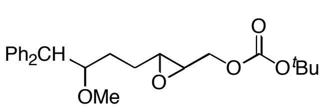
SUPPORTING INFORMATION

An Experimental and Computational Approach to Defining Structure/Reactivity Relationships for Intramolecular Addition Reactions to Bicyclic Epoxonium Ions

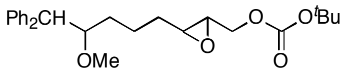
Shuangyi Wan, Hakan Gunaydin, K. N. Houk, and Paul E. Floreancig
Department of Chemistry and Biochemistry
University of California
Los Angeles, CA 90095
and
Department of Chemistry
University of Pittsburgh
Pittsburgh, PA 15260

General Experimental Proton (^{1}H NMR) and carbon (^{13}C NMR) nuclear magnetic resonance spectra were recorded on a Bruker Avance 300 spectrometer at 300 MHz and 75 MHz respectively, a Bruker Avance 500 spectrometer at 500 MHz and 125 MHz, or a Bruker Avance 600 spectrometer at 600 MHz and 151 MHz if specified. The chemical shifts are given in parts per million (ppm) on the delta (δ) scale. The solvent peak was used as a reference value, for ^{1}H NMR: $CDCl_3$ = 7.27 ppm, C_6D_6 = 7.15 ppm, for ^{13}C NMR: $CDCl_3$ = 77.23. Data are reported as follows: (s = singlet; d = doublet; t = triplet; q = quartet; p = pentet; dd = doublet of doublets; ddd = doublet of doublet of doublets; dddd = doublet of doublet of doublet of doublets; dt = doublet of triplets; br = broad; app = apparent). High resolution and low resolution mass spectra were recorded on a VG 7070 spectrometer. Infrared (IR) spectra were collected on a Mattson Cygnus 100 spectrometer. Samples for IR were prepared as a thin film on a NaCl plate by dissolving the compound in CH_2Cl_2 and then evaporating. Optical rotations were measured on a Perkin-Elmer 241 polarimeter. Tetrahydrofuran and diethyl ether were dried by passage through an activated alumina column under positive N_2 pressure. Methylene chloride and benzene were distilled under N_2 from CaH_2. Analytical TLC was performed on E. Merck pre-coated (25 mm) silica gel 60F-254 plates. Visualization was done under UV (254 nm). Flash chromatography was performed ICN SiliTech 32-63 60 Å silica gel. Reagent grade ethyl acetate, diethyl ether, pentane and hexanes (commercial mixture) were purchased from EM Science and used without further purification for chromatography. All reactions were performed in oven or flame-dried glassware under a positive pressure of N_2 with magnetic stirring unless otherwise noted.



1

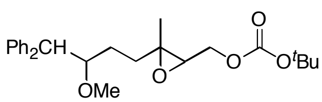
***tert*-Butyl (3-(3-methoxy-4,4-diphenylbutyl)oxiran-2-yl)methyl carbonate (1)**
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38-7.16 (m, 10H), 4.23-4.18 (m, 1H), 3.99-3.92 (m, 3H), 3.14/3.13 (s, 3H), 2.94-2.91 (m, 1H), 2.81-2.77 (m, 1H), 1.79-1.42 (m, 4H), 1.48 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.4, 142.6, 142.3, 142.2, 128.9, 128.9, 128.8, 128.6, 128.5, 126.7, 126.5, 83.1, 83.0, 82.7, 67.2, 67.1, 58.3, 57.9, 56.8, 56.6, 56.5, 56.2, 55.2, 28.8, 28.0, 27.9, 27.4, 27.1; IR (neat) 2981, 2933, 1743, 1495, 1452, 1370, 1281, 1163, 1101, 912, 733, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{25}H_{32}O_5Na$ (M + Na$^+$) 435.2147, found 435.2140.



2

***tert*-Butyl (3-(4-methoxy-5,5-diphenylpentyl)oxiran-2-yl)methyl carbonate (2)**

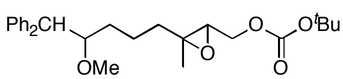
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.16 (m, 10H), 4.23-4.18 (m, 1H), 4.00 (d, *J* = 8.4 Hz, 1H), 3.97-3.88 (m, 2H), 3.16/3.16 (s, 3H), 2.96-2.91 (m, 1H), 2.81-2.79 (m, 1H), 1.59-1.43 (m, 6H), 1.50 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.4, 142.8, 142.4, 129.0, 128.7, 128.6, 128.4, 126.6, 126.4, 83.6, 82.7, 67.2, 58.2, 58.1, 56.6, 56.5, 56.3, 55.2, 55.2, 32.0, 32.0, 31.7, 27.9, 21.6, 21.6; IR (neat) 2980, 2936, 1742, 1495, 1452, 1370, 1280, 1163, 1099, 858, 733, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{26}H_{34}O_5Na$ (M + Na^+) 449.2304, found 449.2308.



3

***tert*-Butyl (3-(3-methoxy-4,4-diphenylbutyl)-3-methyloxiran-2-yl)methyl carbonate (3)**

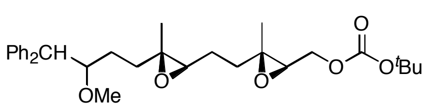
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.17 (m, 10H), 4.20-4.14 (m, 1H), 4.09 (dd, *J* = 11.8, 6.1 Hz, 1H), 3.99-3.91 (m, 2H), 3.15/3.14 (s, 3H), 2.97-2.91 (m, 1H), 1.83-1.42 (m, 4H), 1.50 (s, 9H), 1.20/1.18 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 142.6, 142.4, 142.3, 128.9, 128.9, 128.7, 128.6, 128.4, 126.7, 126.5, 83.2, 83.0, 82.7, 65.7, 65.6, 60.8, 60.6, 59.6, 59.2, 58.1, 57.8, 56.4, 56.2, 33.5, 33.3, 27.9, 27.4, 27.1, 17.1, 16.7; IR (neat) 2980, 2933, 1743, 1495, 1453, 1370, 1327, 1279, 1163, 1098, 859, 738, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{26}H_{34}O_5Na$ (M + Na^+) 449.2304, found 449.2278.



4

***tert*-Butyl (3-(4-methoxy-5,5-diphenylpentyl)-3-methyloxiran-2-yl)methyl carbonate (4)**

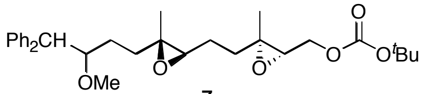
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.17 (m, 10H), 4.18 (dd, *J* = 11.9, 4.8 Hz, 1H), 4.09 (dd, *J* = 11.9, 6.3 Hz, 1H), 4.00 (d, *J* = 8.4 Hz, 1H), 3.93-3.87 (m, 1H), 3.17 (br s, 3H), 2.98-2.94 (m, 1H), 1.64-1.41 (m, 6H), 1.52 (s, 9H), 1.24 (br s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.4, 142.8, 142.4, 128.9, 128.6, 128.5, 128.4, 126.5, 126.4, 83.5, 82.6, 65.7, 60.6, 59.5, 59.4, 58.1, 58.0, 56.2, 38.3, 32.0, 27.9, 20.5, 20.5, 16.8, 16.8; IR (neat) 2934, 1742, 1495, 1452, 1369, 1279, 1255, 1163, 1098, 859, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{27}H_{36}O_5Na$ (M + Na^+) 463.2460, found 463.2462.



6

***tert*-Butyl ((2*R*,3*R*)-3-(2-((2*R*,3*R*)-3-(3-methoxy-4,4-diphenylbutyl)-3-methyloxiran-2-yl)ethyl)-3-methyloxiran-2-yl)methyl carbonate (6)**

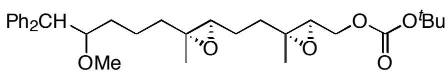
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.17 (m, 10H), 4.27-4.10 (m, 2H), 4.00-3.88 (m, 2H), 3.17/3.16 (s, 3H), 3.02 (t, *J* = 5.8 Hz, 1H), 2.63-2.59 (m, 1H), 1.84-1.38 (m, 8H), 1.52 (s, 9H), 1.32 (s, 3H), 1.14/1.13 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 142.7, 142.4, 142.3, 129.0, 128.9, 128.8, 128.7, 128.6, 128.4, 126.7, 126.5, 83.3, 82.8, 65.6, 61.1, 61.0, 60.3, 59.2, 58.2, 57.9, 56.4, 34.8, 33.9, 33.8, 27.9, 27.4, 24.3, 17.2, 16.9, 16.8, 16.4; IR (neat) 2978, 2932, 1743, 1495, 1453, 1370, 1279, 1256, 1163, 1098, 858, 756, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{31}H_{42}O_6Na$ (M + Na^+) 533.2879, found 533.2859; $[\alpha]_D$ = +17.3 ($CHCl_3$, *c* 1.49).



7

***tert*-Butyl ((2*S*,3*S*)-3-(2-((2*R*,3*R*)-3-(3-methoxy-4,4-diphenylbutyl)-3-methyloxiran-2-yl)ethyl)-3-methyloxiran-2-yl)methyl carbonate (7)**

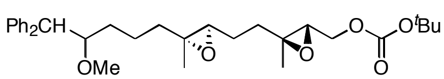
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.18 (m, 10H), 4.29-4.21 (m, 1H), 4.19-4.11 (m, 1H), 4.02-3.92 (m, 2H), 3.18/3.17 (s, 3H), 3.04 (dd, *J* = 6.2, 4.8 Hz, 1H), 2.63-2.59 (m, 1H), 1.82-1.40 (m, 8H), 1.53 (s, 9H), 1.32 (s, 3H), 1.17/1.15 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 142.7, 142.4, 142.2, 129.0, 128.7, 128.6, 128.4, 126.6, 126.4, 83.3, 82.7, 65.6, 63.1, 62.7, 60.9, 60.3, 59.8, 58.1, 57.8, 56.3, 56.1, 35.2, 33.9, 33.8, 27.9, 27.6, 27.3, 24.5, 16.9, 16.8, 16.4; IR (neat) 2977, 2932, 1742, 1495, 1453, 1370, 1279, 1256, 1163, 1097, 858, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{31}H_{42}O_6Na$ (M + Na) 533.2879, found 533.2857; $[\alpha]_D$ = +1.3 ($CHCl_3$, *c* 2.2).



8

***tert*-Butyl ((2*R*,3*R*)-3-(2-((2*R*,3*R*)-3-(4-methoxy-5,5-diphenylpentyl)-3-methyloxiran-2-yl)ethyl)-3-methyloxiran-2-yl)methyl carbonate (8)**

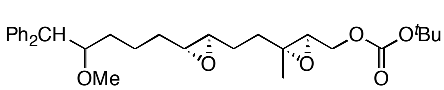
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.18 (m, 10H), 4.30-4.14 (m, 2H), 4.02 (d, *J* = 8.4 Hz, 1H), 3.98-3.85 (m, 1H), 3.19/3.18 (s, 3H), 3.05 (t, *J* = 5.7 Hz, 1H), 2.66-2.63 (m, 1H), 1.81-1.39 (m, 8H), 1.52 (s, 9H), 1.35 (s, 3H), 1.20/1.19 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 142.9, 142.5, 142.4, 129.0, 128.9, 128.7, 128.6, 128.4, 126.6, 126.4, 83.7, 83.6, 82.7, 65.6, 62.9, 62.8, 61.0, 60.3, 59.3, 58.1, 58.0, 56.3, 38.8, 38.8, 34.8, 32.2, 27.9, 24.4, 20.9, 20.8, 17.1, 16.5, 16.5; IR (neat) 3026, 2934, 1743, 1495, 1453, 1370, 1279, 1256, 1163, 1098, 859, 747, 705 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{32}H_{44}O_6Na$ (M + Na^+) 547.3036, found 547.3002; $[\alpha]_D$ = +11.0 ($CHCl_3$, *c* 2.01).



9

***tert*-Butyl ((2*S*,3*S*)-3-(2-((2*R*,3*R*)-3-(4-methoxy-5,5-diphenylpentyl)-3-methyloxiran-2-yl)ethyl)-3-methyloxiran-2-yl)methyl carbonate (9)**

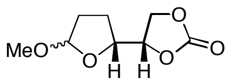
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.07 (m, 10H), 4.29 (dd, *J* = 11.9, 4.8 Hz, 1H), 4.17 (dd, *J* = 11.9, 6.3 Hz, 1H), 4.04 (d, *J* = 8.4 Hz, 1H), 3.99-3.90 (m, 1H), 3.20/3.19 (s, 3H), 3.07 (t, *J* = 5.3 Hz, 1H), 2.67-2.62 (m, 1H), 1.77-1.42 (m, 10H), 1.54 (s, 9H), 1.35 (s, 3H), 1.22 (br s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.4, 142.8, 142.4, 142.4, 128.9, 128.8, 128.6, 128.5, 128.3, 126.5, 126.4, 83.5, 83.5, 82.6, 65.6, 63.0, 62.9, 60.8, 60.8, 60.3, 59.7, 58.0, 57.9, 56.2, 38.7, 38.7, 35.1, 32.1, 27.8, 24.4, 20.8, 20.6, 16.8, 16.5, 16.4; IR (neat) 2979, 2935, 1743, 1495, 1453, 1370, 1279, 1163, 1098, 912, 859, 733, 704 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{32}H_{44}O_6Na$ (M + Na^+) 547.3036, found 547.3031; $[\alpha]_D$ = +0.5 ($CHCl_3$, *c* 1.21).



10

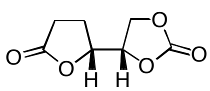
***tert*-Butyl ((2*R*,3*R*)-3-(2-((2*R*,3*R*)-3-(4-methoxy-5,5-diphenylpentyl)oxiran-2-yl)ethyl)-3-methyloxiran-2-yl)methyl carbonate (10)**

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.17 (m, 10H), 4.22 (dd, *J* = 11.8, 4.7 Hz, 1H), 4.14 (dd, *J* = 11.9, 6.0 Hz, 1H), 4.00 (d, *J* = 8.3 Hz, 1H), 3.93-3.88 (m, 1H), 3.17/3.16 (s, 3H), 3.02 (t, *J* = 5.4 Hz, 1H), 2.64-2.60 (m, 2H), 1.75-1.44 (m, 10H), 1.50 (s, 9H), 1.32 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 142.9, 142.4, 129.0, 128.7, 128.6, 128.4, 126.6, 126.4, 83.7, 82.8, 65.6, 60.2, 59.3, 58.8, 58.8, 58.2, 58.1, 56.3, 34.2, 32.2, 27.9, 27.6, 21.7, 17.1; IR (neat) 2978, 2934, 1743, 1495, 1453, 1370, 1279, 1256, 1163, 1097, 859, 746, 704 cm^{-1}; $[\alpha]_D$ = +19.1 ($CHCl_3$, *c* 1.08).

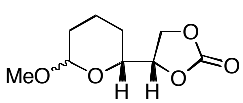


23

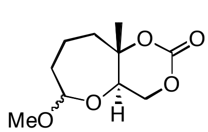
(*S*)-4-((*R*)-Tetrahydro-5-methoxyfuran-2-yl)-1,3-dioxolan-2-one (23)
To **1** (92.0 mg, 0.223 mmol) in dichloroethane/toluene (8.6 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (184 mg), anhydrous $Na_2S_2O_3$ (184 mg), NaOAc (184 mg) and *N*-methylquinolinium hexafluorophosphate (6.4 mg, 22 μmol). The mixture was photoirradiated with gentle aeration for 3 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with Et_2O (40 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (45% - 55% EtOAc in hexanes) to give the product **23** (24.8 mg, 59.0%) in a 1.9:1 diastereomeric ratio as a colorless oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.04 (dd, *J* = 4.5, 1.8 Hz, 66% of 1H), 5.01-4.99 (m, 34% of 1H), 4.67-4.46 (m, 2.4H), 4.39-4.20 (m, 1.6H), 3.33/3.32 (s, 3H), 2.26-1.93 (m, 3H), 1.74-1.66 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.1 (minor), 154.9 (major), 105.7 (minor), 105.6 (major), 79.4 (minor), 77.8 (minor), 77.1 (major), 66.8 (major), 66.4 (minor), 55.1 (major), 32.7 (minor), 31.7 (major), 25.9 (minor), 25.5 (major); IR (neat) 2920, 1807, 1464, 1376, 1170, 1088, 1031, 955 cm^{-1}; HRMS (EI): *m/z* calcd for $C_7H_9O_4$ (M^+ – CH_3O) 157.0501, found 157.0499.



(*S*)-4-((*R*)-Tetrahydro-5-oxofuran-2-yl)-1,3-dioxolan-2-one
^{1}H NMR (300 MHz, $CDCl_3$) δ 4.74 (ddd, *J* = 8.0, 6.7, 5.8 Hz, 1H), 4.64 (t, *J* = 8.9 Hz, 1H), 4.65-4.58 (m, 1H), 4.40 (dd, *J* = 8.9, 5.6 Hz, 1H), 2.68-2.62 (m, 2H), 2.60-2.50 (m, 1H), 2.20-2.10 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.2, 154.0, 78.1, 76.2, 66.7, 27.5, 24.0; IR (neat) 2919, 1778, 1462, 1401, 1328, 1173, 1087, 1048 cm^{-1}; HRMS (EI): *m/z* calcd for $C_7H_8O_5$ (M + H) 173.0450, found 173.0455.



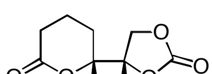
25



26

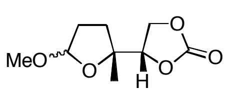
(*S*)-4-((*R*)-Tetrahydro-6-methoxy-2H-pyran-2-yl)-1,3-dioxolan-2-one (25) and (4a*R*,9a*S*)-Hexahydro-6-methoxy-4*H*-[1,3]dioxino[5,4-*b*]oxepin-2-one (26)
To diepoxide **24** (102.0 mg, 0.239 mmol) in dichloroethane/toluene (9.2 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (204 mg), anhydrous $Na_2S_2O_3$ (204 mg), NaOAc (204 mg) and *N*-methylquinolinium hexafluorophosphate (6.9 mg, 24 μmol). The mixture was photoirradiated with gentle aeration for 3 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with Et_2O (50 mL). The filtrate was concentrated and the resulting yellowish-green residue was dissolved in CH_2Cl_2 (2.0 mL). To this solution were added Et_3N (0.22 mL, 1.6 mmol), Ac_2O (57 μL, 0.6 mmol) and DMAP (2.4 mg, 20 μmol) sequentially. The mixture was stirred at room temperature for 3 h, then concentrated and purified by column chromatography (20% - 50% EtOAc in hexanes) to provide the cyclization products, which were further purified by column chromatography (4% – 10% EtOAc in CH_2Cl_2) to give **25** (14.6 mg, 30.2%, dr = 2:1) and **26** (10.6 mg, 21.9%, dr = 3.4:1) as colorless oils. **25**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.74 (app d, *J* = 2.5 Hz, 66% of 1H), 4.62-4.45 (m, 3H), 4.37 (dd, *J* = 9.3, 2.2 Hz, 34% of 1H), 3.96 (ddd, *J* = 11.8, 4.4, 1.9 Hz, 66% of 1H), 3.66 (ddd, *J* = 11.2, 5.6, 2.2 Hz, 34% of 1H), 3.46 (s, 34% of 3H), 3.36 (s, 66% of 3H), 1.98-1.16 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.1

(major), 155.0 (minor), 103.4 (minor), 98.4 (major), 78.0 (major), 77.4 (minor), 75.3 (minor), 68.2 (major), 66.4 (minor), 66.1 (major), 56.4 (minor), 55.0 (major), 30.9 (minor), 29.5 (major), 26.6 (minor), 26.5 (major), 21.2 (minor), 17.2 (major); IR (neat) 2952, 2851, 1799, 1389, 1174, 1078, 1031 cm^{-1}; HRMS (EI): *m/z* calcd for $C_8H_{11}O_4$ (M^+) 171.0657, found 171.0650. **26**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.74 (t, *J* = 4.2 Hz, 23% of 1H), 4.66 (dd, *J* = 8.6, 5.7 Hz, 77% of 1H), 4.42 (dd, *J* = 10.6, 5.8 Hz, 23% of 1H), 4.36-4.29 (m, 77% of 1H), 4.22-4.06 (m, 2.8H), 3.79 (dt, *J* = 9.7, 9.7, 5.8 Hz, 23% of 1H), 3.42 (s, 23% of 3H), 3.36 (s, 77% of 3H), 2.37-2.14 (m, 3H), 1.96-1.93 (m, 23% of 1H), 1.77-1.42 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5 (minor), 148.1 (major), 104.9 (minor), 102.8 (major), 81.2 (minor), 80.8 (major), 69.3 (major), 69.2 (minor), 68.2 (minor), 61.5 (major), 56.4 (minor), 55.8 (major), 35.5 (major), 34.2 (major), 33.5 (minor), 27.9 (minor), 17.7 (major), 16.6 (minor); IR (neat) 2943, 1760, 1403, 1382, 1224, 1140, 1057 cm^{-1}; HRMS (EI): *m/z* calcd for $C_8H_{11}O_4$ (M^+) 171.0657, found 171.0651; an analytical sample of the major diastereomer was obtained through purifying the above mixture by column chromatography (35% - 45% EtOAc in hexanes): ^{1}H NMR (300 MHz, C_6D_6) δ 4.02 (dd, *J* = 8.9, 5.8 Hz, 1H), 3.60 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.42 (t, *J* = 10.2 Hz, 1H), 3.34-3.27 (m, 2H), 2.85 (s, 3H), 1.76-1.67 (m, 1H), 1.57-1.47 (m, 1H), 1.10 (dddd, *J* = 15.3, 11.6, 8.9, 1.0 Hz, 1H), 0.95-0.85 (m, 2H), 0.82-0.71 (m, 1H).

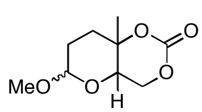


(*R*)-Tetrahydro-6-((*S*)-2-oxo-1,3-dioxolan-4-yl)pyran-2-one

To a solution of acetal **25** (6.0 mg, 30 μmol) in CH_2Cl_2 (0.6 mL) at 0 °C were added *m*-chloroperbenzoic acid (pure, 6.7 mg, 39 μmol) and BF_3·OEt_2 (4.5 μL, 36 μmol) sequentially. After stirring at 0 °C for 10 min and then at room temperature for 1.5 h, the mixture was cooled to 0 °C and Et_3N (20.7 μL, 148 μmol) was added dropwise. The mixture was stirred at 0 °C for 1 h, then concentrated, and the resulting residue was purified by column chromatography (15% - 25% EtOAc in CH_2Cl_2) to give the desired lactone (4.6 mg, 83.6%) as a colorless liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.69-4.59 (m, 2H), 4.56-4.41 (m, 2H), 2.69 (dddd, *J* = 18.0, 6.8, 4.8, 1.1 Hz, 1H), 2.54 (ddd, *J* = 17.9, 9.3, 7.0 Hz, 1H), 2.24-2.16 (m, 1H), 2.08-1.90 (m, 2H), 1.64 (dtd, *J* = 13.8, 11.0, 5.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 154.2, 79.0, 76.3, 66.9, 29.8, 24.6, 18.2; IR (neat) 2919, 1790, 1732, 1376, 1239, 1166, 1056 cm^{-1}; HRMS (EI): *m/z* calcd for $C_8H_{10}O_5$ (M^+) 186.0528, found 186.0536.



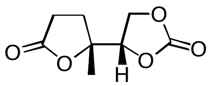
26



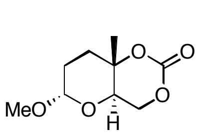
27,28

4-(Tetrahydro-5-methoxy-2-methylfuran-2-yl)-1,3-dioxolan-2-one (26) and **Hexahydro-6-methoxy-8a-methylpyrano[3,2-d][1,3]dioxin-2-one (27 and 28)**

To **3** (100 mg, 0.294 mmol) in dichloroethane/toluene (9.0 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (200 mg), anhydrous $Na_2S_2O_3$ (200 mg), NaOAc (200 mg) and *N*-methylquinolinium hexafluorophosphate (6.8 mg, 23 μmol). The mixture was photoirradiated with gentle aeration for 2.5 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (30 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (30% - 45% EtOAc in hexanes) to provide a mixture of **26** and **27** (19 mg, 40%, pale yellow oil) with a molar ratio of 4.8:1 and **28** (3.8 mg, 8.0%) as a white solid. For the mixture of **26** and **27**: IR (neat) 2929, 2835, 1791, 1755, 1463, 1375, 1170, 1084, 1034, 951 cm^{-1}. **8**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.78 (app d, *J* = 2.0 Hz, 1H), 4.66 (dd, *J* = 12.1, 2.7 Hz, 1H), 4.34 (dd, *J* =12.1, 0.4 Hz, 1H), 3.86 (app d, *J* = 2.0 Hz, 1H), 3.41 (s, 3H), 2.12-2.00 (m, 1H), 1.94 (dd, *J* = 12.8, 4.0 Hz, 1H), 1.87-1.81 (m, 1H), 1.67-1.61 (m, 1H), 1.45 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5, 98.3, 78.6, 69.3, 63.2, 55.4, 29.4, 25.3, 25.1; IR (neat) 2932, 1748, 1212, 1178, 1130, 1060, 1024 cm^{-1}; HRMS (EI): *m/z* calcd for $C_9H_{15}O_5$ (M + H$^+$) 203.0919, found 203.0929.

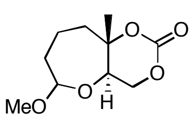


(*S*)-4-((*R*)-Tetrahydro-2-methyl-5-oxofuran-2-yl)-1,3-dioxolan-2-one To a solution of the acetal mixture **26** and **27** (18.9 mg, 93.5 μmol) in acetone (3.0 mL) at 0 °C was added Jones reagent (0.3 mL). The mixture was stirred at 0 °C for 15 min and then at room temperature for 3 h. After that time, the reaction was quenched with isopropyl alcohol (1 drop), concentrated and purified by column chromatography (2% - 20% EtOAc in CH_2Cl_2) to give the unreacted acetal **7** (2.9 mg, 15.3%) as colorless needles and the title lactone (11.0 mg, ~74.8% based on unreacted acetal): ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.72 (dd, *J* = 8.4, 6.2 Hz, 1H), 4.57 (t, *J* = 9.0 Hz, 1H), 4.36 (dd, *J* = 9.2, 6.1 Hz, 1H), 2.70 (t. *J* = 8.8 Hz, 2H), 2.34-2.24 (m, 1H), 2.19-2.09 (m, 1H), 1.47 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.0, 154.1, 83.9, 78.7, 65.4, 30.0, 28.2, 20.8; IR (neat) 2920, 1789, 1463, 1267, 1167, 1082 cm^{-1}; HRMS (EI): *m/z* calcd for $C_8H_{11}O_5$ (M + H$^+$) 187.0606, found 187.0612.



27

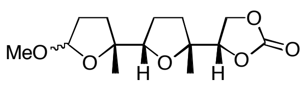
(4a*R*,6*S*,8a*S*)-Hexahydro-6-methoxy-8a-methylpyrano[3,2-d][1,3]dioxin-2-one (27)
^{1}H NMR (300 MHz, $CDCl_3$) δ 4.73 (app d, *J* = 2.4 Hz, 1H), 4.37 (dd, *J* = 8.4, 4.4 Hz, 1H), 4.18 (d, *J* = 8.2 Hz, 1H), 4.16 (d, *J* = 4.7 Hz, 1H), 3.38 (s, 3H), 2.13-2.04 (m, 1H), 1.95-1.76 (m, 3H), 1.50 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.1, 98.3, 77.5, 67.0, 62.7, 55.3, 31.1, 27.8, 17.5; IR (neat) 2917, 1757, 1464, 1196, 1111, 1068 cm^{-1}; HRMS (EI): *m/z* calcd for $C_9H_{15}O_5$ (M + H$^+$) 203.0919, found 203.0930.



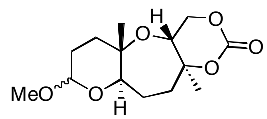
29

(4a*R*,9a*S*)-Hexahydro-6-methoxy-9a-methyl-4*H*-[1,3]dioxino[5,4-b]oxepin-2-one (29)
To *tert*-butyl carbonate **4** (65.8 mg, 149 μmol) in dichloroethane/toluene (5.7 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (132 mg), anhydrous $Na_2S_2O_3$ (132 mg, 0.832 mmol), NaOAc (132 mg, 1.60 mmol) and *N*-methylquinolinium hexafluorophosphate (4.3 mg, 15 μmol). The mixture was photoirradiated with gentle aeration for 2 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (20 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (5% - 15% EtOAc in CH_2Cl_2) to provide the desired compound **29** (23.7 mg, 73.4%) as a mixture of two diastereomers in a 1.2:1 ratio: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.76 (t, *J* = 3.8 Hz, 46% of 1H), 4.66 (dd, *J* = 8.8, 5.8 Hz, 54% of 1H), 4.34 (dd, *J* = 10.8, 6.4 Hz, 46% of 1H), 4.29-4.18 (m, 54% of 2H), 4.19 (t, *J* = 10.8 Hz, 46% of 1H), 3.88 (dd, *J* = 10.6, 6.4 Hz, 46% of 1H), 3.42 (s, 46% of 3H), 3.35 (s, 54% of 3H), 2.23-2.01 (m, ~1.5H), 1.96-1.92 (m, 46% of 1H), 1.75-1.58 (m, ~3.5H), 1.51 (s, 46% of 3H), 1.48 (s, 54% of 3H), 1.45-1.35 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.2, 148.0, 104.3, 102.8, 84.2, 83.0, 68.1, 66.7, 66.3, 62.7, 56.3, 55.7, 43.2, 41.7, 34.5, 34.4, 19.6, 19.3, 18.3, 16.7; IR (neat) 2941, 1755, 1464, 1384, 1252, 1199, 1128, 1091, 1050, 969; HRMS (EI): *m/z* calcd for $C_9H_{13}O_4Na$ (M^+ – CH_3O) 185.0814, found 185.0811; an analytical sample of the slightly major diastereomer was obtained through purifying the above mixture by column chromatography (35% - 40% EtOAc in hexanes): ^{1}H NMR (300 MHz, C_6D_6) δ 4.00 (dd, *J* = 8.8, 5.9 Hz, 1H), 3.60 (d, *J* = 10.4 Hz, 1H), 3.58 (d, *J* =

6.8 Hz, 1H), 3.44 (dd, *J* = 10.4, 6.8 Hz, 1H), 2.85 (s, 3H), 1.56-1.48 (m, 2H), 1.19-1.10 (m, 2H), 0.98-0.88 (m, 1H), 0.94 (s, 3H), 0.75-0.66 (m, 1H).



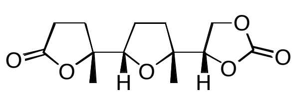
30



31

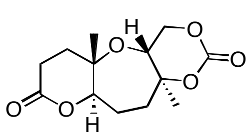
(*S*)-4-((2*R*,5*S*)-Tetrahydro-5-((*R*)-tetrahydro-5-methoxy-2-methylfuran-2-yl)-2-methylfuran-2-yl)-1,3-dioxolan-2-one (30) and (4a*R*,5a*S*,9a*R*,11a*S*)-8-Methoxy-5a,11a-dimethyldecahydro-1,3,5,9-tetraoxadibenzo[a,d]cyclohepten-2-one (31)

To diepoxide **6** (129 mg, 0.253 mmol) in dichloroethane/toluene (9.7 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (258 mg), anhydrous $Na_2S_2O_3$ (258 mg, 1.63 mmol), NaOAc (258 mg, 3.15 mmol) and *N*-methylquinolinium hexafluorophosphate (7.3 mg, 25 μmol). The mixture was photoirradiated with gentle aeration for 4.5 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (40 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (35% - 50% EtOAc in hexanes) to provide a mixture of **12** and **13** (28.7 mg, 39.6%) as a colorless oil: IR (neat) 2926, 1796, 1754, 1460, 1374, 1166, 1085, 1036, 1006, 952 cm^{-1}.



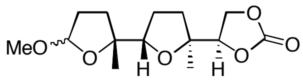
(*S*)-4-((2*R*,5*S*)-Tetrahydro-5-((*R*)-tetrahydro-2-methyl-5-oxofuran-2-yl)-2-methylfuran-2-yl)-1,3-dioxolan-2-one

A mixture of acetals **30** and **31** (21 mg, 73 μmol) in acetone (2.1 mL) at 0 °C was treated dropwise with Jones reagent (0.2 mL). The mixture was stirred at 0 °C for 10 min, then at room temperature for 1.5 h and purified without workup by column chromatography (50% - 90% EtOAc in hexanes) to give the unreacted acetal **31** (3.2 mg, 15.4%, nearly pure) and the title lactone (13.8 mg, ~80%). ^1H NMR (300 MHz, $CDCl_3$) δ 4.60 (dd, *J* = 8.4, 6.2 Hz, 1H), 4.50 (t, *J* = 8.6 Hz, 1H), 4.38 (dd, *J* = 8.7, 6.2 Hz, 1H), 4.08 (dd, *J* = 8.6, 6.3 Hz, 1H), 2.64-2.58 (m, 2H), 2.31-2.19 (m, 1H), 2.10-2.04 (m, 2H), 1.94-1.73 (m, 3H), 1.39 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.7, 155.1, 86.9, 83.5, 83.2, 79.5, 66.0, 34.1, 29.3, 29.2, 26.7, 23.8, 21.0; IR (neat) 2958, 2924, 2853, 1790, 1770, 1456, 1382, 1248, 1166, 1085, 1020, 944, 770, 728 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{13}H_{18}O_6$ (M) 270.1103, found 270.1094; $[\alpha]_D$ = +4.5 ($CHCl_3$, *c* 0.24).

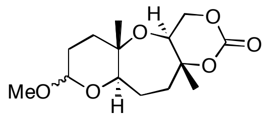


4a*R*,5a*S*,9a*R*,11a*S*)-5a,11a-Dimethyloctahydro-1,3,5,9-tetraoxadibenzo[a,d]cycloheptene-2,8-dione

A solution of acetal **31** (2.9 mg, 11 μmol) in CH_2Cl_2 (0.5 mL) at 0 °C was treated with *m*-chloroperbenzoic acid (2.5 mg, 15 μmol) and $BF_3·OEt_2$ (1.9 μL, 13 μmol) sequentially. After stirred at 0 °C for 10 min and then at room temperature for 30 min, the mixture was cooled to 0 °C and Et_3N (7.8 μL, 56 μmol) was added dropwise. The mixture was stirred at 0 °C for 30 min, and purified by column chromatography (10% - 20% EtOAc in CH_2Cl_2) to give the desired lactone (1.8 mg, 67%) as colorless needles: ^1H NMR (300 MHz, $CDCl_3$) δ 4.28 (dd, *J* = 8.6, 5.1 Hz, 1H), 4.21-4.14 (m, 1H), 4.09 (dd, *J* = 10.1, 8.6 Hz, 1H), 4.06 (dd, *J* = 11.0, 2.9 Hz, 1H), 2.80 (ddd, *J* = 18.3, 9.4, 5.5 Hz, 1H), 2.64 (ddd, *J* = 18.3, 8.7, 7.4 Hz, 1H), 2.35-2.28 (m, 1H), 2.17-1.88 (m, 5H), 1.50 (s, 3H), 1.38 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 148.2, 83.1, 82.7, 77.4, 75.7, 66.5, 65.2, 37.2, 34.3, 27.5, 24.8, 22.4, 16.0; IR (neat) 2923, 1747, 1463, 1408, 1229, 1124, 1068 cm^{-1}; ^1H NMR (300 MHz, C_6D_6) δ 3.44 (d, *J* = 7.7 Hz, 1H), 3.43 (d, *J* = 9.6 Hz, 1H), 3.18 (dd, *J* = 9.6, 7.7 Hz, 1H), 2.93 (dd, *J* = 10.9, 3.0 Hz, 1H), 2.02-1.97 (m, 2H), 1.65 (td, J = 15.3, 4.8 Hz, 1H), 1.43-1.26 (m, 3H), 1.17-1.04 (m, 2H), 0.81 (s, 3H), 0.48 (s, 3H); HRMS (ESI): m/z calcd for $C_{13}H_{18}O_6Na$ (M + Na^+) 293.1001, found 293.1020; $[\alpha]_D$ = +101 ($CHCl_3$, c 0.15).



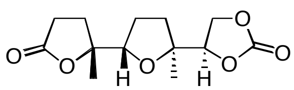
32



33

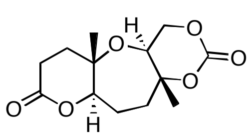
(*R*)-4-((2*S*,5*S*)-tetrahydro-5-((*R*)-tetrahydro-5-methoxy-2-methylfuran-2-yl)-2-methylfuran-2-yl)-1,3-dioxolan-2-one (32) and (4aS,5a*S*,9a*R*,11a*R*)-8-Methoxy-5a,11a-dimethyldecahydro-1,3,5,9-tetraoxadibenzo[a,d]cyclohepten-2-one (33)

To diepoxide **7** (150 mg, 0.294 mmol) in dichloroethane/toluene (11.3 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (300 mg), anhydrous $Na_2S_2O_3$ (300 mg, 1.90 mmol), NaOAc (300 mg, 3.66 mmol) and *N*-methylquinolinium hexafluorophosphate (8.5 mg, 29 μmol). The mixture was photoirradiated with gentle aeration for 4.5 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (40 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (35% - 50% EtOAc in hexanes) to provide a mixture of **32** and **33** (51.2 mg, 60.9%): IR (neat) 2925, 1797, 1750, 1462, 1384, 1259, 1167, 1120 cm^{-1}.



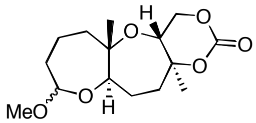
(*R*)-4-((2*S*,5*S*)-tetrahydro-5-((*R*)-tetrahydro-2-methyl-5-oxofuran-2-yl)-2-methylfuran-2-yl)-1,3-dioxolan-2-one

A mixture of acetals **32** and **33** (34.9 mg, 122 μmol) in acetone (1.8 mL) at 0 °C was treated dropwise with Jones reagent (0.3 mL). The mixture was stirred at 0 °C for 10 min, then at room temperature for 1.5 h and purified without workup by column chromatography (50% - 90% EtOAc in hexanes) to give the unreacted acetal **16** (4.9 mg, nearly pure) and the title lactone (22.1 mg, ~81%). For the title lactone: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.58 (dd, J = 8.3, 6.1 Hz, 1H), 4.48 (t, J = 8.4 Hz, 1H), 4.32 (dd, J = 8.8, 6.1 Hz, 1H), 4.08 (dd, J = 8.8, 5.6 Hz, 1H), 2.65-2.59 (m, 2H), 2.24 (ddd, J = 12.9, 9.6, 6.9 Hz, 1H), 2.07-1.81 (m, 5H), 1.38 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.7, 155.0, 86.9, 85.1, 83.1, 80.3, 66.2, 34.5, 29.4, 29.1, 27.0, 23.4, 21.3; IR (neat) 2979, 2880, 1790, 1767, 1454, 1382, 1170, 1111, 1085, 944 cm^{-1}; HRMS (EI): m/z calcd for $C_{13}H_{18}O_6$ (M) 270.1103, found 270.1095; $[\alpha]_D$ = -11.3 ($CHCl_3$, c 1.03).



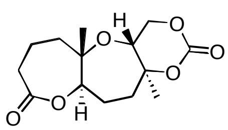
(4a*S*,5a*S*,9a*R*,11a*R*)-5a,11a-Dimethyloctahydro-1,3,5,9-tetraoxadibenzo[a,d]cycloheptene-2,8-dione

A solution of acetal **33** (4.5 mg, 16 μmol) in CH_2Cl_2 (0.5 mL) at 0 °C was treated with *m*-chloroperbenzoic acid (3.5 mg, 20 μmol) and $BF_3{\cdot}OEt_2$ (2.7 μL, 19 μmol) sequentially. After stirring at 0 °C for 10 min and at room temperature for 20 min, the mixture was cooled to 0 °C and Et_3N (10.9 μL, 78.5 μmol) was added dropwise. The mixture was stirred at 0 °C for 30 min and purified by column chromatography (15% - 25% EtOAc in CH_2Cl_2) to give the desired lactone (3.0 mg, 71%) as colorless needles: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.40 (app dd, J = 10.0, 2.2 Hz, 1H), 4.23 (dd, J = 10.5, 6.1 Hz, 1H), 4.09 (t, J = 10.2, Hz, 1H), 4.00 (dd, J = 10.1, 6.1 Hz, 1H), 2.88 (ddd, J = 18.3, 11.2, 4.7 Hz, 1H), 2.72 (ddd, J = 18.3, 9.6, 5.5 Hz, 1H); 2.21-1.76 (m, 6H), 1.49 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 147.4, 83.8, 81.4, 77.4, 66.6, 64.9, 39.1, 30.8, 28.1, 24.4, 20.5, 19.5; IR (neat) 2924, 1748, 1463, 1408, 1229, 1124, 1068 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{13}H_{18}O_6Na$ (M + Na) 293.1001, found 293.0988; $[\alpha]_D$ = +48.7 ($CHCl_3$, *c* 0.23).


34

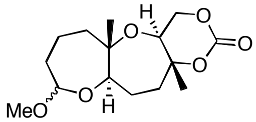
(4a*R*,5a*S*,10a*R*,12a*S*)-9-Methoxy-5a,12a-dimethyldecahydro-1,3,5,10-tetraoxabenzo[b]heptalen-2-one (34)

To diepoxide **8** (145 mg, 276 μmol) in dichloroethane/toluene (10.6 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (290 mg), anhydrous $Na_2S_2O_3$ (290 mg, 1.83 mmol), NaOAc (290 mg, 3.53 mmol) and *N*-methylquinolinium hexafluorophosphate (8.0 mg, 28 μmol). The mixture was photoirradiated with gentle aeration for 2 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (40 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (25% - 35% EtOAc in hexanes) to provide **34** (44.8 mg, 54.0%, pale yellow liquid) as two diastereomers in an approximately 1:1 ratio: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.54-4.48 (m, 1H), 4.25-3.98 (m, 3H), 3.92-3.85 (m, 0.5H), 3.56 (dd, *J* = 11.2, 2.4 Hz, 0.5H), 3.40/3.37 (s, 3H), 3.24 (dd, *J* = 10.8, 3.8 Hz, 0.5H), 2.26-1.94 (m, 2.5H), 1.91-1.72 (m, 3.5H), 1.65-1.52 (m, 3.5H), 1.47/1.44 (s, 3H), 1.33/1.29 (s, 3H), 1.21-1.18 (m, 0.5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.0, 148.6, 105.2, 102.6, 83.3, 83.2, 81.4, 80.3, 79.7, 75.6, 67.0, 67.0, 65.2, 63.8, 56.1, 55.9, 44.6, 43.3, 37.5, 37.0, 35.3, 33.6, 27.6, 26.2, 22.3, 21.6, 19.2, 17.7, 17.0, 16.7; IR (neat) 2940, 1759, 1454, 1384, 1209, 1111, 1053, 1008, 921; HRMS (ESI): *m*/*z* calcd for $C_{15}H_{24}O_6Na$ (M + Na$^+$) 323.1471, found 323.1500; $[\alpha]_D$ = +31.5 ($CHCl_3$, *c* 1.45).


38

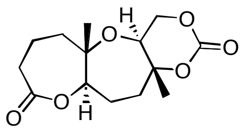
(4a*R*,5a*S*,10a*R*,12a*S*)-5a,12a-Dimethyldecahydro-1,3,5,10-tetraoxabenzo[b]heptalene-2,9-dione (38)

A solution of acetal **34** (15.6 mg, 51.9 μmol) in CH_2Cl_2 (0.5 mL) at 0 °C was treated with *m*-chloroperbenzoic acid (11.6 mg, 67.5 μmol) and $BF_3{\cdot}OEt_2$ (7.2 μL, 57 μmol) sequentially. After stirring at 0 °C for 10 min, then at room temperature for 1 h, the mixture was cooled to 0 °C and Et_3N (36.2 μL, 256 μmol) was added dropwise. The mixture was stirred at 0 °C for 1.5 h, then quenched with a mixture of saturated $NaHCO_3$/ saturated $Na_2S_2O_3$ (4 mL, 1:1, v/v). The mixture was poured onto water (5 mL) and extracted with Et_2O (3 x 25 mL). The extracts were dried over $MgSO_4$, filtered and concentrated, and the resulting residue was purified by column chromatography (10% - 20% EtOAc in CH_2Cl_2) to give lactone **21** (9.9 mg, 67%) as a white crystalline solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.26-4.20 (m, 2H), 4.14-4.06 (m, 2H), 2.70-2.55 (m, 2H), 2.35-2.23 (m, 2H), 1.99-1.81 (m, 4H), 1.77-1.68 (m, 2H), 1.46 (s, 3H), 1.31 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.3, 148.6, 84.3, 82.2, 78.9, 66.8, 64.3, 43.2, 36.2, 33.6, 26.6, 22.0, 20.0, 15.8; IR (neat) 2989, 2941, 2871, 1748, 1727, 1501, 1454, 1365, 1328, 1272, 1212, 1098, 1040 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{14}H_{20}O_6Na$ (M + Na) 307.1158, found 307.1158; $[\alpha]_D$ = +50.4 ($CHCl_3$, *c* 0.42).


35

(4a*S*,5a*S*,10a*R*,12a*R*)-9-Methoxy-5a,12a-dimethyldecahydro-1,3,5,10-tetraoxabenzo[b]heptalen-2-one (35)

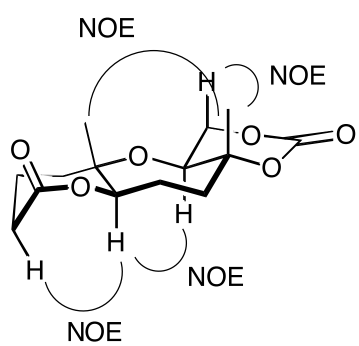
To diepoxide **9** (48.2 mg, 91.9 μmol) in dichloroethane/toluene (3.5 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (96 mg), anhydrous $Na_2S_2O_3$ (96 mg), NaOAc (96 mg) and *N*-methylquinolinium hexafluorophosphate (2.6 mg, 9.2 μmol). The mixture was photoirradiated with gentle aeration for 3 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (30 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (25% - 35% EtOAc in hexanes) to provide **23** (22 mg, 79%, pale yellow needles) as two diastereomers in about 1:1 ratio: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.69 (dd, *J* = 3.8, 2.2 Hz, 0.5H), 4.54 (dd, *J* = 8.9, 5.7 Hz, 0.5H), 4.17 (dd, *J* = 10.7, 6.6 Hz, 1H), 4.02 (t, *J* = 10.7 Hz, 1H), 3.90 (dd, *J* = 10.7, 6.6 Hz, 1H), 3.90-3.85 (m, 0.5H), 3.52 (dd, *J* = 10.1, 0.8 Hz, 0.5H), 3.40/3.37 (s, 3H), 2.08-2.00 (m, 2H), 1.89-1.53 (m, 8H), 1.44 (s, 3H), 1.21/1.17 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.6, 148.0, 102.7, 102.4, 83.6, 83.6, 81.3, 80.6, 78.8, 74.1, 67.0 (2C), 64.0, 63.9, 56.0, 55.8, 40.5, 40.2, 39.8, 39.5, 33.7, 33.4, 27.3 (2C), 20.8, 20.3, 19.4, 19.3, 19.3, 17.5; IR (neat) 2940, 1755, 1461, 1382, 1246, 1223, 1116, 1051, 913 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{15}H_{24}O_6Na$ (M + Na^+) 323.1471, found 323.1462; $[\alpha]_D$ = +26.6 ($CHCl_3$, *c* 0.55).

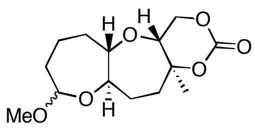


(4a*S*,5a*S*,10a*R*,12a*R*)-5a,12a-Dimethyldecahydro-1,3,5,10-tetraoxabenzo[b]heptalene-2,9-dione

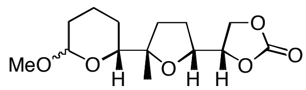
A solution of acetal **35** (19.0 mg, 63.2 μmol) in CH_2Cl_2 (2.0 mL) at 0 °C was treated with *m*-chloroperbenzoic acid (14.2 mg, 82.2 μmol) and $BF_3·OEt_2$ (9.5 μL, 76 μmol) sequentially. The mixture was stirred at 0 °C for 10 min, and then at room temperature for 1 h. After that time, the mixture was cooled to 0 °C and Et_3N (44.0 μL, 316 μmol) was added dropwise. The mixture was stirred at 0 °C for 30 min, then concentrated, and the resulting residue was purified by column chromatography (15% - 25% EtOAc in CH_2Cl_2) to give the desired lactone (14.4 mg, 80.0%) as colorless needles: ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.47 (dd, *J* = 10.4 Hz, 1H), 4.20 (dd, *J* = 10.7, 6.4 Hz, 1H), 4.05 (t, *J* = 10.7 Hz, 1H), 3.98 (dd, *J* = 10.5, 6.4 Hz, 1H), 2.70 (dt, *J* = 14.1, 14.1, 2.2 Hz, 1H), 2.64 (ddd, *J* = 14.1, 5.8, 1.3 Hz, 1H), 2.12 (ddd, *J* = 13.6, 5.9, 2.0 Hz, 1H), 2.07-1.98 (m, 3H), 1.90 (dddd, *J* = 14.7, 5.8, 2.6, 1.0 Hz, 1H), 1.84 (app dt, *J* = 13.6, 13.6, 1.7 Hz, 1H), 1.78-1.70 (m, 2H), 1.48 (s, 3H), 1.14 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.4, 147.8, 83.6, 82.7, 79.0, 66.8, 64.6, 39.3, 38.3, 33.4, 26.4, 20.4, 19.14, 19.10; IR (neat) 2984, 2941, 1747, 1732, 1444, 1388, 1274, 1252, 1200, 1116, 1100, 1070, 1049 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{14}H_{20}O_6$ (M^+) 284.1260, found 284.1254; $[\alpha]_D$ = +17.2 ($CHCl_3$, *c* 0.52).

Key observations from NOESY spectrum





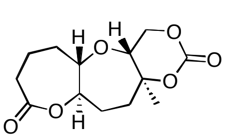
36



37

(4a*R*,5a*S*,10a*R*,12a*S*)-9-Methoxy-12a-methyldecahydro-1,3,5,10-tetraoxabenzo[b]heptalen-2-one (36) and **(*S*)-4-((2*R*,5*S*)-tetrahydro-5-((*R*)-tetrahydro-6-methoxy-2*H*-pyran-2-yl)-2-methylfuran-2-yl)-1,3-dioxolan-2-one (37)**

To diepoxide **10** (52.8 mg, 103 μmol) in dichloroethane/toluene (4.0 mL, 5:1, v/v) in a borosilicate flask at room temperature were added activated 4Å molecular sieves (106 mg), anhydrous $Na_2S_2O_3$ (106 mg,), NaOAc (106 mg) and *N*-methylquinolinium hexafluorophosphate (3.0 mg, 10 μmol). The mixture was photoirradiated with gentle aeration for 4 h while stirring at room temperature. The reaction mixture was filtered through a small plug of silica gel and the residue was washed with EtOAc (20 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography (5% - 20% EtOAc in CH_2Cl_2) to provide the *endo*, *endo* product **36** (8.8 mg, 30%) as a colorless oil and the *exo*, *exo* product **37** (7.3 mg, 25%) as a white solid. **36** (dr = 2.3:1): ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.56 (dd, *J* = 8.8, 5.8 Hz, 70% of 1H), 4.49-4.46 (m, 30% of 1H), 4.39-4.34 (m, 1H), 4.11 (t, *J* = 10.6 Hz, 70% of 1H), 4.10 (t, *J* = 10.6 Hz, 30% of 1H), 3.94 (dd, *J* = 11.3, 6.5 Hz, 70% of 1H), 3.84 (dd, *J* = 11.0, 6.3 Hz, 30% of 1H), 3.68 (dt, *J* = 8.5, 4.5 Hz, 70% of 1H), 3.62-3.59 (m, 30% of 1H), 3.49-3.47 (m, 30% of 1H), 3.42 (s, 30% of 3H), 3.38 (s, 70% of 3H), 3.37-3.33 (70% of 1H), 2.22-1.97 (m, 4H), 1.92-1.78 (m, 2H), 1.65-1.59 (m, 2H), 1.46 (s, 30% of 3H), 1.43 (s, 70% of 3H), 1.38-1.33 (m, 1H), 1.28-1.25 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 149.0 (minor), 148.9 (major), 106.9 (minor), 102.6 (major), 86.4 (major), 83.3 (minor), 82.6 (minor), 82.4 (major), 79.5 (minor), 75.2 (minor), 74.0 (major), 73.2 (minor), 73.1 (major), 66.5 (major), 56.1 (minor), 55.9 (major), 39.5 (minor), 36.7 (major), 35.9 (minor), 35.6 (major), 34.9 (minor), 33.5 (major), 29.7 (major), 28.6 (minor), 28.0 (minor), 21.0 (major), 18.9 (major),17.9 (minor); IR (neat) 2939, 1755, 1455, 1384, 1255, 1205, 1109, 1042, 999 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{14}H_{22}O_6$ (M^+) 286.1416, found 286.1414; $[\alpha]_D$ = +11.8 ($CHCl_3$, *c* 0.85). **37** (dr = 2:1): ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.71 (br s, 67% of 1H), 4.61-4.56 (m, 1H), 4.54-4.50 (m, 1H), 4.44-4.41 (m, 1H), 4.32 (dd, *J* = 9.5, 2.0 Hz, 33% of 1H), 4.02 (dd, *J* = 7.1, 4.9 Hz, 33% of 1H), 3.98 (dd, *J* = 7.4, 4.4 Hz, 67% of 1H), 3.73-3.70 (ddd, *J* = 11.6, 4.2, 2.0 Hz, 67% of 1H), 3.48 (s, 33% of 3H), 3.40 (ddd, *J* = 11.3, 4.7, 1.9 Hz, 67% of 1H), 3.33 (s, 67% of 3H), 2.05-1.95 (m, 4H), 1.90-1.78 (m, 3H), 1.73-1.65 (m, 1H), 1.60 (s, 3H), 1.55-1.48 (m, 1H), 1.31-1.22 (m, 1H), 1.28 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.3 (major), 151.1 (minor), 103.7 (minor), 98.7 (major), 82.2 (major, 2C), 81.9 (minor), 79.6 (minor), 79.2 (major), 69.7 (major), 66.0 (major), 56.3 (minor), 54.7 (major), 35.1 (major), 34.7 (minor), 31.3 (minor), 29.9 (major), 27.6 (minor), 27.3 (major), 26.7 (minor), 26.3 (major), 22.0 (minor), 20.9 (minor), 20.5 (major), 17.8 (major); IR (neat) 2943, 1798, 1455, 1374, 1166, 1033, 949 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{14}H_{22}O_6$ (M^+) 286.1416, found 286.1419; $[\alpha]_D$ = -24.1 ($CHCl_3$, *c* 0.71).



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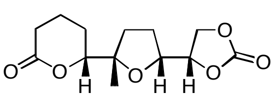
(4a*R*,5a*S*,10a*R*,12a*S*)-12a-Methyldecahydro-1,3,5,10-tetraoxa-benzo[b]heptalene-2,9-dione (39)

To a solution of acetal **36** (8.0 mg, 28 μmol) in CH_2Cl_2 (0.5 mL) at 0 °C were added *m*-chloroperbenzoic acid (6.2 mg, 36 μmol) and $BF_3·OEt_2$ (4.2 μL, 33 μmol) sequentially. After stirring at room temperature for 30 min, the mixture was cooled to 0 °C and Et_3N (19.4 μL, 140 μmol) was added dropwise. The mixture was stirred at 0 °C for 30 min, then concentrated, and the resulting residue was purified by column chromatography (10% - 20% EtOAc in CH_2Cl_2) to give lactone **39** (5.3 mg, 70.2%) as a white crystalline solid: ^{1}H NMR

(600 MHz, $CDCl_3$) δ 4.43-4.39 (m, 1H), 4.40 (dd, *J* = 10.4, 6.5 Hz, 1H), 4.13 (dd, *J* = 11.2, 10.5 Hz, 1H), 3.86 (dd, *J* = 11.3, 6.5 Hz, 1H), 3.53 (ddd, *J* = 10.6, 8.0, 3.4 Hz, 1H), 2.70-2.61 (m, 2H), 2.22-2.17 (m, 3H), 2.07-2.01 (m, 2H), 1.92 (ddd, *J* = 15.4, 9.6, 2.2 Hz, 1H),1.77-1.73 (m, 2H), 1.48 (s, 3H); ^{1}H NMR (500 MHz, C_6D_6) δ 3.58 (dd, *J* = 10.0, 6.6 Hz, 1H), 3.46 (dd, *J* = 11.2, 10.2 Hz, 1H), 3.38-3.34 (m, 1H), 2.66 (dd, *J* = 11.2, 6.6 Hz, 1H), 2.58 (ddd, *J* = 11.2, 7.8, 3.3 Hz, 1H), 2.22-2.18 (m, 1H), 1.73-1.65 (m, 2H), 1.52 (dddd, *J* = 15.8, 8.8, 3.8, 1.4 Hz, 1H), 1.42-1.38 (m, 1H), 1.30 (ddd, *J* = 14.4, 8.8, 1.4 Hz, 1H), 1.22 (dddd, *J* = 17.1, 11.6, 5.2, 1.5 Hz, 1H), 1.16-1.11 (m, 2H), 0.92-0.84 (m, 1H), 0.80 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.9, 148.2, 85.5, 81.9, 81.2, 78.4, 66.5, 35.7, 34.5, 33.6, 27.5, 21.0, 19.2; IR (neat) 2922, 2850, 1747, 1453, 1387, 1273, 1204, 1106, 1058, 1015 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{13}H_{18}O_6$ (M^+) 270.1103, found 270.1111; $[\alpha]_D$ = +12.7 ($CHCl_3$, *c* 0.26).



(*R*)-Tetrahydro-6-((2*S*,5*R*)-tetrahydro-5-methyl-5-((*S*)-2-oxo-1,3-dioxolan-4-yl)furan-2-yl)pyran-2-one

To a solution of acetal **37** (6.8 mg, 24 μmol) in CH_2Cl_2 (0.5 mL) at 0 °C were added *m*-chloroperbenzoic acid (5.3 mg, 31 μmol) and $BF_3{\cdot}OEt_2$ (4.0 μL, 28 μmol) sequentially. After stirred at room temperature for 30 min, the mixture was cooled to 0 °C and Et_3N (16.5 μL, 118 μmol) was added dropwise. The mixture was stirred at 0 °C for 30 min, then concentrated, and the resulting residue was purified by column chromatography (15% - 25% EtOAc in CH_2Cl_2) to give the desired lactone (5.2 mg, 81%) as a white solid: ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.64 (dd, *J* = 8.4, 6.0 Hz, 1H), 4.52 (t, *J* = 8.8 Hz, 1H), 4.45 (dd, *J* = 8.8, 6.0 Hz, 1H), 4.30 (dd, *J* = 11.4, 4.6, 3.0 Hz, 1H), 4.10 (dt, *J* = 7.2, 4.6 Hz, 1H), 2.62 (dddd, *J* = 17.8, 6.6, 4.8, 1.4 Hz, 1H), 2.46 (ddd, *J* = 17.8, 9.3, 7.0 Hz, 1H), 2.19-2.12 (m, 1H), 2.07-2.02 (m, 1H), 2.01-1.93 (m, 3H), 1.90-1.85 (m, 1H), 1.84-1.79 (m, 1H), 1.28 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.1, 155.2, 83.0, 81.1, 80.7, 79.0, 66.1, 34.5, 29.9, 26.4, 24.8, 20.7, 18.5; IR (neat) 2957, 2929, 1789, 1731, 1242, 1173, 1084, 1049, 1018, 771 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{13}H_{18}O_6$ (M^+) 270.1103, found 270.1104; $[\alpha]_D$ = -42.8 ($CHCl_3$, *c* 0.50).

Coordinates for all of the TSs.

The geometries of the stationary points (ground state structures and transition state structures) were optimized at B3LYP/6-31G(d) level. Ground state structures have all positive frequencies and TS structures have one imaginary frequencies corresponding to motions along reaction coordinates.

TS1

Electronic Energy: -654.801107 Hartree

O	2.908183	0.145304	0.385906
C	3.731982	1.219492	-0.205787
C	1.585963	0.027196	-1.819110
C	1.730256	-0.455806	-0.385927
C	0.930360	0.283134	0.641281
C	0.663739	-0.223573	2.013053
C	1.819736	-1.963716	-0.253651
H	0.655100	-0.374024	-2.227483
H	1.540319	1.116977	-1.895697
H	0.822569	1.345457	0.447786
H	0.859678	-2.416380	-0.521607
H	2.584529	-2.350543	-0.932382
H	2.087100	-2.263247	0.761544
H	0.308757	-1.256026	2.003755
H	4.203080	0.863055	-1.123202
H	4.486940	1.422182	0.552894
H	1.604521	-0.200945	2.578965
H	3.149027	2.123092	-0.402547
H	2.412686	-0.338478	-2.434160
H	-0.054617	0.406745	2.540696
O	-2.774376	1.282710	0.380185
C	-2.217261	0.124852	0.007286
O	-0.982847	-0.085203	-0.188393
O	-3.161469	-0.801566	-0.158610
C	-2.776717	-2.170608	-0.571903
H	-2.106165	-2.606530	0.169923
H	-3.721562	-2.705578	-0.609583
H	-2.302950	-2.136762	-1.553741
C	-1.953179	2.502158	0.496612
H	-2.681310	3.304532	0.582247
H	-1.339429	2.452121	1.398376
H	-1.340451	2.629130	-0.397970

TS2

Electronic Energy: -654.807793 Hartree

C	-0.701275	1.632401	-1.841478
O	-0.009792	0.337241	-1.790982
C	0.631331	-0.033091	-0.502573
C	2.114849	-0.254321	-0.711291
C	-0.268882	-1.229777	-0.281408
C	0.007893	-2.470642	-1.047088
C	-1.640867	-1.001252	0.256233
O	0.674942	-1.750694	1.579590
C	0.537546	-2.176865	2.764099
O	0.412318	-3.458939	3.117028
C	0.483945	-4.519778	2.091065
O	0.535429	-1.404974	3.855198
C	0.721248	0.054459	3.721670
H	-2.026354	-1.896077	0.750279
H	-1.695131	-0.149511	0.935461
H	0.438136	0.732371	0.254221
H	1.031900	-2.816778	-0.894331
H	-0.700156	-3.269340	-0.824050
H	-0.083858	-2.211843	-2.113542
H	2.301953	-0.930524	-1.549125
H	-0.989286	1.757408	-2.884684
H	-0.022995	2.435820	-1.539080
H	2.591119	0.702204	-0.945165
H	-1.595735	1.634631	-1.208551
H	-2.310088	-0.808464	-0.596479
H	2.574505	-0.661881	0.192554
H	1.349101	-4.357088	1.446789
H	0.588917	-5.435806	2.666239
H	-0.440973	-4.533839	1.512123
H	0.797346	0.403677	4.747874
H	1.636715	0.263233	3.166364
H	-0.146753	0.494983	3.227694

TS3_exo

Electronic Energy: -691.912189235 Hartree

O -1.804015 -1.086175 0.167616
C -3.037005 -0.826979 -0.614784
C -3.632731 0.546424 -0.364710
C -2.563700 1.634738 -0.503288
C -1.495664 1.444133 0.581553

C -0.919886 0.033674 0.669771
C -0.253554 -0.515202 -0.522756
C 0.656794 -1.730199 -0.523955
C -0.403177 -0.331270 2.045105
H -0.576902 -0.120724 -1.477675
H -4.083487 0.586798 0.634436
H -3.009677 2.626612 -0.386198
H -4.443811 0.689043 -1.087650
H -2.123762 1.613600 -1.509622
H -1.932871 1.687891 1.556371
H -0.646615 2.121764 0.434329
H 0.803876 -2.088018 -1.546589
O 1.944349 -1.422080 0.040638
H 0.242749 -2.532710 0.085243
H 0.352859 0.400359 2.349923
H -1.224864 -0.283143 2.765352
H 0.037902 -1.328630 2.088325
H -2.776392 -0.967334 -1.668218
H -3.692041 -1.643215 -0.303233
C 2.320289 -0.171740 -0.242781
O 3.563335 0.042695 0.083925
O 1.528679 0.639489 -0.737188
C 4.086452 1.381212 -0.152580
H 5.119226 1.335174 0.185923
H 4.032278 1.613632 -1.217162
H 3.515826 2.108727 0.427072

TS3_endo

Electronic Energy: -691.918988090 Hartree

O 1.860390 1.146094 0.310337
C 2.911233 1.040794 -0.714536
C 3.467838 -0.373441 -0.830704
C 2.385886 -1.438084 -1.052978
C 1.492095 -1.650113 0.193008
C 0.903184 -0.422472 0.803950
C 0.522402 0.697433 -0.070927
C -0.557816 1.710029 0.285524
C 0.729649 -0.366881 2.274929
H 0.468907 0.448616 -1.132133
H 4.052446 -0.613057 0.066083
H 2.859098 -2.400368 -1.268542
H 4.169339 -0.374253 -1.672806
H 1.781161 -1.193832 -1.934745
H 2.038064 -2.209842 0.958897

H 0.612694 -2.259115 -0.076317
H -0.253080 2.691321 -0.084141
O -1.784769 1.422266 -0.390615
H -0.729517 1.775802 1.362408
H -0.121728 -1.023454 2.506817
H 1.606970 -0.769330 2.788777
H 0.500981 0.629135 2.654742
H 2.479648 1.393323 -1.657299
H 3.667185 1.751485 -0.375797
C -2.321244 0.208532 -0.137783
O -3.529799 0.164501 -0.653134
O -1.740781 -0.688185 0.457119
C -4.247266 -1.085944 -0.501349
H -5.200669 -0.921818 -0.999795
H -3.692957 -1.896545 -0.978510
H -4.394636 -1.304586 0.557898

TS4_trans_exo

Electronic Energy: -806.440823512 Hartree

O -1.418502 -0.431474 0.255836
C -2.697312 0.022304 -0.469949
C -2.897493 1.519268 -0.360454
C -1.604458 2.296206 -0.621429
C -0.577666 1.977286 0.470550
C -0.314868 0.485473 0.663732
C 0.235795 -0.245021 -0.495681
C 0.832834 -1.639836 -0.421936
C 0.157474 0.128042 2.059064
H -0.021082 0.146959 -1.471275
H -3.284722 1.737995 0.641430
H -1.808518 3.370772 -0.614776
H -3.681308 1.792159 -1.074785
H -1.211948 2.071452 -1.622625
H -0.933150 2.385931 1.423188
H 0.389572 2.448756 0.261812
H 0.864716 -2.092611 -1.416493
O 2.176732 -1.601701 0.098543
H 0.264869 -2.278526 0.252505
H 1.048547 0.715284 2.306058
H -0.624727 0.384983 2.779070
H 0.393769 -0.931822 2.172041
H -2.517907 -0.315089 -1.499904
C 2.796968 -0.470605 -0.235548
O 4.063926 -0.500989 0.062273

O 2.176383 0.470091 -0.751025
C 4.841618 0.696313 -0.228522
H 5.851570 0.450766 0.092308
H 4.809007 0.904043 -1.299031
H 4.444355 1.540256 0.337807
O -3.718145 -0.636308 0.128294
C -3.856082 -2.033309 -0.174951
H -4.821011 -2.334998 0.232079
H -3.846535 -2.195630 -1.259906
H -3.059508 -2.614360 0.299867

TS4_trans_endo

Electronic Energy: -806.447709573 Hartree

O -1.514269 -0.446508 0.455266
C -2.588468 -0.135915 -0.561221
C -2.710533 1.368142 -0.758856
C -1.396779 2.078148 -1.114469
C -0.394976 2.164447 0.062043
C -0.076777 0.889080 0.762420
C -0.145360 -0.371117 -0.001167
C 0.673987 -1.590545 0.399441
C 0.134908 0.929661 2.229093
H -0.080935 -0.233821 -1.083977
H -3.149261 1.789342 0.153348
H -1.619249 3.107238 -1.411177
H -3.439873 1.521018 -1.561376
H -0.930271 1.611240 -1.990287
H -0.725223 2.909174 0.793752
H 0.582292 2.510870 -0.314652
H 0.152704 -2.489551 0.065255
O 1.938314 -1.624642 -0.276062
H 0.828419 -1.657460 1.478992
H 0.991982 1.589417 2.417959
H -0.733063 1.393279 2.711572
H 0.331800 -0.039888 2.683532
H -2.256695 -0.661811 -1.471373
C 2.722630 -0.539574 -0.141819
O 3.909236 -0.806777 -0.638389
O 2.361687 0.525451 0.348086
C 4.880378 0.269652 -0.609978
H 5.773091 -0.150059 -1.069511
H 4.511356 1.122090 -1.183622
H 5.076738 0.566872 0.421767
O -3.751337 -0.619888 -0.062516

C -3.919101 -2.046881 -0.092452
H -4.957119 -2.231977 0.182995
H -3.730835 -2.435617 -1.100964
H -3.254361 -2.532035 0.628491

TS4_cis_exo

Electronic Energy: -806.443127663 Hartree

O 1.467844 0.178356 0.944665
C 2.795512 0.137066 0.195422
C 3.106456 -1.265374 -0.293084
C 1.927390 -1.878452 -1.053099
C 0.744105 -2.068354 -0.096915
C 0.364568 -0.821989 0.697585
C -0.020481 0.377810 -0.062306
C -0.746010 1.572927 0.530136
C -0.324769 -1.126956 2.011475
H 0.405421 0.481612 -1.050667
H 3.366175 -1.890281 0.569707
H 2.212359 -2.852787 -1.460271
H 3.997040 -1.191400 -0.924434
H 1.660956 -1.251137 -1.911751
H 0.986719 -2.862061 0.619025
H -0.157618 -2.387504 -0.632361
H -0.689667 2.422993 -0.155174
O -2.133750 1.278615 0.774581
H -0.323764 1.851996 1.494539
H -1.213317 -1.738237 1.820204
H 0.349930 -1.707370 2.647204
H -0.627791 -0.230753 2.555698
C -2.633035 0.451743 -0.149183
O -3.928960 0.346797 -0.048291
O -1.899338 -0.126416 -0.957878
C -4.590749 -0.549660 -0.985276
H -5.643428 -0.499842 -0.715428
H -4.430385 -0.197722 -2.005526
H -4.202515 -1.562518 -0.865762
H 3.463852 0.476973 0.993946
O 2.753552 1.014950 -0.855753
C 2.983252 2.396904 -0.533009
H 3.950030 2.517135 -0.031696
H 2.995858 2.931594 -1.482905
H 2.187919 2.796906 0.106589

TS4_cis_endo

Electronic Energy: -806.450332320 Hartree

O 1.584817 0.089738 1.002323
C 2.763452 0.267130 0.071709
C 3.007227 -1.011904 -0.721314
C 1.784396 -1.513009 -1.500725
C 0.690560 -2.107994 -0.582267
C 0.256767 -1.257602 0.561849
C 0.261121 0.203715 0.399354
C -0.670964 1.108463 1.194531
C -0.103612 -1.917336 1.839370
H 0.325449 0.552632 -0.630921
H 3.362310 -1.786816 -0.031519
H 2.095887 -2.311640 -2.179941
H 3.827122 -0.796138 -1.413654
H 1.383528 -0.713716 -2.131881
H 0.995952 -3.092315 -0.212761
H -0.236147 -2.262402 -1.160404
H -0.162194 2.052766 1.395845
O -1.826658 1.469021 0.428889
H -0.979035 0.666126 2.144959
H -1.051174 -2.445641 1.661365
H 0.642090 -2.672175 2.106779
H -0.247402 -1.227416 2.670426
C -2.582735 0.444443 -0.014538
O -3.692397 0.927899 -0.529422
O -2.255608 -0.733094 0.046300
C -4.614085 -0.039417 -1.090676
H -5.448809 0.553324 -1.459951
H -4.134413 -0.585108 -1.905762
H -4.943083 -0.734686 -0.316183
H 3.556745 0.460261 0.802042
O 2.548710 1.321518 -0.771683
C 2.800066 2.631098 -0.227753
H 3.837235 2.706257 0.116700
H 2.632051 3.333306 -1.044068
H 2.119820 2.857196 0.600432

TS5_trans_exo

Electronic Energy: -767.119183783 Hartree

O 1.398326 -0.442959 -0.391191
C 2.686244 0.062576 0.319098
C 2.855958 1.556942 0.142029
C 1.557756 2.329729 0.398367
C 0.511357 1.949876 -0.655541
C 0.279615 0.454055 -0.743311
C -0.224099 -0.304388 0.406322
C -0.857307 -1.671307 0.246228
H 0.043917 0.049632 1.393419
H 3.220584 1.738491 -0.875573
H 1.749759 3.404863 0.342586
H 3.649725 1.870100 0.828544
H 1.186964 2.139967 1.414962
H 0.840901 2.303781 -1.638574
H -0.459162 2.417666 -0.453134
H -0.961168 -2.167475 1.214816
O -2.160489 -1.556195 -0.355830
H -0.270730 -2.294393 -0.429011
H 2.509176 -0.236781 1.360718
C -2.795198 -0.460197 0.070874
O -4.045056 -0.451254 -0.294689
O -2.199452 0.411890 0.715557
C -4.834690 0.714140 0.080261
H -5.828741 0.508166 -0.310509
H -4.851890 0.811991 1.166676
H -4.410831 1.609126 -0.378253
O 3.705216 -0.606064 -0.259545
C 3.871839 -1.987410 0.103008
H 4.840148 -2.286912 -0.297057
H 3.870697 -2.102034 1.193782
H 3.083651 -2.601285 -0.342946
H -0.110207 0.127645 -1.708858

TS5_trans_endo

Electronic Energy: -767.120970326 Hartree

O -1.454655 -0.463902 0.486912
C -2.635508 0.017389 -0.371015
C -2.721480 1.534719 -0.332802
C -1.409161 2.263650 -0.657466
C -0.344450 2.108810 0.449773

C -0.005494 0.707959 0.797179
C -0.135783 -0.418577 -0.116632
C 0.698027 -1.654914 0.180276
H -0.162931 -0.183388 -1.183026
H -3.089856 1.821426 0.658979
H -1.615013 3.332760 -0.761267
H -3.494875 1.821032 -1.053565
H -1.012177 1.938572 -1.627096
H -0.648782 2.639937 1.357585
H 0.607093 2.562448 0.130306
H 0.266719 -2.536283 -0.296736
O 2.011613 -1.530369 -0.384155
H 0.765681 -1.826935 1.259634
H -2.397684 -0.383914 -1.368348
C 2.737728 -0.458758 -0.020251
O 3.969053 -0.612079 -0.443874
O 2.302852 0.514238 0.594405
C 4.894008 0.473208 -0.170512
H 5.838653 0.144714 -0.599132
H 4.545632 1.389599 -0.650543
H 4.984203 0.621397 0.906869
O -3.746165 -0.518897 0.174187
C -3.968105 -1.923435 -0.047361
H -4.977172 -2.125535 0.310470
H -3.900937 -2.158814 -1.116421
H -3.248614 -2.521710 0.518883
H 0.219781 0.452462 1.826473

TS5_cis_exo

Electronic Energy: -767.121585241 Hartree

O 1.421256 -0.296728 -1.035370
C 2.772929 -0.087101 -0.326920
C 3.043266 1.390840 -0.113438
C 1.866558 2.101958 0.563786
C 0.650213 2.078431 -0.370118
C 0.315499 0.692294 -0.882827
C -0.024250 -0.408857 0.017040
C -0.800075 -1.618858 -0.458845
H 0.414201 -0.404797 1.005836
H 3.257304 1.853503 -1.084071
H 2.133340 3.140959 0.776223
H 3.953636 1.459885 0.489935
H 1.643514 1.633323 1.529035
H 0.841734 2.722665 -1.235525

H -0.250780 2.462483 0.122582
H -0.843718 -2.379158 0.325659
O -2.140305 -1.250860 -0.827260
H -0.351179 -2.040090 -1.359011
C -2.654338 -0.360728 0.034118
O -3.933738 -0.196914 -0.160493
O -1.944338 0.210277 0.865707
C -4.607051 0.774318 0.689320
H -5.643336 0.756363 0.358772
H -4.522850 0.469856 1.733642
H -4.166075 1.761886 0.543399
H 3.428150 -0.549637 -1.072277
O 2.772091 -0.759057 0.861830
C 3.039002 -2.171940 0.793452
H 3.999568 -2.354806 0.299826
H 3.085291 -2.520168 1.825218
H 2.243667 -2.702468 0.257724
H -0.232705 0.694319 -1.825699

TS5_cis_endo

Electronic Energy: -767.123442672 Hartree

O -1.491047 -0.265733 1.062646
C -2.780258 -0.077317 0.255690
C -2.955997 1.391806 -0.105891
C -1.749426 2.021373 -0.815232
C -0.545616 2.207148 0.133917
C -0.108958 0.972815 0.829386
C -0.252512 -0.370476 0.292947
C 0.676499 -1.446230 0.832192
H -0.440051 -0.468271 -0.775666
H -3.188748 1.949079 0.809219
H -2.030008 3.012493 -1.182622
H -3.840099 1.446200 -0.749168
H -1.472843 1.432677 -1.696339
H -0.753889 2.984558 0.876437
H 0.341616 2.542862 -0.426190
H 0.231944 -2.435089 0.710143
O 1.894479 -1.490610 0.077368
H 0.894180 -1.279038 1.892315
C 2.616553 -0.355303 0.023751
O 3.783403 -0.618299 -0.516407
O 2.226441 0.744763 0.405651
C 4.688554 0.503286 -0.685207
H 5.577932 0.074203 -1.142395

H 4.237190 1.252294 -1.338660
H 4.921921 0.941887 0.286490
H -3.507719 -0.418759 0.999487
O -2.749262 -0.857023 -0.860605
C -3.090561 -2.246465 -0.678832
H -4.091424 -2.335804 -0.243447
H -3.080593 -2.690273 -1.673983
H -2.361958 -2.754253 -0.038030
H 0.260760 1.036622 1.846864

TS6_exo

Electronic Energy: -652.599425127 Hartree

O 2.120065 0.930672 -0.092684
C 3.092903 0.335510 -1.034126
C 2.746874 -1.150755 -1.129200
C 2.092231 -1.474184 0.230748
C 1.437174 -0.173387 0.686977
C 0.431281 0.384747 -0.225893
C -0.390776 1.634474 0.011580
C 1.382607 0.089029 2.169783
H 2.840747 -1.772892 0.970431
H 1.352042 -2.276844 0.162093
H 3.640373 -1.755214 -1.303997
H 2.064228 -1.344334 -1.962335
H 0.366575 -0.047972 -1.217848
H 0.675702 -0.613200 2.624630
H 2.367018 -0.077594 2.615432
H 1.065678 1.105835 2.409733
H -0.397316 1.928041 1.063549
O -1.741911 1.424112 -0.424957
H 4.065916 0.521890 -0.572100
H 3.016738 0.892816 -1.968796
H -0.008656 2.458821 -0.592596
C -2.175618 0.189194 -0.135761
O -3.459086 0.080075 -0.332241
O -1.398211 -0.692175 0.241561
C -4.051103 -1.230879 -0.105671
H -5.104756 -1.100217 -0.342492
H -3.587915 -1.964207 -0.767819
H -3.916670 -1.518896 0.938061

TS6_endo

Electronic Energy: -652.595022419 Hartree

O 2.103605 1.080385 0.085335
C 3.193991 0.475063 -0.670672
C 3.277862 -0.995563 -0.270226
C 1.816216 -1.557590 -0.196912
C 0.978238 -0.495285 0.426718
C 0.769731 0.695896 -0.405994
C -0.244042 1.777203 -0.020569
C 0.574664 -0.588329 1.845377
H 1.800287 -2.477256 0.393718
H 1.444184 -1.768794 -1.205526
H 3.744654 -1.084189 0.715744
H 3.872152 -1.581869 -0.977408
H 0.794773 0.495193 -1.476263
H -0.219458 -1.350820 1.884481
H 1.394273 -0.971668 2.463269
H 0.178797 0.338535 2.259678
H 0.143973 2.354212 0.820608
O -1.526663 1.302073 0.409813
H 4.081336 1.039798 -0.378255
H 3.004995 0.616888 -1.740131
H -0.387738 2.454277 -0.867839
C -2.036749 0.189849 -0.158935
O -3.325024 0.139806 0.096474
O -1.378017 -0.643006 -0.767409
C -4.031413 -1.032903 -0.384197
H -5.062600 -0.883393 -0.070301
H -3.958705 -1.090927 -1.471544
H -3.613120 -1.934097 0.068518

TS7_trans_exo

Electronic Energy: -767.130911209 Hartree

O -1.701204 0.146569 0.759548
C -2.718053 0.043042 -0.380391
C -2.183831 0.967029 -1.473642
C -1.237547 1.959415 -0.778651
C -0.710648 1.231298 0.456948
C 0.008094 -0.023039 0.204055
C 0.662254 -0.894305 1.256140
C -0.379470 2.089506 1.652522
H -1.770773 2.858601 -0.456308

H -0.412686 2.277889 -1.422907
H -3.007199 1.471794 -1.984821
H -1.672211 0.351723 -2.220041
H -0.097850 -0.466249 -0.779653
H 0.488481 2.712097 1.410432
H -1.221473 2.748171 1.882210
H -0.150437 1.505325 2.546028
H 0.827795 -0.357785 2.192617
O 1.923205 -1.385713 0.772256
H 0.053230 -1.778969 1.445177
C 2.563837 -0.474503 0.030641
O 3.786332 -0.843970 -0.228306
O 1.999725 0.557451 -0.347291
C 4.574867 0.038344 -1.076692
H 5.534700 -0.463697 -1.176919
H 4.087163 0.150233 -2.046417
H 4.688997 1.008952 -0.591553
H -3.630158 0.395123 0.116320
O -2.802692 -1.239333 -0.812220
C -3.583901 -2.134527 0.002547
H -4.594439 -1.736266 0.147091
H -3.634426 -3.073712 -0.547806
H -3.105270 -2.296826 0.973801

TS7_trans_endo

Electronic Energy: -767.131811311 Hartree

O -1.705878 0.008189 0.999603
C -2.787546 -0.073757 -0.034838
C -2.727720 1.239613 -0.809008
C -1.230079 1.577866 -1.119193
C -0.474789 1.278992 0.130001
C -0.356328 -0.152323 0.443635
C 0.617600 -0.682620 1.485062
C -0.102741 2.372162 1.049389
H -1.140181 2.629989 -1.401025
H -0.872175 0.946808 -1.938611
H -3.152432 2.034349 -0.188835
H -3.306556 1.176048 -1.733795
H -0.375189 -0.804975 -0.430172
H 0.828478 2.797020 0.644326
H -0.853178 3.169028 1.043565
H 0.096314 2.045540 2.070366
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C 2.478990 -0.353728 0.072627
O 3.622066 -0.909519 -0.264027
O 2.064284 0.741558 -0.282141
C 4.468310 -0.145388 -1.159247
H 5.337600 -0.778136 -1.327668
H 3.942096 0.051226 -2.095374
H 4.757644 0.795027 -0.686532
H -3.680648 -0.161343 0.594564
O -2.616099 -1.138321 -0.874065
C -2.997861 -2.422440 -0.341473
H -2.869024 -3.135479 -1.155301
H -2.362272 -2.704430 0.504680
H -4.047140 -2.405067 -0.026675

TS7_cis_exo

Electronic Energy: -767.129973828 Hartree

O -1.535977 -0.609226 0.020554
C -2.564893 0.066941 -0.889130
C -1.987583 1.458192 -1.123424
C -1.234812 1.793110 0.179190
C -0.721926 0.451836 0.704387
C 0.207262 -0.256069 -0.187163
C 0.889993 -1.574389 0.111950
C -0.650554 0.291839 2.202007
H -1.913203 2.230577 0.916041
H -0.406845 2.490893 0.024174
H -2.793193 2.164436 -1.332721
H -1.322241 1.450182 -1.992194
H 0.286333 0.103433 -1.206662
H 0.115604 0.968210 2.595577
H -1.612480 0.563861 2.644727
H -0.407091 -0.726984 2.510668
H 0.878001 -1.808942 1.178452
O 2.251918 -1.539082 -0.343860
H -2.609614 -0.598062 -1.758980
H 0.412846 -2.383049 -0.443364
C 2.814594 -0.339876 -0.146468
O 4.099662 -0.377077 -0.360217
O 2.138089 0.641990 0.173921
C 4.824739 0.879143 -0.233399
H 5.856596 0.624546 -0.465418
H 4.430367 1.606141 -0.945282
H 4.733543 1.254732 0.786929

O -3.730336 0.141423 -0.198805
C -4.477292 -1.083315 -0.073395
H -3.956630 -1.790594 0.578860
H -5.433642 -0.805808 0.369029
H -4.642366 -1.532268 -1.059912

TS7_cis_endo

Electronic Energy: -767.127253140 Hartree

O -1.593857 -0.449339 0.467221
C -2.555445 0.009364 -0.609675
C -2.361807 1.519348 -0.743131
C -0.850371 1.889349 -0.633855
C -0.267627 1.009829 0.423071
C -0.206097 -0.413358 0.050251
C 0.660599 -1.405426 0.810575
C -0.038968 1.534949 1.784411
H -0.746559 2.946873 -0.378825
H -0.339019 1.701653 -1.584881
H -2.920248 1.984364 0.075094
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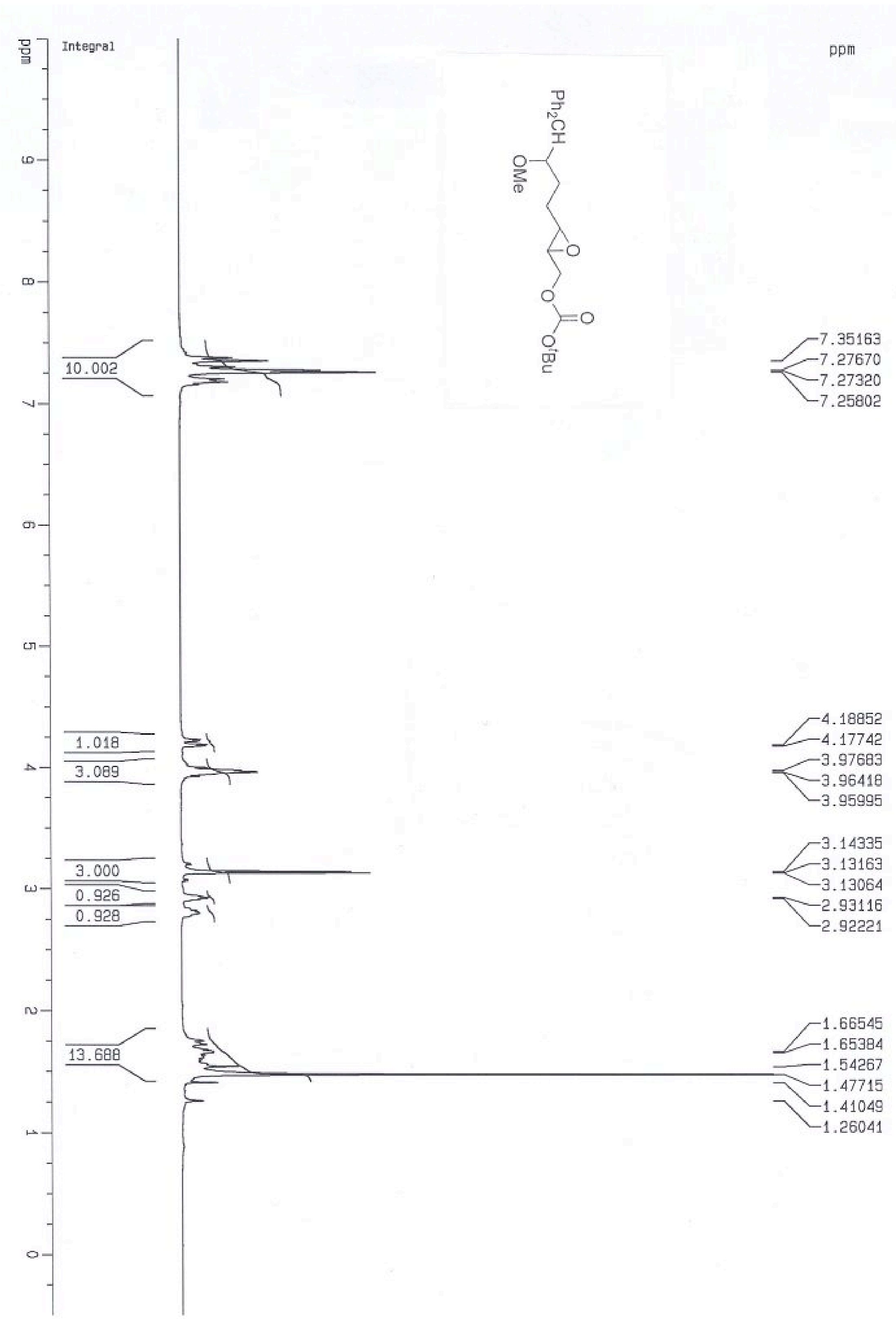
H -0.102832 -0.582407 -1.026865
H 0.883588 2.131147 1.730872
H -0.844750 2.216522 2.077350
H 0.096107 0.764162 2.543088
H 0.793166 -1.127534 1.859407
O 1.939326 -1.568157 0.184694
H -2.266237 -0.561295 -1.506179
H 0.194753 -2.391441 0.765518
C 2.649131 -0.448299 -0.054722
O 3.860161 -0.787773 -0.433744
O 2.211193 0.692427 0.047211
C 4.757685 0.297535 -0.780342
H 5.685864 -0.191889 -1.068751
H 4.345632 0.872427 -1.611918
H 4.911353 0.944595 0.085112
O -3.813406 -0.250969 -0.191477
C -4.233061 -1.626859 -0.256223
H -5.295060 -1.626066 -0.012998
H -4.085047 -2.025925 -1.266929
H -3.685131 -2.230675 0.473155

Full Reference for Gaussian 03, Revision C.02

Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Montgomery, Jr., J. A.; Vreven, T.; Kudin, K. N.; Burant, J. C.; Millam, J. M.; Iyengar, S. S.; Tomasi, J.; Barone, V.; Mennucci, B.; Cossi, M.; Scalmani, G.; Rega, N.; Petersson, G. A.; Nakatsuji, H.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Klene, M.; Li, X.; Knox, J. E.; Hratchian, H. P.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Ayala, P. Y.; Morokuma, K.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Zakrzewski, V. G.; Dapprich, S.; Daniels, A. D.; Strain, M. C.; Farkas, O.; Malick, D. K.; Rabuck, A. D.; Raghavachari, K.; Foresman, J. B.; Ortiz, J. V.; Cui, Q.; Baboul, A. G.; Clifford, S.; Cioslowski, J.; Stefanov, B. B.; Liu, G.; Liashenko, A.; Piskorz, P.; Komaromi, I.; Martin, R. L.; Fox, D. J.; Keith, T.; Al-Laham, M. A.; Peng, C. Y.; Nanayakkara, A.; Challacombe, M.; Gill, P. M. W.; Johnson, B.; Chen, W.; Wong, M. W.; Gonzalez, C.; and Pople, J. A.; Gaussian, Inc., Wallingford CT, 2004.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O
O^tBu
7.35163
7.27670
7.27320
7.25802
4.18852
4.17742
3.97683
3.96418
3.95995
3.14335
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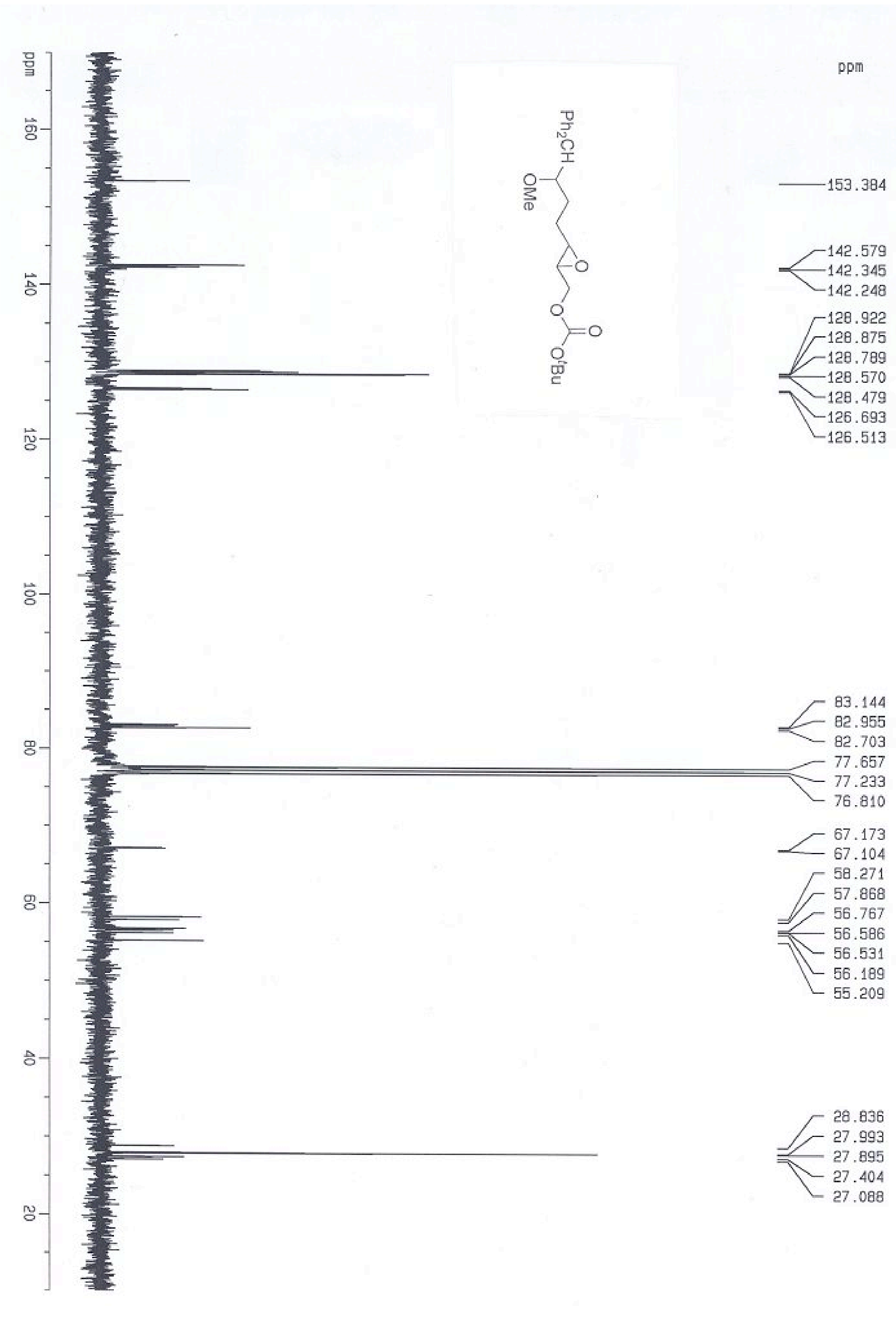
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58.271
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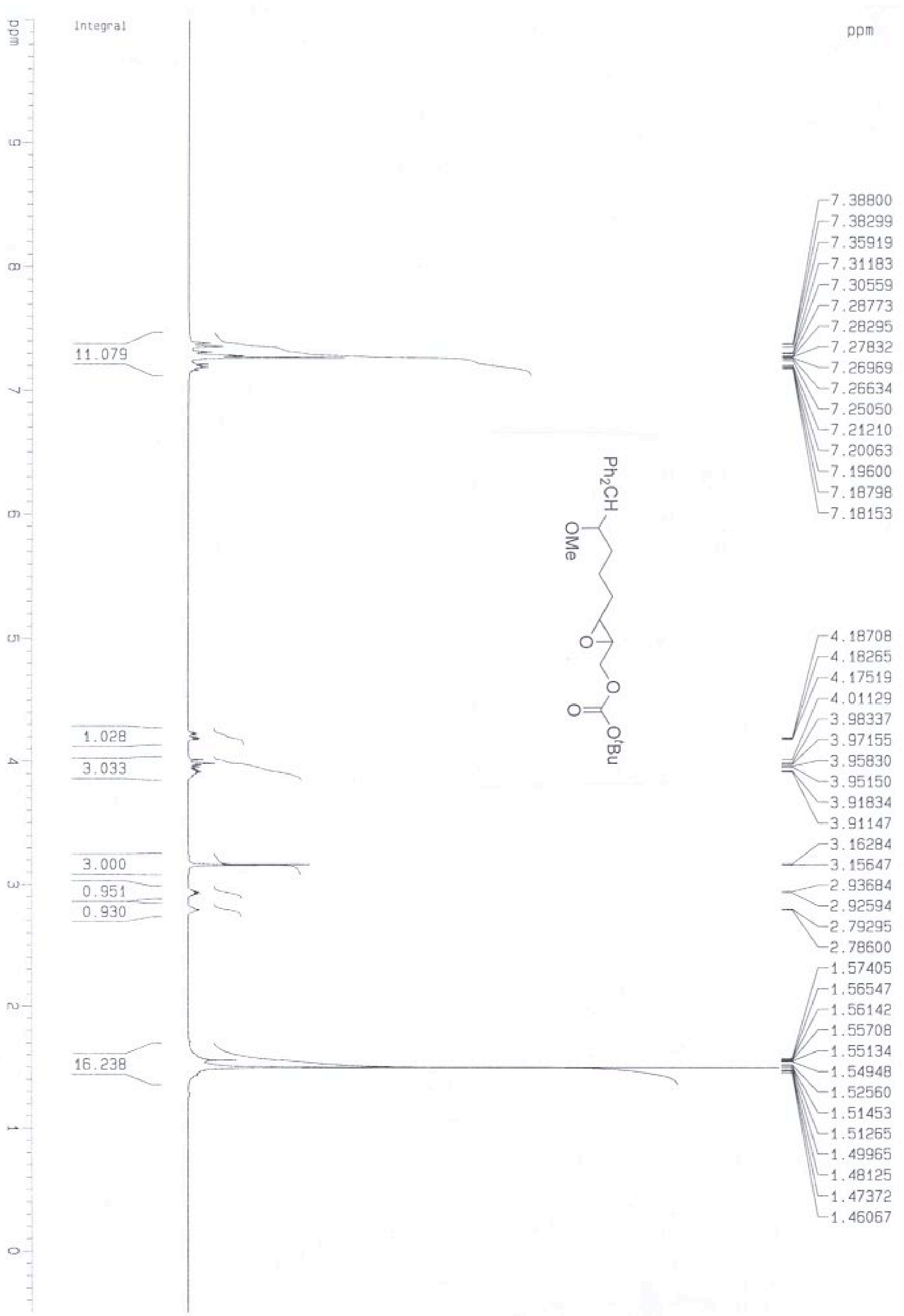
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7.26969
7.26634
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7.19600
7.18798
7.18153
4.18708
4.18265
4.17519
4.01129
3.98337
3.97155
3.95830
3.95150
3.91834
3.91147
3.16284
3.15647
2.93684
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2.79295
2.78600
1.57405
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1.56142
1.55708
1.55134
1.54948
1.52560
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Ph2CH
OMe
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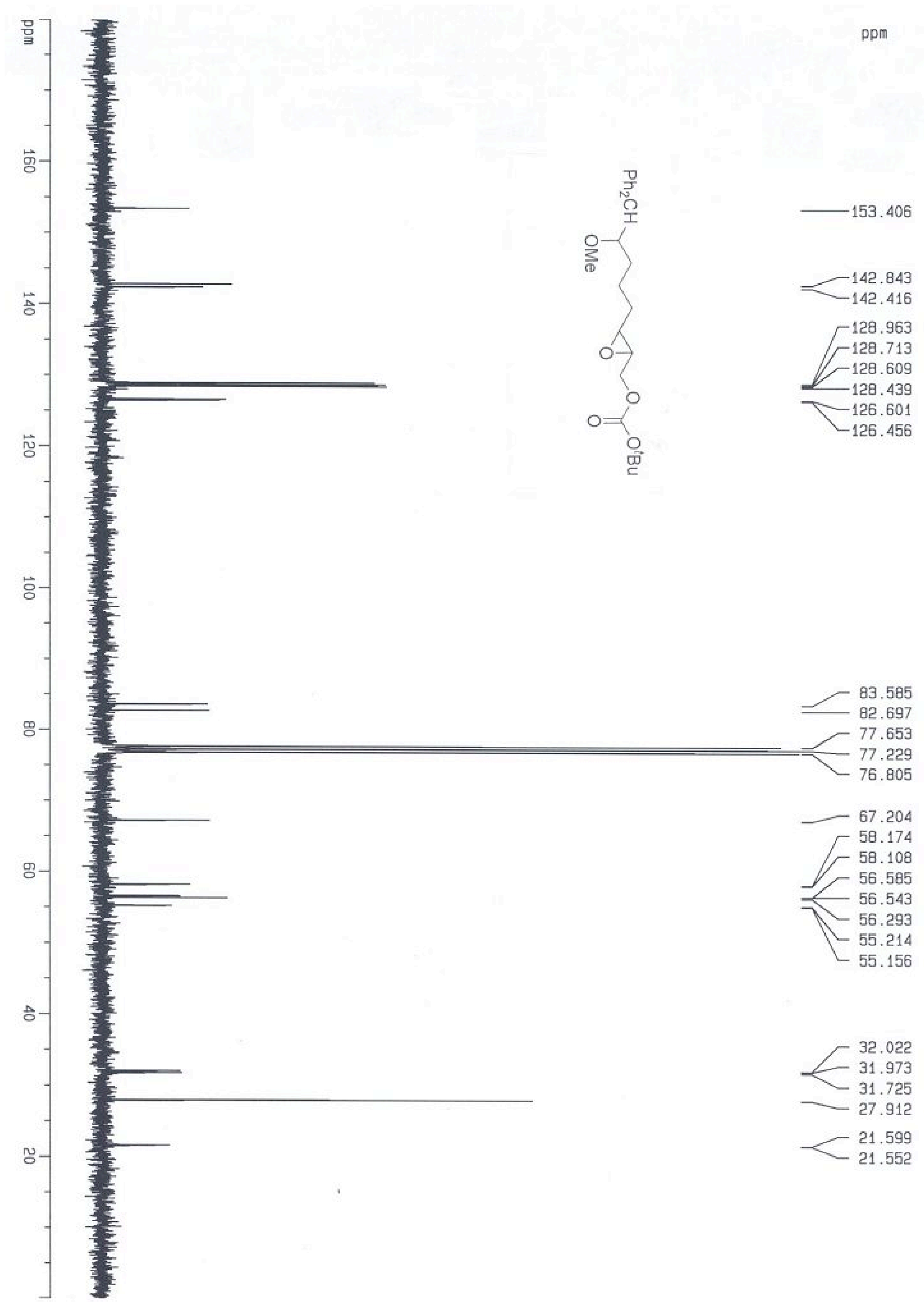
O
O'Bu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76.805
67.204
58.174
58.108
56.585
56.543
56.293
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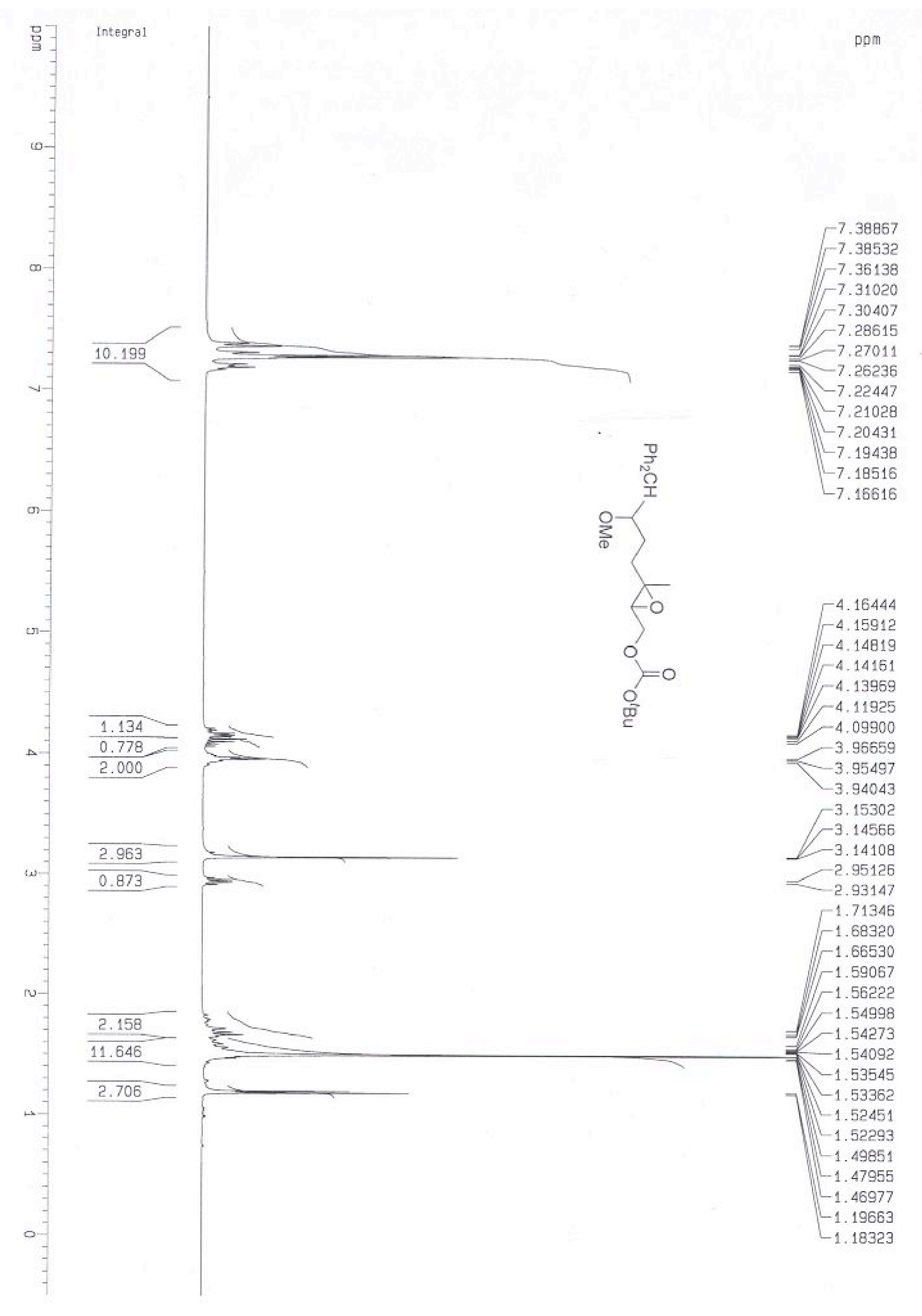
Ph2CH
OMe
O
O
O
OtBu



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OMe
O
O
O
OtBu
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7.38532
7.36138
7.31020
7.30407
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7.18516
7.16616
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4.14161
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1.54998
1.54273
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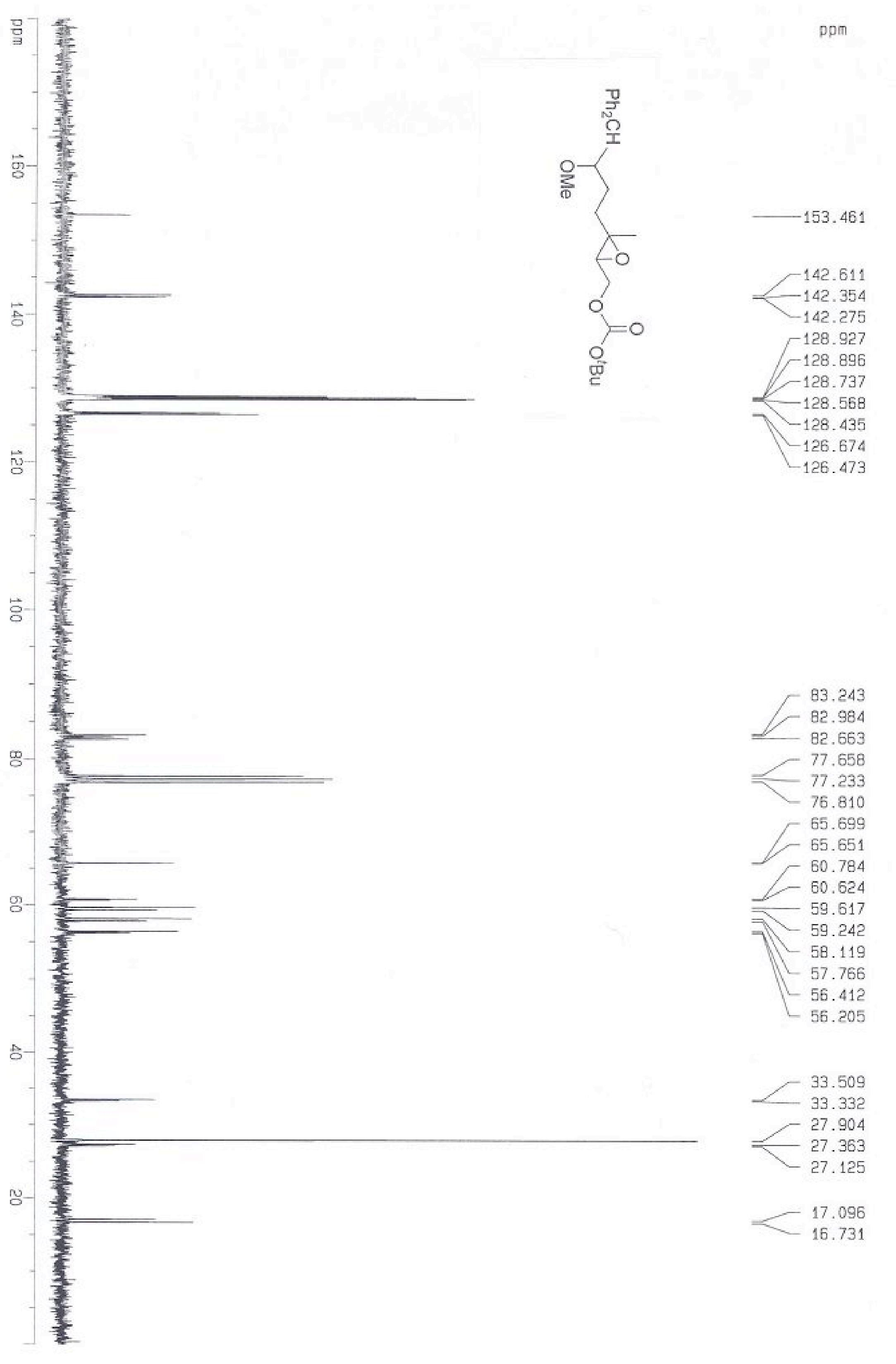
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77.658
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59.617
59.242
58.119
57.766
56.412
56.205
33.509
33.332
27.904
27.363
27.125
17.096
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O
O
O
O
O'Bu
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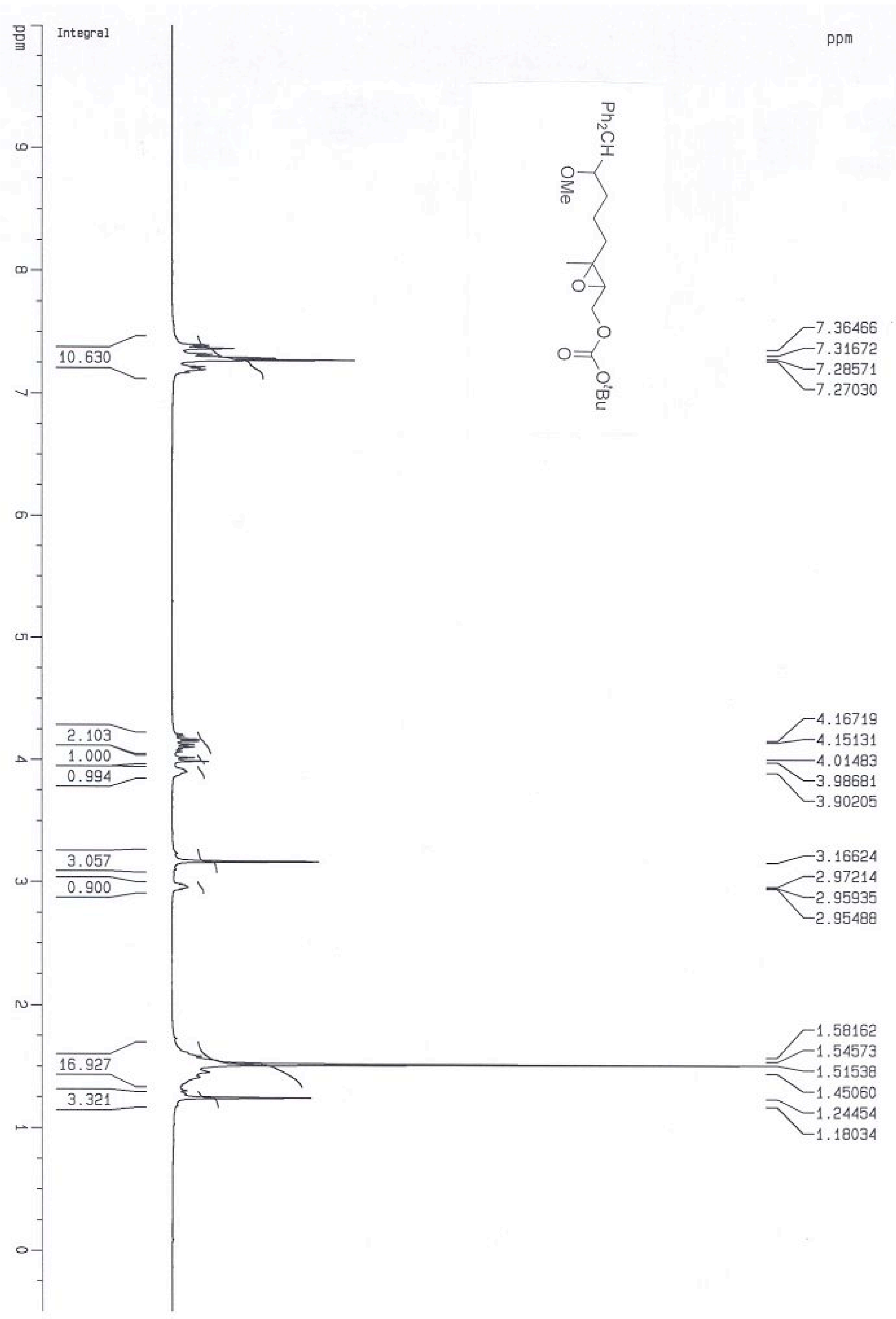

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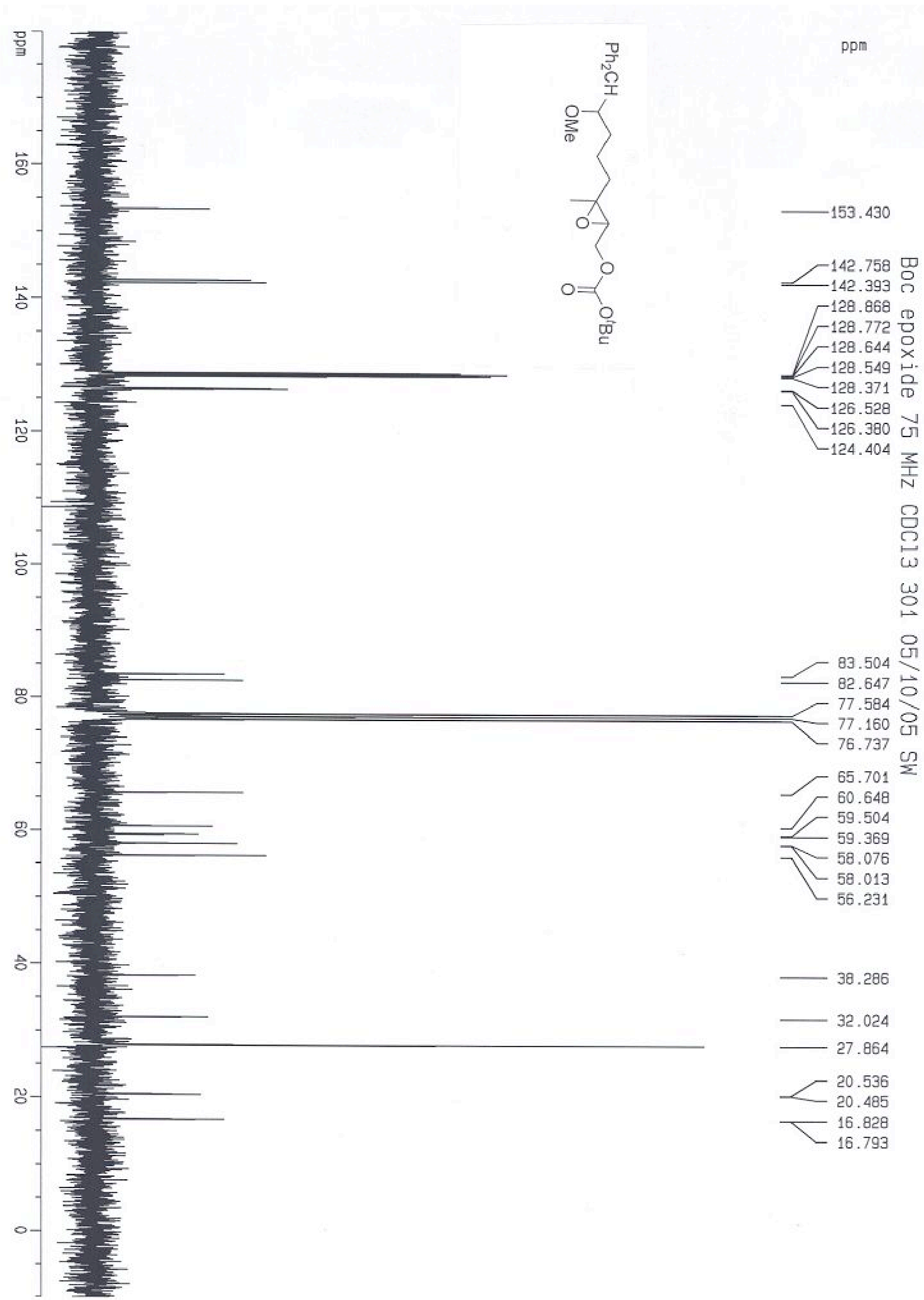
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59.369
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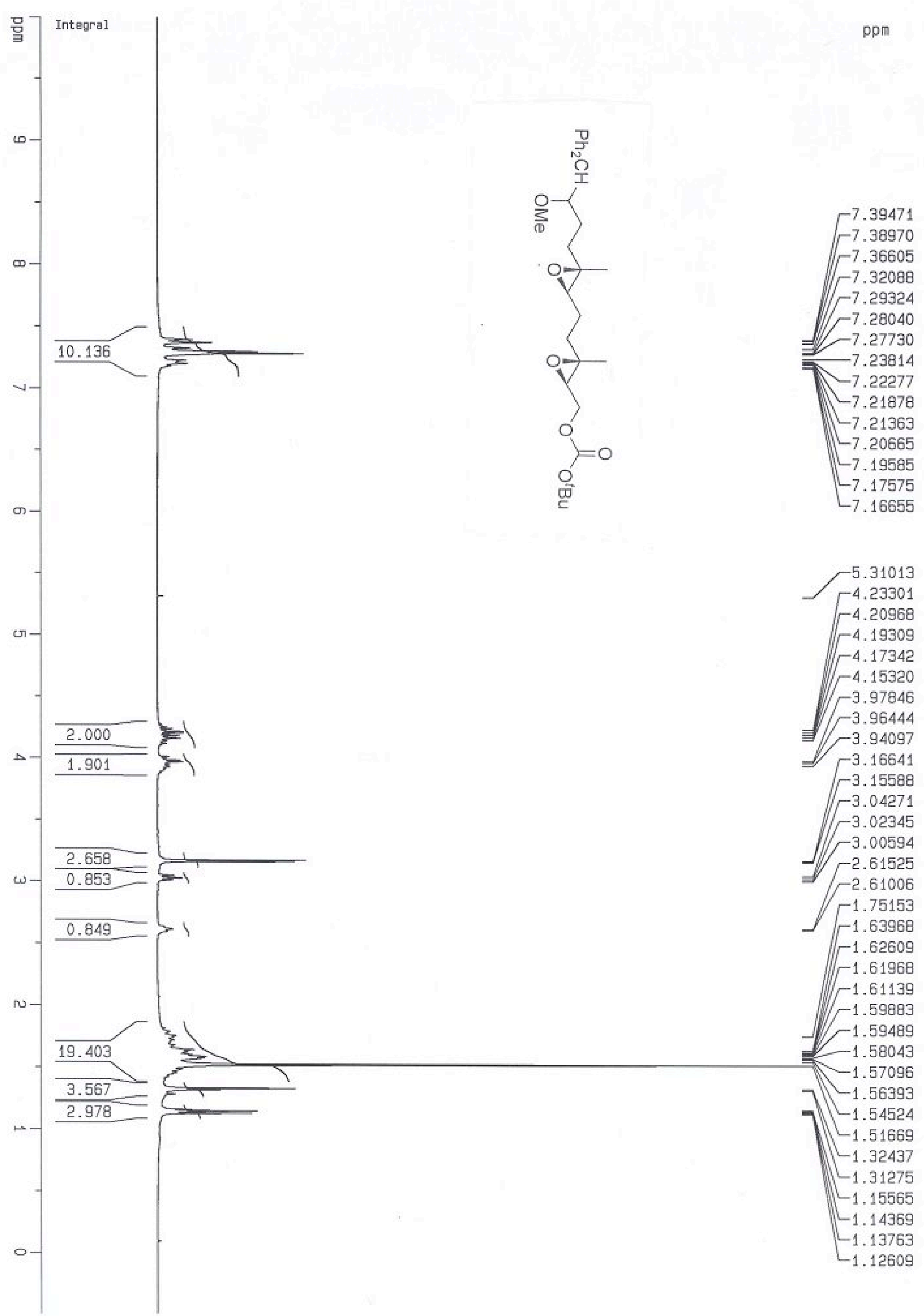
O
OtBu



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O
O^tBu
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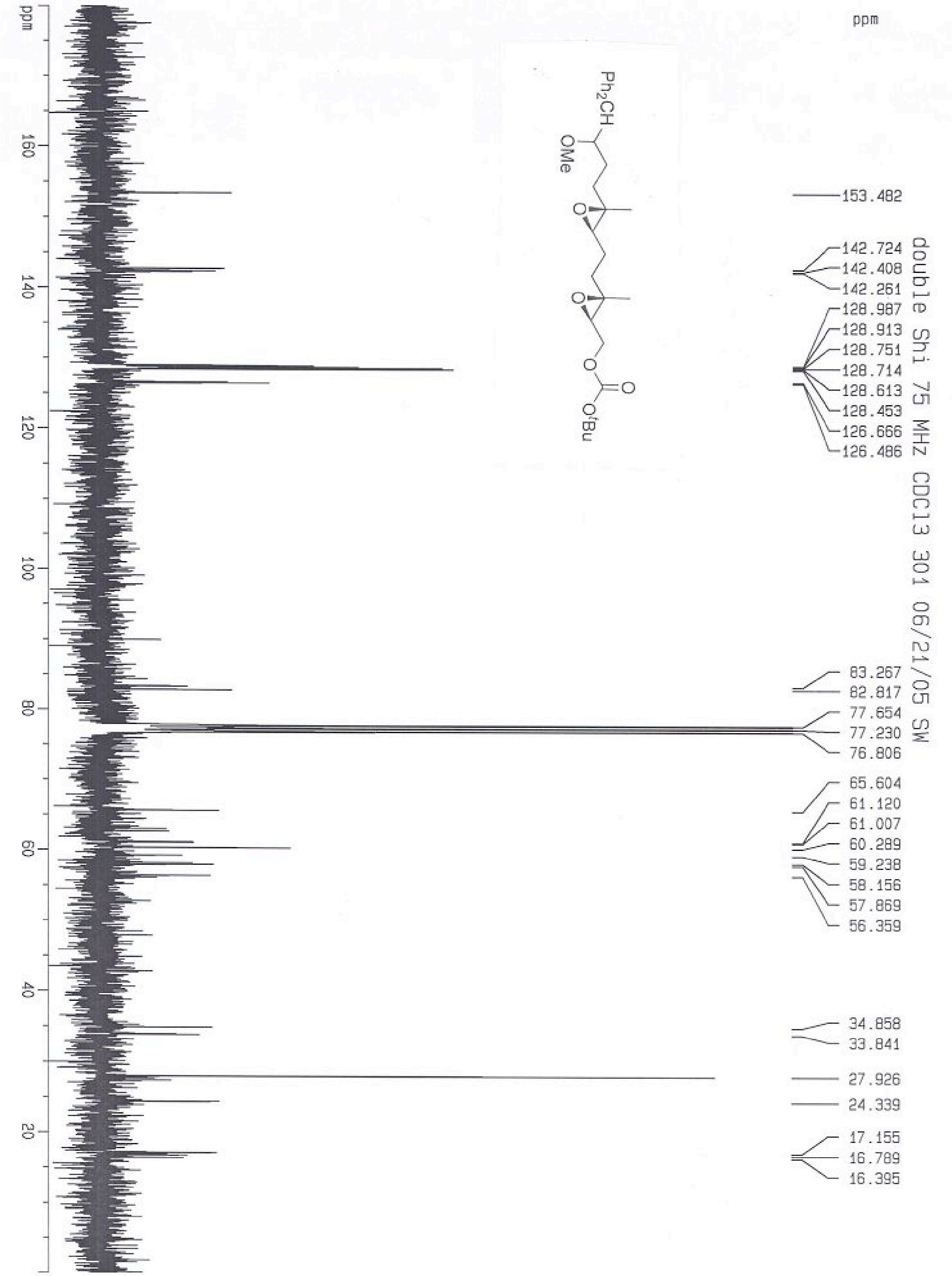
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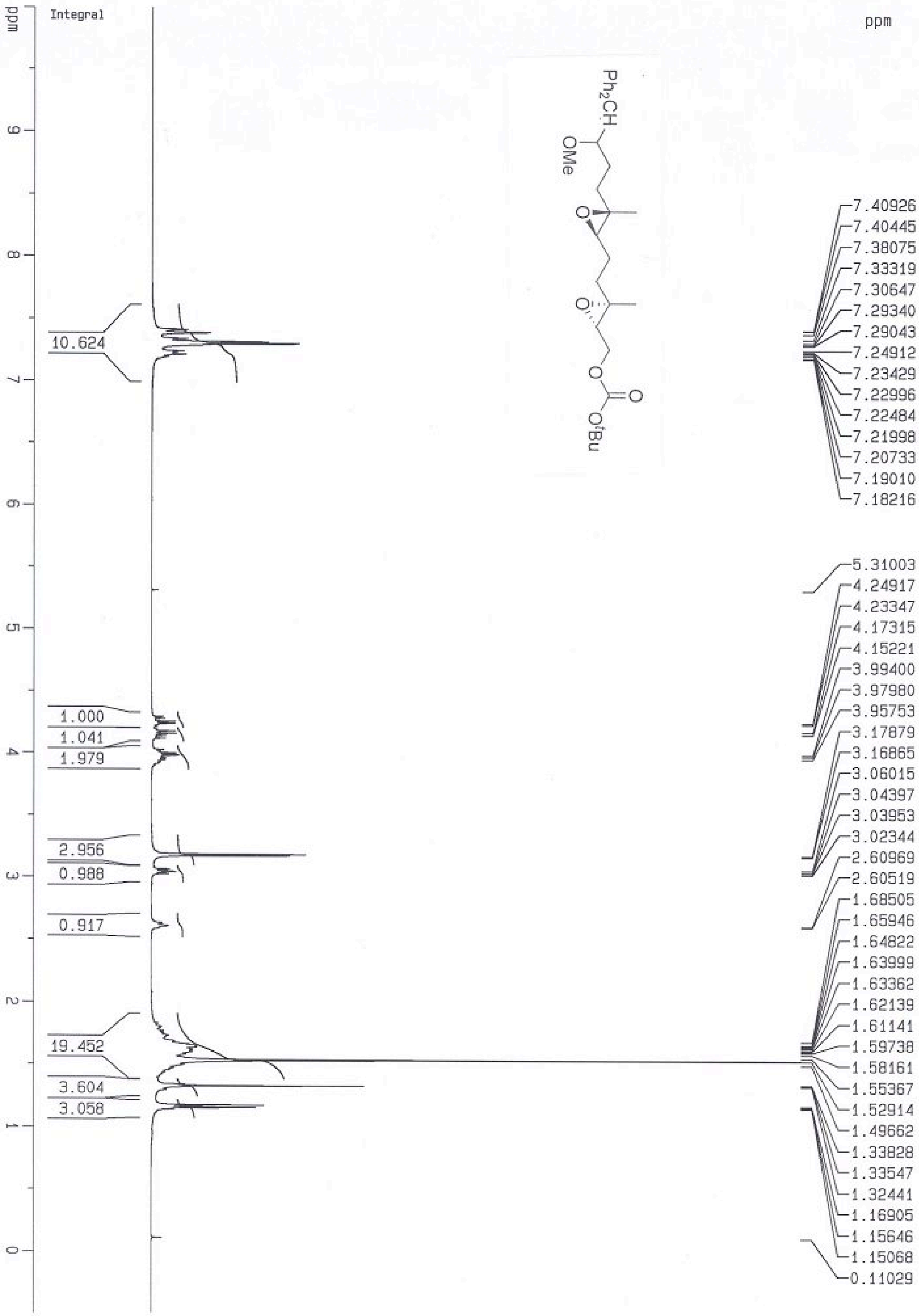
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3.99400
3.97980
3.95753
3.17879
3.16865
3.06015
3.04397
3.03953
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2.60519
1.68505
1.65946
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1.63999
1.63362
1.62139
1.61141
1.59738
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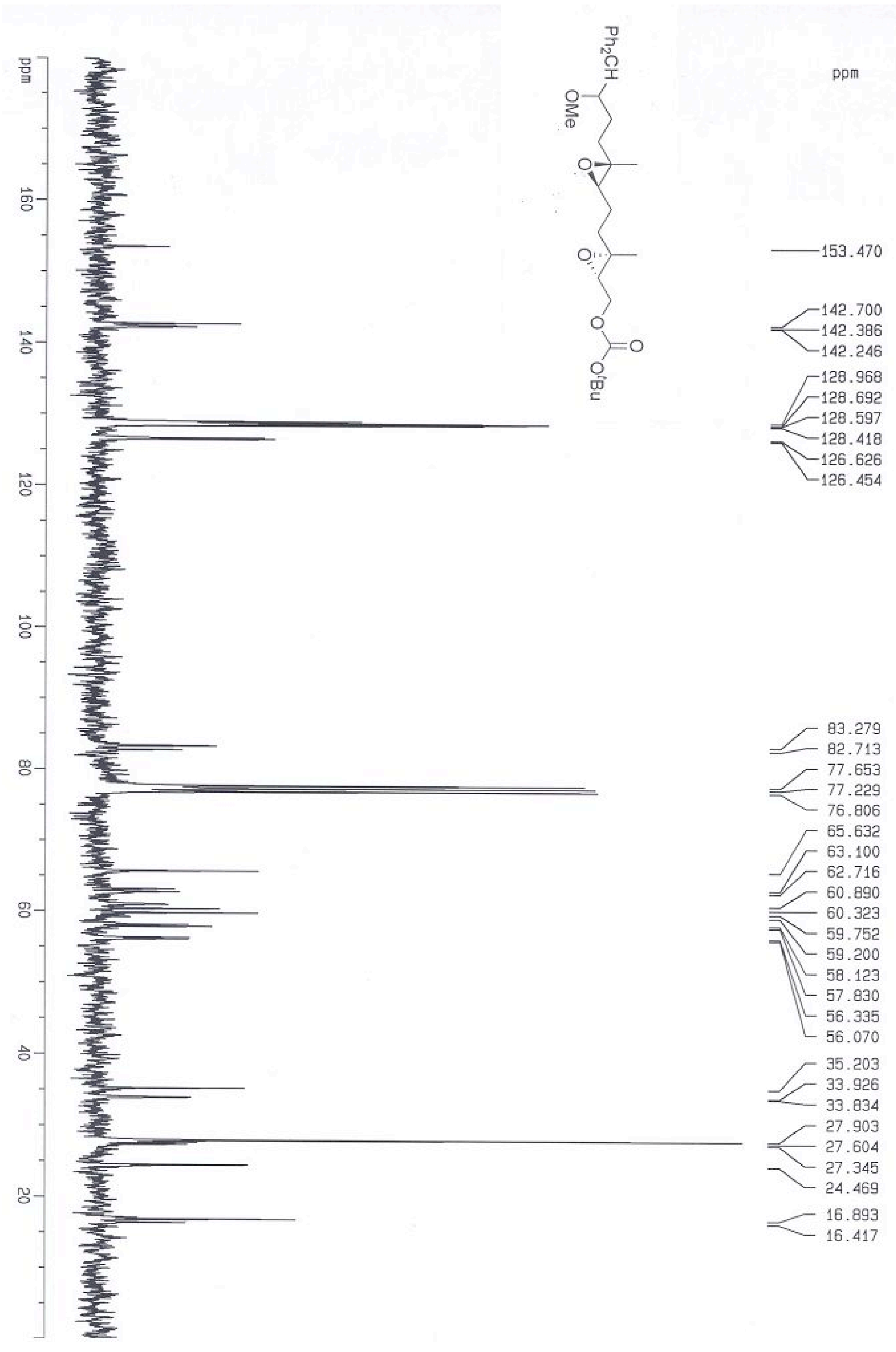
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80
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OMe
O
O
O
O
O'Bu
ppm
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142.386
142.246
128.968
128.692
128.597
128.418
126.626
126.454
83.279
82.713
77.653
77.229
76.806
65.632
63.100
62.716
60.890
60.323
59.752
59.200
58.123
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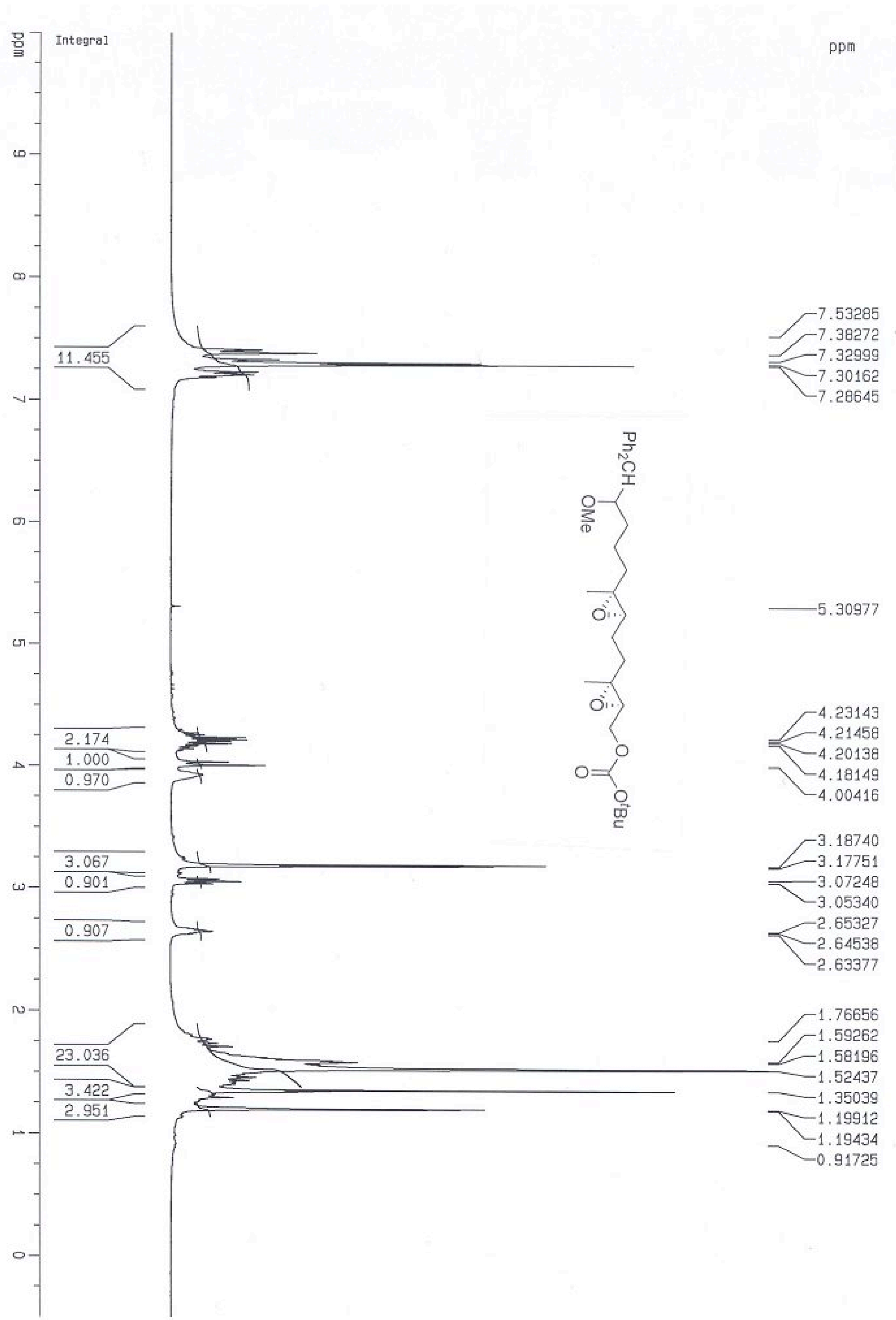
16.417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7.38272
7.32999
7.30162
7.28645
5.30977
4.23143
4.21458
4.20138
4.18149
4.00416
3.18740
3.17751
3.07248
3.05340
2.65327
2.64538
2.63377
1.76656
1.59262
1.58196
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OMe
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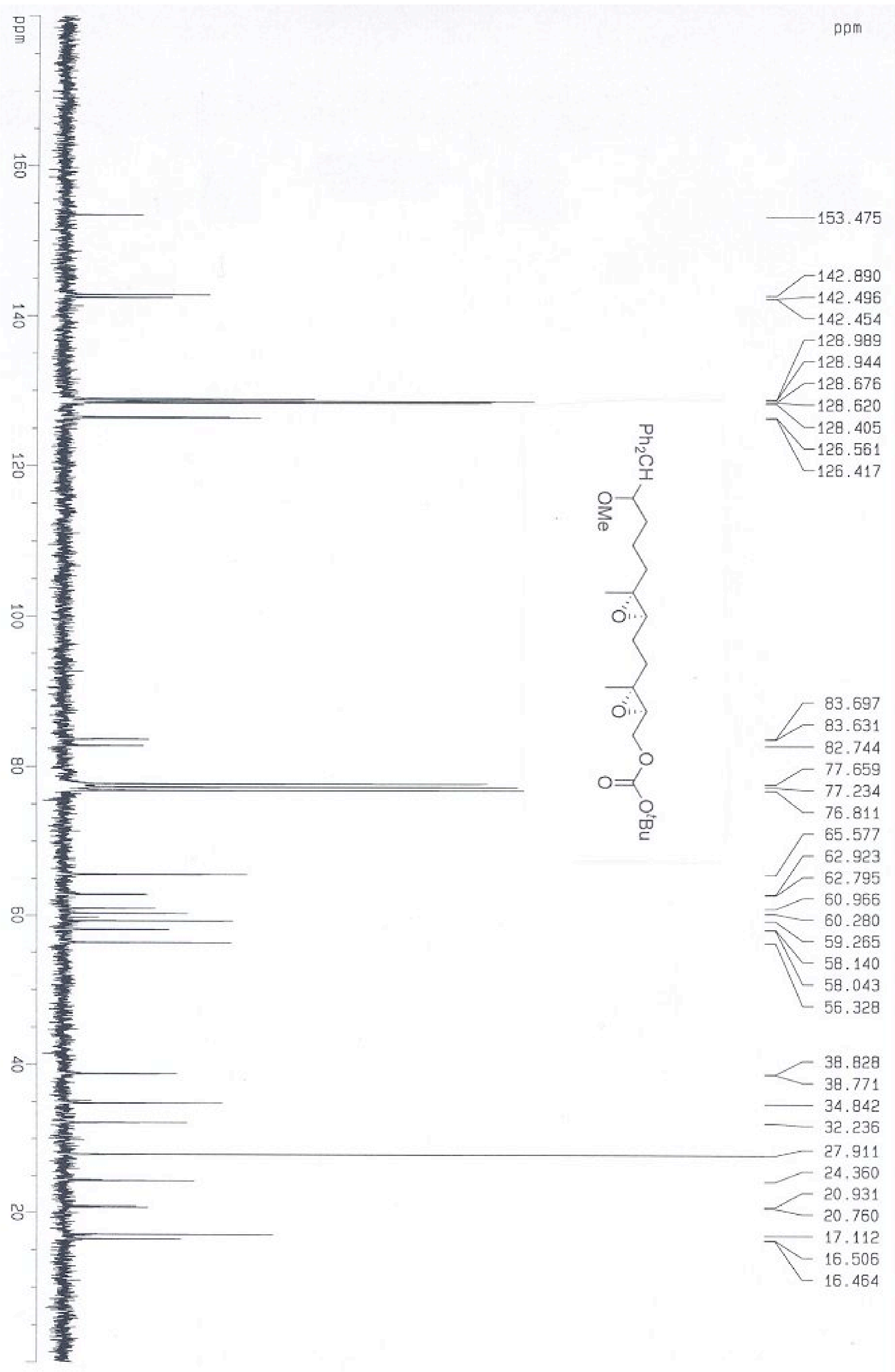
O
O
O
O^tBu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83.631
82.744
77.659
77.234
76.811
65.577
62.923
62.795
60.966
60.280
59.265
58.140
58.043
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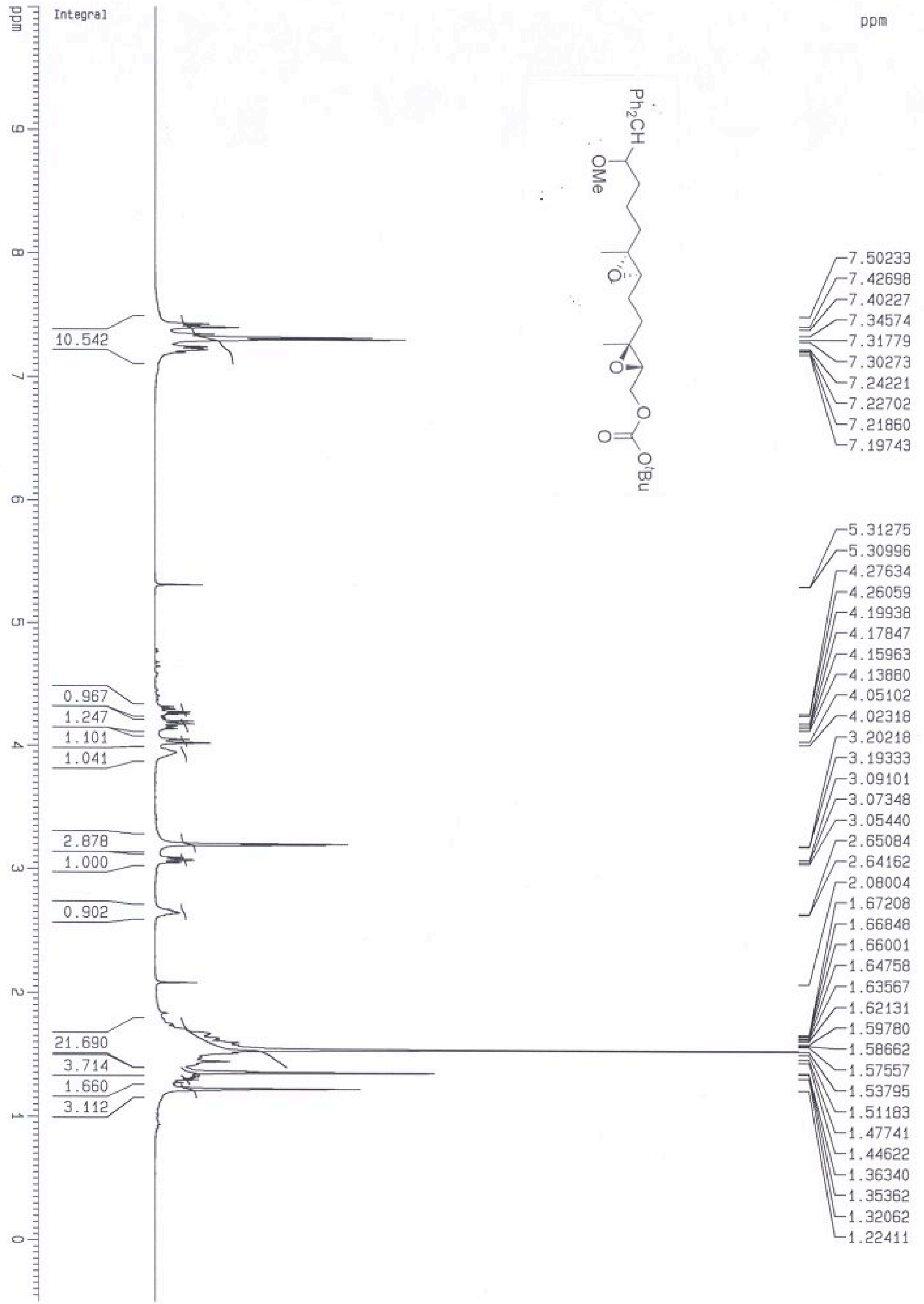
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Integral
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7.40227
7.34574
7.31779
7.30273
7.24221
7.22702
7.21860
7.19743
5.31275
5.30996
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4.26059
4.19938
4.17847
4.15963
4.13880
4.05102
4.02318
3.20218
3.19333
3.09101
3.07348
3.05440
2.65084
2.64162
2.08004
1.67208
1.66848
1.66001
1.64758
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1.62131
1.59780
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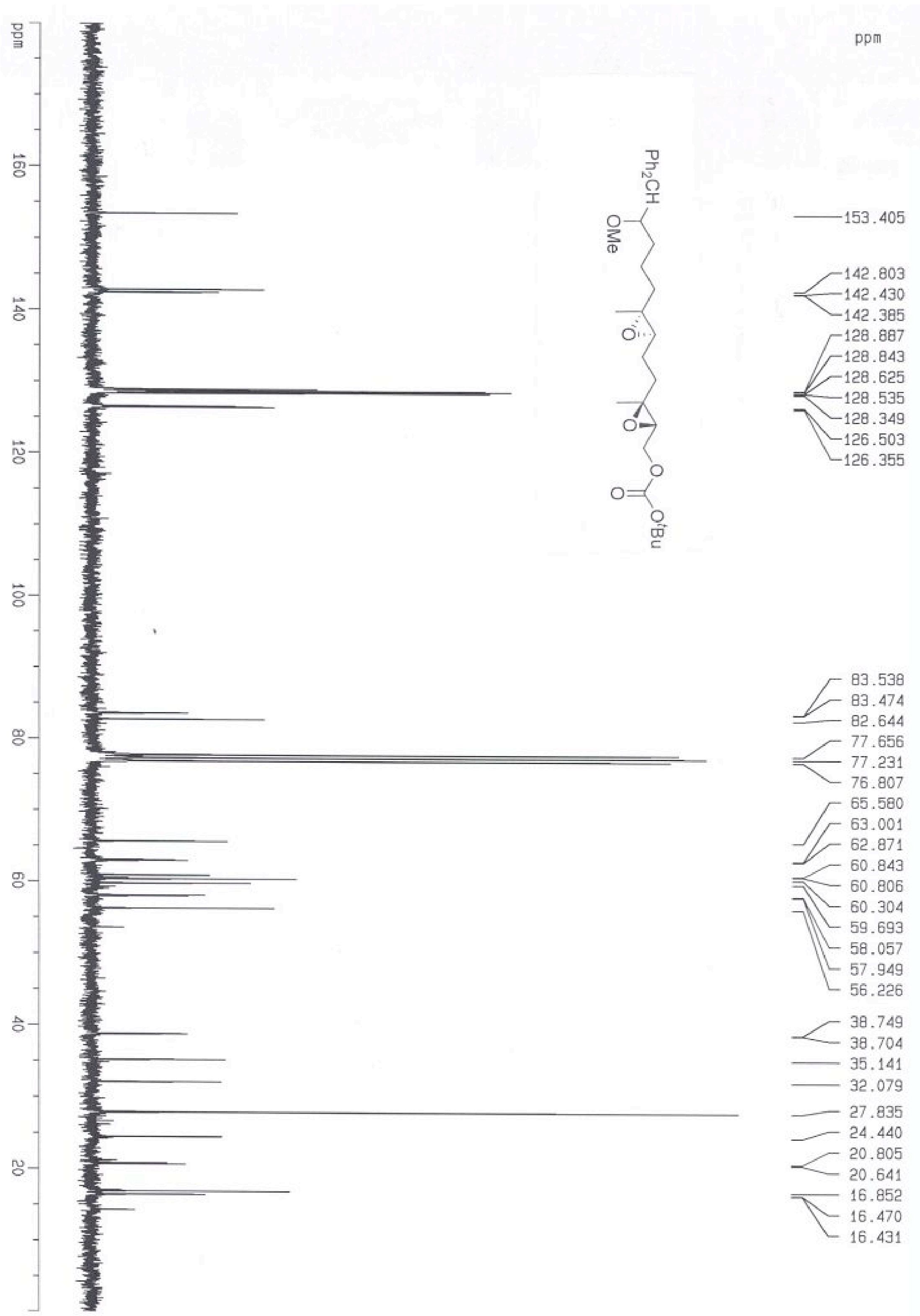
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77.656
77.231
76.807
65.580
63.001
62.871
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60.806
60.304
59.693
58.057
57.949
56.226
38.749
38.704
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32.079
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20.805
20.641
16.852
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OMe
O
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O
O
OtBu
ppm
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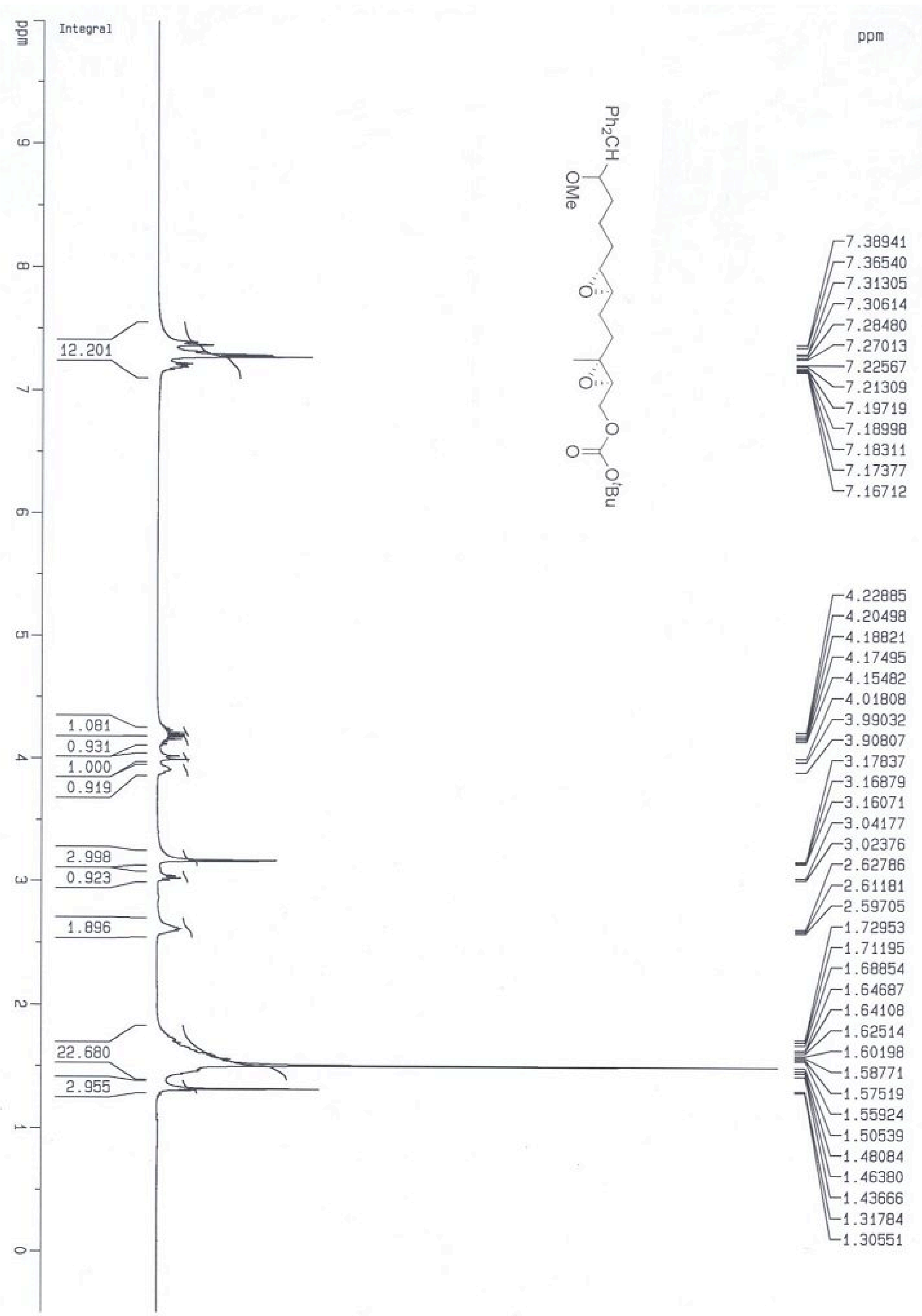
40
20



ppm
Integral
ppm
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OMe
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O
O
O^tBu
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7.28480
7.27013
7.22567
7.21309
7.19719
7.18998
7.18311
7.17377
7.16712
4.22885
4.20498
4.18821
4.17495
4.15482
4.01808
3.99032
3.90807
3.17837
3.16879
3.16071
3.04177
3.02376
2.62786
2.61181
2.59705
1.72953
1.71195
1.68854
1.64687
1.64108
1.62514
1.60198
1.58771
1.57519
1.55924
1.50539
1.48084
1.46380
1.43666
1.31784
1.30551
12.201
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1.000
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3
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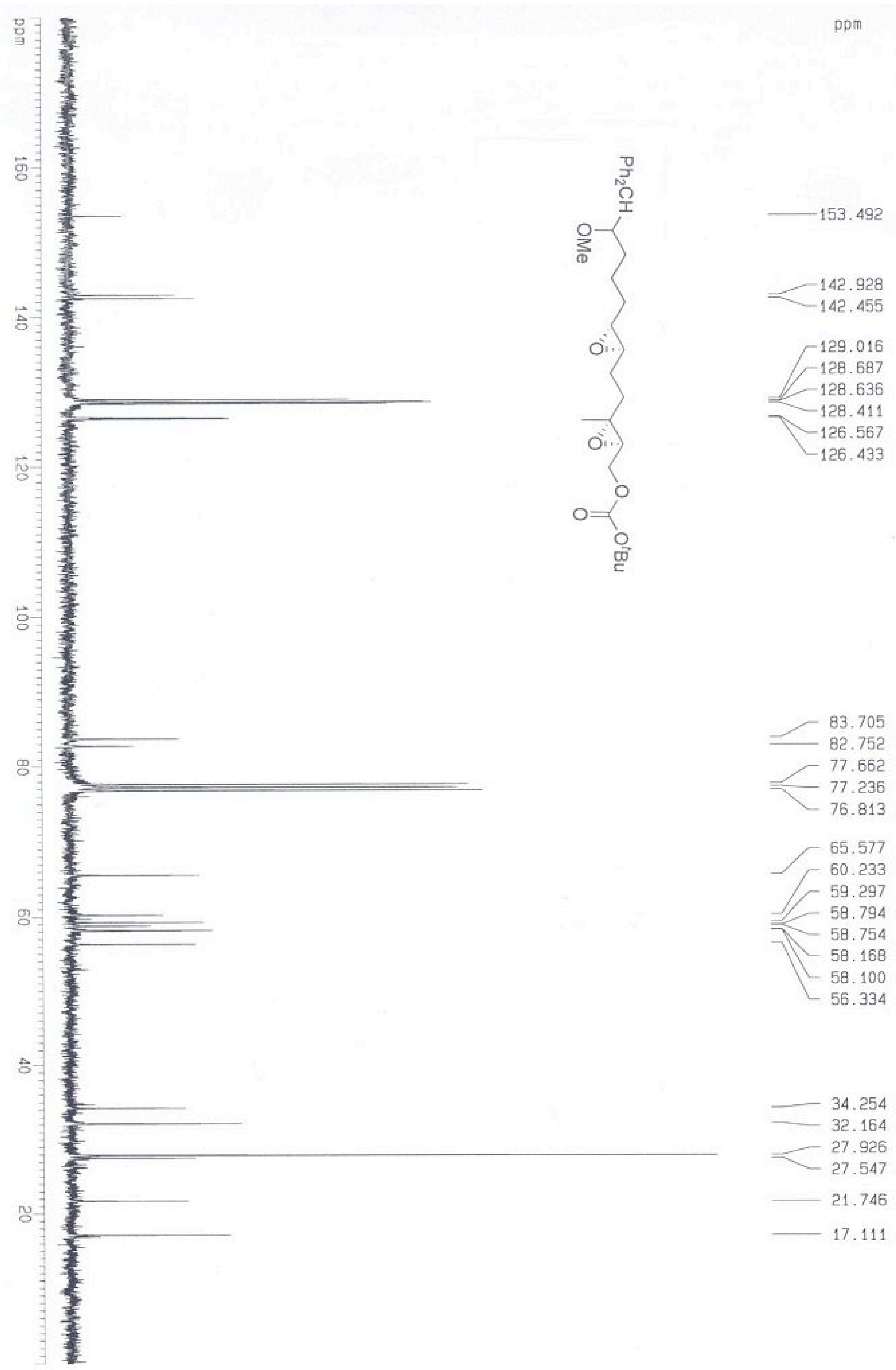
1
0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77.236
76.813
65.577
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58.794
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58.100
56.334
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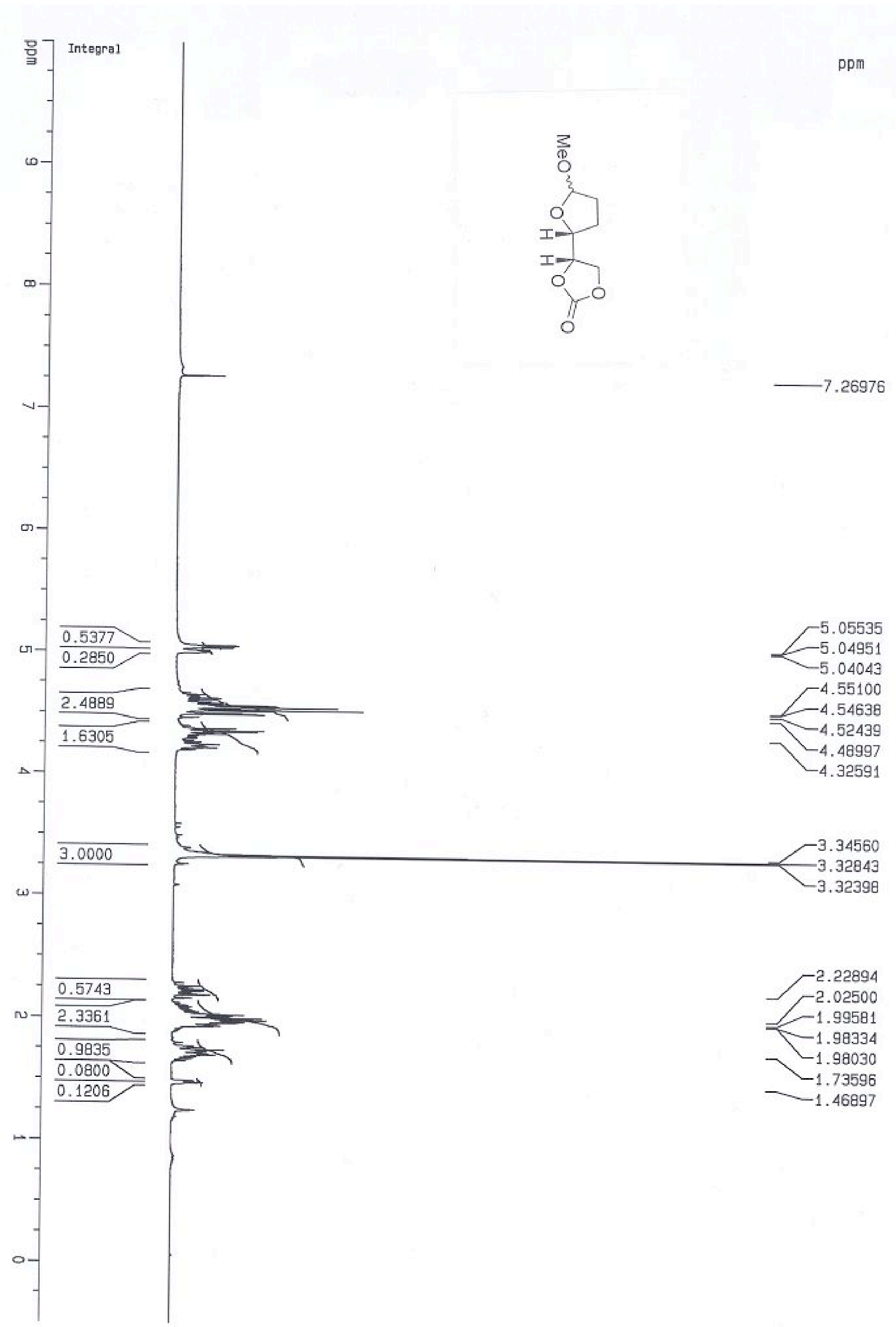
Ph_2CH
OMe
O
O
O
O
O^{t}Bu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2.3361
0.9835
0.0800
0.1206
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5.04951
5.04043
4.55100
4.54638
4.52439
4.48997
4.32591
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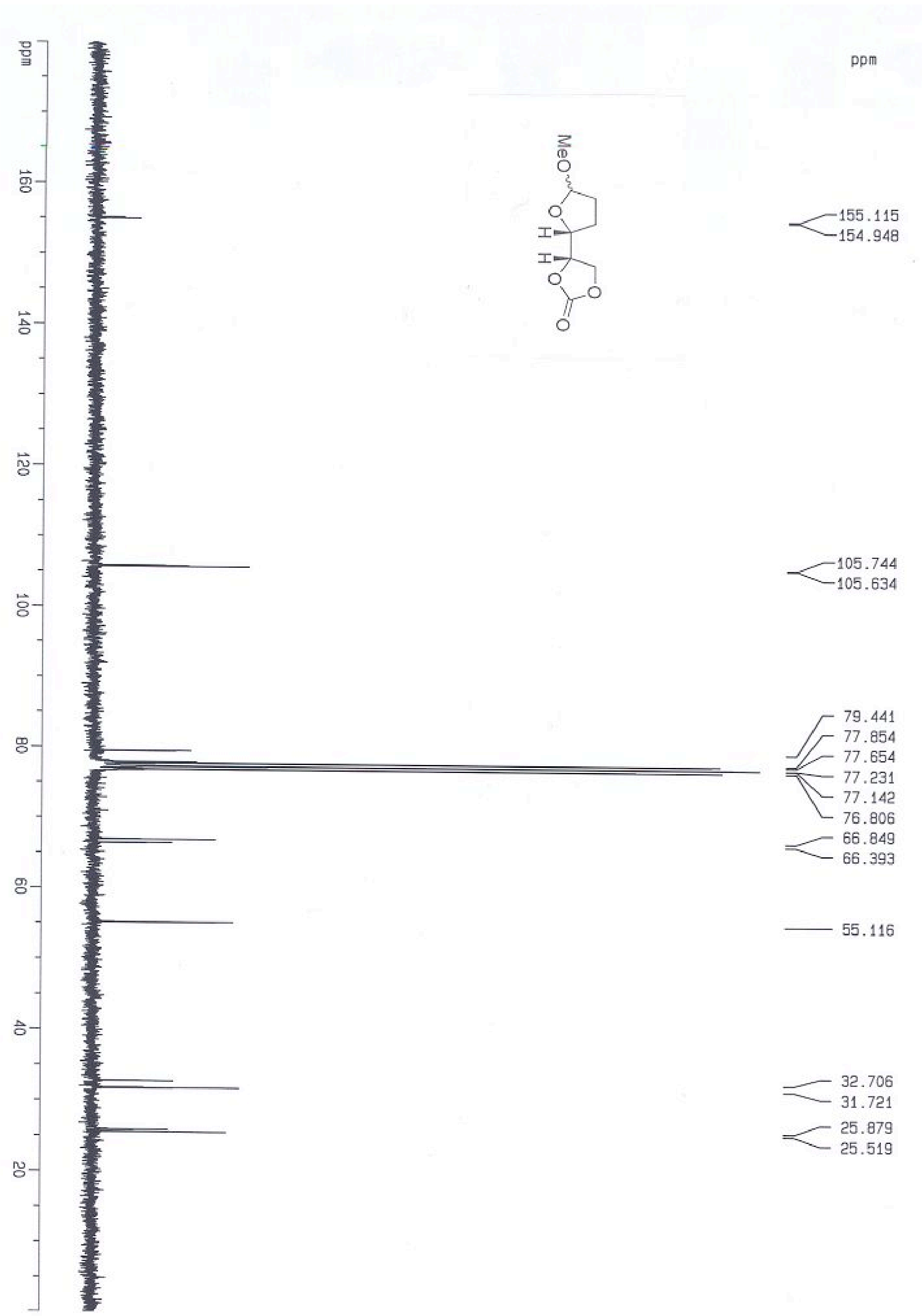
1.46897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O
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O
O
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120
100
80
60
40
20
ppm
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77.142
76.806
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66.393
55.116
32.706
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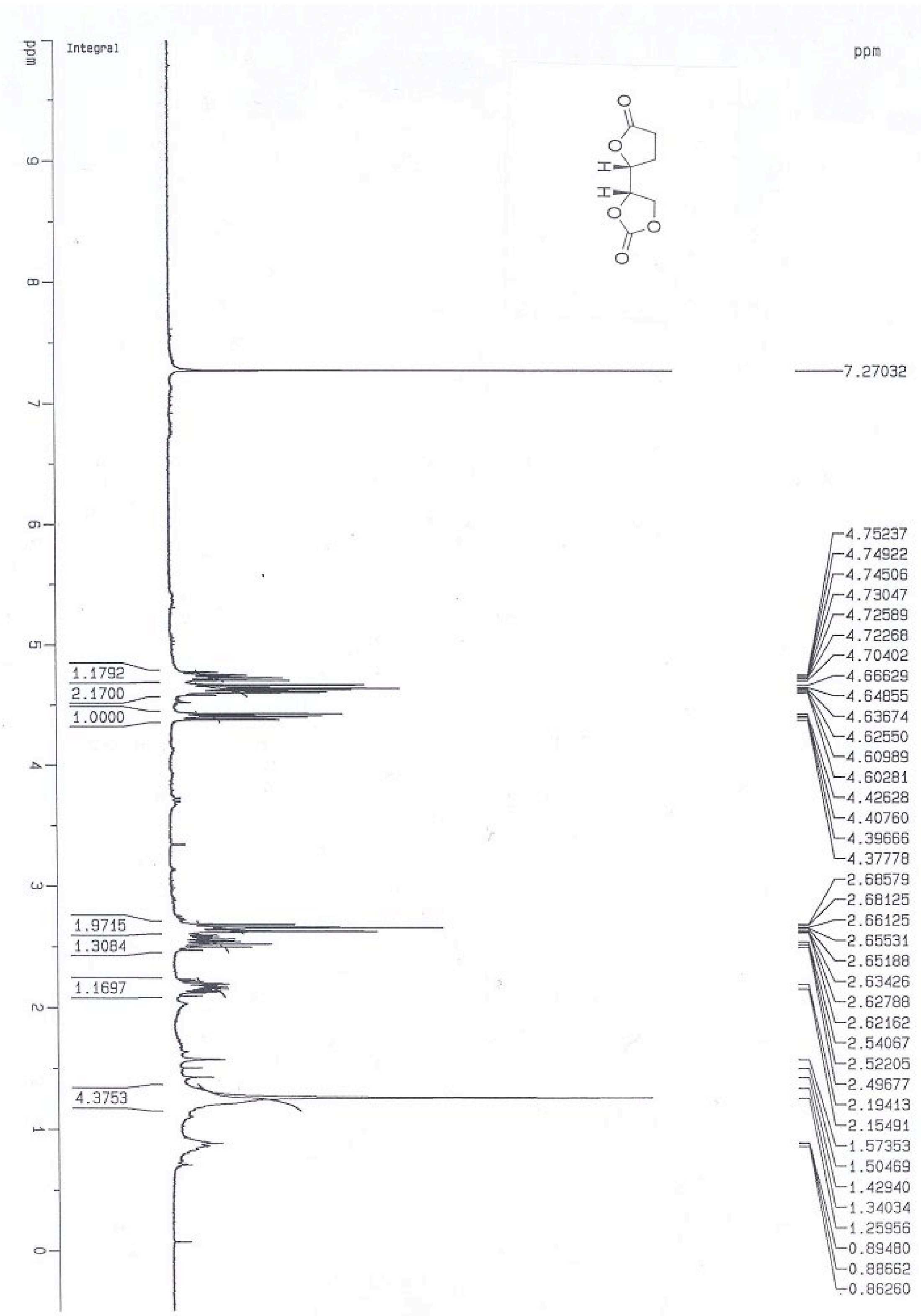
25.879
25.519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4.63674
4.62550
4.60989
4.60281
4.42628
4.40760
4.39666
4.37778
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2.52205
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1.50469
1.42940
1.34034
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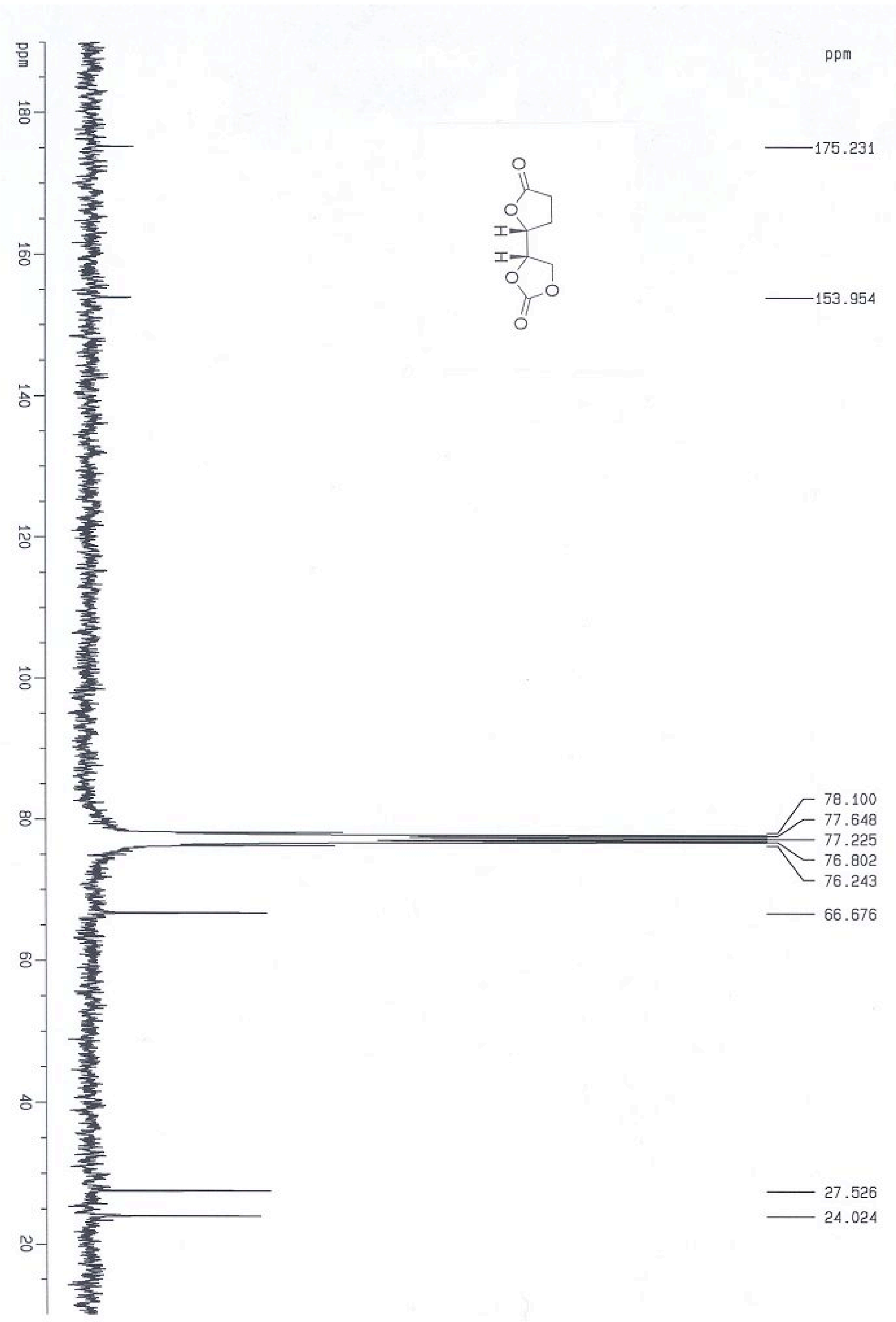
1.1697
4.3753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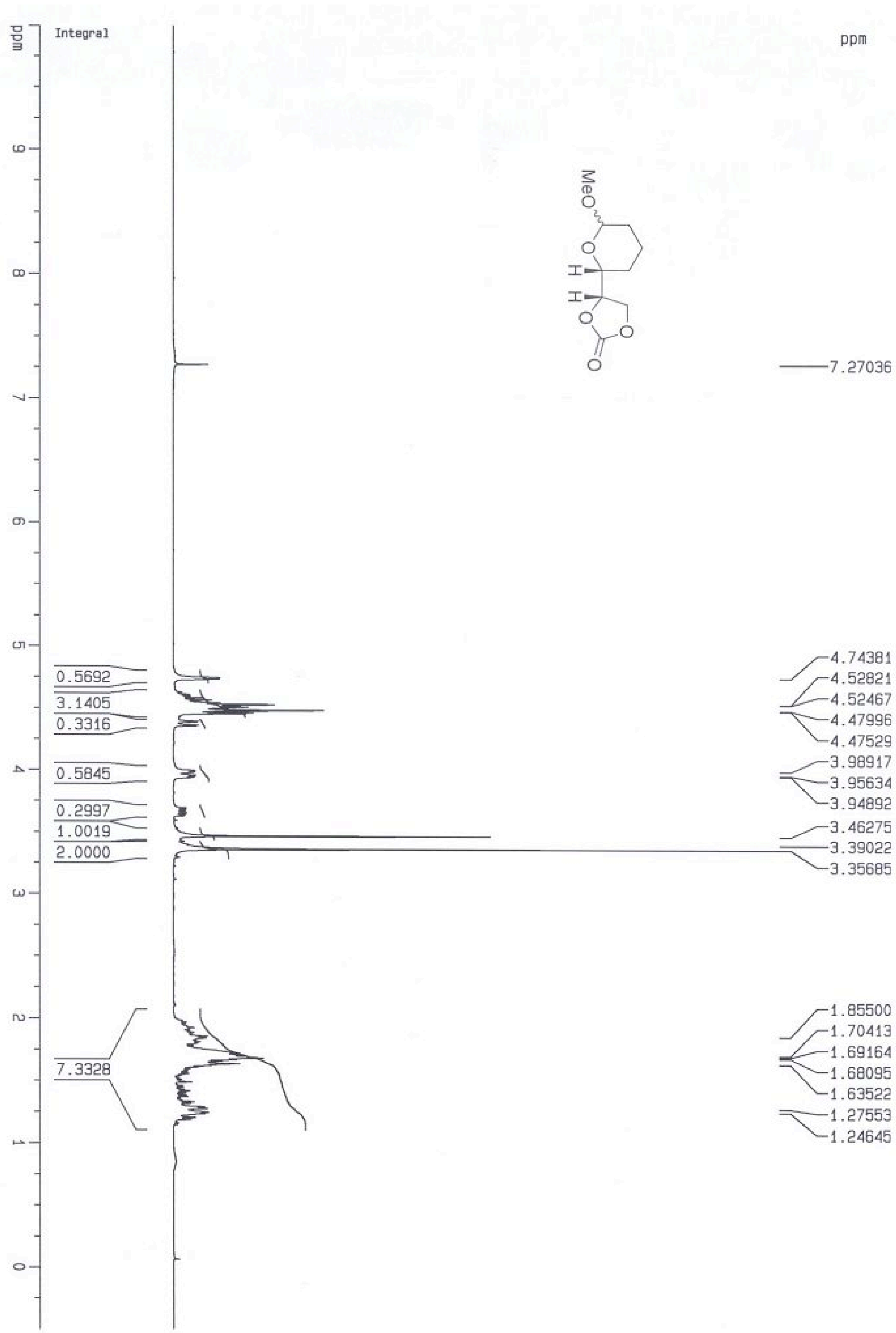
24.024



Integral
ppm
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H
H
O
O
O
O
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0.5845
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2.0000
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4.52467
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4.47529
3.98917
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3.94892
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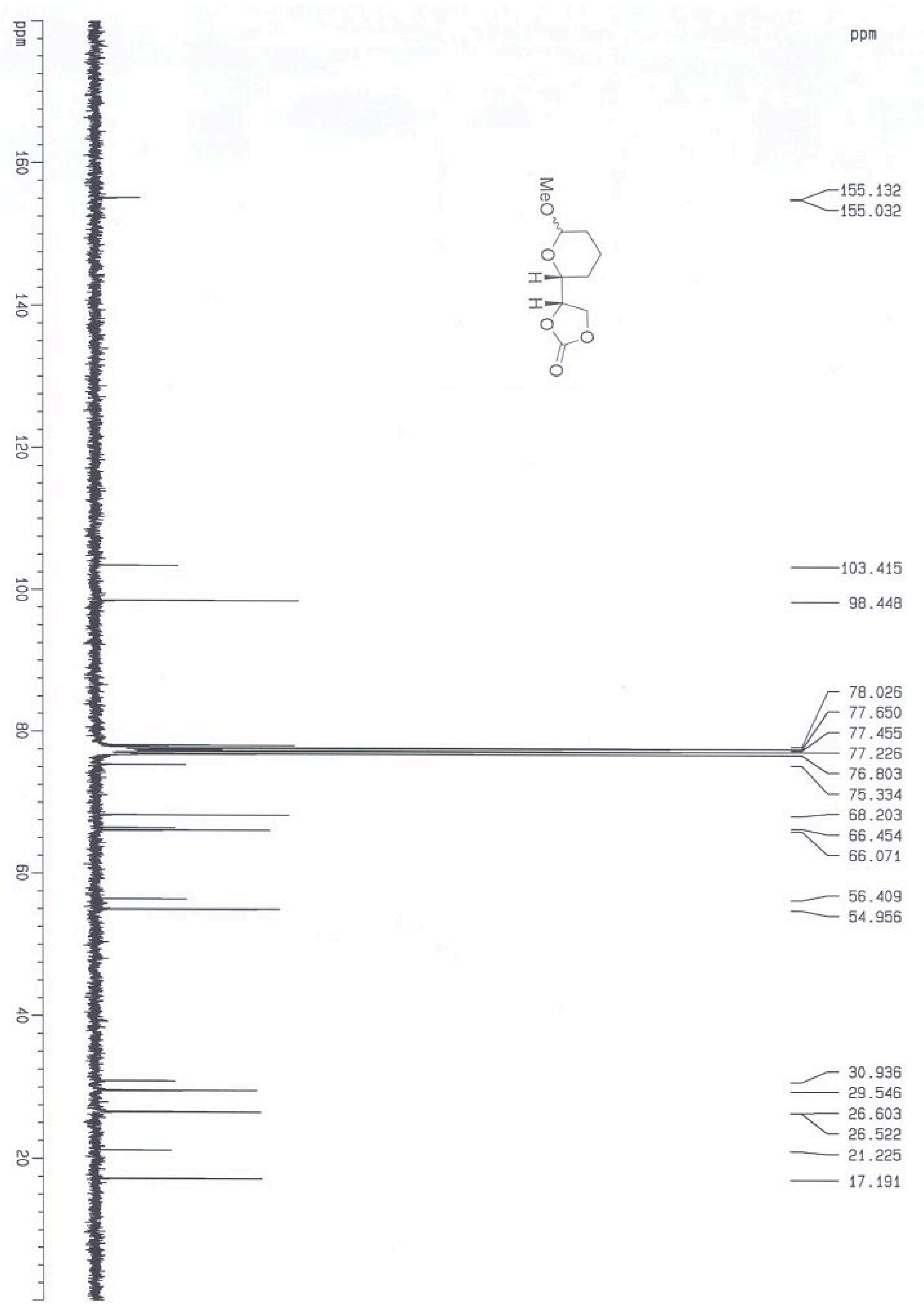
1.27553
1.24645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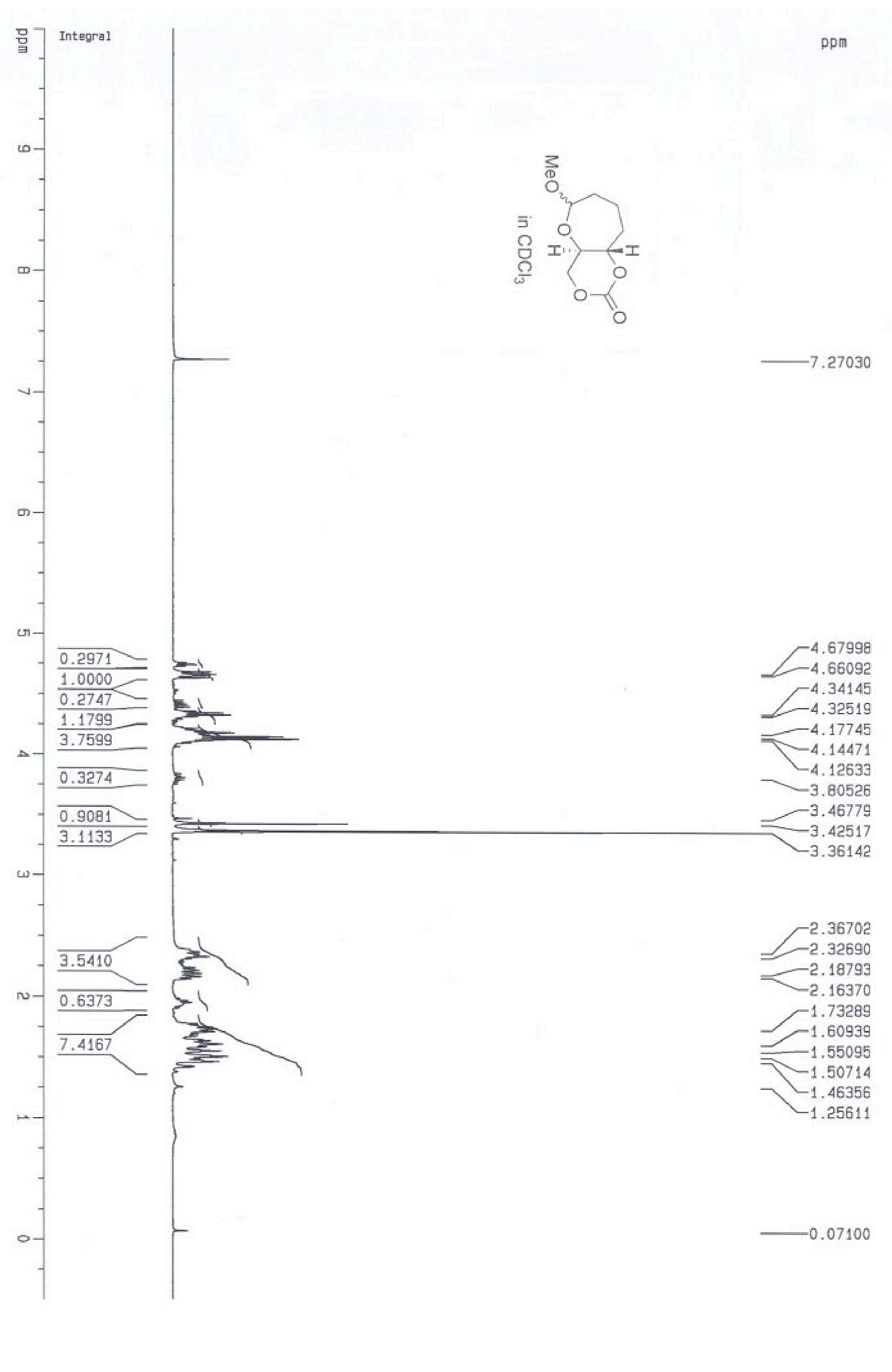

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Integral
ppm
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3.5410
0.6373
7.4167
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4.66092
4.34145
4.32519
4.17745
4.14471
4.12633
3.80526
3.46779
3.42517
3.36142
2.36702
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2.16370
1.73289
1.60939
1.55095
1.50714
1.46356
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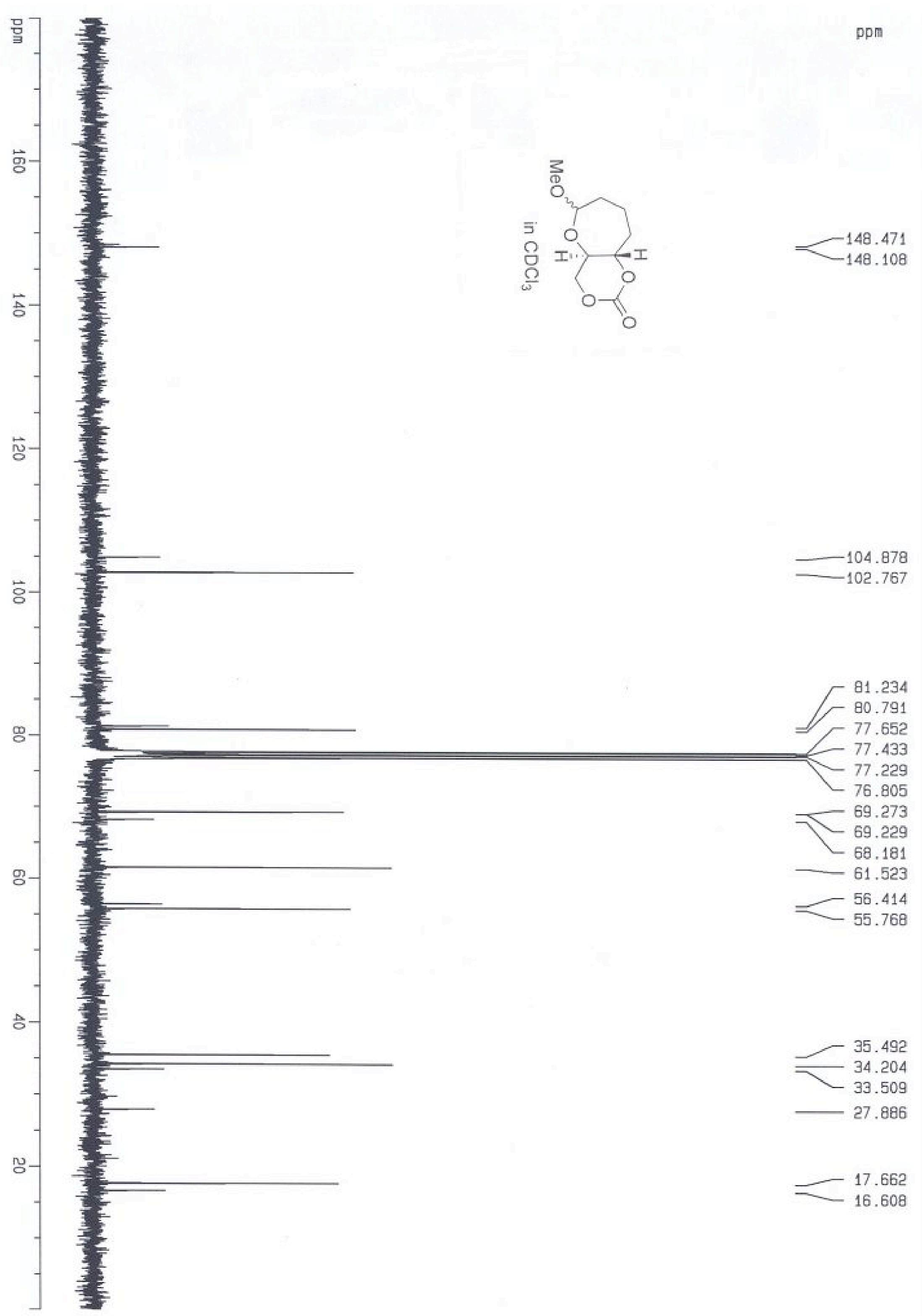
0.07100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O
in $CDCl_3$
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77.433
77.229
76.805
69.273
69.229
68.181
61.523
56.414
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34.204
33.509
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80
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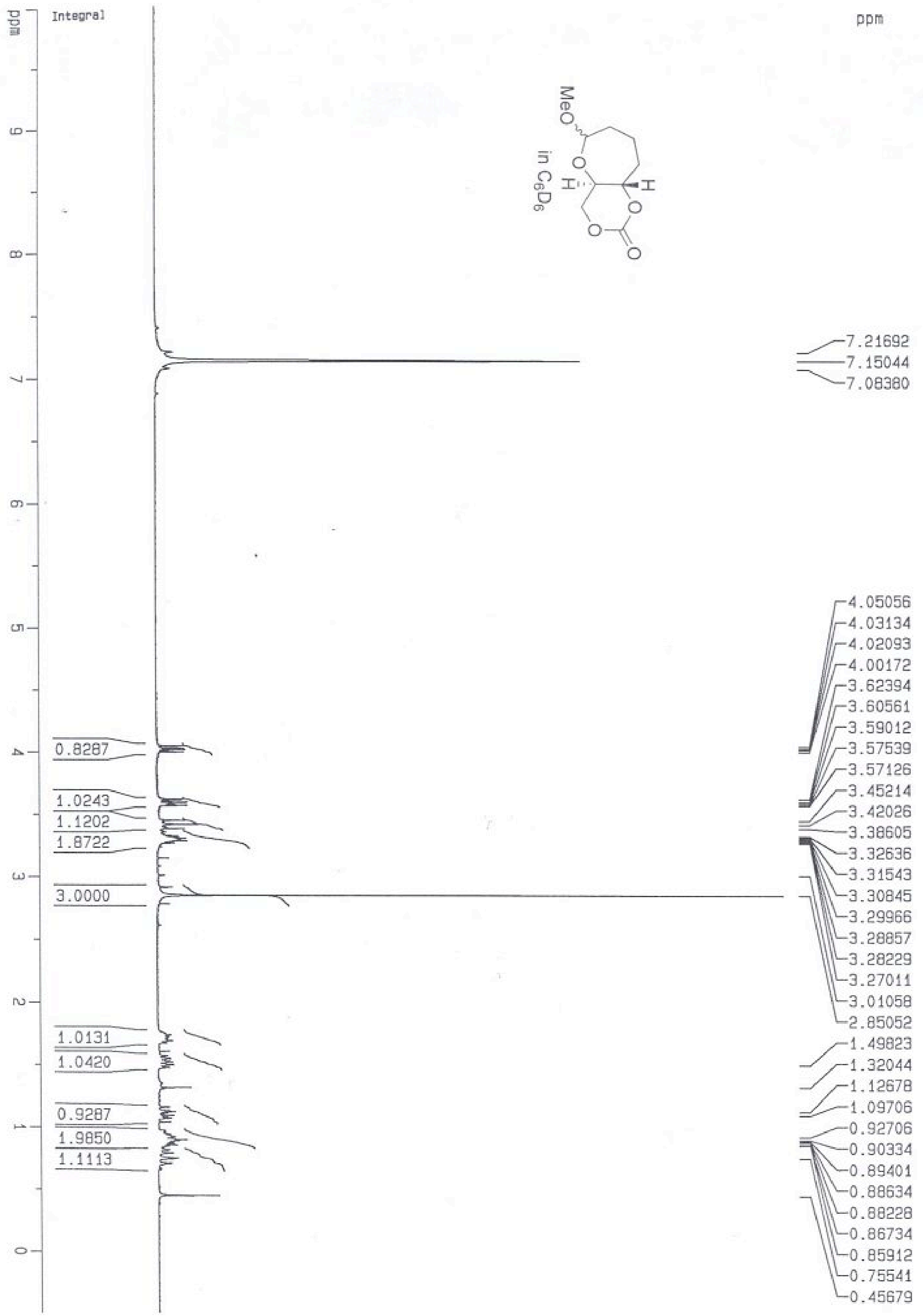
40
20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H
H
O
O
O
in C_6D_6
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1.1113
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7.15044
7.08360
4.05056
4.03134
4.02093
4.00172
3.62394
3.60561
3.59012
3.57539
3.57126
3.45214
3.42026
3.38605
3.32636
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3.01058
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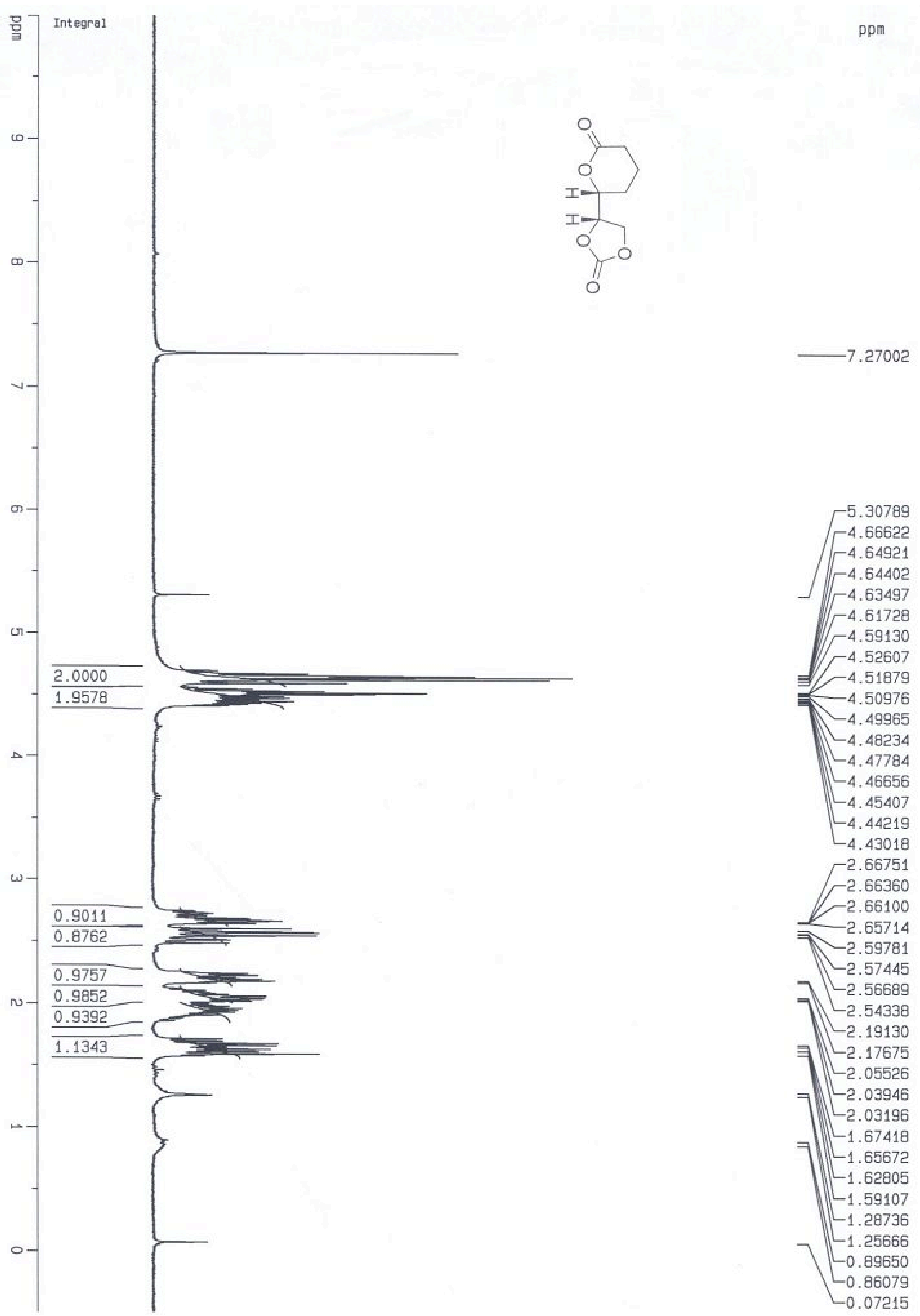
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4.64402
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4.61728
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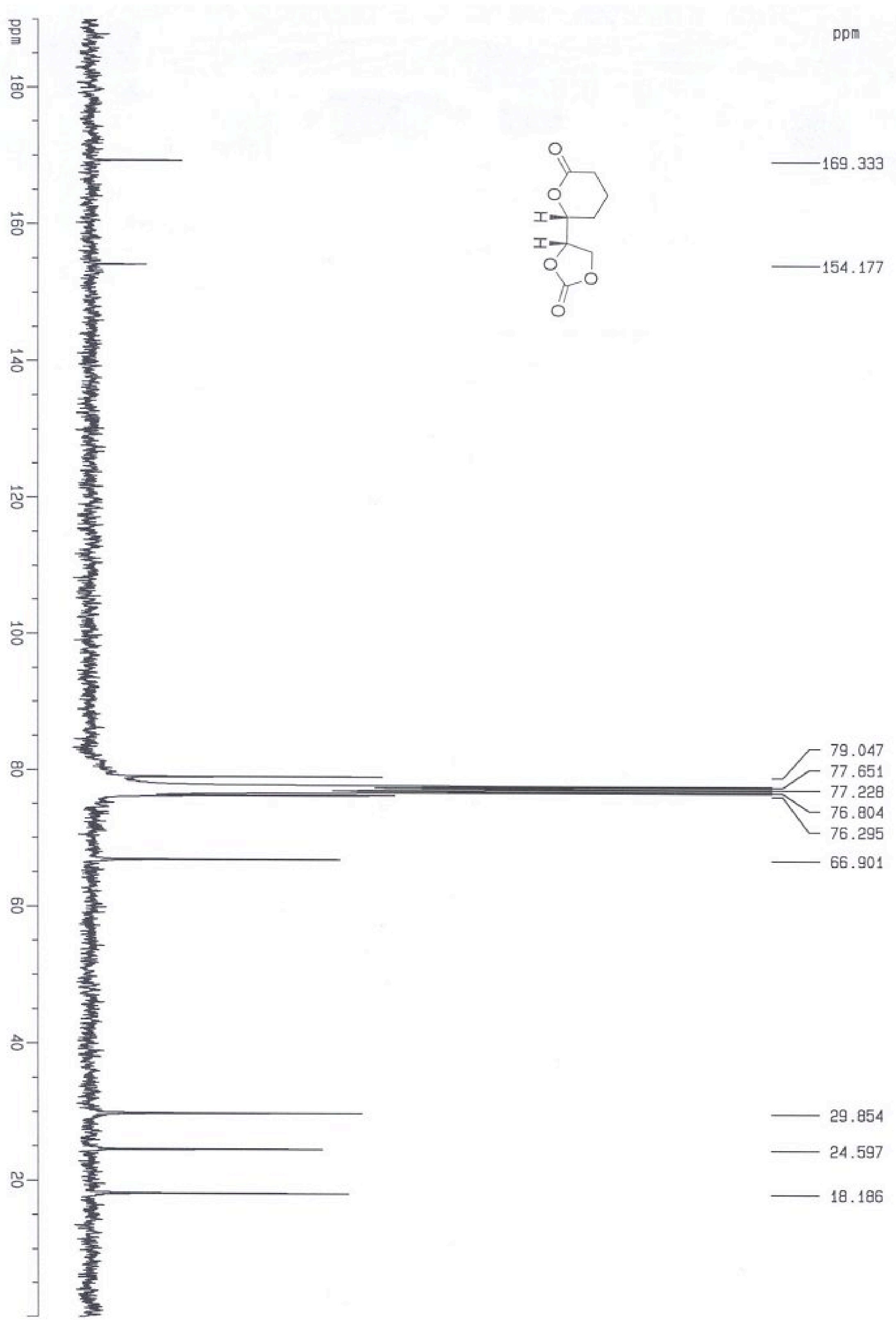
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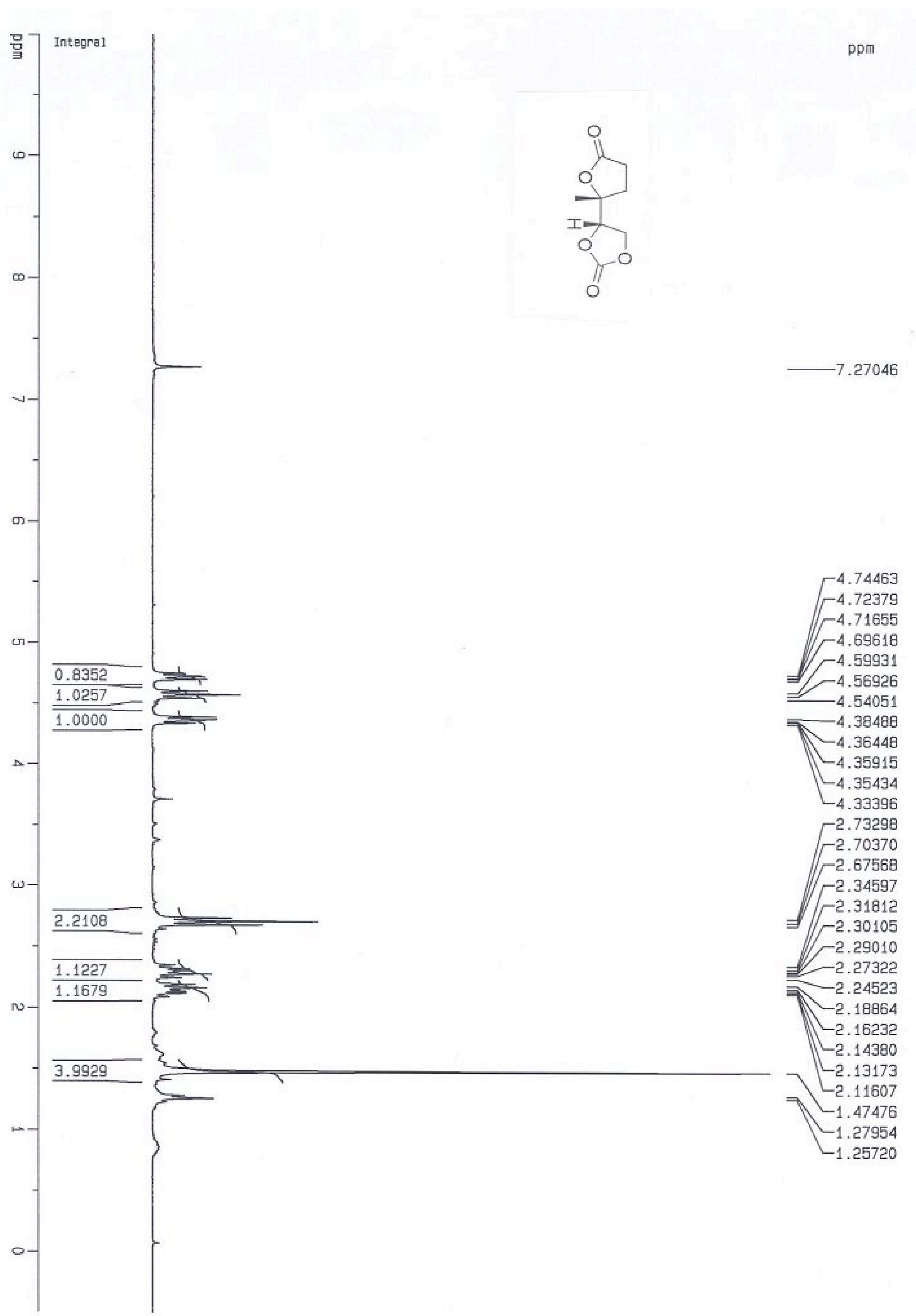

ppm
Integral
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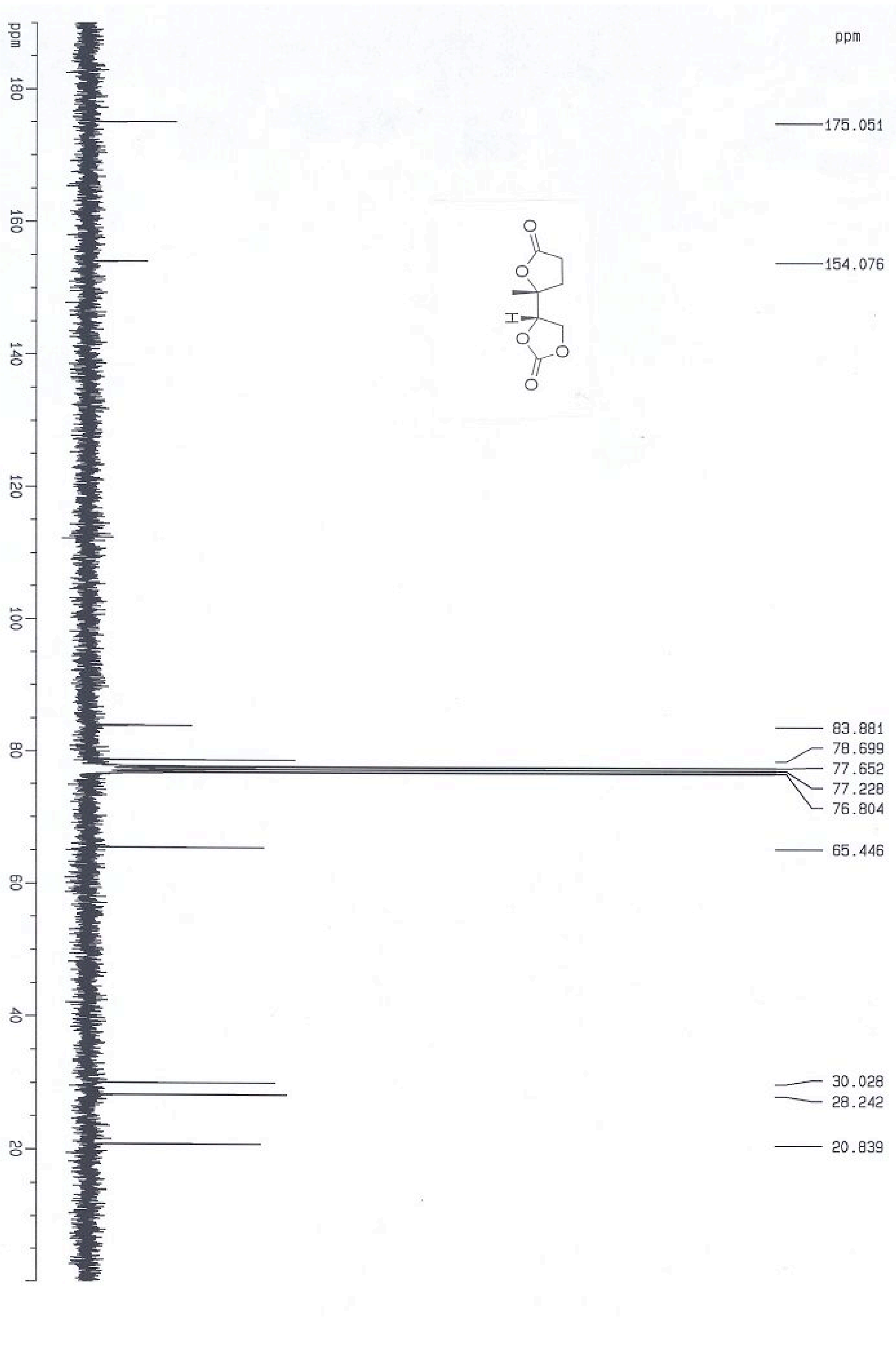
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40
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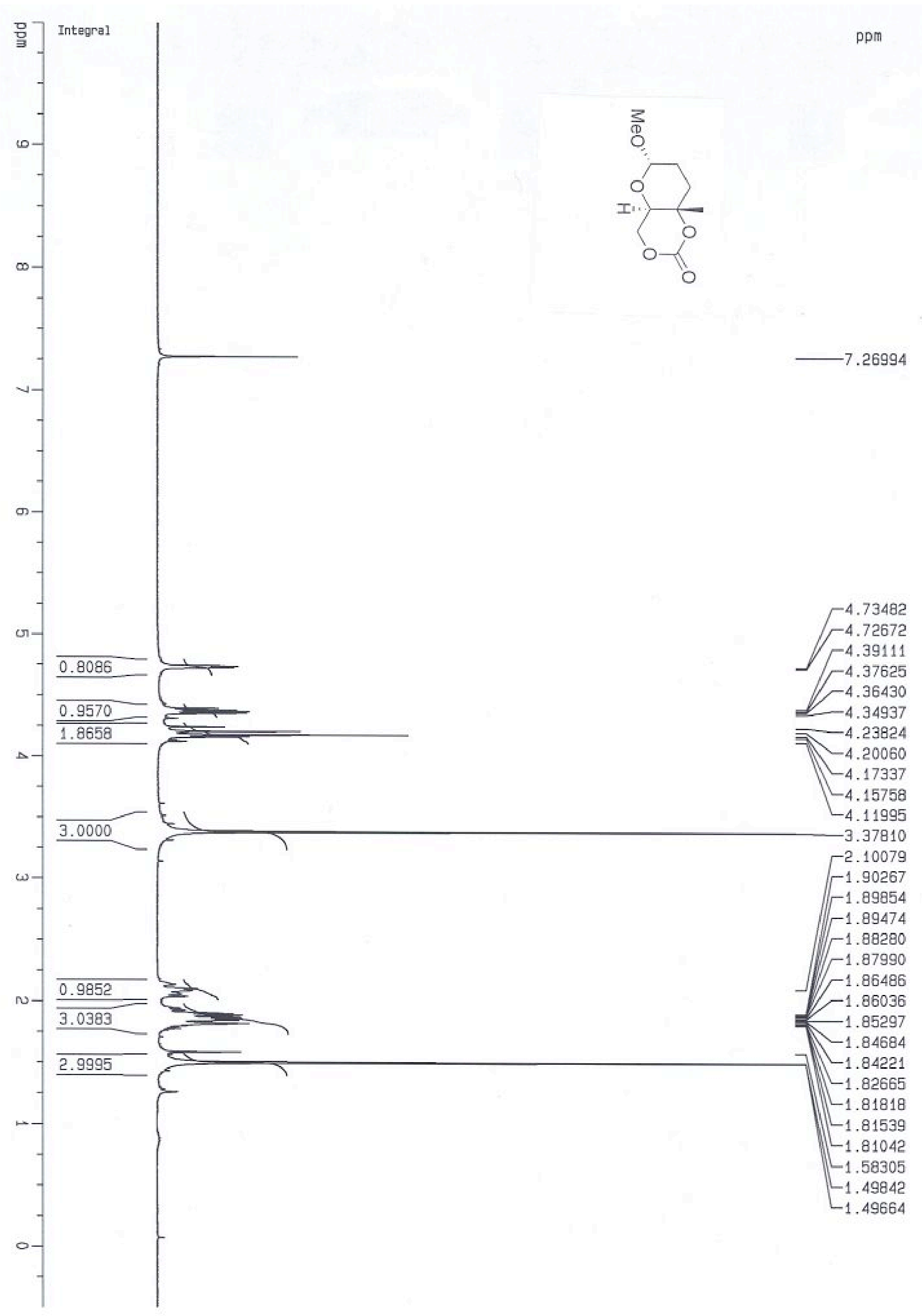

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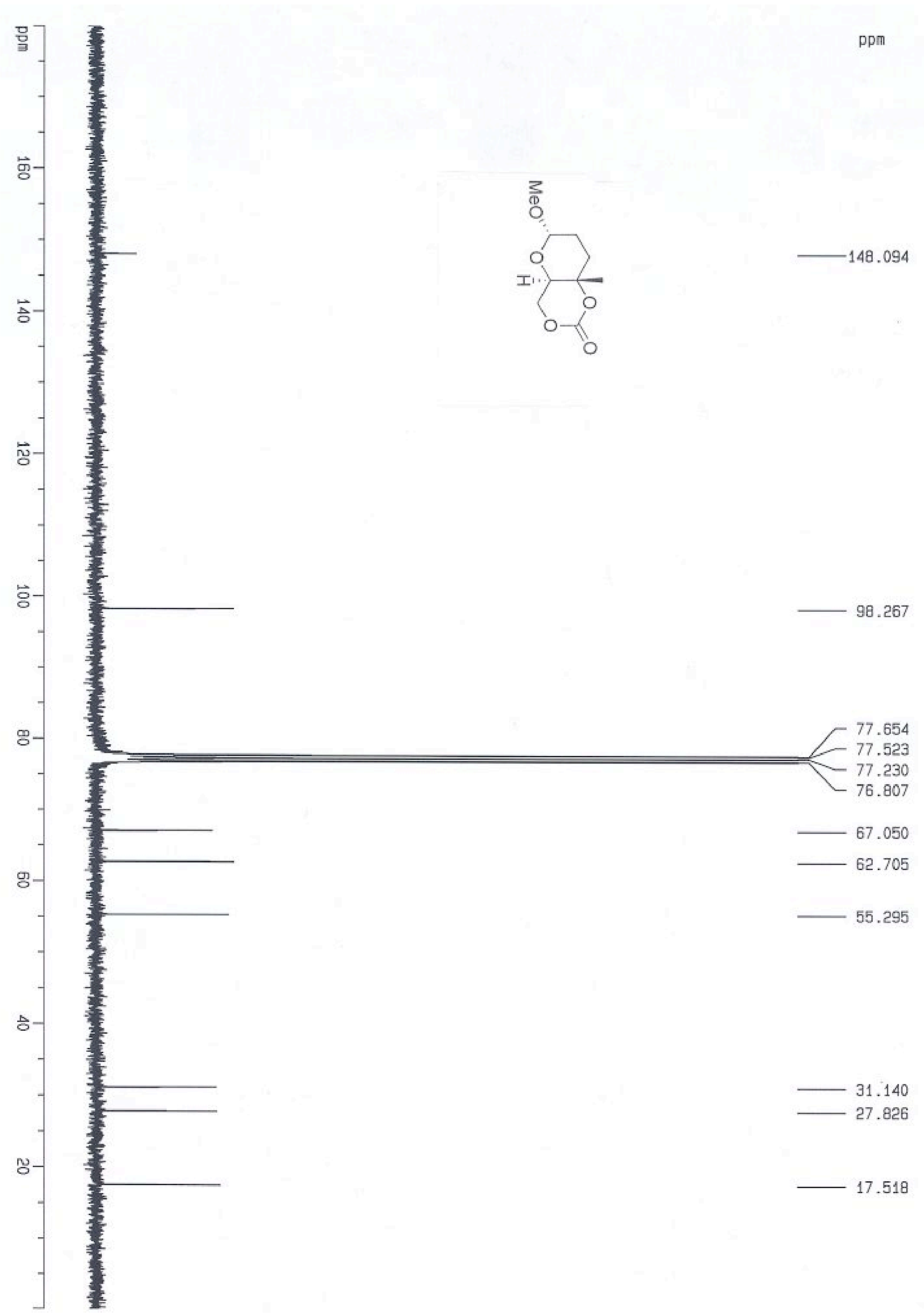
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H
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140
120
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60
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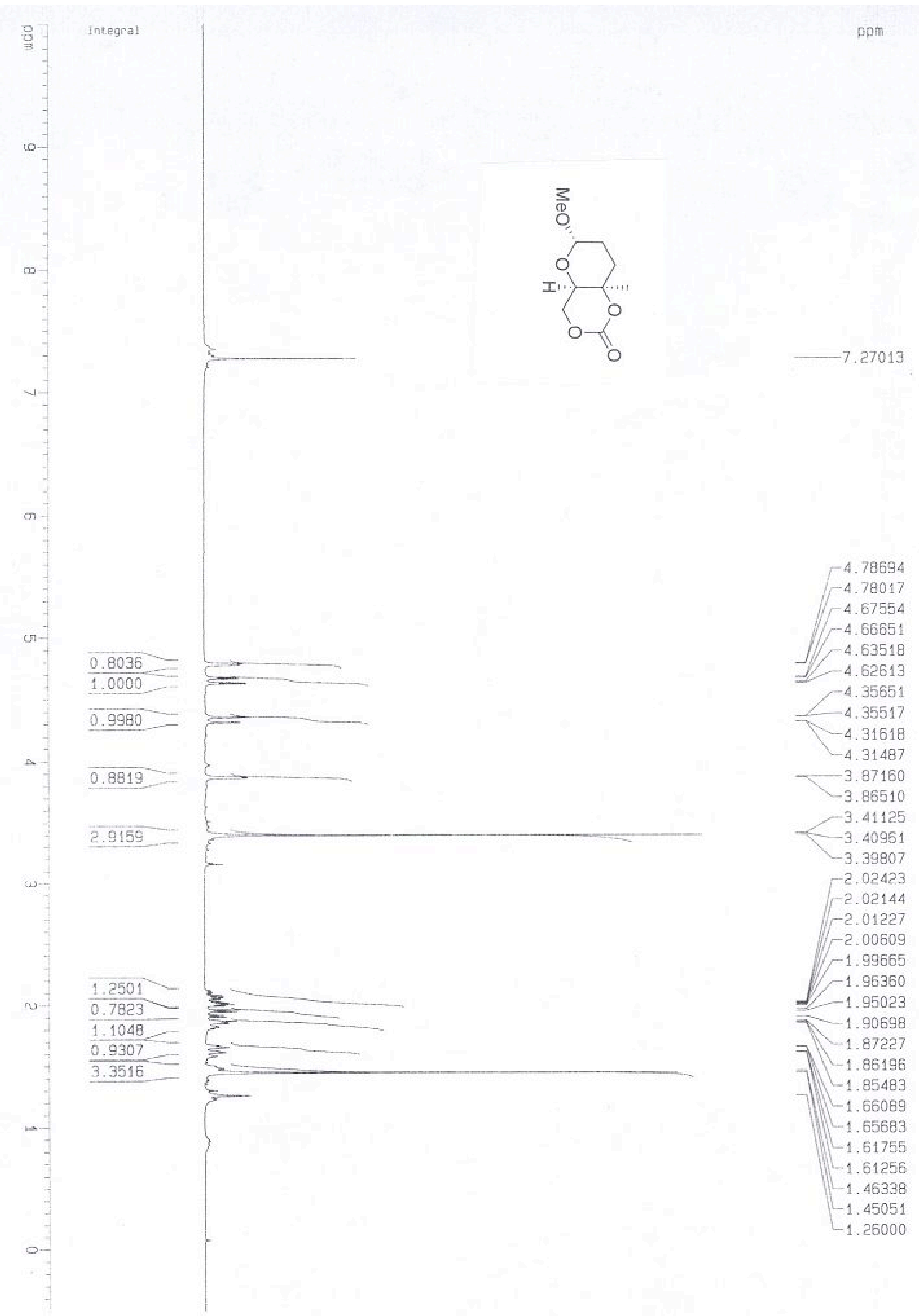
20
ppm



ppm
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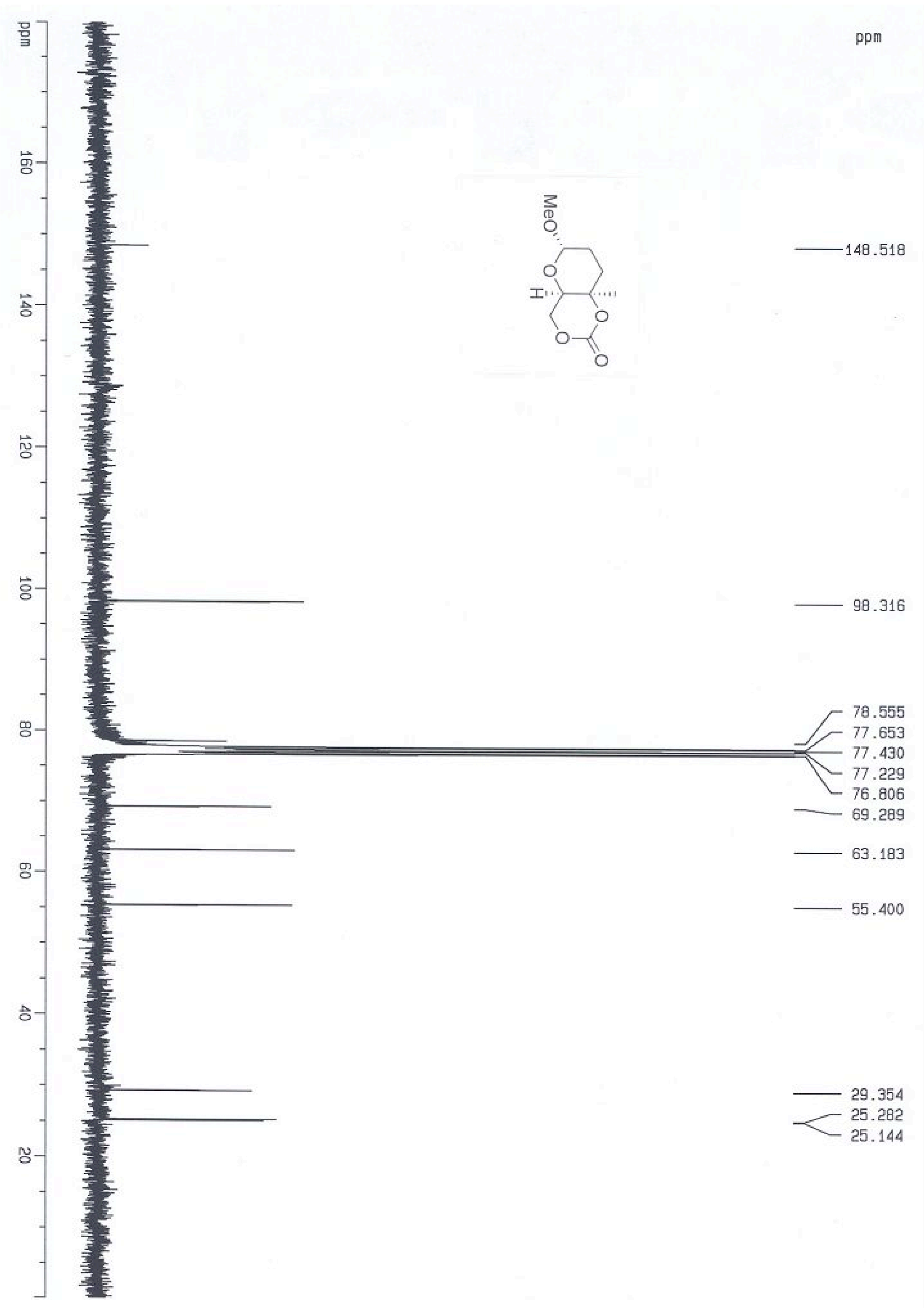
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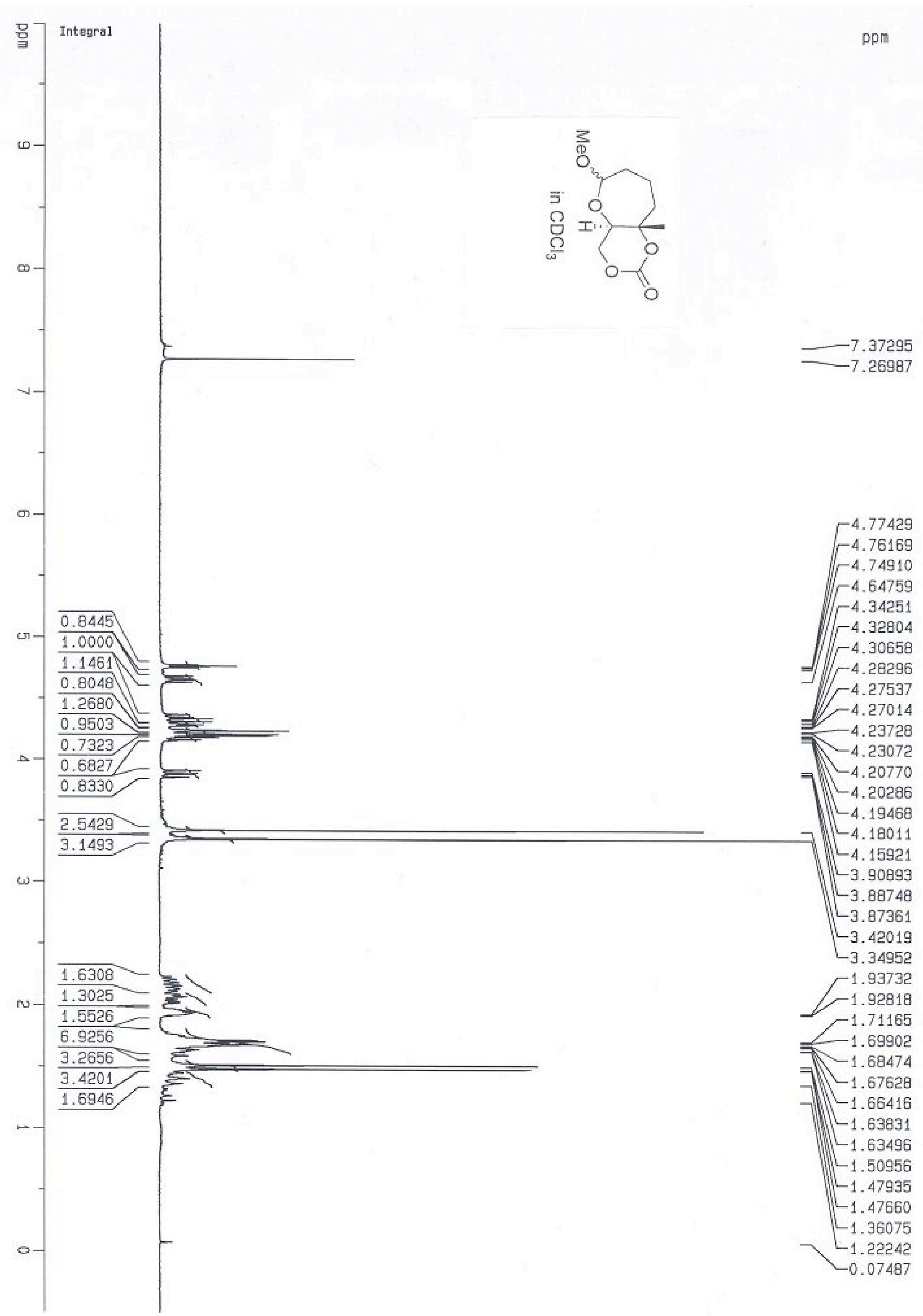
O
O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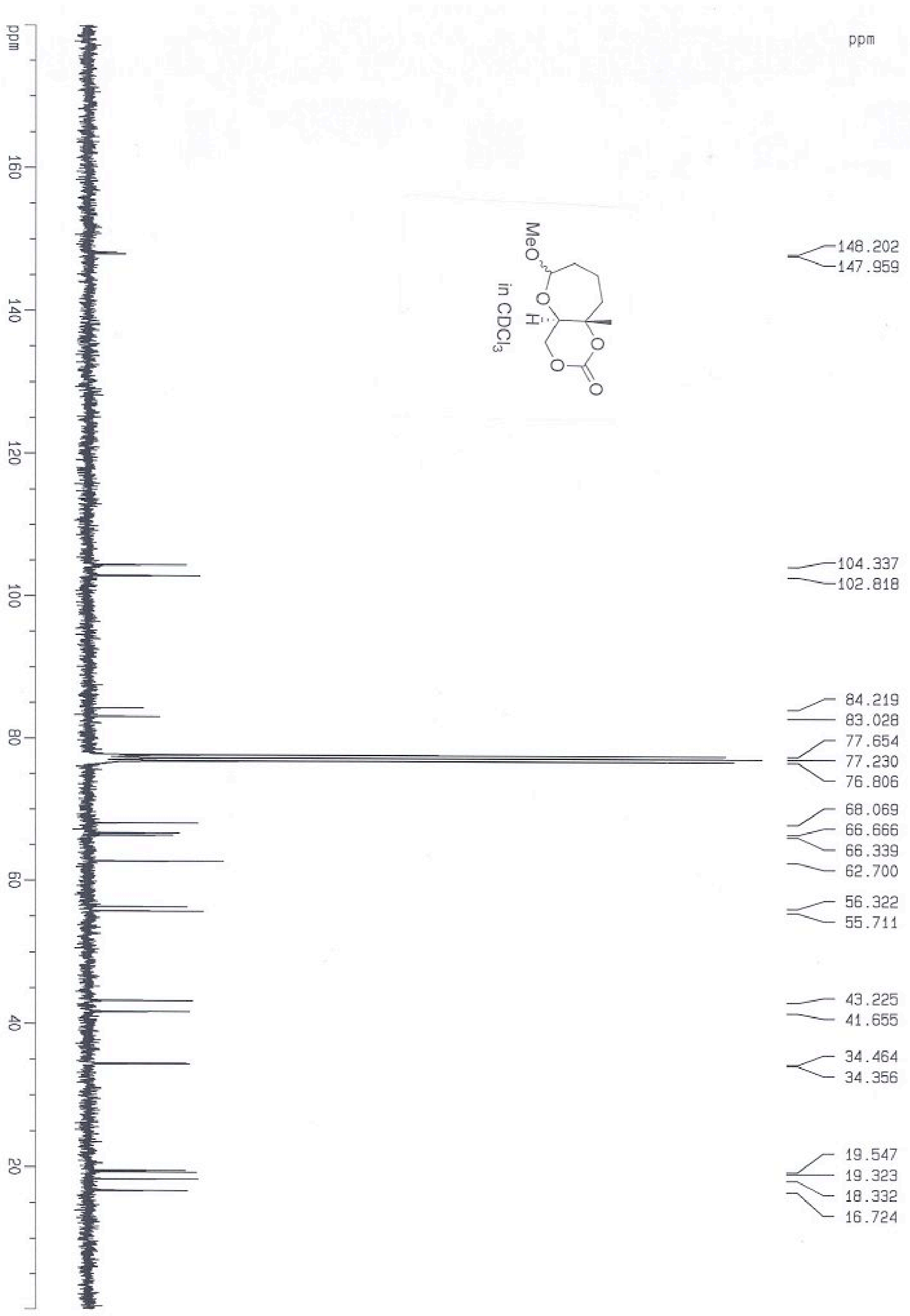
3.4201
1.6946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102.818
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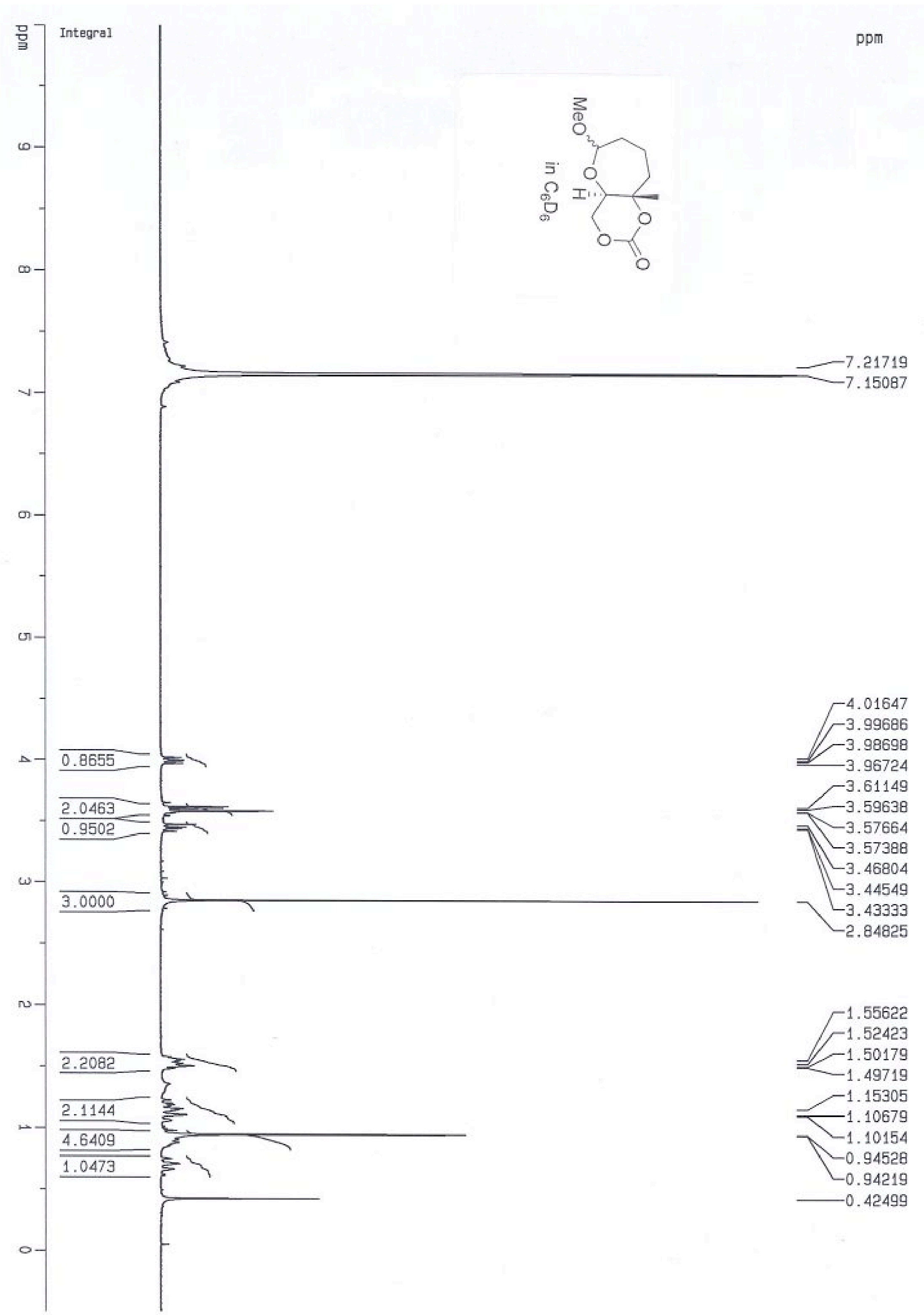
77.230
76.806
68.069
66.666
66.339
62.700
56.322
55.711
43.225
41.655
34.464
34.356
19.547
19.323
18.332
16.724
MeO
in CDCl3
ppm
160
140
120
100
80
60
40
20


Integral
ppm
MeO
in C6D6
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7.15087
4.01647
3.99686
3.98698
3.96724
3.61149
3.59638
3.57664
3.57388
3.46804
3.44549
3.43333
2.84825
1.55622
1.52423
1.50179
1.49719
1.15305
1.10679
1.10154
0.94528
0.94219
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0.8655
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0.9502
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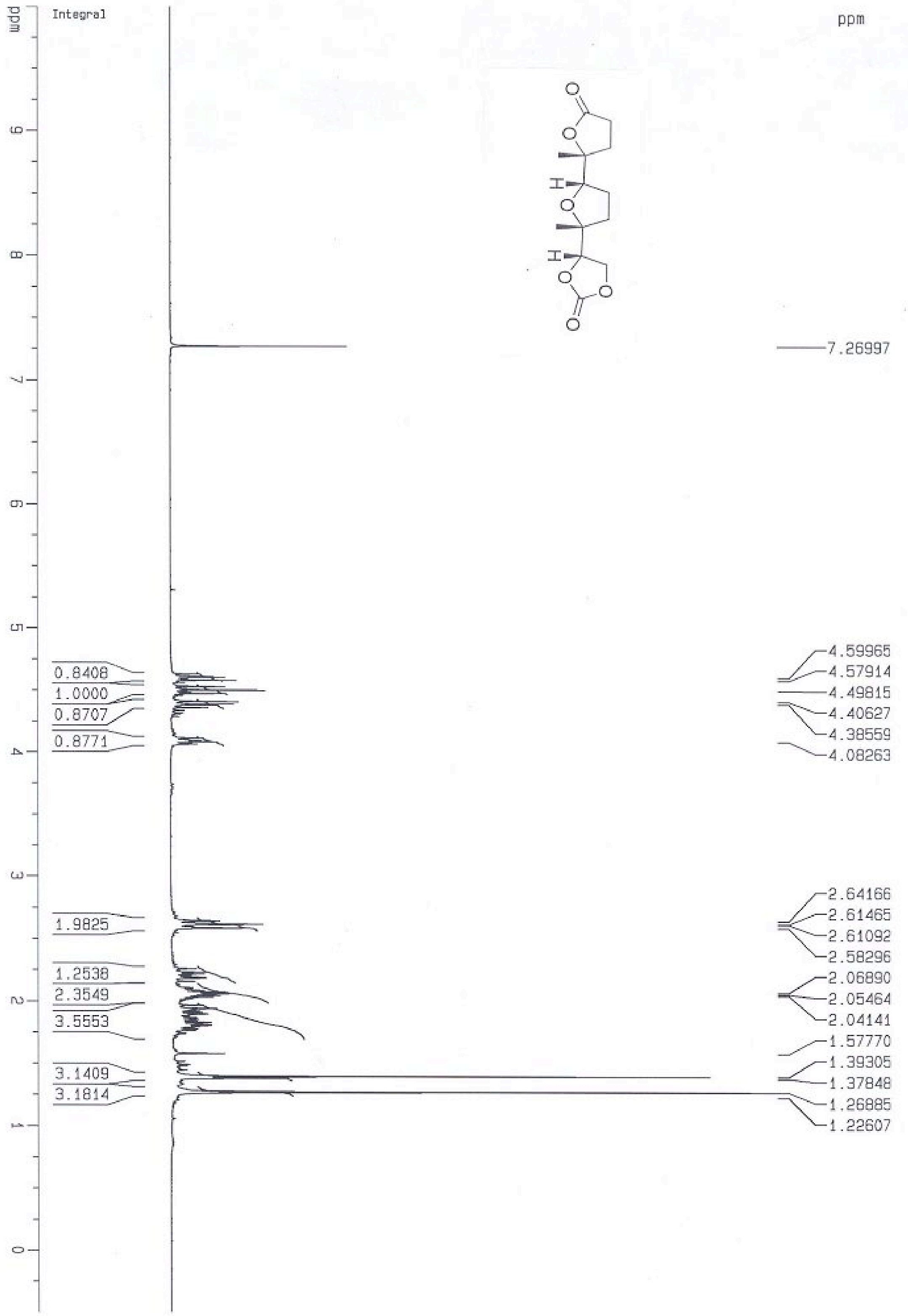
2.2082
2.1144
4.6409
1.0473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4.38559
4.08263
1.9825
1.2538
2.3549
3.5553
3.1409
3.1814
2.64166
2.61465
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2.58296
2.06890
2.05464
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1.39305
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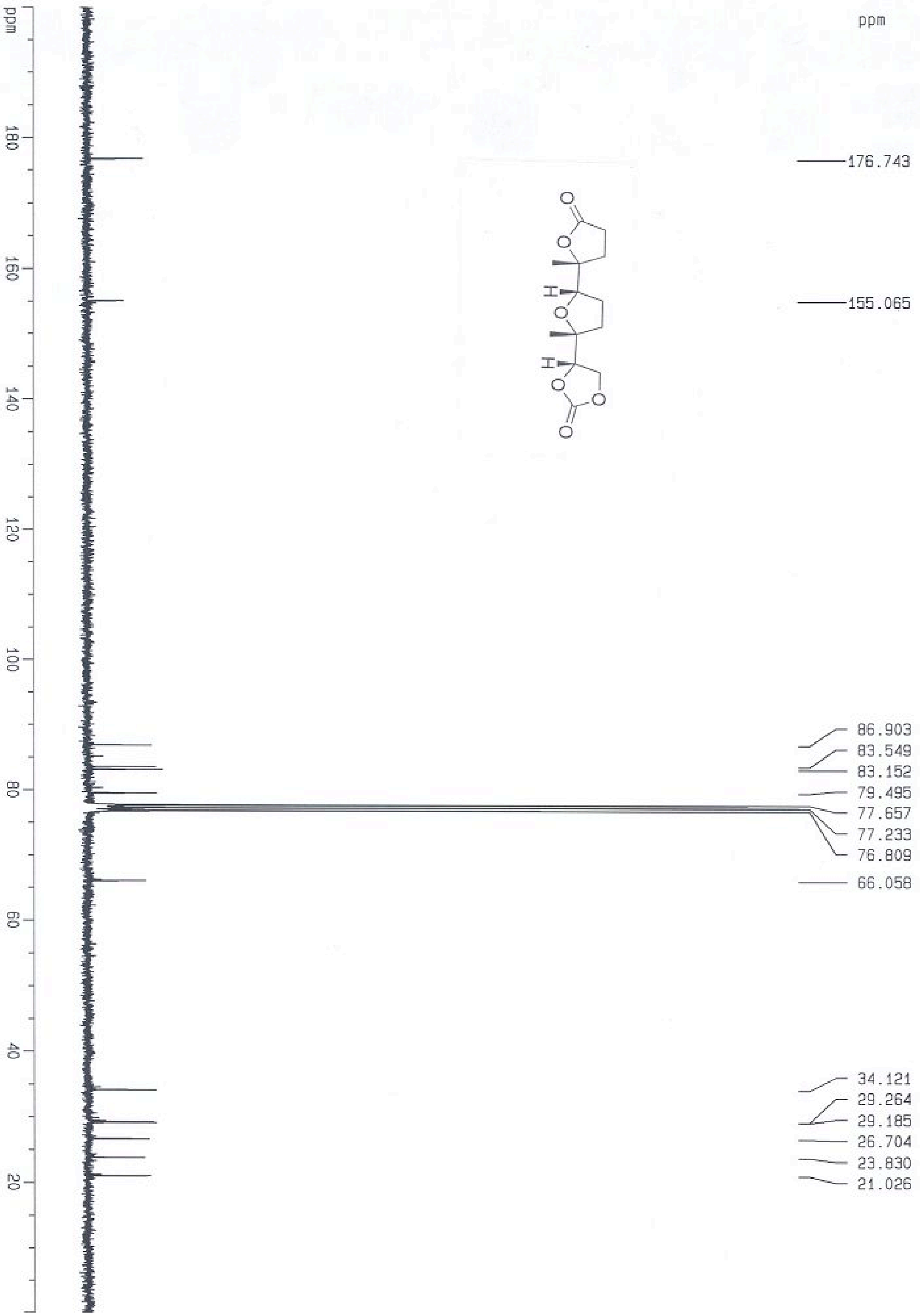
1.26885
1.22607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77.657
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76.809
66.058
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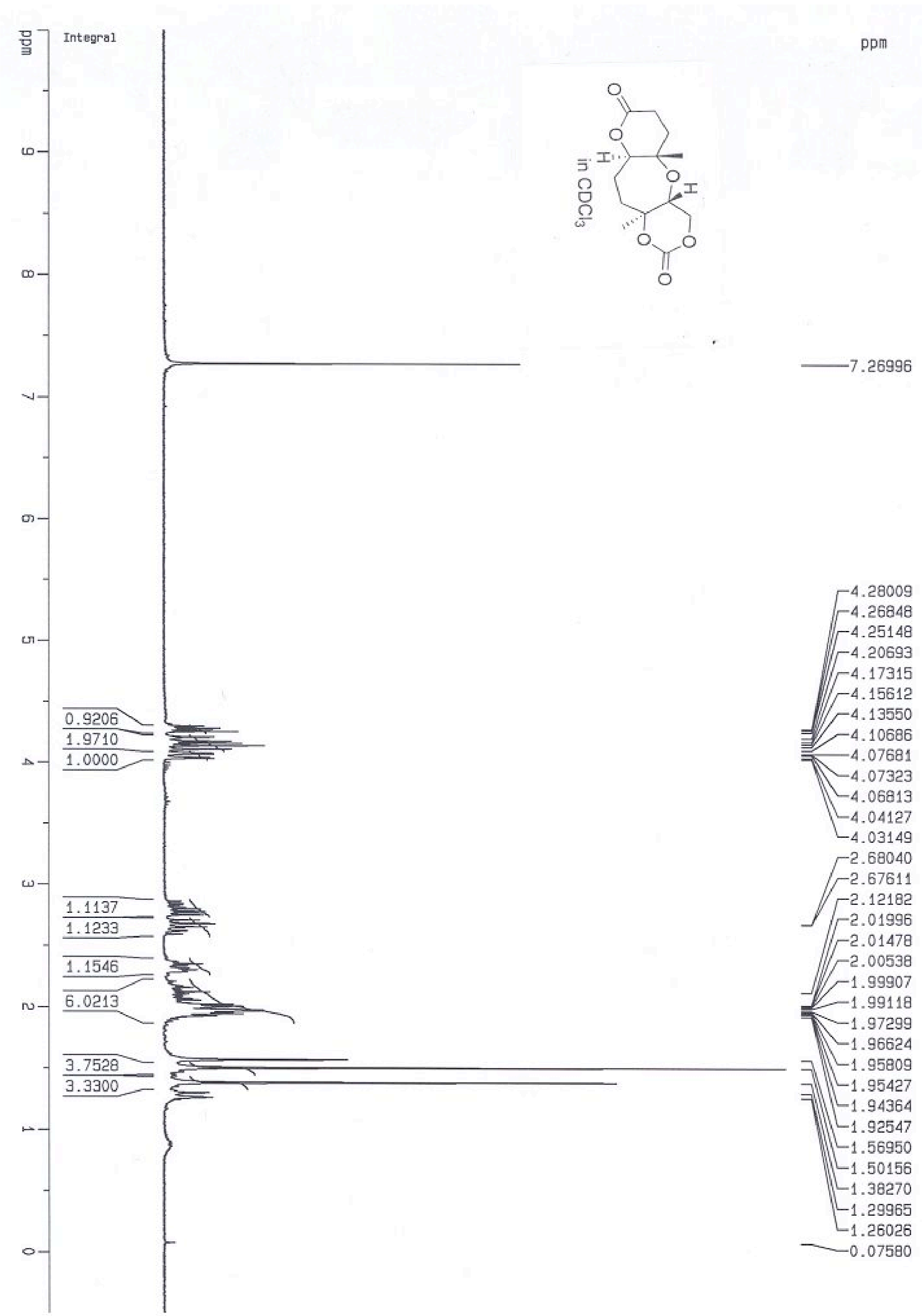
29.185
26.704
23.830
21.026
ppm
180
160
140
120
100
80
60
40
20


ppm
Integral
ppm
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4.26848
4.25148
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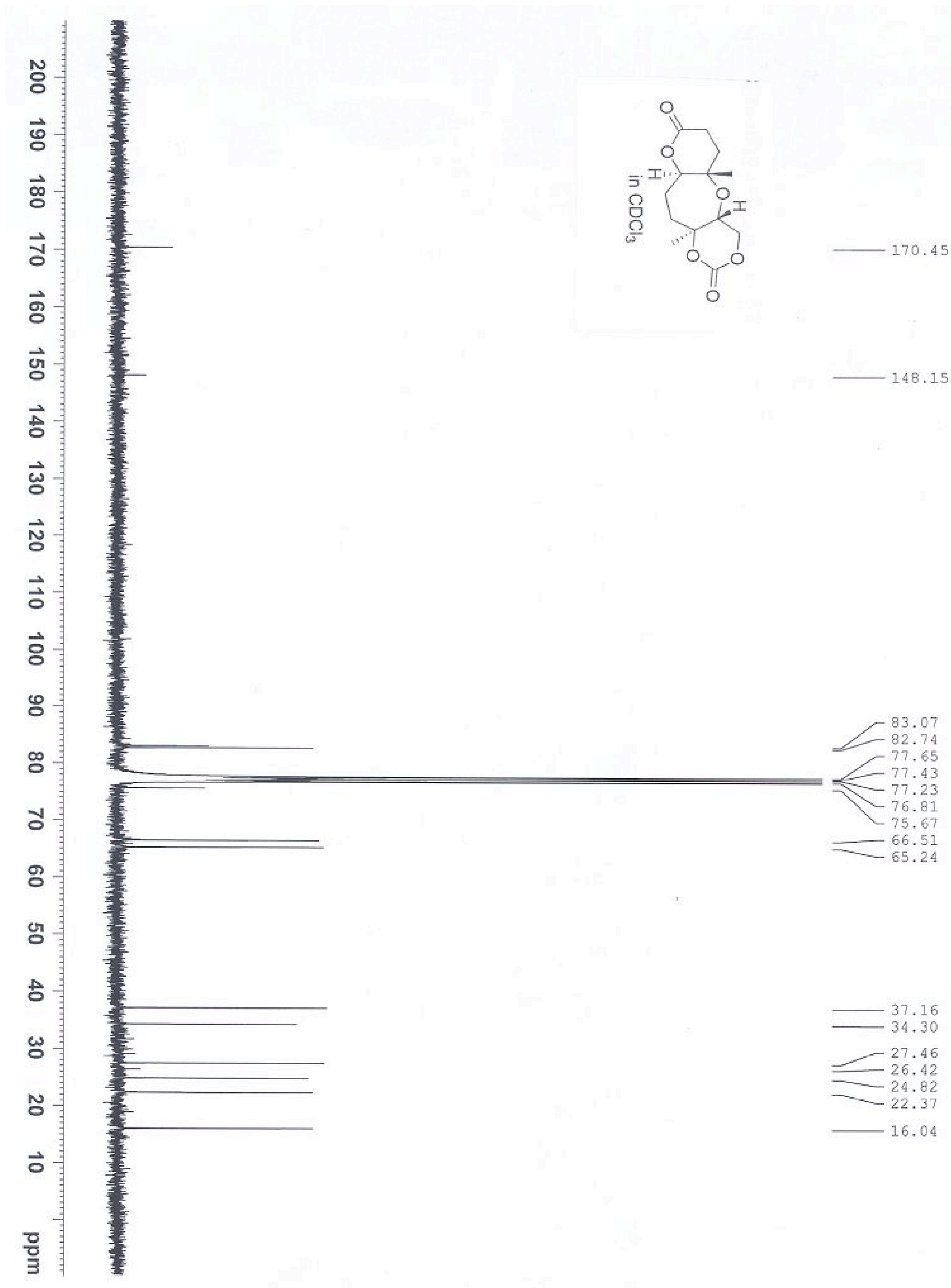
4.17315
4.15612
4.13550
4.10686
4.07681
4.07323
4.06813
4.04127
4.03149
2.68040
2.67611
2.12182
2.01996
2.01478
2.00538
1.99907
1.99118
1.97299
1.96624
1.95809
1.95427
1.94364
1.92547
1.56950
1.50156
1.38270
1.29965
1.26026
0.07580
0.9206
1.9710
1.0000
1.1137
1.1233
1.1546
6.0213
3.7528
3.3300


in CDCl3
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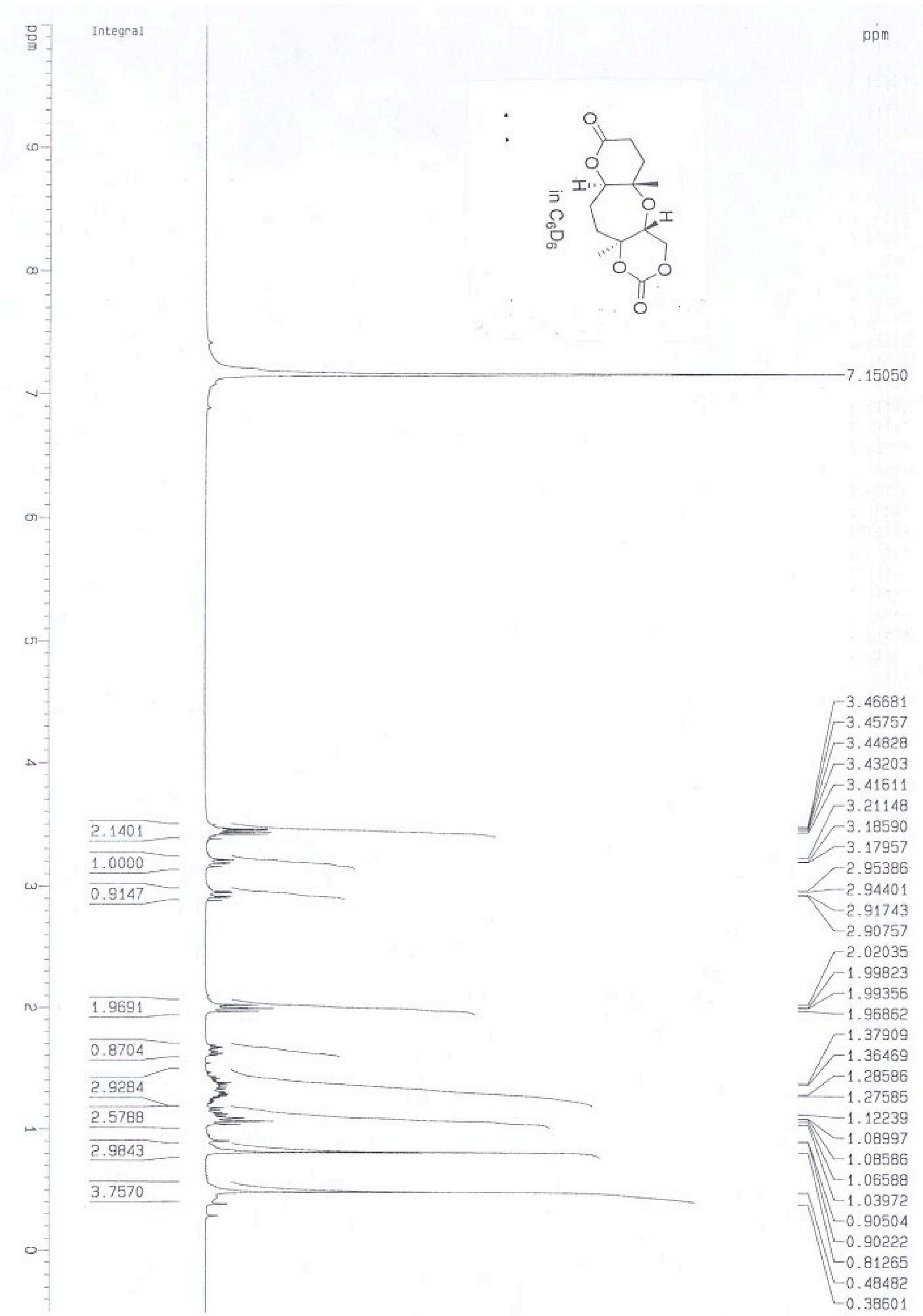
148.15
83.07
82.74
77.65
77.43
77.23
76.81
75.67
66.51
65.24
37.16
34.30
27.46
26.42
24.82
22.37
16.04
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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in C_6D_6
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3.44828
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3.41611
3.21148
3.18590
3.17957
2.95386
2.94401
2.91743
2.90757
2.02035
1.99823
1.99356
1.96862
1.37909
1.36469
1.28586
1.27585
1.12239
1.08997
1.08586
1.06588
1.03972
0.90504
0.90222
0.81265
0.48482
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1.0000
0.9147
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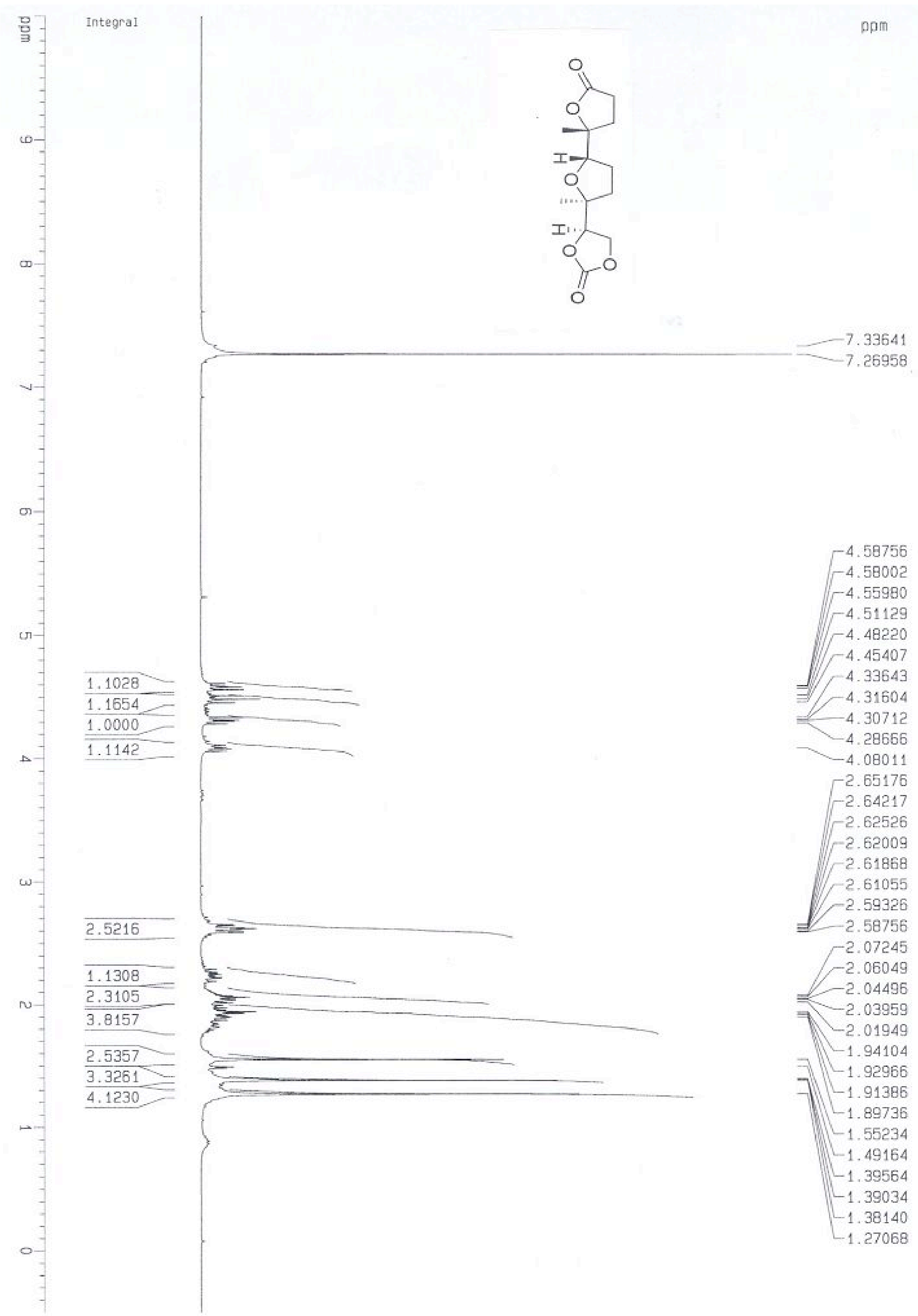
0.8704
2.9284
2.5788
2.9843
3.7570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Integral
ppm
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4.58002
4.55980
4.51129
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4.45407
4.33643
4.31604
4.30712
4.28666
4.08011
2.65176
2.64217
2.62526
2.62009
2.61868
2.61055
2.59326
2.58756
2.07245
2.06049
2.04496
2.03959
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1.94104
1.92966
1.91386
1.89736
1.55234
1.49164
1.39564
1.39034
1.38140
1.27068
1.1028
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3.8157
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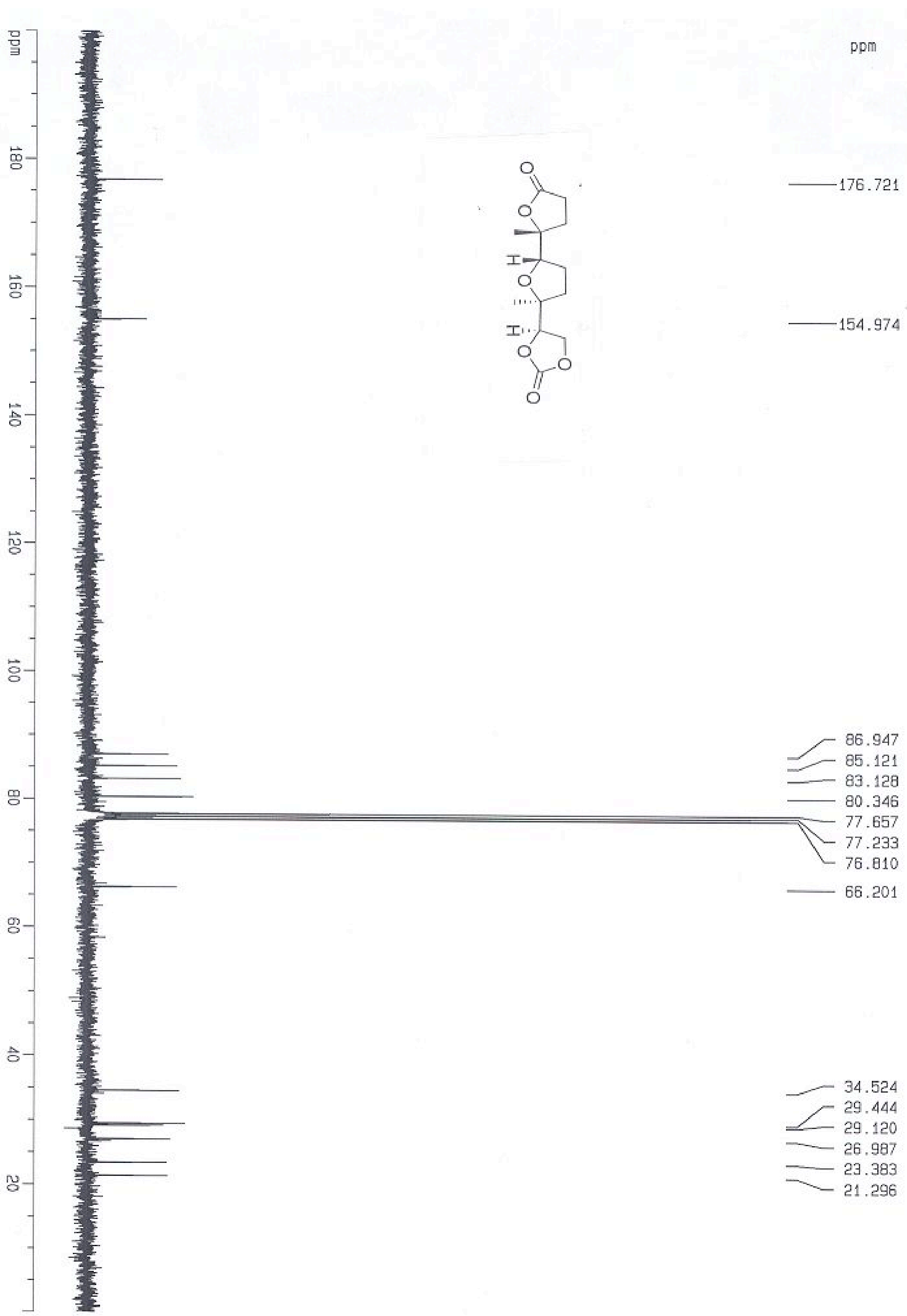
3.3261
4.1230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77.657
77.233
76.810
66.201
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29.120
26.987
23.383
21.296
ppm
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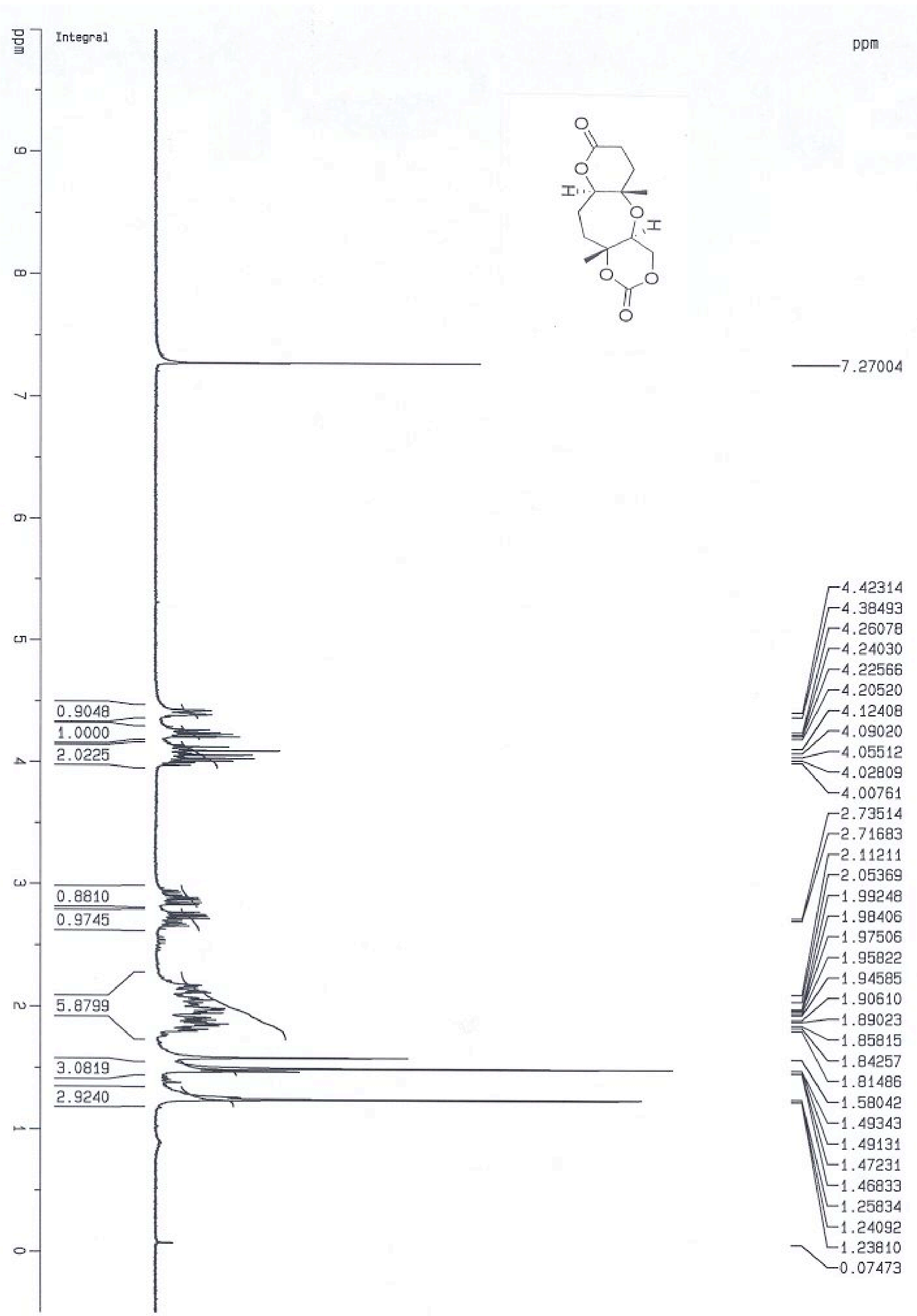
160
140
120
100
80
60
40
20


Integral
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4.26078
4.24030
4.22566
4.20520
4.12408
4.09020
4.05512
4.02809
4.00761
2.73514
2.71683
2.11211
2.05369
1.99248
1.98406
1.97506
1.95822
1.94585
1.90610
1.89023
1.85815
1.84257
1.81486
1.58042
1.49343
1.49131
1.47231
1.46833
1.25834
1.24092
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0.9048
1.0000
2.0225
0.8810
0.9745
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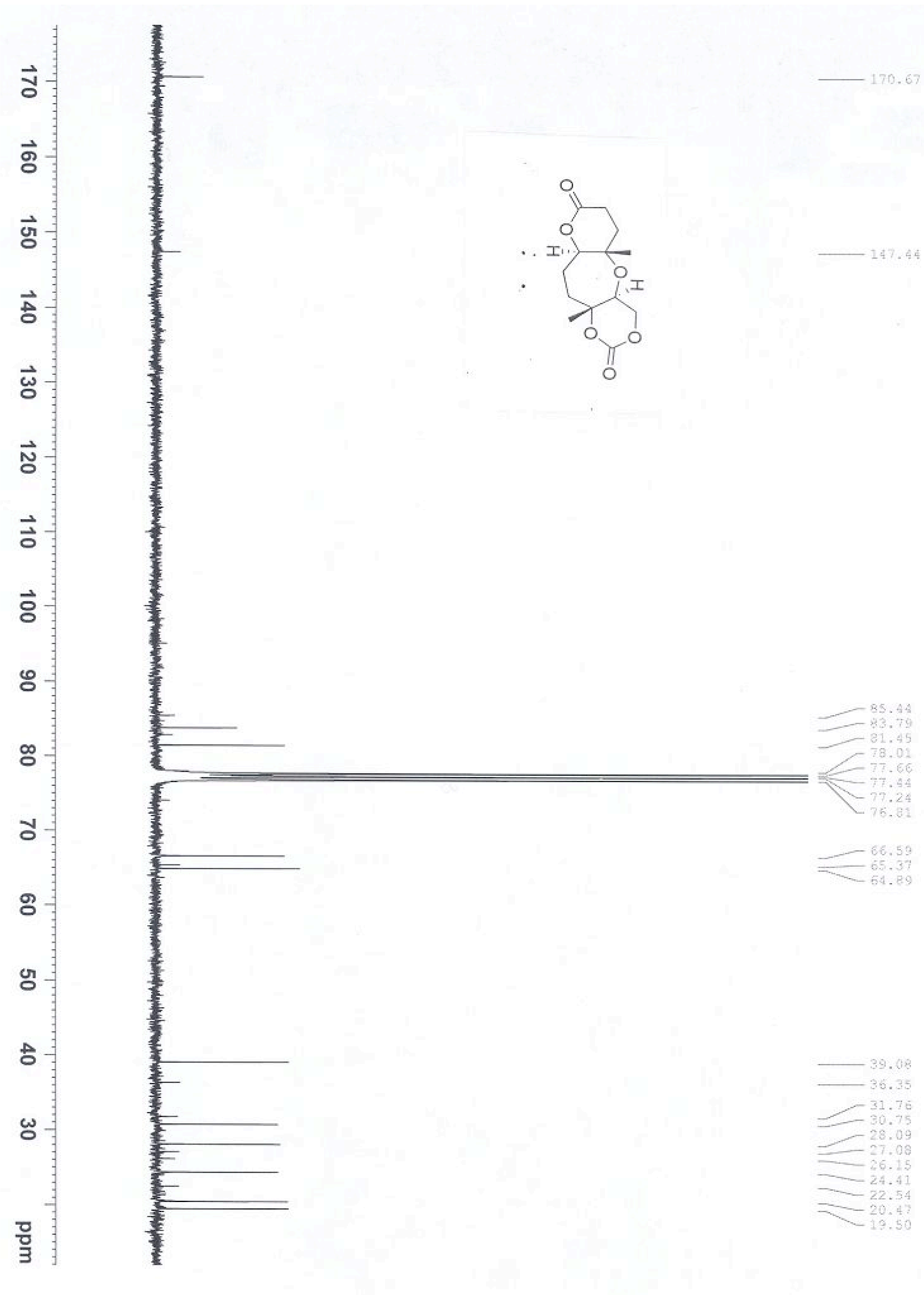
3.0819
2.9240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78.01
77.66
77.44
77.24
76.81
66.59
65.37
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31.76
30.75
28.09
27.08
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24.41
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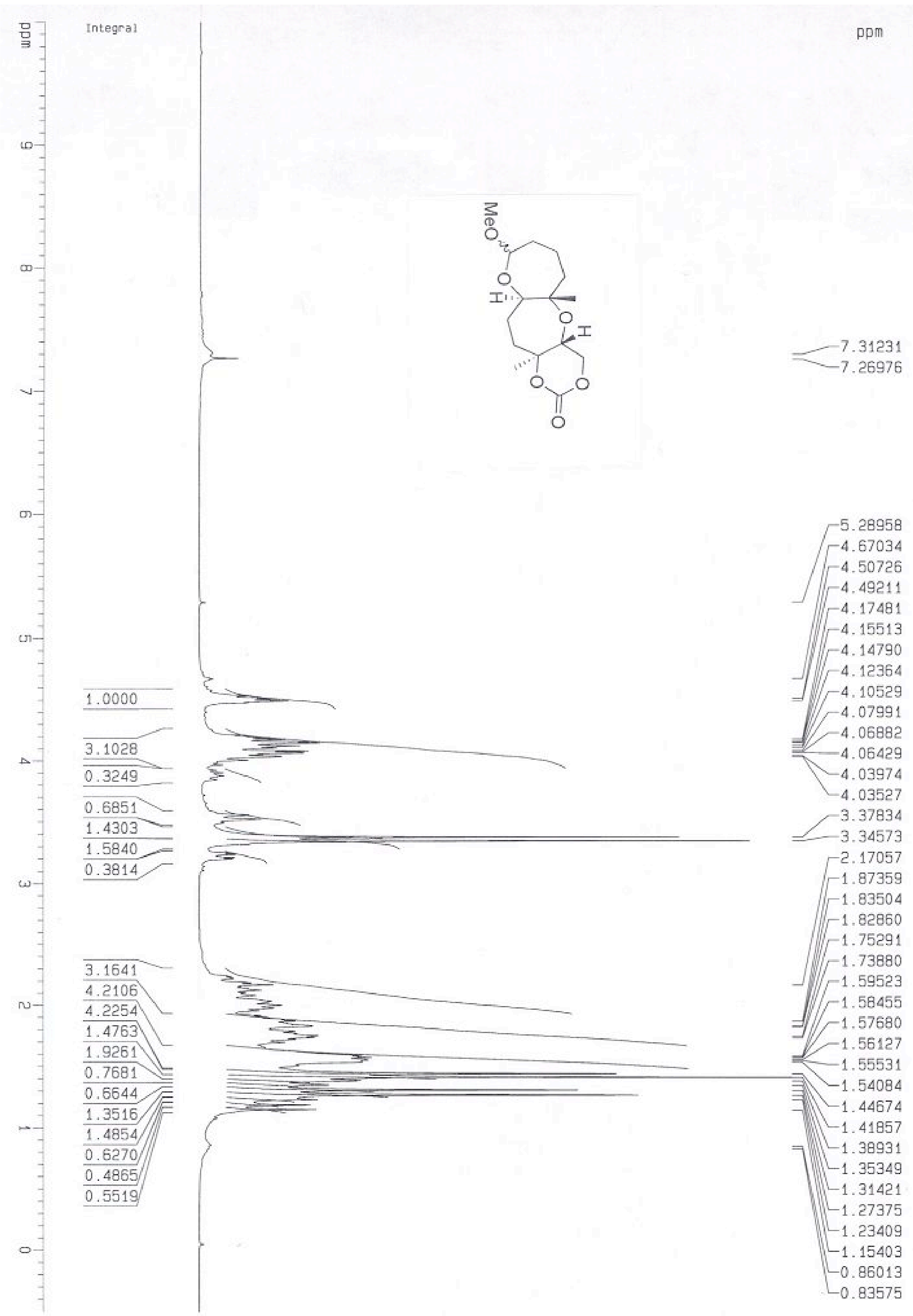
20.47
19.50
ppm


Integral
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0.6851
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3.1641
4.2106
4.2254
1.4763
1.9261
0.7681
0.6644
1.3516
1.4854
0.6270
0.4865
0.5519
7.31231
7.26976
5.28958
4.67034
4.50726
4.49211
4.17481
4.15513
4.14790
4.12364
4.10529
4.07991
4.06882
4.06429
4.03974
4.03527
3.37834
3.34573
2.17057
1.87359
1.83504
1.82860
1.75291
1.73880
1.59523
1.58455
1.57680
1.56127
1.55531
1.54084
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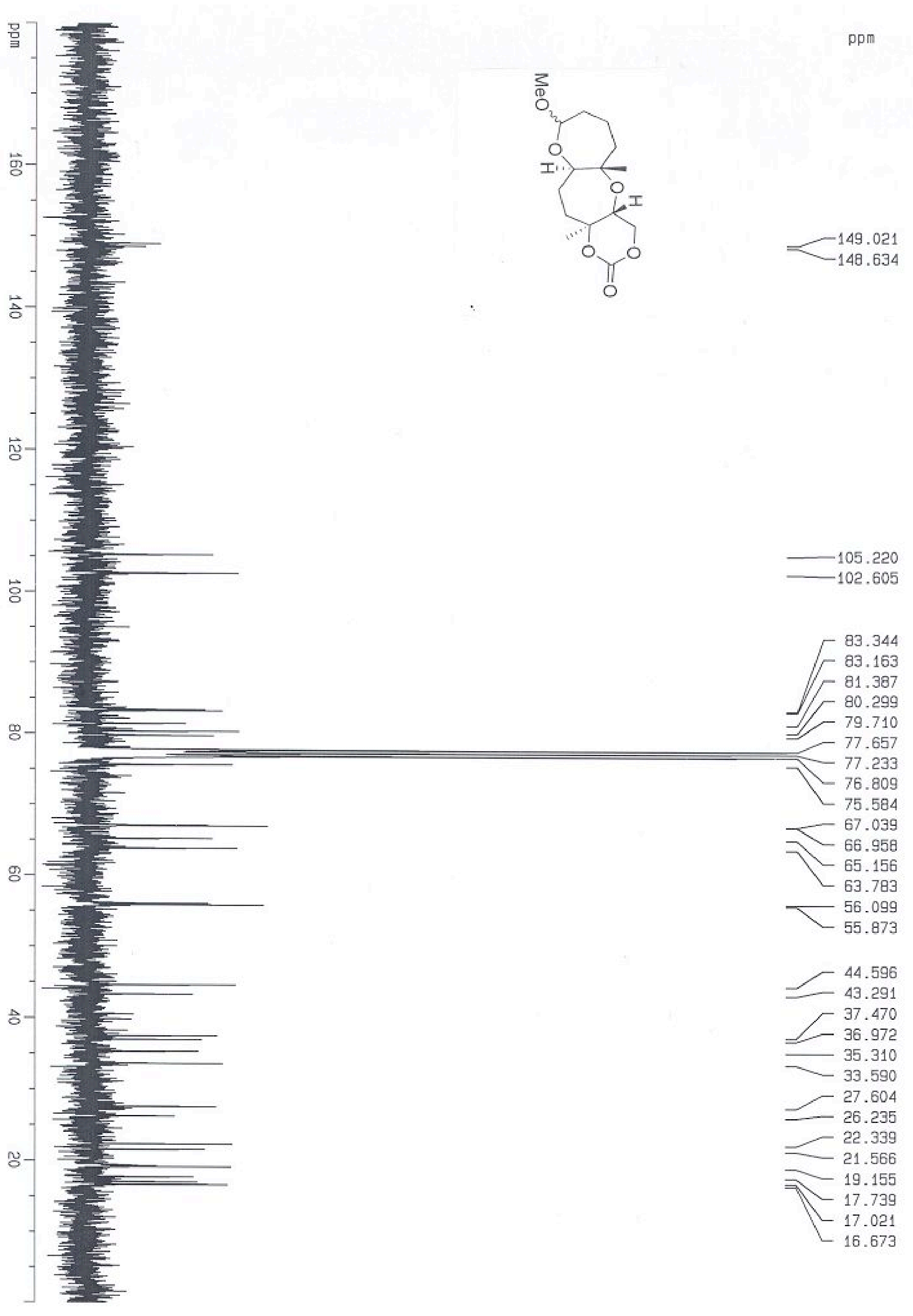
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81.387
80.299
79.710
77.657
77.233
76.809
75.584
67.039
66.958
65.156
63.783
56.099
55.873
44.596
43.291
37.470
36.972
35.310
33.590
27.604
26.235
22.339
21.566
19.155
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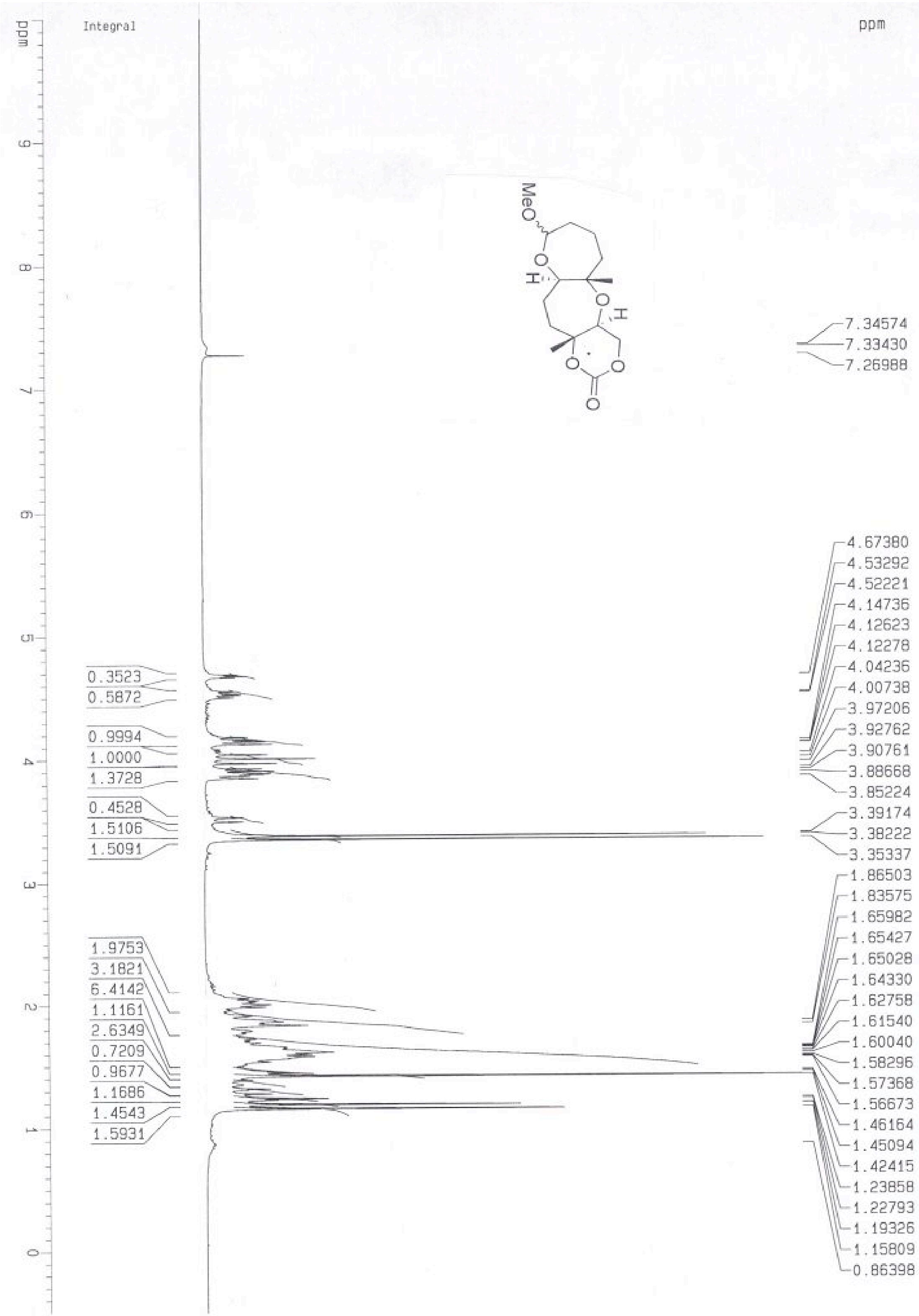
17.021
16.673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Integral
ppm
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4.14736
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4.12278
4.04236
4.00738
3.97206
3.92762
3.90761
3.88668
3.85224
3.39174
3.38222
3.35337
1.86503
1.83575
1.65982
1.65427
1.65028
1.64330
1.62758
1.61540
1.60040
1.58296
1.57368
1.56673
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1.45094
1.42415
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1.22793
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1.15809
0.86398
0.3523
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0.9994
1.0000
1.3728
0.4528
1.5106
1.5091
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6.4142
1.1161
2.6349
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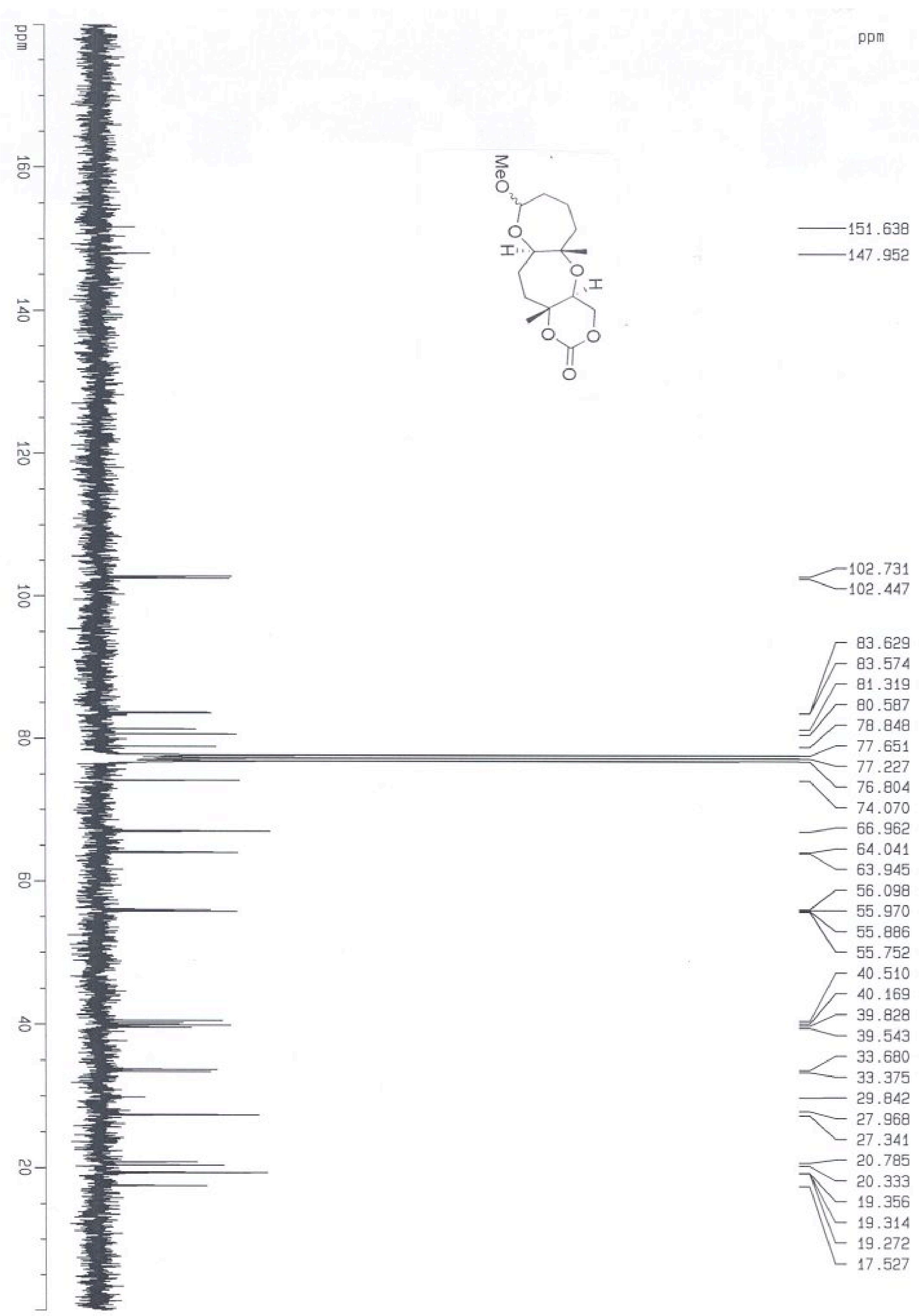
1.5931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77.227
76.804
74.070
66.962
64.041
63.945
56.098
55.970
55.886
55.752
40.510
40.169
39.828
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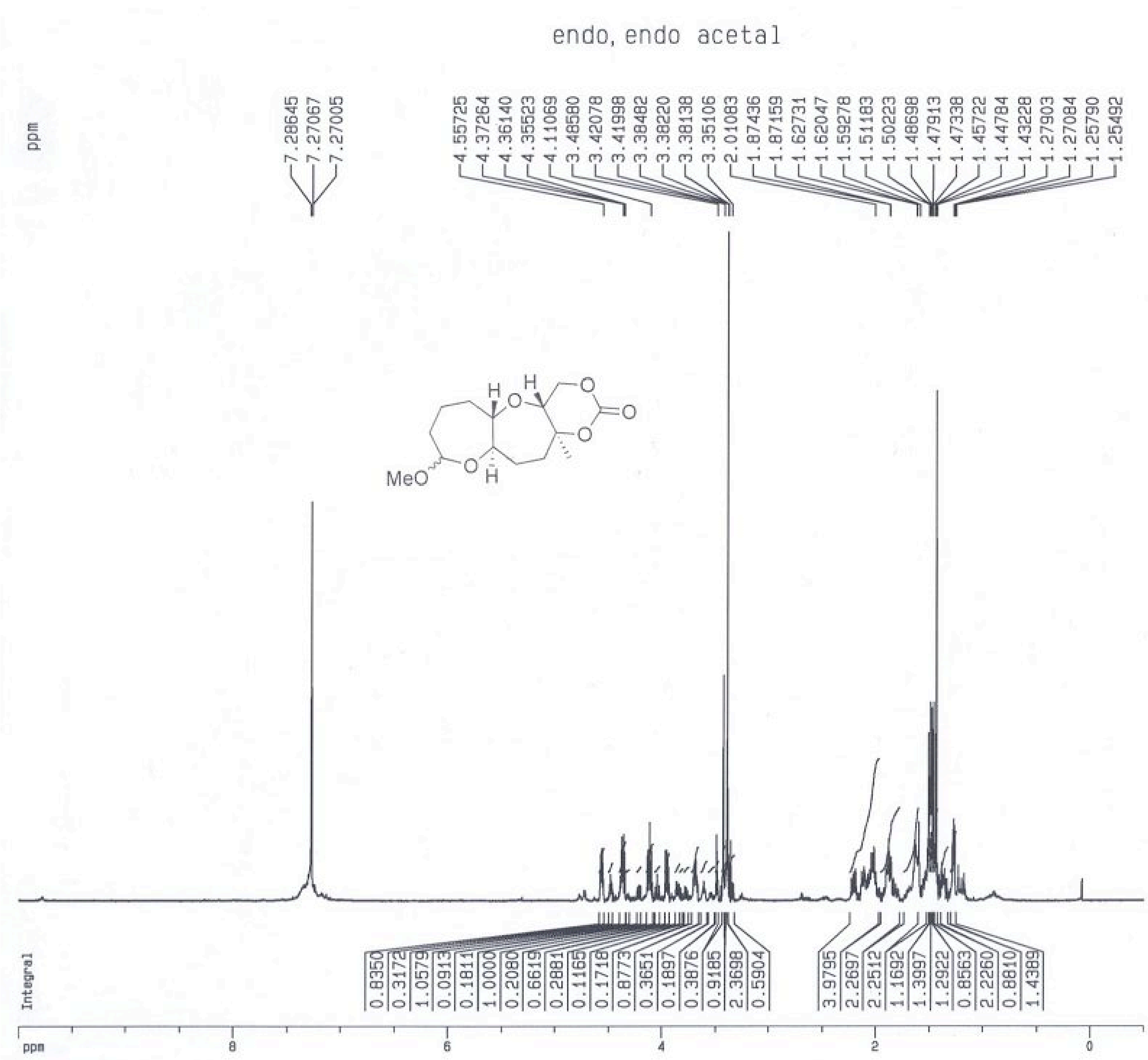
20.333
19.356
19.314
19.272
17.527



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EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
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Time	23.08
INSTRUM	spect
PROBHD	5 mm TBI 1H/
PULPROG	zg
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8992.806 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	114
DW	55.600 usec
DE	6.00 usec
TE	290.0 K
D1	2.00000000 sec
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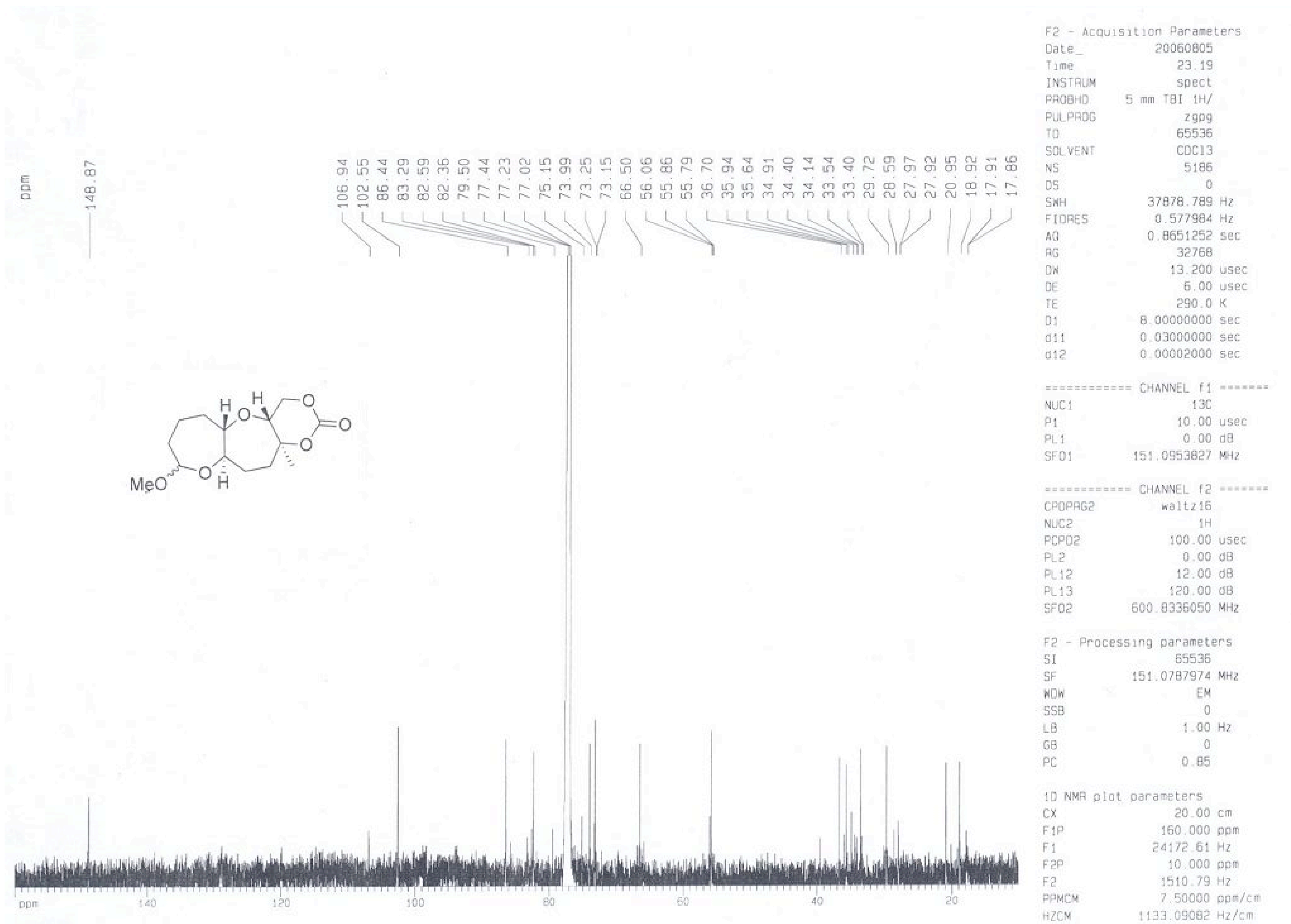
NUC1	1H
P1	3.00 usec
PL1	0.00 dB
SFO1	600.8336050 MHz
F2 - Processing parameters	
SI	65536
SF	600.8300261 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00
1D NMR plot parameters	
CX	20.00 cm
F1P	10.000 ppm
F1	6008.30 Hz
F2P	-0.500 ppm
F2	-300.42 Hz
PPMCM	0.52500 ppm/cm
HZCM	315.43576 Hz/cm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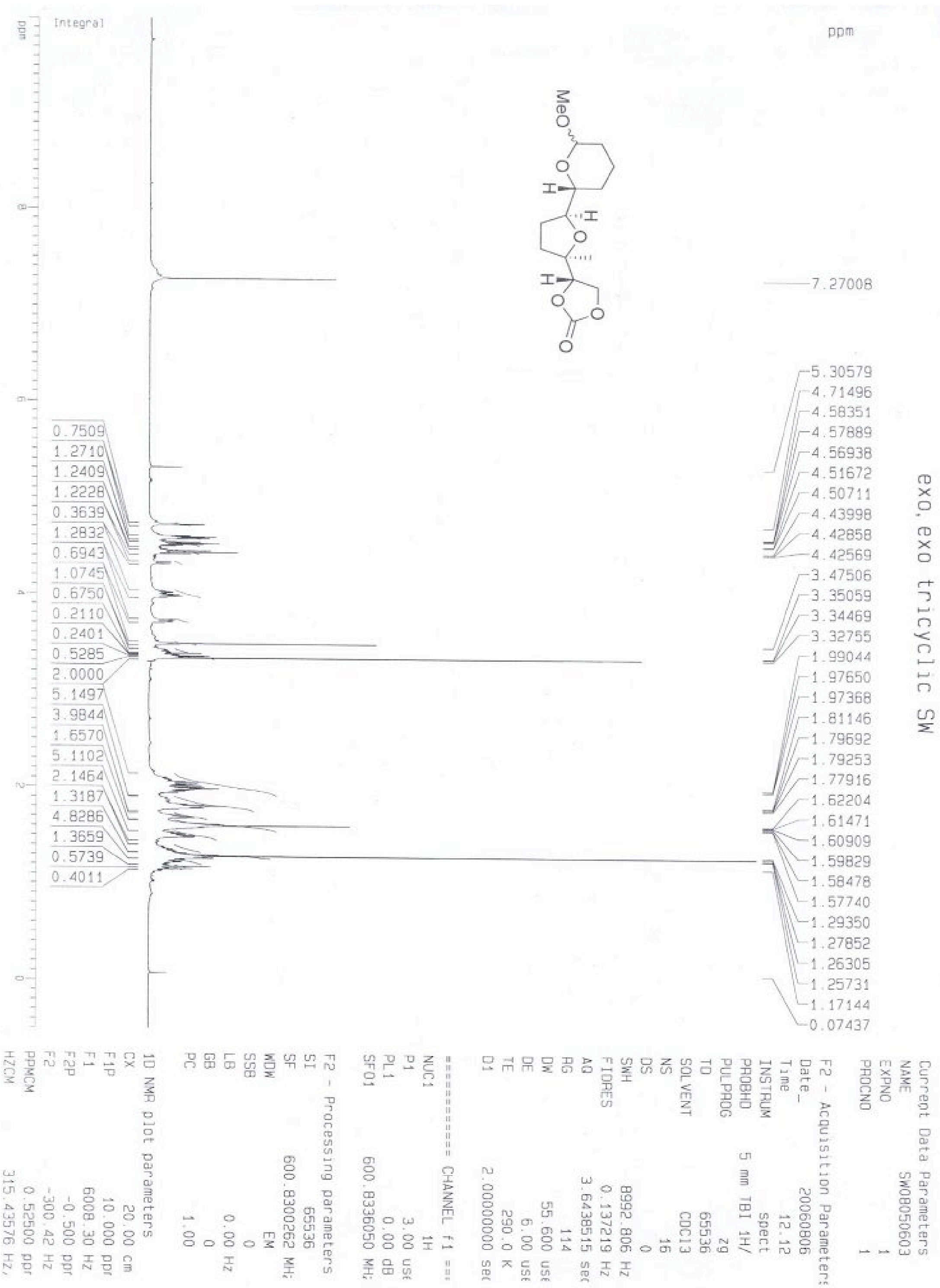
77.44
77.23
77.02
75.15
73.99
73.25
73.15
66.50
56.06
55.86
55.79
36.70
35.94
35.64
34.91
34.40
34.14
33.54
33.40
29.72
28.59
27.97
27.92
20.95
18.92
17.91
17.86
F2 - Acquisition Parameters
Date_ 20060805
Time 23.19
INSTRUM spect
PROBHD 5 mm TBI 1H/
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 5186
DS 0
SWH 37878.789 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 32768
DW 13.200 usec
DE 6.00 usec
TE 290.0 K
D1 8.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
============ CHANNEL f1 =======
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 151.0953827 MHz
============ CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 12.00 dB
PL13 120.00 dB
SFO2 600.8336050 MHz
F2 - Processing parameters
SI 65536
SF 151.0787974 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.85
1D NMR plot parameters
CX 20.00 cm
F1P 160.000 ppm
F1 24172.61 Hz
F2P 10.000 ppm
F2 1510.79 Hz
PPMCM 7.50000 ppm/cm
HZCM 1133.09082 Hz/cm
ppm 140 120 100 80 60 40 20



exo,exo tricyclic SW
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Integral
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H
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4.57889
4.56938
4.51672
4.50711
4.43998
4.42858
4.42569
3.47506
3.35059
3.34469
3.32755
1.99044
1.97650
1.97368
1.81146
1.79692
1.79253
1.77916
1.62204
1.61471
1.60909
1.59829
1.58478
1.57740
1.29350
1.27852
1.26305
1.25731
1.17144
0.07437
0.7509
1.2710
1.2409
1.2228
0.3639
1.2832
0.6943
1.0745
0.6750
0.2110
0.2401
0.5285
2.0000
5.1497
3.9844
1.6570
5.1102
2.1464
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4.8286
1.3659
0.5739
0.4011
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NAME SW0850603
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060806
Time 12.12
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PROBHD 5 mm TBI 1H/
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 114
DW 55.600 usec
DE 6.00 usec
TE 290.0 K
D1 2.0000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 0.00 dB
SFO1 600.8336050 MHz
F2 - Processing parameters
SI 65536
SF 600.8300262 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
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F1 6008.30 Hz
F2P -0.500 ppm
F2 -300.42 Hz
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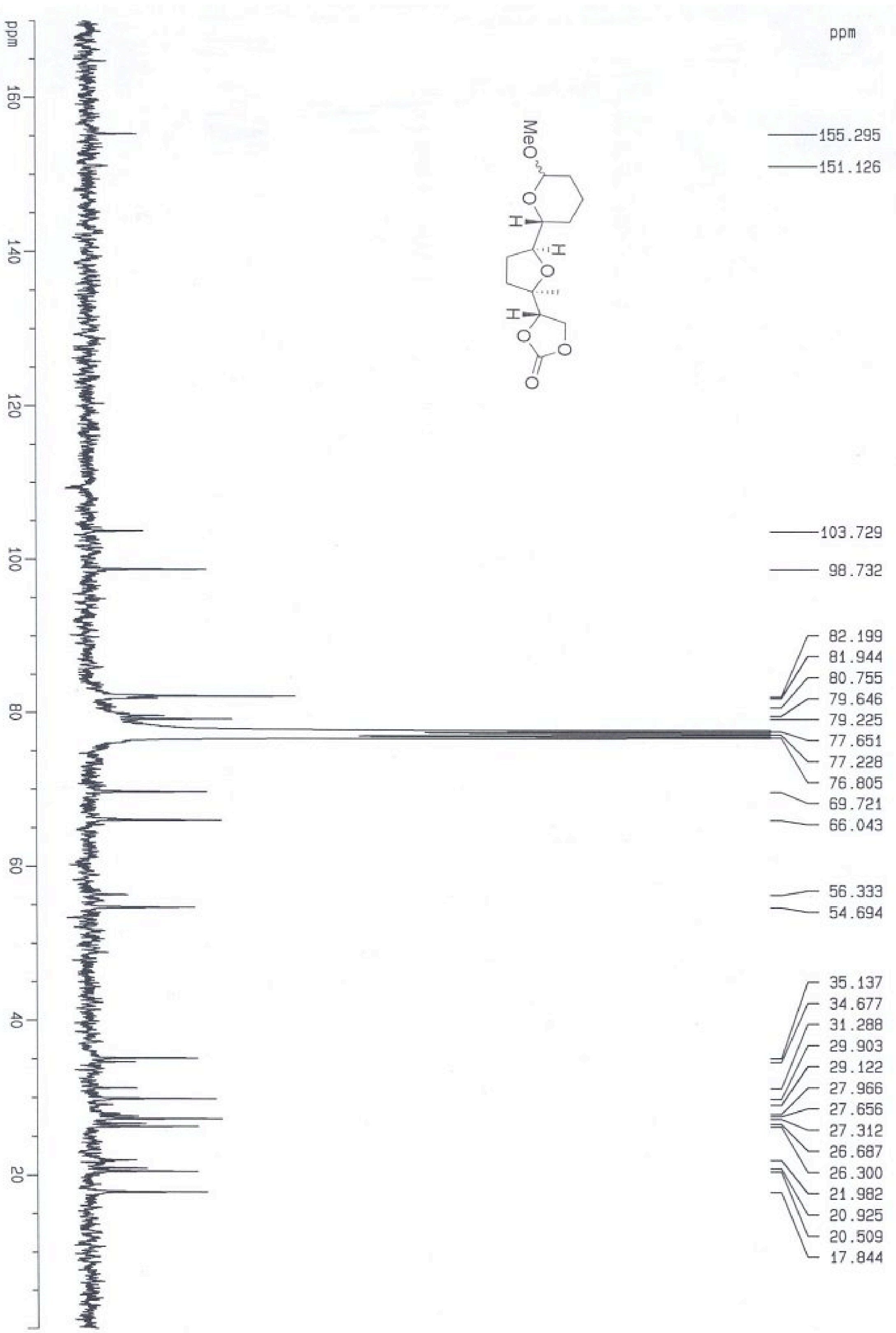
HZCM 315.43576 Hz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103.729
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82.199
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79.646
79.225
77.651
77.228
76.805
69.721
66.043
56.333
54.694
35.137
34.677
31.288
29.903
29.122
27.966
27.656
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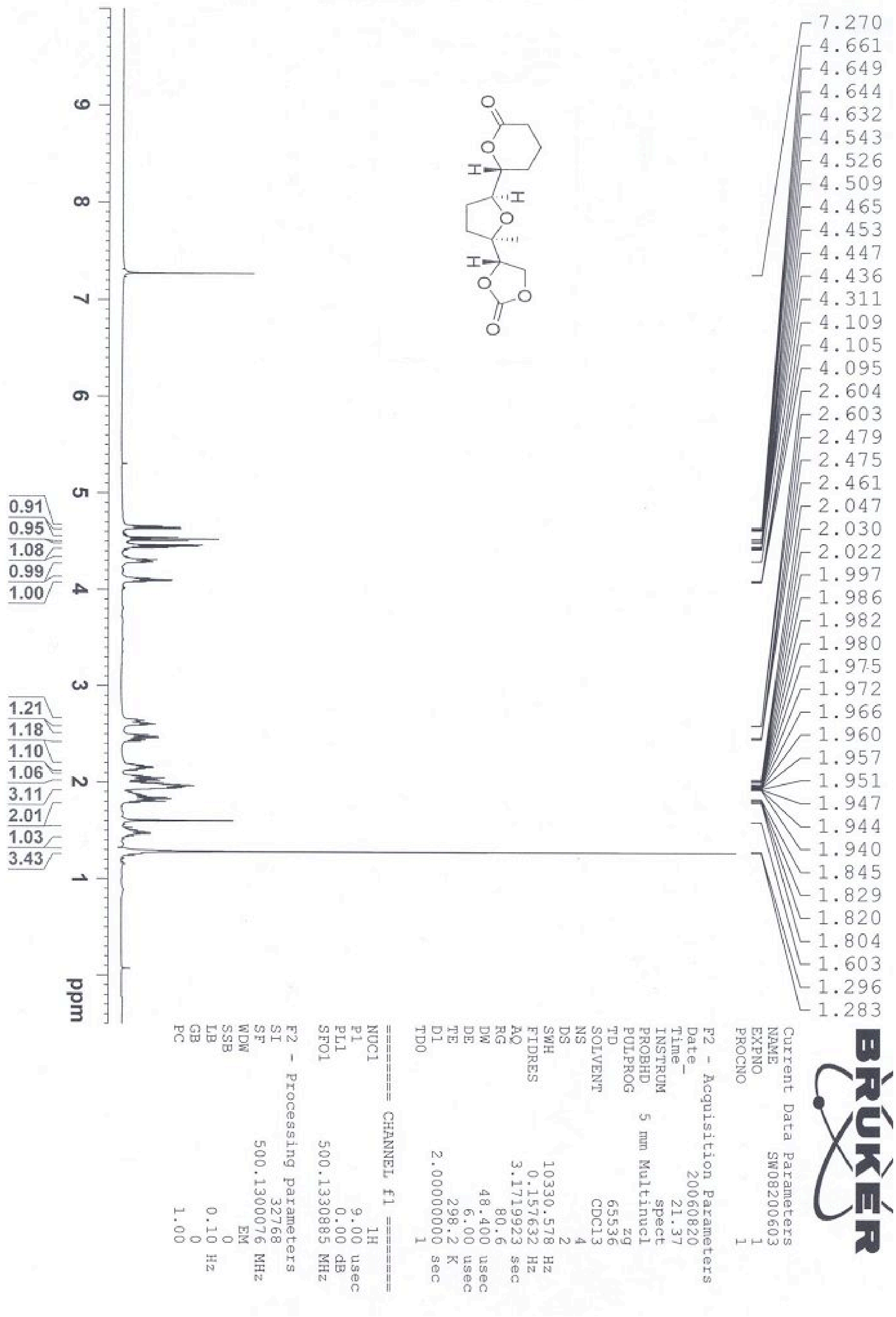
O
H
O
O
O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4.311
4.109
4.105
4.095
2.604
2.603
2.479
2.475
2.461
2.047
2.030
2.022
1.997
1.986
1.982
1.980
1.975
1.972
1.966
1.960
1.957
1.951
1.947
1.944
1.940
1.845
1.829
1.820
1.804
1.603
1.296
1.283
0.91
0.95
1.08
0.99
1.00
1.21
1.18
1.10
1.06
3.11
2.01
1.03
3.43
ppm
BRUKER
Current Data Parameters
NAME SW08200603
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060820
Time 21.37
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 80.6
DW 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.0000000 sec
TD0 1
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NUC1 1H
P1 9.00 usec
PL1 0.00 dB
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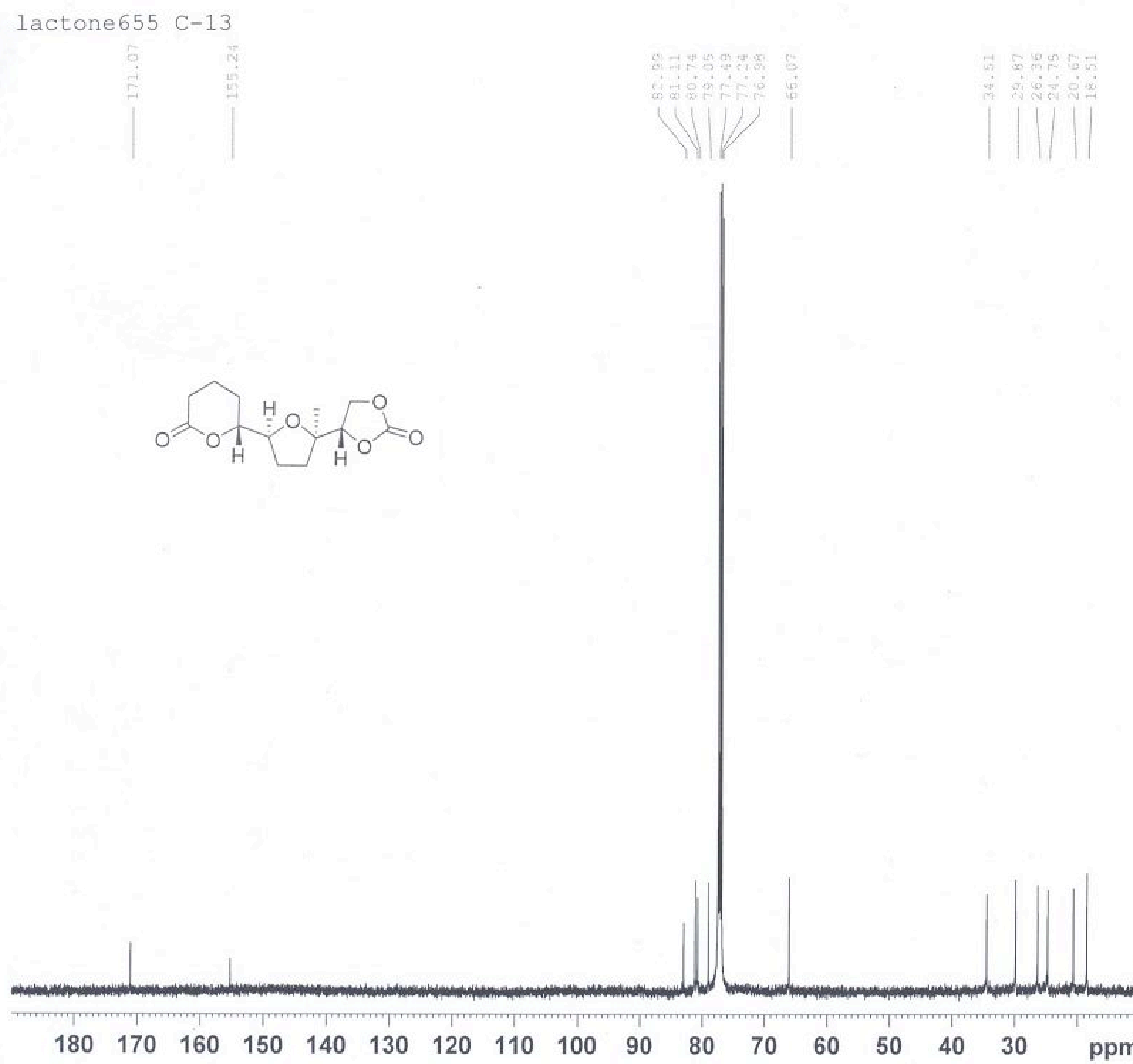
F2 - Processing parameters
SI 32768
SF 500.1300076 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



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NAME lactone655C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 14.13
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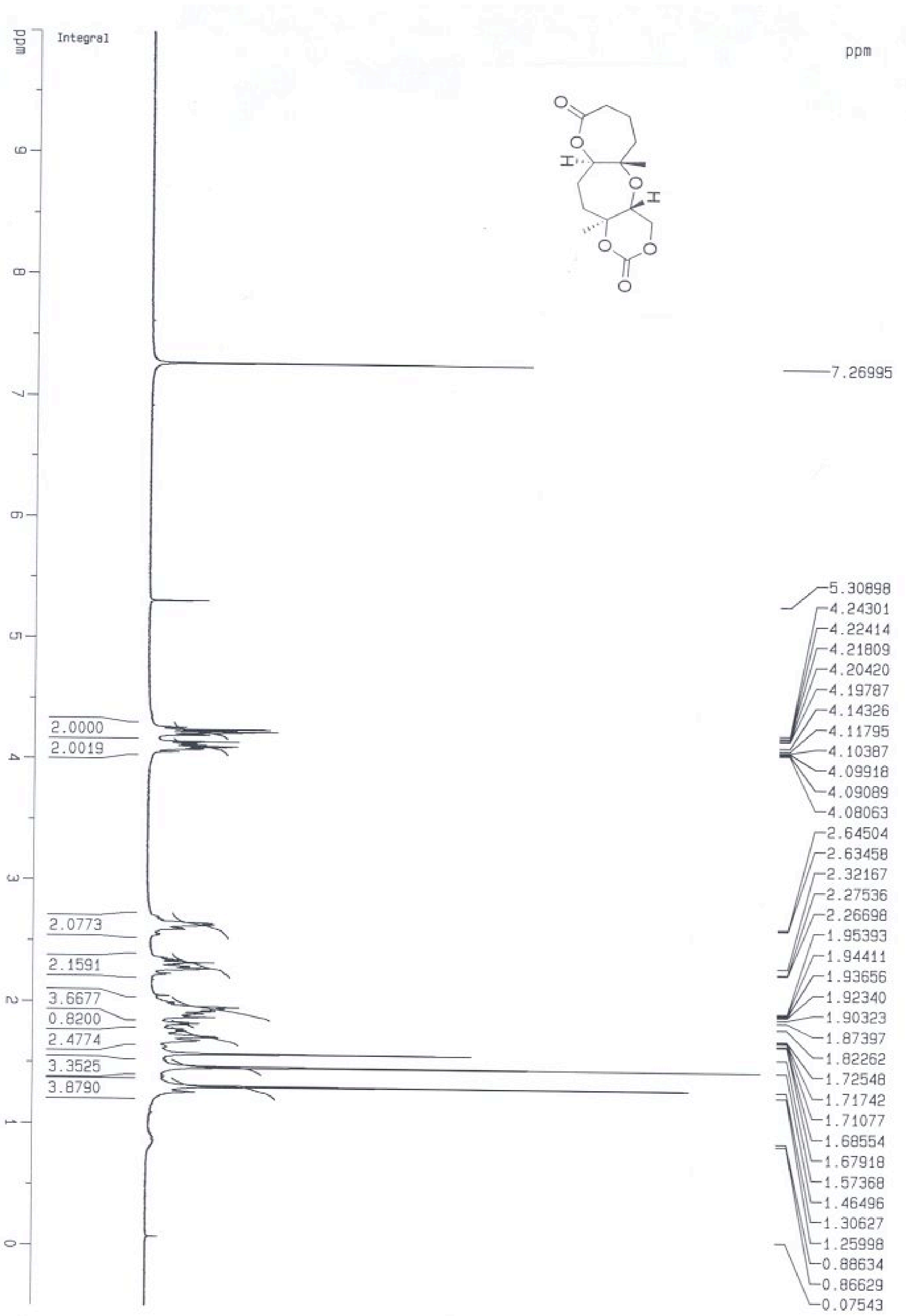
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 9400
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 8192
DW 16.650 usec
DE 6.00 usec
TE 298.2 K
D1 6.00000000 sec
d11 0.03000000 sec
DELTA 5.90000010 sec
TD0 1

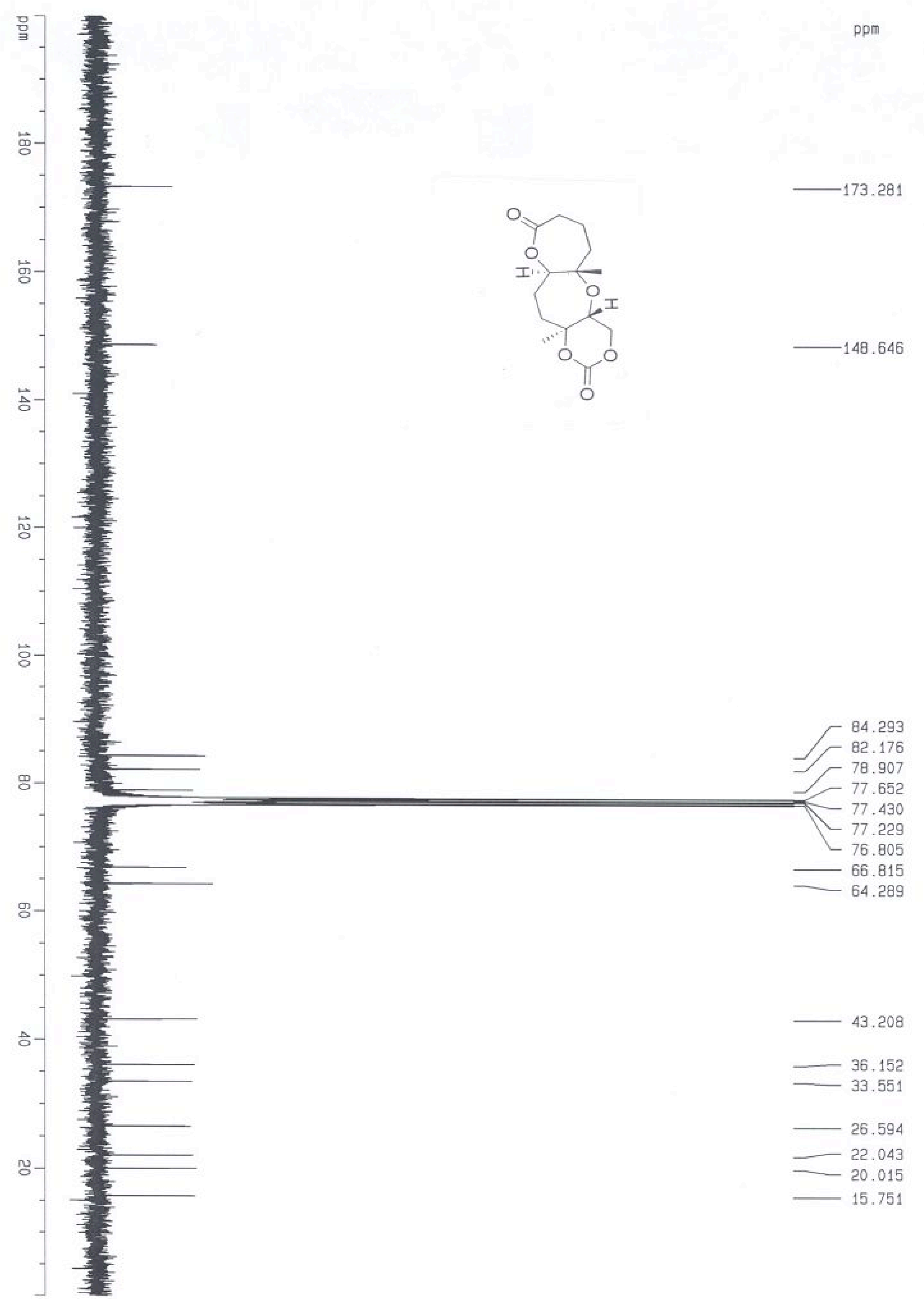
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -2.00 dB
SFO1 125.7703643 MHz

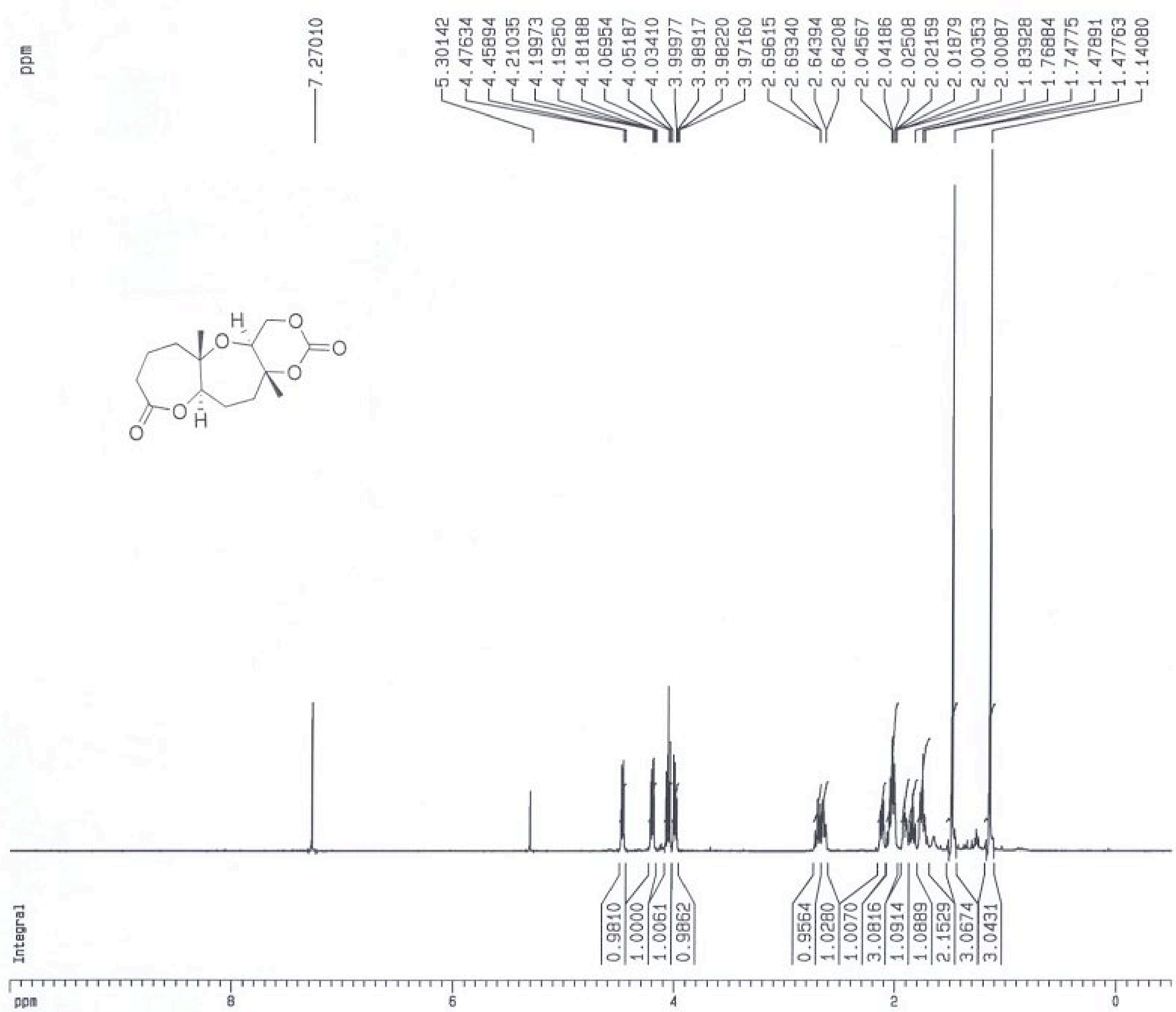
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 20.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577638 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

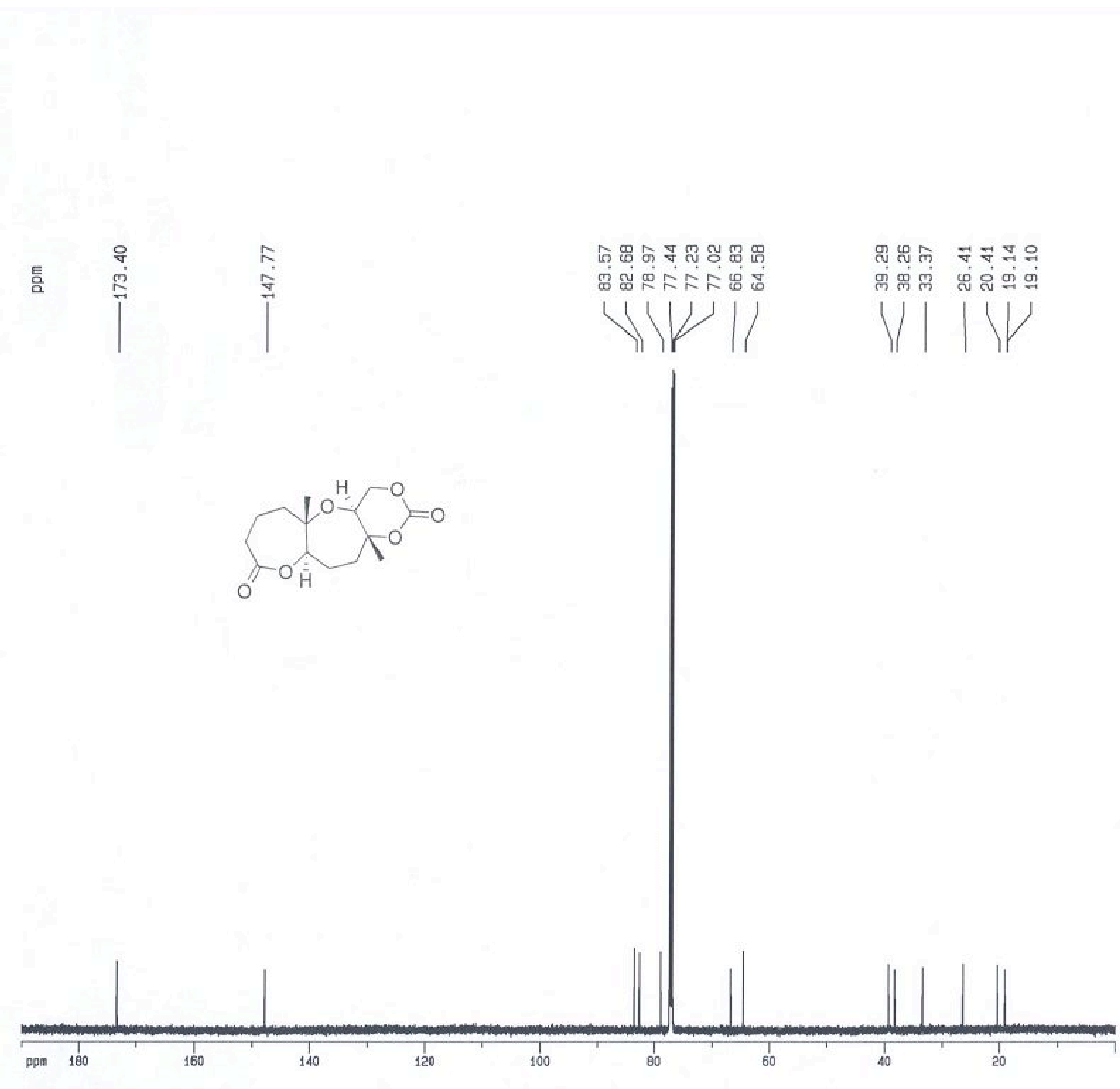


Integral
ppm
7.26995
5.30898
4.24301
4.22414
4.21809
4.20420
4.19787
4.14326
4.11795
4.10387
4.09918
4.09089
4.08063
2.64504
2.63458
2.32167
2.27536
2.26698
1.95393
1.94411
1.93656
1.92340
1.90323
1.87397
1.82262
1.72548
1.71742
1.71077
1.68554
1.67918
1.57368
1.46496
1.30627
1.25998
0.88634
0.86629
0.07543
2.0000
2.0019
2.0773
2.1591
3.6677
0.8200
2.4774
3.3525
3.8790


ppm
173.281
148.646
84.293
82.176
78.907
77.652
77.430
77.229
76.805
66.815
64.289
43.208
36.152
33.551
26.594
22.043
20.015
15.751



Current Data Parameters	
NAME	SW776H
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20060603
Time	13.13
INSTRUM	spect
PROBHD	5 mm TBI 1H/
PULPROG	zg
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8992.806 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	101.6
DW	55.600 usec
DE	6.00 usec
TE	290.0 K
D1	2.00000000 sec
============ CHANNEL f1 ========	
NUC1	1H
P1	3.00 usec
PL1	0.00 dB
SFO1	600.8336050 MHz
F2 - Processing parameters	
SI	65536
SF	600.8300265 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00
1D NMR plot parameters	
CX	20.00 cm
F1P	10.000 ppm
F1	6008.30 Hz
F2P	-0.500 ppm
F2	-300.42 Hz
PPMCM	0.52500 ppm/cm
HZCM	315.43576 Hz/cm



Current Data Parameters
NAME SW776C13
EXPNO 1
PROCNO 1

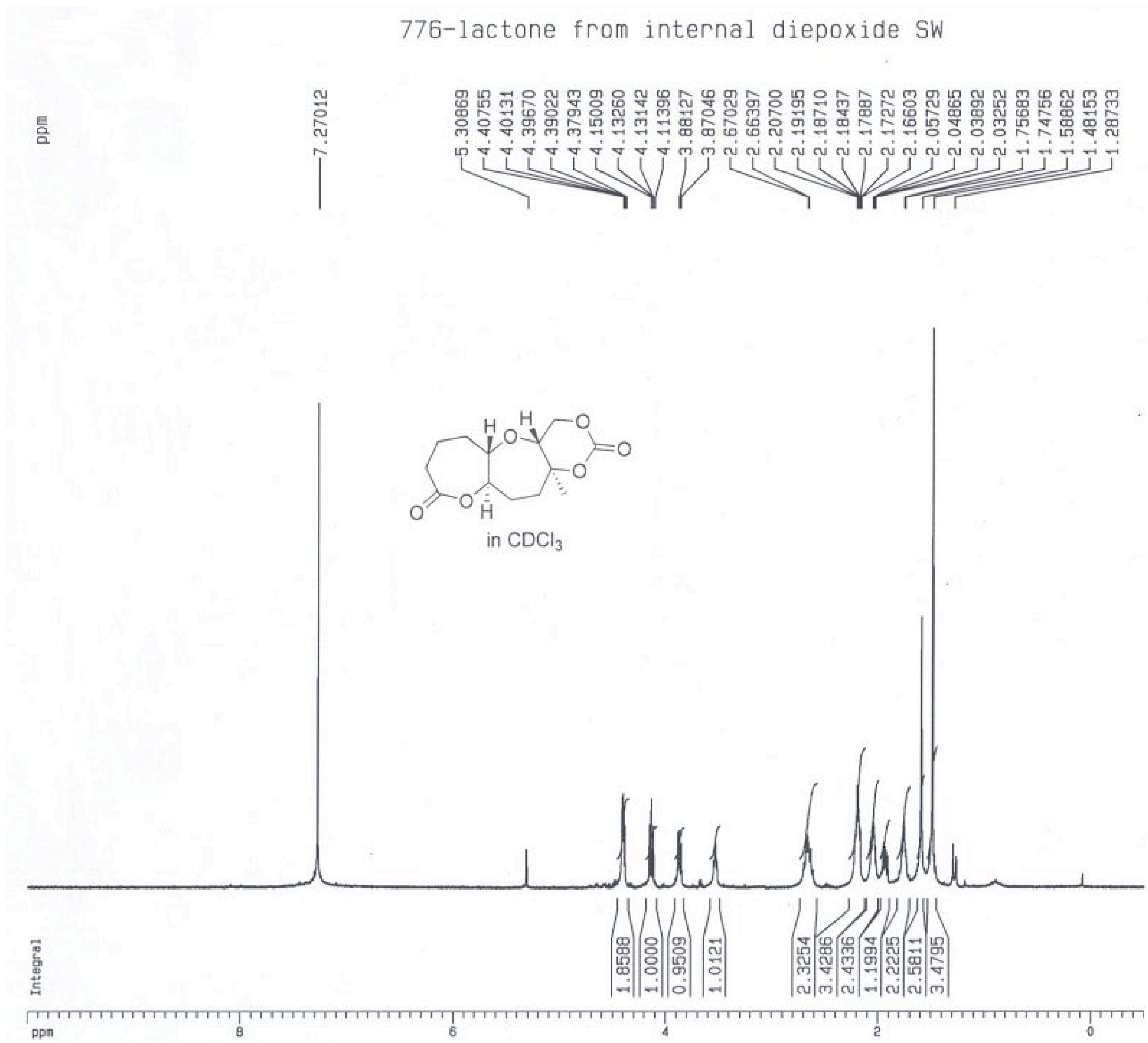
F2 - Acquisition Parameters
Date_ 20060603
Time 13.40
INSTRUM spect
PROBHD 5 mm TBI 1H/
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 779
DS 0
SWH 37878.789 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 32768
DW 13.200 usec
DE 6.00 usec
TE 290.0 K
D1 8.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

============ CHANNEL f1 ============
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 151.0953827 MHz

============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 12.00 dB
PL13 120.00 dB
SFO2 600.8336050 MHz

F2 - Processing parameters
SI 65536
SF 151.0788008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 190.000 ppm
F1 28704.97 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 1435.24854 Hz/cm



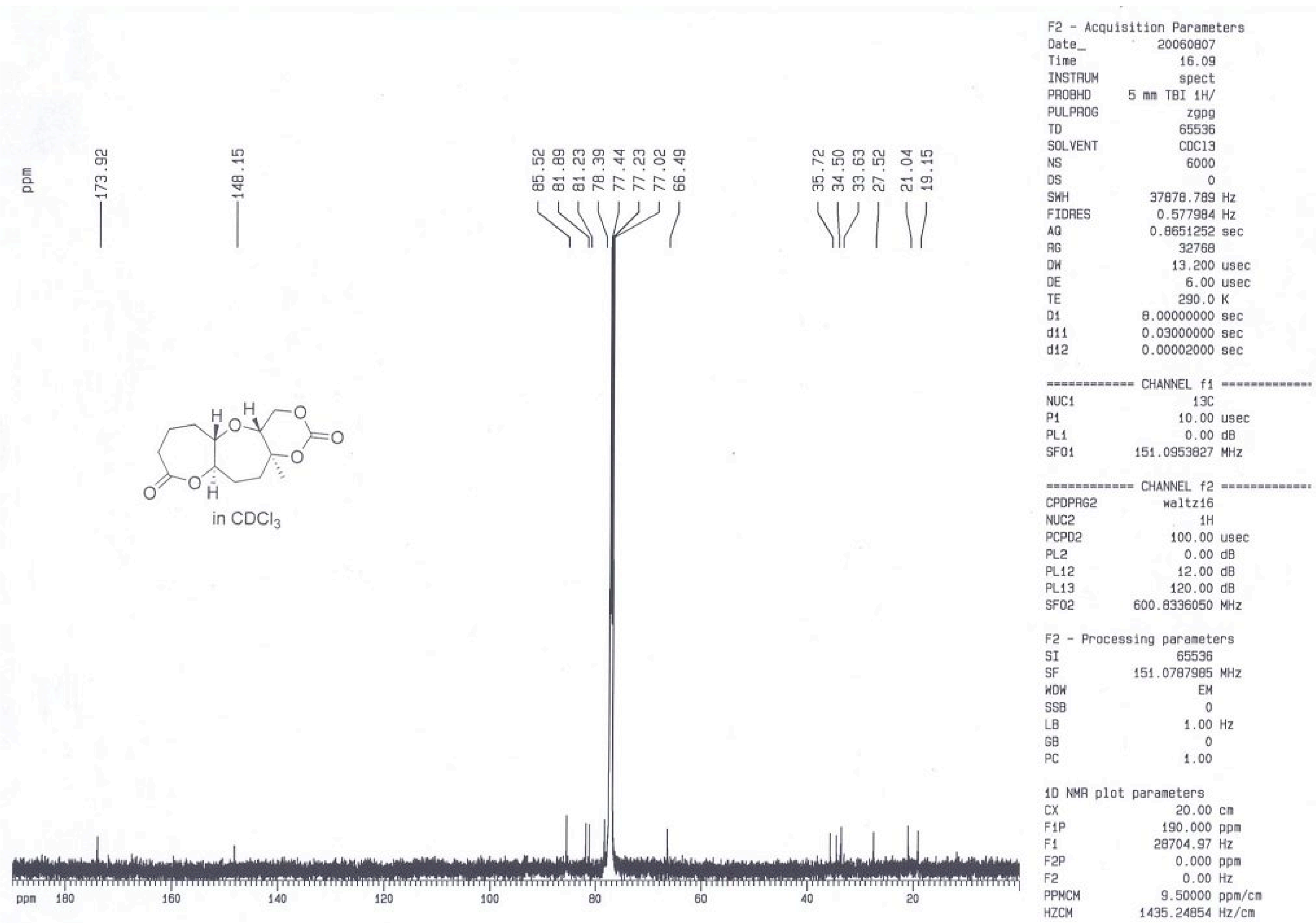
Current Data Parameters
NAME SW08070602
EXPNO 1
PROCNO 1

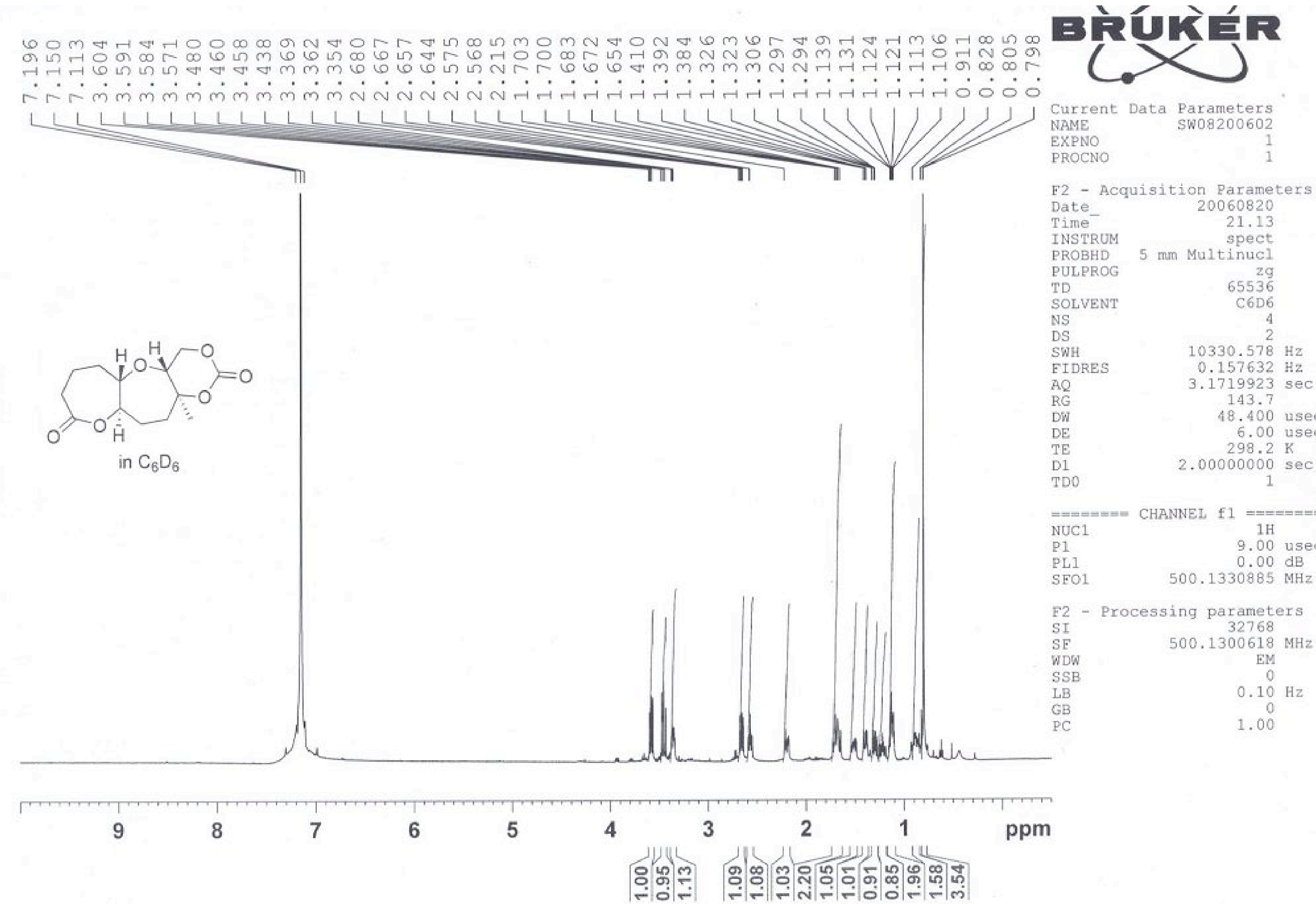
F2 - Acquisition Parameters
Date_ 20060807
Time 15.51
INSTRUM spect
PROBHD 5 mm TBI 1H/
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 322.5
DW 55.600 usec
DE 6.00 usec
TE 290.0 K
D1 2.00000000 sec

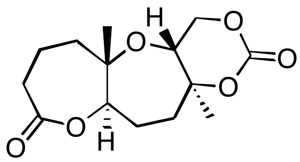
============ CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 0.00 dB
SFO1 600.8336050 MHz

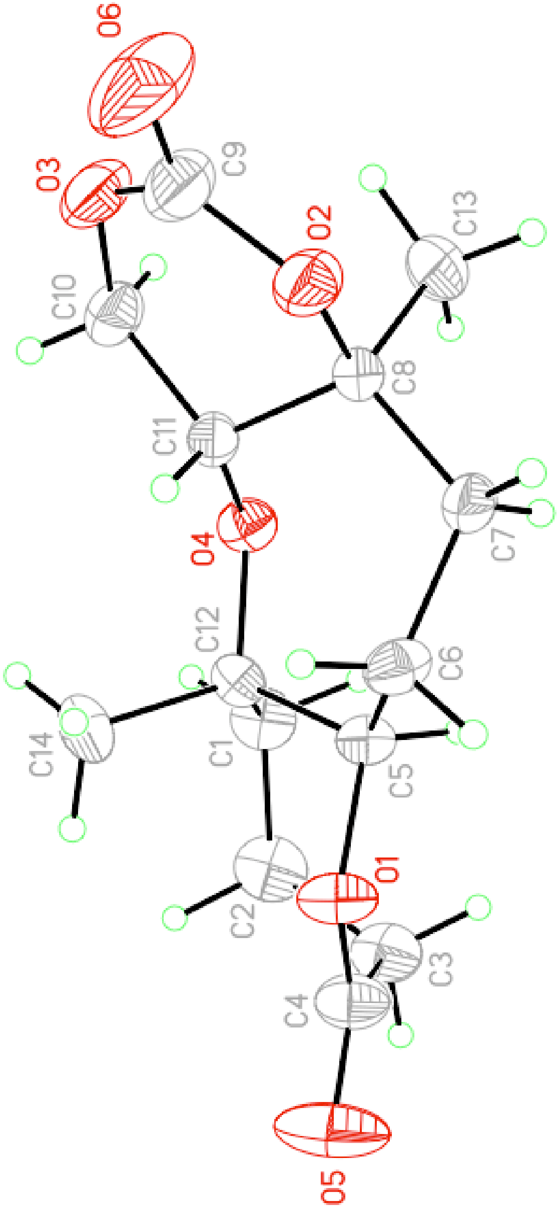
F2 - Processing parameters
SI 65536
SF 600.8300262 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

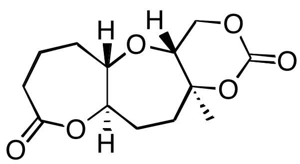
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 6008.30 Hz
F2P -0.500 ppm
F2 -300.42 Hz
PPMCM 0.52500 ppm/cm
HZCM 315.43576 Hz/cm

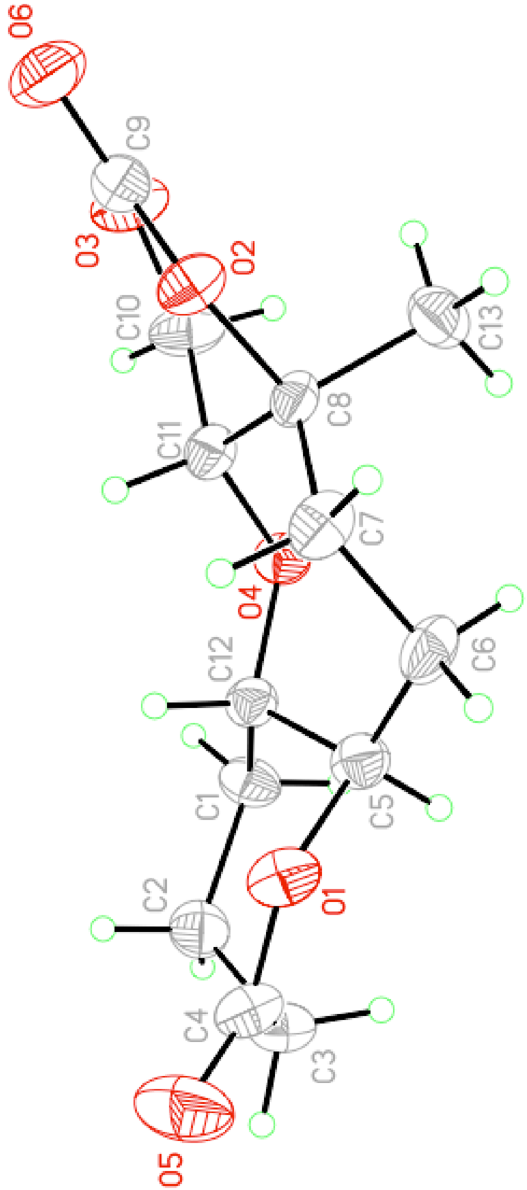

ppm
173.92
148.15
85.52
81.89
81.23
78.39
77.44
77.23
77.02
66.49
35.72
34.50
33.63
27.52
21.04
19.15
in CDCl3
ppm 180 160 140 120 100 80 60 40 20
F2 - Acquisition Parameters
Date_ 20060807
Time 16.09
INSTRUM spect
PROBHD 5 mm TBI 1H/
PULPROG zgpg
TD 65536
SOLVENT CDC13
NS 6000
DS 0
SWH 37878.789 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 32768
DW 13.200 usec
DE 6.00 usec
TE 290.0 K
D1 8.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
============ CHANNEL f1 ============
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 151.0953827 MHz
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 12.00 dB
PL13 120.00 dB
SFO2 600.8336050 MHz
F2 - Processing parameters
SI 65536
SF 151.0787985 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 190.000 ppm
F1 28704.97 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 1435.24854 Hz/cm


in C_6D_6
7.196
7.150
7.113
3.604
3.591
3.584
3.571
3.480
3.460
3.458
3.438
3.369
3.362
3.354
2.680
2.667
2.657
2.644
2.575
2.568
2.215
1.703
1.700
1.683
1.672
1.654
1.410
1.392
1.384
1.326
1.323
1.306
1.297
1.294
1.139
1.131
1.124
1.121
1.113
1.106
0.911
0.828
0.805
0.798
BRUKER
Current Data Parameters
NAME SW08200602
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060820
Time 21.13
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 65536
SOLVENT C6D6
NS 4
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 143.7
DW 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300618 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
0.95
1.13
1.09
1.08
1.03
2.20
1.05
1.01
0.91
0.85
1.96
1.58
3.54




O6
C9
O3
O2
C13
C10
C8
C11
C7
O4
C12
C6
C14
C1
C5
O1
C2
C3
C4
O5




O6
C9
O3
O2
C10
C13
C8
C11
C7
O4
C12
C6
C1
C5
O1
C2
C4
C3
O5