

General. All reactions were performed under nitrogen or argon atmosphere with flame-dried glassware. All solvents were distilled prior to use; diethyl ether and THF over sodium and benzophenone. Toluene and dichloromethane (DCM) were dried over calcium hydride. Column chromatography was performed with 60 silica gel (230-400 mesh, Merck). ^1H and ^{13}C NMR were recorded on Bruker instruments at 500 and 125 MHz, respectively, except where noted. IR spectra were recorded with a Bomen instrument and optical rotations were measured with a Perkin-Elmer model 241 polarimeter. Melting points are uncorrected. The high-resolution mass spectra were recorded at The University of Ottawa Mass Spectrum Center.

1S-[2-(*tert*-Butyl-diphenyl-silanyloxy)-ethyl]-vinyl-6-isopropenyl-3-methyl-cyclohex-2-enol (5): To a solution of vinyl iodide (184.6 mg, 0.423 mmol) in ether (4 mL) at -90°C *t*-BuLi (1.32 M in pentane 0.56 mL, 0.423 mmol) was added and the mixture was stirred for 10 minutes. During this time, a cloudy white precipitate was formed inside the flask. A solution of the ketone **4** (42.3 mg, 0.282 mmol) in ether (5 mL) at -90°C was transferred via canula into the reaction mixture. The solution was allowed to stir while warming to -40°C at which time it was quenched with a saturated aqueous solution of ammonium chloride. It was then extracted with ethyl acetate (3x 15 mL), dried (MgSO_4), filtered and concentrated. The residue was purified by flash chromatography (10% ethyl acetate in hexanes) to give a colourless oil (115.3 mg, 0.25 mmol, 91%). IR (neat, cm^{-1}) 3526, 1434, 1102; ^1H NMR (500 MHz, CDCl_3) δ 7.67-7.65 (m, 4H), 7.42-7.35 (m, 6H), 5.15 (d, $J = 6.6$ Hz, 2H), 4.90 (s, 1H), 4.84 (s, 1H), 4.73 (s, 1H), 3.81-3.78 (m, 2H), 2.37-2.23 (m, 3H), 2.11 (s, 1H), 1.99-1.97 (m, 2H), 1.94-1.86 (m, 1H), 1.71 (s, 3H), 1.65 (s, 3H), 1.56 (s, 1H), 1.04 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 151.2, 147.2, 138.3, 135.5 (2 C), 135.5, 133.8, 129.5 (4 C), 127.6 (4 C), 126.5, 112.9, 110.0, 73.9, 63.8, 48.0, 35.0, 30.8, 26.8 (3 C), 24.6, 24.5, 23.4, 19.1; HRMS (EI) m/z ($\text{M}^+ - \text{C}_4\text{H}_{11}\text{O}$) calcd 385.1988, obsd 385.1989 $[\alpha]_{\text{D}} = +10.5^\circ$, $c = 3.82$ in CH_2Cl_2 .

(1R, 6S, 7R)-7-[(2-*tert*-butyldiphenylsiloxy) ethyl]-10-methylene-4-methylbicyclo [4.4.0]^{1,6} deca-4-en-6-ol (6): A solution of **5** (93.5 mg, 0.203 mmol) in degassed toluene (3 mL) and DBU (0.152 mL, 1.01 mmol) was heated at 220°C in a sealed tube for 5 hrs under argon. The tube was cooled to rt and the solution was concentrated. The yellowish oil was purified by flash chromatography (10% ethyl acetate in hexanes) to afford a colourless oil **6** (55.2 mg, 0.120 mmol, 60%). IR (neat, cm^{-1}) 2930, 1111, 702; ^1H NMR (500 MHz, CDCl_3) δ 7.68-7.65 (m, 4H), 7.42-7.35 (m, 6H), 5.69 (s, 1H), 4.87 (s, 1H), 4.63 (s, 1H), 3.79-3.75 (m, 1H), 3.69-3.66 (m, 1H), 2.34 (s, 1H, OH), 2.28 (dq, $J = 2.1, 13.1$ Hz, 1H), 2.06-1.97 (m, 5H), 1.73-1.65 (m, 6H), 1.53-1.52 (m, 1H), 1.40-1.24 (m, 3H), 1.05 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 149.5, 138.8, 135.6 (4 C), 134.0 (2 C), 129.5, 127.6 (4 C), 124.5, 124.4, 107.5, 71.6, 62.6, 48.2, 41.7, 35.8, 32.2, 30.6, 30.0, 26.9 (3 C), 23.7, 20.5, 19.2; HRMS (EI) m/z $[\alpha]_{\text{D}} = -66.3^\circ$, $c = 2.79$ in CH_2Cl_2 .

(1R, 6S, 7R)-7-[(2-Hydroxy) ethyl]-10-methylene-4-methylbicyclo [4.4.0]^{1,6} deca-4-en-6-ol (7): To a solution of **6** (107.6 mg, 0.234 mmol) in THF (7 mL) TBAF (0.257 mL, 0.257 mmol) was added and the solution was stirred for 1.5 hours. The mixture was concentrated and purified by flash chromatography (25% hexanes in ethyl acetate) to afford a colourless oil **7** (52.0 mg, 0.234 mmol, 100%) which was used immediately for the next reaction.

(1R, 4R, 5R, 6S, 7R)-4,5-Epoxy-7-[(2-hydroxy) ethyl]-10-methylene-4-methylbicyclo [4.4.0]^{1,6} decan-6-ol (8): To a solution of **7** (12.2 mg, 0.055 mmol) in DCM (1.5 ml) was added VO(acac)₂ (65.9 μ L, 0.01M solution in DCM). The reaction mixture turned from faintly green to deep purple immediately upon addition of *tert*-butyl hydroperoxide (8.3 μ L, 0.060 mmol). After stirring for 1 hour the mixture had turned light yellow and was deemed complete (TLC). The solution was washed with an aqueous saturated solution of Na₂S₂O₃ (3x 5ml), dried over MgSO₄, filtered and concentrated. The residue was purified by flash chromatography (20% hexanes in ethyl acetate) to produce an oil **8** (12.4 mg, 0.052 mmol, 95%). IR (neat, cm⁻¹) 2935, 1059; ¹H NMR (500 MHz, CDCl₃) δ 4.80 (s, 1H), 4.51 (s 1H), 3.78-3.73 (qt, *J* = 5.6 Hz, 1H), 3.65-3.60 (m, 1H), 3.11 (s, 1H), 2.50 (br.s, 1H), 2.33 (dq, *J* = 2.1, 13.1 Hz, 1H), 2.12-1.91 (m, 4H), 1.85-1.80 (m, 1H), 1.76-1.54 (m, 5H), 1.44-1.33 (m, 2H), 1.32 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 147.9, 107.3, 70.6, 63.7, 61.2, 60.4, 48.5, 40.9, 35.6, 32.4, 30.1, 29.4, 23.3, 17.3; HRMS (EI) *m/z* (M⁺) calcd 238.1569, obsd 238.1555, [α]_D = -66.2°, *c* = 1.56 in CH₂Cl₂.

(1R, 4R, 5R, 6S, 7R, 10S)-4,5-Epoxy-7-[(2-hydroxy) ethyl]-4,10-methylbicyclo [4.4.0]^{1,6} decan-6-ol (9): To a solution of **8** (12.0 mg, 0.050 mmol) in DCM (1.5 mL) a balloon of H₂ with a stopcock was attached. The flask was evacuated and replenished with hydrogen 3 times. A solution of Crabtree's catalyst was then added (252 μ L, 0.005 M solution in DCM). After stirring for 2 hours the reaction was complete by TLC. The solution was filtered through celite and washed with ethyl acetate and concentrated. The solid was purified by flash chromatography (50% ethyl acetate in benzene) to give a white solid (10.9 mg, 0.045 mmol, 90%). IR (neat, cm⁻¹) 2926, 1056; ¹H NMR (500 MHz, CDCl₃) δ 3.78-3.74 (qt, *J* = 5.6 Hz, 1H), 3.65-3.60 (m, 1H), 3.05 (s, 1H), 2.55 (br.s, 1H), 2.05 (dd, *J* = 4.3, 15.2 Hz, 2H), 1.96-1.90 (m, 1H), 1.69-1.42 (m, 8H), 1.30 (s, 3H), 1.08-0.94 (m, 2H), 0.80-0.79 (d, *J* = 6.6 Hz, 3H), 0.61-0.56 (t, *J* = 11.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 68.8, 64.1, 61.7, 60.5, 50.5, 41.0, 35.3, 32.6, 30.6, 29.7, 27.5, 23.4, 19.7, 17.1; HRMS (EI) *m/z* (M⁺) calcd 240.1725, obsd 240.1678, mp: 79-81°C, [α]_D = -1.25°, *c* = 2.27 in CH₂Cl₂.

(1R, 4R, 5R, 6S, 7R, 10S)-4,5-Epoxy-6,7-[7, 11-(2H)-furan-12-one]-4,10-dimethylbicyclo [4.4.0]^{1,6} decane (13): To a solution of **9** (9.5 mg, 0.040 mmol) in DCM (1.4 mL), TPAP (1 mg, 0.002 mmol), NMO (7 mg, 0.060 mmol) and molecular sieves 4Å (20.0 mg,) were added. After stirring for 1.5 hours, the reaction mixture was concentrated to dryness and placed directly on a column and flashed with 30% hexanes in ethyl acetate yielding a white solid (8.5 mg, 0.036 mmol, 90%). IR (neat, cm⁻¹) 1768, 1175; ¹H NMR (500 MHz, CDCl₃) δ 2.93 (dd, *J* = 6.7, 17.2 Hz, 1H), 2.78 (s, 1H), 2.25 (dd, *J* = 6.7, 8.2 Hz, 1H), 2.18 (d, *J* = 17.2 Hz, 1H), 2.15-2.12 (m, 1H), 1.90-1.84 (m, 1H), 1.67-1.60 (m, 2H), 1.54-1.47 (m, 2H), 1.38 (td, *J* = 4.62, 12.3 Hz, 1H), 1.32 (s, 1H), 1.11-1.04 (m, 1H), 0.88 (d, *J* = 6.7 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 176.1, 83.6, 62.4, 58.2, 46.8, 38.8, 36.6, 31.1, 31.0, 27.8, 27.7, 25.4, 22.8, 19.5, 17.8; HRMS (EI) *m/z* (M⁺) calcd 236.1412, obsd 236.1404, mp: 64-66°C [α]_D = -15.6°, *c* = 1.85 in CH₂Cl₂.

(1R, 4R, 5R, 6S, 7R, 10S, 11R)-4,5-Epoxy-6,7-[7, 11-(2H)-furan-12-one]-4,10,11-trimethylbicyclo [4.4.0]^{1,6} decane (14): To a solution of **13** (53.7 mg, 0.227 mmol) in THF (3ml) at -78°C was added LDA freshly prepared (1.0 M solution in THF, 272 μ L, 0.272 mmol). After stirring for 25 minutes CH₃I (141 μ L, 2.27 mmol) was added and the resulting solution was stirred for 5 minutes. The reaction was quenched with NH₄Cl (saturated aqueous solution) and

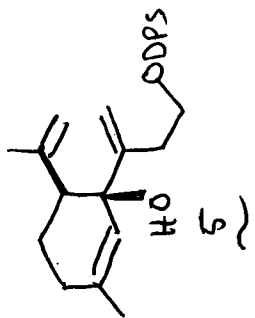
extracted with ethyl acetate (3 x 7 mL). After drying over MgSO_4 , filtering and concentrating the product was purified by flash chromatography (50% hexanes in ethyl acetate) to give a white solid **14** (40.9 mg, 0.163 mmol, 72%), IR (neat, cm^{-1}) 2926, 1771; ^1H NMR (500 MHz, CDCl_3) δ 2.85 (s, 1H), 2.44-2.40 (q, $J = 7.9$ Hz, 1H), 2.16-2.14 (m, 1H), 2.12-2.07 (m, 1H), 1.86-1.79 (m, 1H), 1.65-1.50 (m, 3H), 1.47-1.45 (d, $J = 7.9$ Hz, 3H), 1.44-1.36 (m, 2H), 1.33-1.28 (m, 4H), 1.16-1.12 (m, 1H), 0.90-0.89 (d, $J = 6.7$ Hz, 3H), 0.88-0.85 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 179.9, 83.4, 64.3, 58.5, 45.5, 44.0, 43.7, 30.9, 26.3, 26.1, 25.9, 22.7, 20.3, 18.9, 18.0; HRMS (EI) m/z (M^+) calcd 250.1569, obsd 250.1552, mp: 63-64°C, $[\alpha]_{\text{D}} = -16.3^\circ$, $c = 0.97$ in CH_2Cl_2 .

(1R, 4R, 5R, 6S, 7R, 10S, 11S)-4,5-Epoxy-6,7-[7, 11-(2H)-furan-12-one]-4,10,11-trimethylbicyclo [4.4.0]^{1,6} decane (15): To a solution of **14** (11.3 mg, 0.045 mmol) in THF (2.0 mL) at -78°C was added LDA (0.45 mL, 1.0 M solution). After stirring for 25 minutes at the same temperature, a saturated aqueous solution of ammonium chloride was poured into the mixture. The resulting mixture was extracted with ethyl acetate (3x, 5 mL), dried (MgSO_4), filtered and concentrated. The residue was purified by flash chromatography (40% hexanes in ethyl acetate) to afford a white solid **15** (11.0 mg, 0.044 mmol, 97%). IR (neat, cm^{-1}) 2926, 1768; ^1H NMR (500 MHz, CDCl_3) δ 3.09-3.06 (q, $J = 6.8$ Hz, 1H), 2.83 (s, 1H), 2.20-2.10 (m, 2H), 1.73-1.58 (m, 3H), 1.50-1.43 (m, 3H), 1.30 (s, 3H), 1.28-1.20 (m, 1H), 1.15-1.13 (d, $J = 7.1$ Hz, 3H), 1.06-1.01 (m, 1H), 0.87-0.86 (d, $J = 6.6$ Hz, 3H), 0.83-0.82 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 178.4, 81.3, 62.2, 57.9, 47.1, 42.9, 38.7, 31.5, 30.8, 28.2, 22.9, 22.7, 19.4, 17.6, 9.3; HRMS (EI) m/z (M^+) calcd 250.1569, obsd 250.1565, mp: 74-75°C, $[\alpha]_{\text{D}} = -13.6^\circ$, $c = 2.46$ in CH_2Cl_2 .

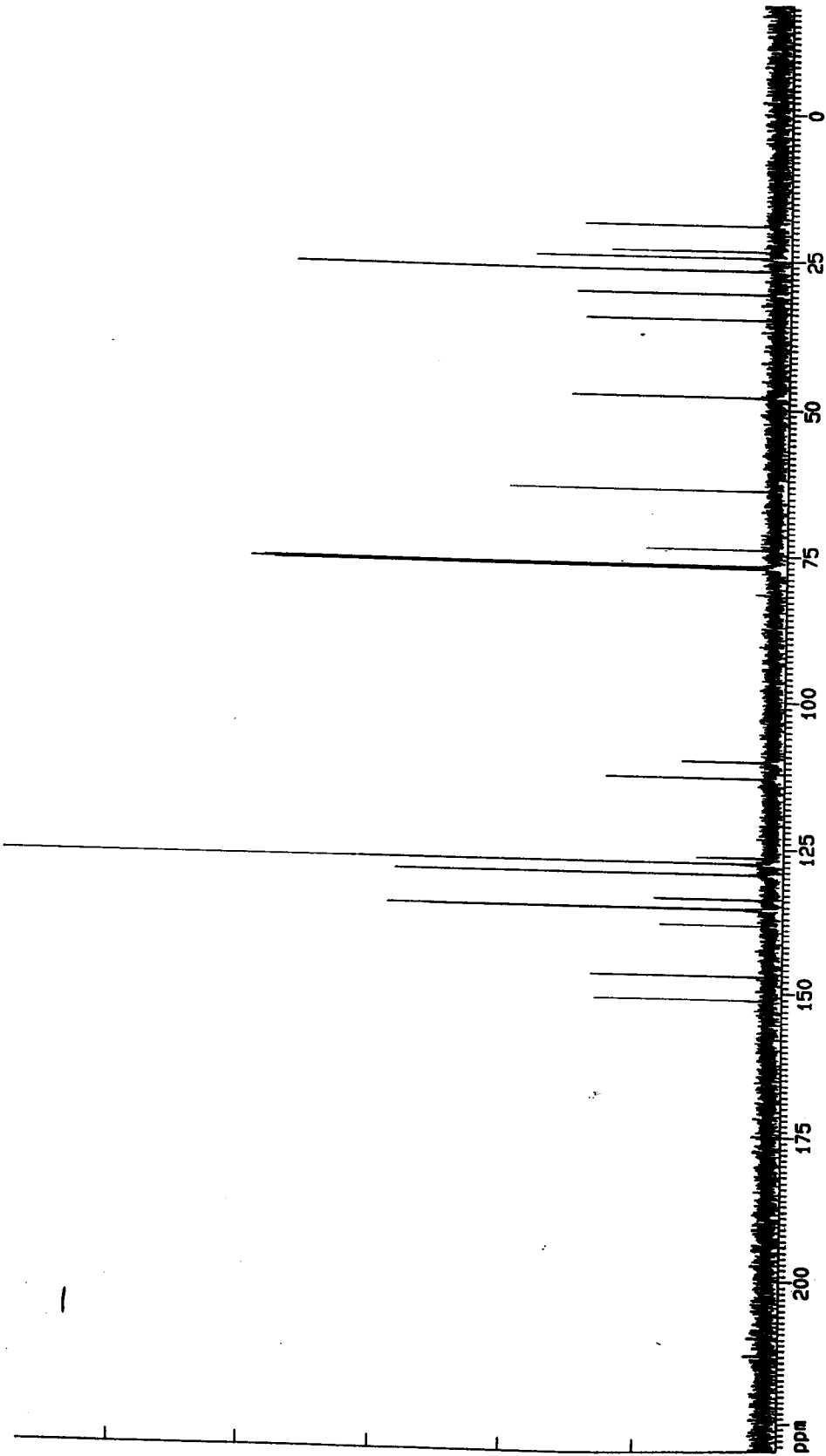
(1R, 6S, 7S, 10S, 11S)-6,7 [7, 11-(2H)-Furan-12-one]4,10,11-triethylbicyclo [4.4.0]^{1,6} decan-4-ene (16): To THF (1.4 mL) at -78°C was added WCl_6 (82.9 mg, 0.22 mmol). Five minutes later, $n\text{-BuLi}$ (1.9 M in hexanes, 0.33 mL, 0.628 mmol) was added over 5 minutes. After stirring for 10 minutes at -78°C , the mixture was slowly warmed to room temperature over 1 hour. After several colour changes (greenish brown, light yellow, dark brown), LiI (0.80 mg, 0.006 mmol) was added and 5 minutes later a solution of **15** (19.4 mg, 0.078 mmol) in THF (1 mL) was added. The mixture was stirred for 2.5 hours and then poured into a cold aqueous solution of NaHCO_3 and then extracted with ethyl acetate (2x 10 mL). After drying over MgSO_4 , filtering and concentrating, the oily product was purified by flash chromatography (25% ethyl acetate in hexanes) to afford **16** as a colourless oil (11.0 mg, 0.047 mmol, 67%). IR (neat, cm^{-1}) 2930, 1764; ^1H NMR (500 MHz, CDCl_3) δ 5.61 (s, 1H), 3.14-3.09 (q, $J = 6.3$ Hz, 1H), 2.11-1.96 (m, 3H), 1.89-1.84 (m, 1H), 1.72-1.61 (m, 6H), 1.54-1.49 (m, 1H), 1.44-1.38 (m, 1H), 1.22-1.14 (m, 1H), 1.13-1.12 (d, $J = 7.2$ Hz, 3H), 1.06-0.98 (m, 1H), 0.92-0.91 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 179.2, 142.1, 121.7, 83.1, 46.5, 42.7, 39.6, 32.4, 30.8, 29.6, 23.7, 23.4, 20.9, 19.5, 9.4; HRMS (EI) m/z (M^+) calcd 234.1620, obsd 234.1600. $[\alpha]_{\text{D}} = -42.2^\circ$, $c = 0.35$ in CH_2Cl_2 .

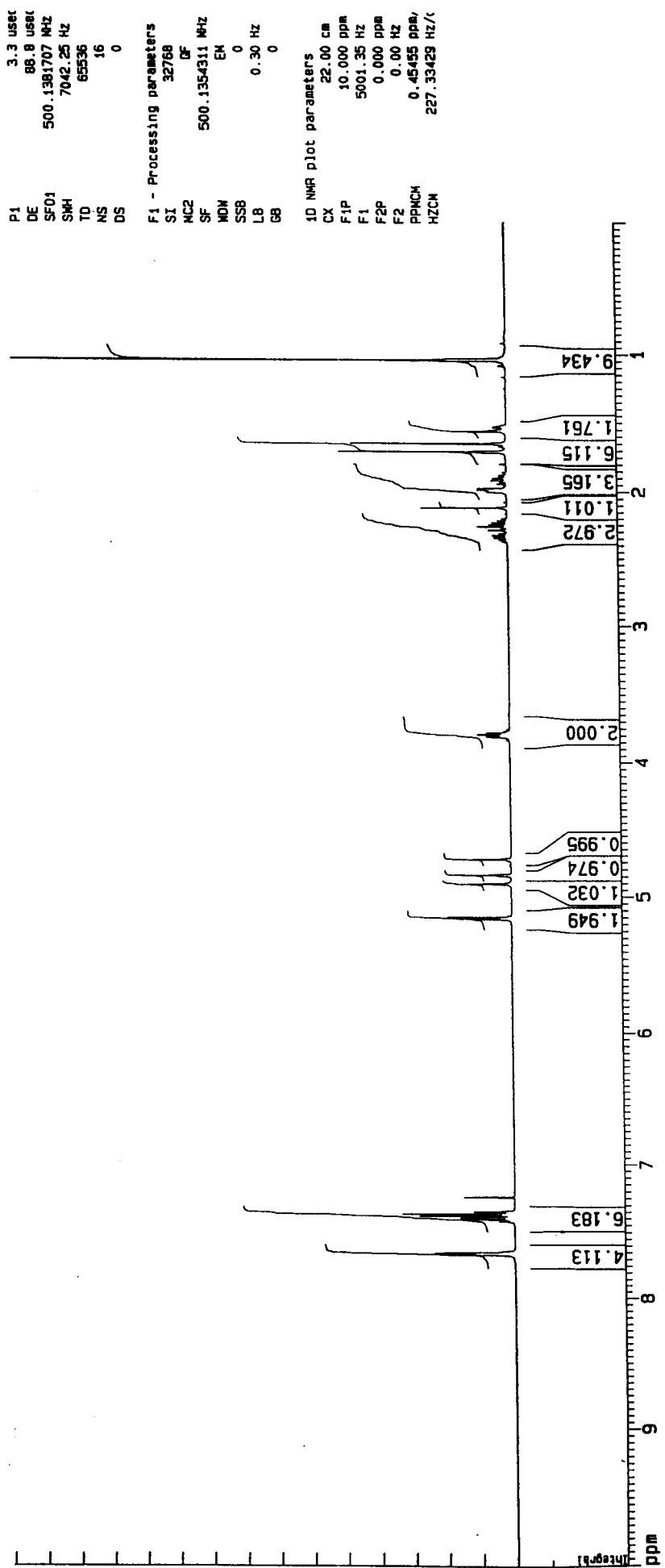
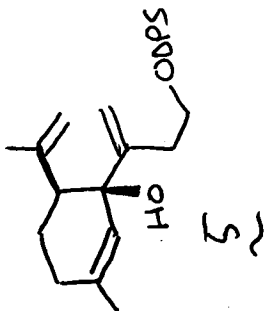
(+)-Arteannuin M (17): To a solution of **16** (6.8 mg, 0.029 mmol) in THF (1.0 mL) was added NMO (4.0 mg, 0.033 mmol) a solution of OsO_4 in H_2O (4% by weight, 18.5 μL , 0.003 mmol). After stirring for 2 days, the solution was washed with brine and extracted with ethyl acetate (2x 5 mL). After concentration, the residue was purified by flash chromatography (30% hexanes in ethyl acetate) to give an oil (5.1 mg, 0.019 mmol, 66%). IR (neat, cm^{-1}) 3347, 1766; ^1H NMR (500 MHz, CDCl_3) δ 3.43 (s, 1H), 3.06 (dq, $J = 6.9, 6.9$ Hz, 1H), 2.65-2.60 (m, 1H), 1.77-1.67 (m, 3H), 1.62-1.56 (m, 2H), 1.53-1.47 (m, 2H), 1.37-1.29 (m, 1H), 1.36 (s, 3H), 1.23 (br.s 1H),

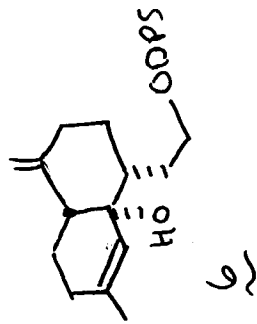
1.11 (d, $J = 6.9$ Hz, 3H), 1.08-1.03 (m, 1H), 0.90 (d, $J = 6.7$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 179.1, 86.2, 74.2, 72.6, 41.7, 39.1, 38.8, 34.3, 32.3, 29.9, 26.6, 23.9, 22.1, 20.1, 9.3; HRMS (EI) m/z ($\text{M}^+ - \text{H}_2\text{O}$) calcd 250.1569, obsd 250.1569 $[\alpha]_{\text{D}} = -34.3^\circ$, $c = 0.26$ in CH_2Cl_2 .



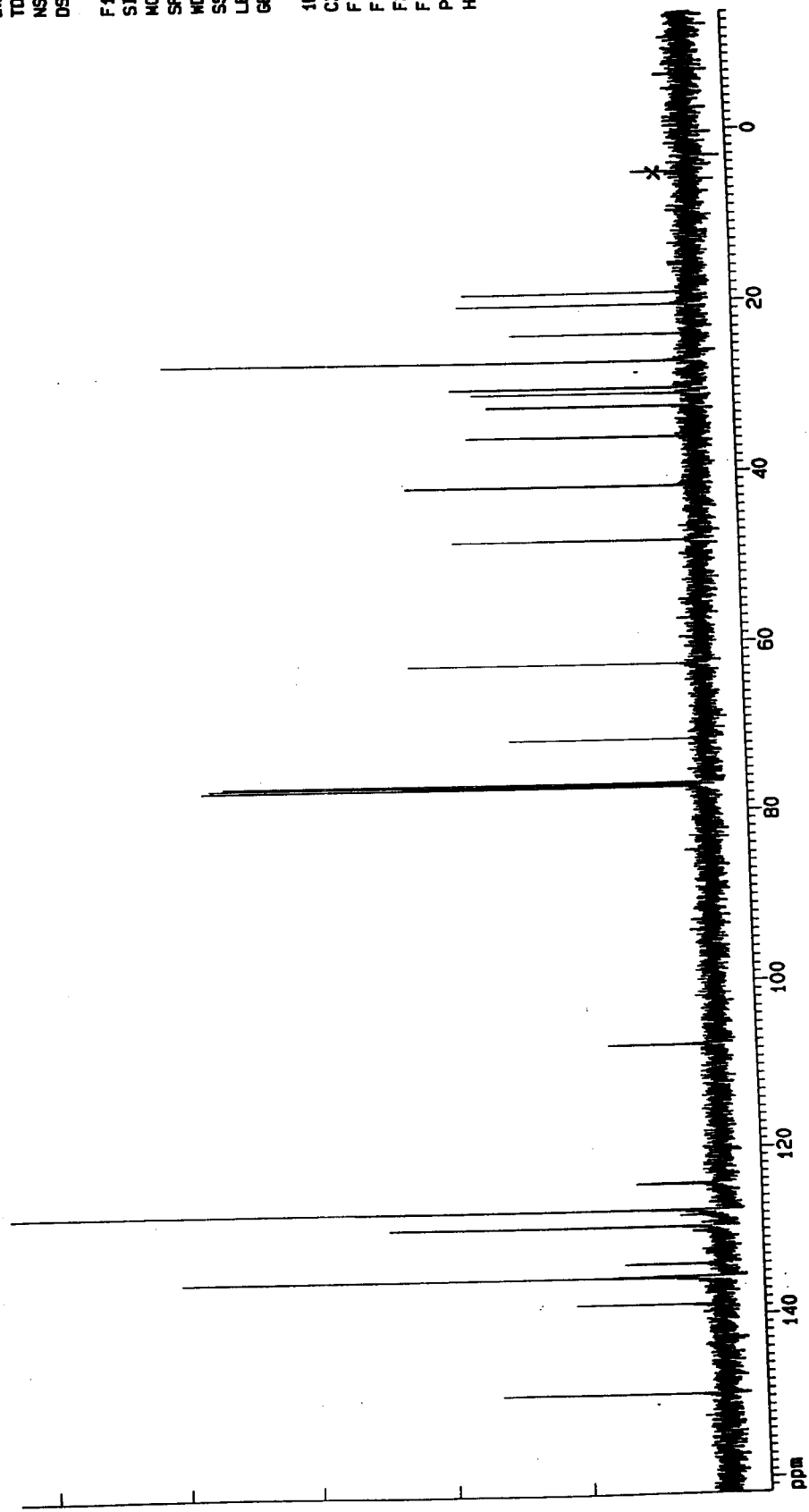
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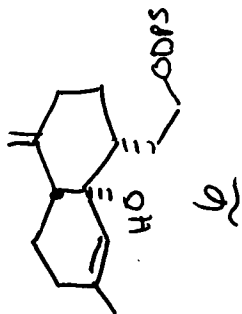




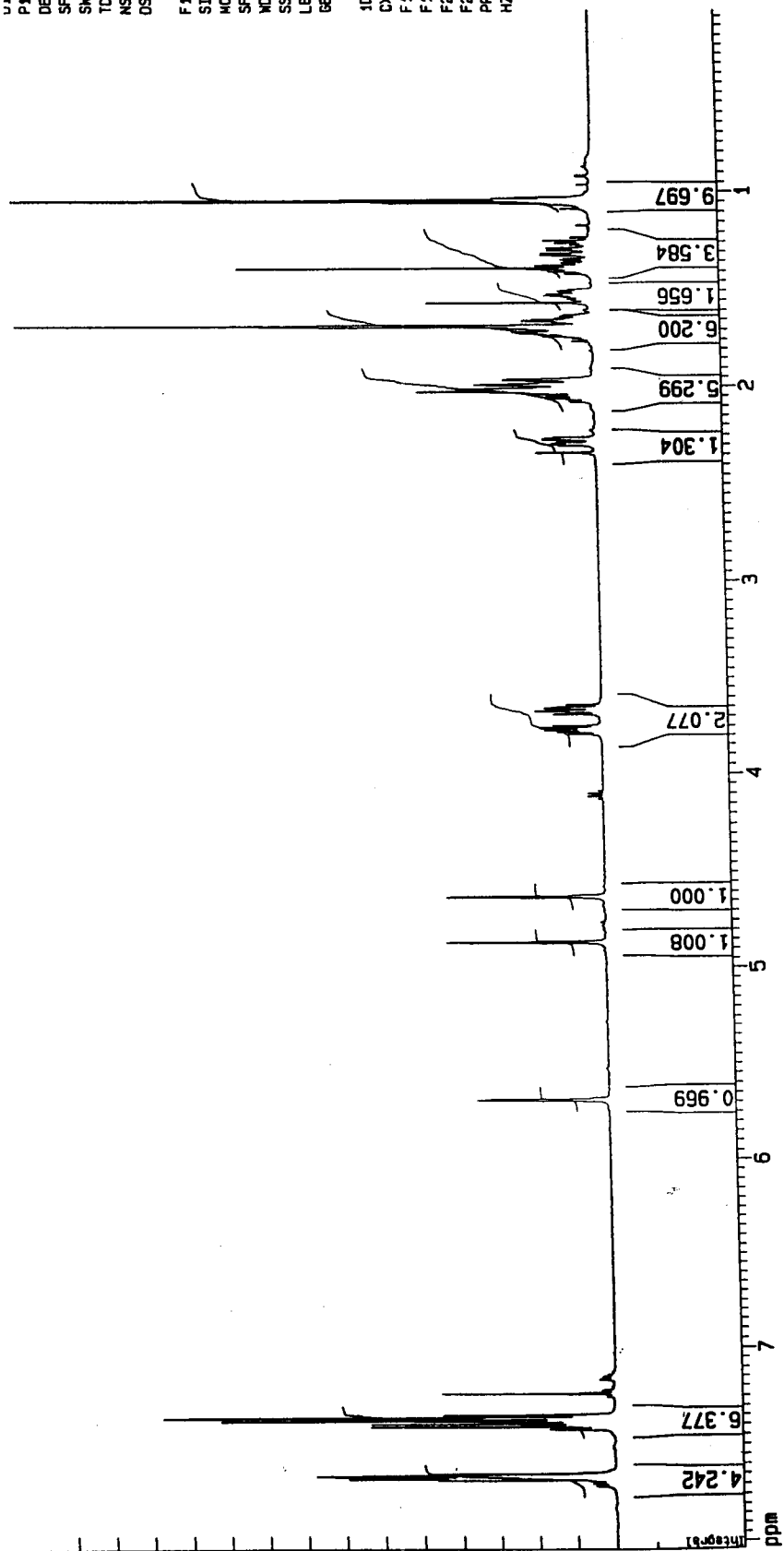


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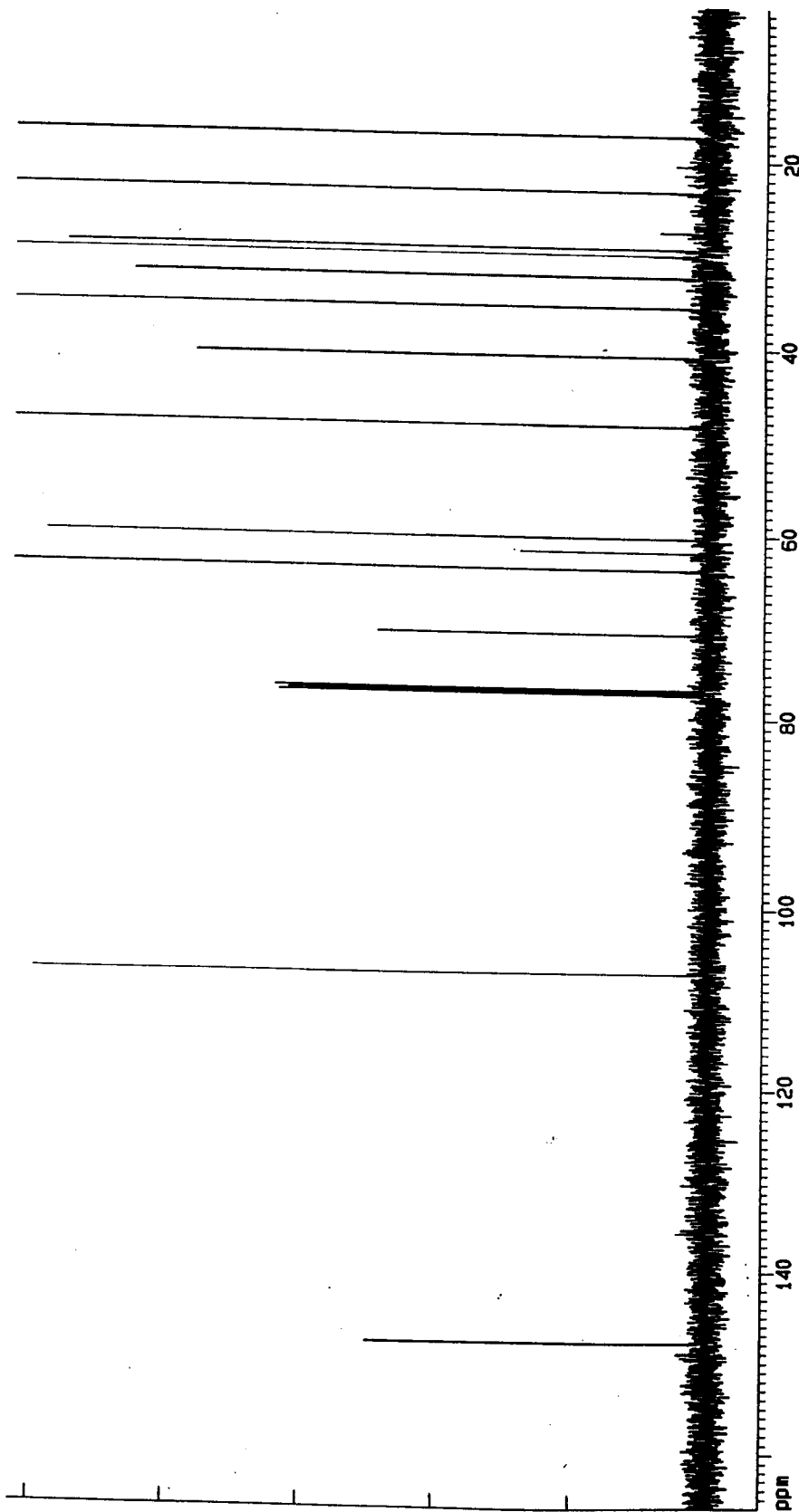
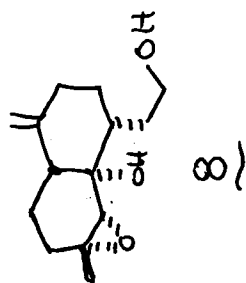




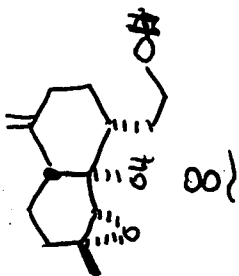
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154



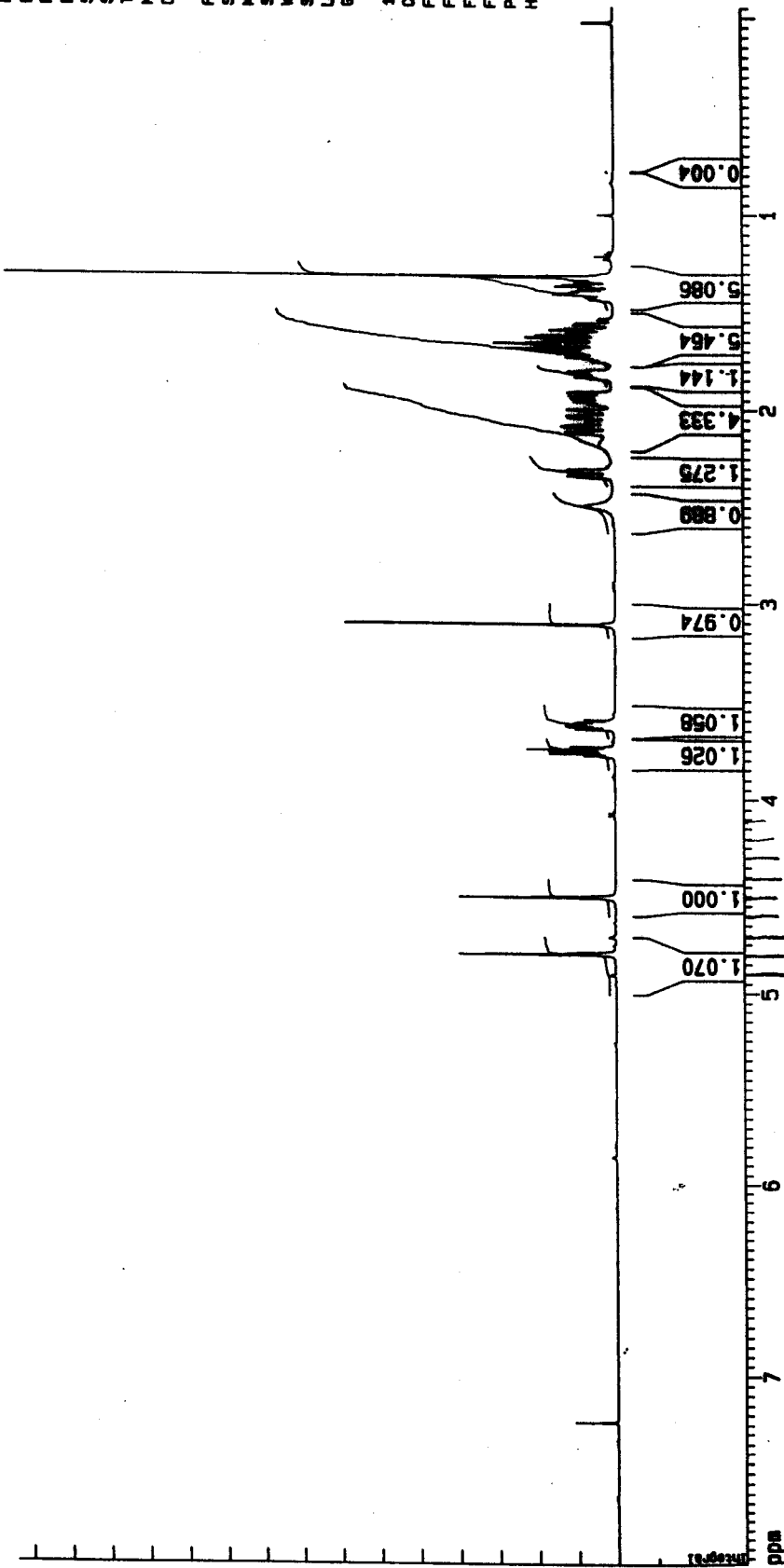
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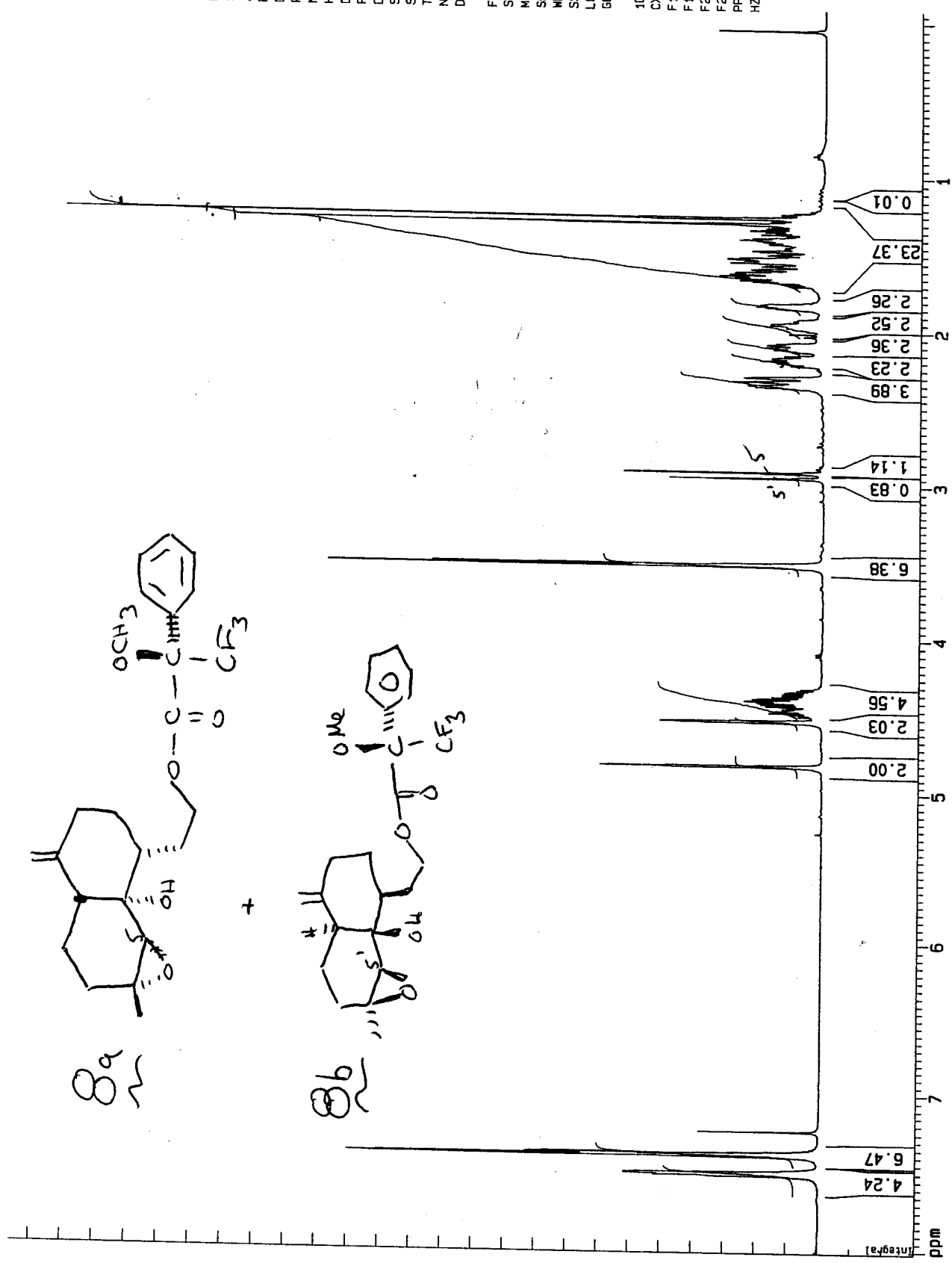


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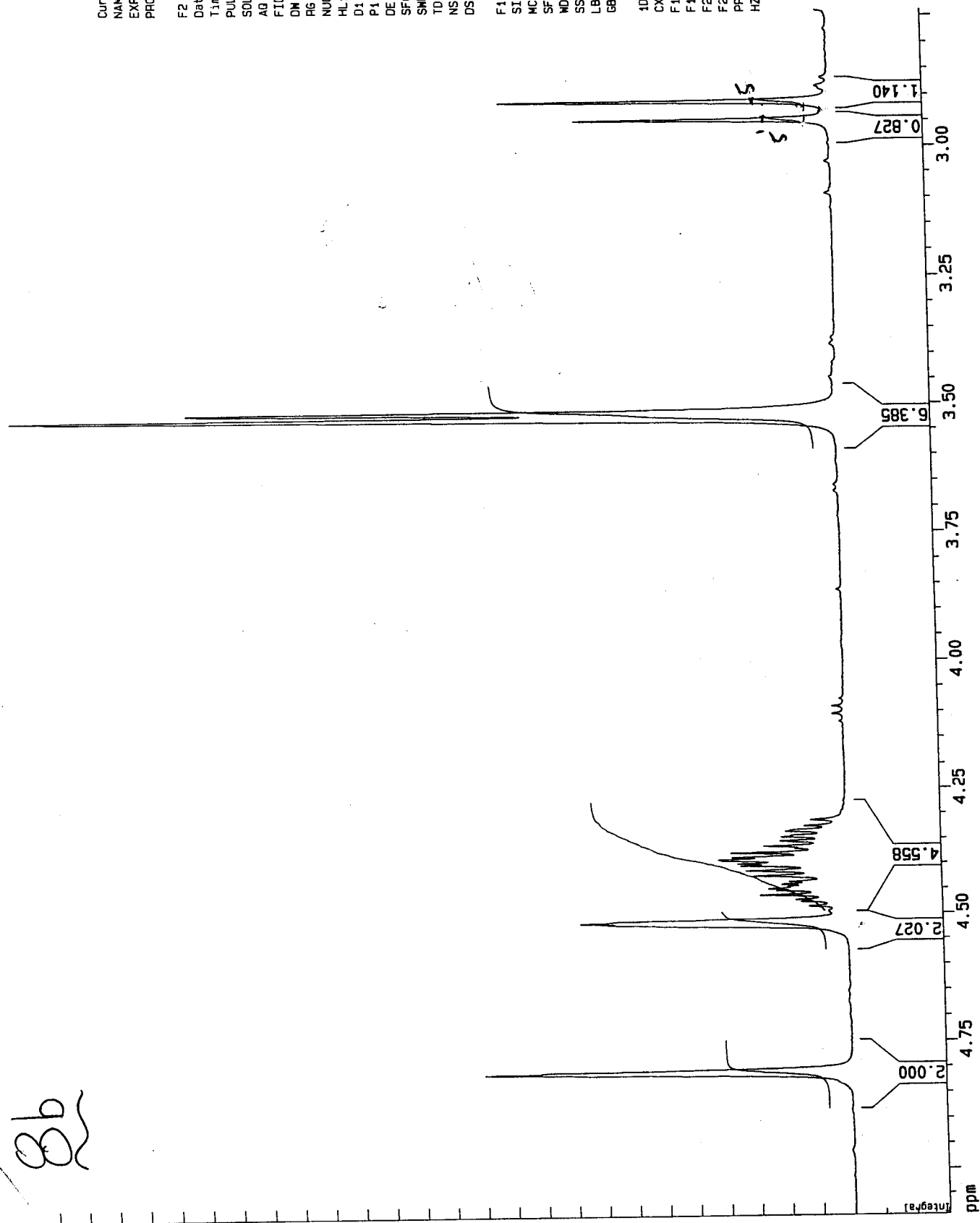
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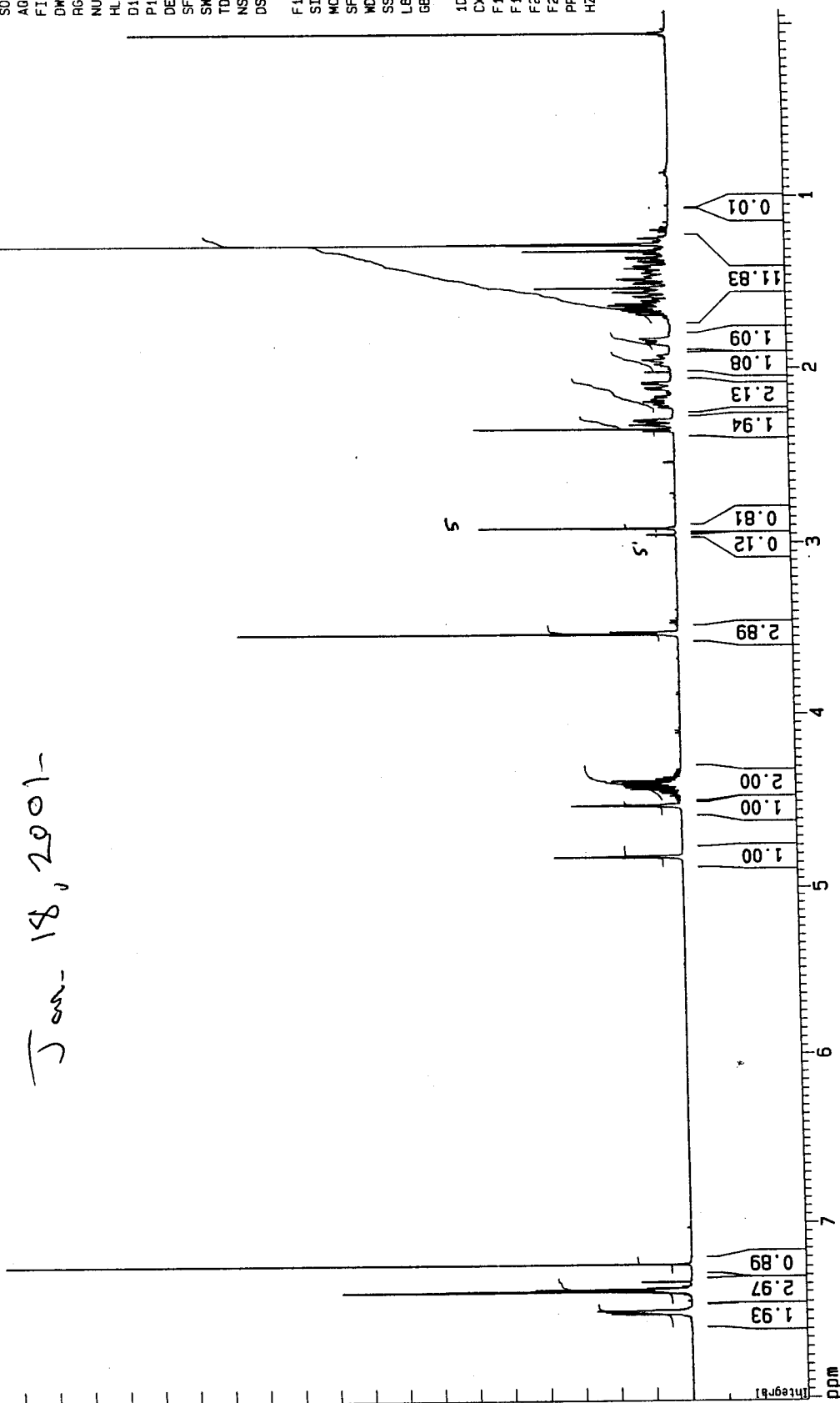
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2001 18, 2001



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 F1 4008.59 Hz
 F2P -0.084 ppm
 F2 -42.01 Hz
 PPMCM 0.36814 ppm/
 HZCM 184.11804 Hz/c



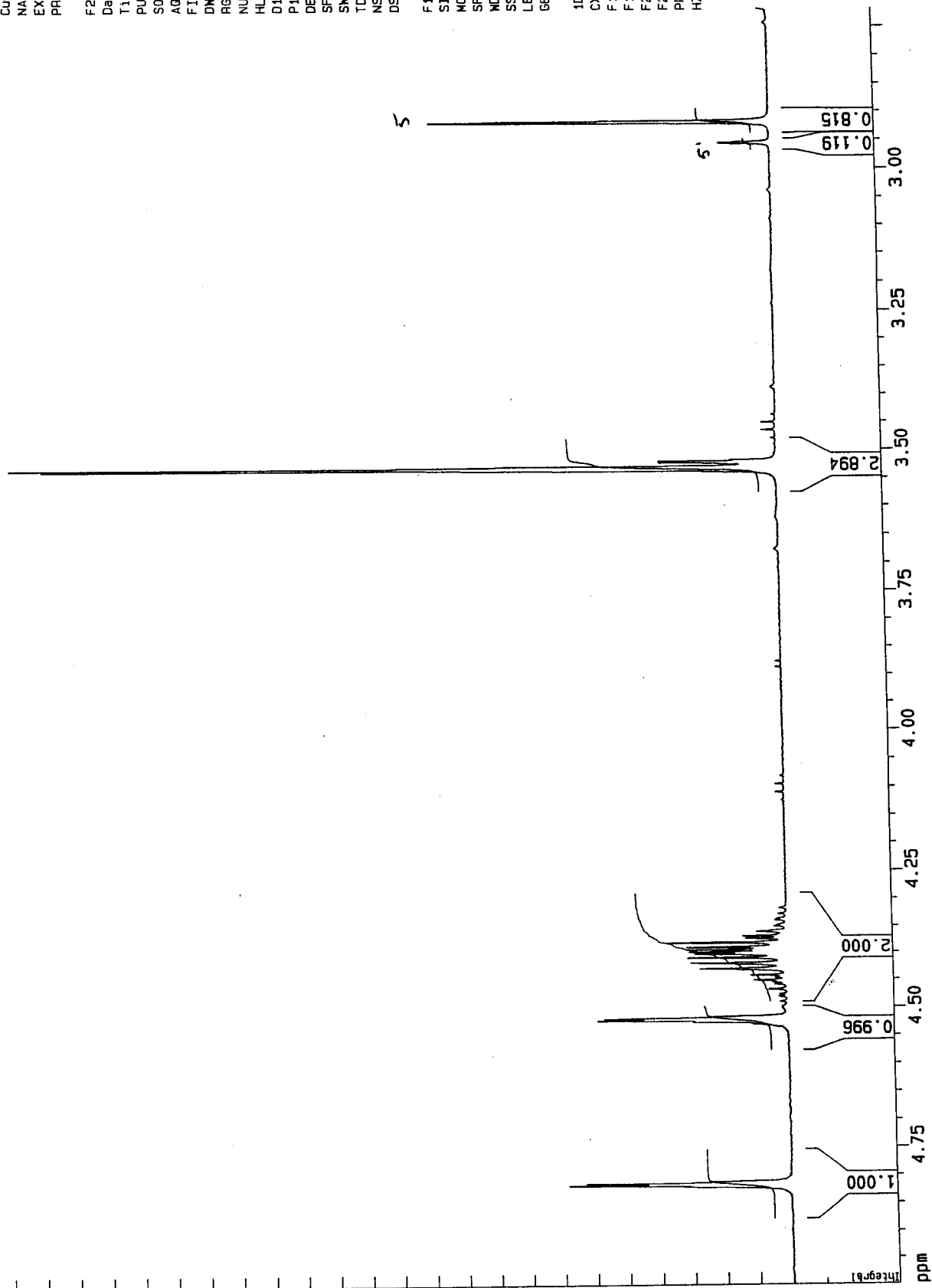
Jan-18, 2001

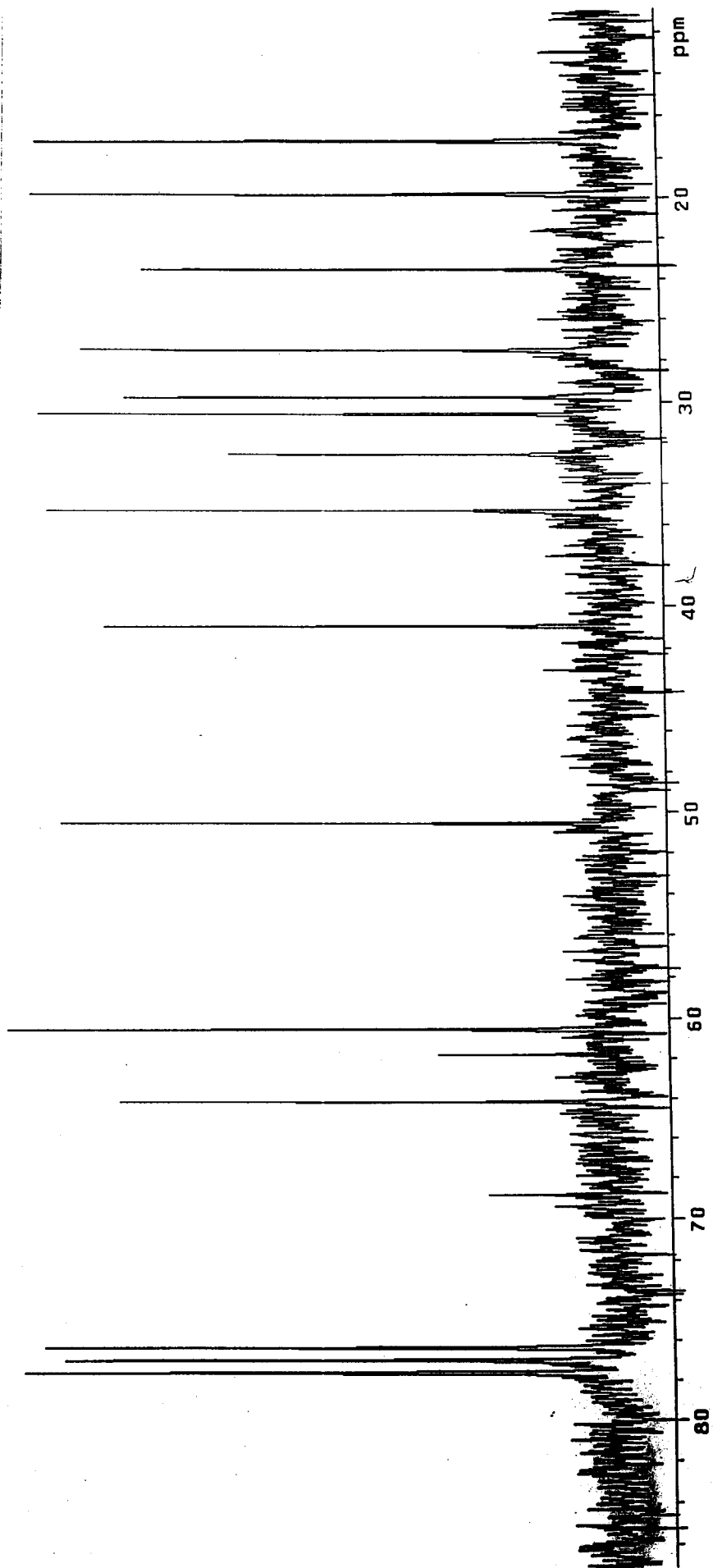
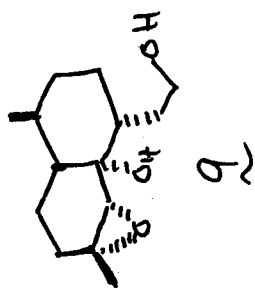
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NAME dhd_328
EXPNO 1
PROCNO 1

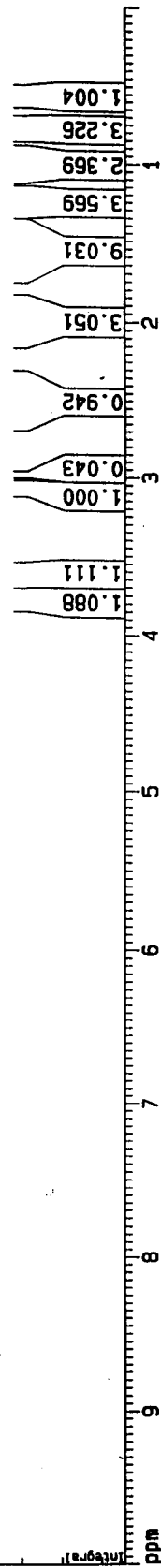
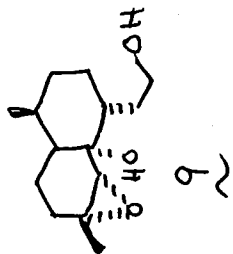
F2 - Acquisition Parameters
Date 20010116
Time 4.25
PULPROG zg
SOLVENT CDCl3
AG 4.5530805 sec
FIDRES 0.107456 Hz
AQ 71.0 usec
RG 512
NUCLEUS 1H
HL1 0 dB
D1 0.0100000 sec
P1 3.0 usec
DE 88.8 usec
SF01 500.1351707 MHz
SWH 7042.25 Hz
TD 65536
NS 16
DS 0

F1 - Processing Parameters
SI 32768
MC2 GF
SF 500.1354311 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0

1D NMR plot parameters
CX 22.00 cm
F1P 5.000 ppm
F1 2500.48 Hz
F2P 2.719 ppm
F2 1359.75 Hz
PPMCM 0.10368 ppm/
HZCM 51.85186 Hz/c



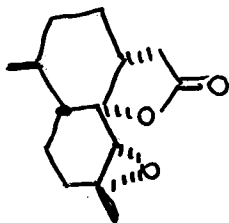




SOLVENT: CDCl₃
 AQ: 4.6530805 sec
 FIDRES: 0.107456 Hz
 DM: 71.0 usec
 RG: 2048
 NUCLEUS: ¹H
 PL1: 0 dB
 D1: 0.0100000 sec
 P1: 3.0 usec
 DE: 88.8 usec
 SF01: 500.1354311 MHz
 SHH: 7042.25 Hz
 TD: 65536
 NS: 16
 DS: 0

F1 - Processing parameters
 SI: 32768
 MC2: 0
 SF: 500.1354311 MHz
 WDW: EM
 SSB: 0
 LB: 0.00 Hz
 GB: 0

1D NMR plot parameters
 CX: 22.00 cm
 F1P: 3.874 ppm
 F1: 1937.46 Hz
 F2P: 2.911 ppm
 F2: 1455.67 Hz
 PPMCM: 0.04379 ppm/
 HZCM: 21.89938 Hz/c



13

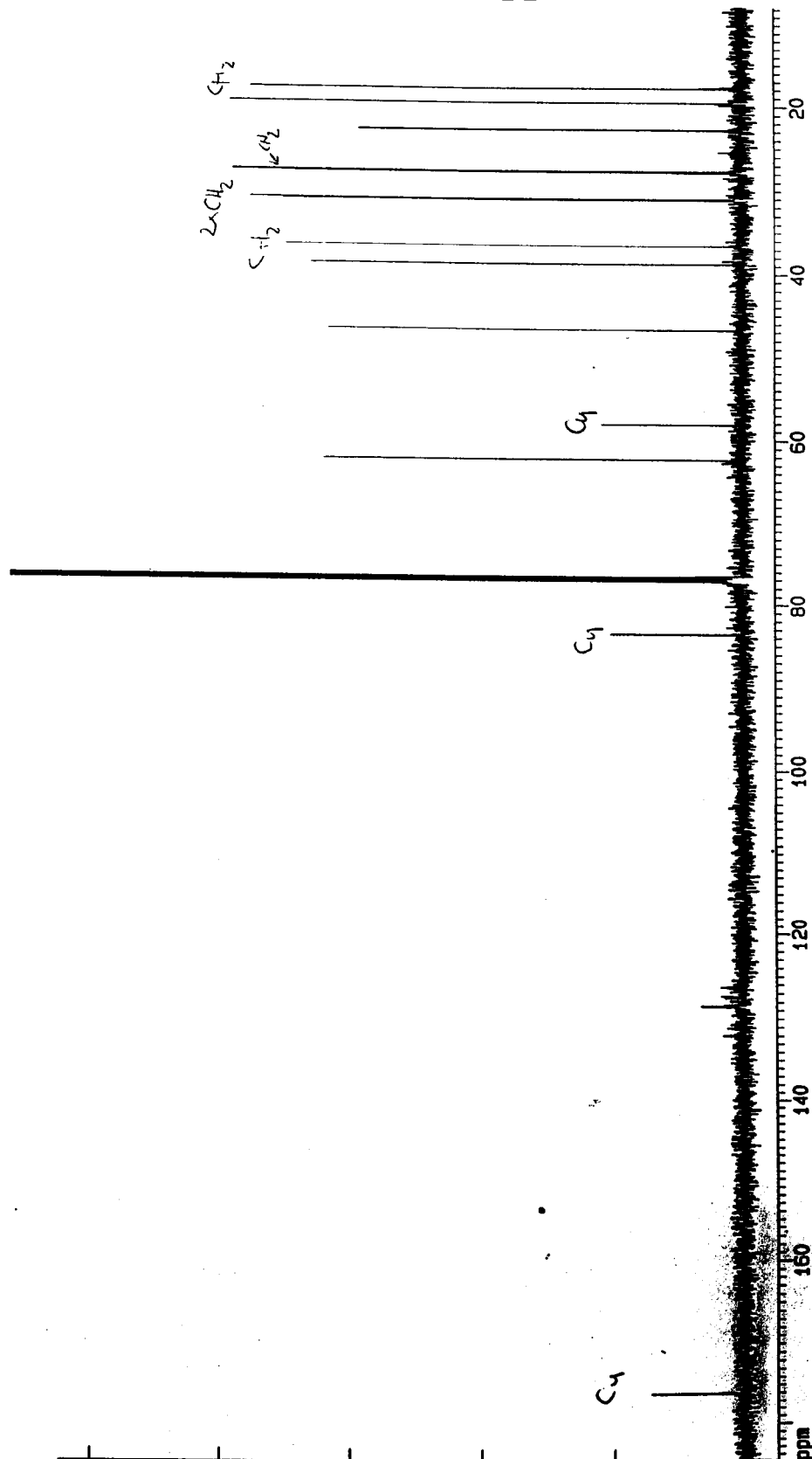
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1.0000000
P1 5.0
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SF01 125.772464
SMH 31250.00
TD 65536
NS 1654
OS 0

F1 - Processing parameter
SI 32768
MC2 GF
SF 125.7591571
MDW EM
SSB 0
LB 1.00
GB 0

1D NMR plot parameters
CX 22.00
F1P 184.790
F1 23239.01
F2P 7.704
F2 968.90
PPMCH 8.04934
HZCM 1012.27783

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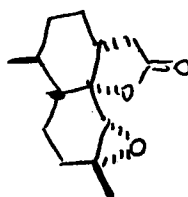


Current Data Parameters
NAME dnd_325
EXPNO 1
PROCNO 1

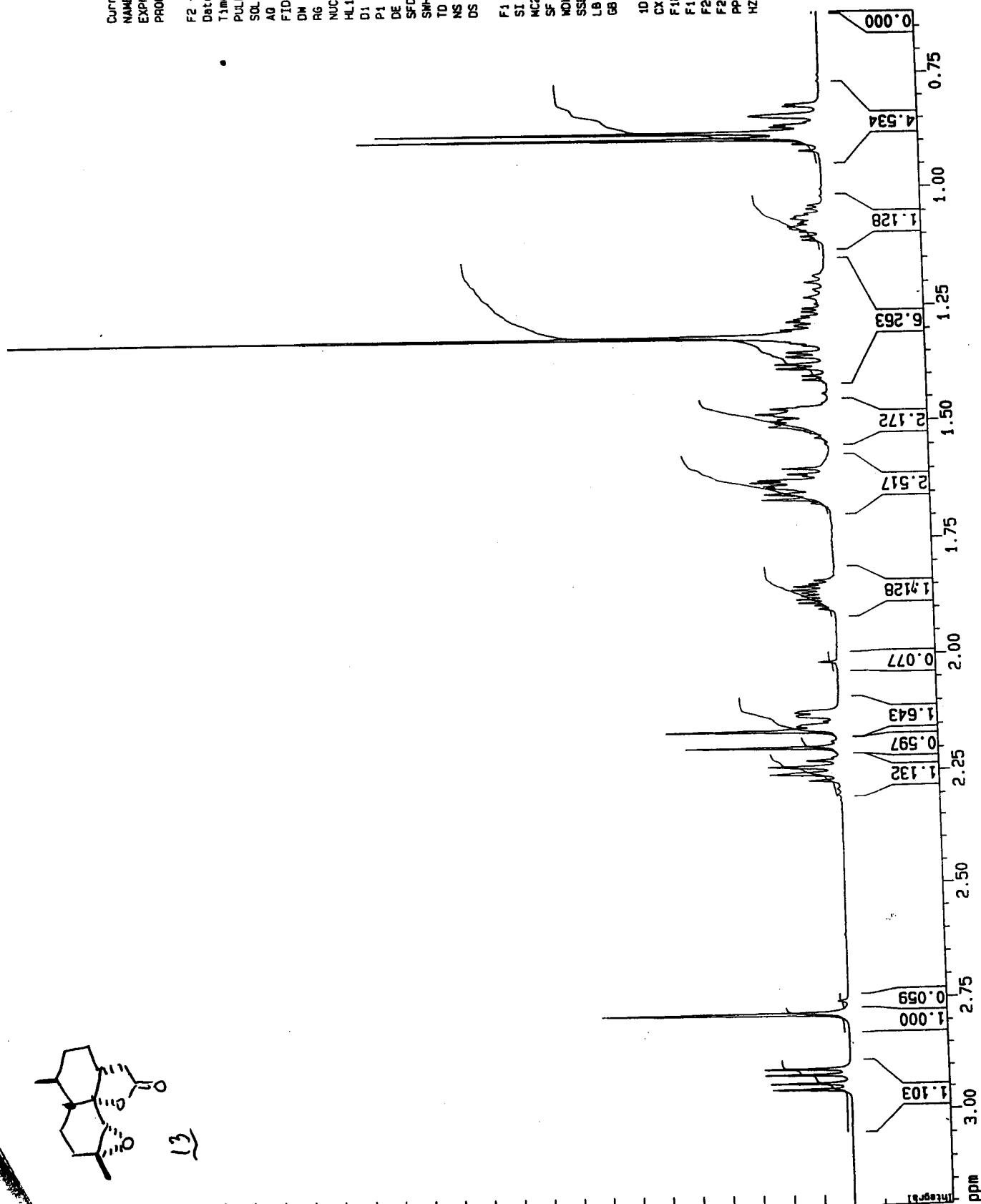
F2 - Acquisition Parameters
Date 20010112
Time 3.44
PULPROG zg
SOLVENT CDC13
AQ 4.6530805 sec
FIDRES 0.107456 Hz
AQ 71.0 usec
RG 4096
NUCLEUS 1H
HL1 0 dB
D1 0.010000 sec
P1 3.0 usec
DE 88.8 usec
SFO1 500.1381707 MHz
SWH 7042.25 Hz
TD 65536
NS 16
DS 0

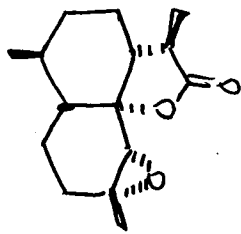
F1 - Processing parameters
SI 32768
MC2 GF
SF 500.1354311 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0

1D NMR plot parameters
CX 22.00 cm
F1P 3.207 ppm
F1 1603.97 Hz
F2P 0.817 ppm
F2 308.57 Hz
PPMCH 0.11773 ppm/
HZCM 58.88197 Hz/



13





14

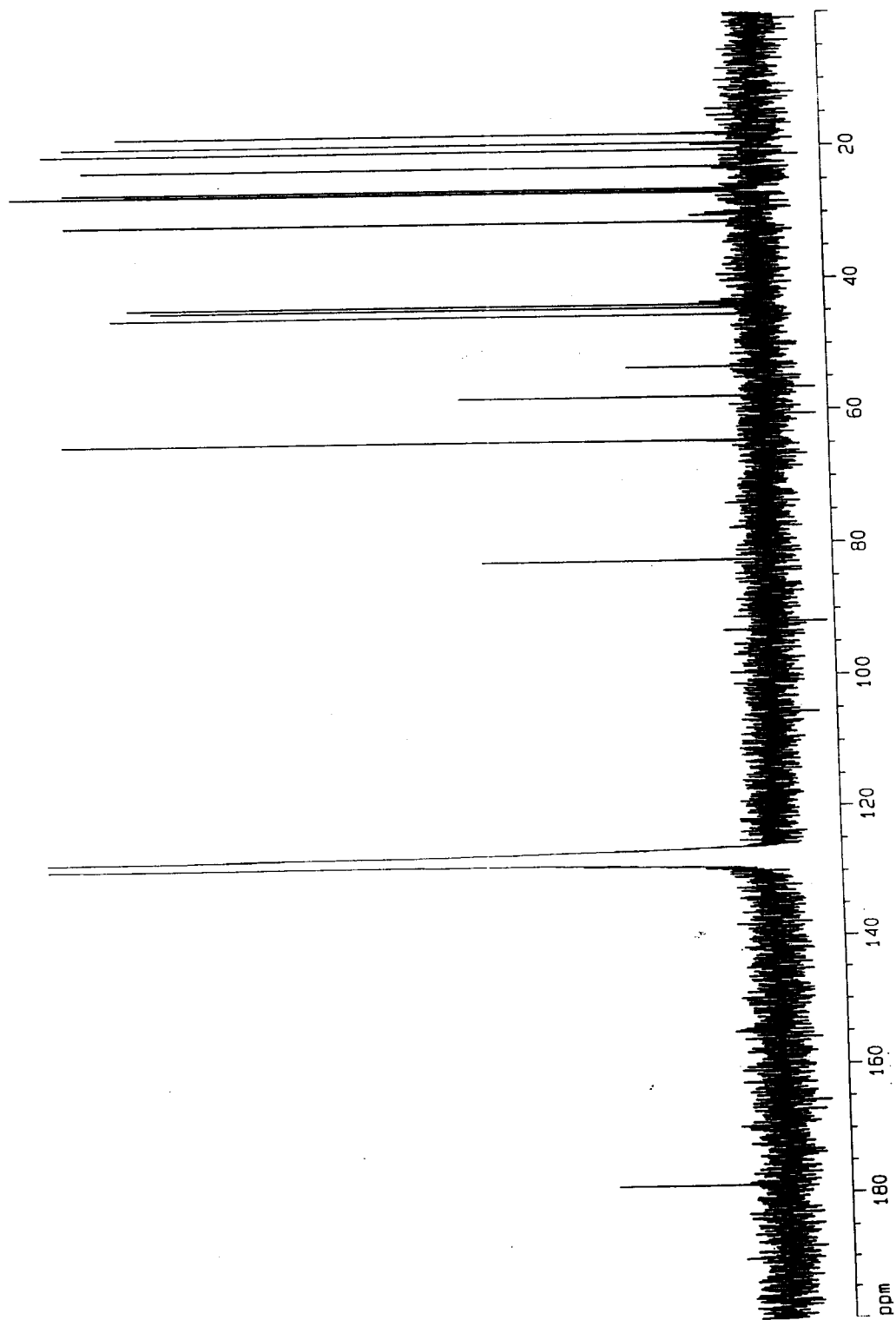
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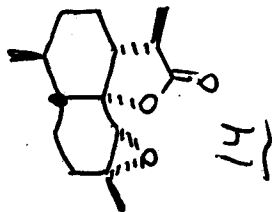
===== CHANNEL f1 =====
NUC1      13C
P1         5.10 usec
PL1        -6.00 dB
SF01       75.4752653 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      70.00 usec
PL2        -3.00 dB
PL12       15.00 dB
PL13       15.00 dB
SF02       300.1314860 MHz

F2 - Processing parameters
SI         65536
SF         75.4677190 MHz
MDM        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
CY         800.00 cm
F1P        200.000 ppm
F1         15093.54 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCH      10.00000 ppm/cm
HZCM       754.67719 Hz/cm
  
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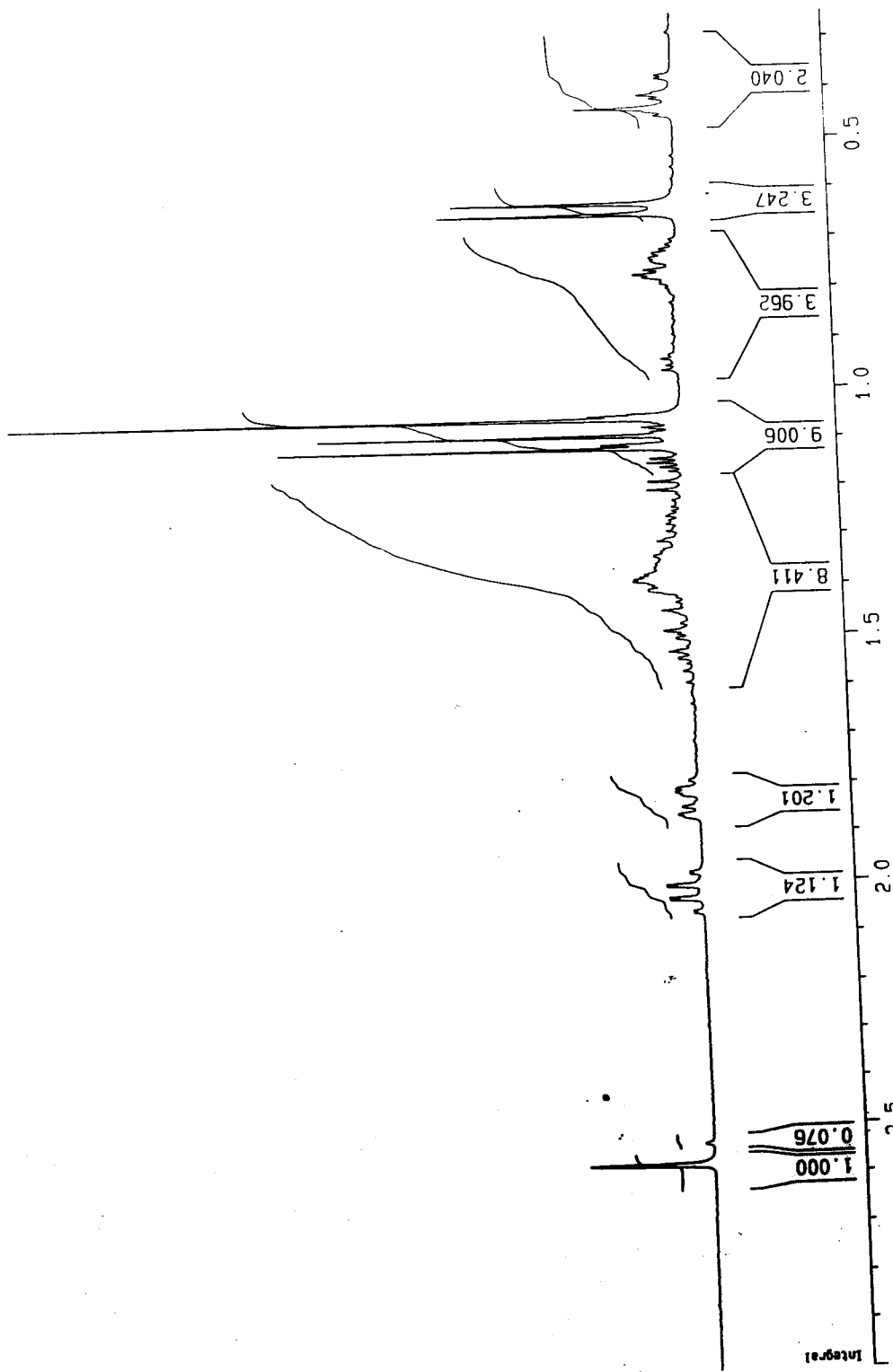


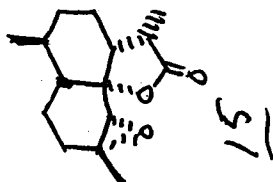
SOLVENT CDCl₃
 NS 16
 DS 0
 SWH 5081.301 Hz
 FIDRES 0.165407 Hz
 AQ 3.0228980 sec
 RG 128
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 -3.00 dB
 SF01 300.1319477 MHz

F2 - Processing parameters
 SI 65536
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 3.000 ppm
 F1 900.39 Hz
 F2P 0.250 ppm
 F2 75.03 Hz
 PPMCM 0.13750 ppm/cm
 HZCM 41.26788 Hz/cm

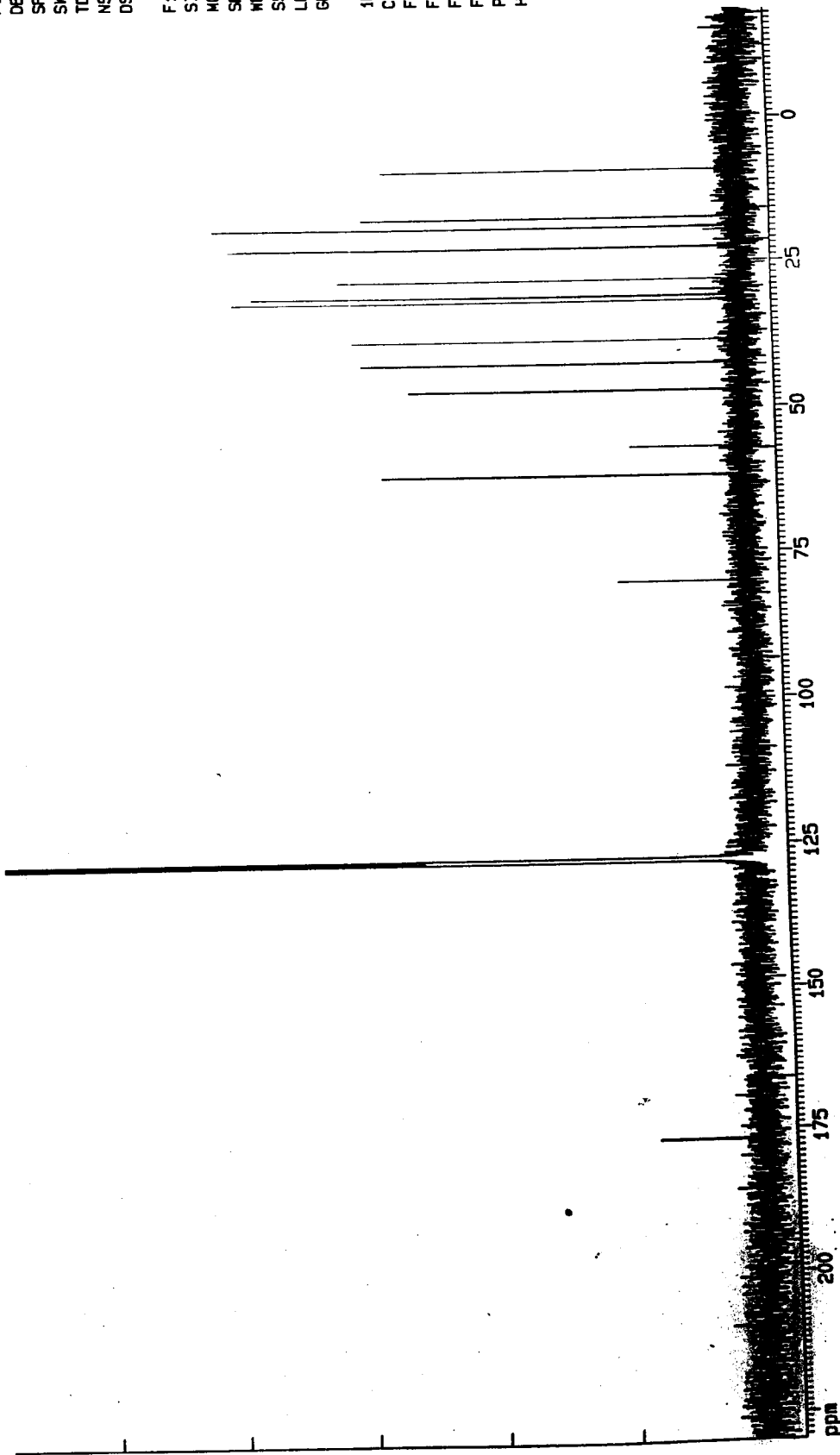


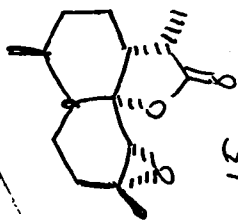


P1 3.0
DE 20.0
SF01 125.7724464
SMH 31250.00
TD 65536
NS 20000
DS 0

F1 - Processing param
SI 32768
MC2 OF
SF 125.7591571
WDW EM
SSB 0
LB 1.00
GB 0

1D NMR plot parameters
CX 22.00
F1P 229.918
F1 28914.29
F2P -18.573
F2 -2335.70
PPMCM 11.29504
HZCM 1420.45435





15

Current Data Parameters
NAME dhv_331
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20010120
Time 6.04

PULPROG zg
SOLVENT CDCl3
AQ 4.6530805 sec
FIDRES 0.107455 Hz
DQ 71.0 usec
RG 2048

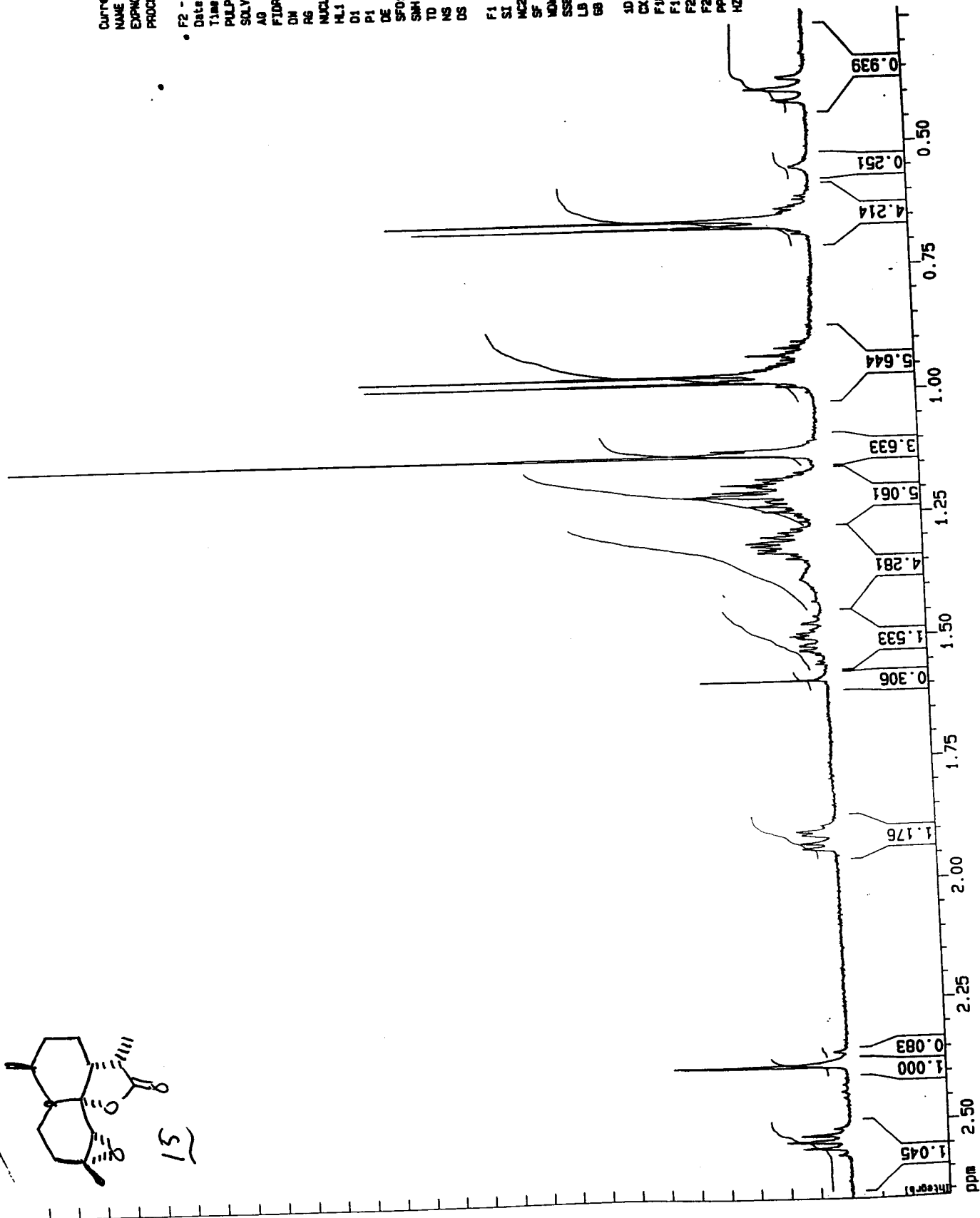
NUCLEUS 1H
HL1 0 dB
D1 0.0100000 sec
P1 3.0 usec
DE 88.8 usec

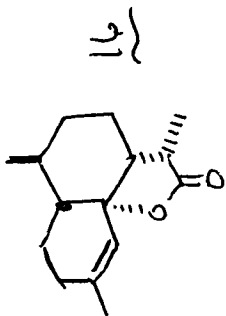
SFO1 500.1361707 MHz
SH 7042.25 Hz
TD 65536
NS 16
DS 0

F1 - Processing parameters

SI 32768
IC 0
SF 500.1354311 MHz
WDW EN
SSB 0
LB 0.00 Hz
GB 0

3D NMR plot parameters
CX 22.00 cm
F1P 2.853 ppm
F1 1331.74 Hz
F2P 0.232 ppm
F2 115.84 Hz
PPMCH 0.11051 ppm
HZCH 55.26948 Hz/K

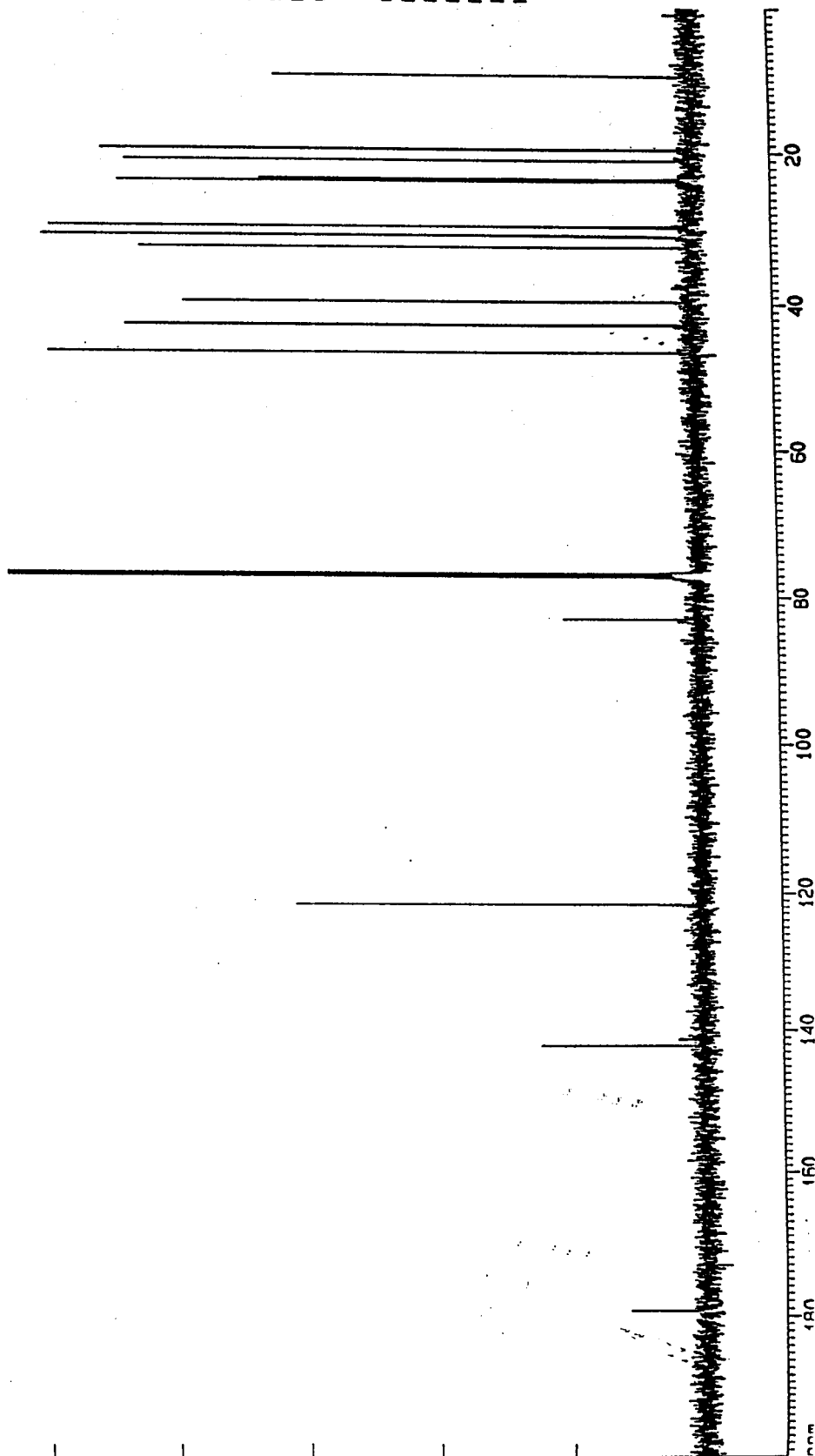




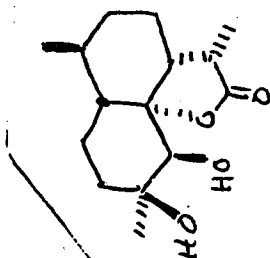
DE 20.0
SF01 125.772464
SNH 31250.00
TO 65536
NS 17000
DS 0

F1 - Processing paramet
SI 32758
MC2 QF
SF 125.7591571
NDW EM
SSB 0
LB 1.00
GB 0

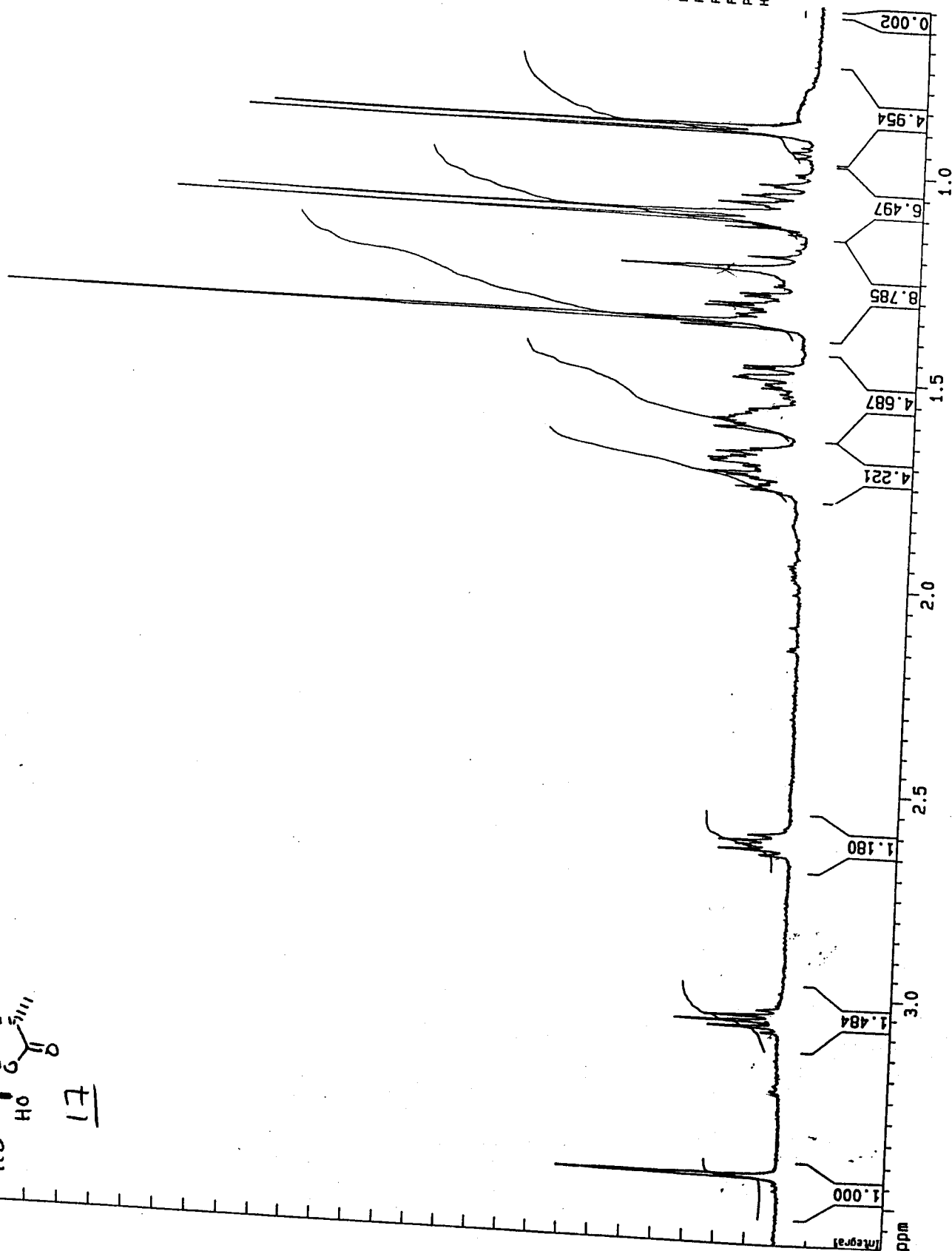
1D NMR plot parameters
CX 22.00
F1P 200.000
F1 25151.83
F2P 0.000
F2 0.00
PPMCH 9.09091
HZCH 1143.26501







17

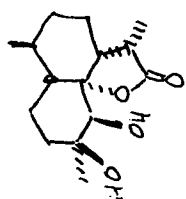


Current Data Parameters
 NAME dh0_3033
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20010114
 Time 3.48
 PULPROG zg
 SOLVENT CDCl3
 AQ 4.6530805 sec
 FIDRES 0.107456 Hz
 DN 71.0 user
 RG 16384
 NUCLEUS 1H
 HL1 0 dB
 D1 0.0100000 sec
 P1 3.0 user
 DE 88.8 user
 SF01 500.1381707 MHz
 SMH 7042.25 Hz
 TD 65536
 NS 64
 DS 0

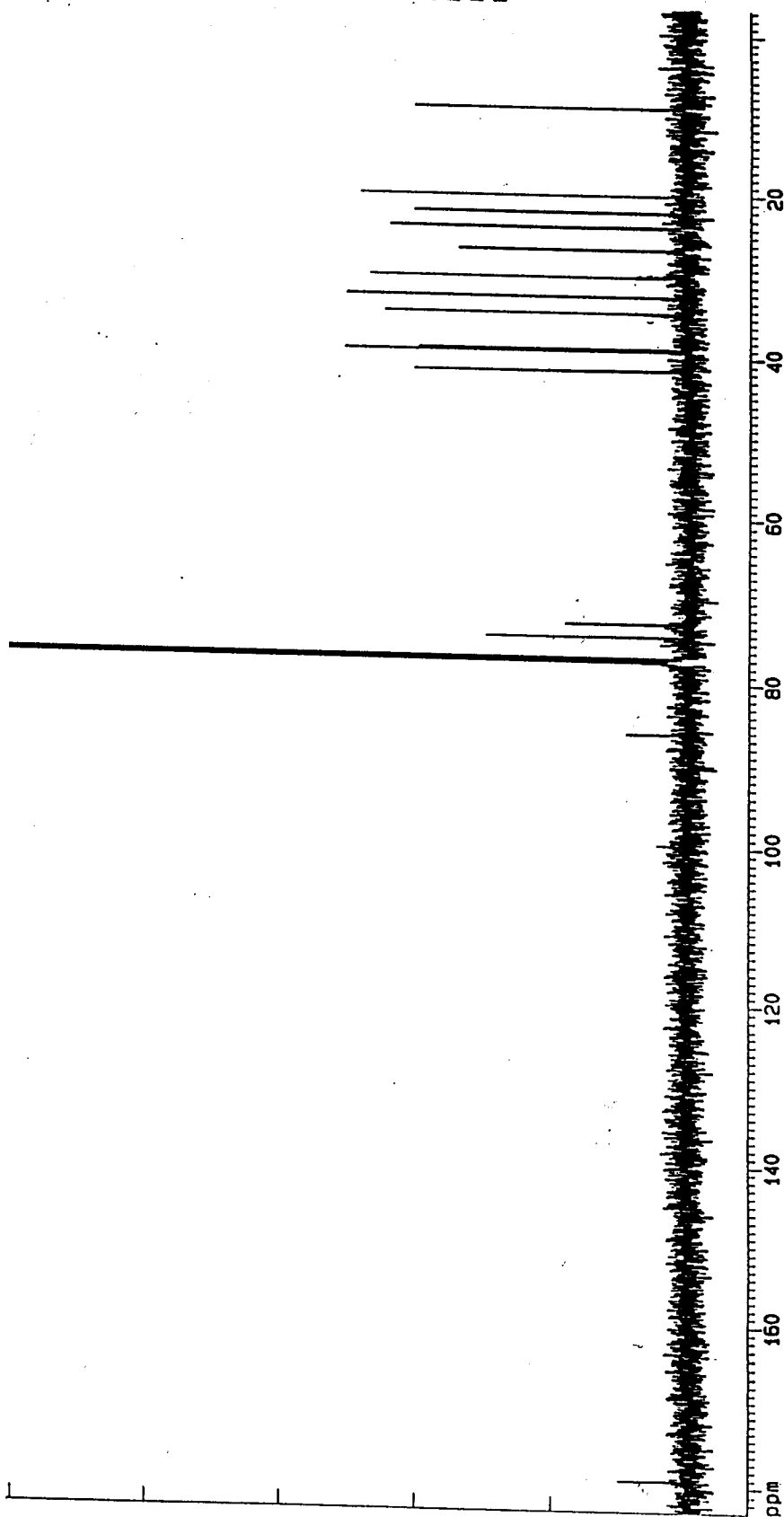
F1 - Processing parameters
 SI 32768
 MC2 OF
 SF 500.1354311 MHz
 HDW EM
 SSB 0
 LB 0.00 Hz
 GB 0

1D NMR plot parameters
 CX 22.00 cm
 F1P 3.595 ppm
 F1 1798.20 Hz
 F2P 0.596 ppm
 F2 298.30 Hz
 PP4M 0.13632 ppm/
 HZCM 68.17721 Hz/

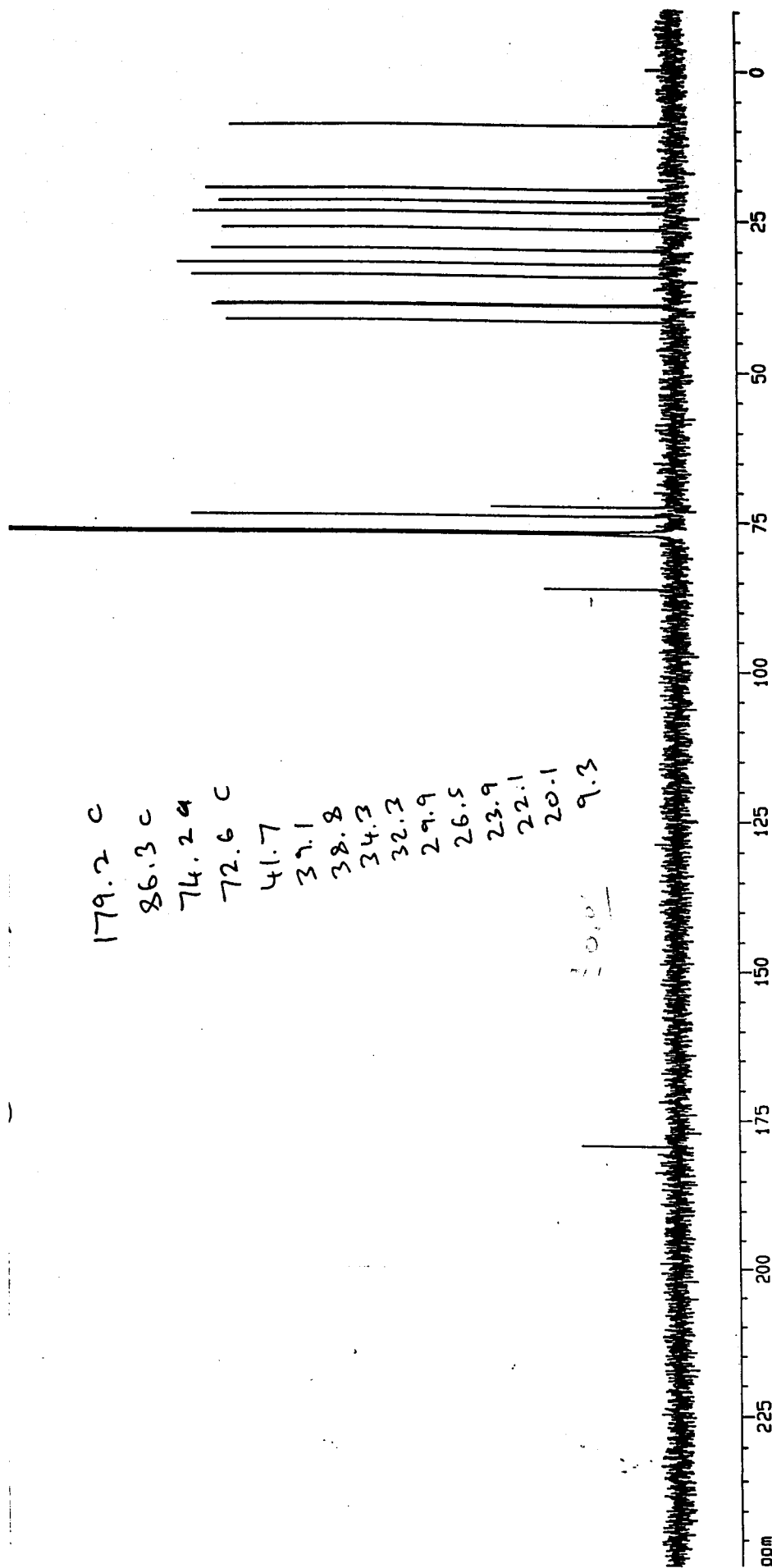
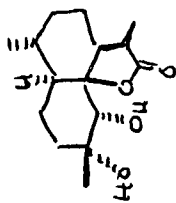


17

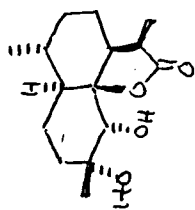
SWH	31250.00
TD	65536
NS	1792
DS	0
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SI	32768
MC2	GF
SF	125.7591480
WDW	EM
SSB	0
LB	1.00
GB	0
1D NMR plot parameters	
CX	22.00
F1P	182.978
F1	23011.20
F2P	-3.301
F2	-415.19
PPMCH	8.46725
HZCH	1054.83582



Natural (-)-AKTEANNIN M



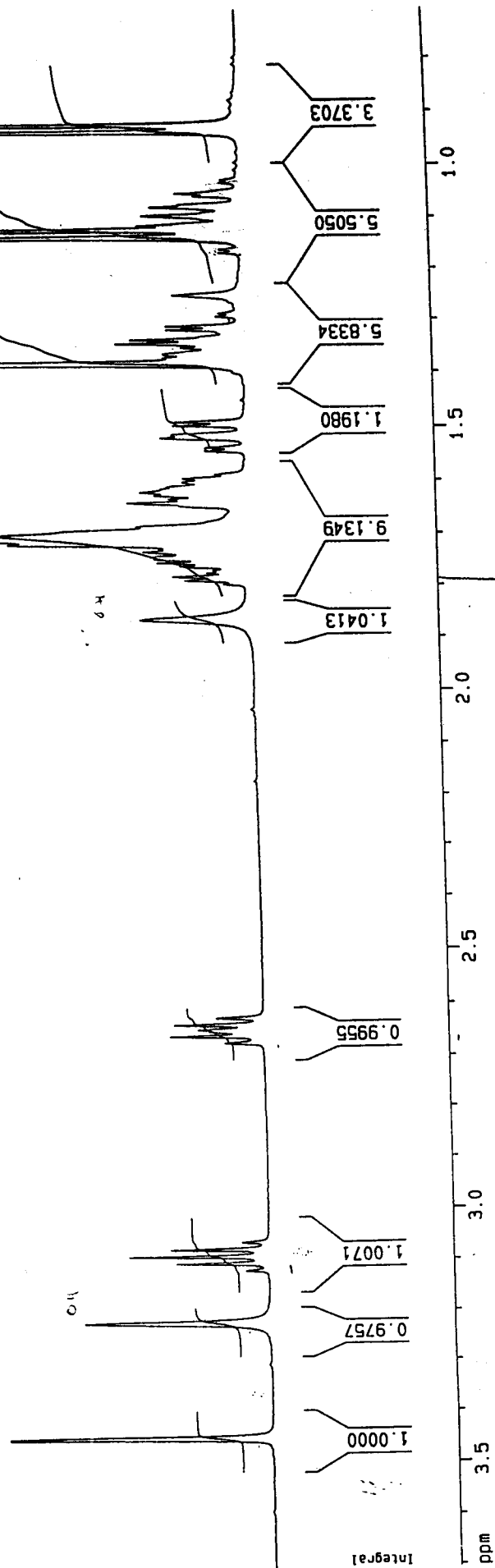
Natural ARTEANNIUM H



artemisia annua, 7.1mg shaken with 0.5ml Na₂CO₃

①

3.45 s (1H)
 3.22 s (1H) OH
 3.09 d q 6.8, 6.8 (4H)
 2.6 s ddd 10.9, 6.8, 6.1 (1H)
 1.87 br (1H) OH
 1.39 s (3H)
 1.13 d 7.1 (3H)
 0.93 d 6.6 (3H)



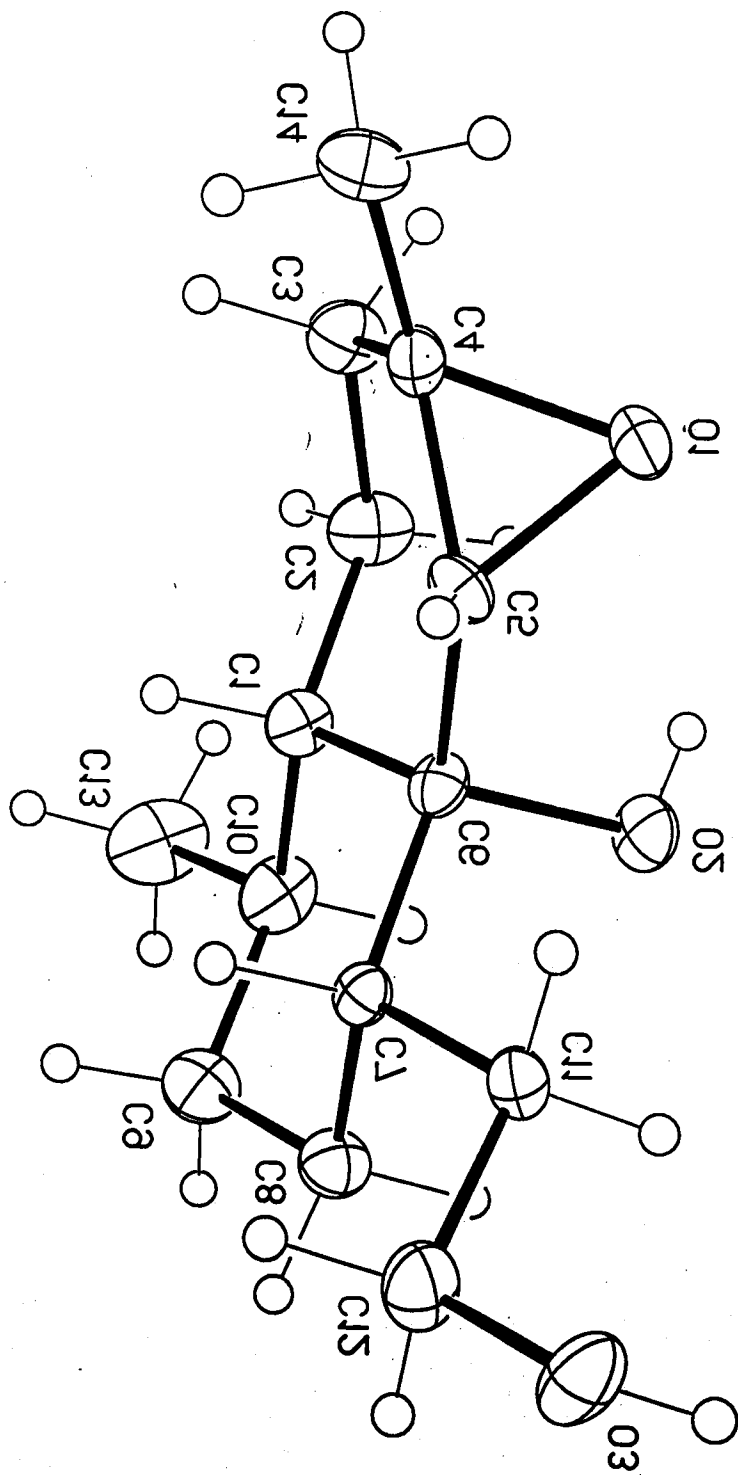


Table 1. Crystal data and structure refinement for lb004.

Identification code	lb004
Empirical formula	C14 H24 O3
Formula weight	240.33
Temperature	203(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 5.2738(8) Å alpha = 90 deg. b = 10.6132(16) Å beta = 90 deg. c = 24.204(4) Å gamma = 90 deg.
Volume	1354.7(4) Å ³
Z, Calculated density	4, 1.178 Mg/m ³
Absorption coefficient	0.081 mm ⁻¹
F(000)	528
Crystal size	0.10 x 0.02 x 0.02 mm
Theta range for data collection	1.68 to 20.78 deg.
Limiting indices	-5<=h<=5, 0<=k<=10, 0<=l<=24
Reflections collected / unique	10721 / 1409 [R(int) = 0.1449]
Completeness to theta = 20.78	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1409 / 0 / 155
Goodness-of-fit on F ²	1.032
Final R indices [I>2sigma(I)]	R1 = 0.0450, wR2 = 0.0890
R indices (all data)	R1 = 0.0698, wR2 = 0.0934
Absolute structure parameter	0(3)
Extinction coefficient	0.023(3)
Largest diff. peak and hole	0.151 and -0.220 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for lb004.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	2233(6)	6842(3)	9550(1)	38(1)
O(2)	4076(5)	8888(3)	9057(1)	37(1)
O(3)	-2387(6)	12070(3)	9876(1)	41(1)
C(1)	1989(9)	7902(4)	8266(2)	31(1)
C(2)	3366(10)	6659(4)	8335(2)	44(2)
C(3)	1773(11)	5703(4)	8658(2)	43(2)
C(4)	534(8)	6231(5)	9161(2)	29(1)
C(5)	428(9)	7606(4)	9240(2)	26(1)
C(6)	1635(8)	8536(4)	8836(2)	27(1)
C(7)	122(8)	9765(4)	8798(2)	23(1)
C(8)	1294(9)	10636(4)	8363(2)	36(1)
C(9)	1608(9)	9993(4)	7808(2)	42(1)
C(10)	3187(10)	8798(5)	7845(2)	42(1)
C(11)	-200(9)	10442(4)	9351(2)	30(1)
C(12)	-2078(10)	11503(4)	9345(2)	42(1)
C(13)	3575(11)	8229(5)	7280(2)	65(2)
C(14)	-1352(9)	5376(4)	9443(2)	44(1)

Table 3. Bond lengths [Å] and angles [deg] for lb004.

O(1)-C(4)	1.454(5)
O(1)-C(5)	1.458(5)
O(2)-C(6)	1.443(5)
O(3)-C(12)	1.427(4)
C(1)-C(2)	1.515(6)
C(1)-C(10)	1.529(6)
C(1)-C(6)	1.547(5)
C(2)-C(3)	1.532(6)
C(3)-C(4)	1.490(6)
C(4)-C(5)	1.473(6)
C(4)-C(14)	1.509(6)
C(5)-C(6)	1.529(5)
C(6)-C(7)	1.531(5)
C(7)-C(11)	1.529(5)
C(7)-C(8)	1.532(5)
C(8)-C(9)	1.514(5)
C(9)-C(10)	1.519(6)
C(10)-C(13)	1.511(5)
C(11)-C(12)	1.500(5)
C(4)-O(1)-C(5)	60.8(3)
C(2)-C(1)-C(10)	114.6(4)
C(2)-C(1)-C(6)	109.8(4)
C(10)-C(1)-C(6)	111.9(4)
C(1)-C(2)-C(3)	111.7(4)
C(4)-C(3)-C(2)	114.1(4)
O(1)-C(4)-C(5)	59.7(3)
O(1)-C(4)-C(3)	115.3(4)
C(5)-C(4)-C(3)	119.8(4)
O(1)-C(4)-C(14)	112.4(4)
C(5)-C(4)-C(14)	120.7(4)
C(3)-C(4)-C(14)	115.6(4)
O(1)-C(5)-C(4)	59.5(2)
O(1)-C(5)-C(6)	114.6(4)
C(4)-C(5)-C(6)	122.7(4)
O(2)-C(6)-C(5)	107.5(3)
O(2)-C(6)-C(7)	105.5(3)
C(5)-C(6)-C(7)	111.8(3)
O(2)-C(6)-C(1)	109.6(3)
C(5)-C(6)-C(1)	109.9(4)
C(7)-C(6)-C(1)	112.3(3)
C(11)-C(7)-C(6)	113.9(3)
C(11)-C(7)-C(8)	111.3(3)
C(6)-C(7)-C(8)	110.2(4)
C(9)-C(8)-C(7)	112.4(4)
C(8)-C(9)-C(10)	112.6(4)
C(13)-C(10)-C(9)	110.8(4)
C(13)-C(10)-C(1)	114.2(4)
C(9)-C(10)-C(1)	109.4(4)
C(12)-C(11)-C(7)	114.7(3)
O(3)-C(12)-C(11)	112.5(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for lb004.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	46(2)	33(2)	34(2)	2(2)	-16(2)	2(2)
O(2)	21(2)	43(2)	47(2)	-3(2)	-3(2)	3(2)
O(3)	34(2)	41(2)	47(2)	-21(2)	1(2)	2(2)
C(1)	34(3)	31(3)	27(3)	-2(2)	3(3)	0(3)
C(2)	55(4)	38(4)	39(3)	-4(3)	13(3)	1(3)
C(3)	54(4)	37(3)	36(3)	-4(3)	7(3)	6(3)
C(4)	28(3)	33(3)	26(3)	-1(2)	-4(3)	-5(3)
C(5)	23(3)	31(3)	24(3)	6(2)	5(3)	7(3)
C(6)	19(3)	33(3)	29(3)	0(2)	1(2)	-8(3)
C(7)	18(3)	24(3)	26(3)	0(2)	2(2)	-3(3)
C(8)	42(4)	34(3)	32(3)	-2(2)	2(3)	-2(3)
C(9)	47(4)	44(4)	34(3)	10(3)	6(3)	-5(3)
C(10)	57(4)	46(3)	24(3)	-4(2)	8(3)	-8(3)
C(11)	33(3)	29(3)	28(3)	1(2)	-5(2)	0(3)
C(12)	41(3)	48(4)	37(3)	-16(3)	-6(3)	8(3)
C(13)	90(5)	65(4)	40(3)	-5(3)	24(3)	-13(4)
C(14)	54(4)	39(3)	39(3)	12(3)	6(3)	-2(3)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for lb004.

	x	y	z	U(eq)
H(2B)	4989	8253	9081	56
H(3C)	-1001	12344	9985	61
H(1A)	271	7700	8127	37
H(2A)	4968	6803	8530	53
H(2C)	3765	6313	7970	53
H(3A)	459	5365	8413	51
H(3B)	2866	5000	8769	51
H(5A)	-1045	7925	9452	31
H(7A)	-1597	9542	8666	27
H(8A)	211	11379	8317	43
H(8B)	2956	10923	8493	43
H(9A)	2421	10578	7550	50
H(9B)	-70	9783	7661	50
H(10A)	4877	9039	7989	51
H(11A)	-733	9825	9629	36
H(11B)	1451	10776	9466	36
H(12A)	-1512	12145	9082	51
H(12B)	-3721	11183	9218	51
H(13A)	4327	8852	7036	97
H(13B)	1953	7963	7131	97
H(13C)	4694	7507	7308	97
H(14A)	-972	4506	9352	66
H(14B)	-3051	5581	9318	66
H(14C)	-1244	5492	9840	66

Table 6. Torsion angles [deg] for lb004.

C(10)-C(1)-C(2)-C(3)	-167.3(4)
C(6)-C(1)-C(2)-C(3)	65.8(5)
C(1)-C(2)-C(3)-C(4)	-45.1(6)
C(5)-O(1)-C(4)-C(3)	111.1(5)
C(5)-O(1)-C(4)-C(14)	-113.5(4)
C(2)-C(3)-C(4)-O(1)	-54.7(6)
C(2)-C(3)-C(4)-C(5)	13.4(6)
C(2)-C(3)-C(4)-C(14)	171.4(4)
C(4)-O(1)-C(5)-C(6)	-114.8(4)
C(3)-C(4)-C(5)-O(1)	-103.6(5)
C(14)-C(4)-C(5)-O(1)	99.6(4)
O(1)-C(4)-C(5)-C(6)	101.3(4)
C(3)-C(4)-C(5)-C(6)	-2.4(7)
C(14)-C(4)-C(5)-C(6)	-159.2(4)
O(1)-C(5)-C(6)-O(2)	-29.5(5)
C(4)-C(5)-C(6)-O(2)	-97.8(5)
O(1)-C(5)-C(6)-C(7)	-144.8(3)
C(4)-C(5)-C(6)-C(7)	146.9(4)
O(1)-C(5)-C(6)-C(1)	89.8(4)
C(4)-C(5)-C(6)-C(1)	21.5(6)
C(2)-C(1)-C(6)-O(2)	66.3(5)
C(10)-C(1)-C(6)-O(2)	-62.2(5)
C(2)-C(1)-C(6)-C(5)	-51.7(5)
C(10)-C(1)-C(6)-C(5)	179.8(4)
C(2)-C(1)-C(6)-C(7)	-176.8(4)
C(10)-C(1)-C(6)-C(7)	54.7(5)
O(2)-C(6)-C(7)-C(11)	-58.9(4)
C(5)-C(6)-C(7)-C(11)	57.7(5)
C(1)-C(6)-C(7)-C(11)	-178.2(4)
O(2)-C(6)-C(7)-C(8)	67.0(4)
C(5)-C(6)-C(7)-C(8)	-176.4(3)
C(1)-C(6)-C(7)-C(8)	-52.3(5)
C(11)-C(7)-C(8)-C(9)	-179.4(4)
C(6)-C(7)-C(8)-C(9)	53.3(5)
C(7)-C(8)-C(9)-C(10)	-56.6(5)
C(8)-C(9)-C(10)-C(13)	-176.8(4)
C(8)-C(9)-C(10)-C(1)	56.4(5)
C(2)-C(1)-C(10)-C(13)	54.2(6)
C(6)-C(1)-C(10)-C(13)	-179.9(4)
C(2)-C(1)-C(10)-C(9)	179.0(4)
C(6)-C(1)-C(10)-C(9)	-55.2(5)
C(6)-C(7)-C(11)-C(12)	-168.7(4)
C(8)-C(7)-C(11)-C(12)	66.0(5)
C(7)-C(11)-C(12)-O(3)	177.6(4)

Symmetry transformations used to generate equivalent atoms: