

SUPPORTING INFORMATION

Title: Intermolecular [4+2] Cycloadditions of a Reactive Cyclopentadienone

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General Information:

All reactions were carried out in oven dried nitrogen stream cooled glassware. Toluene was distilled over CaH_2 and stored over 4A° molecular sieves and used for a month. THF was freshly distilled over sodium-benzophenone and methylene chloride was over CaH_2 .

Chromatographic separation was performed on Silicycle ultra pure silica gel (230-400 mesh) and also on 60 A° mesh from Aldrich. Analytical thin layer chromatography was done on silica gel with UV indicator from Sigma-Aldrich.

Melting points were determined with a Fisher-Johns melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. $^1\text{H-NMR}$ were recorded on a Bruker ARS-250 (250 MHz), DRX-300 (300 MHz), and DRX-500 (500 MHz) spectrometer and are reported in ppm (δ) from tetramethylsilane (TMS: $\delta = 0.0\text{ppm}$). Data are reported according to the following sequence: chemical shift, multiplicity (s =singlet, d =doublet, t =triplet, q= quartet, m= multiplet, dd= doublet of doublet, ddd= doublet of doublet of doublet, bm= broad multiplet), coupling constants in Hz, and integration. $^{13}\text{C-NMR}$ spectra were performed on a Bruker ARS-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 (CDCl_3 : $\delta 77\text{ ppm}$).

Compounds **DA-17**¹ and **DA-18**¹ are reported in the literature.

Experimental : General procedure

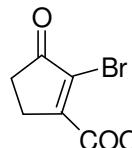
Diels-Alder reactions in THF: Bromo-enone ester **11** was taken into a 10mL flask equipped with a stir bar and a condenser. A solution of this compound was made in THF in 0.10-0.04 M concentration. Desired amount of diene and triethylamine were added into this solution and heated to gentle reflux under nitrogen atmosphere. Reaction was monitored by TLC. Usually a pale yellow color reaction mixture was obtained when the reaction was complete. After completion of the reaction, THF was removed under vacuum. The crude product was dissolved in dichloromethane, washed with 1×5mL 1M HCl, 1×5mL water, 1×5mL brine, dried over MgSO_4 and concentrated. Purification of

this crude product by flash chromatography on silica gel with 15-25% EtOAC/hexanes gave the desired Diels-Alder adduct.

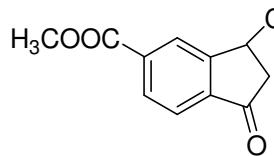
Diels-Alder reactions in toluene: Reactions were usually carried out in 20-40mg scale of compound **11** and reproducible. Into a 10mL flask enone ester **11** was taken and a solution of this was made in toluene in 0.04 M concentration. Desired amount of diene and triethylamine were added into this solution and heated to reflux under nitrogen atmosphere at bath temperature 120-130°C. Reaction was monitored by TLC. A nice light yellow color was an indicative of a successful reaction. After completion of the reaction, toluene was removed under vacuum. Purification of this crude product by flash chromatography on silica gel with 15-20% EtOAC/hexanes provided the desired product.

Diels-Alder reaction with Danishefsky diene: A solution of Bromo-enone-ester **11** (8.1mg, 0.037mmol) and Et₃N.HBr (0.007 mmol, 20 mol%) was made in d₆-benzene under nitrogen atmosphere. Danishefsky diene (0.185mmol) and Et₃N (0.111 mmol) were added to the reaction mixture and transferred to an NMR tube. The reaction was monitored by ¹H-NMR at 80 °C. After 5.5 hours all the SM (**11**) was consumed. The reaction mixture was cooled down to room temperture and poured into 5% HCl. The aqueous medium was extracted with EtOAC (3 × 10mL). The combined organic layers was washed with NaHCO₃ solution, brine, dried over Na₂SO₄ and concentrated. Purification by flash chromatography on silica gel with Hexanes/Et₂O (1: 2) provided 53% of the DA adduct.

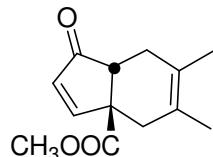
Analytical Data section:



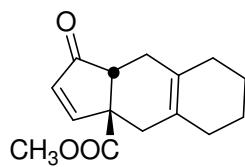
(11) White solid, mp 66-67 °C, IR (neat) 3000, 2970, 2361, 2333, 1718 cm⁻¹; ¹H-NMR (300 MHz, CDCl₃): δ 2.63-2.66 (m, 2H), 2.88-2.91 (m, 2H), 3.92 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 28.3, 32.6, 52.4, 130.9, 157.5, 163.8, 201.3; HRMS calculated for C₇H₇BrO₃⁺ 217.957307 amu, found 217.9597 amu.



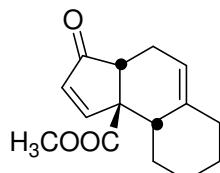
(14) Solid, mp 98-99°C, IR (neat) 3020, 2400, 1724, 1615, 1523, 1437, 1301, 1255, 1210 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 2.94-3.00 (dd, *J*= 19.1, 8.2 Hz, 1H), 3.20-3.26 (dd, *J*= 19.2, 3.7 Hz, 1H), 3.82 (s, 3H), 3.97 (s, 3H), 4.36-4.39 (dd, *J*= 8.1, 3.6, 1H), 7.80-7.81 (d, *J*=8.1 Hz, 1H), 8.31-8.34 (dd, *J*= 8.1, 1.6, 1H), 8.4 (d, *J*=1.5Hz, 1H), 8.34-; ¹³C NMR (125 MHz, CDCl₃) δ 39.7, 43.5, 52.5, 52.9, 125.4, 126.8, 131.2, 135.7, 136.6, 155.0, 165.9, 171.5, 202.9; HRMS calculated for C₁₃H₁₂O₅Na⁺ 271.057693 amu, found 271.05614 amu.



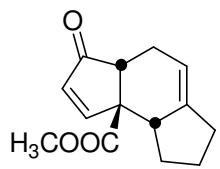
(DA-15) White solid, mp 60-61 °C, 73% yield, IR (neat) 2987, 2952, 2858, 1715, 1437, 1294, 1220, 1132 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ : 1.59 (s, 3H), 1.64 (s, 3H), 2.17-2.20 (d, *J*= 14.7 Hz, 1H), 2.22-2.28 (bm, 1H), 2.34-2.40 (dd, *J*= 14.7, 3.1 Hz, 1H), 2.3-2.6 (bd, *J*= 14.7 Hz, 1H), 2.85-2.90 (dd, *J*= 6.1, 3.1, 1H), 3.75 (s, 3H), 6.14-6.14 (d, *J*=5.6 Hz, 1H), 7.44-7.45 (d, *J*= 5.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 19.18, 19.22, 31.0, 37.5, 48.3, 52.6, 56.9, 124.0, 128.2, 134.4, 163.4, 174.2, 210.1; HRMS calculated for C₁₃H₁₆O₃Na⁺ 234.099163 amu, observed mass 243.09964 amu



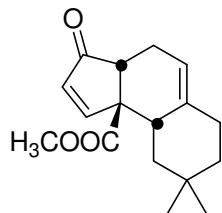
(DA-16) Oil, 82% yield, IR (neat) 2930, 715, 1436, 1252 cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 1.45-1.55 (m, 4H), 1.67-2.50 (m, 4H), 2.25-2.35 (m, 2H), 2.86-2.90 (dd, $J= 12, 6.6\text{Hz}$, 1H), 3.75 (s, 3H), 6.13-6.15 (d, $J= 5.6\text{ Hz}$, 1H), 7.44-7.46 (d, $J= 5.6\text{ Hz}$, 1H) ; ^{13}C -NMR (125 MHz, CDCl_3) δ 22.8, 22.9, 29.63, 30.3, 30.2, 36.4, 48.2, 52.6, 56.8, 126.6, 130.7, 134.1, 163.5, 174.2, 210.1; HRMS calculated for $(\text{C}_{15}\text{H}_{18}\text{O}_3 + \text{O}_2 + \text{Na}^+)$ 301.104643 amu, found 301.10599 amu.



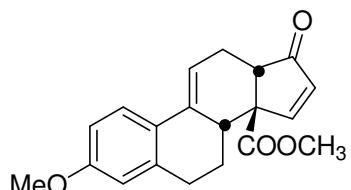
(DA-19) Oil, 77% yield in Toluene, 63% in THF , IR (neat) 2936, 2867, 1717, 1435, 1236, cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 1.35-1.27 (m, 1H), 1.50-1.40 (m, 2H), 1.61-1.53 (m, 1H), 1.83-1.71(m, 2H), 2.15-2.02 (m, 2H), 2.29-2.22 (m, 1H), 2.65-2.60 (ddd, $J= 17.0, 6.5, 2.0\text{ Hz}$, 1H), 2.73-2.68 (m, 1H), 2.76-2.73 (dd, $J= 7.2, 2.1\text{ Hz}$, 1H), 3.79 (s, 3H), 5.51-5.47 (m, 1H), 6.25-6.24 (d, $J= 5.8\text{Hz}$, 1H), 7.70-7.69 (d, $J= 5.8\text{Hz}$, 1H); ^{13}C -NMR (62.5 MHz, CDCl_3) δ 23.0, 23.1, 26.1, 30.9, 42.1, 49.3, 52.6, 58.6, 118.8, 134.2, 138.8, 162.1, 174.6, 209.0; HRMS calculated for $\text{C}_{15}\text{H}_{18}\text{O}_3\text{Na}^+$ 301.104643 amu, found 301.10412 amu.



(DA-20) Oil, 56% yield in Toluene, 33% yield in THF, IR (neat) 2952, 1715, 1434, 1233 cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 2.02-1.96 (m, 1H), 2.24-2.08 (m, 3H), 2.68-2.63 (ddd, 14.7, 7.3, 1.9Hz, 1H), 2.83-2.82 (dd, $J= 6.2, 2.0\text{ Hz}$, 1H), 3.77 (s, 3H), 6.00-5.78 (m, 1H), 6.22-6.21 (d, $J= 5.8\text{Hz}$, 1H), 7.60-7.59 (d, $J=5.8\text{Hz}$, 1H); ^{13}C NMR (62.5 MHz, CDCl_3) δ 25.1, 26.2, 28.1, 31.6, 45.3, 50.0, 52.6, 59.4, 116.5, 135.6, 144.7, 161.6, 174.5, 209.9; HRMS calculated for $\text{C}_{14}\text{H}_{16}\text{O}_3\text{Na}^+$ 255.099163amu, found 287.08989 amu.



(DA-21) Oil, 80% yield, IR (neat), 2953, 2927, 2863, 1717, 1237 cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 0.96 (s, 3H), 0.97 (s, 3H), 1.28-1.16 (m, 2H), 1.43-1.32 (m, 2H), 2.10-1.98 (m, 2H), 2.25-2.16 (m, 1H), 2.65-2.59 (ddd, J = 15.7, 7.9, 1.9 Hz, 1H), 2.73-2.70 (dd, J = 6.8, 2.0 Hz, 1H), 2.86-2.80 (m, 1H), 3.77 (s, 3H), 5.50-5.49 (b, 1H), 6.24-6.23 (5.8 Hz), 7.66-7.64 (d, J = 5.9 Hz, 1H); ^{13}C -NMR (75 MHz, CDCl_3) δ 23.5, 27.5, 28.5, 30.1, 37.3, 38.4, 38.9, 49.7, 52.6, 58.8, 118.8, 134.7, 138.9, 162.1, 174.6, 209.3,



(DA-22) White semi solid, 80% yield, IR (neat) 3020, 2949, 2839, 1713, 1607, 1572, 1496, 1435, 1248 cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 1.64-1.56 (m, 1H), 2.07-2.02 (m, 1H), 2.41-2.36 (m, 1H), 2.73-2.62 (m, 2H), 2.85-2.80 (m, 2H), 3.14-3.09 (m, 1H), 3.77 (s, 3H), 3.81 (s, 3H), 6.18-6.16 (m, 1H), 6.22-6.20 (d, J = 5.8 Hz, 1H), 6.61 (dd, J = 2.61 Hz, 1H), 6.71-6.68 (dd, J = 8.6, 2.7 Hz, 1H), 7.40-7.39 (d, J = 5.7 Hz, 1H), 7.62-7.61 (d, J = 5.8 Hz, 1H); ^{13}C -NMR (75 MHz, CDCl_3) δ 23.8, 25.0, 30.0, 41.5, 50.7, 52.7, 55.2, 60.0, 112.66, 112.72, 119.0, 125.0, 126.9, 135.1, 136.0, 138.7, 158.7, 161.5, 174.3, 209.6; HRMS calculated 347.125378 amu, found 347.12457 amu.

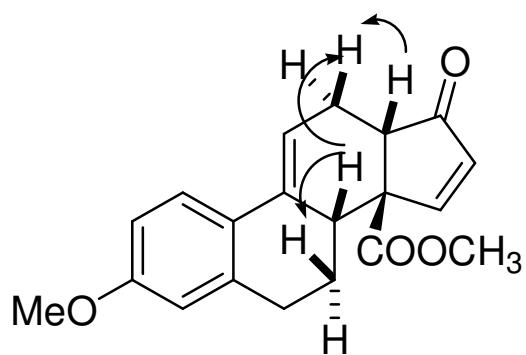
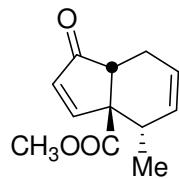
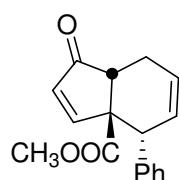


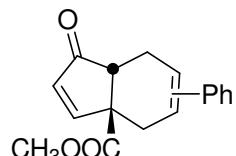
Figure 1: The nOe effect in Diels-Alder adduct **22**



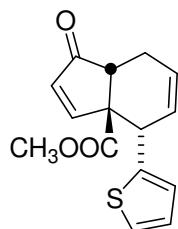
(DA-23) Oil, 13% yield; IR (neat): 3036, 2954, 2879, 2848, 1715, 1595, 1457, 1436, 1254, 1231 cm^{-1} ; $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 1.18 (d, J = 9.0 Hz, 3H), 2.19-2.25 (m, 1H), 2.64-2.69 (ddd, J = 15.0, 7.5, 2.2 Hz, 1H), 2.75-2.77 (dd, J = 7.5, 2.0 Hz, 1H), 2.80-2.85 (m, 1H), 3.77 (s, 3H), 5.42-5.45 (m, 1H), 5.81-5.86 (m, 1H), 6.25-6.26 (d, J = 5.5 Hz, 1H), 7.63-7.65 (d, J = 5.5 Hz, 1H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 15.9, 24.4, 35.5, 50.1, 52.6, 60.3, 128.0, 132.5, 135.7, 161.5, 174.4, 209.4; Elemental analysis: Expt C, 69.83%; H, 6.59% Calculated C 69.88%, H, 6.84%.



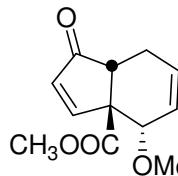
(DA-24) Oil, 66% yield; IR (neat): 3032, 2952, 2847, 1717, 1598, 1454, 1254, 1230 cm^{-1} ; $^1\text{H-NMR}$ (250 MHz, CDCl_3): δ 2.10-2.40 (m, 1H), 2.75-2.87 (m, 2H), 3.68 (s, 3H), 4.05-4.09 (q, J = 2.9 Hz, 1H), 5.87-5.93 (m, 1H), 5.99-6.04 (m, 1H), 6.20-6.23 (d, J = 5.9 Hz, 1H), 7.09-7.15 (m, 2H), 7.18-7.21 (d, J = 5.9 Hz, 1H), 7.29-7.40 (m, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 24.2, 47.0, 49.5, 52.4, 61.5, 127.4, 128.5, 129.0, 129.1, 130.7, 134.8, 139.1, 162.2, 174.0, 209.0; Elemental analysis: Expt C, 75.97%; H, 6.23% Calculated C 76.10%, H, 6.01%



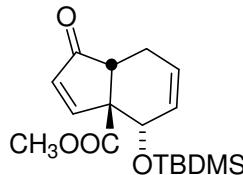
(DA-25) Oil, 80% yield, 1:1 mixture of two regeoisomers; IR (neat): 3032, 2952, 2900, 2844, 1714, 1593, 1495, 1436, 1249, 1220 cm^{-1} ; $^1\text{H-NMR}$ (250 MHz, CDCl_3): δ 2.35-3.15 (m, 5H, from each regeoisomers), 3.79 (s, 3H, from each regeoisomers), 5.9-5.97 (m, 1H from one regioisomer), 6.15-6.23 (3H, from both isomers), 7.17-7.37 (m, 5H, from each regeoisomers), 7.47-7.52 (d, 2H, J = 5.6 Hz, from each regeoisomers); $^{13}\text{C NMR}$ (62.5 MHz, CDCl_3): δ 25.4, 27.8, 31.7, 33.7, 47.5, 48.4, 52.78, 52.83, 56.4, 56.97, 120.6, 124.3, 125.2, 125.5, 127.26, 127.3, 128.35, 128.40, 134.63, 134.9, 140.18, 140.54, 163.4, 163.9, 173.9, 173.9, 209.28, 209.8, from both the regeoisomers; HRMS calculated for C17H16O3Na^+ to be 291.099163 amu, found 291.09932 amu.



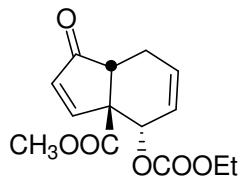
(DA-26) Oil, 78% yield; IR (neat): 3044, 2952, 2902, 2361, 2329, 1716, 1435, 1297, 1231 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 2.98-2.40 (m, 1H), 2.74-2.85 (m, 2H), 3.75 (s, 3H), 4.47-4.44 (dd, $J= 5.7, 3.0$ Hz, 1H), 5.89-6.06 (m, 2H), 6.26-6.24 (d, $J= 5.6$ Hz, 1H), 6.85-6.82 (d, $J= 3.6$, 1H), 7.02-6.99 (q, $J= 3.6$ Hz, 1H), 7.25-7.23 (dd, $J= 5.1, 1.2$ Hz, 1H), 7.32-7.29 (d, $J= 5.6$ Hz, 1H); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 24.5, 42.0, 49.6, 52.7, 61.8, 124.8, 126.1, 127.1, 129.1, 130.7, 135.47, 142.06, 162.0, 173.9, 208.8; Elemental analysis: Expt C, 65.72%; H, 4.99% Calculated C 65.67%, H, 5.14%



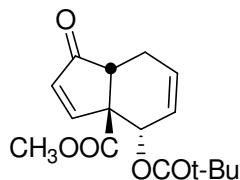
(DA-27) Oil, 84 % yield in Toluene, 52% yield in THF, IR (neat): 3046, 2952, 2829, 1716, 1593, 1437, 1256 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 2.22-2.17 (m, 1H), 2.69-2.63 (m, 2H), 3.47 (s, 3H), 3.81 (s, 3H), 4.48-4.47 (dd, $J= 4.3, 2.2$ Hz, 1H), 5.84-5.76 (m, 2H), 6.33-6.32 (d, $J= 5.8$ Hz, 1H), 7.73-7.72 (d, $J= 5.8$ Hz, 1H); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 24.3, 48.4, 52.8, 58.1, 61.1, 80.0, 126.8, 129.6, 135.9, 162.4, 173.9, 208.8; HRMS (EI) calculated for $\text{C}_{12}\text{H}_{14}\text{O}_4^+$ 222.08921 amu, found 222.0779 amu.



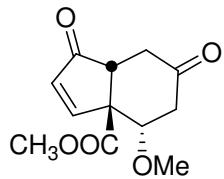
(DA-28) Oil, 79% Yield, IR (neat): 2953, 2856, 1718, 1254, 1119 cm^{-1} ; $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 0.08 (s, 3H), 0.10 (s, 3H), 0.92 (s, 9H), 2.23-2.18 (m, 1H), 2.67-2.62 (m, 2H), 3.77 (s, 3H), 4.85-4.84 (q, $J= 4.72, 2.32$ Hz, 1H), 5.61-5.58 (m, 1H), 5.76-5.71 (m, 1H), 6.32-6.31 (d, $J= 5.8$ Hz, 1H), 7.80-7.79 (d, $J= 5.8$ Hz 1H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 5.4, -4.6, 17.9, 24.3, 25.6, 30.9, 48.1, 52.6, 62.5, 72.3, 125.9, 133.1, 135.8, 162.9, 174.0, 207.0; HRMS calculated for $\text{C}_{17}\text{H}_{26}\text{O}_4\text{SiNa}^+$ 345.149255 amu, found 345.14820 amu.



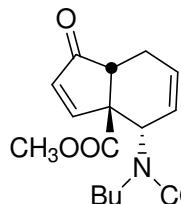
(DA-29) Oil, 72% yield; IR (neat): 2956, 2361, 1750, 1718, 1437, 1472, 1257 cm⁻¹; ¹H-NMR (300 MHz, CDCl₃): δ 1.31-1.36 (t, *J*= 7.2 Hz, 3H), 2.20-2.34 (m, 1H), 2.66-2.74 (ddd, *J*= 16.7, 6.9, 2.1 Hz, 1H), 2.79-2.83 (dd, *J*= 6.9, 2.1 Hz, 1H), 3.78 (s, 3H), 4.20-4.28 (q, *J*= 7.2 Hz, 2H), 5.63-5.68 (dt, *J*= 9.6, 2.7 Hz, 1H), 5.83-5.92 (m, 1H), 6.34 (d, *J*= 5.7 Hz, 1H), 7.72 (d, *J*= 5.7 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 14.2, 23.9, 47.4, 53.1, 60.0, 64.6, 75.4, 127.8, 128.1, 136.4, 154.4, 160.6, 172.4, 208.0; Elemental analysis: Expt C, 60.16%; H, 5.57% Calculated C 59.99%, H, 5.75%.



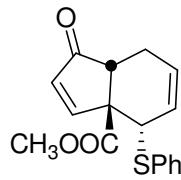
(DA-30) White Solid, mp °C, 61% yield; IR (neat): 2974, 1736, 1716, 1480, 1485, 1437, 1255, 1279, 1234, 1147 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 1.23 (s, 9H), 2.36-2.28 (m, 1H), 2.72-2.66 (ddd, *J*= 15.6, 7.5, 2.5 Hz, 1H), 2.91-2.88 (dd, *J*= 7.0, 2.5 Hz, 1H), 3.76 (s, 3H), 5.60-5.56 (dt, *J*= 10.0, 2.5 Hz, 1H), 5.78-5.76 (q, *J*= 2.5 Hz, 1H), 5.91-5.86 (m, 1H), 6.34 (d, *J*= 6.0 Hz, 1H), 7.66 (d, *J*= 6.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): 23.9, 27.0, 38.9, 47.1, 53.0, 60.2, 71.4, 128.0, 128.4, 136.1, 160.6, 172.4, 177.2, 208.1; Elemental analysis: Expt C, 65.57%; H, 6.85% Calculated C 65.74%, H, 6.90%



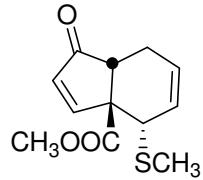
(DA-31) Oil, 53% yield, IR (neat): 3076, 2955, 2836, 1792, 1718, 1457, 1437, 1232, 1100 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 2.18 (dd, *J*= 18, 10 Hz, 1H), 2.55-2.67 (m, 2H), 2.78-2.95 (m, 2H), 3.42 (s, 3H), 3.83 (s, 3H), 4.34-4.28 (dd, *J*= 8.0, 3.4 Hz, 1H), 6.44-6.41 (d, *J*= 5.8 Hz, 1H), 7.76-7.74 (d, *J*= 5.8 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 37.8, 39.8, 45.7, 53.2, 57.7, 59.0, 77.8, 134.6, 161.0, 173.0, 205.8, 206.5;



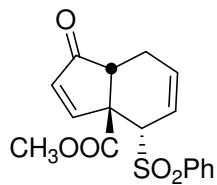
(DA-32) Oil, 84% yield, IR (neat liq) 2957, 2874, 2362, 1718, 1653, 1413, 1239 cm^{-1} ; $^1\text{H-NMR}$ (250 MHz, CDCl_3): δ 0.95-0.90 (t, $J= 7.3\text{Hz}$, 3H), 1.33-1.24 (m, 2H), 1.68-1.40 (m, 2H), 2.14 (s, 3H), 2.38-2.34 (m, 1H), 2.91-2.72 (m, 3H), 3.17-3.11 (m, 1H), 3.78 (s, 3H), 5.43-5.40 (q, $J=2.7\text{ Hz}$, 1H), 5.71-5.64 (dt, $J= 9.8, 3.0\text{ Hz}$, 1H), 6.03-5.96 (m, 1H), 6.23-6.21 (d, $J= 5.9\text{Hz}$, 1H), 7.57-7.54 (d, $J=5.9\text{ Hz}$, 1H). $^{13}\text{C NMR}$ (62.5 MHz, CDCl_3) δ 13.6, 20.2, 21.9, 22.4, 33.4, 48.26, 48.35, 52.9, 56.6, 60.0, 125.6, 129.5, 133.5, 162.0, 171.6, 173.5, 207.9. HRMS calculated for $(\text{C}_{17}\text{H}_{23}\text{NO}_4^- + \text{Na}^+)$ 328.151927 amu, found 328.15191 amu.



(DA-33) Oil, 84% yield; IR (neat) 3053, 2952, 2847, 1717, 1585, 1479, 1438, 1300, 1233 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 2.42-2.27 (m, 1H), 2.73-2.63 (m, 1H), 2.81-2.76 (dd, $=5.3, 2.9, 1\text{ Hz}$, 1H), 3.63 (s, 3H), 4.47-4.44 (m, 1H), 5.83-5.77 (m, 1H), 5.94-5.88 (m, 1H), 6.33-6.32 (d, $J= 5.8\text{ Hz}$, 1H), 7.46-7.25 (m, 5H), 7.77-7.74 (d, $J= 5.8\text{ Hz}$, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 23.8, 49.7, 49.9, 52.8, 60.6, 127.5, 129.3, 129.9, 130.2, 131.9, 134.4, 135.9, 139.1, 161.5, 173.2, 208.2; HRMS calculated for $(\text{C}_{17}\text{H}_{16}\text{O}_3\text{S} + \text{O} + \text{Na})^+$ 339.066148 amu, observed 339.06514 amu.



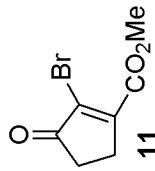
(DA-34) Oil 67% yield; IR (neat): 30.42, 2953, 2920, 2847, 1716, 1594, 1435, 1232 cm^{-1} ; $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 2.16 (s, 3H), 2.26-2.33 (m, 1H), 2.62-2.67 (ddd, $J= 19.2, 6.5, 3.0\text{ Hz}$, 1H) 2.76-2.78 (dd, $J=7.5, 3.0\text{ Hz}$, 1H), 3.79 (s, 3H), 3.92-3.94 (m, 1H), 5.74-5.77 (m, 1H), 5.89-5.92 (m, 1H), 6.3 (d, $J= 5.5\text{Hz}$, 1H), 7.72 (d, $J= 5.5\text{ Hz}$, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 16.1, 23.8, 47.6, 49.8, 52.9, 60.8, 129.0, 130.1, 135.5, 161.7, 173.55, 208.3; Elemental analysis: Expt C, 60.27%; H, 5.76% Calculated C 60.48%, H, 5.92%.



White solid, mp 160-161°C, 74% Yield, IR (neat) cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 2.15-2.05 (m, 1H), 2.81-2.67 (m, 2H), 5.84-5.78 (dt, $J= 3.1$ Hz, 1H), 6.09-6.00 (m, 1H), 6.42-6.39 (d, $J= 5.8$ Hz, 1H), 7.73-7.56 (m, 3H), 7.96-7.92 (m, 2H), 7.96-7.92 (m, 2H), 8.13-8.01 (d, $J= 5.8$ Hz, 1H); $^{13}\text{C NMR}$ (62.5 MHz, CDCl_3) δ 23.9, 50.1, 53.6, 57.6, 65.5, 121.4, 128.4, 129.5, 131.9, 134.2, 135.9, 139.0, 160.6, 173.1, 206.7. X-Ray crystallography data on this compound is provided.

Reference:

1. Hirao, K; Yamashita, A; Ando, A.; Ijima, H.; Yamamoto, Y.; Hamada, T. and Yonemitsu, O *J. Chem. Research (S)*, **1987**, 162-163.



Current Data Parameters
 NAME MG-3-013d-300
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20040824
 Time 23:46

INSTRUM drx300
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 1024
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 D31 0.0000000 sec

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

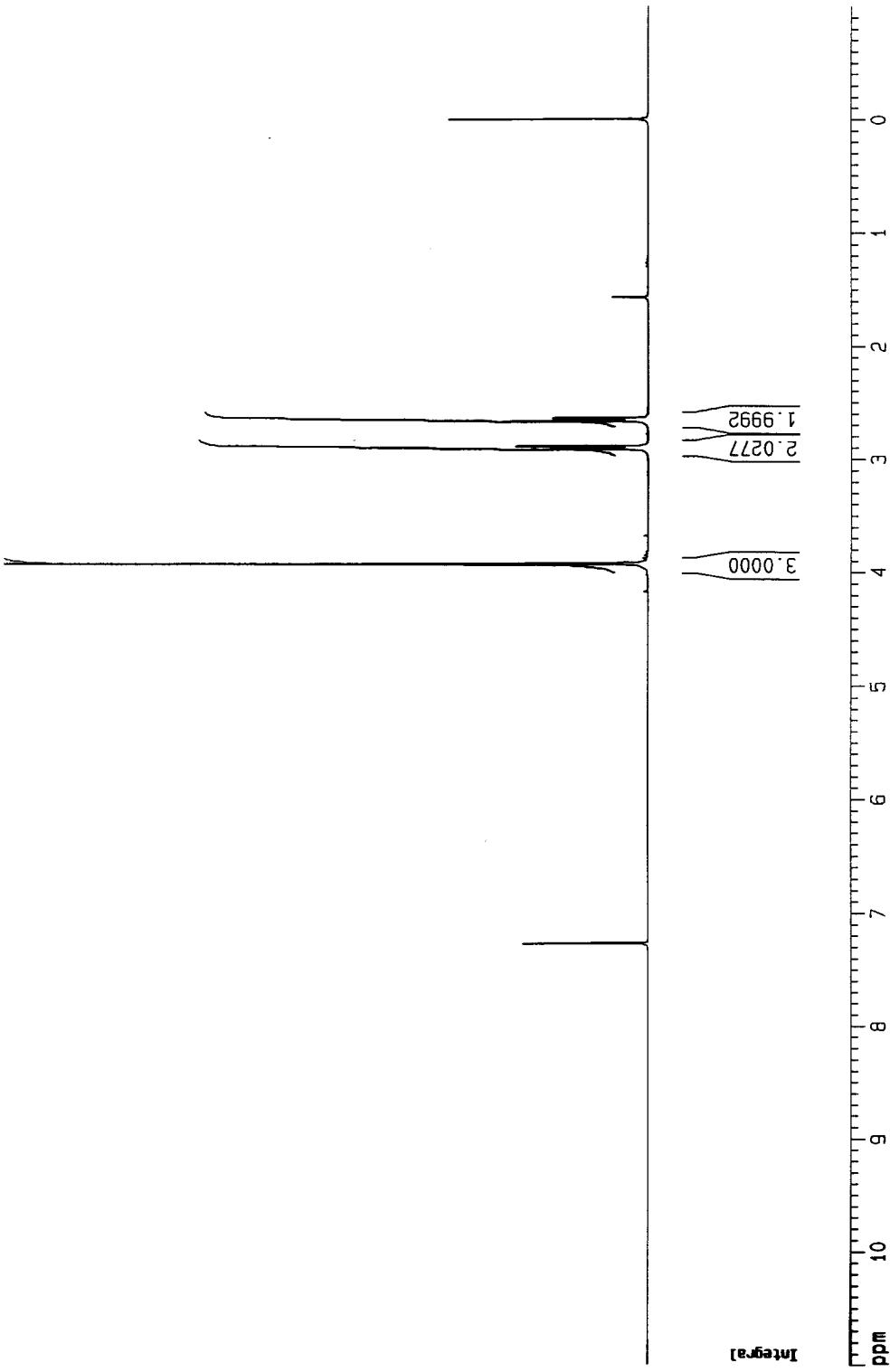
NUC1 1H
 P1 7.05 usec
 PL1 0.00 dB
 SF01 300.1318534 MHz

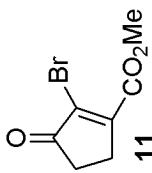
F2 - Processing parameters
 SI 32768
 SF 300.1300047 MHz
 MDW EM

SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.30

1D NMR plot parameters
 CX 20.00 cm

CY 12.50 cm
 F1P 11.000 ppm
 F1 3301.43 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 Hz/cm 180.07799 Hz/cm





Current Data Parameters

NAME	MG-3-013d-300
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	20040826
Time	11:06
INSTRUM	drx300
PROBHD	5 mm Multinuc1
PULPROG	2gdc30
TD	65536
SOLVENT	CDCl3
NS	572
DS	4
SWH	10032.393 Hz
FOIDES	0.297360 Hz
AQ	1.740308 sec
RG	22528
DM	26.550 usec
DE	6.00 usec
TE	297.1 K
D1	1.2999995 sec
d11	0.03000000 sec
D31	0.00000000 sec

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

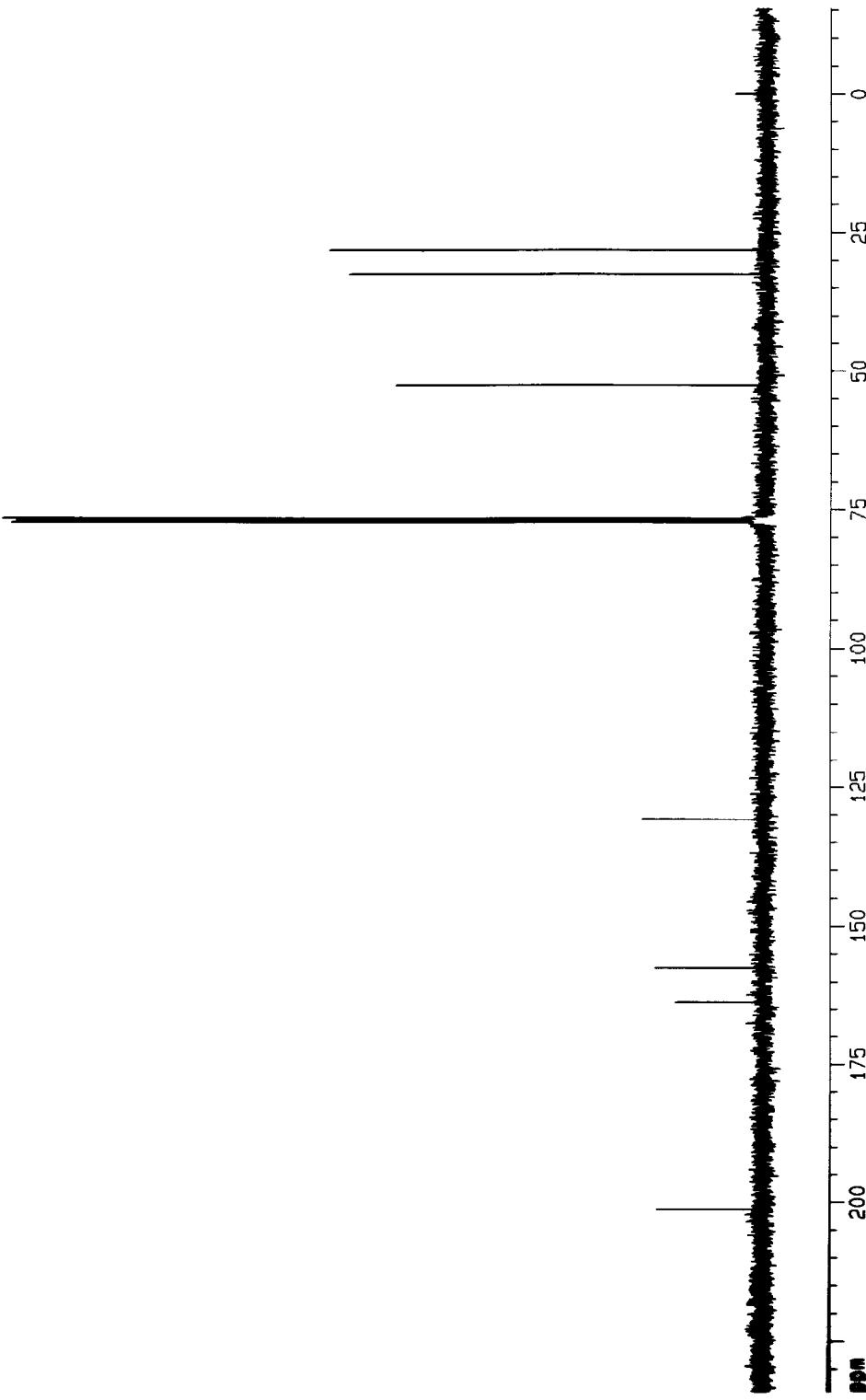
NUC1	13C
P1	8.50 usec
PL1	5.00 dB
SF01	75.4760107 MHz

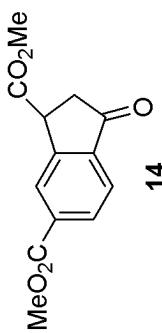
===== CHANNEL f2 =====

OPDPRG2	Waltz16
NUC2	1H
PCPDP2	100.00 usec
PL2	120.00 dB
PL12	25.60 dB
SFQ2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677514 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

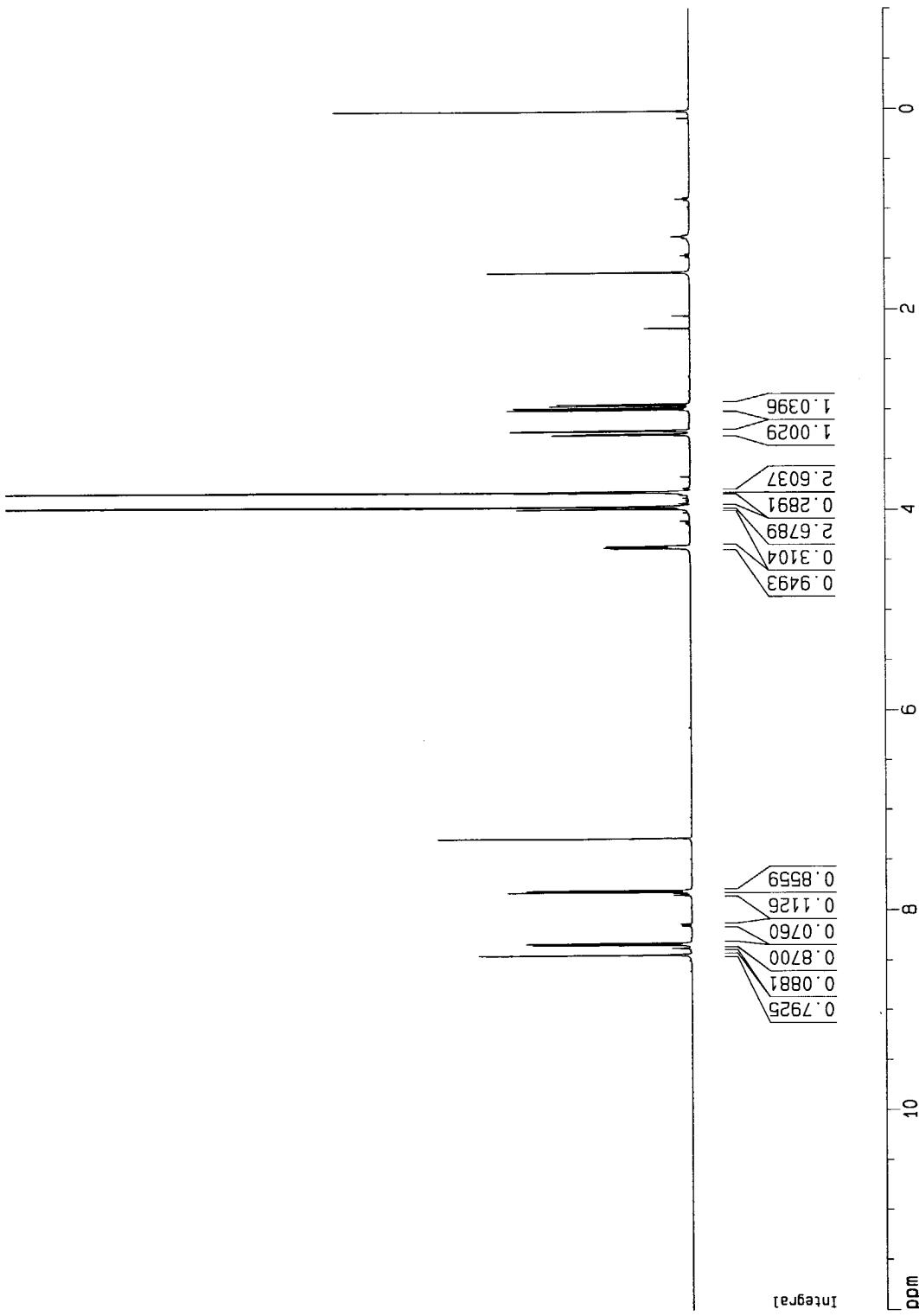


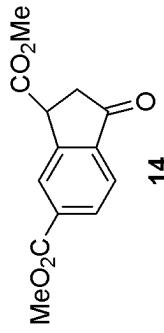


Current Data Parameters
 NAME MG-IV-048b-500
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date	20050506
Time	17.09
INSTRUM	DRX500
PROBHD	5 mm Multinuc
PULPROG	zg30
TD	57344
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FINRES	0.180151 Hz
AQ	2.7754996 sec
RG	161.3
DW	48.400 usec
DE	6.00 usec
TE	296.7 K
DM	1.0000000 sec





Current Data Parameters
 NAME MG-IV-048b-500
 EXPNO 2
 PROCN0 1

F2 - Acquisition Parameters
 Date 20050506
 Time 17.12

INSTRUM DRX500
 PROBHD 5 mm Multinuc1
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 8310
 DS 4
 SWH 39681.812 Hz
 FIDRES 0.60596 Hz
 AQ 0.8258188 sec
 RG 16384
 DW 12.600 usec
 DE 6.00 usec
 TE 298.0 K
 D1 2.0000000 sec
 d11 0.03000000 sec

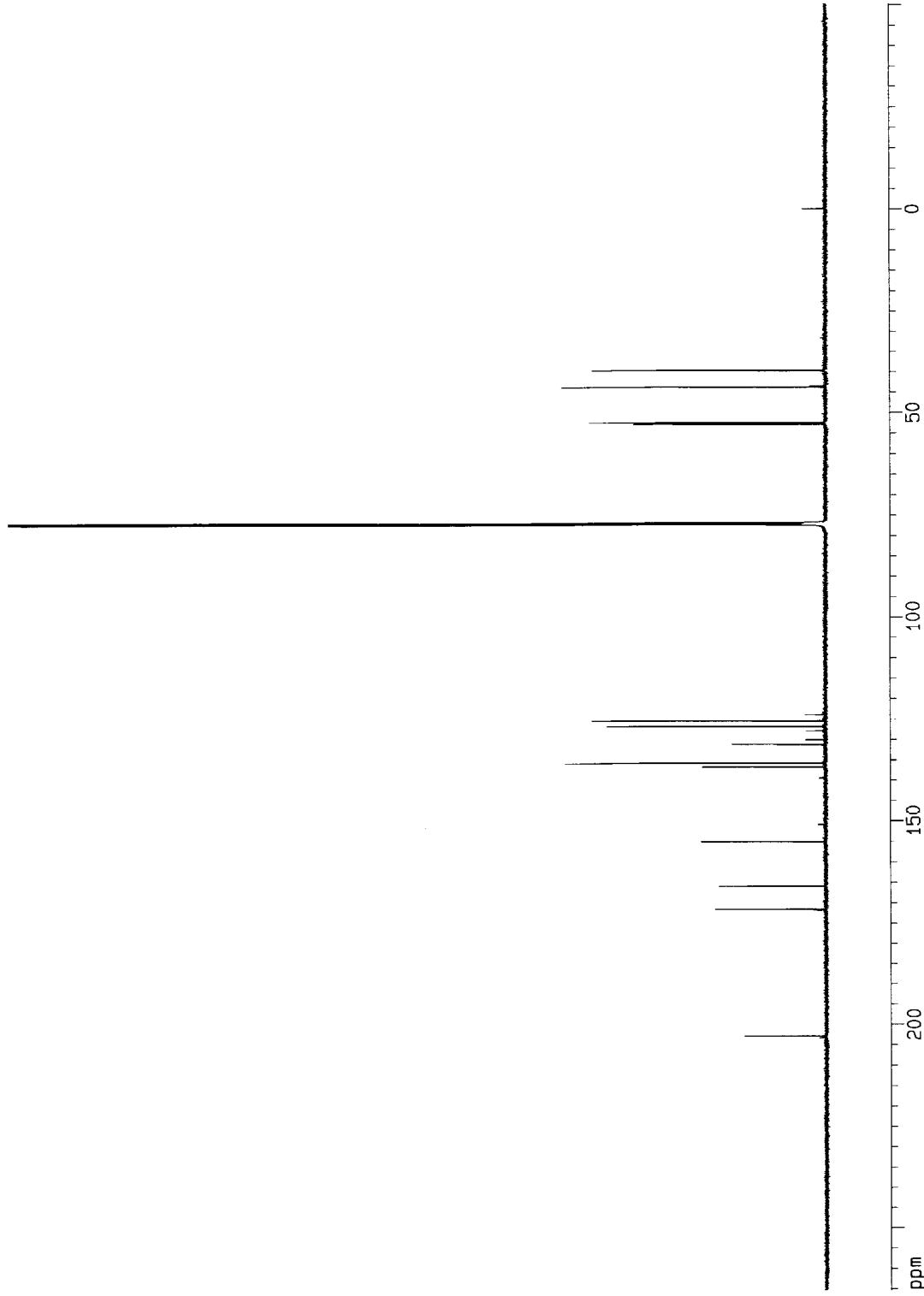
===== CHANNEL f1 =====

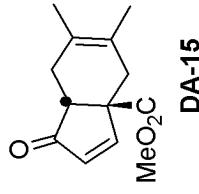
NUC1 13C
 P1 8.10 usec
 PL1 3.00 dB
 SF01 125.7713108 MHz
 CPDPG2 Waltz16
 NUC2 1H
 POPD2 88.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 SF02 500.1320005 MHz

===== CHANNEL f2 =====

SI 32768
 SF 125.7577335 MHz
 WDN EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

F2 - Processing parameters
 ST 20.00 cm
 CY 24.00 cm
 F1P 285.258 ppm
 F1 33358.26 Hz
 F2P -50.284 ppm
 F2 -6323.5 Hz
 PPMCM 15.77708 ppm/cm
 HZCM 1984.09045 Hz/cm





Current Data Parameters

NAME	MG-2-034a-500
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20031220
Time	12.05
INSTRUM	DRX500
PROBHD	5 mm Multinuc1
PULPROG	Z930
TD	57344
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.180151 Hz
AQ	2.7754996 sec
RG	114
DW	40.400 usec
DE	6.00 usec
TE	296.7 K
D1	1.00000000 sec

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

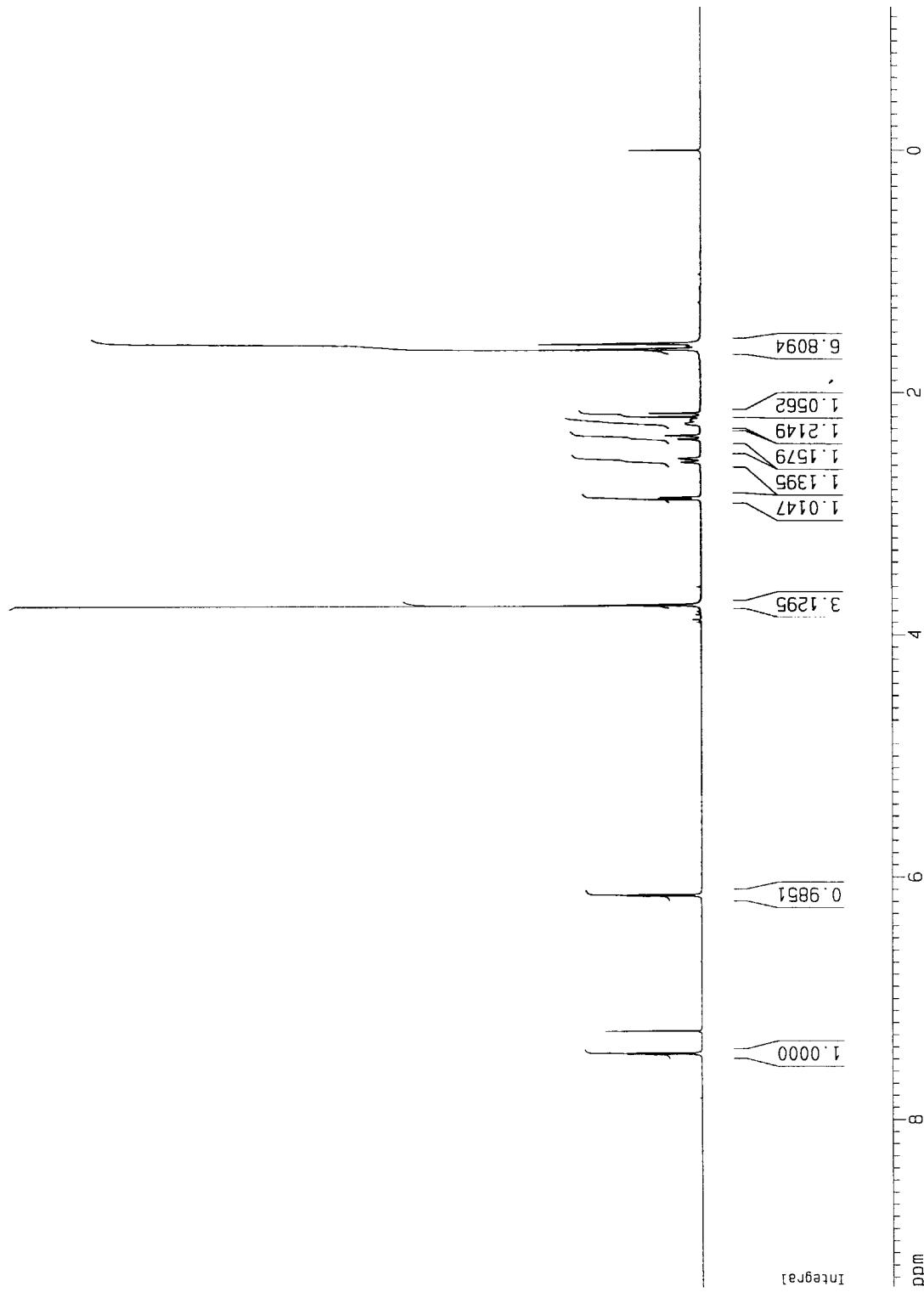
NUC1	1H
P1	13.25 usec
PL1	-3.00 dB
SFO1	500.1330885 MHz

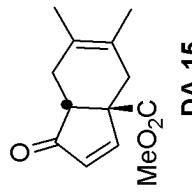
F2 - Processing parameters

SI	32768
SF	500.1330093 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	9.379 ppm
F1	4690.87 Hz
F2P	-1.182 ppm
F2	-590.93 Hz
PPNOM	0.52804 ppm/cr
HZCM	264.08994 Hz/cm





Current Data Parameters

NAME	MG-2-034a-500
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date _	20031220
Time	12.07
INSTRUM	DXR300
PROBHD	5 mm Multinuc1
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl ₃
NS	4994
DS	4
SWH	39681.812 Hz
FIDRES	0.605396 Hz
AQ	0.8958188 sec
RG	16384
DW	12.600 usec
DE	6.00 usec
TE	298.0 K
D1	2.0000000 sec
d11	0.0300000 sec

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

NUC1	¹³ C
P1	8.10 usec
PL1	3.00 dB
SF01	125.7713108 MHz

===== CHANNEL f2 =====

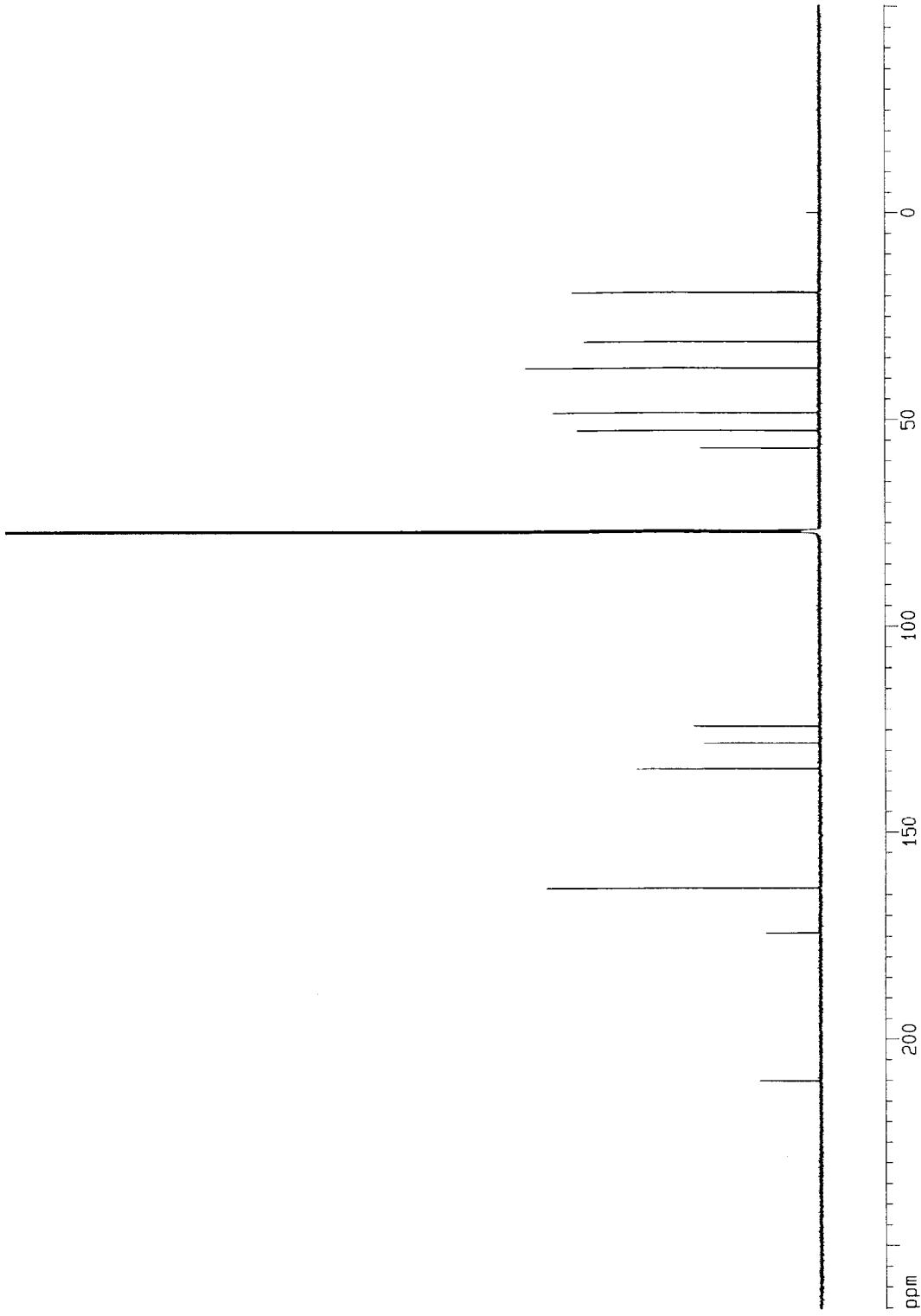
CPDPFG2	wait:16
NUC2	¹ H
PCPD2	88.00 usec
PL2	0.00 dB
PL12	21.00 dB
SF02	500.1320005 MHz

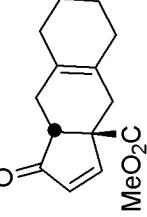
F2 - Processing parameters

SI	32768
SF	125.7377335 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR pilot parameters

CX	20.00 cm
CY	15.00 cm
F1P	265.25B ppm
F1	33358.26 Hz
F2P	-50.284 ppm
F2	-6323.55 Hz
PPCM	15.77708 ppm/cm
H2CM	1984.09045 Hz/cm



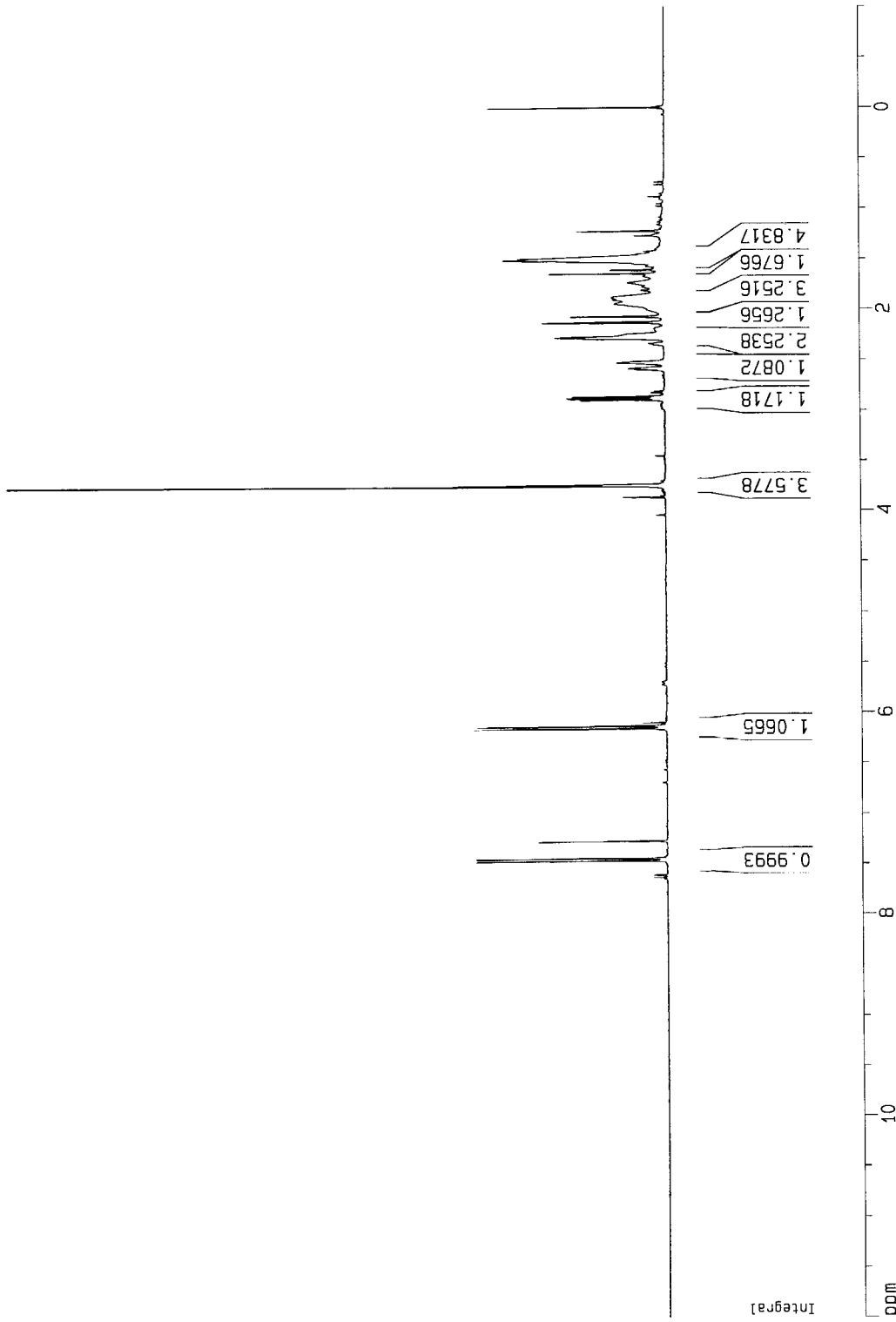


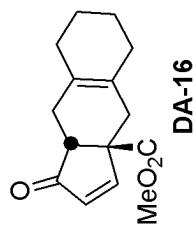
Current Data Parameters
 NAME MG-4-01Ba-250
 EXPNO 1
 PROCNQ 1

F2 - Acquisition Parameters
 Date_ 20050401
 Time 0.52
 INSTRUM grx250
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5208.333 Hz
 FIDRES 0.156946 Hz
 AQ 3.1457779 sec
 RG 715
 DW 96.000 use
 DE 137.14 use
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.70 use
 SF01 250.1315321 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 250.1300043 MHz
 WDDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.50

1D NMR plot parameters
 CX 20.00 cm
 CY 30.00 cm
 F1P 12.000 ppm
 F1 3001.56 Hz
 F2P -1.000 ppm
 F2 -250.13 Hz
 PPMQ 0.65000 ppm
 HZCM 162.58450 Hz





Current Data Parameters
 NAME MG-4-015a-500
 EXPNO 2
 PROCN 1

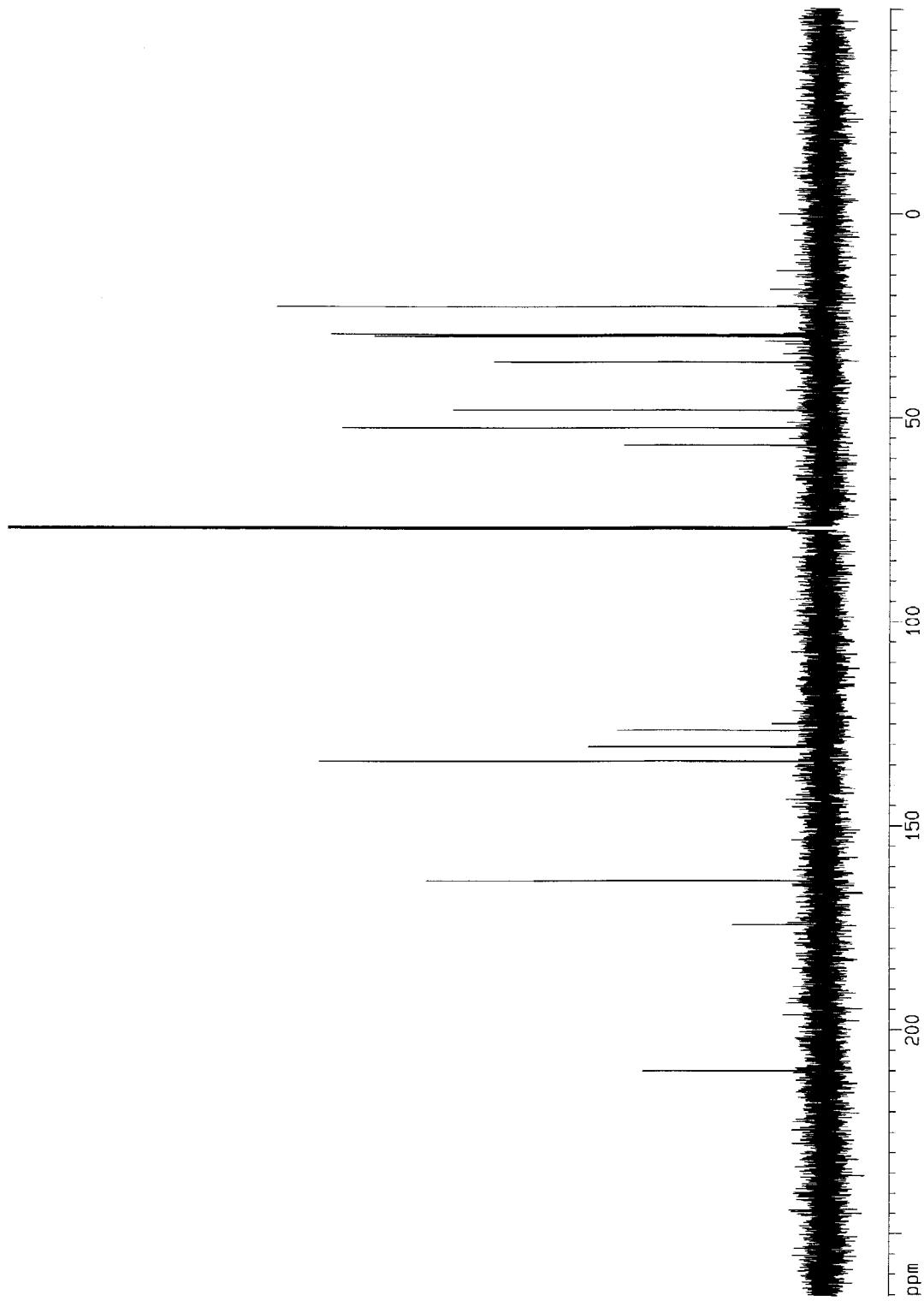
F2 - Acquisition Parameters
 Date 20050329
 Time 1.25
 INSTRUM DR500
 PROBHD 5 mm Multinucl
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 182
 DS 4
 SWH 39681.812 Hz
 FIDRES 0.603496 Hz
 AQ 0.825188 sec
 RG 16384
 DM 12.600 usec
 DE 6.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec

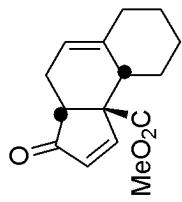
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.10 usec
 PL1 3.00 dB
 SF01 125.771310B MHz

===== CHANNEL f2 =====
 CPDPFG2
 NUC2 1H
 PCP02 88.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 SF02 500.132005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577922 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR pilot parameters
 CX 20.00 cm
 CY 35.00 cm
 F1P 265.268 ppm
 F1 3335.46 Hz
 F2P -50.274 ppm
 F2 -6322.34 Hz
 PPMCM 15.7708 ppm/cm
 HZCM 1994.09033 Hz/cm





DA-19

Current Data Parameters

NAME	MG-2-110a-250
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

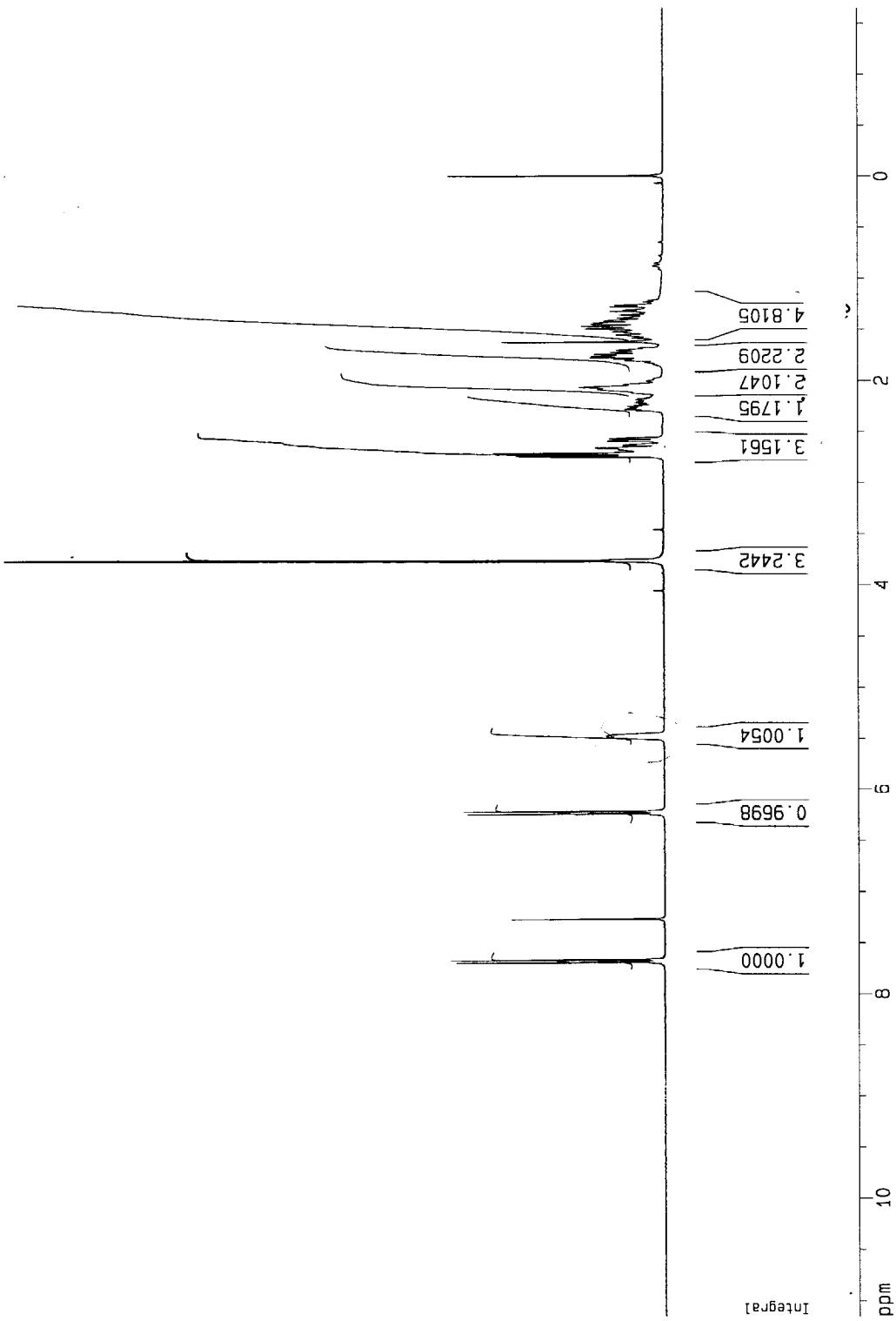
Date -	20040528
Time -	17.18
INSTRUM	airx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDC13
NS	16
DS	2
SWH	5208.333 Hz
TDRES	0.156946 Hz
AQ	3.145779 sec
RG	1430
DW	96.000 use
DE	137.14 use
TE	300.0 K
01	1.0000000 sec
P1	8.70 use
SF01	250.1315321 MHz
NUCLEUS	1H

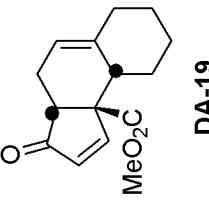
F2 - Processing parameters

SI	16384
SF	250.1300055 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	11.155 ppm
F1	2790.11 Hz
F2P	-1.647 ppm
F2	-412.05 Hz
PPMCM	0.64010 ppm
HZCM	160.10803 Hz /





DA-19

Current Data Parameters
 NAME MG-2-110a-250
 EXPNO 2
 PRICNO 1

F2 - Acquisition Parameters

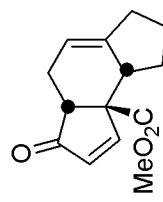
Date 20040528
 Time 17.22
 INSTRUM ar250
 PRBHD 5 mm QNP 1H
 PULPROG zgdc30
 TO 36864
 SOLVENT CDCl3
 NS 722
 DS 4
 SWH 17241.379 Hz
 FIDRES 0.467702 Hz
 AQ 1.0691060 sec
 RG 22800
 DW 29.000 use
 DE 41.43 use
 TE 300.0 K
 D12 0.00002000 sec
 DL5 23.00 dB
 CPDPRG Waitz16
 P31 103.00 use
 D1 1.0000000 sec
 P1 5.35 use
 SF01 62.9023694 MHz
 NUCLEUS 13C
 D11 0.03000000 sec

F2 - Processing parameters

SI 32768
 SF 62.8952408 MHz
 WIDM 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 250.405 ppm
 F1 15749.28 Hz
 F2P -23.724 ppm
 F2 -1492.10 Hz
 PPICM 13.70643 ppm
 H2PP 862.06891 Hz /



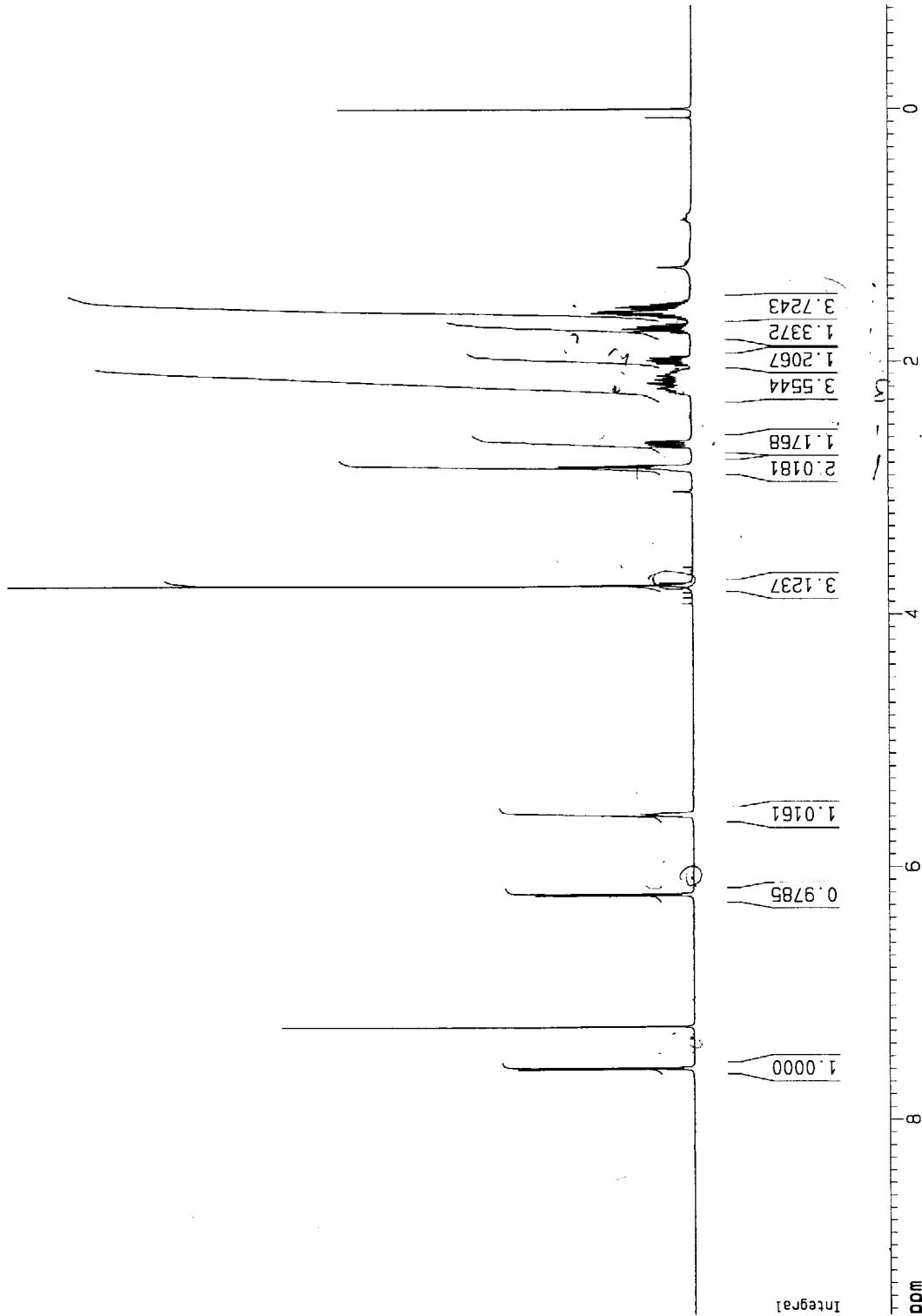
Current Data Parameters
 NAME MG-2-111a-500
 EXPNO 1
 PROCN0 1

F2 - Acquisition Parameters
 Date_ 20040606
 Time 12.51
 INSTRUM DRX500
 PROBHD 5 mm Multinucl1
 PULPROG P1
 TD 57344
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.18051 Hz
 AQ 2.7754896 sec
 RG 161.3
 DW 48.400 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.25 usec
 PL1 -3.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300109 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 30.00 cm
 F1P 9.549 ppm
 F1 4775.63 Hz
 F2P -0.820 ppm
 F2 -410.00 Hz
 PPMCM 0.51843 ppm/cr
 HZCM 259.28125 Hz/cm



Current Data Parameters
 NAME MG-2-111a-250
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

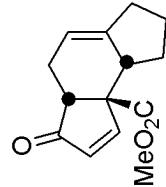
Date 20000601
 Time 13.19
 INSTRUM δ x250
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 36864
 SOLVENT CDC13
 NS 2007
 DS 4
 SWH 17241.379 Hz
 FIDRES 0.487702 Hz
 AQ 1.0691060 sec
 RG 22800
 DW 29.0000 usec
 DE 41.43 usec
 TE 300.0 K
 D12 0.00002000 sec
 DL5 23.00 dB
 CRDPRG
 P31 103.00 usec
 D1 1.00000000 sec
 P1 5.35 usec
 SF01 62.9023694 MHz
 NUCLEUS ^{13}C
 D11 0.03000000 sec

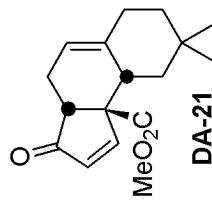
F2 - Processing parameters

SJ 32768
 SF 62.8952403 MHz
 MDW EH
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 ppm
 CY 30.00 ppm
 F1P 233.411 ppm
 F1 14660.45 Hz
 F2P 6.889 ppm
 F2 433.28 Hz
 PPMCM 11.32611 ppm
 HZCM 712.35852 Hz/





Current Data Parameters
 NAME MG-3-073a-300
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 20050124
 Time 15:56
 INSTRUM drx300
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542560 sec
 RG 228.1
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 D31 0.0000000 sec

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

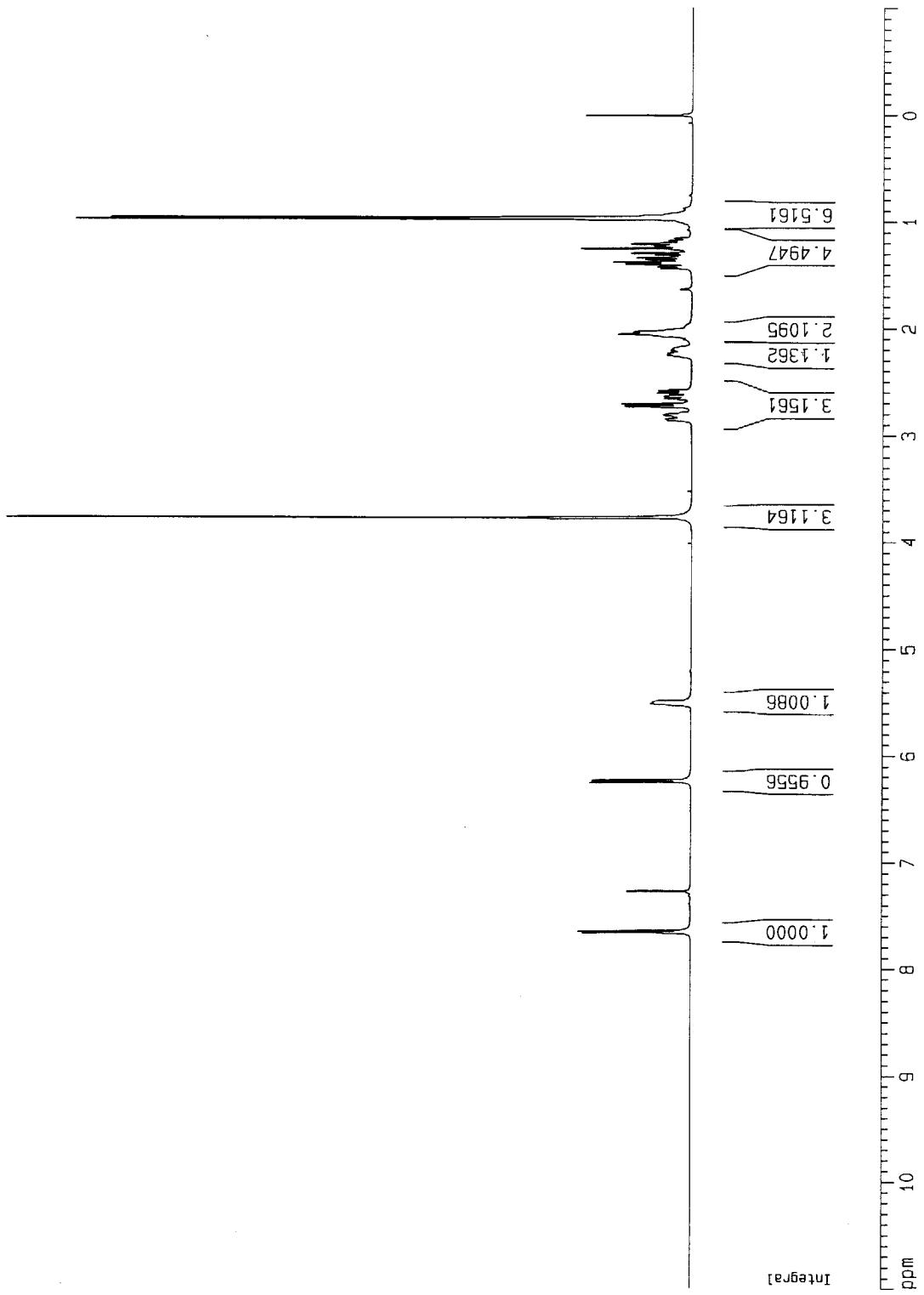
NUC1 1H
 P1 7.05 usec
 PL1 0.00 QB
 SF01 300.1318534 MHz

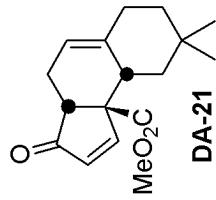
F2 - Processing parameters

SI 32768
 SF 300.1300022 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.30

1D NMR plot parameters

CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 3301.43 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.07800 Hz/cm





Current Date Parameters
 NAME MG-3-073a-300
 EXNO 2
 PROCHO 1

F2 - Acquisition Parameters

DATE 20050124
 TIME 15:58
 INSTRUM drx300
 PROBHD 5 mm Multinuc1
 PULPROG 296c30
 TD 65536
 SOLVENT CDCl3
 NS 198
 DS 4
 SWH 10032.393 Hz
 FIDRES 0.287360 Hz
 AQ 1.7400308 usec
 RG 222228
 DW 26.550 usec
 DE 6.00 usec
 TE 297.1 K
 D1 1.2099995 sec
 d11 0.0300000 sec
 d31 0.0000000 sec

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

NUC1 13C
 P1 8.50 usec
 PL1 5.00 dB
 SF01 75.4760107 MHz

===== CHANNEL f2 =====

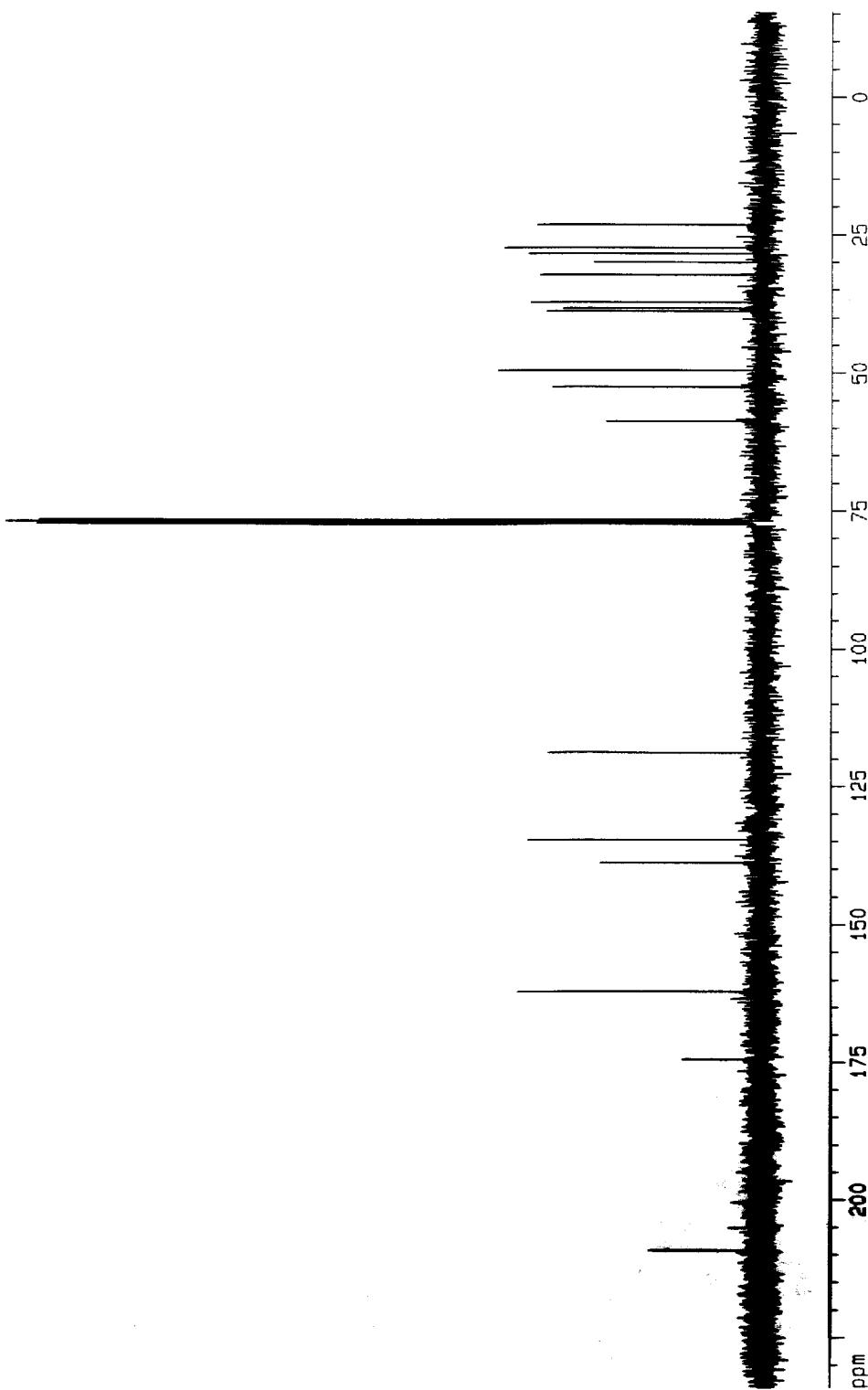
CPDPG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 25.60 dB
 SF02 300.1312005 MHz

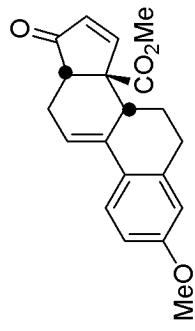
F2 - Processing parameters

SI 32768
 SF 75.4677508 MHz
 MDW 1H
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 11.00 cm
 F1P 234.220 ppm
 F1 17676.09 Hz
 F2P -15.322 ppm
 F2 -1156.30 Hz
 PPMCM 12.47711 ppm/cm
 HZCM 941.61957 Hz/cm





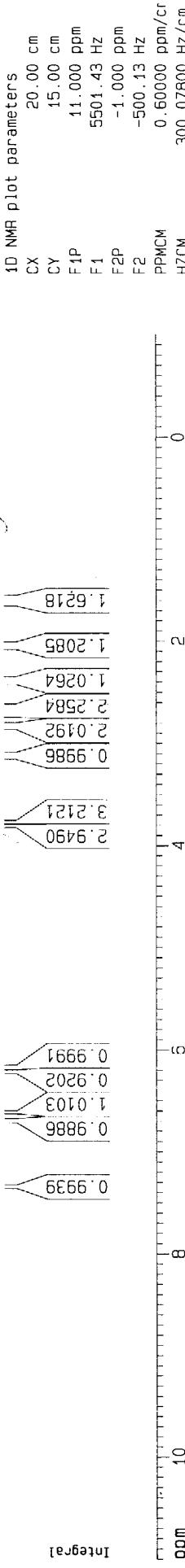
DA-22

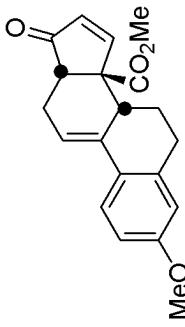
Current Data Parameters
 NAME MG-3-061a-500
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20041102
 Time 20.39
 INSTRUM DRX500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 57344
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 F1ORES 0.180151 Hz
 AQ 2.7754996 sec
 RG 101.6
 DW 40.400 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.25 usec
 PL1 -3.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.13300125 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.40





DA-22

Current Data Parameters
 NAME MG-3-050a-300
 EXPTNO 2
 PROCNO 1

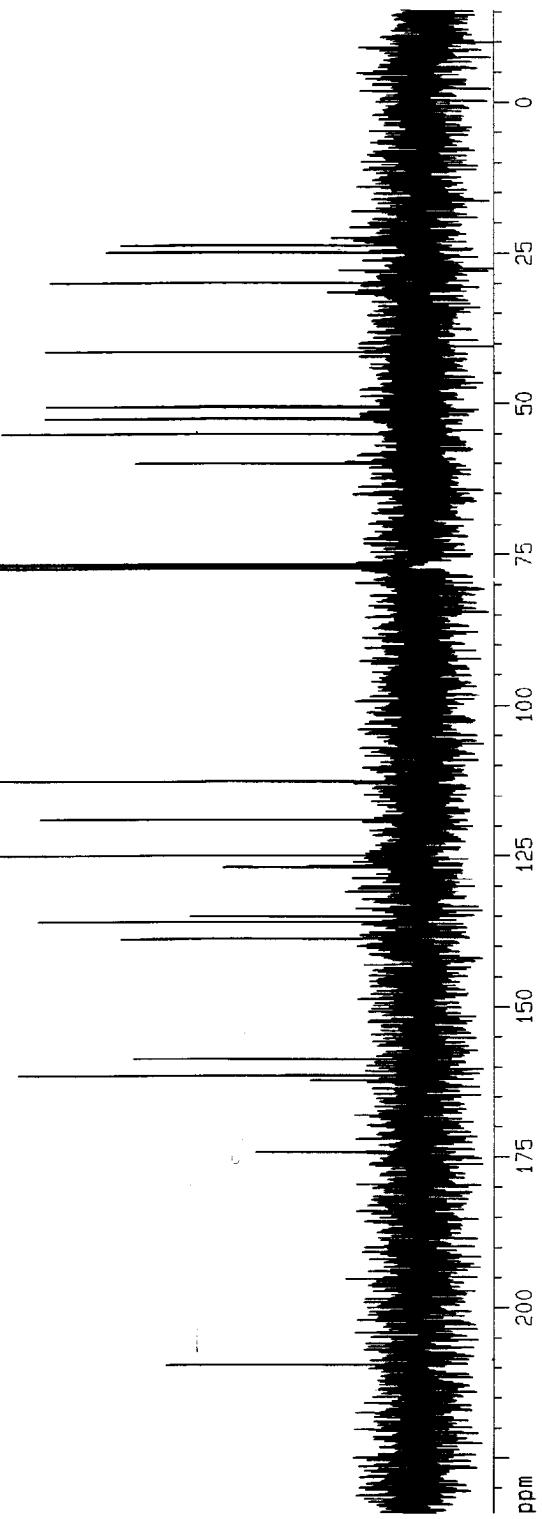
F2 - Acquisition Parameters
 Date 20041101
 Time 22:58
 INSTRUM drx300
 PROBOD 5 mm Multinuc1
 PROBPROG zgdc30
 TO 65536
 SOLVENT CDCl3
 NS 78
 DS 4
 SWH 18832.393 Hz
 FIDRES 0.287360 Hz
 AQ 1.7400308 sec
 RG 22528
 DW 26.550 usec
 DE 6.00 usec
 TE 297.1 K
 D1 1.2999995 sec
 D11 0.0300000 sec
 D31 0.0000000 sec

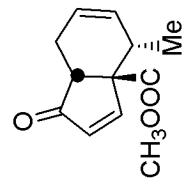
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.50 usec
 PL1 5.00 dB
 SF01 75.4760107 MHz

===== CHANNEL f2 =====
 CPDRG2 waltz16
 NUC2 1H
 P0P02 100.00 usec
 PL2 120.00 dB
 PL12 25.60 dB
 SFQ2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677514 MHz
 MDW EH
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 20.00 cm
 F1P 234.213 ppm
 F1 17675.51 Hz
 F2P -15.329 ppm
 F2 -1156.88 Hz
 PPMCM 12.4771 ppm/cm
 HZCM 941.6957 Hz/cm

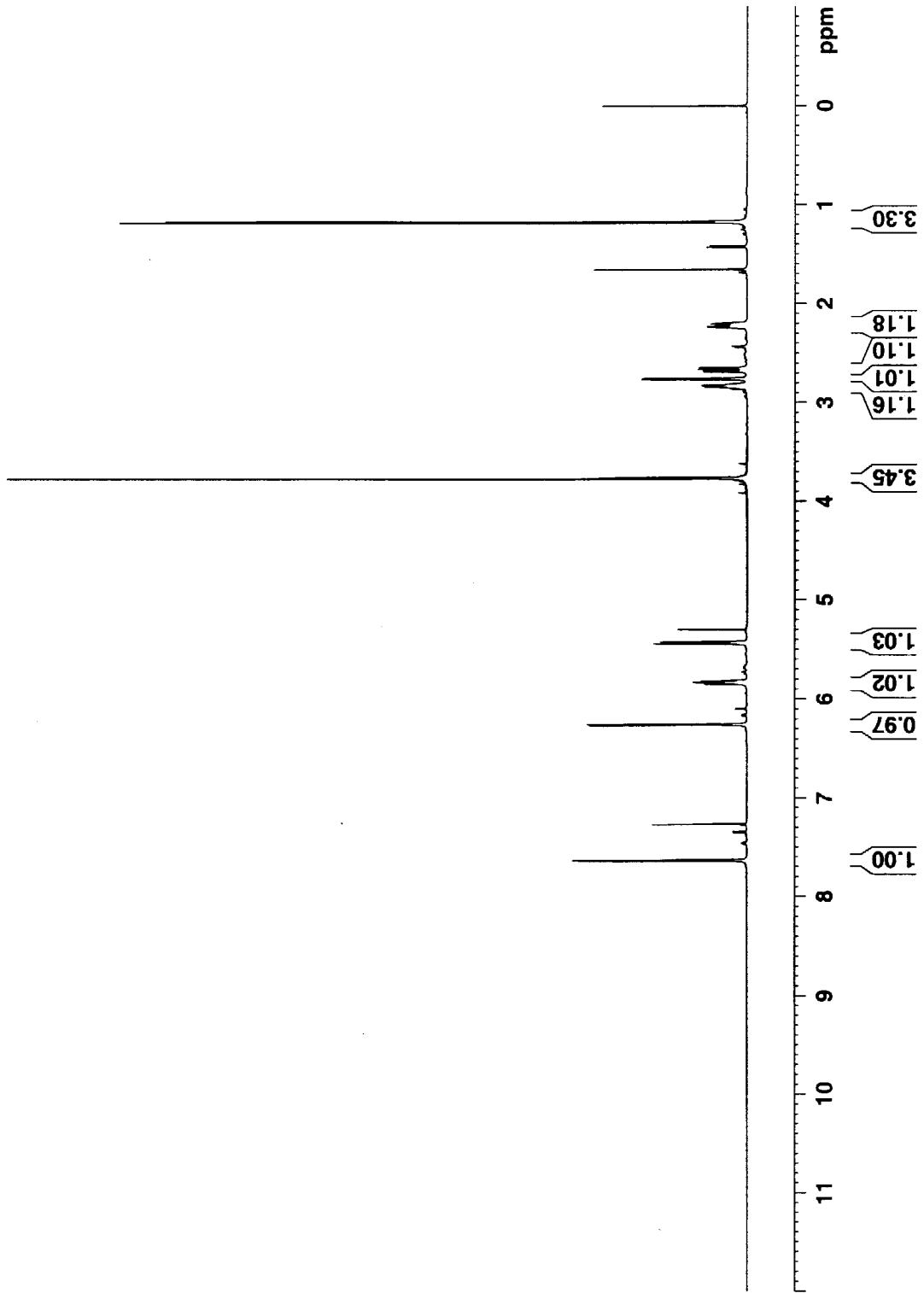


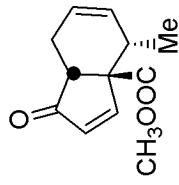


Current Data Parameters
 NAME MG-V-84a-A3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20060210
 Time 22:43
 INSTRUM DRX500
 PROPHD 5 mm Multinucl
 PULPROG zg30pad
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.171923 sec
 RG 1.14
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 D31 0.0000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 0.00 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300071 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.40

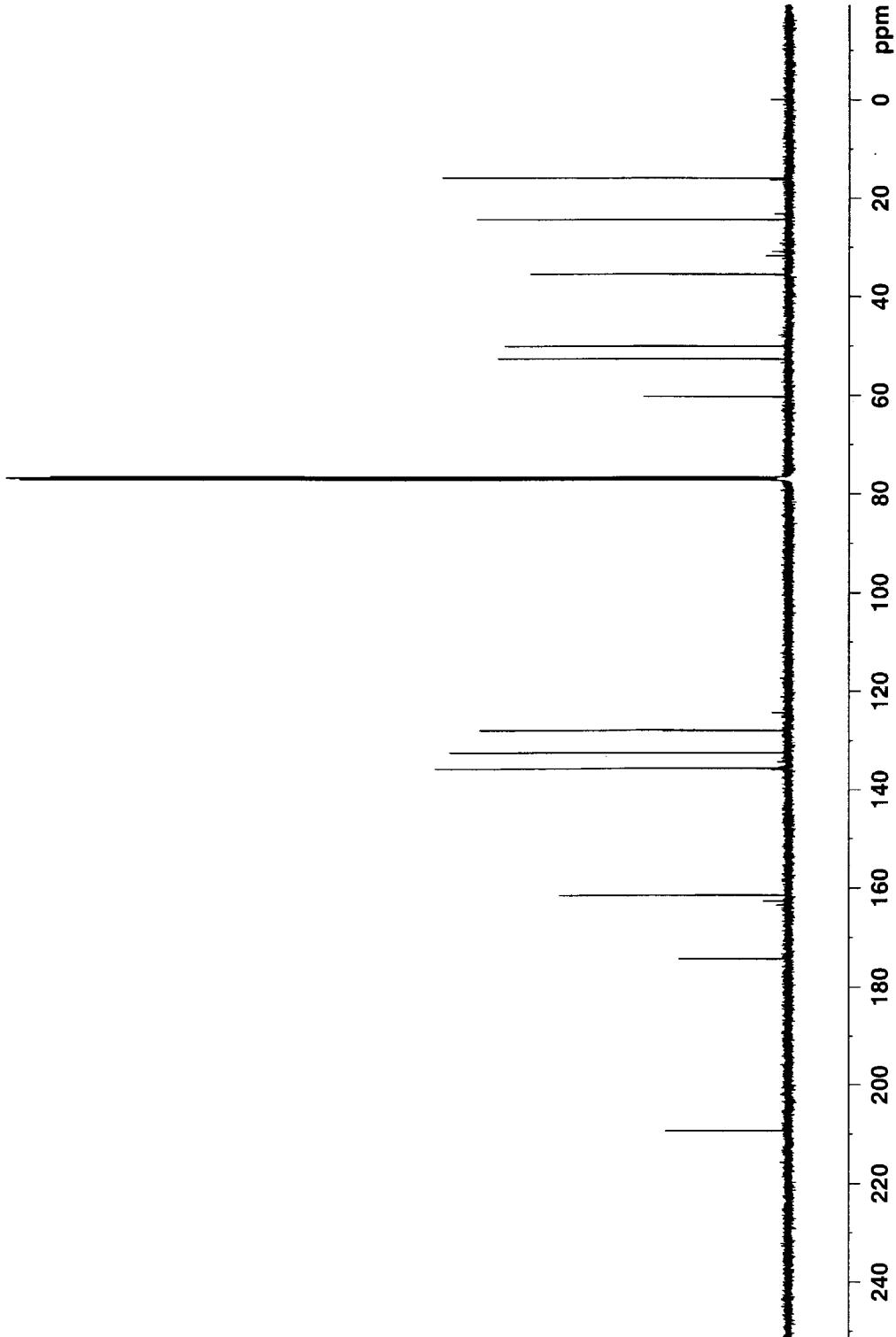


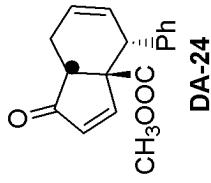


Current Data Parameters
 NAME MG-V-84a-A3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20060210
 Time 23:07
 INSTRUM DRX500
 PROBHD 5 mm Multinucl
 PULPROG zgdc30
 TD 65536
 SOLVENT CPC13
 NS 428
 DS 4
 SWH 34013.605 Hz
 FIDRES 0.519006 Hz
 AQ 0.9634292 sec
 RG 32768
 DW 14.700 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d1 0.0300000 sec
 D31 0.0000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.10 usec
 PL1 3.00 dB
 SFO1 125.7733786 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 88.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 SFO2 500.1320005 MHz
 F2 - Processing parameters
 SI 32768
 SF 125.7577938 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





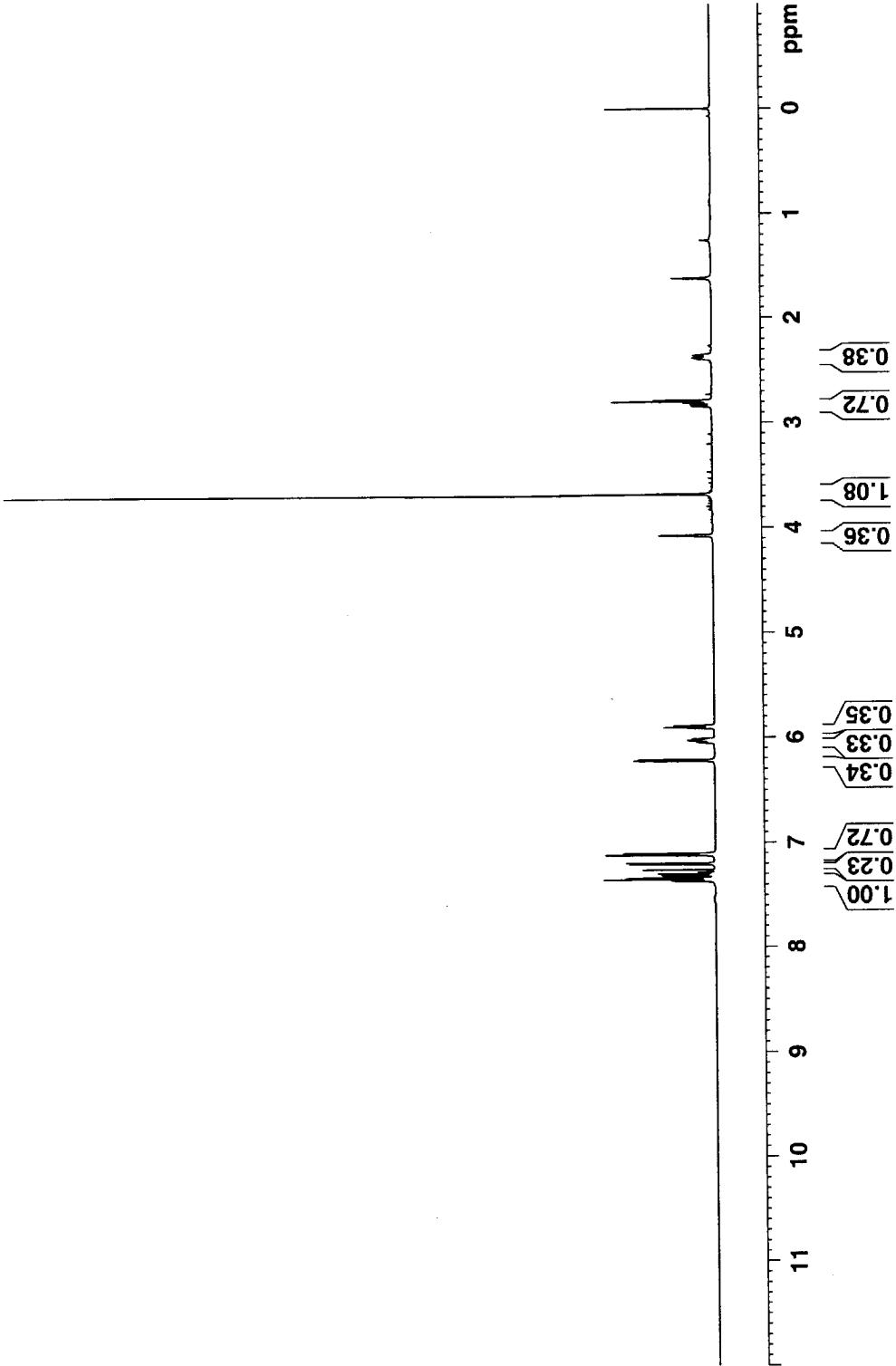
Current Data Parameters
 NAME MG-IV-77a-A3
 EXPNO 1
 PROCNO

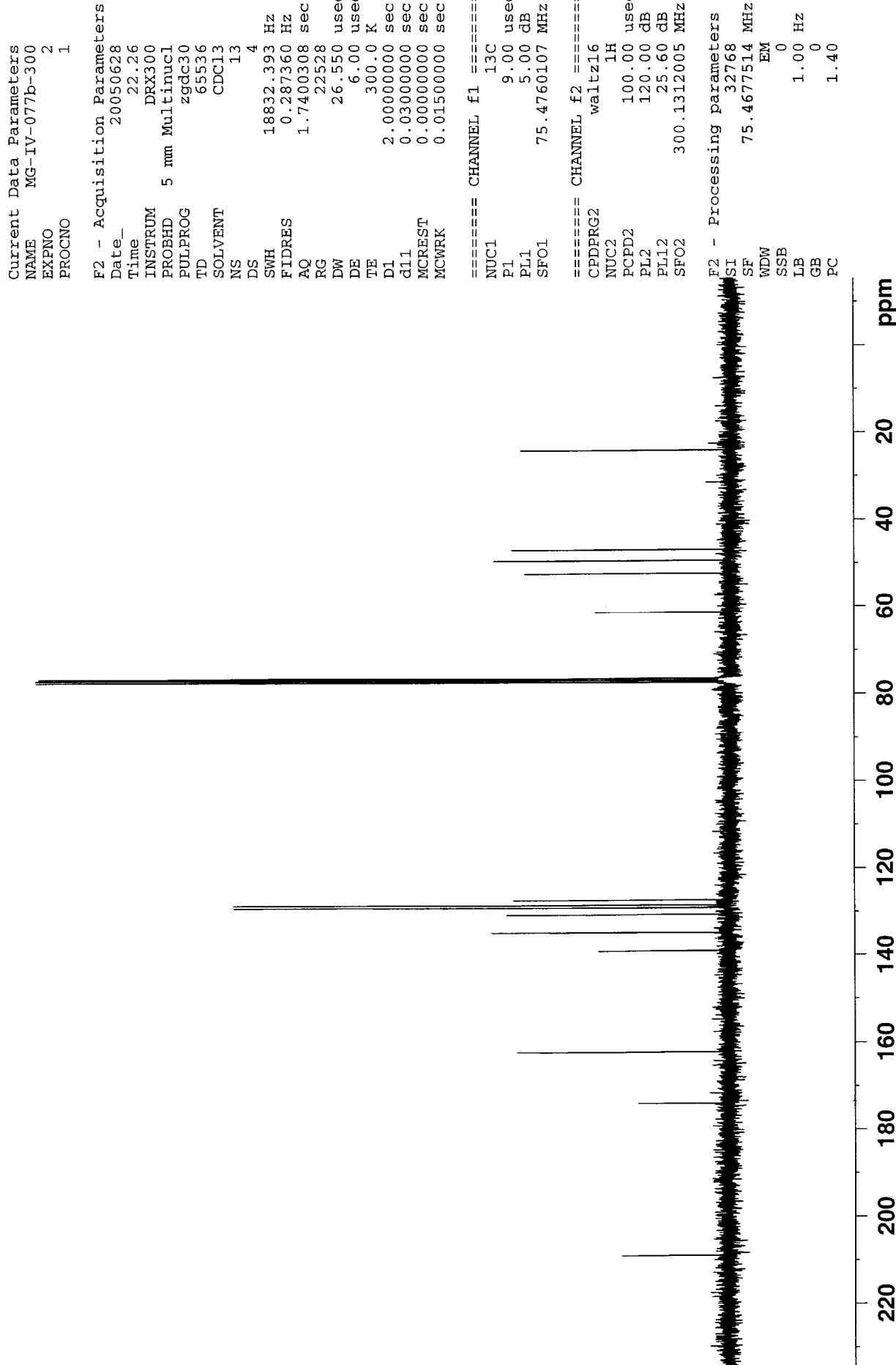
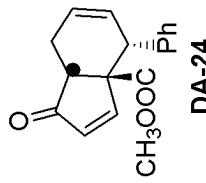
F2 - Acquisition Parameters
 Date 20060208
 Time 14.38
 INSTRUM DRX500
 PROBHD 5 mm MultiInuc1
 PULPROG zg30pad
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.171923 sec
 RG 128
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 D31 0.0000000 sec

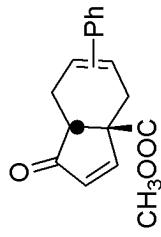
===== CHANNEL f1 =====

NUC1 1H
 P1 11.50 usec
 PLL 0.00 dB
 SFO1 500.13330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300115 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.40





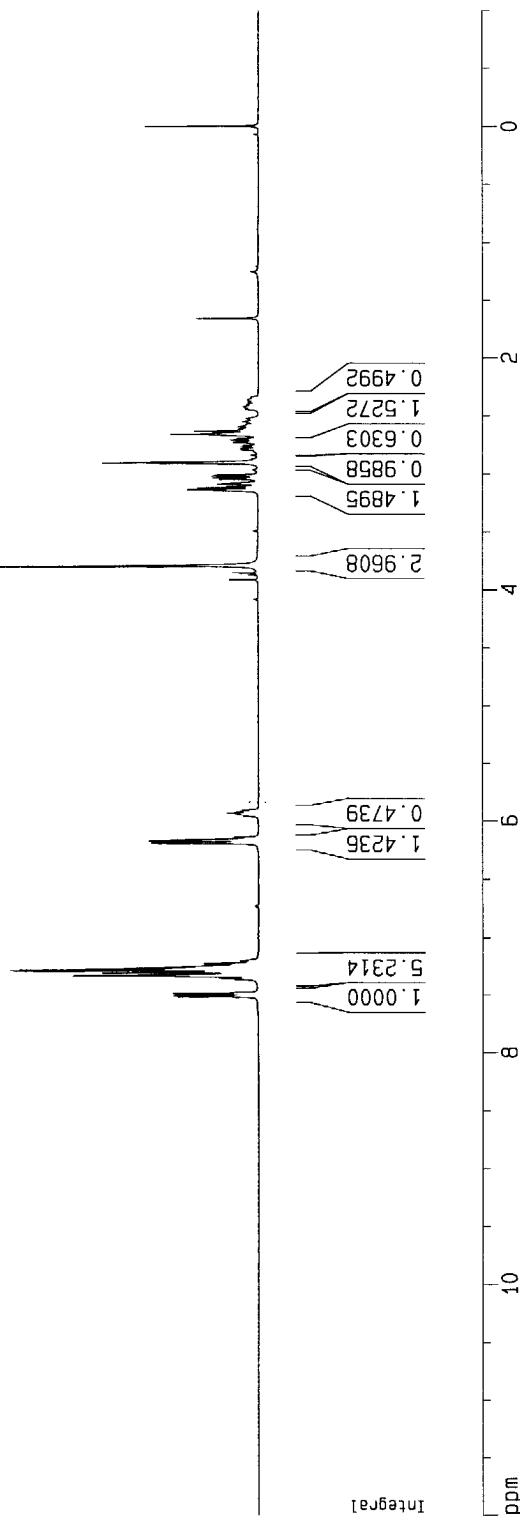


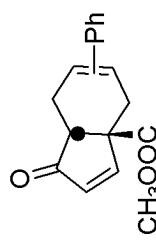
DA-25

Current Data Parameters
 NAME MG-4-062a-250
 EXPNO 1
 PROCN0

F2 - Acquisition Parameters
 Date_ 20050525
 Time 23.48
 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 1024
 DW 96.000 use
 DE 137.14 use
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.70 use
 SF01 250.1315321 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 250.1300075 MHz
 MDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.50





DA-25

Current Data Parameters
 NAME MG-4-082a-250
 EXPNO 2
 PROCN 1

F2 - Acquisition Parameters

Date 20050525
 Time 23.52
 INSTRUM ar250
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 36864
 SOLVENT CDCl3
 NS 536
 DS 4
 SWH 17241.379 Hz
 FIDRES 0.467702 Hz
 AQ 1.0691060 sec
 RG 22800
 DW 29.000 use
 DE 41.43 use
 TE 300.0 K
 D12 0.0000200 sec
 DL5 23.00 dB
 CPDPRG waltz16
 P31 103.00 use
 D1 2.0000000 sec
 P1 6.00 use
 SF01 62.9023694 MHz
 NUCLEUS 13C
 D11 0.03000000 sec

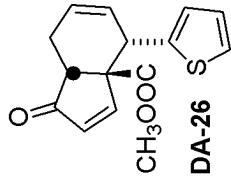
F2 - Processing parameters

SI 32768
 SF 62.8952434 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 RC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 20.00 cm
 F1P 250.363 ppm
 F1 15746.65 Hz
 F2P -23.765 ppm
 F2 -1494.73 Hz
 PPMW 13.70643 ppm
 HZCM 862.06897 Hz/



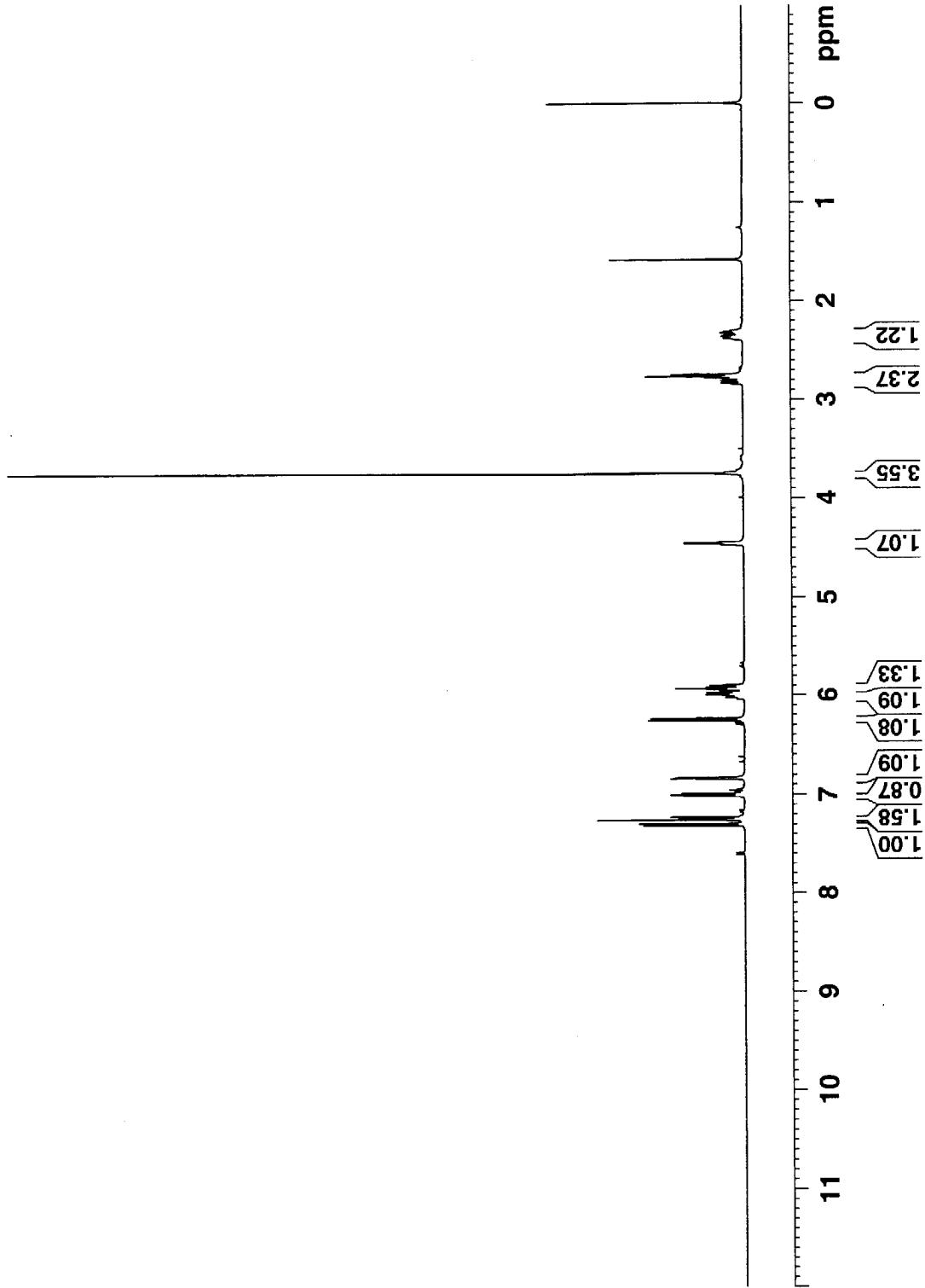


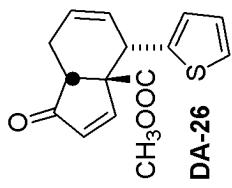
Current Data Parameters
 NAME MG-IV-142ba-A2
 EXPNO 1
 PROCN0

F2 - Acquisition Parameters
 Date_ 20050908
 Time 10.58
 INSTRUM DRX300
 PROBHD 5 mm Multinucl
 PULPROG zg30pdd
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 645.1
 DW 81.000 use
 DE 6.00 use
 TB 300.0 K
 D1 1.0000000 sec
 D31 0.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.05 use
 PL1 0.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300054 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.30





Current Data Parameters
 NAME MG-IV-142b-A2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date 20050907
 Time 12.11
 INSTRUM DRX300
 PROBHD 5 mm Multinucl
 PULPROG zgdd30pad
 TD 65536
 SOLVENT CDCl3
 NS 561
 DS 4
 SWH 18832.393 Hz
 FIDRES 0.287360 Hz
 AQ 1.7400308 sec
 RG 22538
 DW 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 D31 0.0000000 sec

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

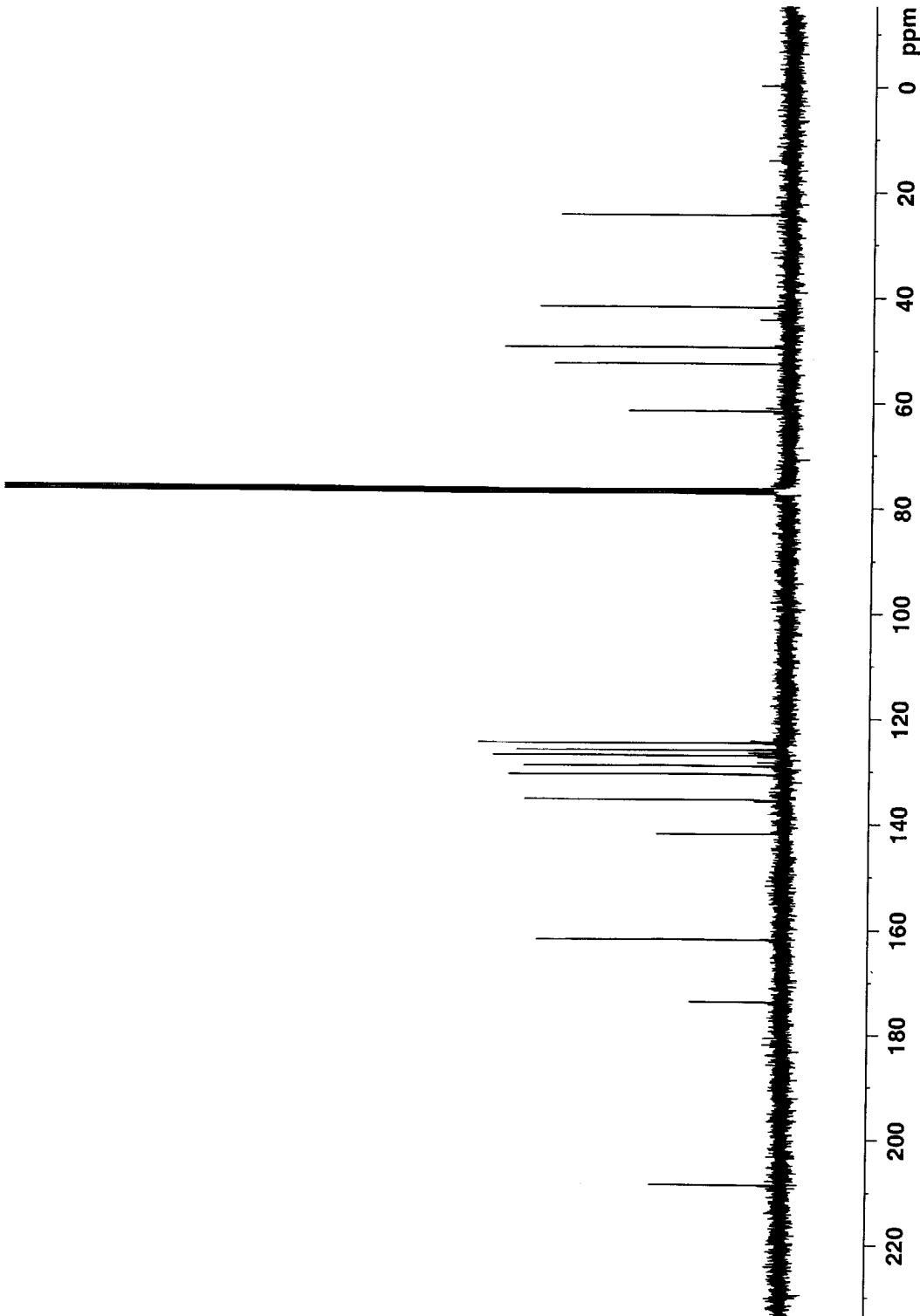
NUC1 13C
 P1 9.00 usec
 PL1 5.00 dB
 SFO1 75.4760107 MHz

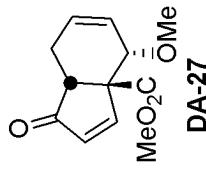
===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 25.60 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768
 SF 75.4677508 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
 NAME MG-3-05a-500
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 20040902
 Time 22.56
 INSTRUM DRX500
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TD 57344
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.180151 Hz
 AQ 2.7754996 sec
 RG 128
 DW 48.400 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.0000000 sec

==== CHANNEL f1 =====

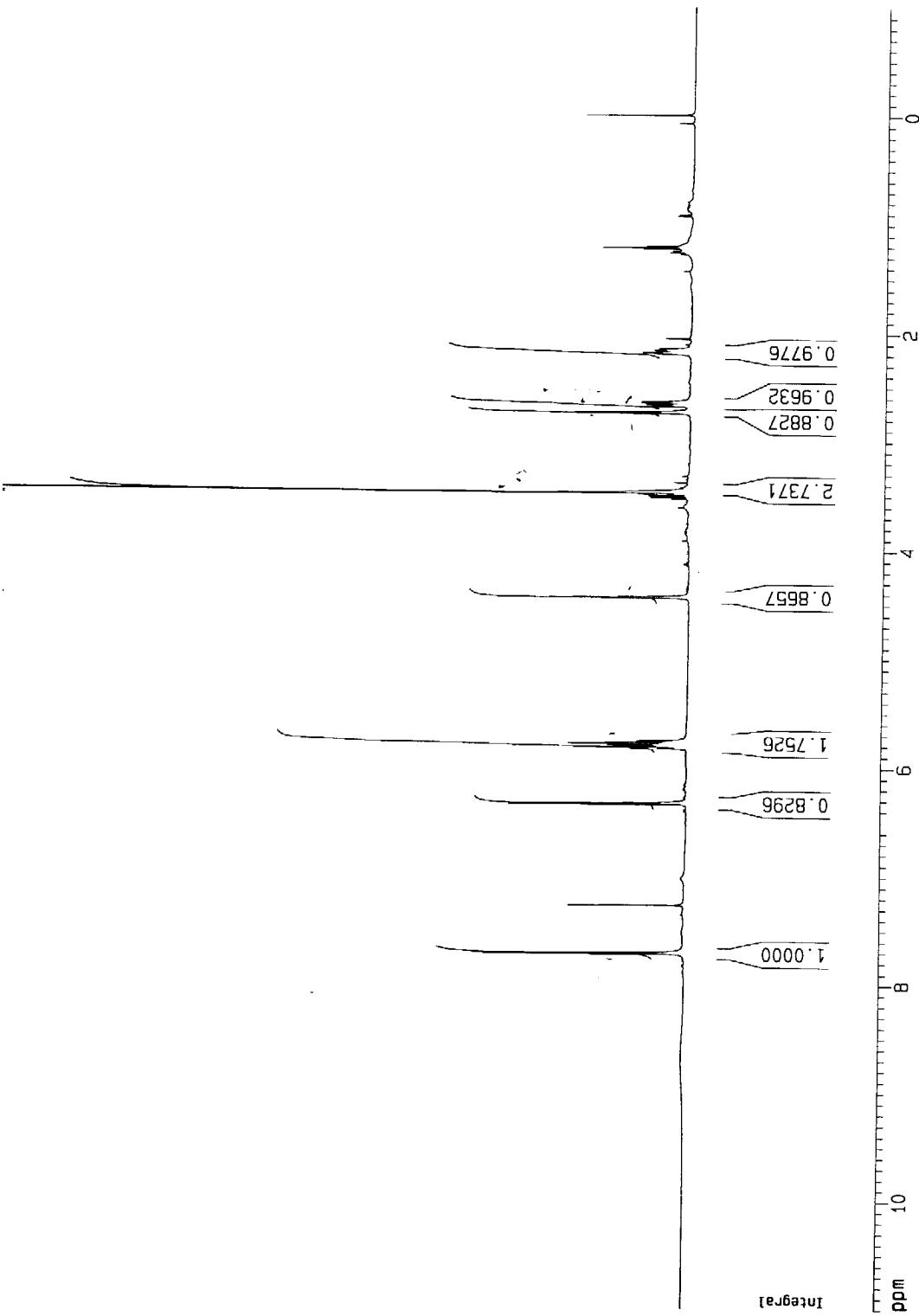
NUC1 1H
 P1 13.25 usec
 PL1 -3.00 dB
 SF01 500.13300885 MHz

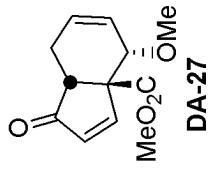
F2 - Processing parameters

SI 32768
 SF 500.13000081 MHz
 MDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 20.00 cm
 F1P 11.000 ppm
 F1 5601.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cr
 HZCM 300.07800 Hz/cm





Current Date Parameters
 NAME: MG-3-025-300
 EXPNO: 2
 PROCNO: 1

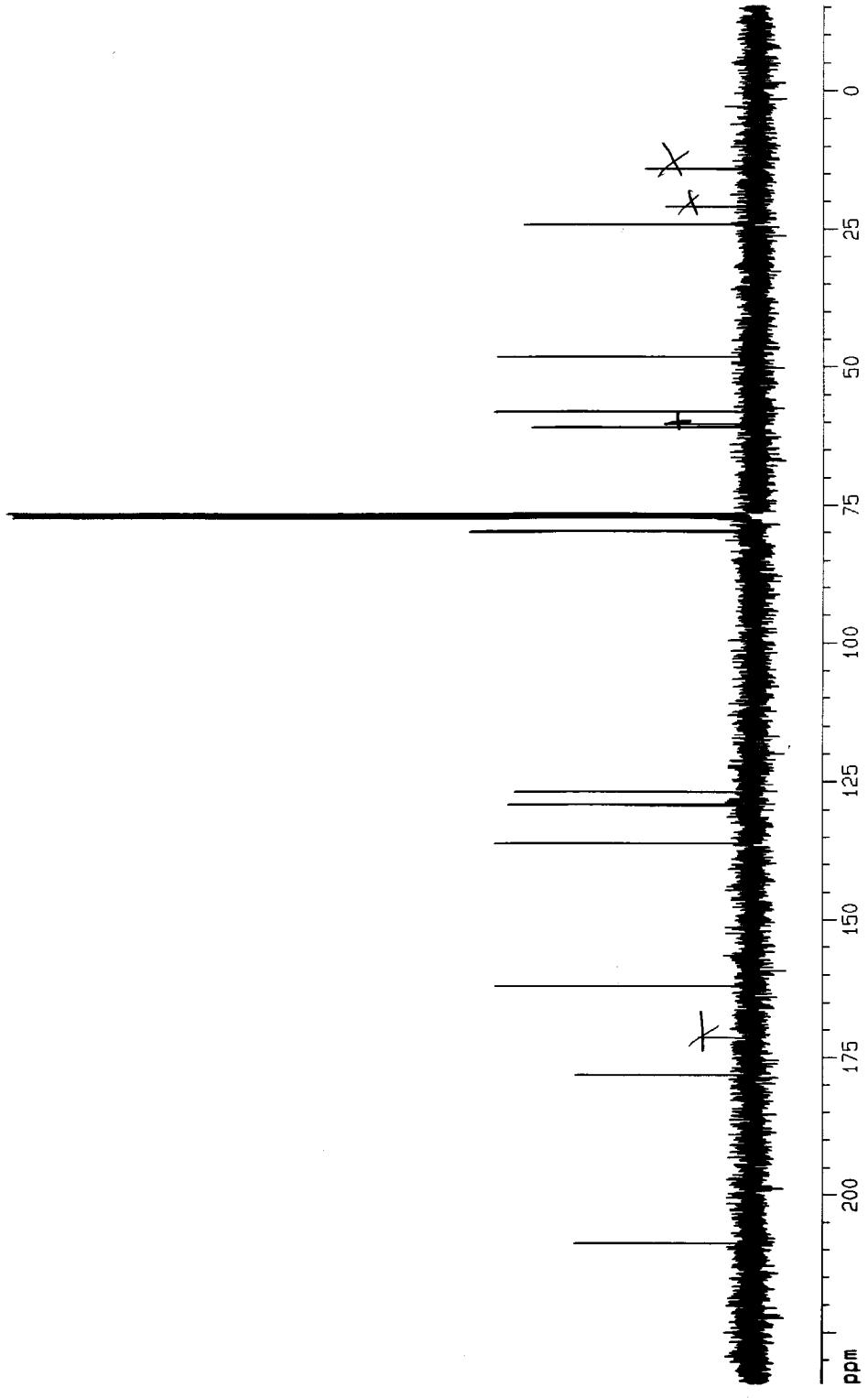
F2 - Acquisition Parameters
 Date- 20040502
 Time 13:20
 INSTRUM drx300
 PROBHD 5 mm Multinuc1
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 121
 DS 4
 SWH 18832.393 Hz
 FIDRES 0.287350 Hz
 AQ 1.740308 sec
 RG 22528
 DN 26.550 usec
 DE 6.00 usec
 TE 297.1 K
 D1 1.2999995 sec
 D11 0.0300000 sec
 D31 0.0000000 sec

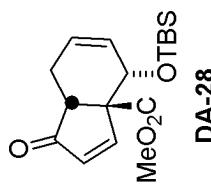
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.50 usec
 PL1 5.00 dB
 SF01 75.4760107 MHz

===== CHANNEL f2 =====
 CPDPFG2 w1t216
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 25.60 dB
 SFQ2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677520 MHz
 NDN EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 11.00 cm
 F1P 234.205 ppm
 F1 17674.94 Hz
 F2P -175.337 ppm
 F2 -1157.45 Hz
 PRPCM 12.47711 ppm/cm
 HZCM 941.61957 Hz/cm





DA-28

Current Data Parameters
NAME MG-4-024a-300
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date -	2005/04/06
Time	12.38
INSTRUM	drx300
PROBHD	5 mm Multinuc 1
PROBPG	zq30
TD	32768
SOLVENT	CDC13
NS	16
DS	2
SWH	61.72-8300 Hz
FIDRES	0.188380 Hz
AQ	2.0545580 sec

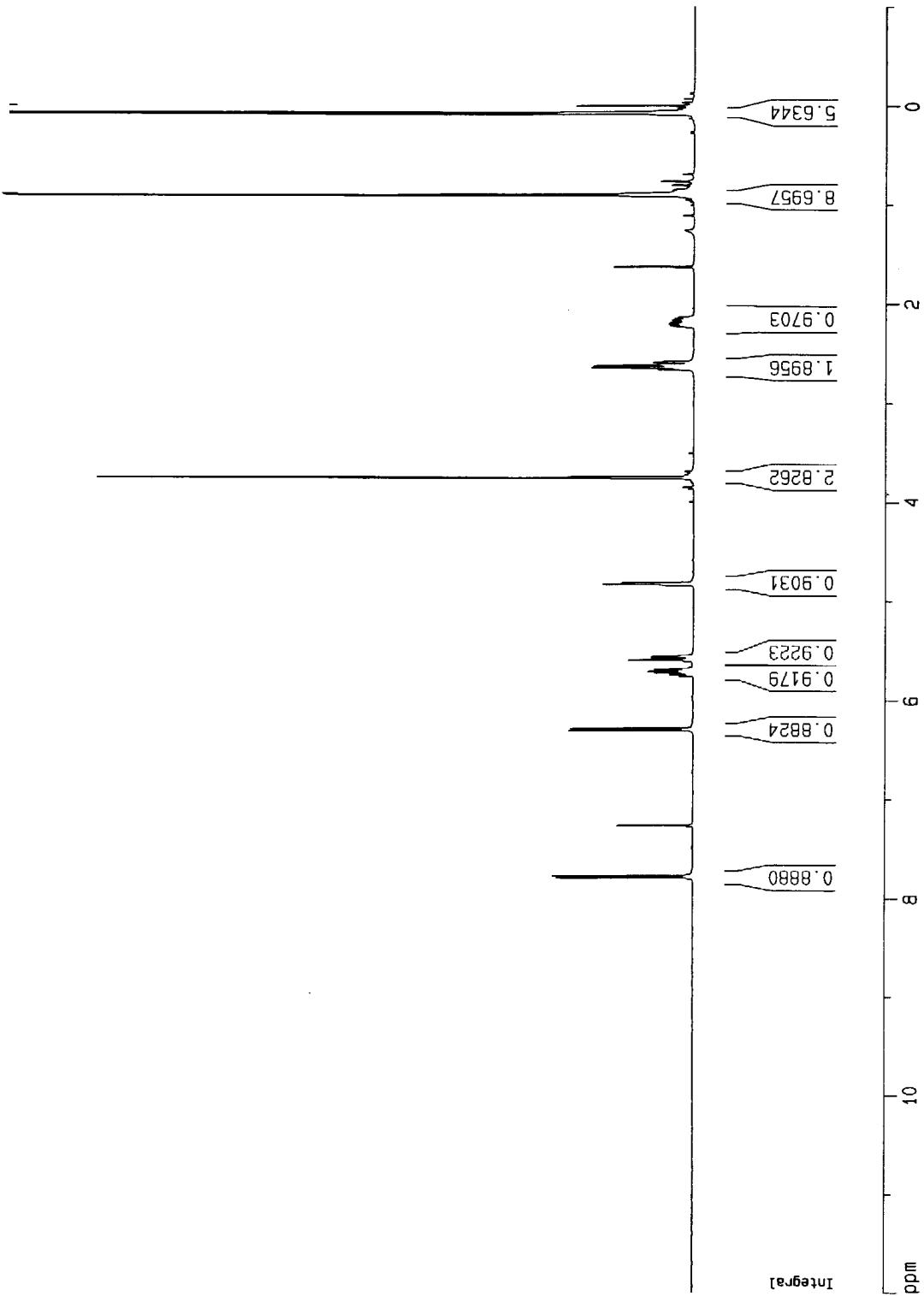
RG	203.2
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec
D31	0.0000000 sec

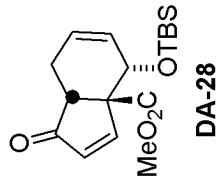
===== CHANNEL f1 =====

NUC1	1H
P1	7.05 usec
PL1	0.00 dB
SF01	300.1318534 MHz

SI	32768
SF	300.1300037
MDW	MHz
SSB	EM
LB	0
LB	0.30
GB	0
PC	1.30

1D NMR plot parameters	
CX	20.00 cm
CY	40.00 cm
F1P	12,000 ppm
F1	3601.56 Hz
F2P	-1,000 ppm
F2	-300.13 Hz
PPCM	0.6500 ppm/Hz
H7CM	195.08450 Hz/cm





Current Data Parameters

NAME	MG-4-024a-500
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20050407
Time	20:41
INSTRUM	DRX500
PROBHD	5 mm Multinuc1
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	495
DS	4
SWH	39681.812 Hz
EDDRES	0.60596 Hz
AQ	0.8258188 sec
RG	16384
DW	12.600 usec
DE	6.00 usec
TE	298.0 K
D1	2.0000000 sec
d11	0.03000000 sec

***** CHANNEL f1 *****

NUC1	¹³ C
P1	8.10 usec
PL1	3.00 dB
SF01	125.7713108 MHz

***** CHANNEL f2 *****

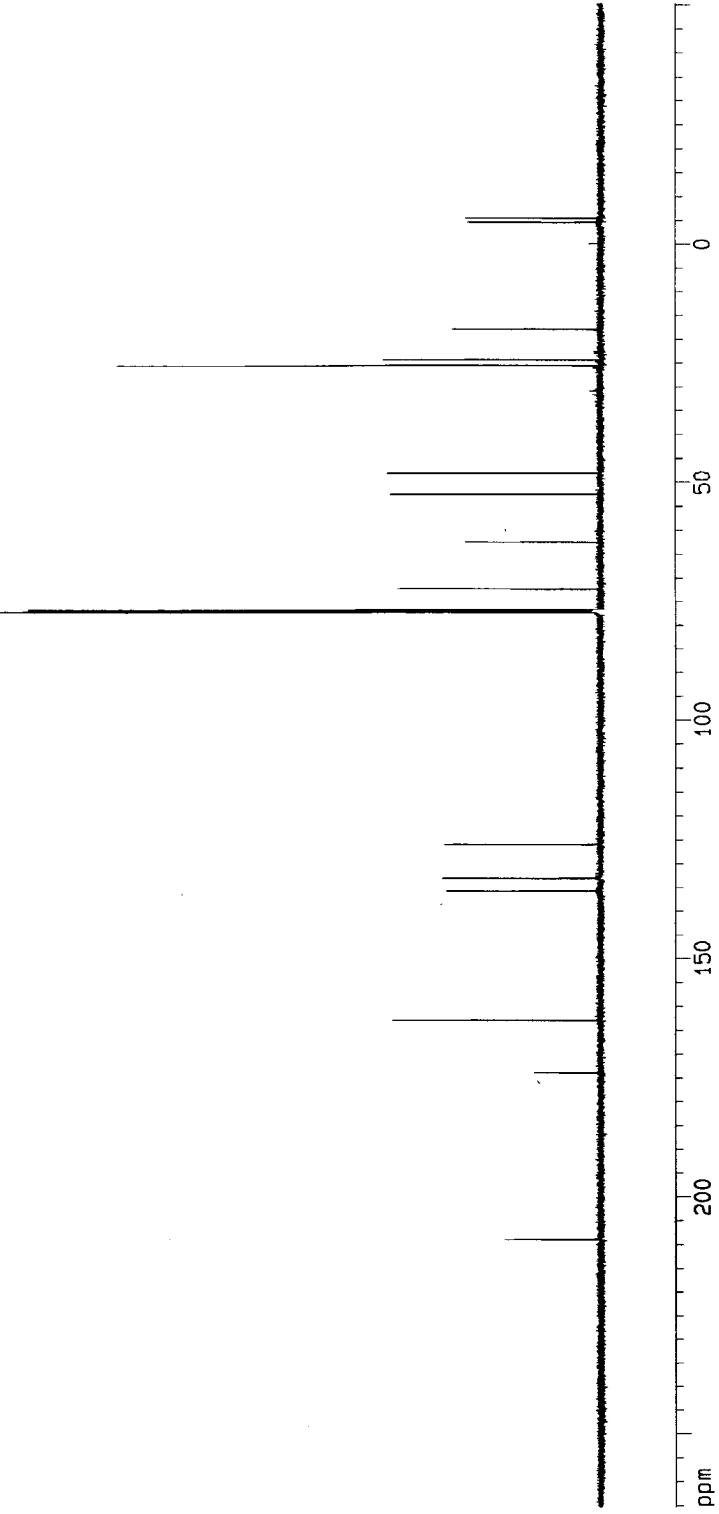
CPDPG2	Waltz16
NUC2	¹ H
PCPD2	88.00 usec
PL2	0.00 dB
PL12	21.00 dB
SF02	500.1320005 MHz

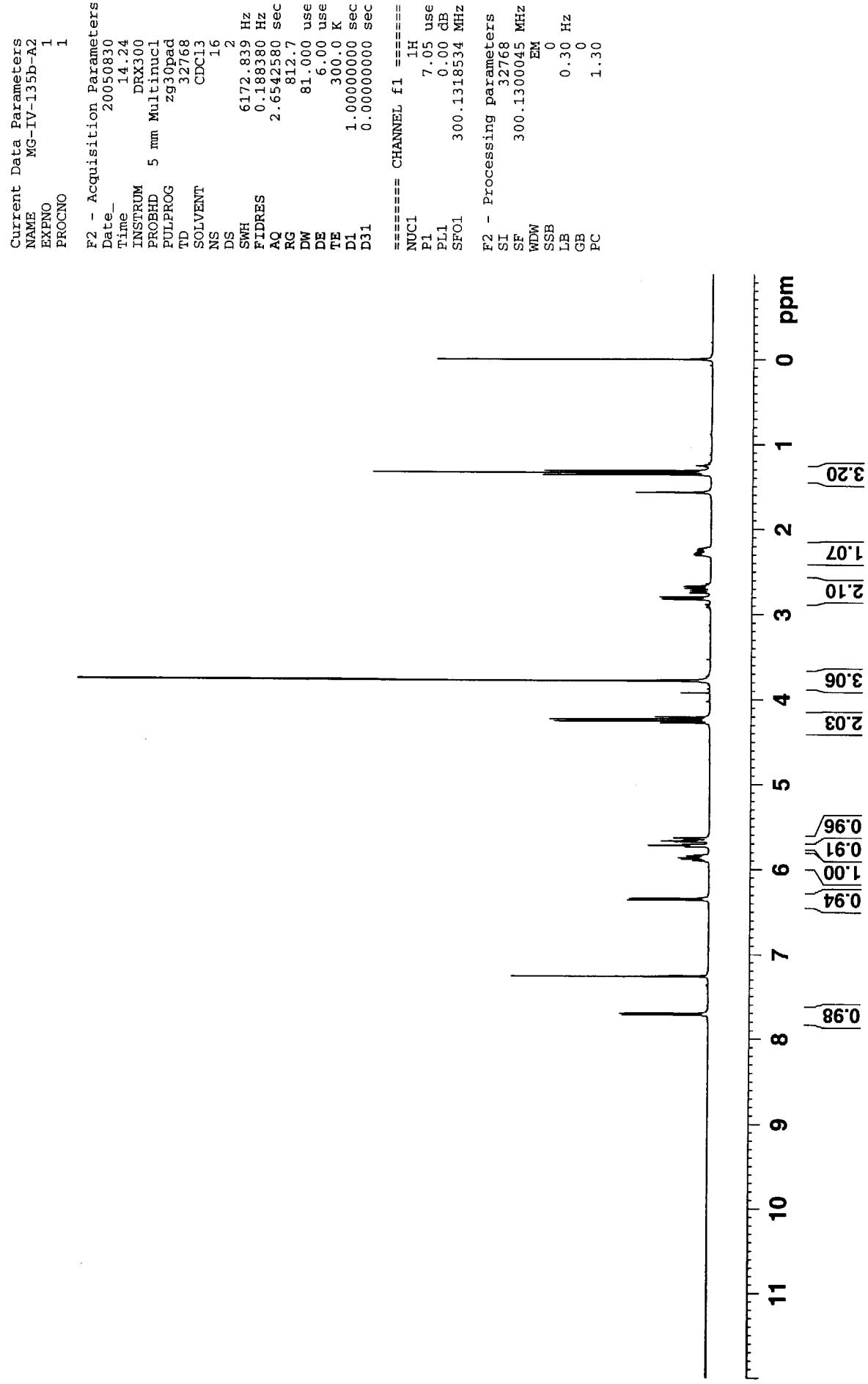
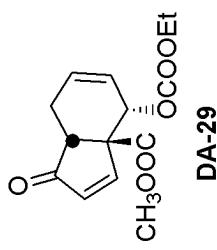
F2 - Processing parameters

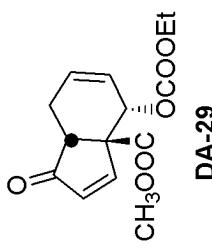
SI	32768
SF	125.7577335 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR print parameters

CX	20.00 cm
CY	8.00 cm
FI	265.258 ppm
F1	33358.26 Hz
F2P	-50.284 ppm
F2	-6323.55 Hz
PPMCH	15.7708 ppm/cm
HZCM	1984.09045 Hz/cm







Current Data Parameters

NAME	MG-IV-135b-A2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Time	20050830
INSTRUM	DRX300
PROBHD	5 mm Multinucl
PULPROG	2gdc30pad
TD	65536
SOVNT	CDCl3
NS	923
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	2.0000000 sec
D11	0.03000000 sec
D31	0.0000000 sec

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

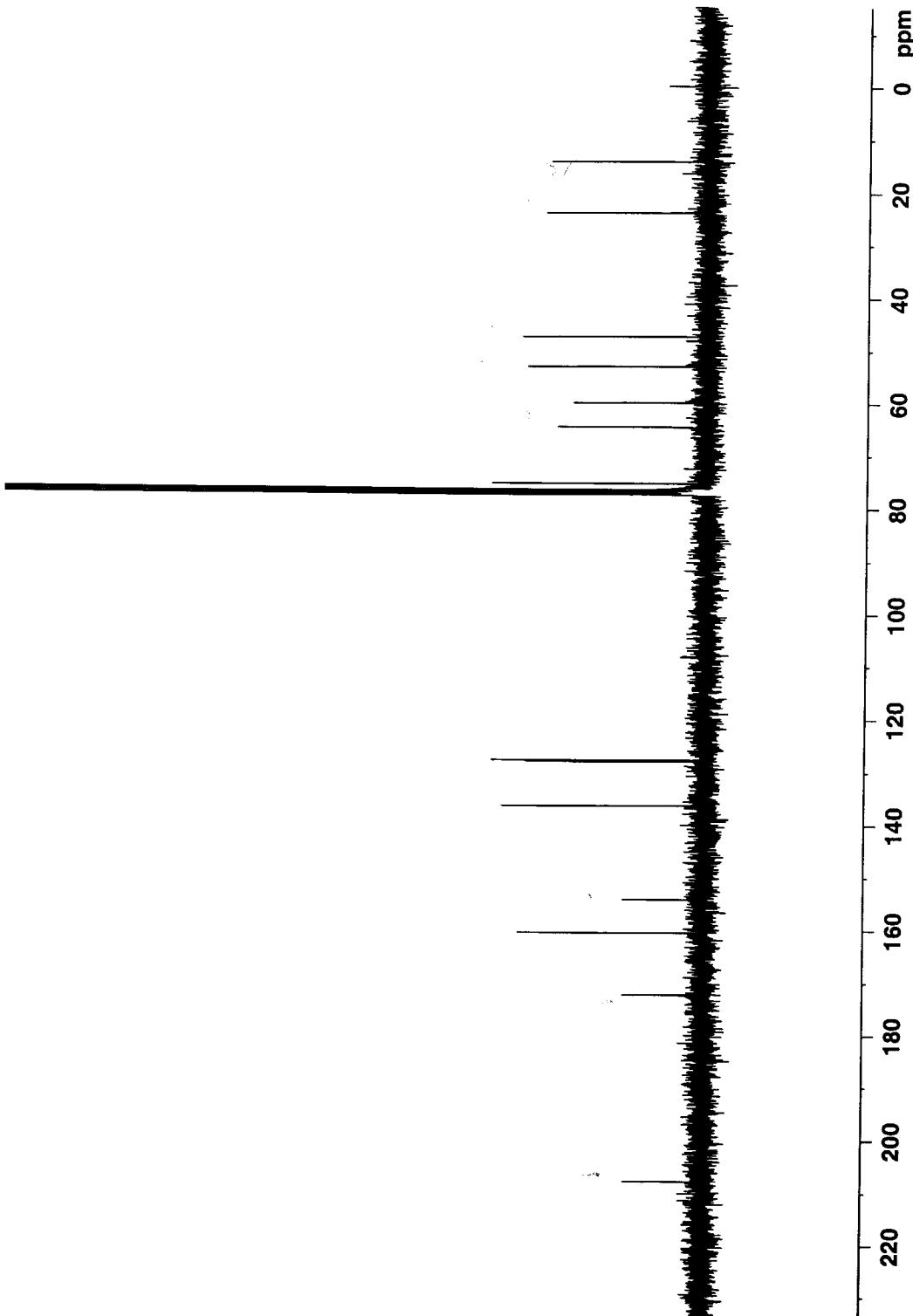
NUC1	13C
P1	9.00 usec
PL1	5.00 dB
SFO1	75.4760107 MHz

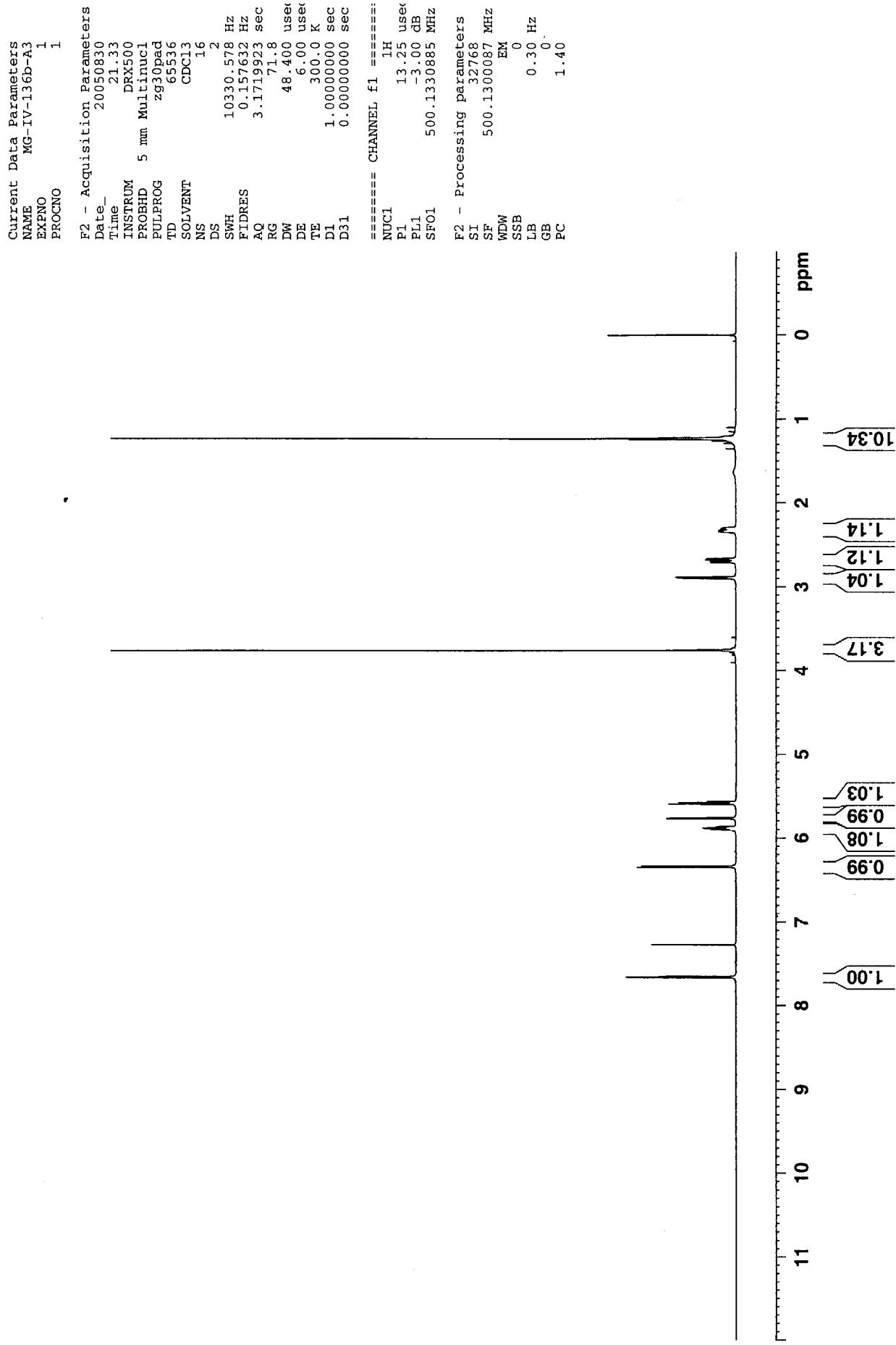
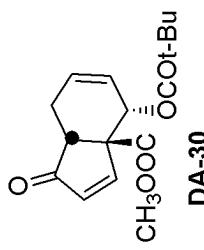
===== CHANNEL f2 =====

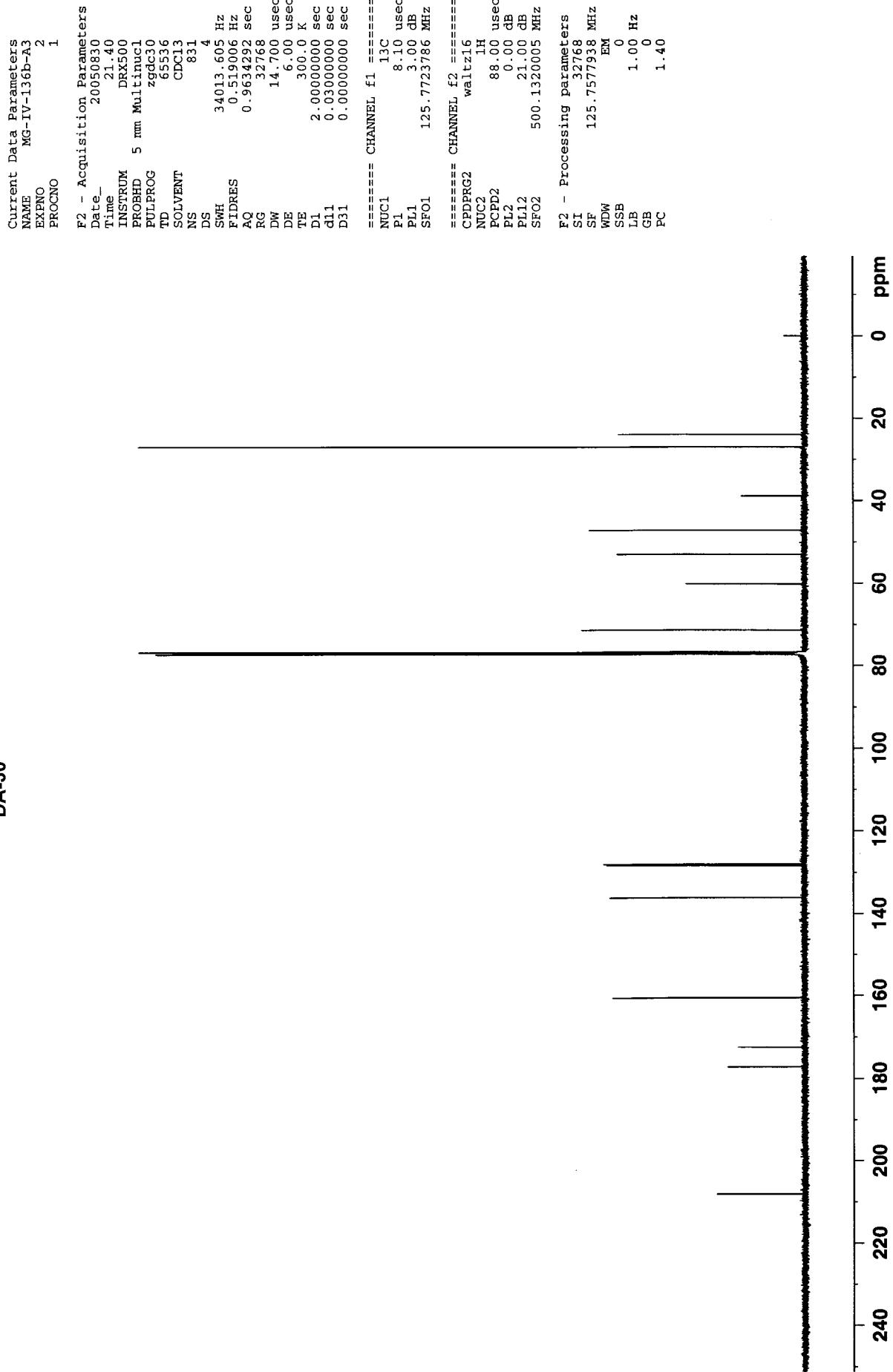
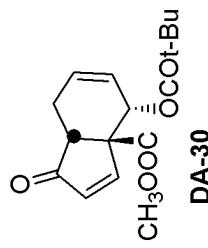
CPDPG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	25.60 dB
SFO2	300.1312005 MHz

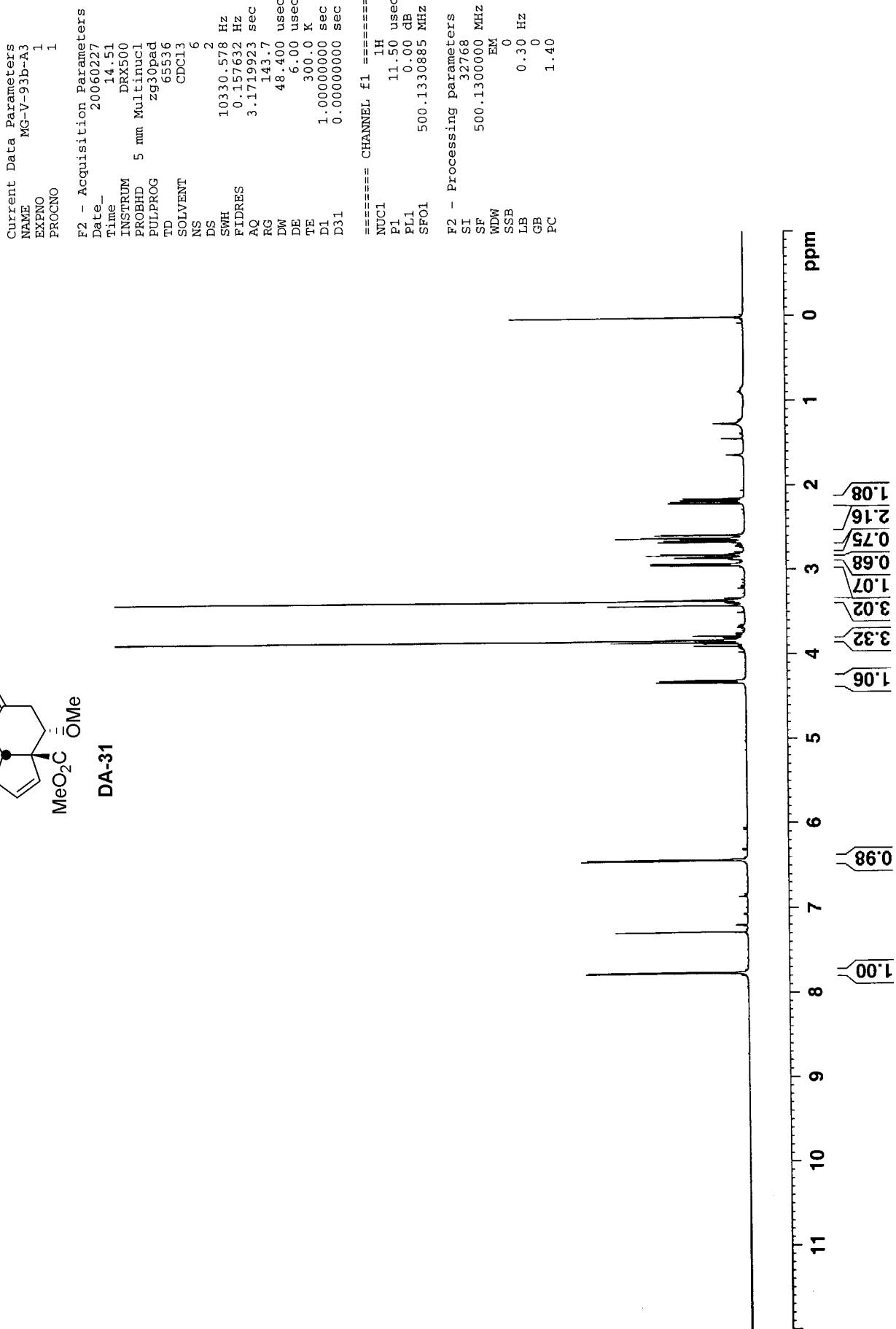
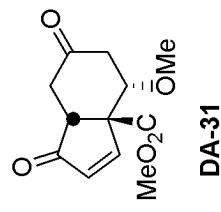
F2 - Processing parameters

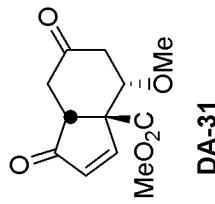
SI	32768
SF	75.4677497 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40











Current Data Parameters
 NAME MG-V-93b-A3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20060227
 Time 14.53
 INSTRUM DRX500
 PROBHD Multirruc1
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 669
 DS 4
 SWH 34013.605 Hz
 FIDRES 0.519006 Hz
 AQ 0.9634292 sec
 RG 32768
 DW 14.700 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.000000 sec
 d11 0.0300000 sec
 D31 0.0000000 sec

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

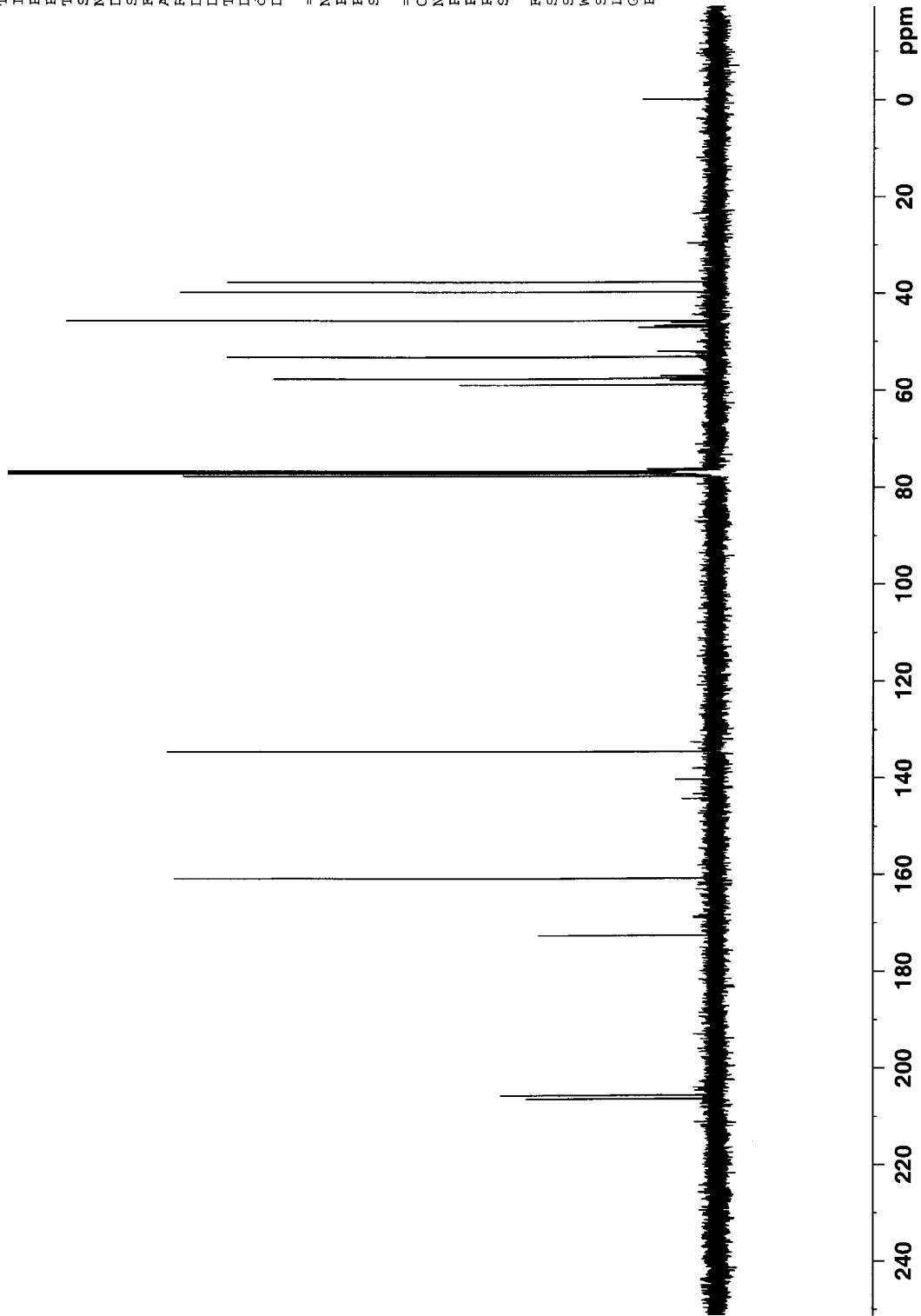
NUC1 13C
 P1 8.10 usec
 PL1 3.00 dB
 SFO1 125.7723786 MHz

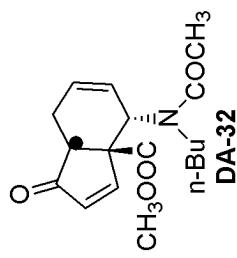
===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 88.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SP 125.7577938 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

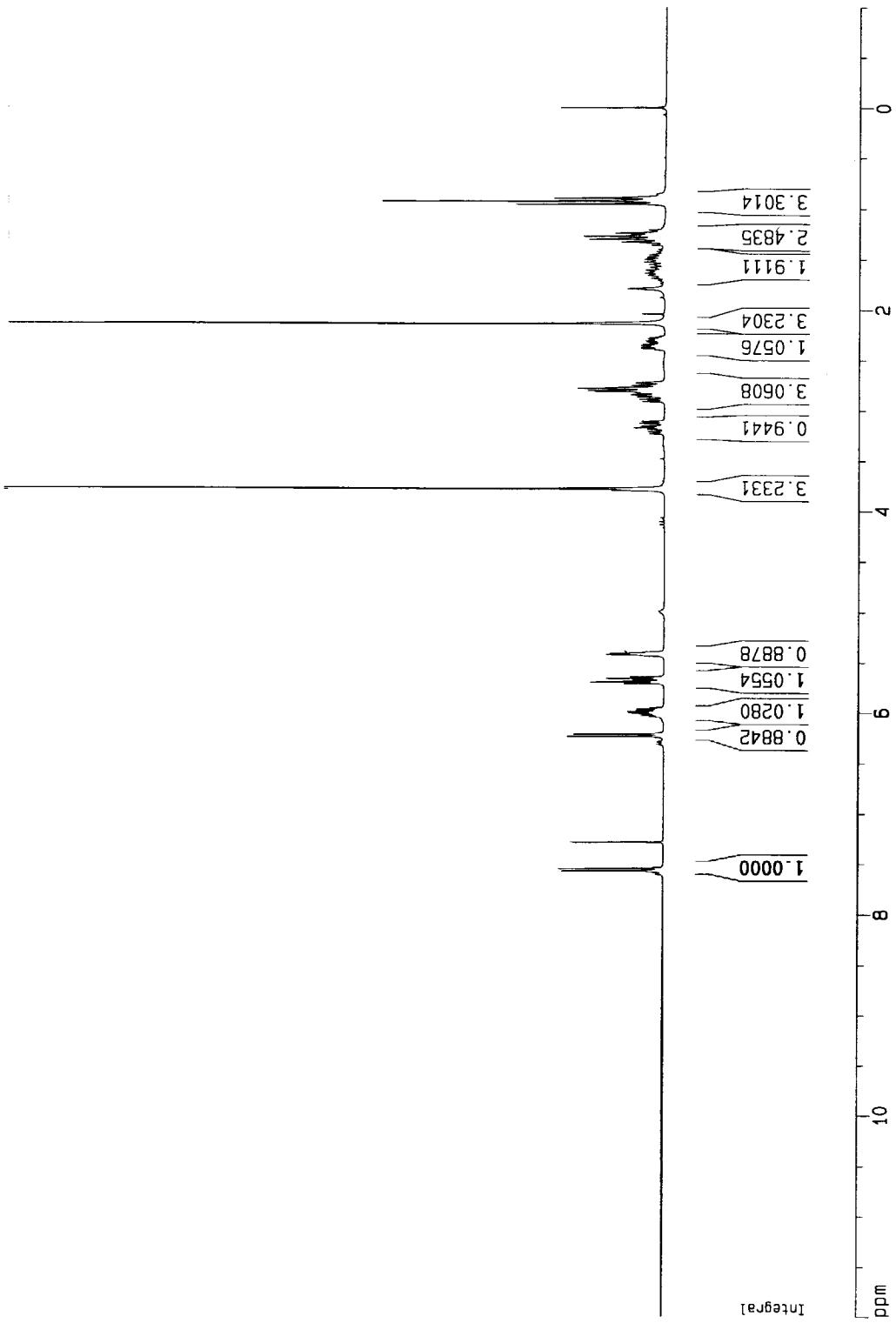


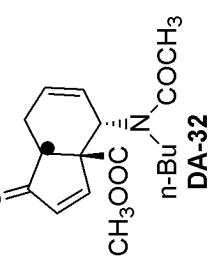


Current Data Parameters
NAME MG-4-0666b-250
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Data 20050520

DATE -	20050520
TIME	23.03
INSTRUM	arX250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDC13
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.458946 Hz
AQ	3.1457779 sec
RG	715
DW	96.000 use
DE	137.14 use
TE	300.0 K
D1	1.00000000 sec
P1	8.70 use
SF01	250.1315321 MHz
NUCLEUS	1H

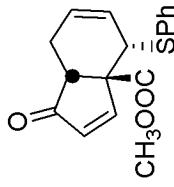




Current Data Parameters

NAME	MG-4-0660-250
EXPNO	2
PROCNO	1





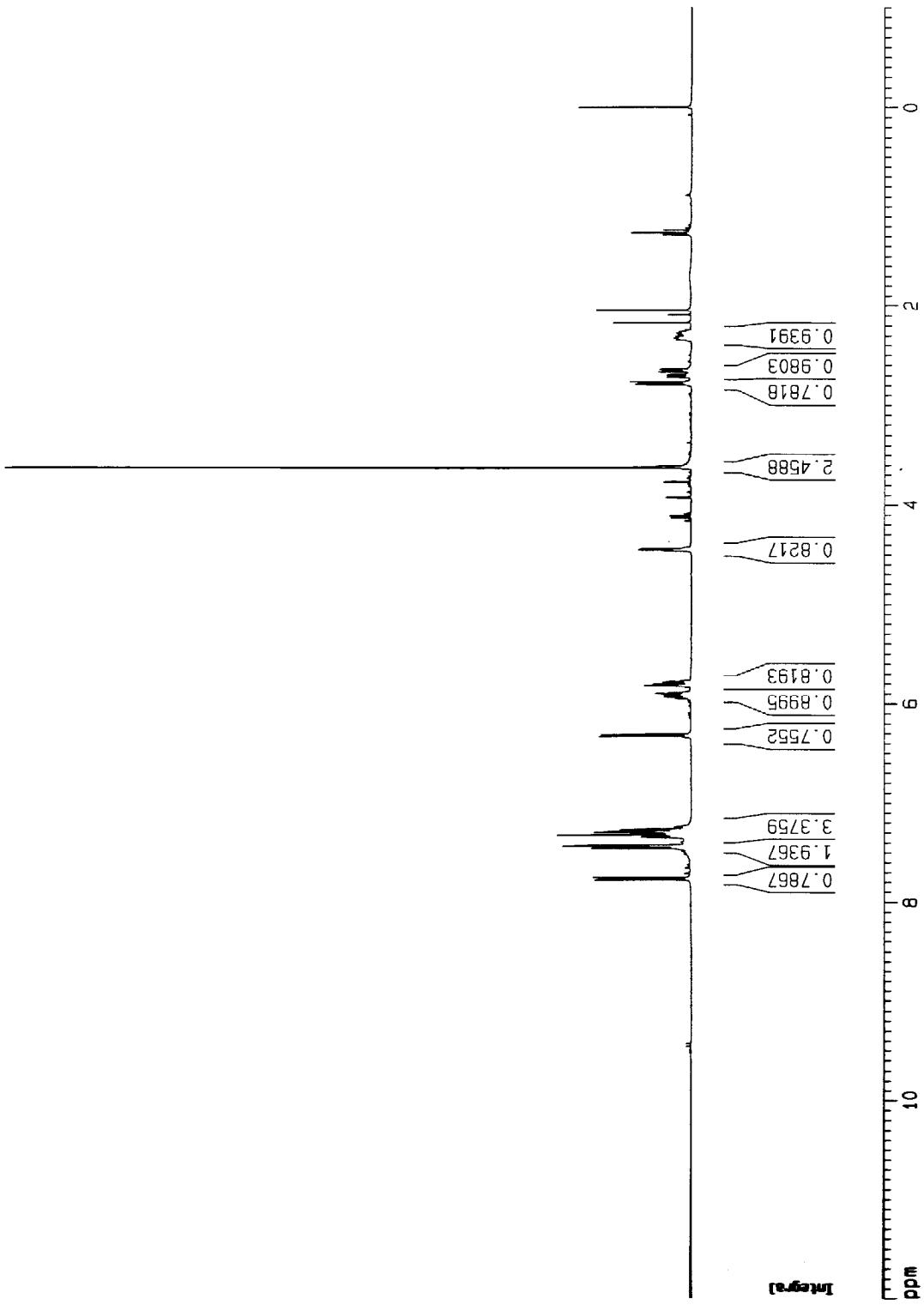
Current Data Parameters
 NAME MG-4-035a-300
 EXPNO 1
 PROCN0

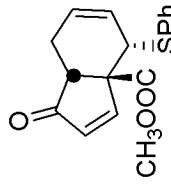
F2 - Acquisition Parameters
 Date 20050429
 Time 14.42
 INSTRUM drx300
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 256
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 D31 0.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.05 usec
 PL1 0.00 dB
 SF01 300.1318534 MHz

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

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 12.000 ppm
 F1 3601.56 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.65000 ppm/cm
 HZCM 195.08450 Hz/cm





DA-33

Current Data Parameters
 NAME MG-4-35a-300
 EXPNO 2
 PROCN0 1

F2 - Acquisition Parameters

Date_ 20050422
 Time 22.49
 INSTRUM drx300
 PROBHD 5 mm Multinucl
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 321
 DS 4
 SWH 18832.393 Hz
 FIDRES 0.287360 Hz
 AQ 1.7400308 sec
 RG 22528
 DW 6.00 usec
 DE 26.550 usec
 TE 297.1 K
 P1 1.7999995 sec
 d11 0.0300000 sec
 d31 0.0000000 sec

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

NUC1 ^{13}C
 P1 8.50 usec
 PL1 5.00 dB
 SF01 75.4760107 MHz

===== CHANNEL f2 =====

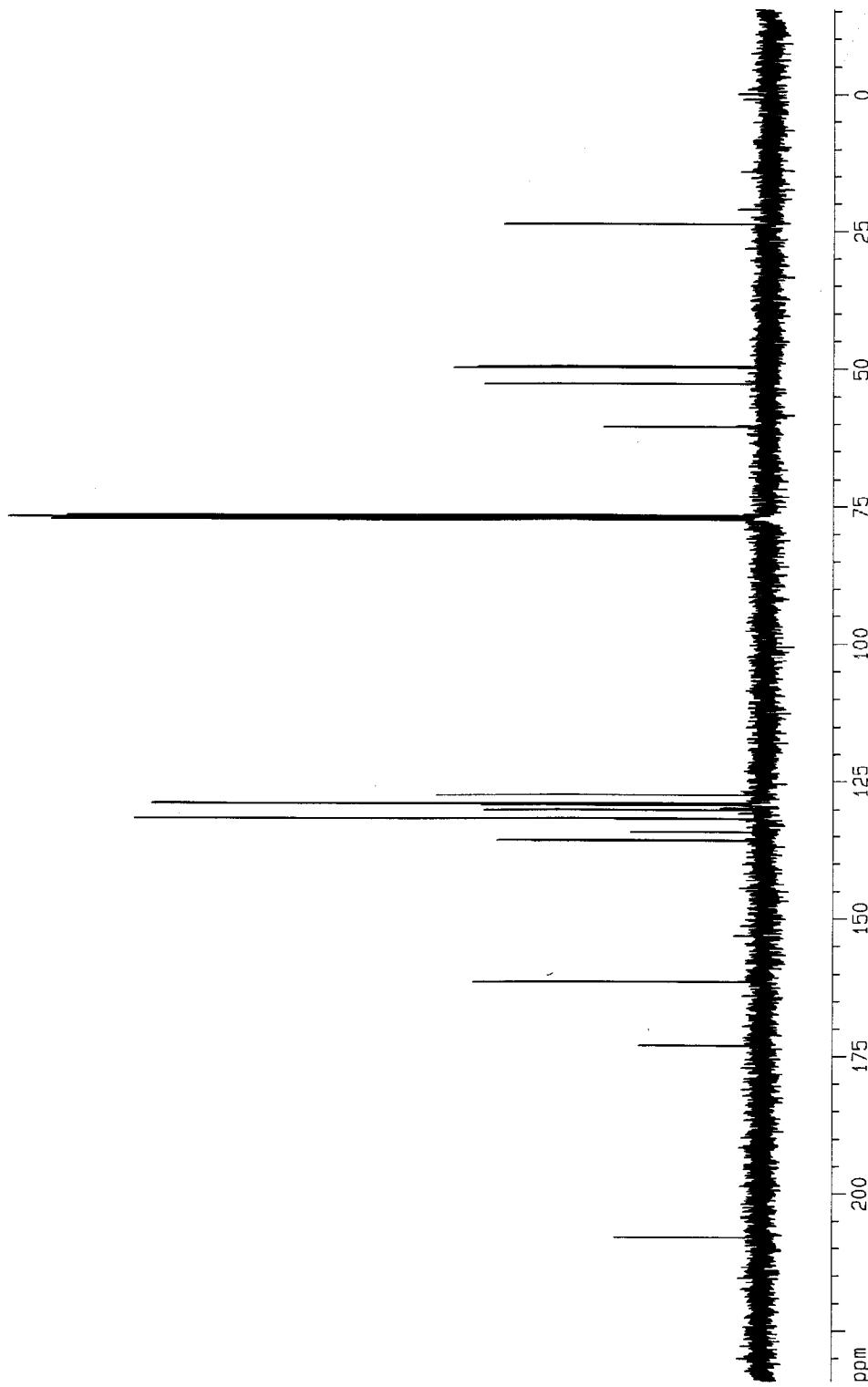
CPDPG2 waltz16
 NUC2 ^1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 25.60 dB
 SF02 300.1312005 MHz

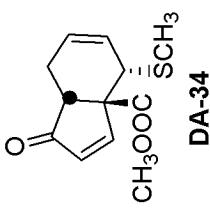
F2 - Processing parameters

SI 32768
 SF 75.4677508 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 ppm
 CY 11.00 ppm
 F1P 234.220 ppm
 F1 17676.09 Hz
 F2P -15.322 ppm
 F2 -1156.30 Hz
 FPMW 12.47711 ppm/cm
 HZCM 941.61957 Hz/cm



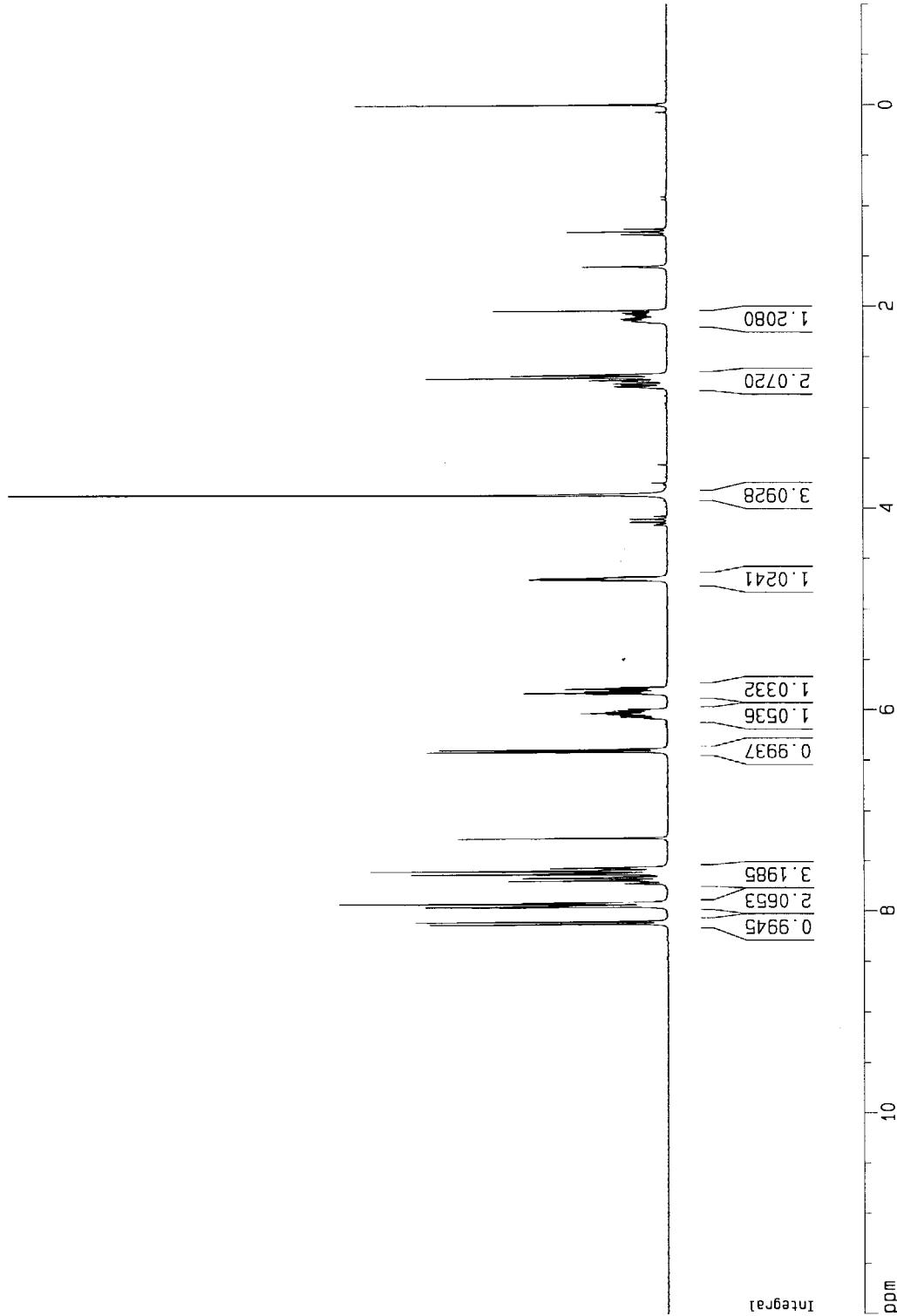


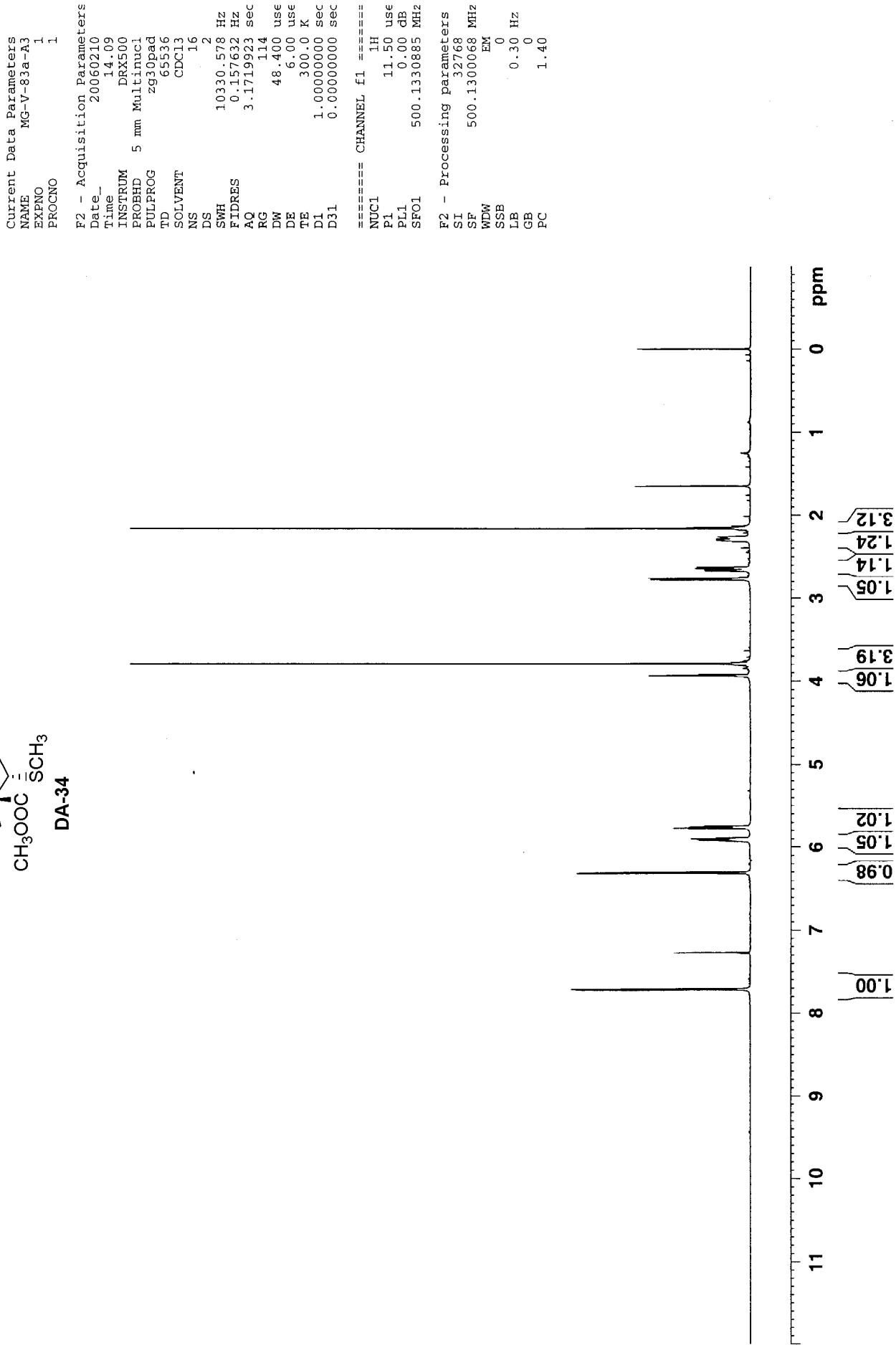
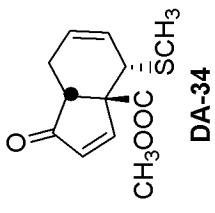
Current Data Parameters
 NAME MG-IV-046a-250
 EXPNO 1
 PROCNO

F2 - Acquisition Parameters
 Date - 20050505
 Time 0.35
 INSTRUM arx250
 PROBHD 5 mm QNP 1H
 PULPROG PULPROG
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 2048
 DW 96.000 use
 DE 137.14 use
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.70 use
 SFO1 250.1315321 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 250.1300049 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.50

1D NMR plot parameters
 CX 20.00 cm
 CY 30.00 cm
 F1P 12.000 ppm
 F1 3001.56 Hz
 F2P -1.000 ppm
 F2 -250.13 Hz
 FPMCM 0.65000 ppm
 HZCM 162.58452 Hz /



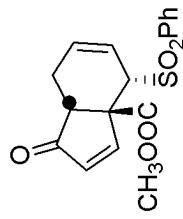


Current Data Parameters

NAME MG-IV-046a-250
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050505
Time 0.35
INSTRUM arX250
PROBHD 5 mm QNP 1H
PULPROG PULPROG
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5208.333 Hz
FIDRES 0.158946 Hz
AQ 3.1457779 sec
RG 2048
DW 96.000 use
DE 137.14 use
TE 300.0 K
D1 1.0000000 sec
P1 8.70 use
SF01 250.1315321 MHz
NUCLEUS 1H

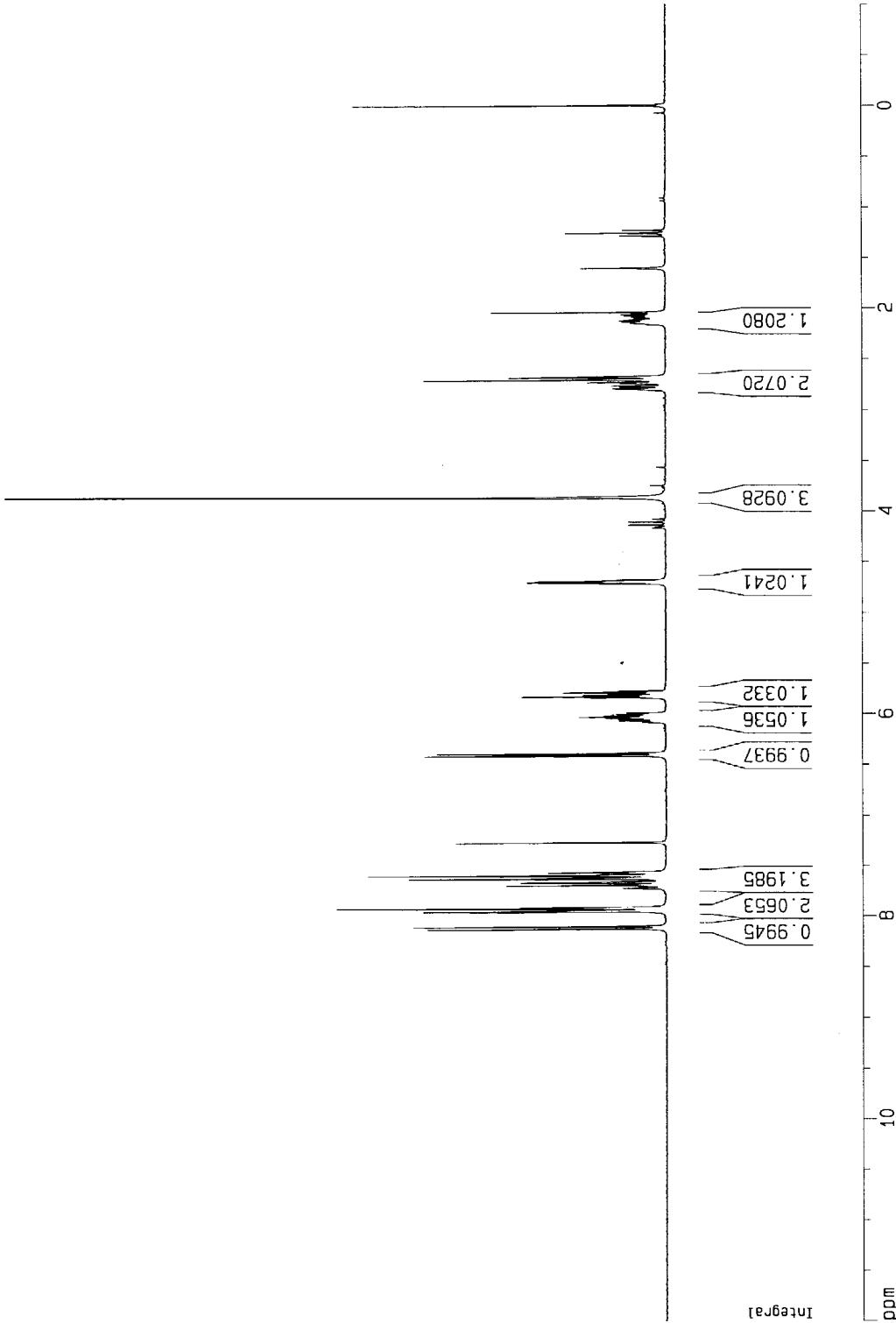


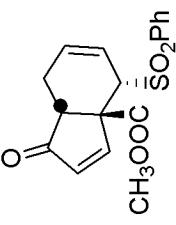
F2 - Processing parameters

SI 16384
SF 250.1300049 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.50

1D NMR plot parameters

CX 20.00 cm
CY 30.00 cm
F1P 12.000 ppm
F1 3001.56 Hz
F2P -1.000 ppm
F2 -250.13 Hz
PPMCM 0.65000 ppm
HZCM 162.58452 Hz/





Current Data Parameters
 NAME MG-IV-046a-250
 EXPNO 2
 PROBNO 1

F2 - Acquisition Parameters
 Date_ 20050505
 Time 0.39
 INSTRUM arx50
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 36664
 SOLVENT CDCl3
 NS 209
 DS 4
 SWH 17241.379 Hz
 FIDRES 0.467702 Hz
 AQ 1.0693060 sec
 RG 22800
 DW 29.000 use
 DE 41.43 use
 TE 300.0 K
 D12 0.00002000 sec
 QL5 23.00 dB
 CPDPG6 waltz16
 P31 103.00 use
 D1 2.00000000 sec
 P1 6.00 use
 SF01 62.9023694 MHz
 NUCLEUS 13C
 D11 0.03000000 sec

F2 - Processing parameters
 SI 32768
 SF 62.8952408 MHz
 MDW 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 250.405 ppm
 F1 15749.28 Hz
 F2P -23.724 ppm
 F2 -1492.10 Hz
 PPMCM 13.70643 ppm
 HZCM 862.06891 Hz /

