

01 006404 D
08-04 23

SUPPLEMENTARY MATERIAL

Experimental Procedure

All reactions were carried out under a nitrogen atmosphere. Common solvents were purified before use. Tetrahydrofuran (THF) and diethyl ether (Et₂O) were purified by distillation from potassium-benzophenone ketyl. Dichloromethane (CH₂Cl₂), benzene, and toluene were distilled from calcium hydride. All reagents were reagent grade and purified when necessary. NaHMDS was used from newly opened 100 mL bottles, purchased from Aldrich, Inc. Reactions were monitored by thin layer chromatography (TLC) using 250 mm Whatman precoated silica gel plates. Flash column chromatography was performed over Fisher or EM Science Laboratories silica gel (230-400 mesh). Melting points were measured on a Thomas Hoover capillary melting point apparatus and are uncorrected. Carbon and proton NMR spectra were recorded on Brüker DRX-500 or DRX-400 spectrometer. ¹H NMR chemical shifts are reported as δ values (ppm) relative to internal tetramethylsilane and splitting patterns are designated as: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Coupling constants are given in hertz (Hz). Infrared spectra (IR) were recorded with Nicolet 20 SXB FTIR spectrometer and are reported in reciprocal centimeter (cm⁻¹). Mass spectra were obtained on either a Kratos MS-30 or a Kratos VG 70-250S mass spectrometer at The Ohio State University Campus Chemical Instrumentation Center.

Materials

Dienes were made using our reported procedure¹ and redistilled prior to use. Aldehydes **2a-i** and methyl pyruvate were purchased from Aldrich and distilled prior to use. Imines **11a** and **11b** were made by known procedures.²

General procedure for the hetero-Diels-Alder reaction of diene with aldehydes

To a 25 mL flame-dried flask under a nitrogen atmosphere were added freshly distilled diene **1** (227 mg, 1 mmol, 1.0 equiv.) and 2 mL CHCl₃. Aldehyde **2** (1.5 mmol, 1.5 equiv.) was added dropwise via a gas-tight syringe. The reaction mixture was stirred at room temperature for indicated time or until the diene was fully consumed, as monitored by either TLC or NMR. The reaction was then diluted with 15 mL CH₂Cl₂, the yellow solution cooled to -78 °C, and treated dropwise with 142 μL acetyl chloride (2 mmol, 2.0 equiv.). After stirring for ca. 30 min saturated sodium bicarbonate was added. The organic layer was separated, and the water phase was diluted with 15 mL water and extracted twice with CH₂Cl₂. The combined organic phase was dried with magnesium sulfate, filtered, and concentrated to give yellow oil, which was subjected to flash chromatography to afford the desired dihydropyrone **7**.

2-phenyl-2,3-dihydro-pyran-4-one (**7a**).

¹H NMR (500 MHz, CDCl₃, ppm) δ 7.59 (d, *J*=6 Hz, 1H), 7.43 (m, 5H), 5.54 (dd, *J*₁=1 Hz *J*₂=6 Hz, 1H), 5.43 (dd, *J*₁=4 Hz *J*₂=14 Hz, 1H), 2.92 (dd, *J*₁=14 Hz *J*₂=17 Hz, 1H), 2.67 (ddd, *J*₁=1 Hz *J*₂=4 Hz, *J*₃=17 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃, ppm) δ

192.2, 163.2, 137.8, 128.9, 128.8, 126.1, 107.4, 81.1, 43.4. IR (Neat, cm^{-1}) 1676, 1595, 1403, 1269, 1228.

2-*o*-nitrophenyl-2,3-dihydro-pyran-4-one (7b).

^1H NMR (500 MHz, CDCl_3 , ppm) d 8.06 (dd, $J_1=1$ Hz $J_2=8$ Hz, 1H), 7.85 (dd, $J_1=1$ Hz $J_2=8$ Hz, 1H), 7.75 (dt, $J_1=1$ Hz $J_2=8$ Hz, 1H), 7.57 (dt, $J_1=1$ Hz $J_2=8$ Hz, 1H), 7.49 (d, $J=6$ Hz, 1H), 6.04 (dd, $J_1=3$ Hz $J_2=14$ Hz, 1H), 5.59 (dd, $J_1=1$ Hz $J_2=6$ Hz, 1H), 2.97 (ddd, $J_1=1$ Hz $J_2=3$ Hz $J_3=17$ Hz, 1H), 2.78 (dd, $J_1=14$ Hz $J_2=17$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3 , ppm) d 190.8, 162.5, 134.0, 133.8, 129.5, 128.0, 124.9, 107.9, 77.1, 43.1. IR (Neat, cm^{-1}) 1684, 1523, 1344, 1269. HRMS m/z [M^+] calcd for $\text{C}_{11}\text{H}_9\text{NO}_4$ 219.0532, found 219.0533.

2-*p*-methoxyphenyl-2,3-dihydro-pyran-4-one (7c).

^1H NMR (500 MHz, CDCl_3 , ppm) d 7.46 (d, $J=6$ Hz, 1H), 7.33 (d, $J=9$ Hz, 2H), 6.94 (d, $J=11$ Hz, 2H), 5.51 (d, $J=6$ Hz, 1H), 5.37 (dd, $J_1=3$ Hz $J_2=14$ Hz, 1H), 3.83 (s, 3H), 2.93 (dd, $J_1=15$ Hz $J_2=17$ Hz, 1H), 2.62 (dd, $J_1=3$ Hz $J_2=17$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3 , ppm) d 192.4, 163.3, 160.1, 129.8, 127.7, 114.2, 107.2, 80.9, 55.3, 43.1. IR (Neat, cm^{-1}) 1676, 1594, 1518, 1253. mp. 48-50 $^\circ\text{C}$.

2-furfuryl-2,3-dihydro-pyran-4-one (7d).

^1H NMR (500 MHz, CDCl_3 , ppm) d 7.48 (dd, $J_1=1$ Hz $J_2=2$ Hz, 1H), 7.38 (d, $J=6$ Hz, 1H), 6.46 (d, $J=4$ Hz, 1H), 6.41 (dd, $J_1=2$ Hz $J_2=3$ Hz, 1H), 5.51 (dd, $J_1=1$ Hz $J_2=6$ Hz, 1H), 5.48 (dd, $J_1=4$ Hz $J_2=13$ Hz, 1H), 3.10 (dd, $J_1=13$ Hz $J_2=17$ Hz, 1H), 2.74 (ddd, $J_1=1$ Hz $J_2=4$ Hz $J_3=17$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3 , ppm) d 191.3, 162.4,

150.0, 143.6, 110.6, 109.7, 107.4, 73.5, 39.5. IR (Neat, cm^{-1}) 1677, 1596, 1403, 1272, 1209.

2-*n*-pentyl-2,3-dihydro-pyran-4-one (7e).

^1H NMR (400 MHz, CDCl_3 , ppm) d 7.36 (d, $J=6$ Hz, 1H), 5.40 (dd, $J_1=1$ Hz $J_2=6$ Hz, 1H), 4.39 (m, 1H), 2.51 (dd, $J_1=13$ Hz $J_2=17$ Hz, 1H), 2.45 (ddd, $J_1=1$ Hz $J_2=4$ Hz $J_3=17$ Hz, 1H), 1.80 (m, 1H), 1.66 (m, 1H), 1.40 (m, 6H), 0.90 (t, $J=7$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3 , ppm) d 192.9, 163.4, 106.9, 79.6, 41.8, 34.3, 31.4, 24.4, 22.5, 14.0. IR (Neat, cm^{-1}) 2932, 1680, 1596, 1272.

2-benzyl-2,3-dihydro-pyran-4-one (7f).

^1H NMR (500 MHz, CDCl_3 , ppm) d 7.33 (m, 3H), 7.28 (m, 1H), 7.22 (m, 2H), 5.40 (dd, $J_1=1$ Hz $J_2=6$ Hz, 1H), 4.64 (m, 1H), 3.12 (dd, $J_1=7$ Hz $J_2=14$ Hz, 1H), 3.00 (dd, $J_1=6$ Hz $J_2=14$ Hz, 1H), 2.54 (dd, $J_1=14$ Hz $J_2=17$ Hz, 1H), 2.46 (ddd, $J_1=1$ Hz $J_2=3$ Hz $J_3=17$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3 , ppm) d 192.4, 163.1, 135.8, 129.5, 128.6, 127.0, 107.1, 79.8, 41.2, 40.7. IR (Neat, cm^{-1}) 1675, 1595, 1405, 1276. HRMS m/z [M^+] calcd for 188.0838, found 188.0838.

2-(1'-methyl)-butyl-2,3-dihydro-pyran-4-one (7g).

The product was identified as a mixture of diastereomers (cis and trans) with 1.3/1 ratio. ^1H NMR (500 MHz, CDCl_3 , ppm) d 7.38 (d, $J=6$ Hz, 1H, trans and cis overlap); 5.40 (d, $J=6$ Hz, 1H, trans and cis overlap); 4.30-4.24 (m, 2H total, cis + trans); 2.63-2.52 (m, 2H total, cis + trans); 2.36 (m, 2H total, cis + trans); 1.87 (m, 1H total); 1.53-1.17 (m; 7H total; cis + trans); 0.98 (d, $J=7$ Hz, 2H); 0.96 (d, $J=7$ Hz); 0.92 (t, $J=7$ Hz,

3H, cis and trans overlap). ^{13}C NMR (125 MHz, CDCl_3 , ppm) d 193.3; 193.2; 163.61; 163.58; 106.74; 106.72; 83.2; 82.9; 39.0; 38.1; 36.3; 36.1; 34.2; 33.9; 20.0; 19.9; 14.5; 14.3; 14.1 (some peaks overlap). IR (Neat, cm^{-1}) 2960; 2933; 2874; 1686; 1678; 1405; 1277; 1224; 1039 (some peaks overlap).

2-3'-cyclohexenyl-2,3-dihydro-pyran-4-one (7h).

The product was identified as a mixture of diastereomers with 1.2/1 ratio. ^1H NMR (400 MHz, CDCl_3 , ppm) d 7.39 (d, $J=6$ Hz, 1H, cis and trans overlap); 5.68 (m, 4H total, cis + trans); 5.42 (dd, $J_1=1$ Hz $J_2=6$ Hz, 1H, cis and trans overlap); 4.32-4.21 (m, 2H total, cis + trans); 2.61 (dd, $J_1=14$ Hz $J_2=16$ Hz, 1H); 2.57 (dd, $J_1=14$ Hz $J_2=16$ Hz, 1H); 2.47 (ddd, $J_1=1$ Hz $J_2=3$ Hz $J_3=16$ Hz, 1H); 2.44 (ddd, $J_1=1$ Hz $J_2=3$ Hz $J_3=16$ Hz, 1H); 2.25-1.72 (m, 12H total, cis + trans); 1.49-1.25 (m, 2H total, cis + trans). ^{13}C NMR (100 MHz, CDCl_3 , ppm) d 192.5; 163.3; 163.1; 127.0; 126.6; 125.1; 124.8; 106.6; 82.5; 82.3; 38.95; 38.92; 37.2; 37.1; 26.5; 26.3; 24.4; 24.0; 23.6 (some peaks overlap). IR (Neat, cm^{-1}) 3024; 2917; 2840; 1679; 1675; 1406; 1274; 1229; 1037 (some peaks overlap).

2-*t*-butyl-2,3-dihydro-pyran-4-one (7i).

^1H NMR (500 MHz, CDCl_3 , ppm) d 7.41 (dd, $J_1 \sim 0$ Hz $J_2=4$ Hz, 1H), 5.40 (dd, $J_1=1$ Hz $J_2=5$ Hz, 1H), 4.03 (dd, $J_1=3$ Hz $J_2=15$ Hz, 1H), 2.53 (dd, $J_1=15$ Hz $J_2=16$ Hz, 1H), 2.39 (ddd, $J_1=1$ Hz $J_2=3$ Hz, $J_3=16$ Hz, 1H), 1.00 (s, 9H). ^{13}C NMR (125 MHz, CDCl_3 , ppm) d 193.6, 163.8, 106.6, 86.9, 37.2, 33.8, 25.4. IR (Neat, cm^{-1}) 2963, 1679, 1596, 1405, 1284, 1272. HRMS m/z [M^+] calcd for 154.0994, found 154.0992.

HDA reaction of diene with methyl pyruvate

To a 25 mL flame-dried flask maintained under a nitrogen atmosphere was added 227 mg (1 mmol, 1.0 equiv.) freshly distilled diene **1a** and 2 mL CH₂Cl₂. The reaction solution was cooled to -78 °C and methyl pyruvate (1.5 mmol, 1.5 equiv.) was added dropwise via a gas-tight syringe. The reaction mixture was then brought to -40 °C and stirred at that temperature until the diene was consumed, as monitored by TLC and NMR. After addition of 15 mL of CH₂Cl₂, the resulting yellow solution was cooled to -78 °C and treated via syringe with 142 µL acetyl chloride (2 mmol, 2.0 equiv.). After stirring for additional 30 min, the reaction was quenched with saturated sodium bicarbonate. The organic layer was separated, and the water phase diluted with 15 mL water and extracted twice with CH₂Cl₂. The combined organic phase was dried with magnesium sulfate, filtered, and concentrated. The resulting yellow oil was purified by flash chromatography to give the desired dihydropyrone **10**.

2-methyl-4-oxo-3,4-dihydro-2H-pyran-2-carboxylic acid methyl ester (**10**).

¹H NMR (500 MHz, CDCl₃, ppm) δ 7.36 (d, *J*=6 Hz, 1H), 5.44 (dd, *J*₁=1 Hz *J*₂=6 Hz, 1H), 3.78 (s, 3H), 3.02 (dd, *J*₁=1 Hz *J*₂=17 Hz, 1H), 2.70 (d, *J*=17 Hz, 1H), 1.67 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm) δ 189.9, 171.4, 161.7, 107.3, 82.8, 53.1, 44.6, 24.1. IR (Neat, cm⁻¹) 1745, 1680, 1599, 1276, 1236.

HDA Reactions with Imines

4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid methyl ester (**12**)

To a 25 ml flame-dried flask was added freshly distilled diene **1a** (227 mg, 1.0 mmol, 1.0 equiv.) and 1.0 mL CH₂Cl₂. The resulting solution was cooled to -78 °C and treated dropwise with 196 mg (1.2 mmol, 1.2 equiv.) of dienophile **11** in 1.0 mL CH₂Cl₂. The reaction was stirred at -78 °C for 3 hours and quenched with 15 mL of a 4:1 THF/1N HCl solution. The mixture was allowed to warm up to room temperature and stirred for 2 hours, then quenched with 10 mL of saturated sodium bicarbonate solution. The organic layer was separated and the aqueous layer extracted three times with CH₂Cl₂. The combined organic phase was dried over magnesium sulfate, filtered, and concentrated to give yellow oil, which was purified by flash chromatography over silica gel (50% ethyl acetate/hexane) to afford **12** as a colorless oil (178 mg, 77%).

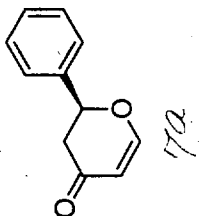
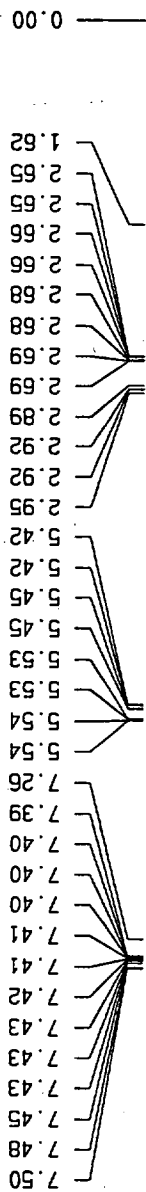
¹H NMR (500 MHz, CDCl₃, ppm) δ 7.96 (br d, *J*=7 Hz, 1H); 7.27 (m, 3H); 7.22 (m, 2H); 5.73 (d, *J*=7 Hz, 1H); 5.40 (d, *J*=7 Hz, 1H); 3.85 (s, 3H); 3.15 (dd, *J*₁=7 Hz, *J*₂=17 Hz, 1H); 2.82 (d, *J*=17 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃, ppm) δ 191.8; 142.3; 138.3; 128.9; 128.0; 125.9; 108.0; 55.9; 54.3; 41.8 (1 CH overlaps with others). IR (Neat, cm⁻¹) 1792; 1672; 1605; 1401; 1340; 1320; 1302; 1208.

.4-oxo-1-phenyl-3,4-dihydro-2H-pyridine-2-carboxylic acid methyl ester (14)

¹H NMR (400 MHz, CDCl₃, ppm) δ 7.47 (d, *J*=8 Hz, 1H); 7.39 (t, *J*=9 Hz, 2H); 6.83 (d, *J*=9 Hz, 2H); 5.68 (d, *J*=6 Hz, 1H); 5.39 (d, *J*=8 Hz, 1H); 3.85 (s, 3H); 3.77 (s, 3H); 3.12 (dd, *J*₁=8 Hz, *J*₂=17 Hz, 1H); 2.78 (d, *J*=17 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃, ppm) δ 192.2; 159.3; 153.3; 142.2; 130.4; 127.3; 114.1; 107.8; 55.3; 55.2; 54.2; 41.8. IR (Neat, cm⁻¹) 1732; 1670; 1607; 1514; 1338; 1321; 1208.

References

1. Kozmin, S. A.; Janey, J. M.; Rawal, V. H. *J. Org. Chem.* **1999**, *64*, 3039-3052.
2. (a) Georg, G. I.; Harriman, G. C. B.; Peterson, S. A. *J. Org. Chem.* **1995**, *60*, 7366. (b) Vidal, J.; Damestoy, S.; Guy, L.; Hannachi, J-C.; Aubry, A.; Collet, A. *Chem. Eur. J.* **1997**, *3*, 1691.

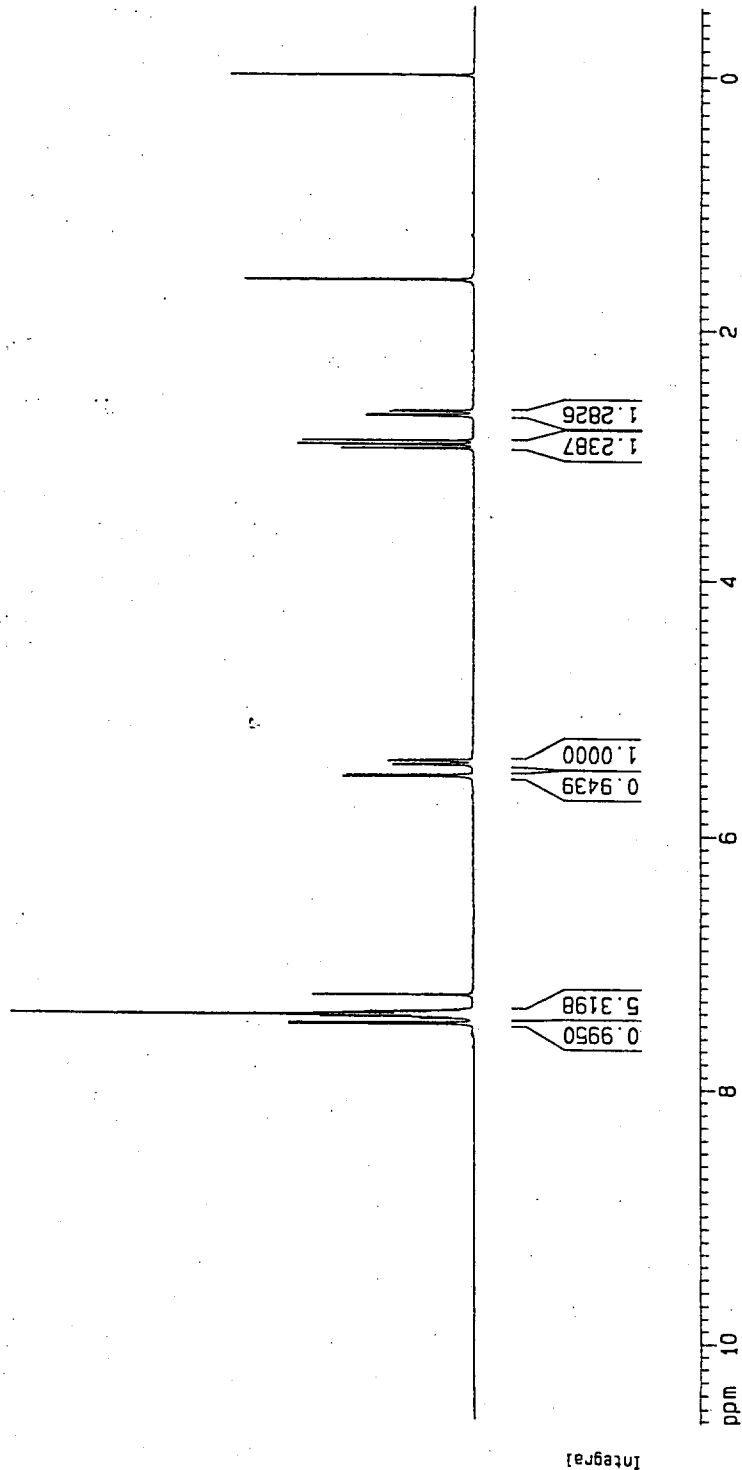


Current Data Parameters
 NAME 0827-4
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980827
 Time 17.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DW 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300122 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

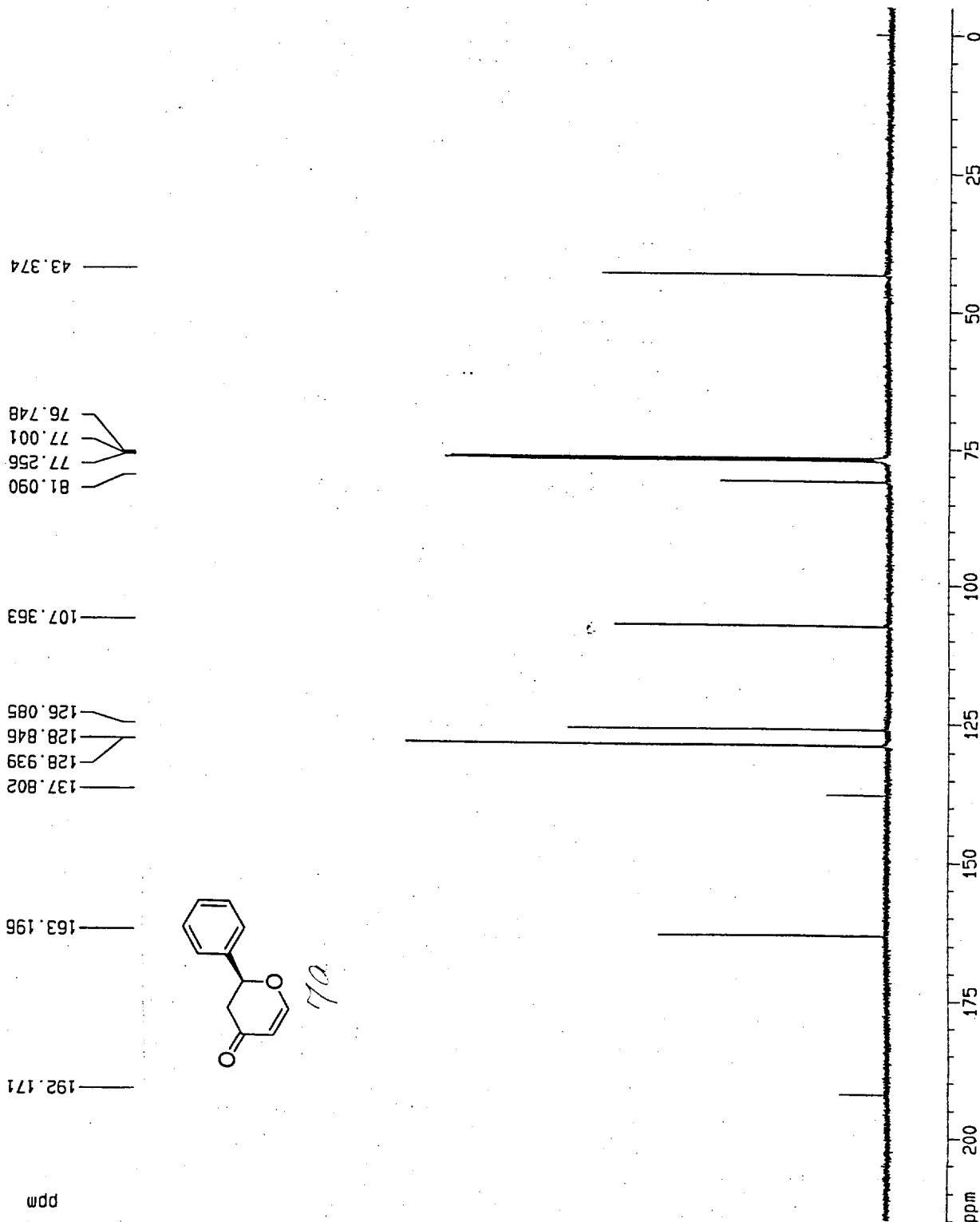
1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm



ppm

Integral

97/10/17, #5, 122.5 mg



Current Data Parameters
 NAME 0827-4
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980827
 Time 18.34
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1602
 DS 0
 SMH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 1024
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.00000000 sec
 P1 5.00 usec
 SFO1 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577946 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33582 Hz/cm

Current Data Parameters
 NAME 1028-7
 EXPNO 1
 PROCNO 1

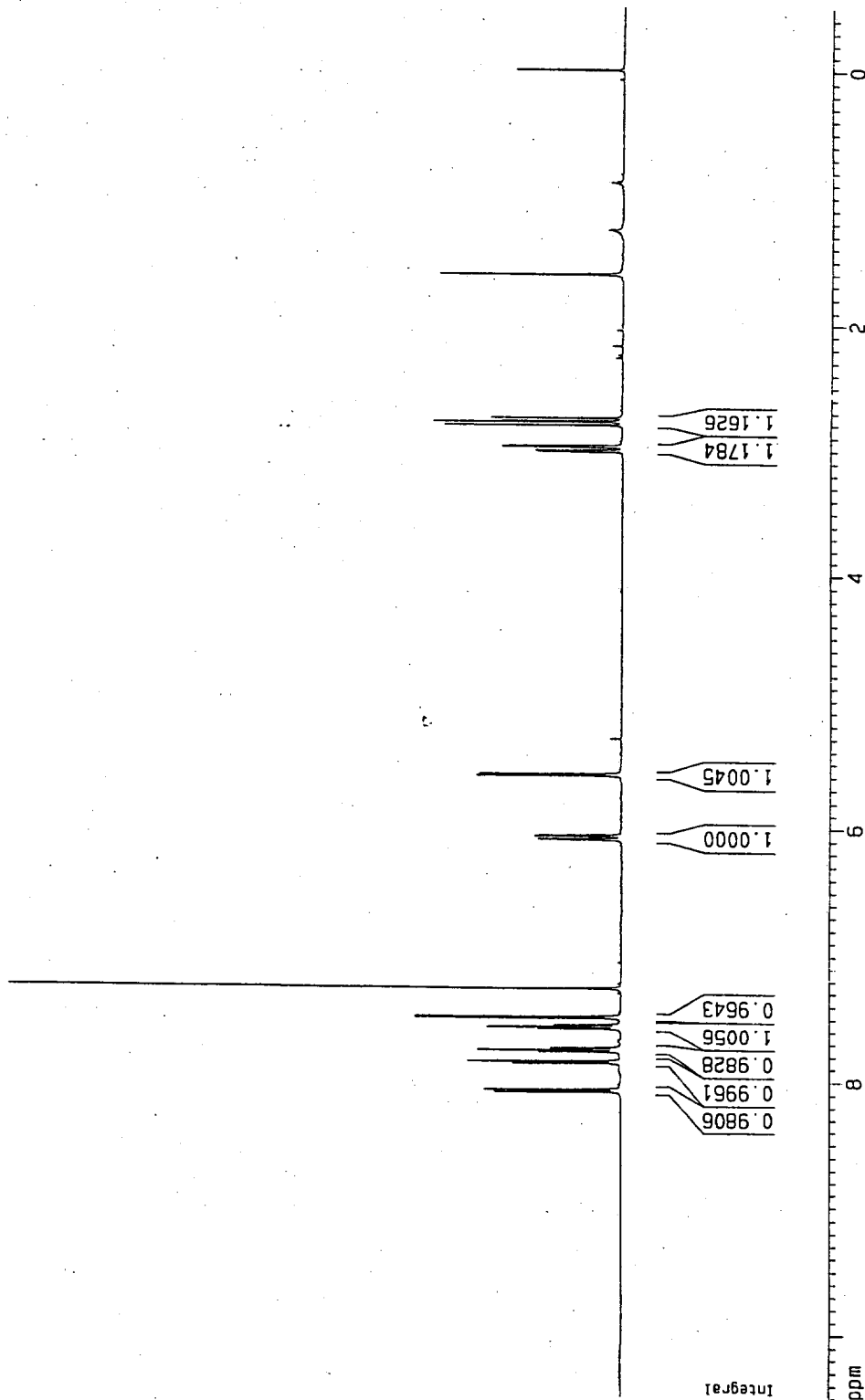
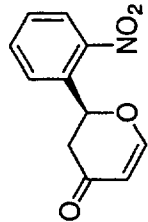
F2 - Acquisition Parameters
 Date_ 981028
 Time 18.45
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 4
 DS 0
 SMH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DM 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 B1 1.0000000 sec
 P1 8.80 usec
 SFO1 500.1325364 MHz
 NU01 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300102 MHz
 HDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

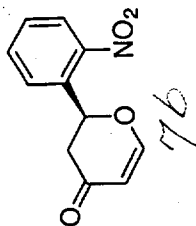
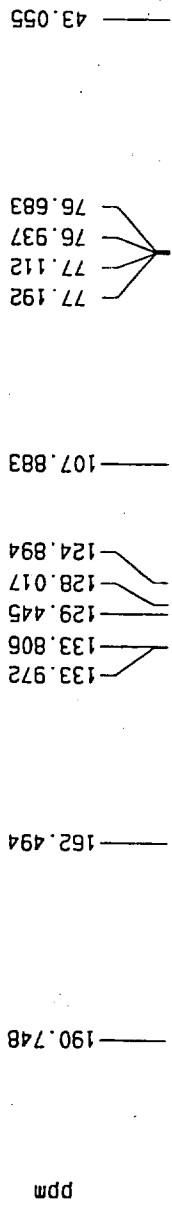
1D NMR plot parameters
 CX 20.00 cm
 F1P 10.500 ppm
 F1 5251.37 Hz
 F2P -0.500 ppm
 F2 -250.07 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

0.000
 1.607
 2.746
 2.774
 2.780
 2.808
 2.970
 2.973
 2.977
 2.979

5.583
 5.585
 5.595
 5.597
 7.267
 7.484
 7.497
 7.565
 7.753
 7.840
 7.842
 7.856
 7.858
 8.056
 8.058
 8.073
 8.075



97/10/17, #5, 122.5 mg



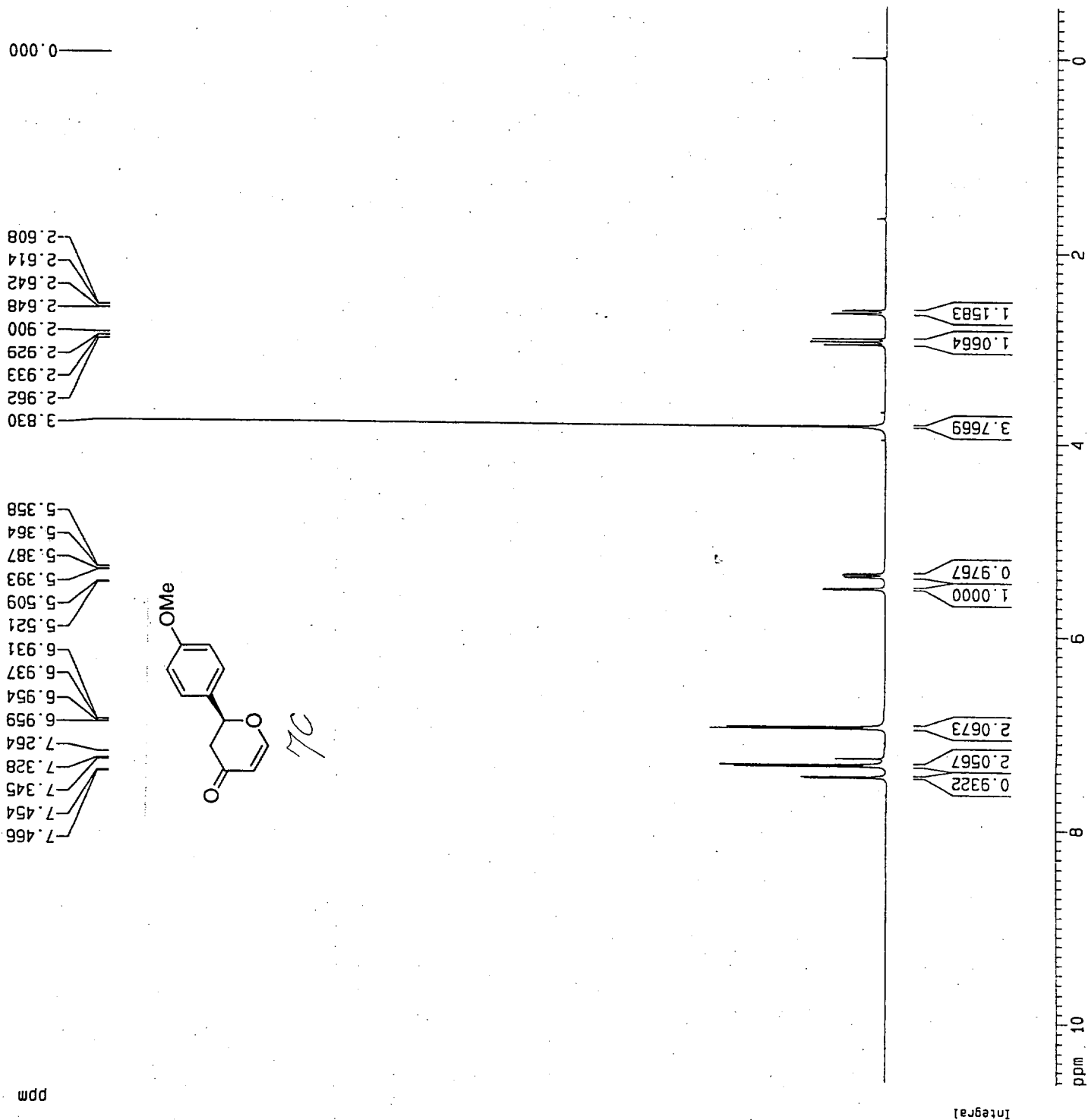
Current Data Parameters
 NAME 1028-7
 EXPNO 1
 PROCNO 1
 F2 Acquisition Parameters
 Date 981028
 Time 18.55
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 1661
 DS 0
 SWH 39662.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 2048
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.00000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 EM 0.00 dB
 F2 Processing parameters
 SI 32768
 SF 125.7578019 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33562 Hz/cm

Current Data Parameters
 NAME 1111-3
 EXPNO 1
 PROCNO 1

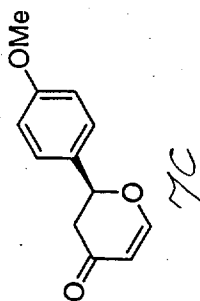
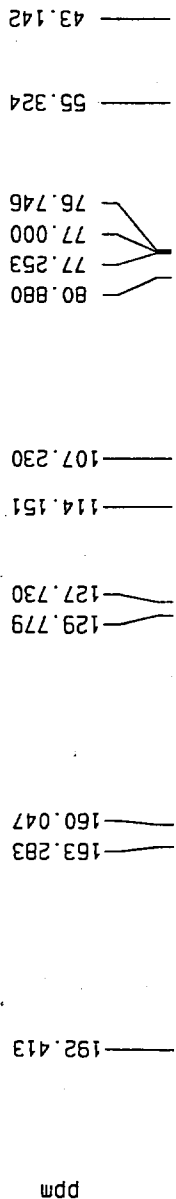
F2 - Acquisition Parameters
 Date_ 981112
 Time 15.37
 INSTRUM spect
 PROBHD 5 mm ONP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DM 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300115 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm



97/10/17, #5, 122.5 mg

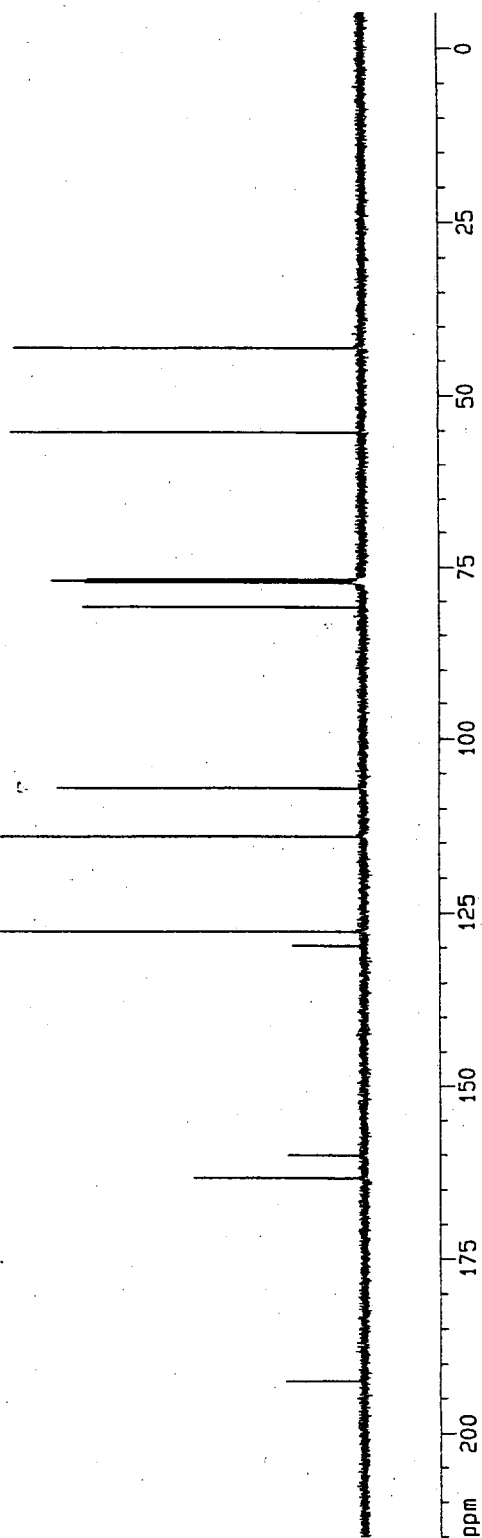


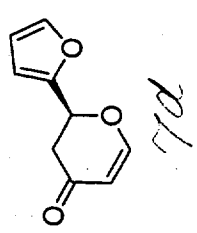
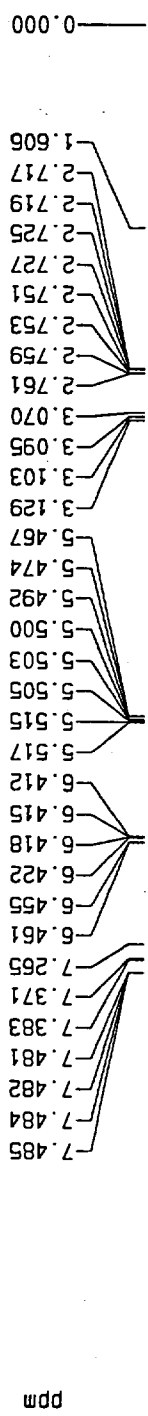
Current Data Parameters
 NAME 1111-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981112
 Time 15.46
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPRDG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 321
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 1024
 DM 12.600 usec
 DE 7.50 usec
 TE 300.10 K
 d11 0.03000000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.00000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577958 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPHCM 11.00000 ppm/cm
 HZCN 1363.33582 Hz/cm



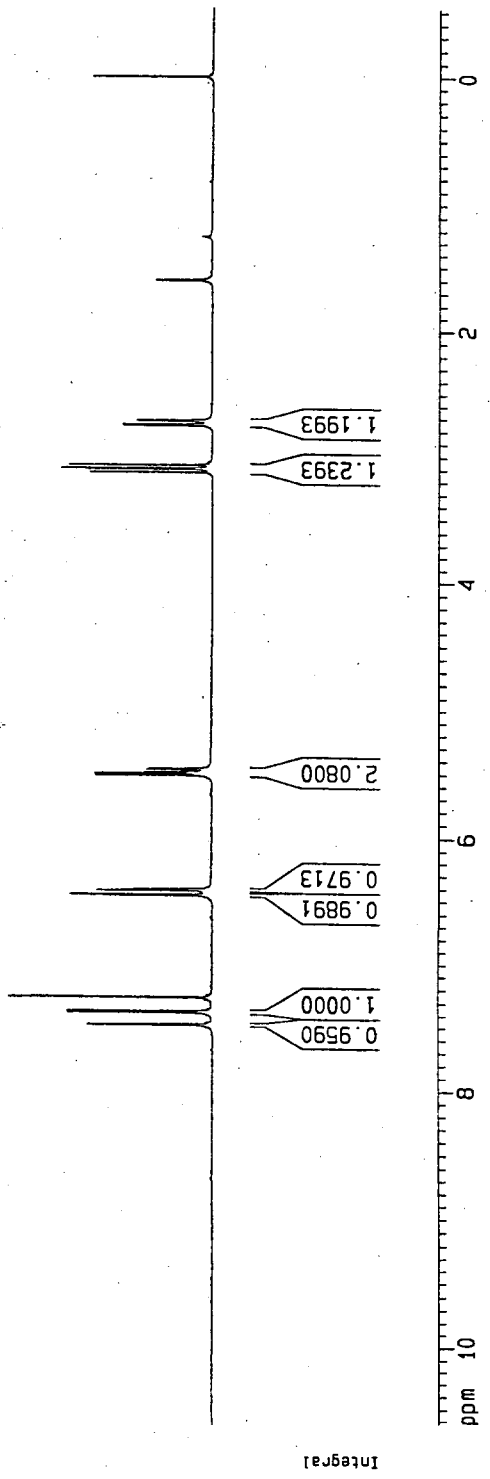


Current Data Parameters
 NAME 1117-5
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981117
 Time 15.20
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DW 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PL1 -6.00 dB

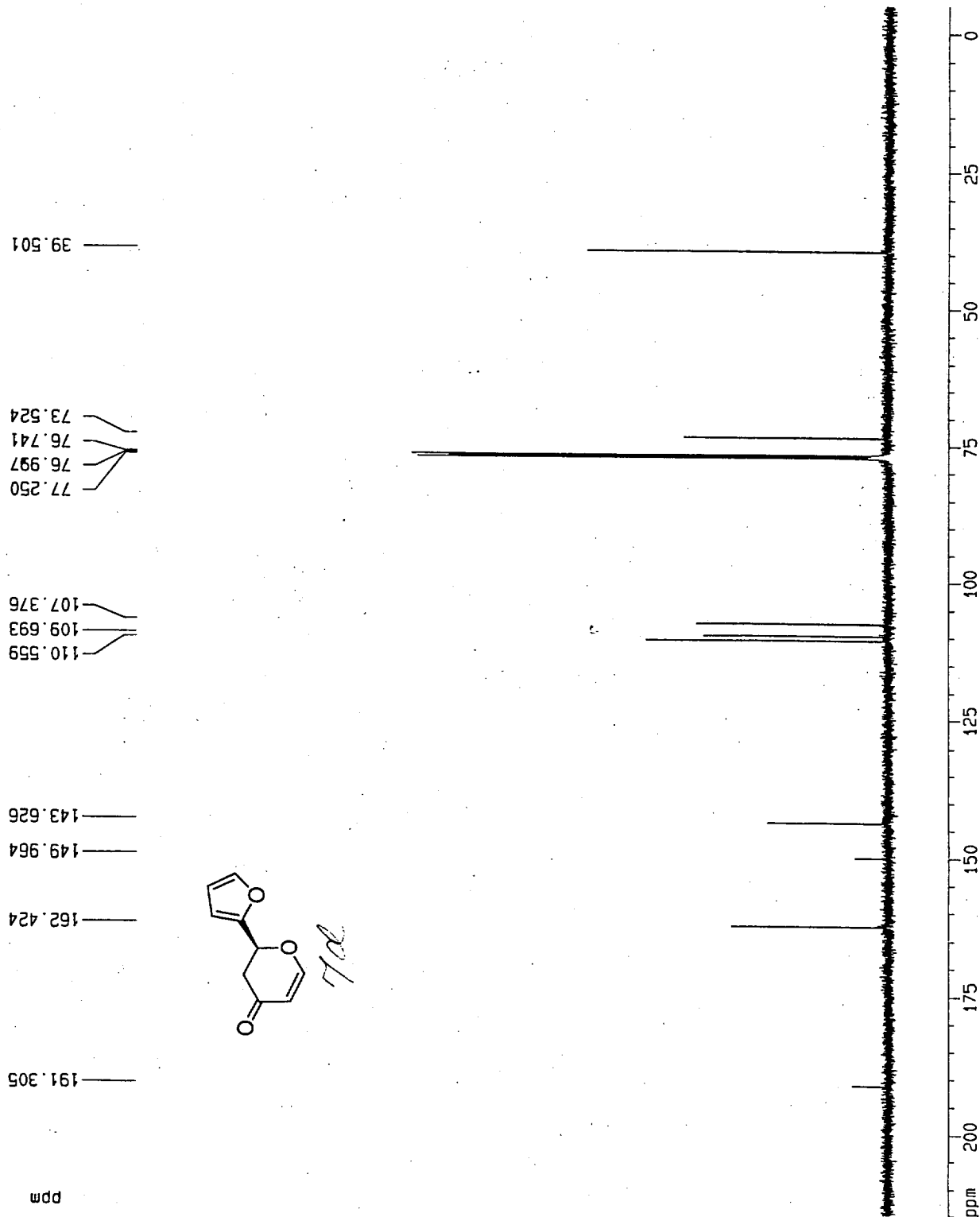
F2 - Processing parameters
 SI 16384
 SF 500.1300105 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm



Integral

97/10/17. #5. 122.5 mg



Current Data Parameters
 NAME 1117-5
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981117
 Time 15.27
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 662
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 2048
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 P1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577946 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

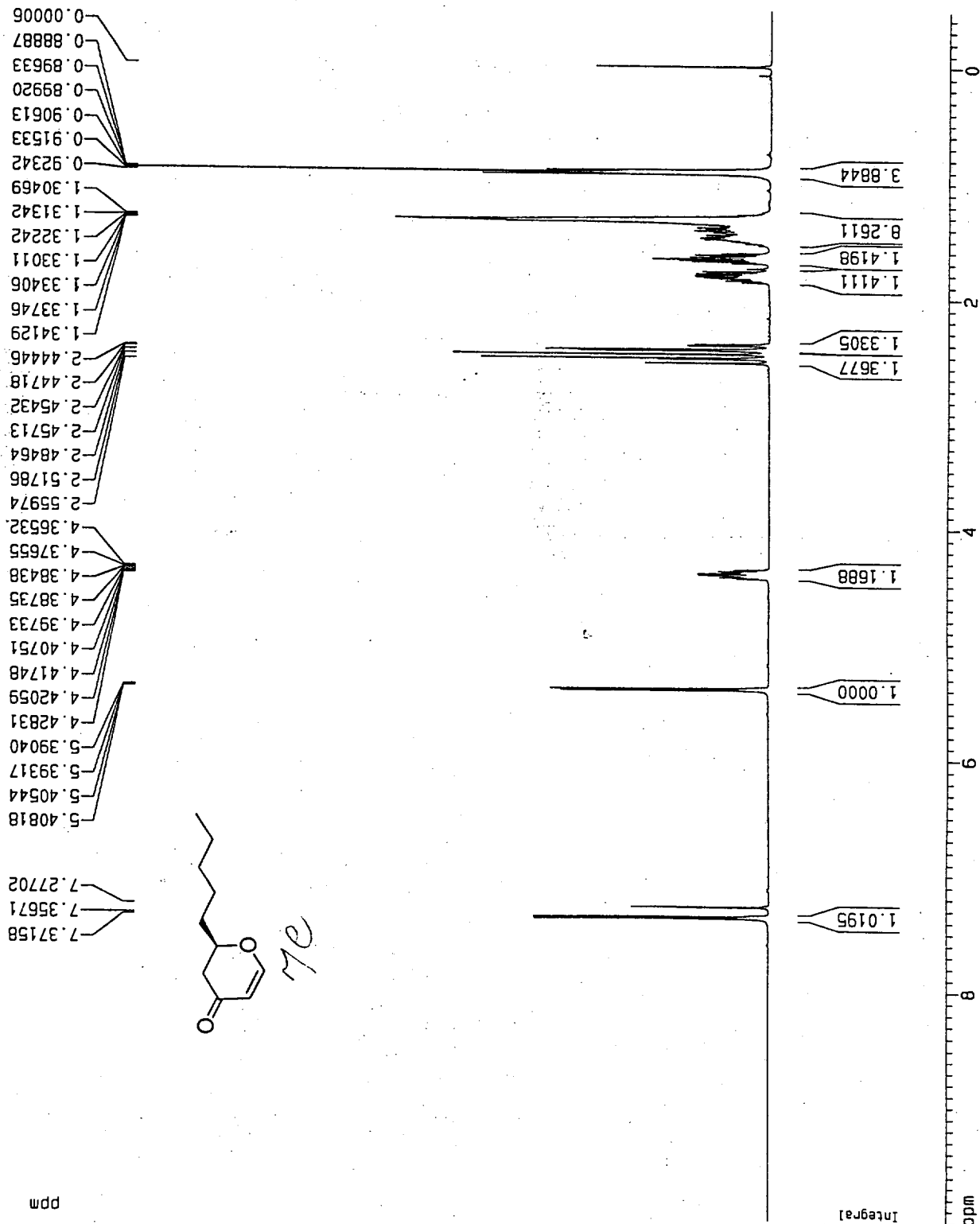
1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33582 Hz/cm

Current Data Parameters
 NAME 0713-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980713
 Time 18.22
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDC13
 NS 4
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.146719 Hz
 AQ 3.4079220 sec
 RG 64
 DW 104.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.50 usec
 SFO1 400.1319246 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300028 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.007 ppm
 F1 4004.18 Hz
 F2P -0.476 ppm
 F2 -190.45 Hz
 PPMCM 0.52416 ppm/cm
 HZCM 209.73154 Hz/cm

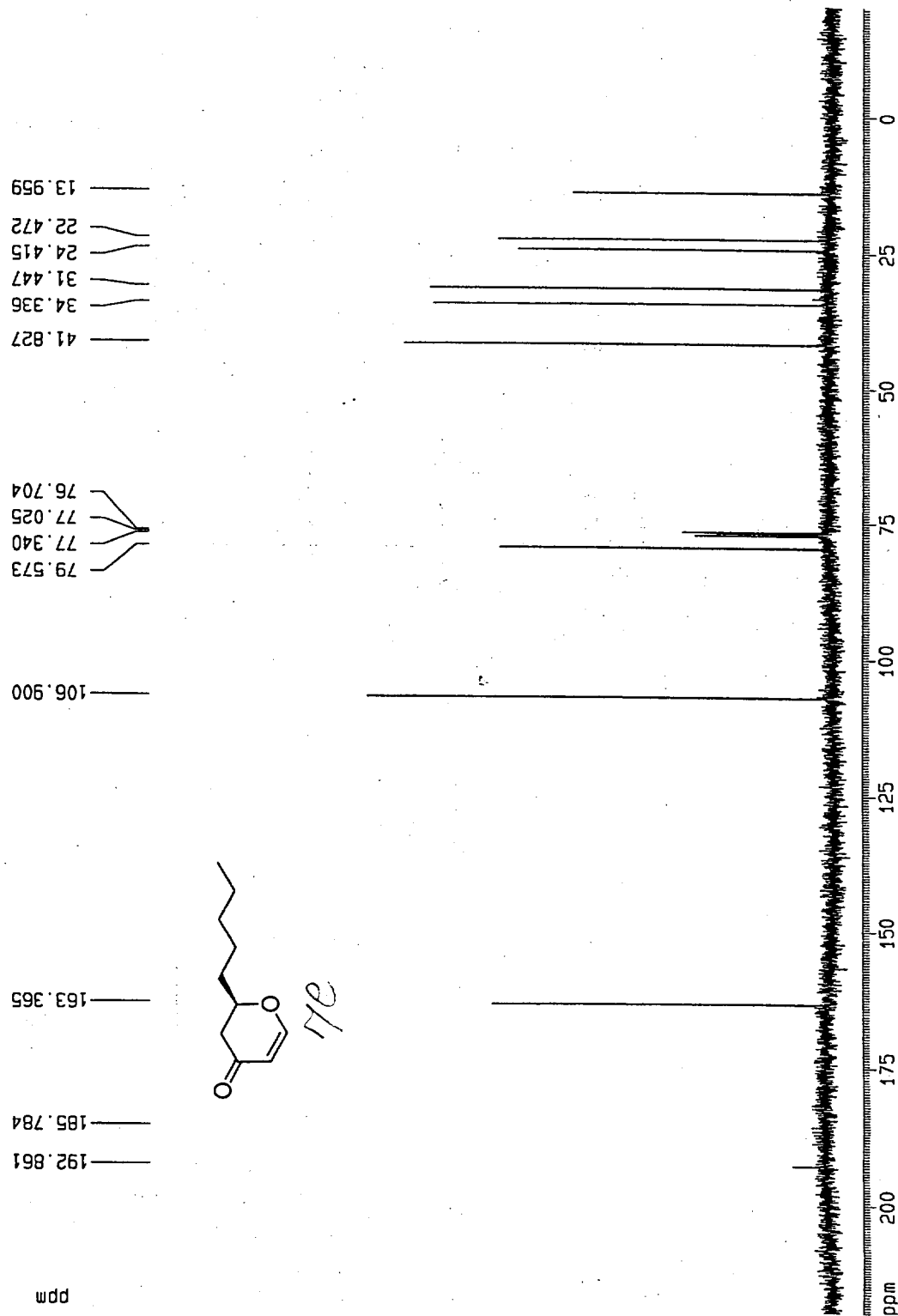


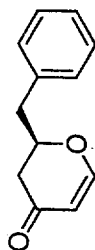
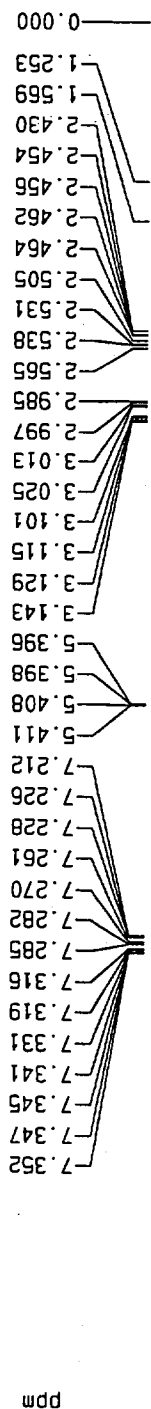
Current Data Parameters
 NAME 0713-5-c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980713
 Time 19.34
 INSTRUM spect
 PROBRD 5 mm QNP 1H
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 1802
 DS 2
 SWH 31746.031 Hz
 FIDRES 0.968812 Hz
 AQ 0.5161460 sec
 RG 4597.6
 DM 15.750 usec
 DE 22.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 19.00 dB
 CPDPRG2 waltz16
 PCPD2 101.00 usec
 SF02 400.1324710 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 1.00000000 sec
 P1 8.00 usec
 SF01 100.6254358 MHz
 NUC1 13C
 PL1 -2.00 dB

F2 - Processing parameters
 SI 16384
 SF 100.6127717 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 220.000 ppm
 F1 22134.81 Hz
 F2P -20.000 ppm
 F2 -2012.25 Hz
 PPMCM 12.00000 ppm/cm
 HZCM 1207.35327 Hz/cm



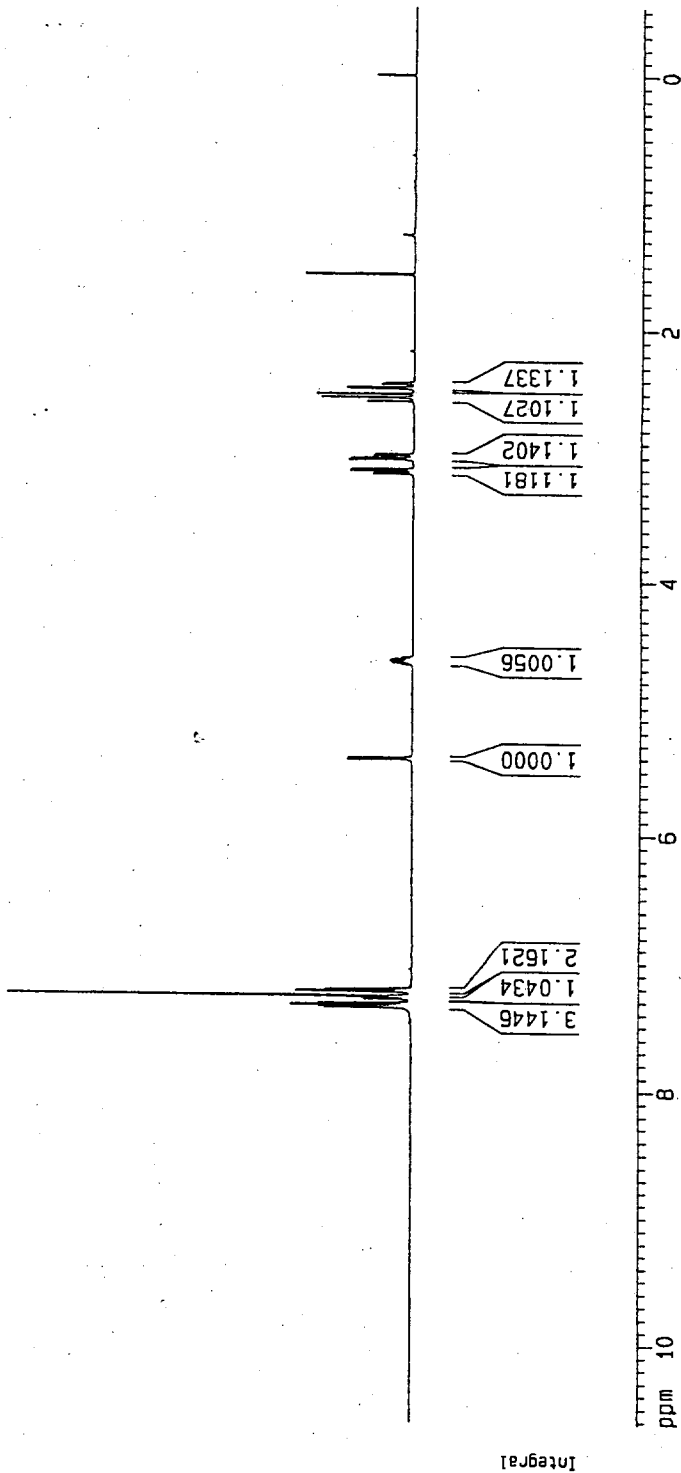


Current Data Parameters
 NAME 1223-3
 EXPNO 1
 PROCNO 1

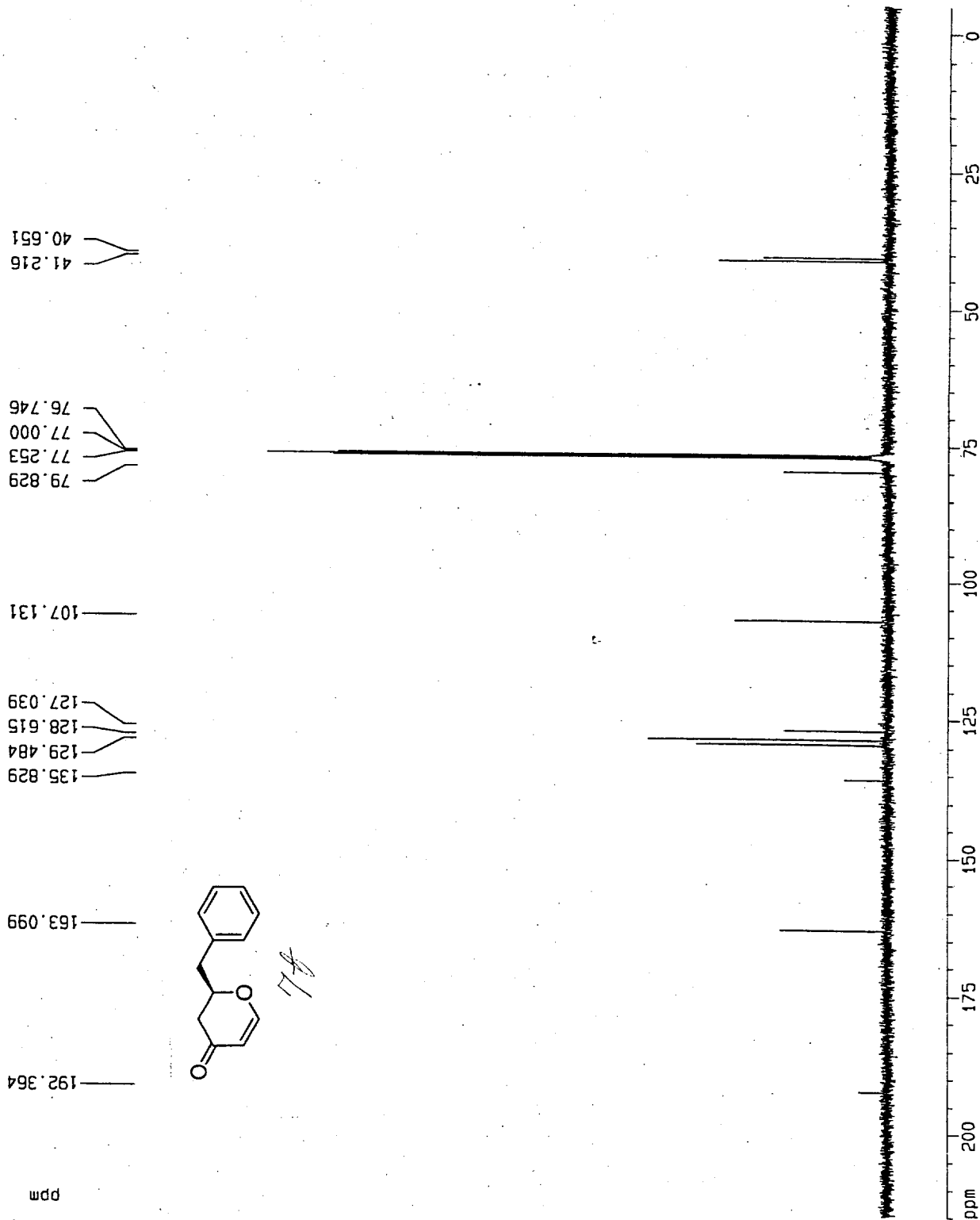
F2 - Acquisition Parameters
 Date_ 981223
 Time 17.14
 INSTRUM spect
 PRBHD 5 mm QNP 1H
 PULPROG zg
 TO 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 256
 DM 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300129 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FJP 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm



97/10/17, #5, 122.5 mg



Current Data Parameters
 NAME 1223-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981223
 Time 17.21
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TO 65536
 SOLVENT CDCl3
 NS 607
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.60507 Hz
 AQ 0.8258036 sec
 RG 1024
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 20.00 dB
 CPDPRG2 Waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.0000000 sec
 P1 5.00 usec
 SFD1 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577934 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

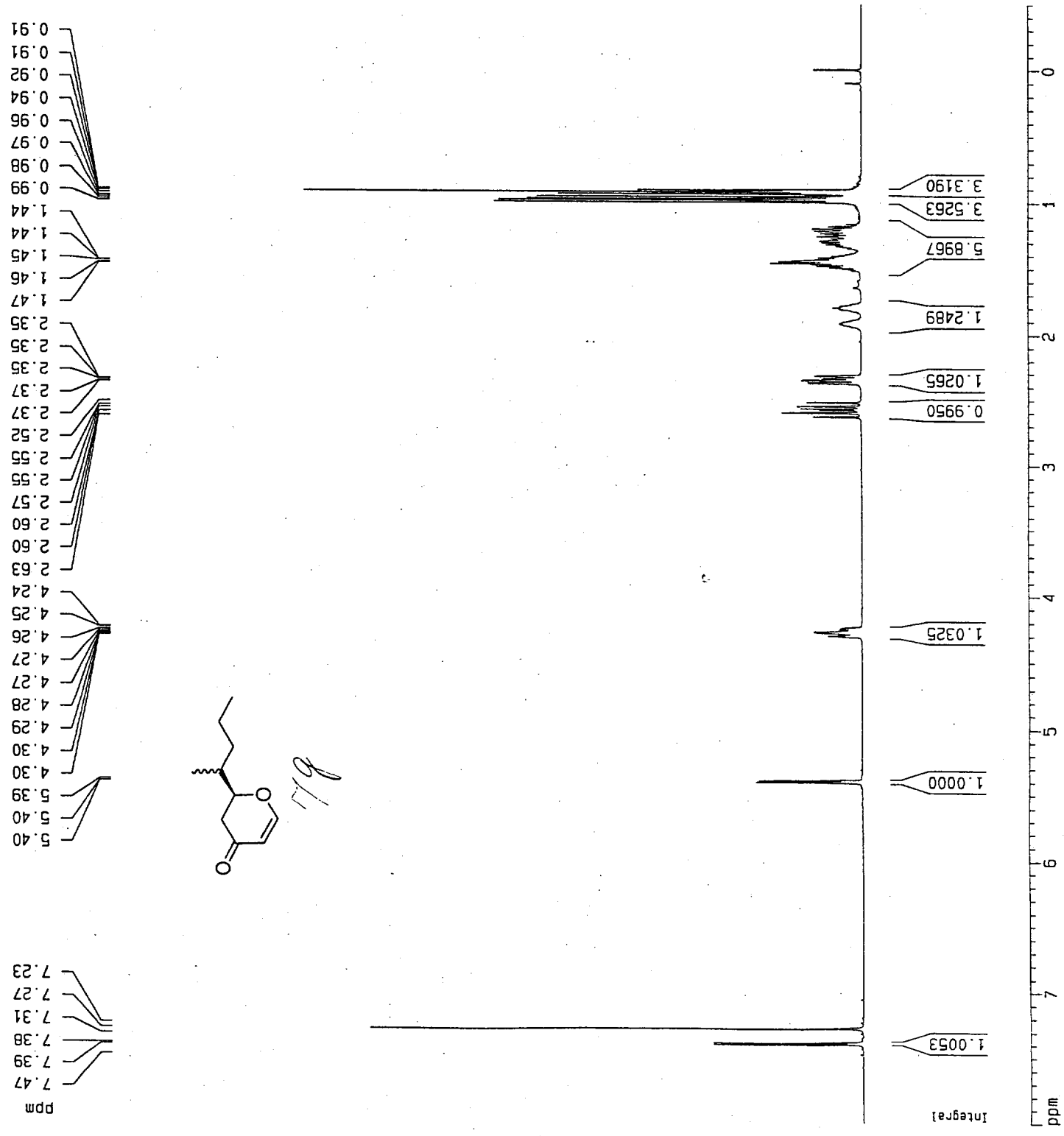
1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCH 11.00000 ppm/cm
 HZCM 1383.33569 Hz/cm

Current Data Parameters
 NAME 1103-2
 EXPNO 1
 PROCNO 1

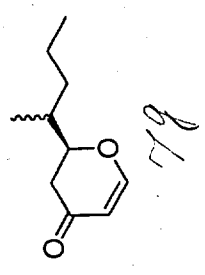
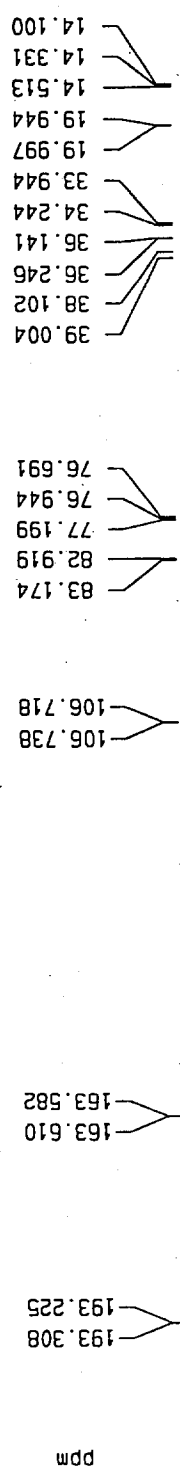
F2 - Acquisition Parameters
 Date_ 981103
 Time 17.40
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5560.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DW 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300102 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P -0.500 ppm
 F2 -250.07 Hz
 PPMCM 0.42500 ppm/cm
 HZCM 212.55525 Hz/cm



97/10/17, #5, 122.5 mg

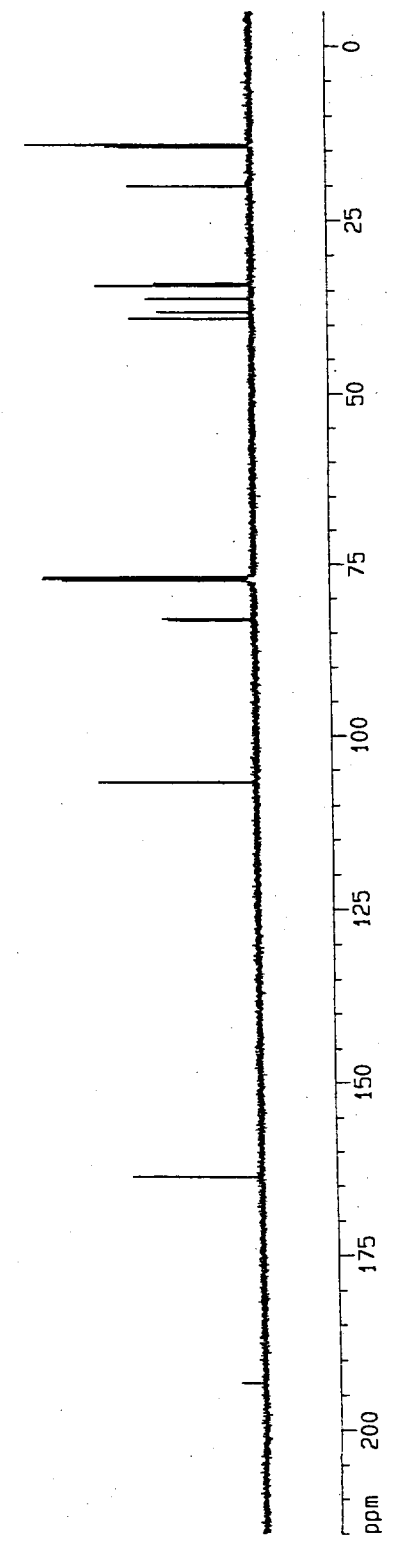


Current Data Parameters
 NAME 1204-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981204
 Time 17.51
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDC13
 NS 237
 DS 0
 SMH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 2048
 DM 12.500 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.00000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7578019 MHz
 EM
 WDM 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPHCM 11.00000 ppm/cm
 HZCM 1303.33582 Hz/cm



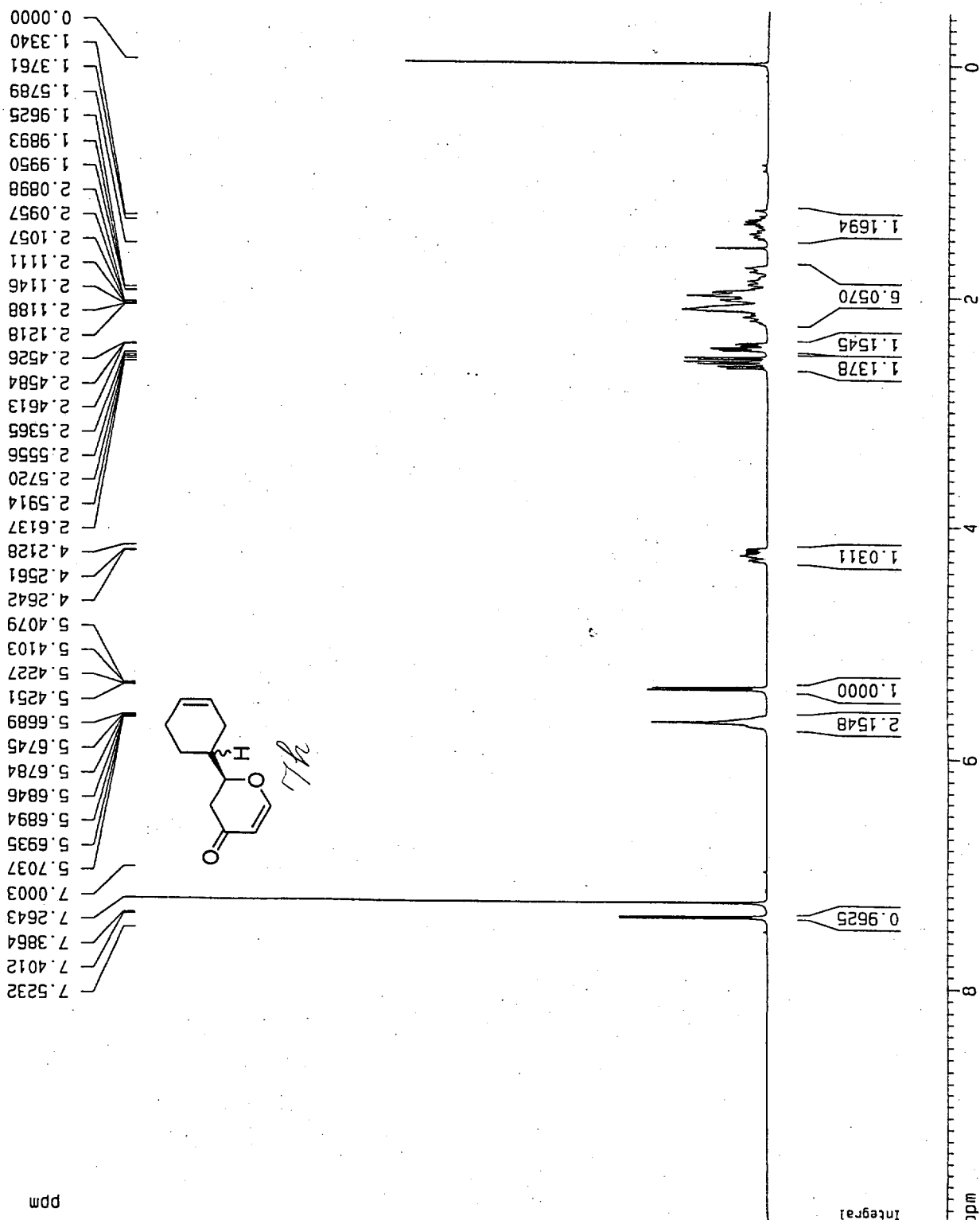
Current Data Parameters
 NAME 1120-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981120
 Time 18.10
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9058956 sec
 RG 228.1
 DM 119.200 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 8.50 usec
 PL1 0.00 dB
 SF01 400.1319246 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300080 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.007 ppm
 F1 4004.18 Hz
 F2P -0.476 ppm
 F2 -190.45 Hz
 PPMCM 0.52416 ppm/cm
 HZCM 209.73154 Hz/cm



Current Data Parameters
 NAME 1120-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981120
 Time 18.43
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 32768
 SOLVENT CDC13
 NS 226
 DS 2
 SWH 27173.912 Hz
 FIDRES 0.629282 Hz
 AQ 0.6029812 sec
 RG 4597.6
 DW 18.400 usec
 DE 26.29 usec
 TE 300.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.00 usec
 PL1 -2.00 dB
 SF01 100.6254358 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 101.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 400.1324710 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6127958 MHz
 NDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -20.000 ppm
 F2 -2012.26 Hz
 PPMCM 12.00000 ppm/cm
 HZCM 1207.35352 Hz/cm

23.639
 24.019
 24.425
 26.294
 26.491
 37.141
 37.190
 38.922
 38.952

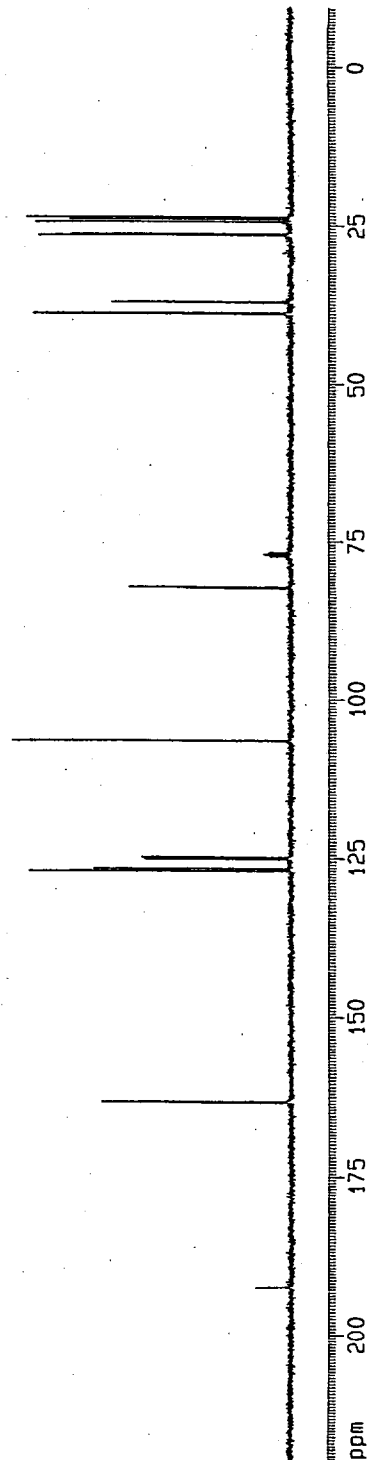
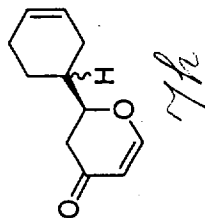
76.682
 77.001
 77.317
 82.326
 82.542

106.580
 124.821
 125.103
 126.619
 126.998

163.257
 163.116

192.468

ppm

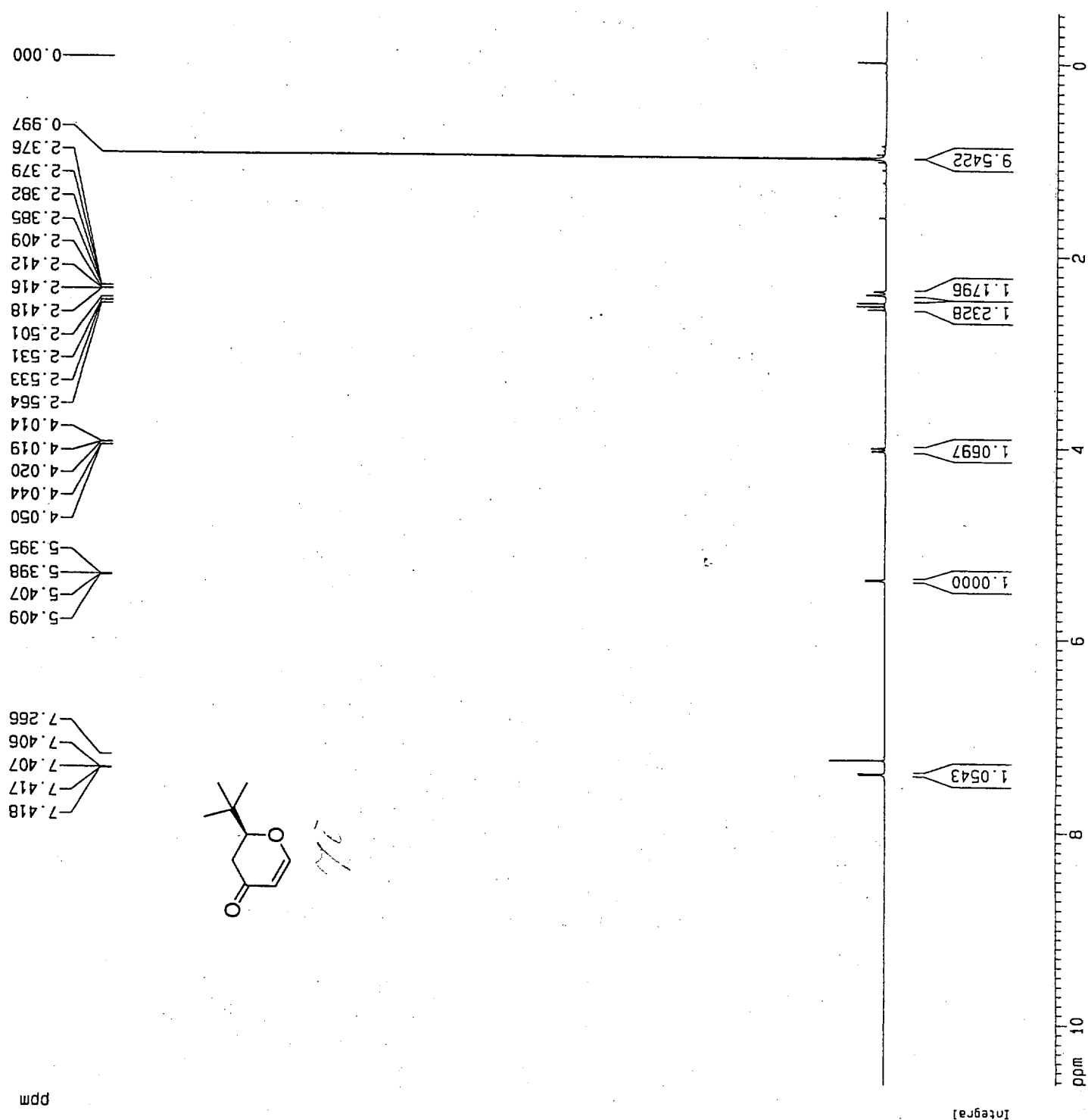


Current Data Parameters
 NAME 1125-1
 EXPNO 1
 PROCNO 1

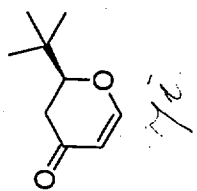
F2 - Acquisition Parameters
 Date_ 981125
 Time 10.34
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DW 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300102 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm



97/10/17, #5, 122.5 mg

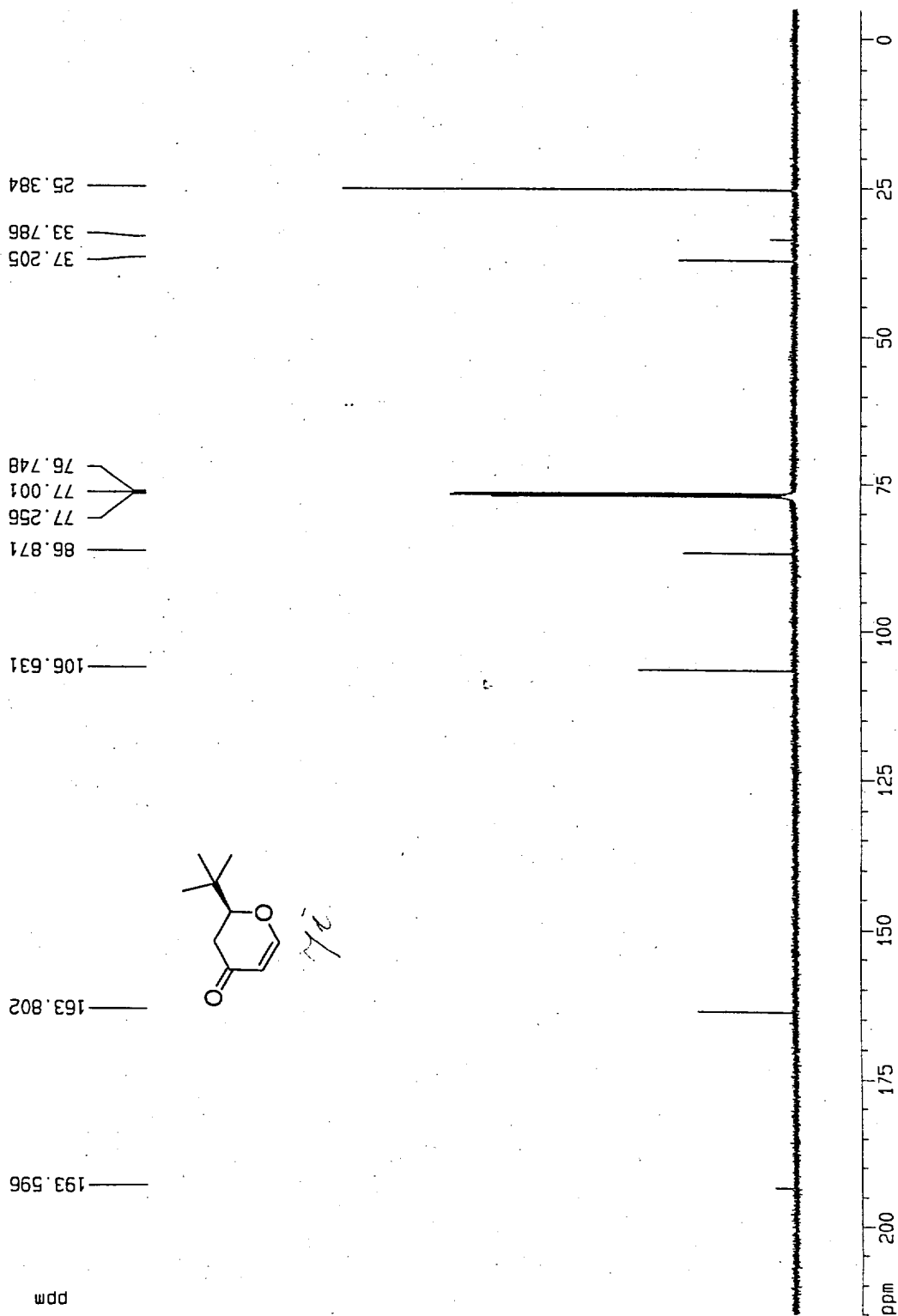


Current Data Parameters
 NAME 1125-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981125
 Time 10.42
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TO 65536
 SOLVENT CDCl3
 NS 602
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 2048
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577934 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33569 Hz/cm



Current Data Parameters
 NAME 102B-9
 EXPNO 1
 PROCNO 1

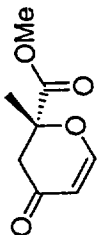
F2 - Acquisition Parameters
 Date_ 981028
 Time 20.26
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 5580.357 Hz
 FIDRES 0.170299 Hz
 AQ 2.9360628 sec
 RG 128
 DW 89.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 EI 8.80 usec
 SF01 500.1325364 MHz
 NUC1 1H
 PC 1.00
 REFL -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300092 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm

1.673
 2.685
 2.719
 3.001
 3.003
 3.784

7.269
 7.358
 7.370



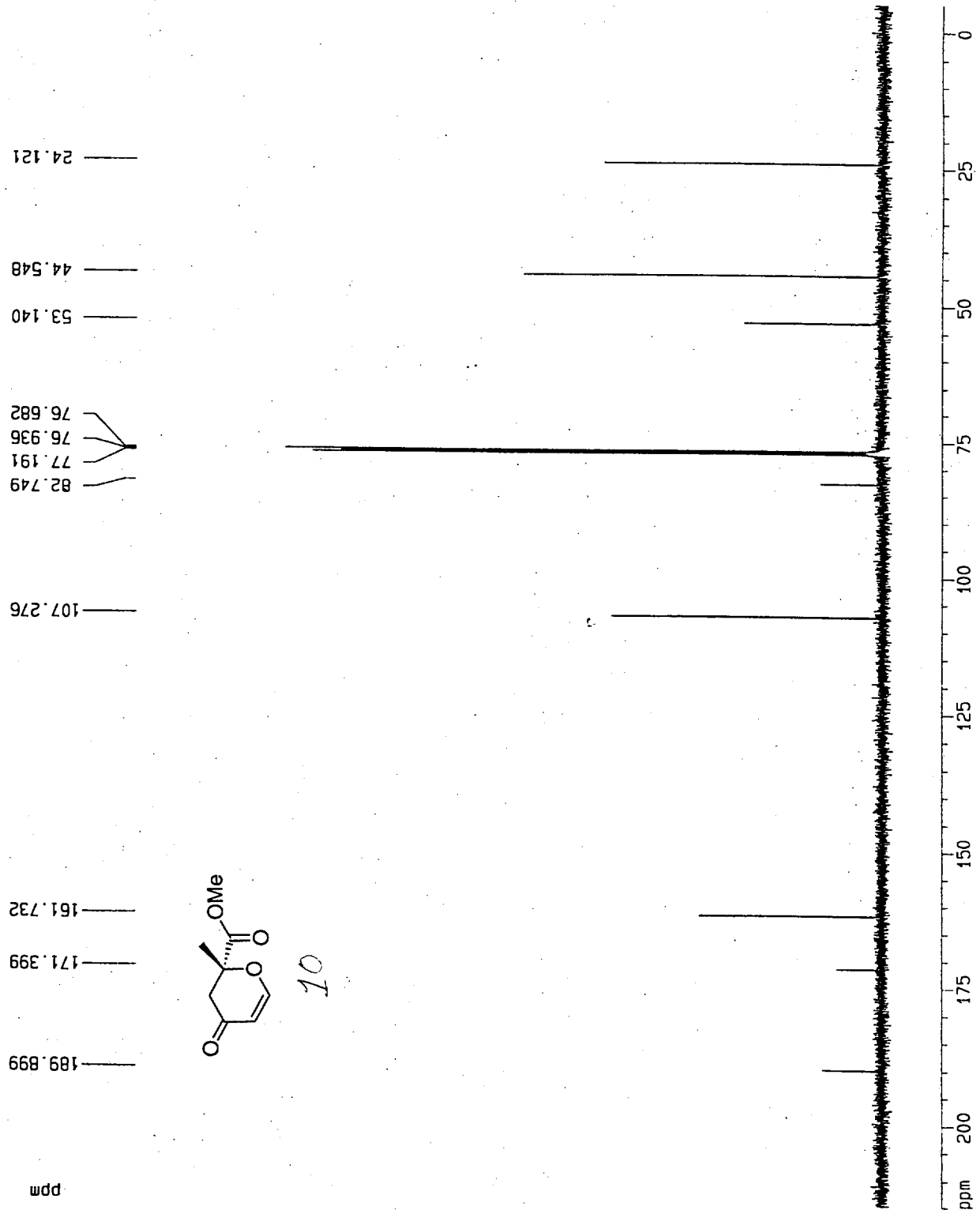
10

Integral

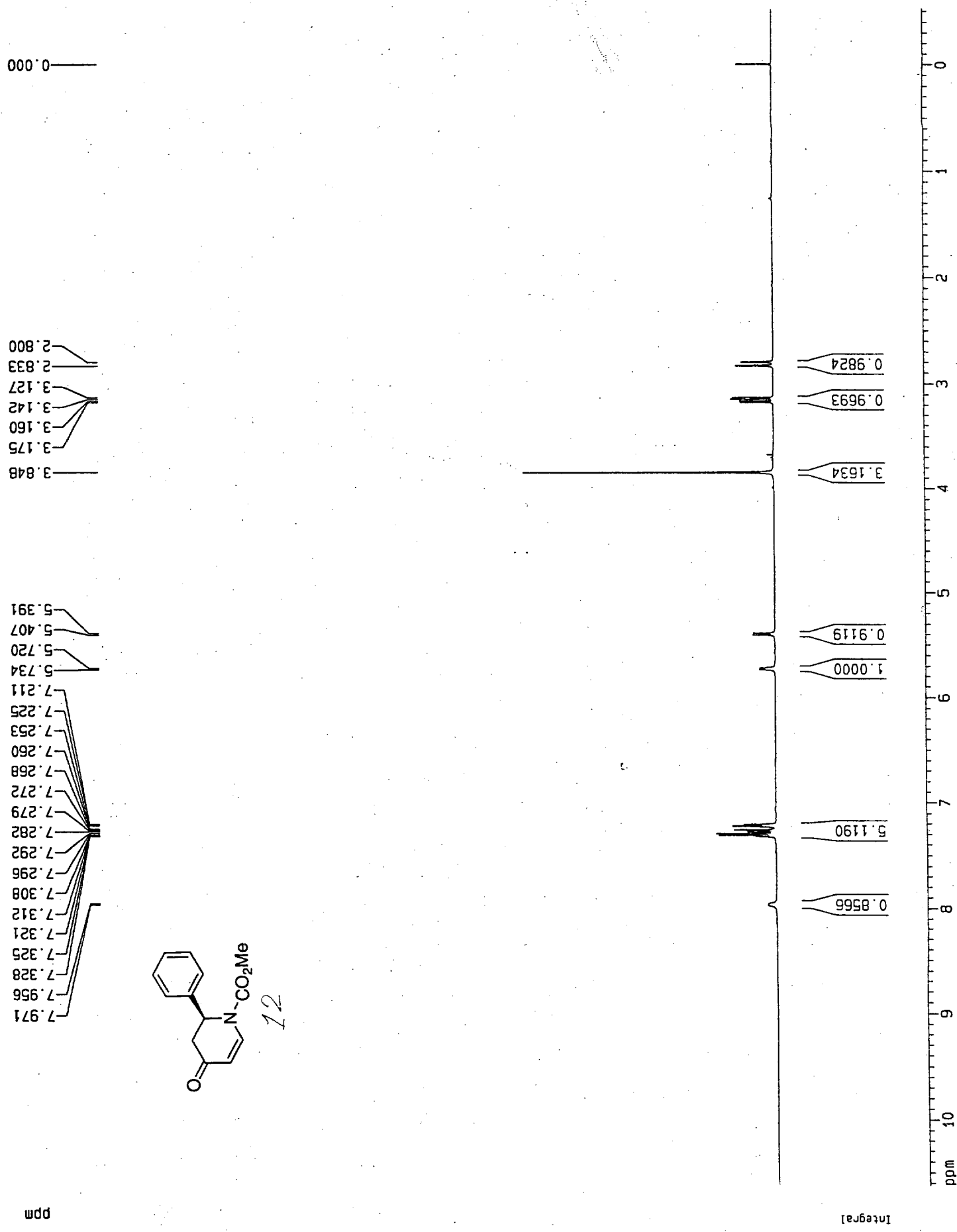


ppm

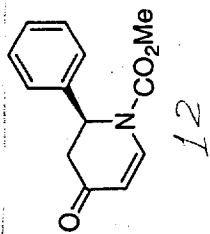
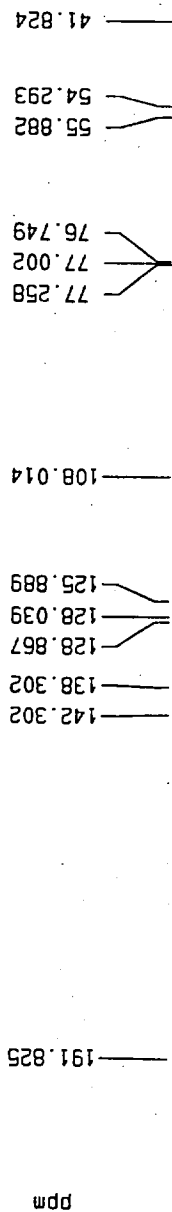
97/10/17, #5, 122.5 mg



Current Data Parameters
 NAME 1028-9
 EXPNO 1
 PROCNO 1
 F2 Acquisition Parameters
 Date_ 981028
 Time 20.54
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 448
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.606507 Hz
 AQ 0.8258036 sec
 RG 2048
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 20.00 dB
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB
 F2 Processing parameters
 SI 32768
 SF 125.7578019 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCH 11.00000 ppm/cm
 HZCM 1383.33582 Hz/cm



97/10/17. #5, 122.5 mg

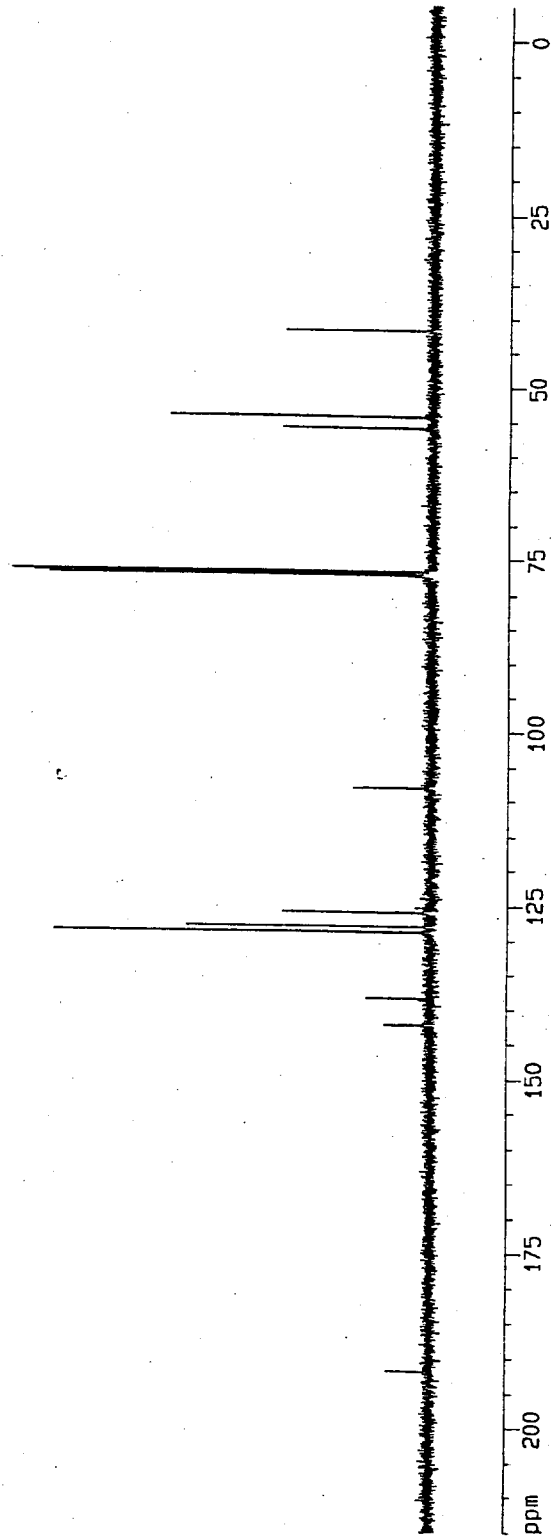


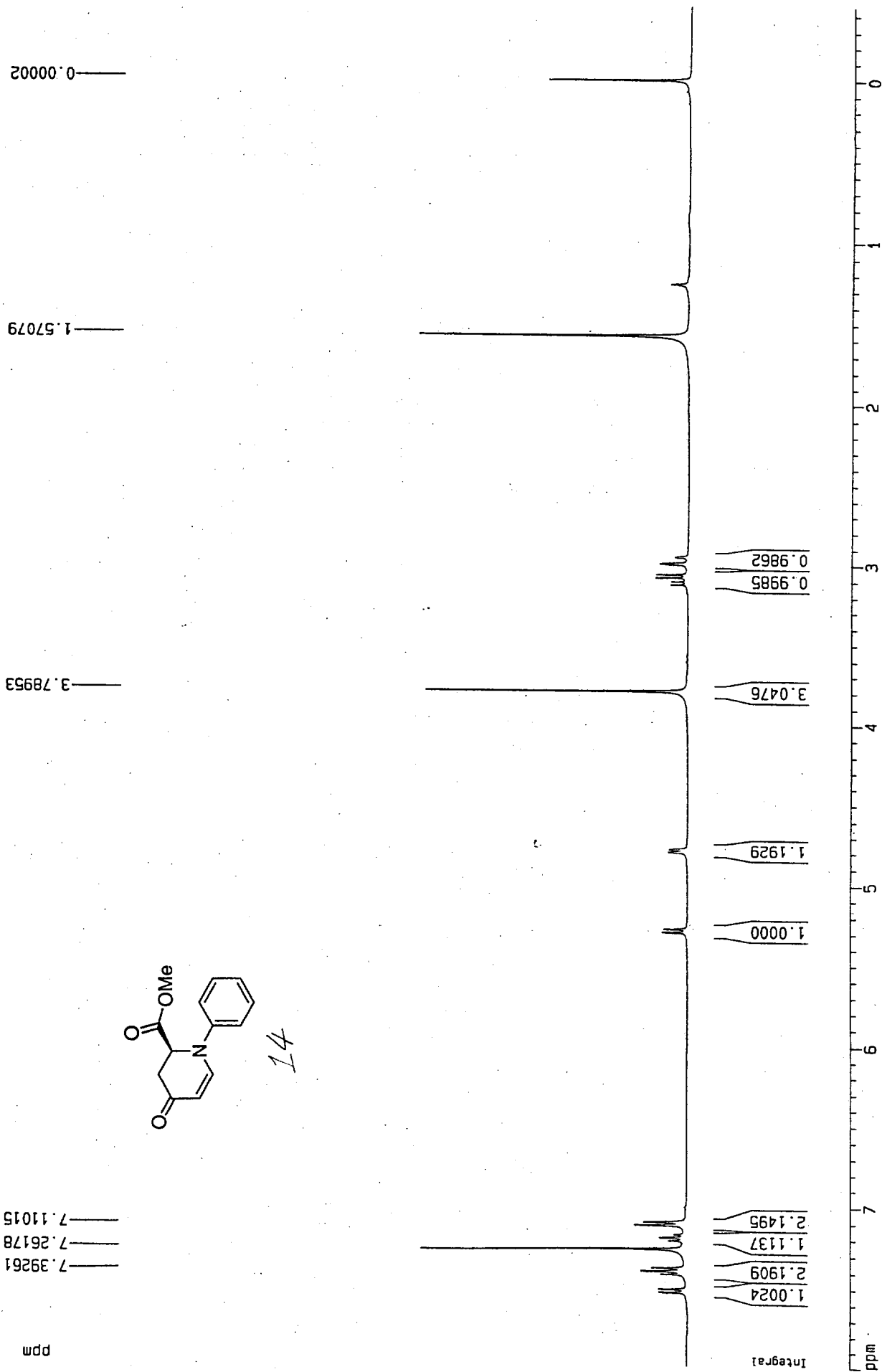
Current Data Parameters
 NAME 1231-4
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 981231
 Time 14.55
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 269
 DS 0
 SMH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 1024
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

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

1D NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1363.33569 Hz/cm





97/10/17, #5, 122.5 mg

