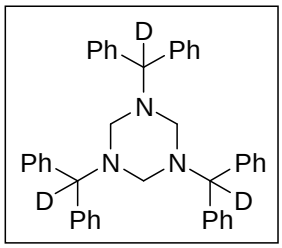
Supporting Information

Rhenium-Catalyzed Regio- and Stereoselective Addition of Imines to Terminal Alkynes Leading to *N*-Alkylideneallylamines

Yoshiya Fukumoto,* Masato Daijo, and Naoto Chatani

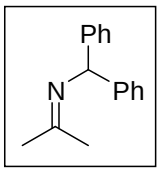
Department of Applied Chemistry, Faculty of Engineering, Osaka University, Suita, Osaka 565-0871, Japan

General Information. ^{1}H NMR and ^{13}C NMR spectra were recorded on a JEOL JNM-ECS400 spectrometer using $CDCl_3$ as solvent. Data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, br = broad singlet, m = multiplet, c = complex), coupling constant (Hz), integration, and interpretation. Infrared spectra (IR) were obtained on a Horiba FT-720 spectrometer; absorption data are reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak). Mass spectra were obtained using a Shimadzu GCMS-QP 2010 instrument with an ionization voltage of 70 eV. Medium-pressure column chromatography was carried out on a Biotage Flash Purification System Isolera, equipped with a 254 nm UV detector. Elemental analyses were performed by the Elemental Analysis Section of Osaka University. High resolution mass spectra (HRMS) were obtained on a JEOL JMS-DX303. Analytical gas chromatography (GC) was carried out on a Shimadzu GC-2014 gas chromatograph, equipped with a flame ionization detector. Toluene (Kanto Chemical) was purified by passage through activated alumina using a GlassContour Solvent Dispensing System. Alkynes **1a** (TCI), **1b** (Aldrich), **1d** (Wako), **1e** (Aldrich), **1i** (Wako), **1j** (Aldrich), 3,3-dimethyl-1-butyne (TCI) and trimethylsilylacetylene (TCI) were purchased and purified by distillation before use. $P(C_6F_5)_3$ was commercially available from TCI and was purified by recrystallization before use. $ReBr(CO)_5$ purchased from Aldrich, and were used without further purification. Imine **2a'** was commercially available from Aldrich and was purified by recrystallization before use. Alkynes, **1c**,[1] **1f**,[2] **1g**,[2] **1h**,[3] **1a-*d***[4] and Imines **2a**,[5] **2a''**,[6] **2b**,[7] **2c**,[8] **2d**,[9] **2e**,[10] and **2f**[8] were prepared according to the procedures described in the literature.



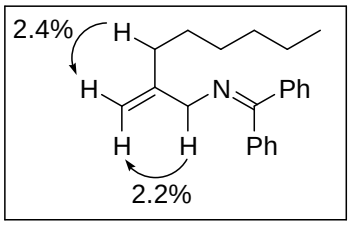
Procedure for the Synthesis of 1,3,5-trideuteriodiphenylmethylhexahydro-1,3,5-triazine (pre-2a-*d*). *N*-Deuteriodiphenylmethylamine (1.29 g, 6.96 mmol, prepared by the literature method[11]), benzene (25 mL) and 37 % HCHO *aq* (1.5 mL) were added to the 50 mL flask. The reaction mixture was warmed to 110 °C under Dean-Stark conditions. After 12h, the reaction mixture was cooled to room temperature, and filtered through a filter paper. The resulting white solids were purified by crystallization from toluene. Mp = 208 ºC. ^{1}H NMR ($CDCl_3$): δ 4.68 (s, 0.03H, CH), 6.99-7.03 (c, 6H, Ph), 7.05-7.09 (c, 12H, Ph), 7.23-7.36 (c, 12H, Ph). ^{13}C NMR ($CDCl_3$): δ 69.4 (t, *J* = 78 Hz, CD), 71.7 (CH_2N), 126.6 (Ph), 127.4 (Ph), 128.2 (Ph), 142.2 (Ph). IR (KBr): 3082 w (=C-H), 3060 w (=C-H), 3023 w (=C-H), 3001 w (=C-H), 2790 (C-D) cm^{-1}. MS: *m*/*z* (relative intensity): 169 (10), 168 (100), 166 (29), 153 (13), 107 (13) 83 (15). HRMS Calcd for $C_{42}H_{36}D_3N_3Na$ (M^++Na): 611.3227. Found: 611.3235.

Procedure for the Synthesis of *N*-(Propan-2-ylidene)diphenylmethylamine (2g). 2g was synthesized according to the literature procedure.[12] A 50 mL two-necked flask was flame-dried and purged with N_2. After cooling to room temperature, molecular sieves 4A (9.70 g, purchased from Wako) were added to the flask, followed by ether (8 mL), *N*-diphenylmethylamine (3.17 g, 17 mmol), and acetone (1.30 g, 22 mmol). The reaction mixture was stirred at room temperature. After 18 h, the reaction mixture was filtered through a pad of Celite. The filtrate was evaporated to give white solids, which was crystallized from hexane. Mp = 70 ºC. ^{1}H NMR ($CDCl_3$): δ 1.91 (s, 3H, CH_3), 2.13 (s, 3H, CH_3), 5.62 (s, 1H, CH), 7.17-7.23 (c, 2H, Ph), 7.27-7.35 (c, 8H, Ph). ^{13}C NMR ($CDCl_3$): δ 19.1 (CH_3), 29.7 (CH_3), 67.9 (CH), 126.6 (Ph), 127.6 (Ph), 128.3 (Ph), 144.4 (Ph), 167.4 (C=N). IR (KBr): 3081 m (=C-H), 3062 m (=C-H), 3021 m (=C-H), 2989 m (C-H), 2944 w (C-H), 2910 m (C-H), 2886 m (C-H), 1654 s (C=N) cm^{-1}. MS: *m*/*z* (relative intensity): 223 (9, M^+), 168 (15), 167 (100), 165 (28), 152 (16), 83 (18). HRMS Calcd for $C_{16}H_{17}N$ (M^+): 223.1361. Found: 223.1355.



Typical Procedure for the Rhenium-Catalyzed Addition of Imines to Terminal Alkynes. A 25 mL Schlenk tube was dried for 2 h in an oven at 120 ºC and then purged with N_2. After cooling to room temperature, $ReBr(CO)_5$ (40.6 mg, 0.1 mmol), $P(C_6F_5)_3$ (106.4 mg, 0.2 mmol), and the imine (1.5 mmol) were added to the tube. The mixture was dried for 5 min under reduced pressure. After purging the system with N_2 gas again, toluene (2 mL) and the alkyne (1 mmol) were added to the tube. The reaction mixture was heated in an oil-bath at 110 ºC for 24 h. After cooling to room temperature, the volatiles were removed *in vacuo.* The residue was purified by column chromatography on NH_2 silica-gel (Fuji Silysia Chemical LTD. NH-DM1020 (70-230 mesh)) and the product was obtained as a colorless oil. An Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography (YMC-DispoPackAT NH2-25: 40g) followed by bulb-to-bulb distillation.

***N*-Diphenylmethylidene-2-methylideneoct-1-ylamine (3a). 3a** was isolated by NH_2 silica-gel column chromatography using hexane as the eluent (R_f = 0.17 in hexane; R_f = 0.46 in hexane/EtOAc = 20/1) in 73% yield as colorless oil. An analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (140 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.87 (t, *J* = 7.0 Hz, 3H, CH_3), 1.20-1.31 (m, 6H, CH_2), 1.39 (quint, *J* = 7.6 Hz, 2H, =CCH_2CH_2), 2.07 (t, *J* = 7.6 Hz, 2H, =CCH_2), 3.95 (s, 2H, CH_2N), 4.84 (s, 1H, *E*-CH), 4.91 (s, 1H, *Z*-CH), 7.16-7.20 (c, 2H, Ph), 7.30-7.47 (c, 6H, Ph), 7.64-7.66 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 14.1 (CH_3), 22.6 (CH_2), 27.6 (CH_2), 29.0 (CH_2), 31.7 (CH_2), 35.0 (=C*C*H_2), 58.2 (CH_2N), 109.5 (=CH_2), 127.7 (Ph), 128.0 (Ph), 128.4 (Ph), 128.4 (Ph), 128.5 (Ph), 129.9 (Ph), 136.5 (Ph), 139.8 (Ph), 148.5 (=C), 168.6 (N=C). IR (neat): 3079 (=C-H), 3059 m (=C-H), 2956 s (C-H), 2927 s (C-H), 1624 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 305 (11, M^+), 304 (17), 262 (23), 250 (23), 249 (19), 248 (47), 235 (18), 234 (26), 228 (17), 220 (12), 206 (21), 194 (21), 193 (14), 182 (35), 180 (17), 167 (12), 166 (11), 165 (30), 158 (18), 117 (12), 115 (14), 104 (40), 91 (100), 81 (12), 77 (36), 69 (12), 67 (13), 65 (12), 55 (28), 53 (11), 51 (13). Anal. Calcd for $C_{22}H_{27}N$: C, 86.51; H, 8.91; N, 4.59. Found: C, 86.30; H, 8.96; N, 4.60.



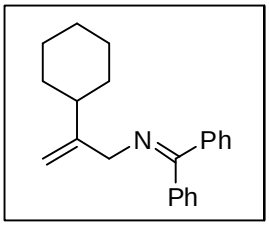
2-methylideneoct-1-ylamine hydrochloride ($3a_{NH2}$). Hydrolysis of **3a** was carried out according to the literature procedure.[13] To **3a** (152 mg, 0.50 mmol) was added MeOH (5 mL) and 1M HCl *aq* (5 mL). The reaction mixture was stirred for 12 h at room temperature. The volatiles were removed *in vacuo*. The residue was washed with ether, and filtered through a filter paper. $\mathbf{3a_{NH2}}$ (76 mg) was obtained as white solids in 85% yield. Mp = 103-104ºC. ^{1}H NMR ($CDCl_3$): δ 0.88 (t, *J* = 6.8 Hz, 3H, CH_3), 1.28-1.32 (c, 6H, CH_2), 1.41-1.46 (c, 2H, =CCH_2CH_2), 2.13 (t, *J* = 7.6 Hz, 2H, =CCH_2), 3.53 (q, *J* = 5.6 Hz, 2H, CH_2N), 5.12 (s, 1H, =CH), 5.20 (s, 1H, =CH), 8.60 (bs, 2H, NH_2). ^{13}C NMR ($CDCl_3$): δ 14.1 (CH_3), 22.6 (CH_2), 27.3 (CH_2), 28.9 (CH_2), 31.7 (=CCH_2CH_2), 34.3 (=CCH_2), 43.6 (CH_2N), 114.2 (=CH_2). 141.1 (=C). IR (KBr): 3032 m (=C-H), 2958 s (C-H), 2925 s (C-H) cm^{-1}. MS: *m*/*z* (relative intensity): 141 (1, M^+-HCl), 98 (10), 95 (10), 81 (12), 70 (39), 56 (100). HRMS Calcd for $C_9H_{19}N$ (M^+-HCl): 141.1517. Found: 141.1519.

***Z*-Deuterio-*N*-diphenylmethylidene-2-methylideneoct-1-ylamine (3a-*Z*-*d*).** Colorless oil. ^{1}H NMR ($CDCl_3$): δ 0.87 (t, *J* = 7.8 Hz, 3H, CH_3), 1.23-1.31 (c, 6H, CH_2), 1.39 (quint, *J* = 7.2 Hz, 2H, =CCH_2CH_2), 2.07 (t, *J* = 7.2 Hz, 2H, =CCH_2), 3.95 (s, 2H, CH_2N), 4.83 (d, *J* = 1.6 Hz, 1H, =CH), 4.92 (s, 0.01H, =CH), 7.16-7.20 (c, 2H, Ph), 7.30-7.47 (c, 6H, Ph), 7.64-7.66 (c, 2H, Ph).

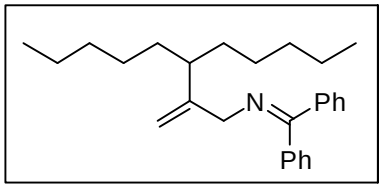
***E*-Deuterio-*N*-diphenylmethylidene-2-methylideneoct-1-ylamine (3a-*E*-*d*).** Colorless oil. ^{1}H NMR ($CDCl_3$): δ 0.87 (t, *J* = 6.8 Hz, 3H, CH_3), 1.25-1.30 (c, 6H, CH_2), 1.39 (quint, *J* = 7.2 Hz, 2H, =CCH_2CH_2), 2.08 (t, *J* = 7.2 Hz, 2H, =CCH_2), 3.95 (s, 2H, CH_2N), 4.84 (s, 0.1H, =CH), 4.91 (s, 1H, =CH), 7.16-7.20 (c, 2H, Ph), 7.30-7.46 (c, 6H, Ph), 7.64-7.67 (c, 2H, Ph).

***N*-Benzylidene-2-methylideneoct-1-ylamine (3a'). 3a'** was isolated by flash column chromatography on SiO_2 (Silicycle SiliaFlash F60 (230-400 mesh)) using hexane/AcOEt (20/1) as the eluent (R_f = 0.20 in hexane/EtOAc = 20/1) in 23% yield as colorless oil. An Analytically pure sample was obtained by bulb-to-bulb distillation (90 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.88 (t, *J* = 7.0 Hz, 3H, CH_3), 1.27-1.36 (c, 6H, CH_2), 1.50 (quint, *J* = 7.4 Hz, 2H, =CCH_2CH_2), 2.12 (t, *J* = 7.4 Hz, 2H, =CCH_2), 4.18 (s, 2H, CH_2N), 4.87 (s, 1H, =CH), 4.92 (s, 1H, =CH), 7.39-7.44 (c, 3H, Ph), 7.75-7.79 (c, 2H, Ph), 8.28 (s, 1H, N=CH). ^{13}C NMR ($CDCl_3$): δ 14.1 (CH_3), 22.6 (CH_2), 27.6 (CH_2), 29.0 (CH_2), 31.7 (CH_2), 34.7 (=CCH_2), 65.8 (CH_2N), 110.3 (=CH_2), 128.1 (Ph), 128.5 (Ph), 130.6 (Ph), 136.1 (Ph), 147.7 (=C), 161.9 (N=C). IR (neat): 3083 w (=C-H), 3064 w (=C-H), 3026 w (=C-H), 2956 m (C-H), 2927 s (C-H), 2856 m (C-H), 1645 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 229 (1, M^+), 186 (10), 172 (100), 159 (11), 158 (45), 144 (15), 129 (18), 118 (41), 117 (40), 106 (50), 105 (10), 104 (57), 95 (10), 91 (92), 90 (31), 89 (19), 86 (11), 82 (19), 81 (21), 79 (22), 77 (22), 69 (21), 68 (20), 67 (22), 65 (17), 56 (17), 55 (39), 53 (15), 51 (10). HRMS Calcd for $C_{16}H_{23}N$ (M^+): 229.1830. Found: 229. 1834.

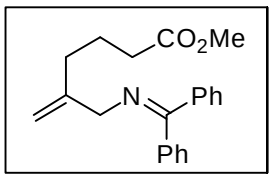
2-Cyclohexyl-*N*-diphenylmethylideneprop-2-en-1-amine (3b). 3b was isolated by NH_2 silica-gel column chromatography (R_f = 0.13 in hexane; R_f = 0.40 in hexane/EtOAc = 20/1) in 72% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (145 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 1.09-1.29 (c, 5H, CH_2), 1.64-1.74 (c, 5H, CH_2), 1.93 (t, *J* = 11.6 Hz, 1H, =CCH), 3.99 (s, 2H, CH_2N), 4.86 (s, 1H, =CH), 4.89 (d, *J* = 1.8 Hz, 1H, =CH), 7.16-7.18 (c, 2H, Ph), 7.31-7.46 (c, 6H, Ph), 7.64-7.66 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 26.3 (CH_2), 26.7 (CH_2), 32.3 (CH_2), 43.1 (=CCH), 57.2 (CH_2N), 107.7 (=CH_2), 127.6 (Ph), 128.0 (Ph), 128.3 (Ph), 128.4 (Ph), 128.4 (Ph), 129.9 (Ph), 136.5 (Ph), 139.9 (Ph), 153.6 (=C), 168.6 (N=C). IR (neat): 3079 m (=C-H), 3058 m (=C-H), 3023 m (=C-H), 2925 s (C-H), 2850 s (C-H), 1623 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 303 (44, M^+), 302 (51), 260 (10), 248 (12), 227 (11), 226 (61), 220 (21), 206 (12), 194 (26), 183 (14), 182 (90), 180 (19), 167 (31), 166 (14), 165 (40), 144 (10), 136 (12), 122 (10), 117 (13), 116 (10), 115 (19), 107 (29), 104 (51), 94 (12), 93 (23), 91 (100), 81 (23), 80 (11), 79 (45), 78 (11), 77 (52), 67 (32), 65 (18), 55 (28), 53 (12), 51 (16). Anal. Calcd for $C_{22}H_{25}N$: C, 87.08; H, 8.30; N, 4.62. Found: C, 86.88; H, 8.35; N, 4.48.



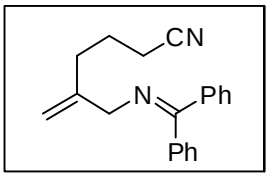
***N*-Diphenylmethylidene-2-methylidene-3-pentyloct-1-ylamine (3c). 3c** was isolated by NH_2 silica-gel column chromatography (R_f = 0.11 in hexane; R_f = 0.29 in hexane/EtOAc = 20/1) in 66% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (150 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.85 (t, *J* = 6.8 Hz, 6H, CH_3), 1.12-1.28 (c, 16H, CH_2), 2.01 (quint, *J* = 6.9 Hz, 1H, =CCH), 3.93 (s, 2H, CH_2N), 4.83 (d, *J* = 1.8 Hz, 1H, =CH), 4.94 (d, *J* = 1.8 Hz, 1H, =CH), 7.16-7.20 (c, 2H, Ph), 7.27-7.44 (c, 6H, Ph), 7.65-7.68 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 14.1 (CH_3), 22.6 (CH_2), 27.1 (CH_2), 32.0 (CH_2), 34.1 (CH_2), 45.5 (=C*C*H), 55.9 (CH_2N), 109.8 (=CH_2), 127.2 (Ph), 127.5 (Ph), 128.0 (Ph), 128.3 (Ph), 128.4 (Ph), 129.9 (Ph), 136.5 (Ph), 139.8 (Ph), 150.6 (=C), 168.7 (N=C). IR (neat): 3082 m (=C-H), 3060 m (=C-H), 3023 m (=C-H), 2953 s (C-H), 2927 s (C-H), 2856 s (C-H), 1624 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 319 (15), 318 (47), 305 (22), 304 (42), 264 (27), 251 (11), 250 (34), 248 (33), 234 (11), 220 (10), 206 (16), 194 (24), 183 (15), 182 (100), 180 (14), 167 (33), 165 (28), 123 (10), 117 (13), 115 (14), 104 (40), 95 (28), 91 (97), 82 (14), 81 (52), 79 (19), 77 (28), 69 (23), 68 (11), 67 (32), 65 (12), 55 (47). Anal. Calcd for $C_{27}H_{37}N$: C, 86.34; H, 9.93; N, 3.73. Found: C, 86.40; H, 10.13; N, 3.68.



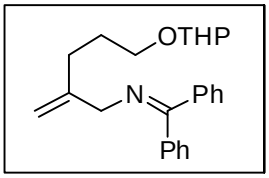
Methyl 5-(*N*-diphenylmethylideneaminomethyl)hex-5-enoate (3d). 3d was isolated by NH_2 silica-gel column chromatography (R_f = 0.26 in hexane/EtOAc = 5/1) in 70% yield as a colorless oil. Analytically pure sample was obtained by bulb-to-bulb distillation (145 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 1.76 (quint, *J* = 7.6 Hz, 2H, =CCH_2CH_2), 2.14 (t, *J* = 7.6 Hz, 2H, =CCH_2), 2.30 (t, *J* = 7.6 Hz, 2H, CH_2C=O), 3.65 (s, 3H, OCH_3), 3.94 (s, 2H, CH_2N), 4.87 (s, 1H, =CH), 4.96 (s, 1H, =CH), 7.16-7.18 (c, 2H, Ph), 7.31-7.47 (c, 6H, Ph), 7.63-7.65 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 27.8 (CH_2), 33.5 (CH_2), 34.2 (CH_2), 51.4 (OCH_3), 58.0



(CH_2N), 110.5 ($=CH_2$), 127.6 (Ph), 128.0 (Ph), 128.4 (Ph x 3), 130.0 (Ph), 136.4 (Ph), 139.7 (Ph), 147.1 (=C), 168.7 (N=C), 174.0 (C=O). IR (neat): 3080 w (=C-H), 3059 w (=C-H), 3023 w (=C-H), 2949 m (C-H), 1736 s (C=O), 1623 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 321 (15, M^+), 320 (13, M^+-1), 266 (22), 262 (14), 249 (12), 248 (73), 235 (18), 234 (45), 220 (13), 206 (15), 194 (13), 193 (11), 167 (11), 166 (10), 165 (36), 158 (12), 131 (10), 129 (12), 117 (11), 116 (11), 115 (16), 108 (15), 104 (31), 91 (100), 81 (21), 79 (16), 77 (36), 67 (15), 65 (13), 55 (14), 53 (11), 51 (13). Anal. Calcd for $C_{21}H_{23}NO_2$: C, 78.47; H, 7.21; N, 4.36. Found: C, 78.29; H, 7.29; N, 4.35.

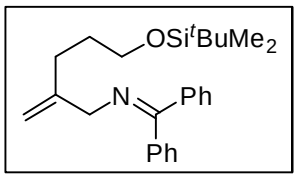


5-(*N*-Diphenylmethylideneaminomethyl)hex-5-enenitrile (3e). 3e was isolated by NH_2 silica-gel column chromatography (R_f = 0.29 in hexane/EtOAc = 5/1) in 70% yield as a colorless oil. Analytically pure sample was obtained by bulb-to-bulb distillation (145 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 1.80 (quint, *J* = 7.3 Hz, 2H, $=CCH_2CH_2$), 2.27 (t, *J* = 7.3 Hz, 2H, CH_2CN), 2.32 (t, *J* = 7.3 Hz, 2H, $=CCH_2$), 3.93 (s, 2H, CH_2N), 4.90 (s, 1H, =CH), 5.01 (s, 1H, =CH), 7.15-7.17 (c, 2H, Ph), 7.32-7.48 (c, 6H, Ph), 7.62-7.64 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 16.4 (CH_2), 23.3 (CH_2), 33.6 (CH_2), 57.9 (CH_2N), 111.7 ($=CH_2$), 119.6 (C≡N), 127.5 (Ph), 128.0 (Ph), 128.4 (Ph), 128.5 (Ph), 128.5 (Ph), 130.1 (Ph), 136.3 (Ph), 139.5 (Ph), 145.7 (=C), 169.0 (N=C). IR (neat): 3080 m (=C-H), 3059 m (=C-H), 3024 m (=C-H), 2935 m (C-H), 2247 m (C≡N), 1623 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 288 (14, M^+), 287 (23), 249 (21), 248 (100), 235 (15), 234 (22), 233 (11), 211 (12), 194 (11), 193 (10), 180 (13), 166 (12), 165 (35), 158 (12), 117 (11), 116 (11), 115 (18), 104 (33), 91 (99), 81 (11), 77 (50), 67 (28), 65 (18), 53 (13), 51 (23). Anal. Calcd for $C_{20}H_{20}N_2$: C, 83.30; H, 6.99; N, 9.71. Found: C, 83.49; H, 6.72; N, 9.53.

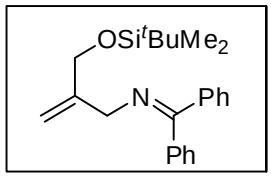


***N*-Diphenylmethylidene-2-methylidene-5-(tetrahydro-2*H*-pyran-2-yloxy)pent-1-ylamine (3f). 3f** was isolated by NH_2 silica-gel column chromatography (R_f = 0.17 in hexane/EtOAc = 20/1) in 76% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane/EtOAc = 20/1 as the eluent, followed by bulb-to-bulb distillation (150 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 1.49-1.60 (c, 4H, CH_2), 1.67-1.85 (c, 4H, CH_2), 2.18 (m, 2H, $=CCH_2$), 3.39 (m, 1H, CH_aH_bO), 3.48 (m, 1H, CH_aH_bO), 3.73 (m, 1H, CH_aH_bO), 3.84 (m, 1H, CH_aH_bO), 3.96 (s, 2H, CH_2N), 4.56 (t, *J* = 3.4 Hz, 1H, OCHO), 4.89 (s, 1H, =CH), 4.96 (d, *J* = 1.2 Hz, 1H, =CH), 7.16-7.19 (c, 2H, Ph), 7.30-7.47 (c, 6H, Ph), 7.64-7.66 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 19.5 (CH_2), 25.4 (CH_2), 27.7 (CH_2), 30.6 (CH_2), 31.4 (CH_2), 58.2 (CH_2N), 62.2 (CH_2O), 67.1 (CH_2O), 98.7 (OCH_2O), 109.8 ($=CH_2$), 127.6 (Ph), 127.9 (Ph), 128.3 (Ph x 3), 129.4 (Ph), 136.4 (Ph), 139.7 (Ph), 147.6 (C), 168.6 (N=C). IR (neat): 3080 m (=C-H), 3056 m (=C-H), 3024 m (=C-H), 2923 s (C-H), 2870 s (C-H), 1623 s (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 363 (2, M^+), 280 (13), 279 (38), 278 (100), 263 (18), 262 (78), 260 (10), 248 (43), 235 (13), 234 (14), 222 (12), 194 (10), 182 (29), 167 (38), 165 (23), 104 (22), 97 (33), 91 (54), 85 (82), 81 (10), 79 (12), 77 (19), 67 (28), 57 (22), 55 (20). HRMS Calcd for $C_{24}H_{29}NO_2$ (M^+): 363.2198. Found: 363.2195.

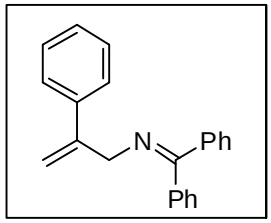
5-*tert*-Butyldimethylsilyloxy-*N*-diphenylmethylidene-2-methylidenepent-1-yl amine (3g). 3g was isolated by NH_2 silica-gel column chromatography (R_f = 0.16 in hexane; R_f = 0.43 in hexane/EtOAc = 20/1) in 73% yield as a colorless oil. Analytically pure sample was obtained by bulb-to-bulb distillation (150 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.03 (s, 6H, $Si(CH_3)_2$), 0.90 (s, 9H, $C(CH_3)_3$), 1.64 (tt, *J* = 6.8, 7.6 Hz, 2H, $=CCH_2CH_2$), 2.14 (t, *J* = 7.6 Hz, 2H, $=CCH_2$), 3.60 (t, *J* = 6.8 Hz, 2H, CH_2O), 3.95 (s, 2H, CH_2N), 4.87 (d, *J* = 1.4 Hz, 1H, =CH), 4.94 (d, *J* = 1.4 Hz, 1H, =CH), 7.16-7.18 (c, 2H, Ph), 7.30-7.46 (c, 6H, Ph), 7.64-7.66 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ -5.3 ($Si(CH_3)_2$), 18.3 (CH_2), 25.9 ($C(CH_3)_3$), 30.8 (CH_2), 31.1 ($C(CH_3)_3$), 58.3 (CH_2N), 62.8 (CH_2O), 109.7 ($=CH_2$), 127.6 (Ph), 128.0 (Ph), 128.2 (Ph), 128.4 (Ph), 128.5 (Ph), 129.9 (Ph), 136.5 (Ph), 139.7 (Ph), 147.9 (=C), 168.6 (N=C). IR (neat): 3080 m (=C-H), 3060 m (=C-H), 3023 w (=C-H), 2952 s (C-H), 2929 s (C-H), 2885 s (C-H), 2856 s (C-H), 1624 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 337 (23), 336 (82, M^+-C_4H_9), 262 (12), 249 (15), 248 (75), 238 (18), 236 (15), 235 (77), 234 (28), 222 (10), 220 (13), 182 (40), 167 (18), 165 (25), 158 (33), 155 (17), 136 (14), 135 (100), 113 (10), 104 (25), 101 (31), 91 (76), 89 (10), 81 (13), 79 (13), 77 (28), 75 (89), 73 (78), 61 (17), 59 (35). Anal. Calcd for $C_{25}H_{35}NOSi$: C, 76.28; H, 8.96; N, 3.56. Found: C, 76.28; H, 8.78; N, 3.45.



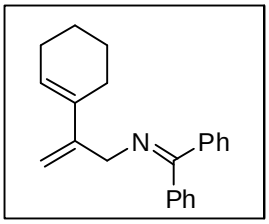
2-*tert*-Butyldimethylsilyloxymethyl-*N*-diphenylmethylideneprop-2-en-1-amine (3h). 3h was isolated by NH_2 silica-gel column chromatography (R_f = 0.19 in hexane; R_f = 0.51 in hexane/EtOAc = 20/1) in 41% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (145 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.04 (s, 6H, $Si(CH_3)_2$), 0.87 (s, 9H, $C(CH_3)_3$), 3.98 (s, 2H, CH_2N), 4.21 (s, 2H, CH_2O), 5.01 (s, 1H, =CH), 5.14 (s, 1H, =CH), 7.15-7.18 (c, 2H, Ph), 7.31-7.47 (c, 6H, Ph), 7.63-7.65 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ -5.4 ($Si(CH_3)_2$), 18.3 (CH_2), 25.9 ($C(CH_3)_3$), 55.6 (CH_2N), 65.1 (CH_2O), 109.4 ($=CH_2$), 127.6 (Ph), 128.0 (Ph), 128.4 (Ph x 3), 129.9 (Ph), 136.4 (Ph), 139.7 (Ph), 147.3 (=C), 168.8 (N=C). IR (neat): 3081 m (=C-H), 3060 m (=C-H), 3024 m (=C-H), 2954 s (C-H), 2927 s (C-H), 2884 s (C-H), 2856 s (C-H), 1623 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 309 (22), 308 (80, M^+-C_4H_9), 185 (10), 184 (18), 165 (13), 135 (26), 129 (129), 128 (25), 127 (42), 115 (20), 113 (22), 91 (99), 77 (11), 73 (100), 59 (15). Anal. Calcd for $C_{23}H_{31}NOSi$: C, 75.56; H, 8.55; N, 3.83. Found: C, 75.69; H, 8.64; N, 3.85.



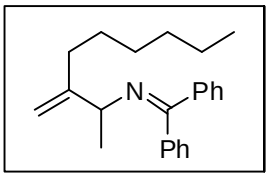
***N*-Diphenylmethylidene-2-phenylprop-2-en-1-amine (3i). 3i** was isolated by NH_2 silica-gel column chromatography (R_f = 0.17 in hexane; R_f = 0.31 in hexane/EtOAc = 20/1) in 67% yield as a pale yellow oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (150 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 4.41 (s, 2H, CH_2N), 5.32 (d, *J* = 1.2 Hz, 1H, =CH), 5.47 (d, *J* = 1.2 Hz, 1H, =CH), 7.20-7.46 (c, 13H, Ph), 7.65-7.67 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 56.9 (CH_2N), 112.8 ($=CH_2$), 126.0 (Ph), 127.4 (Ph), 127.6 (Ph), 128.0 (Ph), 128.2 (Ph), 128.5 (Ph), 128.5 (Ph x 2), 130.0 (Ph), 136.4 (Ph), 139.8 (Ph), 140.4 (Ph), 146.5 (=C), 169.2 (N=C). IR (neat): 3080 s (=C-H), 3057 s (=C-H), 3023 s (=C-H), 2885 m (C-H), 2839 m (C-H), 1621 s (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 297 (15, M^+), 296



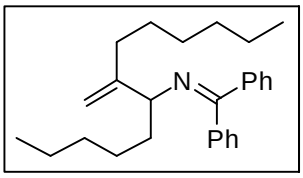
(26, M^+-1), 220 (55), 165 (17), 117 (13), 115 (31), 91 (100), 77 (16). Anal. Calcd for $C_{22}H_{19}N$: C, 88.85; H, 6.44; N, 4.71. Found: C, 88.57; H, 6.44; N, 4.61.



2-Cyclohex-1-en-1-yl-*N*-diphenylmethylideneprop-2-en-1-amine (3j). 3j was isolated by NH_2 silica-gel column chromatography (R_f = 0.14 in hexane; R_f = 0.34 in hexane/EtOAc = 20/1) in 54% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (150 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 1.52-1.58 (m, 2H, CH_2), 1.64-1.70 (m, 2H, CH_2), 2.08-2.09 (m, 2H, CH_2), 2.20-2.23 (m, 2H, CH_2), 4.18 (s, 2H, CH_2N), 5.08 (s, 1H, =CH), 5.13 (s, 1H, =CH), 5.76 (t, *J* = 4.0 Hz, 1H, C=C*H*CH_2), 7.17-7.20 (c, 2H, Ph), 7.30-7.47 (c, 6H, Ph), 7.64-7.66 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 22.1 (CH_2), 22.8 (CH_2), 25.7 (CH_2), 25.9 (CH_2), 55.7 (CH_2N), 109.4 (=CH_2), 124.2 (C=*C*HCH_2), 127.6 (Ph), 128.0 (Ph), 128.4 (Ph), 128.4 (Ph), 128.5 (Ph), 129.8 (Ph), 135.3 (*C*=CHCH_2), 136.6 (Ph), 139.9 (Ph), 146.4 (=C), 168.4 (N=C). IR (neat): 3080 w (=C-H), 3056 w (=C-H), 3023 w (=C-H), 2927 s (C-H), 2857 m (C-H), 2833 m (C-H), 1625 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 301 (47, M^+), 300 (20), 272 (11), 224 (11), 182 (29), 167 (11), 165 (21), 105 (18), 104 (16), 91 (100), 79 (19), 77 (30), 65 (13). Anal. Calcd for $C_{22}H_{23}N$: C, 87.66; H, 8.69; N, 4.65. Found: C, 87.41; H, 7.49; N, 4.43.

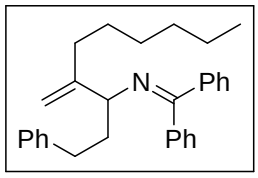


***N*-Diphenylmethylidene-3-methylidenenonan-2-ylamine (4b). 4b** was isolated by NH_2 silica-gel column chromatography (R_f = 0.24 in hexane; R_f = 0.51 in hexane/EtOAc = 20/1) in 73% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (145 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.85-0.89 (m, 3H, CH_3), 1.24-1.33 (c, 11H, CH_2 and CH_3), 2.06 (m, 2H, =CCH_2), 3.92 (m, 1H, CHN), 4.76 (s, 1H, =CH), 4.92 (s, 1H, =CH), 7.14-7.17 (c, 2H, Ph), 7.29-7.46 (c, 6H, Ph), 7.62-7.66 (c, 2H, Ph). ^{13}C NMR ($CDCl_3$): δ 14.1 (CH_3), 21.5 (CH_3), 22.6 (CH_2), 28.0 (CH_2), 29.2 (CH_2), 31.8 (CH_2), 33.1 (CH_2), 61.7 (CH_2N), 108.5 (=CH_2), 127.6 (Ph), 128.0 (Ph), 128.1 (Ph), 128.3 (Ph), 128.4 (Ph), 129.7 (Ph), 137.0 (Ph), 140.1 (Ph), 153.0 (=C), 165.8 (N=C). IR (neat): 3082 m (=C-H), 3060 m (=C-H), 3024 m (=C-H), 2958 s (C-H), 12929 s (C-H), 2856 s (C-H), 1623 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 319 (17, M^+), 318 (21, M^+-1), 304 (15), 263 (18), 262 (32), 251 (16), 250 (75), 249 (13), 248 (39), 242 (15), 234 (16), 208 (21), 207 (11), 206 (25), 194 (11), 193 (10), 183 (14), 182 (100), 167 (19), 166 (22), 165 (43), 115 (10), 109 (15), 105 (66), 104 (54), 103 (11), 95 (13), 91 (51), 82 (12), 81 (11), 79 (17), 77 (42), 69 (25), 68 (11), 67 (25), 55 (36), 53 (11), 51 (11). Anal. Calcd for $C_{23}H_{29}N$: C, 86.47; H, 9.15; N, 4.38. Found: C, 86.42; H, 9.16; N, 4.44.

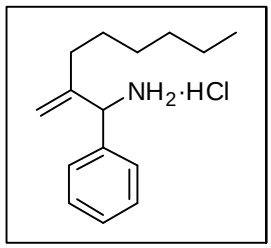


***N*-Diphenylmethylidene-7-methylidenetridec-6-ylamine (4c). 4c** was isolated by NH_2 silica-gel column chromatography (R_f = 0.25 in hexane; R_f = 0.63 in hexane/EtOAc = 20/1) in 75% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH_2 silica-gel column chromatography using hexane as the eluent, followed by bulb-to-bulb distillation (150 °C/1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.83 (t, *J* = 7.1 Hz, 3H, CH_3), 0.86 (t, *J* = 7.3 Hz, 3H, CH_3), 1.07-1.37 (c, 14H, CH_2), 1.67

(quint, J = 7.6 Hz, 2H, =CCH$_2$CH_2), 2.03 (quint, J = 7.6 Hz, 1H, =CCH_aH$_b$), 2.13 (quint, J = 7.6 Hz, 1H, =CCH$_a$$H_b$), 3.75 (t, J = 6.2 Hz, 1H, CHN), 4.74 (s, 1H, =CH), 4.79 (s, 1H, =CH), 7.12-7.14 (c, 2H, Ph), 7.30-7.44 (c, 6H, Ph), 7.63-7.65 (c, 2H, Ph). ^{13}C NMR (CDCl$_3$): δ 14.1 (CH$_3$), 14.1 (CH$_3$), 22.6 (CH$_2$ x 2), 26.3 (CH$_2$), 28.0 (CH$_2$), 29.2 (CH$_2$), 31.8 (CH$_2$ x 2), 32.6 (CH*C*H$_2$), 35.6 (=C*C*H$_2$), 67.4 (*C*H$_2$N), 109.2 (=CH$_2$), 127.8 (Ph), 128.0 (Ph), 128.0 (Ph), 128.1 (Ph), 128.4 (Ph), 129.6 (Ph), 137.2 (Ph), 140.2 (Ph), 152.3 (=C), 165.9 (N=C). IR (neat): 3080 m (=C-H), 3060 m (=C-H), 3029 m (=C-H), 2952 s (C-H), 2929 s (C-H), 2870 s (C-H), 2856 s (C-H), 1623 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 318 (12 M$^+$-C$_4$H$_9$), 304 (78), 250 (41), 234 (28), 206 (12), 182 (100), 165 (30), 117 (12), 104 (30), 91 (62), 77 (20). Anal. Calcd for C$_{27}$H$_{37}$N: C, 86.34; H, 9.93; N, 3.73. Found: C, 86.52; H, 10.21; N, 3.82.

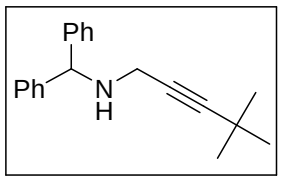


***N*-Diphenylmethylidene-4-methylidene-1-phenyl-dec-3-ylamine (4d). 4d** was isolated by NH$_2$ silica-gel column chromatography (R_f = 0.30 in hexane) in 61% yield as a colorless oil. Analytically pure sample was obtained by medium-pressure NH$_2$ silica-gel column chromatography using hexane as the eluent. ^{1}H NMR (CDCl$_3$): δ 0.86 (t, J = 6.0 Hz, 3H, CH$_3$), 1.24-1.35 (c, 8H, CH$_2$), 1.97-2.04 (c, 3H, c, 1H, CHCH_2 and =CCH_aH$_b$), 2.15 (quint, J = 7.2 Hz, 1H, =CCH$_a$$H_b$), 2.2.45 (quint, J = 7.2 Hz, 1H, PhCH$_a$$H_b$), 2.58 (quint, J = 7.2 Hz, 1H, PhCH_aH$_b$), 3.83 (t, J = 6.4 Hz, 1H, CHN), 4.78 (s, 1H, CH), 4.83 (s, 1H, CH), 7.09-7.15 (c, 5H, Ph), 7.21-7.26 (c, 5H, Ph), 7.31-7.40 (c, 3H, Ph), 7.65-7.68 (c, 2H, Ph). ^{13}C NMR (CDCl$_3$): δ 14.1 (CH$_3$), 22.6 (CH$_2$), 28.0 (CH$_2$), 29.2 (CH$_2$), 31.8 (CH$_2$), 32.7 (CH$_2$), 32.9 (CH$_2$), 37.3 (CH*C*H$_2$), 67.1 (*C*H$_2$N), 109.8 (=CH$_2$), 125.6 (Ph), 127.7 (Ph), 128.0 (Ph), 128.1 (Ph), 128.2 (Ph), 128.2 (Ph), 128.3 (Ph), 128.5 (Ph), 129.7 (Ph), 137.0 (Ph), 140.1 (Ph), 142.5 (Ph), 151.8 (C), 166.5 (N=C). IR (neat): 3080 w (=C-H), 3060 w (=C-H), 3029 w (=C-H), 2952 w (C-H), 2927 m (C-H), 2863 w (C-H), 1641 m (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 296 (42), 277 (15), 227 (13), 217 (21), 234 (28), 198 (10), 129 (44), 117 (10), 110 (13), 69 (100). HRMS Calcd for C$_{30}$H$_{35}$N (M$^+$): 409.2769. Found: 409.2772.

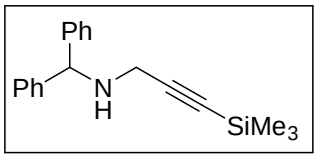


2-Methylidene-1-phenyloct-1-ylamine hydrochloride (4f$_{NH2}$). After the reaction of 1-octyne (**1a**) with imine **2f**, the volatiles were removed in *vacuo*. The residue was passed through NH$_2$ silica-gel column chromatography using hexane as the eluent (R_f = 0.26 in hexane; R_f = 0.66 in hexane/EtOAc = 20/1) to give **4f** along with some unidentified products as an yellow oil. Hydrolysis of **4f** was carried out according to the literature procedure.[13] To the resulting yellow oil was added MeOH (5 mL) and 1M HCl *aq* (5 mL). The reaction mixture was stirred for 12 h at room temperature. The volatiles were removed *in vacuo*. The residue was washed with ether, and filtered through a filter paper. **4f$_{NH2}$** was obtained as white solids in 36% yield. Mp = 182 ºC. ^{1}H NMR (CDCl$_3$): δ 0.84 (t, J = 7.0 Hz, 3H, CH$_3$), 1.16-1.27 (c, 6H, CH$_2$), 1.32 (quint, J = 7.4 Hz, 2H, =CCH$_2$CH_2), 1.81 (m, 2H, CCH$_2$), 4.61 (m, 1H, CHN), 5.02 (s, 1H, =CH), 5.18 (s, 1H, =CH), 7.29-7.33 (c, 3H, Ph), 7.35-7.40 (c, 2H, Ph), 9.00 (br, 2H, NH$_2$). ^{13}C NMR (CDCl$_3$): δ 14.0 (CH$_3$), 22.4 (CH$_2$), 27.1 (CH$_2$), 28.7 (CH$_2$), 31.5 (CH$_2$), 33.3 (=C*C*H$_2$), 59.0 (CHN), 112.1 (=CH$_2$), 128.1 (Ph), 128.8 (Ph), 128.9 (Ph), 134.6 (Ph), 143.9 (=C). IR (KBr): 3043 m (=C-H), 2956 s (C-H), 2929 s (C-H), 1654 w (C=C) cm^{-1}. MS: *m*/*z* (relative intensity): 217 (7), 132 (13), 106 (100). HRMS Calcd for C$_{15}$H$_{23}$N (M$^+$-HCl): 217.1830. Found: 217.1830.

***N*-Diphenylmethyl-4,4-dimethylpent-2-yn-1-amine.** The product was isolated by NH_2 silica-gel column chromatography (R_f = 0.09 in hexane; R_f = 0.33 in hexane/EtOAc = 20/1) in 70% yield as white solids. Analytically pure sample was obtained by bulb-to-bulb distillation (135 °C/1.5 mmHg). Mp = 30 ºC. ^{1}H NMR ($CDCl_3$): δ 1.24 (s, 9H, $(CH_3)_3$), 1.72 (br, 1H, NH), 3.32 (s, 2H, CH_2), 5.09 (s, 1H, CH), 7.18-7.22 (c, 2H, Ph), 7.27-7.31 (c, 4H, Ph), 7.40-7.42 (c, 4H, Ph). ^{13}C NMR ($CDCl_3$): δ 27.4 ($C(CH_3)_3$), 31.2 (CH_3), 36.6 (CH_2), 65.3 (CH), 76.2 (≡C), 92.6 (≡C), 127.0 (Ph), 127.4 (Ph), 128.4 (Ph), 143.4 (Ph). IR (KBr): 3315 m (NH), 3083 w (=C-H), 3062 w (=C-H), 3027 m (=C-H), 2968 m (C-H), 2929 m (C-H), 2906 m (C-H), 2863 m (C-H), 28383 m (C-H), 2227 w (C≡C) cm^{-1}. MS: *m*/*z* (relative intensity): 277 (16, M^+), 276 (14, M^+-1), 262 (17), 235 (28), 234 (76), 220 (27), 217 (15), 200 (12), 194 (11), 182 (22), 167 (19), 165 (27), 158 (14), 117 (16), 115 (22), 104 (36), 91 (100), 81 (11), 77 (39), 67 (11), 65 (12), 57 (10), 55 (30), 51 (15). Anal. Calcd for $C_{20}H_{23}N$: C, 86.59; H, 8.36; N, 5.05. Found: C, 86.59; H, 8.58; N, 4.85.



***N*-Diphenylmethyl-3-trimethylsilylprop-2-yn-1-amine.** The product was isolated by NH_2 silica-gel column chromatography (R_f = 0.12 in hexane; R_f = 0.34 in hexane/EtOAc = 20/1) in 87% yield as a olorless oil. Analytically pure sample was obtained by bulb-to-bulb distillation (135 °C / 1.5 mmHg). ^{1}H NMR ($CDCl_3$): δ 0.20 (d, *J* = 1.6 Hz, 9H, $Si(CH_3)_3$), 1.76 (br, 1H, NH), 3.37 (d, *J* = 1.6 Hz, 2H, CH_2), 5.09 (s, 1H, CH), 7.19-7.23 (c, 2H, Ph), 7.28-7.31 (c, 4H, Ph), 7.40-7.42 (c, 4H, Ph). ^{13}C NMR ($CDCl_3$): δ 0.1 ($Si(CH_3)_3$), 37.3 (CH_2), 65.3 (CH), 88.3 (≡CSi), 104.4 (≡C), 127.1 (Ph), 127.4 (Ph), 128.5 (Ph), 143.2 (Ph). IR (neat): 3322 w (NH), 3086 s (=C-H), 3062 s (=C-H), 3025 s (=C-H), 2958 s (C-H), 2898 m (C-H), 2166 s (C≡C) cm^{-1}. MS: *m*/*z* (relative intensity): 293 (10, M^+), 292 (29, M^+-H), 278 (10), 217 (19), 216 (100), 194 (17), 182 (10), 168 (11), 167 (33), 165 (22), 152 (13), 126 (13), 104 (27), 83 (47), 77 (13), 73 (55). Anal. Calcd for $C_{19}H_{23}NSi$: C, 77.76; H, 7.90; N, 4.77. Found: C, 77.48; H, 7.83; N, 4.63.



References

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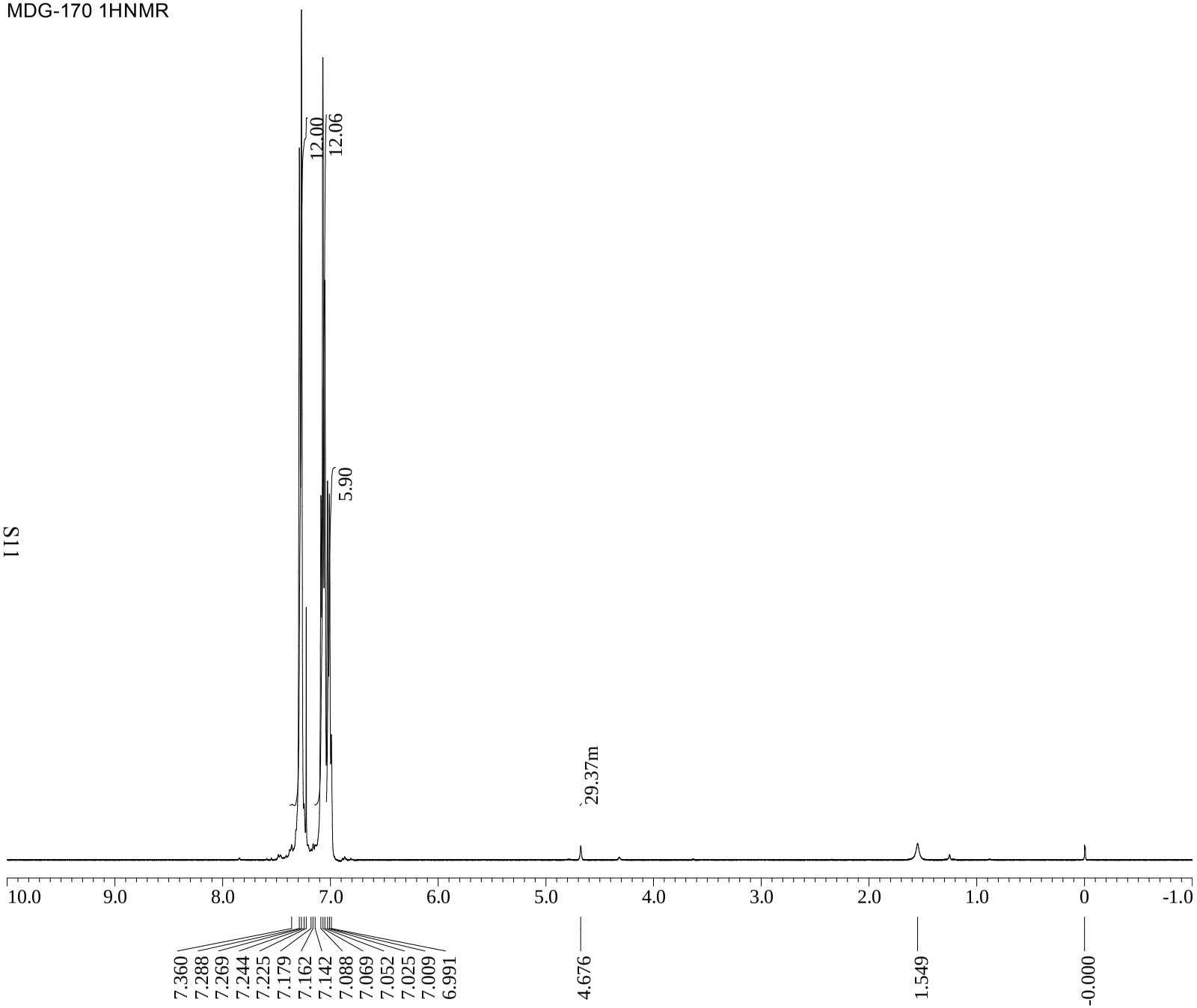
MDG-170 1HNMR



X : parts per Million : Proton

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Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

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X_Freq            = 399.78219838[MHz]
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X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
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Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 17.6[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
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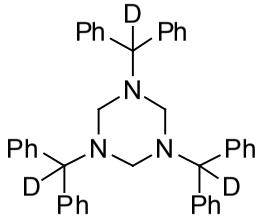
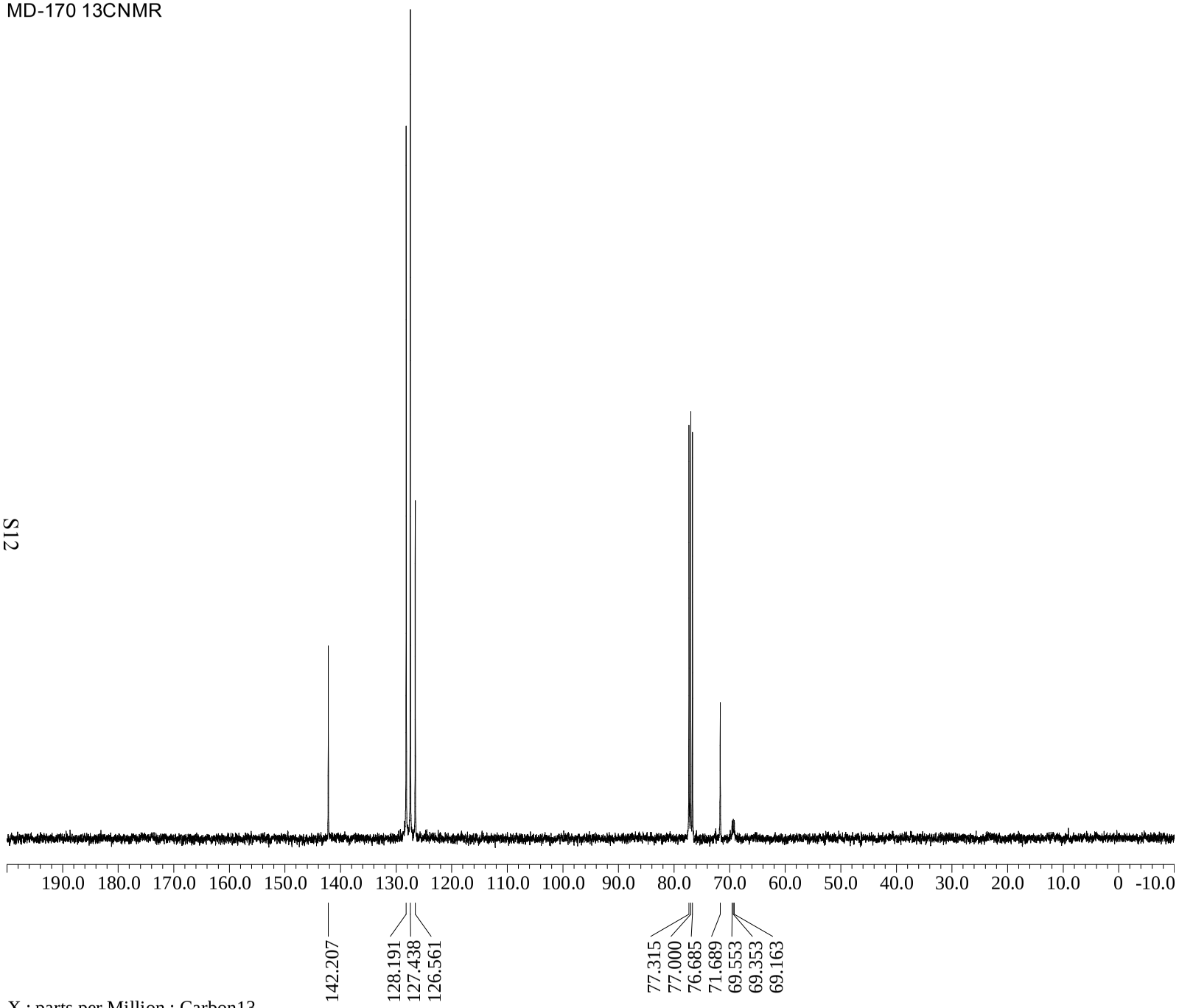


2a-*d*

MD-170 13CNMR



X : parts per Million : Carbon13

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Revision_Time     =  5-MAR-2012 20:08:22
Current_Time      =  5-MAR-2012 20:35:42

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Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 17.8[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
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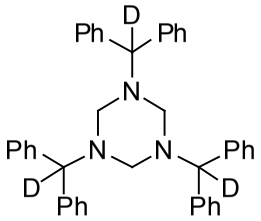
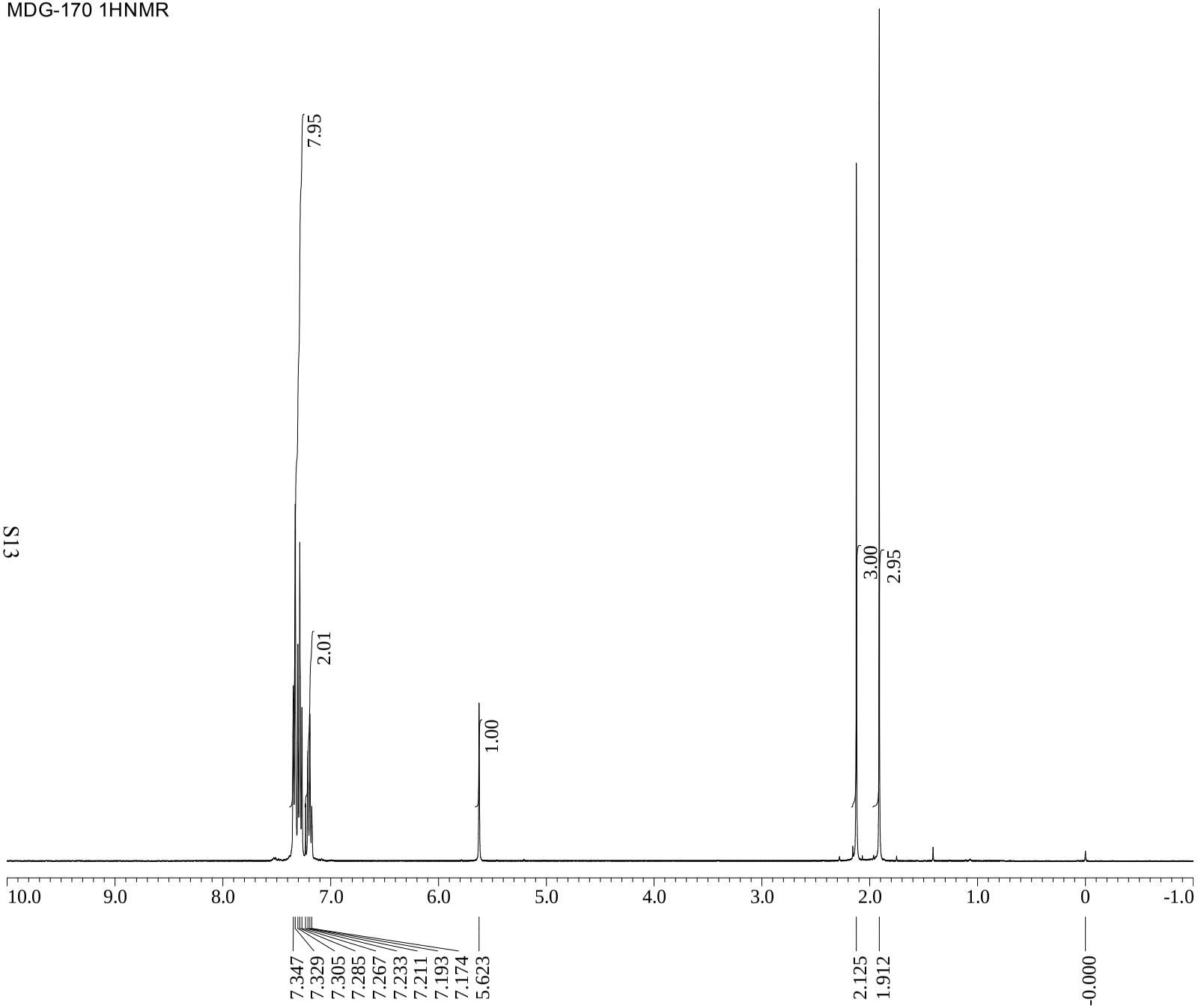


2a-*d*

MDG-170 1HNMR



X : parts per Million : Proton

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Revision_Time     =  5-MAR-2012 12:05:50
Current_Time      =  5-MAR-2012 12:06:24

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Data_Format       = 1D COMPLEX
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Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 30
Temp_Get          = 15.8[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
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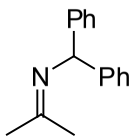
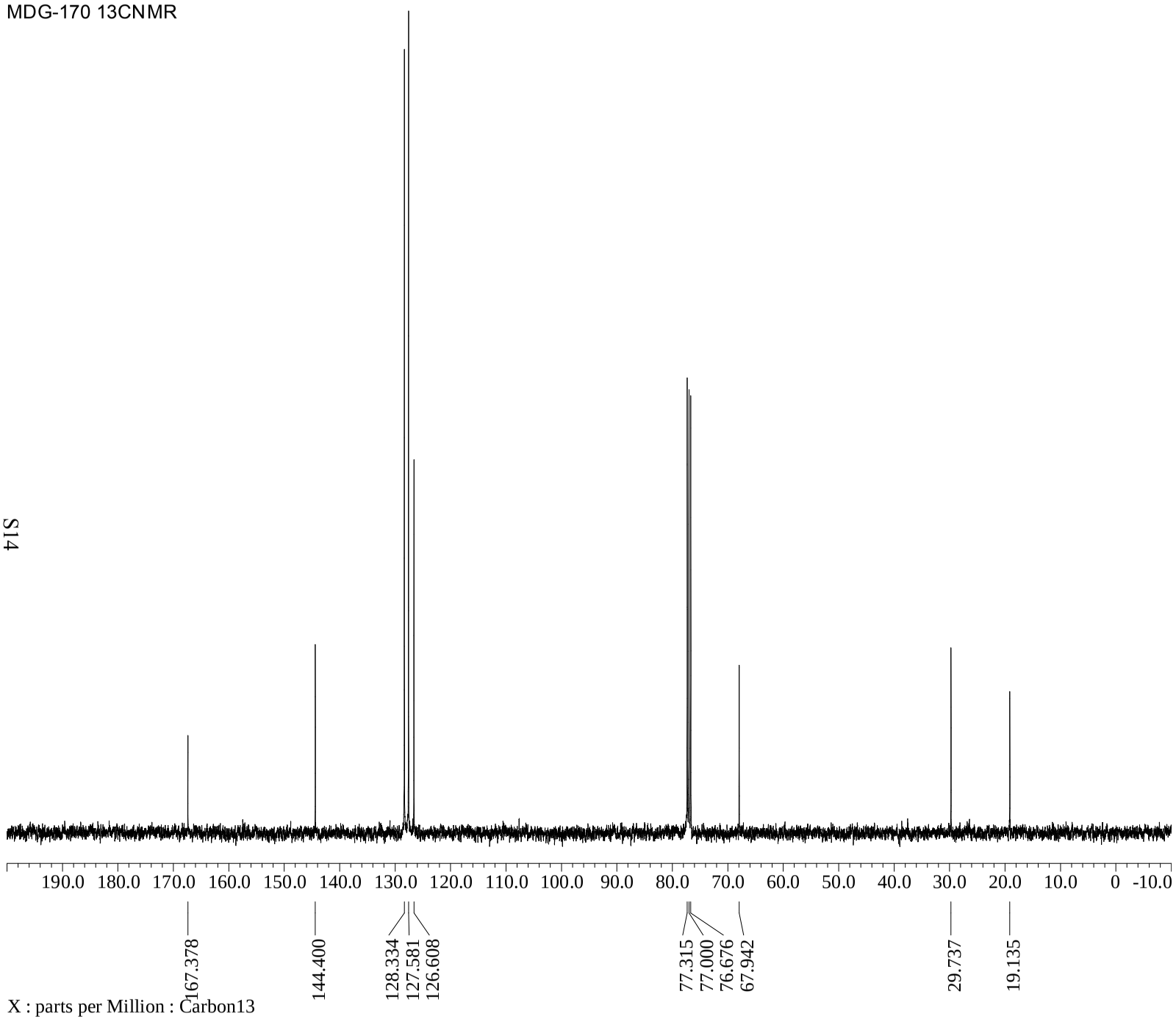


2e

MDG-170 13CNMR



X : parts per Million : Carbon13

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Revision_Time     = 28-FEB-2012 10:54:20
Current_Time      =  5-MAR-2012 12:48:09

Author            = delta
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Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
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X_Sweep_Clipped   = 25.12562814[kHz]
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Temp_Get          = 15.6[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
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X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
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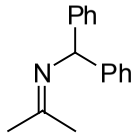
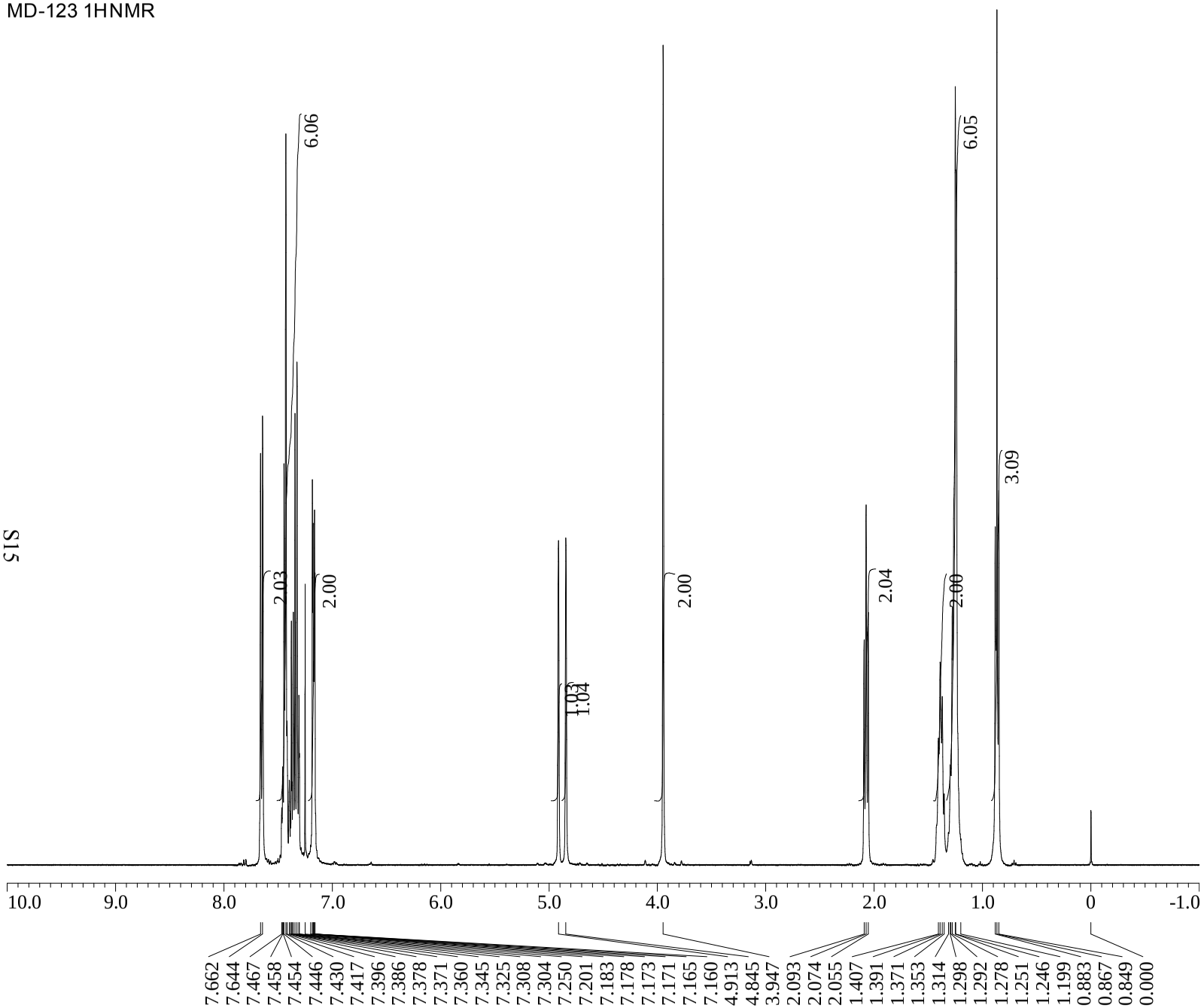


2e

MD-123 1HNMR



X : parts per Million : Proton

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Dim_Units         = [ppm]
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Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

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X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 22.6[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
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X_Atn             = 1[dB]
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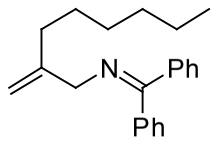
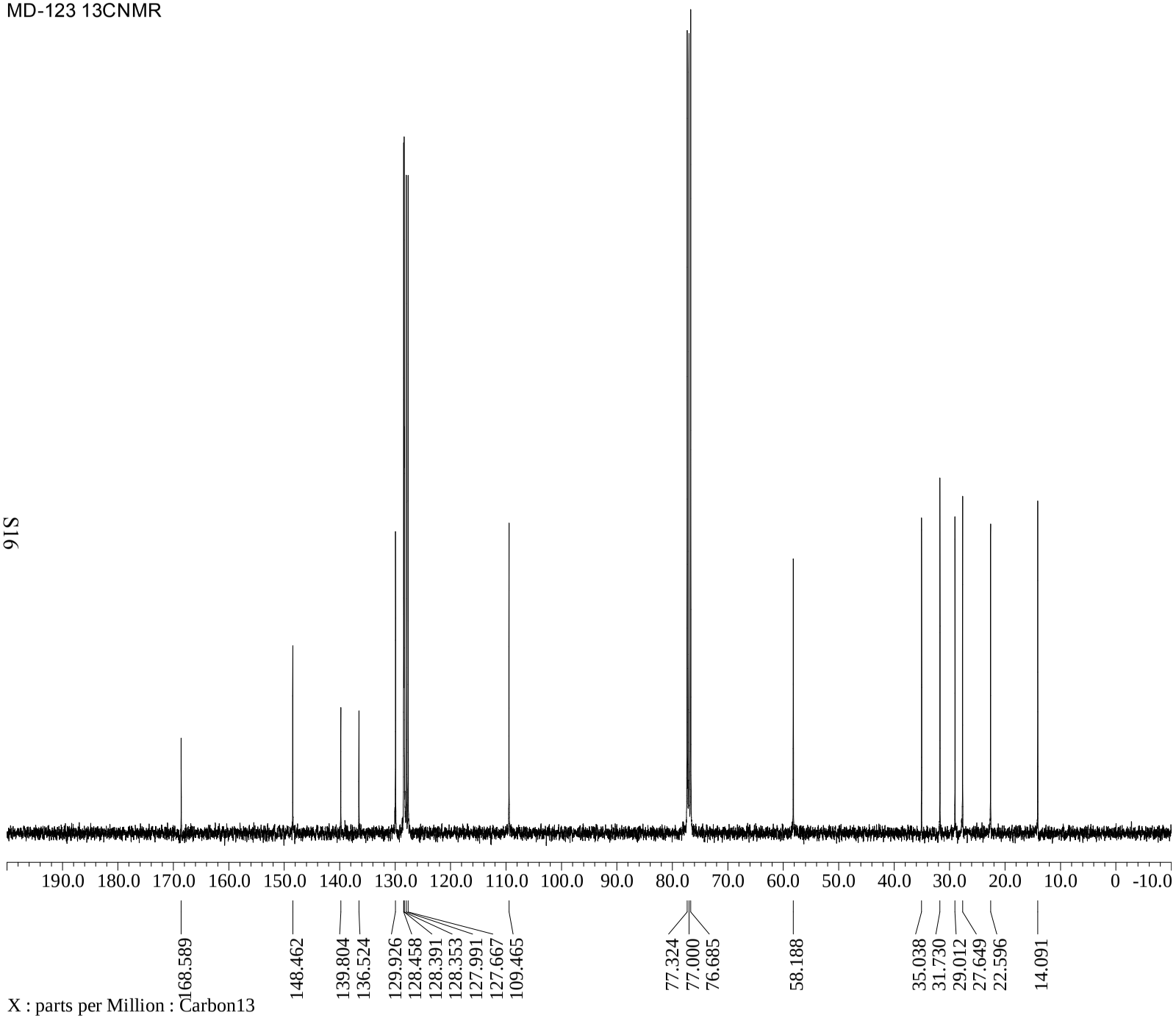


3a

MD-123 13CNMR

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X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

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Temp_Get          = 22.4[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
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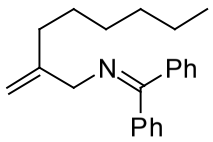
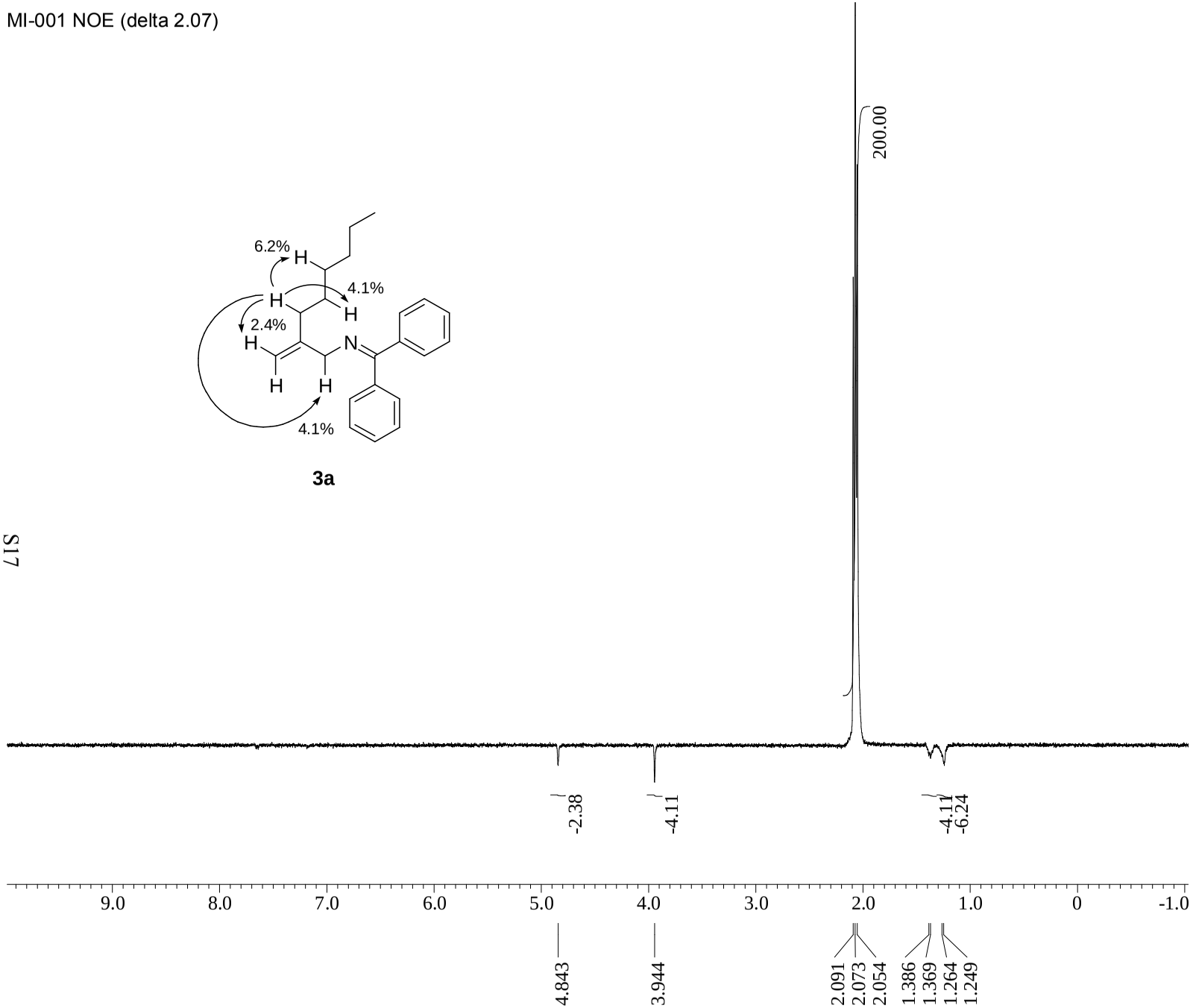


3a

MI-001 NOE (delta 2.07)

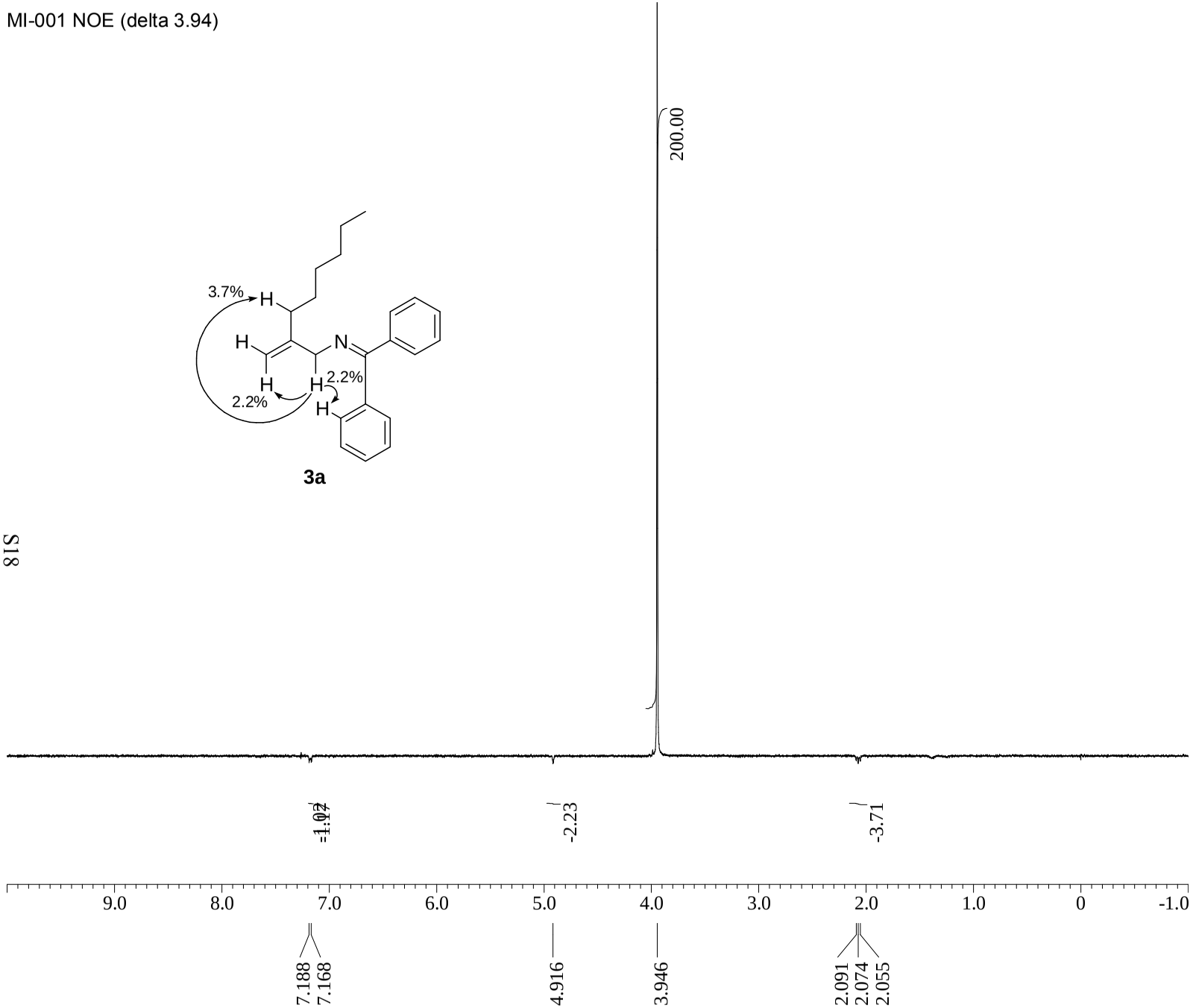
3a




```
Filename          = C:\Documents and Settings\
Experiment        = noe_1d_dpfgse.jxp
Sample_Id         = MI-001-F3-7 NOE
Solvent           = CHLOROFORM-D
Creation_Time     = 18-APR-2012 21:42:32
Revision_Time     = 27-APR-2012 20:57:43
Current_Time      = 27-APR-2012 21:49:42

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.9267584[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 2.062[ppm]
X_Points          = 16384
X_Prescans        = 2
X_Resolution      = 0.51900643[Hz]
X_Sweep           = 8.50340136[kHz]
X_Sweep_Clipped   = 6.80272109[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay  = 7[s]
Recvr_Gain        = 42
Temp_Get          = 19.2[dC]
Mix_Time          = 1.03889[s]
X_Acq_Time        = 1.9267584[s]
X_Atn             = 1[dB]
X_Pulse           = 10[us]
Irr_Mode          = Off
Obs_Sel_180       = 40[ms]
Obs_Sel_Atn       = 59.4[dB]
Obs_Sel_Offset    = 2.062[ppm]
Obs_Sel_Shape     = GAUSS
Obs_Sel_Slp       = 2.062[ppm]
Tri_Mode          = Off
Dante_Presat      = FALSE
Grad_1            = 1[ms]
Grad_1_Amp        = 60[mT/m]
Grad_2            = 1[ms]
Grad_2_Amp        = 90[mT/m]
Grad_3            = 1[ms]
Grad_3_Amp        = 30[mT/m]
Grad_Recover      = 0.1[ms]
Grad_Shape        = SINE
Initial_Wait      = 1[s]
Repetition_Time   = 8.9267584[s]
```

X : parts per Million : Proton

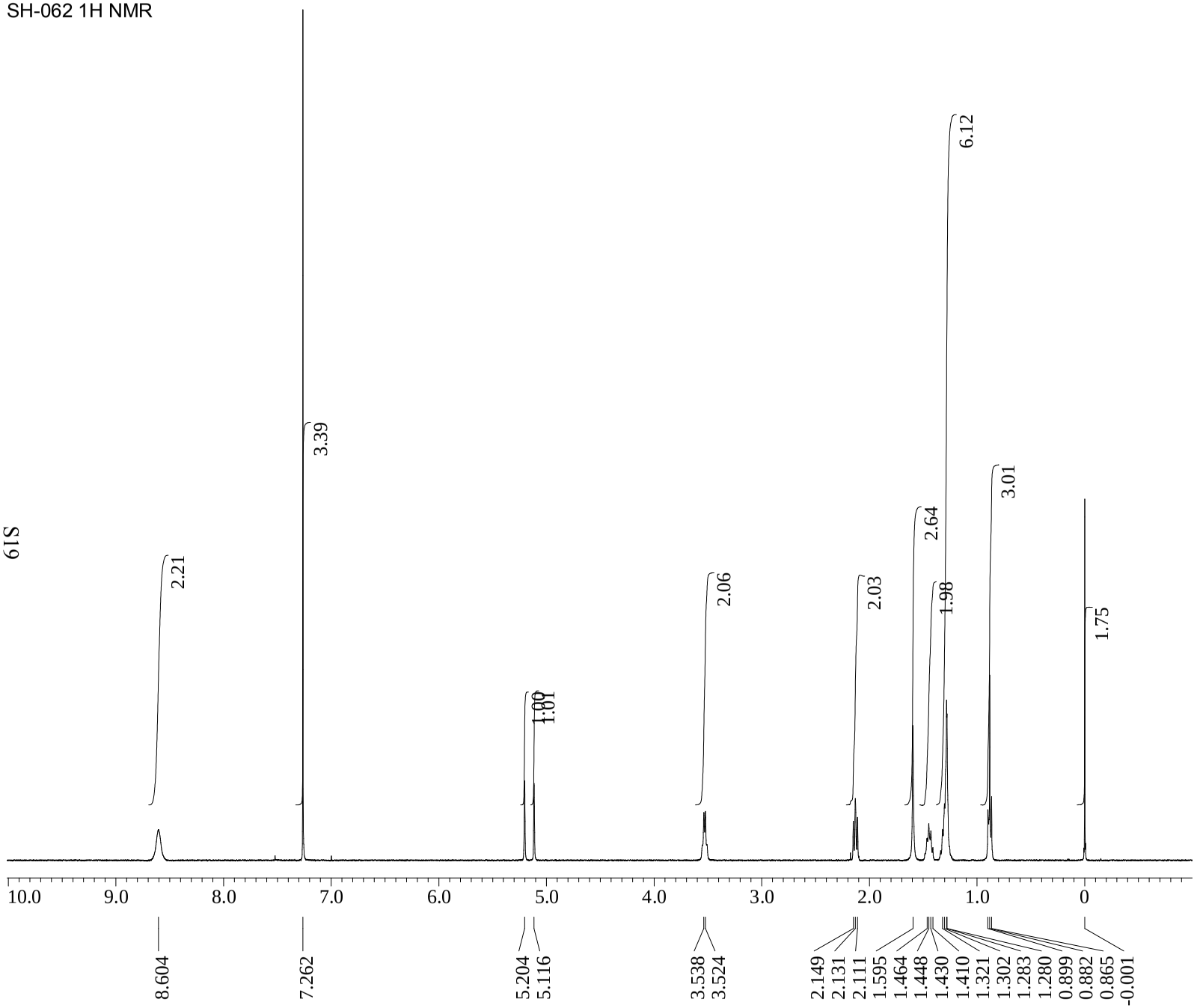
MI-001 NOE (delta 3.94)




```
Filename          = C:\Documents and Settings\
Experiment        = noe_1d_dpfgse.jxp
Sample_Id         = MI-001-F1
Solvent           = CHLOROFORM-D
Creation_Time     = 13-APR-2012 21:28:55
Revision_Time     = 27-APR-2012 21:44:04
Current_Time      = 27-APR-2012 21:44:24

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 3.946[ppm]
X_Points          = 16384
X_Prescans        = 2
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay  = 7[s]
Recvr_Gain        = 50
Temp_Get          = 18.4[dC]
Mix_Time          = 1.00582[s]
X_Acq_Time        = 2.18365952[s]
X_Atn             = 1[dB]
X_Pulse           = 10[us]
Irr_Mode          = Off
Obs_Sel_180       = 40[ms]
Obs_Sel_Atn       = 59.4[dB]
Obs_Sel_Offset    = 3.946[ppm]
Obs_Sel_Shape     = GAUSS
Obs_Sel_Slp       = 3.946[ppm]
Tri_Mode          = Off
Dante_Presat      = FALSE
Grad_1            = 1[ms]
Grad_1_Amp        = 60[mT/m]
Grad_2            = 1[ms]
Grad_2_Amp        = 90[mT/m]
Grad_3            = 1[ms]
Grad_3_Amp        = 30[mT/m]
Grad_Recover      = 0.1[ms]
Grad_Shape        = SINE
Initial_Wait      = 1[s]
Repetition_Time   = 9.18365952[s]
```

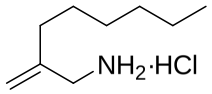
X : parts per Million : Proton

SH-062 1H NMR



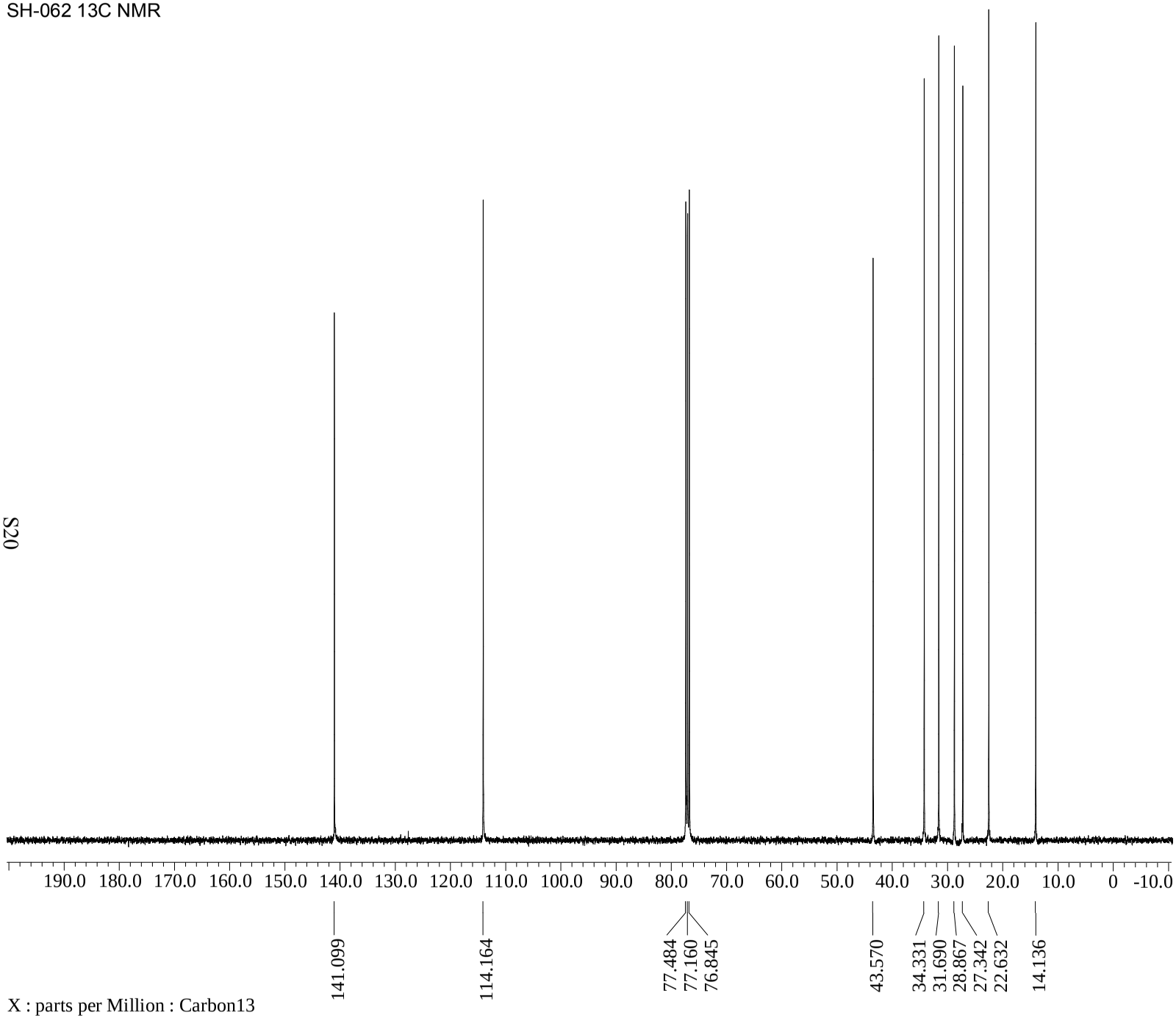
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = SH-063 ether-wash
Solvent           = CHLOROFORM-D
Creation_Time     = 27-APR-2012 17:06:33
Revision_Time     = 27-APR-2012 21:48:29
Current_Time      = 27-APR-2012 21:48:35

Author            = delta
Comment           = SH-063 ether-wash
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 19.5[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



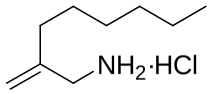
3a$_{NH2}$

SH-062 13C NMR



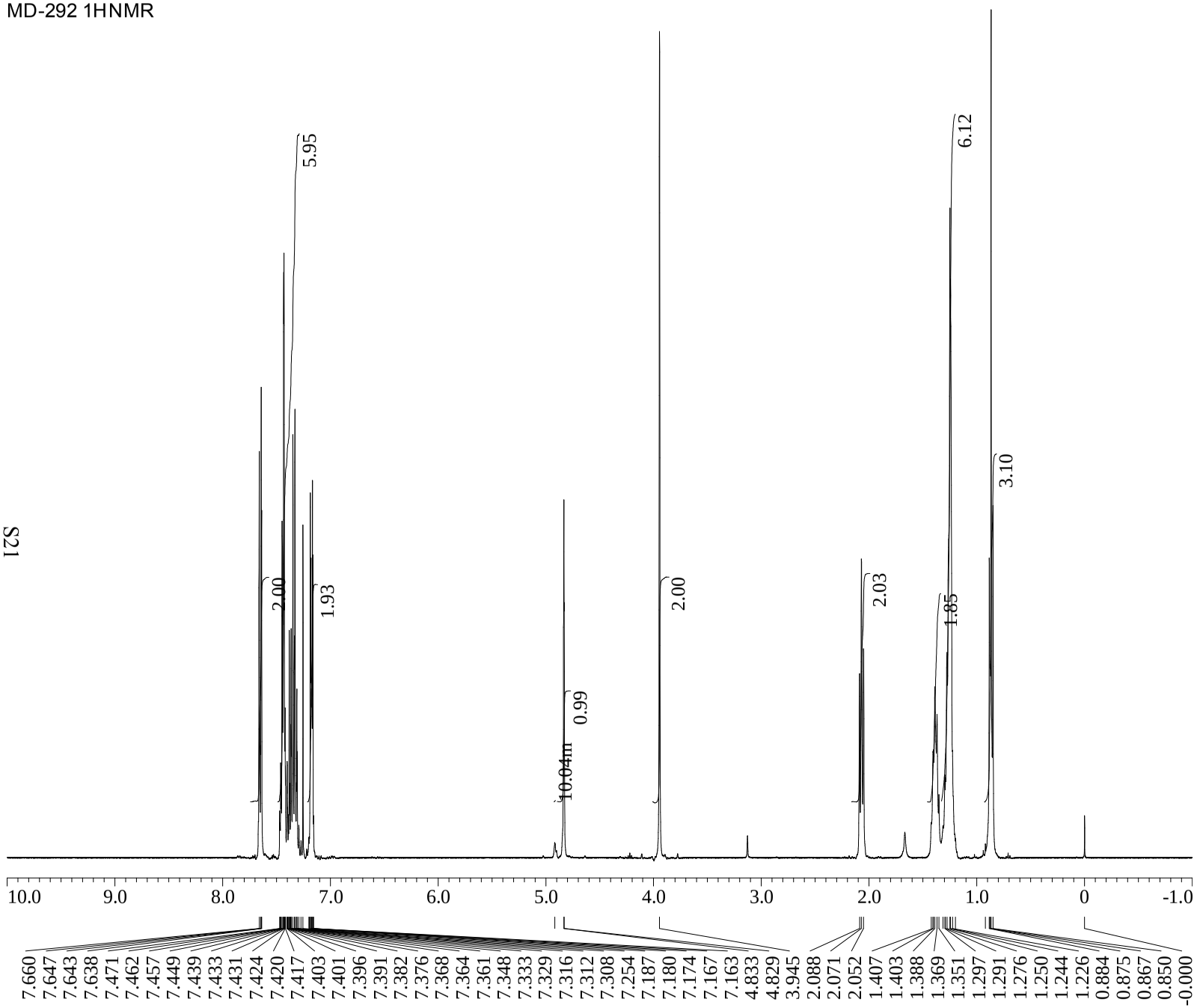
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = SH-063 13C NMR
Solvent           = CHLOROFORM-D
Creation_Time     = 27-APR-2012 17:30:05
Revision_Time     = 27-APR-2012 21:02:59
Current_Time      = 27-APR-2012 21:38:49

Author            = delta
Comment           = SH-063 13C NMR
Data_Format       = 1D REAL
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.4[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



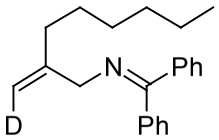
3a$_{NH2}$

MD-292 1HNMR



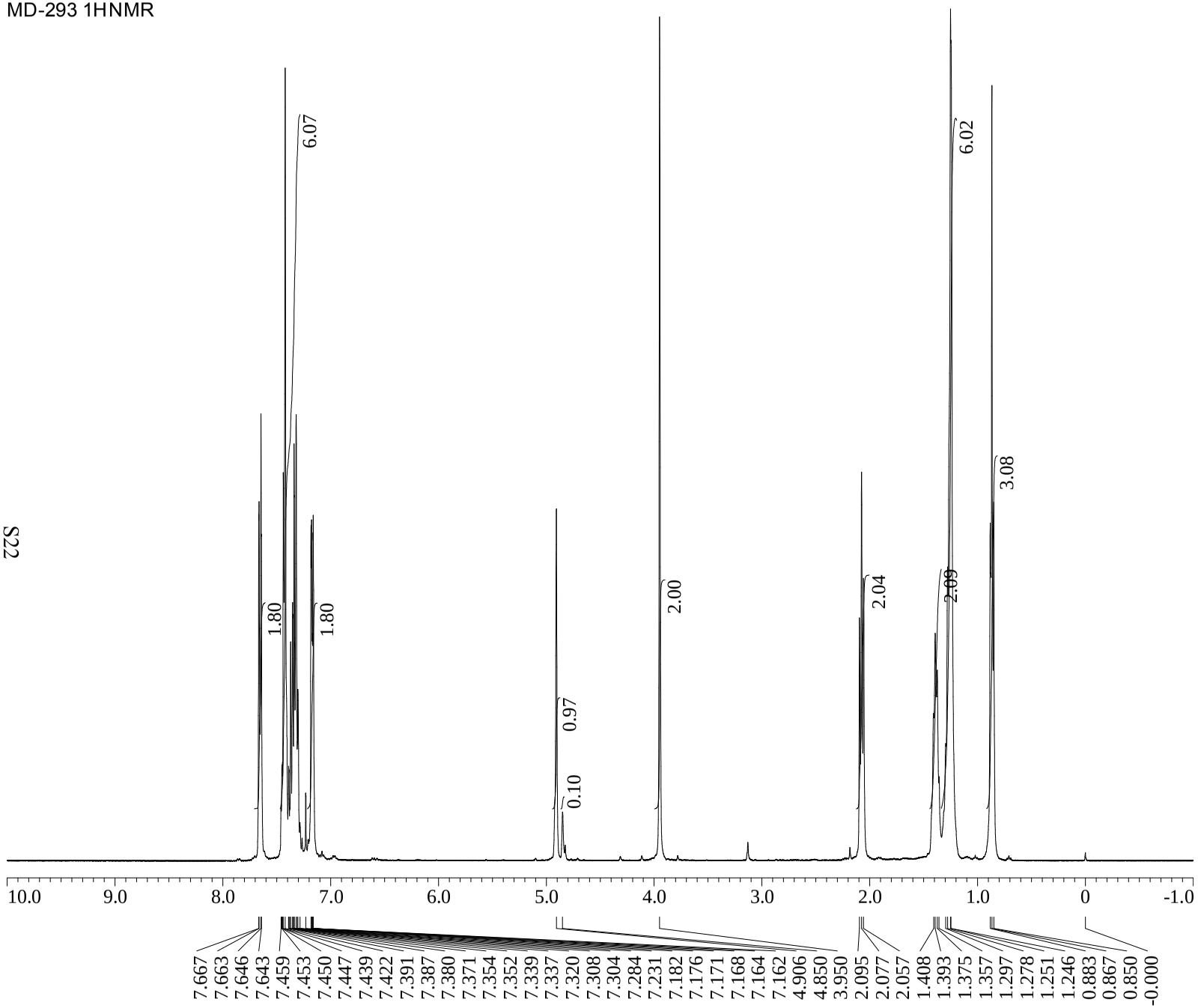
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-292
Solvent           = CHLOROFORM-D
Creation_Time     = 12-FEB-2012 16:03:23
Revision_Time     =  5-MAR-2012 11:00:32
Current_Time      =  5-MAR-2012 11:01:11

Author            = delta
Comment           = MD-292
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 15.9[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



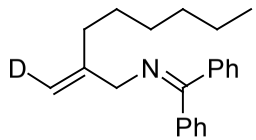
3a-*Z*-*d*

MD-293 1HNMR



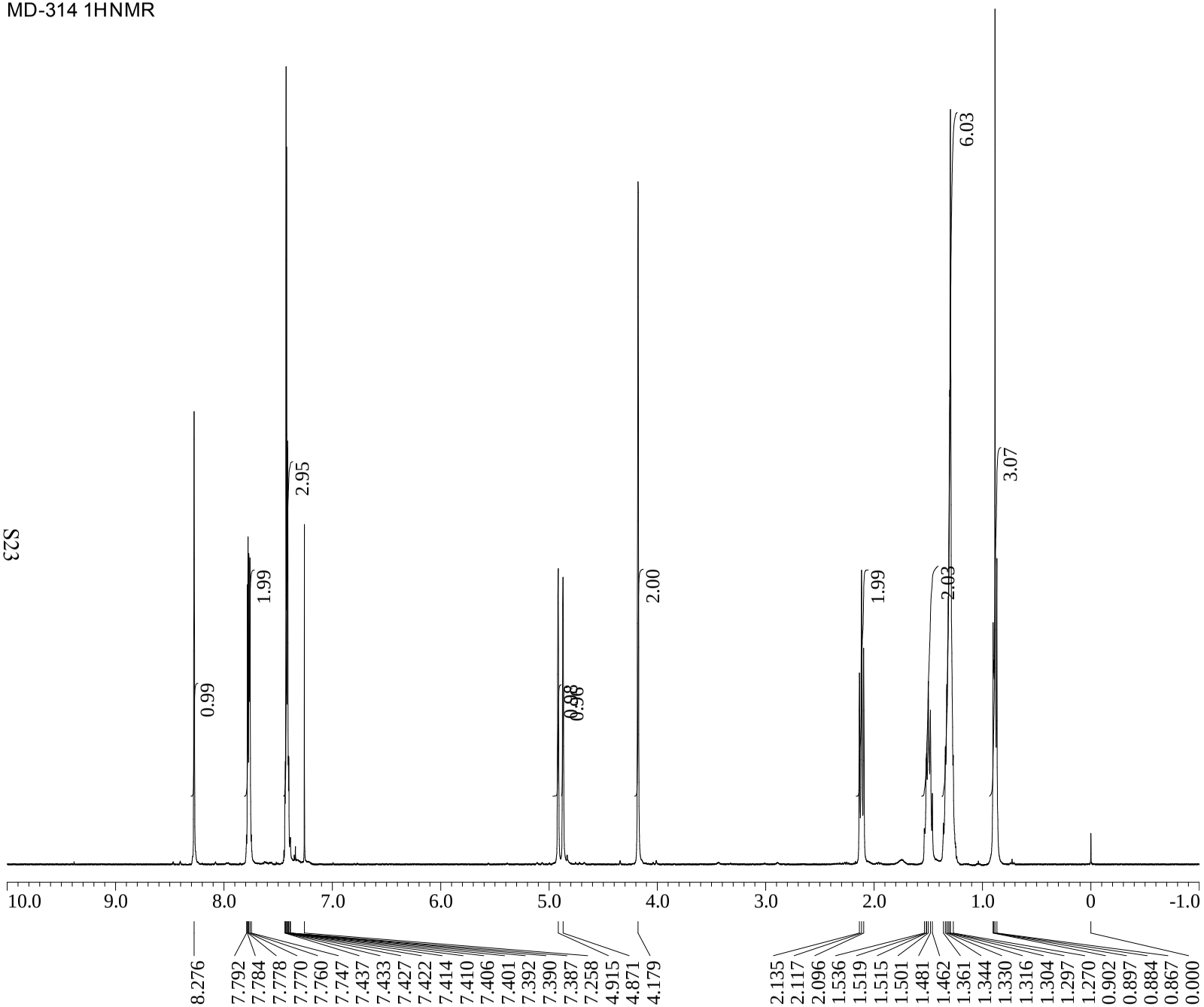
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-293 pure
Solvent           = CHLOROFORM-D
Creation_Time     = 12-FEB-2012 17:27:06
Revision_Time     =  5-MAR-2012 10:57:29
Current_Time      =  5-MAR-2012 10:57:58

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 24
Temp_Get          = 15.3[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



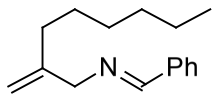
3a-*E*-*d*

MD-314 1HNMR



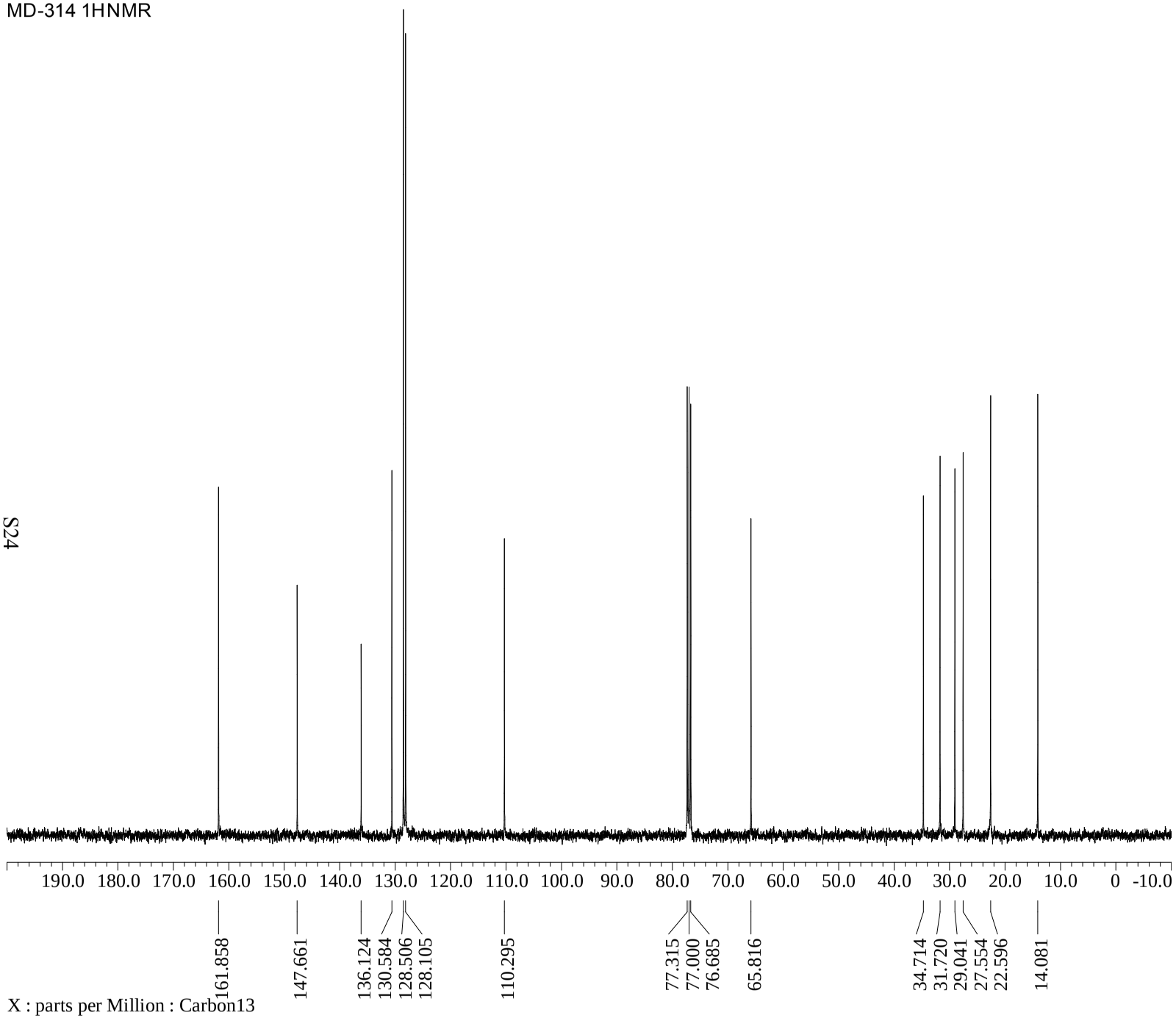
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-314
Solvent           = CHLOROFORM-D
Creation_Time     =  9-MAR-2012 16:36:26
Revision_Time     =  9-MAR-2012 18:17:12
Current_Time      =  9-MAR-2012 18:18:10

Author            = delta
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 16.4[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



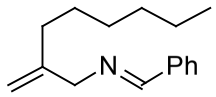
3a'

MD-314 1HNMR



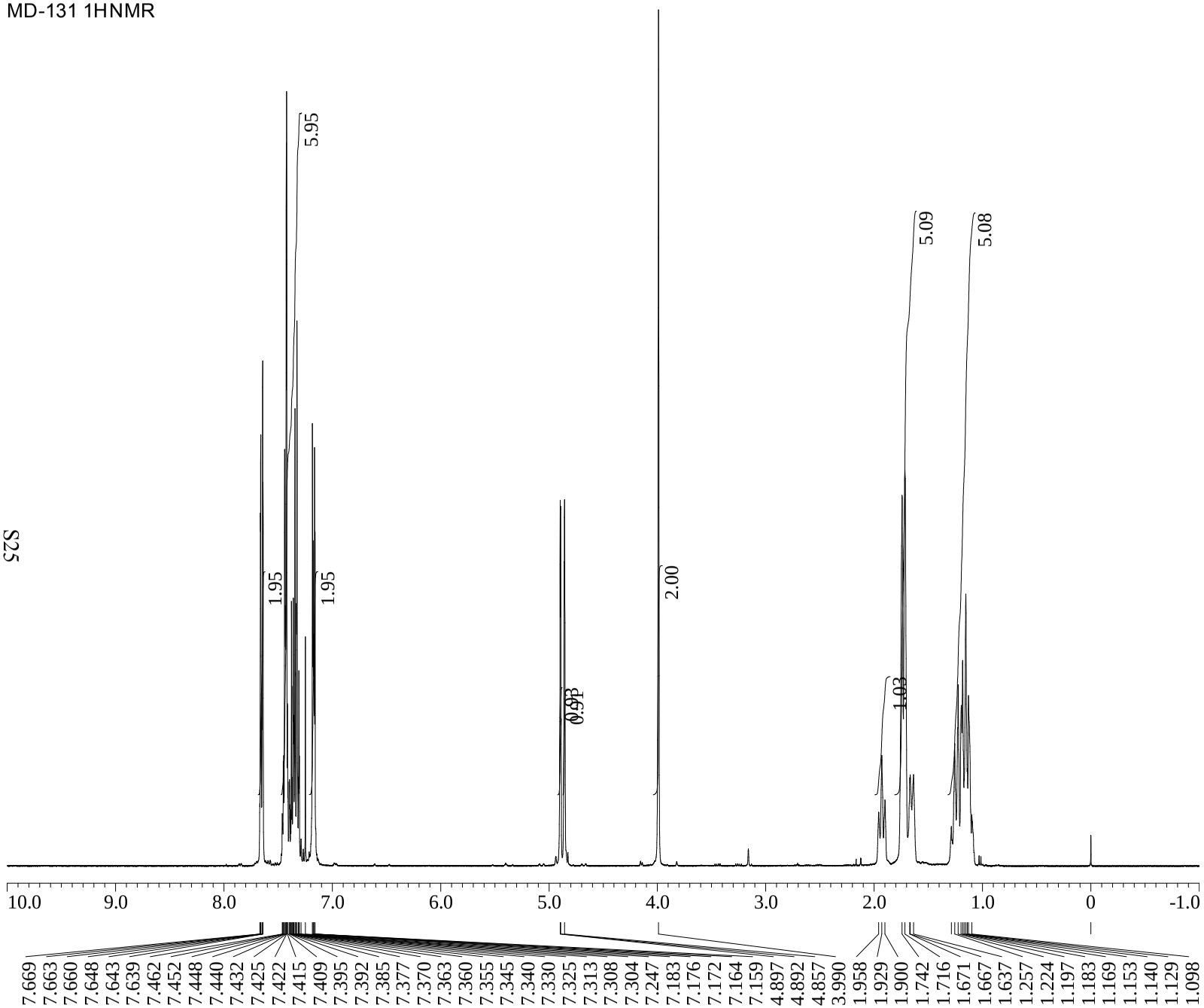
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-314
Solvent           = CHLOROFORM-D
Creation_Time     =  9-MAR-2012 15:05:30
Revision_Time     =  9-MAR-2012 15:13:54
Current_Time      =  9-MAR-2012 18:21:21

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 16.6[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



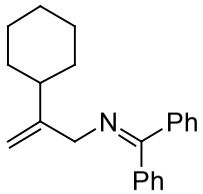
3a'

MD-131 1HNMR



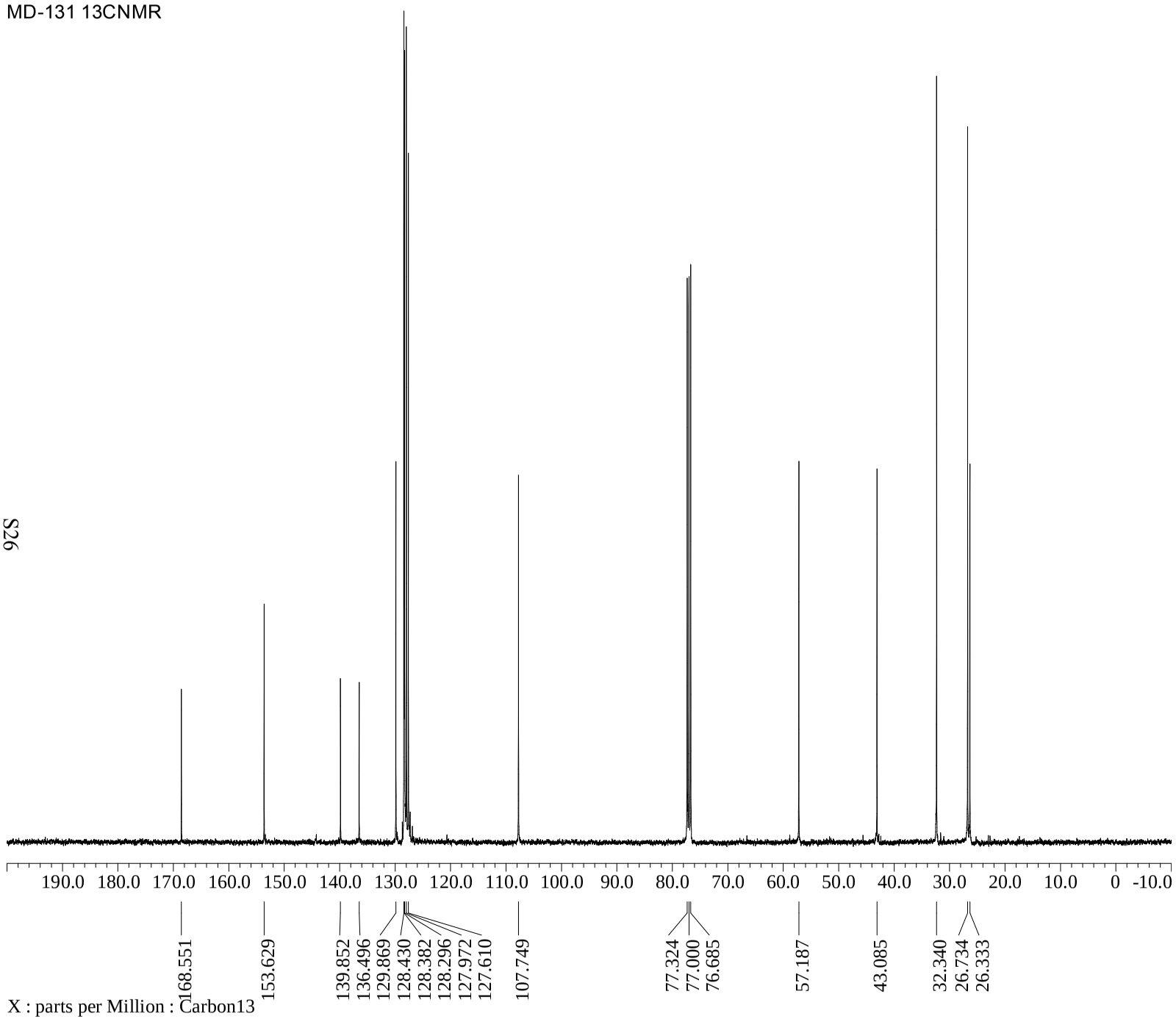
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-235 k2
Solvent           = CHLOROFORM-D
Creation_Time     =  9-NOV-2011 23:45:29
Revision_Time     =  5-MAR-2012 11:02:48
Current_Time      =  5-MAR-2012 11:03:09

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 20.4[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



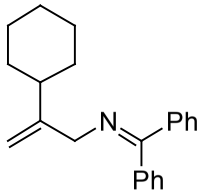
3b

MD-131 13CNMR



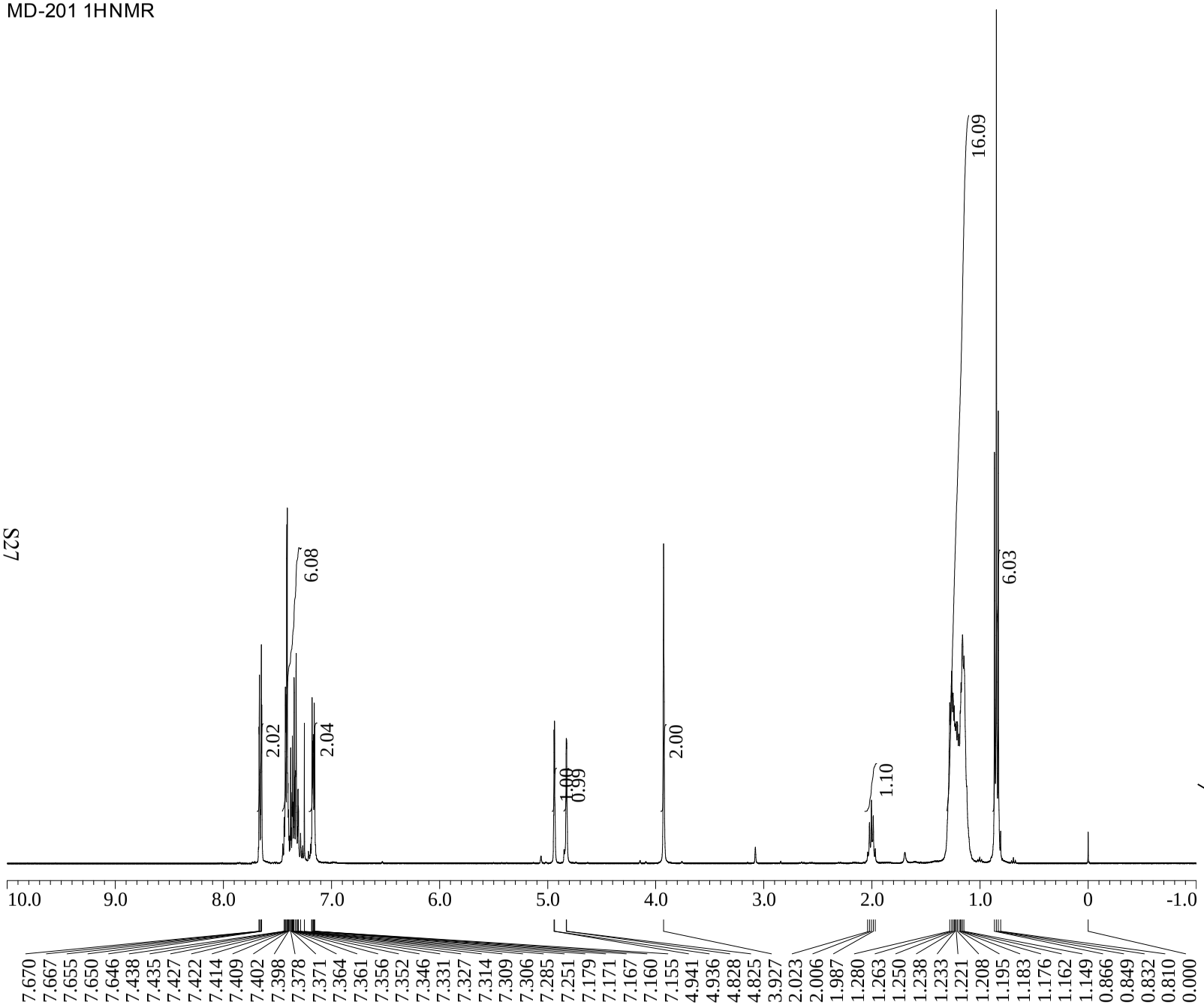
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-235 k3
Solvent           = CHLOROFORM-D
Creation_Time     =  9-NOV-2011 23:54:50
Revision_Time     =  5-MAR-2012 12:16:58
Current_Time      =  5-MAR-2012 12:17:45

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.9[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



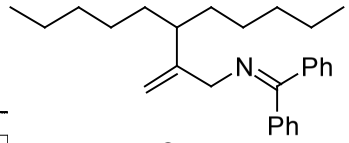
3b

MD-201 1HNMR



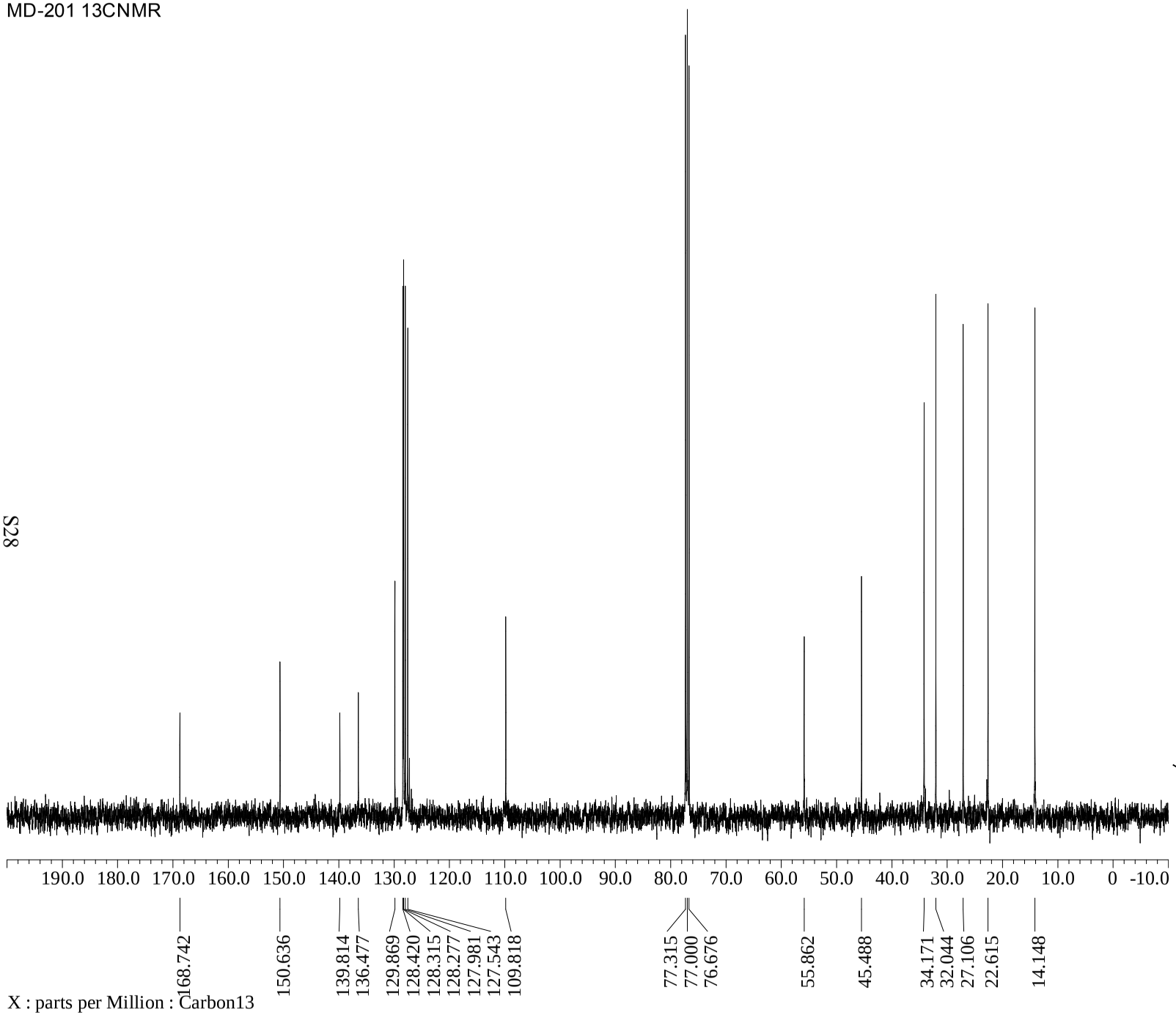
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-306
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2012 00:50:32
Revision_Time     =  5-MAR-2012 11:06:33
Current_Time      =  5-MAR-2012 11:06:59

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 17.4[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

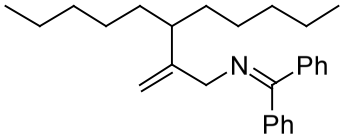


3c

MD-201 13CNMR

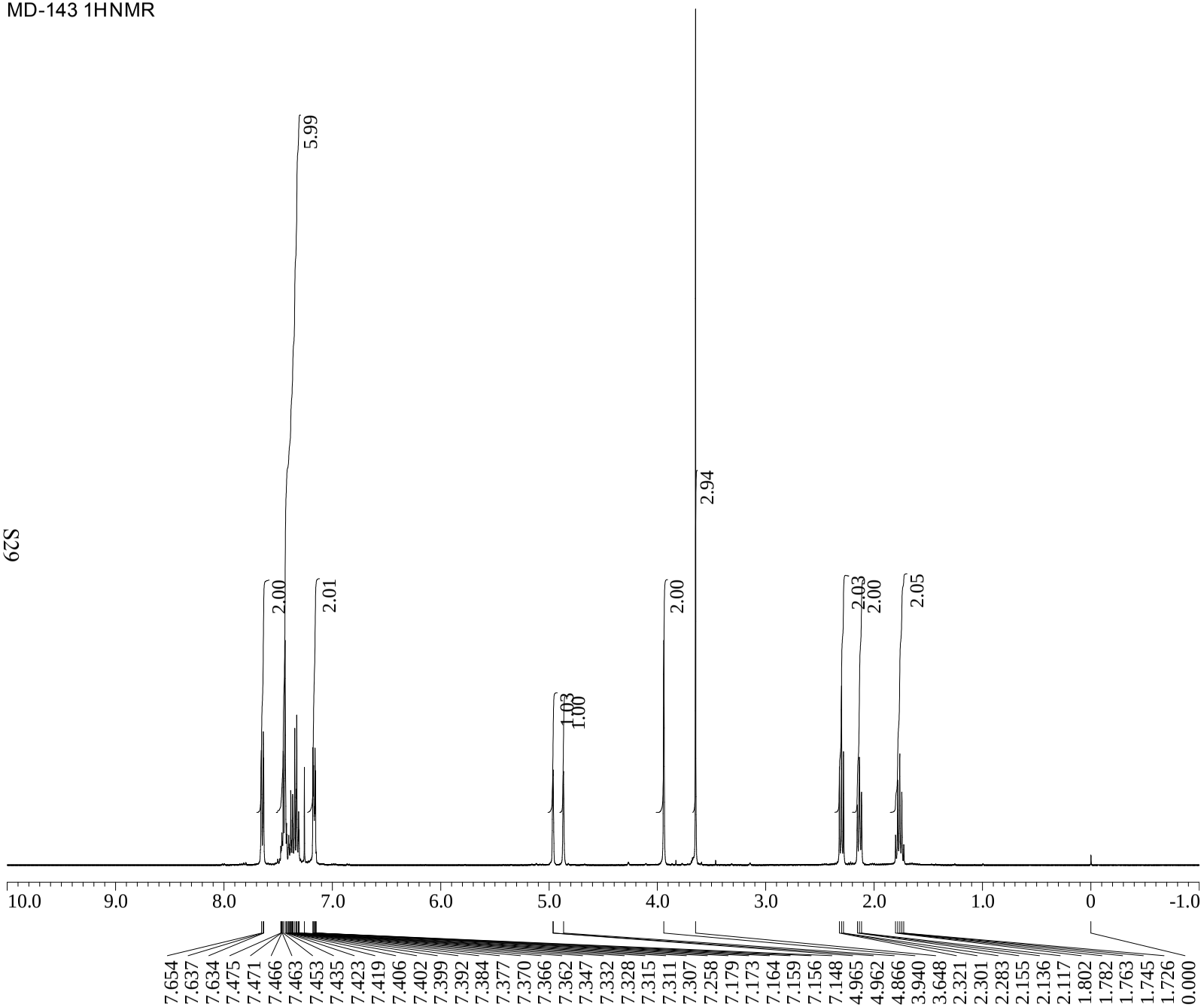



```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-306
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2012 01:09:06
Revision_Time     =  5-MAR-2012 12:30:52
Current_Time      =  5-MAR-2012 12:32:46

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 41
Total_Scans       = 41

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 17.7[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



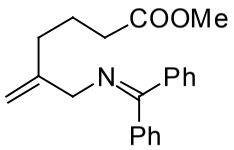
3c

MD-143 1HNMR



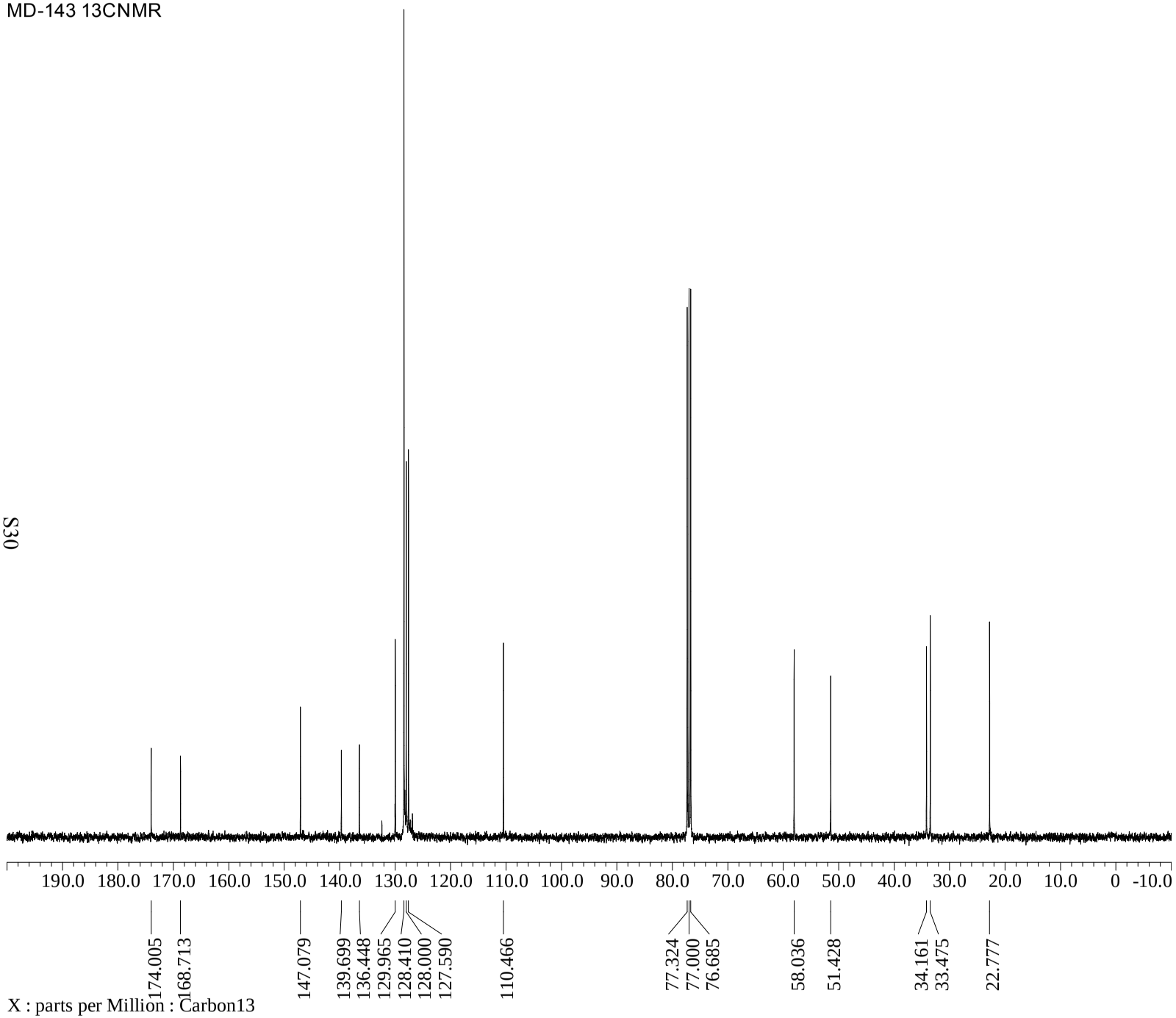
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = R=(CH2)3COOMe
Solvent           = CHLOROFORM-D
Creation_Time     = 16-SEP-2011 20:38:09
Revision_Time     =  5-MAR-2012 11:08:07
Current_Time      =  5-MAR-2012 11:08:29

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 22.8[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



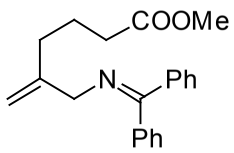
3d

MD-143 13CNMR



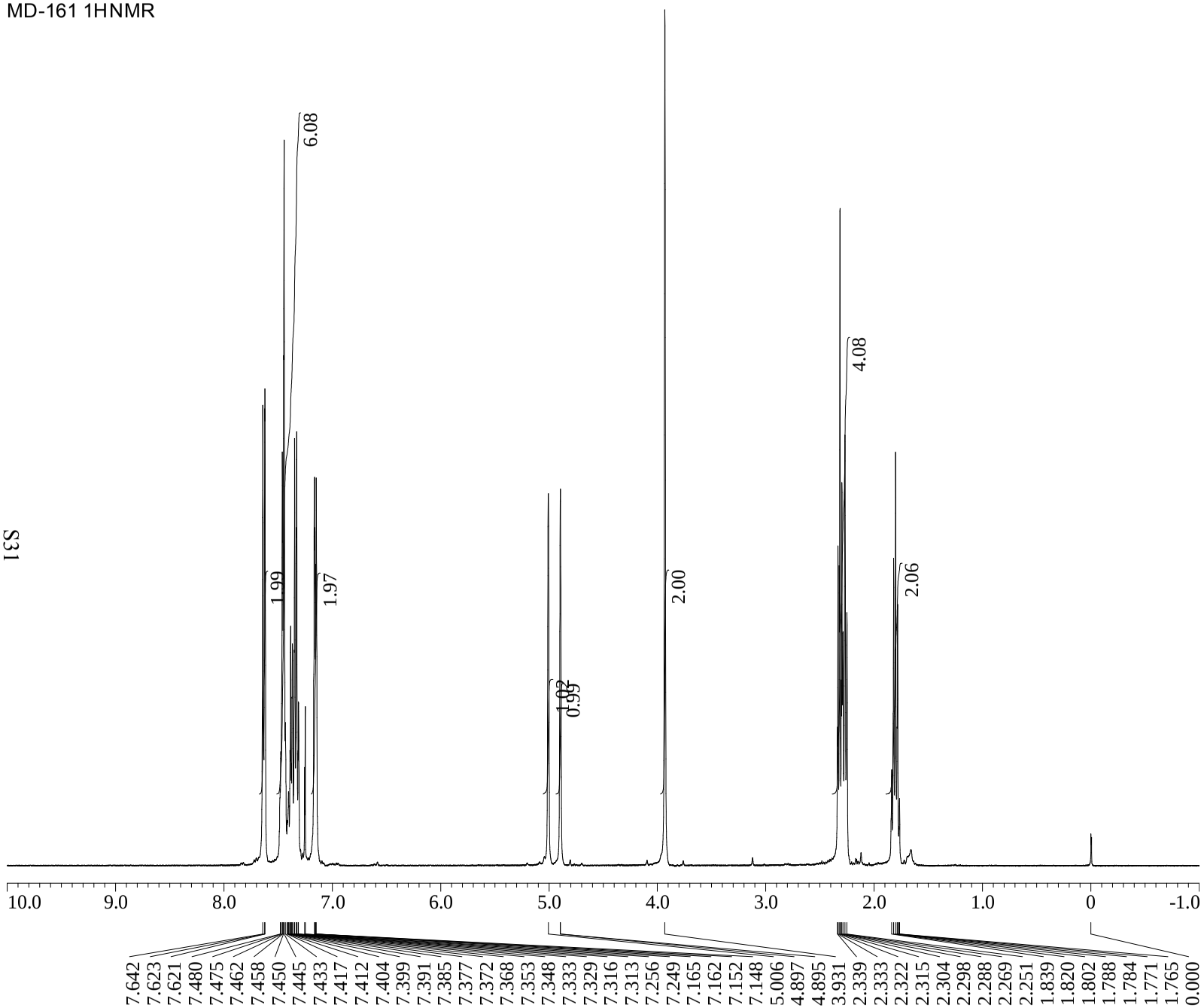
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = R=(CH2)3COOMe
Solvent           = CHLOROFORM-D
Creation_Time     = 21-SEP-2011 13:36:14
Revision_Time     =  5-MAR-2012 12:35:43
Current_Time      =  5-MAR-2012 12:36:05

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 277
Total_Scans       = 277

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.7[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



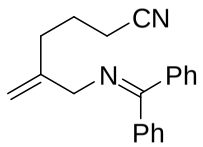
3d

MD-161 1HNMR



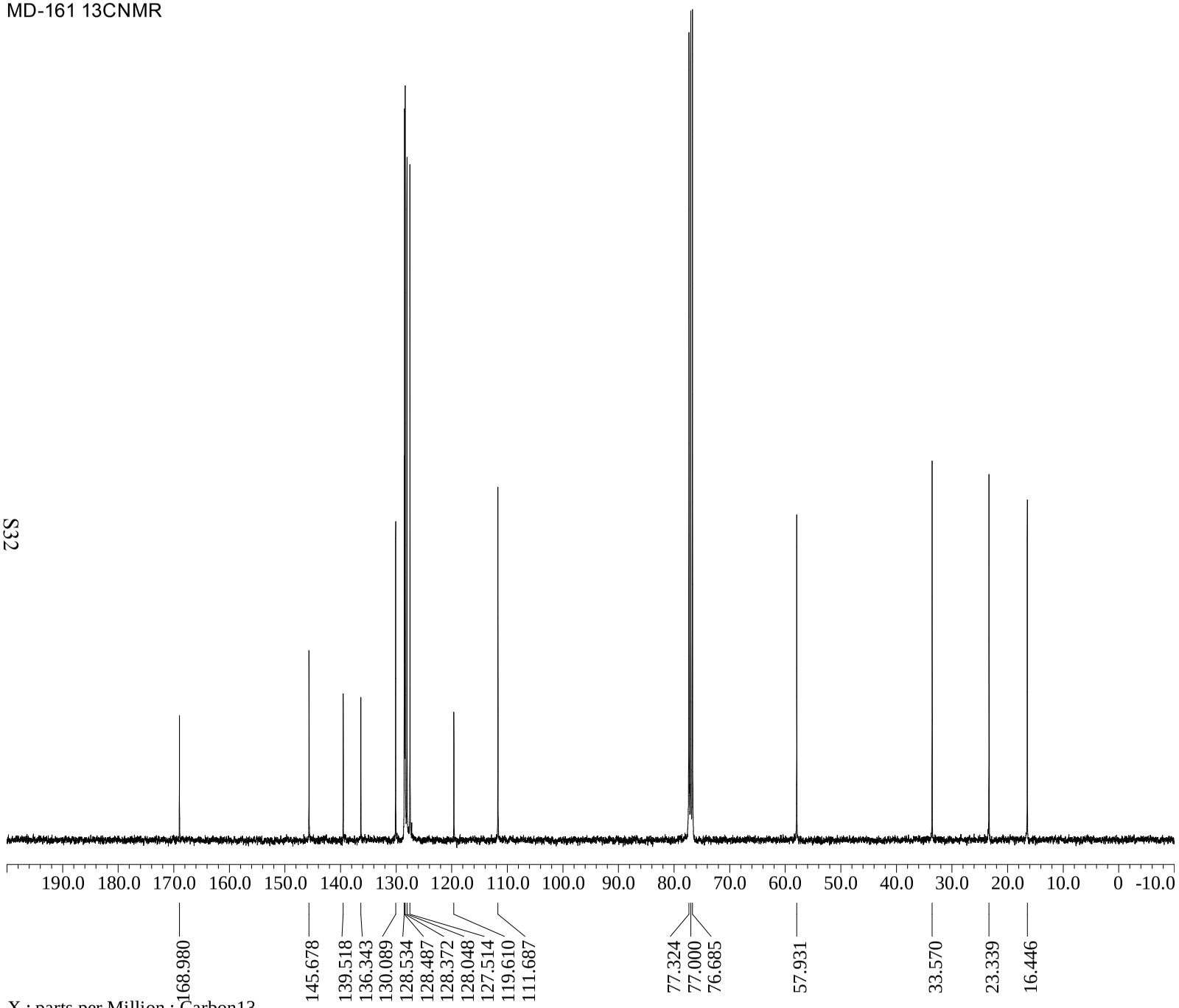
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-236 k3
Solvent           = CHLOROFORM-D
Creation_Time     = 10-NOV-2011 02:49:37
Revision_Time     =  5-MAR-2012 11:09:56
Current_Time      =  5-MAR-2012 11:10:17

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 20.7[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



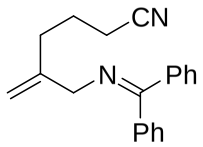
3e

MD-161 13CNMR



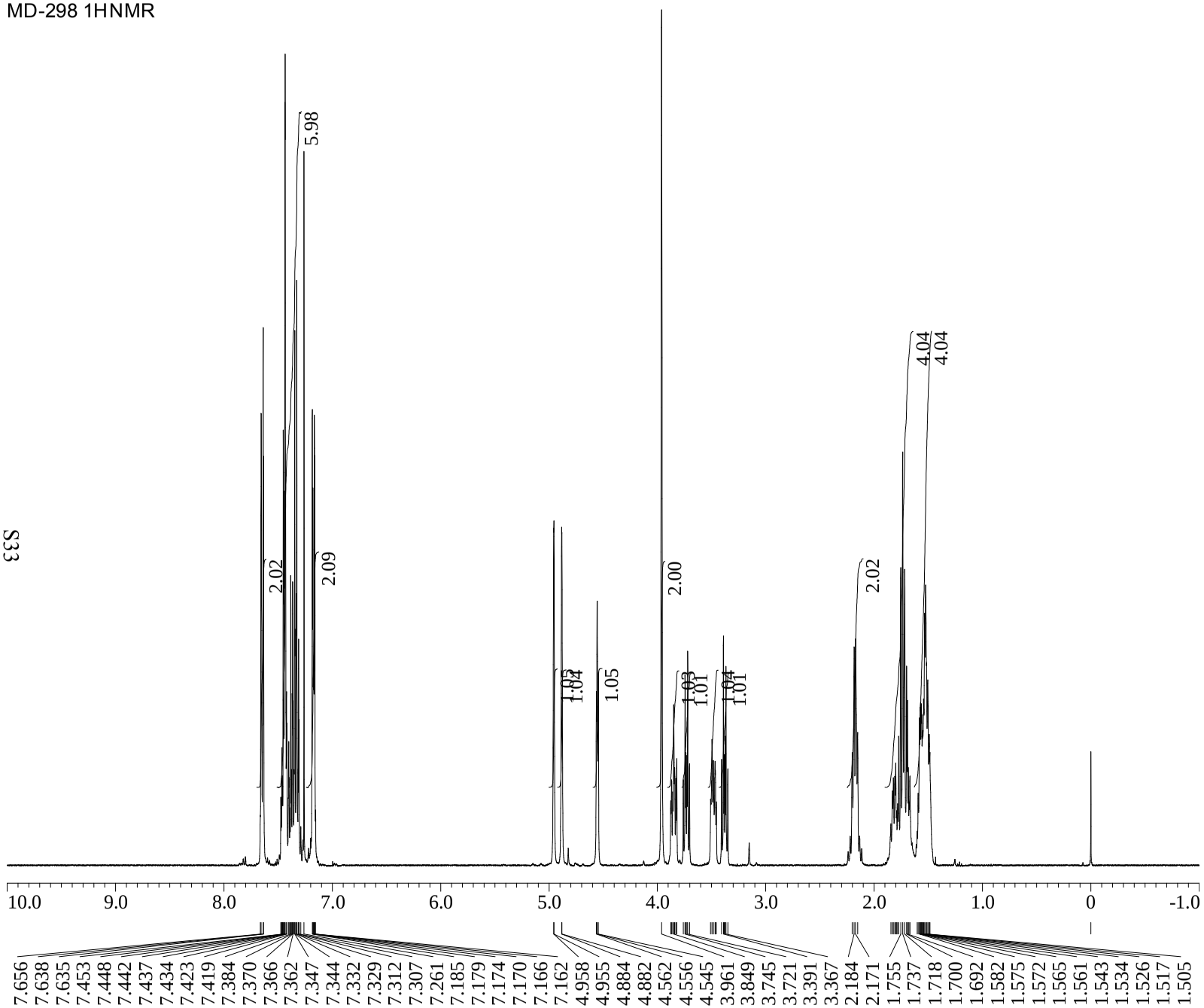
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-236 k3
Solvent           = CHLOROFORM-D
Creation_Time     = 10-NOV-2011 01:56:22
Revision_Time     =  5-MAR-2012 12:37:02
Current_Time      =  5-MAR-2012 12:37:42

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



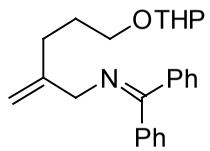
3e

MD-298 1HNMR



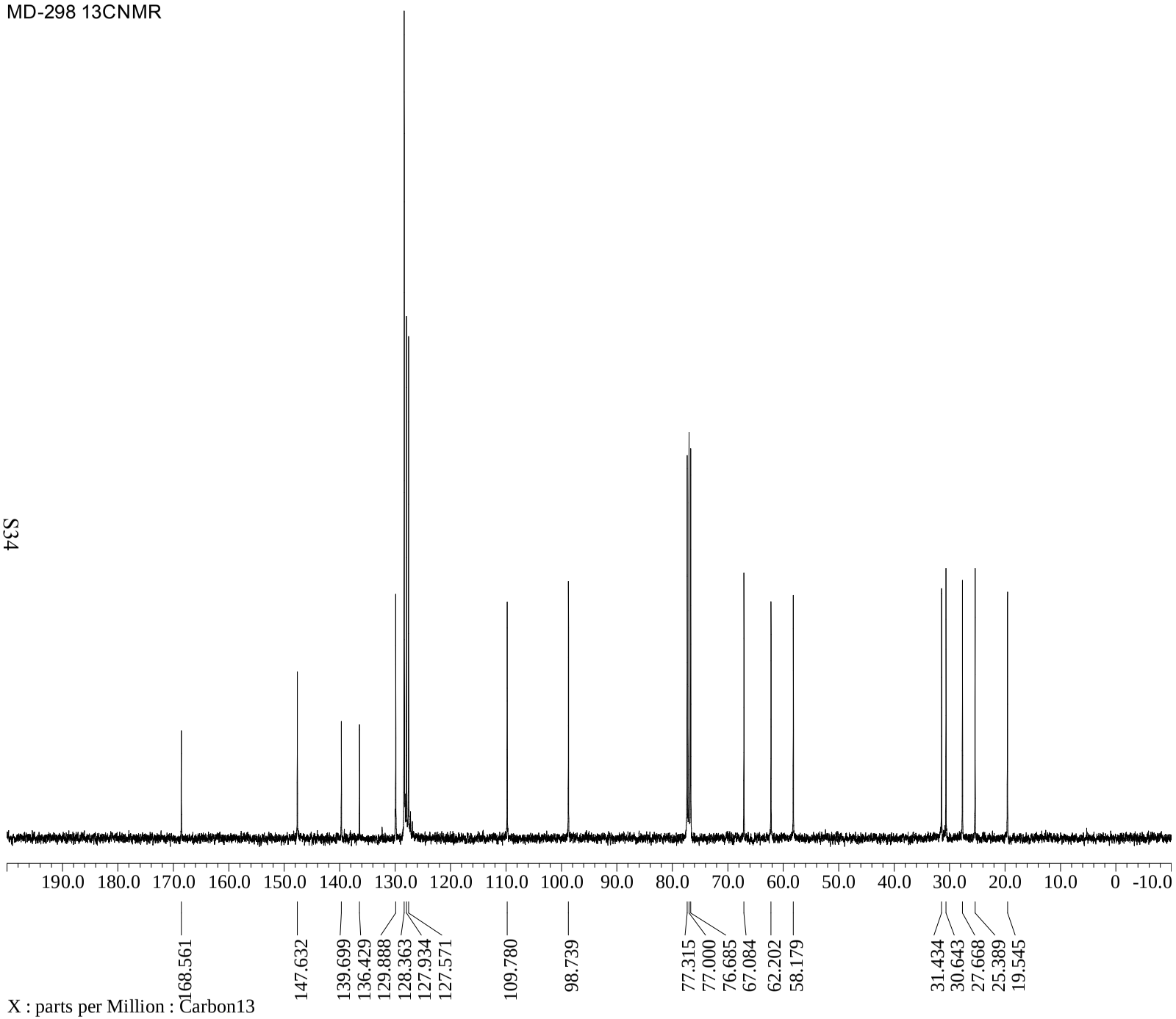
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = (CH2)3OTHP
Solvent           = CHLOROFORM-D
Creation_Time     = 23-FEB-2012 21:40:52
Revision_Time     =  5-MAR-2012 11:11:32
Current_Time      =  5-MAR-2012 11:12:03

Author            = delta
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 16.8[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



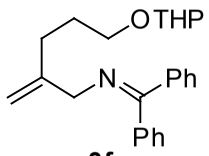
3f

MD-298 13CNMR



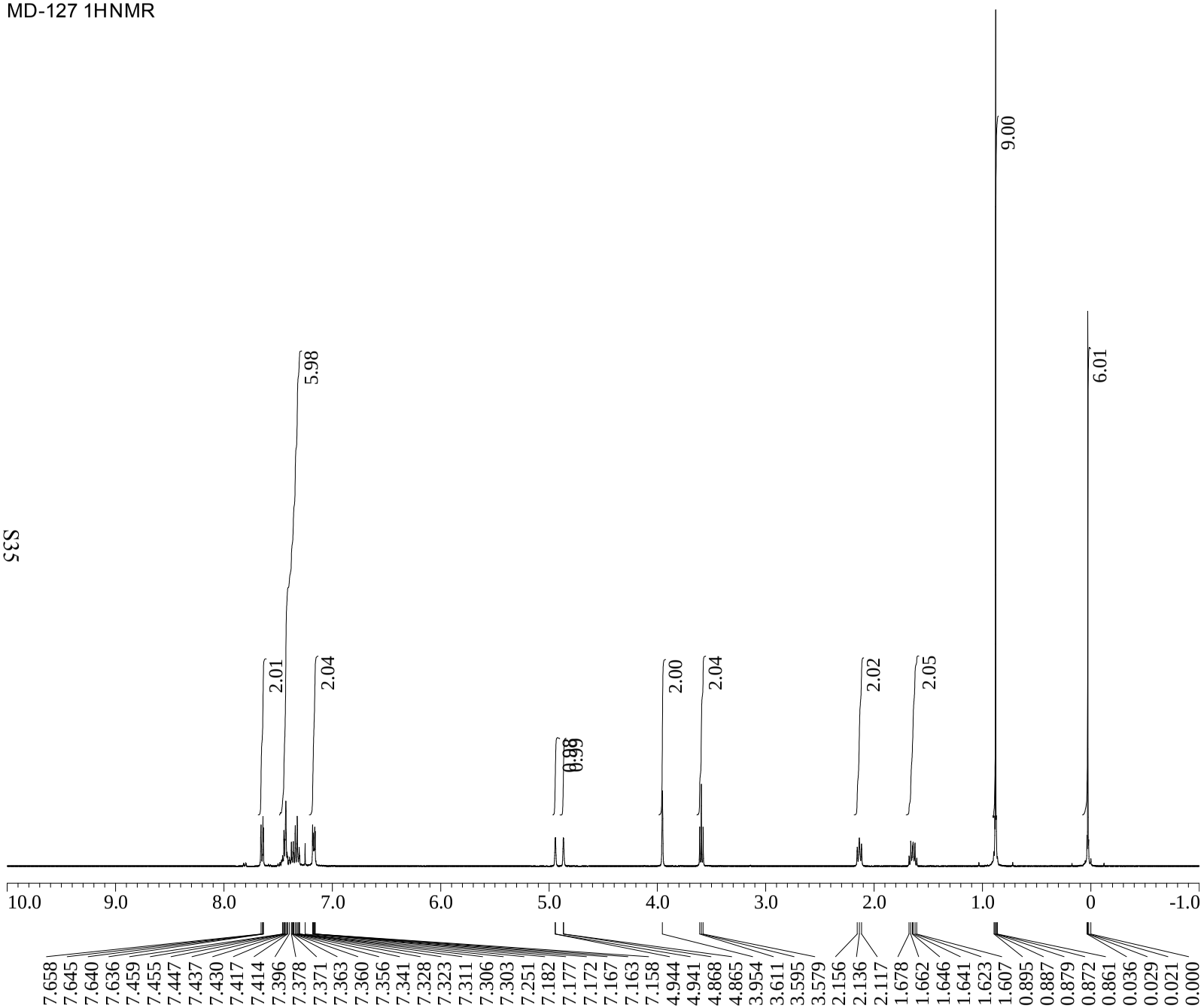
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = (CH2)3OTHP
Solvent           = CHLOROFORM-D
Creation_Time     = 23-FEB-2012 12:55:18
Revision_Time     = 23-FEB-2012 13:04:09
Current_Time      =  5-MAR-2012 12:38:45

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 134
Total_Scans       = 134

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 17.3[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



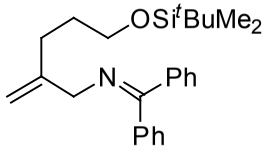
3f

MD-127 1HNMR



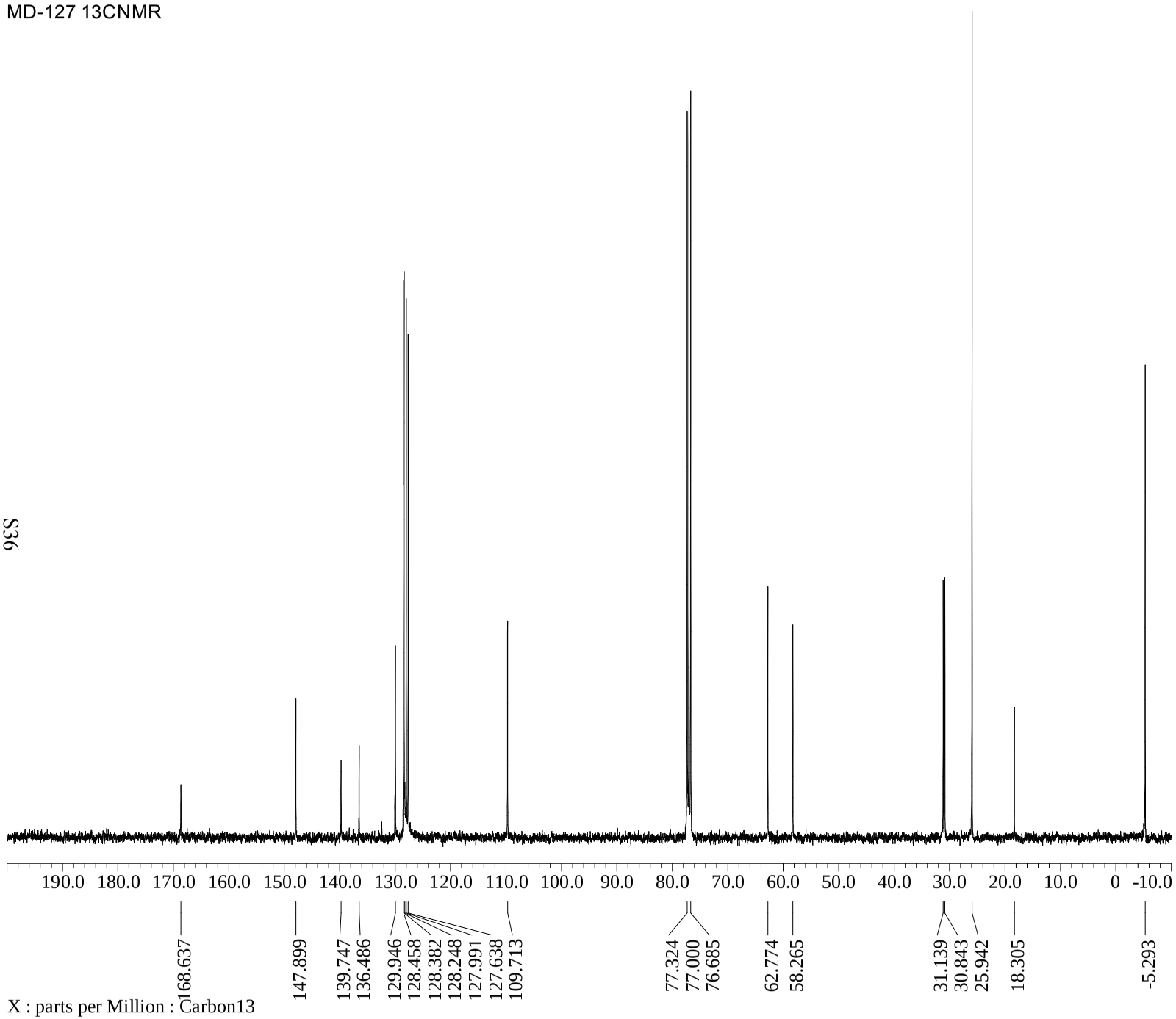
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = R=(CH2)3OSitBuMe2
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2011 19:37:35
Revision_Time     =  5-MAR-2012 11:13:20
Current_Time      =  5-MAR-2012 11:13:39

Author            = delta
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 20.4[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

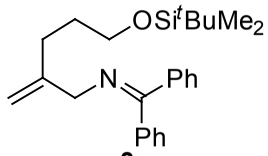


3g

MD-127 13CNMR

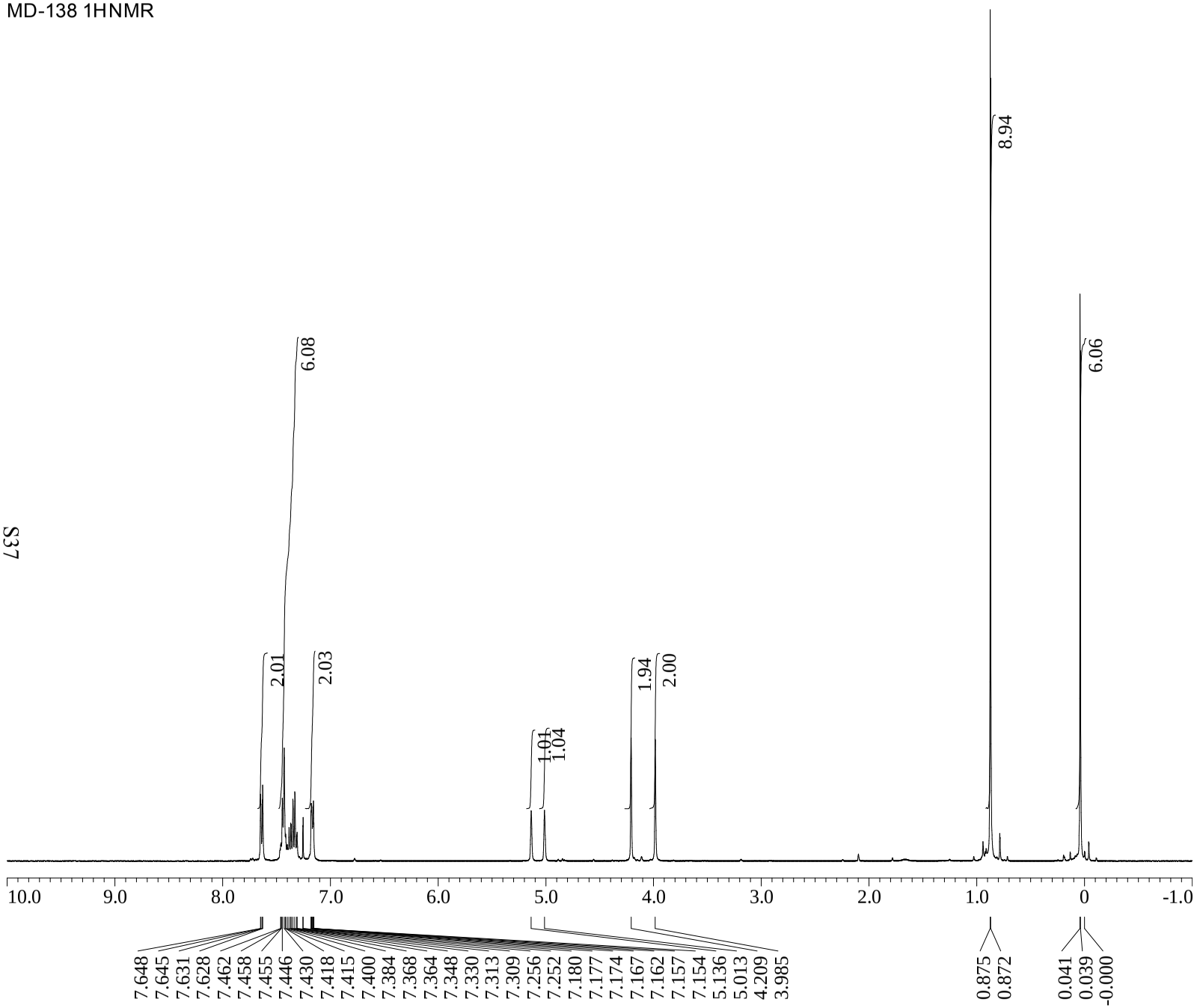



```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = R=(CH2)3OSitBuMe2
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2011 19:39:16
Revision_Time     = 27-SEP-2011 20:07:18
Current_Time      =  5-MAR-2012 12:39:39

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 502.0
Total_Scans       = 502.0

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.7[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



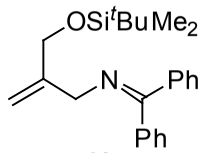
3g

MD-138 1HNMR



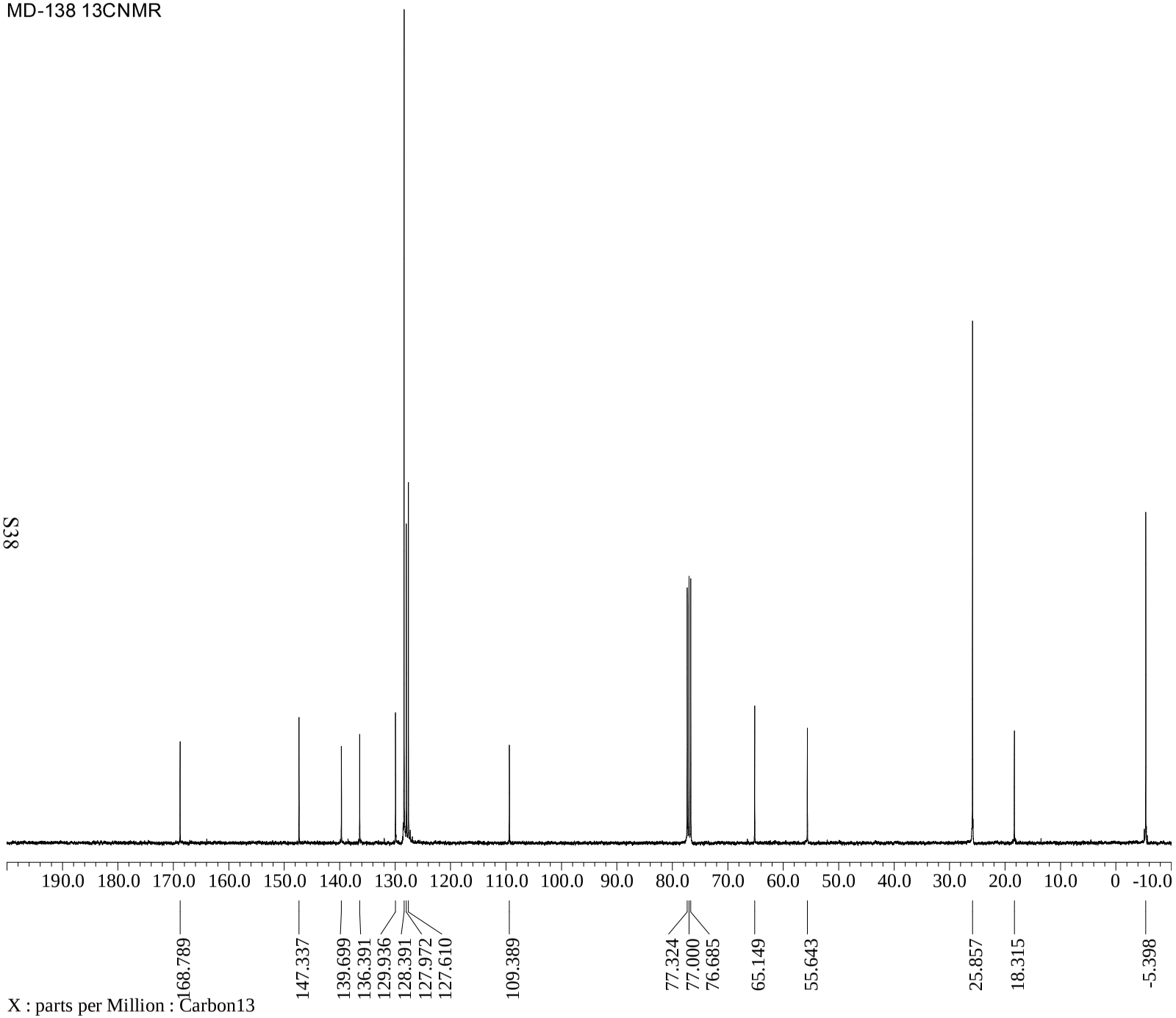
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-294
Solvent           = CHLOROFORM-D
Creation_Time     = 17-FEB-2012 22:20:50
Revision_Time     =  5-MAR-2012 15:40:07
Current_Time      =  5-MAR-2012 15:40:33

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 15.5[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

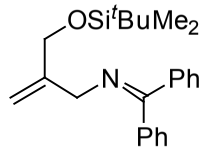


3h

MD-138 13CNMR

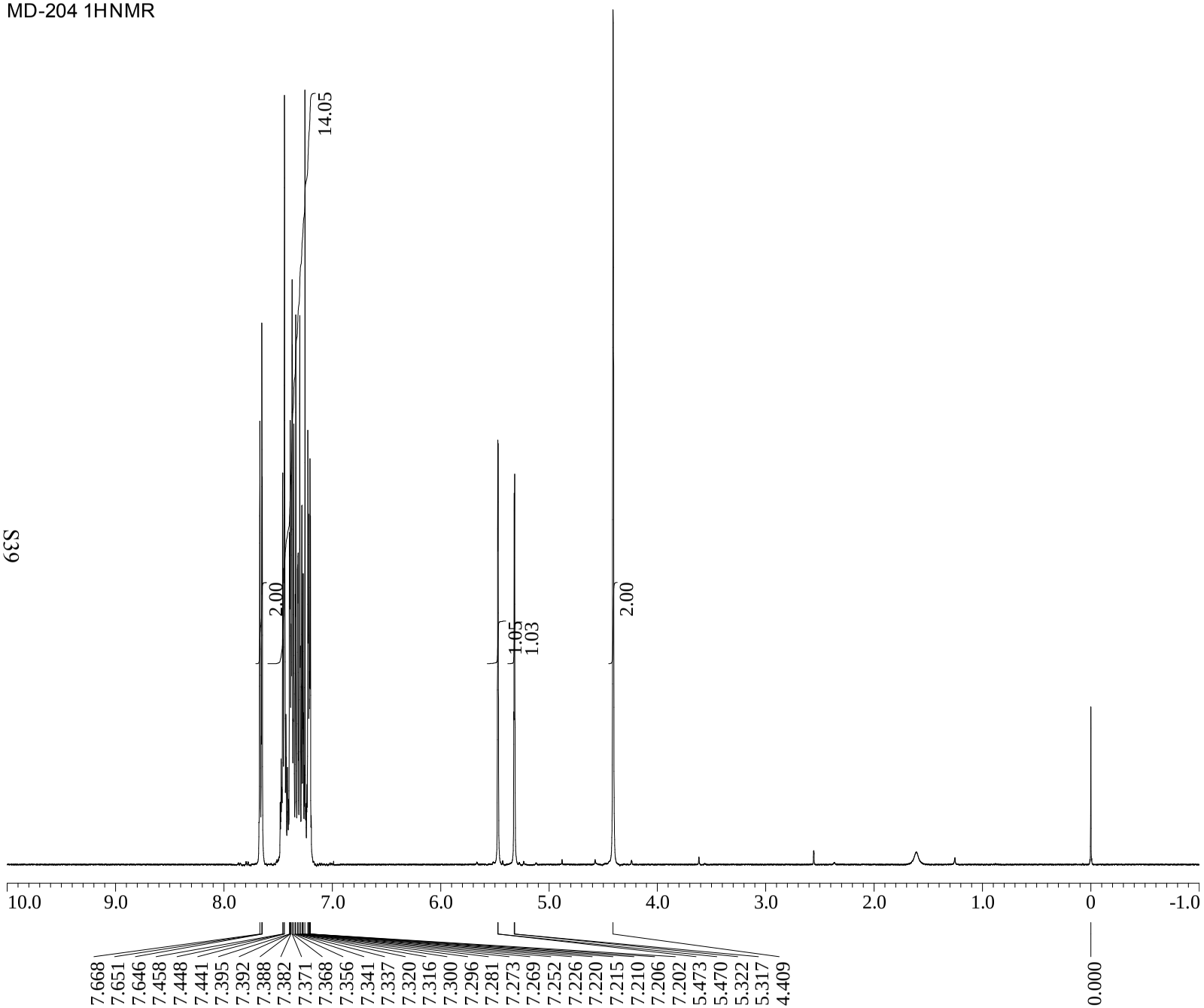



```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-294 pure
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2012 13:05:33
Revision_Time     = 15-FEB-2012 14:28:28
Current_Time      =  5-MAR-2012 12:40:15

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 15.6[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



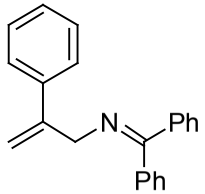
3h

MD-204 1HNMR



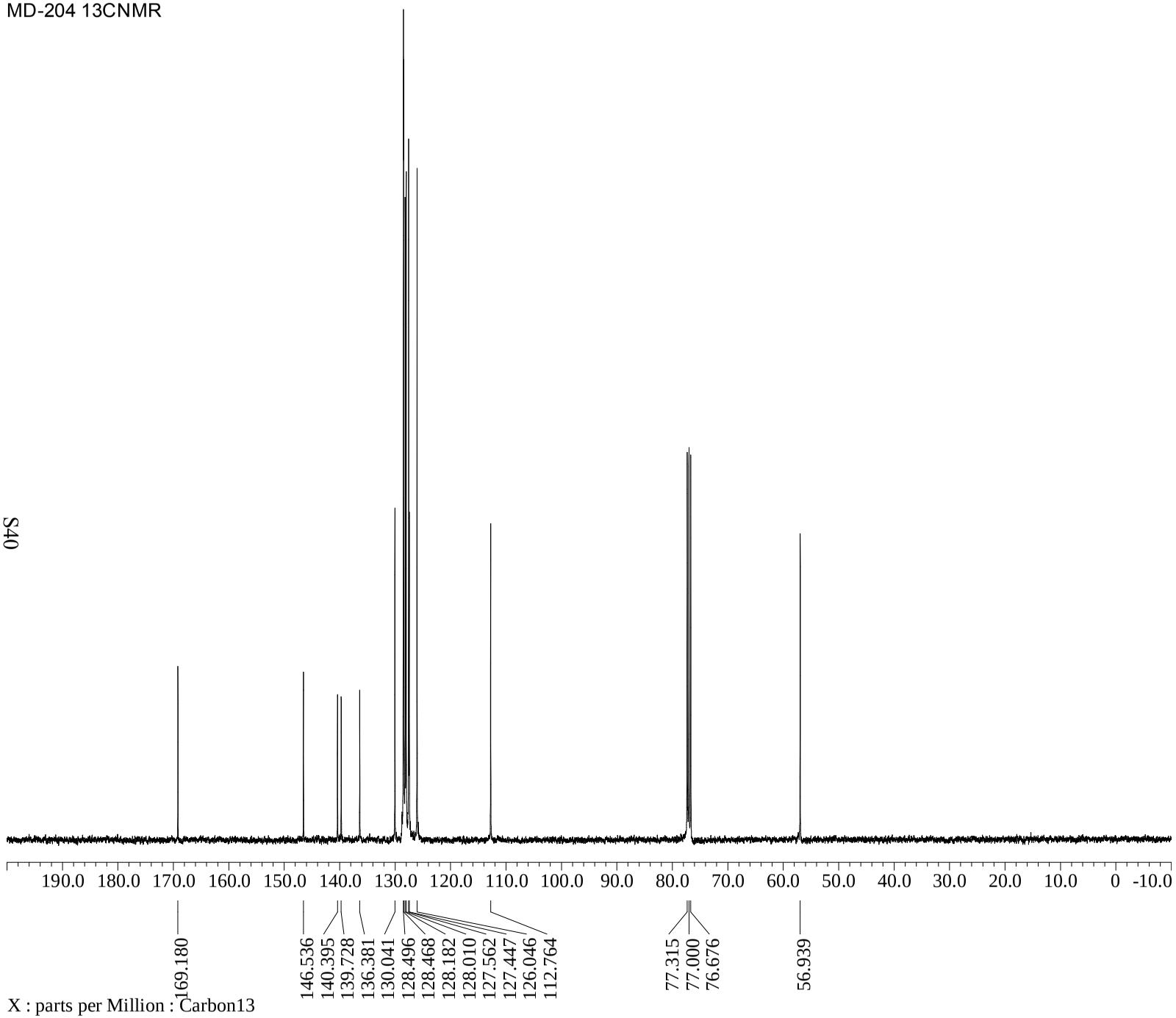
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-305
Solvent           = CHLOROFORM-D
Creation_Time     = 29-FEB-2012 23:30:50
Revision_Time     =  5-MAR-2012 11:20:04
Current_Time      =  5-MAR-2012 11:20:33

Author            = delta
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 16.7[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

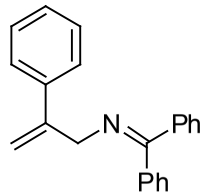


3i

MD-204 13CNMR

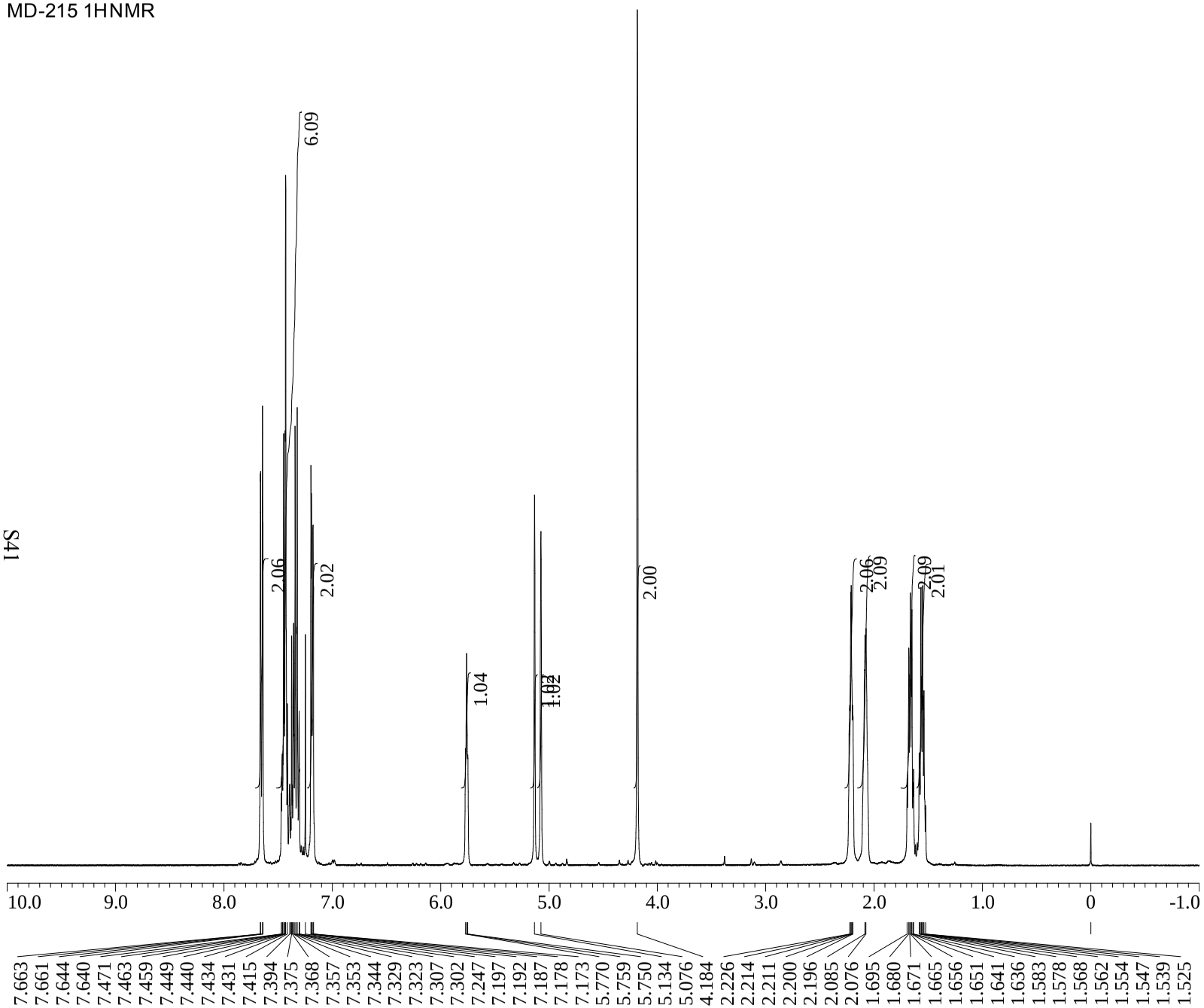



```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-241 k3
Solvent           = CHLOROFORM-D
Creation_Time     = 17-NOV-2011 00:07:12
Revision_Time     =  5-MAR-2012 12:40:38
Current_Time      =  5-MAR-2012 12:41:09

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.6[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



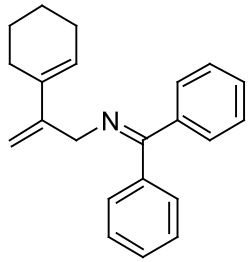
3i

MD-215 1HNMR



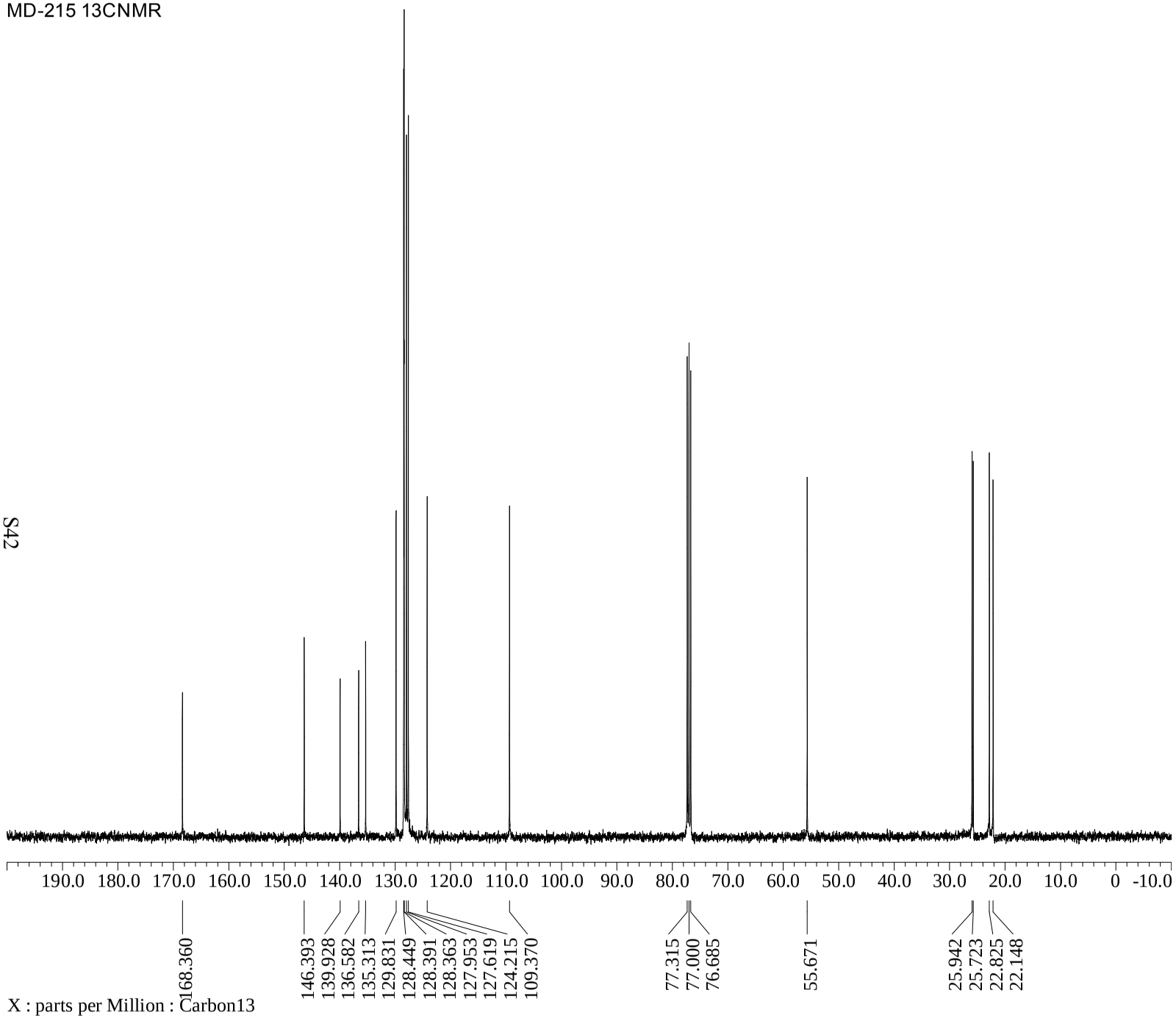
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-238 auto-column F5
Solvent           = CHLOROFORM-D
Creation_Time     = 15-NOV-2011 00:13:51
Revision_Time     =  5-MAR-2012 11:22:17
Current_Time      =  5-MAR-2012 11:22:37

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 21.2[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



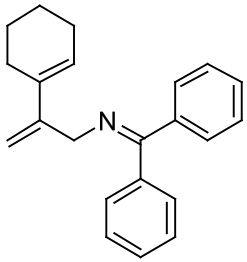
3j

MD-215 13CNMR



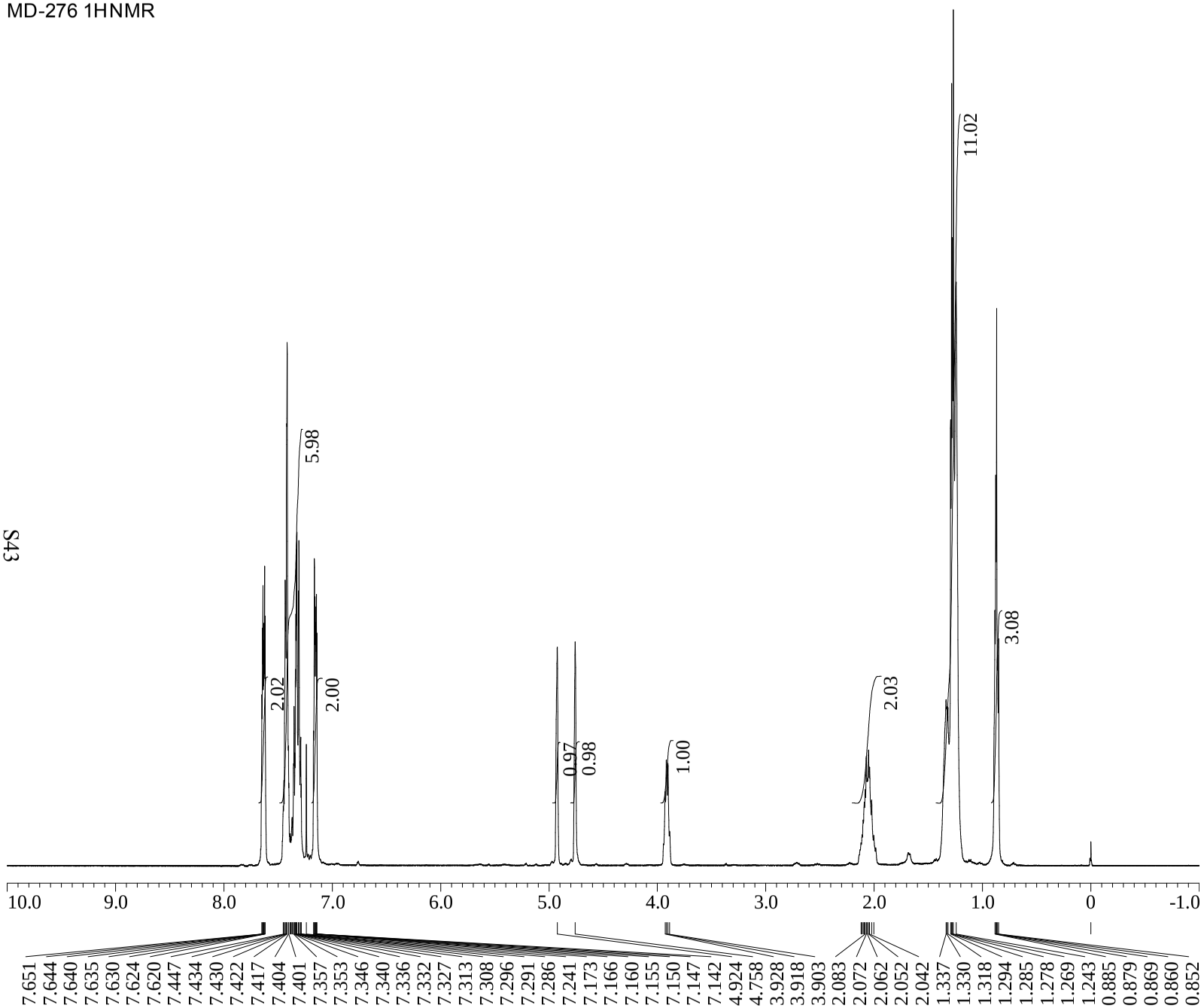
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-238 auto-column F5
Solvent           = CHLOROFORM-D
Creation_Time     = 14-NOV-2011 23:30:40
Revision_Time     =  5-MAR-2012 12:41:43
Current_Time      =  5-MAR-2012 12:42:13

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.5[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



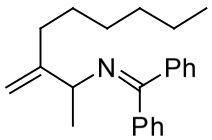
3j

MD-276 1HNMR



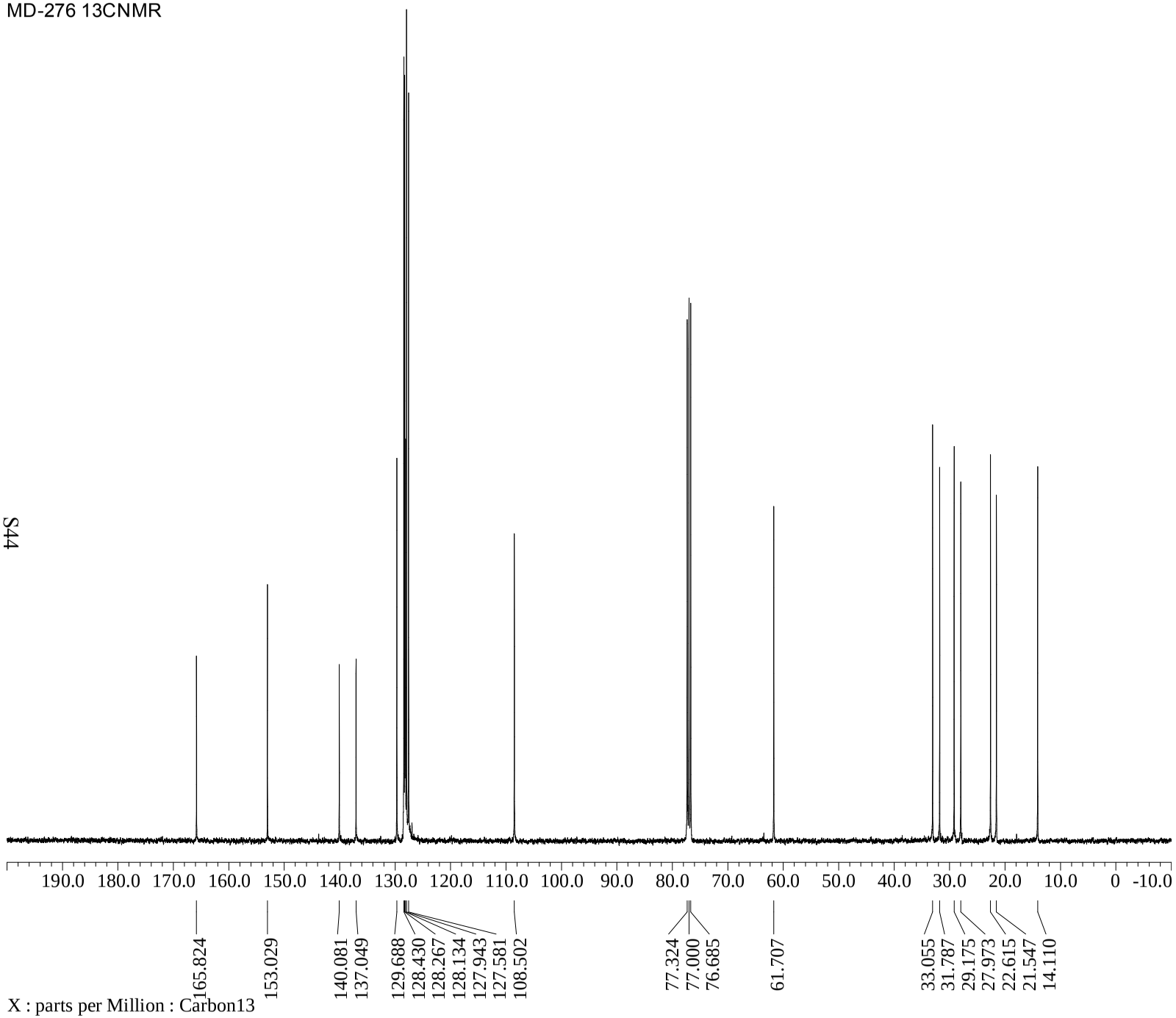
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-252
Solvent           = CHLOROFORM-D
Creation_Time     =  1-DEC-2011 23:02:39
Revision_Time     =  5-MAR-2012 11:24:22
Current_Time      =  5-MAR-2012 11:24:46

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 20.2[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

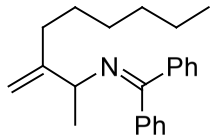


4b

MD-276 13CNMR

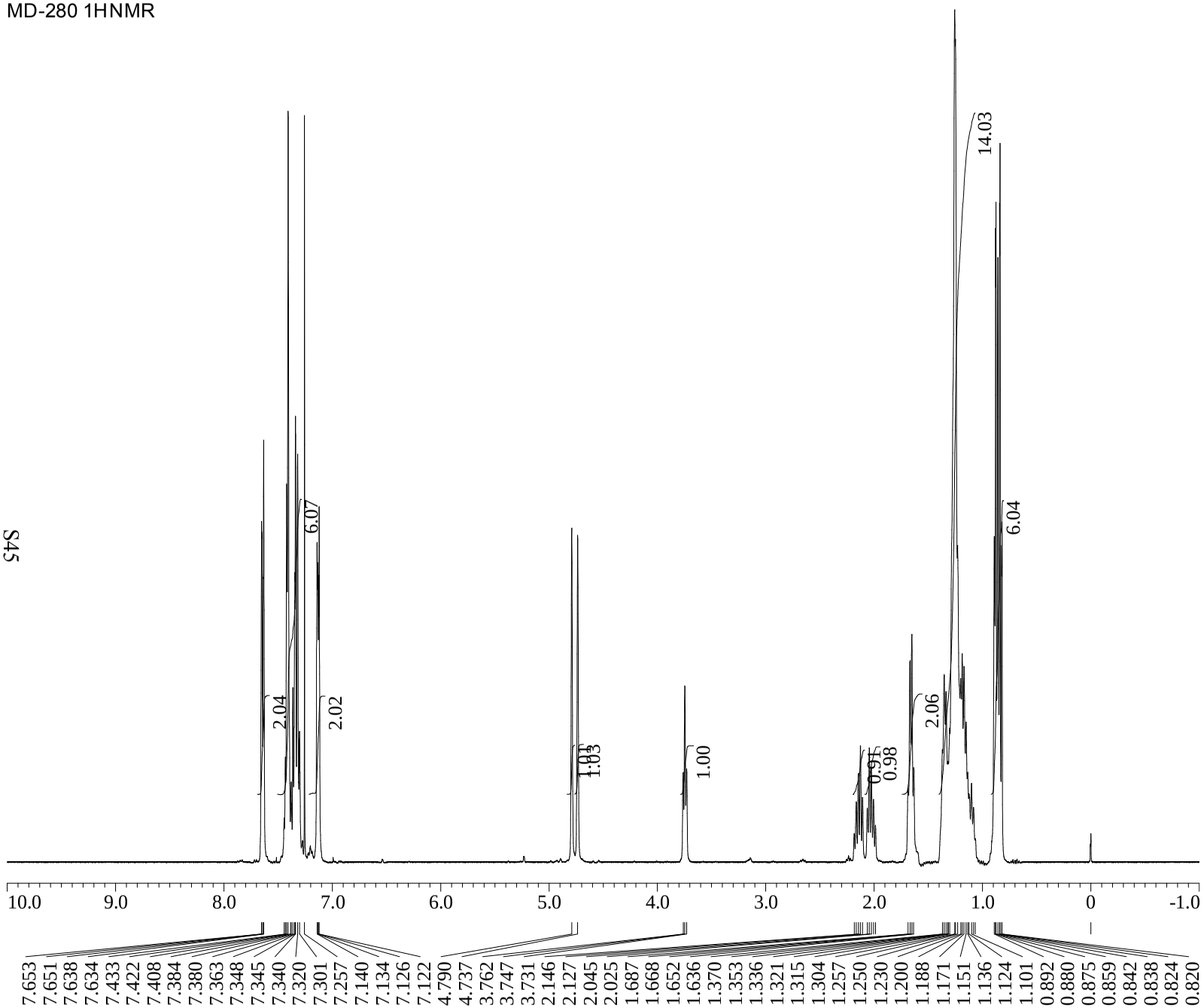



```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-252
Solvent           = CHLOROFORM-D
Creation_Time     =  1-DEC-2011 23:14:35
Revision_Time     =  5-MAR-2012 12:42:44
Current_Time      =  5-MAR-2012 12:43:05

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.5[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



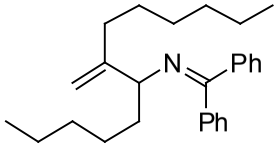
4b

MD-280 1HNMR



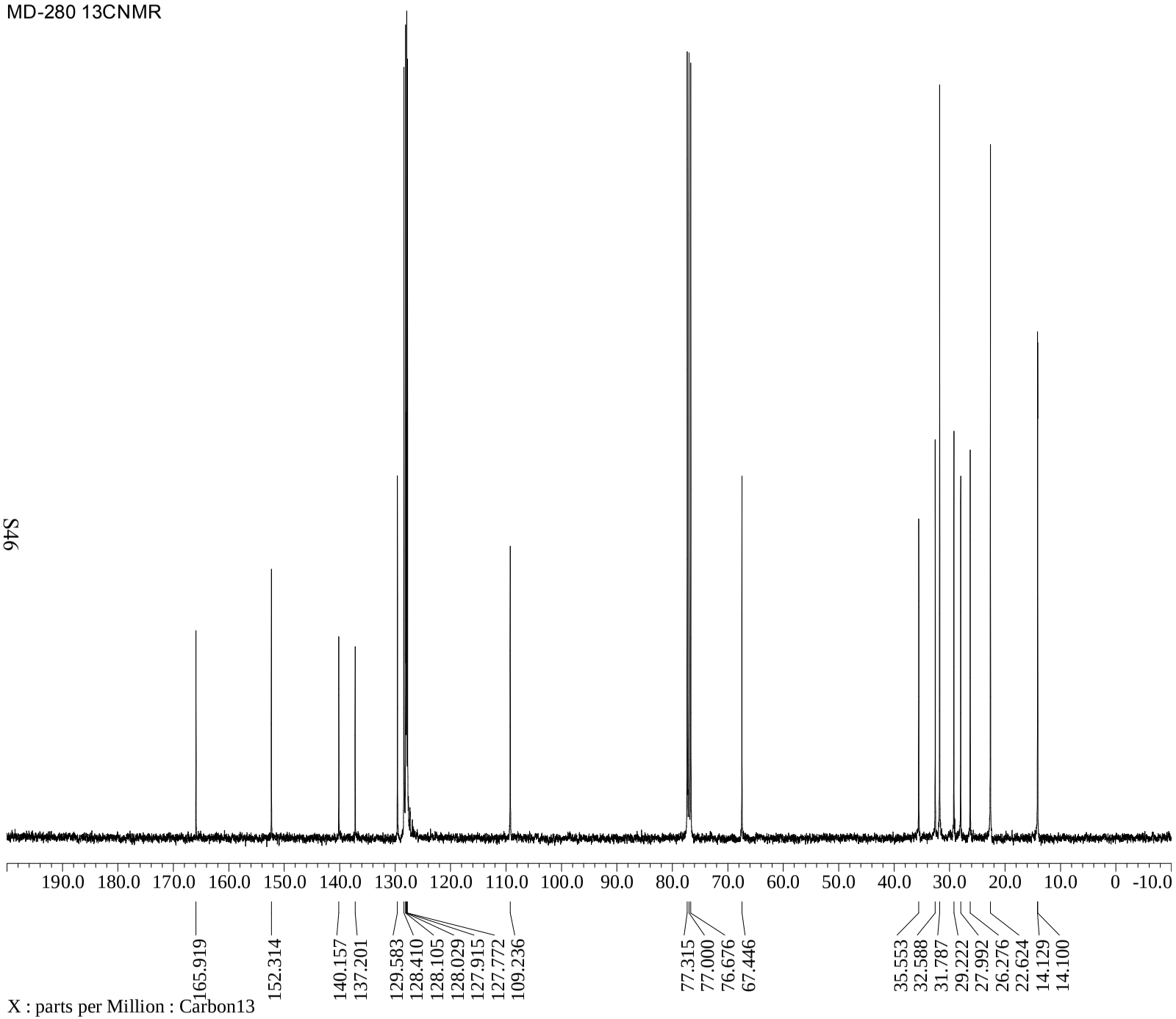
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-280 dis
Solvent           = CHLOROFORM-D
Creation_Time     = 31-JAN-2012 15:47:13
Revision_Time     =  5-MAR-2012 11:25:44
Current_Time      =  5-MAR-2012 11:26:02

Author            = delta
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 15.2[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

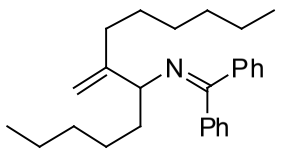


4c

MD-280 13CNMR

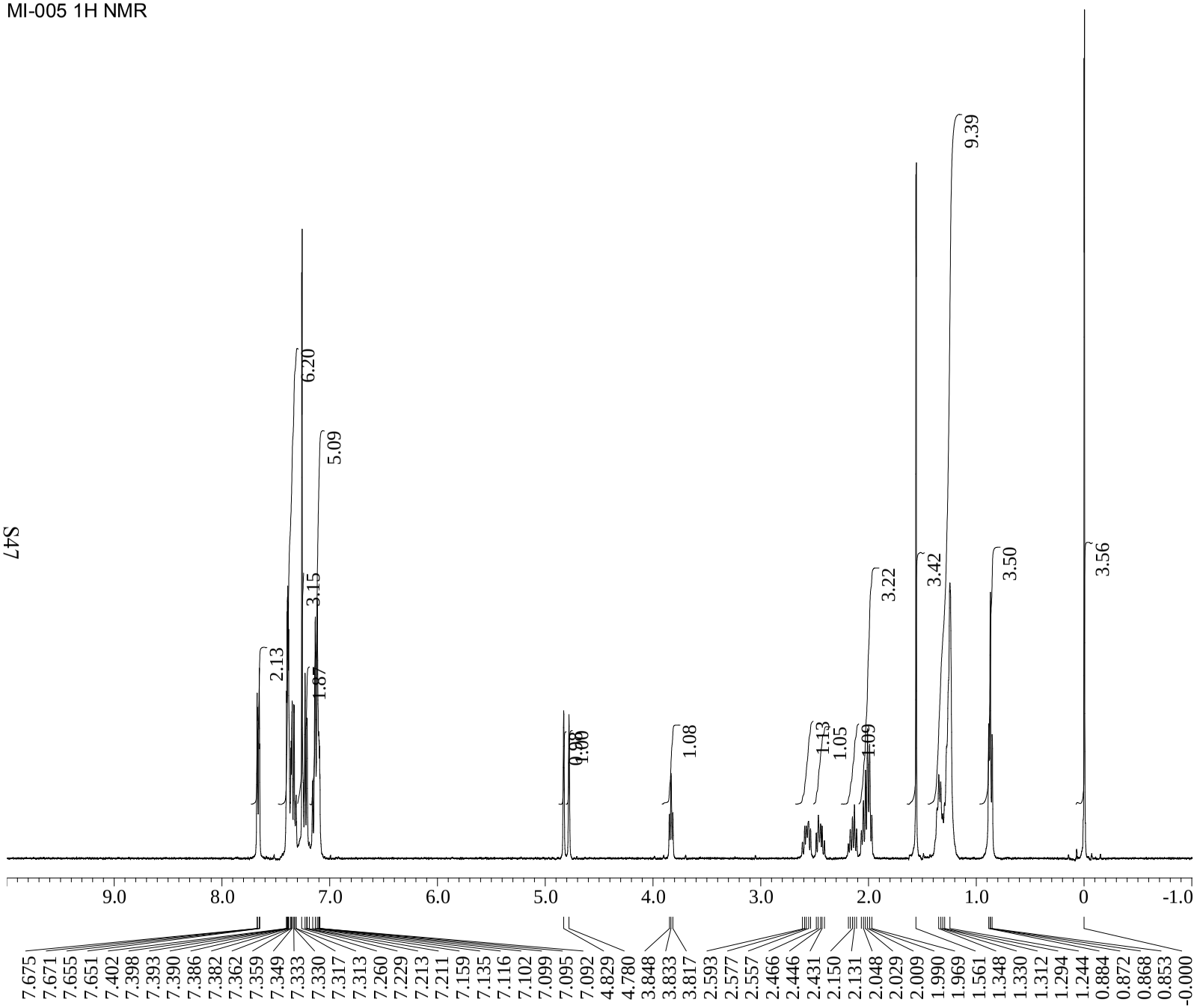



```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-280 dis
Solvent           = CHLOROFORM-D
Creation_Time     = 31-JAN-2012 16:05:05
Revision_Time     = 31-JAN-2012 16:32:51
Current_Time      =  5-MAR-2012 12:43:46

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 15.5[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



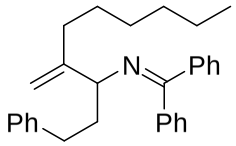
4c

MI-005 1H NMR



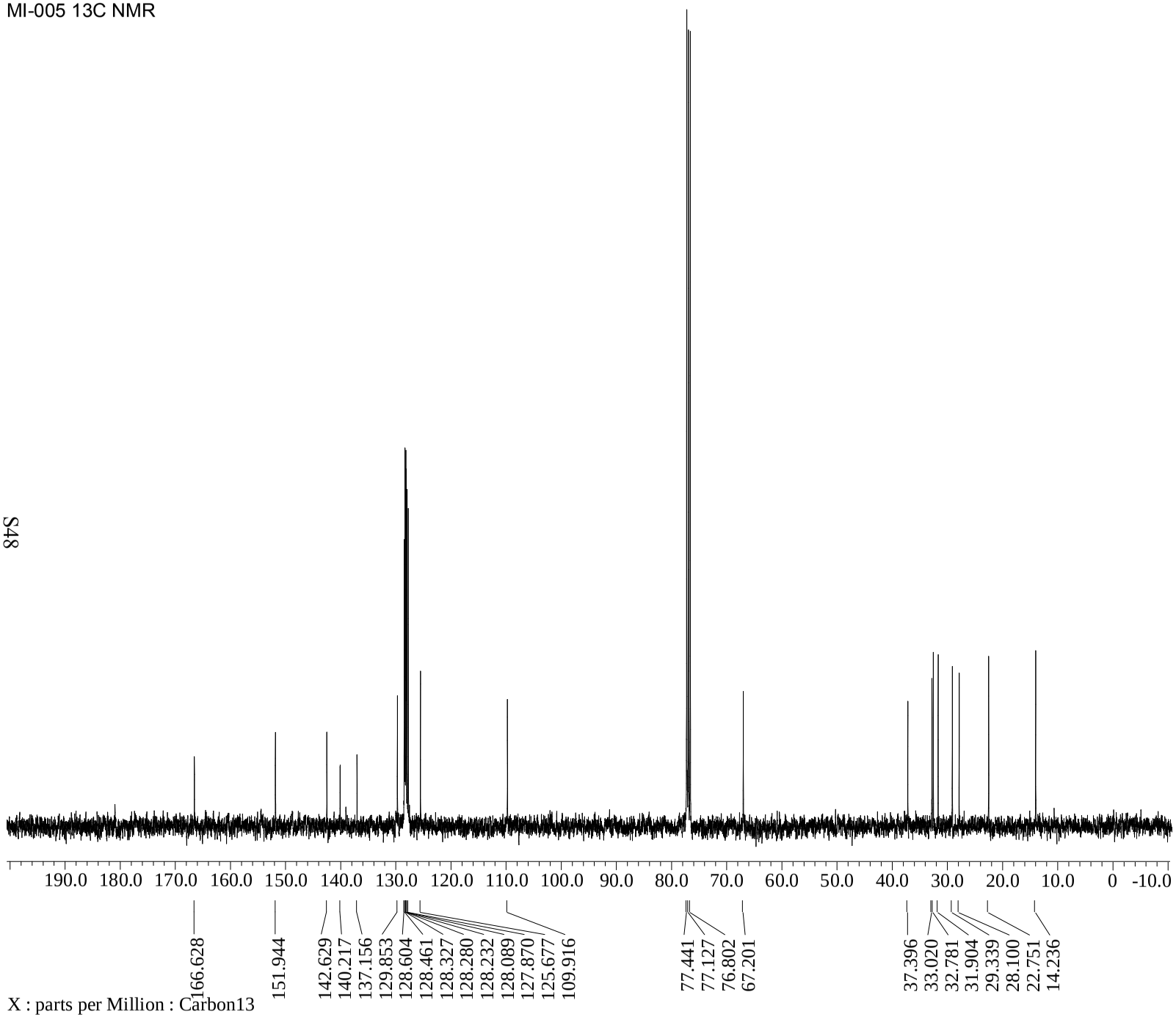
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MI005-F2-after auto column
Solvent           = CHLOROFORM-D
Creation_Time     = 25-APR-2012 16:45:34
Revision_Time     = 27-APR-2012 21:11:53
Current_Time      = 27-APR-2012 21:45:10

Author            = delta
Comment           = MI005-F2-after auto column
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 48
Temp_Get          = 19.5[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



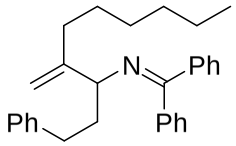
4d

MI-005 13C NMR



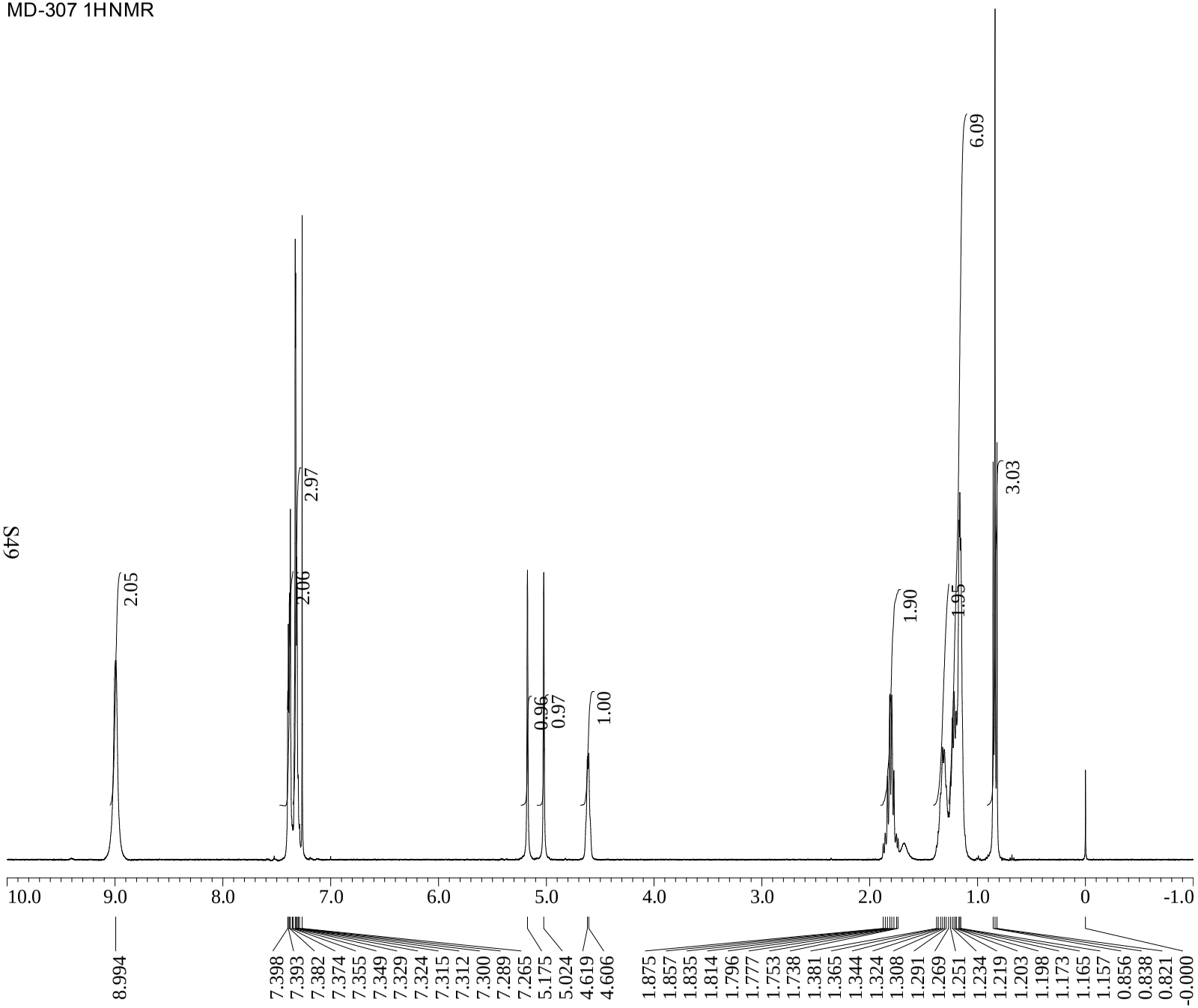
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MI005-F3
Solvent           = CHLOROFORM-D
Creation_Time     = 25-APR-2012 20:32:05
Revision_Time     = 27-APR-2012 21:20:25
Current_Time      = 27-APR-2012 21:38:30

Author            = delta
Comment           = MI005-F3_Carbon
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 95
Total_Scans       = 95

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



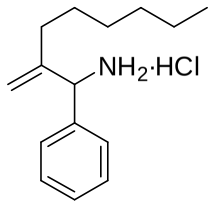
4d

MD-307 1HNMR



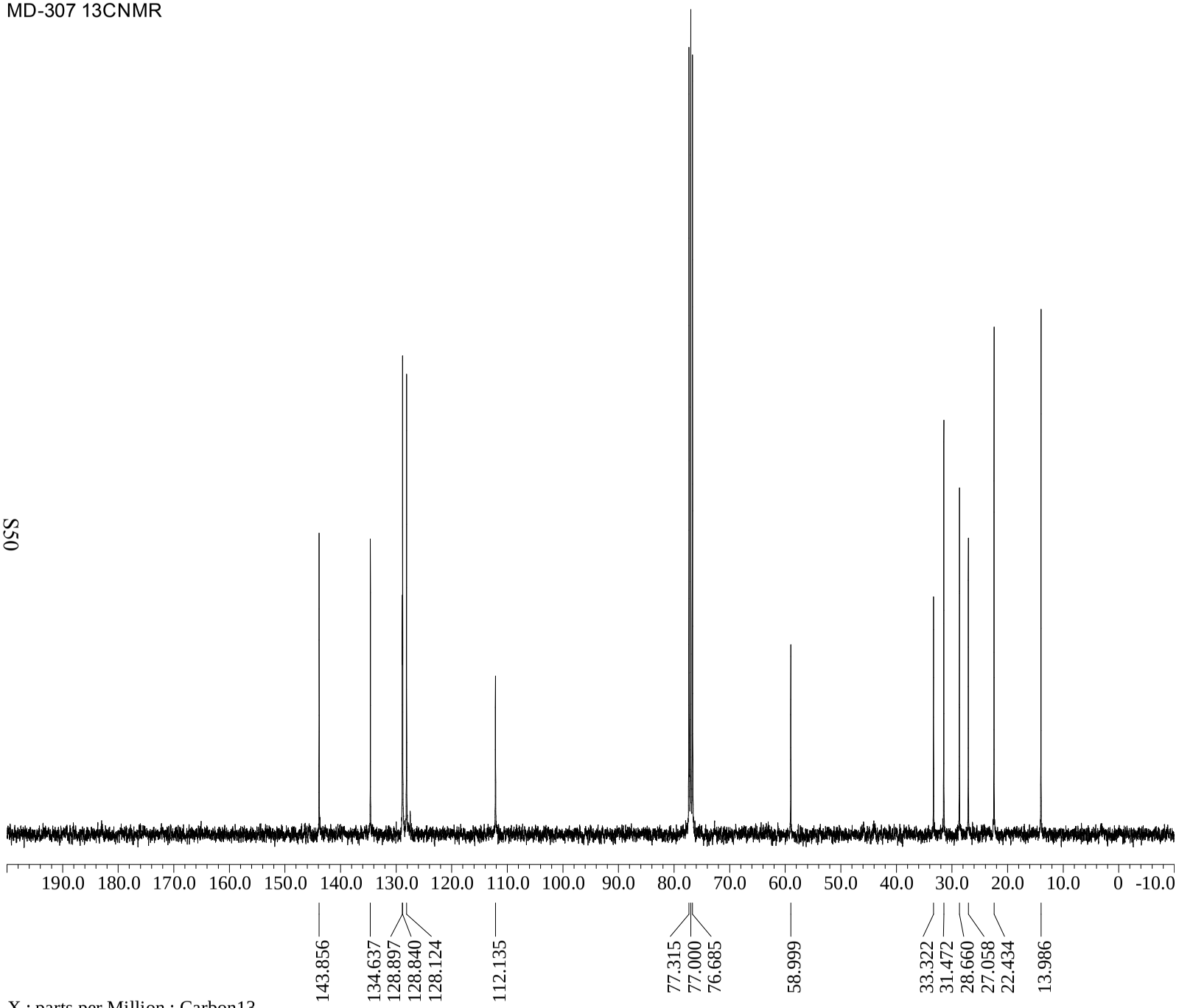
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-307
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2012 20:45:34
Revision_Time     =  5-MAR-2012 11:27:06
Current_Time      =  5-MAR-2012 11:27:37

Author            = delta
Data_Format       = 1D REAL
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 17.8[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



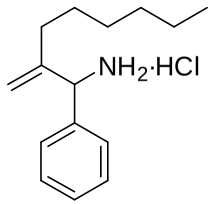
4f$_{NH2}$

MD-307 13CNMR



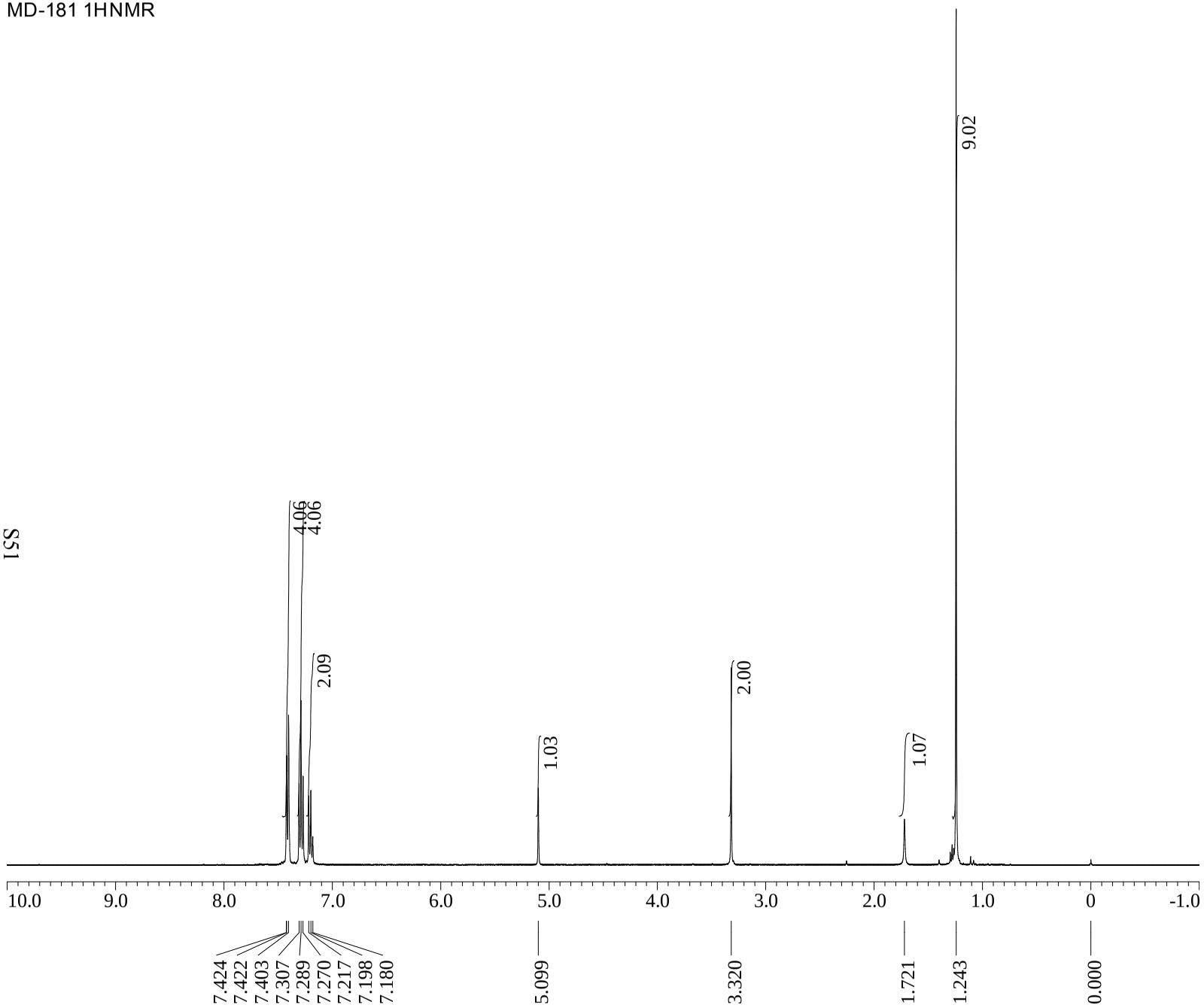
X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-307
Solvent           = CHLOROFORM-D
Creation_Time     =  1-MAR-2012 13:46:57
Revision_Time     =  5-MAR-2012 12:44:30
Current_Time      =  5-MAR-2012 12:44:59

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 207.0
Total_Scans       = 207.0

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 17.2[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



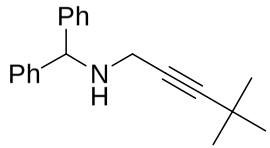
4f$_{NH2}$

MD-181 1HNMR

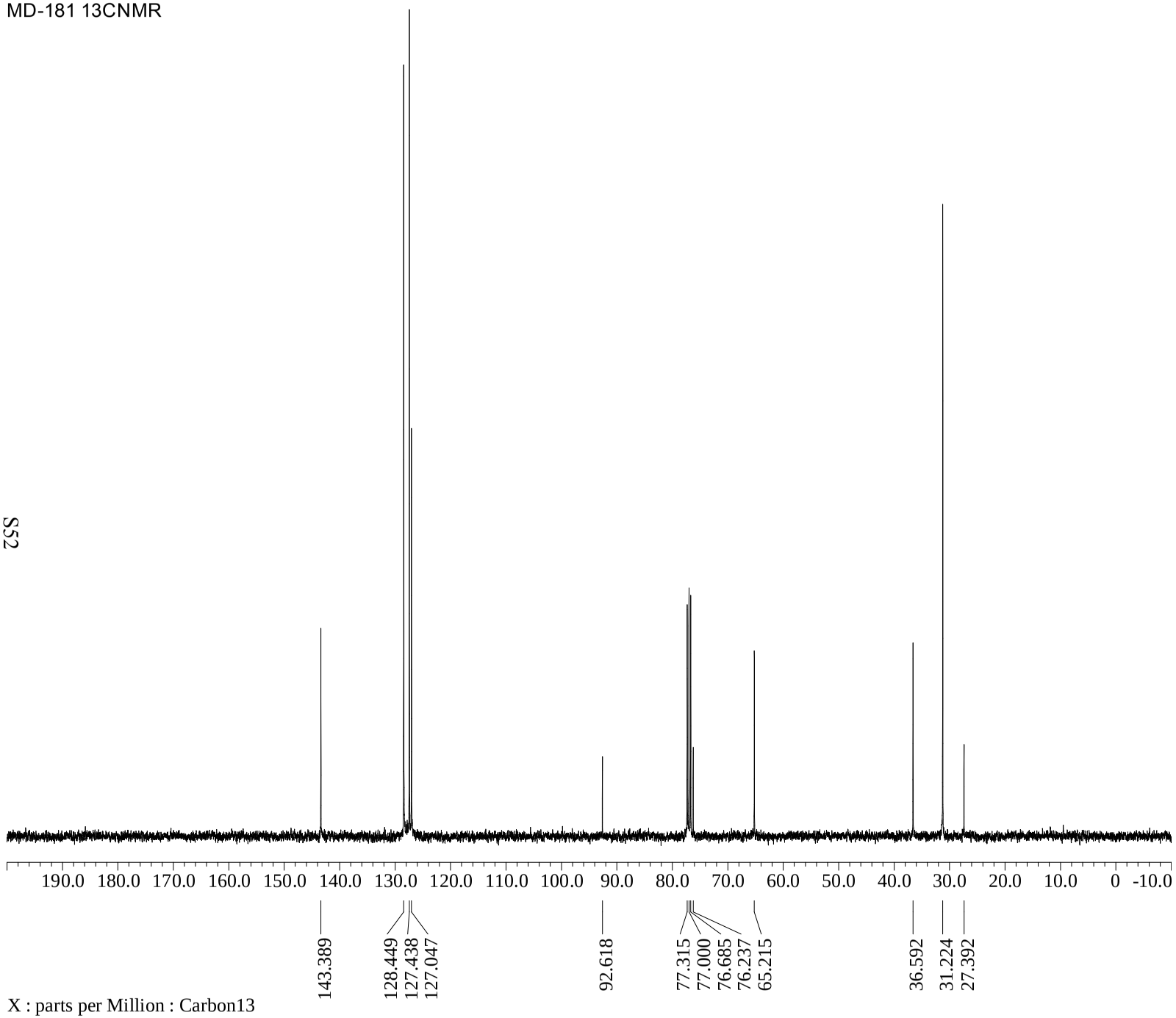


X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = MD-308
Solvent           = CHLOROFORM-D
Creation_Time     =  4-MAR-2012 00:29:42
Revision_Time     =  5-MAR-2012 12:08:23
Current_Time      =  5-MAR-2012 12:08:44

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 17.4[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

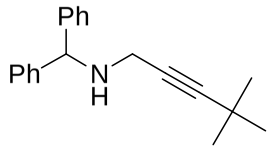


MD-181 13CNMR

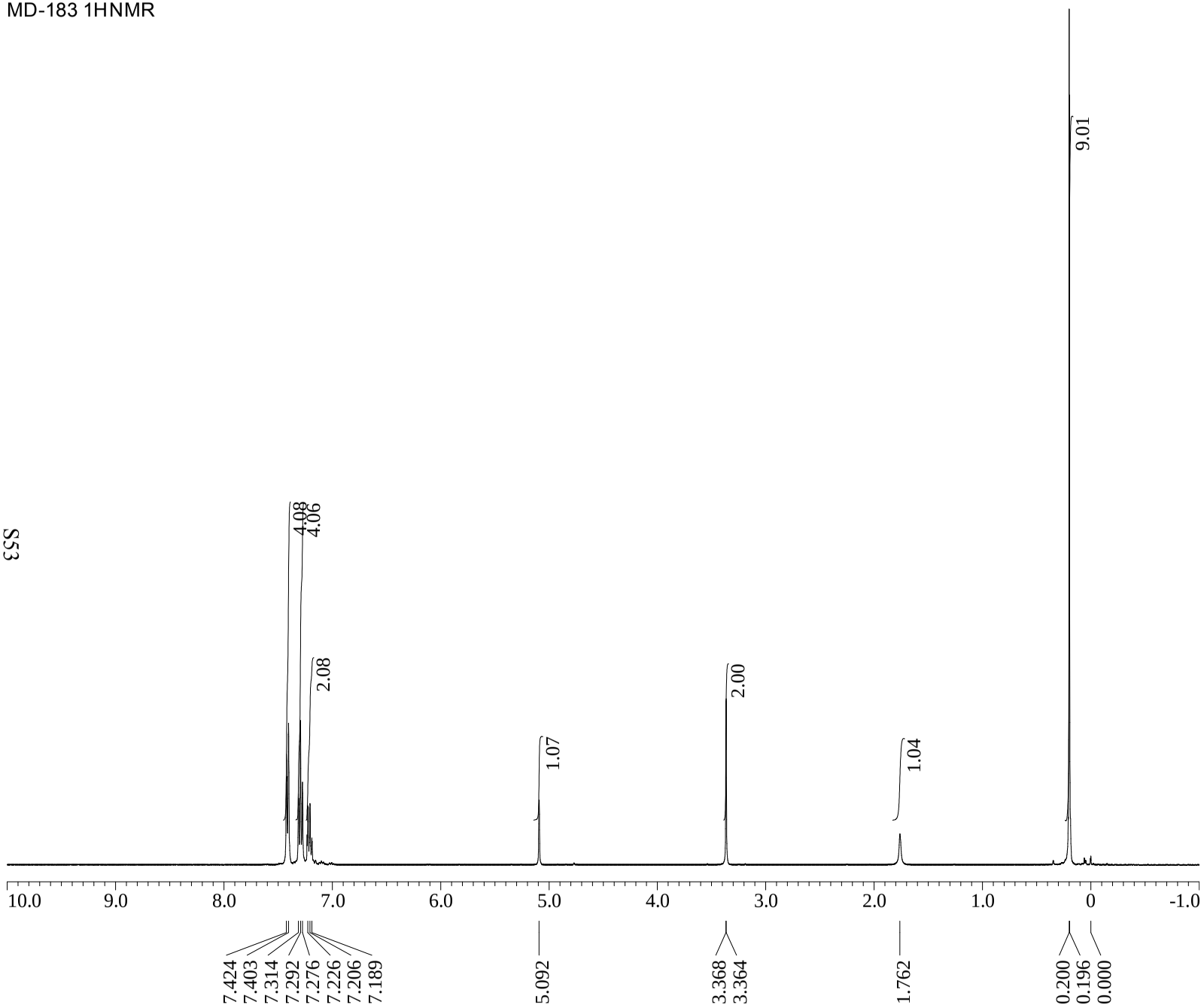


X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = MD-308
Solvent           = CHLOROFORM-D
Creation_Time     =  3-MAR-2012 23:45:32
Revision_Time     =  5-MAR-2012 12:48:50
Current_Time      =  5-MAR-2012 12:49:14

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 17.5[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

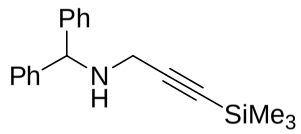


MD-183 1HNMR

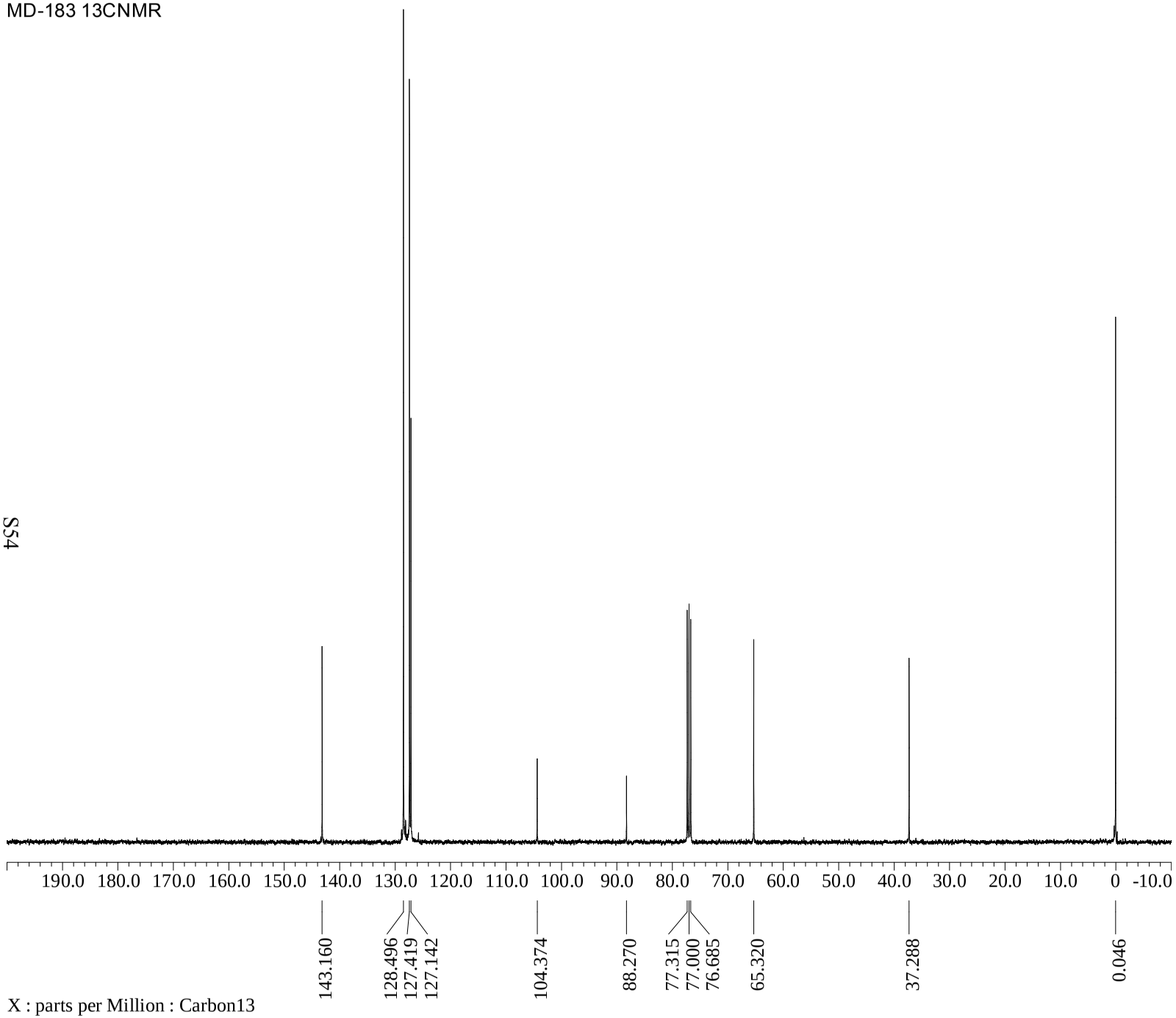


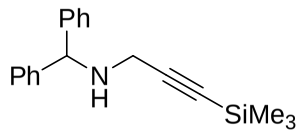
X : parts per Million : Proton

```
Filename          = C:\Documents and Settings\
Experiment        = proton.jxp
Sample_Id         = R=TMS
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2011 20:09:30
Revision_Time     =  5-MAR-2012 15:24:47
Current_Time      =  5-MAR-2012 15:25:13

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 20.6[dC]
X_90_Width        = 10[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



MD-183 13CNMR



X : parts per Million : Carbon13

```
Filename          = C:\Documents and Settings\
Experiment        = carbon.jxp
Sample_Id         = R=TMS
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2011 19:47:16
Revision_Time     =  5-MAR-2012 12:49:40
Current_Time      =  5-MAR-2012 12:50:01

Author            = delta
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 255.0
Total_Scans       = 255.0

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 22[dB]
Irr_Atn_Noe       = 22[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```