Electronic Supplementary Information

Structure-antitumor activity relationships of hybrid acetogenins focusing on connecting groups between heterocycles and the linker moiety

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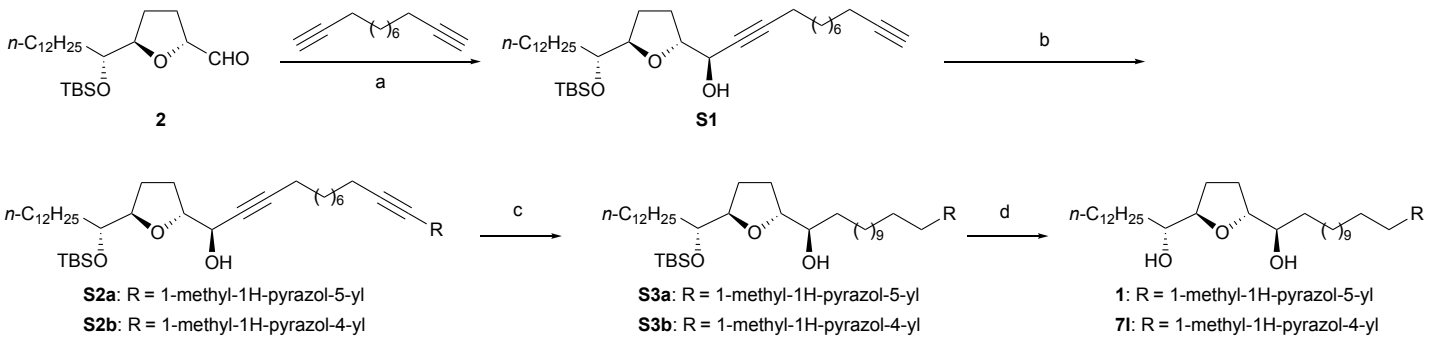


Scheme S1 Synthesis of **1** and **7l**; Reagents and conditions: (a) $Zn(OTf)_2$, Et_3N, (1*R*,2*S*)-*N*-methylephedrine, toluene, rt, 87%, dr = >97:3; (b) R–I, $Pd(PPh_3)_4$, CuI, Et_2NH, 50 °C, **S2a** (93%); **S2b** (86%); (c) 10% Pd–C, H_2 (3 atm), EtOAc, rt, **S3a** (82%); **S3b** (76%); (d) 48% HF aq, THF/CH_3CN, rt, **1** (98%); **7l** (93%).

(1*R*)-{(2*R*,5*R*)-5-[(1*R*)-1-(*tert*-Butyldimethylsilyloxy)tridecyl]tetrahydrofuran-2-yl}trideca-2,12-diyn-1-ol (S1) A flask was charged with $Zn(OTf)_2$ (124 mg, 0.341 mmol). Vacuum (12 mmHg) was applied and the flask was heated to 120 °C for 6 h. After the flask was cooled to rt, the vacuum was released. Then, (1*R*,2*S*)-(–)-*N*-methylephedrine (66.8 mg, 0.373 mmol), toluene (0.4 mL), and Et_3N (0.052 mL, 0.37 mmol) were added to the flask with stirring at rt. After 3 h at the same temperature, a solution of 1,11-dodecadiyne (105 mg, 0.649 mmol) in toluene (0.2 mL) was added to the mixture at the same temperature. After 15 min at the same temperature, **2** (67.0 mg, 0.162 mmol) in toluene (0.3 mL) was added to the reaction mixture and the whole mixture was stirred for 22 h at rt. The reaction was quenched with saturated NH_4Cl and the mixture was extracted with EtOAc. The combined organic layers were washed with brine prior to drying and solvent evaporation. Purification by flash column chromatography over silica gel with *n*-hexane/EtOAc (20:1) as eluent yielded **S1** (76.9 mg, 83%, dr = >97:3) as a colorless oil. $[\alpha]^{24}_D$ +12.1 (*c* 0.850 in $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 0.06 (s, 3H, SiC<u>H</u>$_3$), 0.08 (s, 3H), 0.88 (t, 3H, *J* = 6.7 Hz), 0.89 (s, 9H), 1.26−1.56 (m, 34H), 1.66−1.77 (m, 2H), 1.89−1.94 (m, 1H), 1.94 (t, 1H, *J* = 3.1 Hz), 2.02−2.08 (m, 1H), 2.18 (td, 2H, *J* = 6.7, 3.1 Hz), 2.19 (td, 2H, *J* = 7.0, 4.9 Hz), 2.46 (d, 1H, *J* = 2.5 Hz), 3.56−3.59 (m, 1H), 3.91 (q, 1H, *J* = 6.7 Hz), 4.00 (q, 1H, *J* = 6.7 Hz), 4.14−4.18 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: –4.7, –4.2, 14.1, 18.2, 18.3, 18.6, 22.6, 25.5, 25.9 (3C), 27.6, 28.2, 28.36, 28.42, 28.6, 28.7, 28.9, 29.3 (2C), 29.55 (2C), 29.58 (2C), 29.62, 29.8, 31.9, 32.8, 65.5, 68.1, 74.8, 78.0, 82.4, 82.6, 84.6, 86.2; IR (KBr) cm^{-1}: 3315; MS (FAB) *m/z*: 575 $[M+H]^+$; HRMS (FAB) *m/z*: calcd for $C_{36}H_{67}O_3Si$: 575.4859; found: 575.4866 $[M+H]^+$.

5-[(13*R*)-13-{(2*R*,5*R*)-5-[(1*R*)-1-(*tert*-Butyldimethylsilyloxy)tridecyl]tetrahydrofuran-2-yl}-13-hydroxytridec-1,11-diynyl]-1-methyl-1*H*-pyrazole (S2a)

$Pd(Ph_3P)_4$ (13.9 mg, 12.0 μmol) and CuI (I) (0.6 mg, 3.0 μmol) were added to a solution of 5-iodo-1-methyl-1*H*-pyrazole (25.0 mg, 0.120 mmol) in Et_2NH (0.4 mL) with stirring at rt. After stirring for 5 min at 50 °C, **S1** (34.5 mg, 0.0601 mmol) in Et_2NH (0.3 mL) was added to the mixture at the same temperature over 10 min. After stirring for 24 h at the same temperature, $Pd(Ph_3P)_4$ (7.0 mg, 6.0 μmol) and CuI (I) (0.3 mg, 1.5 μmol) were added to the reaction mixture. After stirring for 21 h at the same temperature, the mixture was diluted with Et_2O, and the catalyst was filtered off prior to solvent evaporation. Purification by flash column

chromatography over silica gel with *n*-hexane/EtOAc (15:1 to 7:1) as eluent yielded **S2a** (36.6 mg, 93%) as a colorless oil. $[\alpha]^{21}_{D}$ +10.2 (*c* 0.800 in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.06 (s, 3H), 0.08 (s, 3H), 0.88 (t, 3H, *J* = 6.7 Hz), 0.89 (s, 9H), 1.26–1.79 (m, 36H), 1.88–1.94 (m, 1H), 2.02–2.07 (m, 1H), 2.20 (td, 2H, *J* = 7.0, 1.8 Hz), 2.45 (t, 2H, *J* = 7.0 Hz), 2.52 (d, 1H, *J* = 3.7 Hz), 3.57 (td, 1H, *J* = 6.4, 3.7 Hz), 3.90–3.93 (m, 4H), 4.00 (dt, 1H, *J* = 7.3, 6.7 Hz), 4.15–4.17 (m, 1H), 6.31 (d, 1H, *J* = 1.8 Hz), 7.40 (d, 1H, *J* = 1.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ: –4.8, –4.3, 14.0, 18.1, 18.6, 19.4, 22.6, 25.4, 25.8 (3C), 27.5, 28.3, 28.4, 28.4, 28.66, 28.74, 28.85, 28.86, 29.2, 29.49, 29.53 (3C), 29.6, 29.7, 31.8, 32.8, 36.9, 65.6, 69.2, 74.9, 78.1, 82.4, 82.6, 86.2, 98.2, 109.2, 126.2, 138.3; IR (KBr) cm^{-1}: 3338; MS (FAB) *m/z*: 655 $[M+H]^+$; HRMS (FAB) *m/z*: calcd for $C_{40}H_{71}N_2O_3Si$: 655.5234; found: 655.5240 $[M+H]^+$.

4-[(13*R*)-13-[(2*R*,5*R*)-5-[(1*R*)-1-(*tert*-Butyldimethylsilyloxy)tridecyl]tetrahydrofuran-2-yl]-13-hydroxy-tridec-1,11-diynyl]-1-methylpyrazole (S2b) The procedure was the same as that used for the preparation of **S2a** by use of 4-iodo-1-methyl-1*H*-pyrazole instead of 5-iodo-1-methyl-1*H*-pyrazole, giving **S2b** (Yield: 86%) as an yellow oil. $[\alpha]^{24}_{D}$ +8.7 (*c* 1.01 in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.06 (s, 3H), 0.08 (s, 3H), 0.88 (t, 3H, *J* = 6.7 Hz), 0.89 (s, 9H), 1.21–1.59 (m, 34H), 1.64–1.81 (m, 2H), 1.87–1.95 (m, 1H), 2.00–2.07 (m, 1H), 2.19 (td, 2H, *J* = 7.0, 1.8 Hz), 2.35 (t, 2H, *J* = 7.0 Hz), 2.55 (br s, 1H), 3.55–3.60 (m, 1H), 3.86 (s, 3H), 3.91 (td, 1H, *J* = 7.9, 6.4 Hz), 4.00 (q, 1H, *J* = 6.7 Hz), 4.15–4.18 (m, 1H), 7.41 (s, 1H), 7.51 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: –4.6, –4.2, 14.1, 18.2, 18.7, 19.4, 22.7, 25.5, 25.9 (3C), 27.6, 28.3, 28.5, 28.7, 28.8, 28.9, 28.97, 28.98, 29.3, 29.58, 29.62 (3C), 29.7, 29.8, 31.9, 32.9, 39.0, 65.6, 71.2, 74.9, 78.1, 82.5, 82.6, 86.3, 90.6, 103.9, 132.1, 141.8; IR (KBr) cm^{-1}: 3429, 2360; MS (FAB) *m/z*: 655 $[M+H]^+$; HRMS (FAB) *m/z*: calcd for $C_{40}H_{71}N_2O_3Si$: 655.5234; found: 655.5256 $[M+H]^+$.

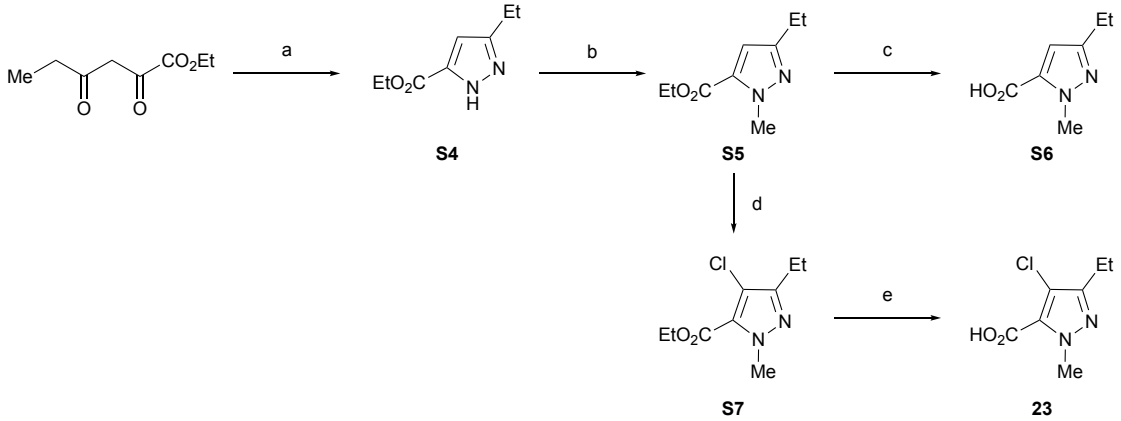
5-[(13*R*)-13-{(2*R*,5*R*)-5-[(1*R*)-1-(*tert*-Butyldimethylsilyloxy)tridecyl]tetrahydrofuran-2-yl}-13-hydroxy-tridecyl]-1-methyl-1*H*-pyrazole (S3a) The procedure was the same as that used for the preparation of **20** by use of S2a instead of **19**, giving **S3a** (Yield: 82%) as a colorless oil. $[\alpha]^{25}_{D}$ +8.0 (*c* 0.74 in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.06 (s, 3H), 0.08 (s, 3H), 0.87–0.89 (m, 12H), 1.26–1.41 (m, 42H), 1.58–1.68 (m, 4H), 1.88–1.95 (m, 2H), 2.42 (br s, 1H), 2.57 (t, 2H, *J* = 7.9 Hz), 3.35–3.39 (m, 1H), 3.55 (td, 1H, *J* = 6.1, 3.7 Hz), 3.75–3.78 (m, 4H), 3.86 (dt, 1H, *J* = 7.9, 6.1 Hz), 6.00 (d, 1H, *J* = 1.8 Hz), 7.37 (d, 1H, *J* = 1.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ: –4.6, –4.2, 14.1, 18.3, 22.7, 25.4, 25.5, 25.6, 25.9 (3C), 28.4 (2C), 28.5, 29.2, 29.3 (2C), 29.5, 29.56 (4C), 29.61 (3C), 29.64 (2C), 29.7, 29.8, 31.9, 33.2, 33.5, 36.0, 74.1, 75.2, 82.3, 82.4, 104.0, 137.9, 143.0; IR (KBr) cm^{-1}: 3464; MS (FAB) *m/z*: 663 $[M+H]^+$; HRMS (FAB) *m/z*: calcd for $C_{40}H_{79}N_2O_3Si$: 663.5860; found: 663.5878 $[M+H]^+$.

4-[(13*R*)-13-{(2*R*,5*R*)-5-[(1*R*)-1-(*tert*-Butyldimethylsilyloxy)tridecyl]tetrahydrofuran-2-yl}-13-hydroxy-tridecyl]-1-methyl-1*H*-pyrazole (S3b) The procedure was the same as that used for the preparation of **20** by use of S2b instead of **19**, giving **S3b** (Yield: 76%) as a colorless oil. $[\alpha]^{24}_{D}$ +9.5 (*c* 0.56 in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.06 (s, 3H), 0.08 (s, 3H), 0.87–0.89 (m, 12H), 1.26–1.68 (m, 46H), 1.88–1.97 (m, 2H), 2.41 (br s, 1H), 2.42 (t, 2H, *J* = 7.6 Hz), 3.35–3.39 (m, 1H), 3.55 (td, 1H, *J* = 6.1, 3.7 Hz), 3.76 (q, 1H, *J* = 6.7

Hz), 3.83–3.88 (m, 4H), 7.12 (s, 1H), 7.29 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: –4.6, –4.2, 14.1, 18.2, 22.6, 24.1, 25.4, 25.6, 25.9 (3C), 28.4, 28.5, 29.2, 29.3, 29.4, 29.57 (4C), 29.61 (4C), 29.64 (2C), 29.7, 29.8, 31.0, 31.9, 33.1, 33.4, 38.7, 74.1, 75.2, 82.2, 82.4, 122.1, 128.0, 138.6; IR (KBr) cm^{-1}: 3431; MS (FAB) *m*/*z*: 663 [*M*+H]$^+$; HRMS (FAB) *m*/*z*: calcd for $C_{40}H_{79}N_2O_3Si$: 663.5860; found: 663.5857 [*M*+H]$^+$.

5-[(13*R*)-13-Hydroxy-13-{(2*R*,5*R*)-5-[(1*R*)-1-hydroxytridecyl]tetrahydrofuran-2-yl}tridecyl]-1-methyl-1*H*-pyrazole (1) The procedure was the same as that used for the preparation of **7d** by use of **S3a** instead of **6d**, giving **1** (Yield: 98%) as a white waxy solid. M.p. 68.3–69.3 °C; $[\alpha]^{24}_D$ +11.4 (*c* 0.880 in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.88 (t, 3H, *J* = 7.0 Hz), 1.26–1.41 (m, 42H), 1.60–1.72 (m, 4H), 1.95–2.01 (m, 2H), 2.57 (t, 2H, *J* = 7.6 Hz), 3.39–3.42 (m, 2H), 3.78 (s, 3H), 3.78–3.82 (m, 2H), 6.01 (d, 1H, *J* = 1.5 Hz), 7.37 (d, 1H, *J* = 1.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.1, 22.7, 25.5, 25.6, 28.4, 28.7, 29.2, 29.3 (2C), 29.5, 29.56 (4C), 29.59 (2C), 29.61 (2C), 29.62 (2C), 29.64 (2C), 29.7 (2C), 31.9, 33.4, 36.0, 74.0 (2C), 82.6 (2C), 104.0, 137.9, 143.0; IR (KBr) cm^{-1}: 3448, 3354; MS (FAB) *m*/*z*: 549 [*M*+H]$^+$; HRMS (FAB) *m*/*z*: calcd for $C_{34}H_{65}N_2O_3$: 549.4995; found: 549.5009 [*M*+H]$^+$.

4-[(13*R*)-13-Hydroxy-13-{(2*R*,5*R*)-5-[(1*R*)-1-hydroxytridecyl]tetrahydrofuran-2-yl}tridecyl]-1-methyl-1*H*-pyrazole (7l) The procedure was the same as that used for the preparation of **7d** by use of **S3b** instead of **6d**, giving **7l** (Yield: 93%) as a white waxy solid. M.p. 75.5–76.4 °C; $[\alpha]^{24}_D$ +10.7 (*c* 0.320 in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.88 (t, 3H, *J* = 6.7 Hz), 1.26–1.72 (m, 46H), 1.98–1.99 (m, 2H), 2.36 (br s, 2H), 2.43 (t, 2H, *J* = 7.6 Hz), 3.40 (q, 2H, *J* = 6.1 Hz), 3.80 (dt, 2H, *J* = 7.9, 6.1 Hz), 3.85 (s, 3H), 7.12 (s, 1H), 7.29 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.1, 22.7, 24.1, 25.6 (2C), 28.7 (2C), 29.2, 29.3, 29.4, 29.57 (4C), 29.61 (4C), 29.65 (2C), 29.7 (2C), 31.0, 31.9, 33.4 (2C), 38.7, 74.0 (2C), 82.6 (2C), 122.1, 128.0, 138.7; IR (KBr) cm^{-1}: 3465, 3438; MS (FAB) *m*/*z*: 549 [*M*+H]$^+$; HRMS (FAB) *m*/*z*: calcd for $C_{34}H_{65}N_2O_3$: 549.4995; found: 549.4993 [*M*+H]$^+$.



Scheme S2 Synthesis of substituted pyrazoles **S6** and **23**; Reagents and conditions: (a) NH_2NH_2 H_2O, AcOH, 100 °C, 90%; (b) Me_2SO_4, THF, 65 °C, 57%; (c) 2N NaOH, rt, 85%; (d) SO_2Cl_2, CH_2Cl_2, rt, quant.; (e) 2N NaOH, rt, 96%.

Ethyl 3-ethyl-1*H*-pyrazole-5-carboxylate (S4) Hydrazine monohydrate (49.0 μL, 1.00 mmol) was added to a solution of ethyl 4-oxohexanoaate (17.2 mg, 0.100 mmol) in AcOH (1.0 mL) with stirring at rt over 10 min. After stirring for 3 h at reflux, water was added to the reaction mixture and the mixture was extracted with $CHCl_3$. The combined organic layer was washed with saturated $NaHCO_3$ prior to drying and solvent evaporation. Purification by column chromatography over silica gel with *n*-hexane/EtOAc (5:1) as eluent yielded **S4** (15.2 mg, 90%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26 (t, 3H, J = 7.3 Hz), 1.34 (t, 3H, J = 7.3 Hz), 2.73 (q, 2H, J = 7.3 Hz), 4.36 (q, 2H, J = 7.3 Hz), 6.60 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 13.2, 14.2, 19.4, 60.8, 105.7, 141.4, 149.2, 162.0; IR (NaCl) cm^{-1}: 1726; MS (EI) *m*/*z*: 168 (61.8) [*M*]$^+$, 153 (12.6), 122 (100.0), 107 (15.5), 94 (19.2); HRMS (EI) *m*/*z*: calcd for $C_8H_{12}N_2O_2$: 168.0899; found: 168.0901 [*M*]$^+$.

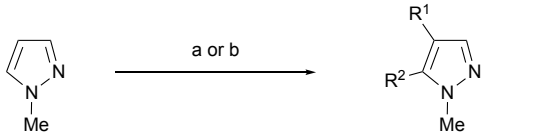
Ethyl 3-ethyl-1-methyl-1*H*-pyrazole-5-carboxylate (S5) Dimethyl sulfate (3.37 mL, 34.7 mmol) was added to a solution of **S4** (583 mg, 3.47 mmol) in THF (35 mL) with stirring at rt. After stirring for 3 h at 65 °C, water was added to the reaction mixture and the mixture was extracted with Et_2O. The combined organic layer was washed with brine prior to drying and solvent evaporation. Purification by column chromatography over silica gel with *n*-hexane/EtOAc (15:1 to 10:1) as eluent yielded **S5** (358 mg, 57%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.24 (t, 3H, J = 7.8 Hz), 1.37 (t, 3H, J = 7.3 Hz), 2.64 (q, 2H, J = 7.8 Hz), 4.11 (s, 3H), 4.33 (q, 2H, J = 7.3 Hz), 6.63 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 13.8, 14.2, 21.2, 39.1, 60.8, 108.9, 132.9, 153.0, 160.0; IR (NaCl) cm^{-1}: 1724; MS (EI) *m*/*z*: 182 (100) [*M*]$^+$, 167 (40.9), 139 (36.6), 109 (15.8); HRMS (EI) *m*/*z*: calcd for $C_9H_{14}N_2O_2$: 182.1055; found: 182.1072 [*M*]$^+$.

3-Ethyl-1-methyl-1*H*-pyrazole-5-carboxylic acid (S6) A solution of **S5** (93.2 mg, 0.511 mmol) in 2N NaOH (5.1 mL) was stirred for 1 h at 65° C. 3N HCl was added to the reaction mixture and the mixture was extracted with Et_2O. The combined organic layer was washed with brine prior to drying and solvent evaporation. The residual solid was recrystallized from MeOH to give **S6** (66.9 mg, 85%) as colorless needles. M.p. 152.0–153.0 °C (MeOH): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26 (t, 3H, J = 7.3 Hz), 2.69 (q, 2H, J = 7.3 Hz), 4.17 (s, 3H), 6.77 (s, 1H), 9.76 (br s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.7, 20.9, 39.1, 110.2, 132.6, 153.3, 163.5; IR (NaCl) cm^{-1}: 1705; MS (EI) *m*/*z*: 154 (97.8) [*M*]$^+$, 139 (100), 109 (10.0); HRMS (EI) *m*/*z*: calcd for $C_7H_{10}N_2O_2$: 154.0742; found: 154.0756 [*M*]$^+$.

Ethyl 4-chloro-3-ethyl-1-methyl-1*H*-pyrazole-5-carboxylate (S7) Sulfuryl chloride (25.0 μL, 0.311 mmol) was added to a solution of **S5** (19.2 mg, 0.105 mmol) in CH_2Cl_2 (1.0 mL) with stirring at rt over 5 min. After stirring for 30 min at the same temperature, water was added to the reaction mixture and the mixture was extracted with Et_2O. The combined organic layer was washed with brine prior to drying and solvent evaporation. Purification by column chromatography over silica gel with *n*-hexane/EtOAc (10:1) as eluent yielded **S7** (22.8 mg, quant.) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.24 (t, 3H, J = 7.8 Hz), 1.41 (t, 3H, J = 7.3 Hz), 2.64 (q, 2H, J = 7.8 Hz), 4.09 (s, 3H), 4.39 (q, 2H, J = 7.3 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 12.8, 14.1, 19.2, 40.5, 61.3, 113.1, 129.0, 150.4, 159.2; IR (NaCl) cm^{-1}: 1719; MS (EI) *m*/*z*: 218 (30.3)

[*M*+2]$^+$, 216 (100.0) [*M*]$^+$, 201 (40.9), 187 (37.5), 173 (49.8); HRMS (EI) *m/z*: calcd for $C_9H_{13}ClN_2O_2$: 216.0666; found: 216.0687 [*M*]$^+$.

4-Chloro-3-ethyl-1-methyl-1*H*-pyrazole-5-carboxylic acid (23) The procedure was the same as that used for the preparation of **S6** by use of **S7** instead of **S5**, giving **23** (Yield: 96%) as colorless needles. M.p. 162.5–163.4 °C (MeOH): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26 (t, 3H, *J* = 7.3 Hz), 2.68 (q, 2H, *J* = 7.3 Hz), 4.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 12.7, 19.1, 40.9, 114.8, 128.2, 150.7, 162.9; IR (NaCl) cm^{-1}: 1707; MS (EI) *m/z*: 190 (24.1) [*M*+2]$^+$, 188 (74.9) [*M*]$^+$, 173 (100.0); HRMS (EI) *m/z*: calcd for $C_7H_9ClN_2O_2$: 188.0353; found: 188.0359 [*M*]$^+$.

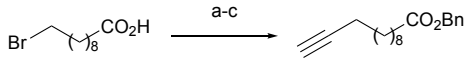


S8a: R^1 = H, R^2= SO_2Cl
S8b: R^1 = SO_2Cl, R^2= H

Scheme S3 Synthesis of pyrazole-sulfonyl chloride **S8a**-**S8b**; Reagents and conditions: (a) *t*-BuLi, –78 °C then SO_2Cl_2, –78 °C, 24%; (b) $ClSO_3H$, rt to 120 °C, 25%.

1-Methyl-1*H*-pyrazole-5-sulfonyl chloride (S8a) *t*-BuLi (1.6 M in *n*-pentane, 3.94 mL, 6.31 mmol) was added to a solution of 1-methyl-1*H*-pyrazole (246 mg, 3.00 mmol) in THF (10 mL) with stirring at –78 °C over 10 min. After stirring for 1 h at the same temperature, sulfuryl chloride (0.362 mL, 4.50 mmol) was added to the reaction mixture at the same temperature. After stirring for 1.5 h at the same temperature, water was added to the reaction mixture at 0 °C and the mixture was extracted with Et_2O. The combined organic layer was washed with brine prior to drying and solvent evaporation. Purification by flash column chromatography over silica gel with *n*-hexane/EtOAc (20:1 to 10:1) as eluent yielded **S8a** (127 mg, 24%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.23 (s, 3H), 7.02 (d, 1H, *J* = 1.8 Hz), 7.57 (d, 1H, *J* = 1.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 39.0, 112.1, 137.9, 141.1; IR (NaCl) cm^{-1}: 1386, 1200; MS (EI) *m/z*: 182 (15.7) [*M*+2]$^+$, 180 (44.8) [*M*]$^+$, 145 (100.0); HRMS (EI) *m/z*: Calcd for $C_4H_5ClN_2O_2S$: 179.9760; Found: 179.9787 [*M*]$^+$.

1-Methyl-1*H*-pyrazole-4-sulfonyl chloride (S8b) A mixture of 1-methyl-1*H*-pyrazole (246 mg, 3.00 mmol) and chlorosulfuric acid (1.60 mL, 24.0 mmol) was stirred at 100 °C for 3 h. After cooling to rt, the mixture was poured into ice-water. The residual solid was collected and washed with water and CH_3CN to give **S8b** (135 mg, 25%) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.01 (s, 3H), 7.97 (s, 1H), 8.03 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 40.0, 126.6, 132.5, 138.5; IR (NaCl) cm^{-1}: 1395, 1370; MS (EI) *m/z*: 182 (4.3) [*M*+2]$^+$, 180 (14.5) [*M*]$^+$, 145 (100.0); HRMS (EI) *m/z*: Calcd for $C_4H_5ClN_2O_2S$: 179.9760; Found: 179.9752 [*M*]$^+$.



17

Scheme S4 Synthesis of benzyl ester **17**; Reagents and conditions: (a) NaI, acetone, reflux; (b) lithium acetylide ethylenediamine complex, HMPA 0 °C ; (c) BnBr, $CsCO_3$, DMF, rt, 51% over 3 steps.

Benzyl dodec-11-ynoate (17) A suspension of 10-bromododecanoic acid (2.00 g, 7.96 mmol) and sodium iodide (2.38 g, 15.9 mmol) in acetone (20 mL) was stirred for 18 h at reflux. After cooling, the precipitate was filtered off and the filtrate was concentrated under reduced pressure. Water was added to the residual mixture and the mixture was extracted with Et_2O. The combined organic layers were washed with saturated $Na_2S_2O_3$ prior to drying and solvent evaporation. The residual solid was recrystallized from Et_2O and *n*-hexane to give a crude of 10-iododododecanoic acid as a white solid. A solution of lithium acetylide ethylenediamine complex (1.38 g, 15.0 mmol) in hexamethylphosphoric triamide (HMPA, 18 mL) was stirred for 40 min at rt. A solution of crude of 10-iododododecanoic acid in HMPA (2 mL) was added to the mixture at 0 °C over 30 min. After stirred for 70 min at the same temperature, water and 3N HCl were added to the reaction mixture and the mixture was extracted with Et_2O. The combined organic layers were washed with brine prior to drying and solvent evaporation. The mixture was filtered through a short pad of silica gel with *n*-hexane/EtOAc (8:1 to 3:1) as eluent to give a crude of dodec-11-ynoic acid as a white solid. Cesium carbonate (740 mg, 2.27 mmol) was added to a suspension of a crude of dodec-11-ynoic acid in DMF (18 mL) with stirring at rt. Benzyl bromide (0.540 mL, 4.54 mmol) was added to the mixture at the same temperature. After stirring for 3 h at the same temperature, water was added to the reaction mixture and the mixture was extracted with Et_2O. The combined organic layers were dried prior to solvent evaporation. Purification by column chromatography over silica gel with *n*-hexane/EtOAc (50:1 to 30:1) as eluent yielded **17** (1.17 g, 51% over 3 steps) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26–1.42 (m, 10H), 1.47–1.54 (m, 2H), 1.61–1.66 (m, 2H), 1.93 (t, 1H, J = 2.4 Hz), 2.16 (td, 2H, J = 7.3, 2.4 Hz), 2.34 (t, 2H, J = 7.3 Hz), 5.11 (s, 2H), 7.29–7.37 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 18.3, 24.8, 28.4, 28.6, 28.9, 29.0, 29.1, 29.2, 34.2, 66.0, 68.0, 84.6, 128.06, 128.07 (2C), 128.4 (2C), 136.1, 173.5; IR (NaCl) cm^{-1}: 3306, 1736; MS (EI) *m/z*: 286 (2.6) $[M]^+$, 195 (2.0), 180 (3.9), 108 (33.2), 91 (100); HRMS (EI) *m/z*: Calcd for $C_{19}H_{26}O_2$: 286.1933; Found: 286.1936 $[M+H]^+$.

Table S1. Logarithm of the $1/GI_{50}$ values of compounds against 39 human cancer cell lines

cell line	solamin	**1**	**7a**	**7e**	**7g**	**7h**	**7i**	**7j**	**7k**	**7b**	**7c**	**23**	**7l**	**7d**	**7f**
HBC-4	4.00	4.00	4.00	5.24	5.00	5.74	4.00	4.29	4.00	4.43	4.80	4.00	4.00	4.00	4.00
BSY-1	4.00	4.49	8.00	5.70	6.30	6.47	4.02	6.09	4.47	5.80	8.00	4.00	4.00	4.00	4.43
HBC-5	4.00	4.16	4.77	5.43	5.51	5.68	4.00	4.59	4.00	4.72	4.85	4.00	4.00	4.00	4.43
MCF-7	4.15	4.57	5.49	5.62	5.30	5.77	4.00	4.64	4.66	5.68	5.42	4.18	4.00	4.77	4.96
MDA-MB-231	4.00	4.49	5.07	5.11	5.05	5.66	4.00	4.00	4.21	4.41	4.82	4.00	4.00	4.00	4.00
U251	4.00	4.00	4.00	5.29	5.00	5.82	4.00	4.16	4.00	4.57	5.26	4.00	4.00	4.00	4.00
SF-268	4.00	4.00	4.00	4.89	4.82	5.44	4.00	4.00	4.00	4.15	5.06	4.00	4.00	4.00	4.00
SF-295	4.40	4.62	5.00	6.15	5.70	5.92	4.24	4.51	4.54	5.44	4.96	4.04	4.62	4.62	5.85
SF-539	4.00	4.43	5.43	4.89	4.92	5.80	4.00	4.00	4.00	4.60	5.57	4.00	4.43	4.00	4.00
SNB-75	4.00	4.00	4.30	4.92	4.80	5.70	4.00	4.00	4.00	4.35	4.48	4.33	4.64	4.00	4.00
SNB-78	4.00	4.00	4.00	5.96	4.92	5.68	4.00	4.35	4.00	4.28	4.52	4.00	4.09	4.00	4.00
HCC2998	4.00	4.10	4.00	5.35	4.92	5.74	4.00	4.33	4.00	4.24	4.59	4.00	4.00	4.00	4.00
KM-12	4.00	4.15	4.00	5.17	5.38	5.85	4.00	4.70	4.00	4.43	4.92	4.00	4.00	4.00	4.00
HT-29	4.00	4.12	4.00	5.47	5.09	5.52	4.00	4.49	4.06	4.27	4.77	4.00	4.00	4.00	4.00
HCT-15	4.00	4.00	4.00	5.24	5.00	5.70	4.00	4.32	4.00	4.17	4.24	4.00	4.00	4.00	4.00
HCT-116	4.00	5.04	8.00	5.77	6.10	5.96	4.19	5.40	6.07	8.00	7.74	4.00	4.15	5.89	5.33
NCI-H23	4.14	6.04	7.28	6.60	6.62	6.19	4.57	4.96	5.42	5.89	5.85	4.00	4.89	4.85	4.89
NCI-H226	4.00	4.28	4.39	5.35	5.01	5.48	4.12	4.55	4.68	4.66	4.74	4.14	4.25	4.36	4.46
NCI-H522	4.24	5.42	5.85	6.23	5.64	5.89	4.54	5.20	5.64	5.72	5.43	4.27	4.92	4.62	4.92
NCI-H460	4.00	4.00	5.09	5.74	5.26	5.77	4.00	4.46	4.00	4.74	5.68	4.00	4.00	4.00	4.00
A549	4.00	4.51	4.89	5.27	5.07	5.06	4.00	4.25	4.05	5.00	5.24	4.00	4.00	4.00	4.00
DMS273	4.00	4.31	5.24	5.64	5.48	5.80	4.00	4.42	4.30	5.08	5.54	4.00	4.00	4.00	4.10
DMS114	5.37	6.26	8.00	7.07	7.46	6.89	5.48	6.39	6.70	8.00	8.00	4.00	5.68	7.08	7.22
LOX-IMVI	4.54	4.00	8.00	5.82	5.85	5.92	4.00	4.89	4.68	5.19	5.52	4.00	5.02	4.00	4.00
OVCAR-3	4.00	4.00	4.11	5.28	5.00	5.82	4.00	4.00	4.00	4.85	5.33	4.00	4.00	4.00	4.00
OVCAR-4	4.00	4.43	4.64	5.23	5.00	5.80	4.00	4.00	4.00	4.66	5.55	4.00	4.00	4.00	4.01
OVCAR-5	4.00	4.00	4.00	5.41	4.85	5.51	4.00	4.00	4.00	4.00	4.34	4.00	4.00	4.00	4.00
OVCAR-8	4.00	4.00	4.51	5.29	5.22	5.80	4.00	4.00	4.00	4.70	5.36	4.00	4.00	4.00	4.00
SK-OV-3	4.00	4.11	4.00	5.04	4.80	5.68	4.00	4.00	4.00	4.28	4.46	4.00	4.00	4.00	4.00
RXF-631L	4.00	4.00	4.00	5.05	4.96	5.62	4.00	4.00	4.00	4.32	5.46	4.00	4.00	4.00	4.00
ACHN	4.00	4.06	4.62	5.41	4.96	5.51	4.00	4.24	4.00	4.77	5.21	4.00	4.00	4.00	4.00
St-4	4.00	4.19	4.74	5.22	5.17	5.74	4.00	4.52	4.02	4.68	4.82	4.00	4.00	4.00	4.00
MKN1	4.00	4.00	4.36	5.02	5.26	5.85	4.00	4.36	4.00	4.96	5.16	4.00	4.00	4.00	4.00
MKN-B	4.89	5.21	8.00	7.01	7.29	6.51	4.68	6.21	5.21	7.36	6.02	4.00	5.27	5.44	7.02
MKN-A	4.85	5.36	8.00	6.80	6.52	6.30	4.74	5.60	4.00	4.85	4.00	4.00	4.72	8.00	6.51
MKN45	4.00	4.00	5.13	5.00	4.96	5.74	4.00	4.00	6.10	4.55	4.00	4.00	4.00	4.00	4.00
MKN74	4.00	4.64	5.41	5.89	5.77	5.89	4.00	4.51	4.46	4.77	4.00	4.00	4.00	7.59	5.01
DU-145	4.00	4.00	4.00	4.77	4.89	5.36	4.00	4.00	4.00	4.00	4.92	4.00	4.00	4.00	4.00
PC-3	4.00	4.00	4.74	5.26	5.66	5.64	4.00	4.55	4.49	4.92	4.96	4.00	4.00	4.00	4.00

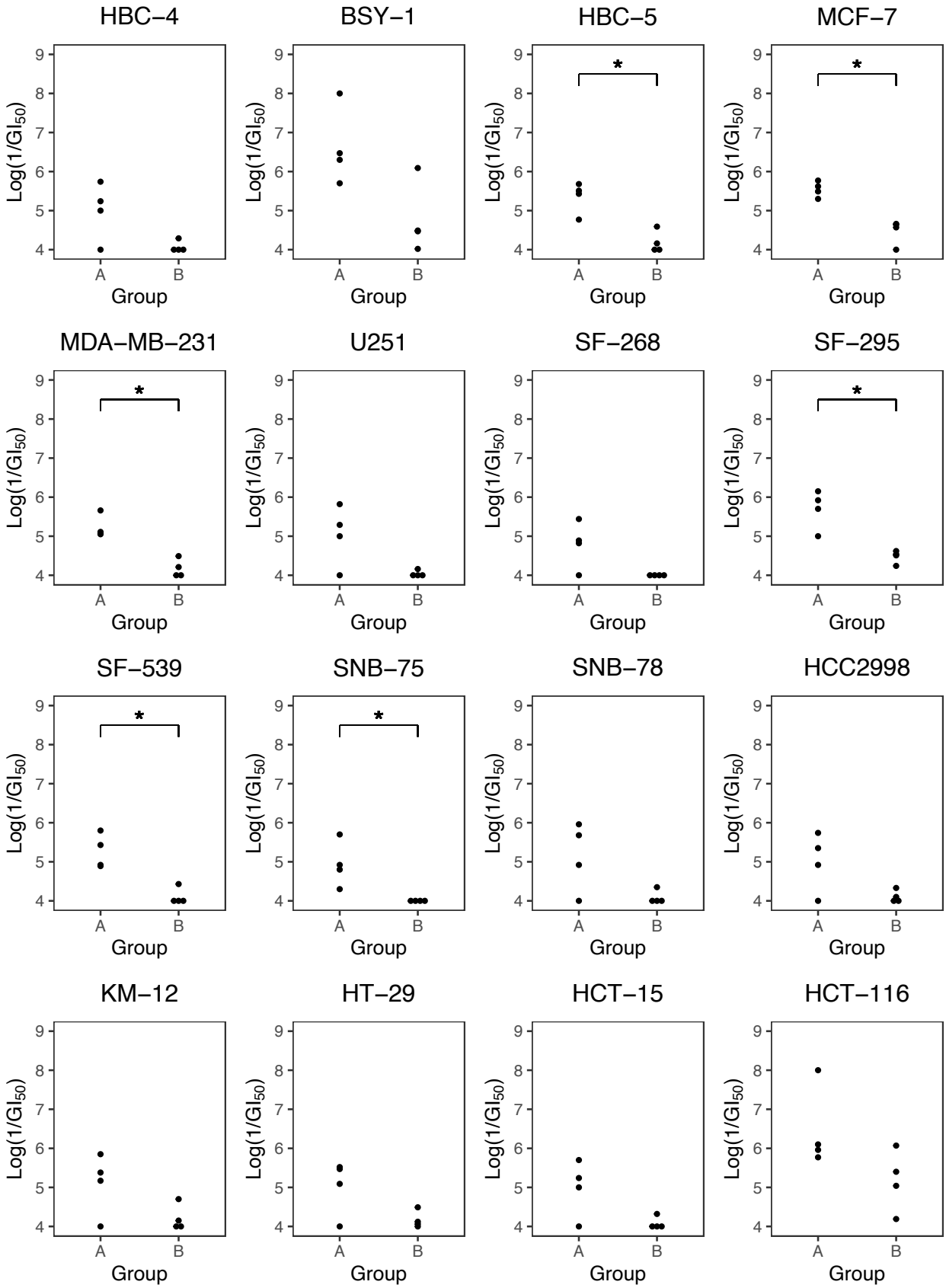


Fig. S1A. Comparison of growth-inhibitory activities of analogs **1**, **7a**, **7e**, and **7g-7k**. Each plot shows distribution of logarithm of $1/GI_{50}$ against each human cancer cell line among the group A (**7a**, **7e**, **7g** and **7h**) and B (**1** and **7i**-**7k**). *P* values were determined by the Mann–Whitney U test (*p <0.05).

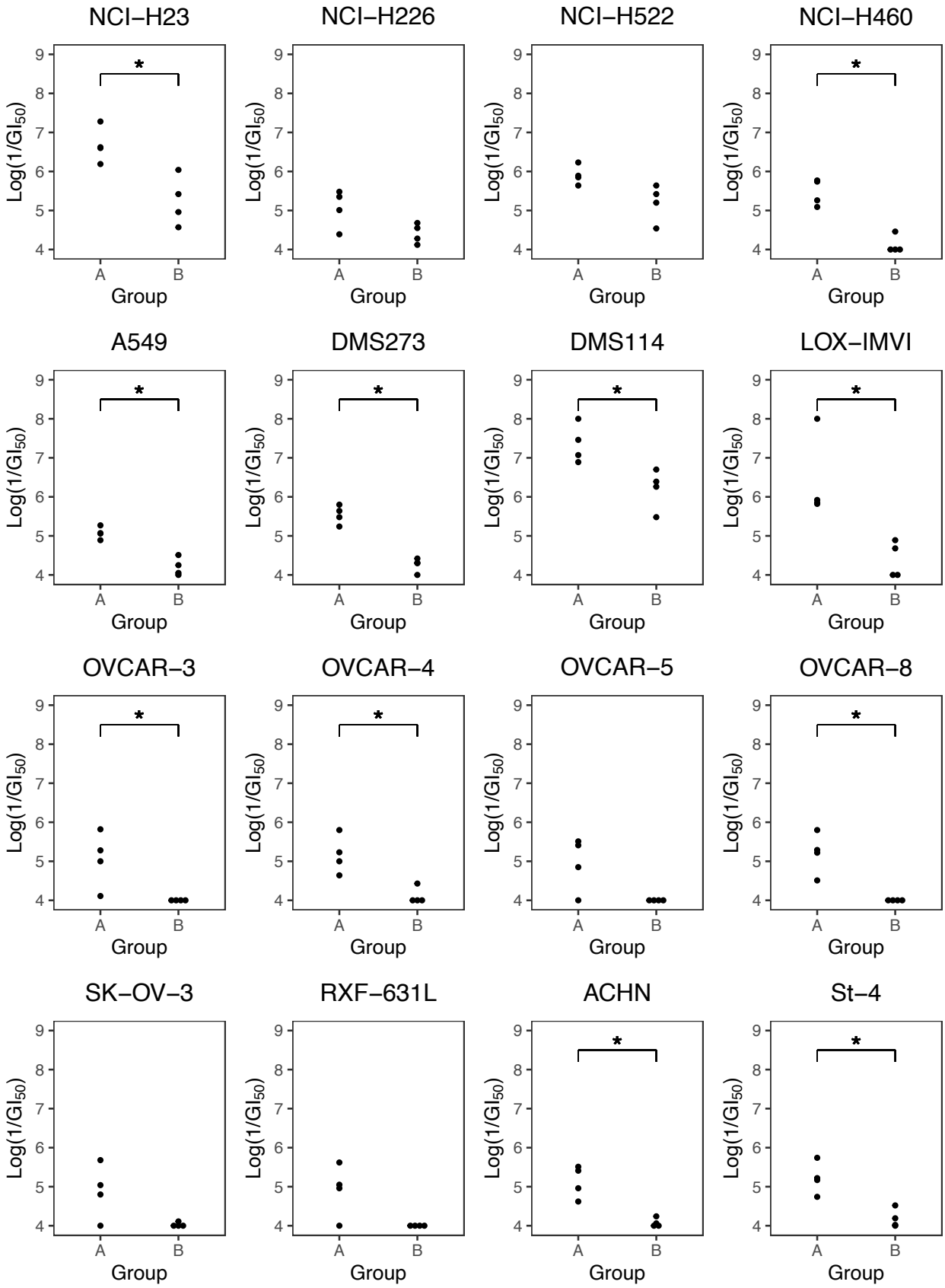


Fig. S1B. Comparison of growth-inhibitory activities of analogs **1**, **7a**, **7e**, and **7g-7k**. Each plot shows distribution of logarithm of $1/GI_{50}$ against each human cancer cell line among the group A (**7a**, **7e**, **7g** and **7h**) and B (**1** and **7i**-**7k**). *P* values were determined by the Mann–Whitney U test (*p <0.05).

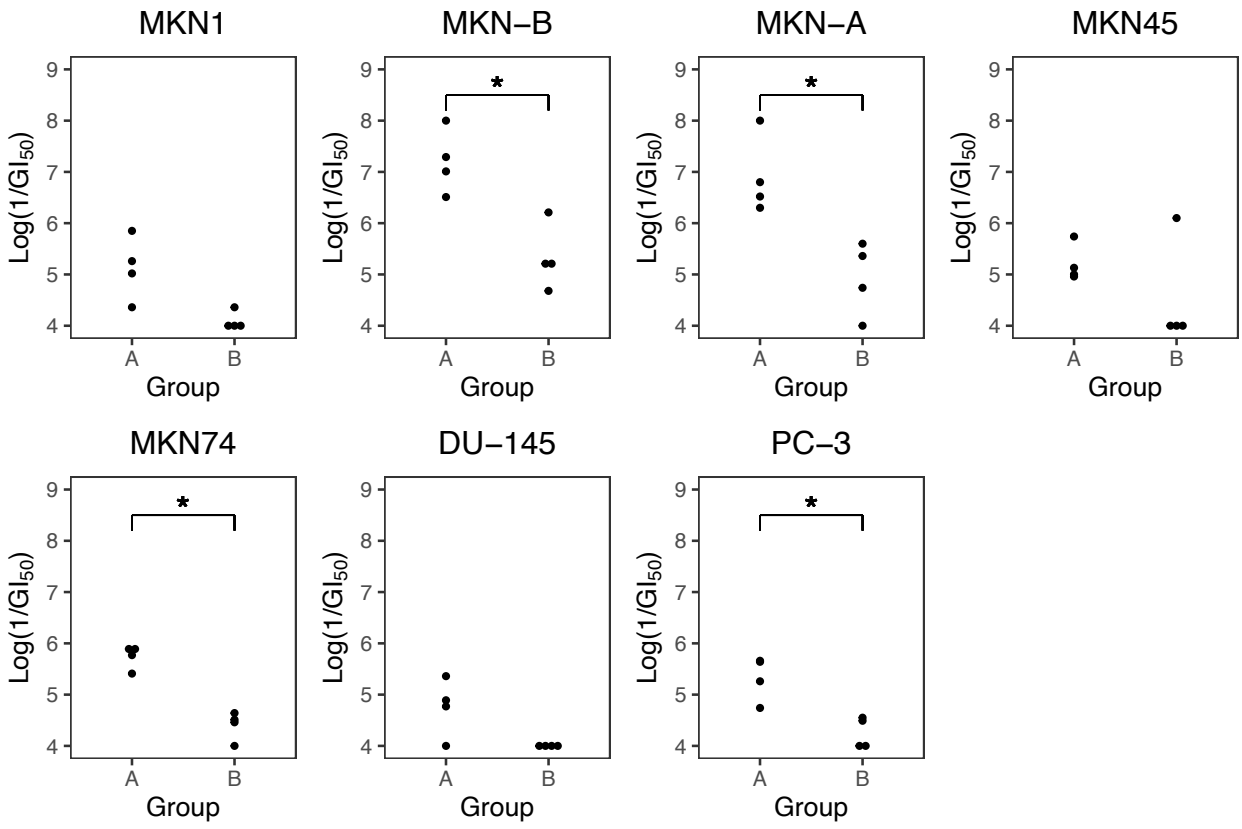


Fig. S1C. Comparison of growth-inhibitory activities of analogs **1**, **7a**, **7e**, and **7g-7k**. Each plot shows distribution of logarithm of $1/GI_{50}$ against each human cancer cell line among the group A (**7a**, **7e**, **7g** and **7h**) and B (**1** and **7i**-**7k**). *P* values were determined by the Mann–Whitney U test (*p <0.05).

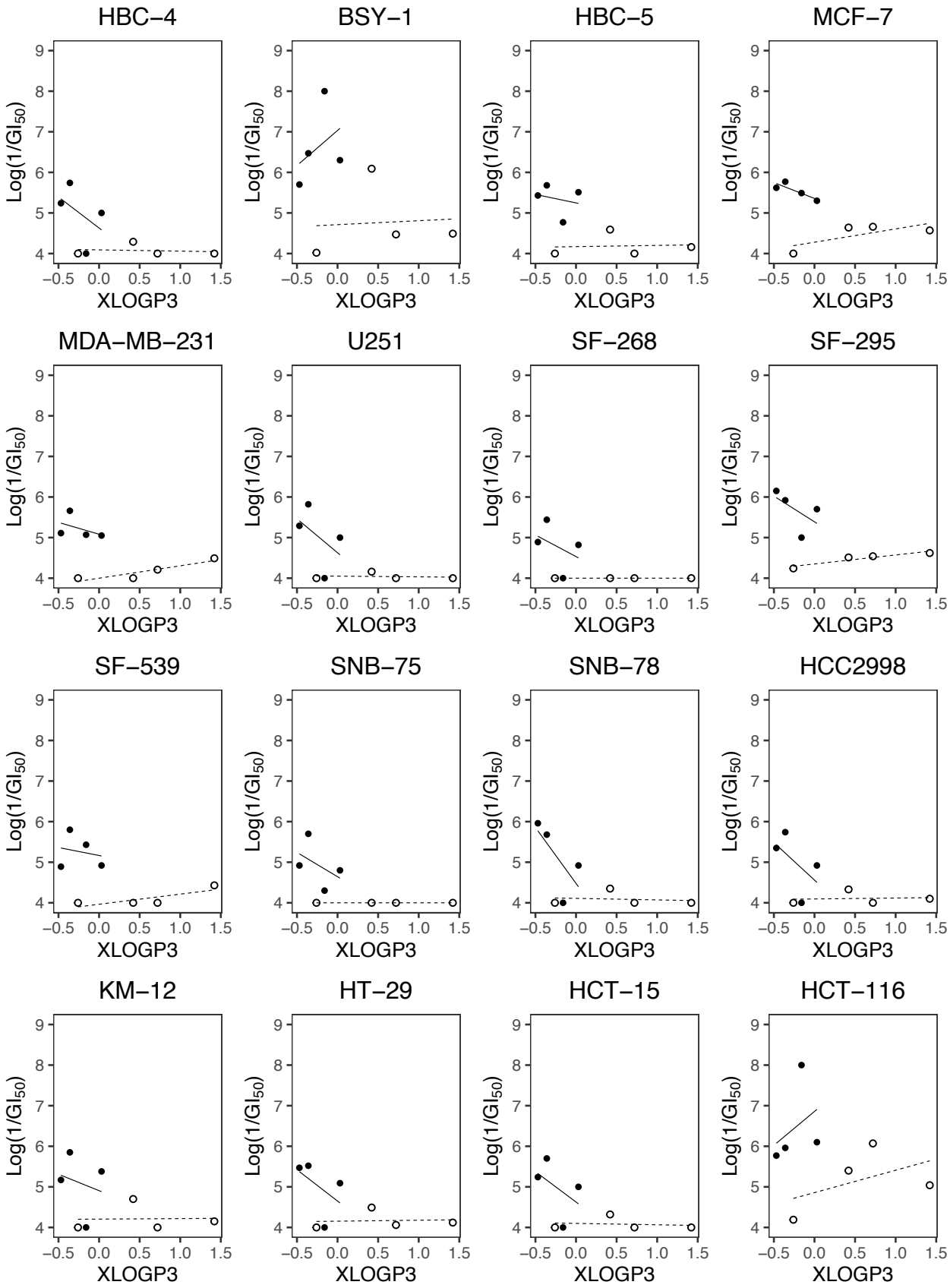


Fig. S2A. Correlation between lipophilicity (XLOGP3) of the partial structure of our analogs and logarithm of $1/GI_{50}$ against each cancer cell line. Solid circles represent compounds of group A (**7a**, **7e**, **7g** and **7h**), and open circles represent compounds of group B (**1** and **7i**-**7k**). Trend lines represent the expected values predicted by the QSAR model.

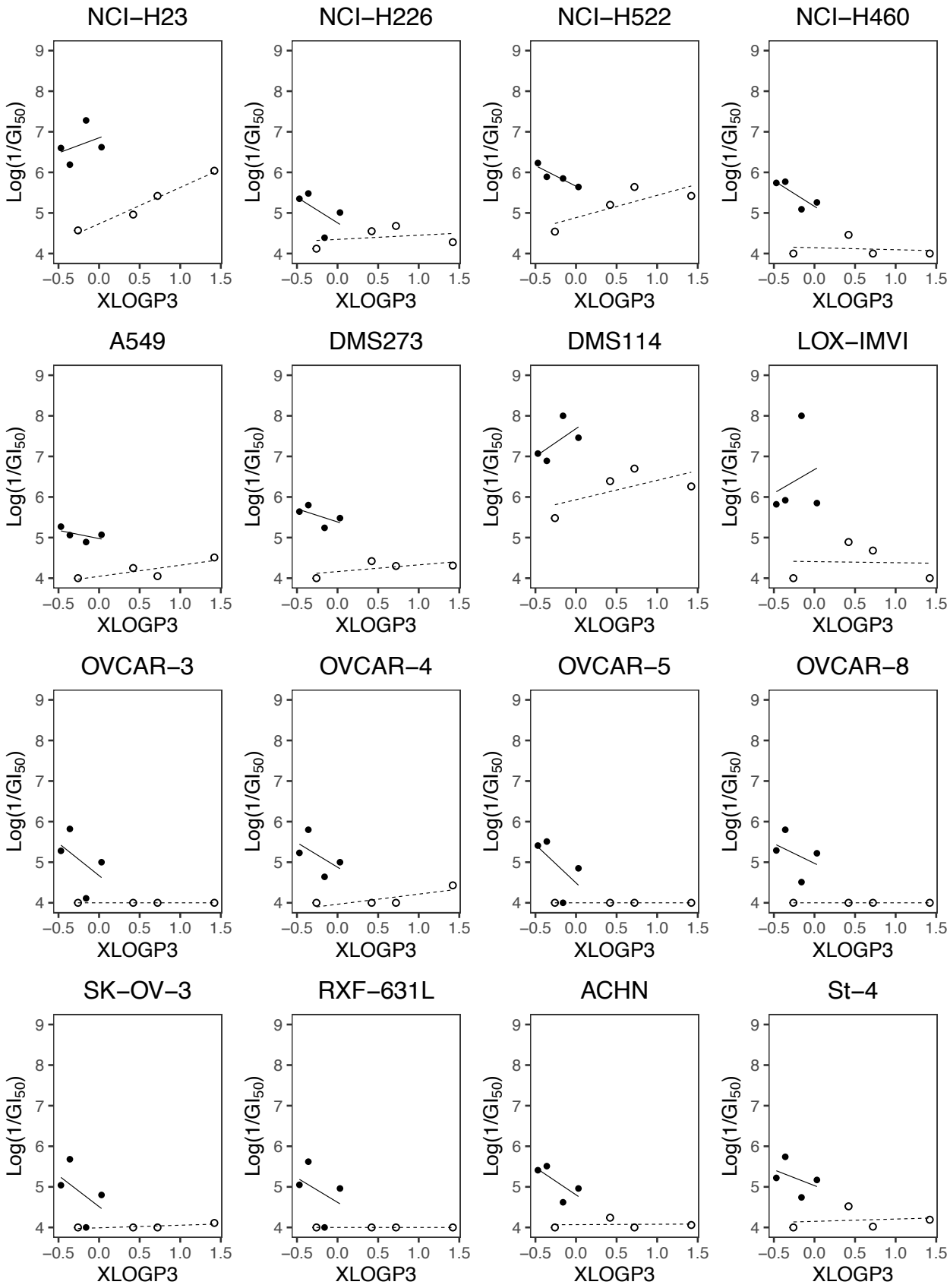


Fig. S2B. Correlation between lipophilicity (XLOGP3) of the partial structure of our analogs and logarithm of $1/GI_{50}$ against each cancer cell line. Solid circles represent compounds of group A (**7a**, **7e**, **7g** and **7h**), and open circles represent compounds of group B (**1** and **7i**-**7k**). Trend lines represent the expected values predicted by the QSAR model.

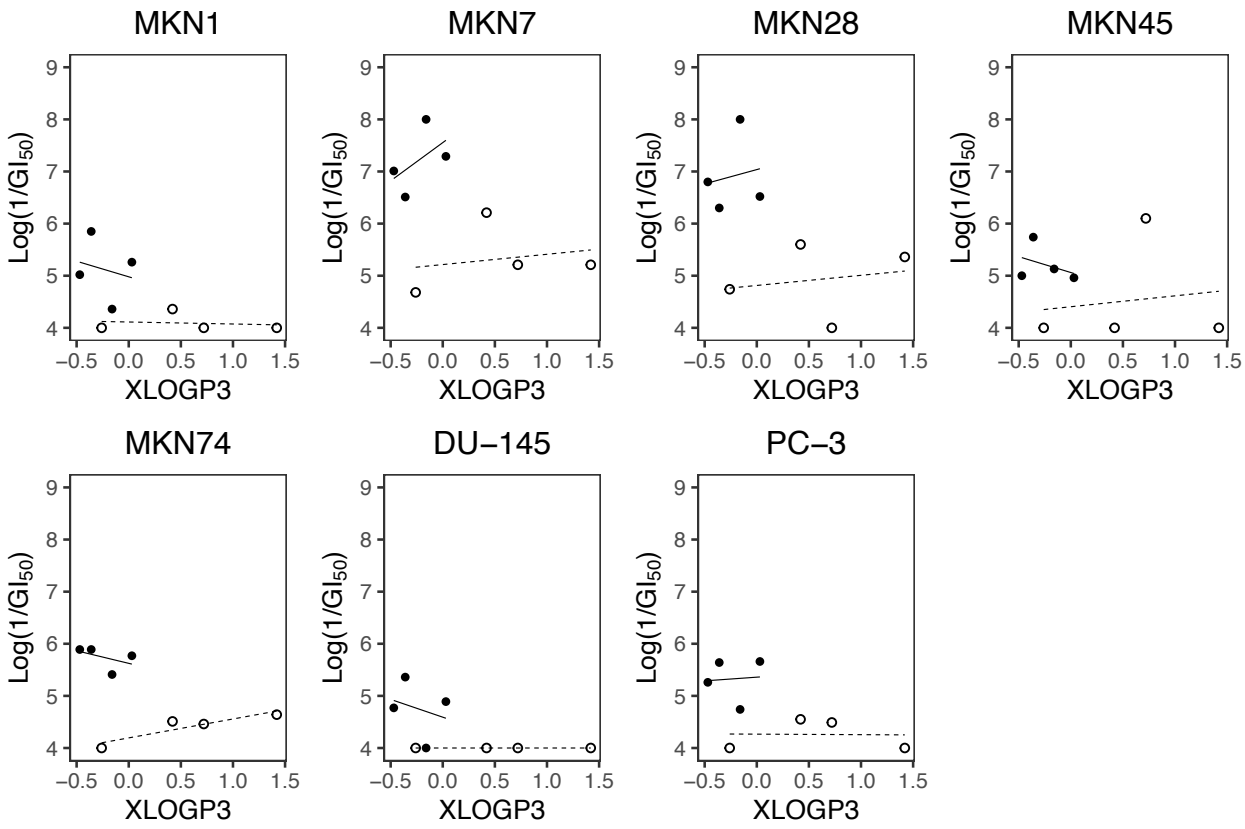


Fig. S2C. Correlation between lipophilicity (XLOGP3) of the partial structure of our analogs and logarithm of $1/GI_{50}$ against each cancer cell line. Solid circles represent compounds of group A (**7a**, **7e**, **7g** and **7h**), and open circles represent compounds of group B (**1** and **7i**-**7k**). Trend lines represent the expected values predicted by the QSAR model.

Table S2A. QSAR models of the relationships between the Log($1/GI_{50}$) and XLOP3 of partial structure of analogs in group A (**7a**, **7e**, **7g**, and **7h**). *n* is the number of compounds, *r* is the correlation coefficient, *s* is the standard deviation, and the figures in parentheses are the 95% confidence intervals.

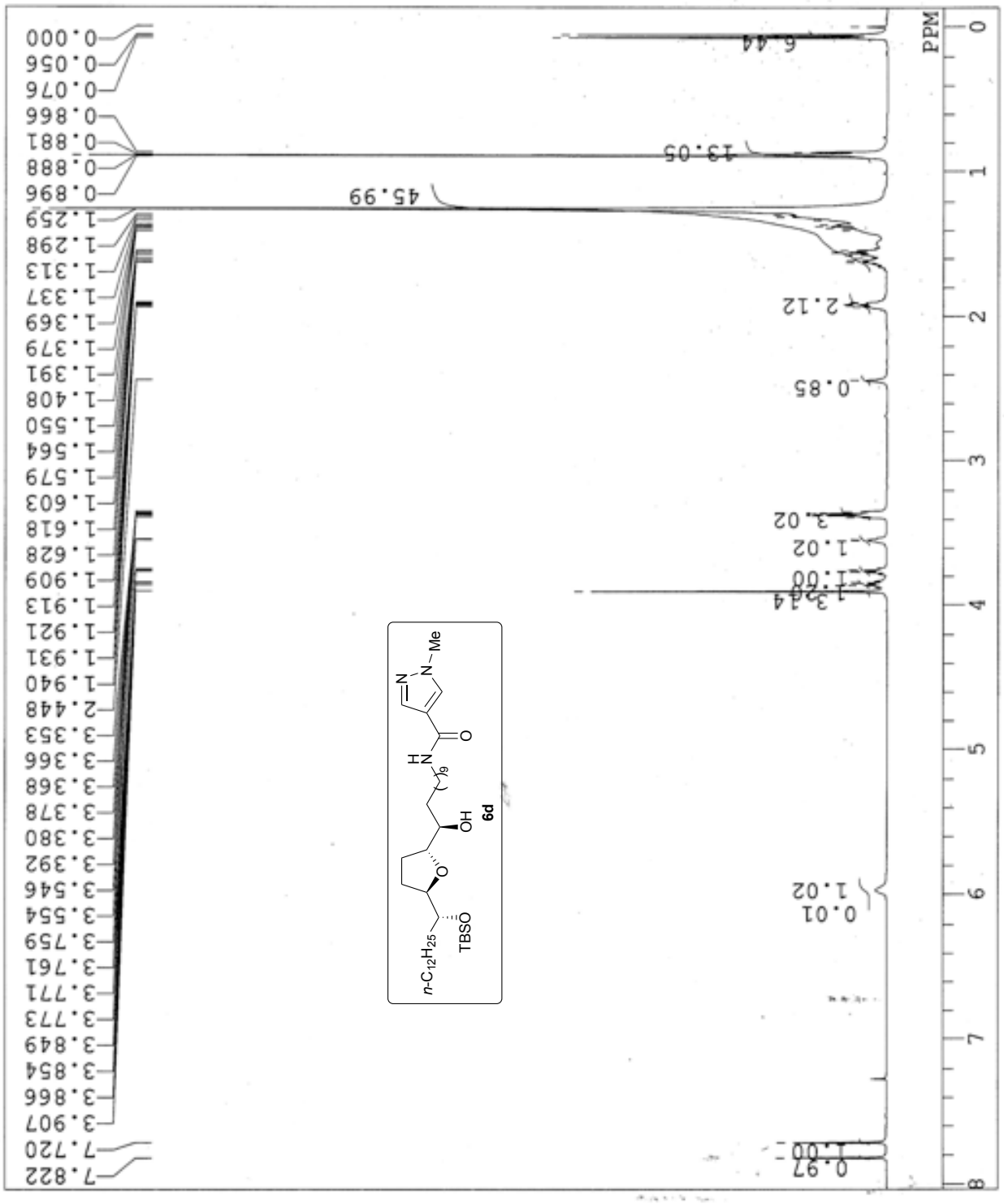
$$\text{Log}\ (1/GI_{50}) = a\ \text{XLOGP3} + b$$

cell line	*a*	*b*	*n*	r^2	*s*
HBC-4	-1.528(±8.930)	4.628(±2.741)	4	0.213	0.795
BSY-1	1.730(±12.405)	7.033(±3.808)	4	0.153	1.104
HBC-5	-0.417(±5.341)	5.247(±1.640)	4	0.054	0.475
MCF-7	-0.783(±1.363)	5.357(±0.418)	4	0.754	0.121
MDA-MB-231	-0.583(±3.618)	5.083(±1.111)	4	0.194	0.322
U251	-1.672(±9.211)	4.626(±2.828)	4	0.234	0.820
SF-268	-1.065(±7.502)	4.532(±2.303)	4	0.157	0.668
SF-295	-1.268(±5.646)	5.388(±1.733)	4	0.318	0.502
SF-539	-0.395(±5.894)	5.165(±1.809)	4	0.040	0.524
SNB-75	-1.198(±7.091)	4.643(±2.177)	4	0.209	0.631
SNB-78	-2.756(±8.700)	4.479(±2.671)	4	0.482	0.774
HCC2998	-1.848(±8.617)	4.559(±2.645)	4	0.299	0.767
KM-12	-0.808(±10.542)	4.906(±3.236)	4	0.052	0.938
HT-29	-1.543(±8.517)	4.650(±2.615)	4	0.233	0.758
HCT-15	-1.495(±8.773)	4.626(±2.693)	4	0.212	0.781
HCT-116	1.669(±13.341)	6.858(±4.096)	4	0.127	1.187
NCI-H23	0.744(±5.778)	6.851(±1.774)	4	0.133	0.514
NCI-H226	-1.256(±5.508)	4.756(±1.691)	4	0.325	0.490
NCI-H522	-1.016(±1.326)	5.659(±0.407)	4	0.845	0.118
NCI-H460	-1.263(±2.722)	5.162(±0.836)	4	0.666	0.242
A549	-0.404(±1.751)	4.976(±0.538)	4	0.330	0.156
DMS273	-0.644(±2.640)	5.385(±0.811)	4	0.355	0.235
DMS114	1.373(±5.319)	7.685(±1.633)	4	0.382	0.473
LOX-IMVI	1.163(±14.283)	6.677(±4.385)	4	0.058	1.271
OVCAR-3	-1.596(±8.552)	4.669(±2.626)	4	0.244	0.761
OVCAR-4	-1.212(±5.590)	4.877(±1.716)	4	0.303	0.497
OVCAR-5	-1.883(±7.613)	4.491(±2.337)	4	0.361	0.677
OVCAR-8	-0.972(±6.677)	4.972(±2.050)	4	0.164	0.594
SK-OV-3	-1.533(±8.340)	4.512(±2.560)	4	0.238	0.742
RXF-631L	-1.205(±8.489)	4.618(±2.606)	4	0.157	0.755
ACHN	-1.342(±3.955)	4.803(±1.214)	4	0.516	0.352
St-4	-0.780(±5.114)	5.030(±1.570)	4	0.177	0.455
MKN1	-0.598(±8.287)	4.979(±2.544)	4	0.046	0.737
MKN-B	1.465(±7.307)	7.554(±2.243)	4	0.271	0.650
MKN-A	0.548(±10.300)	7.037(±3.162)	4	0.026	0.917
MKN45	-0.608(±4.631)	5.061(±1.422)	4	0.138	0.412
MKN74	-0.483(±2.760)	5.624(±0.847)	4	0.221	0.246
DU-145	-0.682(±7.481)	4.591(±2.297)	4	0.071	0.666
PC-3	0.142(±5.919)	5.359(±1.817)	4	0.005	0.527

Table S2B. QSAR models of the relationships between the Log($1/GI_{50}$) and XLOP3 of partial structure of analogs in group B (**1**, and **7i**-**7k**). *n* is the number of compounds, *r* is the correlation coefficient, *s* is the standard deviation, and the figures in parentheses are the 95% confidence intervals.

$$\text{Log}\ (1/GI_{50}) = a\ \text{XLOGP3} + b$$

cell line	*a*	*b*	*n*	r^2	*s*
HBC-4	-0.031(±0.626)	4.090(±0.522)	4	0.022	0.176
BSY-1	0.097(±3.954)	4.712(±3.295)	4	0.006	1.109
HBC-5	0.030(±1.214)	4.170(±1.012)	4	0.006	0.340
MCF-7	0.328(±0.940)	4.279(±0.783)	4	0.531	0.264
MDA-MB-231	0.305(±0.407)	3.999(±0.339)	4	0.839	0.114
U251	-0.017(±0.345)	4.050(±0.288)	4	0.022	0.097
SF-268	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
SF-295	0.222(±0.254)	4.350(±0.212)	4	0.876	0.071
SF-539	0.250(±0.552)	3.964(±0.460)	4	0.654	0.155
SNB-75	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
SNB-78	-0.037(±0.756)	4.109(±0.630)	4	0.022	0.212
HCC2998	0.023(±0.676)	4.094(±0.563)	4	0.011	0.190
KM-12	0.013(±1.452)	4.205(±1.210)	4	0.001	0.407
HT-29	0.023(±0.960)	4.154(±0.800)	4	0.005	0.269
HCT-15	-0.034(±0.691)	4.100(±0.576)	4	0.022	0.194
HCT-116	0.552(±2.980)	4.858(±2.484)	4	0.241	0.836
NCI-H23	0.896(±0.438)	4.732(±0.365)	4	0.975	0.123
NCI-H226	0.103(±1.064)	4.348(±0.887)	4	0.080	0.298
NCI-H522	0.550(±1.228)	4.884(±1.024)	4	0.650	0.344
NCI-H460	-0.049(±0.993)	4.143(±0.828)	4	0.022	0.279
A549	0.274(±0.572)	4.045(±0.477)	4	0.680	0.160
DMS273	0.165(±0.605)	4.163(±0.504)	4	0.408	0.170
DMS114	0.477(±1.740)	5.933(±1.450)	4	0.410	0.488
LOX-IMVI	-0.027(±2.013)	4.408(±1.677)	4	0.002	0.564
OVCAR-3	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
OVCAR-4	0.250(±0.552)	3.964(±0.460)	4	0.654	0.155
OVCAR-5	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
OVCAR-8	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
SK-OV-3	0.064(±0.141)	3.991(±0.118)	4	0.654	0.040
RXF-631L	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
ACHN	0.009(±0.495)	4.070(±0.413)	4	0.003	0.139
St-4	0.057(±1.036)	4.150(±0.864)	4	0.027	0.291
MKN1	-0.038(±0.777)	4.112(±0.648)	4	0.022	0.218
MKN-B	0.197(±2.726)	5.214(±2.272)	4	0.046	0.764
MKN-A	0.195(±3.067)	4.813(±2.556)	4	0.036	0.860
MKN45	0.209(±4.541)	4.405(±3.785)	4	0.019	1.274
MKN74	0.363(±0.514)	4.194(±0.428)	4	0.822	0.144
DU-145	0.000(±0.000)	4.000(±0.000)	4	NaN	0.000
PC-3	-0.010(±1.315)	4.266(±1.096)	4	0.001	0.369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COMNT
DATIM Wed Feb 17 14:17
OBNUC 1H
EXMOD non
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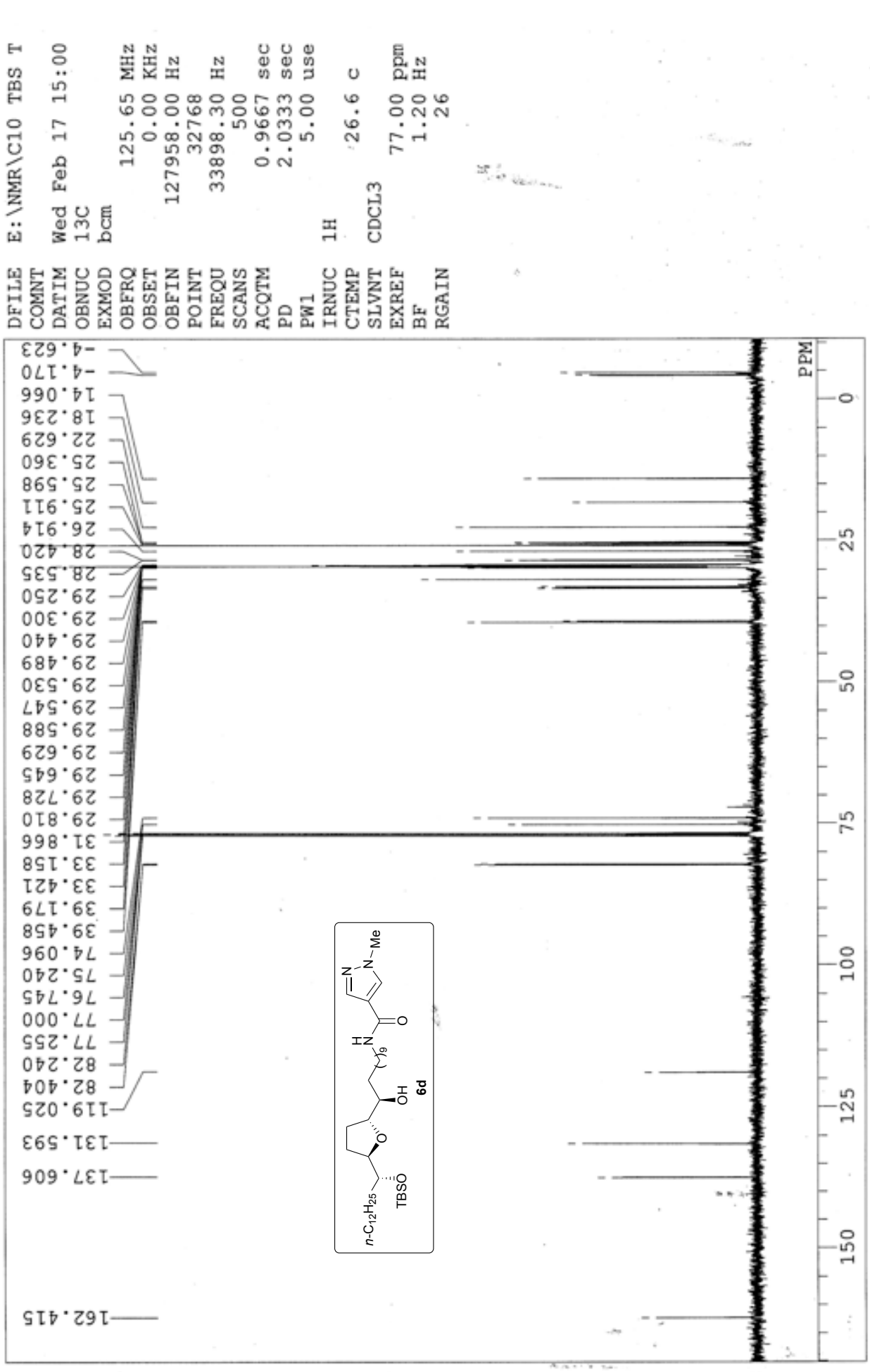
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 8192
FREQU 10000.00 Hz
SCANS 100
ACQTM 0.8192 sec
PD 6.1808 sec
PW1 5.70 use
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 9
PPM
6.44
13.05
45.99
2.12
0.85
3.02
1.02
1.00
3.14
1.02
0.01
1.00
0.97
6d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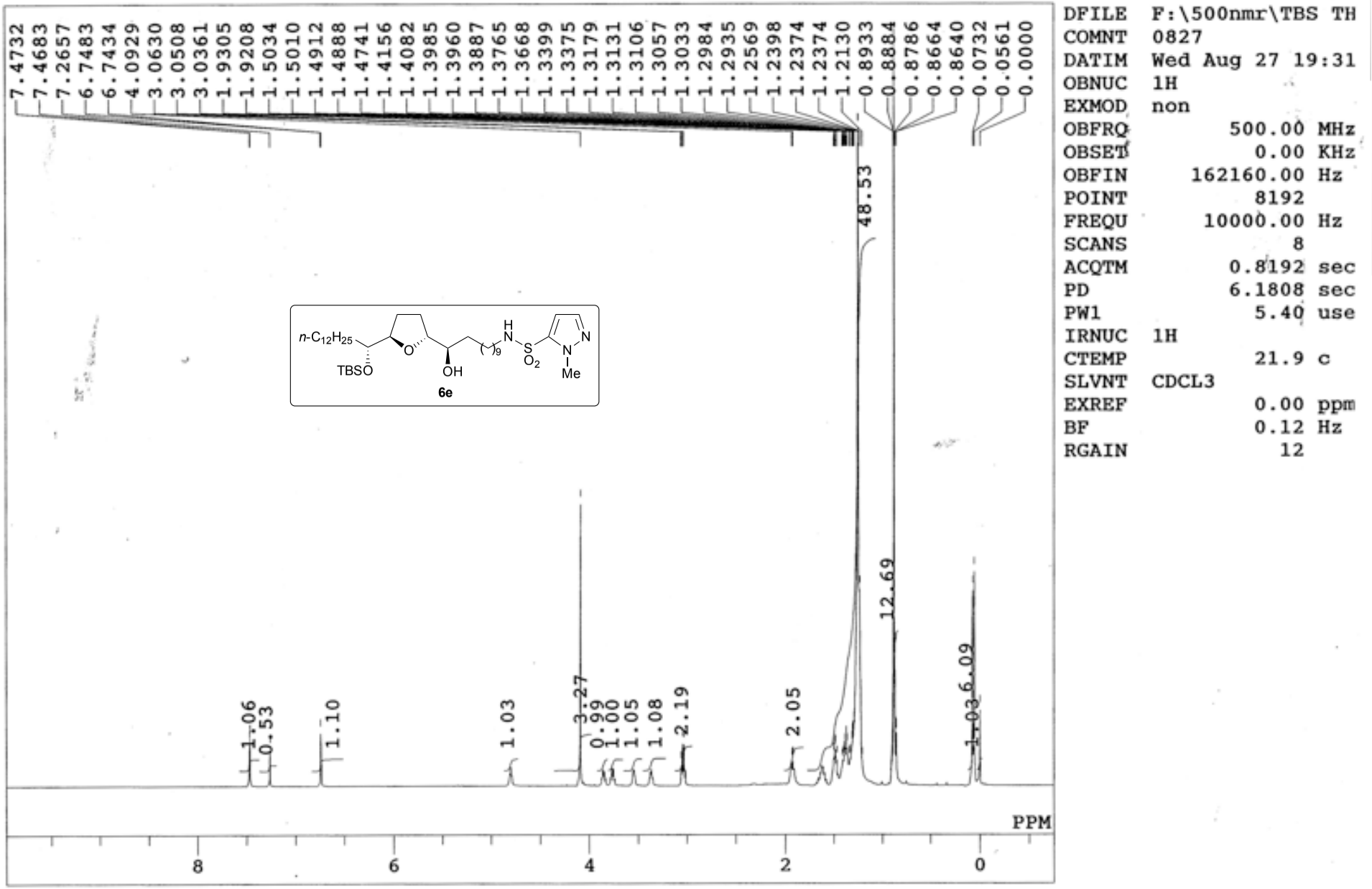
COMNT
DATIM Wed Feb 17 15:00
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 500
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26
-4.623
-4.170
14.066
18.236
22.629
25.360
25.598
25.911
26.914
28.420
28.535
29.250
29.300
29.440
29.489
29.530
29.547
29.588
29.629
29.645
29.728
29.810
31.866
33.158
33.421
39.179
39.458
74.096
75.240
76.745
77.000
77.255
82.240
82.404
119.025
131.593
137.606
162.415
PPM
0
25
50
75
100
125
150
n-C12H25
TBSO
OH
6d
Me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3.0361
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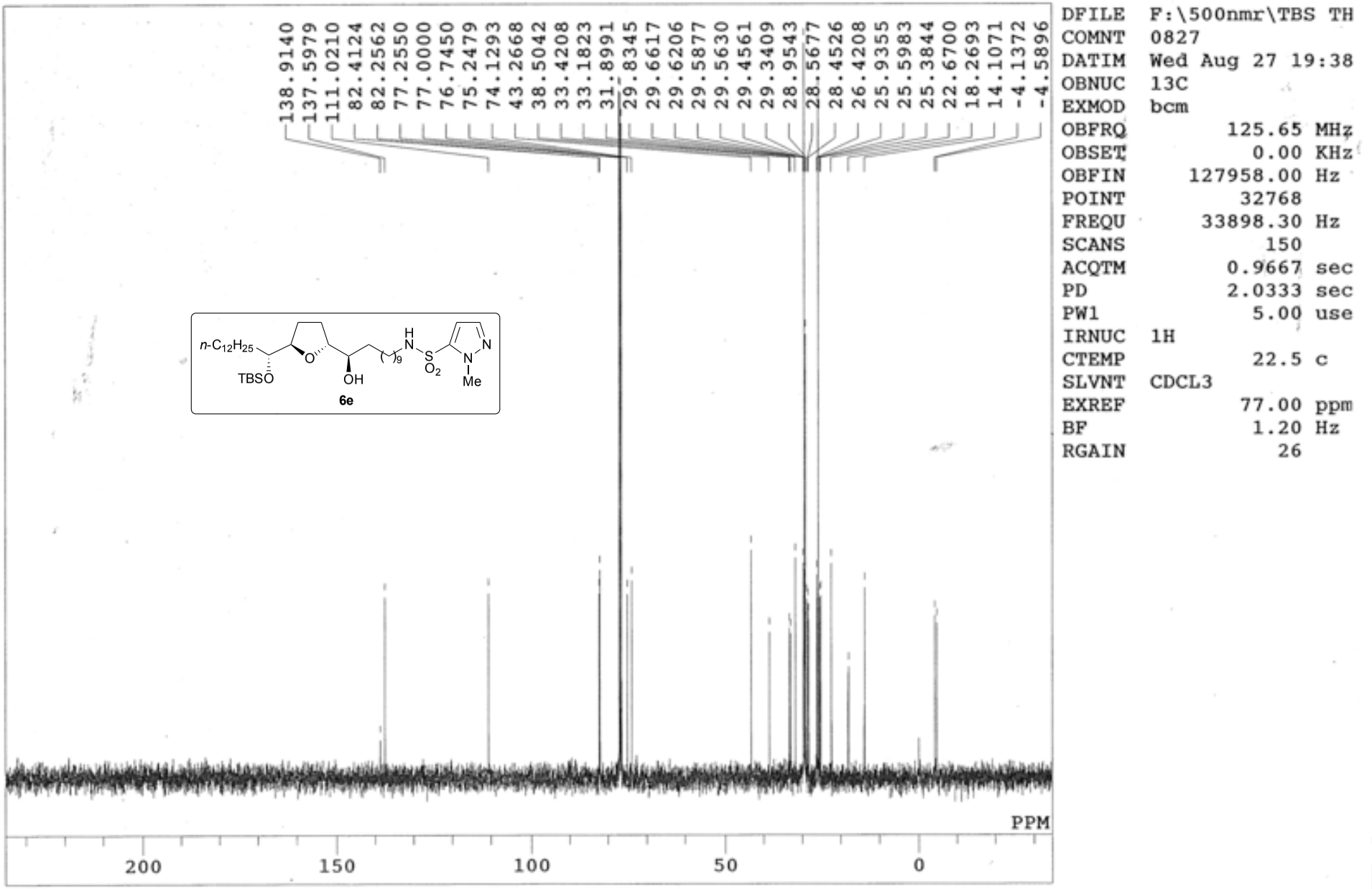
1.9208
1.5034
1.5010
1.4912
1.4888
1.4741
1.4156
1.4082
1.3985
1.3960
1.3887
1.3765
1.3668
1.3399
1.3375
1.3179
1.3131
1.3106
1.3057
1.3033
1.2984
1.2935
1.2569
1.2398
1.2374
1.2374
1.2130
0.8933
0.8884
0.8786
0.8664
0.8640
0.0732
0.0561
0.0000
48.53
12.69
6.09
1.06
0.53
1.10
1.03
3.27
0.99
1.00
1.05
1.08
2.19
2.05
1.03
PPM
8
6
4
2
0
n-C12H25
TBSO
O
OH
H
N
(9)
S
O2
N
N
Me
6e
DFILE F:\500nmr\TBS TH
COMNT 0827
DATIM Wed Aug 27 19:31
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 8192
FREQU 10000.00 Hz
SCANS 8
ACQTM 0.8192 sec
PD 6.1808 sec
PW1 5.40 use
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12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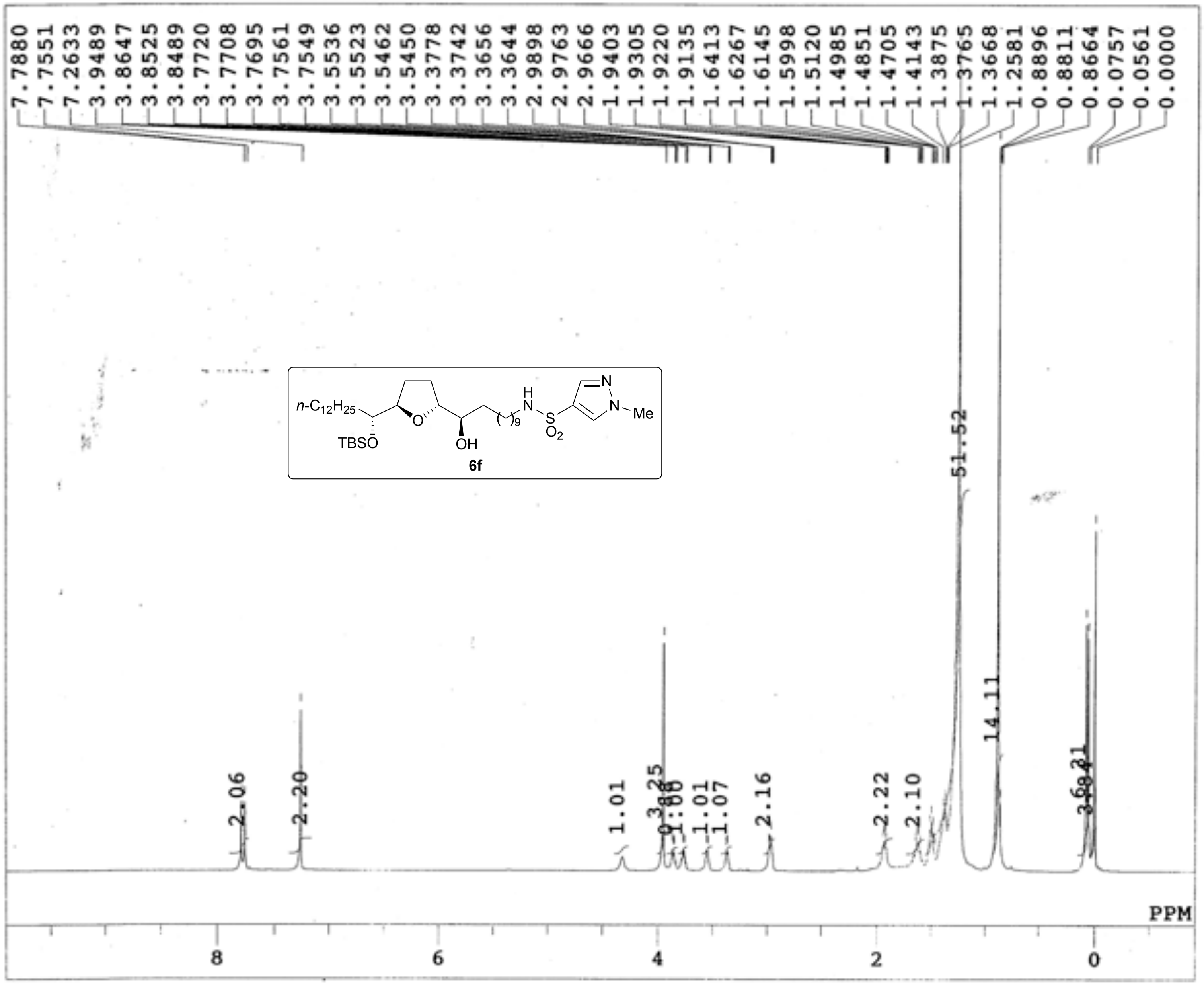
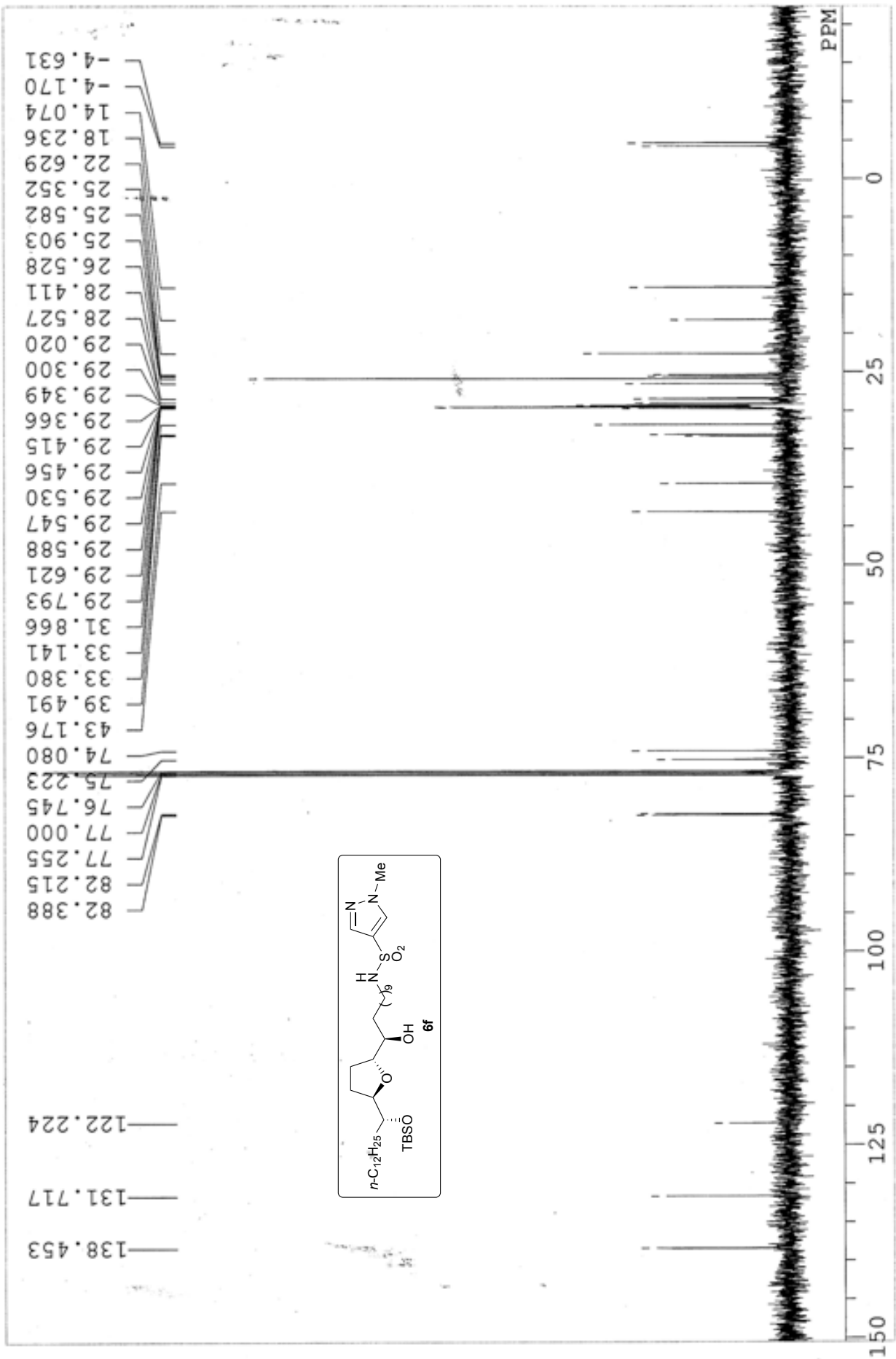
43.2668
38.5042
33.4208
33.1823
31.8991
29.8345
29.6617
29.6206
29.5877
29.5630
29.4561
29.3409
28.9543
28.5677
28.4526
26.4208
25.9355
25.5983
25.3844
22.6700
18.2693
14.1071
-4.1372
-4.5896
DFILE F:\500nmr\TBS TH
COMNT 0827
DATIM Wed Aug 27 19:38
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 150
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26
n-C12H25
TBSO
OH
Me
6e
PPM
200
150
100
50
0



DFILE	F:\500nmr\TBS
COMNT	
DATIM	Wed Dec 10 12:
OBNUC	1H
EXMOD	non
OBFRQ	500.00 M
OBSET	0.00 K
OBFIN	162160.00 H
POINT	16384
FREQU	10000.00 H
SCANS	16
ACQTM	1.6384 s
PD	5.3616 s
PW1	5.40 u
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	0.00 p
BF	1.20 H
RGAIN	14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14.074
18.236
22.629
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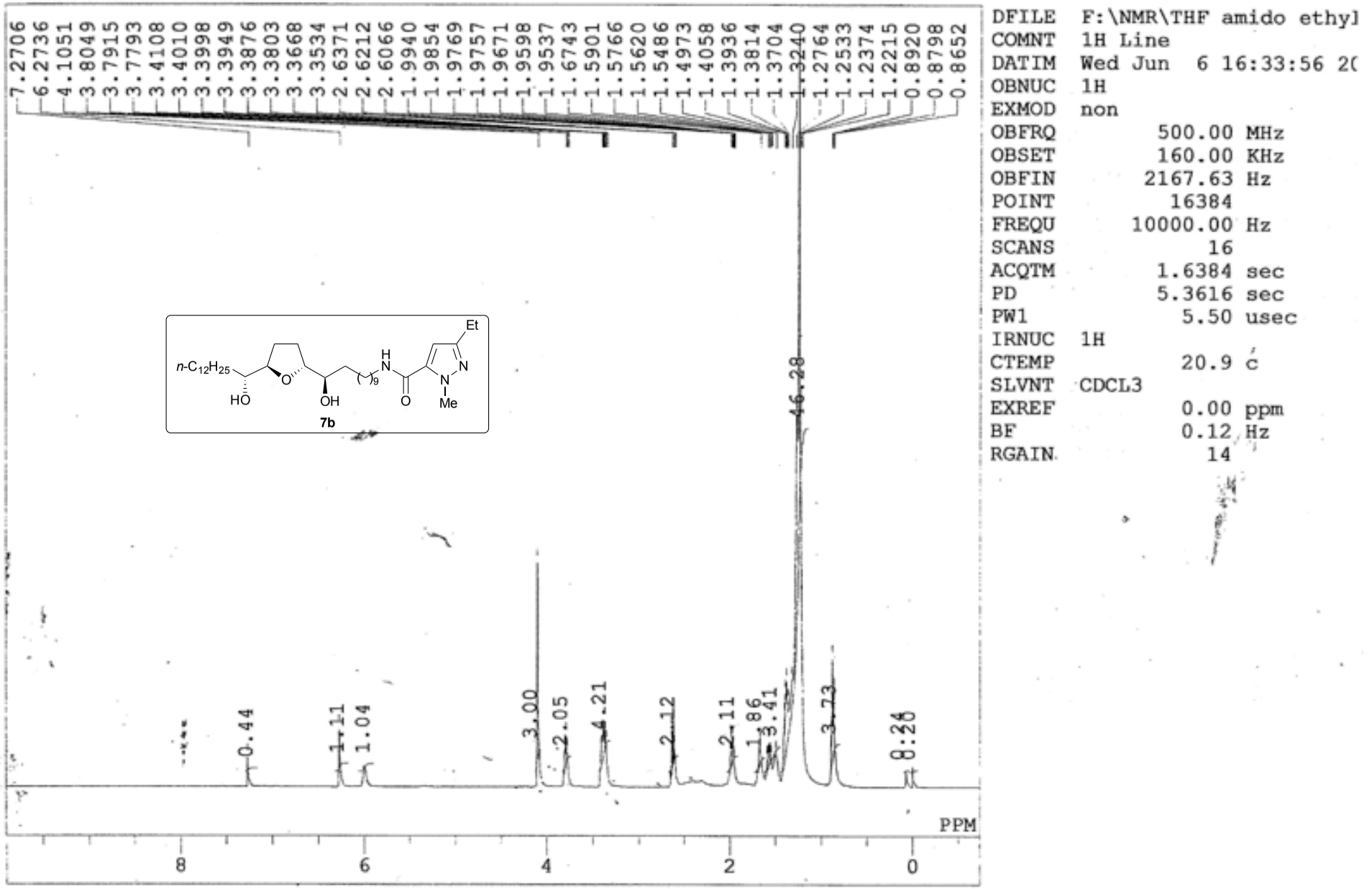
25.582
25.903
26.528
28.411
28.527
29.020
29.300
29.349
29.366
29.415
29.456
29.530
29.547
29.588
29.621
29.793
31.866
33.141
33.380
39.491
43.176
74.080
75.223
76.745
77.000
77.255
82.215
82.388
122.224
131.717
138.453
PPM
0
25
50
75
100
125
150
n-C12H25
TBSO
O
OH
6f
H
N
S
O2
N
N
Me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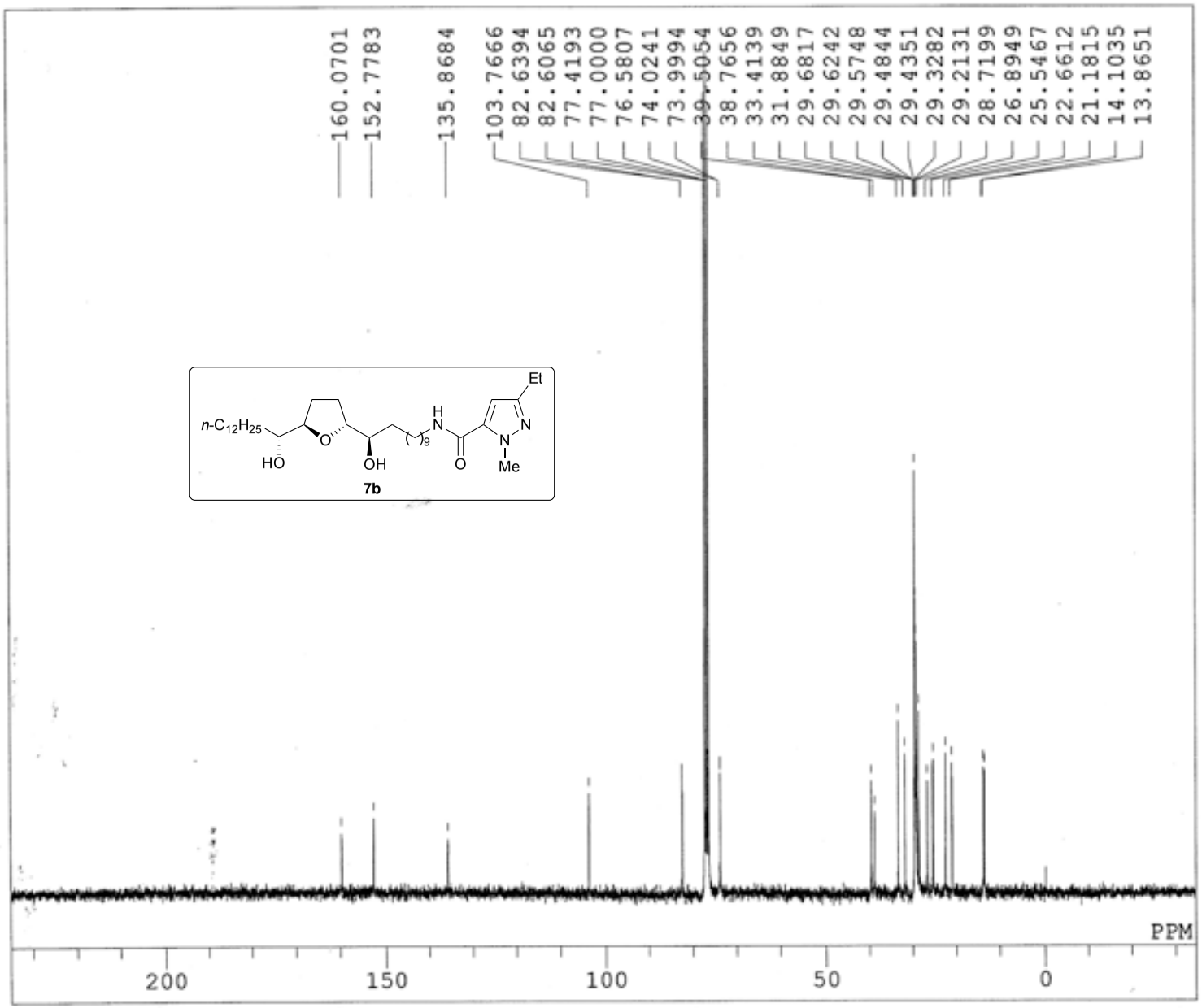
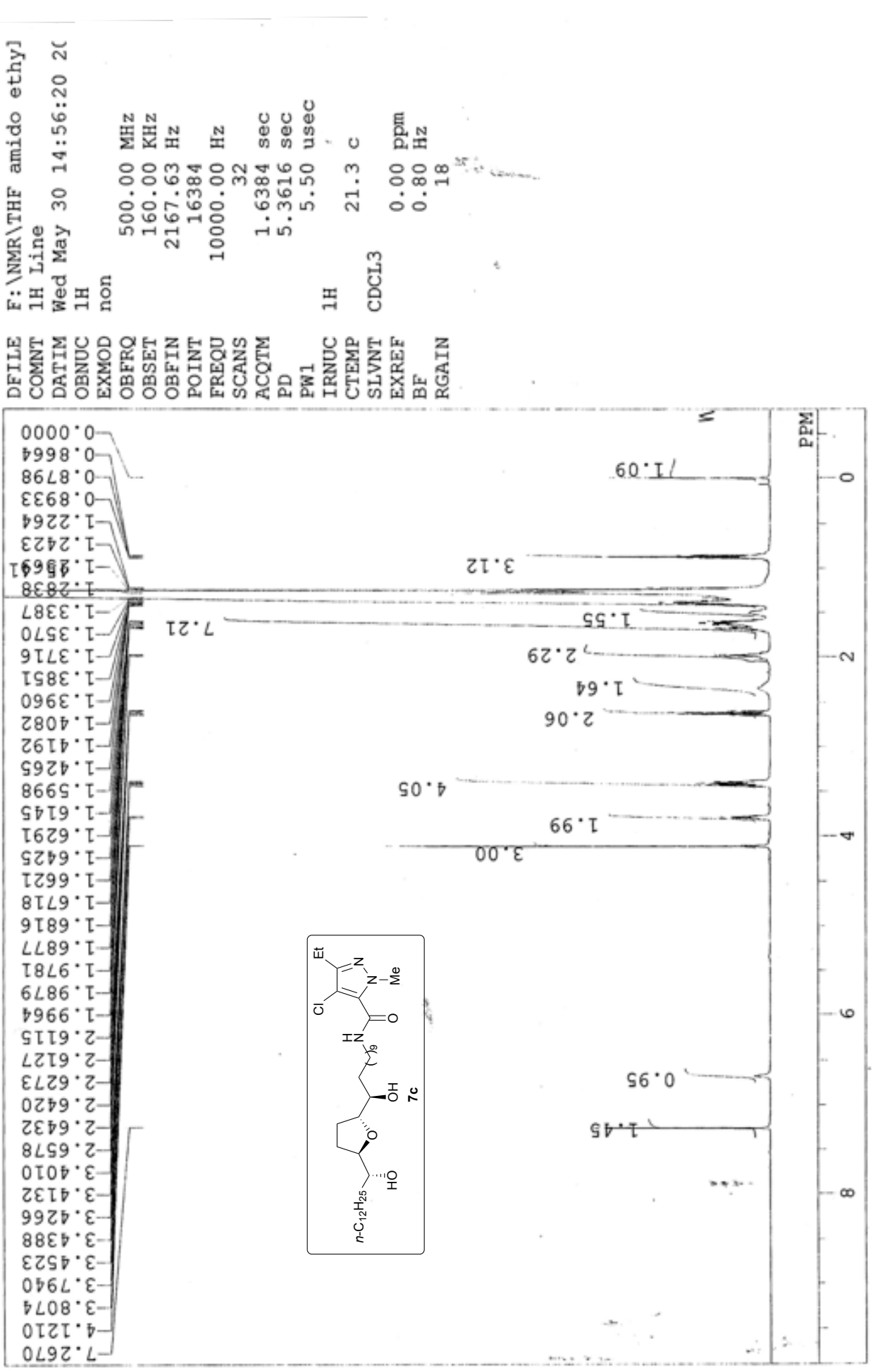
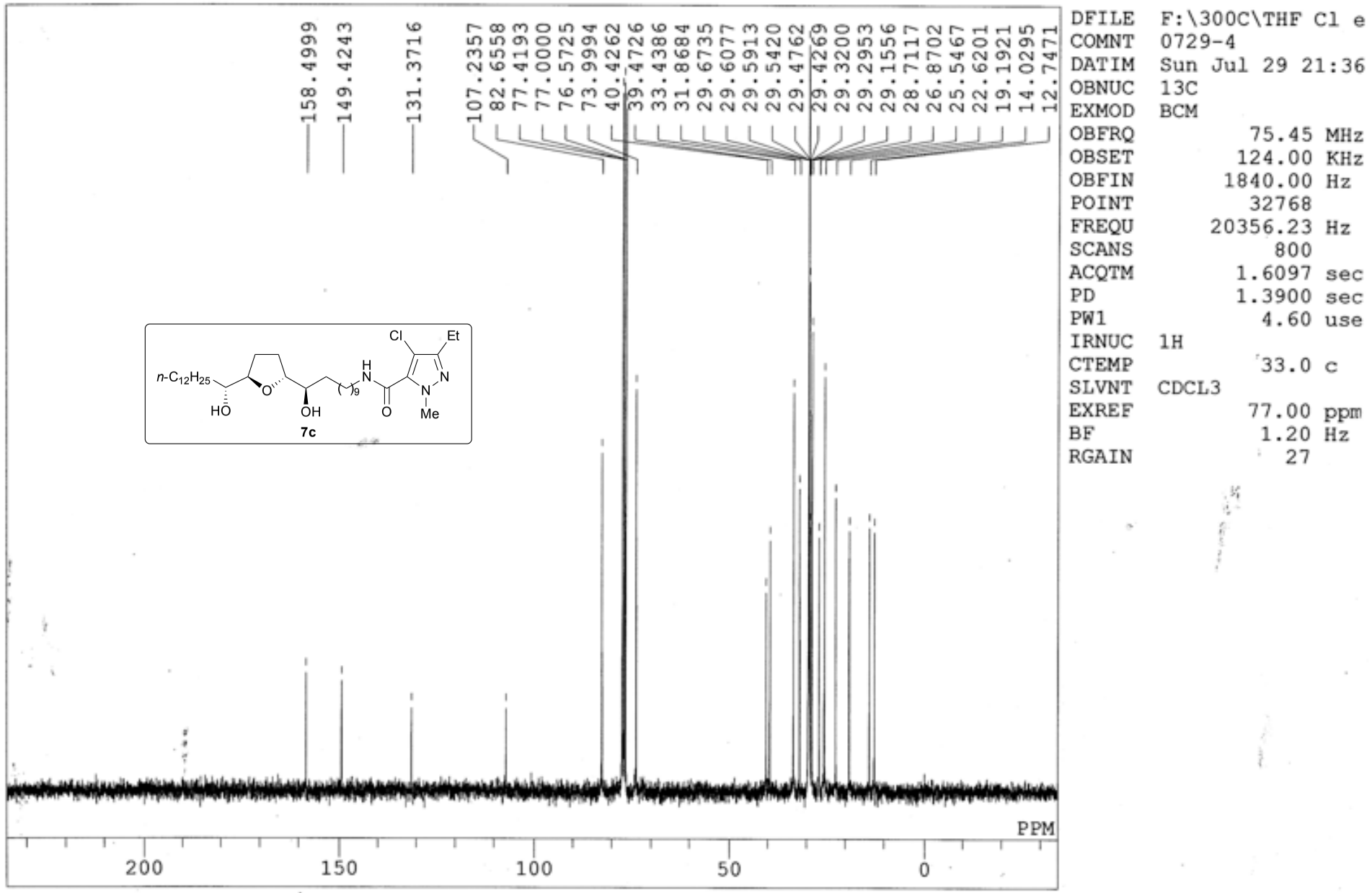
3.3998
3.3949
3.3876
3.3803
3.3668
3.3534
2.6371
2.6212
2.6066
1.9940
1.9854
1.9769
1.9757
1.9671
1.9598
1.9537
1.6743
1.5901
1.5766
1.5620
1.5486
1.4973
1.4058
1.3936
1.3814
1.3704
1.3240
1.2764
1.2533
1.2374
1.2215
0.8920
0.8798
0.8652
46.28
0.44
1.11
1.04
3.00
2.05
4.21
2.12
2.11
1.86
3.41
3.23
8.28
PPM
8
6
4
2
0
n-C12H25
HO
O
OH
H
N
9
O
N
N
Me
Et
7b
DFILE F:\NMR\THF amido ethyl
COMNT 1H Line
DATIM Wed Jun 6 16:33:56 2(
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2167.63 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 5.3616 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14



DFILE F:\300C\THFethyl
COMNT 070613
DATIM Thu Jun 14 01:57
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 2000
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 4.60 use
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24


DFILE F:\NMR\THF amido ethyl
COMNT 1H Line
DATIM Wed May 30 14:56:20 2(
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2167.63 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 32
ACQTM 1.6384 sec
PD 5.3616 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.80 Hz
RGAIN 18
PPM
1.09
3.12
7.21
1.55
2.29
1.64
2.06
4.05
1.99
3.00
0.95
1.45
7c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77.0000
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73.9994
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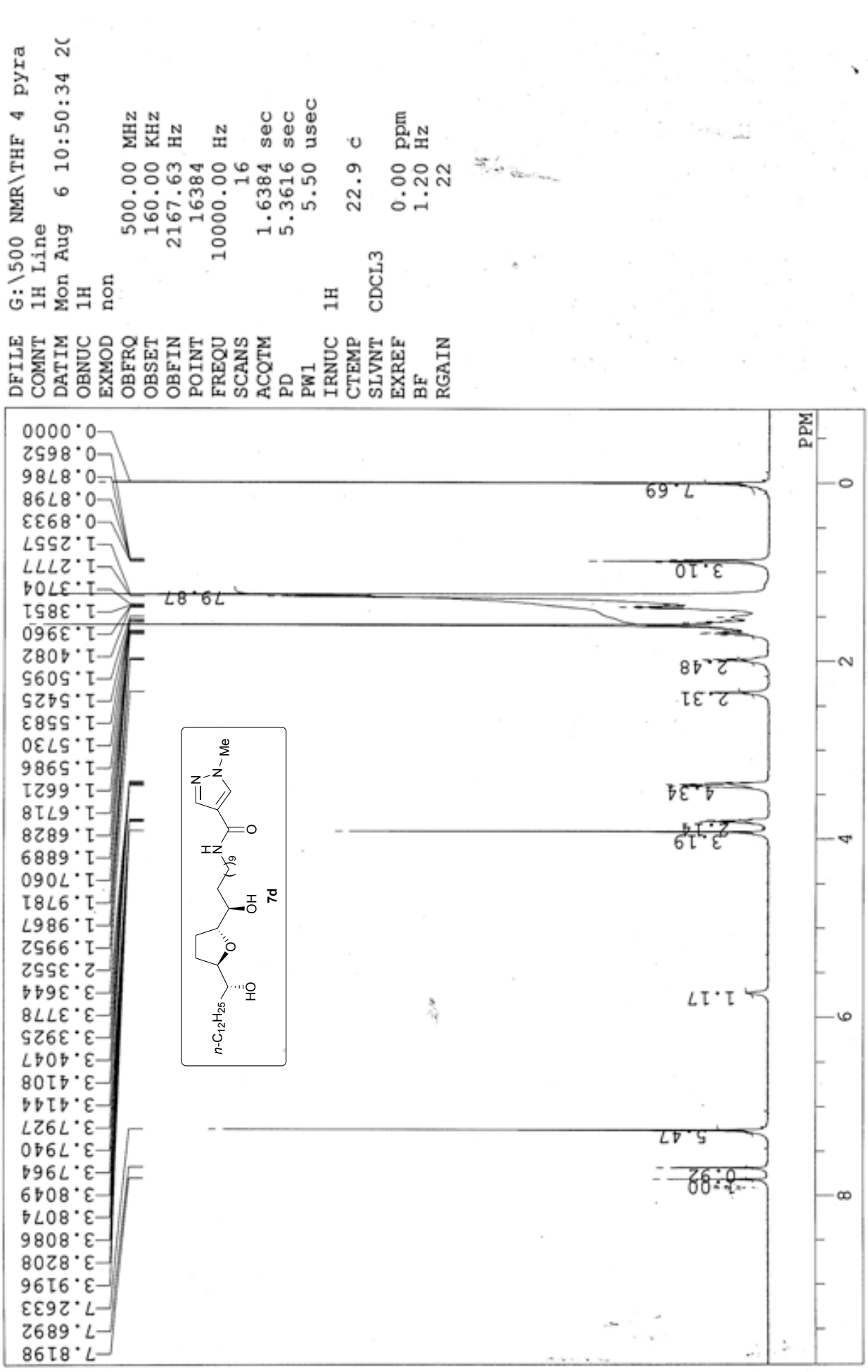
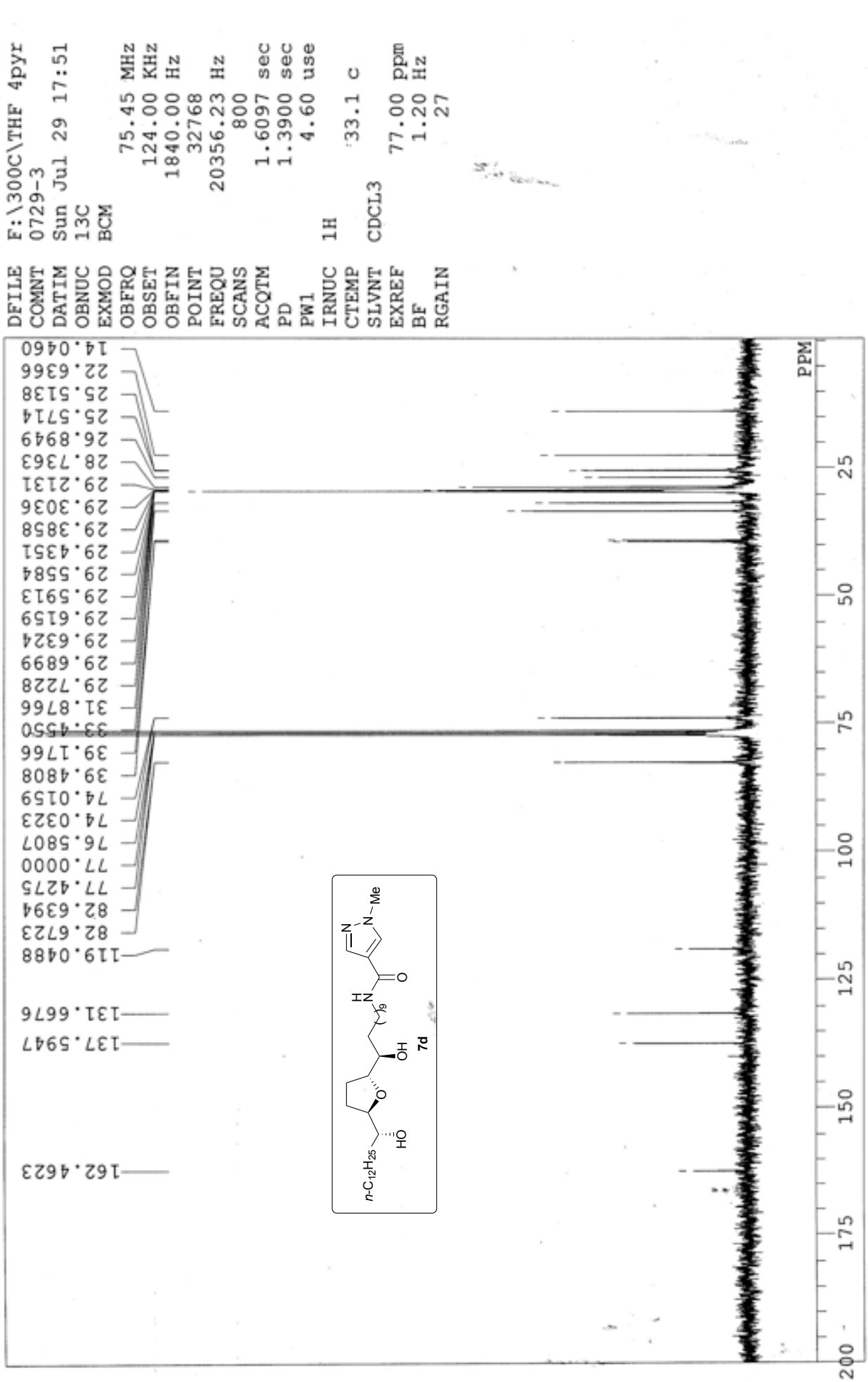
39.4726
33.4386
31.8684
29.6735
29.6077
29.5913
29.5420
29.4762
29.4269
29.3200
29.2953
29.1556
28.7117
26.8702
25.5467
22.6201
19.1921
14.0295
12.7471
DFILE F:\300C\THF Cl e
COMNT 0729-4
DATIM Sun Jul 29 21:36
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 800
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 4.60 use
IRNUC 1H
CTEMP 33.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 27
Cl
Et
n-C12H25
HO
O
OH
H
N
(9)
O
N
N
Me
7c
PPM
200
150
100
50
0


DFILE G:\500 NMR\THF 4 pyra
COMNT 1H Line
DATIM Mon Aug 6 10:50:34 2(
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2167.63 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 5.3616 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 22
PPM
7.69
3.10
79.87
2.48
2.31
4.34
2.14
3.19
1.17
5.47
7d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EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 800
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 4.60 use
IRNUC 1H
CTEMP 33.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 27
14.0460
22.6366
25.5138
25.5714
26.8949
28.7363
29.2131
29.3036
29.3858
29.4351
29.5584
29.5913
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29.6324
29.6899
29.7228
31.8766
33.4550
39.1766
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74.0159
74.0323
76.5807
77.0000
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82.6394
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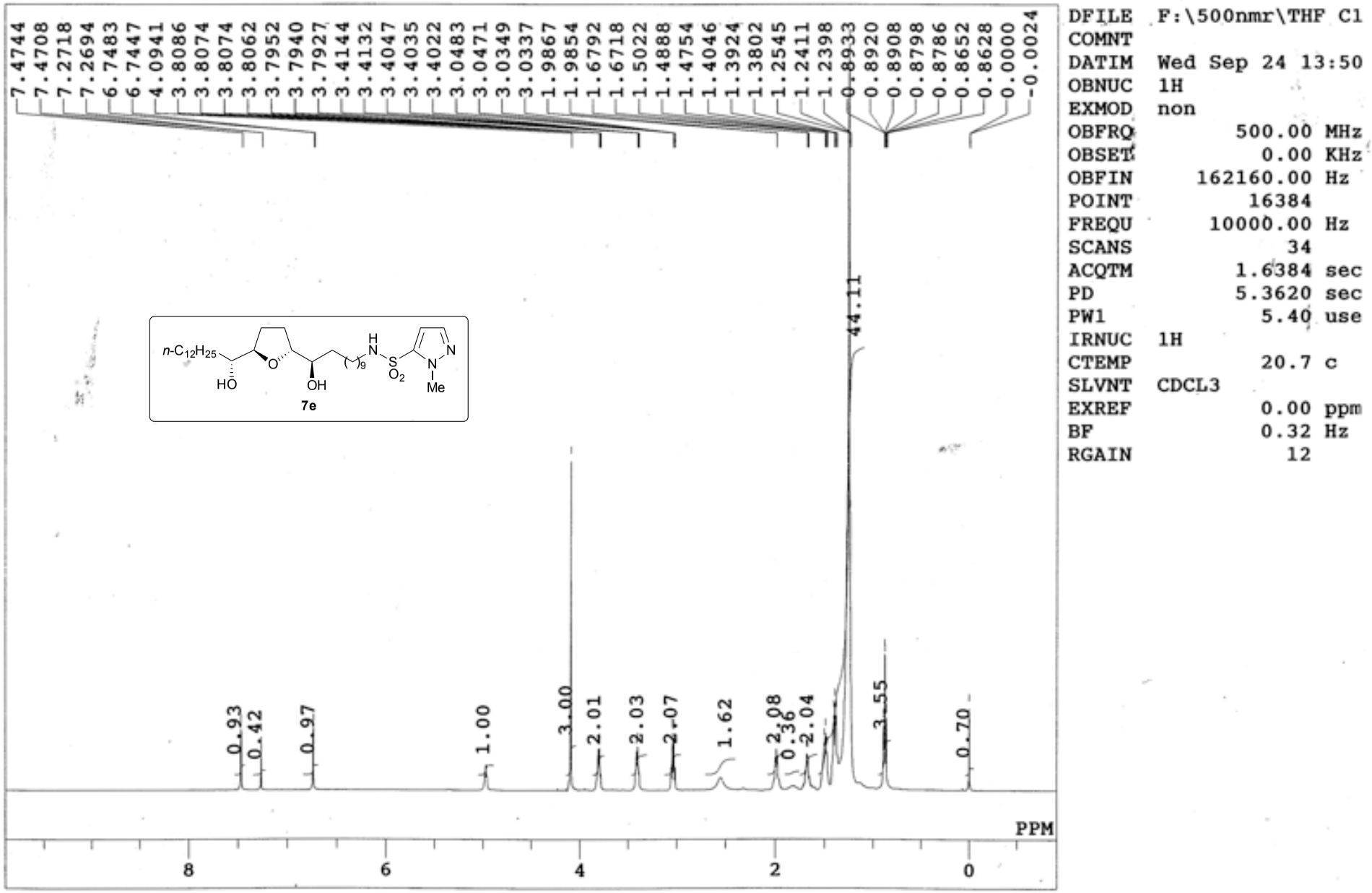
PPM
25
50
75
100
125
150
175
200
7d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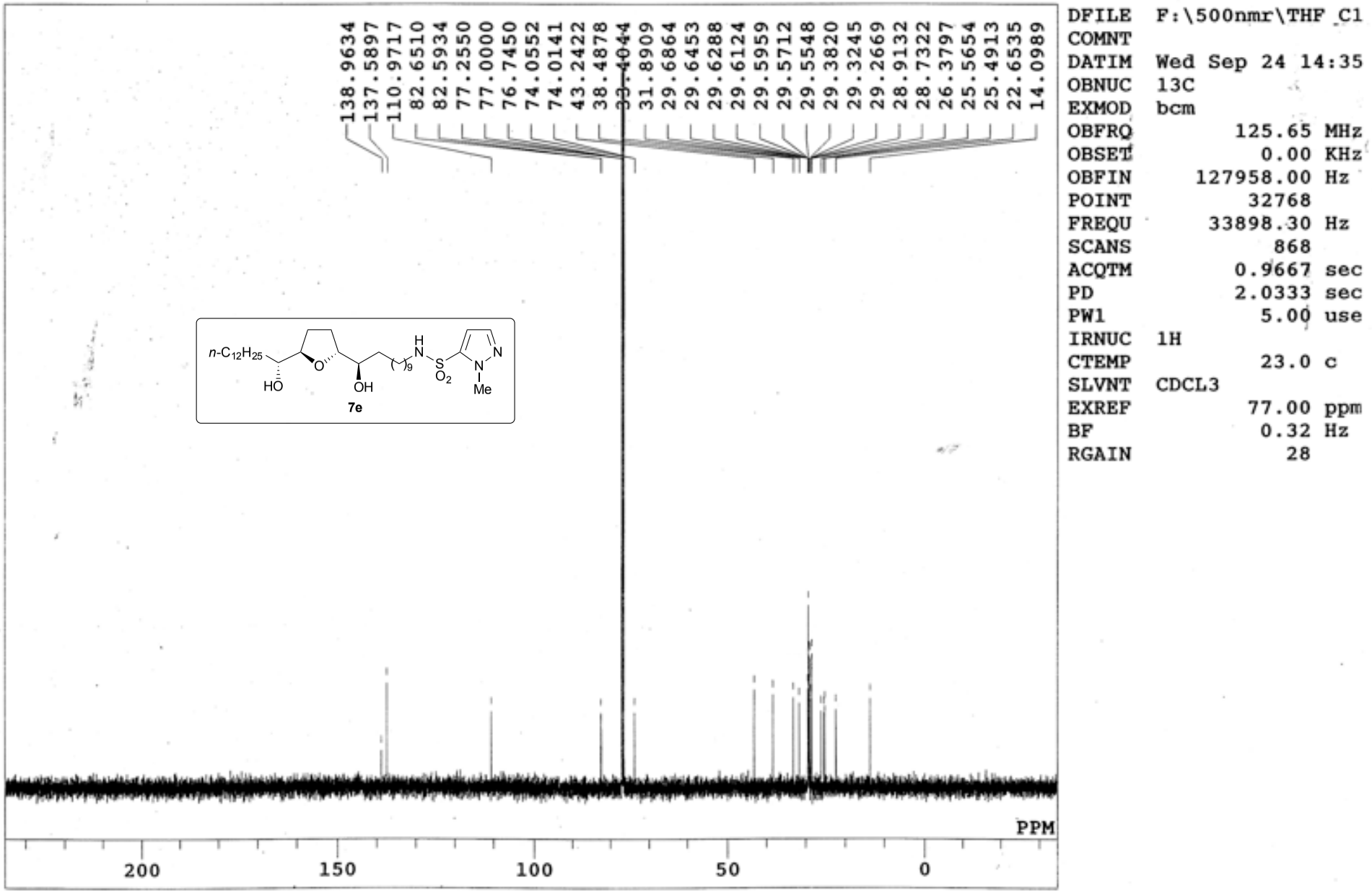
3.8062
3.7952
3.7940
3.7927
3.4144
3.4132
3.4047
3.4035
3.4022
3.0483
3.0471
3.0349
3.0337
1.9867
1.9854
1.6792
1.6718
1.5022
1.4888
1.4754
1.4046
1.3924
1.3802
1.2545
1.2411
1.2398
0.8933
0.8920
0.8908
0.8798
0.8786
0.8652
0.8628
0.0000
-0.0024
44.11
0.93
0.42
0.97
1.00
3.00
2.01
2.03
2.07
1.62
2.08
0.36
2.04
3.55
0.70
PPM
8
6
4
2
0
n-C12H25
HO
O
OH
H
N
9
S
O2
N
N
Me
7e
DFILE F:\500nmr\THF C1
COMNT
DATIM Wed Sep 24 13:50
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 34
ACQTM 1.6384 sec
PD 5.3620 sec
PW1 5.40 use
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.32 Hz
RGAIN 12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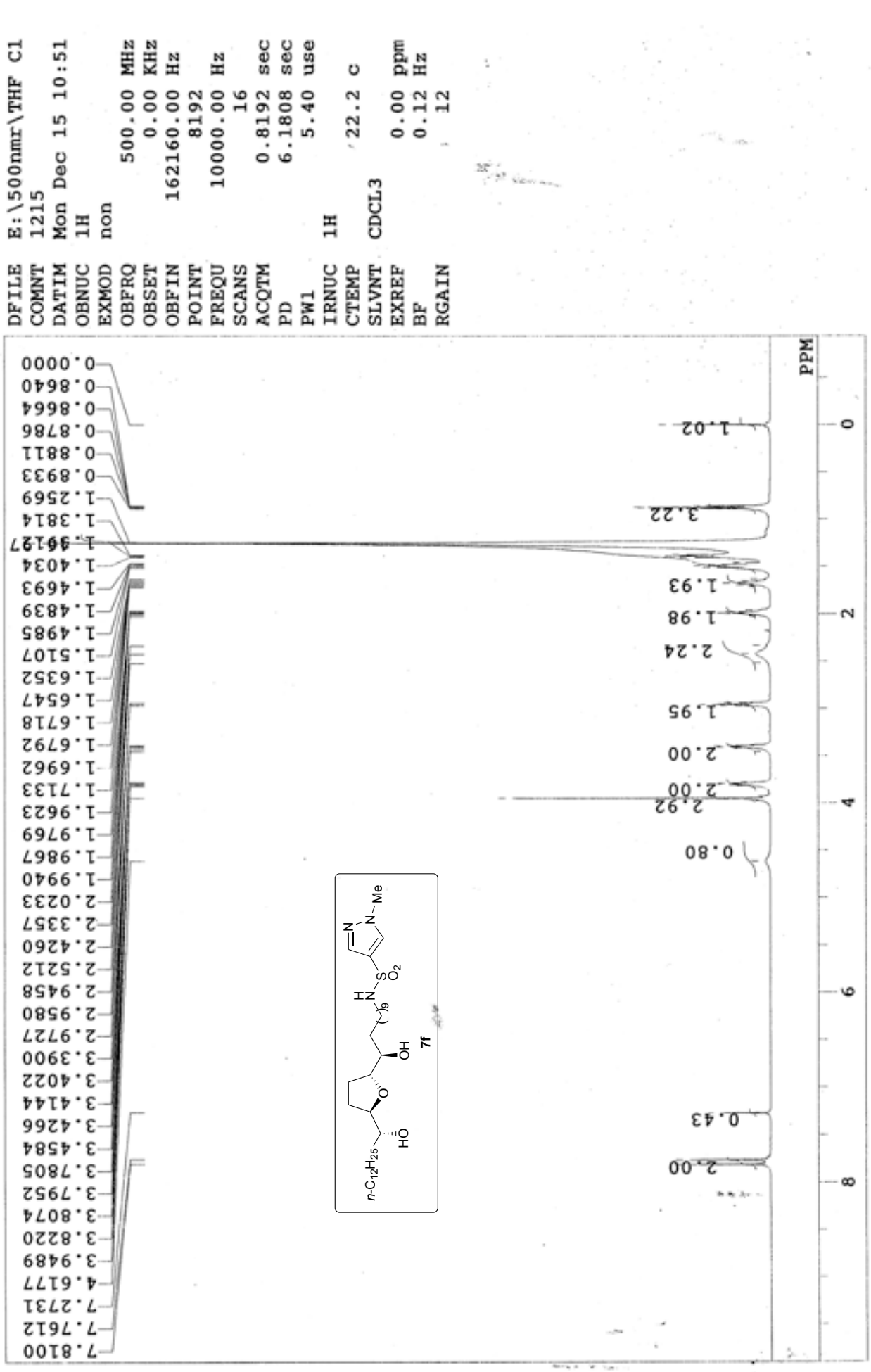
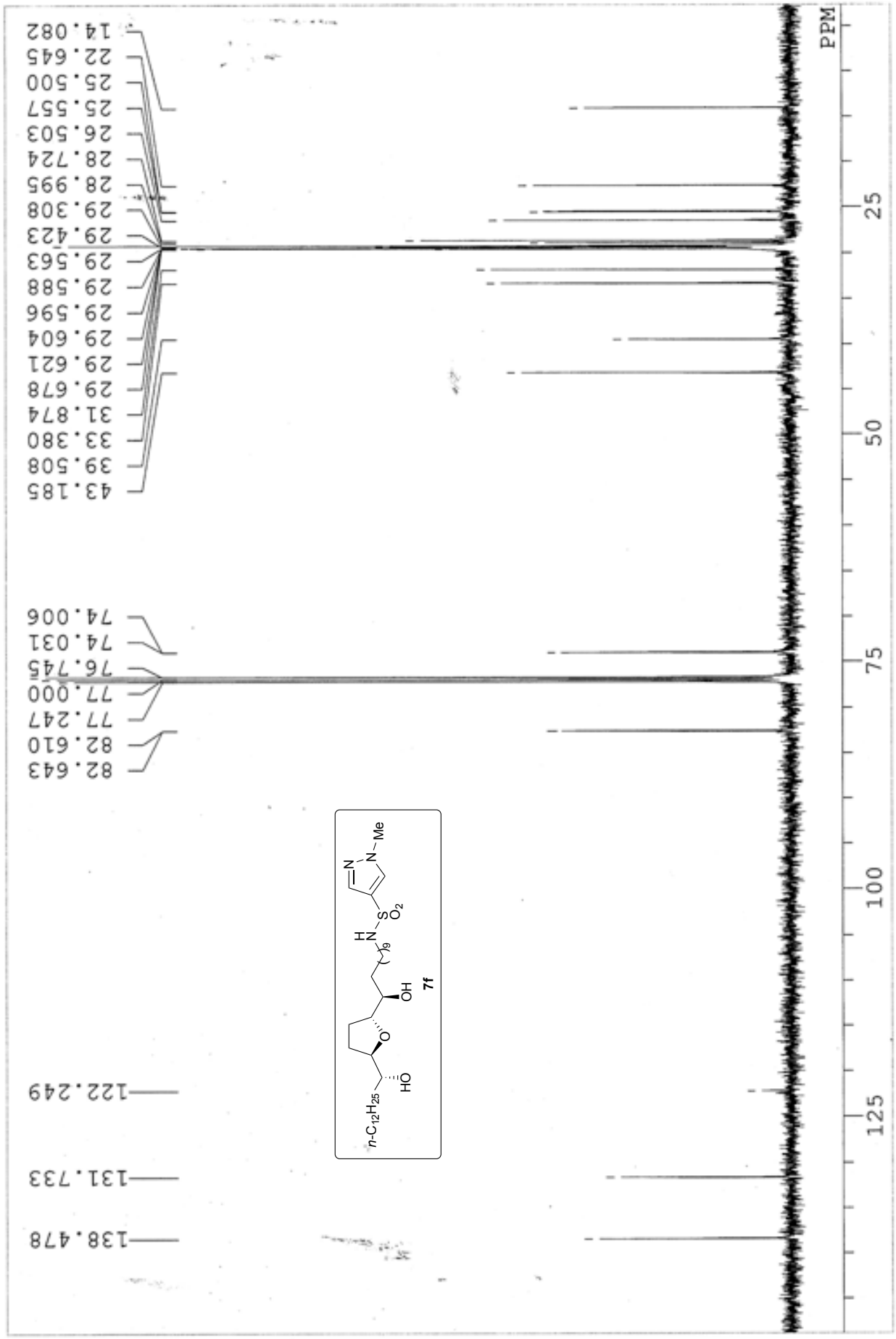
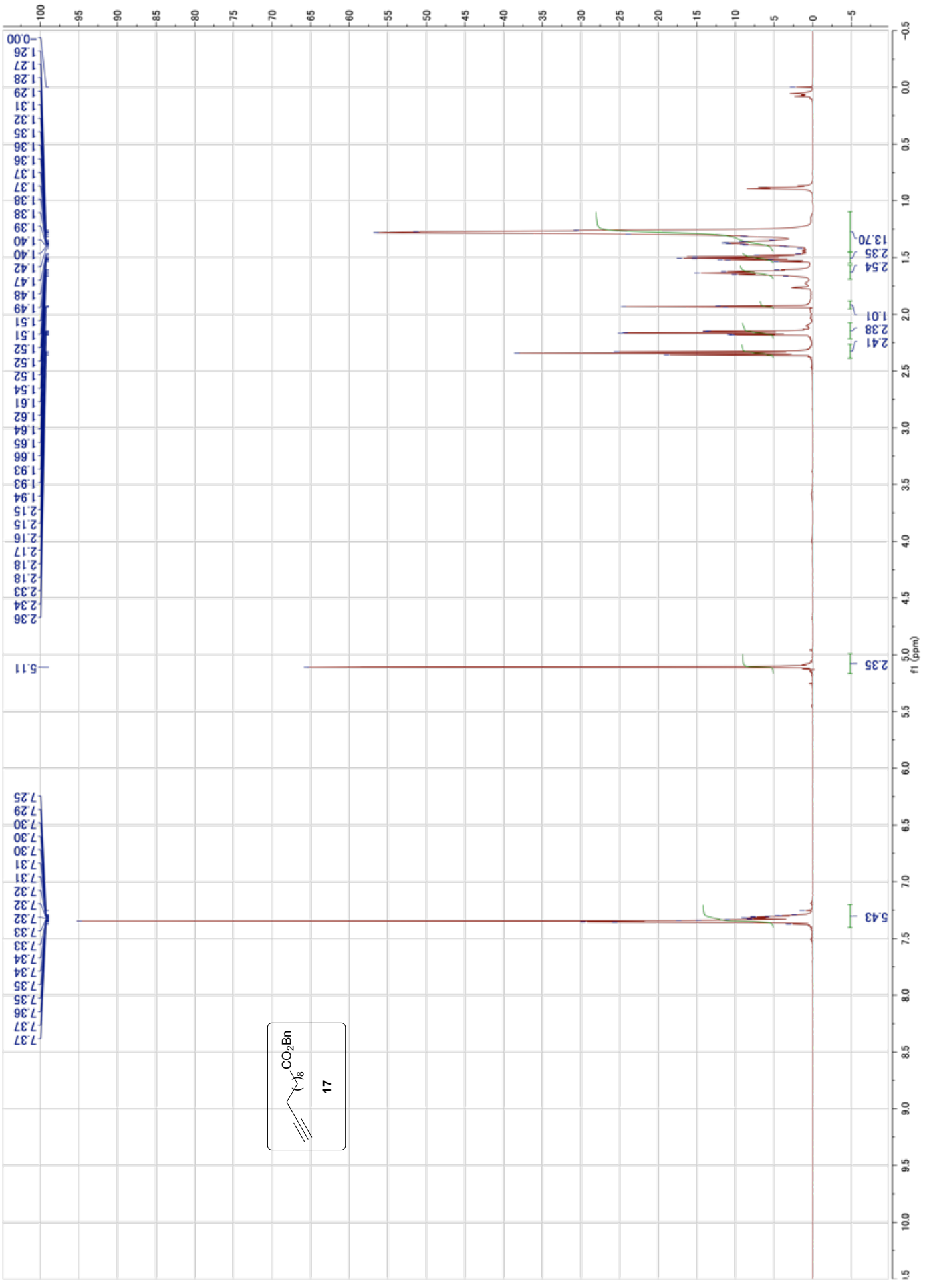
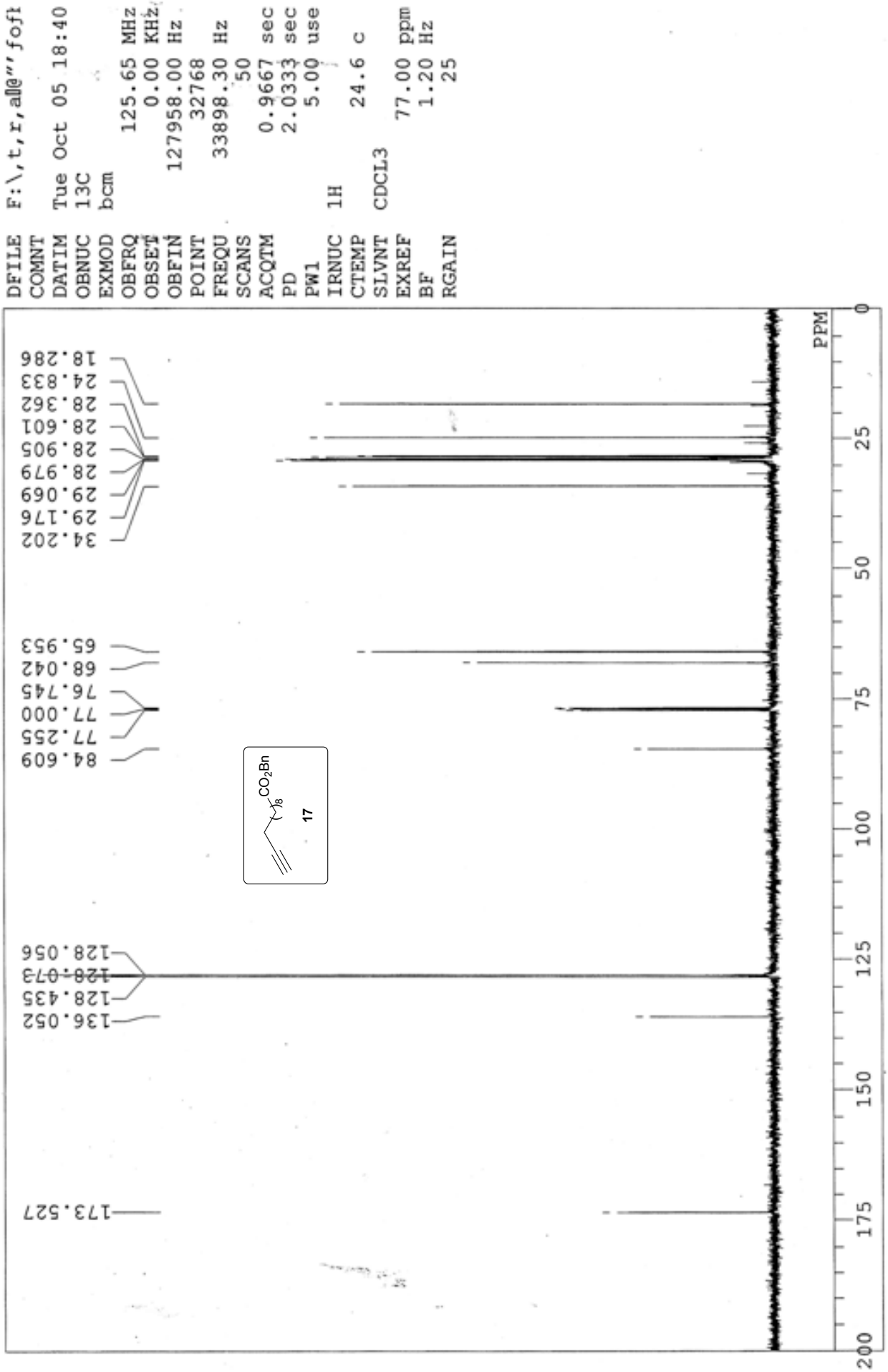
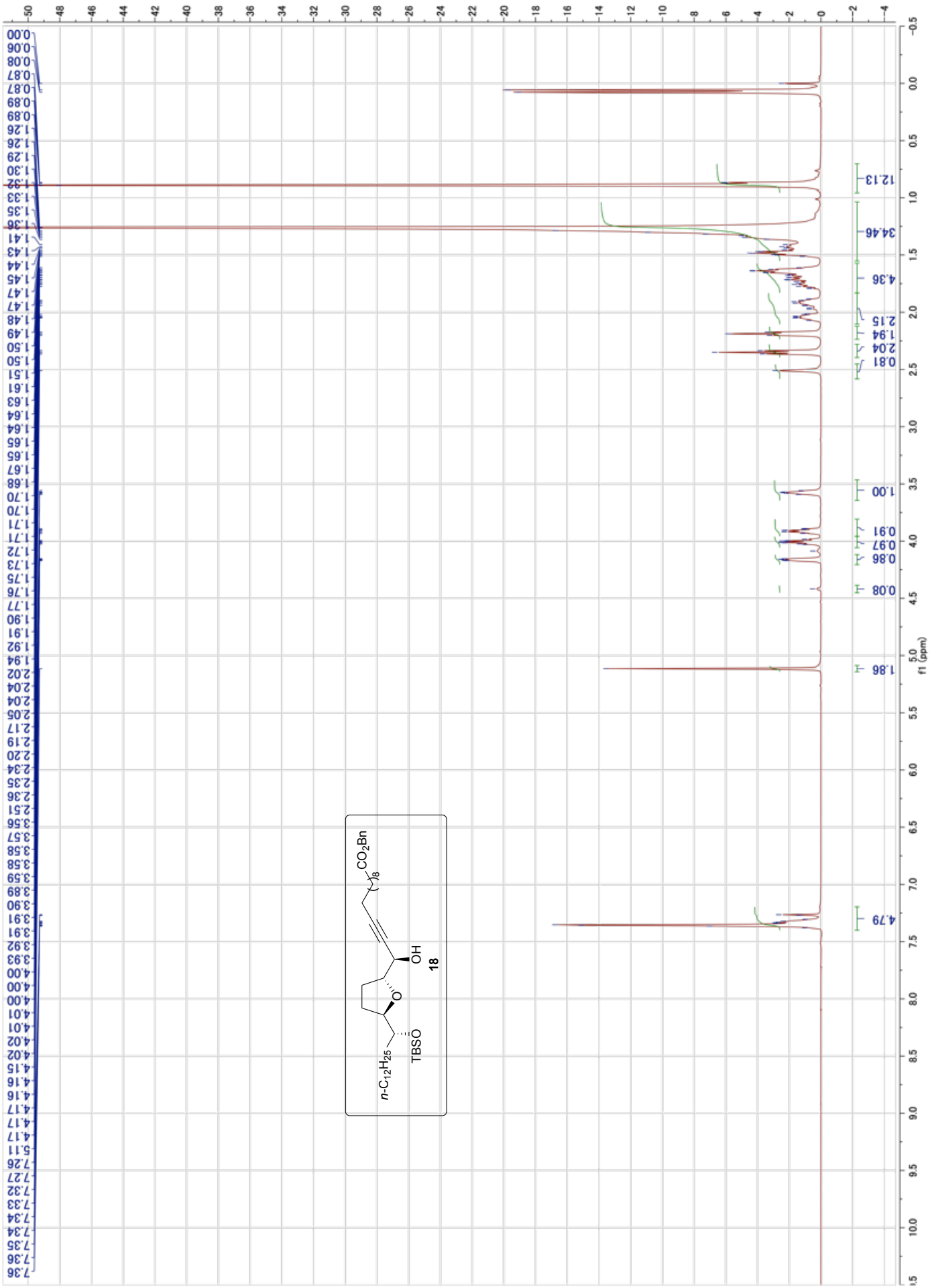
74.0552
74.0141
43.2422
38.4878
33.4044
31.8909
29.6864
29.6453
29.6288
29.6124
29.5959
29.5712
29.5548
29.3820
29.3245
29.2669
28.9132
28.7322
26.3797
25.5654
25.4913
22.6535
14.0989
n-C12H25
HO
O
OH
H
N
(CH2)9
S
O2
N
N
Me
7e
DFILE F:\500nmr\THF_C1
COMNT
DATIM Wed Sep 24 14:35
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 868
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.32 Hz
RGAIN 28
PPM
200
150
100
50
0

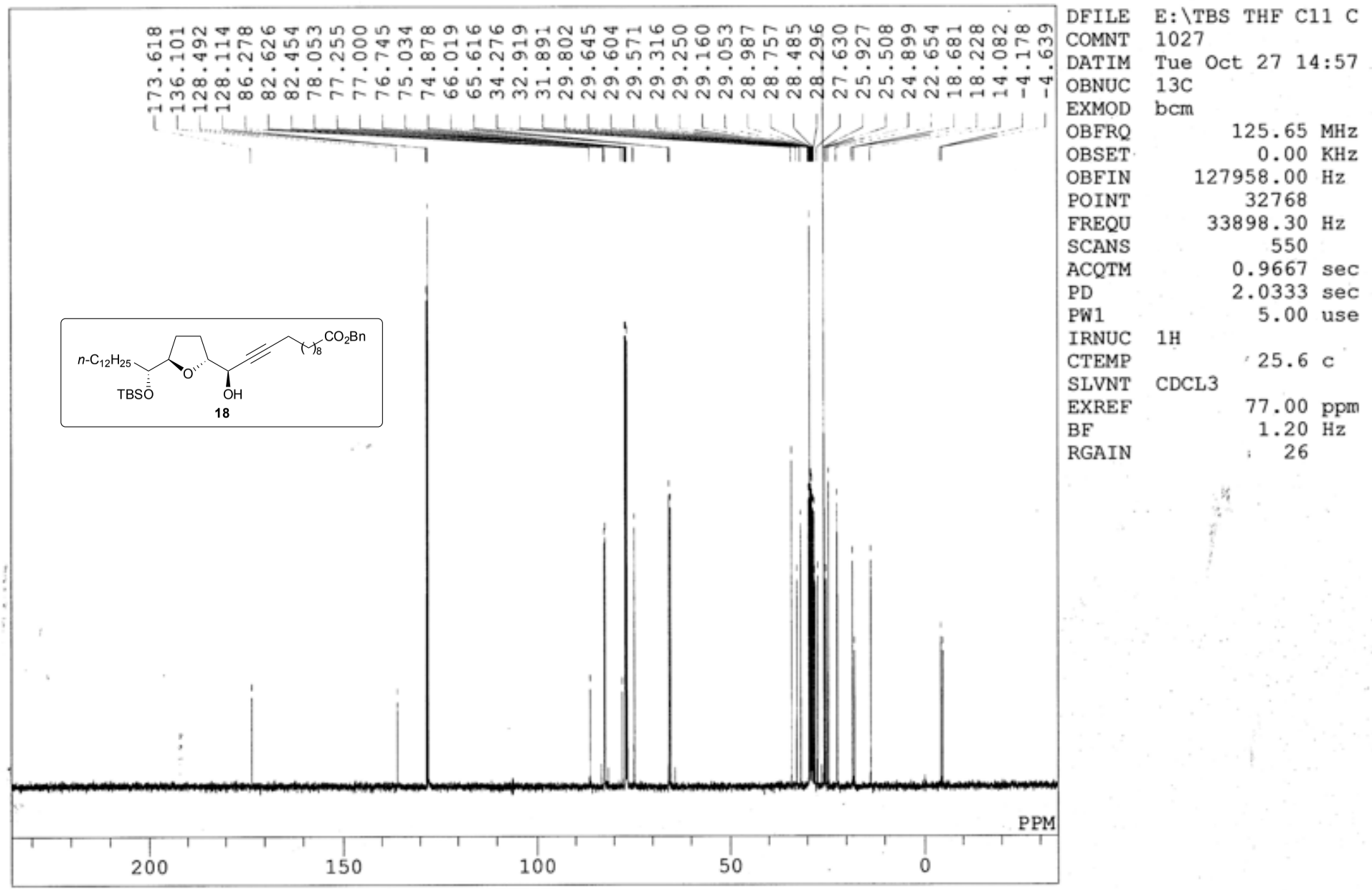

DFILE E:\500nmr\THF C1
COMNT 1215
DATIM Mon Dec 15 10:51
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 8192
FREQU 10000.00 Hz
SCANS 16
ACQTM 0.8192 sec
PD 6.1808 sec
PW1 5.40 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12
PPM
0
2
4
6
8
1.02
3.22
1.93
1.98
2.24
1.95
2.00
2.00
2.92
0.80
0.43
2.00
7f

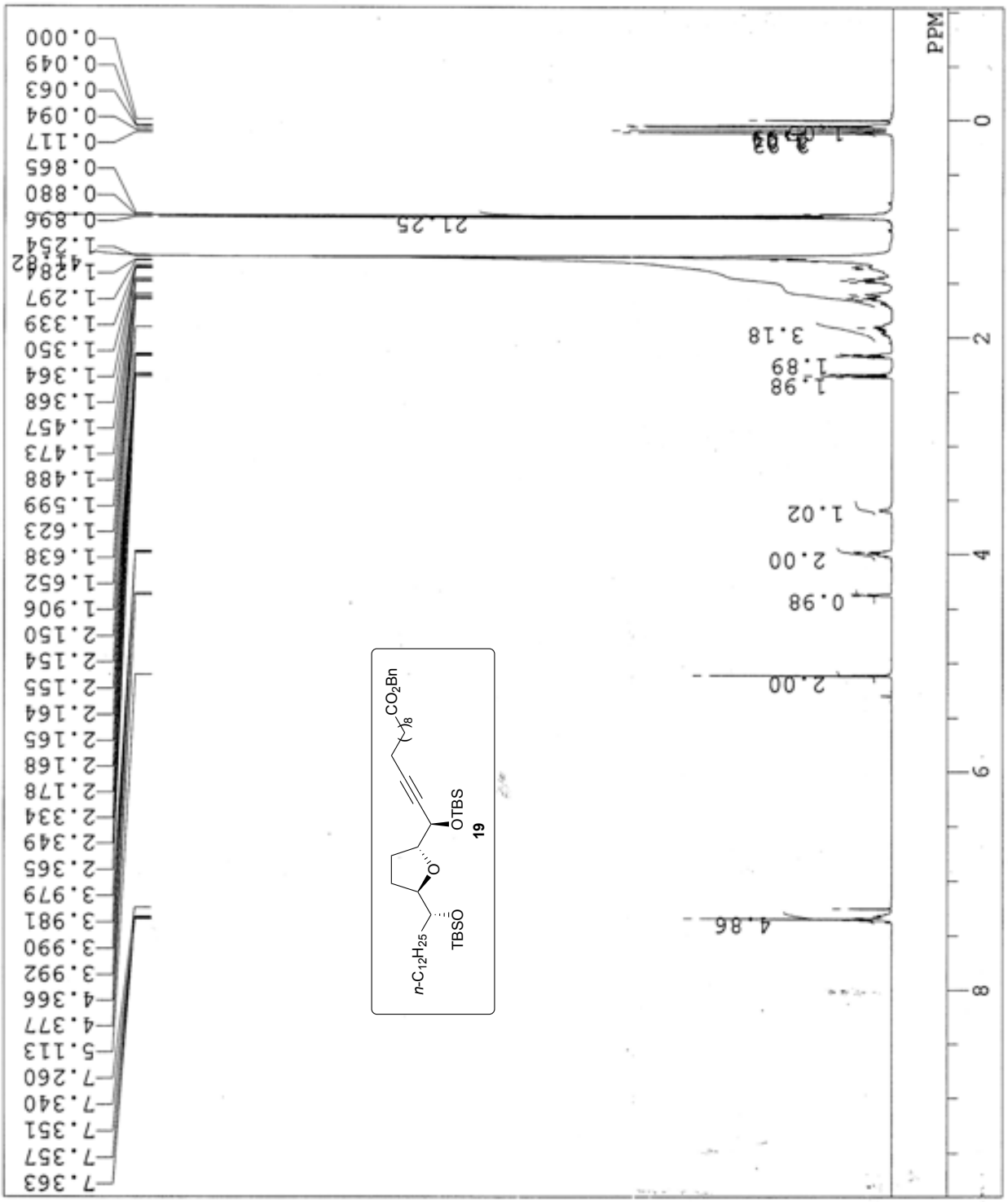

14.082
22.645
25.500
25.557
26.503
28.724
28.995
29.308
29.423
29.563
29.588
29.596
29.604
29.621
29.678
31.874
33.380
39.508
43.185
74.006
74.031
76.745
77.000
77.247
82.610
82.643
122.249
131.733
138.478
PPM
25
50
75
100
125
7f

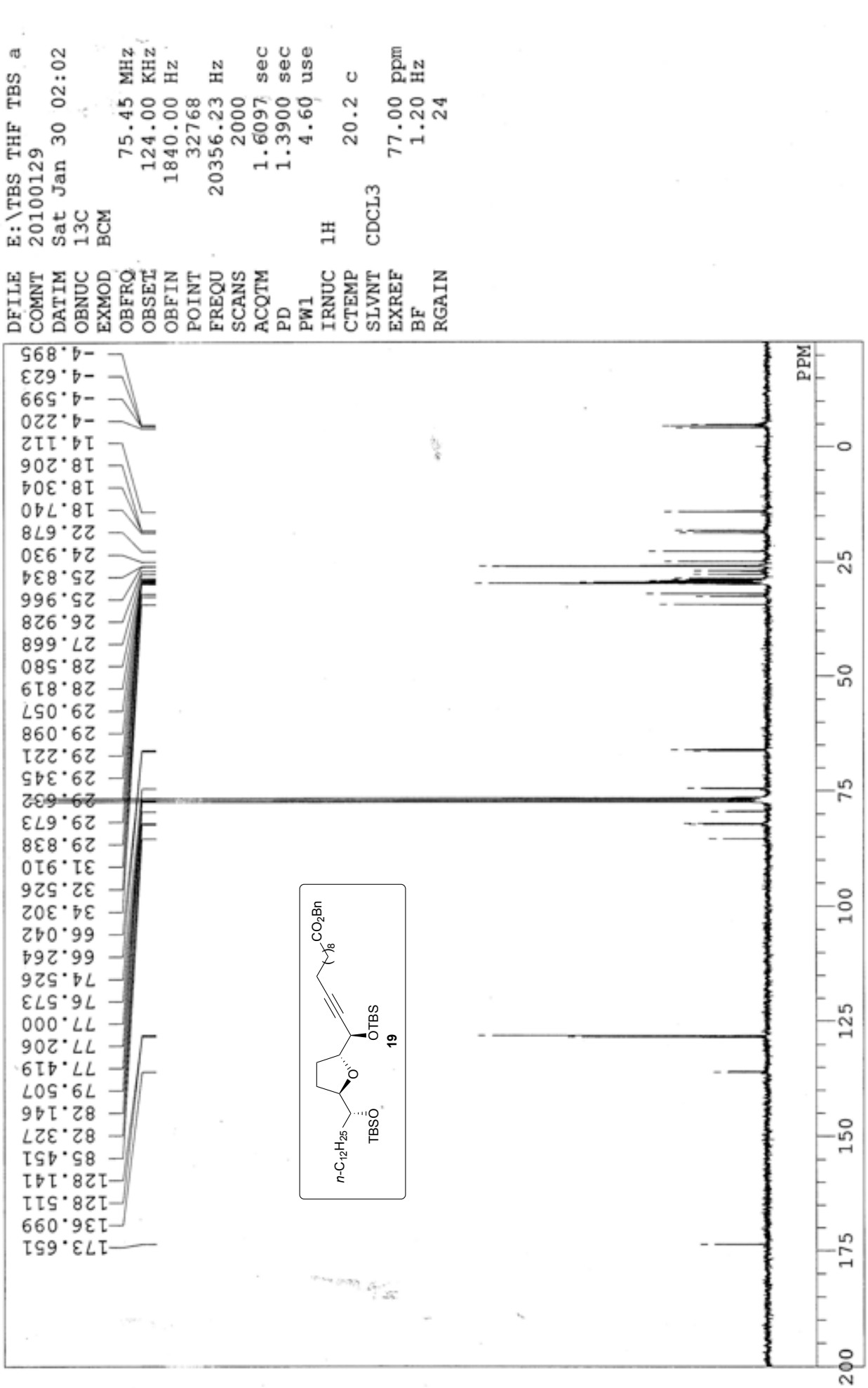

CO2Bn
8
17
f1 (ppm)

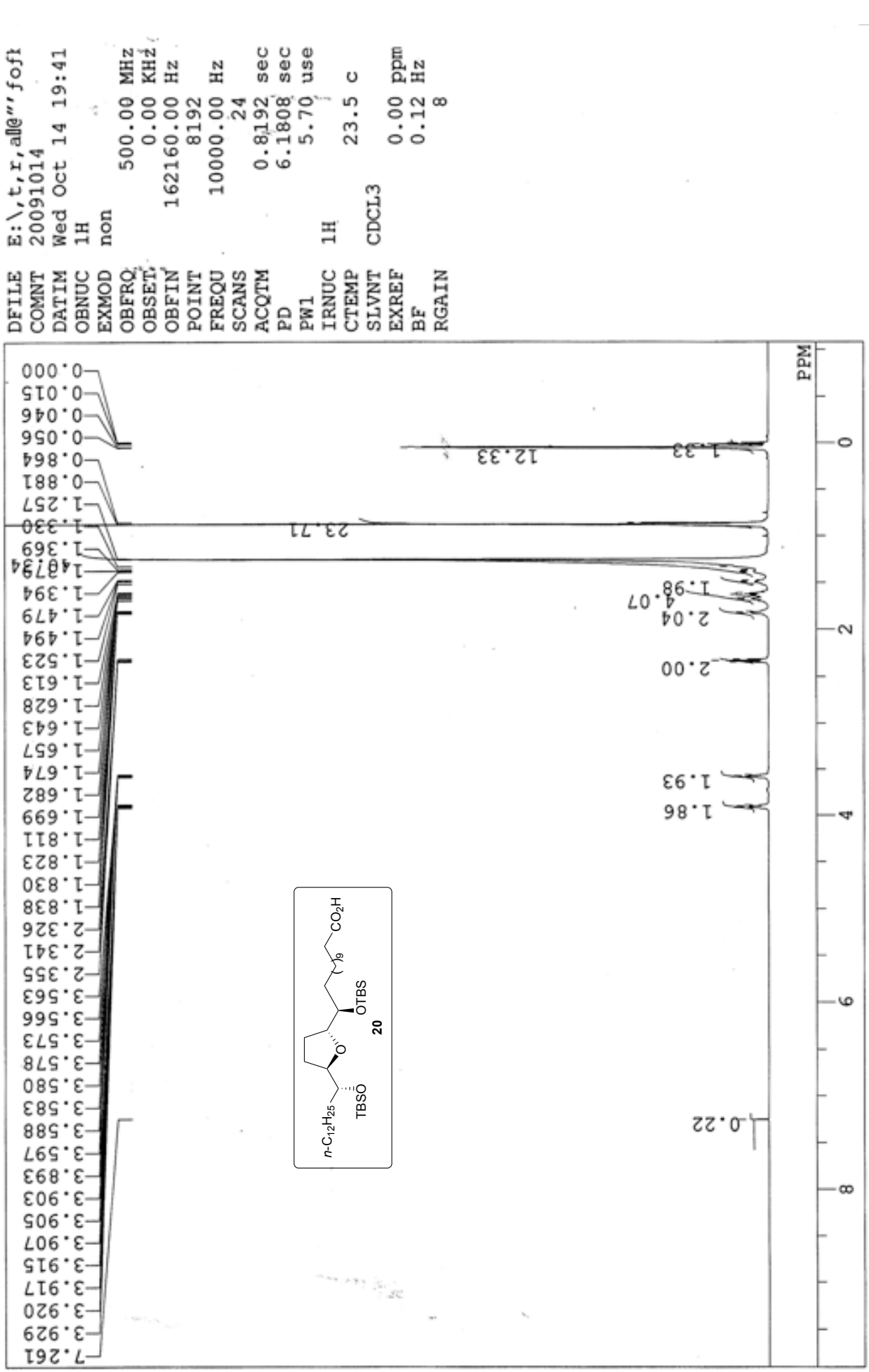

DFILE F:\,t,r,a@"'fofl
COMNT
DATIM Tue Oct 05 18:40
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 50
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 24.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
18.286
24.833
28.362
28.601
28.905
28.979
29.069
29.176
34.202
65.953
68.042
76.745
77.000
77.255
84.609
128.056
128.073
128.435
136.052
173.527
CO2Bn
8
17
PPM
0
25
50
75
100
125
150
175
200

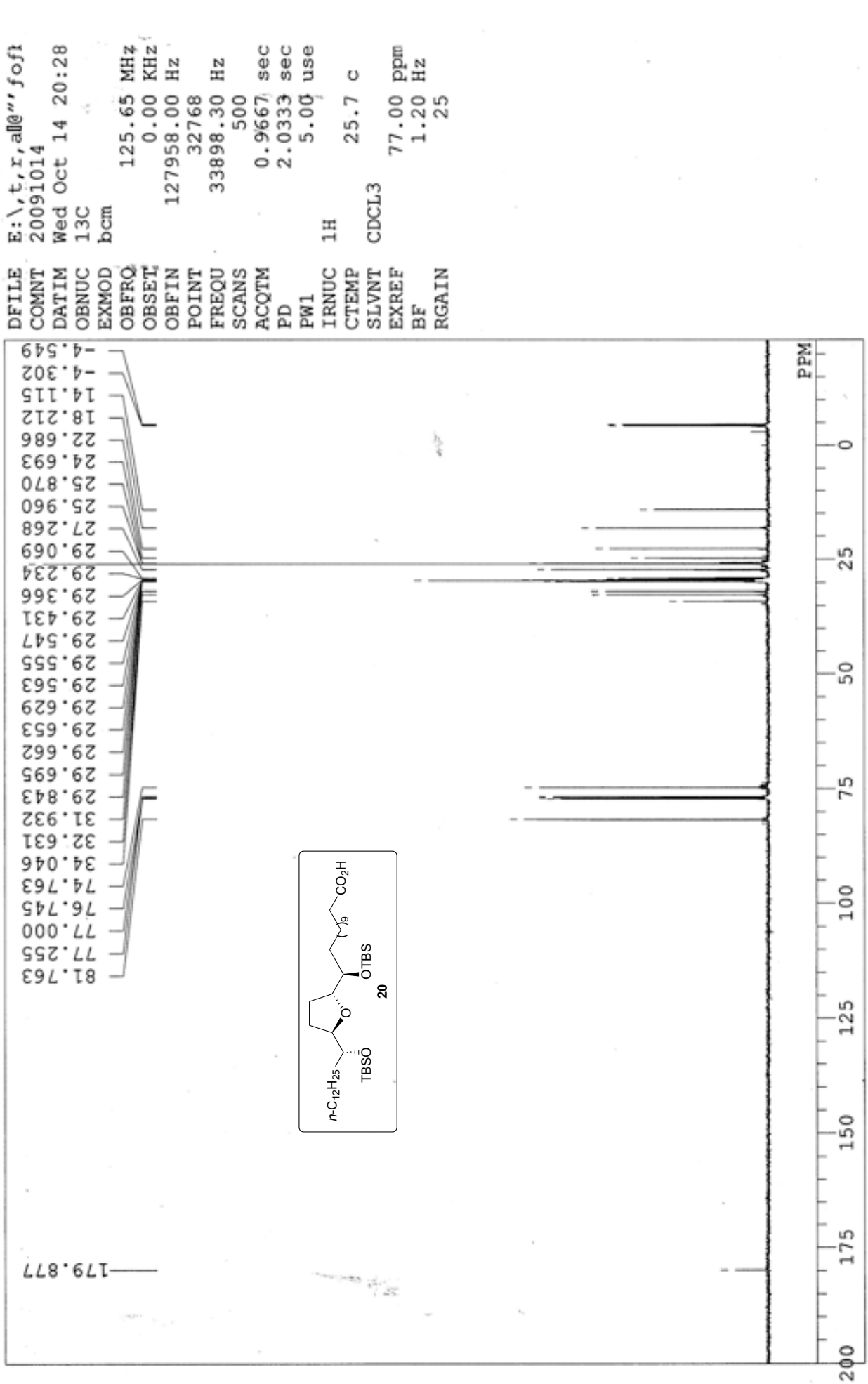

CO2Bn
8
OH
18
O
TBSO
n-C12H25
12.13
34.46
4.36
2.15
1.94
2.04
0.81
1.00
0.91
0.97
0.86
0.08
1.86
4.79
f1 (ppm)

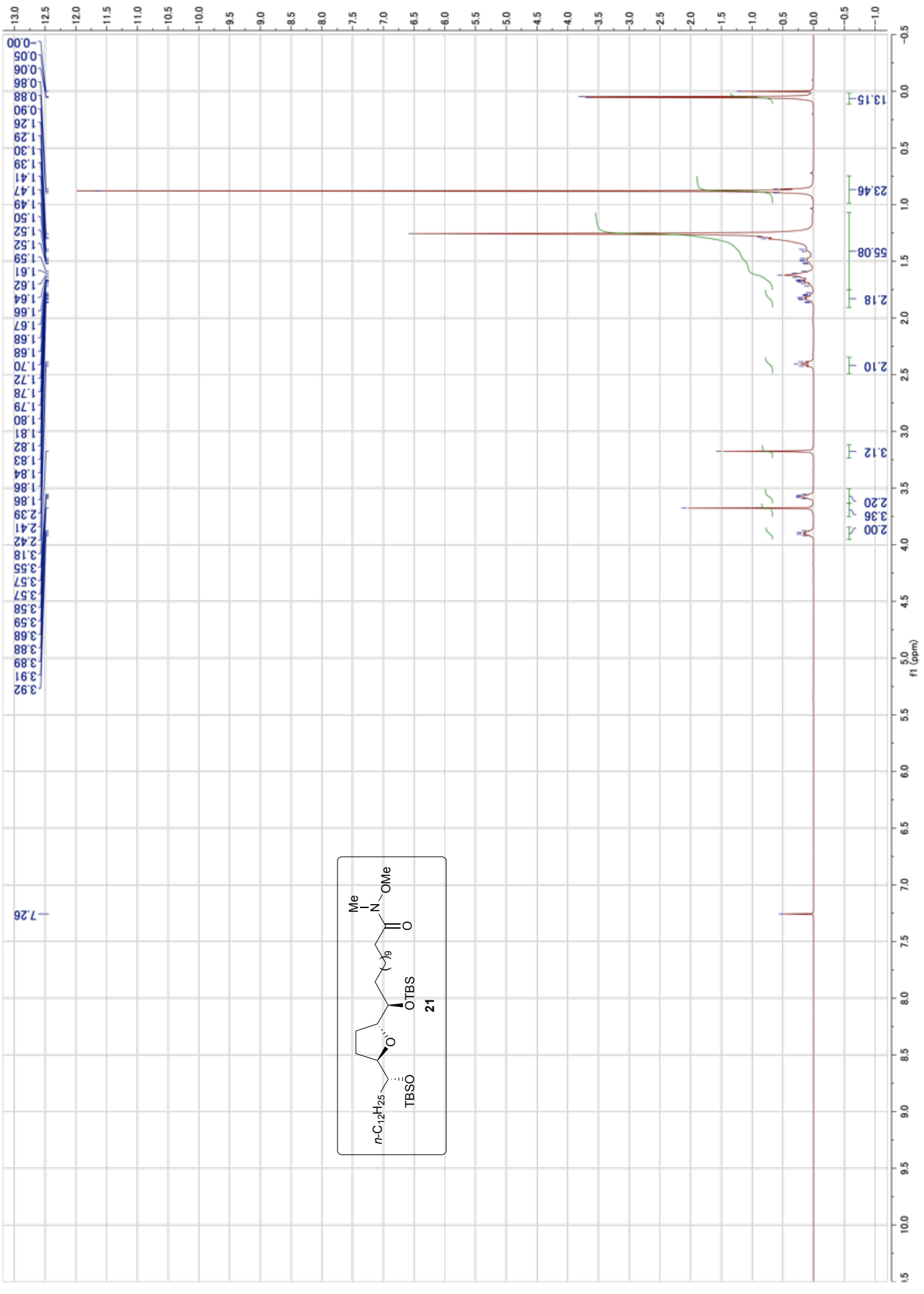

173.618
136.101
128.492
128.114
86.278
82.626
82.454
78.053
77.255
77.000
76.745
75.034
74.878
66.019
65.616
34.276
32.919
31.891
29.802
29.645
29.604
29.571
29.316
29.250
29.160
29.053
28.987
28.757
28.485
28.296
27.630
25.927
25.508
24.899
22.654
18.681
18.228
14.082
-4.178
-4.639
n-C12H25
TBSO
O
OH
CO2Bn
8
18
PPM
200
150
100
50
0
DFILE E:\TBS THF C11 C
COMNT 1027
DATIM Tue Oct 27 14:57
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 550
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 25.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26

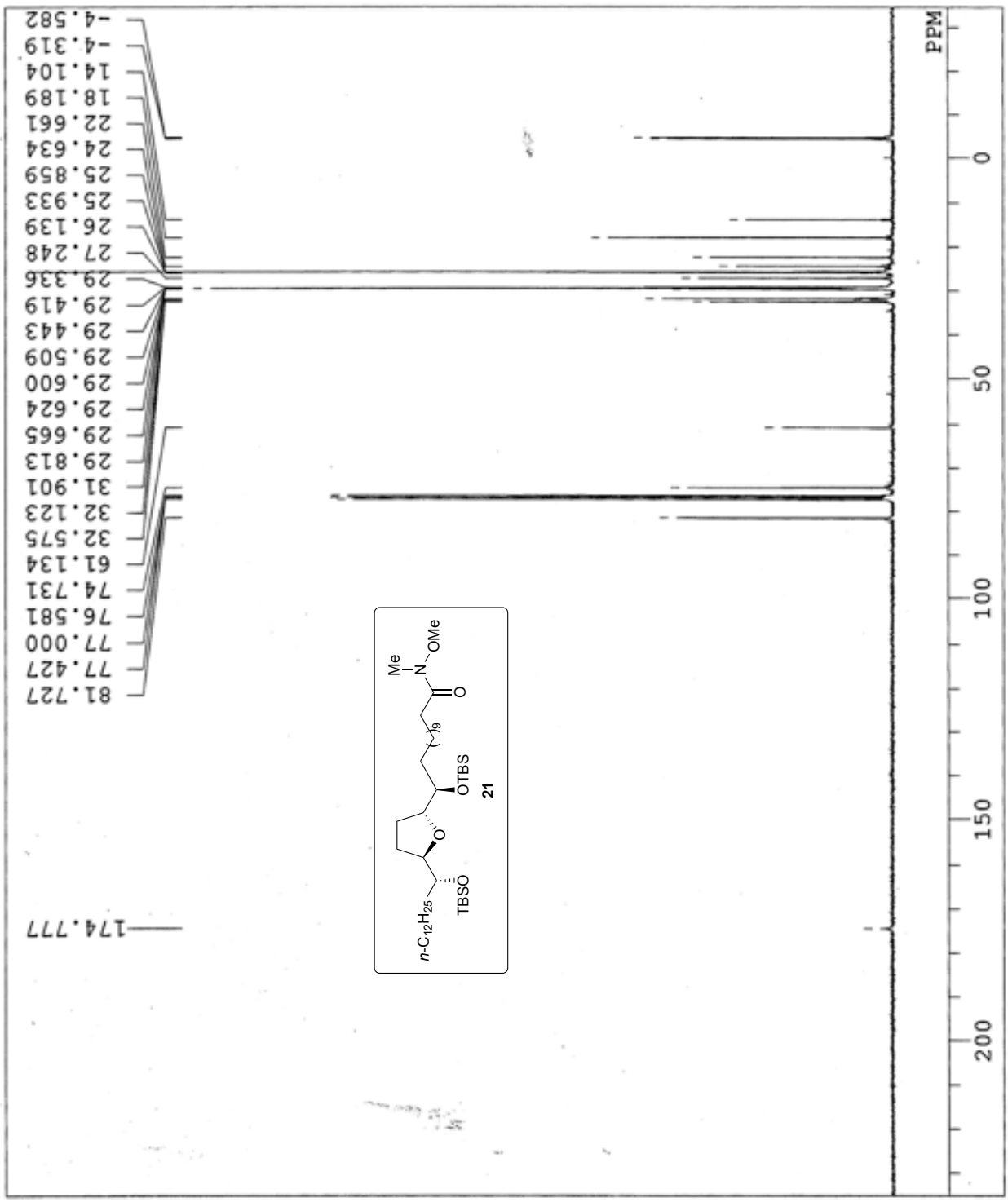

DFILE F:\0414\TBS THF
COMNT
DATIM Mon Aug 23 17:56
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 32
ACQTM 1.6384 sec
PD 5.3616 sec
PW1 5.70 use
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12
PPM
21.25
3.18
1.89
1.98
1.02
2.00
0.98
2.00
4.86
CO2Bn
8
OTBS
19
n-C12H25
TBSO

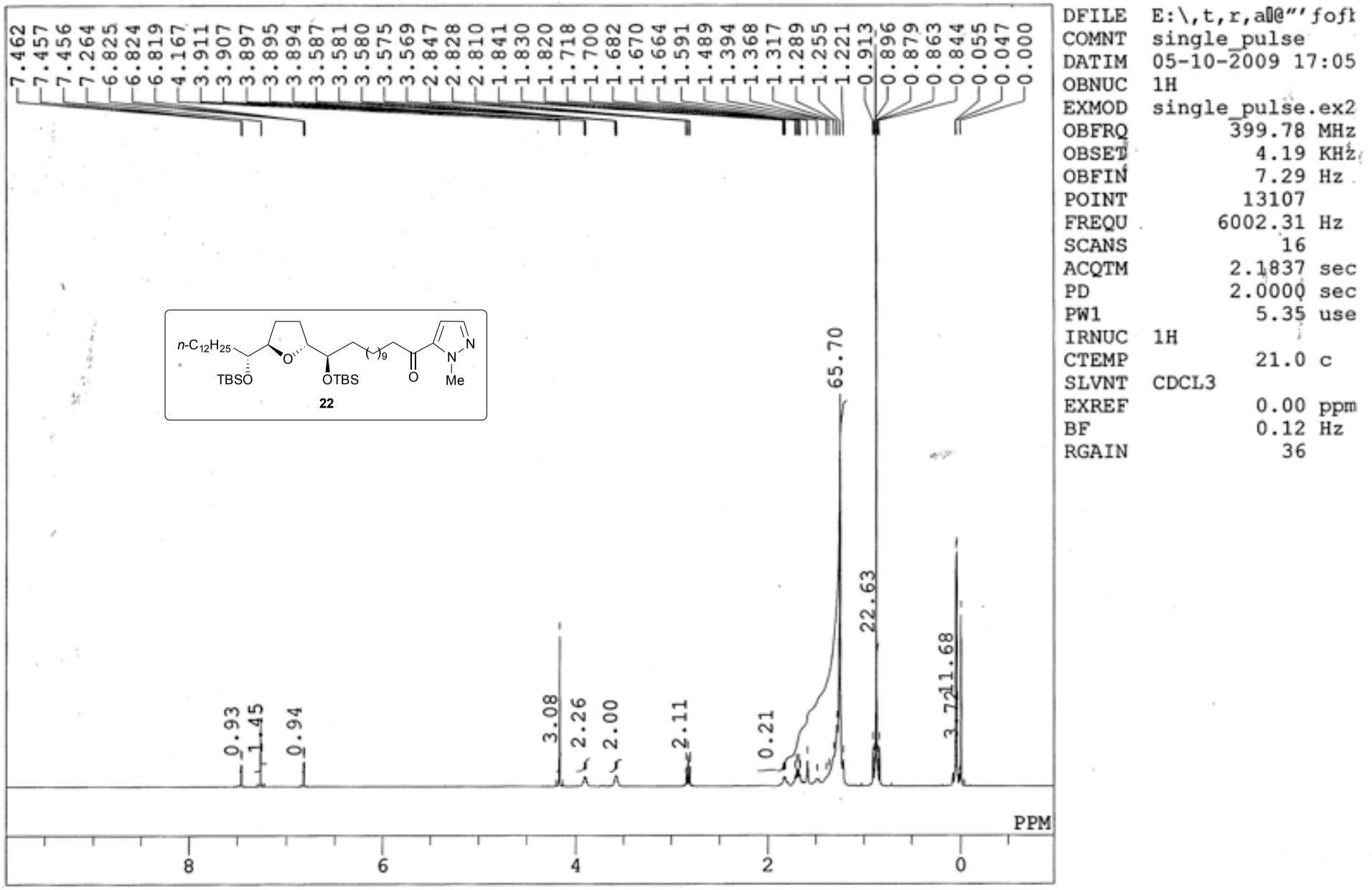

DFILE E:\TBS THF TBS a
COMNT 20100129
DATIM Sat Jan 30 02:02
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 2000
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 4.60 use
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24
-4.895
-4.623
-4.599
-4.220
14.112
18.206
18.304
18.740
22.678
24.930
25.834
25.966
26.928
27.668
28.580
28.819
29.057
29.098
29.221
29.345
29.632
29.673
29.838
31.910
32.526
34.302
66.042
66.264
74.526
76.573
77.000
77.206
77.419
79.507
82.146
82.327
85.451
128.141
128.511
136.099
173.651
PPM
0
25
50
75
100
125
150
175
200
CO_2Bn
8
OTBS
19
O
TBSO
n-$C_{12}H_{25}$

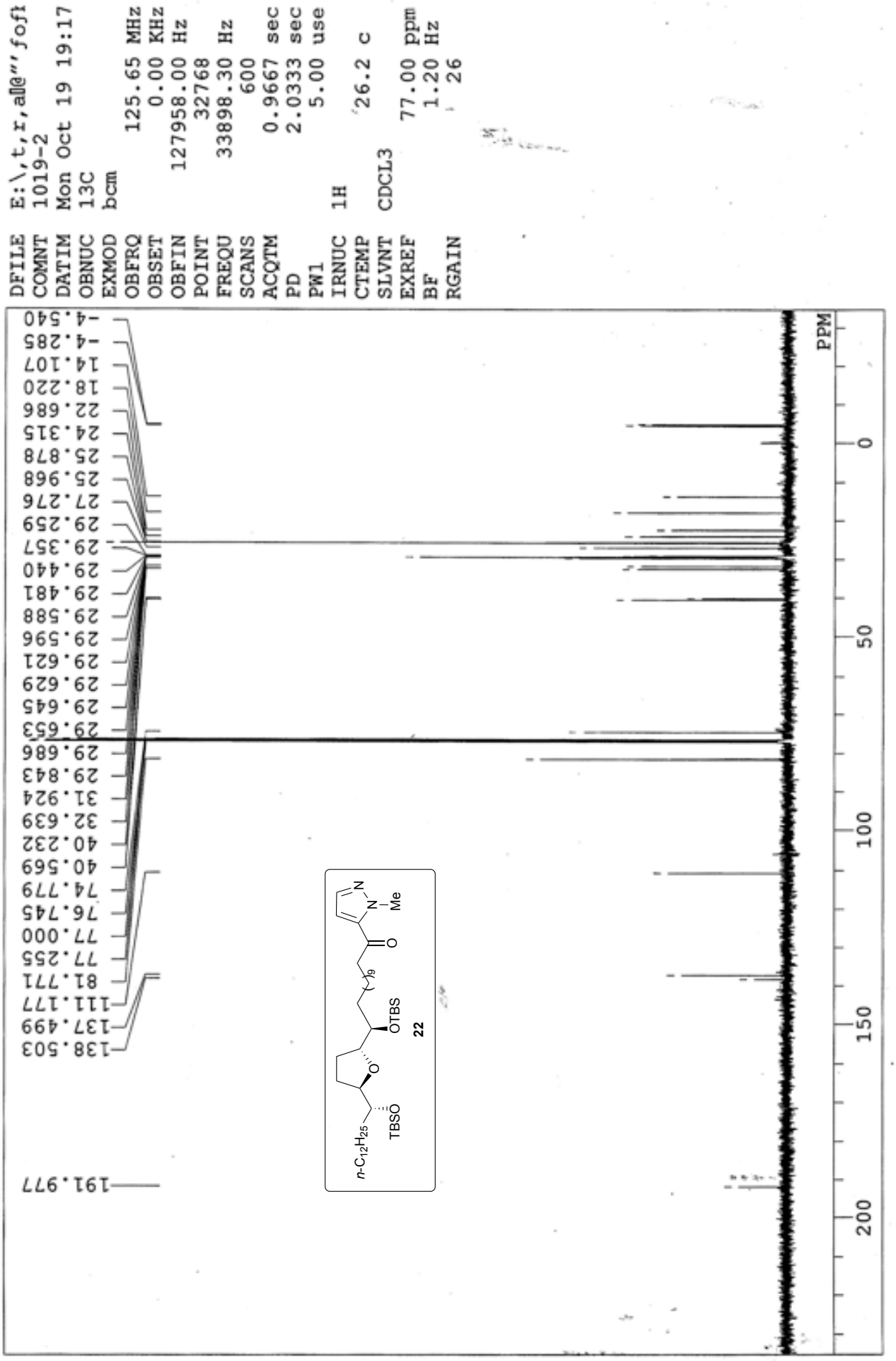

DFILE E:\,t,r,a@"'fofi
COMNT 20091014
DATIM Wed Oct 14 19:41
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 8192
FREQU 10000.00 Hz
SCANS 24
ACQTM 0.8192 sec
PD 6.1808 sec
PW1 5.70 use
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 8
PPM
0.22
1.86
1.93
2.00
2.04
4.07
1.98
23.71
12.33
1.33
20
OTBS
TBSO
CO2H
n-C12H25

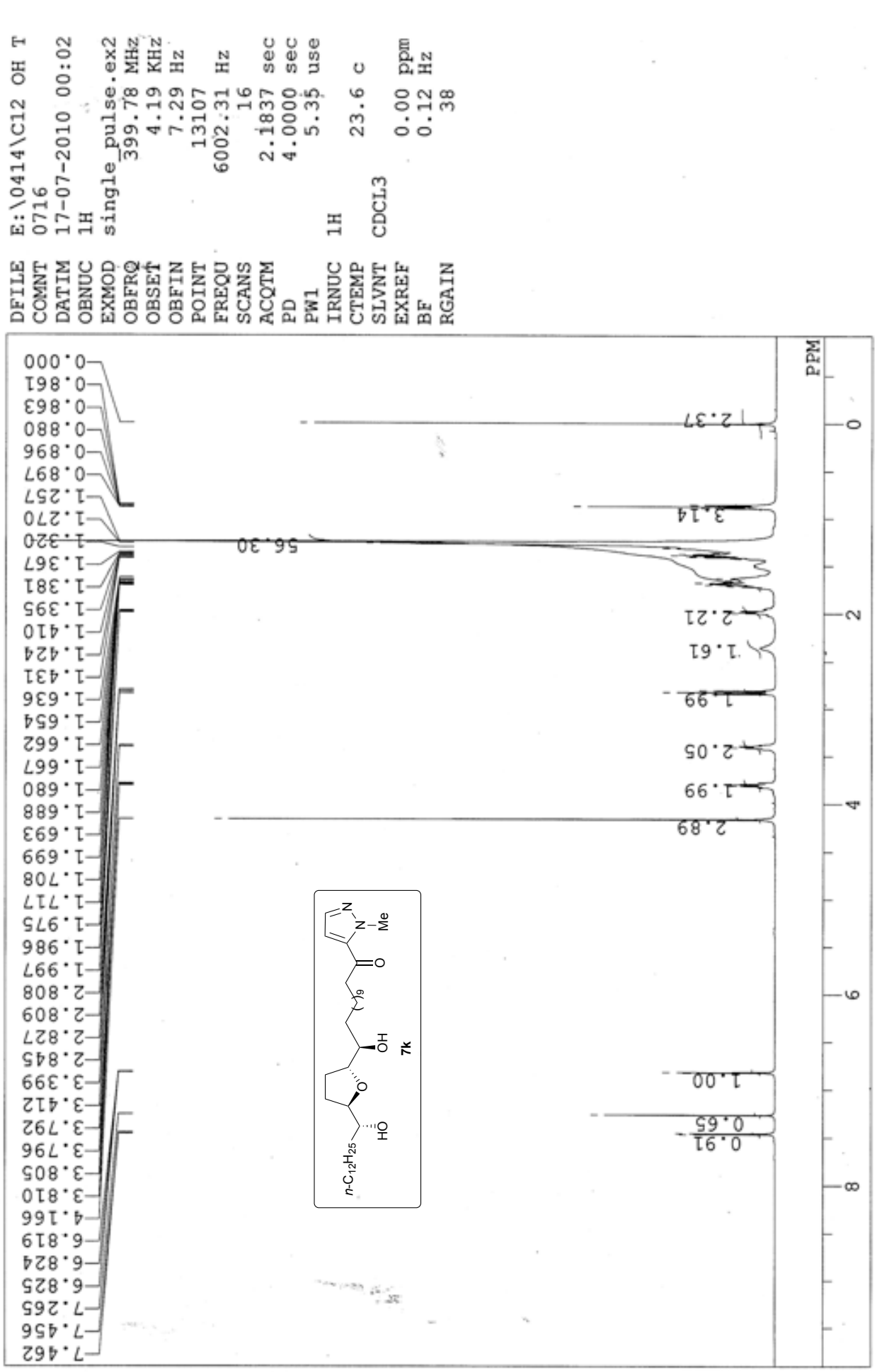

DFILE E:\,t,r,a@"'fofi
COMNT 20091014
DATIM Wed Oct 14 20:28
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 500
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 25.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
179.877
81.763
77.255
77.000
76.745
74.763
34.046
32.631
31.932
29.843
29.695
29.662
29.653
29.629
29.563
29.555
29.547
29.431
29.366
29.234
29.069
27.268
25.960
25.870
24.693
22.686
18.212
14.115
-4.302
-4.549
PPM
200
175
150
125
100
75
50
25
0
TBSO
OTBS
O
CO_2H
9
n-$C_{12}H_{25}$
20

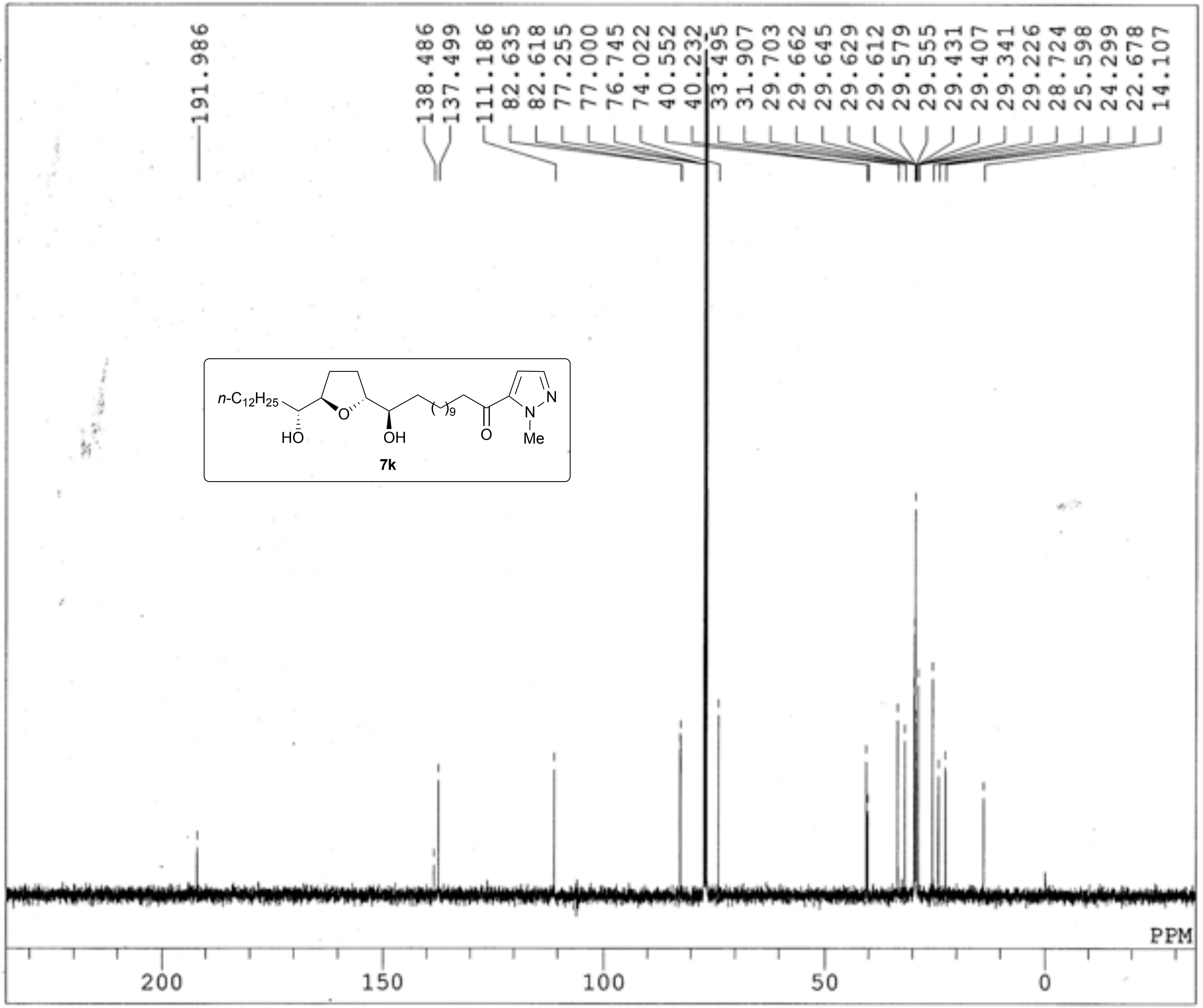

Me
N
OMe
O
9
OTBS
21
O
TBSO
n-C12H25
13.15
23.46
55.08
2.18
2.10
3.12
2.20
3.36
2.00
7.26
f1 (ppm)


DFILE E:\TBS THFTBS C1
COMNT 0123
DATIM Sat Jan 23 03:30
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 4000
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 4.60 use
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24
-4.582
-4.319
14.104
18.189
22.661
24.634
25.859
25.933
26.139
27.248
29.336
29.419
29.443
29.509
29.600
29.624
29.665
29.813
31.901
32.123
32.575
61.134
74.731
76.581
77.000
77.427
81.727
174.777
PPM
0
50
100
150
200
Me
N
OMe
O
9
OTBS
21
O
TBSO
n-C12H25

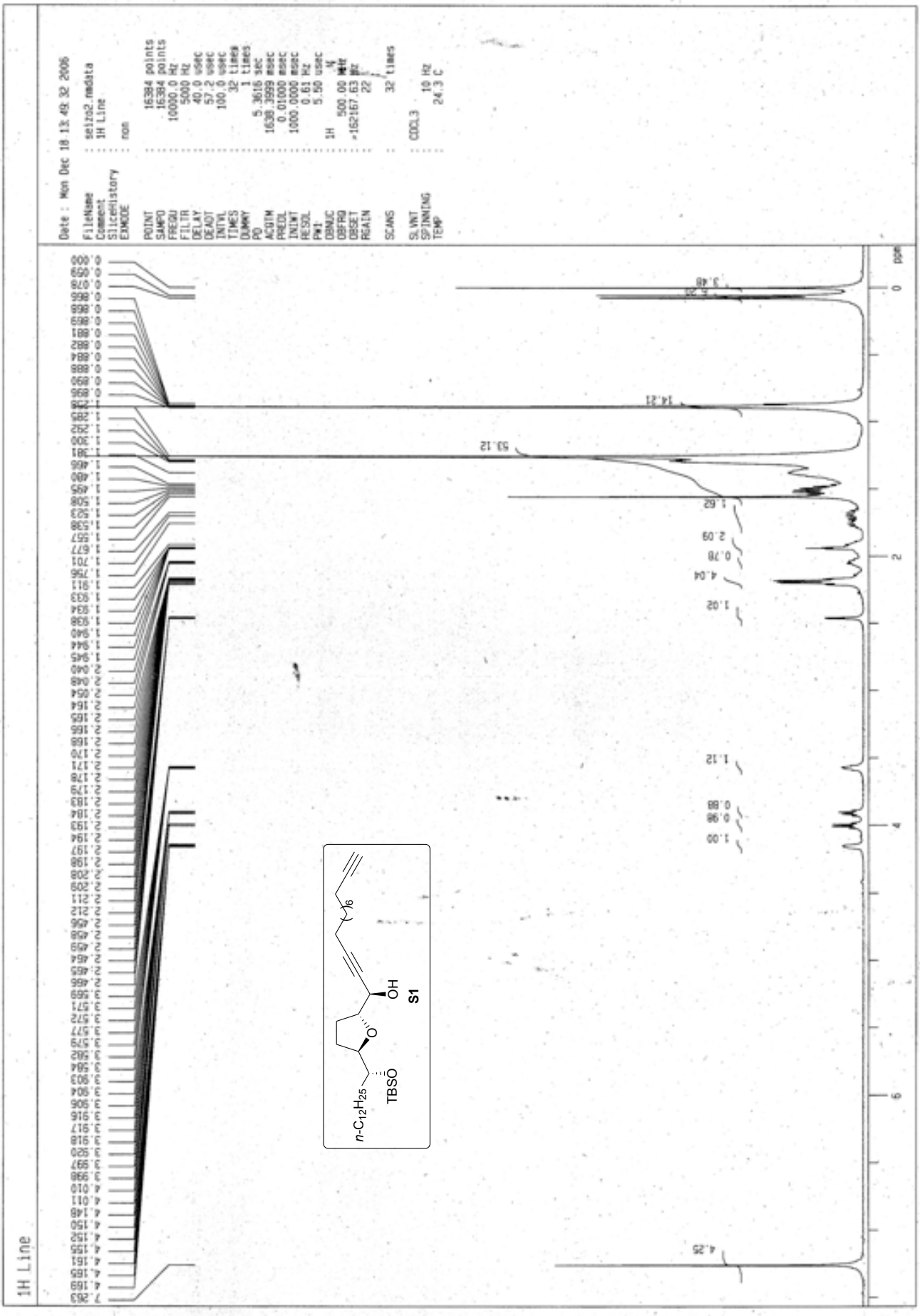

7.462
7.457
7.456
7.264
6.825
6.824
6.819
4.167
3.911
3.907
3.897
3.895
3.894
3.587
3.581
3.580
3.575
3.569
2.847
2.828
2.810
1.841
1.830
1.820
1.718
1.700
1.682
1.670
1.664
1.591
1.489
1.394
1.368
1.317
1.289
1.255
1.221
0.913
0.896
0.879
0.863
0.844
0.055
0.047
0.000
n-C12H25
TBSO
OTBS
O
N
N
Me
22
0.93
1.45
0.94
3.08
2.26
2.00
2.11
0.21
65.70
22.63
3.72
11.68
PPM
8
6
4
2
0
DFILE E:\,t,r,a@"'fofk
COMNT single_pulse
DATIM 05-10-2009 17:05
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.31 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 5.35 use
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

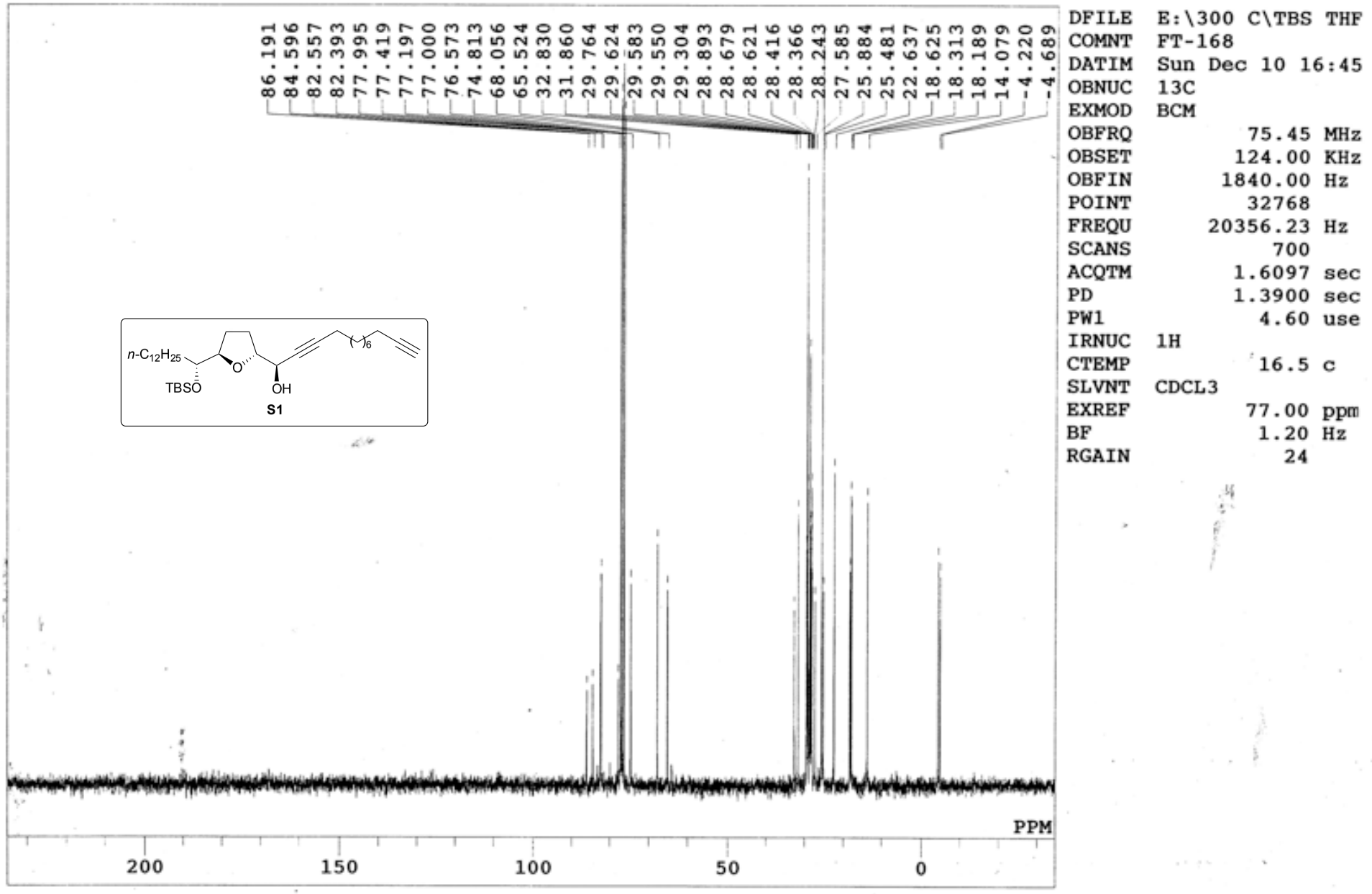

DFILE E:\,t,r,a@"'fofi
COMNT 1019-2
DATIM Mon Oct 19 19:17
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 600
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 26.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26
-4.540
-4.285
14.107
18.220
22.686
24.315
25.878
25.968
27.276
29.259
29.357
29.440
29.481
29.588
29.596
29.621
29.629
29.645
29.653
29.686
29.843
31.924
32.639
40.232
40.569
74.779
76.745
77.000
77.255
81.771
111.177
137.499
138.503
191.977
PPM
0
50
100
150
200
TBSO
OTBS
O
O
N
N
Me
9
n-C12H25
22

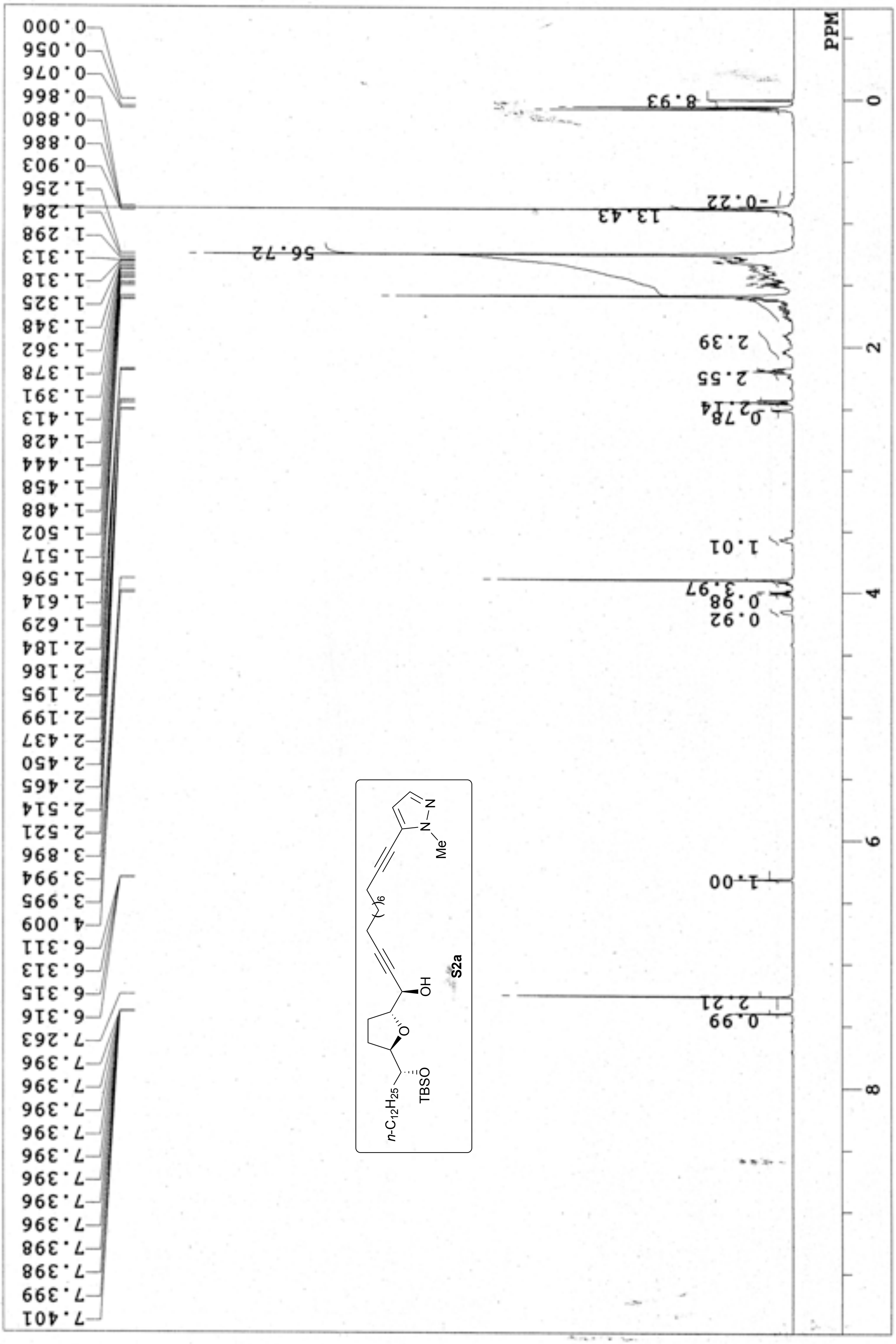

DFILE E:\0414\C12 OH T
COMNT 0716
DATIM 17-07-2010 00:02
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.31 Hz
SCANS 16
ACQTM 2.1837 sec
PD 4.0000 sec
PW1 5.35 use
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
PPM
0
2
4
6
8
2.37
3.14
56.30
2.21
1.61
1.99
2.05
1.99
2.89
1.00
0.65
0.91
7k

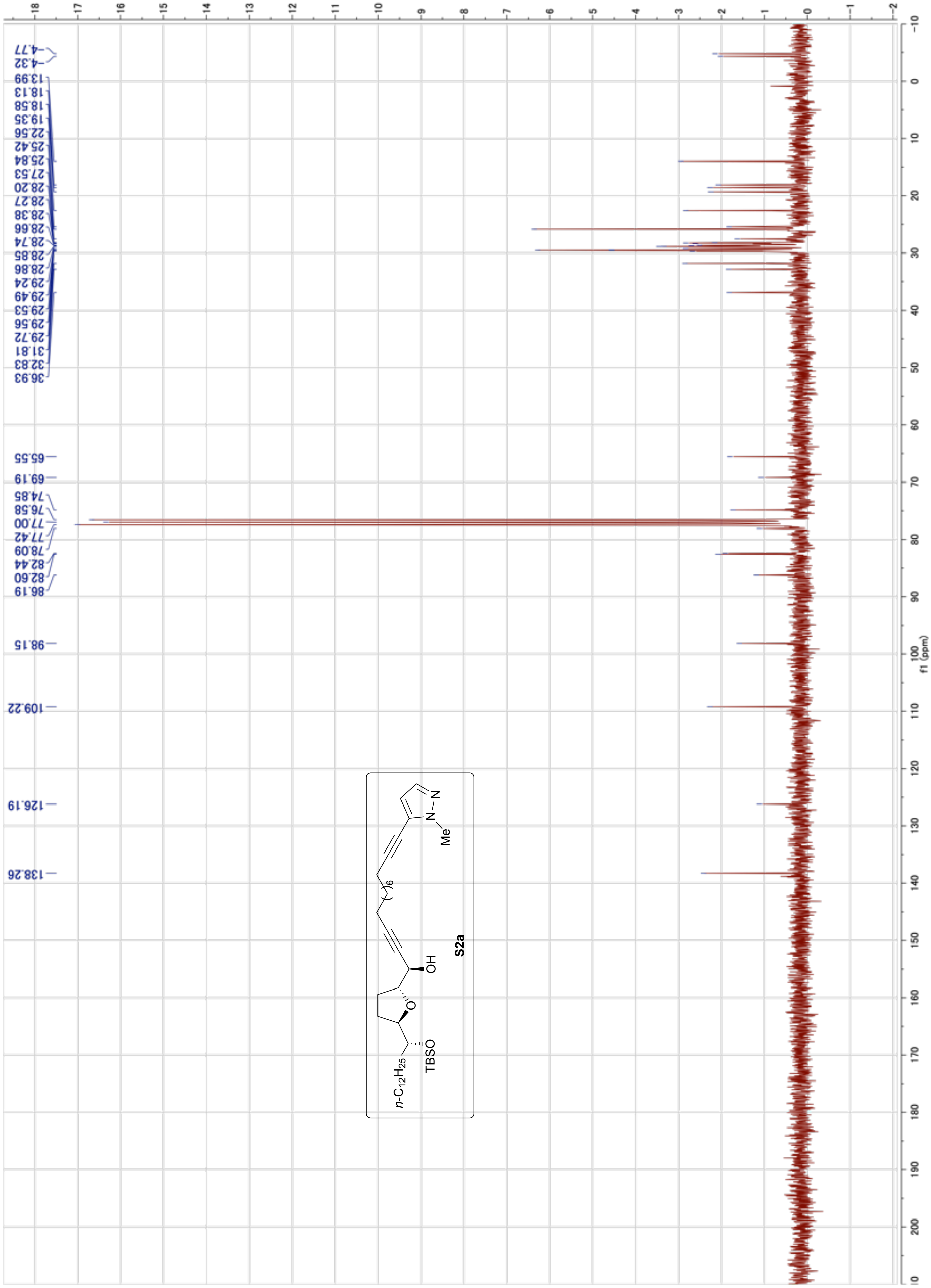


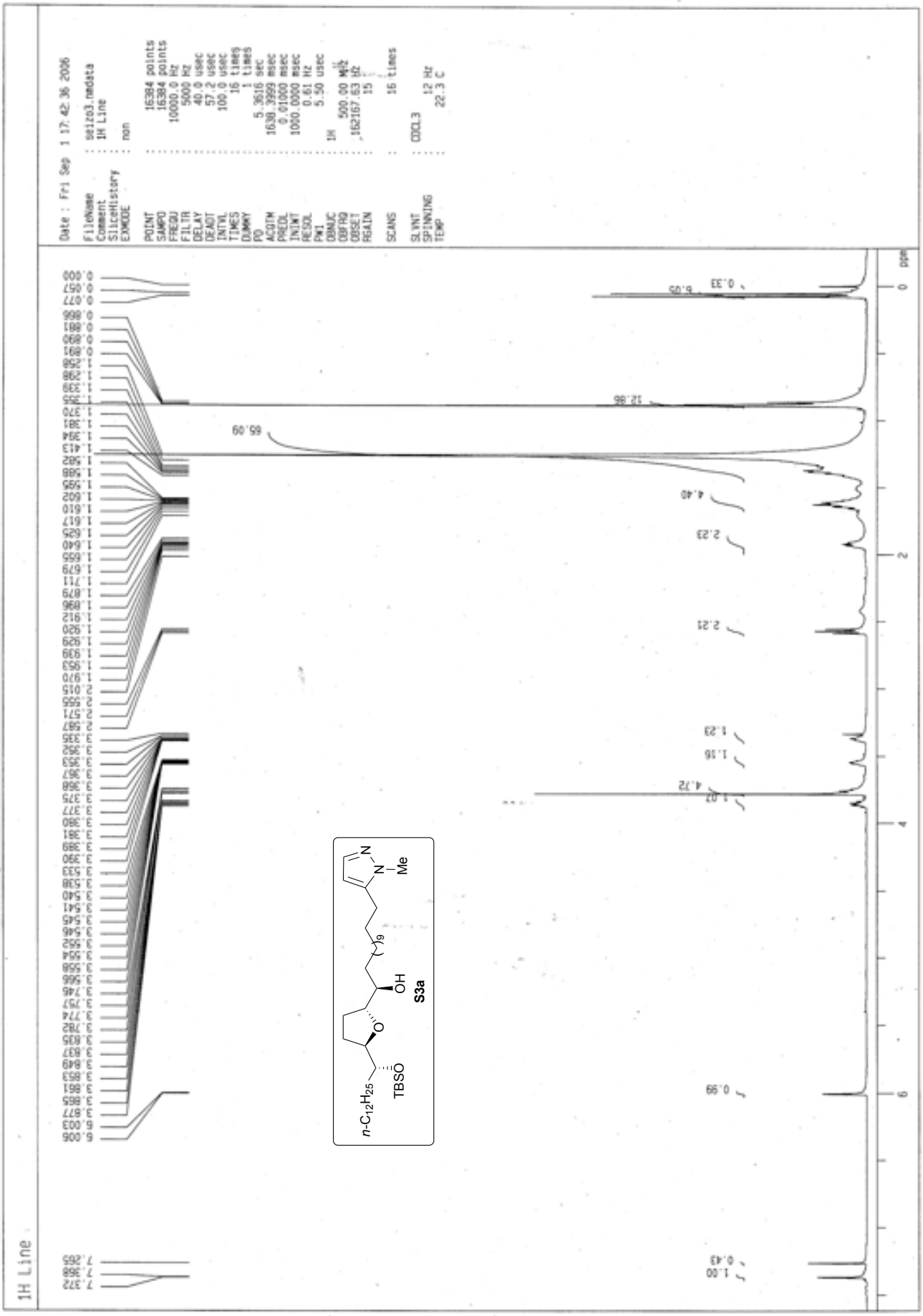
DFILE	E:\,t,r,a@"'fofł
COMNT	1022
DATIM	Thu Oct 22 17:01
OBNUC	13C
EXMOD	bcm
OBFRQ	125.65 MHz
OBSET	0.00 KHz
OBFIN	127958.00 Hz
POINT	32768
FREQU	33898.30 Hz
SCANS	550
ACQTM	0.9667 sec
PD	2.0333 sec
PW1	5.00 use
IRNUC	1H
CTEMP	25.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	26

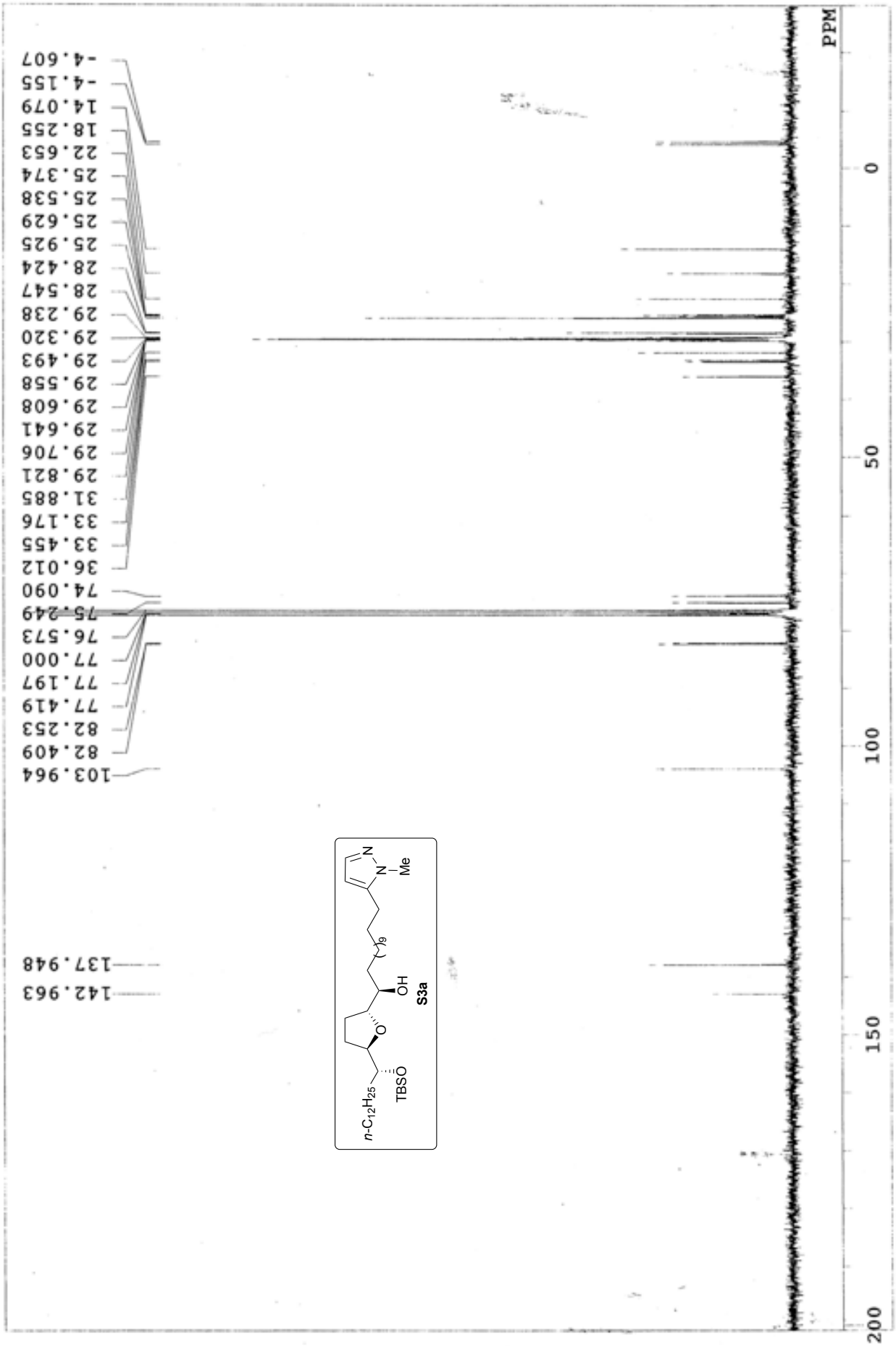

1H Line
Date : Mon Dec 18 13:49:32 2006
FileName : set02.mdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 16384 points
SAMPO : 16384 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 57.2 usec
INTVL : 100.0 usec
TIMES : 32 times
DUMMY : 1 times
PD : 5.3616 sec
ACQTM : 1638.3999 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.61 Hz
PW1 : 5.50 usec
DENUC : N
1H : 500.00 MHz
OBFRQ : +162167.63 Hz
OBSET : 22 /
REGAIN :
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 24.3 C
3.48
3.30
14.21
53.12
1.62
2.09
0.78
4.04
1.02
1.12
0.98
0.88
1.00
4.25
ppm
0
2
4
6
OH
S1
O
TBSO
n-C12H25
6

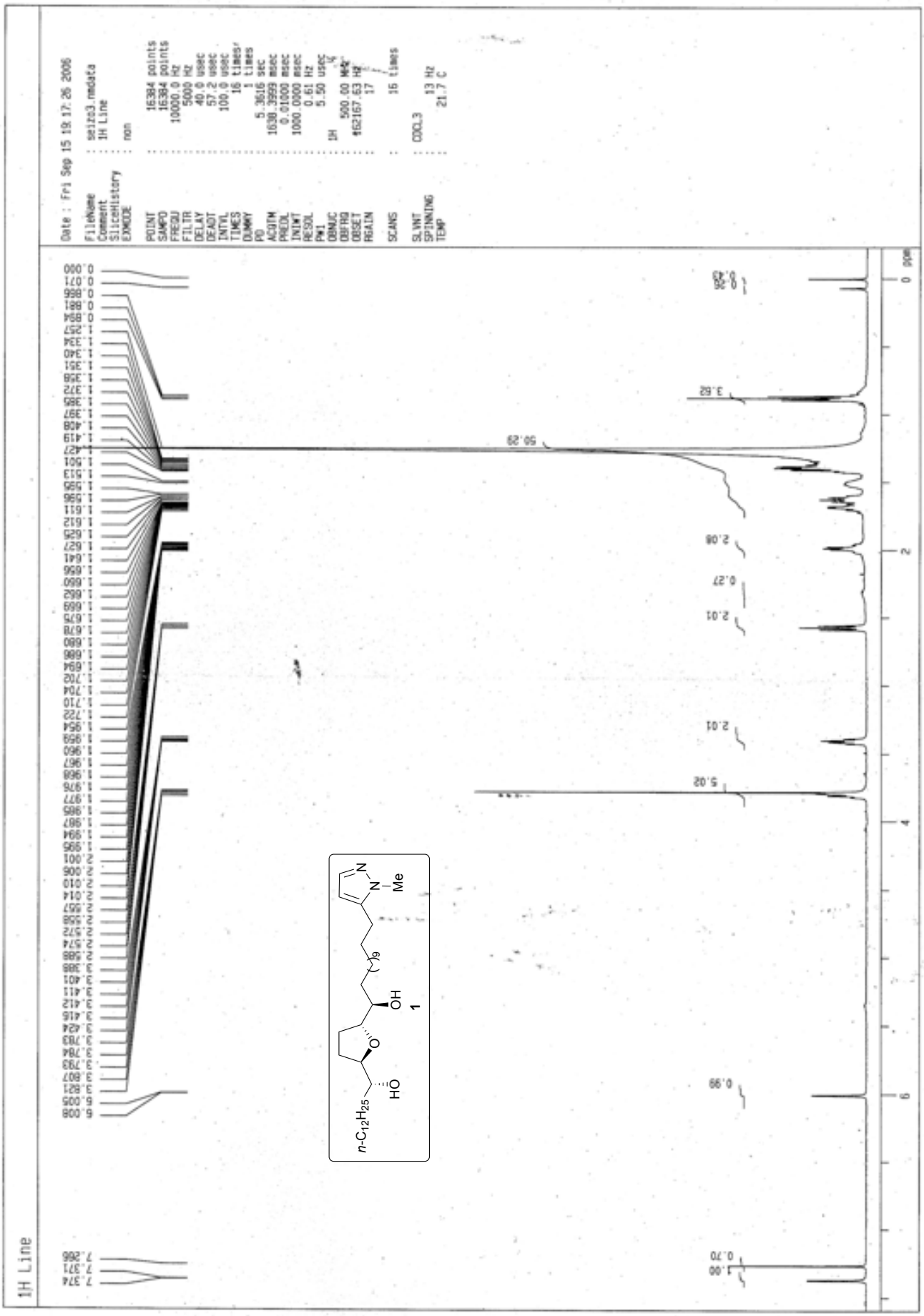

86.191
84.596
82.557
82.393
77.995
77.419
77.197
77.000
76.573
74.813
68.056
65.524
32.830
31.860
29.764
29.624
29.583
29.550
29.304
28.893
28.679
28.621
28.416
28.366
28.243
27.585
25.884
25.481
22.637
18.625
18.313
18.189
14.079
-4.220
-4.689
n-C12H25
TBSO
O
OH
6
S1
PPM
200
150
100
50
0
DFILE E:\300 C\TBS THF
COMNT FT-168
DATIM Sun Dec 10 16:45
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 700
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 4.60 use
IRNUC 1H
CTEMP 16.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24

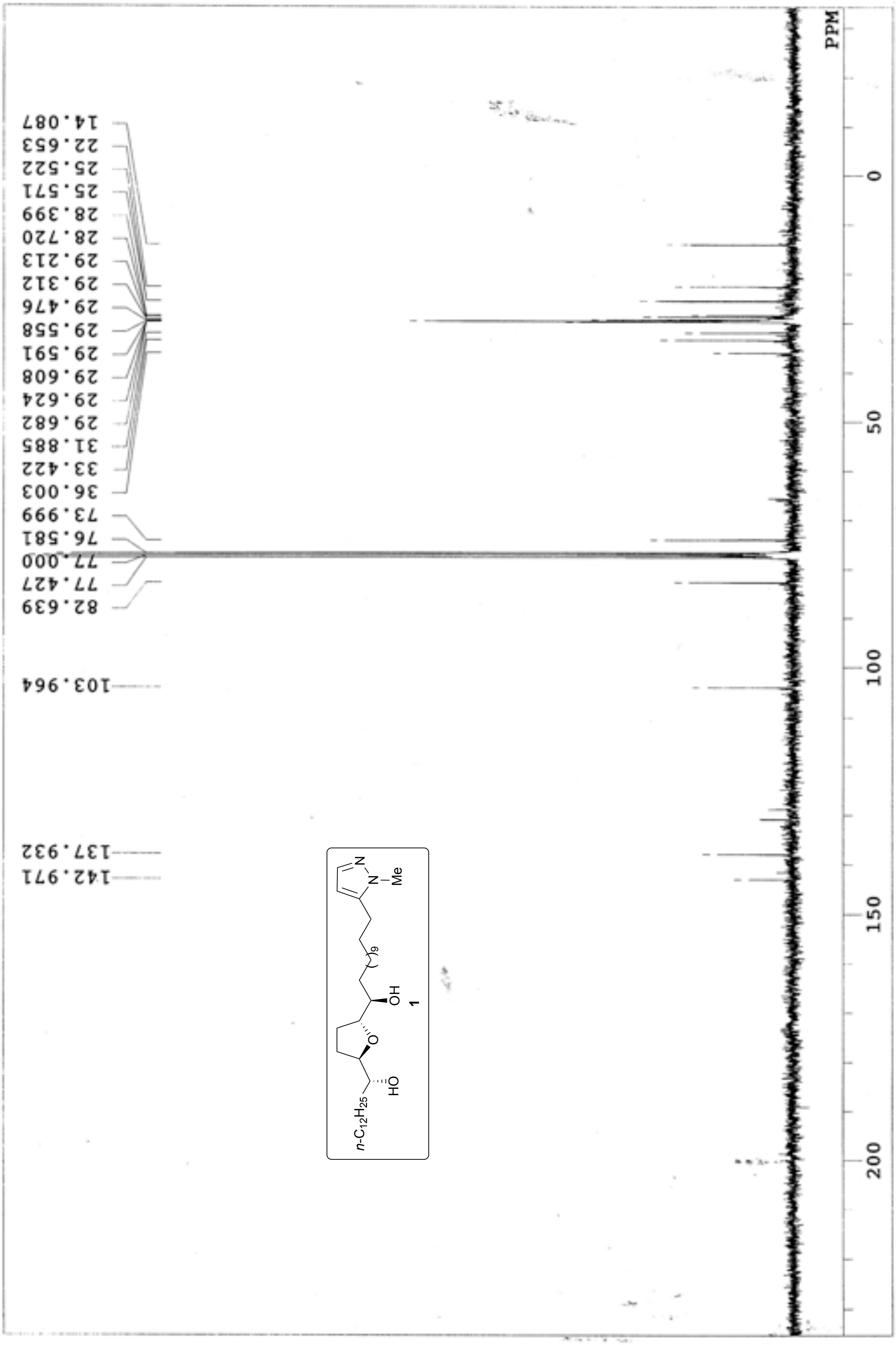

PPM
S2a
TBSO
OH
Me

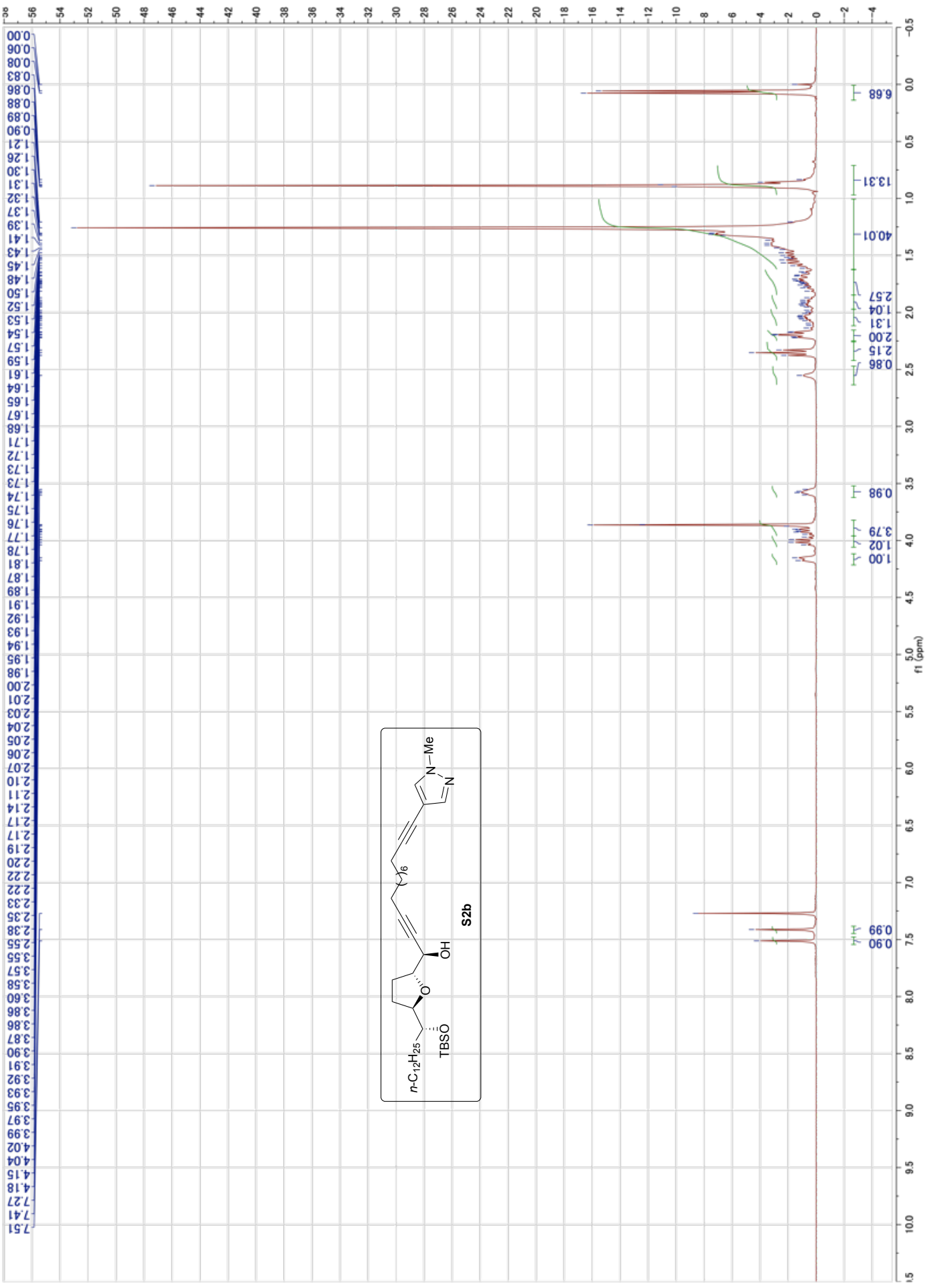

-4.77
-4.32
13.99
18.13
18.58
19.35
22.56
25.42
25.84
27.53
28.20
28.27
28.38
28.66
28.74
28.85
28.86
29.24
29.49
29.53
29.56
29.72
31.81
32.83
36.93
65.55
69.19
74.85
76.58
77.00
77.42
78.09
82.44
82.60
86.19
98.15
109.22
126.19
138.26
f1 (ppm)
n-C12H25
TBSO
OH
Me
S2a

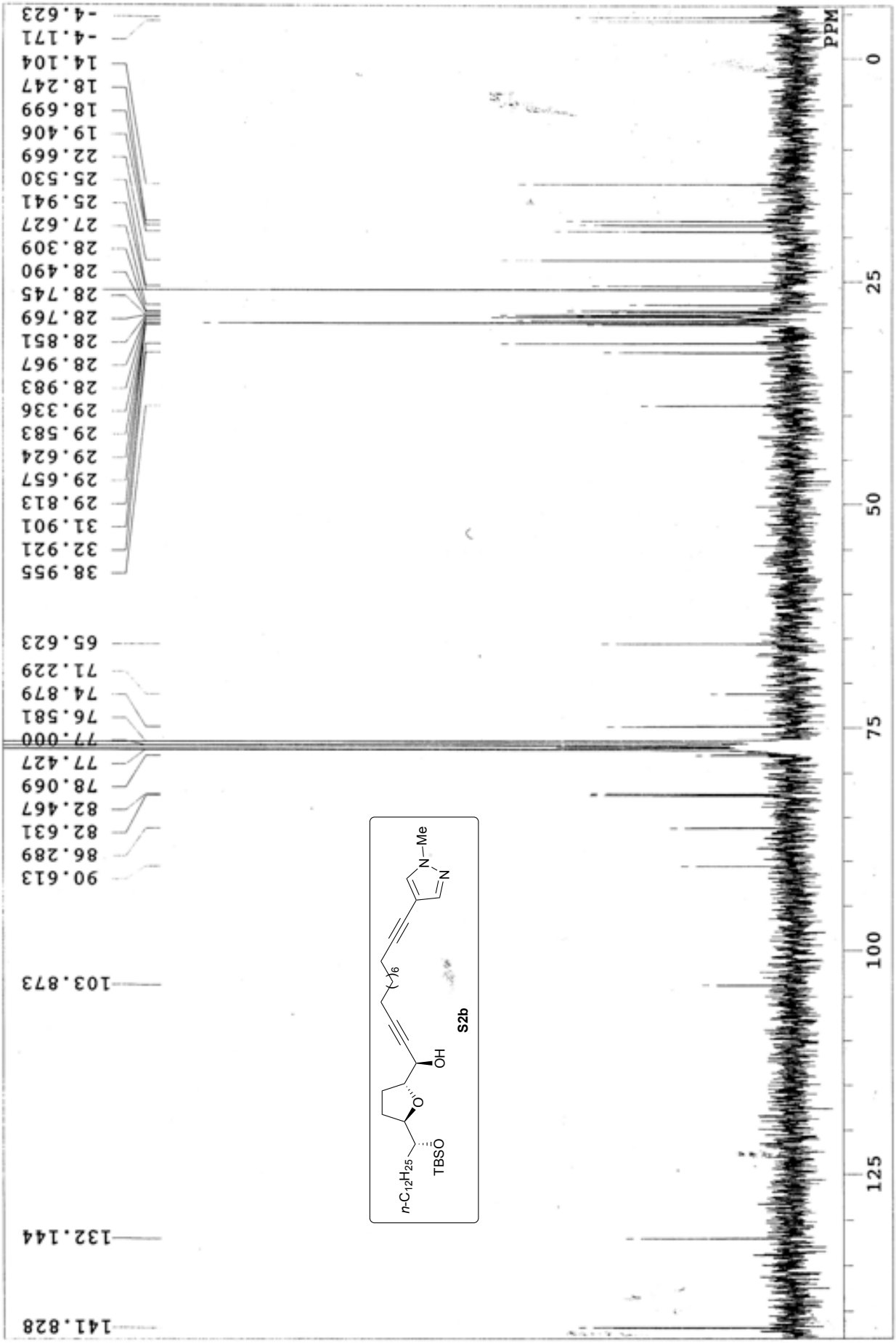

1H Line
Date : Fri Sep 1 17:42:36 2006
FileName : sei203.nedata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT 16384 points
SAMPD 16384 points
FREQU 10000.0 Hz
FILTR 5000 Hz
DELAY 40.0 usec
DEADT 57.2 usec
INTVL 100.0 usec
TIMES 16 times
DUMMY 1 times
PD 5.3616 sec
ACQTM 1638.3999 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 0.61 Hz
PW1 5.50 usec
1H 500.00 MHz
OBNUC
OBFRQ 162167.63 Hz
OBSET 15
REGAIN
SCANS 16 times
SLVNT CDCl3
SPINNING 12 Hz
TEMP 22.3 C
S3a
ppm

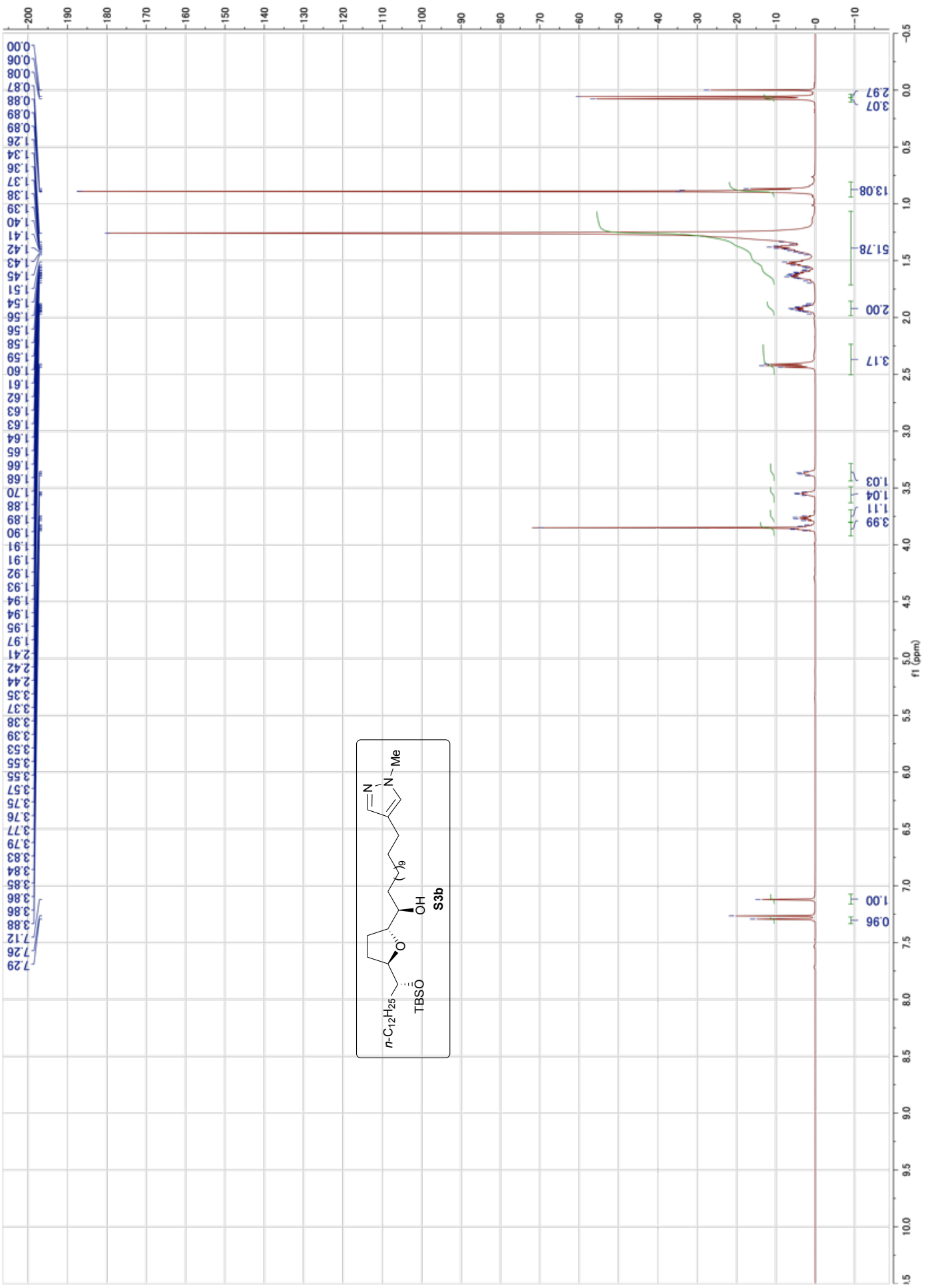

-4.607
-4.155
14.079
18.255
22.653
25.374
25.538
25.629
25.925
28.424
28.547
29.238
29.320
29.493
29.558
29.608
29.641
29.706
29.821
31.885
33.176
33.455
36.012
74.090
75.249
76.573
77.000
77.197
77.419
82.253
82.409
103.964
137.948
142.963
PPM
0
50
100
150
200
S3a
N
N
Me
OH
O
TBSO
n-C12H25

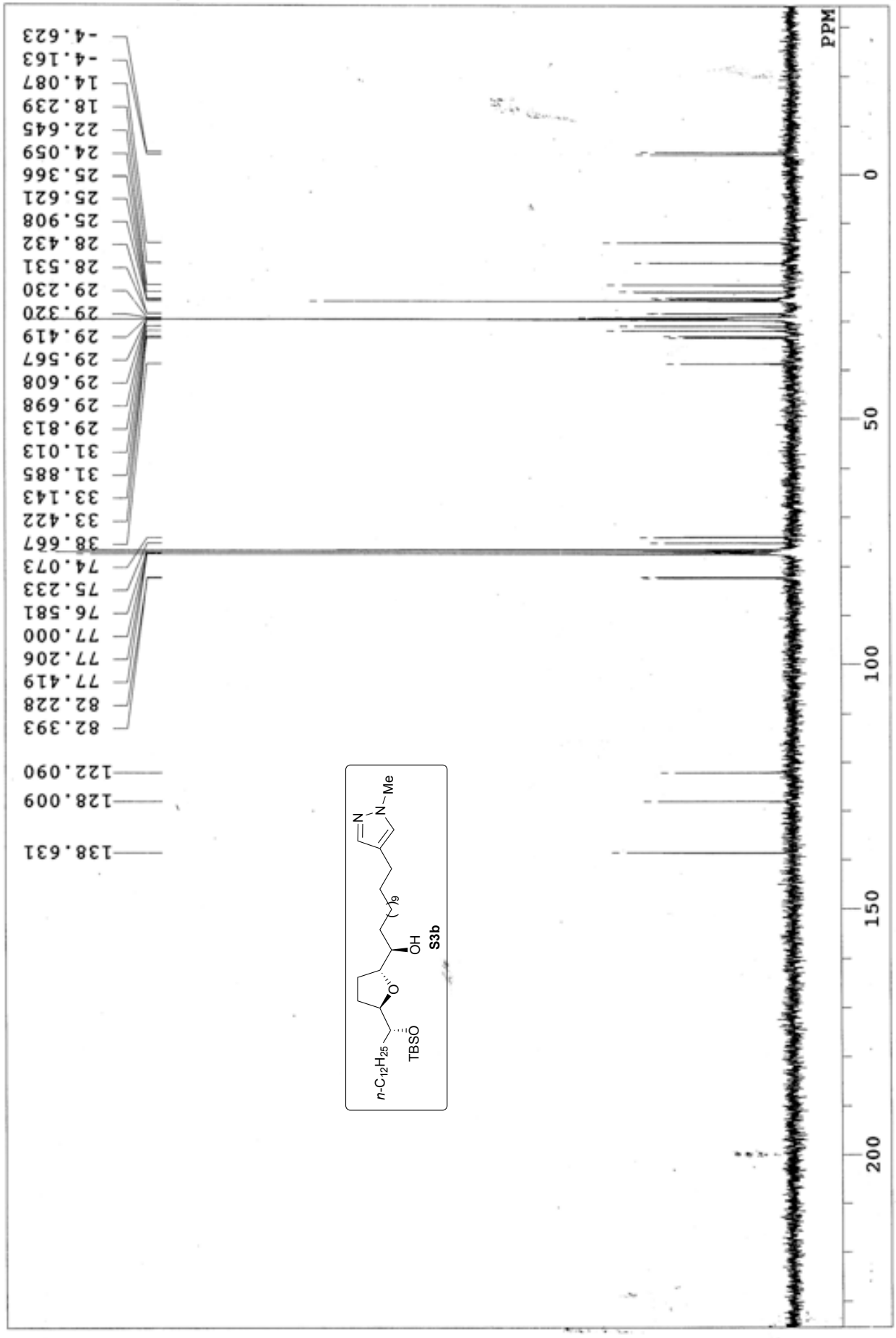

1H Line
Date : Fri Sep 15 19:17:26 2006
FileName : seize3.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 16384 points
SAMPO : 16384 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 57.2 usec
INTVL : 100.0 usec
TIMES : 16 times
DUMMY : 1 times
PD : 5.3616 sec
ACQTM : 1638.3999 msec
PRDEL : 0.01000 msec
INTWT : 1000.0000 msec
RESOL : 0.61 Hz
PW1 : 5.50 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 462167.63 Hz
RGAIN : 17
SCANS : 16 times
SLVNT : CDCl3
SPINNING : 13 Hz
TEMP : 21.7 C
n-C12H25
HO
OH
N
N
Me
1
0.70
1.00
0.99
5.02
2.01
2.01
0.27
2.08
50.29
3.62
0.82
0.86
0 ppm

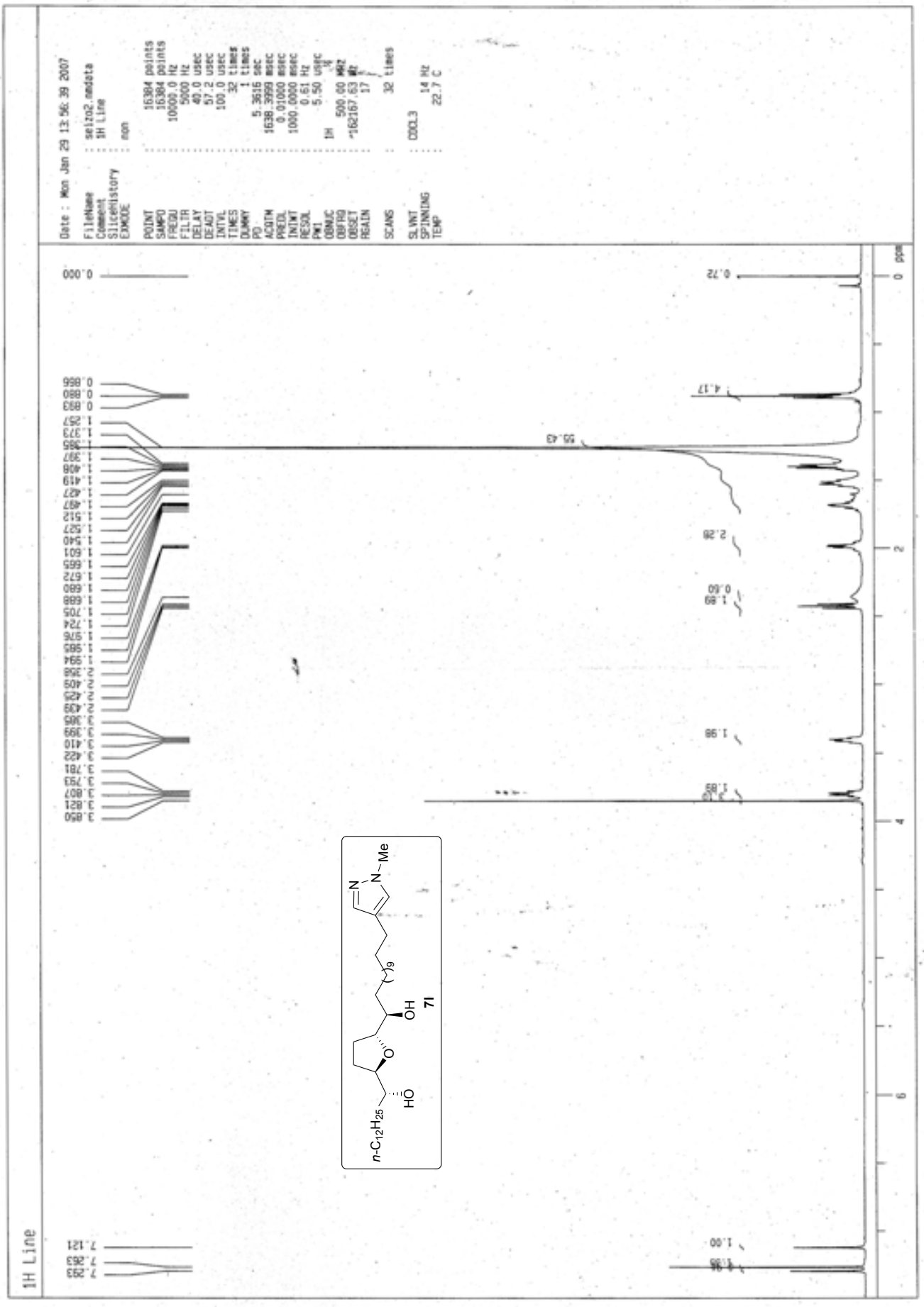

14.087
22.653
25.522
25.571
28.399
28.720
29.213
29.312
29.476
29.558
29.591
29.608
29.624
29.682
31.885
33.422
36.003
73.999
76.581
77.000
77.427
82.639
103.964
137.932
142.971
PPM
0
50
100
150
200
1

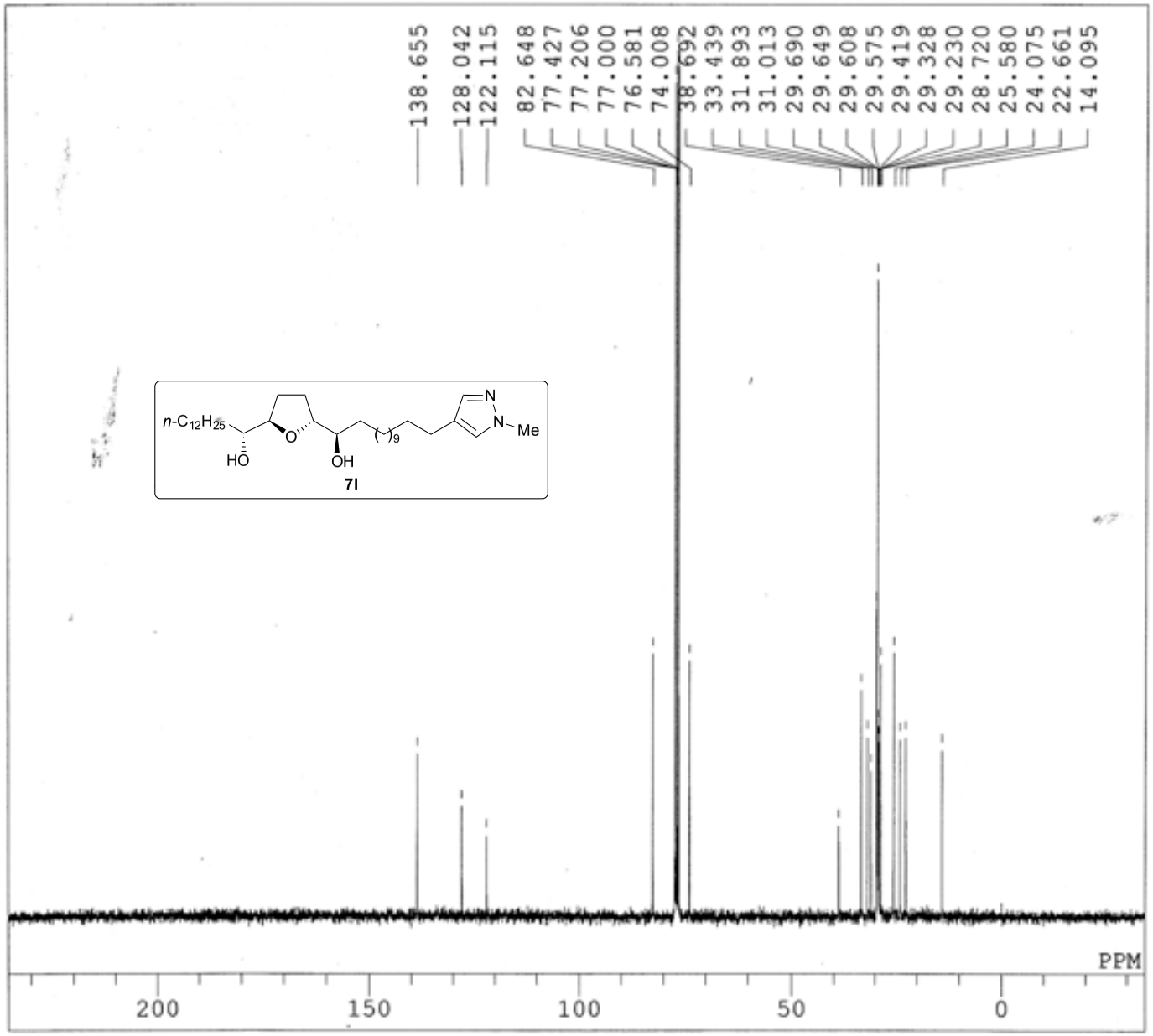

S2b
N–Me
N
OH
O
TBSO
n-C12H25
6.68
13.31
40.01
2.57
1.04
1.31
2.00
2.15
0.86
0.98
3.79
1.02
1.00
0.99
0.90
f1 (ppm)


-4.623
-4.171
14.104
18.247
18.699
19.406
22.669
25.530
25.941
27.627
28.309
28.490
28.745
28.769
28.851
28.967
28.983
29.336
29.583
29.624
29.657
29.813
31.901
32.921
38.955
65.623
71.229
74.879
76.581
77.000
77.427
78.069
82.467
82.631
86.289
90.613
103.873
132.144
141.828
PPM
0
25
50
75
100
125
S2b
N
Me
N
6
OH
O
TBSO
n-C12H25

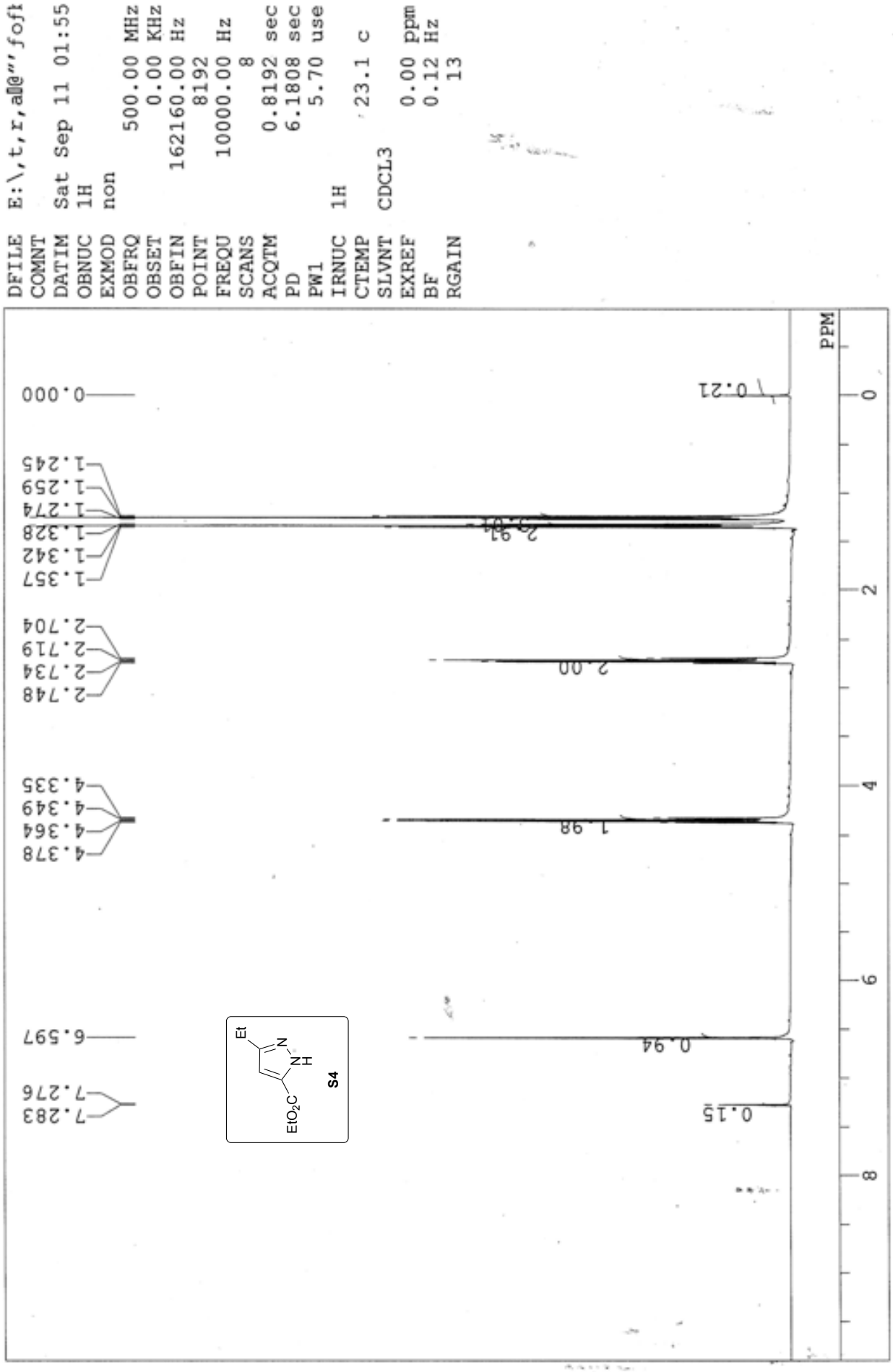

Me
N
N
9
OH
S3b
O
TBSO
n-C12H25
2.97
3.07
13.08
51.78
2.00
3.17
1.03
1.04
1.11
3.99
1.00
0.96
f1 (ppm)

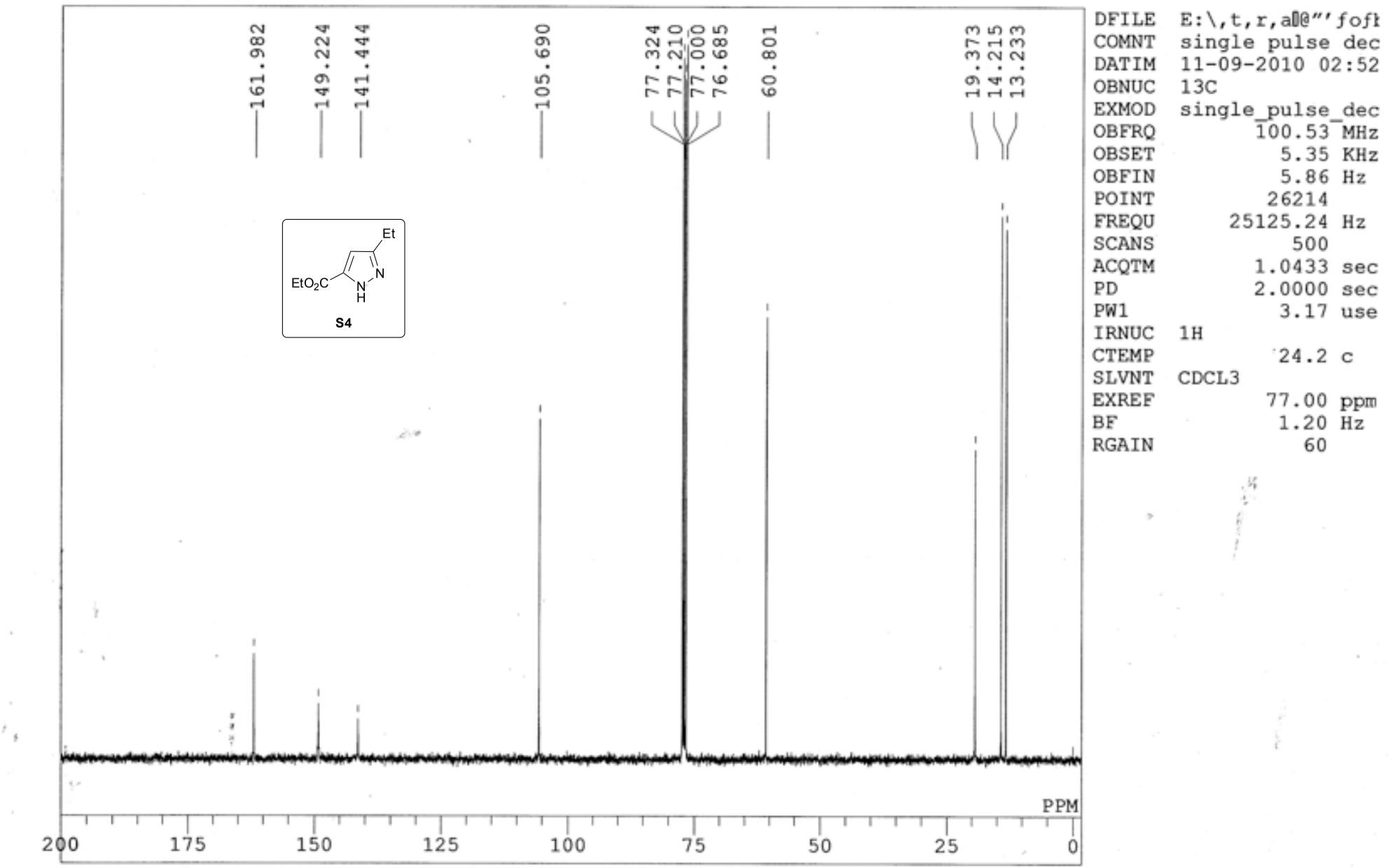

-4.623
-4.163
14.087
18.239
22.645
24.059
25.366
25.621
25.908
28.432
28.531
29.230
29.320
29.419
29.567
29.608
29.698
29.813
31.013
31.885
33.143
33.422
38.667
74.073
75.233
76.581
77.000
77.206
77.419
82.228
82.393
122.090
128.009
138.631
PPM
0
50
100
150
200
S3b
OH
TBSO
n-C12H25
O
N
N
Me
9


1H Line
Date : Mon Jan 29 13:56:39 2007
FileName : seiz02.mdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT 16384 points
SAMPO 16384 points
FREQU 10000.0 Hz
FILTR 5000 Hz
DELAY 40.0 usec
DEADT 57.2 usec
INTVL 100.0 usec
TIMES 32 times
DUMMY 1 times
PD 5.3616 sec
ACQTM 1638.3999 msec
PREDL 0.01000 msec
INTWT 1000.0000 msec
RESOL 0.61 Hz
PW1 5.50 usec
1H
ODFRQ 500.00 MHz
OBSET *162167.63 Hz
REATN 17 /
SCANS 32 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 22.7 C
ppm
n-C12H25
HO
OH
Me
71

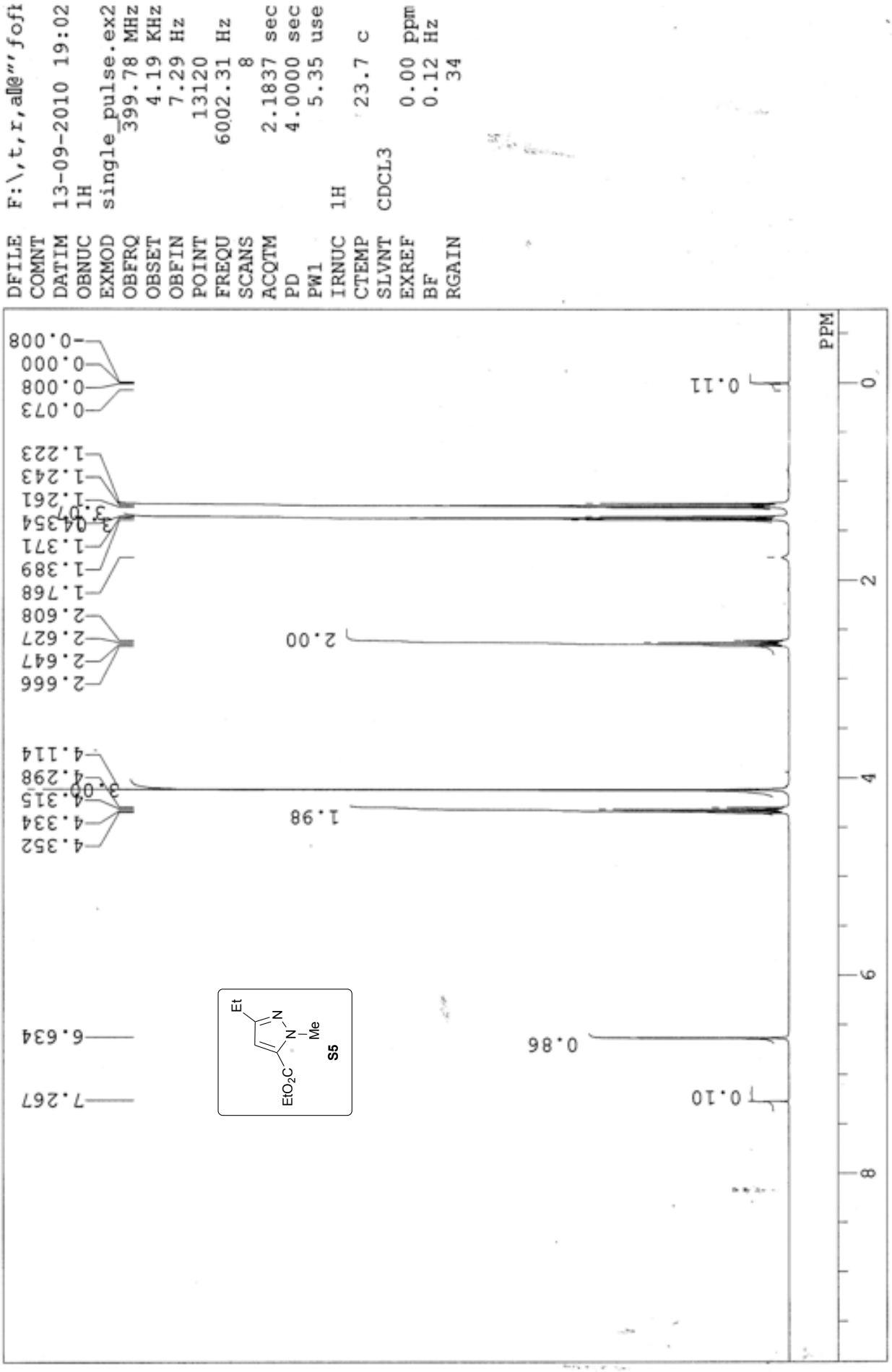


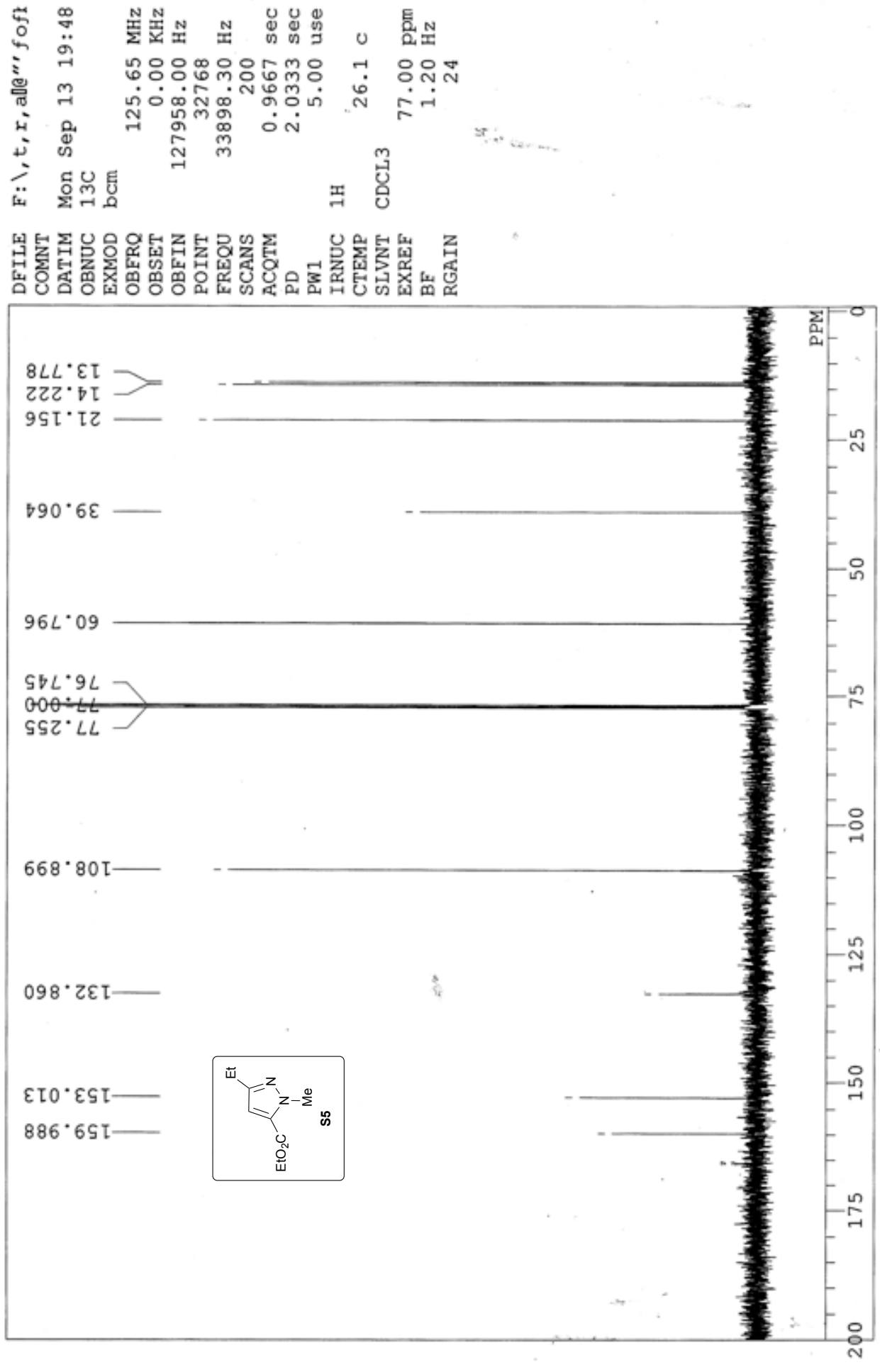

```
DFILE   E:\300 C\THF 4pyra fir
COMNT   THF 4pyra fin
DATIM   Sat Apr 28 05:07:32 2(
OBNUC   13C
EXMOD   BCM
OBFRQ        75.45 MHz
OBSET       124.00 KHz
OBFIN      1840.00 Hz
POINT        32768
FREQU      20356.23 Hz
SCANS         1100
ACQTM       1.6097 sec
PD          1.3900 sec
PW1           4.60 usec
IRNUC   1H
CTEMP         22.6 c
SLVNT   CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           26
```

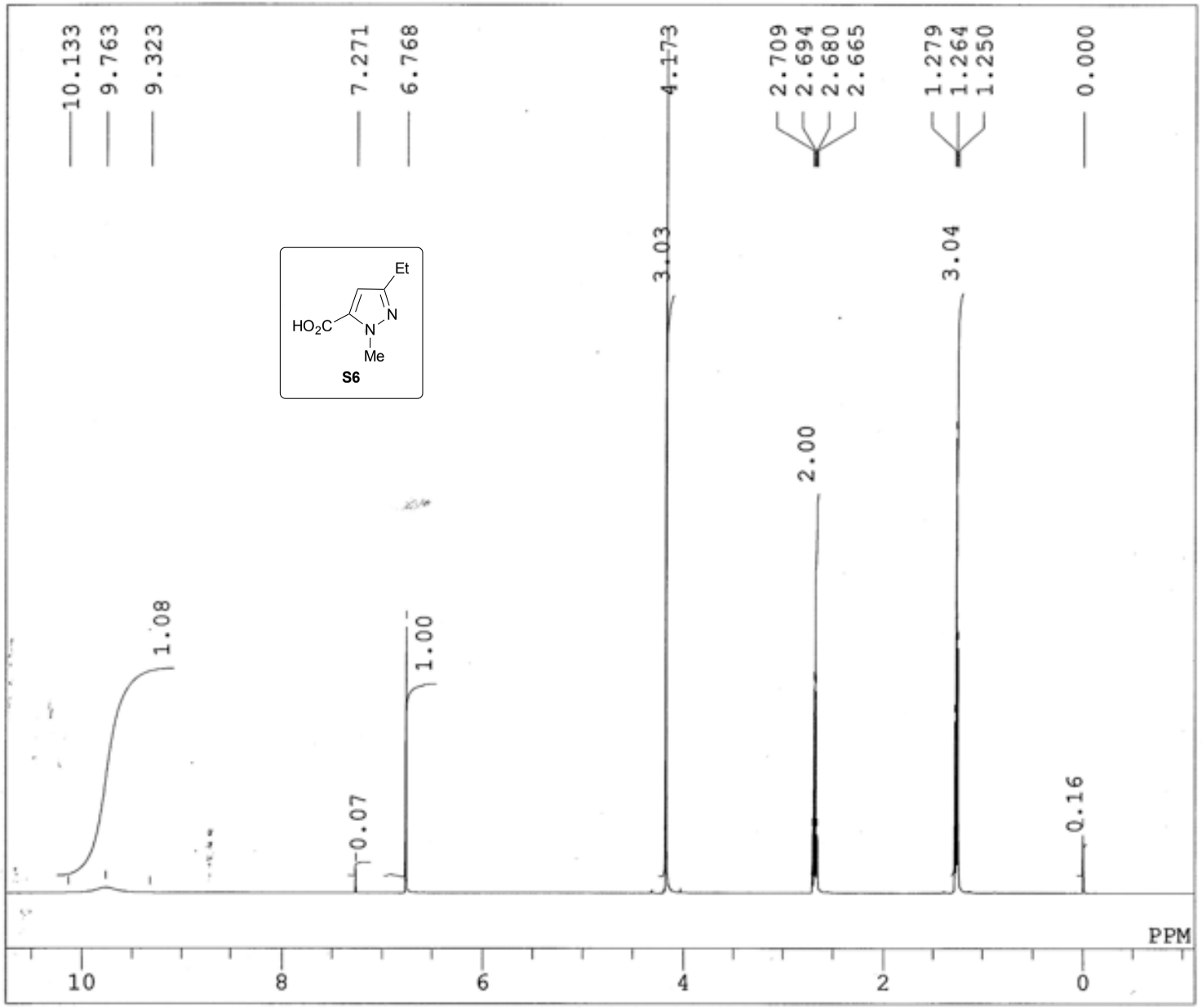

DFILE E:\,t,r,a@"'fofk
COMNT
DATIM Sat Sep 11 01:55
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 8192
FREQU 10000.00 Hz
SCANS 8
ACQTM 0.8192 sec
PD 6.1808 sec
PW1 5.70 use
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
PPM
0.000
1.245
1.259
1.274
1.328
1.342
1.357
2.704
2.719
2.734
2.748
4.335
4.349
4.364
4.378
6.597
7.276
7.283
0.21
2.91
2.00
1.98
0.94
0.15
Et
EtO2C
S4



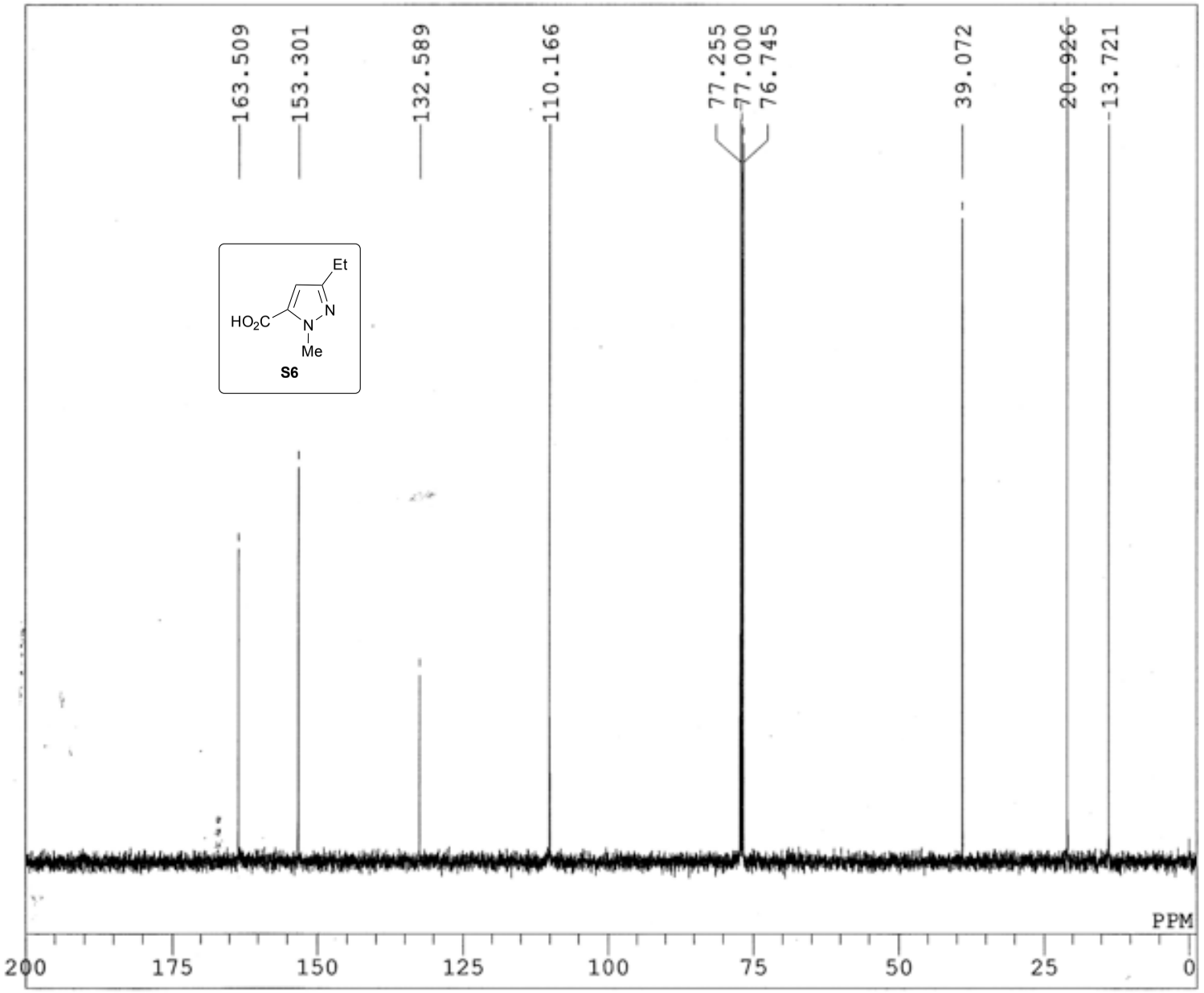
DFILE E:\,t,r,a@"'fofl
COMNT single pulse dec
DATIM 11-09-2010 02:52
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.24 Hz
SCANS 500
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 use
IRNUC 1H
CTEMP 24.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60


DFILE F:\,t,r,a@"'fofi
COMNT
DATIM 13-09-2010 19:02
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13120
FREQU 6002.31 Hz
SCANS 8
ACQTM 2.1837 sec
PD 4.0000 sec
PW1 5.35 use
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
PPM
0.11
2.00
1.98
0.86
0.10
Et
N
N
Me
EtO2C
S5

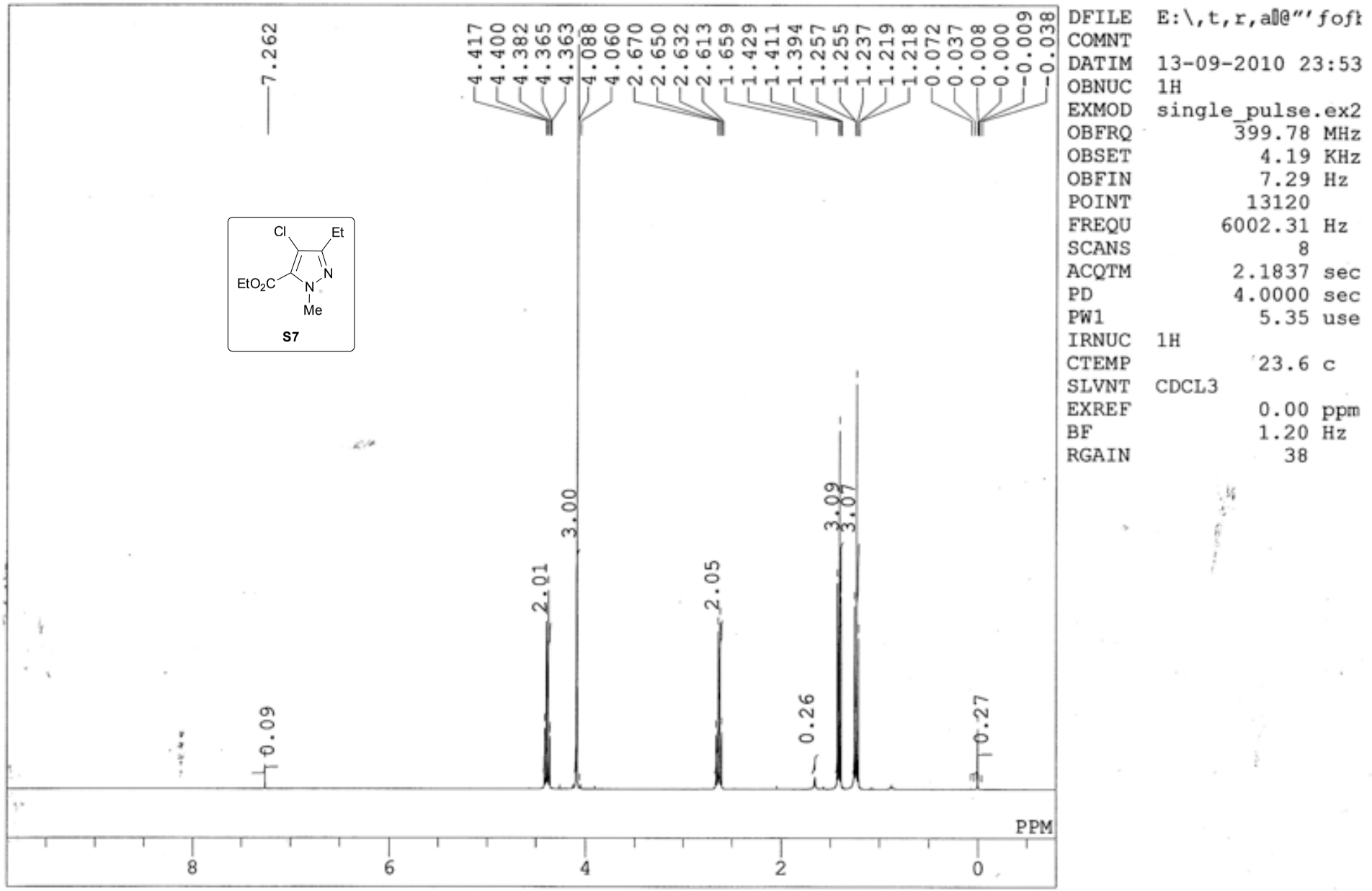

DFILE F:\,t,r,a@"fofi
COMNT
DATIM Mon Sep 13 19:48
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 200
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24
13.778
14.222
21.156
39.064
60.796
76.745
77.000
77.255
108.899
132.860
153.013
159.988
PPM
0
25
50
75
100
125
150
175
200
Et
N
N
Me
EtO2C
S5

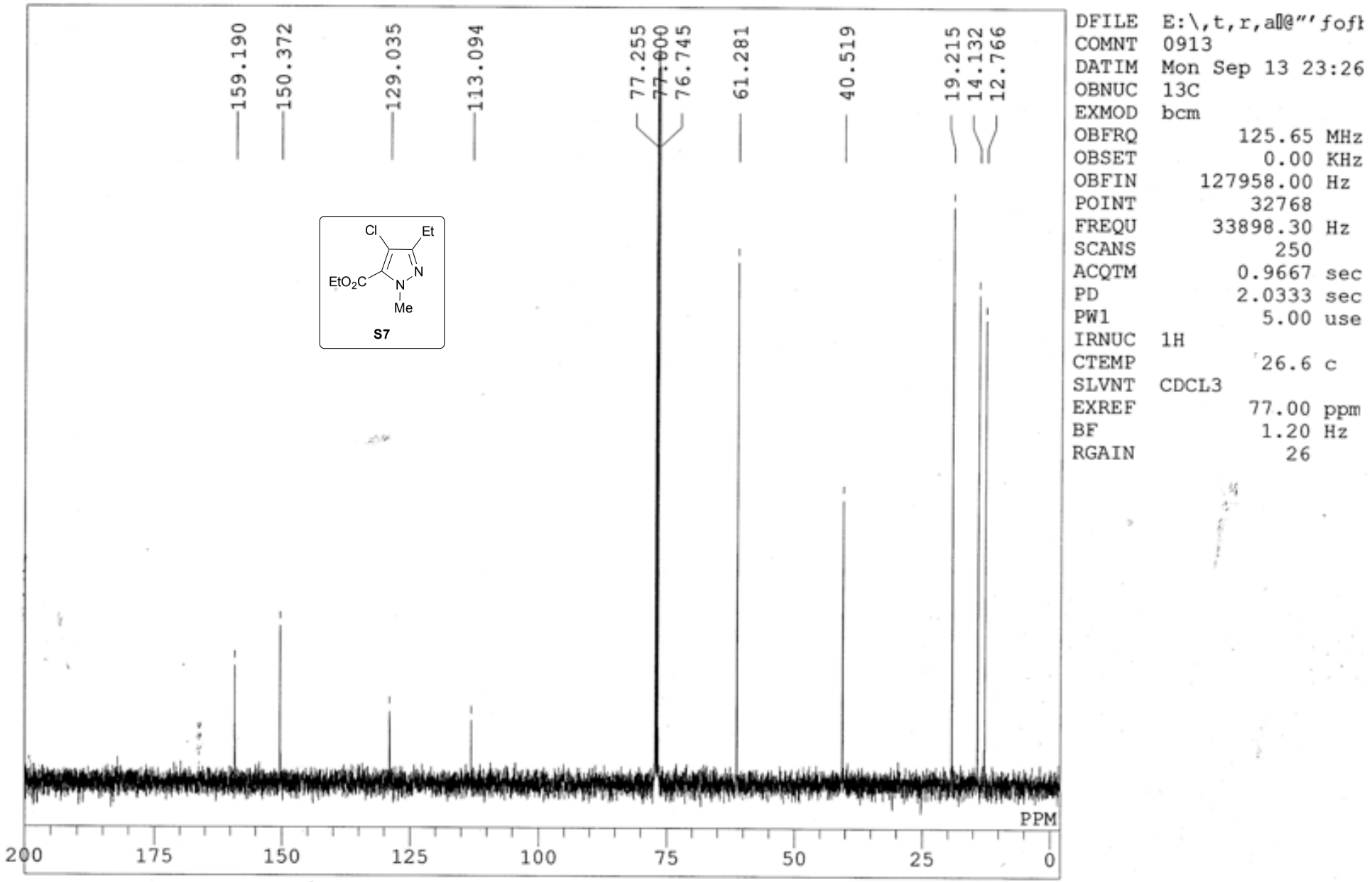


DFILE E:\,t,r,a[@"'foft
COMNT 0910
DATIM Fri Sep 10 22:21
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 8192
FREQU 10000.00 Hz
SCANS 16
ACQTM 0.8192 sec
PD 6.1808 sec
PW1 5.70 use
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13

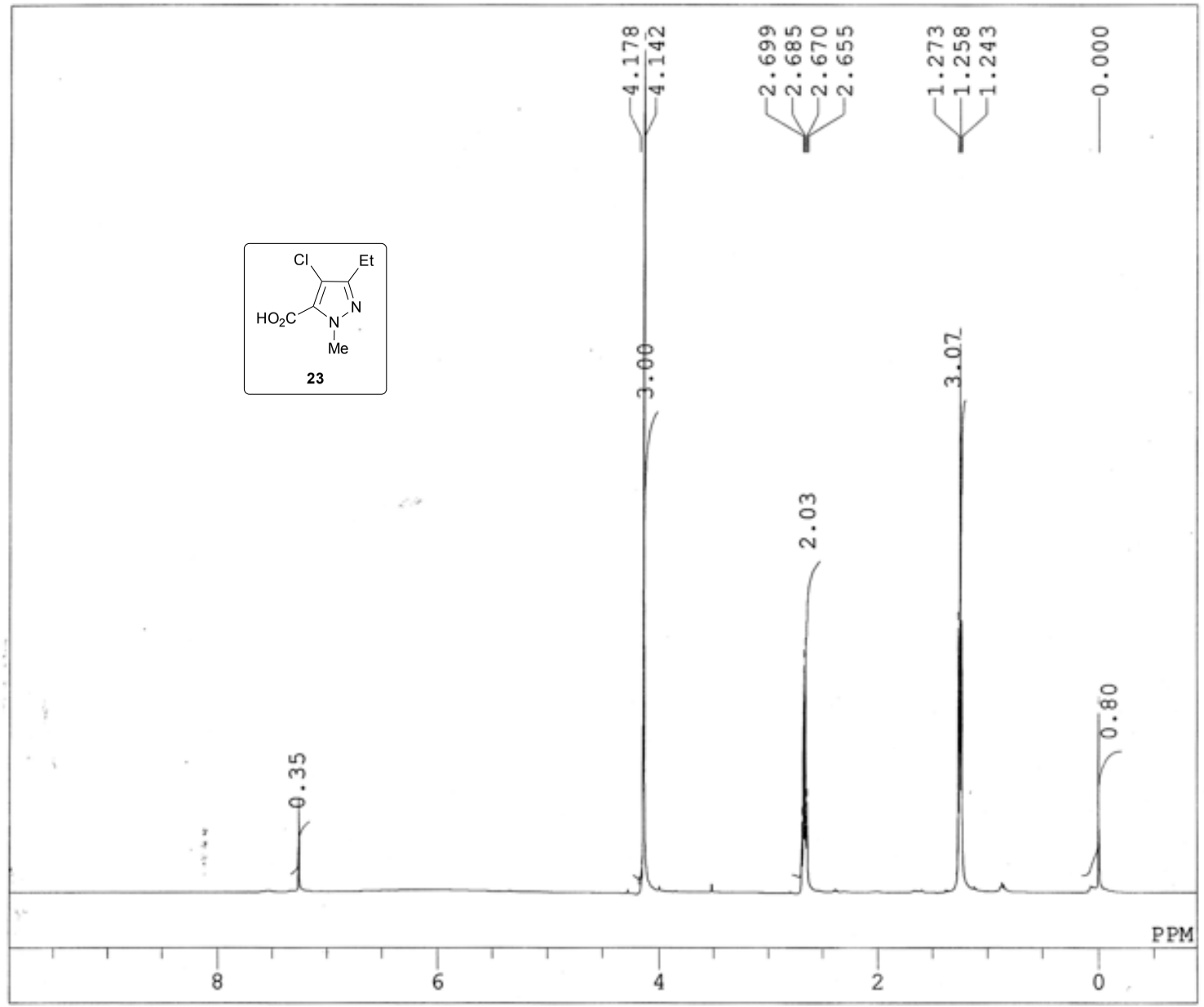



```
DFILE   E:\,t,r,a@"'fofh
COMNT   0910
DATIM   Fri Sep 10 22:44
OBNUC   13C
EXMOD   bcm
OBFRQ         125.65 MHz
OBSET           0.00 KHz
OBFIN      127958.00 Hz
POINT          32768
FREQU       33898.30 Hz
SCANS            200
ACQTM          0.9667 sec
PD             2.0333 sec
PW1            5.00 use
IRNUC   1H
CTEMP           24.6 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              1.20 Hz
RGAIN             26
```

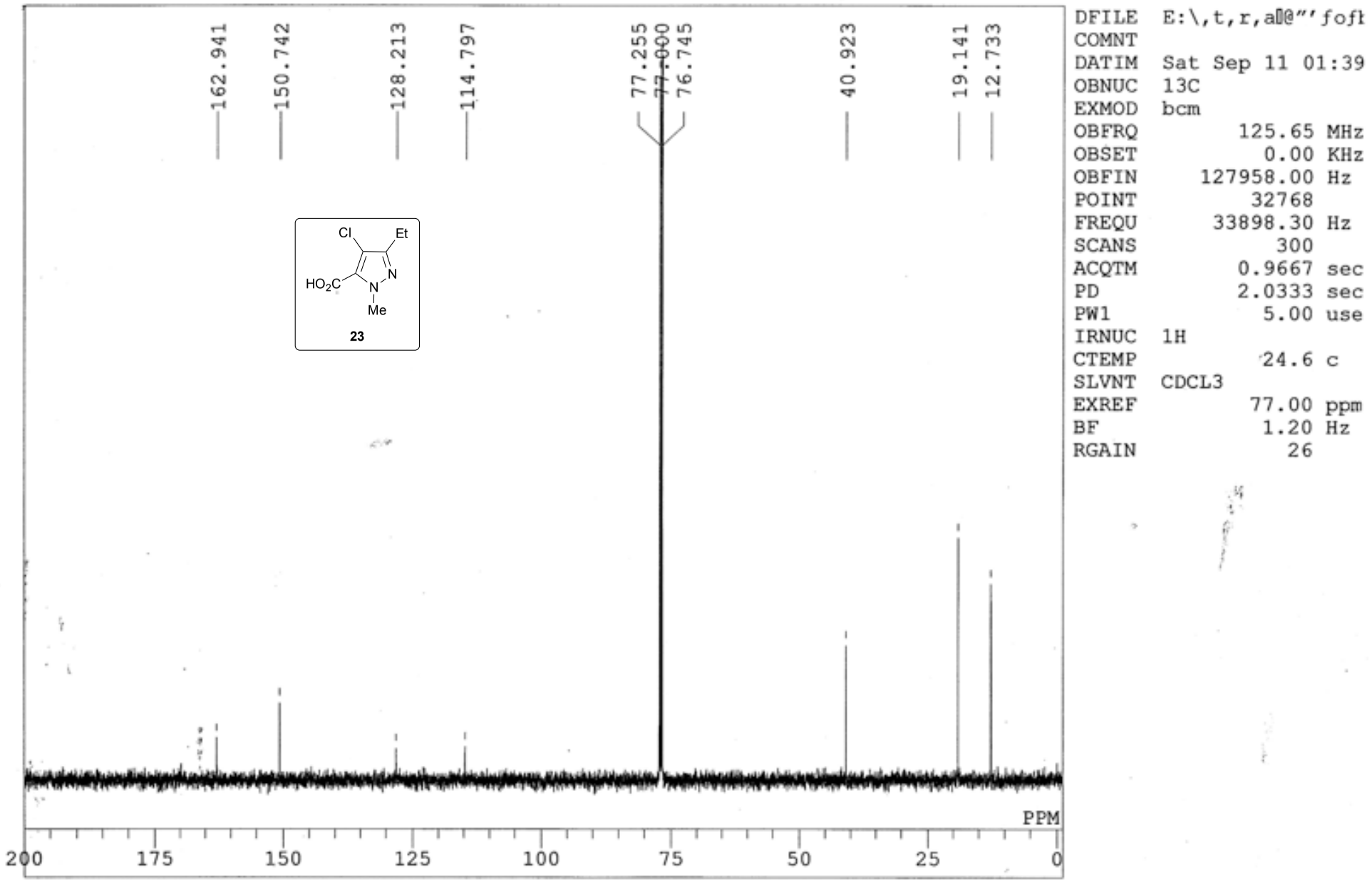

7.262
4.417
4.400
4.382
4.365
4.363
4.088
4.060
2.670
2.650
2.632
2.613
1.659
1.429
1.411
1.394
1.257
1.255
1.237
1.219
1.218
0.072
0.037
0.008
0.000
-0.009
-0.038
Cl
Et
EtO2C
N
N
Me
S7
3.00
2.01
2.05
3.09
3.07
0.09
0.26
0.27
PPM
8
6
4
2
0
DFILE E:\,t,r,a@"'fofk
COMNT
DATIM 13-09-2010 23:53
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13120
FREQU 6002.31 Hz
SCANS 8
ACQTM 2.1837 sec
PD 4.0000 sec
PW1 5.35 use
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 38



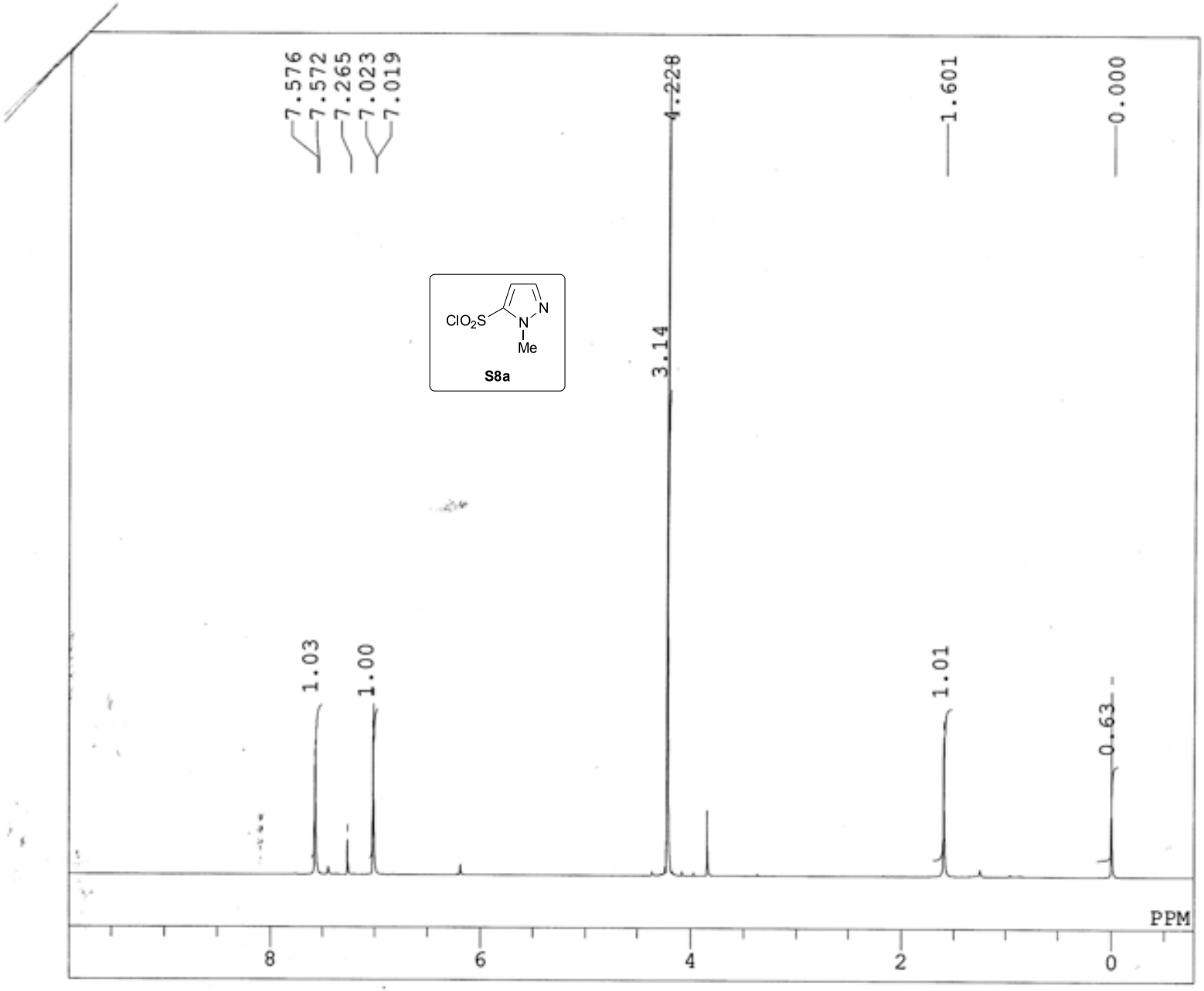

```
DFILE   E:\,t,r,a@"'fofh
COMNT   0913
DATIM   Mon Sep 13 23:26
OBNUC   13C
EXMOD   bcm
OBFRQ         125.65 MHz
OBSET           0.00 KHz
OBFIN      127958.00 Hz
POINT          32768
FREQU       33898.30 Hz
SCANS            250
ACQTM          0.9667 sec
PD             2.0333 sec
PW1              5.00 use
IRNUC   1H
CTEMP           26.6 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF               1.20 Hz
RGAIN              26
```



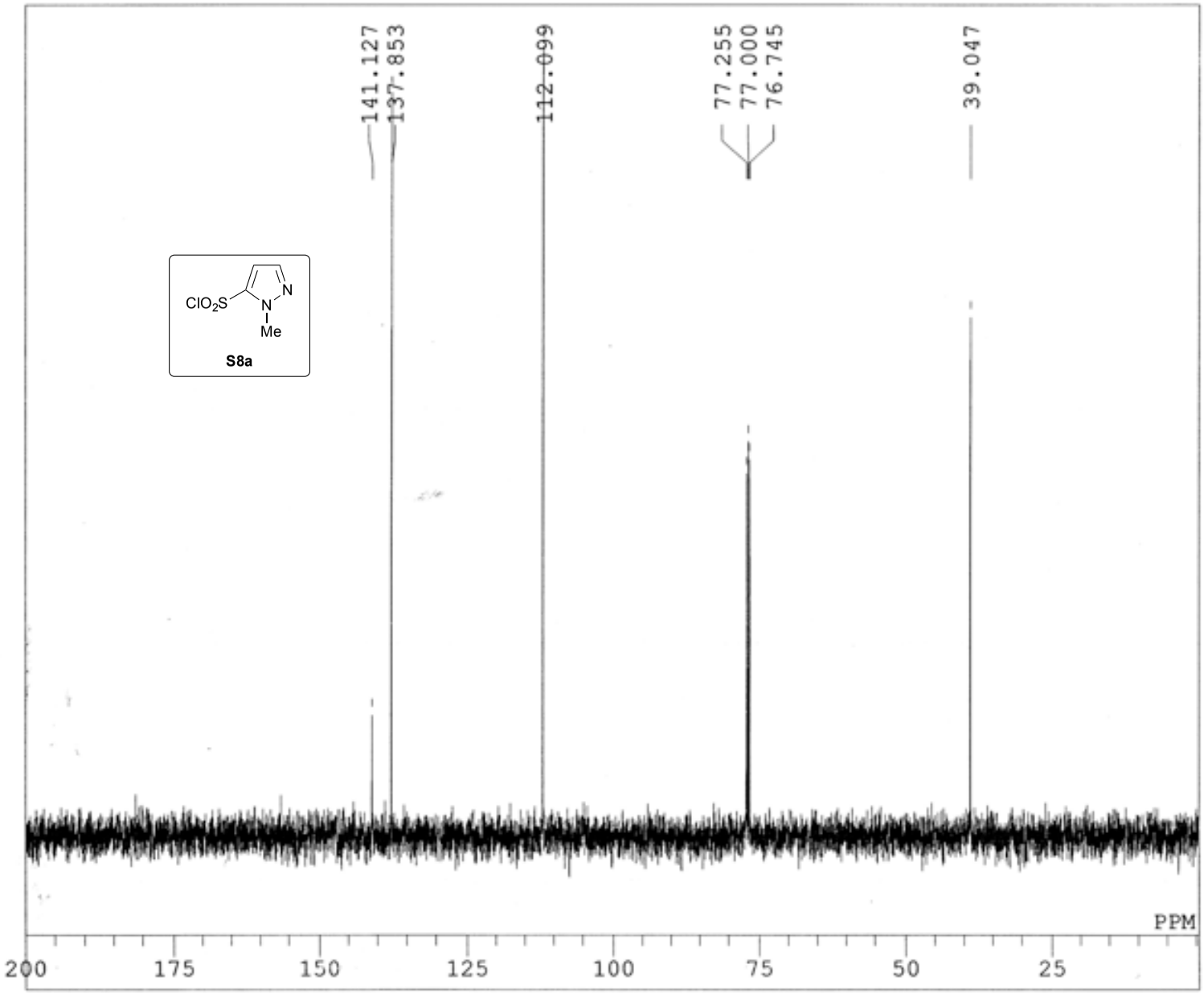

```
DFILE   E:\,t,r,a@"'fofk
COMNT
DATIM   Sat Sep 11 01:08
OBNUC   1H
EXMOD   non
OBFRQ         500.00 MHz
OBSET           0.00 KHz
OBFIN      162160.00 Hz
POINT          16384
FREQU       10000.00 Hz
SCANS             16
ACQTM          1.6384 sec
PD             5.3616 sec
PW1              5.70 use
IRNUC   1H
CTEMP           23.1 c
SLVNT   CDCL3
EXREF             0.00 ppm
BF                1.20 Hz
RGAIN               18
```



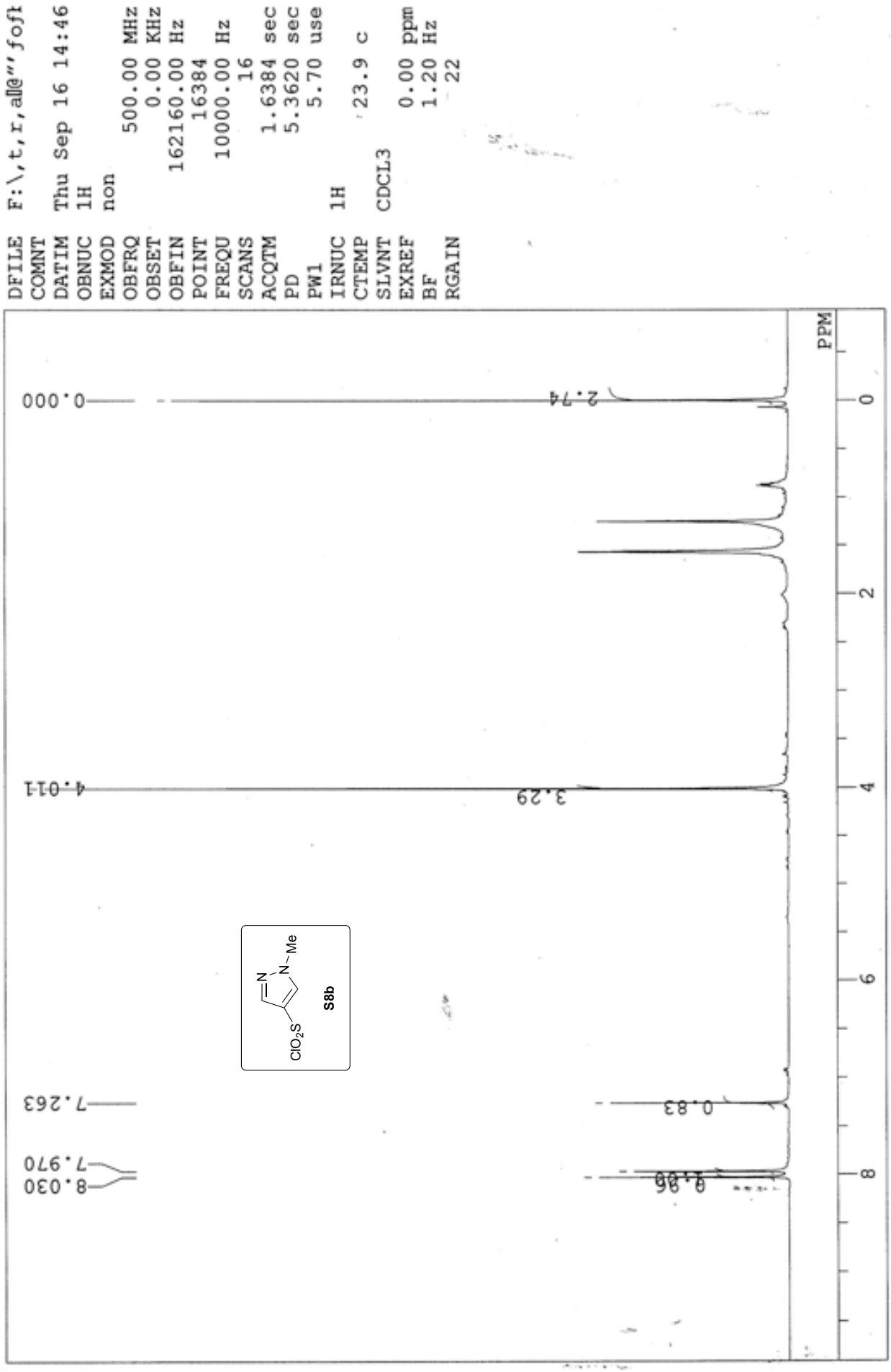
DFILE E:\,t,r,a@"'foft
COMNT
DATIM Sat Sep 11 01:39
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 300
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 24.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26

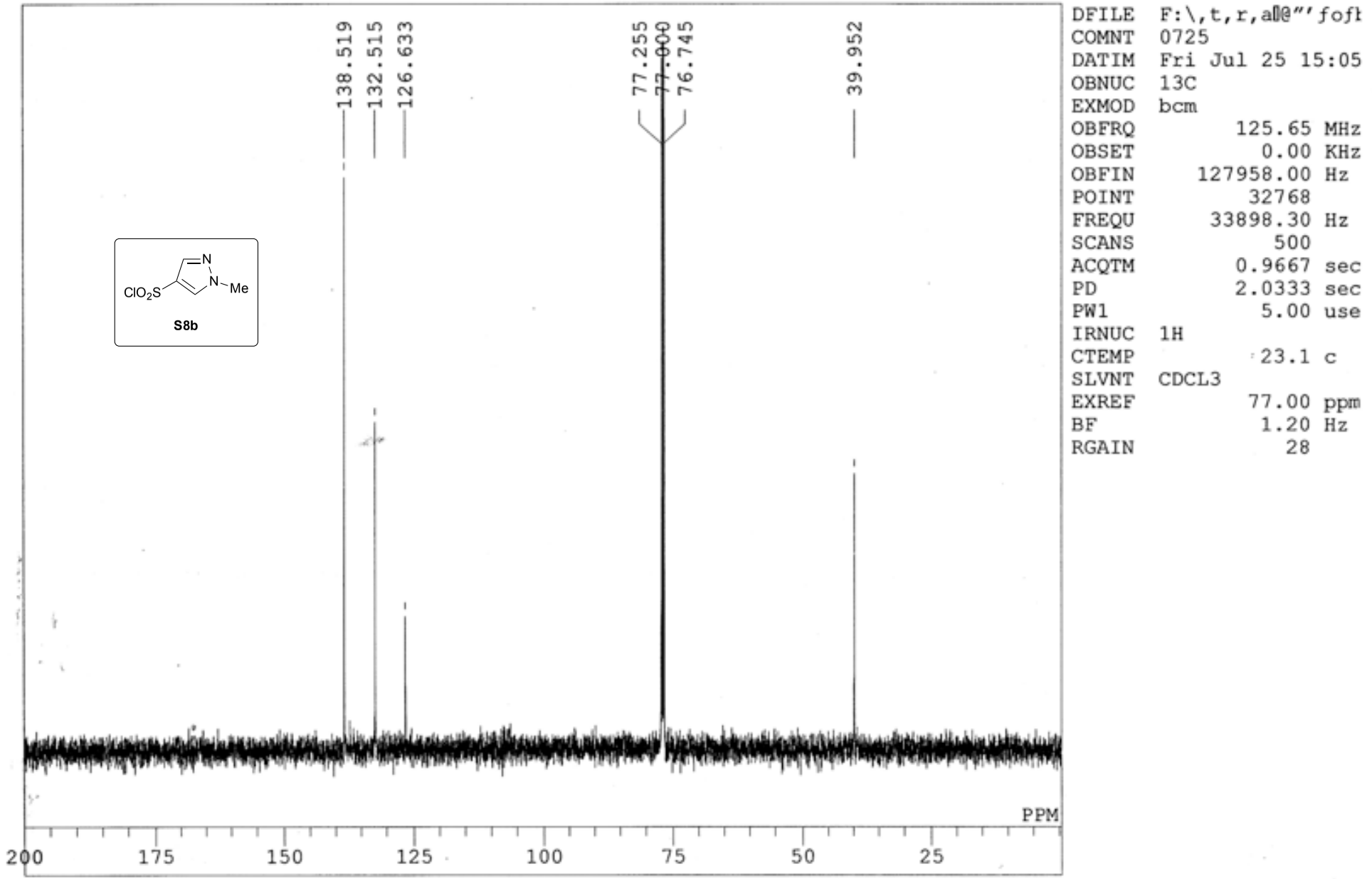


DFILE G:\,t,r,a[@"'fofl
COMNT
DATIM Tue Sep 21 21:30
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 2.0000 sec
PW1 5.70 use
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19




```
DFILE  G:\,t,r,a@"'foft
COMNT  0922
DATIM  Tue Sep 21 23:12
OBNUC  13C
EXMOD  bcm
OBFRQ        125.65 MHz
OBSET          0.00 KHz
OBFIN     127958.00 Hz
POINT         32768
FREQU      33898.30 Hz
SCANS            20
ACQTM        0.9667 sec
PD           2.0333 sec
PW1            5.00 use
IRNUC  1H
CTEMP          24.6 c
SLVNT  CDCL3
EXREF          77.00 ppm
BF             1.20 Hz
RGAIN            26
```


DFILE F:\,t,r,a@"'fofi
COMNT
DATIM Thu Sep 16 14:46
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.00 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 5.3620 sec
PW1 5.70 use
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 22
PPM
0.000
2.74
4.011
3.29
7.263
0.83
8.030
7.970
8.06
S8b
N
N
Me
ClO2S



DFILE F:\,t,r,a@"'fofb
COMNT 0725
DATIM Fri Jul 25 15:05
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 500
ACQTM 0.9667 sec
PD 2.0333 sec
PW1 5.00 use
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 28