Supporting Information

Toward the total synthesis of elisapterosin B: A $Hg(OTf)_2$-promoted diasteroselective intramolecular Friedel-Crafts alkylation reaction

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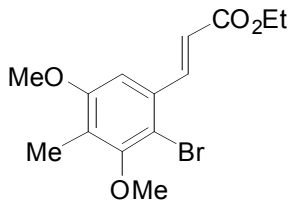
General information:

All reactions were carried out under an atmosphere of nitrogen in flame-dried glassware. THF was distilled over sodium-benzophenone before use. Acetonitrile and dichloromethane were distilled over calcium hydride. Chromatographic separations were carried out using Sorbent® standard silica gel (230-400 mesh). Analytical thin layer chromatography was performed on Sigma-Aldrich silica gel TLC plates with UV indicator.

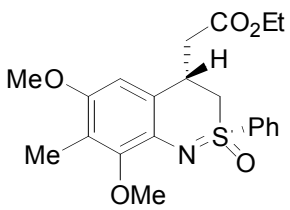
Infrared spectra were recorded (film) on a Perkin Elmer 1600 series FT-IR spectrometer. Optical rotations were measured on a Jasco DIP-370 digital polarimeter. ^{1}H NMR were recorded on a Bruker ARX-250 (250 MHz), DRX-300 (300 MHz), DRX-500 (500 MHz) spectrometer and are reported in ppm (δ) from tetramethylsilane (TMS: δ 0.0 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet, ddd = doublet of doublet of doublet), coupling constants (Hz), and integration. ^{13}C NMR spectra were recorded on a Bruker ARX-250 (62.5 MHz), DRX-300 (75 MHz), and DRX-500 (125 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with solvent resonance as the internal standard (CDCl3: δ 77.0 ppm).

Experimental Procedure and Analytical Data

(*E*)-ethyl 3-(2-bromo-3,5-dimethoxy-4-methylphenyl)-acrylate (**10**):



To triethyl phosphonoacetate (23.06 mL, 25.90 g, 115.55 mmol) in 200 mL of THF at 0 °C, *n*-BuLi (2.3 M in Hexanes, 46.37 mL, 106.66 mmol) was added dropwise. Then a solution of starting material aldehyde **7** (22.93 g, 88.88 mmol) in 100 mL of THF was added dropwise. The reaction was allowed to warm to room temperature and stirred for another 4 h before being quenched with 100 mL of water. The mixture was extracted with ethyl acetate (3x 100 mL). The combined organic layers were washed with brine and dried with Na_2SO_4. Filtration and concentration gave 31.6 g of crude product. The crude product was subjected to flash silica gel chromatography (20:80, EtOAc:hexanes) to obtain the pale yellow product in 100% yield. IR: 3089, 3072, 2995, 2974, 2946, 2901, 2844, 1712, 1626, 1581, 1552, 1471, 1438, 1385, 1328, 1270, 1246 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.08 (d, *J* = 15.9 Hz, 1H), 6.87 (s,1H), 6.35 (d, *J* = 15.9 Hz, 1H), 4.29 (q, *J* = 7.2 Hz, 2H), 3.85 (s, 3H), 3.78 (s, 3H), 2.21 (s, 3H), 1.35 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.4, 157.7, 156.1, 143.4, 132.7, 124.1, 120.2, 112.3, 104.6, 60.6, 60.3, 55.7, 14.3, 9.9; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{14}H_{17}BrNO_4Na^+$ 351.0202; Found 351.0201.

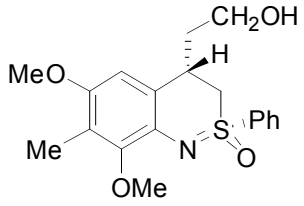


ethyl [(4*S*)-6,8-dimethoxy-7-methyl-2-oxido-2-phenyl-3,4-dihydro-2λ^4,1-benzo-thiazin-4-yl]acetate (**6**):

A 300 mL flame dried round-bottomed flask was charged with **10** (29.15 g, 88.87 mmol), sulfoximine (*S*)-**11** (1.2 equiv, 16.55 g, 106.64 mmol), Cs_2CO_3 (1.4 equiv, 40.54 g, 124.4 mmol),

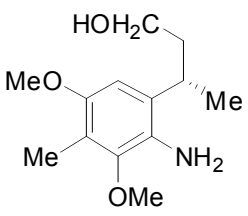
BINAP (0.075 equiv, 4.150g, 6.66 mol) and $Pd(OAc)_2$ (0.05 equiv, 0.998 g, 4.44 mmol) and freshly distilled toluene (180 mL). After degassing with N_2 for 10 minutes, the reaction mixture was refluxed for 48 h. It was cooled, filtered through Celite and concentrated. The crude product was filtered through a short pad of silica gel with ethyl acetate. The eluent was dried with $MgSO_4$, filtered through Celite, concentrated and transferred to a flame-dried round-bottomed flask. After removal of solvents from this crude product on a vacuum pump, 600 mL of freshly distilled THF was added. The solution was cooled down to -78 ^oC for 20 minutes. Then LiHMDS (2 equiv, 1.0 M in THF, 178 mL) was added dropwise over 30 minutes. The reaction was stirred for another 1 h at -78 ^oC before quenched with 200 mL of water. The reaction was extracted with ethyl acetate (3x 200 mL), and the organics washed with brine, dried with Na_2SO_4, and concentrated. Flash silica gel chromatography (50:50, EtOAc:hexanes) afforded 30.45 g of pale yellow semisolid **6** in 85% yield over two steps. IR: 3068, 2991, 2925, 2831, 1724, 1601, 1565, 1467, 1442, 1409, 1250, 1115 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 8.12 (td, *J* = 1.6, 6.9 Hz, 2H), 7.65 (tt, *J* = 1.4, 7.0 Hz, 1H), 7.54 (tt, *J* = 1.6, 7.0 Hz, 2H), 6.47 (s, 1H), 4.05 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 3.80-3.70 (m, 4H), 3.60-3.35 (m, 2H), 2.90-2.65 (m, 2H), 2.18 (s, 3H), 1.16 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 171.1, 152.4, 152.1, 139.6, 133.5, 131.6, 129.3, 128.8, 123.1, 120.2, 103.9, 60.7, 60.4, 55.9, 53.2, 37.0, 32.8, 14.1, 9.0; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{21}H_{25}NO_5SNa^+$ 426.1346; Found 426.1338.

2-[(4*S*)-6,8-dimethoxy-7-methyl-2-oxido-2-phenyl-3,4-dihydro-2λ^4,1-benzothiazin-4-yl]ethanol:



To a solution of **6** (11.58 g, 28.72 mmol) in THF at 0 ^oC in a flame dried flask under N_2, $LiAH_4$ (1.2 equiv, 1.309 g, 34.47 mmol) was added. The reaction was stirred at this temperature for another 30 minutes before being quenched with ethyl acetate (20 mL) and water (80-100 mL). The stirred mixture was warmed to room temperature. The reaction was extracted with ethyl acetate (3 x 100 mL), washed with brine, dried with Na_2SO_4, and concentrated, and the crude product was purified by silica gel chromatography (75:25, EtOAc:hexanes) to give a pale yellow semisolid (9.874 g, 95%). IR: 3444, 3064, 2991, 2938, 2831, 1605, 1569, 1442, 1405, 1254, 1111 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.05 (td, *J* = 1.5, 7.2 Hz, 2H), 7.61 (tt, *J* = 1.5, 7.2 Hz, 1H), 7.52 (tt, *J* = 1.2, 6.9 Hz, 2H), 6.48 (s, 1H), 3.85 (s, 3H), 3.75 (s, 3H), 3.65-3.48 (m, 3H), 3.48-3.32 (m, 1H), 3.19 (dd, *J* = 8.7, 13.2 Hz, 1H), 2.72 (s, 1H), 2.30-2.10 (m, 4H), 1.80-1.60 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 152.2, 151.7, 139.5, 133.5, 131.3, 129.2, 128.6, 124.2, 119.6, 104.5, 60.3, 59.4, 55.9, 53.4, 34.4, 32.6, 8.9; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{19}H_{23}NO_4SNa^+$ 384.1240; Found 384.1230.

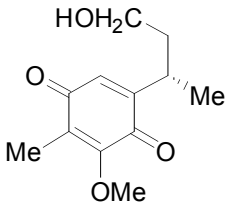
(*S*)-3-(2-amino-3,5-dimethoxy-4-methylphenyl)butan-1-ol (**12**):



A flame dried round-bottomed flask was charged with Na (10 equiv, 230 mg, 10 mmol) and a magnetic stir bar, and equipped with condenser and N_2 balloon. With vigorous stirring, the flask was heated until the Na was complete melted. While vigorously stirring, Hg (4.37 g) was added onto Na the sand bath was removed. With

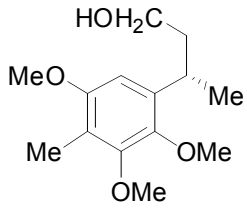
vigorous stirring, the 5% Na/Hg amalgam (10 equiv) was broken into small pieces and cooled down to room temperature. To this same flask, MeOH (16 mL) and then Na_2HPO_4 (10 equiv, 1.42 g) were added. After 10 minutes, a solution of benzothiazine (1 mmol in 4 mL of THF) was added. The yellowish mixture was stirred for 5-8 h and turned to light grey. After filtration through Celite and removal of solvent, the crude product was diluted with water (3 mL) and saturated NH_4Cl (3 mL). The mixture was extracted with EtOAc (3x 10 mL), dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (80:20, EtOAc:hexanes) to give **12** (216 mg, 90%); pale yellow oil; IR: 3361, 2935, 2833, 1582, 1486, 1461, 1418, 1363, 1287, 1230, 1192, 1130 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 6.44 (s, 1H), 3.76 (s, 3H), 3.71 (s, 3H), 3.66-3.44 (m, 4H), 3.35-3.23 (m, 1H), 3.22-3.04 (m, 1H), 2.14 (s, 3H), 2.00-1.80 (m, 1H), 1.65-1.45 (m, 1H), 1.30 (d, J = 7.0 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 152.2, 147.6, 130.8, 130.6, 117.7, 104.4, 60.7, 59.9, 56.3, 41.6, 29.0, 21.0, 9.2; HRMS: m/z $[M + Na]^+$ calcd for $C_{13}H_{21}O_3Na^+$ 262.1414, found 262.1411.

(*S*)-5-(4-hydroxybutan-2-yl)-3-methoxy-2-methylcyclohexa-2,5-diene-1,4-dione (13):



To a solution of ceric ammonium nitrate (CAN, 4 equiv, 2.193 g, 4 mmol) in water (33 mL) and MeCN (28 mL) at room temperature, a solution of aniline (1 mmol) in MeCN (5 mL) was added dropwise over 0.5 h, then water (33 mL) was added. The mixture was extracted with EtOAc (3x 40 mL). The combined organic layers were washed with water (2x 35 mL) and brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (75:25, EtOAc:hexanes) to give **13** as a pale yellow oil (170 mg, 76%); IR: 3407, 2942, 2876, 1667, 1646, 1605, 1446, 1373, 1340, 1315, 1270, 1201, 1136 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 6.42 (s, 1H), 3.95 (s, 3H), 3.65-3.45(m, 2H), 3.20-2.95 (m, 1H), 2.15 (s, 1H), 1.89 (s, 3H), 1.68 (q, J = 6.5 Hz, 2H), 1.12 (d, J = 7.0 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 188.8, 183.5, 156.3, 152.1, 131.2, 128.7, 60.9, 60.6, 39.4, 28.4, 19.6, 8.6; HRMS: m/z $[M + Na]^+$ calcd for $C_{12}H_{16}O_4Na$ 247.0941, found 247.0940.

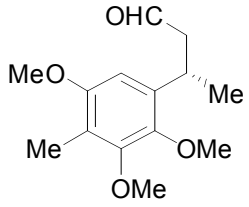
(*S*)-3-(2,3,5-trimethoxy-4-methylphenyl)butan-1-ol (14):



To a solution of quinone **13** (166 mg, 0.74 mmol) and Bu_4NBr (0.5 equiv, 119 mg, 0.37 mol) in THF (10.9 mL) at room temperature under N_2, a solution of $Na_2S_2O_4$ (6 equiv, 85%, 909 mg, 4.44 mmol) in water (5.4 mL) was added. After stirring for 15 minutes, a solution of KOH (30 equiv, 1.245 g, 22.2 mmol) in water (3.5 mL) was added. After 15 minutes, Me_2SO_4 (10 equiv, 933 mg, 0.704 mL, 7.4 mmol) was added. The reaction was stirred for another 10 h. The mixture was extracted with EtOAc (3x 15 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (40:60, EtOAc:hexanes) to give **14** as a pale yellow oil (151 mg, 83%). IR: 3416, 2954, 2929, 2864, 1605, 1581, 1479, 1462, 1397, 1336, 1230, 1189, 1123 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$): δ 6.43 (s, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 3.79 (s, 3H), 3.60-3.45 (m, 1H), 3.45-3.25 (m, 2H), 2.45 (dd, J = 4.5, 7.3 Hz, 1H), 2.11 (s, 3H),

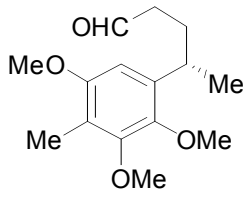
2.00-1.80 (m, 1H), 1.63-1.42 (m, 1H), 1.29 (d, J = 7.0 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 154.7, 151.6, 144.6, 136.4, 118.5, 103.1, 61.2, 60.8, 60.3, 55.7, 41.33, 28.0, 21.6, 8.7; HRMS: m/z $[M + Na]^+$ calcd for $C_{14}H_{22}O_4Na$ 277.1410, found 277.1411.

(*S*)-3-(2,3,5-trimethoxy-4-methylphenyl)butanal:



To a solution of $(ClCO)_2$ (1.3 equiv, 350 mL, 0.518 mg, 4.078 mmol) in CH_2Cl_2 (20 mL) at -78 °C, DMSO (2.6 equiv, 0.579 mL, 8.156 mmol) was added dropwise. After stirring for 5 minutes, a solution of **14** (798 mg, 3.137 mmol) in CH_2Cl_2 (10 mL) was added dropwise. The reaction was stirred for another 15 minutes before Et_3N (5 equiv, 2.3 mL, 15.685 mmol) was added dropwise. The reaction was warmed to room temperature and quenched with water (30 mL).The mixture was extracted with CH_2Cl_2 (3x 25 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (20:80, EtOAc:hexanes) to give a pale yellow oil (752 mg, 95%). IR: 2966, 2933, 2827, 1728, 1605, 1581, 1487, 1458, 1401, 1344, 1225, 1123 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$): δ 9.70 (s, 1H), 6.41 (s, 1H), 3.82 (s, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 3.71 (dd, J = 7.3, 14.4 Hz, 1H), 2.80-2.55 (m, 2H), 2.10 (s ,3H), 1.30 (d, J = 7.0 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 201.9, 154.1, 151.9, 144.3, 135.5, 118.9, 103.4, 60.7, 59.9, 55.6, 51.1, 27.7, 21.4, 8.6; HRMS: m/z $[M + Na]^+$ calcd for $C_{14}H_{20}O_4Na$ 275.1254, found 275.1255.

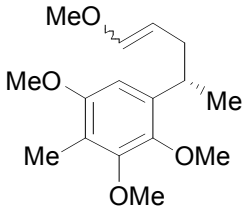
(*S*)-4-(2,3,5-trimethoxy-4-methylphenyl)pentanal (**15**):



To a solution of diisopropylamine (1.42 equiv, 1.219 mL, 8.7 mmol) in THF (20 mL) *n*-BuLi (2.5 M in Hexanes, 3.408 mL, 8.52 mmol) was added dropwise at -20 °C and the solution was stirred for 15 minutes. This colorless LDA solution was added into a solution of methoxymethyl triphenylphosphonium chloride (1.5 equiv, 3.085 g, 9.0 mmol) in THF (30 mL) at 0 °C. Upon addition of LDA, the reaction turned red and was stirred for another 10 minutes and cooled down to -78 °C. To this solution, a solution of (*S*)-3-(2,3,5-trimethoxy-4-methylphenyl)butanal (1.505 g, 5.965 mmol) in THF (10 mL) was added dropwise. The reaction was stirred at -78 °C for another 0.5 h and warmed to room temperature. The reaction was quenched by addition of water (20 mL) and extracted with EtOAc (3x 25 mL) and concentrated. The crude intermediate was used in the next step. The crude intermediate ((*S*)-1,3,4-trimethoxy-5-(5-methoxypent-4-en-2-yl)-2-methylbenzene) was dissolved in acetone (60 mL) and water (2 mL). *p*-Toluenesulfonic acid (3.0 equiv, 17.895 mmol) was added. After 15 minutes, water was added and the reaction was warmed to room temperature and quenched with water (180 mL).The mixture was extracted with EtOAc (3x 100 mL). The combined organic layers were washed with saturated $NaHCO_3$ and brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (10:90, EtOAc:hexanes) to give **15** as a pale yellow oil (1.375 g, 89% over two steps). IR: 2958, 2933. 2868, 2831, 2721, 1724, 1605, 1581, 1487, 1458, 1401, 1225, 1123 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.70 (s, 1H), 6.40 (s, 1H), 3.81 (s, 3H), 3.79 (s, 3H), 3.77 (s, 3H), 3.30-3.10 (m, 1H), 2.48-2.35 (m, 1H), 2.35-

2.26 (m, 1H), 2.11 (s, 3H), 1.99-1.89 (m, 1H), 1.89-1.78 (m, 1H), 1.26 (d, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 202.4, 154.3, 151.8, 144.8, 136.4, 118.6, 103.1, 60.8, 60.1, 55.7, 42.2, 31.6, 29.8, 21.8, 8.7; HRMS: m/z $[M + Na]^+$ calcd for $C_{15}H_{22}O_4Na$ 289.1410, found 289.1408.

(*S*)-1,3,4-trimethoxy-5-(5-methoxypent-4-en-2-yl)-2-methylbenzene:

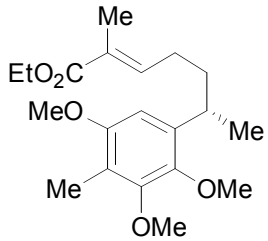


A mixture of two diastereomers, dr = 1.6: 1; IR: 2995, 2958, 2929, 2827, 1654, 1605, 1585, 1487, 1462, 1405, 1225, 1127 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.49 (s, 1H, minor), 6.43 (s, 1H, major), 6.29 (d, J = 12.5 Hz, 1H, major), 5.88 (d, J = 6.5 Hz, 1H, minor), 4.68 (td, J = 6.0, 12.5 Hz, 1H, major), 4.31 (q, J = 6.5 Hz, 1H, minor), 3.83 (s, 6H), 3.81 (s, 6H), 3.80 (s, 3H, minor), 3.79 (s, 3H, major), 3.57 (s, 3H, minor), 3.48 (s, 3H, major), 3.24-3.12 (m, 2H), 2.42-2.26 (m, 2H), 2.26-2.17 (m, 1H, major), 2.16-2.06 (m, 7H), 1.22 (d, J = 7.0 Hz, 3H, minor), 1.21 (d, J = 7.0 Hz, 3H, major); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.1, 154.1, 151.8, 151.7, 147.8, 146.6, 144.6, 137.9, 137.7, 118.1, 118.0, 105.4, 103.6, 103.6, 101.3, 61.0, 61.0, 60.2, 60.2, 59.4, 55.8, 55.8, 55.7, 36.0, 33.3, 32.5, 31.5, 30.3, 21.7, 20.9, 8.7; HRMS: m/z $[M + Na]^+$ calcd for $C_{16}H_{24}O_4Na$ 303.1567, found 303.1564.

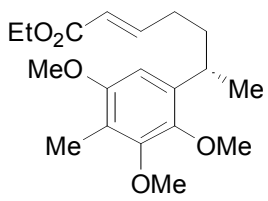
General Procedure Roush Modified Horner-Wadsworth-Emmons Reaction

A flame-dried flask was charged with LiCl (1.2 equiv, 1.2 mmol) and MeCN (5 mL), followed by addition of triethyl 2-phosphonopropionate (1.2 equiv, 1.2 mmol) (or triethyl phosphonoacetate (1.2 equiv, 1.2 mmol)) and DBU (1.1 equiv, 1.1 mmol). Then a solution of **15** (1 equiv, 1 mmol) in MeCN (5 mL) was added. The reaction was stirred at room temperature for 0.5 h before being diluted with Et_2O (30 mL), washed with H_2O (2x 20 mL) and dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (15:85, EtOAc:hexanes) to give pale yellow oil enoate.

(*S*,*E*)-ethyl 2-methyl-6-(2,3,5-trimethoxy-4-methylphenyl)hept-2-enoate:



pale yellow oil, 93%; IR: 2958, 2933, 2868, 2831, 1708, 1650, 1605, 1585, 1487, 1458, 1405, 1266, 1225, 1131 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.76 (qt, J = 1.5, 7.2 Hz, 1H), 6.42 (s, 1H), 4.17 (q, J = 7.2 Hz, 2H), 3.81 (s, 3H), 3.79 (s, 3H), 3.78 (s, 3H), 3.28-3.10 (m, 1H), 2.22-2.00 (m, 5H), 1.82-1.62 (m, 5H), 1.28 (t, J = 7.2 Hz, 3H), 1.23 (d, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.1, 154.2, 151.7, 144.7, 142.1, 137.2, 127.6, 118.2, 103.2, 60.8, 60.2, 60.0, 55.6, 36.3, 32.0, 27.0, 21.9, 14.2, 12.2, 8.6; HRMS: m/z $[M + Na]^+$ calcd for $C_{20}H_{30}O_5Na$ 373.1985, found 373.1975.



(*S*,*E*)-ethyl 6-(2,3,5-trimethoxy-4-methylphenyl)hept-2-enoate:

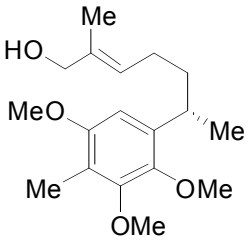
pale yellow oil, 87%; IR: 2966, 2933, 2864, 2831, 1716, 1654, 1487, 1467, 1401, 1262, 1189, 1127 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.95 (td, J = 7.0. 15.5 Hz, 1H), 6.40 (s, 1H), 5.79 (d, J

= 15.5Hz, 1H), 4.15 (q, *J* = 7.5 Hz, 2H), 3.81 (s, 3H), 3.79 (s, 3H), 3.77 (s, 3H), 3.22-3.10 (m, 1H), 2.24-2.05 (m, 5H), 1.80-1.65 (m, 2H), 1.27 (t, *J* = 7.5 Hz, 3H), 1.23 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.6, 154.2, 151.8, 149.0, 144.7, 136.9, 121.1, 118.3, 103.2, 60.8, 60.0, 59.9, 55.6, 35.9, 31.9, 30.4, 21.8, 14.2, 8.6; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{19}H_{28}O_5Na$ 359.1829, found 359.1822.

General Procedure for DIBAL Reduction, Synthesis of 5a and 5b

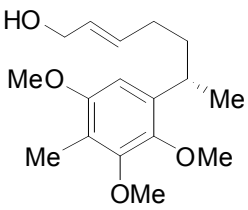
To a solution of enoate (1 mmol) in toluene (7 mL) at -78 °C under N_2, DIBAL (3 equiv, 1.0 M in toluene, 3 mmol) was added dropwise. After stirring for 2 hours, the reaction was quenched with EtOAc (5 mL) and water (10 mL). The mixture was extracted with EtOAc (3x 10 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (20:80, EtOAc:hexanes) to give **5a** or **5b**.

(*S*,*E*)-2-methyl-6-(2,3,5-trimethoxy-4-methylphenyl)hept-2-en-1-ol (**5a**):



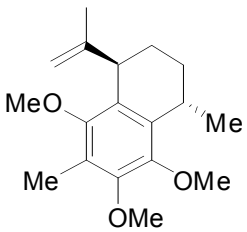
97%, colorless oil; IR: 3420, 2954, 2929, 2852, 1605, 1581, 1487, 1458, 1405, 1230, 1131 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.42 (s, 1H), 5.39 (t, *J* = 7.0 Hz, 1H), 3.96 (s, 2H), 3.81 (s, 3H), 3.79 (s, 3H), 3.78 (s, 3H), 3.22-3.13 (m, 1H), 2.11 (s, 3H), 2.07-1.88 (m, 2H), 1.70-1.58 (m, 3H), 1.56 (s, 3H), 1.22 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.2, 151.7, 144.6, 137.7, 134.7, 126.3, 118.0, 103.3, 68.9, 60.9, 60.1, 55.7, 37.2, 31.6, 25.8, 22.2, 13.5, 8.6; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{18}H_{28}O_4Na$ 331.1880, found 331.1874.

(*S*,*E*)-6-(2,3,5-trimethoxy-4-methylphenyl)hept-2-en-1-ol (**5b**):



91%, colorless oil; IR: 3420, 2954, 2933, 2848, 1605, 1581, 1487, 1450, 1397, 1344, 1230, 1127, 1037 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.42 (s, 1H), 5.66 (td, *J* = 6.5, 15.0 Hz, 1H), 5.56 (td, *J* = 6.0, 15.5 Hz, 1H), 4.04 (d, *J* = 6.0 Hz, 2H), 3.81(s, 3H), 3.79 (s, 3H), 3.77 (s, 3H), 3.25-3.12 (m, 1H), 2.11 (s, 3H), 2.06-1.90 (m, 2H), 1.80 (s, 1H), 1.72-1.59 (m, 2H), 1.21 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.2, 151.7, 144.6, 137.6, 133.1, 129.0, 118.1, 103.3, 63.7, 61.0, 60.2, 55.7, 36.9, 31.5, 30.4, 22.2, 8.7; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{17}H_{26}O_4Na$ 317.1723, found 317.1720.

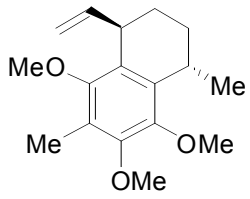
(1*R*,4*S*)-5,6,8-trimethoxy-4,7-dimethyl-1-(prop-1-en-2-yl)-1,2,3,4-tetrahydronaphthalene (**4**):



A flame-dried flask was charged with $Hg(OTf)_2$ (0.005 equiv, 0.1 M in MeCN, 0.0139 mL, 0.001394 mmol). After removal of MeCN on vacuum pump, the flask was vented twice with N_2 and charged with 4.6 mL of toluene and followed by addition of a solution of **5a** (86 mg, 0.279 mmol) in toluene (1 mL). The mixture was heated at 45 °C

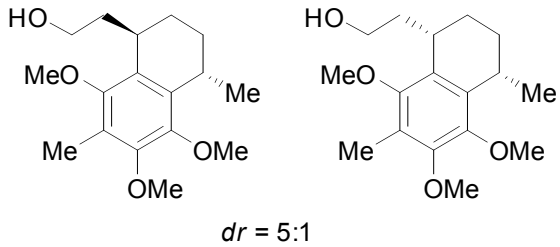
for 4 h. The reaction was cooled down to room temperature and filtered through Celite and concentrated. The crude product (NMR shows *dr* = 9:1) was carefully purified by silica gel column (5:95, EtOAc:hexanes) to give **4** as a colorless oil (44 mg, 54%); two diastereomers, *dr* = 10:1; IR: 3068, 2933, 2860, 2823, 1646, 1589, 1454, 1405, 1332, 1279, 1246, 1103, 1066 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 4.81 (s, 1H, minor), 4.79 (S, 1H, major), 4.43 (s, 1H, minor), 3.94 (s, 1H, major), 3.85 (s, 3H, major), 3.84 (s, 3H, minor), 3.82 (s, 3H, minor), 3.81 (s, 3H, major), 3.67 (s, 3H, major), 3.63 (s, 3H, minor), 3.54-3.48 (m, 2H), 3.40-3.31 (m, 1H, minor), 3.17-3.00 (m, 1H, major), 2.18 (s, 3H, minor), 2.16 (s, 3H, major), 1.97-1.85 (m, 10H), 1.66-1.60 (m, 2H), 1.44-1.36 (m, 2H), 1.26 (d, *J* = 7.0 Hz, 3H, minor), 1.19 (d, *J* = 7.0 Hz, 3H, major); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.4, 152.3, 150.2, 150.0, 150.0, 149.8, 147.4, 146.9, 134.6, 134.3, 128.0, 127.8, 122.2, 121.9, 111.6, 110.8, 60.4, 60.3, 60.1, 60.0, 59.8, 59.8, 41.7, 39.8, 28.1, 27.2, 25.2, 24.5, 23.3, 23.0, 22.3, 22.1, 20.5, 20.4, 9.6, 9.4; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{18}H_{26}O_3Na^+$ 313.1774, found 313.1772.

(1*R*,4*S*)-5,6,8-trimethoxy-4,7-dimethyl-1-vinyl-1,2,3,4-tetrahydronaphthalene (16):



A flame-dried flask was charged with $Hg(OTf)_2$ (0.02 equiv, 0.1 M in MeCN, 0.0543 mL, 0.00543 mmol). After removal of MeCN on vacuum pump, the flask was vented twice with N_2 and charged with 4.5 mL of toluene and followed by addition of a solution of **5b** (79.6 mg, 0.270 mmol) in toluene (0.9 mL). The mixture was heated at 70 °C for 15 minutes. The reaction was cooled down to room temperature and filtered through Celite and concentrated. The crude product (NMR shows *dr* = 6.3:1) was carefully purified by silica gel chromatography (5:95, EtOAc:hexanes) to give **16** (55 mg, 73%). Major isomer: IR: 3077, 2938, 2864, 2827, 1634, 1589, 1458, 1405, 1319, 1246, 1103, 1066 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 5. 97 (ddd, *J* = 6.0, 10.2, 16.8 Hz, 1H), 5.00 (td, *J* = 1.6, 10.5 Hz, 1H), 4.62 (td, *J* = 1.5, 17.1 Hz, 1H), 3.85 (s, 3H), 3.81 (s, 3H), 3.80-3.74 (m, 1H), 3.68 (s, 3H), 3.20-3.00 (m, 1H), 2.18 (s, 3H), 2.10-1.80 (m, 2H), 1.70-1.60 (m, 1H), 1.50-1.40 (m, 1H), 1.19 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 152.7, 150.1, 147.3, 143.1, 134.2, 126.3, 122.4, 113.9, 60.6, 60.4, 59.8, 36.4, 27.3, 24.6, 22.4, 9.6; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{17}H_{24}O_3Na$ 299.1618, found 299.1623.

2-((1*R*,4*S*)-5,6,8-trimethoxy-4,7-dimethyl-1,2,3,4-tetrahydronaphthalen-1-yl)ethanol (**17**):

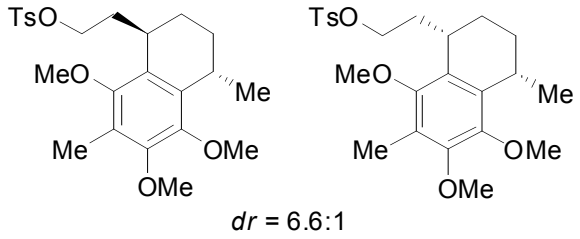


dr = 5:1

To a solution of **16** (98 mg, 0.355 mmol, *dr* = 5:1) in THF (3 mL) at 0 °C, BH_3·THF (2 equiv, 1.0 M in THF, 0.709 mL, 0.709 mmol) was added dropwise. The reaction was warmed to room temperature and stirred for another 1h and cooled down to 0 °C before an aqueous NaOH solution (5.2 equiv, 2M, 0.922 mL, 1.844 mmol) was added carefully, followed by addition of H_2O_2 (5 equiv, 30% in water, 0.183 mL, 1.773

mmol). The reaction was stirred for another 2 h at room temperature and quenched with water (3 mL). The mixture was extracted with EtOAc (3x 6 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (30:70, EtOAc:hexanes) to give 17 as a colorless oil (74 mg, 71%, *dr* = 5:1). IR: 3314, 2929, 2864, 2819, 1589, 1454, 1405, 1303, 1254, 1119, 1099, 1066 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 3.85 (s, 3H, major), 3.82 (s, 3H, minor), 3.81 (s, 3H, minor). 3.79 (s, 3H, major), 3.70 (s, 3H, major), 3.69-3.53 (m, 7H), 3.20-3.07 (m, 2H), 3.07-2.97 (m, 2H), 2.39 (s, 2H, broad), 2.18 (s, 3H, minor), 2.17 (s, 3H, major), 2.00-1.86 (m, 4H), 1.85-1.55 (m, 6H), 1.55-1.47 (m, 2H), 1.26 (d, *J* = 7.0 Hz, 3H, minor), 1.18 (d, *J* = 7.0 Hz, 3H, major); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.1, 151.9, 149.8, 149.6, 148.0, 147.4, 134.1, 133.7, 129.6, 129.3, 122.3, 121.8, 61.3, 61.0, 60.6, 60.5, 60.3, 59.9, 59.8, 39.6, 39.2, 28.8, 28.7, 27.6, 27.3, 27.2, 27.0, 24.7, 23.4, 22.6, 21.5, 9.5, 9.3; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{17}H_{26}O_4Na$ 317.1723, found 317.1723.

2-((1*R*,4*S*)-5,6,8-trimethoxy-4,7-dimethyl-1,2,3,4-tetrahydronaphthalen-1-yl)ethyl 4-methylbenzenesulfonate (**18**):



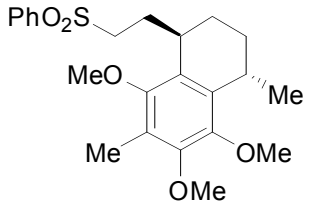
dr = 6.6:1

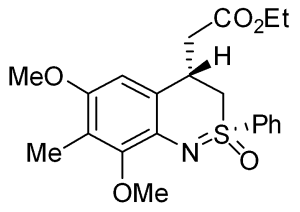
To a solution of **17** (77 mg, 0.261 mmol) in CH_2Cl_2 (2.6 mL) at 0 °C, DMAP (0.1 equiv, 3.2 mg, 0.0262 mmol), Et_3N (5 equiv, 0.184 mL, 1.31 mmol) and TsCl (1.3 equiv, 65 mg, 0.341 mmol) were added. The reaction was warmed to room temperature and stirred for 2 h before being quenched with water (3 mL). The mixture was extracted with CH_2Cl_2 (3x 5 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (20:80, EtOAc:hexanes) to give **18** as a white semisolid (94 mg, 84%, two diastereomers mixture, *dr* = 6.6: 1). IR: 2933, 2868, 2827, 1597, 1462, 1401, 1356, 1299, 1254, 1193, 1176, 1099, 1070 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.82 (d, *J* = 8.5 Hz, 2H, major), 7.81 (d, *J* = 8.5 Hz, 2H, minor), 7.35 (d, *J* = 8.5 Hz, 4H), 4.22-4.10 (m, 4H), 3.83 (s, 3H, major), 3.81 (s, 3H, minor), 3.79 (s, 3H, minor), 3.78 (s, 3H, major), 3.64 (s, 3H, major), 3.61 (s, 3H, minor), 3.14-3.04 (m, 2H), 3.03-2.87 (m, 2H), 2.45 (s, 6H), 2.18 (s, 3H, minor), 2.15 (s, 3H, major), 2.00-1.90 (m, 2H), 1.88-1.72 (m, 4H), 1.72-1.62 (m, 2H), 1.62-1.52 (m, 2H), 1.52-1.40 (m, 2H), 1.20 (d, *J* = 7.0 Hz, 3H, minor), 1.17 (d, *J* = 7.0 Hz, 3H, major); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.4, 152.3, 149.9, 149.8, 147.4, 147.1, 144.5, 134.5, 133.6, 133.3, 129.7, 128.8, 128.7, 127.8, 122.7, 122.4, 70.0, 69.6, 60.3, 60.0, 60.0, 59.7, 35.3, 34.4, 29.4, 29.0, 27.7, 27.6, 27.1, 25.0, 24.5, 22.3, 22.1, 21.6, 20.3, 9.4, 9.3; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{24}H_{32}O_6SNa$ 471.1812, found 471.1809.

(1*R*,4*S*)-5,6,8-trimethoxy-4,7-dimethyl-1-(2-(phenylsulfonyl)ethyl)-1,2,3,4-tetrahydronaphthalene (**19**):

A mixture of tosylate **18** (145 mg, 0.323 mmol), thiophenol (2 equiv, 0.066 mL, 0.646 mmol), and anhydrous K_2CO_3 (3 equiv, 134 mg, 0.97 mmol) in dry DMSO (3.2 mL)

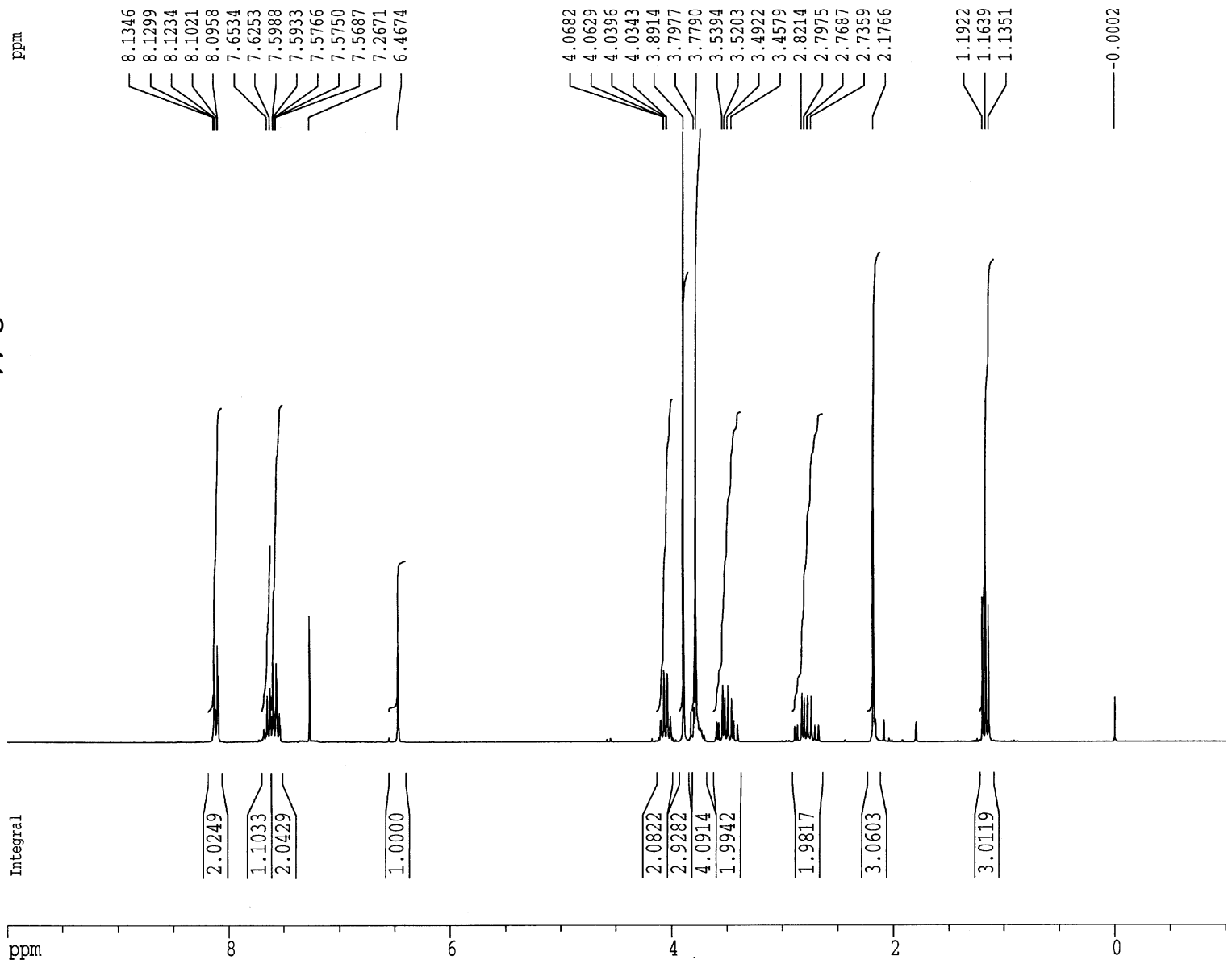
under N_2 were stirred at 30 ^oC for 3 h. The reaction mixture was poured into water (15 mL). The mixture was extracted with Et_2O (3x 15 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. To a solution of crude sulfide in dry CH_2Cl_2 (3 mL) at 0 ^oC under N_2, was added *m*CPBA (3 equiv, 70-75%, 167 mg, 0.969 mmol). The reaction was stirred for another 17 h and quenched with water (4 mL). The mixture was extracted with CH_2Cl_2 (3x 5 mL). The combined organic layers were washed with brine, dried with $MgSO_4$ and concentrated. The crude product was purified by silica gel chromatography (20:80, EtOAc:hexanes) to give **19** as a white solid (85 mg, 63%, two diastereomers mixture, *dr* = 6.6: 1). After recrystalization from CH_2Cl_2/MeOH, colorless needle crystals were obtained (the major isomer); m.p. 121-123 ^oC, IR: 2954, 2933, 2864, 2827, 1585, 1462, 1446, 1401, 1307, 1242, 1140, 1066 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.91 (d, *J* = 7.5 Hz, 2H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.55 (d, *J* = 7.5 Hz, 2H), 3.83 (s, 3H), 3.78 (s, 3H), 3.59 (s, 3H), 3.30-3.15 (m, 2H), 3.13-3.03 (m, 1H), 2.93-2.80 (m, 1H), 2.13 (s, 3H), 2.04-1.93 (m, 1H), 1.90-1.68 (m, 3H), 1.56-1.50 (m, 1H), 1.49-1.41 (m, 1H), 1.15 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.4, 150.1, 147.2, 139.3, 133.6, 133.5, 129.2, 128.1, 127.9, 122.7, 60.4, 60.4, 59.8, 55.4, 31.6, 28.2, 27.1, 24.4, 22.3, 20.7, 9.5; HRMS: *m*/*z* $[M + Na]^+$ calcd for $C_{23}H_{30}O_5SNa$ 441.1706, found 441.1703.





6

1H NMR



Current Data Parameters

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PROCNO	1

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F2 - Processing parameters

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1D NMR plot parameters

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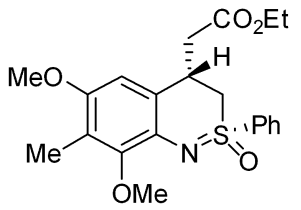

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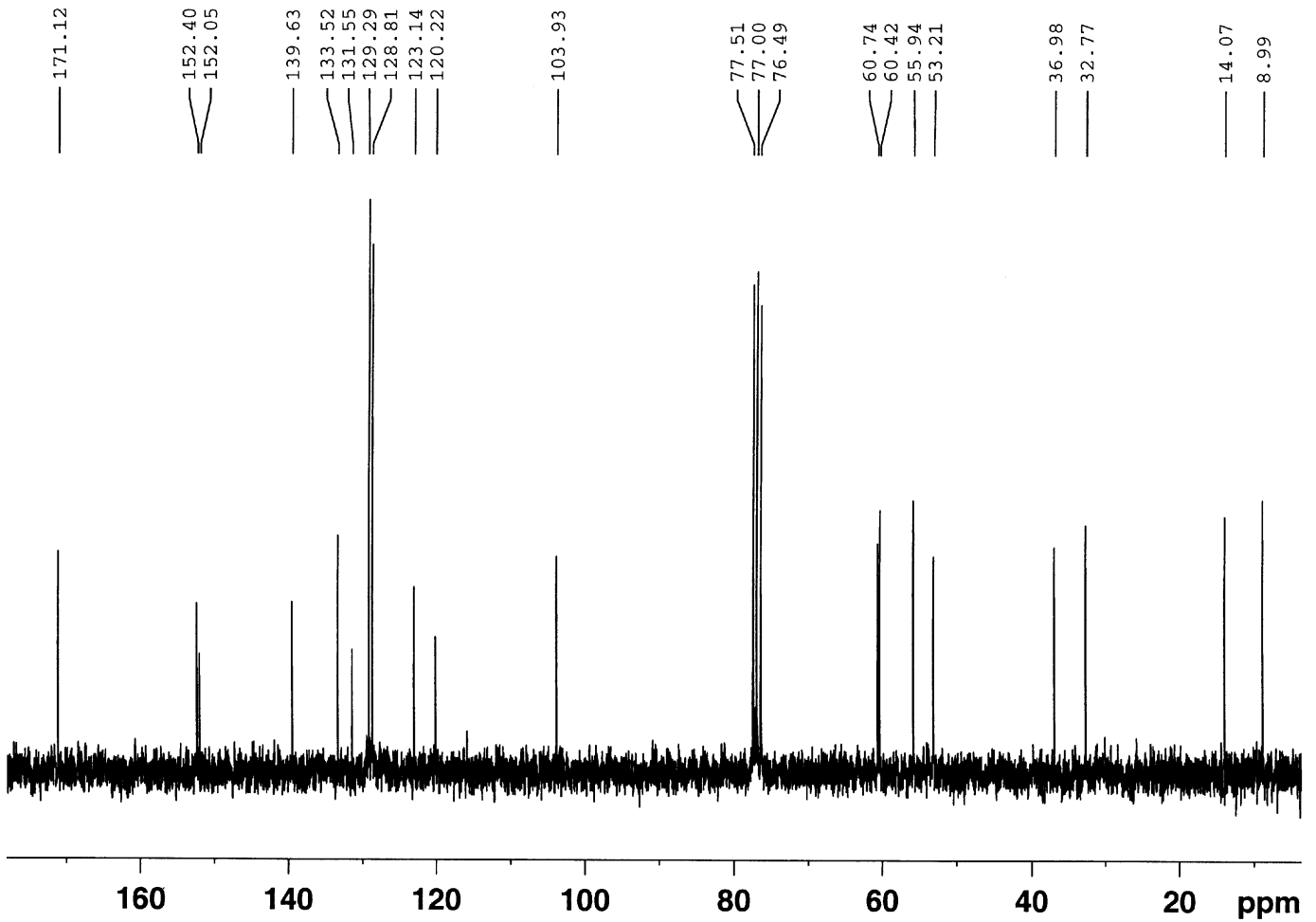

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F2 - Processing parameters

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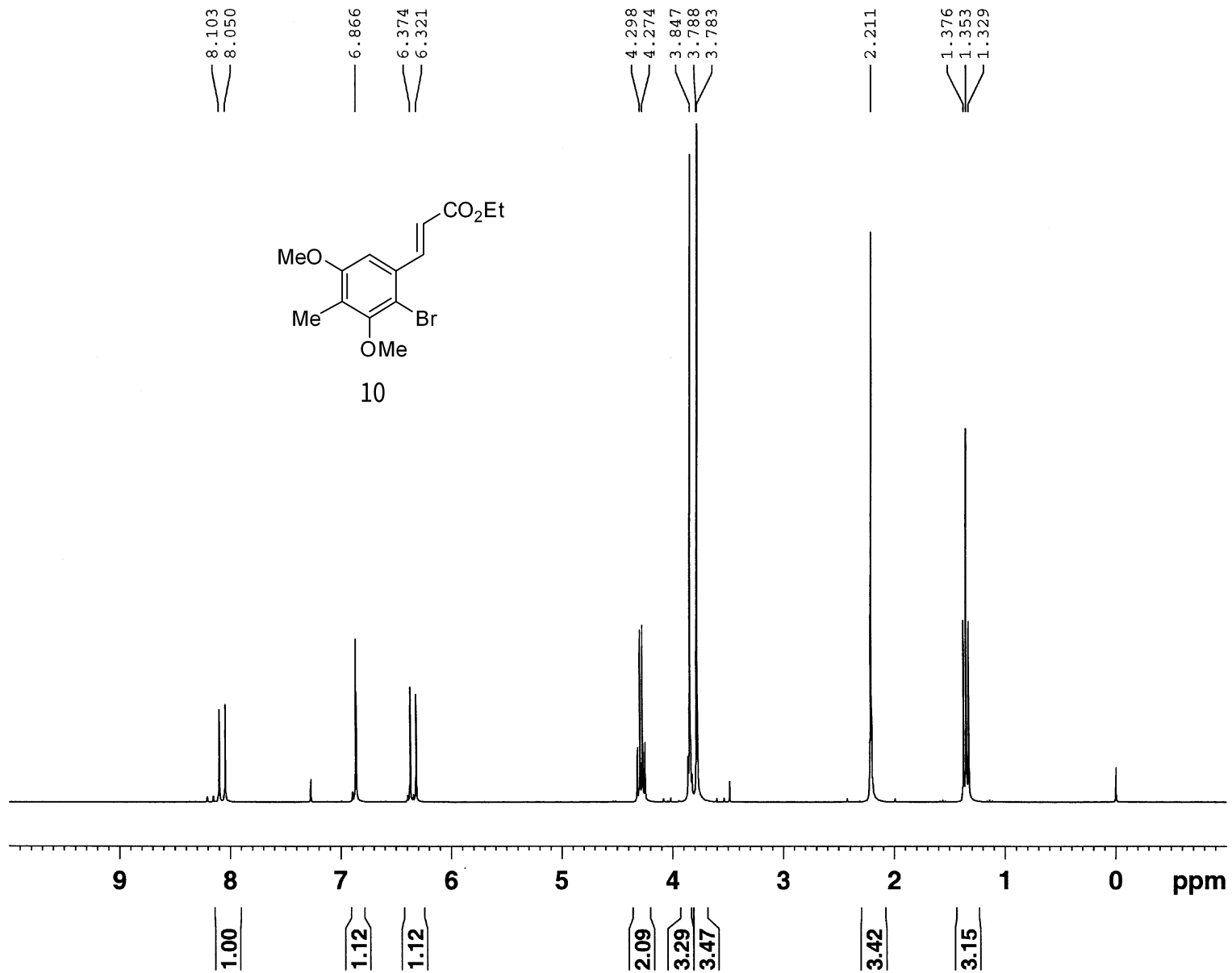
1H NMR

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PL1                0.00 dB
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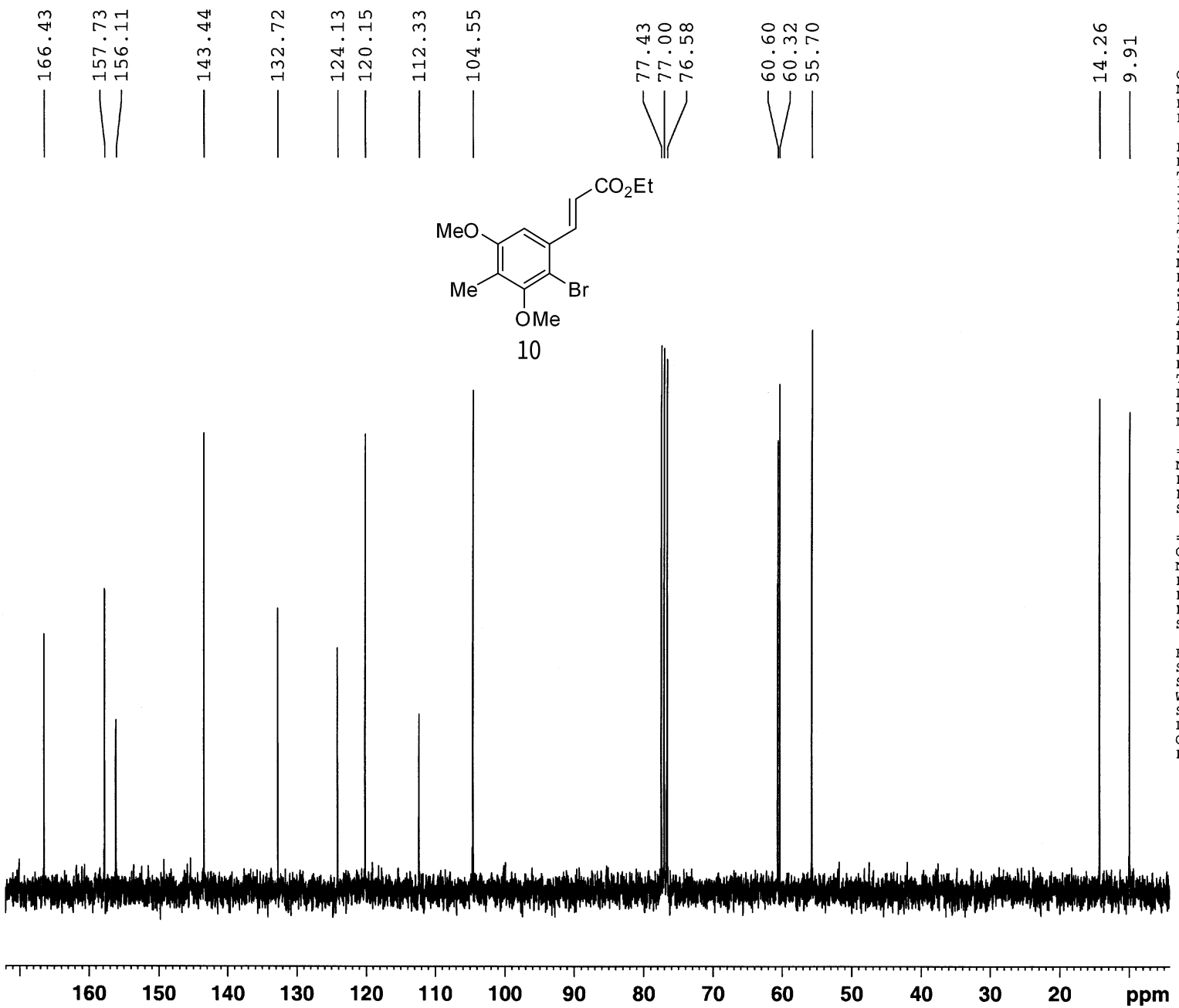
13C NMR

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PL2                 120.00 dB
PL12                 21.41 dB
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F2 - Processing parameters
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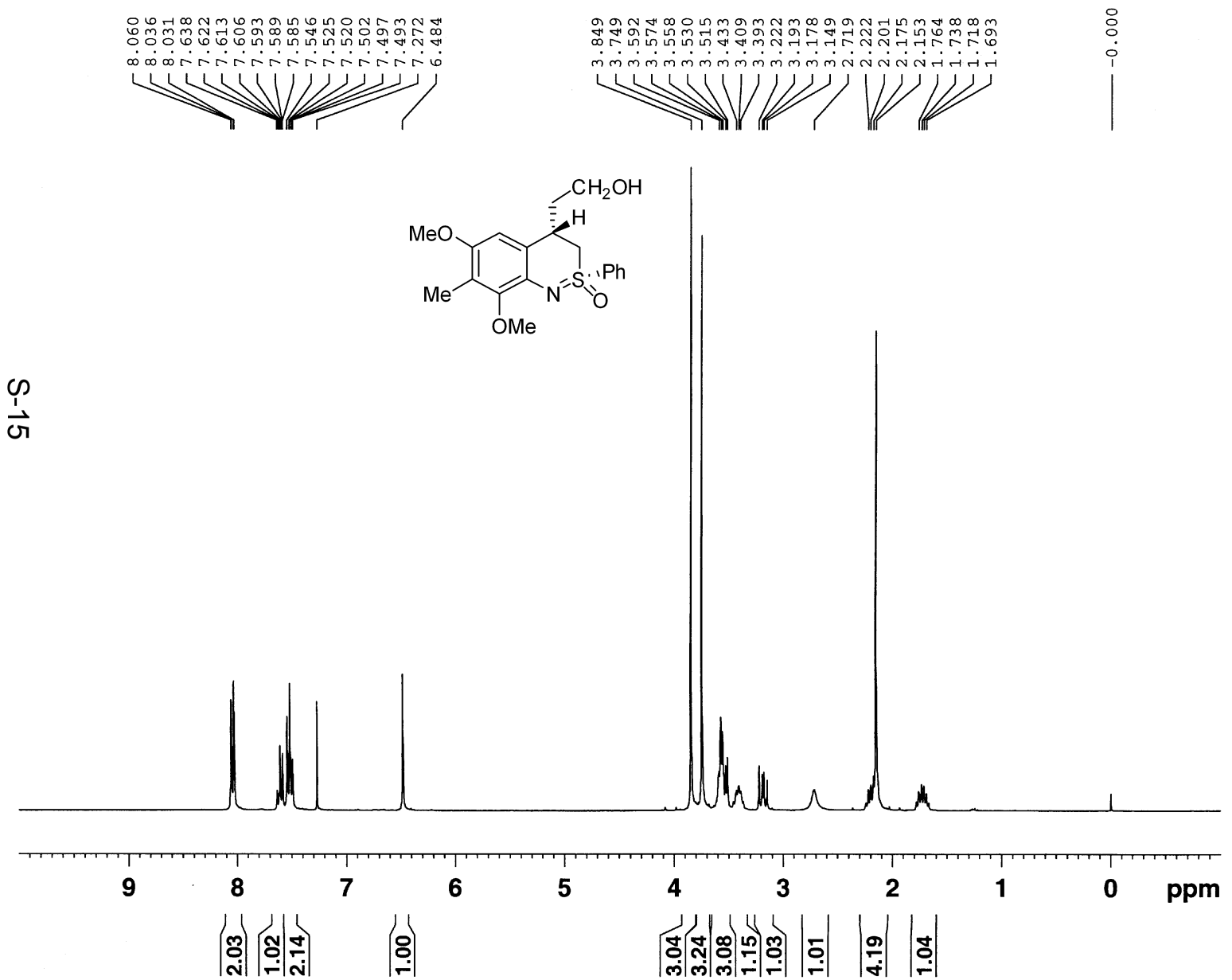
PC                     1.40
```

1H NMR



S-15

Current Data Parameters

NAME	wy-VI-8-A2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090504
Time	23.25
INSTRUM	DRX300
PROBHD	5 mm Multinucl
PULPROG	zg30pad
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	28.5
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
D31	0.00000000 sec

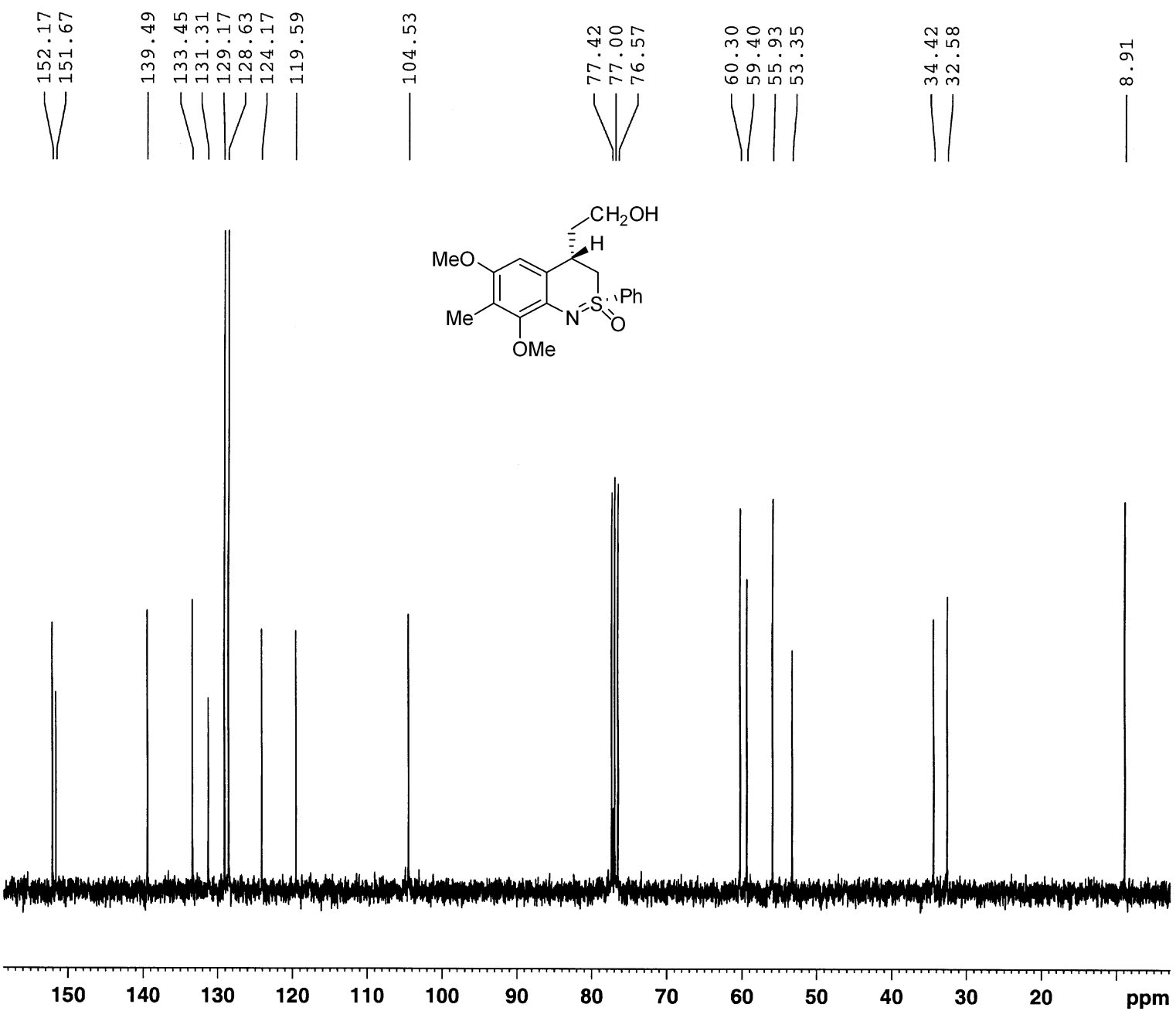
======== CHANNEL f1 ========

NUC1	1H
P1	7.05 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300026 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

13C NMR



Current Data Parameters
NAME wy-VI-8-A2
EXPNO 2
PROCNO 1

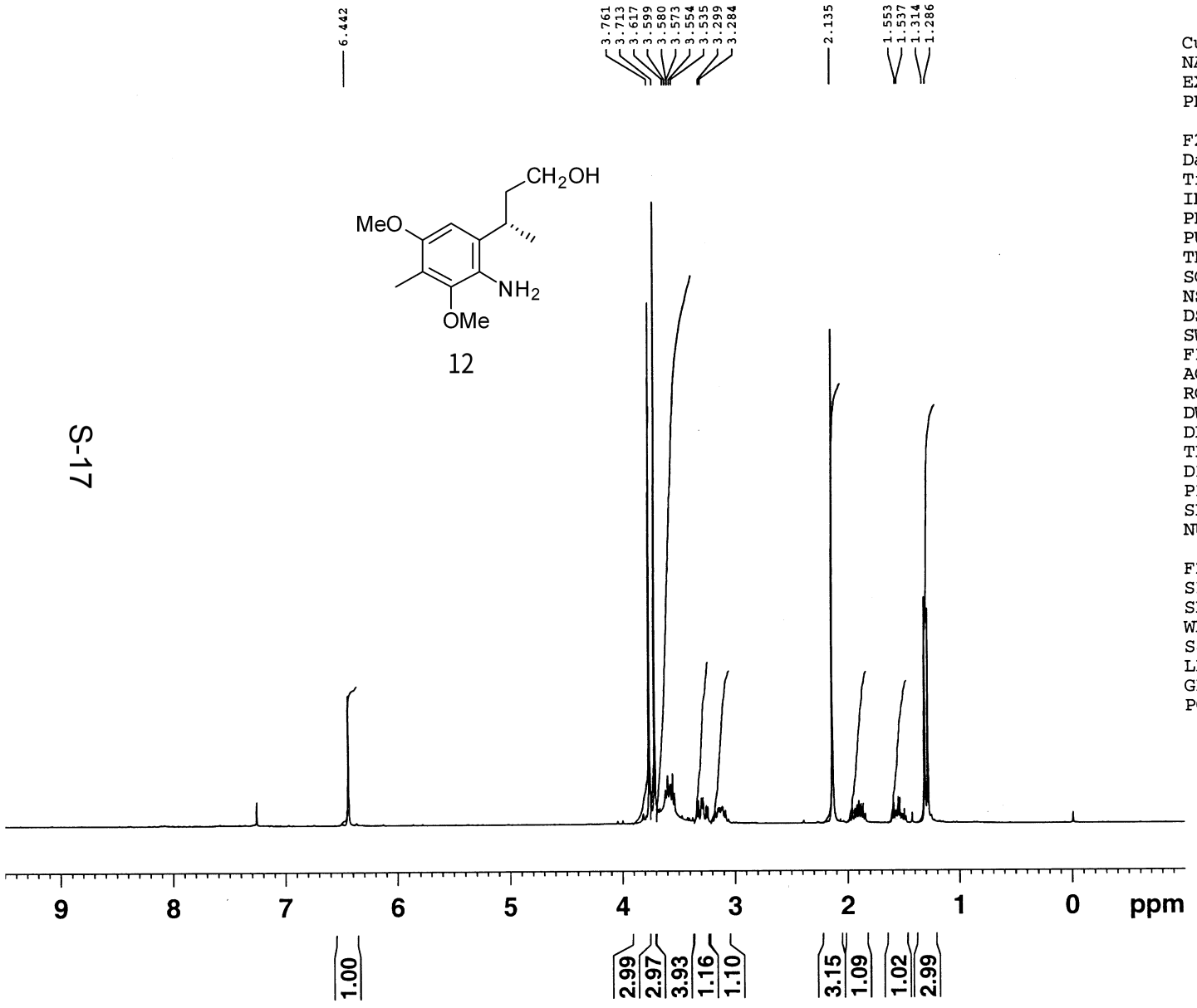
F2 - Acquisition Parameters
Date_ 20090504
Time 23.32
INSTRUM DRX300
PROBHD 5 mm Multinucl
PULPROG zgdc30pad
TD 65536
SOLVENT CDCl3
NS 51
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 22528
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D31 0.00000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 5.00 dB
SFO1 75.4760107 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 21.41 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677623 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H NMR



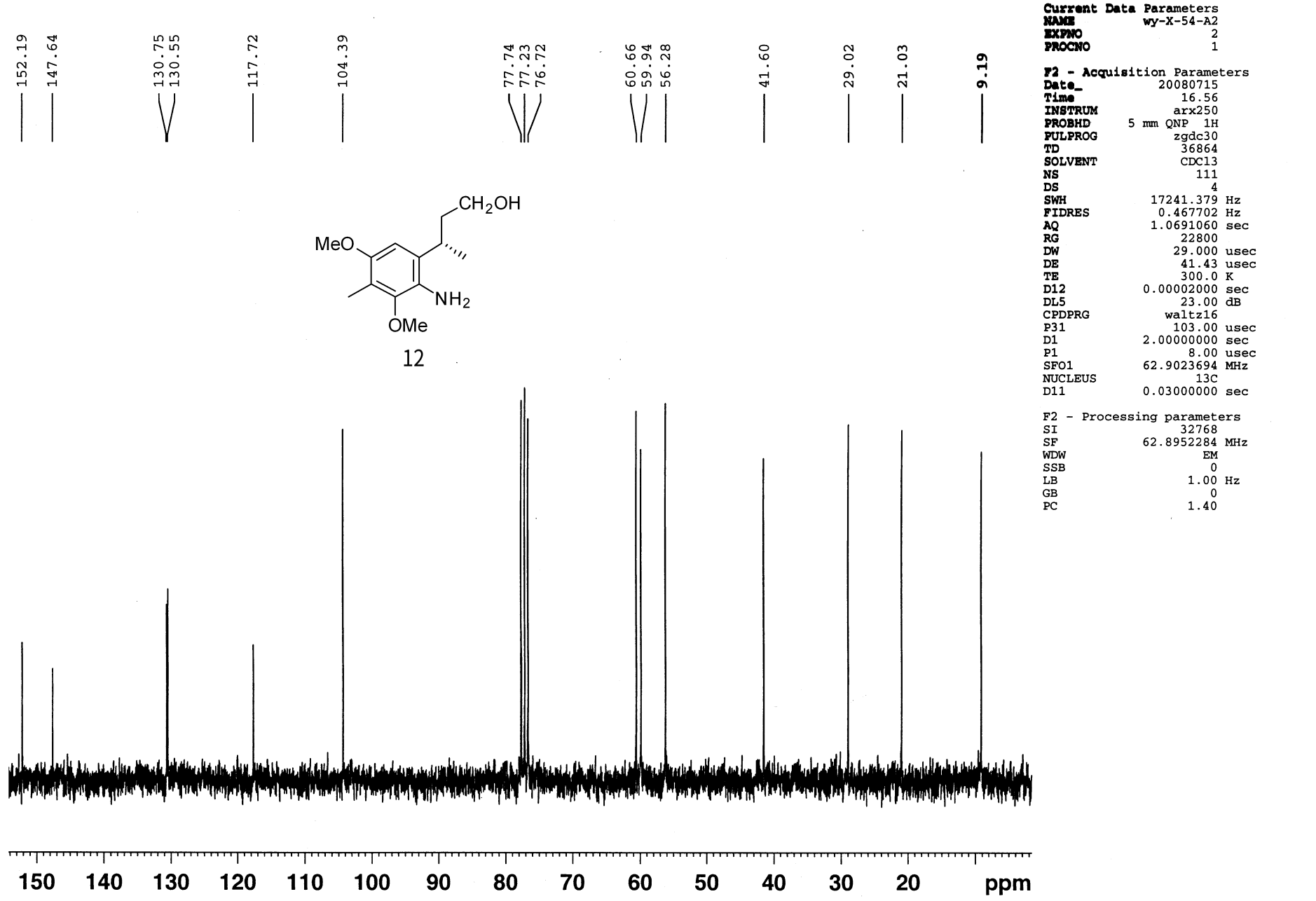
S-17

```
Current Data Parameters
NAME          wy-X-54-A2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080715
Time               16.53
INSTRUM          arx250
PROBHD     5 mm QNP  1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            5208.333 Hz
FIDRES        0.158946 Hz
AQ           3.1457779 sec
RG                  512
DW               96.000 usec
DE               137.14 usec
TE                300.0 K
D1           1.00000000 sec
P1                 9.50 usec
SFO1       250.1315321 MHz
NUCLEUS              1H

F2 - Processing parameters
SI                16384
SF       250.1300073 MHz
WDW                  EM
SSB                   0
LB                 0.20 Hz
GB                    0
PC                 1.50
```

13C NMR



Current Data Parameters

NAME	wy-X-54-A2
EXPNO	2
PROCNO	1

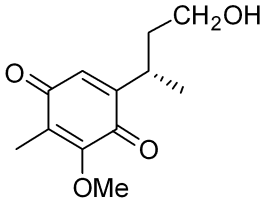
F2 - Acquisition Parameters

Date_	20080715
Time	16.56
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	111
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 usec
DE	41.43 usec
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 usec
D1	2.00000000 sec
P1	8.00 usec
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952284 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1H NMR



13

Current Data Parameters

NAME	wy-X-55-A
EXPNO	1
PROCNO	1

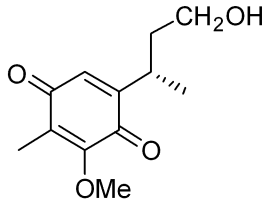
F2 - Acquisition Parameters

Date_	20080716
Time	14.43
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	256
DW	96.000 usec
DE	137.14 usec
TE	300.0 K
D1	1.00000000 sec
P1	9.50 usec
SFO1	250.1315321 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	250.1300076 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

13C NMR

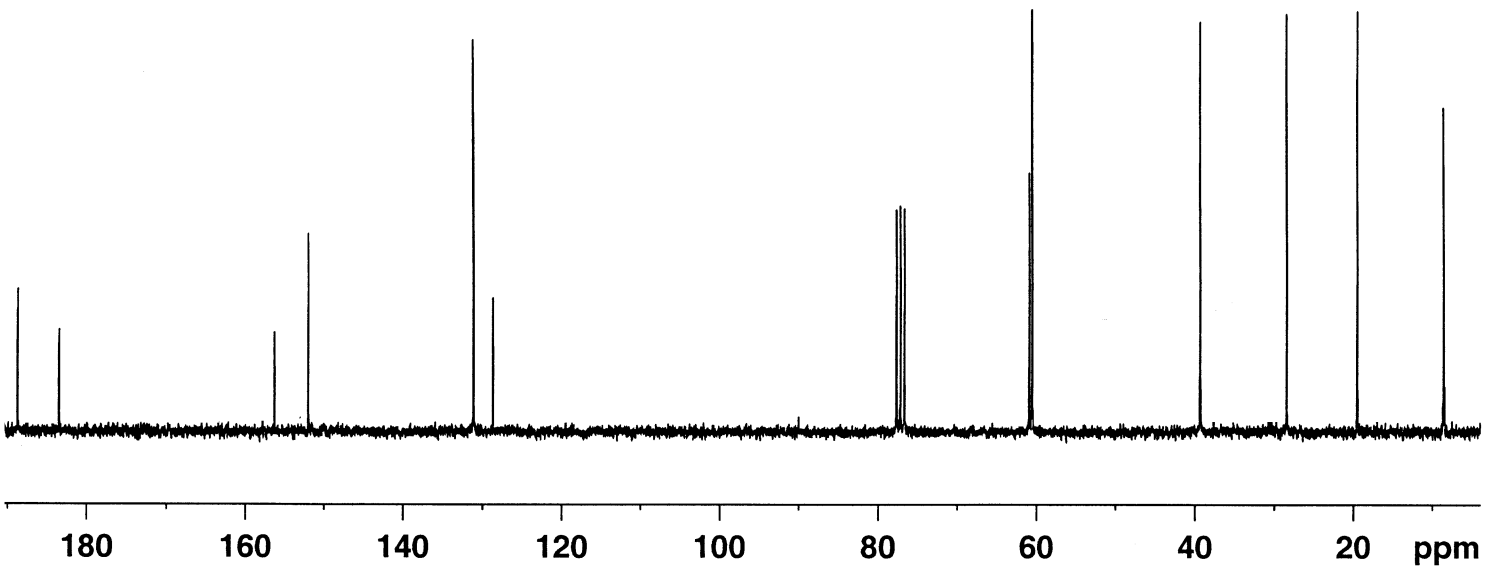


13

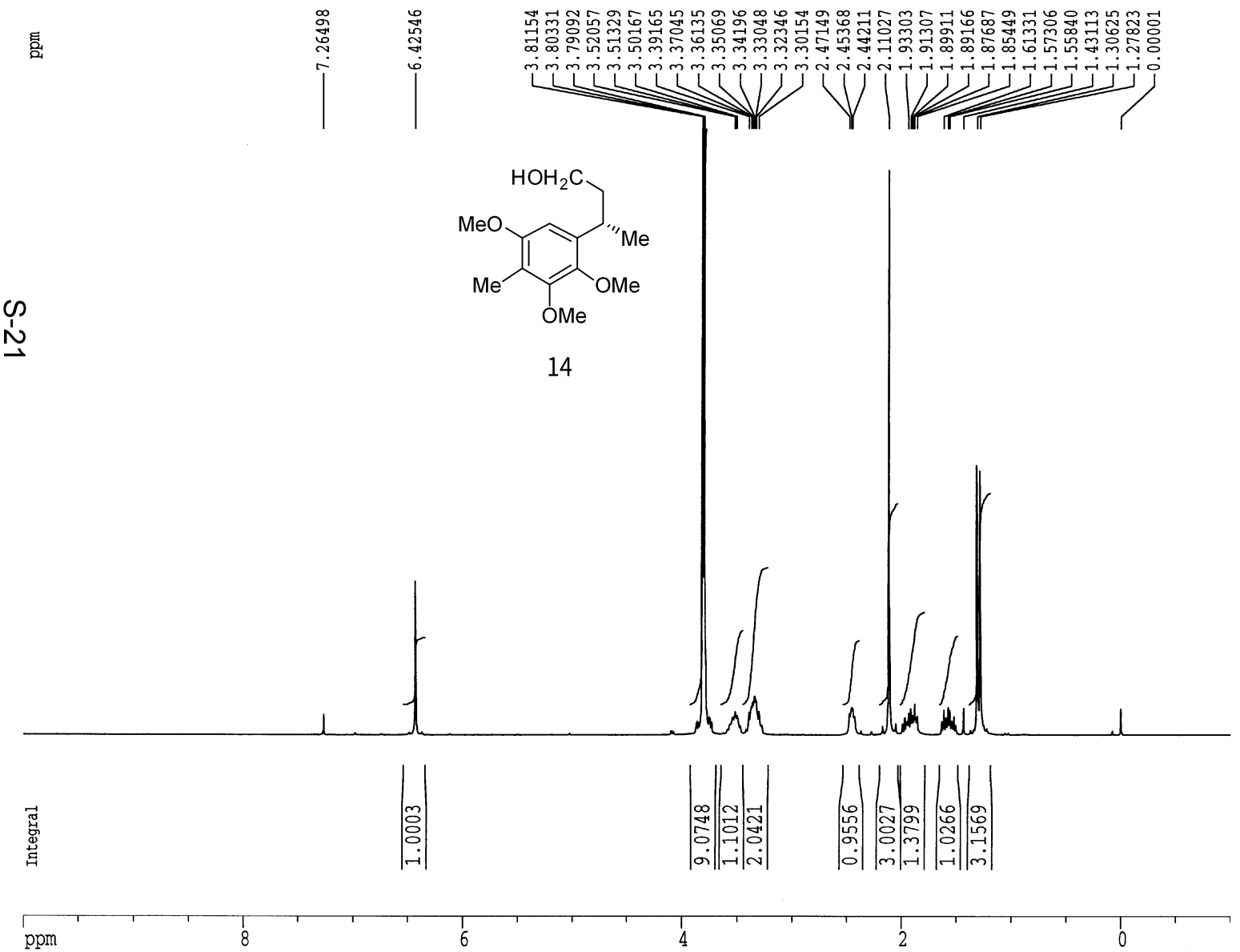

```
Current Data Parameters
NAME                  wy-X-55-A
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080716
Time                      14.59
INSTRUM                  arx250
PROBHD        5 mm QNP  1H
PULPROG                 zgdc30
TD                       36864
SOLVENT                  CDCl3
NS                         319
DS                           4
SWH                  17241.379 Hz
FIDRES                0.467702 Hz
AQ                    1.0691060 sec
RG                       22800
DW                      29.000 usec
DE                      41.43 usec
TE                      300.0 K
D12                0.00002000 sec
DL5                     23.00 dB
CPDPRG                 waltz16
P31                     103.00 usec
D1                 2.00000000 sec
P1                       8.00 usec
SFO1                62.9023694 MHz
NUCLEUS                    13C
D11                0.03000000 sec

F2 - Processing parameters
SI                       32768
SF                  62.8952300 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



1H NMR



Current Data Parameters

NAME	wy-X-78-A
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080731
Time	11.31
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	512
DW	96.000 usec
DE	137.14 usec
TE	300.0 K
D1	1.00000000 sec
P1	9.50 usec
SFO1	250.1315321 MHz
NUCLEUS	1H

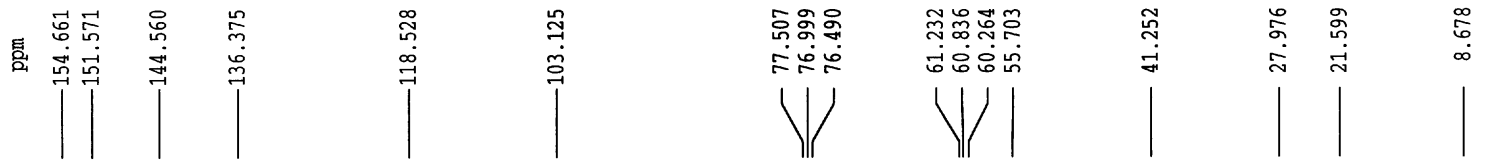
F2 - Processing parameters

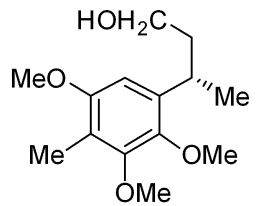
SI	16384
SF	250.1300062 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

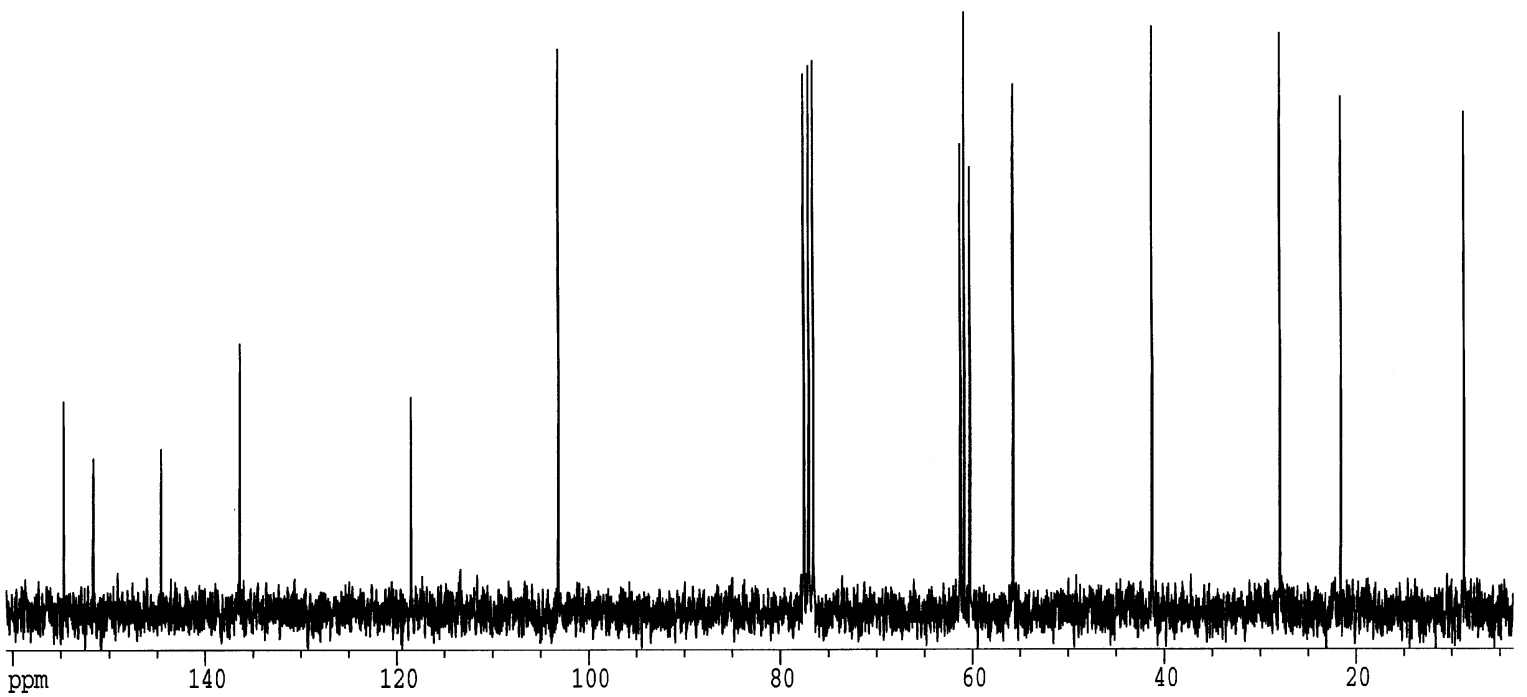
CX	20.00 cm
CY	12.50 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-1.000 ppm
F2	-250.13 Hz
PPMCM	0.55000 ppm/cm
HZCM	137.57150 Hz/cm

13C NMR





14



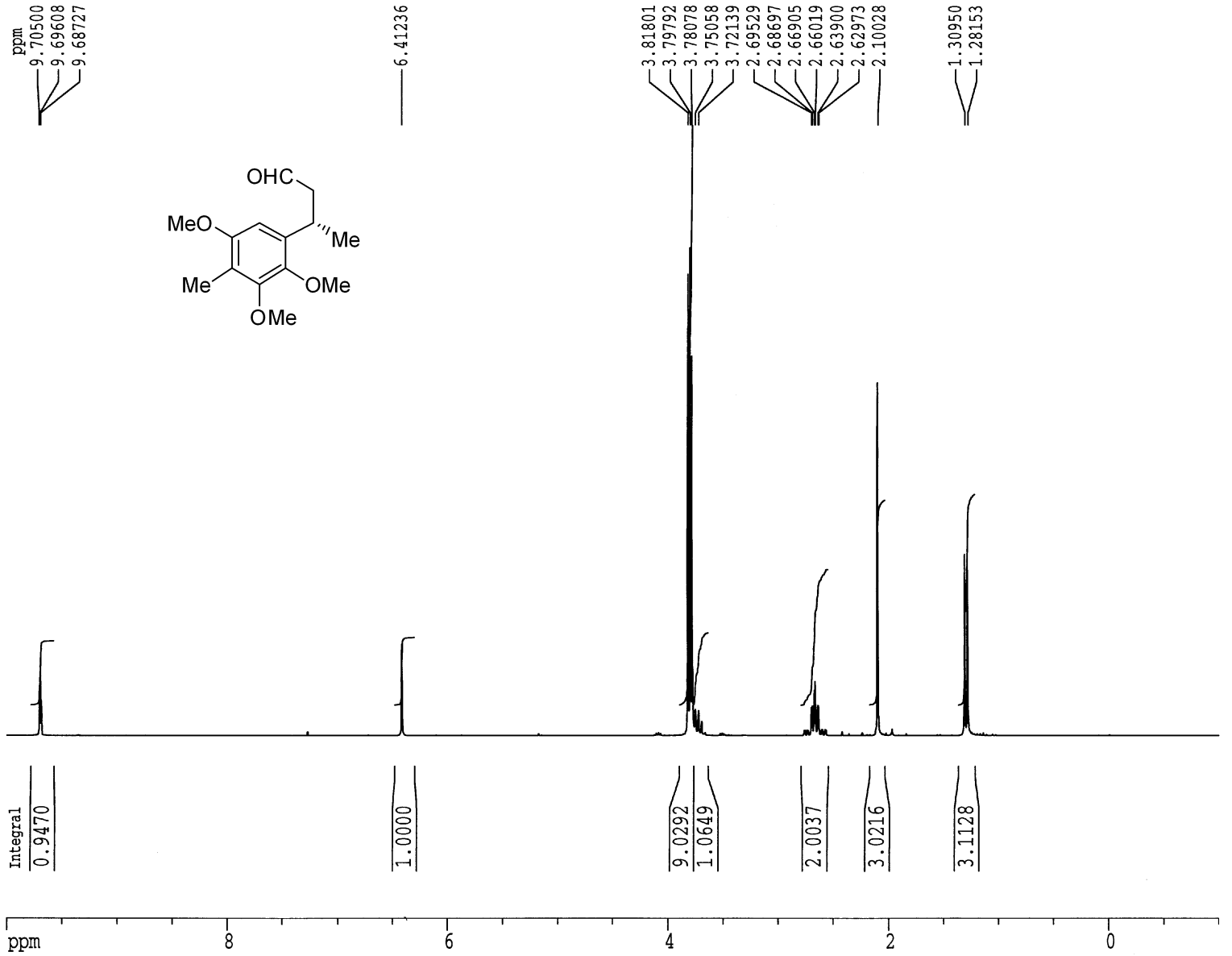
Current Data Parameters
NAME wy-X-78-A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080731
Time 11.35
INSTRUM arx250
PROBHD 5 mm QNP 1H
PULPROG zgdc30
TD 36864
SOLVENT CDCl3
NS 110
DS 4
SWH 17241.379 Hz
FIDRES 0.467702 Hz
AQ 1.0691060 sec
RG 22800
DW 29.000 usec
DE 41.43 usec
TE 300.0 K
D12 0.00002000 sec
DL5 23.00 dB
CPDPRG waltz16
P31 103.00 usec
D1 2.00000000 sec
P1 8.00 usec
SFO1 62.9023694 MHz
NUCLEUS 13C
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 62.8952424 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 160.650 ppm
F1 10104.11 Hz
F2P 3.606 ppm
F2 226.79 Hz
PPMCM 7.85220 ppm/cm
HZCM 493.86630 Hz/cm

1H NMR



Current Data Parameters

NAME	wy-X-124-A1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080925
Time	15.24
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.158946 Hz
AQ	3.1457779 sec
RG	128
DW	96.000 usec
DE	137.14 usec
TE	300.0 K
D1	1.00000000 sec
P1	9.50 usec
SFO1	250.1315321 MHz
NUCLEUS	1H

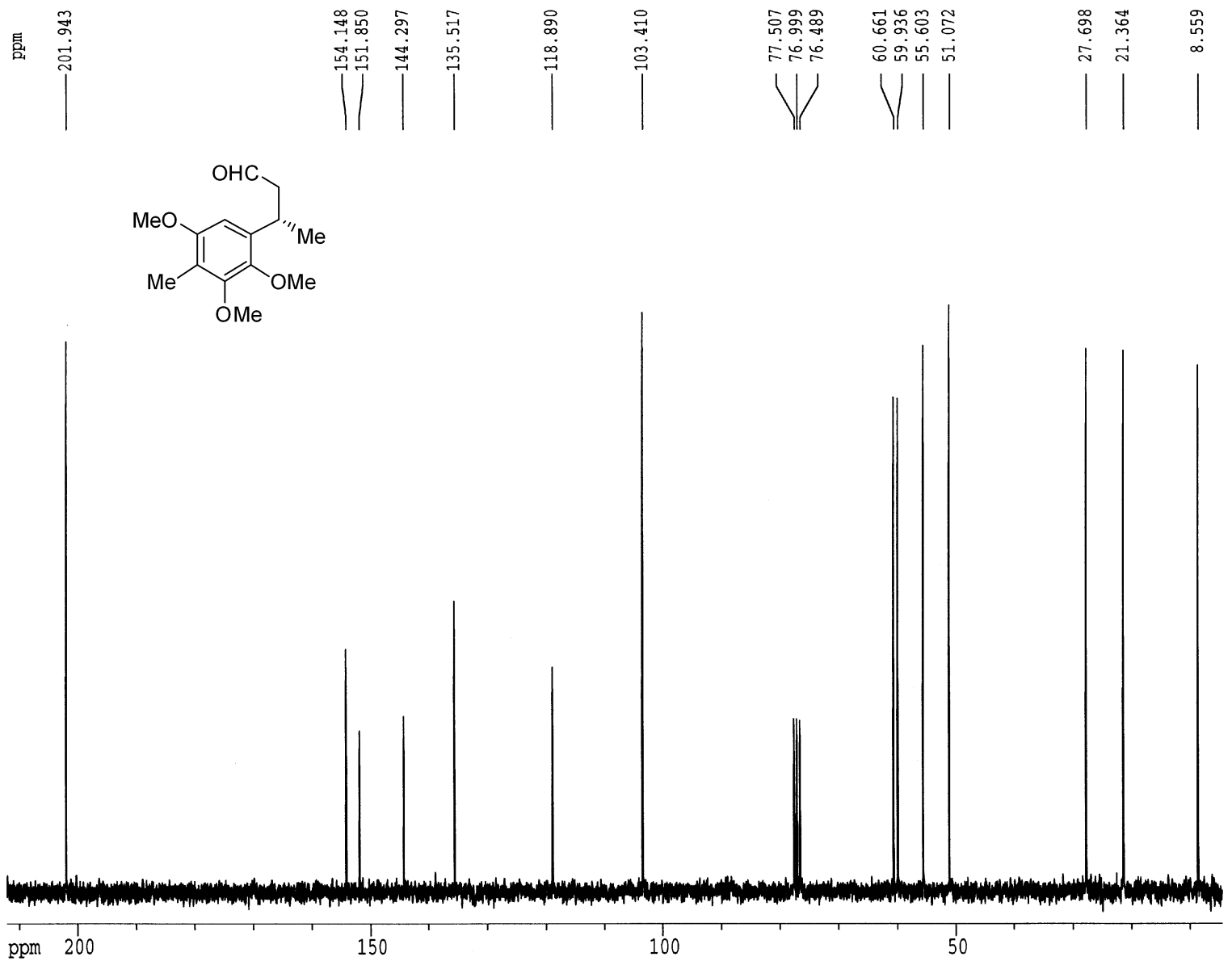
F2 - Processing parameters

SI	16384
SF	250.1300048 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	8.00 cm
F1P	10.000 ppm
F1	2501.30 Hz
F2P	-1.000 ppm
F2	-250.13 Hz
PPMCM	0.55000 ppm/cm
HZCM	137.57152 Hz/cm

13C NMR



Current Data Parameters

NAME	wy-X-124-A1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080925
Time	15.28
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	36864
SOLVENT	CDCl3
NS	51
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.0691060 sec
RG	22800
DW	29.000 usec
DE	41.43 usec
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 usec
D1	2.00000000 sec
P1	8.00 usec
SFO1	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

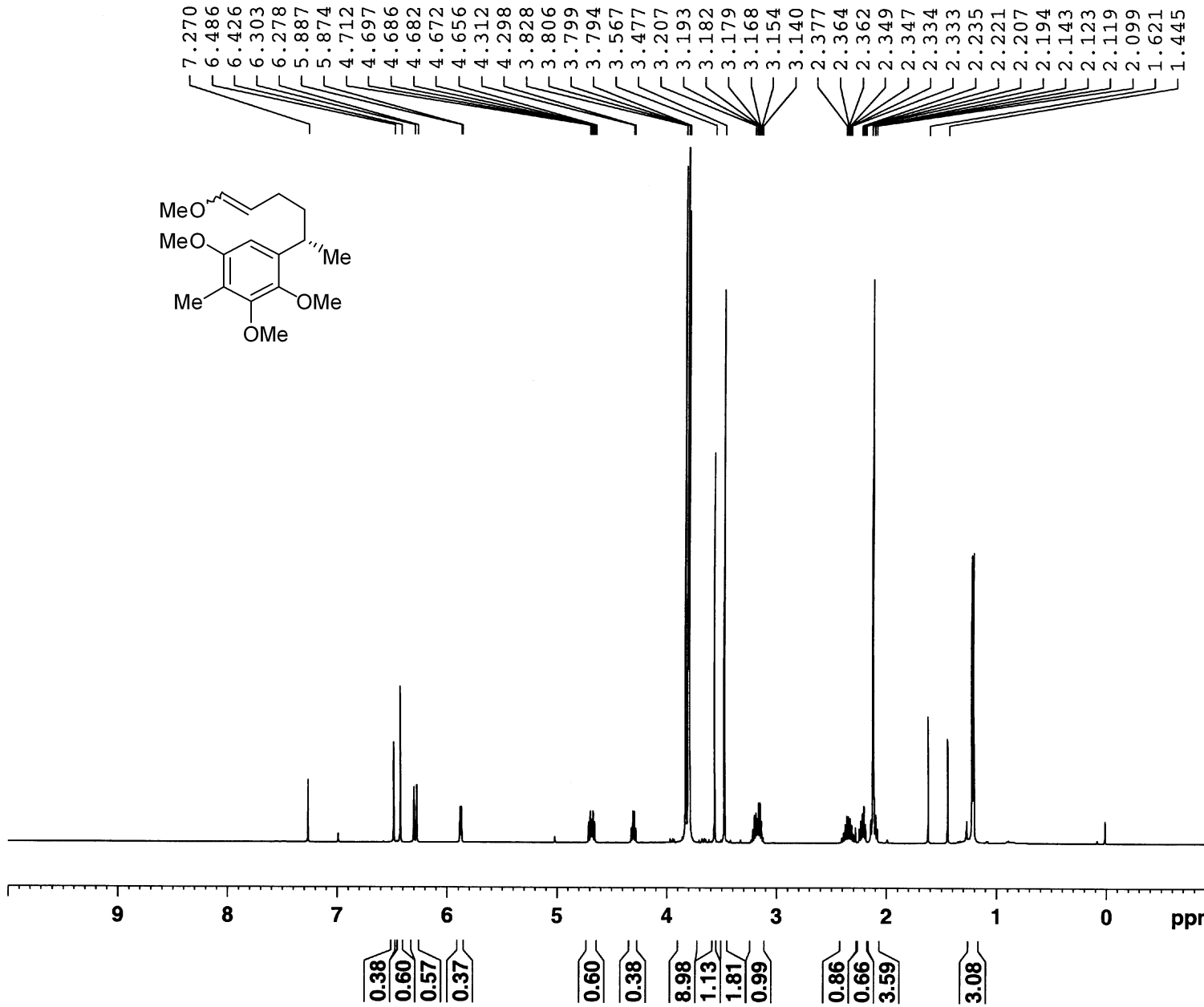
F2 - Processing parameters

SI	32768
SF	62.8952482 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	212.128 ppm
F1	13341.84 Hz
F2P	4.455 ppm
F2	280.19 Hz
PPMCM	10.38365 ppm/cm
HZCM	653.08252 Hz/cm

1H NMR



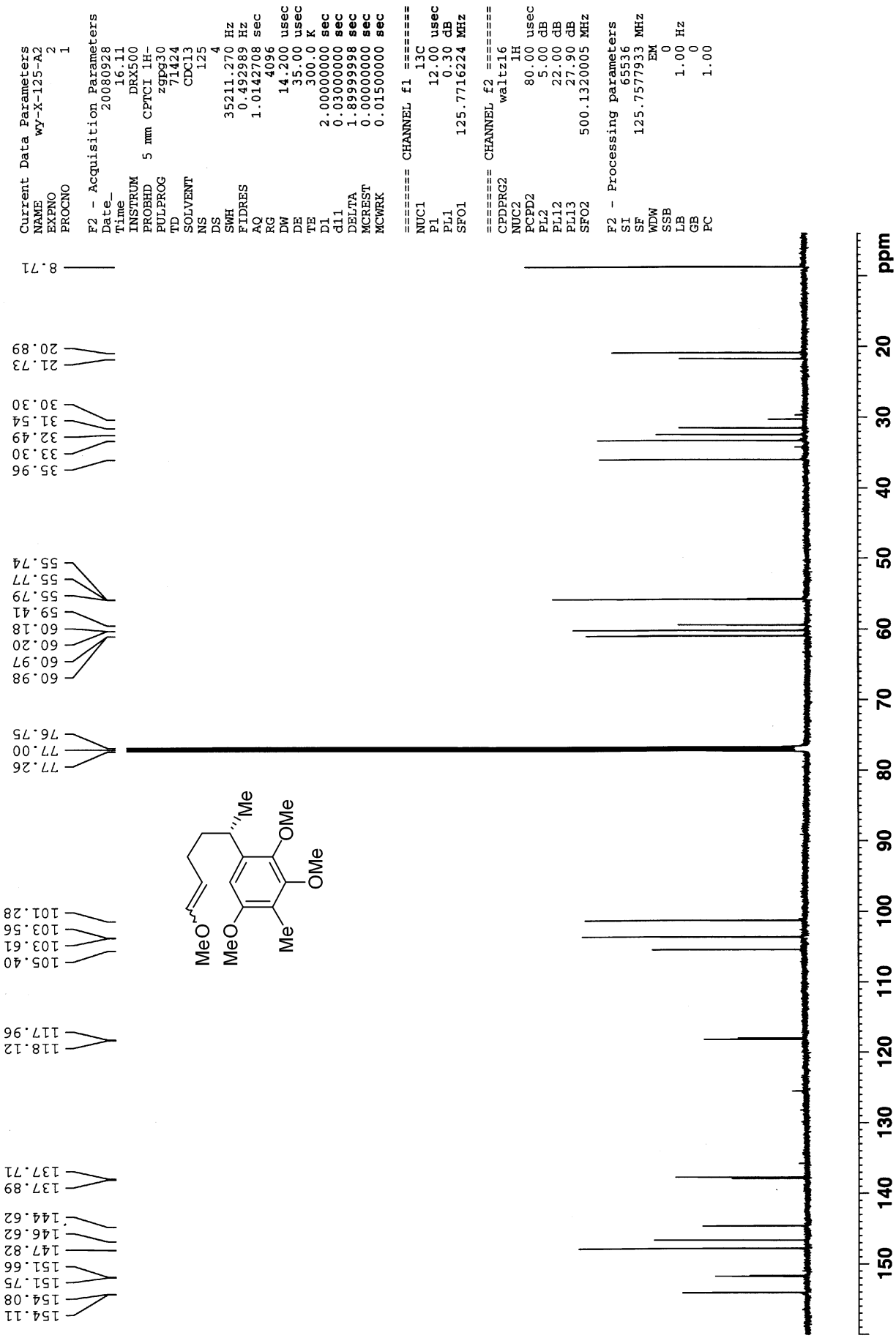
Current Data Parameters
NAME wy-X-125-A2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080928
Time 16.07
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 14.3
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

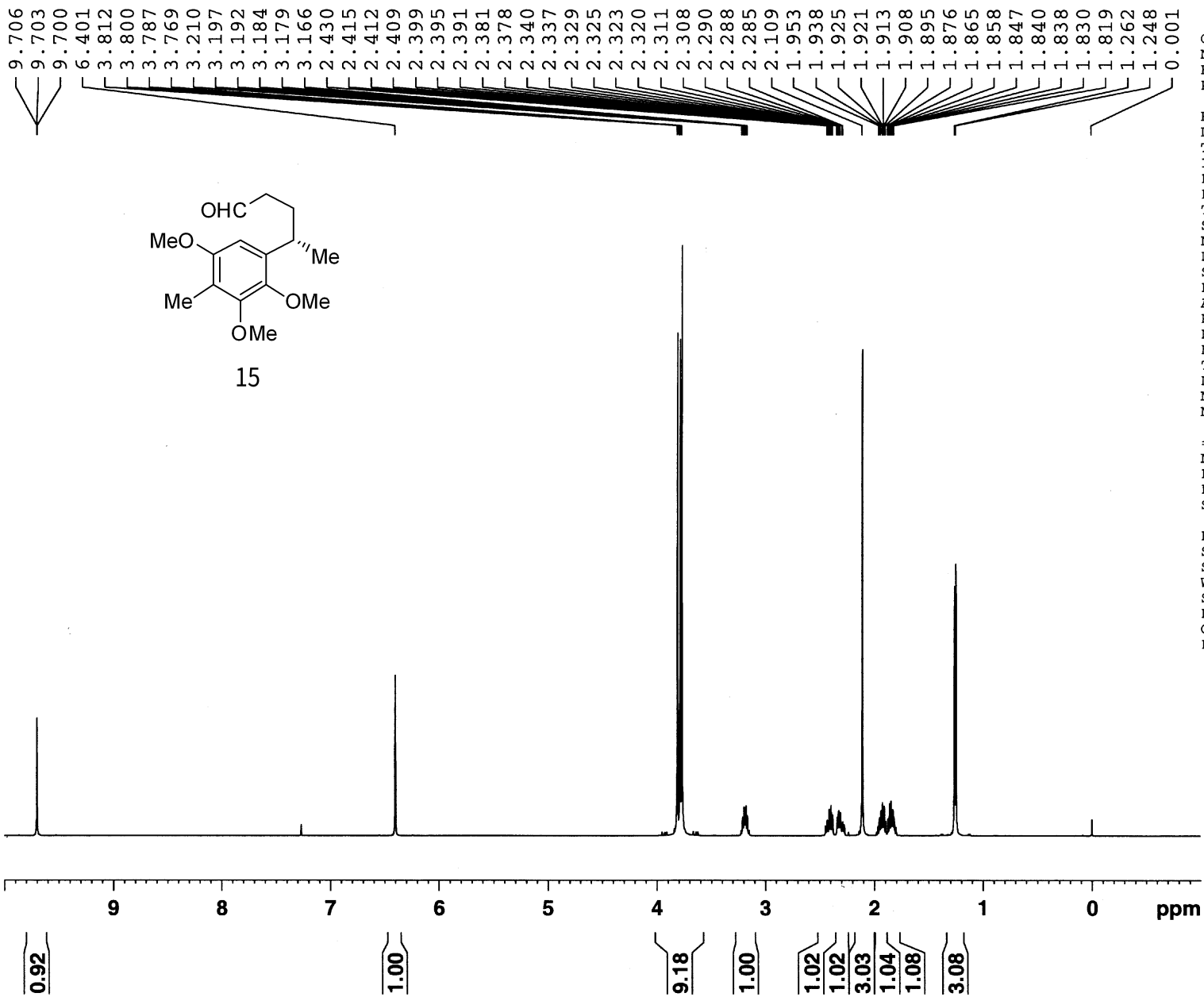
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C NMR


Current Data Parameters
NAME wy-X-125-A2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080928
Time 16.11
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 125
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.01142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577933 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
8.71
21.73
20.89
30.30
31.54
32.49
33.30
35.96
55.74
55.77
55.79
59.41
60.18
60.20
60.97
60.98
76.75
77.00
77.26
101.28
103.56
103.61
105.40
117.96
118.12
137.71
137.89
144.62
146.62
147.82
151.66
151.75
154.08
154.11
MeO
MeO
Me
OMe
OMe
Me
ppm

1H NMR



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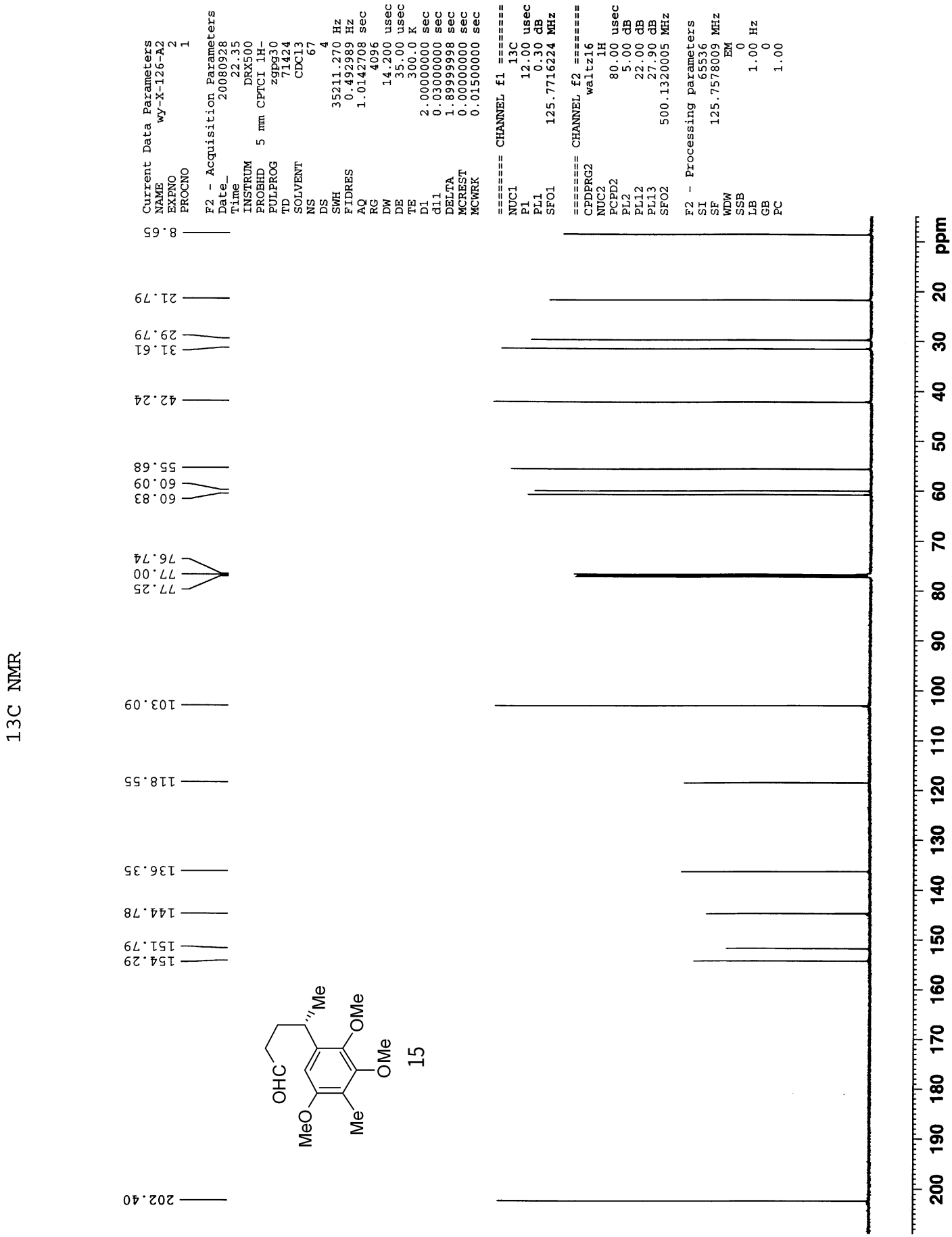
Current Data Parameters
NAME wy-X-126-A2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080928
Time 22.31
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 9
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz

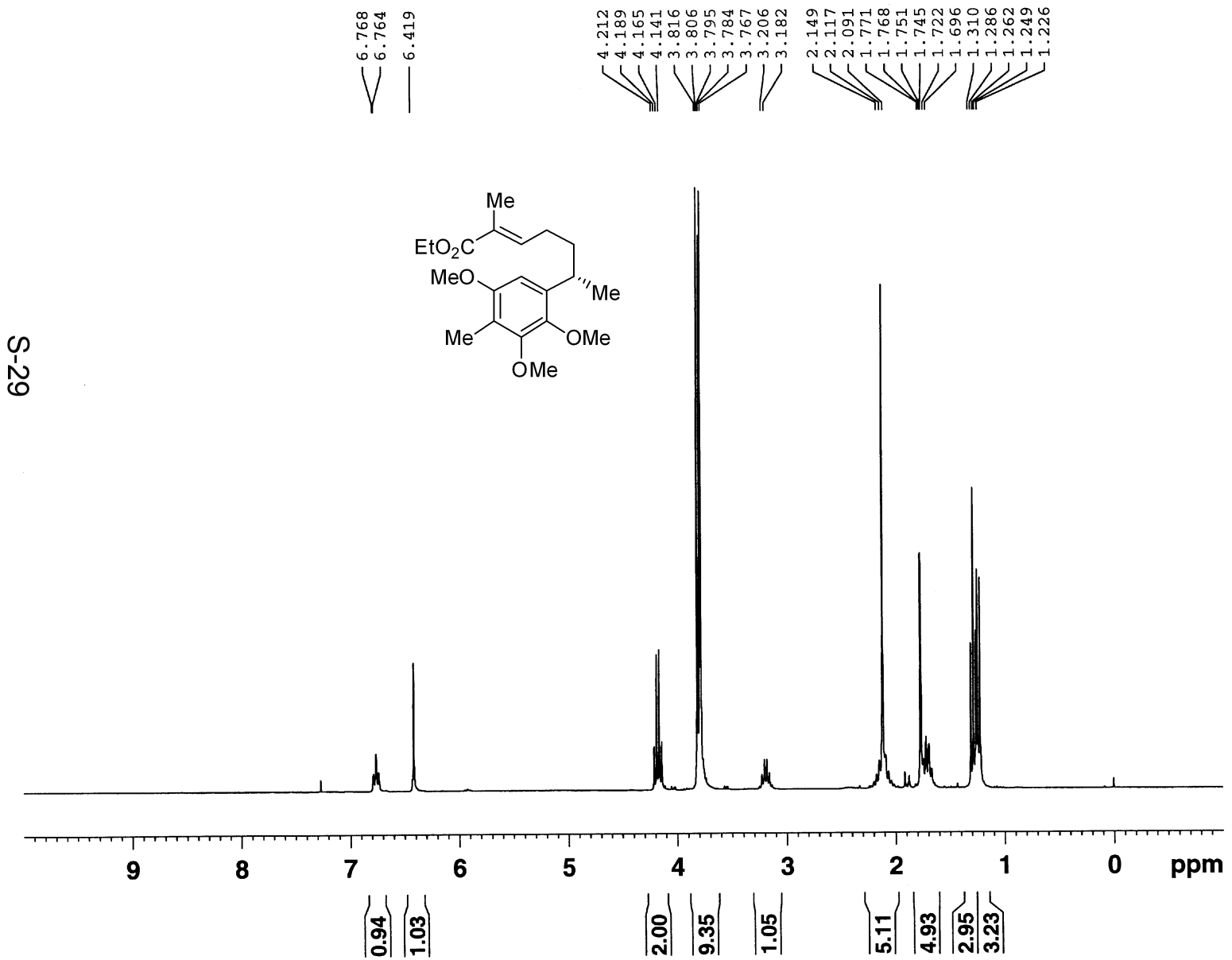
F2 - Processing parameters
SI 32768
SF 500.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C NMR


Current Data Parameters
NAME wy-X-126-A2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080928
Time 22.35
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 67
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7578009 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
8.65
21.79
29.79
31.61
42.24
55.68
60.09
60.83
76.74
77.00
77.25
103.09
118.55
136.35
144.78
151.79
154.29
202.40
ppm
OHC
Me
OMe
OMe
MeO
Me

15

1H NMR



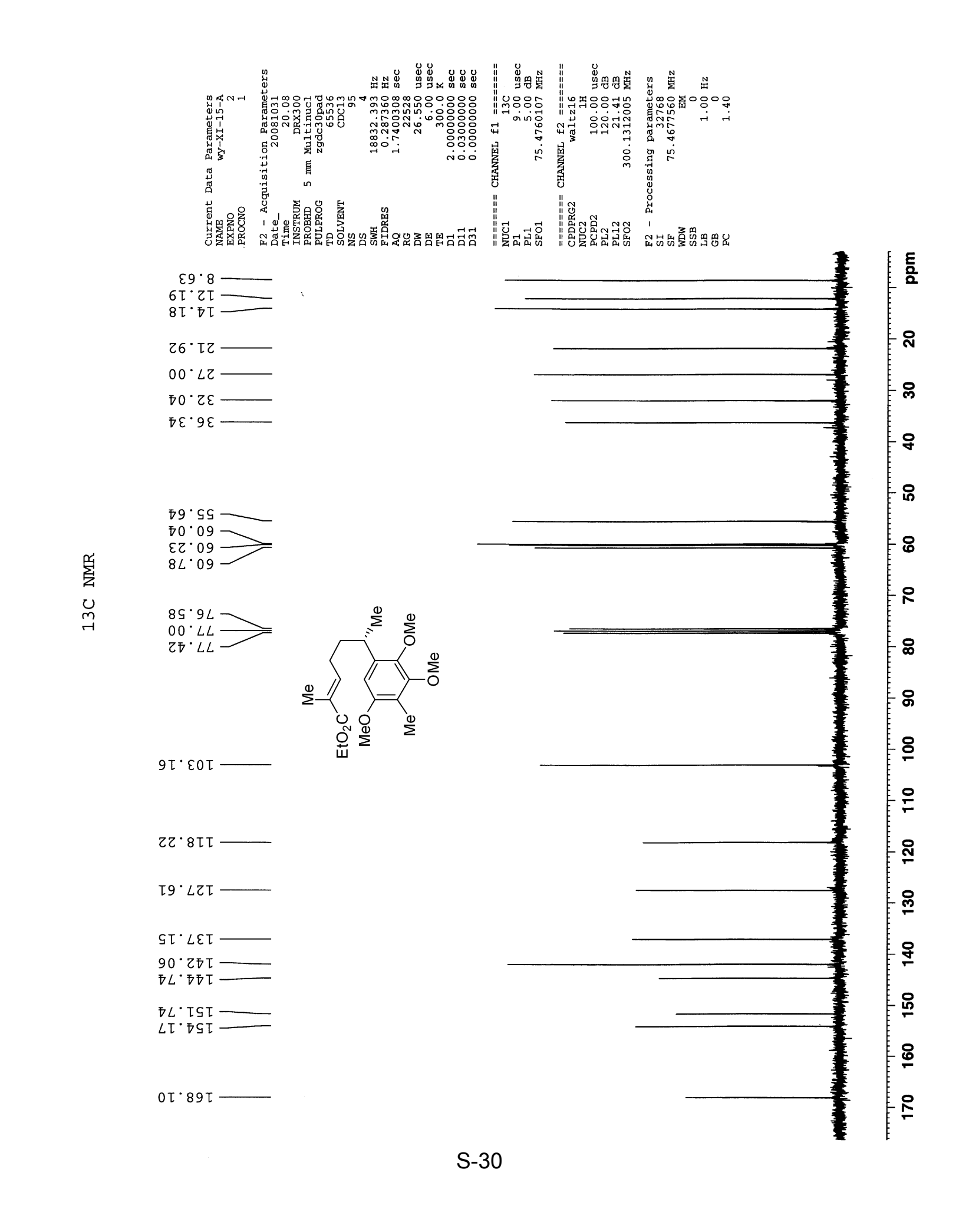
Current Data Parameters
NAME wy-XI-15-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081031
Time 20.00
INSTRUM DRX300
PROBHD 5 mm Multinucl
PULPROG zg30pad
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 40.3
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D31 0.00000000 sec

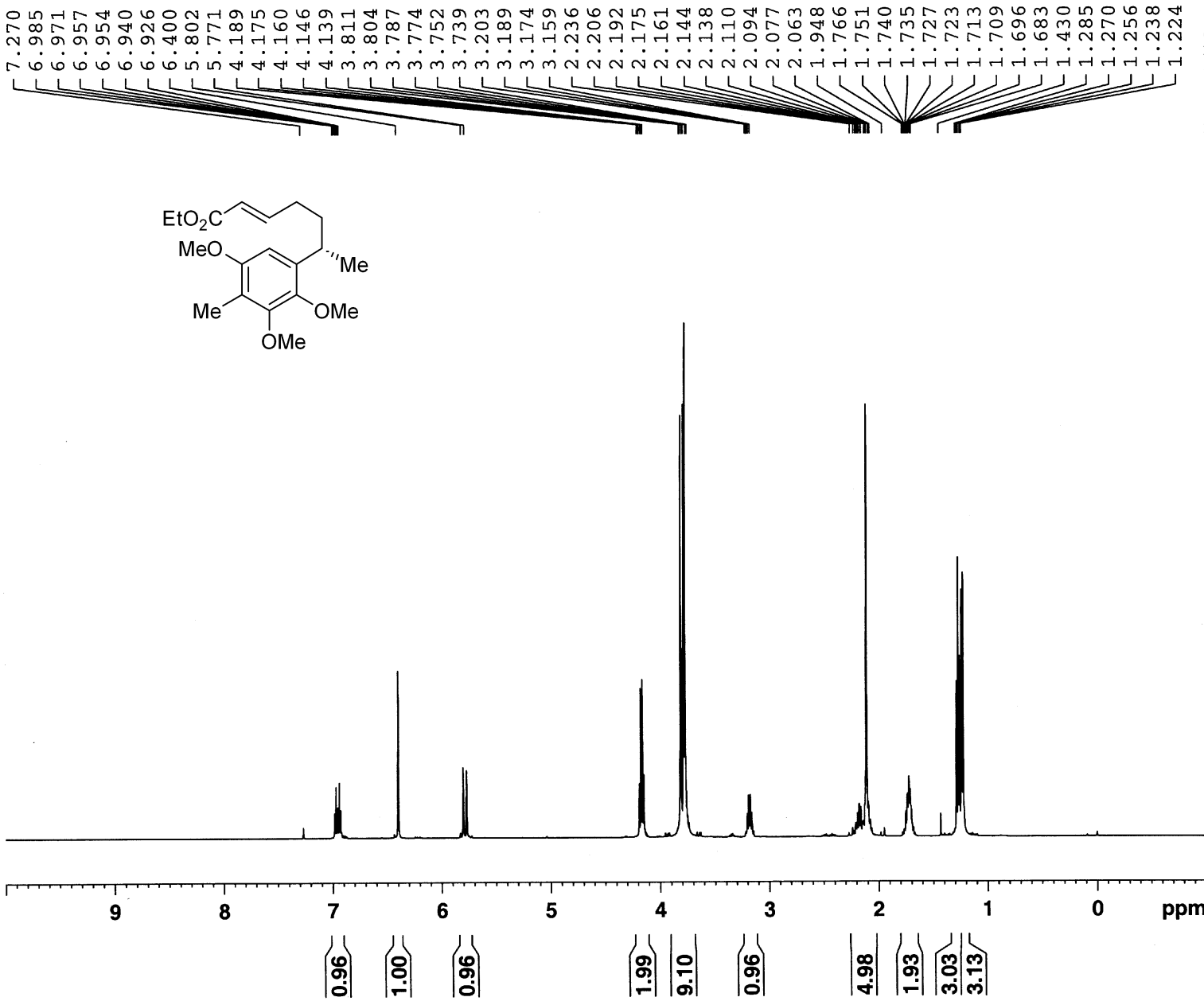
======== CHANNEL f1 ========
NUC1 1H
P1 7.05 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300022 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

13C NMR


Current Data Parameters
NAME wy-XI-15-A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081031
Time 20.08
INSTRUM DRX300
PROBHD 5 mm Multinuc1
PULPROG zgdc30pad
TD 65536
SOLVENT CDC13
NS 95
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.74400308 sec
RG 22528
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D31 0.00000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 5.00 dB
SFO1 75.4760107 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 21.41 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
8.63
12.19
14.18
21.92
27.00
32.04
36.34
55.64
60.04
60.23
60.78
76.58
77.00
77.42
103.16
118.22
127.61
137.15
142.06
144.74
151.74
154.17
168.10
Me
EtO2C
MeO
Me
OMe
OMe
Me
ppm

1H NMR



Current Data Parameters

NAME	wy-XI-18-A
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20081106
Time	12.06
INSTRUM	DRX500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	5
DW	48.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

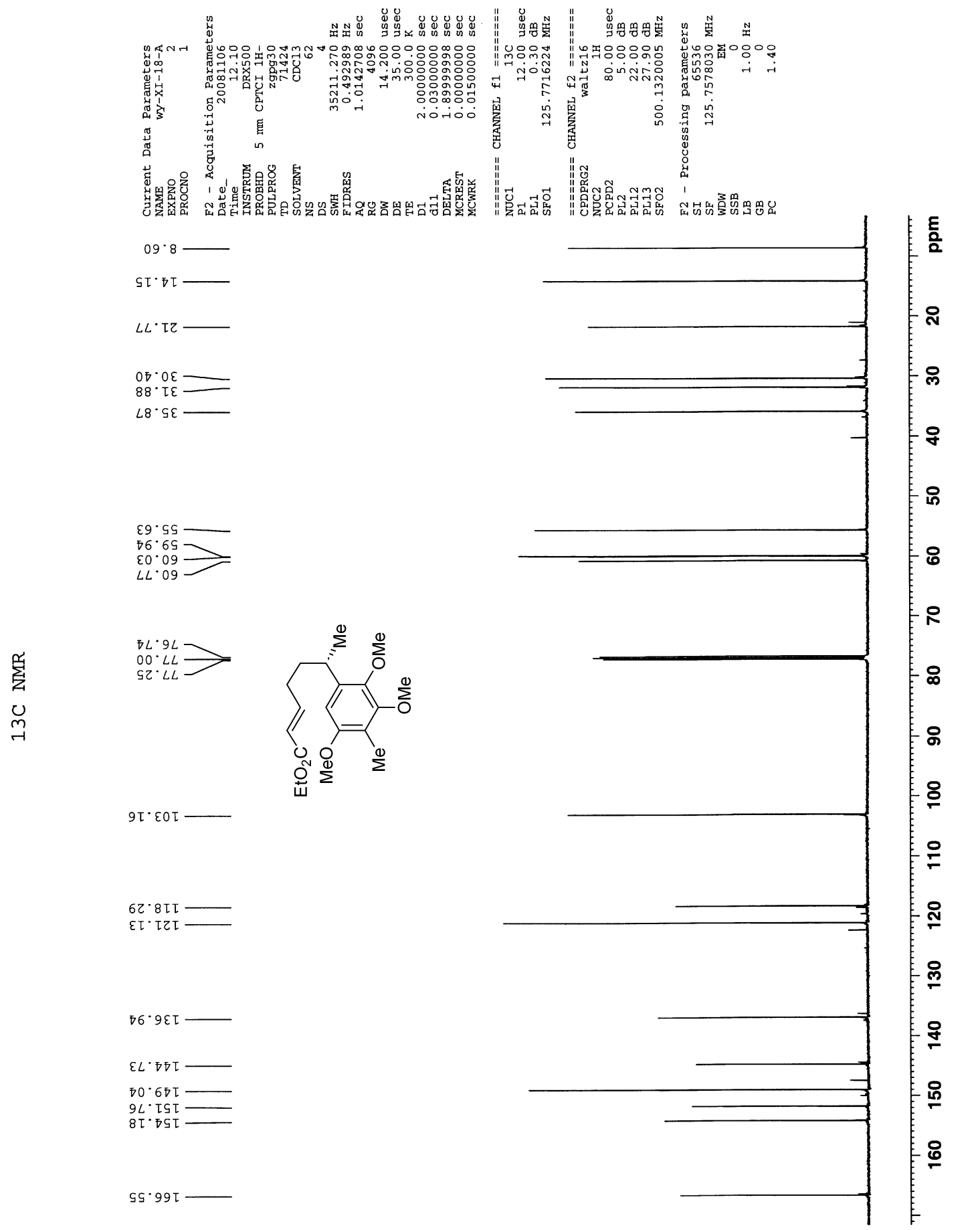
======== CHANNEL f1 ========

NUC1	1H
P1	8.00 usec
PL1	4.30 dB
SFO1	500.1335009 MHz

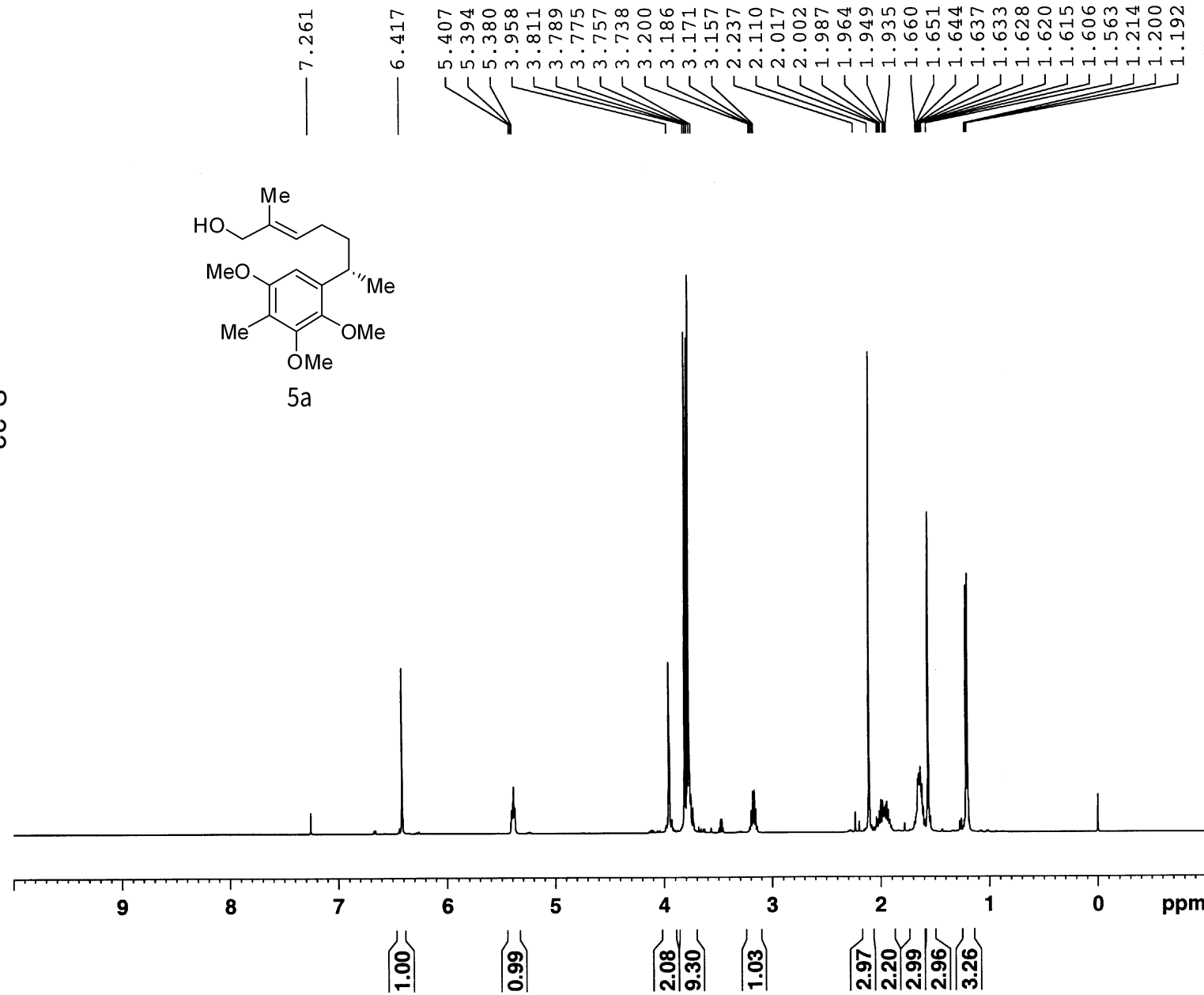
F2 - Processing parameters

SI	32768
SF	500.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

13C NMR


Current Data Parameters
NAME wy-XI-18-A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081106
Time 12.10
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 62
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.01142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7578030 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
8.60
14.15
21.77
30.40
31.88
35.87
55.63
59.94
60.03
60.77
76.74
77.00
77.25
103.16
118.29
121.13
136.94
144.73
149.04
151.76
154.18
166.55
EtO2C
MeO
Me
OMe
OMe
Me
ppm

1H NMR



5a

Current Data Parameters

NAME	wy-XI-20-A
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20081106
Time	21.31
INSTRUM	DRX500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	8
DW	48.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

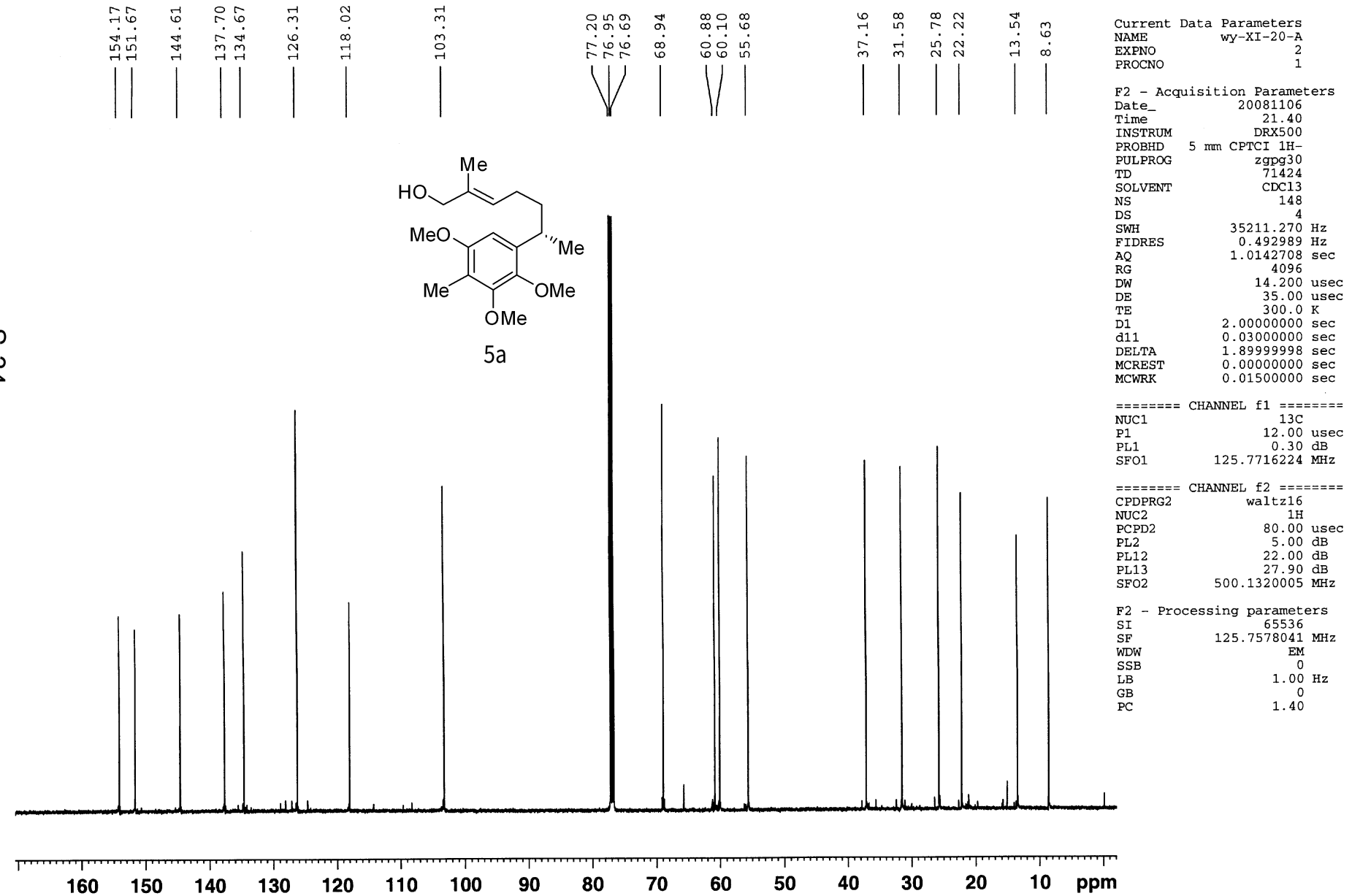
======== CHANNEL f1 ========

NUC1	1H
P1	8.00 usec
PL1	4.30 dB
SFO1	500.1335009 MHz

F2 - Processing parameters

SI	32768
SF	500.1300135 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

13C NMR




```
Current Data Parameters
NAME                 wy-XI-20-A
EXPNO                         2
PROCNO                        1

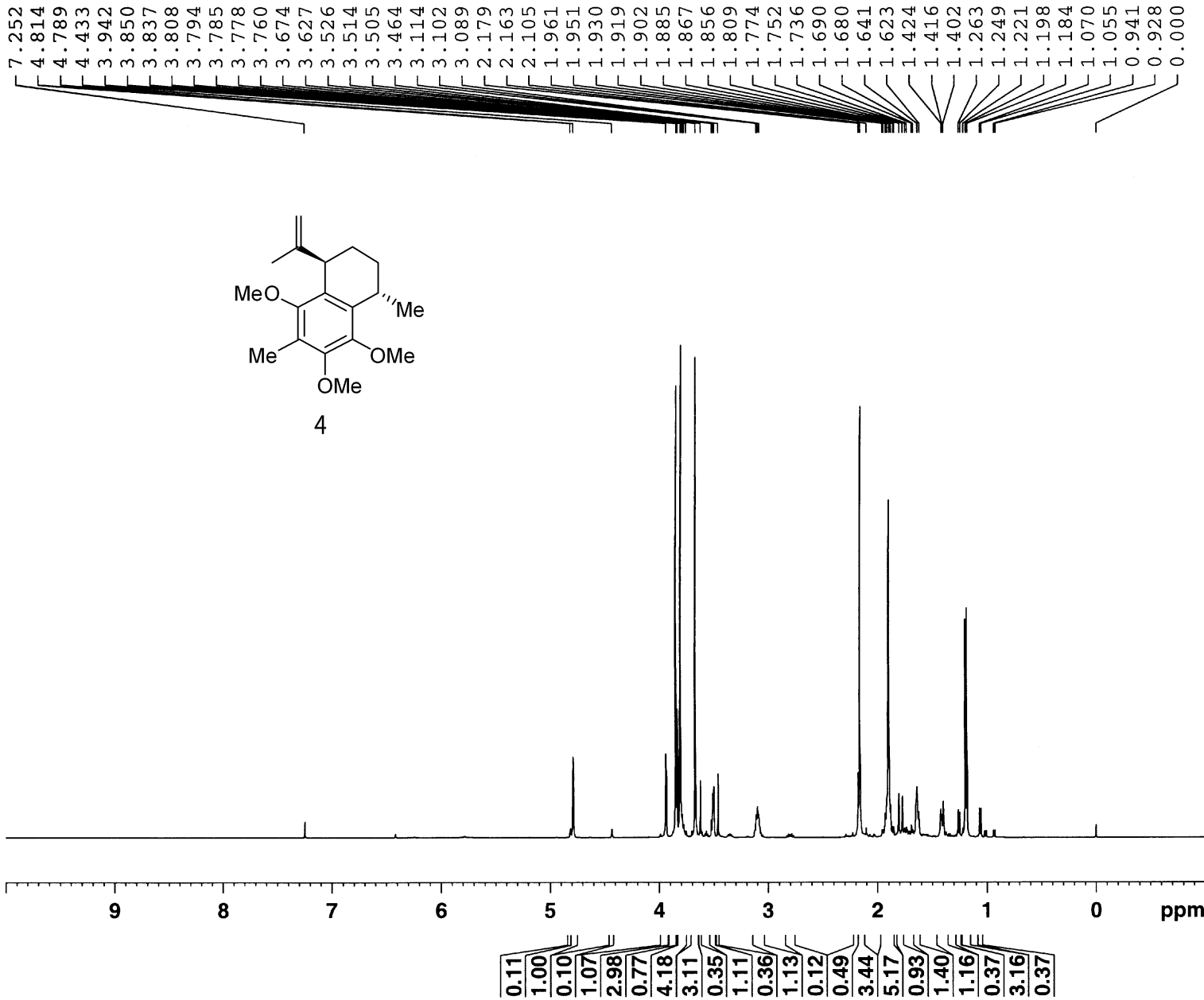
F2 - Acquisition Parameters
Date_                  20081106
Time                      21.40
INSTRUM                  DRX500
PROBHD    5 mm CPTCI 1H-
PULPROG                 zgpg30
TD                       71424
SOLVENT                  CDCl3
NS                         148
DS                           4
SWH                35211.270 Hz
FIDRES              0.492989 Hz
AQ                  1.0142708 sec
RG                        4096
DW                    14.200 usec
DE                    35.00 usec
TE                    300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                    12.00 usec
PL1                    0.30 dB
SFO1              125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    5.00 dB
PL12                  22.00 dB
PL13                  27.90 dB
SFO2              500.1320005 MHz

F2 - Processing parameters
SI                         65536
SF                125.7578041 MHz
WDW                           EM
SSB                            0
LB                     1.00 Hz
GB                             0
PC                          1.40
```

1H NMR



4

Current Data Parameters

NAME	wy-XI-80-A
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090119
Time	12.46
INSTRUM	DRX500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	5
DW	48.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

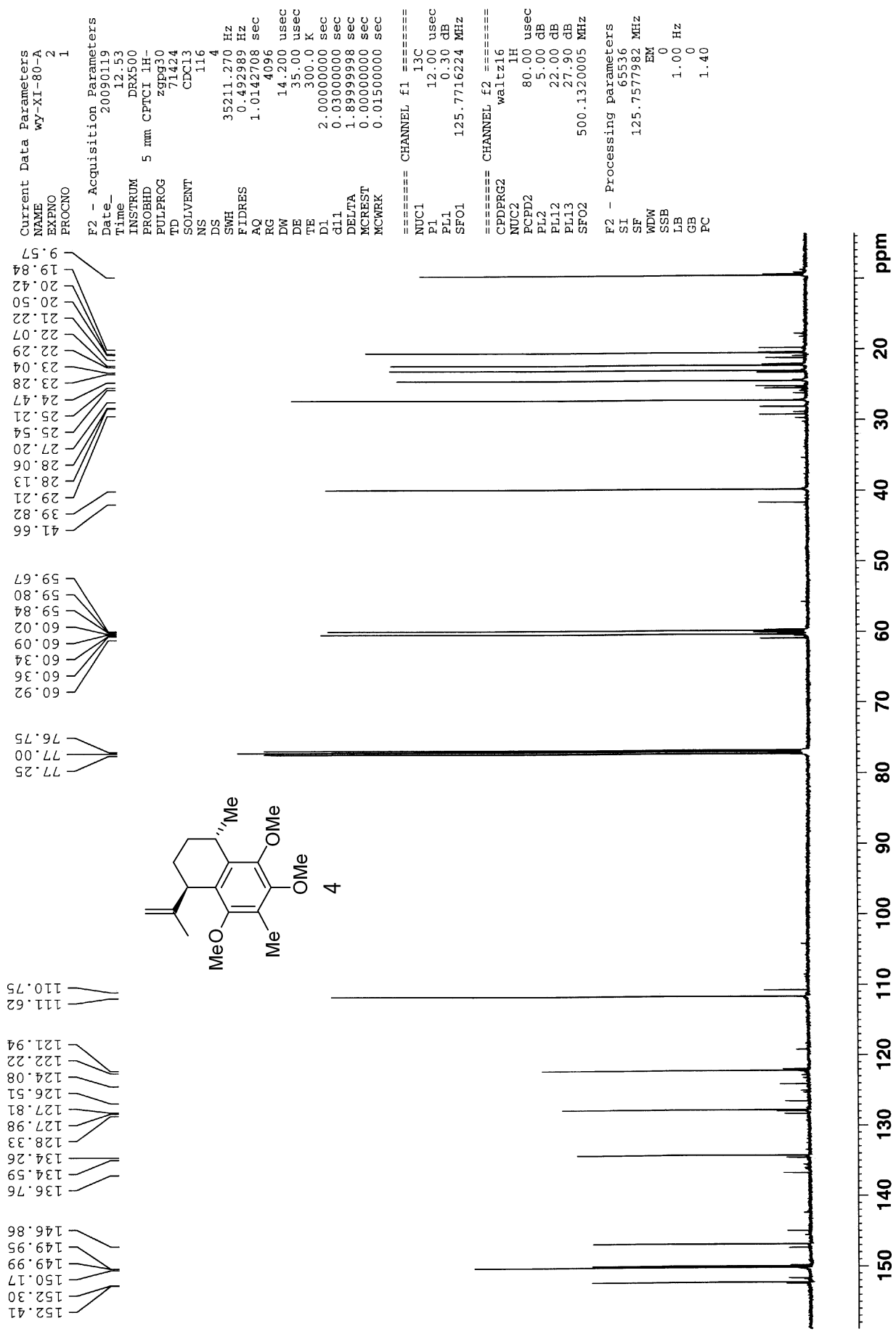
======== CHANNEL f1 ========

NUC1	1H
P1	8.00 usec
PL1	4.30 dB
SFO1	500.1335009 MHz

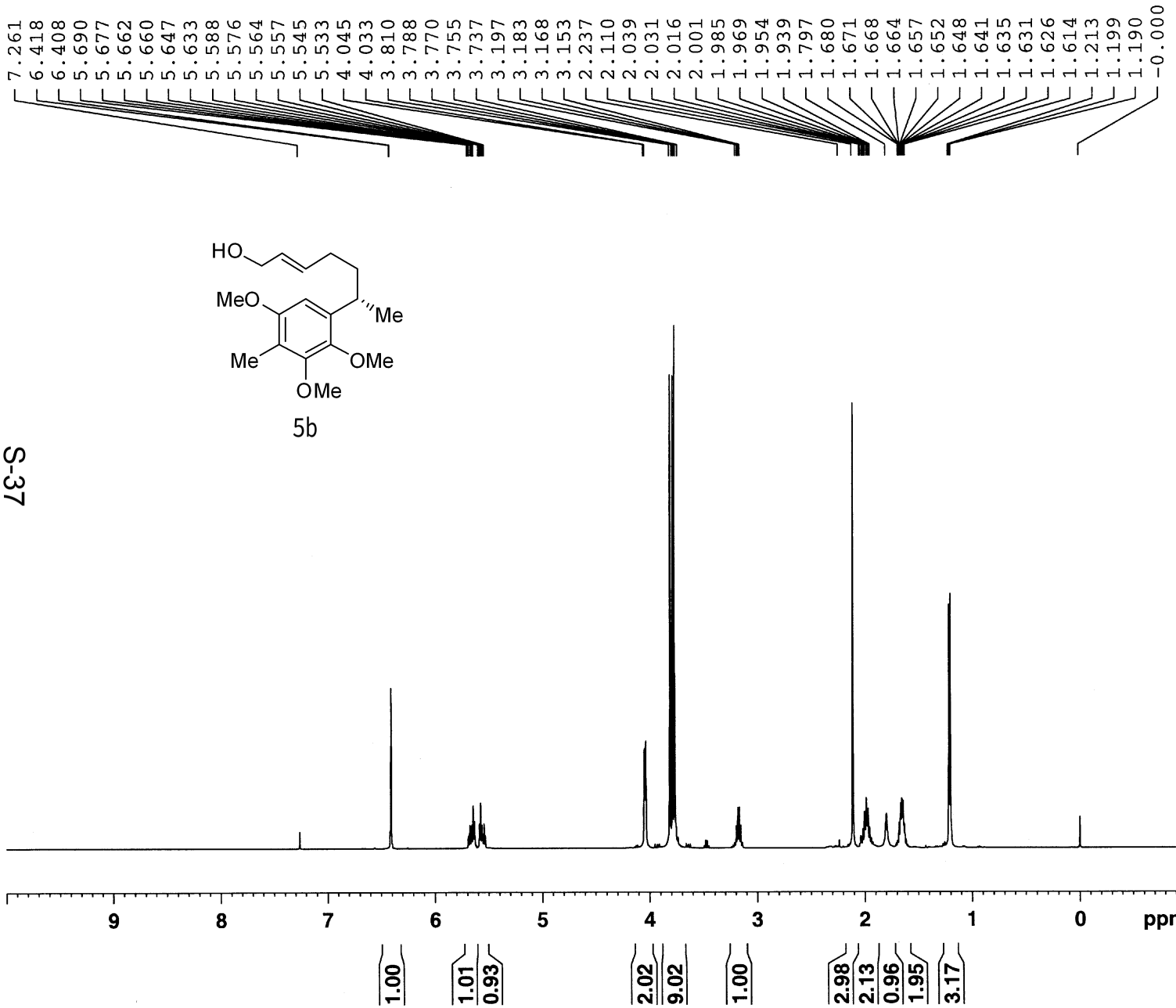
F2 - Processing parameters

SI	32768
SF	500.1300182 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

13C NMR


Current Data Parameters
NAME wy-XI-80-A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090119
Time 12.53
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 116
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577982 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
9.57
19.84
20.42
20.50
21.22
22.07
22.29
23.04
23.28
24.47
25.21
25.54
27.20
28.06
28.13
29.21
39.82
41.66
59.67
59.80
59.84
60.02
60.09
60.34
60.36
60.92
76.75
77.00
77.25
110.75
111.62
121.94
122.22
124.08
126.51
127.81
127.98
128.33
134.26
134.59
136.76
146.86
149.95
149.99
150.17
152.30
152.41
Me
OMe
OMe
MeO
Me
4
ppm

1H NMR



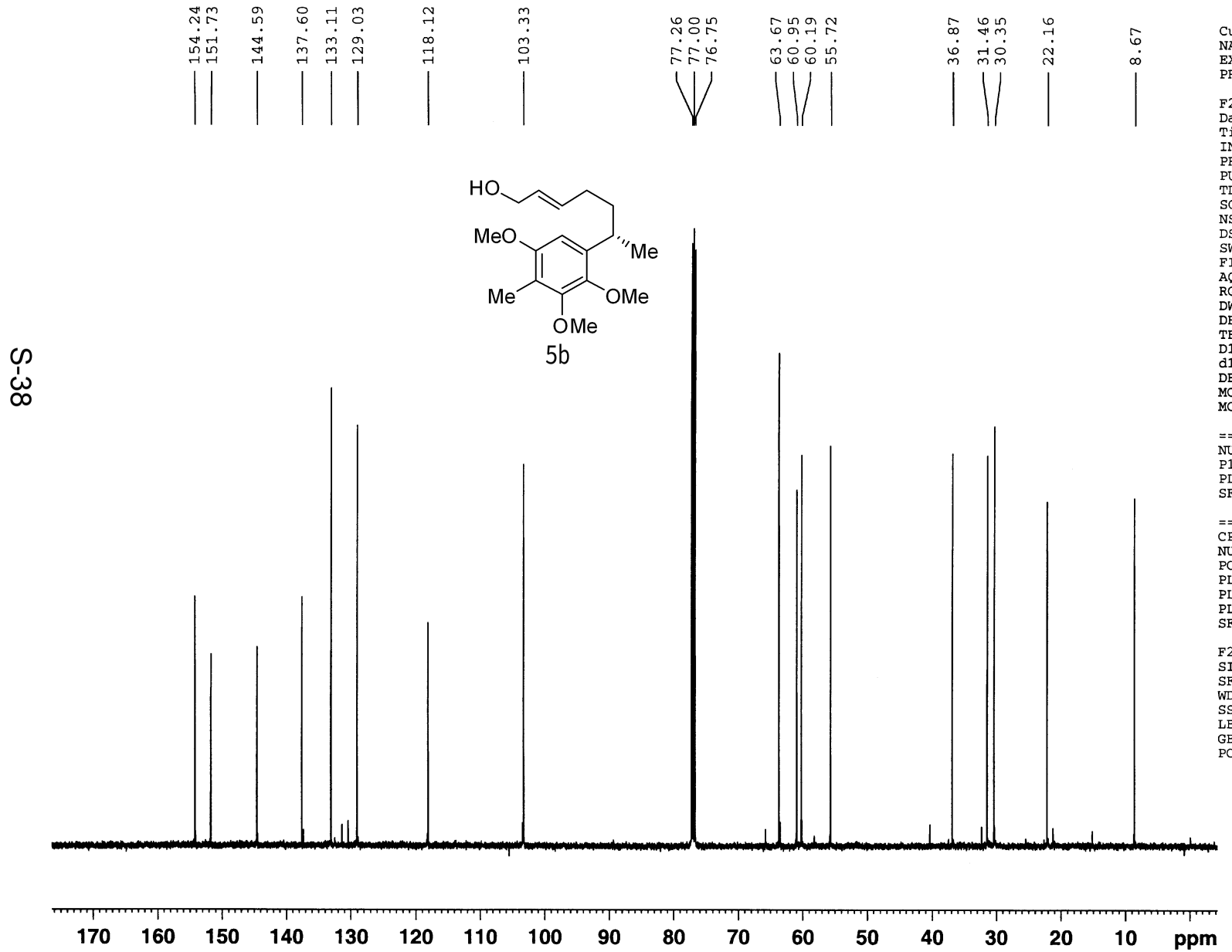
Current Data Parameters
NAME wy-XI-21-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081106
Time 21.25
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

13C NMR




```
Current Data Parameters
NAME          wy-XI-21-A
EXPNO                  2
PROCNO                 1

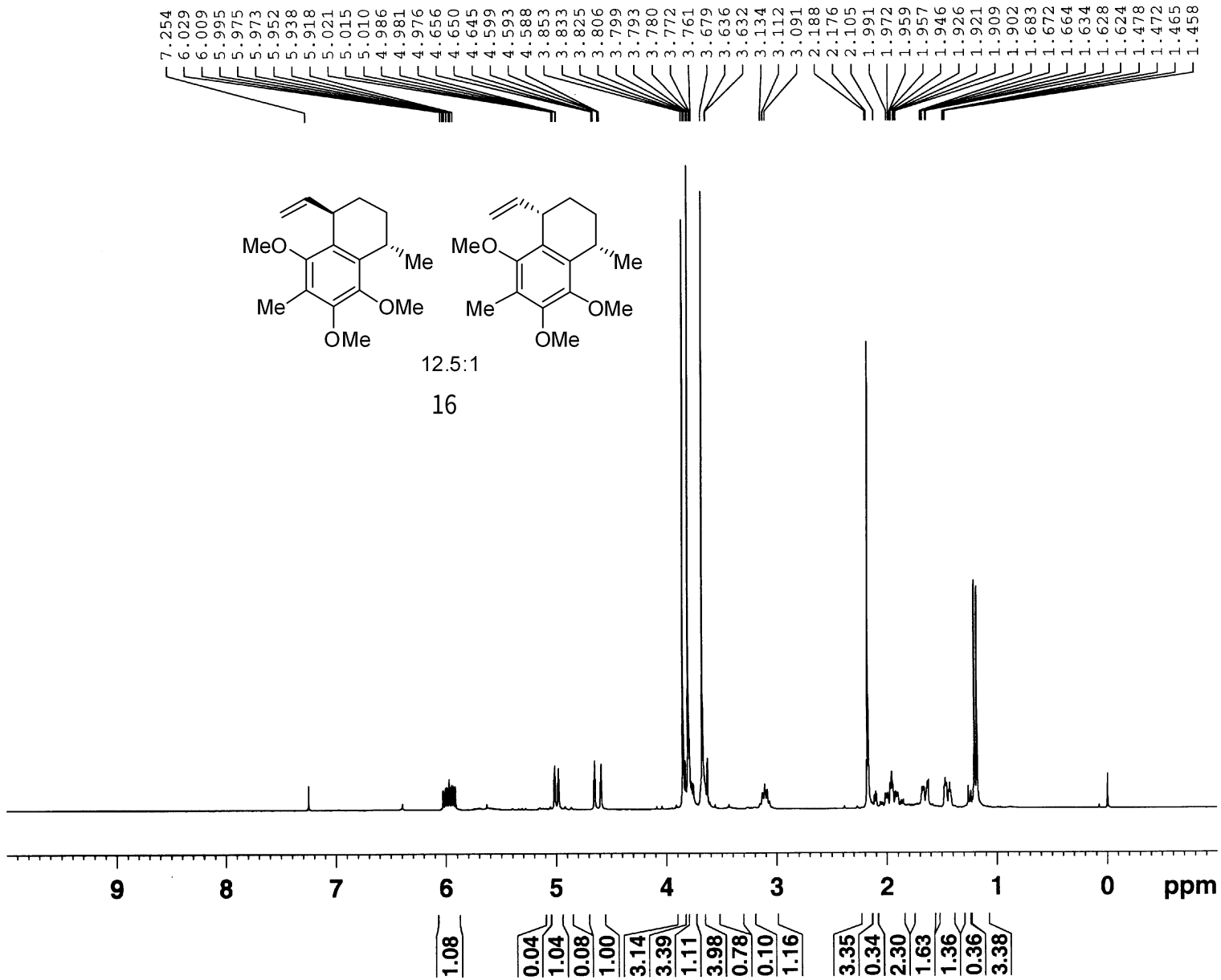
F2 - Acquisition Parameters
Date_           20081106
Time               21.28
INSTRUM           DRX500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgpg30
TD                71424
SOLVENT            CDCl3
NS                    26
DS                     4
SWH           35211.270 Hz
FIDRES         0.492989 Hz
AQ            1.0142708 sec
RG                  4096
DW                14.200 usec
DE                35.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                12.00 usec
PL1                0.30 dB
SFO1         125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                5.00 dB
PL12              22.00 dB
PL13              27.90 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 65536
SF          125.7577993 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```

1H NMR



Current Data Parameters

NAME	wy-XI-64-A
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090110
Time	9.39
INSTRUM	DRX300
PROBHD	5 mm Multinucl
PULPROG	zg30pad
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	101.6
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
D31	0.00000000 sec

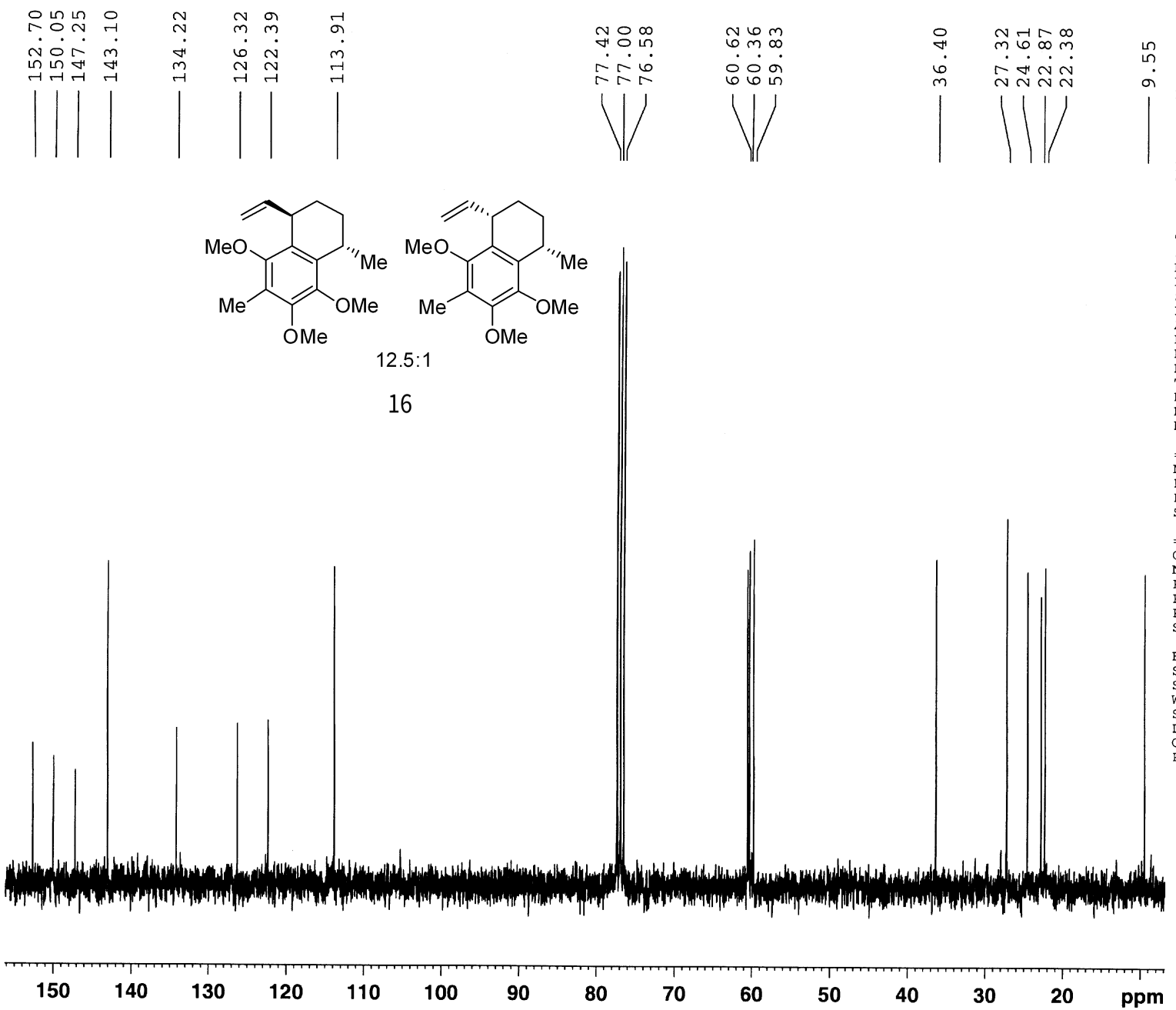
======== CHANNEL f1 ========

NUC1	1H
P1	7.05 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300081 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

13C NMR



Current Data Parameters
NAME wy-XI-64-A
EXPNO 2
PROCNO 1

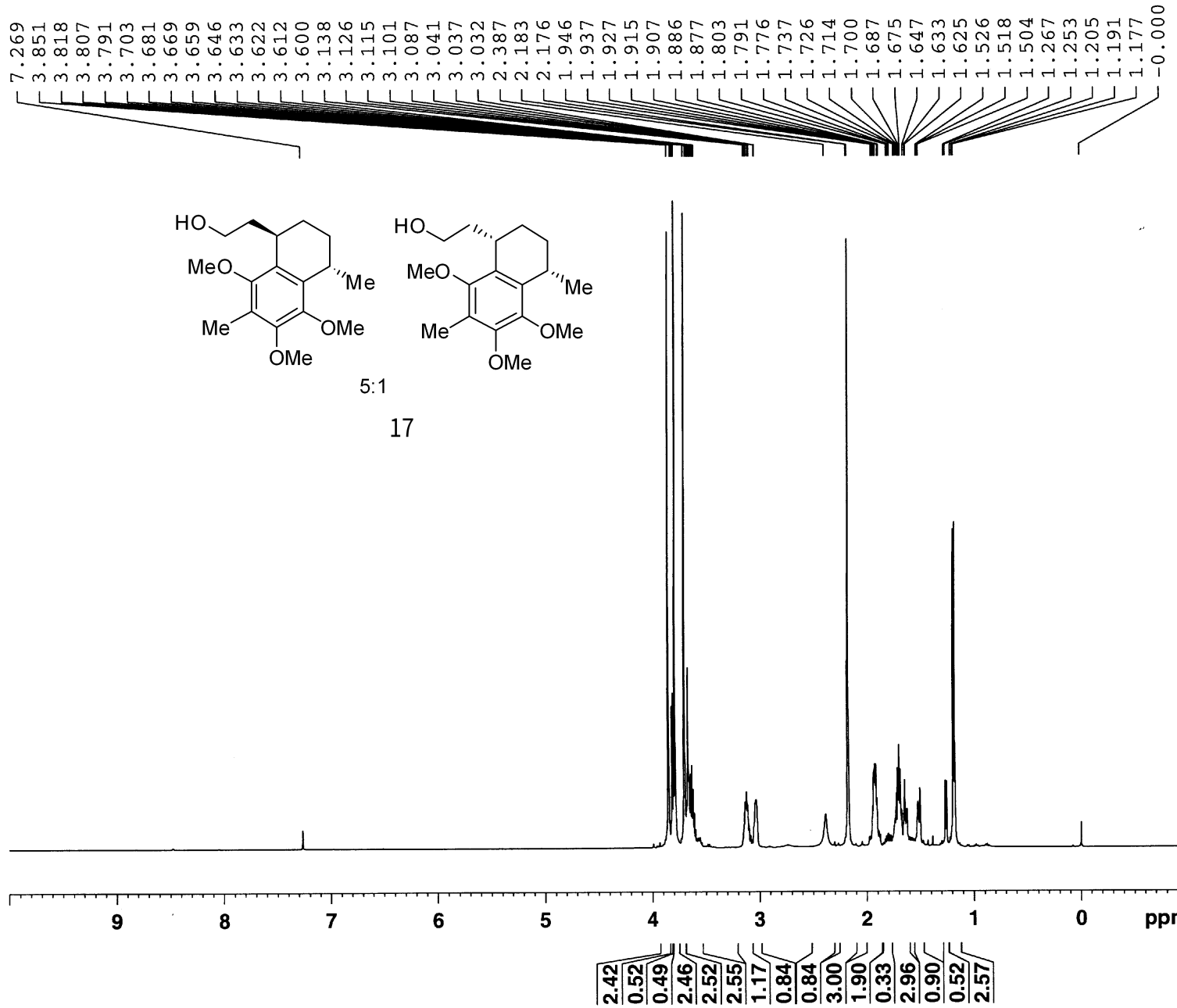
F2 - Acquisition Parameters
Date_ 20090110
Time 9.44
INSTRUM DRX300
PROBHD 5 mm Multinucl
PULPROG zgdc30pad
TD 65536
SOLVENT CDCl3
NS 58
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 22528
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D31 0.00000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 5.00 dB
SFO1 75.4760107 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 21.41 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677520 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.30

1H NMR



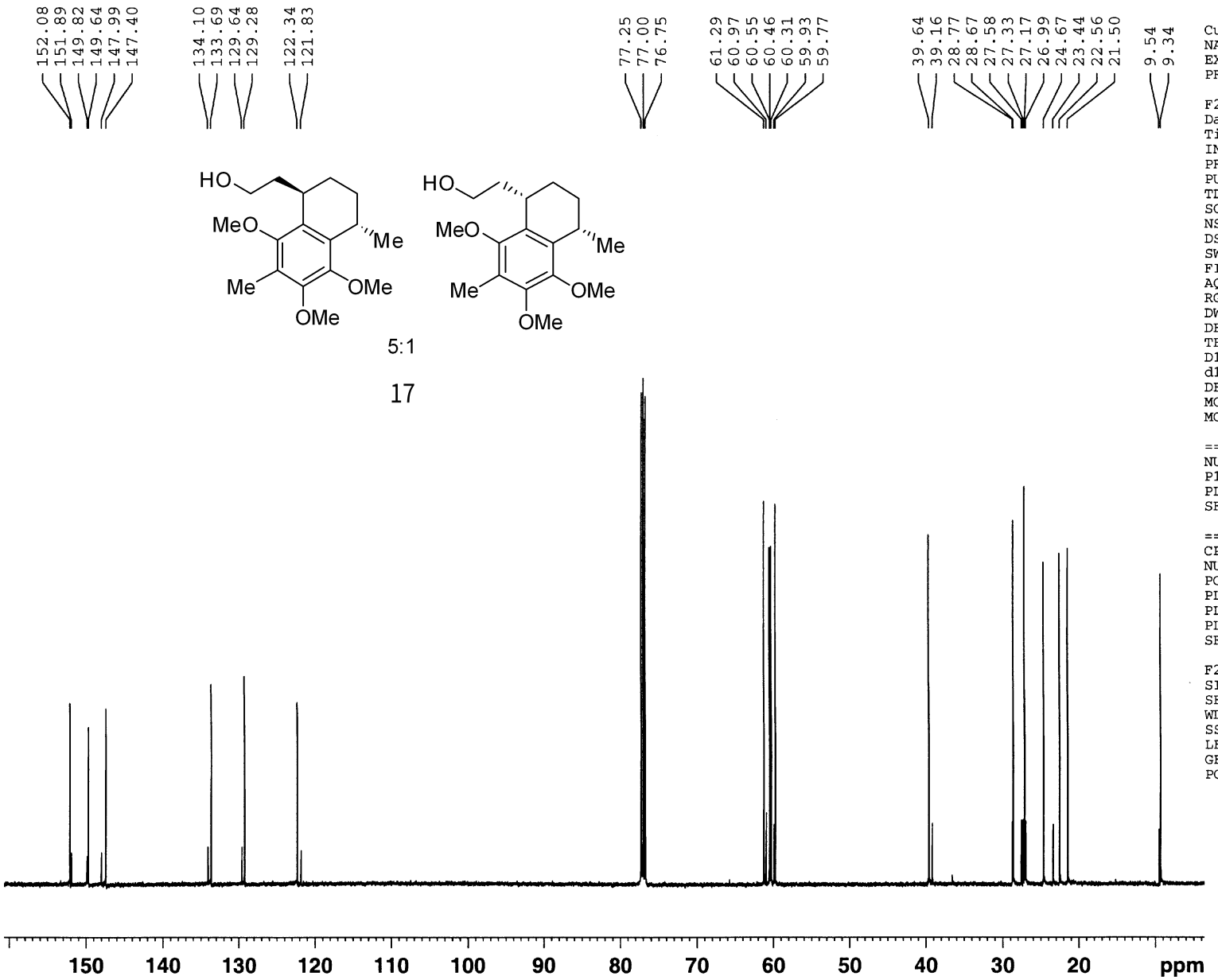
Current Data Parameters
NAME wy-XI-72-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090112
Time 14.35
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C NMR




```
Current Data Parameters
NAME                wy-XI-72-A
EXPNO                        2
PROCNO                       1

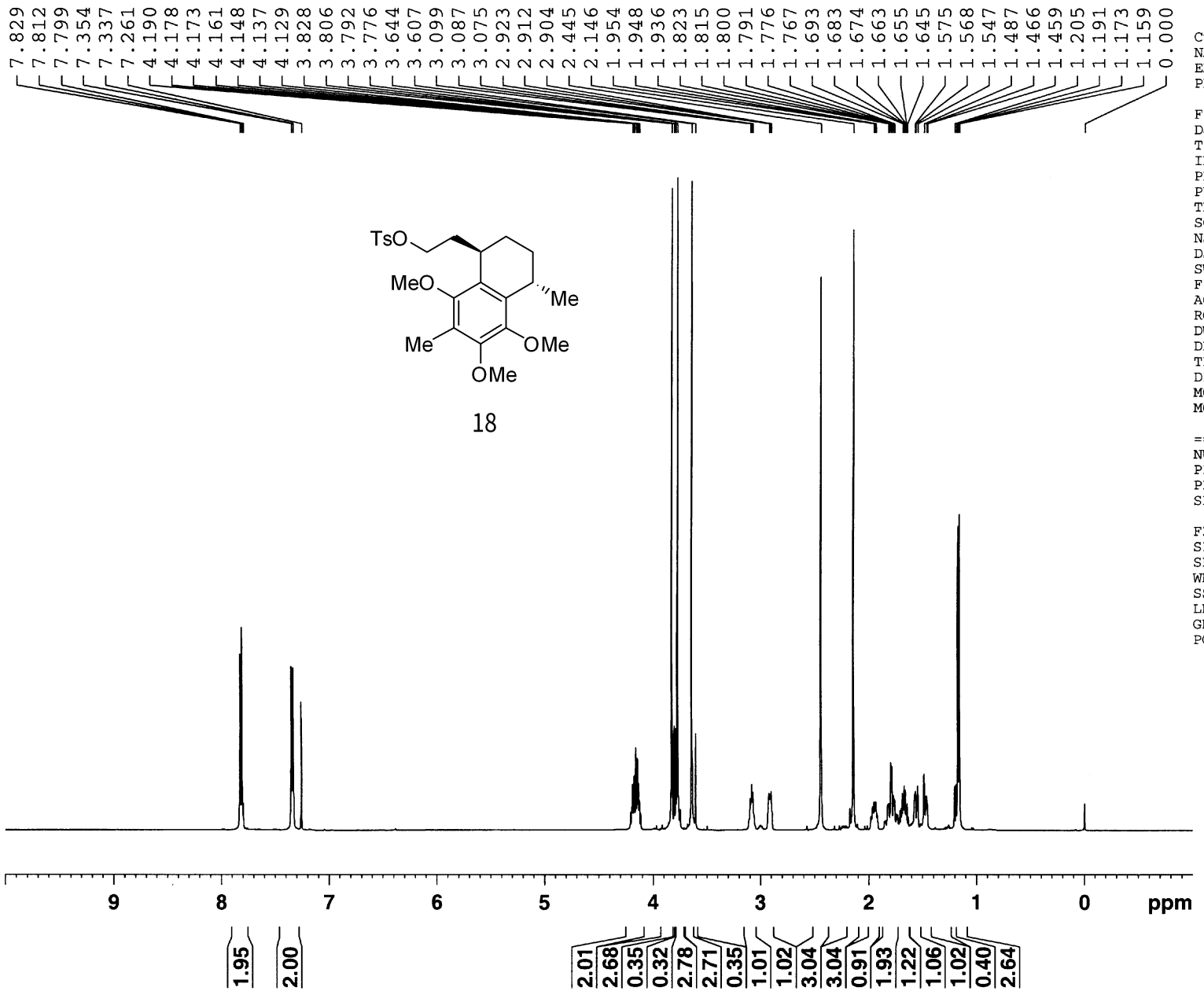
F2 - Acquisition Parameters
Date_                 20090112
Time                     14.40
INSTRUM                DRX500
PROBHD    5 mm CPTCI 1H-
PULPROG                zgpg30
TD                      71424
SOLVENT                 CDCl3
NS                         91
DS                          4
SWH                35211.270 Hz
FIDRES             0.492989 Hz
AQ                 1.0142708 sec
RG                       4096
DW                     14.200 usec
DE                     35.00 usec
TE                     300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                     12.00 usec
PL1                     0.30 dB
SFO1             125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     5.00 dB
PL12                   22.00 dB
PL13                   27.90 dB
SFO2             500.1320005 MHz

F2 - Processing parameters
SI                         65536
SF               125.7577976 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                          1.00
```

1H NMR



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Current Data Parameters

NAME	wy-XI-84-A
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090126
Time	15.52
INSTRUM	DRX500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	5
DW	48.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

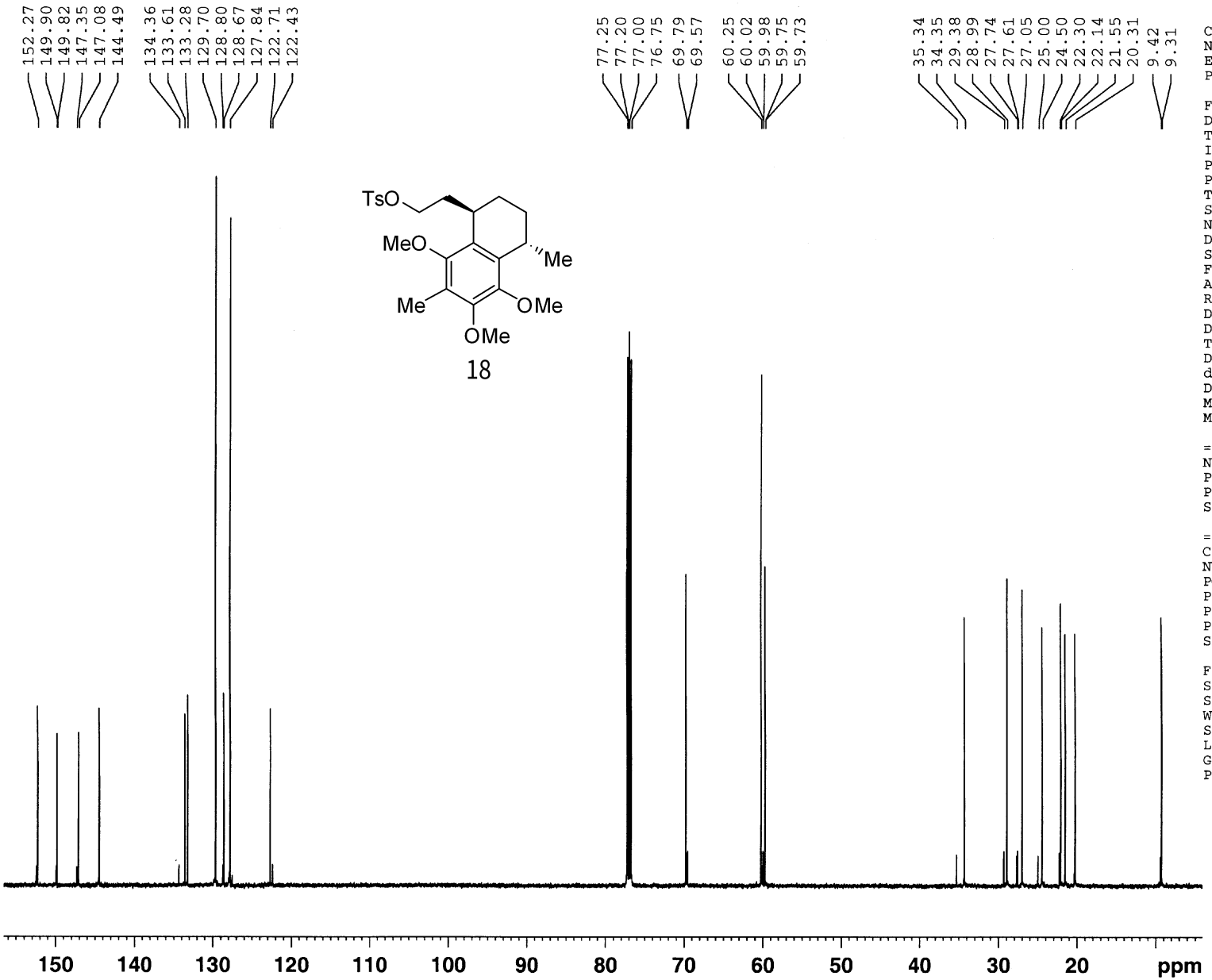
======== CHANNEL f1 ========

NUC1	1H
P1	8.00 usec
PL1	4.30 dB
SFO1	500.1335009 MHz

F2 - Processing parameters

SI	32768
SF	500.1300138 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

13C NMR



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```
Current Data Parameters
NAME          wy-XI-84-A
EXPNO                  2
PROCNO                 1

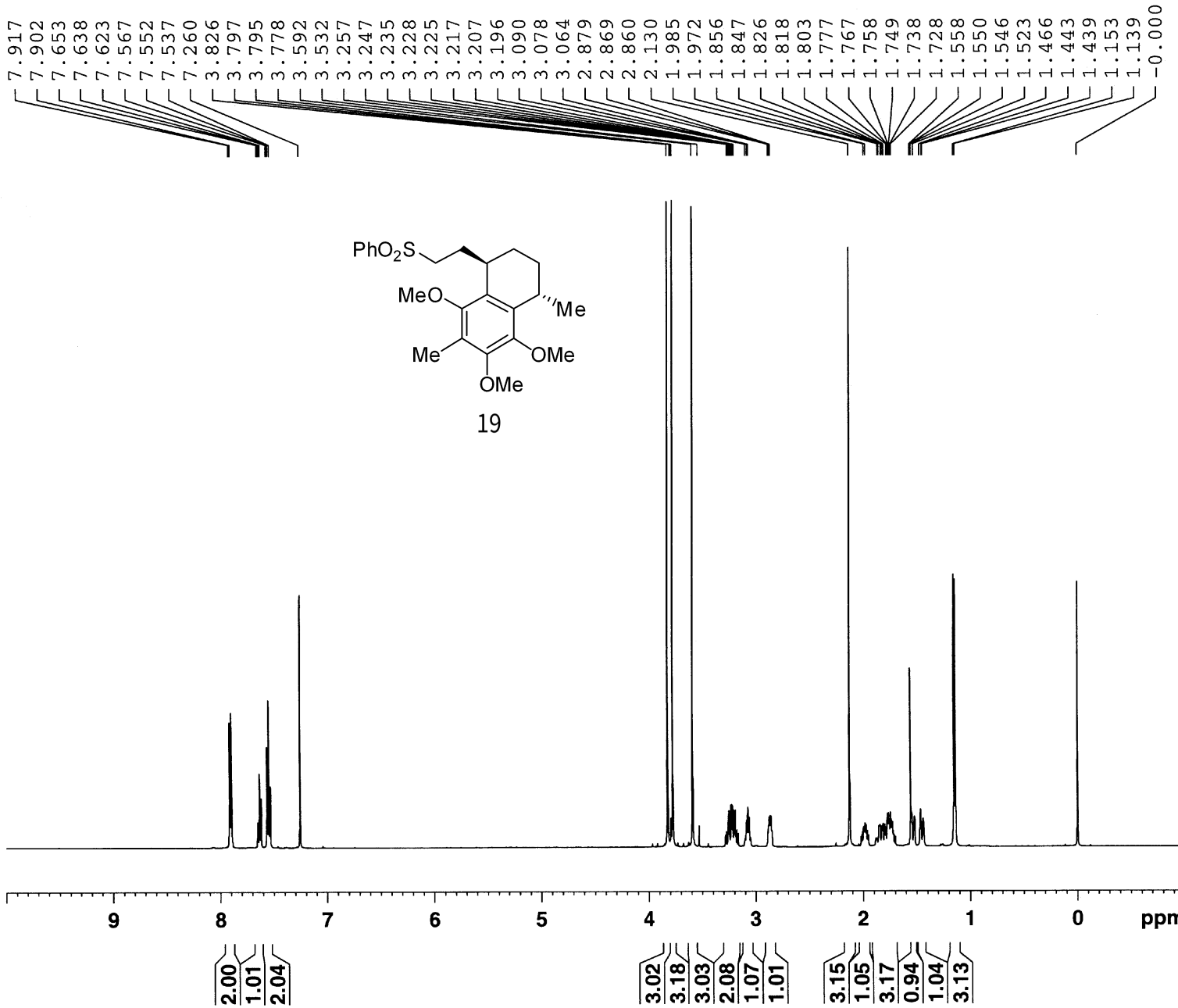
F2 - Acquisition Parameters
Date_           20090126
Time               15.56
INSTRUM           DRX500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgpg30
TD                71424
SOLVENT           CDCl3
NS                   72
DS                    4
SWH           35211.270 Hz
FIDRES         0.492989 Hz
AQ             1.0142708 sec
RG                 4096
DW                14.200 usec
DE                35.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                12.00 usec
PL1                0.30 dB
SFO1        125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2                5.00 dB
PL12              22.00 dB
PL13              27.90 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 65536
SF          125.7578009 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.00
```

1H NMR



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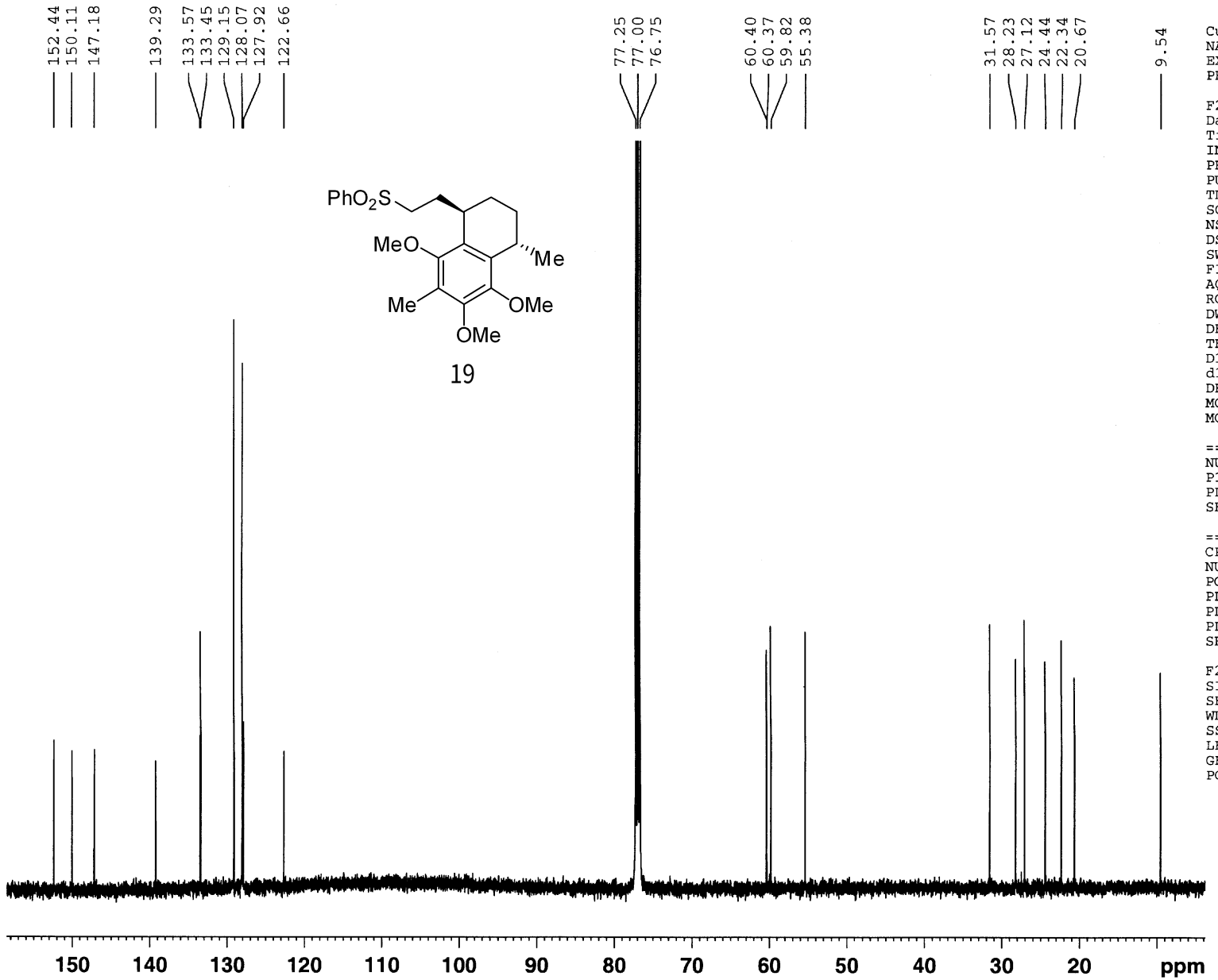
Current Data Parameters
NAME wy-XI-82-Crystl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090128
Time 14.11
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 14.3
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

13C NMR



Current Data Parameters
NAME wy-XI-82-Crystl
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090128
Time 14.15
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDC13
NS 505
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 65536
SF 125.7577923 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40