Stereoselective Synthesis of (-) Acanthoic Acid

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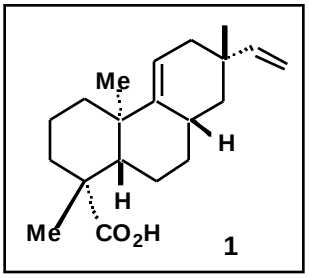
Supporting Information

General techniques. Organic solutions were concentrated by rotary evaporation below 45 °C at about 20 mmHg. All nonaqueous reactions were carried out using flame-dried glassware, under an argon atmosphere in dry, freshly distilled solvents under anhydrous conditions, unless otherwise noted. THF and Et_2O were distilled from sodium/benzophenone; CH_2Cl_2 and toluene from calcium hydride; and benzene from potassium. Pyridine, triethylamine and boron trifluoride etherate were distilled from calcium hydride prior to use. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous materials, unless otherwise stated. Reactions were monitored by thin-layer chromatography carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV light as visualizing agent and *p*-anisaldehyde solution and heat as developing agents. E. Merck silica gel (60, particle size 0.040-0.063 mm) was used for flash chromatography. Preparative thin-layer chromatography separations were carried out on 0.25 or 0.50 mm E. Merck silica gel plates (60F-254). NMR spectra were recorded on a Varian 400 MHz instrument and calibrated using residual undeuterated solvent as an internal reference. IR spectra were recorded on a Nicolet Avatar 320 FT-IR spectrometer. Optical rotations were recorded on a Perkin-Elmer 241 polarimeter. High resolution mass spectra (HRMS) were recorded on a VG 7070 HS mass spectrometer under chemical ionization (CI) conditions or on a VG ZAB-ZSE mass spectrometer under fast atom bombardment (FAB) conditions.



Acanthoic acid (1). To a solution of alkene **21** (16.8 mg, 0.05 mmol) in N, N-dimethylformamide (2 ml) added lithium bromide (5.0 mg, 0.06 mmol) and the mixture was refluxed at 160 °C for 1 hr. The reaction mixture was then cooled to 25 °C, diluted with H_2O (5 ml) and extracted with ethyl acetate (3 x 10 ml). The organic layer was collected, dried ($MgSO_4$) and concentrated and residue was chromatographed (silica, 15-20 % ethyl ether in hexane) to afford acanthoic acid (**1**) (14.9 mg, 0.05 mmol, 93%). **1:** white solid; R_f = 0.20 (silica, 30% ethyl ether in hexanes); $[\alpha]^{25}_D$: -26.0 (c= 0.33, benzene); IR (film) ν_{max} 3080.6, 2928.9, 2857.6, 1693.6, 1638.2, 1464.7, 1413.8, 1376.4, 1263.1, 1179.3, 1095.9, 1027.5, 999.2, 909.2, 801.7; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.82 (dd, 1H, J= 10.5, 17.5 Hz), 5.40 (m, 1H), 4.92 (d, 1H, J= 17.5 Hz), 4.86 (d, 1H, J= 10.5 Hz), 2.30 (bs, 1H), 2.16-1.2 (m, 14H), 1.24 (s, 3H), 1.00-1.10 (m, 2H), 0.99 (s, 3H), 0.95 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 185.0, 150.3, 149.9, 116.7, 109.2, 47.9, 41.8, 41.7, 38.3, 38.2, 37.4, 34.8, 31.8, 28.6, 28.5, 27.7, 22.4, 22.1, 20.3, 18.9; HRMS, calcd for $C_{20}H_{30}O_2$ ($M+Cs^+$) 435.1298, found 435.1302.

X-Ray data of compound **16**

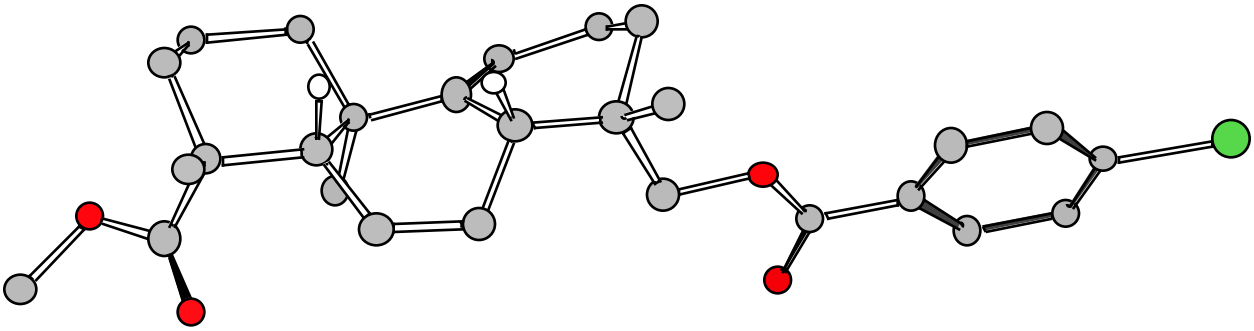


Table 1. Crystal data and structure refinement for TAOT

Empirical formula	$C_{27}\ H_{34}\ BrO_4$
Formula weight	502.45
Temperature	23 C
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 11.00(2)Å alpha = 90° b = 19.19(2)Å beta = 109.82(9)° c = 12.459(13)Å gamma = 90°
Crystal size	0.50 x 0.50 x 0.15 mm
Density (calculated)	1.349 Mg/m^3
Absorption coefficient	1.691 mm^{-1}
Volume, Z, F(000)	2474(5) $Å^3$, 4, 1052
θ range for data collection	1.74 to 22.50°
Limiting indices	$0 \le h \le 11$, $0 \le k \le 20$, $-13 \le l \le 12$
Reflections collected	3502, 1796 observed [I>2σ(I)]
Independent reflections	3318 (R_{int} = 0.1414)
Absorption correction	Semi-empirical from psi-scans
Max. and min. transmission	0.9479 and 0.7019
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3318 / 1 / 257
Goodness-of-fit on F^2	1.034
Final R indices [I>2σ(I)]	R1 = 0.1129, wR2 = 0.2667
R indices (all data)	R1 = 0.1942, wR2 = 0.3406
Absolute structure parameter	-0.08(4)
Largest diff. peak and hole	0.738 and -1.052 $eÅ^{-3}$
Scan speed, range, type	10°/minute, 0.7°, Wyckoff
Background range, % time	1.0°, 25% each side

Table 2. Atomic coordinates [x10^4] and equivalent isotropic displacement parameters [A^2 x 10^3] for TAOT U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	8405(13)	2734(6)	816(8)	74(9)
C(2)	8509(14)	3436(7)	1125(10)	53(6)
C(3)	8062(14)	3949(5)	289(13)	58(7)
C(4)	7512(15)	3759(6)	-855(11)	49(6)
C(5)	7408(12)	3057(6)	-1164(8)	58(7)
C(6)	7855(9)	2545(5)	-328(9)	47(6)
C(7)	7787(15)	1807(5)	-654(13)	35(5)
O(1)	7574(16)	1615(9)	-1648(14)	68(5)
O(2)	8029(15)	1378(9)	171(13)	57(4)
O(3)	8620(25)	-2925(12)	-1086(23)	122(9)
O(4)	7014(17)	-3555(10)	-1053(15)	71(5)
Br(1)	8205(3)	4885(1)	676(3)	83(1)
C(8)	8056(27)	632(13)	-75(22)	64(7)
C(9)	7978(23)	256(13)	936(19)	48(6)
C(10)	9131(27)	475(16)	2022(23)	79(8)
C(11)	6785(24)	503(15)	1267(22)	67(7)
C(12)	5619(32)	329(18)	293(28)	97(10)
C(13)	5581(25)	-427(13)	-155(21)	58(7)
C(14)	6614(23)	-821(13)	186(20)	55(6)
C(15)	7983(22)	-513(12)	858(20)	43(6)
C(16)	8907(20)	-826(11)	292(17)	39(5)
C(17)	9013(26)	-1614(15)	370(24)	72(8)
C(18)	7825(19)	-1976(11)	511(18)	37(5)
C(19)	6543(22)	-1576(13)	-165(19)	51(6)
C(20)	6270(26)	-1626(15)	-1451(21)	75(8)
C(21)	5405(25)	-1914(14)	129(24)	69(7)
C(22)	5349(23)	-2740(14)	-38(22)	69(7)
C(23)	6644(24)	-3062(16)	734(23)	72(8)
C(24)	7818(22)	-2806(13)	419(19)	51(6)
C(25)	8980(25)	-3051(16)	1302(23)	74(8)
C(26)	7873(23)	-3080(13)	-650(20)	47(6)
C(27)	7065(31)	-3908(21)	-2076(28)	102(11)
C(1')	7019(14)	3324(7)	4237(10)	75(9)
C(2')	6872(16)	2627(8)	3895(10)	80(9)
C(3')	7284(16)	2098(6)	4706(14)	62(7)
C(4')	7844(15)	2265(6)	5859(13)	51(6)
C(5')	7992(13)	2961(7)	6201(9)	72(8)
C(6')	7579(11)	3491(6)	5391(11)	49(6)
C(7')	7773(19)	4223(6)	5745(17)	73(8)
O(1')	8054(19)	4397(11)	6737(17)	85(6)
O(2')	7517(17)	4655(9)	4905(15)	69(5)
O(3')	8879(31)	8804(13)	6739(24)	165(14)
O(4')	7655(19)	9646(10)	5912(17)	79(6)
Br(2)	7085(4)	1170(1)	4275(3)	103(1)
C(8')	7776(30)	5400(15)	5187(25)	78(8)
C(9')	7786(28)	5743(16)	4082(24)	74(8)
C(10')	8943(29)	5517(17)	3808(26)	92(10)
C(11')	6569(31)	5576(20)	3156(29)	103(11)
C(12')	5407(34)	5792(19)	3318(31)	108(12)
C(13')	5492(31)	6514(17)	3927(26)	88(9)
C(14')	6641(24)	6877(14)	4277(20)	59(7)
C(15')	7783(29)	6565(17)	4337(27)	85(9)

C(16')	8908(29)	6792(16)	5391(25)	85(9)
C(17')	9066(31)	7615(18)	5372(26)	91(9)
C(18')	7929(22)	7946(12)	4603(21)	50(6)
C(19')	6632(24)	7669(14)	4632(21)	58(7)
C(20')	6519(27)	7689(15)	5817(22)	78(8)
C(21')	5492(28)	8042(15)	3764(25)	82(9)
C(22')	5500(33)	8865(19)	3898(32)	106(11)
C(23')	6827(28)	9093(18)	3837(27)	86(9)
C(24')	7959(29)	8787(17)	4673(26)	82(9)
C(25')	9205(23)	8992(14)	4436(21)	65(7)
C(26')	8155(30)	9058(18)	5868(28)	77(8)
C(27')	7931(27)	9970(20)	7016(25)	87(9)

Table 3. Bond lengths [Å] and angles [°] for TAOT

C(1)-C(6)	1.40
C(1)-C(2)	1.40
C(2)-C(3)	1.40
C(3)-C(4)	1.39
C(3)-Br(1)	1.854(10)
C(4)-C(5)	1.40
C(5)-C(6)	1.40
C(6)-C(7)	1.47
C(7)-O(1)	1.24(2)
C(7)-O(2)	1.27(2)
O(2)-C(8)	1.47(3)
O(3)-C(26)	1.17(3)
O(4)-C(26)	1.29(3)
O(4)-C(27)	1.46(4)
C(8)-C(9)	1.48(3)
C(9)-C(15)	1.48(3)
C(9)-C(10)	1.57(3)
C(9)-C(11)	1.58(4)
C(11)-C(12)	1.47(4)
C(12)-C(13)	1.55(4)
C(13)-C(14)	1.31(3)
C(14)-C(19)	1.51(3)
C(14)-C(15)	1.57(3)
C(15)-C(16)	1.54(3)
C(16)-C(17)	1.52(3)
C(17)-C(18)	1.54(3)
C(18)-C(19)	1.58(3)
C(18)-C(24)	1.60(3)
C(19)-C(20)	1.53(3)
C(19)-C(21)	1.56(3)
C(21)-C(22)	1.60(4)
C(22)-C(23)	1.55(3)
C(23)-C(24)	1.55(4)
C(24)-C(25)	1.45(3)
C(24)-C(26)	1.45(3)
C(1')-C(2')	1.40
C(1')-C(6')	1.40
C(2')-C(3')	1.40
C(3')-C(4')	1.39

C(3')-Br(2)	1.853(12)
C(4')-C(5')	1.39
C(5')-C(6')	1.40
C(6')-C(7')	1.47
C(7')-O(1')	1.21(2)
C(7')-O(2')	1.29(2)
O(2')-C(8')	1.48(3)
O(3')-C(26')	1.21(3)
O(4')-C(26')	1.26(4)
O(4')-C(27')	1.44(3)
C(8')-C(9')	1.53(4)
C(9')-C(11')	1.48(4)
C(9')-C(10')	1.49(4)
C(9')-C(15')	1.61(4)
C(11')-C(12')	1.42(4)
C(12')-C(13')	1.57(5)
C(13')-C(14')	1.38(4)
C(14')-C(15')	1.37(4)
C(14')-C(19')	1.58(4)
C(15')-C(16')	1.53(4)
C(16')-C(17')	1.59(4)
C(17')-C(18')	1.44(4)
C(18')-C(19')	1.53(3)
C(18')-C(24')	1.62(4)
C(19')-C(20')	1.52(4)
C(19')-C(21')	1.53(4)
C(21')-C(22')	1.59(5)
C(22')-C(23')	1.55(4)
C(23')-C(24')	1.45(4)
C(24')-C(26')	1.52(4)
C(24')-C(25')	1.55(4)
C(6)-C(1)-C(2)	120.0
C(3)-C(2)-C(1)	120.0
C(4)-C(3)-C(2)	120.0
C(4)-C(3)-Br(1)	119.2(8)
C(2)-C(3)-Br(1)	120.8(8)
C(3)-C(4)-C(5)	120.0
C(6)-C(5)-C(4)	120.0
C(5)-C(6)-C(1)	120.0
C(5)-C(6)-C(7)	120.1
C(1)-C(6)-C(7)	119.9
O(1)-C(7)-O(2)	122.2(14)
O(1)-C(7)-C(6)	122.7(12)
O(2)-C(7)-C(6)	115.0(10)
C(7)-O(2)-C(8)	118(2)
C(26)-O(4)-C(27)	115(2)
O(2)-C(8)-C(9)	107(2)
C(8)-C(9)-C(15)	115(2)
C(8)-C(9)-C(10)	110(2)
C(15)-C(9)-C(10)	108(2)
C(8)-C(9)-C(11)	112(2)
C(15)-C(9)-C(11)	110(2)
C(10)-C(9)-C(11)	101(2)
C(12)-C(11)-C(9)	107(2)
C(11)-C(12)-C(13)	115(3)
C(14)-C(13)-C(12)	121(3)
C(13)-C(14)-C(19)	120(2)
C(13)-C(14)-C(15)	122(2)

C(19)-C(14)-C(15)	118(2)
C(9)-C(15)-C(16)	116(2)
C(9)-C(15)-C(14)	113(2)
C(16)-C(15)-C(14)	106(2)
C(17)-C(16)-C(15)	114(2)
C(16)-C(17)-C(18)	114(2)
C(17)-C(18)-C(19)	111(2)
C(17)-C(18)-C(24)	115(2)
C(19)-C(18)-C(24)	118(2)
C(14)-C(19)-C(20)	110(2)
C(14)-C(19)-C(21)	107(2)
C(20)-C(19)-C(21)	109(2)
C(14)-C(19)-C(18)	111(2)
C(20)-C(19)-C(18)	111(2)
C(21)-C(19)-C(18)	108(2)
C(19)-C(21)-C(22)	112(2)
C(23)-C(22)-C(21)	109(2)
C(24)-C(23)-C(22)	113(2)
C(25)-C(24)-C(26)	106(2)
C(25)-C(24)-C(23)	108(2)
C(26)-C(24)-C(23)	115(2)
C(25)-C(24)-C(18)	107(2)
C(26)-C(24)-C(18)	115(2)
C(23)-C(24)-C(18)	106(2)
O(3)-C(26)-O(4)	123(3)
O(3)-C(26)-C(24)	126(2)
O(4)-C(26)-C(24)	111(2)
C(2')-C(1')-C(6')	120.0
C(1')-C(2')-C(3')	120.0
C(4')-C(3')-C(2')	120.0
C(4')-C(3')-Br(2)	119.2(9)
C(2')-C(3')-Br(2)	120.8(9)
C(3')-C(4')-C(5')	120.0
C(4')-C(5')-C(6')	120.0
C(5')-C(6')-C(1')	120.0
C(5')-C(6')-C(7')	120.1
C(1')-C(6')-C(7')	119.9
O(1')-C(7')-O(2')	124(2)
O(1')-C(7')-C(6')	122.0(14)
O(2')-C(7')-C(6')	113.7(11)
C(7')-O(2')-C(8')	117(2)
C(26')-O(4')-C(27')	118(3)
O(2')-C(8')-C(9')	105(2)
C(11')-C(9')-C(10')	112(3)
C(11')-C(9')-C(8')	109(3)
C(10')-C(9')-C(8')	111(3)
C(11')-C(9')-C(15')	108(3)
C(10')-C(9')-C(15')	113(3)
C(8')-C(9')-C(15')	104(3)
C(12')-C(11')-C(9')	117(3)
C(11')-C(12')-C(13')	115(3)
C(14')-C(13')-C(12')	120(3)
C(15')-C(14')-C(13')	121(3)
C(15')-C(14')-C(19')	120(3)
C(13')-C(14')-C(19')	119(3)
C(14')-C(15')-C(16')	112(3)
C(14')-C(15')-C(9')	119(3)
C(16')-C(15')-C(9')	113(3)

C(15')-C(16')-C(17')	109(3)
C(18')-C(17')-C(16')	112(3)
C(17')-C(18')-C(19')	116(2)
C(17')-C(18')-C(24')	114(2)
C(19')-C(18')-C(24')	110(2)
C(20')-C(19')-C(21')	111(2)
C(20')-C(19')-C(18')	113(2)
C(21')-C(19')-C(18')	112(2)
C(20')-C(19')-C(14')	108(2)
C(21')-C(19')-C(14')	109(2)
C(18')-C(19')-C(14')	103(2)
C(19')-C(21')-C(22')	115(3)
C(23')-C(22')-C(21')	104(3)
C(24')-C(23')-C(22')	116(3)
C(23')-C(24')-C(26')	112(3)
C(23')-C(24')-C(25')	111(3)
C(26')-C(24')-C(25')	105(2)
C(23')-C(24')-C(18')	112(3)
C(26')-C(24')-C(18')	113(3)
C(25')-C(24')-C(18')	104(2)
O(3')-C(26')-O(4')	119(3)
O(3')-C(26')-C(24')	125(3)
O(4')-C(26')-C(24')	115(3)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for TAOT&N. The factor exponent takes the form: $-2\pi^2[(ha^*)^2U_{11}+...+ 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
O(3)	151(20)	80(16)	179(23)	-42(16)	115(19)	-29(15)
O(4)	80(12)	51(13)	82(12)	-28(10)	29(10)	-17(9)
Br(1)	85(2)	32(2)	132(3)	-13(2)	37(2)	0(2)
O(3')	193(28)	78(17)	143(23)	-35(16)	-49(21)	40(18)
O(4')	99(15)	47(13)	96(15)	7(10)	39(12)	21(10)
Br(2)	132(3)	39(2)	134(3)	-19(2)	39(2)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for TAOT

	x	y	z	U(eq)
H(1)	8713(19)	2382(7)	1391(10)	88
H(2)	8887(20)	3567(9)	1912(10)	64
H(4)	7204(21)	4112(7)	-1431(14)	58
H(5)	7030(17)	2927(8)	-1952(8)	69
H(8C)	8850(27)	514(13)	-209(22)	77
H(8D)	7331(27)	510(13)	-747(22)	77
H(10D)	9074(27)	229(16)	2675(23)	119
H(10E)	9093(27)	968(16)	2142(23)	119
H(10F)	9933(27)	363(16)	1916(23)	119
H(11C)	6773(24)	265(15)	1950(22)	80
H(11D)	6829(24)	1001(15)	1409(22)	80
H(12C)	5557(32)	649(18)	-327(28)	116
H(12D)	4866(32)	403(18)	518(28)	116
H(13B)	4818(25)	-602(13)	-670(21)	69

H(15B)	8242(22)	-696(12)	1638(20)	51
H(16C)	9359(20)	-556(11)	-66(17)	46
H(17B)	9772(26)	-1736(15)	1013(24)	87
H(17C)	9138(26)	-1790(15)	-315(24)	87
H(18B)	7925(19)	-1892(11)	1312(18)	44
H(20D)	6224(26)	-2107(15)	-1672(21)	112
H(20E)	6952(26)	-1400(15)	-1637(21)	112
H(20F)	5463(26)	-1401(15)	-1850(21)	112
H(21C)	4597(25)	-1712(14)	-355(24)	83
H(21D)	5502(25)	-1807(14)	915(24)	83
H(22C)	4639(23)	-2930(14)	164(22)	83
H(22D)	5206(23)	-2852(14)	-830(22)	83
H(23C)	6779(24)	-2942(16)	1523(23)	86
H(23D)	6589(24)	-3565(16)	668(23)	86
H(25D)	8990(25)	-2884(16)	2031(23)	110
H(25E)	9727(25)	-2880(16)	1152(23)	110
H(25F)	8991(25)	-3551(16)	1306(23)	110
H(27D)	6392(31)	-4251(21)	-2313(28)	152
H(27E)	7889(31)	-4131(21)	-1911(28)	152
H(27F)	6947(31)	-3573(21)	-2675(28)	152
H(1')	6736(20)	3688(9)	3680(12)	90
H(2')	6486(23)	2512(11)	3102(11)	96
H(4')	8128(22)	1901(8)	6417(16)	61
H(5')	8377(19)	3077(10)	6995(9)	86
H(8A)	8603(30)	5461(15)	5789(25)	93
H(8B)	7105(30)	5601(15)	5434(25)	93
H(10A)	9711(29)	5635(17)	4430(26)	138
H(10B)	8952(29)	5749(17)	3128(26)	138
H(10C)	8911(29)	5022(17)	3690(26)	138
H(11A)	6600(31)	5788(20)	2459(29)	124
H(11B)	6530(31)	5076(20)	3043(29)	124
H(12A)	5160(34)	5441(19)	3765(31)	129
H(12B)	4728(34)	5816(19)	2579(31)	129
H(13A)	4769(31)	6697(17)	4053(26)	106
H(15A)	7988(29)	6777(17)	3704(27)	102
H(16A)	9699(29)	6567(16)	5393(25)	103
H(16B)	8742(29)	6654(16)	6077(25)	103
H(17A)	9804(31)	7854(18)	5806(26)	109
H(18A)	7934(22)	7835(12)	3836(21)	60
H(20A)	7243(27)	7452(15)	6348(22)	117
H(20B)	5733(27)	7463(15)	5798(22)	117
H(20C)	6508(27)	8165(15)	6052(22)	117
H(21A)	4697(28)	7862(15)	3830(25)	98
H(21B)	5489(28)	7931(15)	3003(25)	98
H(22A)	4798(33)	9075(19)	3287(32)	127
H(22B)	5421(33)	8996(19)	4623(32)	127
H(23A)	6844(28)	8980(18)	3083(27)	103
H(23B)	6891(28)	9595(18)	3919(27)	103
H(25A)	9939(23)	8784(14)	5001(21)	97
H(25B)	9298(23)	9489(14)	4466(21)	97
H(25C)	9152(23)	8829(14)	3693(21)	97
H(27A)	7491(27)	10409(20)	6927(25)	130
H(27B)	8845(27)	10043(20)	7355(25)	130
H(27C)	7640(27)	9671(20)	7499(25)	130

Experimental R1=($\sum||F_o|-|F_c||/\sum|F_o|$), wR2=$\sum w(F_o^2-F_c^2)^2/\sum w[(F_o^2)^2]_{1/2}$, S=$[\sum w(F_o^2-F_c^2)^2/(n-p)]^{1/2}$

Phenyl rings were treated as regular hexagons of D_{6h} symmetry with C-C =

1.395 Å and C-C-C = 120°. Unit cell dimensions and standard deviations were obtained by least squares fit to 16 reflections (14<2 <26°).

References

SHELXTL-PC.& G.M.Sheldrick, Siemens Analytical X-ray Instruments Inc. Madison, WI. (1990). H.D.Flack. Acta Crystallographica, A39, 876-881 (1983).

h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
-1	0	1	187.31	21.98	4.58	0.014	9.52
1	9	0	11224.33	5492.79	4.42	0.228	2.09
-3	14	5	-93.15	273.07	4.08	0.051	1.18
-2	5	1	649.08	247.09	3.91	0.048	3.14
1	9	1	28888.57	17067.81	3.29	0.401	2.03
-1	9	1	60034.12	36897.02	3.05	0.590	2.08
-2	2	7	331.36	109.37	3.00	0.032	1.75
1	2	11	-204.61	2.23	2.98	0.005	1.02
1	5	0	18363.22	11442.89	2.93	0.328	3.60
-6	10	10	-156.03	101.36	2.93	0.031	1.00
0	12	3	1498.62	855.68	2.91	0.090	1.48
1	0	0	700.98	376.68	2.87	0.060	10.35
3	0	5	-71.53	57.43	2.86	0.023	1.69
2	1	8	90.16	356.22	2.84	0.058	1.30
11	0	0	340.68	27.89	2.83	0.016	0.94
-4	4	10	-149.05	14.52	2.83	0.012	1.20
9	3	4	278.13	16.88	2.81	0.013	0.95
-1	16	3	296.76	714.14	2.81	0.082	1.15
-1	10	2	162.69	11.54	2.77	0.010	1.83
11	3	0	-261.83	1.14	2.77	0.003	0.93
-7	5	5	-76.52	111.15	2.77	0.032	1.40
5	15	4	-148.71	87.60	2.75	0.029	0.97
1	15	3	523.32	215.94	2.75	0.045	1.19
1	1	8	9.98	216.98	2.71	0.045	1.38
0	1	5	52.18	196.97	2.69	0.043	2.33
8	8	1	239.20	21.71	2.69	0.014	1.10
5	13	5	428.84	141.58	2.69	0.037	0.99
2	5	5	-18.30	135.38	2.68	0.036	1.71
-10	3	10	-224.90	9.82	2.68	0.010	0.94
1	4	0	4661.34	2964.77	2.67	0.167	4.35
-5	12	8	-68.53	164.16	2.67	0.039	1.07
1	5	8	-3.66	194.00	2.67	0.043	1.30
-11	1	5	144.39	507.13	2.67	0.069	1.00
8	2	4	-180.65	13.12	2.66	0.011	1.05
2	2	8	-28.94	161.35	2.66	0.039	1.29
-8	8	8	-124.43	95.62	2.61	0.030	1.07
0	8	4	392.67	175.69	2.61	0.041	1.86
-4	10	10	-161.36	24.94	2.59	0.015	1.04
0	14	3	622.70	319.96	2.56	0.055	1.29
4	14	0	1026.68	566.40	2.56	0.073	1.21
5	6	5	-57.89	131.83	2.55	0.035	1.24
-9	8	4	-64.87	135.79	2.55	0.036	1.09
1	14	1	4230.83	2715.46	2.55	0.160	1.34
3	3	6	110.45	351.03	2.54	0.058	1.46
-7	2	10	9.65	236.98	2.54	0.047	1.11
0	12	0	1768.25	1080.90	2.54	0.101	1.60
9	3	2	-168.34	19.40	2.52	0.014	1.05
-2	12	5	1064.94	608.80	2.52	0.076	1.34
-1	16	5	109.46	403.03	2.52	0.062	1.08
-10	9	6	-186.64	32.39	2.51	0.017	0.96

X-Ray data of compound **17**

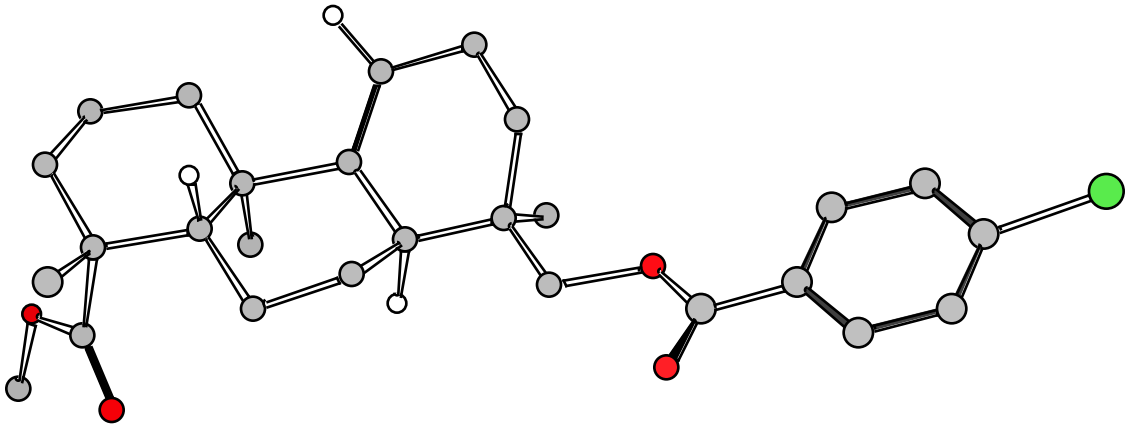


Table 1. Crystal data and structure refinement for TAOT2

Empirical formula	$C_{27}\ H_{34.50}BrO_4$
Formula weight	502.96
Temperature	22 C
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 10.801(6)Å alpha = 90° b = 22.481(11)Å beta = 109.45(4)° c = 10.819(5)Å gamma = 90°
Crystal size	0.80 x 0.50 x 0.06 mm
Density (calculated)	1.349 Mg/m^3
Absorption coefficient	1.689 mm^{-1}
Volume, Z, F(000)	2477(2) $Å^3$, 4, 1054
θ range for data collection	1.81 to 22.50°
Limiting indices	-11 ≤ h ≤ 10, 0 ≤ k <21, -10 ≤ l ≤ 10
Reflections collected	2728, 1179 observed [I>2σ(I)]
Independent reflections	2579 (R_{int} = 0.0747)
Absorption correction None	
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2578 / 1 / 257
Goodness-of-fit on F^2	1.035
Final R indices [I>2σ(I)]	R1 = 0.1126, wR2 = 0.2667
R indices (all data)	R1 = 0.2329, wR2 = 0.3694
Absolute structure parameter	0.00(6)
Largest diff. peak and hole	0.625 and -0.627 $eÅ^3$
Scan speed, range, type	5°/minute, 0.6°, Wyckoff
Background range, % time	0.6°, 25% each side

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for TAOT2 U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	-4359(19)	-2240(9)	-3220(19)	75(12)
C(2)	-3091(20)	-2403(9)	-3148(21)	82(13)
C(3)	-2423(15)	-2074(11)	-3820(21)	41(10)
C(4)	-3023(19)	-1583(11)	-4566(22)	74(12)
C(5)	-4291(19)	-1419(9)	-4638(19)	60(11)
C(6)	-4959(15)	-1748(8)	-3965(16)	35(9)
C(7)	-6309(17)	-1589(13)	-4075(26)	53(11)
Br(1)	-675(5)	-2235(3)	-3662(5)	99(2)
O(1)	-6805(25)	-1163(12)	-4847(24)	64(8)
O(2)	-6846(29)	-1821(14)	-3383(28)	93(10)
O(3)	-14596(22)	-191(16)	-6693(24)	83(9)
O(4)	-15258(20)	631(10)	-7812(21)	55(7)
C(8)	-8113(36)	-947(17)	-4814(36)	56(11)
C(9)	-8475(36)	-399(17)	-5678(34)	51(11)
C(10)	-8369(35)	-484(16)	-6955(32)	54(11)
C(11)	-7453(34)	88(16)	-5041(33)	54(11)
C(12)	-7697(34)	666(16)	-5729(36)	61(11)
C(13)	-9105(35)	830(19)	-6190(35)	67(12)
C(14)	-10088(31)	429(15)	-6317(29)	36(9)
C(15)	-9801(33)	-199(20)	-5809(32)	51(10)
C(16)	-10307(25)	-260(15)	-4665(26)	32(8)
C(17)	-11794(35)	-127(18)	-5067(37)	69(11)
C(18)	-12019(31)	506(15)	-5530(31)	46(9)
C(19)	-11587(30)	663(16)	-6633(30)	36(9)
C(20)	-12310(32)	330(16)	-7958(30)	54(10)
C(21)	-11675(32)	1324(15)	-6890(35)	54(10)
C(22)	-13040(30)	1557(16)	-7173(31)	46(9)
C(23)	-13583(34)	1381(16)	-6137(36)	60(11)
C(24)	-13460(32)	742(16)	-5770(31)	45(9)
C(25)	-13813(40)	665(20)	-4496(39)	87(13)
C(26)	-14501(39)	327(21)	-6854(40)	49(11)
C(27)	-16213(41)	293(20)	-8779(39)	87(14)
C(1')	-10498(21)	2(8)	-11208(21)	60(11)
C(2')	-11807(22)	128(9)	-11368(23)	81(13)
C(3')	-12670(17)	-332(12)	-11360(21)	65(12)
C(4')	-12225(21)	-919(11)	-11192(23)	78(13)
C(5')	-10916(22)	-1045(8)	-11032(22)	69(12)
C(6')	-10053(17)	-585(9)	-11040(16)	65(13)
C(7')	-8687(19)	-719(14)	-10915(28)	62(12)
Br(2)	-14430(4)	-182(3)	-11569(5)	103(2)
O(1')	-8284(25)	-1227(12)	-10352(24)	62(8)
O(2')	-7976(26)	-324(13)	-11199(25)	84(9)
O(3')	-1068(22)	-2486(14)	-10054(23)	80(10)
O(4')	-445(23)	-3407(14)	-9330(21)	72(8)
C(8')	-6860(39)	-1338(20)	-10035(41)	69(13)
C(9')	-6619(33)	-1947(16)	-9411(31)	41(10)
C(10')	-7605(40)	-2403(19)	-10277(39)	81(14)
C(11')	-6778(37)	-1936(17)	-8042(33)	54(11)
C(12')	-6533(42)	-2497(19)	-7386(43)	88(15)
C(13')	-5361(32)	-2842(16)	-7555(32)	54(10)
C(14')	-4756(41)	-2677(19)	-8398(39)	66(13)
C(15')	-5170(35)	-2170(20)	-9266(34)	56(11)

C(16')	-4155(39)	-1651(21)	-8846(43)	89(14)
C(17')	-2814(37)	-1912(17)	-8821(38)	66(11)
C(18')	-2454(35)	-2483(17)	-8035(37)	63(11)
C(19')	-3505(34)	-2953(16)	-8371(32)	44(10)
C(20')	-3739(31)	-3239(16)	-9773(29)	50(10)
C(21')	-3031(33)	-3461(16)	-7390(33)	53(10)
C(22')	-1607(30)	-3724(16)	-7224(34)	49(10)
C(23')	-695(34)	-3245(17)	-6982(32)	55(10)
C(24')	-1012(34)	-2712(16)	-7885(34)	52(10)
C(25')	18(37)	-2203(20)	-7376(37)	80(13)
C(26')	-867(38)	-2861(19)	-9073(41)	55(11)
C(27')	-271(38)	-3534(18)	-10485(38)	78(13)

Table 3. Bond lengths [Å] and angles [°] for TAOT2

C(1)-C(6)	1.39
C(1)-C(2)	1.40
C(2)-C(3)	1.40
C(3)-C(4)	1.39
C(3)-Br(1)	1.87(2)
C(4)-C(5)	1.39
C(5)-C(6)	1.39
C(6)-C(7)	1.47
C(7)-O(2)	1.21(3)
C(7)-O(1)	1.27(3)
O(1)-C(8)	1.51(4)
O(3)-C(26)	1.19(5)
O(4)-C(26)	1.28(4)
O(4)-C(27)	1.42(4)
C(8)-C(9)	1.52(5)
C(9)-C(10)	1.44(5)
C(9)-C(15)	1.46(5)
C(9)-C(11)	1.55(4)
C(11)-C(12)	1.48(5)
C(12)-C(13)	1.48(4)
C(13)-C(14)	1.36(4)
C(14)-C(15)	1.51(5)
C(14)-C(19)	1.63(4)
C(15)-C(16)	1.52(4)
C(16)-C(17)	1.55(4)
C(17)-C(18)	1.50(5)
C(18)-C(19)	1.46(4)
C(18)-C(24)	1.58(4)
C(19)-C(21)	1.51(5)
C(19)-C(20)	1.58(4)
C(21)-C(22)	1.50(4)
C(22)-C(23)	1.48(4)
C(23)-C(24)	1.48(5)
C(24)-C(25)	1.56(5)
C(24)-C(26)	1.62(5)
C(1')-C(2')	1.39
C(1')-C(6')	1.40
C(2')-C(3')	1.40
C(3')-C(4')	1.39

C(3')-Br(2)	1.87(2)
C(4')-C(5')	1.39
C(5')-C(6')	1.39
C(6')-C(7')	1.47
C(7')-O(2')	1.28(4)
C(7')-O(1')	1.30(4)
O(1')-C(8')	1.48(4)
O(3')-C(26')	1.32(4)
O(4')-C(27')	1.35(4)
O(4')-C(26')	1.37(4)
C(8')-C(9')	1.51(5)
C(9')-C(11')	1.55(5)
C(9')-C(10')	1.55(5)
C(9')-C(15')	1.60(5)
C(11')-C(12')	1.43(5)
C(12')-C(13')	1.55(5)
C(13')-C(14')	1.34(5)
C(14')-C(15')	1.45(5)
C(14')-C(19')	1.48(5)
C(15')-C(16')	1.56(5)
C(16')-C(17')	1.55(5)
C(17')-C(18')	1.52(5)
C(18')-C(19')	1.50(5)
C(18')-C(24')	1.60(5)
C(19')-C(21')	1.53(5)
C(19')-C(20')	1.59(4)
C(21')-C(22')	1.60(4)
C(22')-C(23')	1.42(4)
C(23')-C(24')	1.51(5)
C(24')-C(26')	1.39(5)
C(24')-C(25')	1.56(5)
C(6)-C(1)-C(2)	120.0
C(1)-C(2)-C(3)	120.0
C(4)-C(3)-C(2)	120.0
C(4)-C(3)-Br(1)	117.8(13)
C(2)-C(3)-Br(1)	122.1(13)
C(3)-C(4)-C(5)	120.0
C(4)-C(5)-C(6)	120.0
C(5)-C(6)-C(1)	120.0
C(5)-C(6)-C(7)	120.1
C(1)-C(6)-C(7)	119.9
O(2)-C(7)-O(1)	123(2)
O(2)-C(7)-C(6)	121(2)
O(1)-C(7)-C(6)	115(2)
C(7)-O(1)-C(8)	115(2)
C(26)-O(4)-C(27)	115(3)
O(1)-C(8)-C(9)	107(3)
C(10)-C(9)-C(15)	110(3)
C(10)-C(9)-C(8)	114(3)
C(15)-C(9)-C(8)	111(3)
C(10)-C(9)-C(11)	104(3)
C(15)-C(9)-C(11)	111(3)
C(8)-C(9)-C(11)	108(3)
C(12)-C(11)-C(9)	115(3)
C(11)-C(12)-C(13)	112(3)
C(14)-C(13)-C(12)	123(4)
C(13)-C(14)-C(15)	122(3)
C(13)-C(14)-C(19)	120(3)

C(15)-C(14)-C(19)	117(3)
C(9)-C(15)-C(14)	114(3)
C(9)-C(15)-C(16)	120(3)
C(14)-C(15)-C(16)	107(3)
C(15)-C(16)-C(17)	112(3)
C(18)-C(17)-C(16)	108(3)
C(19)-C(18)-C(17)	116(3)
C(19)-C(18)-C(24)	110(3)
C(17)-C(18)-C(24)	115(3)
C(18)-C(19)-C(21)	112(3)
C(18)-C(19)-C(20)	116(3)
C(21)-C(19)-C(20)	109(3)
C(18)-C(19)-C(14)	108(3)
C(21)-C(19)-C(14)	111(3)
C(20)-C(19)-C(14)	101(2)
C(22)-C(21)-C(19)	112(3)
C(23)-C(22)-C(21)	111(3)
C(22)-C(23)-C(24)	116(3)
C(23)-C(24)-C(25)	109(3)
C(23)-C(24)-C(18)	111(3)
C(25)-C(24)-C(18)	110(3)
C(23)-C(24)-C(26)	113(3)
C(25)-C(24)-C(26)	105(3)
C(18)-C(24)-C(26)	109(3)
O(3)-C(26)-O(4)	125(4)
O(3)-C(26)-C(24)	122(4)
O(4)-C(26)-C(24)	112(3)
C(2')-C(1')-C(6')	120.0
C(1')-C(2')-C(3')	120.0
C(4')-C(3')-C(2')	120.0
C(4')-C(3')-Br(2)	118.7(14)
C(2')-C(3')-Br(2)	121.3(14)
C(3')-C(4')-C(5')	120.0
C(6')-C(5')-C(4')	120.0
C(5')-C(6')-C(1')	120.0
C(5')-C(6')-C(7')	120.1
C(1')-C(6')-C(7')	119.9
O(2')-C(7')-O(1')	126(2)
O(2')-C(7')-C(6')	120(2)
O(1')-C(7')-C(6')	113(2)
C(7')-O(1')-C(8')	115(3)
C(27')-O(4')-C(26')	122(3)
O(1')-C(8')-C(9')	105(3)
C(8')-C(9')-C(11')	111(3)
C(8')-C(9')-C(10')	110(3)
C(11')-C(9')-C(10')	109(3)
C(8')-C(9')-C(15')	110(3)
C(11')-C(9')-C(15')	109(3)
C(10')-C(9')-C(15')	108(3)
C(12')-C(11')-C(9')	114(3)
C(11')-C(12')-C(13')	114(4)
C(14')-C(13')-C(12')	123(4)
C(13')-C(14')-C(15')	123(4)
C(13')-C(14')-C(19')	121(4)
C(15')-C(14')-C(19')	115(4)
C(14')-C(15')-C(16')	111(3)
C(14')-C(15')-C(9')	113(3)
C(16')-C(15')-C(9')	111(3)

C(17')-C(16')-C(15')	106(3)
C(18')-C(17')-C(16')	113(3)
C(19')-C(18')-C(17')	115(3)
C(19')-C(18')-C(24')	116(3)
C(17')-C(18')-C(24')	113(3)
C(14')-C(19')-C(18')	109(3)
C(14')-C(19')-C(21')	115(3)
C(18')-C(19')-C(21')	108(3)
C(14')-C(19')-C(20')	107(3)
C(18')-C(19')-C(20')	113(3)
C(21')-C(19')-C(20')	106(3)
C(19')-C(21')-C(22')	116(3)
C(23')-C(22')-C(21')	109(3)
C(22')-C(23')-C(24')	119(3)
C(26')-C(24')-C(23')	110(3)
C(26')-C(24')-C(25')	103(3)
C(23')-C(24')-C(25')	112(3)
C(26')-C(24')-C(18')	113(3)
C(23')-C(24')-C(18')	109(3)
C(25')-C(24')-C(18')	111(3)
O(3')-C(26')-O(4')	113(3)
O(3')-C(26')-C(24')	124(4)
O(4')-C(26')-C(24')	124(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for TAOT2. The factor exponent takes the form: $-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + 2hka*b*U_{12}]

	U11	U22	U33	U23	U13	U12
Br(1)	69(3)	140(5)	90(4)	5(4)	29(3)	33(4)
O(3)	42(15)	106(27)	77(18)	33(19)	-12(13)	13(17)
O(4)	37(14)	58(17)	47(15)	14(14)	-17(12)	3(14)
Br(2)	50(3)	174(6)	83(3)	18(4)	18(2)	44(4)
O(3')	46(15)	150(29)	44(16)	-5(18)	15(13)	-13(17)
O(4')	70(17)	127(26)	35(14)	3(16)	37(13)	23(17)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for TAOT2

	x	y	z	U(eq)
H(1)	-4818(26)	-2466(12)	-2757(28)	90
H(2)	-2678(27)	-2742(12)	-2635(30)	98
H(4)	-2563(26)	-1356(14)	-5029(31)	88
H(5)	-4704(27)	-1080(11)	-5151(28)	73
H(8A)	-8054(36)	-848(17)	-3923(36)	67
H(8B)	-8774(36)	-1253(17)	-5138(36)	67
H(10A)	-7496(35)	-612(16)	-6866(32)	81
H(10B)	-8553(35)	-116(16)	-7431(32)	81
H(10C)	-8988(35)	-781(16)	-7422(32)	81
H(11A)	-7435(34)	149(16)	-4148(33)	65
H(11B)	-6592(34)	-54(16)	-5001(33)	65
H(12A)	-7389(34)	647(16)	-6474(36)	74
H(12B)	-7199(34)	973(16)	-5141(36)	74
H(13A)	-9330(35)	1225(19)	-6405(35)	81
H(15A)	-10384(33)	-449(20)	-6501(32)	62

H(16A)	-9832(25)	13(15)	-3975(26)	38
H(16B)	-10142(25)	-661(15)	-4318(26)	38
H(17A)	-12281(35)	-394(18)	-5764(37)	83
H(17B)	-12094(35)	-185(18)	-4325(37)	83
H(18A)	-11462(31)	743(15)	-4794(31)	55
H(20A)	-11960(32)	460(16)	-8619(30)	81
H(20B)	-13232(32)	417(16)	-8232(30)	81
H(20C)	-12179(32)	-91(16)	-7830(30)	81
H(21A)	-11088(32)	1529(15)	-6131(35)	64
H(21B)	-11386(32)	1409(15)	-7630(35)	64
H(22A)	-13030(30)	1988(16)	-7231(31)	55
H(22B)	-13600(30)	1404(16)	-8011(31)	55
H(23A)	-14507(34)	1486(16)	-6426(36)	72
H(23B)	-13147(34)	1613(16)	-5357(36)	72
H(25A)	-13734(40)	253(20)	-4245(39)	130
H(25B)	-14699(40)	795(20)	-4653(39)	130
H(25C)	-13225(40)	899(20)	-3806(39)	130
H(27A)	-16733(41)	554(20)	-9457(39)	130
H(27B)	-16771(41)	92(20)	-8384(39)	130
H(27C)	-15781(41)	5(20)	-9150(39)	130
H(1A)	-9904(27)	318(9)	-11213(33)	72
H(2A)	-12113(30)	532(10)	-11484(35)	97
H(4A)	-12819(27)	-1236(13)	-11186(35)	94
H(5A)	-10610(30)	-1449(8)	-10916(33)	83
H(8C)	-6351(39)	-1039(20)	-9432(41)	83
H(8D)	-6622(39)	-1330(20)	-10824(41)	83
H(10D)	-7442(40)	-2787(19)	-9868(39)	121
H(10E)	-7499(40)	-2424(19)	-11122(39)	121
H(10F)	-8484(40)	-2280(19)	-10377(39)	121
H(11C)	-6178(37)	-1644(17)	-7503(33)	64
H(11D)	-7664(37)	-1810(17)	-8138(33)	64
H(12C)	-7316(42)	-2740(19)	-7715(43)	105
H(12D)	-6367(42)	-2431(19)	-6458(43)	105
H(13B)	-5063(32)	-3180(16)	-7048(32)	65
H(16C)	-4414(39)	-1325(21)	-9467(43)	107
H(16D)	-4096(39)	-1504(21)	-7985(43)	107
H(17C)	-2847(37)	-1989(17)	-9714(38)	79
H(17D)	-2132(37)	-1619(17)	-8449(38)	79
H(18B)	-2385(35)	-2362(17)	-7144(37)	76
H(20D)	-2941(31)	-3423(16)	-9790(29)	75
H(20E)	-3997(31)	-2934(16)	-10431(29)	75
H(20F)	-4419(31)	-3534(16)	-9946(29)	75
H(21C)	-3663(33)	-3783(16)	-7654(33)	64
H(21D)	-3029(33)	-3322(16)	-6540(33)	64
H(22C)	-1344(30)	-4003(16)	-6498(34)	59
H(22D)	-1628(30)	-3934(16)	-8014(34)	59
H(23C)	86(34)	-3260(17)	-6278(32)	66
H(25D)	4(37)	-2066(20)	-6540(37)	120
H(25E)	-196(37)	-1879(20)	-7989(37)	120
H(25F)	877(37)	-2350(20)	-7286(37)	120
H(26A)	-1783(38)	-2993(19)	-9417(41)	66
H(27D)	37(38)	-3936(18)	-10467(38)	117
H(27E)	364(38)	-3266(18)	-10620(38)	117
H(27F)	-1091(38)	-3491(18)	-11186(38)	117

Experimental

$R1=(\sum||F_o|-|F_c||/\sum|F_o|)$, $wR2=\sum w(F_o^2-F_c^2)^2/\sum w[(F_o^2)^2]^{1/2}$, $S=[\sum w(F_o^2-F_c^2)^2/(n-p)]^{1/2}$

Phenyl rings were treated as regular hexagons of D_{6h} symmetry with C-C = 1.395 Å and C-C-C = 120°. Unit cell dimensions and standard deviations were obtained by least squares fit to 14 reflections (14<2θ<22°).

References SHELXTL-PC.& G.M.Sheldrick, Siemens Analytical X-ray Instr. Inc. Madison, WI. (1990). H.D.Flack.Acta Crystallographica, A39, 876-881(1983).

	h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
*	5	12	0	-452.12	47.83	3.54	0.031	1.38
	3	5	6	160.38	879.26	3.29	0.131	1.29
	6	10	2	4304.55	2244.03	3.27	0.209	1.23
	-1	20	3	563.02	4.83	3.14	0.010	1.07
	4	1	6	486.25	1385.35	3.03	0.165	1.23
	3	6	1	3304.76	1854.08	2.99	0.190	2.31
	-10	8	5	-493.07	2.62	2.96	0.007	0.99
	2	14	1	-172.32	189.38	2.95	0.061	1.49
	-3	19	2	-32.42	521.83	2.91	0.101	1.12
	4	17	0	102.37	759.92	2.90	0.122	1.17
	-1	16	2	-394.11	12.04	2.88	0.015	1.36
	2	2	6	168.91	696.11	2.87	0.117	1.46
	-7	13	3	499.89	38.35	2.86	0.027	1.15
	3	4	6	902.54	332.32	2.85	0.081	1.31
	-2	18	5	-479.42	1.34	2.84	0.005	1.08
	2	1	6	-114.31	269.56	2.81	0.073	1.47
	3	8	6	547.67	68.00	2.80	0.036	1.22
	2	5	3	607.38	1277.47	2.79	0.158	2.17
	5	7	5	650.03	143.59	2.76	0.053	1.16
	5	4	0	494.78	147.83	2.76	0.054	1.91
	1	9	6	131.37	625.21	2.73	0.111	1.34
	-6	1	2	235.45	662.42	2.67	0.114	1.79
	5	0	3	5551.73	3335.92	2.67	0.255	1.54
	2	15	5	506.72	53.90	2.66	0.032	1.13
	5	7	1	2366.40	1372.84	2.62	0.164	1.62
	-4	12	4	-332.69	9.64	2.62	0.014	1.43
	-2	7	3	472.60	191.80	2.60	0.061	2.33
	1	5	8	545.96	111.16	2.58	0.047	1.17
	5	1	5	-218.38	170.55	2.57	0.058	1.25
	3	11	3	1409.26	2636.11	2.56	0.227	1.46
	5	15	0	-29.00	435.15	2.55	0.092	1.21
	1	11	1	632.97	1241.97	2.54	0.156	1.94
	-8	3	5	320.75	911.52	2.52	0.133	1.27
	4	4	6	-272.98	158.75	2.52	0.056	1.21
	-4	19	3	-162.08	333.71	2.51	0.081	1.07
	-4	9	2	-6.82	294.67	2.50	0.076	1.82
	-10	10	4	-300.28	129.68	2.50	0.050	0.97
	1	16	4	498.19	86.97	2.49	0.041	1.20
	-8	12	1	999.79	420.93	2.47	0.091	1.08
	8	3	3	421.41	0.14	2.44	0.002	1.07
	-2	14	1	151.85	538.96	2.44	0.103	1.54
	1	10	4	-220.09	50.49	2.43	0.031	1.61
	-4	5	4	-141.61	88.85	2.42	0.042	1.98
	1	12	6	-64.83	331.86	2.38	0.081	1.21
	-2	19	1	-184.26	234.99	2.38	0.068	1.15
	4	17	2	440.18	41.94	2.36	0.029	1.11
	2	0	7	-276.39	72.57	2.36	0.038	1.29
	3	5	1	946.90	543.30	2.34	0.103	2.46
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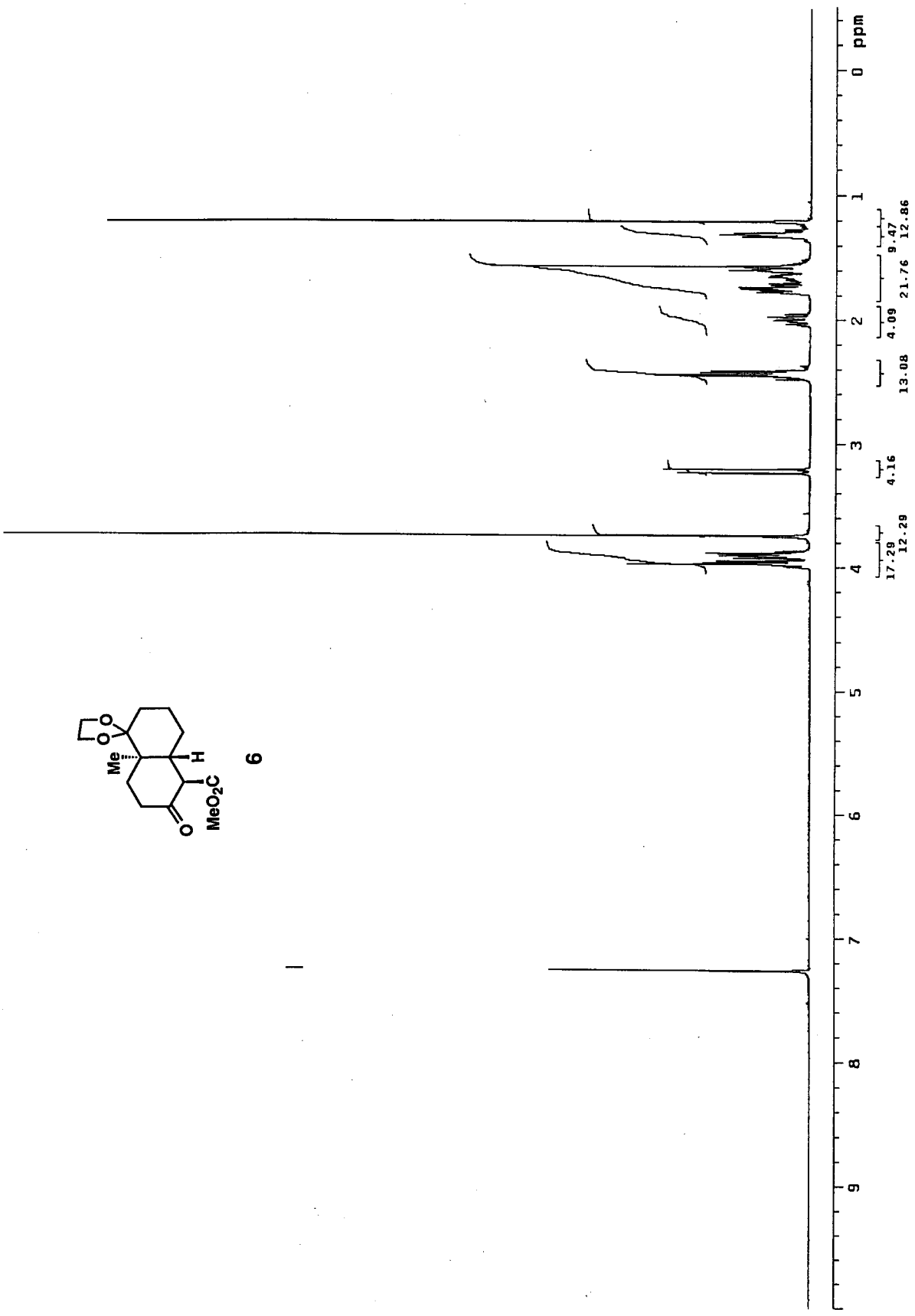
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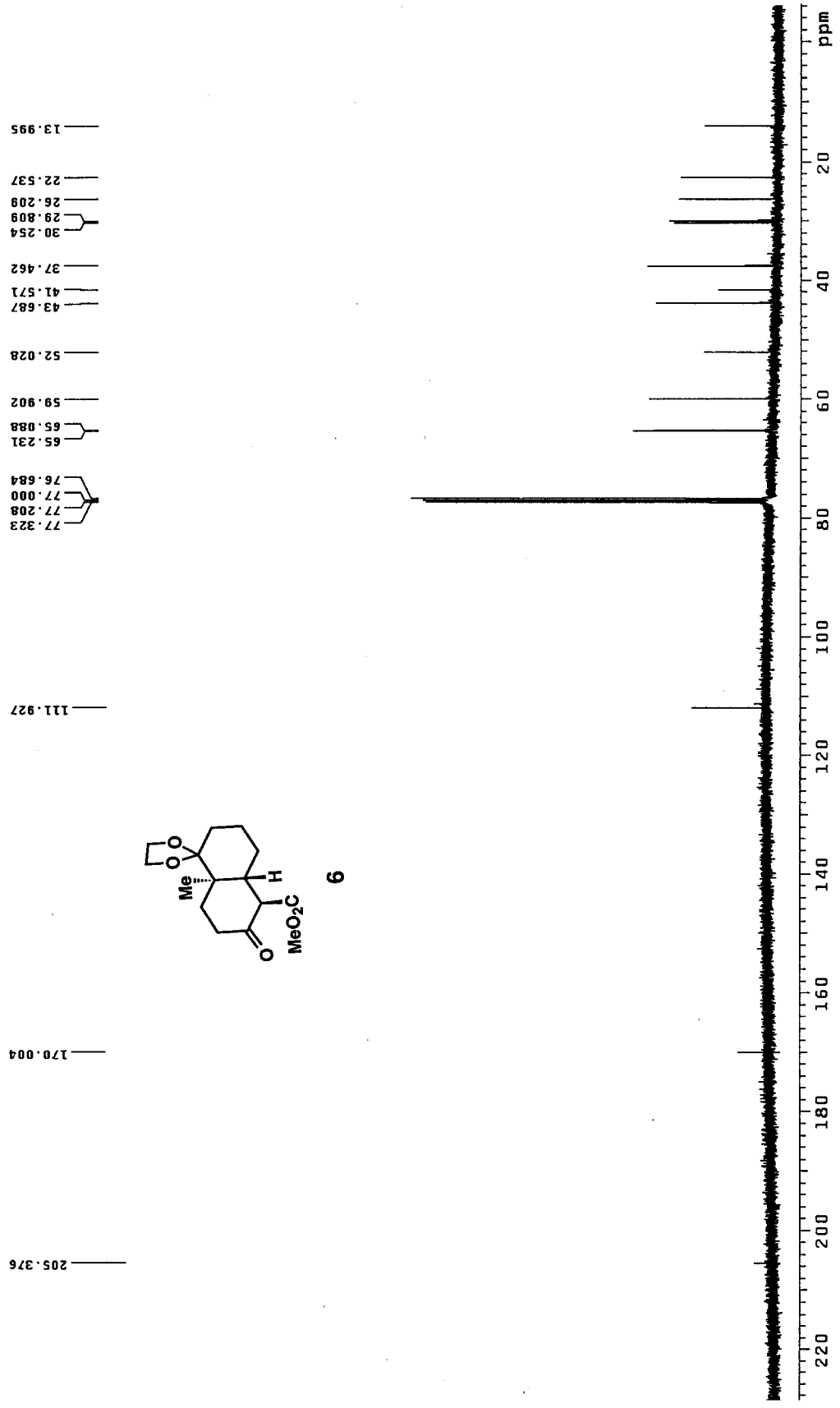
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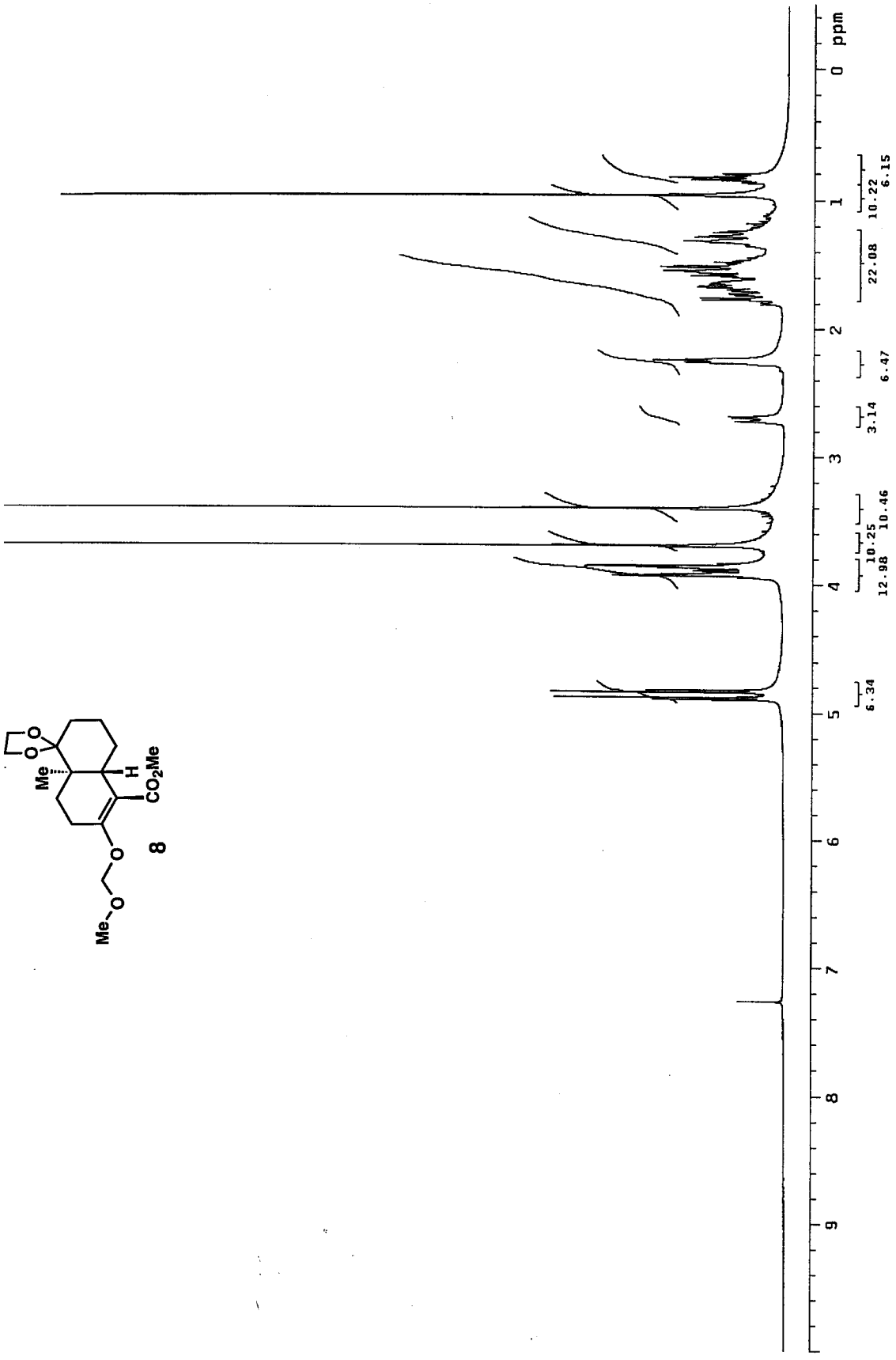
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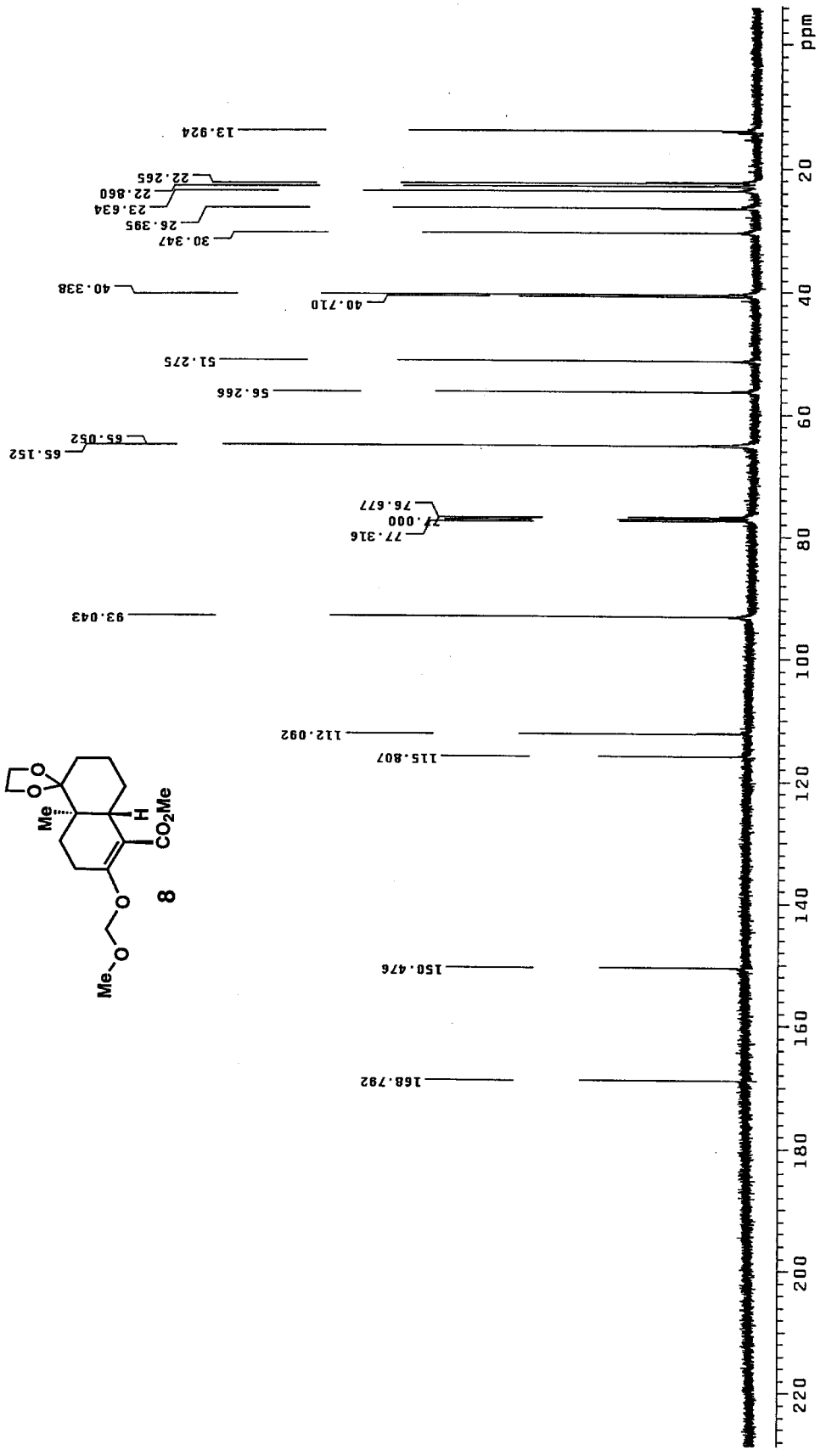
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Me
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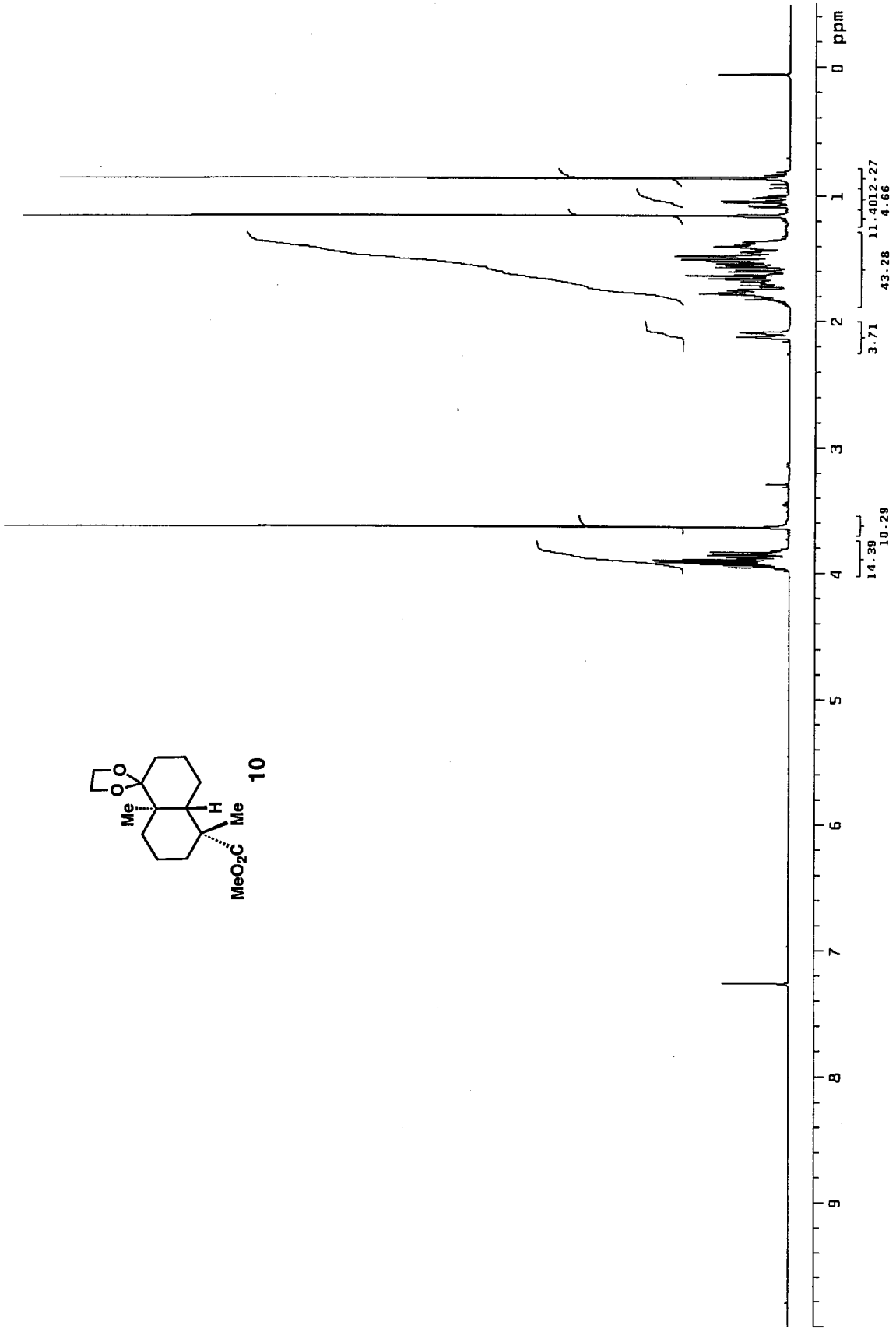
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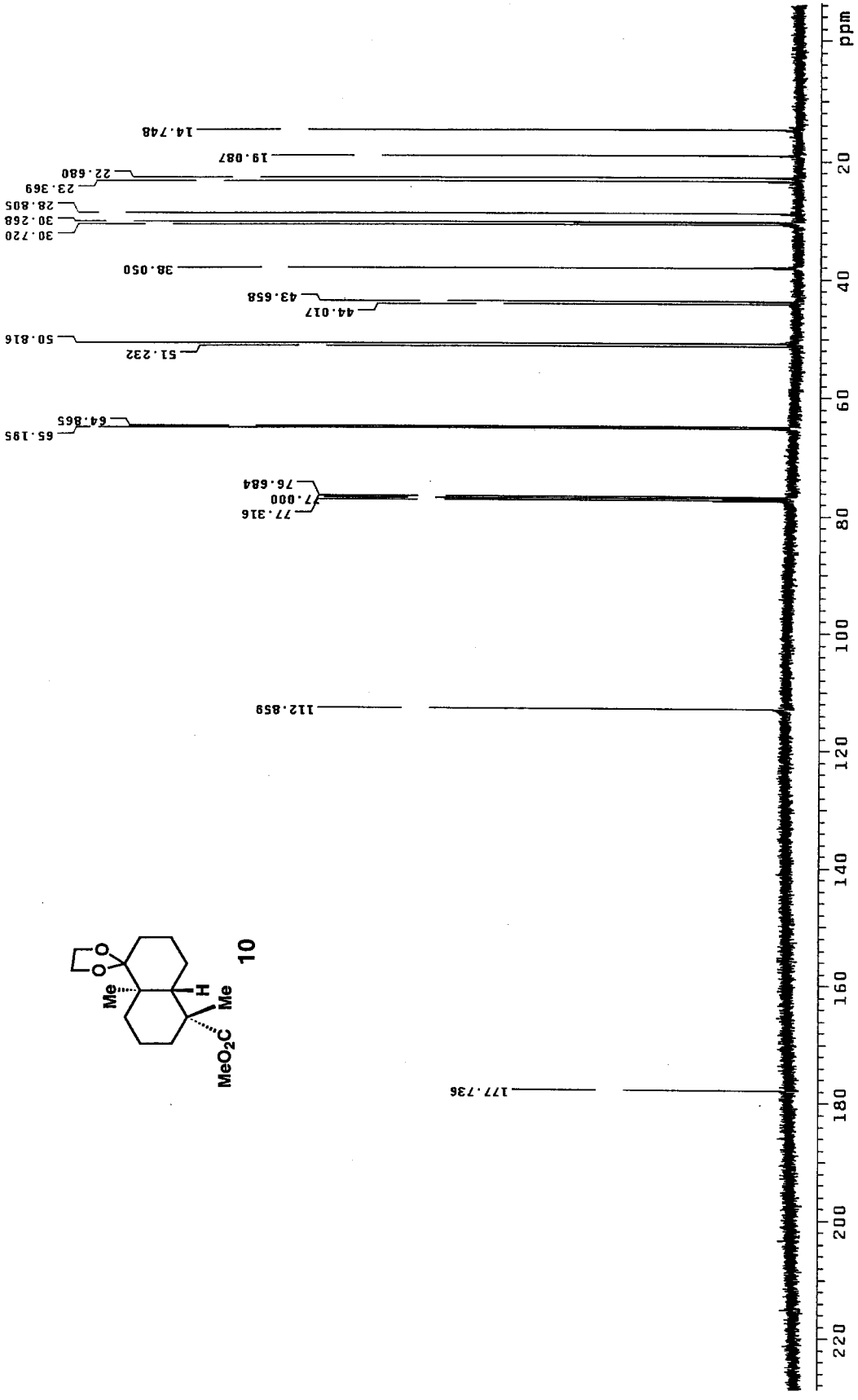
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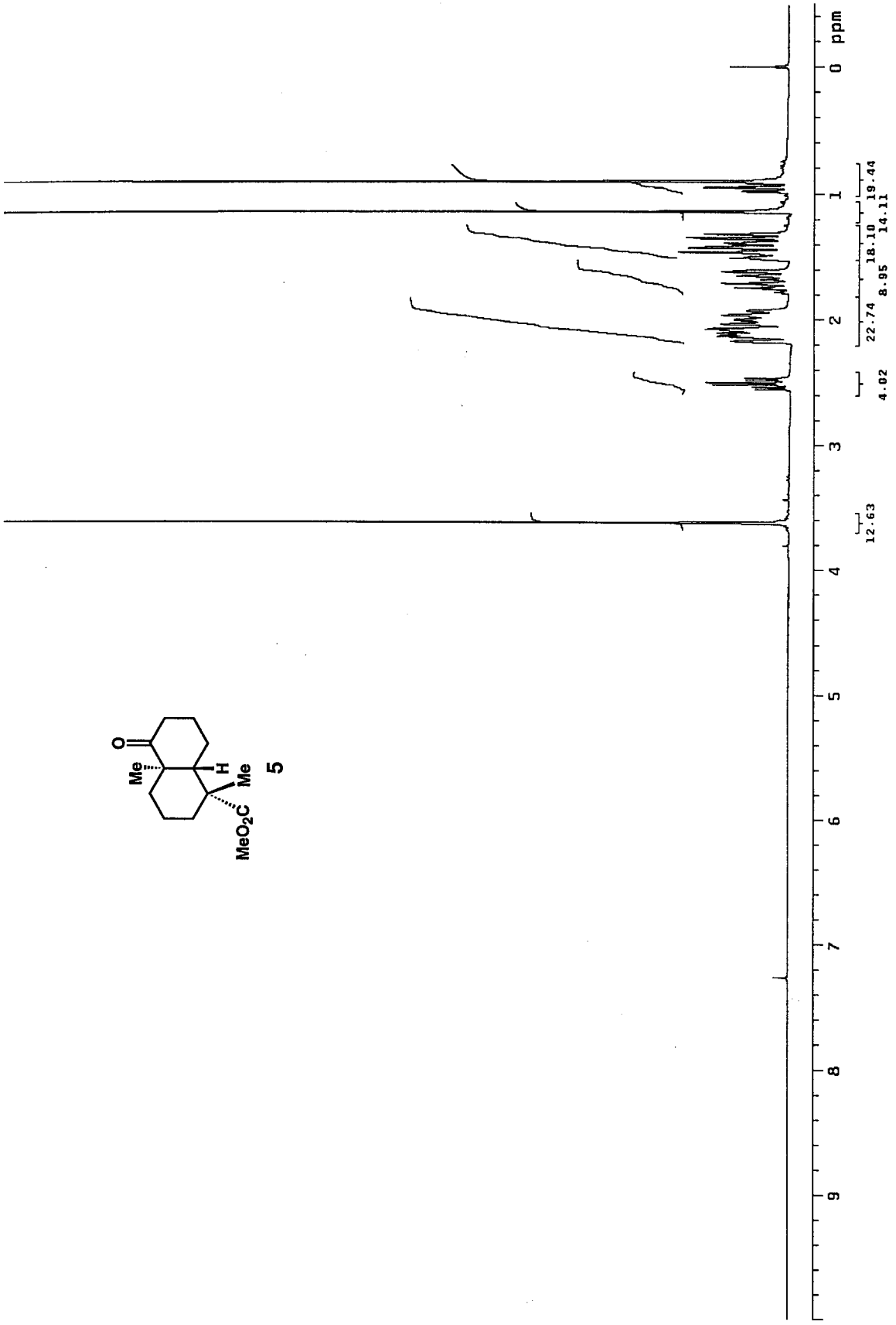
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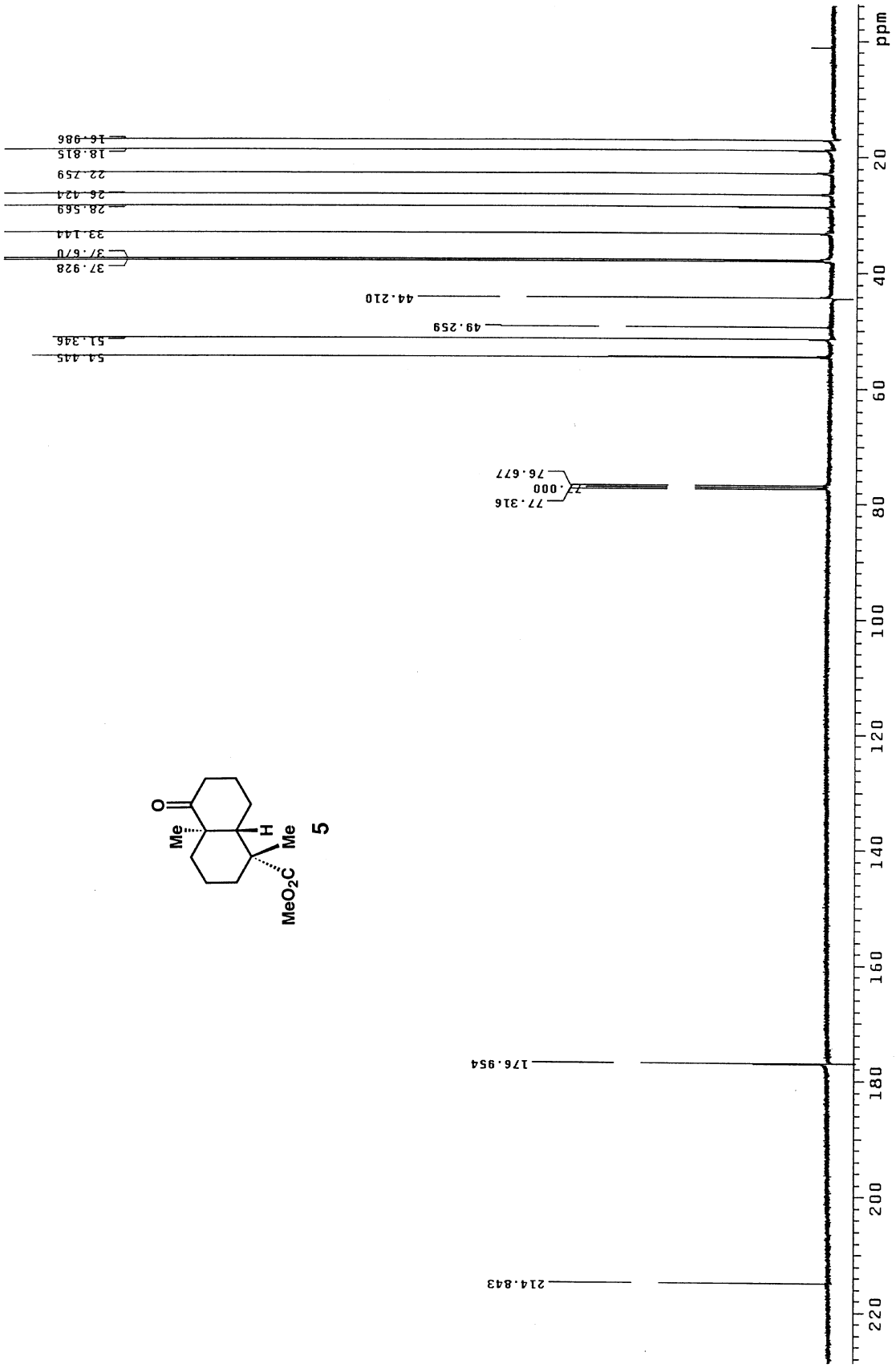
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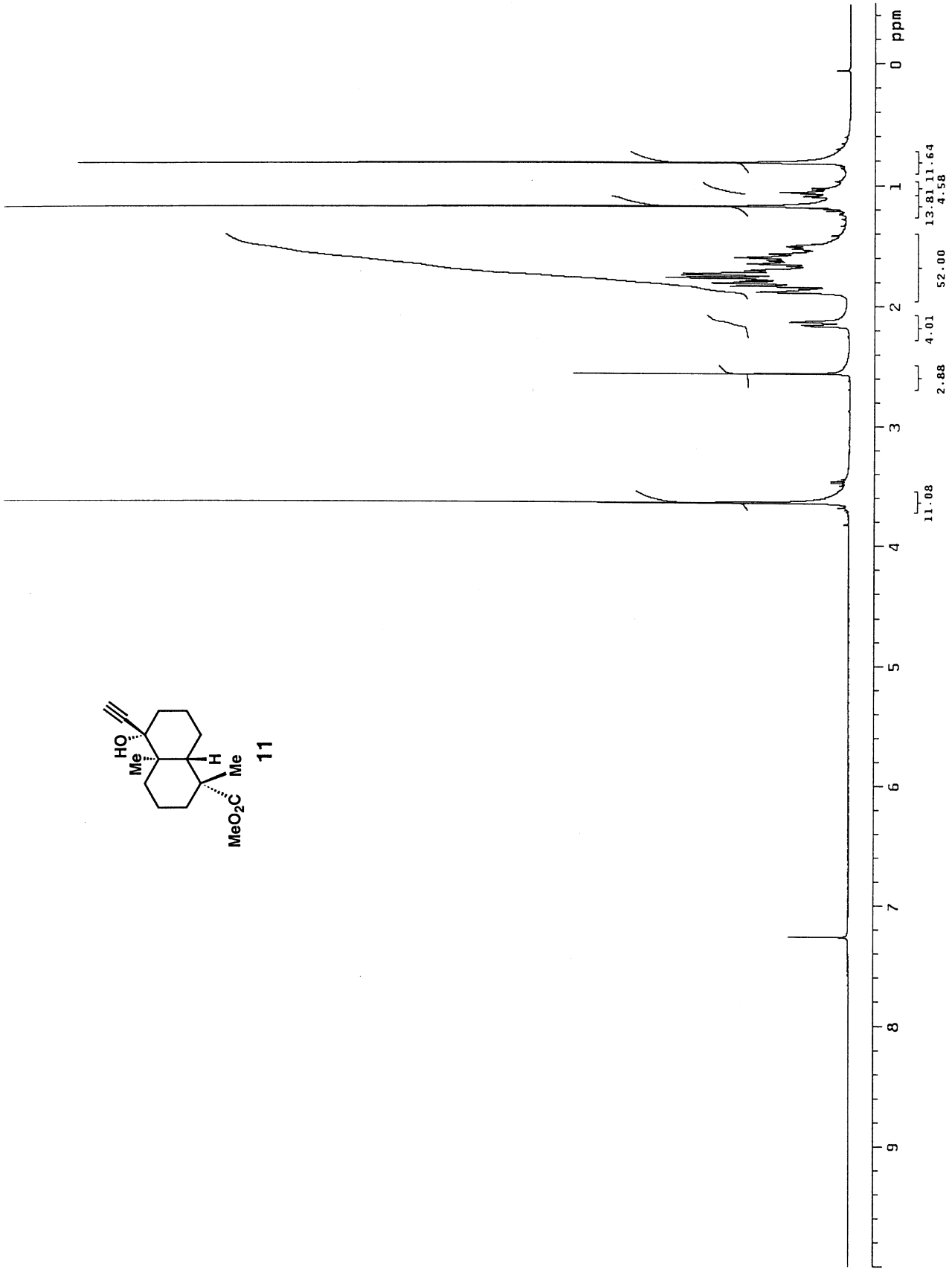
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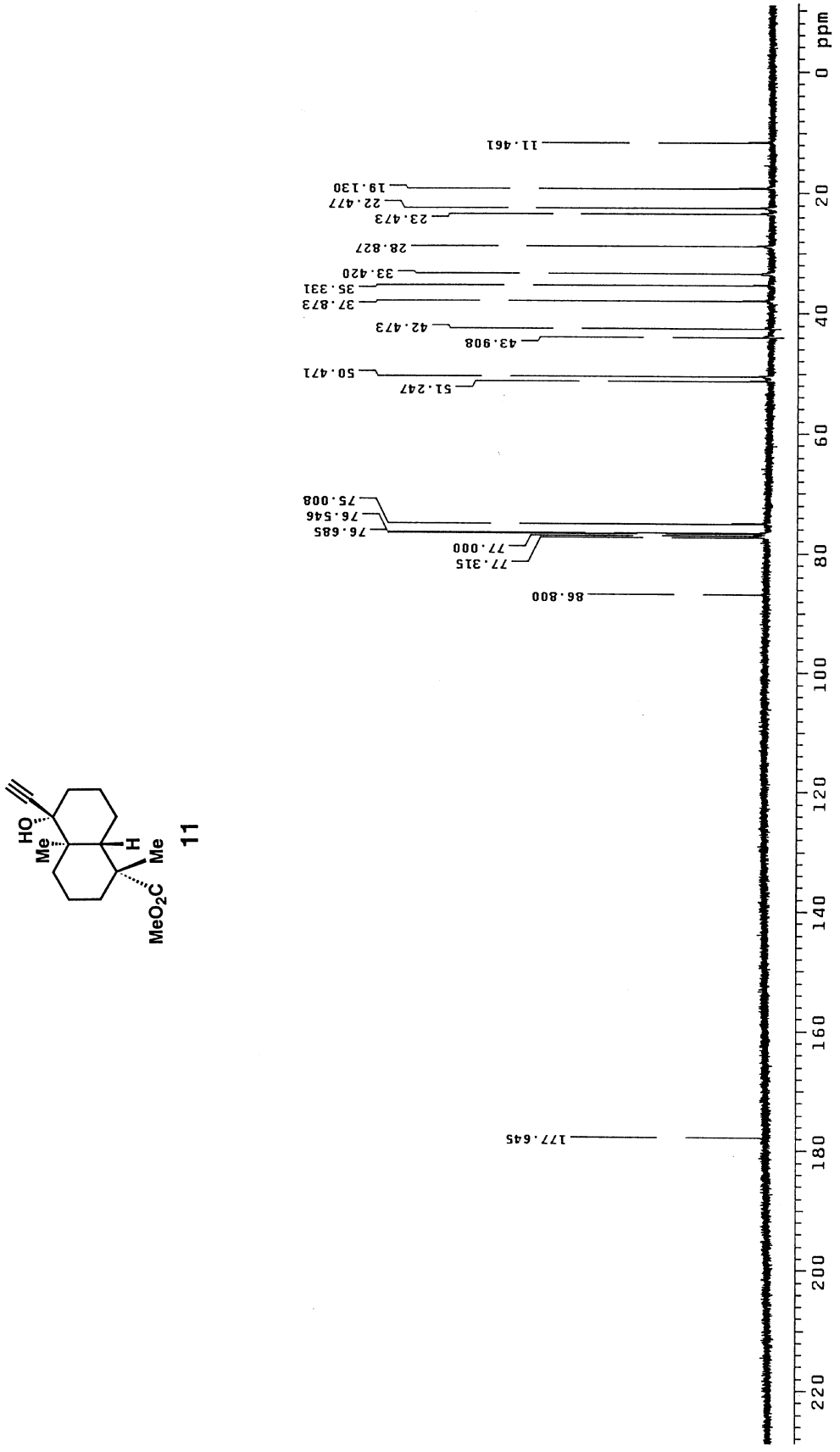
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HO
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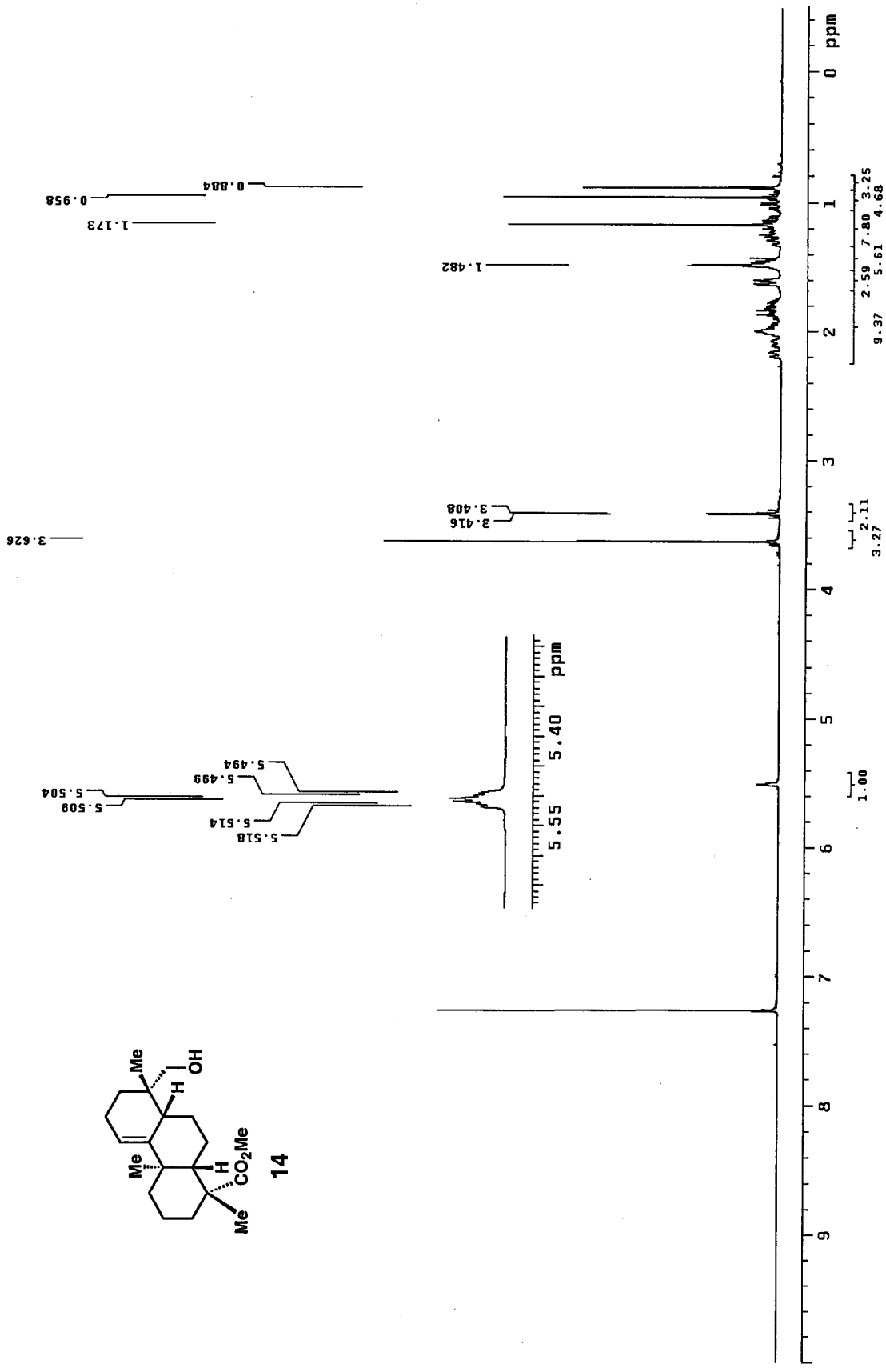
MeO2C

11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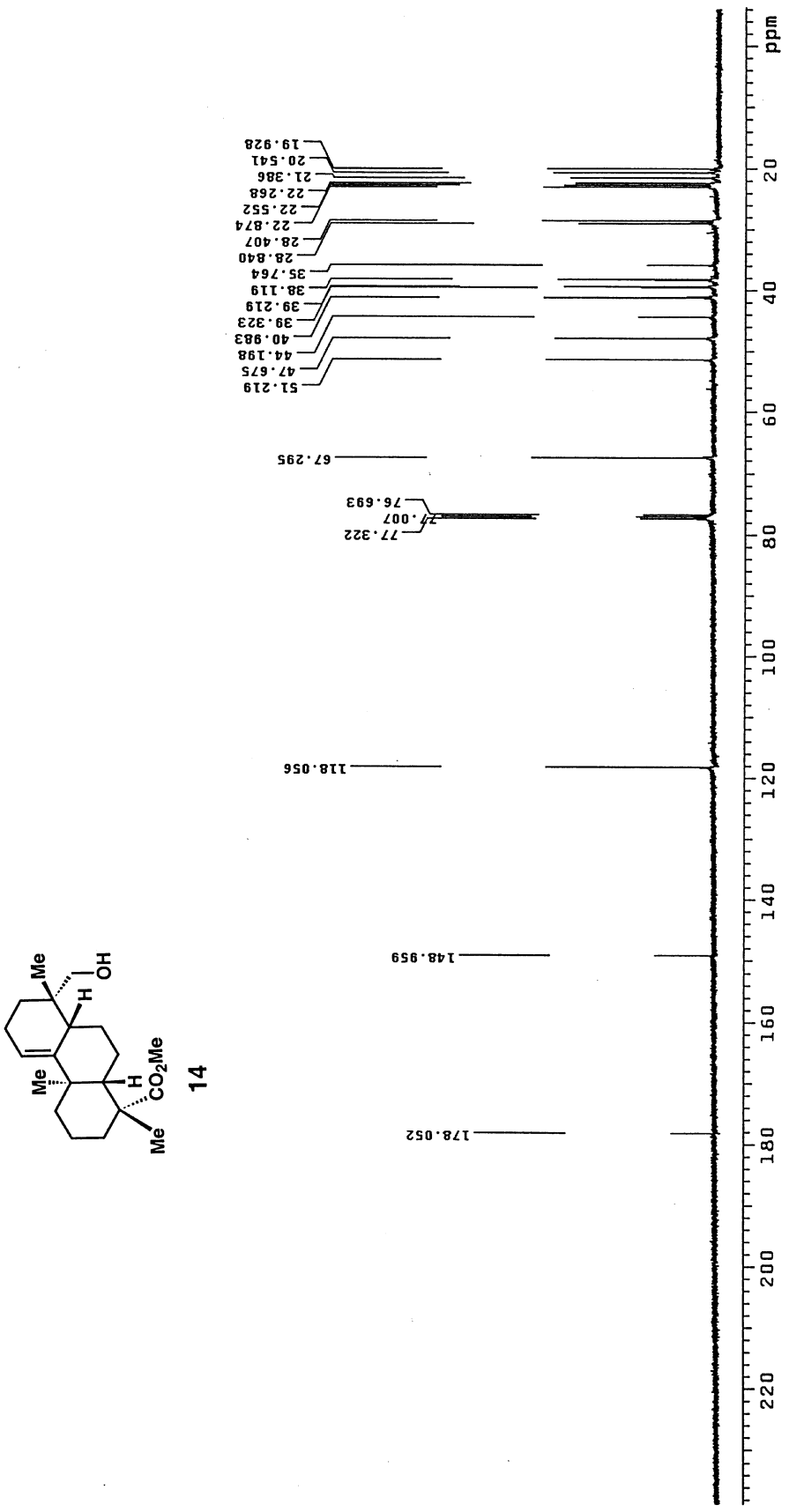
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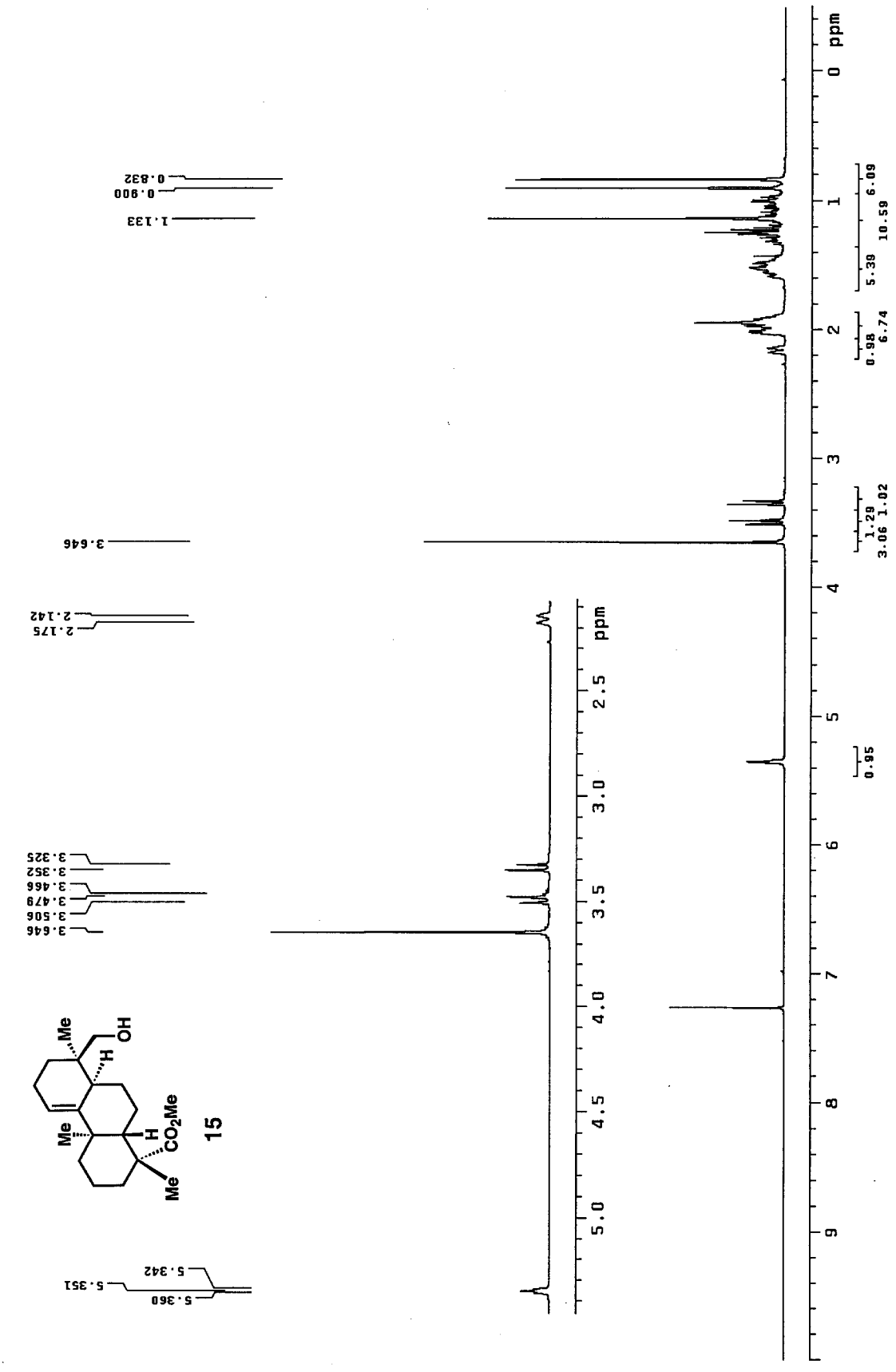
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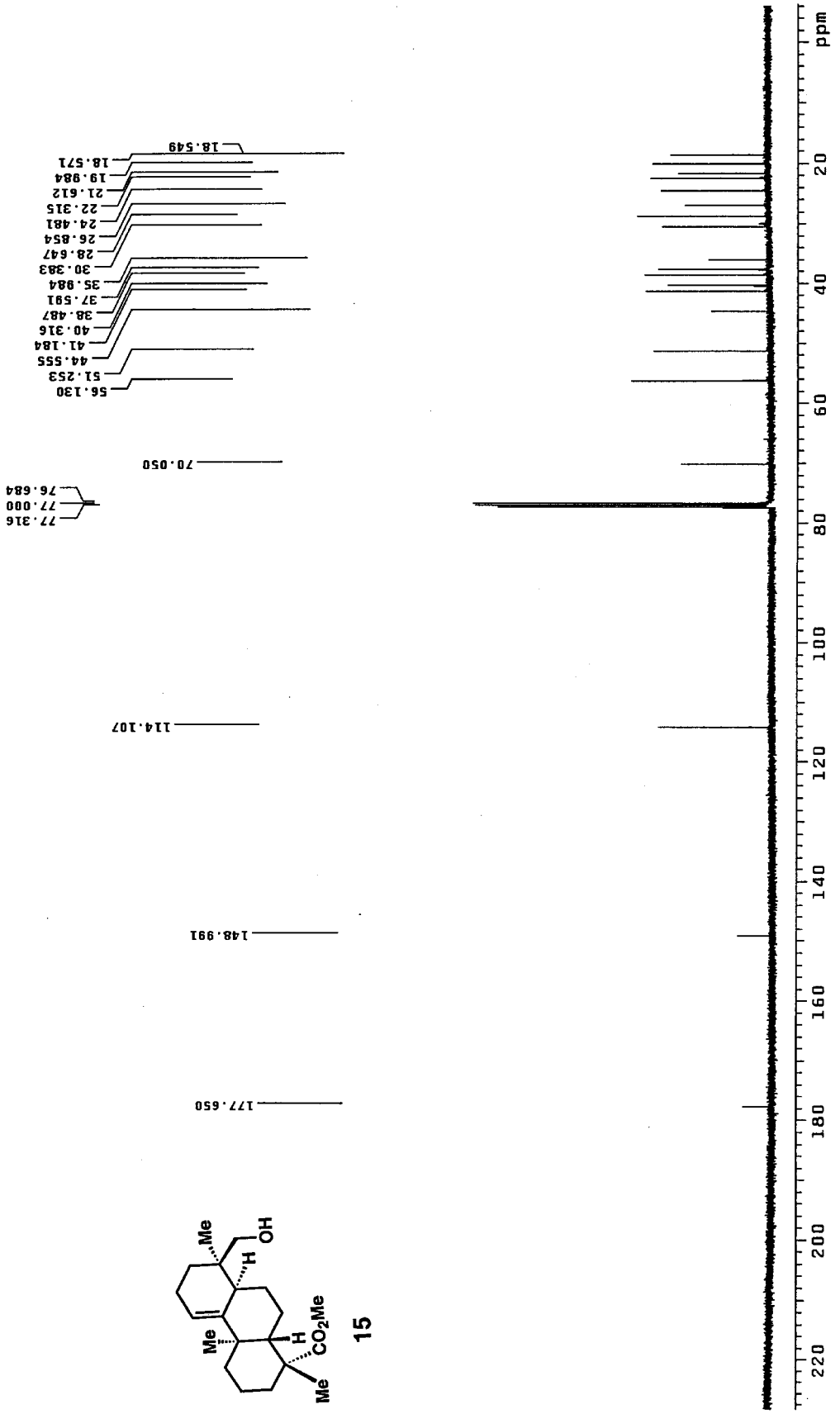
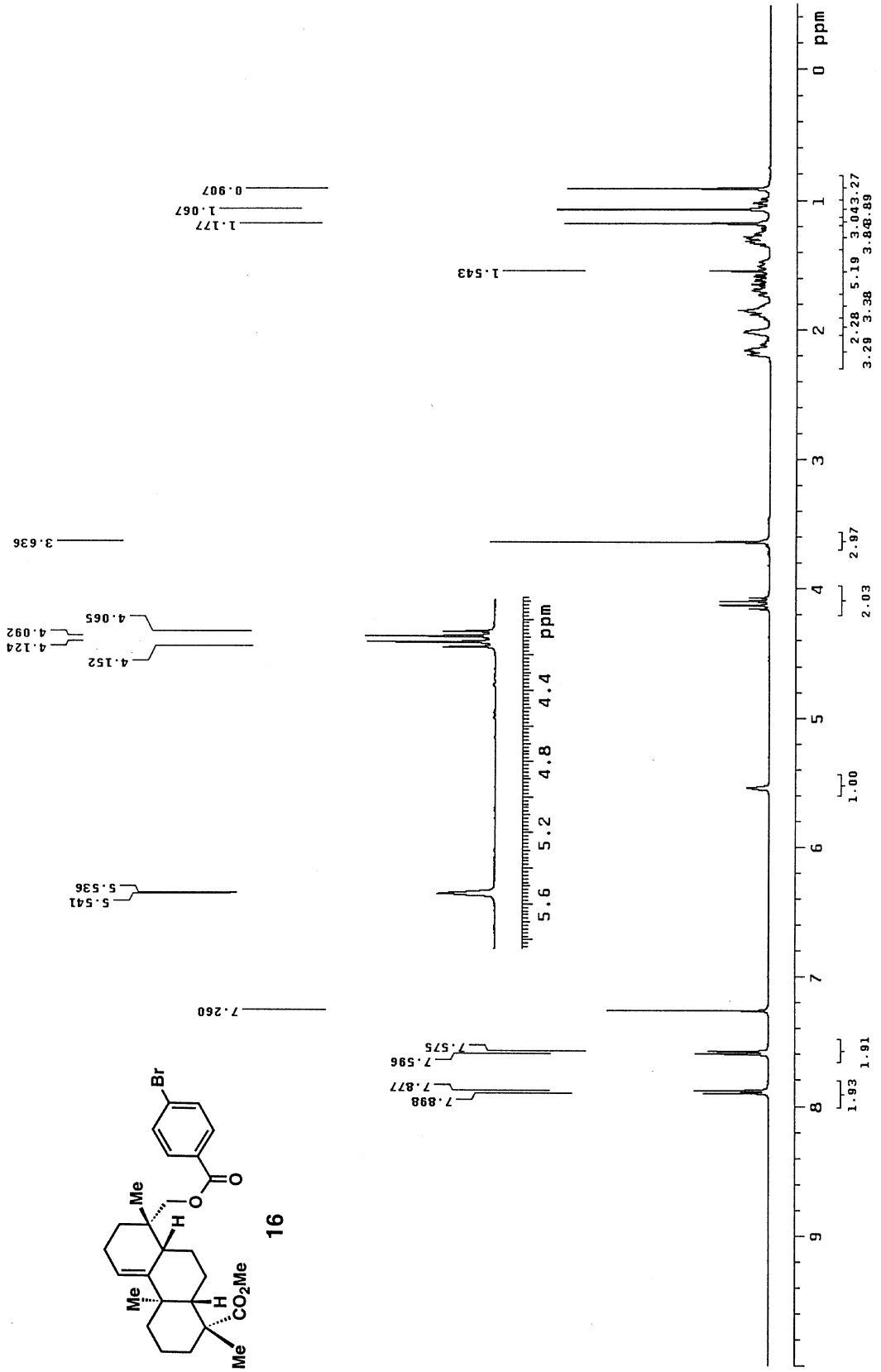
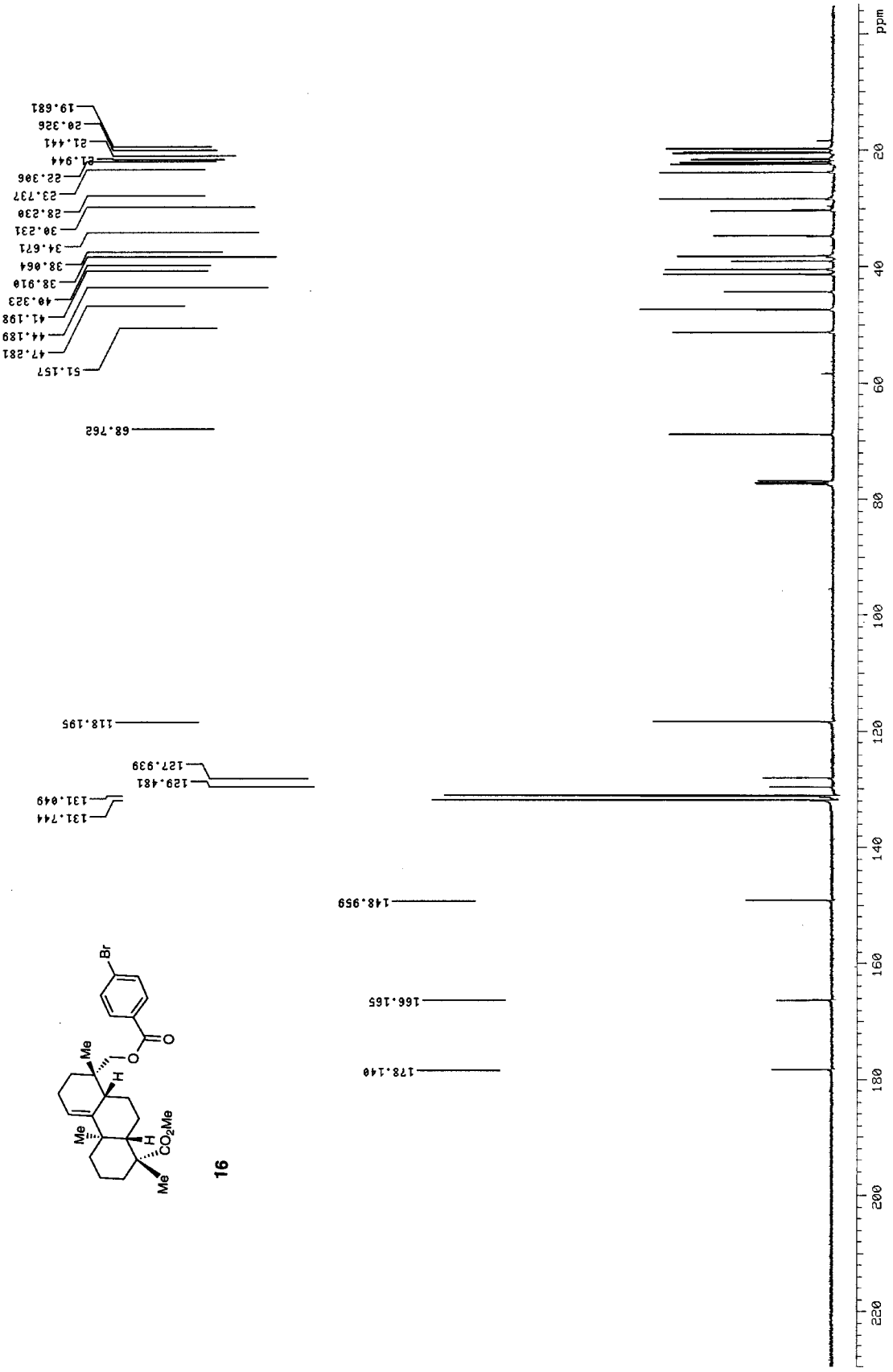
2.142
3.646
1.133
0.900
0.832
ppm
5.0
4.5
4.0
3.5
3.0
2.5
ppm
0
1
2
3
4
5
6
7
8
9
0.95
1.29
3.06
1.02
0.98
6.74
5.39
10.59
6.09

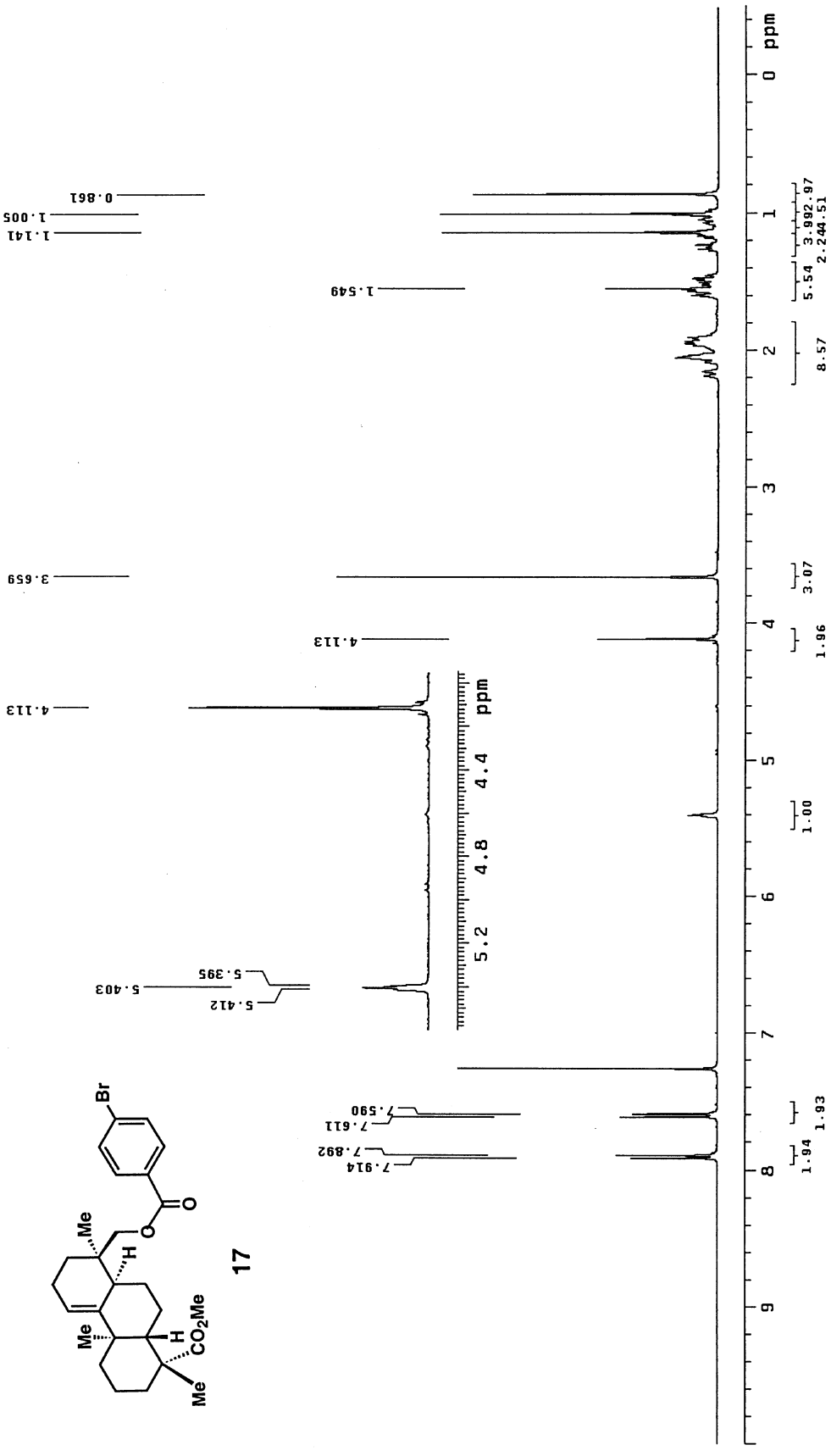

177.650
148.991
114.107
70.050
77.316
77.000
76.684
56.130
51.253
44.555
41.184
40.316
38.487
37.591
35.984
30.383
28.647
26.854
24.481
22.315
21.612
19.984
18.571
18.549
ppm
Me
OH
H
Me
H
CO2Me
Me

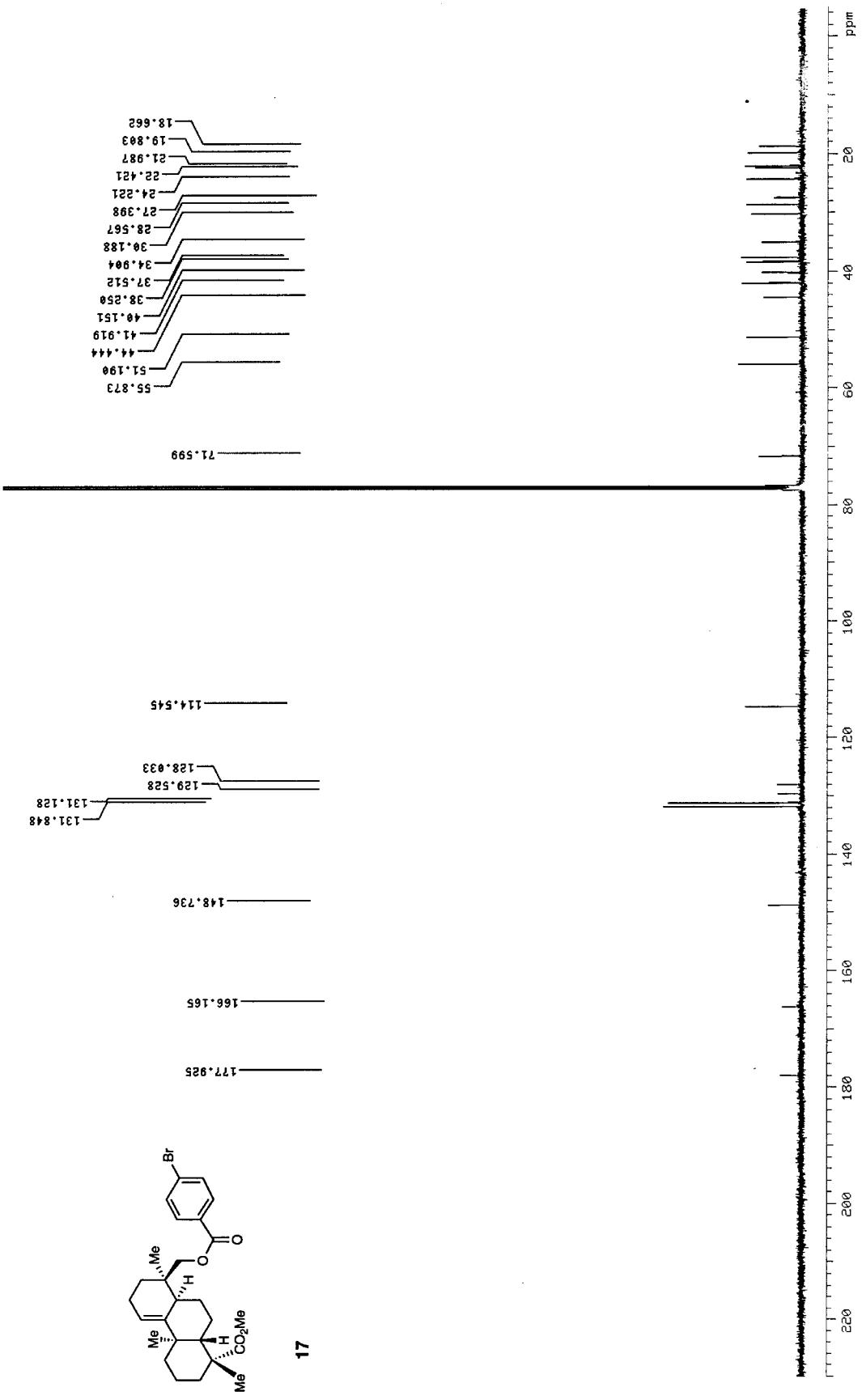
15

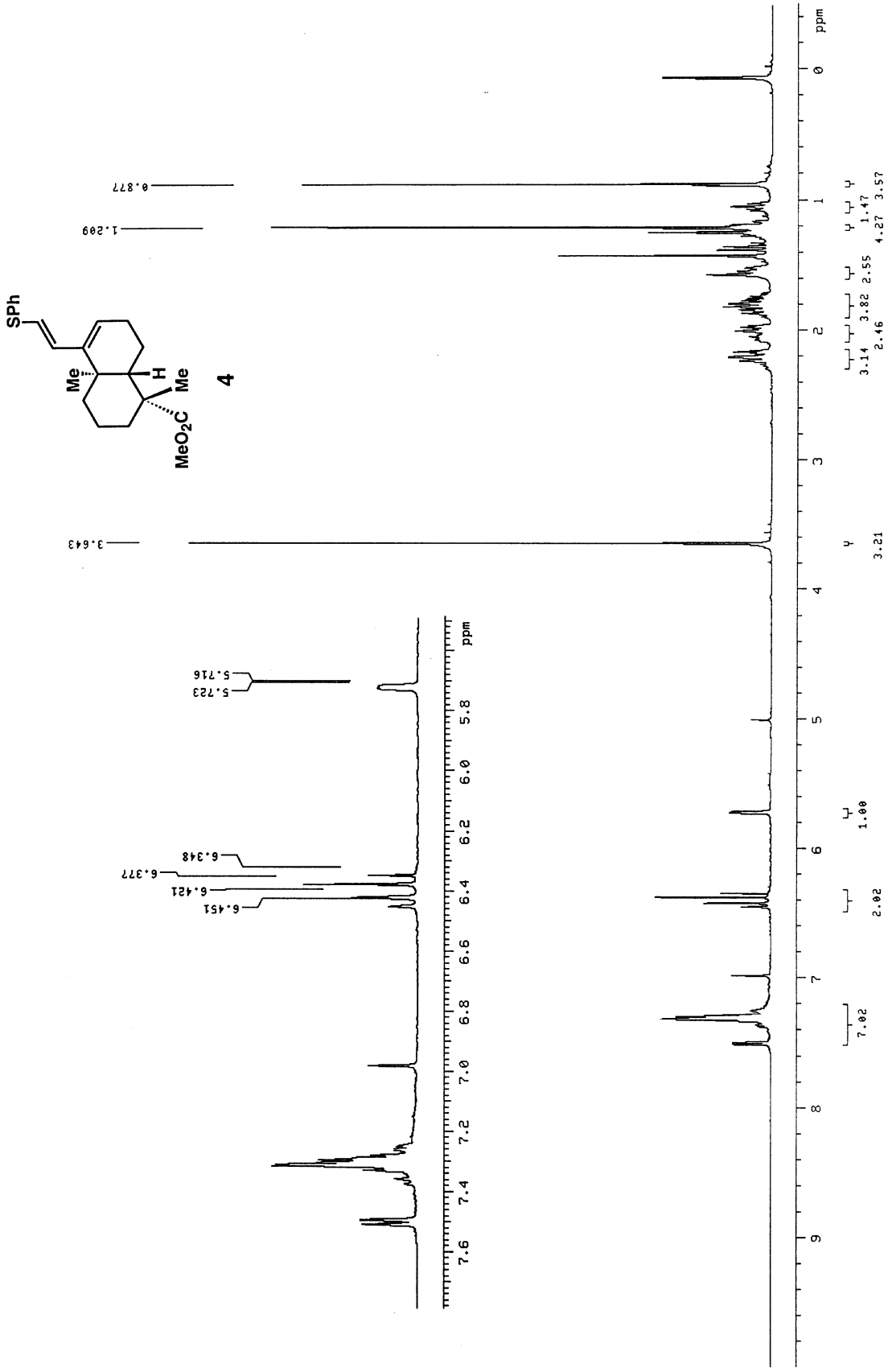

Br
O
O
H
Me
Me
H
CO2Me
Me
16
7.898
7.877
7.596
7.575
7.260
5.541
5.536
4.152
4.124
4.092
4.065
3.636
1.543
1.177
1.067
0.907
5.6 5.2 4.8 4.4 ppm
0 ppm
1.91
1.93
1.00
2.03
2.97
3.29 2.28 3.38 5.19 3.848.89 3.043.27

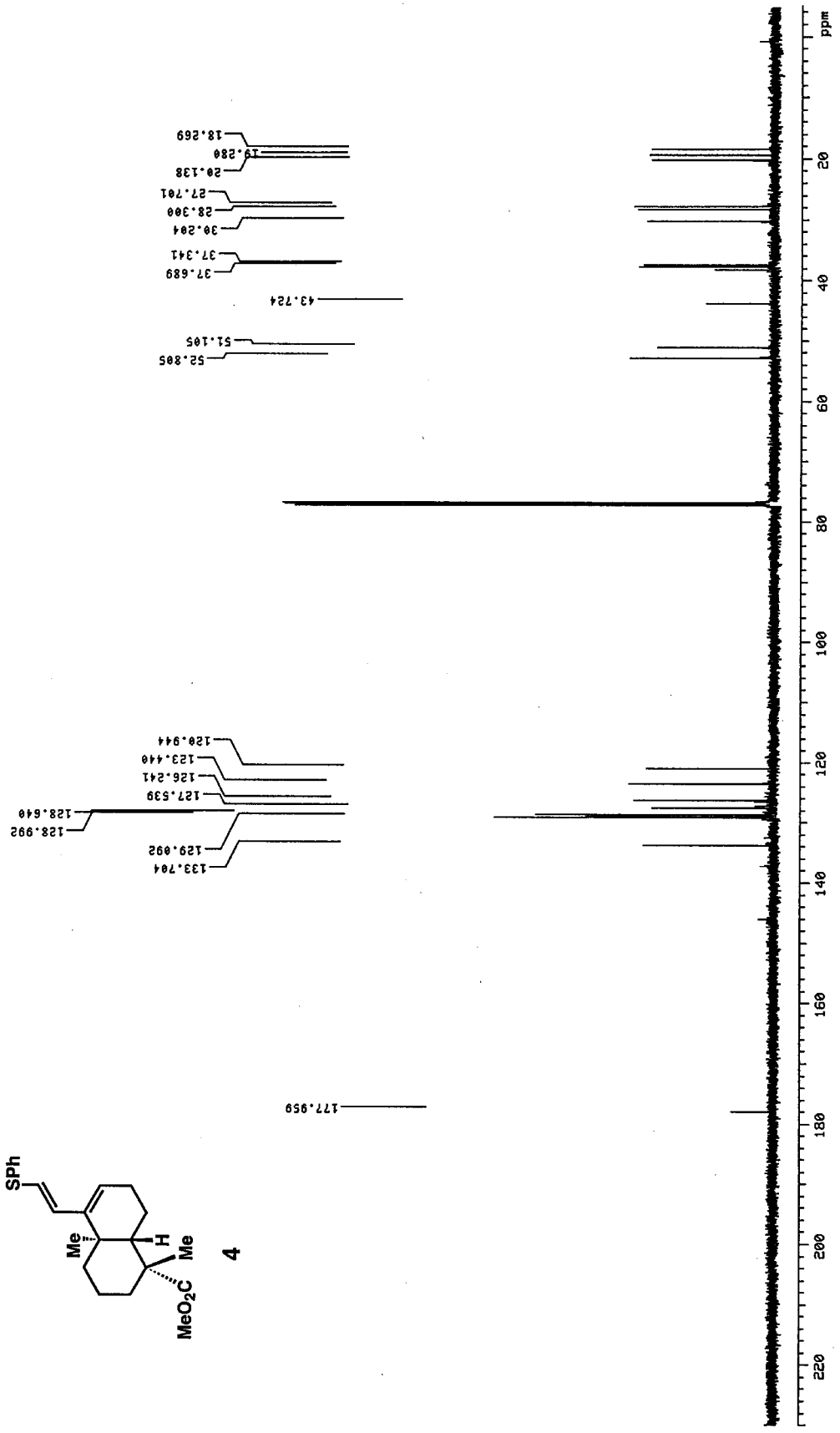

19.681
20.326
21.441
21.944
22.306
23.737
28.230
30.231
34.671
38.064
38.910
40.323
41.198
44.189
47.281
51.157
68.762
118.195
127.939
129.481
131.049
131.744
148.959
166.165
178.140
ppm
20
40
60
80
100
120
140
160
180
200
220
Br
O
O
Me
H
Me
H
CO2Me
Me

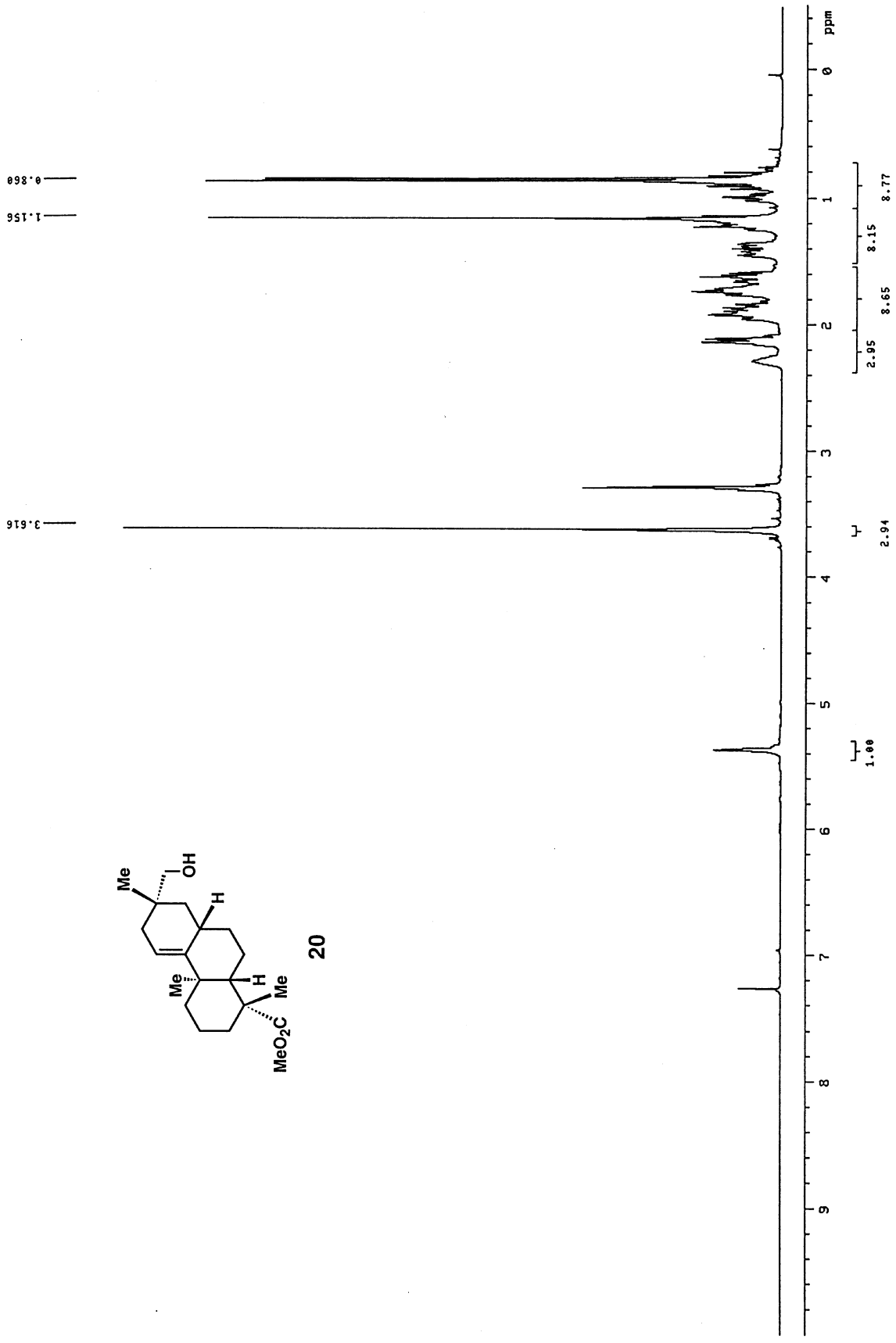
16


Br
O
O
Me
H
Me
H
CO2Me
Me
17
7.914
7.892
7.611
7.590
5.412
5.395
5.403
4.113
3.659
4.113
1.549
0.861
1.005
1.141
5.2
4.8
4.4
ppm
0 ppm
1.94
1.93
1.00
1.96
3.07
8.57
5.54
3.992.97
2.244.51

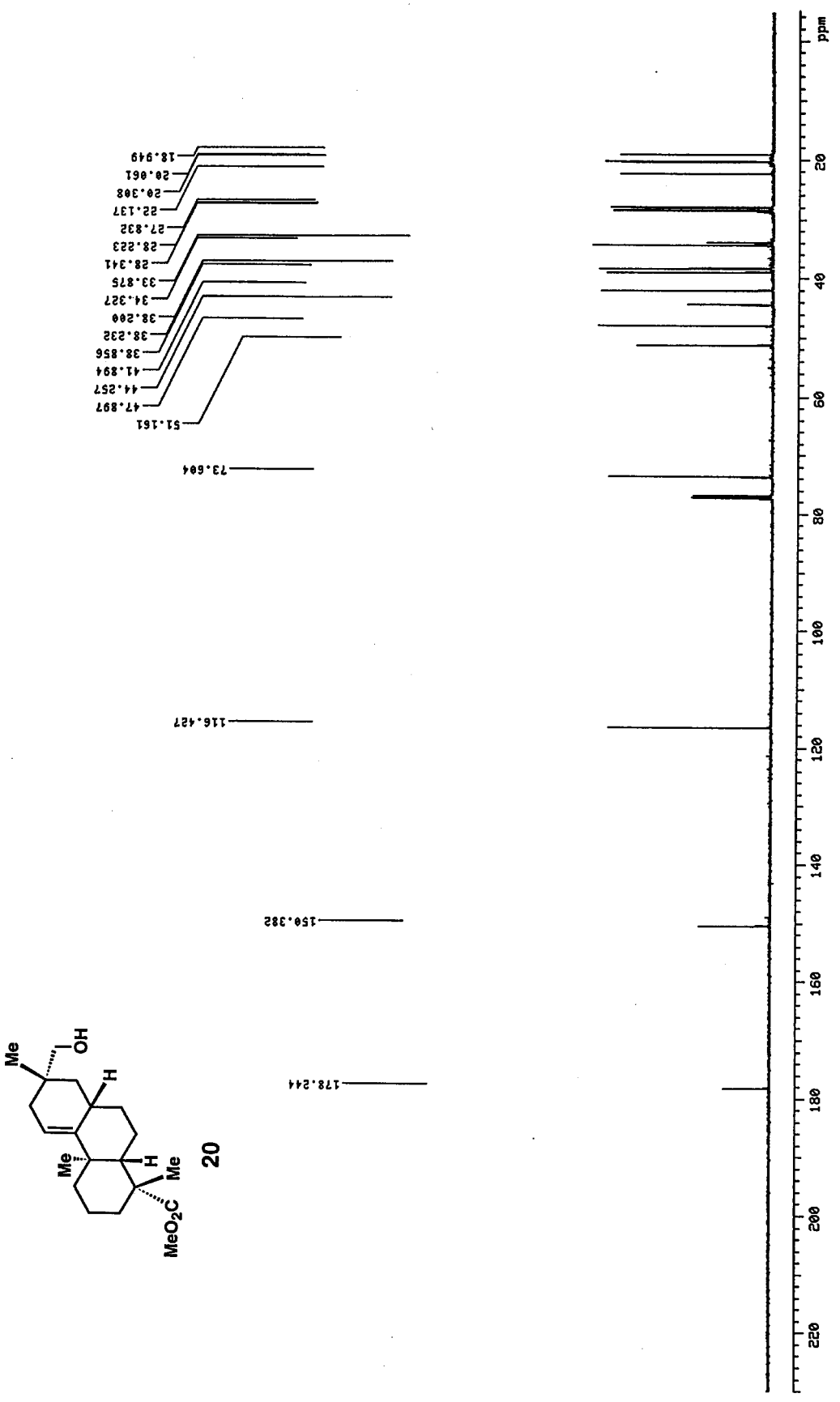

18.662
19.803
21.987
22.421
24.221
27.398
28.567
30.188
34.904
37.512
38.250
40.151
41.919
44.444
51.190
55.873
71.599
114.545
128.033
129.528
131.128
131.848
148.736
166.165
177.925
ppm
20
40
60
80
100
120
140
160
180
200
220
Br
O
O
Me
H
Me
H
CO2Me
Me
17

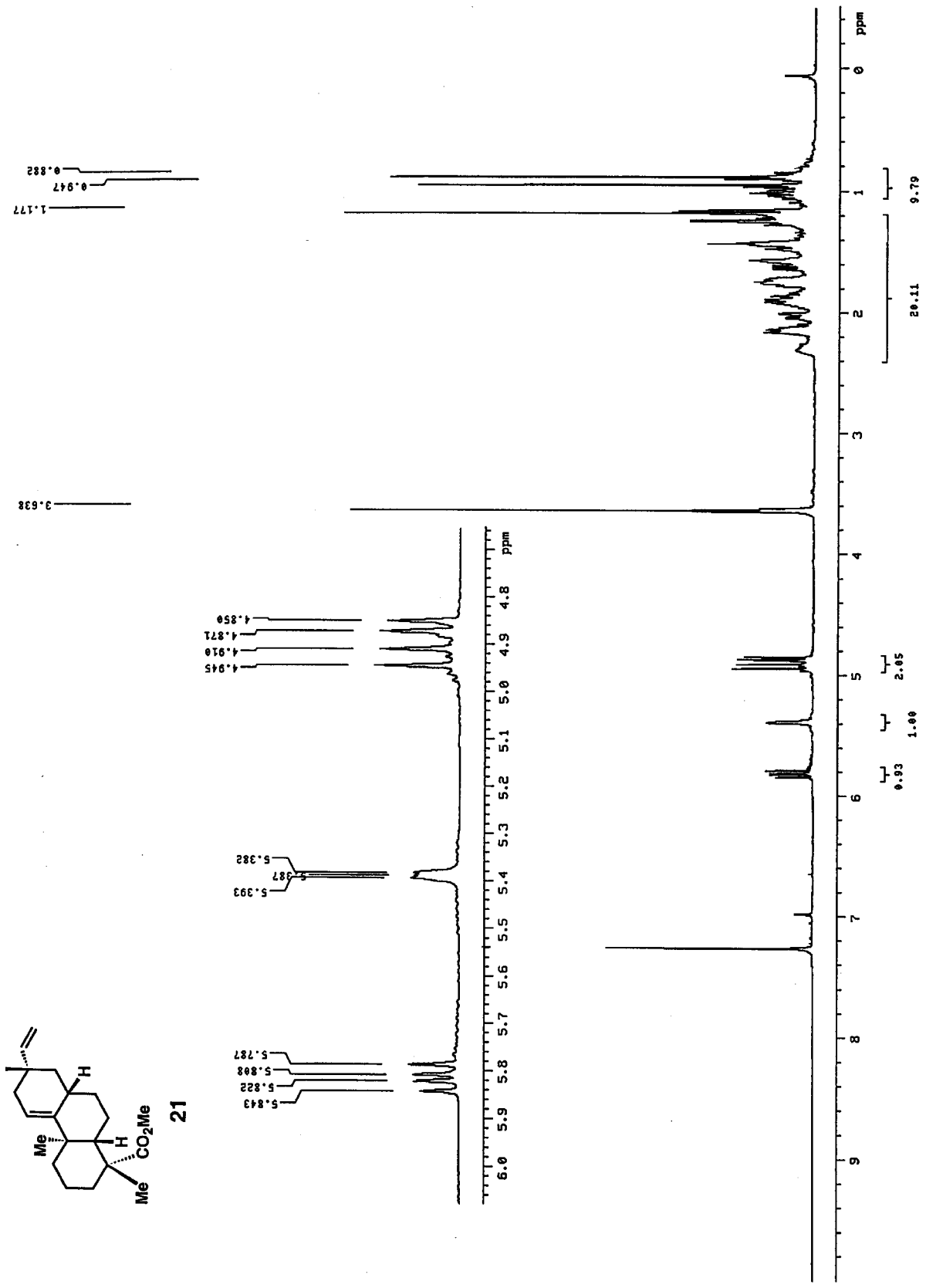

SPh
Me
H
Me
MeO2C
4
0.877
1.209
3.643
5.723
5.716
6.348
6.377
6.421
6.451
ppm
7.02
2.02
1.00
3.21
3.14
3.82
2.55
2.46
1.47
4.27
3.57

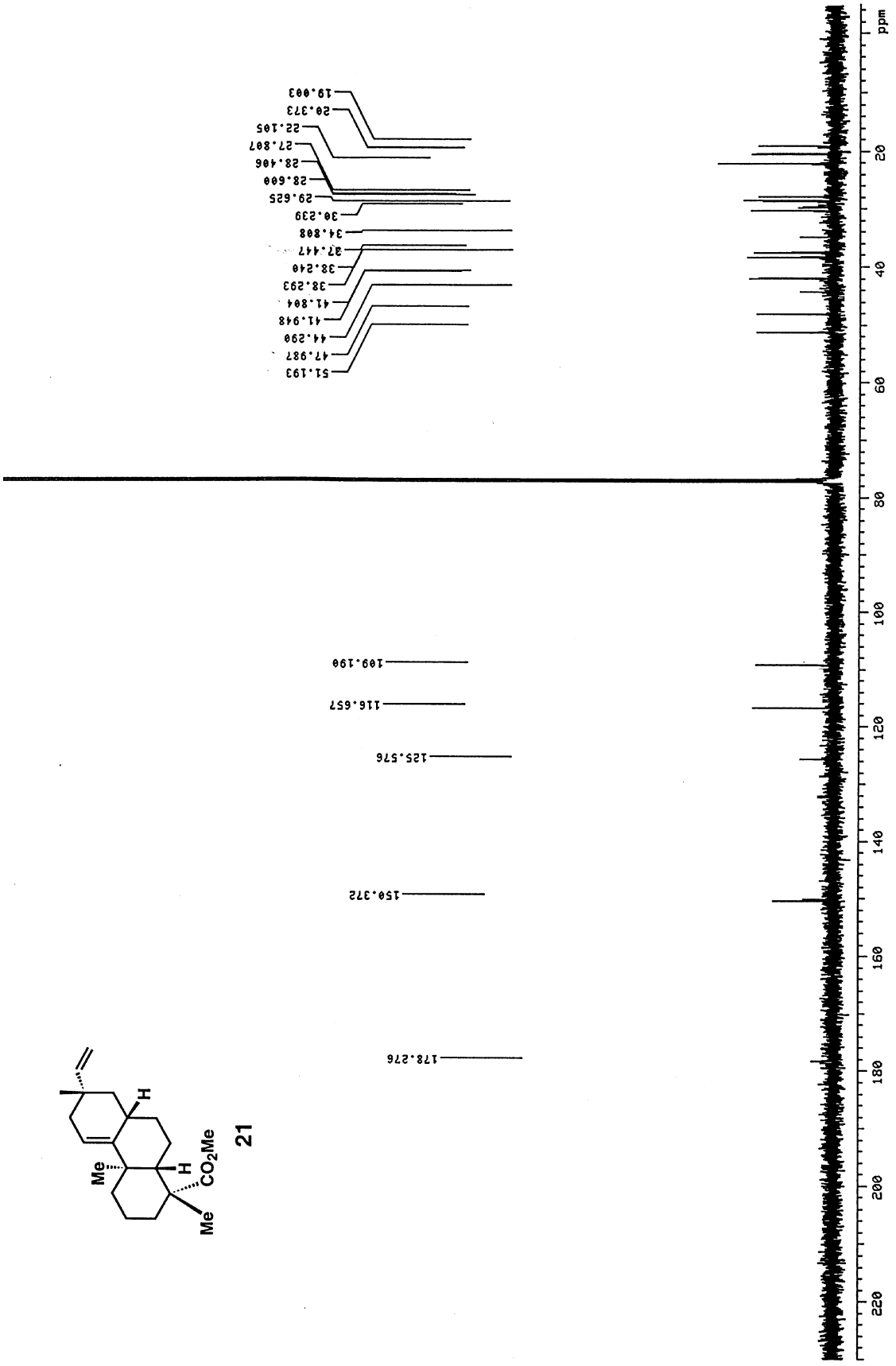

18.269
19.280
20.138
27.701
28.300
30.204
37.341
37.689
43.724
51.105
52.805
120.944
123.440
126.241
127.539
128.640
128.992
129.092
133.704
177.959
ppm
20
40
60
80
100
120
140
160
180
200
220
SPh
Me
H
Me
MeO2C
4

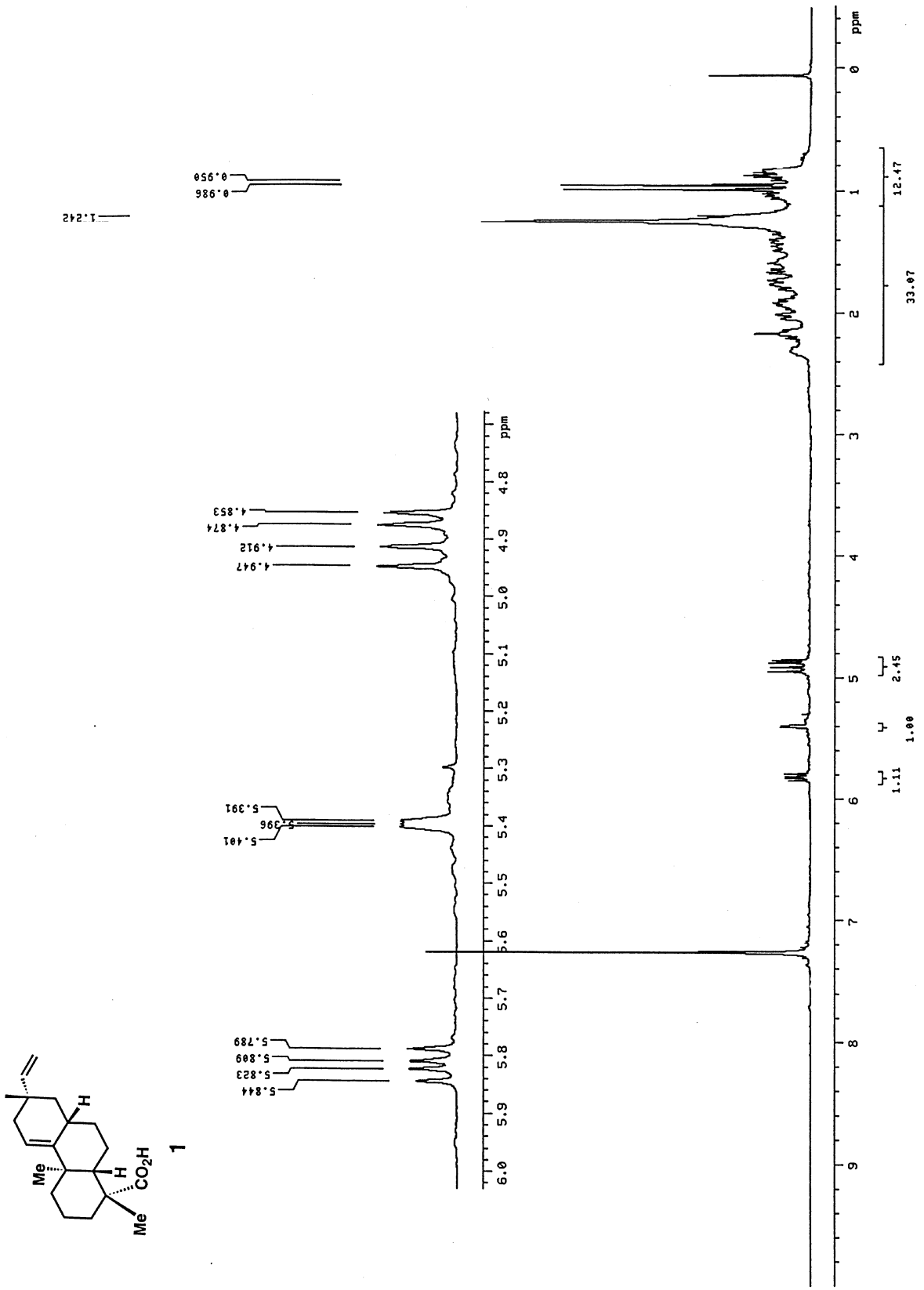

0.860
1.156
3.616
ppm
0
1
2
3
4
5
6
7
8
9
2.95
8.65
8.15
8.77
2.94
1.00
Me
OH
H
Me
H
Me
MeO2C

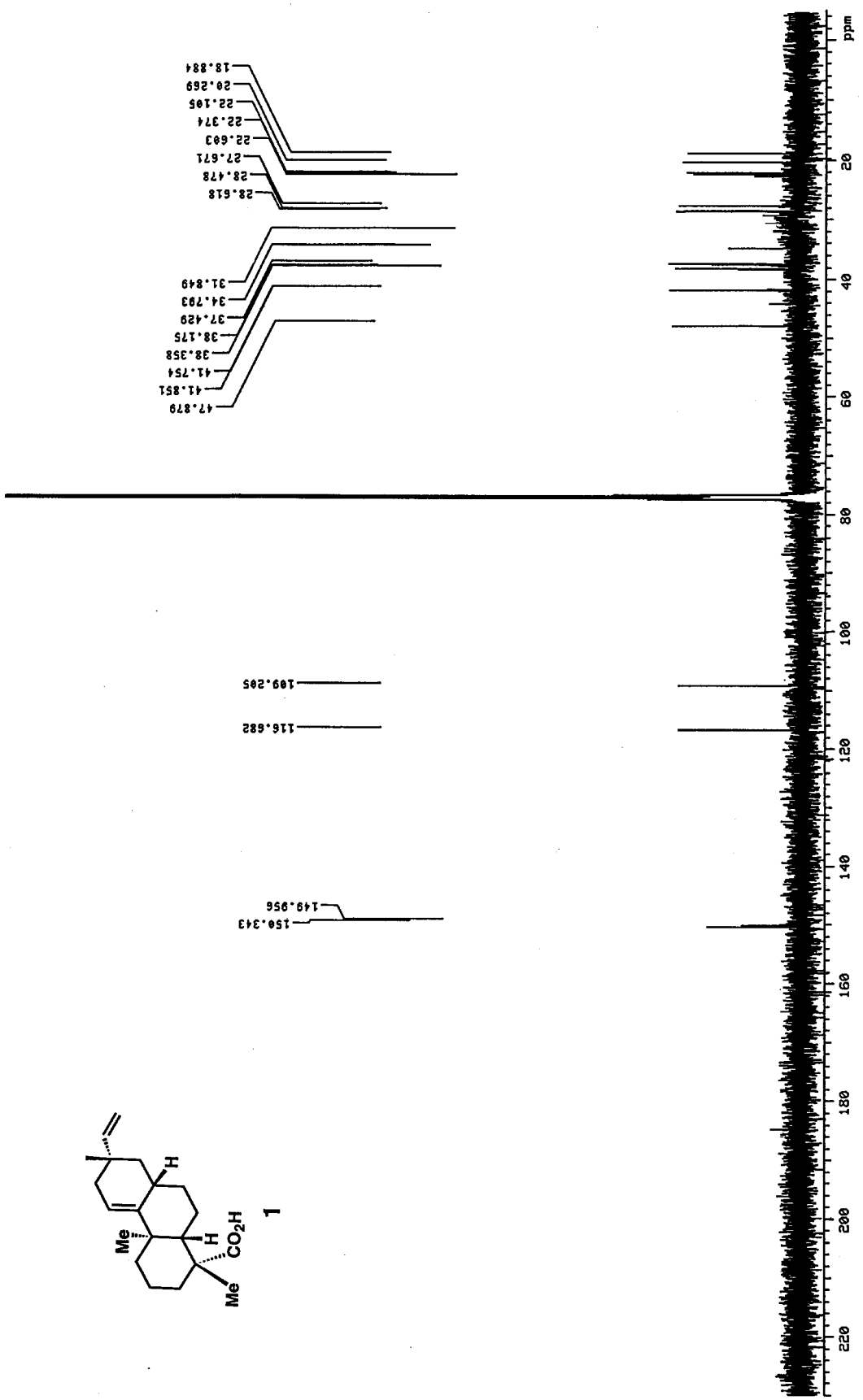
20


178.244
150.382
116.427
73.604
51.161
47.897
44.257
41.894
38.856
38.232
38.200
34.327
33.875
28.341
28.223
27.832
22.137
20.308
20.061
18.949
Me
OH
H
Me
H
Me
MeO2C
20
ppm
20
40
60
80
100
120
140
160
180
200
220


21
Me
H
H
CO2Me
Me
5.843
5.822
5.808
5.787
5.393
5.387
5.382
4.945
4.918
4.871
4.858
3.638
1.177
0.947
0.882
ppm
9.79
20.11
2.05
1.00
0.93


ppm
20
40
60
80
100
120
140
160
180
200
220
19.003
20.373
22.105
27.807
28.406
28.600
29.625
30.239
34.808
37.447
38.240
38.293
41.804
41.948
44.290
47.987
51.193
109.190
116.657
125.576
150.372
178.276
H
Me
H
CO2Me
Me
21


Me
H
H
CO2H
Me
1
5.844
5.823
5.809
5.789
5.401
5.396
5.391
4.947
4.912
4.874
4.853
1.242
0.986
0.950
ppm
6.0
5.9
5.8
5.7
5.6
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
ppm
0
1
2
3
4
5
6
7
8
9
1.11
1.00
2.45
33.07
12.47


18.884
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22.105
22.374
22.603
27.671
28.478
28.618
31.849
34.793
37.429
38.175
38.358
41.754
41.851
47.879
116.682
149.956
150.343
ppm
20
40
60
80
100
120
140
160
180
200
220
Me
H
H
CO2H
Me
1