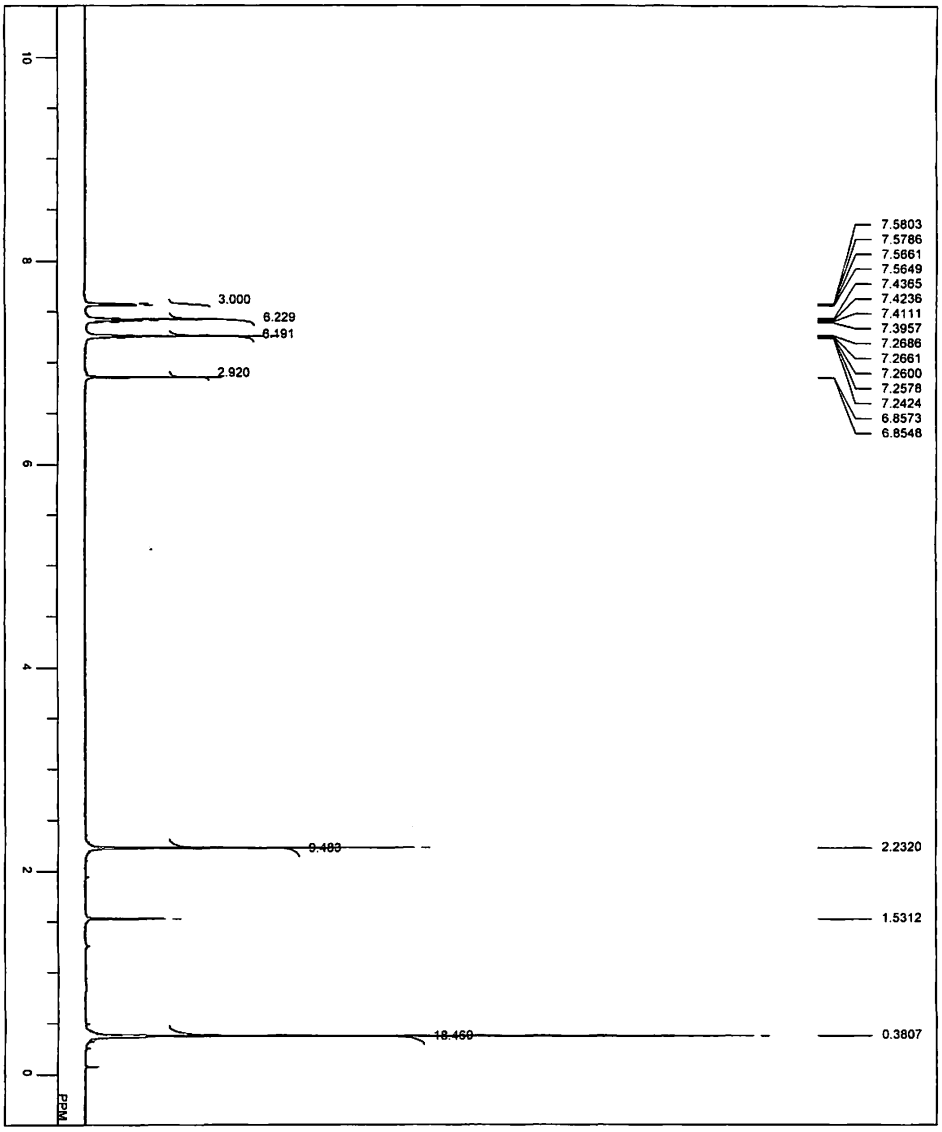
^{13}C NMR



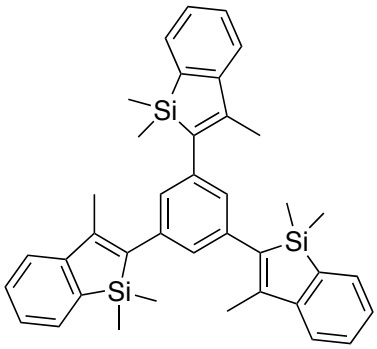
13an


Si
Si
Si

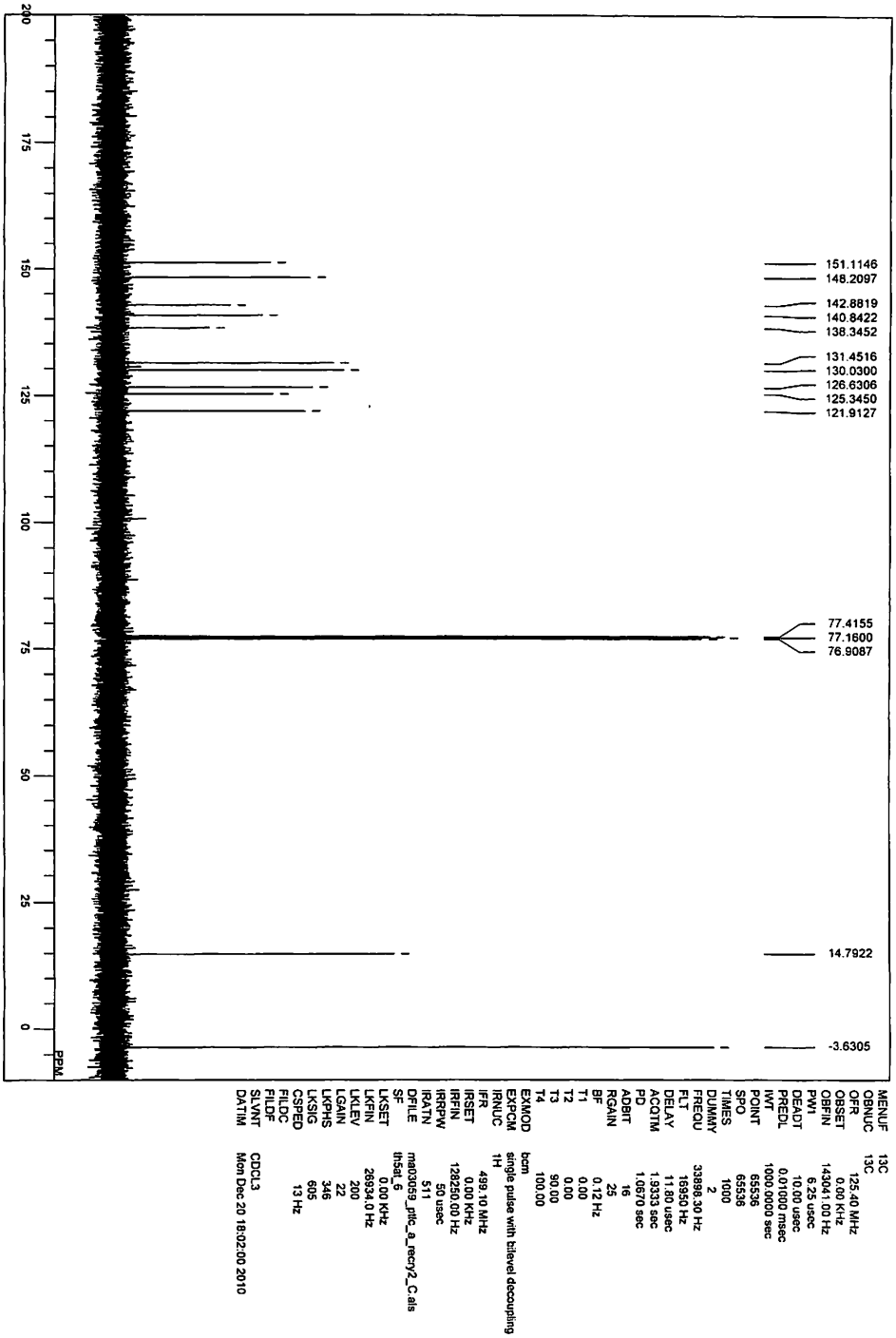
^{1}H NMR


7.5803
7.5786
7.5661
7.5649
7.4365
7.4236
7.4111
7.3957
7.2686
7.2661
7.2600
7.2578
7.2424
6.8573
6.8548
3.000
6.229
6.191
2.920
9.483
18.469
2.2320
1.5312
0.3807
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128685.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ma030699_pllc_a_recry2_2.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 346
LKSIG 698
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Wed Jan 12 17:07:39 2011

13an


Si
Si
Si

^{13}C NMR


151.1146
148.2097
142.8819
140.8422
138.3452
131.4516
130.0300
126.6306
125.3450
121.9127
77.4155
77.1600
76.9087
14.7922
-3.6305
PPM
MENUF
OBNUC
OFR
OBSET
OBFIN
PW1
DEADT
PREDL
IWT
POINT
SPO
TIMES
DUMMY
FREQU
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IFR
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC
FILDF
SLVNT
DATIM
13C
13C
125.40 MHz
0.60 KHz
143041.00 Hz
6.25 usec
10.00 usec
0.01000 msec
1000.0000 sec
65536
65536
1000
2
33898.30 Hz
16950 Hz
11.80 usec
1.9333 sec
1.0670 sec
16
25
0.12 Hz
0.00
0.00
90.00
100.00
bcm
single pulse with bilevel decoupling
1H
499.10 MHz
0.00 KHz
128250.00 Hz
50 usec
511
ma030059_ptfc_a_recy2_C.als
th5at_6
0.00 KHz
26934.0 Hz
200
22
346
605
13 Hz
CDCL3
Mon Dec 20 18:02:00 2010