Supporting Information

Facile carbohydrate-based stereocontrolled divergent synthesis of (+)-pericosines A and B

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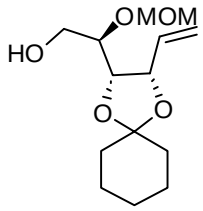
Experimental Section

General:

All melting points are uncorrected. All reactions were carried out in anhydrous solvents unless specified. THF and diethyl ether were distilled from sodium-benzophenone under argon. DCM and hexanes were distilled from CaH_2. ^1H NMR spectra were obtained at 300 or 400 MHz (as indicated). ^{13}C NMR spectra were obtained at 75.5 or 100.6 MHz, using a Bruker NMR spectrometer. Chemical shifts (δ) are reported in *ppm* relative to $CDCl_3$ (7.26 and 77.0 ppm). Mass spectra (EI-MS) and high resolution mass spectra (HRMS) were determined on a Finnigan/Thermo Quest MAT 95XL mass spectrometer. Infrared spectra were recorded using a JASCO FT/IR 410 spectrometer. Flash Column chromatography was performed using MN silica gel 60 (70–230 mesh) purchased from Macherey-Nagel.

Synthesis of Pericosine A

(*R*)-2-(methoxymethoxy)-2-((2*S*,3*S*)-3-vinyl-1,4-dioxaspiro[4.5]decan-2-yl)ethanol 11



11

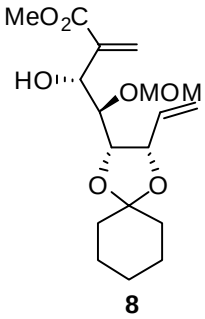
A solution of diol **6** (2.84 g, 12.46 mmol) in dry DCM was cooled to 0 °C, then to it imidazole (1.86 gm, 27.46 mmol) was added and stirred for 30 min at the same temperature, after which TBSCl (2.81 gm, 18.70 mmol) was added. The reaction mixture was stirred for 1 h at 0 °C and quenched by adding water to it. Extraction was done by DCM; combined organic layer was dried over anhydrous Na_2SO_4. Organic solvent was evaporated under reduced pressure to afford crude product which was used for the next reaction without purification.

To a solution of TBS ether (300 mg, 0.87 mmol) in dry DCM (10 mL) was added Hunig's base (381 μL, 2.18 mmol) and MOMBr (143 μL, 1.75 mmol), the resulting mixture was refluxed for 24 h. Reaction was cooled to rt and quenched by adding water to it. Extraction was done by DCM, combined organic layer was dried over anhydrous Na_2SO_4 and concentrated to obtain crude product which was used for next reaction without further purification.

The THF (10 mL) solution of MOM ether was added TBAF (1 M solution in THF, 2 mL). The reaction mixture was stirred for 45 min at rt and quenched by adding saturated

aqueous solution of $NaHCO_3$. Extraction was done by DCM, dried over anhydrous $MgSO_4$, and combined organic layer was evaporated under reduced pressure to give crude product which was purified by column chromatography [SiO_2, ethyl acetate-hexanes (1:5)] to furnish alcohol **11** (215 mg, 0.87 mmol, 91%) as gum. $[\alpha]_D^{24.2}$ +61.8 (*c* 0.17, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.81 (m, 1H), 5.31 (d, J = 17.1 Hz, 1H), 5.15 (d, J = 10.5 Hz, 1H), 4.57 (m, 3H), 4.06 (m, 1H), 3.80 (d, J = 12.1 Hz, 1H), 3.58 (m, 1H), 3.47 (m, 1H), 3.35 (s, 3H), 1.55 (m, 8H), 1.32 (m, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 134.0, 117.3, 109.3, 97.5, 81.1, 78.2, 76.5, 63.8, 55.8, 37.5, 34.7, 25.0, 23.9, 23.6. IR (neat) ν_{max} 3461, 2934, 2857, 1448, 1366, 1108, 1034, 927, 847 cm^{-1}. MS-EI (*m/z*) 272 (M^+, 10), 167 (56), 138 (42), 125 (59), 81 (56), 55 (100). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_{14}H_{24}O_5$, 272. 1624; found 272.1627.

(3*R*,4*R*)-methyl 3-hydroxy-4-(methoxymethoxy)-2-methylene-4-((2*S*,3*S*)-3-vinyl-1,4-dioxaspiro[4.5]decan-2-yl)butanoate 8



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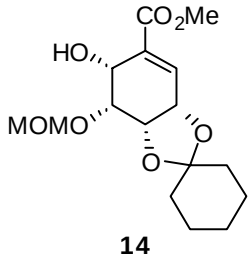
Preparation of aldehyde 7: A solution of DCM (30 mL) and oxalyl chloride (634 μL, 7.34 mmol) was placed under inert atmosphere and DMSO (1.04 mL, 14.68 mmol) in DCM (5 mL) was added at –78 °C. The reaction mixture was stirred for 15 min at the same temperature and alcohol (1.00 g, 3.67 mmol) was added within 5 min. Stirring was continued for an additional 40 min and then triethyl amine (5.11 mL, 36.72 mmol) was added. The reaction mixture was warmed to rt and additionally stirred overnight. Water (50 mL) was added to it, aqueous layer was separated and extracted with fresh portion of DCM and combined organic layer was dried over anhydrous Na_2SO_4 and concentrated to get crude aldehyde **7**, which was used for next reaction without further purification.

Preparation of Iodoacrylate- The mixture of I_2 (2.50 g, 1.01 mmol) and DABCO (1.25 g, 1.14 mmol) was dissolved in DMF (2.5 mL) and stirred for 30 min at rt, then to it methyl acrylate (875 μL, 9.72 mmol) was added and stirred for additional 30 min. Reaction was quenched by adding water to it, the reaction mass was added to the pentane (50 mL) with good stirring and to this saturated aqueous solution of sodium thiosulfate (30 mL) was added. The pentane layer was separated; the remaining aqueous layer was

extracted by additional pentane. Combined organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated to give crude iodoacrylate which was used for next reaction without further purification.

To a THF (6 mL) solution of the ligand **12** (758 mg, 2.20 mmol) added anhydrous $CrCl_2$ (266 mg, 2.20 mmol), anhydrous $NiCl_2$ (140 mg, 1.1 mmol) inside the glove box and the resulting mixture was stirred for 1 h. To this solution successively added a THF (2 mL) solution of the aldehyde **7** (270 mg, 1.0 mmol) and iodoacrylate (422 mg, 2 mmol) in THF(2 mL) and the mixture left for stirring at room temperature for 3 h at which time TLC indicated complete consumption of the starting material. Saturated bicarbonate solution was added and the solution further stirred for 30 min and then mixture was filtered to get rid of all the solid materials. The filtrate was diluted with EtOAc (15 mL) and the organic phase was washed with brine (3 × 10 mL) followed by drying ($MgSO_4$) and concentration under reduced pressure to give a crude residue. The residue was purified by column chromatography [SiO_2, ethyl acetate-hexanes (1:5)] to furnish **8** (300 mg, 0.84 mmol, 84%) as oil. $[\alpha]_D^{24.2}$ +13.4 (*c* 0.25, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.30 (s, 1H), 5.94 (m, 2H), 5.32 (d, J = 17.2 Hz, 1H), 5.22 (d, J = 10.2 Hz, 1H), 4.78 (m, 1H), 4.66 (s, 2H), 4.58 (dd, J = 7.0 and 6.4 Hz, 1H), 4.15 (dd, J = 8.4 and 5.9 Hz 1H), 3.92 (dd, J = 8.3 and 4.0 Hz, 1H), 3.76 (s, 3H, merged with a multiplet, 1H)), 3.37 (s, 3H), 3.33 (s, 3H), 1.52 (m, 10H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 167.1, 138.8, 135.0, 126.3, 117.9, 109.4, 98.5, 80.7, 78.9, 76.2, 71.4, 56.4, 51.8, 37.9, 34.7, 25.1, 23.9, 23.8. IR (neat) ν_{max} 2931, 2854, 1723, 1439, 1365, 1241, 1149, 1131, 1110, 1085, 1033, 937 cm^{-1}. MS-EI (*m/z*) 356 (M^+, 2), 294 (39), 251 (72), 209 (40), 185 (46), 179 (50), 167 (86), 157 (60). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_{18}H_{28}O_7$ 356.1835, found 356.1833.

(3a*S*,4*R*,5*R*,7a*S*)-methyl-5-hydroxy-4-(methoxymethoxy)-3a,4,5,7a-tetrahydrospiro[benzo[*d*][1,3]dioxole-2,1'-cyclohexane]-6-carboxylate 14

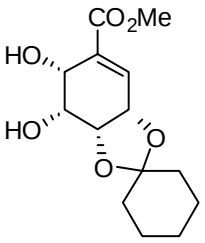


14

To a solution of Compound **8** (50 mg, 0.14 mmol) in degassed toluene (60 mL) added a Hoveyda-Grubbs (II) catalyst (6.3 mg, 10.2 μmol) and the resulting solution was refluxed for 18 h. The solvent were evaporated under reduced pressure and the residue was purified by column chromatography [SiO_2, ethyl acetate-

hexanes (1:5)] to furnish **14** (38 mg, 0.12 mmol, 86%) as a thick gum. $[\alpha]_D^{24.2}$ –34.9 (*c* 0.2, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79 (d, *J* = 2.6 Hz, 1H), 4.97 (d, *J* = 7.0 Hz, 1H), 4.83 (d, *J* = 7.0 Hz, 1H), 4.75 (m, 2H), 4.65 (b, *J* = 5.4 Hz, 1H), 3.81 (s, 3H), 3.80 (m, 1H), 3.52 (d, *J* = 10.2 Hz, 1H), 3.46 (s, 3H), 1.61 (m, 8H). 1.37 (m, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 166.0, 135.9, 131.8, 112.1, 95.9, 75.5, 73.0, 71.2, 63.3, 55.9, 52.4, 37.6, 35.7, 24.8, 23.8, 23.6. IR (neat) ν_{max} 3521, 2934, 2858,1723, 1439, 1367, 1303, 1247, 1146, 1107, 1082, 1056,1032, 935 cm^{-1}. MS-EI (*m/z*) 328 (M^+, 2), 285 (16), 152 (4), 137 (5), 99 (4), 84 (10). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_{16}H_{24}O_7$ 328.1522, found 328.1529.

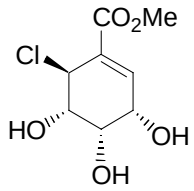
(3a*R*,4*R*,5*R*,7a*S*)-methyl 4,5-dihydroxy-3a,4,5,7a-tetrahydrospiro[benzo[*d*][1,3]dioxole-2,1'-cyclohexane]-6-carboxylate 10



10

A mixture of MOM ether **14** (150 mg, 0.45 mmol) and zirconocene dichloride (133 mg, 0.45 mmol) in dry isopropanol (20 mL) was heated at reflux. After the completion of reaction (TLC analysis), it was cooled to room temperature, the reaction mixture was filtered through short celite pad and concentrated under reduced pressure. The residue was treated with EtOAc (10 mL). The organic layer was washed with water (10 mL), brine (10 mL), dried over anhydrous Na_2SO_4 and evaporated under reduced pressure to give product. The crude product was purified by PTLC (50% ethyl acetate-hexanes) with three times elution afforded diol **10** (108 mg, 0.38 mmol, 83%) as a colorless gum. $[\alpha]_D^{24.1}$ –10.2 (*c* 0.25, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 (d, *J* = 2.9 Hz, 1H), 4.71 (m, 1H), 4.59 (m, 2H), 3.82 (s, 3H merged with a multiplet, 1H), 3.21 (d, *J* = 9.8 Hz, 1H), 3.10 (d, *J* = 11.4 Hz, 1H), 1.59 (m, 8H), 1.24 (m, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 165.9, 136.8, 131.3, 111.9, 72.6, 67.1, 65.3, 52.4, 37.7, 35.4, 29.7, 24.7, 23.8, 23.5. IR (neat) ν_{max} 3467, 2923, 2851, 1720, 1437, 1245, 1113, 1031, 935 cm^{-1}. MS-EI (*m/z*) 284 (M^+, 19), 255 (21), 241 (100), 137 (41), 109 (31), 55 (41). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_{14}H_{20}O_6$, 284.1260, found 284.1258.

(3*S*,4*S*,5*S*,6*S*)-methyl 6-chloro-3,4,5-trihydroxycyclohex-1-enecarboxylate 1



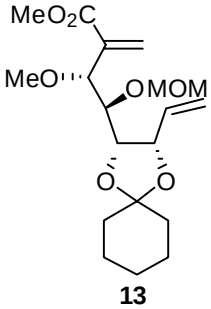
1: (+) Pericosine A

1-Chlorocarbonyl-1-methylethyl acetate (8.80 μL, 0.06 mmol) was added dropwise at 0 °C, under a nitrogen atmosphere, to a solution of diol **10** (11.70 mg, 0.04 mmol) in dry MeCN (5 mL). The mixture was stirred for 15 min at 0 °C and then at room temperature for further 2 h. The solvent was removed under reduced pressure, the residue extracted with EtOAc, washed once with 3% aqueous $NaHCO_3$, dried over Na_2SO_4, and concentrated to give the crude chloroacetate. The crude product was used for next reaction without further purification.

A solution of chloroacetate in dry MeOH (5 mL) was treated with two drops of acetyl chloride and the mixture stirred at room temperature for 12 h. The solvent was removed under reduced pressure and the residue purified by PTLC (75% ethyl acetate-hexanes) to give pericosine A **1** (7.86 mg, 0.03 mmol, 86%) as an oil. $[\alpha]_D^{26.5}$ +105.5 (*c* 0.30, EtOH); lit.[4b] $[\alpha]_D^{20}$ +94.3 (*c* 0.26, EtOH) (synthetic); lit.[4d] $[\alpha]_D^{20}$ +104 (*c* 0.04, EtOH) (synthetic); lit.[5] $[\alpha]_D^{20}$ +100 (*c* 0.94, EtOH) (synthetic); lit.[1,2a] $[\alpha]_D$ +57 (*c* 3.16, EtOH) (natural product). ^{1}H NMR (400 MHz, Acetone-d_6) δ 6.89 (d, J = 4.0 Hz, 1H), 4.88 (m, 2H), 4.36 (m, 1H), 4.18 (d, J = 6.0 Hz, 1H), 4.12 (d, J = 8.6 Hz, 2H), 4.04 (d, J = 3.2 Hz, 1H), 3.77 (s, 3H). ^{13}C NMR (100.6 MHz, Acetone-d_6) δ 165.3, 141.0, 129.6, 74.8, 67.7, 66.2, 56.9, 51.5. IR (neat) ν_{max} 3390, 2923, 1717, 1437, 1264, 1094, 806 cm^{-1}. MS-EI (*m/z*) 222 (M^+, 0.07), 157 (34), 131 (63), 109 (50), 95 (87), 53 (100). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_8H_{11}ClO_5$, 222.0295, found 222.0293.

Synthesis of Pericosine B

(3*R*,4*R*)-methyl 3-methoxy-4-(methoxymethoxy)-2-methylene-4-((2*S*,3*S*)-3-vinyl-1,4-dioxaspiro[4.5]decan-2-yl)butanoate 13

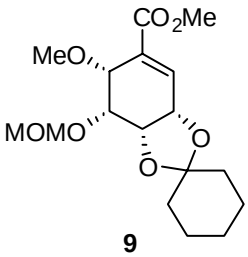


13

To a solution of compound **8** (90 mg, 0.25 mmol) in dry ether (10 mL) added a MeI (0.5 mL, excess) and KOH (10 mg) and the resulting mixture was refluxed for 2 h. All the solid materials were removed by filtration over celite and the filtrate was concentrated to afford a crude residue. The residue was purified by column chromatography [SiO_2,

ethyl acetate-hexanes (1:9)] to furnish **13** (70mg, 0.19 mmol, 76%) as a thick gum. $[\alpha]_D^{24.0}$ –8.4 (*c* 0.25, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.39 (s, 1H), 5.99 (m, 1H), 5.92 (s, 1H), 5.30 (d, *J* = 17.2 Hz, 1H), 5.20 (d, *J* = 10.7 Hz, 1H), 4.74 (d, *J* = 6.3 Hz, 1H), 4.67 (d, *J* = 6.3 Hz, 1H), 4.58 (t, *J* = 6.6 Hz, 1H), 4.47 (d, *J* = 3.6 Hz, 1H). 4.25 (dd, *J* = 7.2 and 6.2 Hz, 1H). 3.92 (dd, *J* = 7.5 and 3.7 Hz, 1H), 3.76 (s, 3H), 3.36 (s, 3H), 3.33 (s, 3H), 1.46 (m, 10H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 166.9, 137.1, 135.5, 127.2, 117.3, 109.0, 97.9, 80.6, 78.9, 77.9, 75.9, 57.6, 56.3, 51.8, 37.9, 34.7, 25.2, 24.0, 23.8. IR (neat) ν_{max} 2931, 2854, 1723, 1439, 1365, 1241, 1149, 1131, 1110, 1085, 1033, 937 cm^{-1}. MS-EI (*m/z*) 370 (M^+, 0.5), 250 (5), 207 (12), 142 (20), 129 (100), 75 (90). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_{19}H_{30}O_7$, 370.1992, found 370.1998.

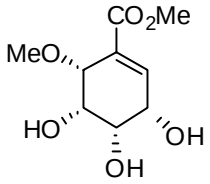
(3a*S*,4*R*,5*R*,7a*S*)-methyl 5-methoxy-4-(methoxymethoxy)-3a,4,5,7a-tetrahydrospiro[benzo[d][1,3]dioxole-2,1'-cyclohexane]-6-carboxylate 9



9

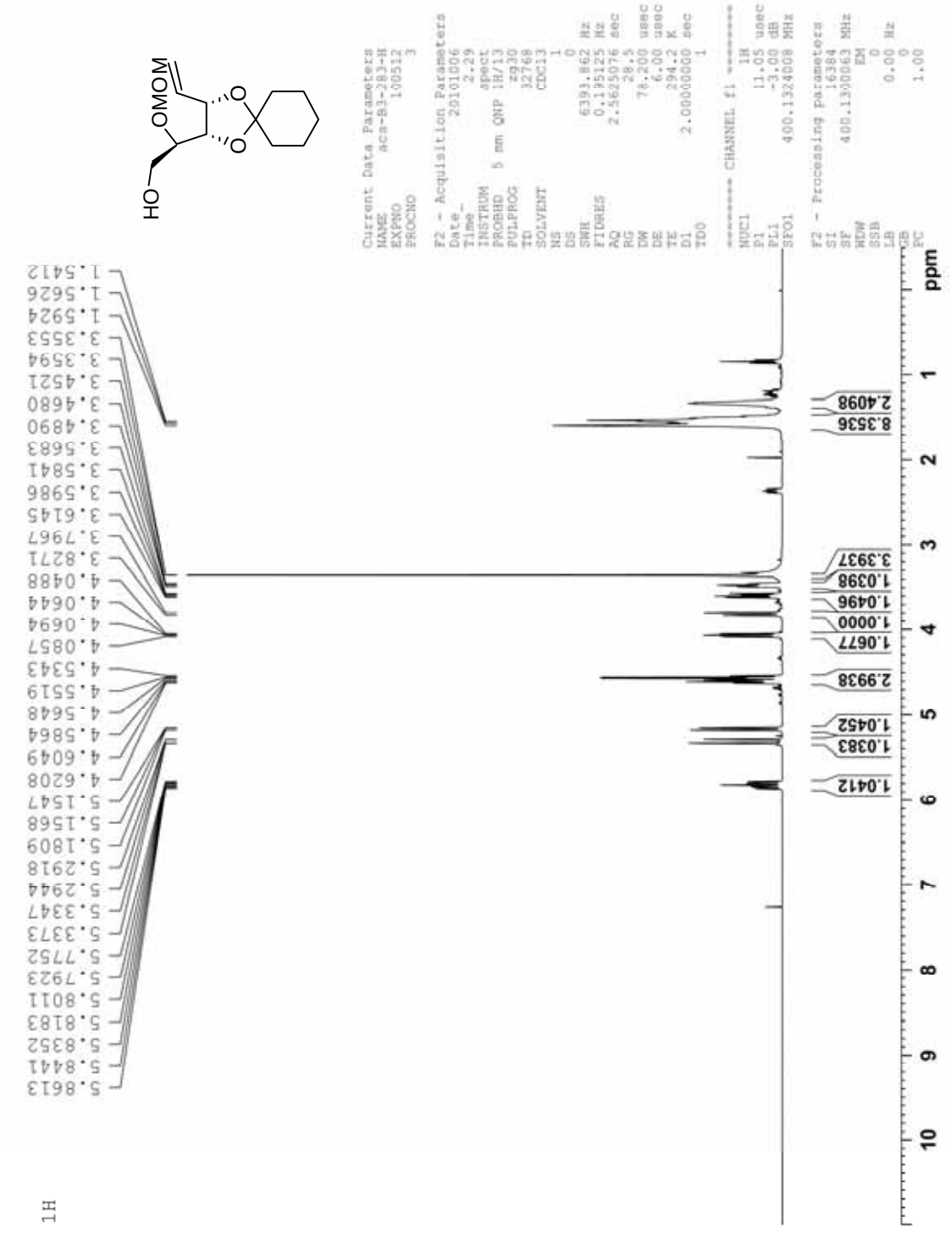
To a solution of compound **13** (38 mg, 0.102 mmol) in degassed toluene (40 mL) added a Hoveyda-Grubbs (II) catalyst (6.3 mg, 10.2 μmol) and the resulting solution was refluxed for 18 h. The solvent were evaporated under reduced pressure and the residue was purified by column chromatography [SiO_2, ethyl acetate-hexanes (1:5)] to furnish **9** (30 mg, 0.88 mmol, 86%) as a thick gum. $[\alpha]_D^{24.1}$ –18.5 (*c* 0.28, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.78 (d, *J* = 2.8 Hz, 1H), 4.85 (s, 2H), 4.64 (m, 1H), 4.56 (m, 1H), 4.35 (d, *J* = 4.1 Hz, 1H), 3.79 (s, 3H merged with a multiplet, 1H), 3.57 (s, 3H), 3.47 (s, 3H). 1.54 (m, 10 H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 166.5, 137.3, 130.2, 112.1, 95.1, 72.8, 72.6, 71.8, 61.0, 55.8, 52.1, 37.2, 35.7, 24.9, 23.8, 23.7. IR (neat) ν_{max} 2931, 2854, 1723, 1439, 1365, 1241, 1149, 1131, 1110, 1085, 1033, 937 cm^{-1}. MS-EI (*m/z*) 342 (M^+, 13), 299 (48), 183 (10), 151 (20), 123 (8), 55 (12). HRMS-EI (*m/z*) $[M]^+$ calcd. for $C_{17}H_{26}O_7$ 342.1679, found 342.1682.

(3*S*,4*S*,5*S*,6*R*)-methyl 3,4,5-trihydroxy-6-methoxycyclohex-1-enecarboxylate 2



2:(+) Pericosine B

To a solution of compound **9** (26 mg, 0.076 mmol) in DCM (2 mL) added a mixture of TFA and water (9:1, 0.5 mL) and the resulting solution was stirred for 18 h at room temperature. The solvents were evaporated under reduced pressure and the residue was purified by preparative TLC (10% methenol-ethyl acetate, R_f 0.5) to furnish (+)-pericosine B **2** as a white solid (16 mg, 73 μmol, 96%). Mp 84–86 °C, $[\alpha]_D^{24.1}$ +30.3 (*c* 0.41, EtOH); lit.[3] $[\alpha]_D^{21}$ +30.6 (*c* 0.8, EtOH) (synthetic); lit.[5] $[\alpha]_D^{20}$ +32.1 (*c* 0.88, EtOH) (synthetic); lit.[4a] $[\alpha]_D^{25}$ –32.6 (*c* 0.35, EtOH) (synthetic enantiomer); lit.[1,2a] $[\alpha]_D$ +22.3 (*c* 0.82, EtOH) (natural product). ^{1}H NMR (400 MHz, Acetone-d_6) δ 6.71 (dd, J = 2.4 and 1.1 Hz, 1H), 4.71 (b, 1H), 4.46 (m, 2H), 4.23 (d, J = 4.3 Hz, 1H), 4.20 (m, 1H), 3.98 (m, 1H), 3.84 (m, 1H), 3.75 (s, 3H), 3.58 (s, 3H). ^{13}C NMR (100.6 MHz, Acetone-d_6) δ 166.1, 141.2, 129.6, 75.8, 71.5, 68.9, 68.4, 60.7, 51.2. IR (neat) ν_{max} 3415, 1680, 1438, 1256, 1205, 1133, 1067, 799 cm^{-1}. MS-EI (*m/z*) 219 (M^++H, 0.3), 171 (10), 158 (12), 139 (39), 126 (80), 97 (18), 69 (15), 41 (11), 17 (15). HRMS-EI (*m/z*) $[M+H]^+$ calcd. for $C_9H_{14}O_6$ 218.0790, found 219.0867.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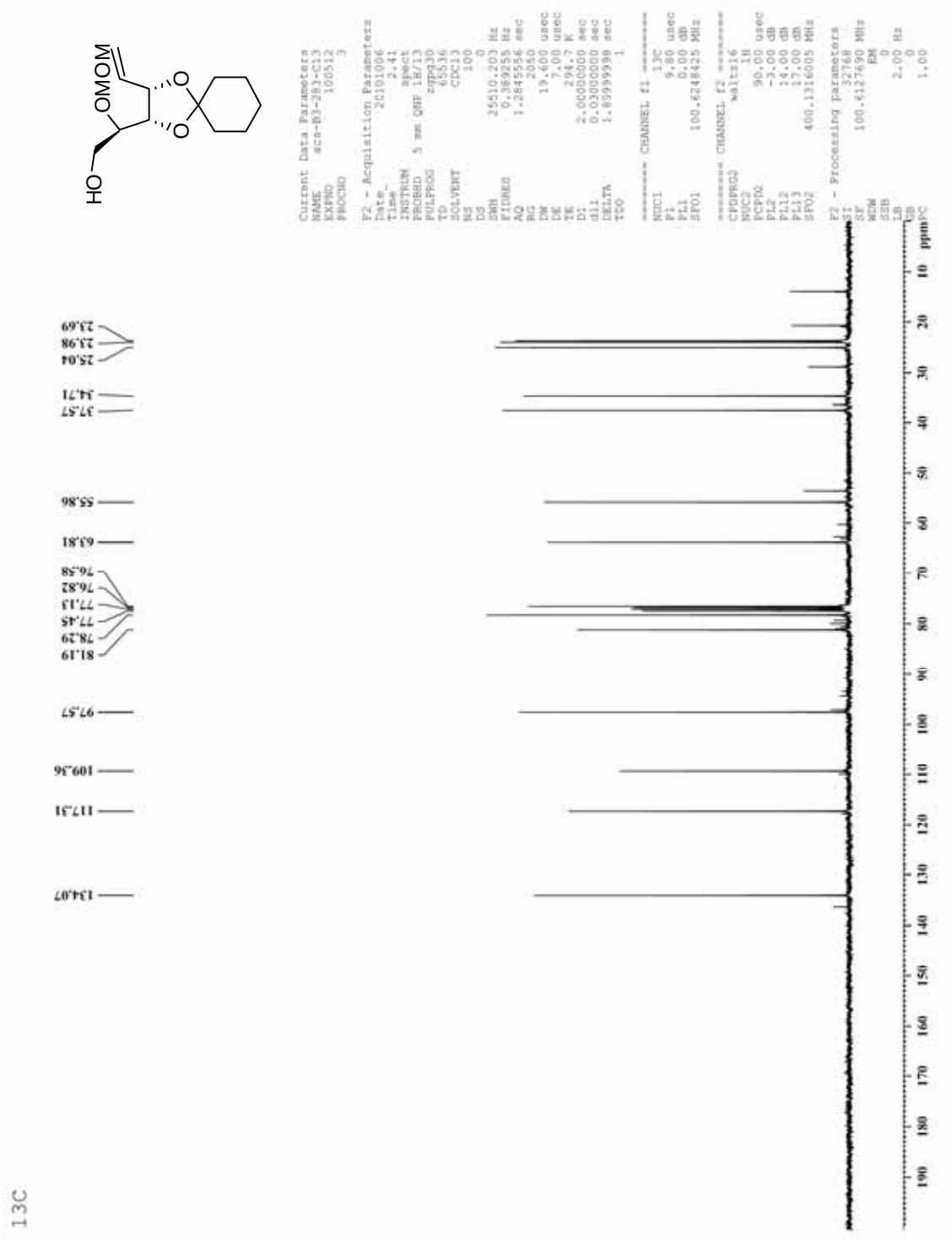
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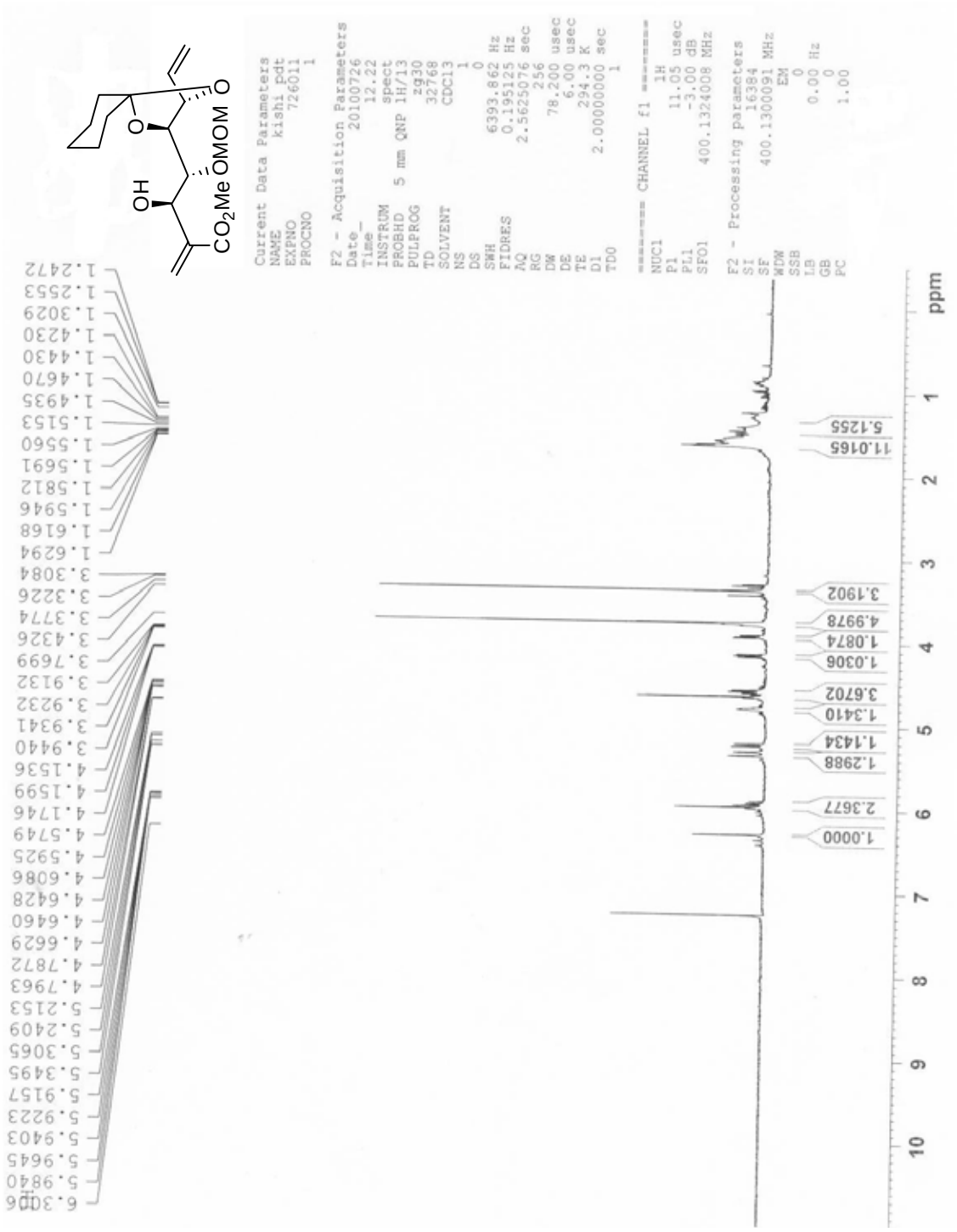
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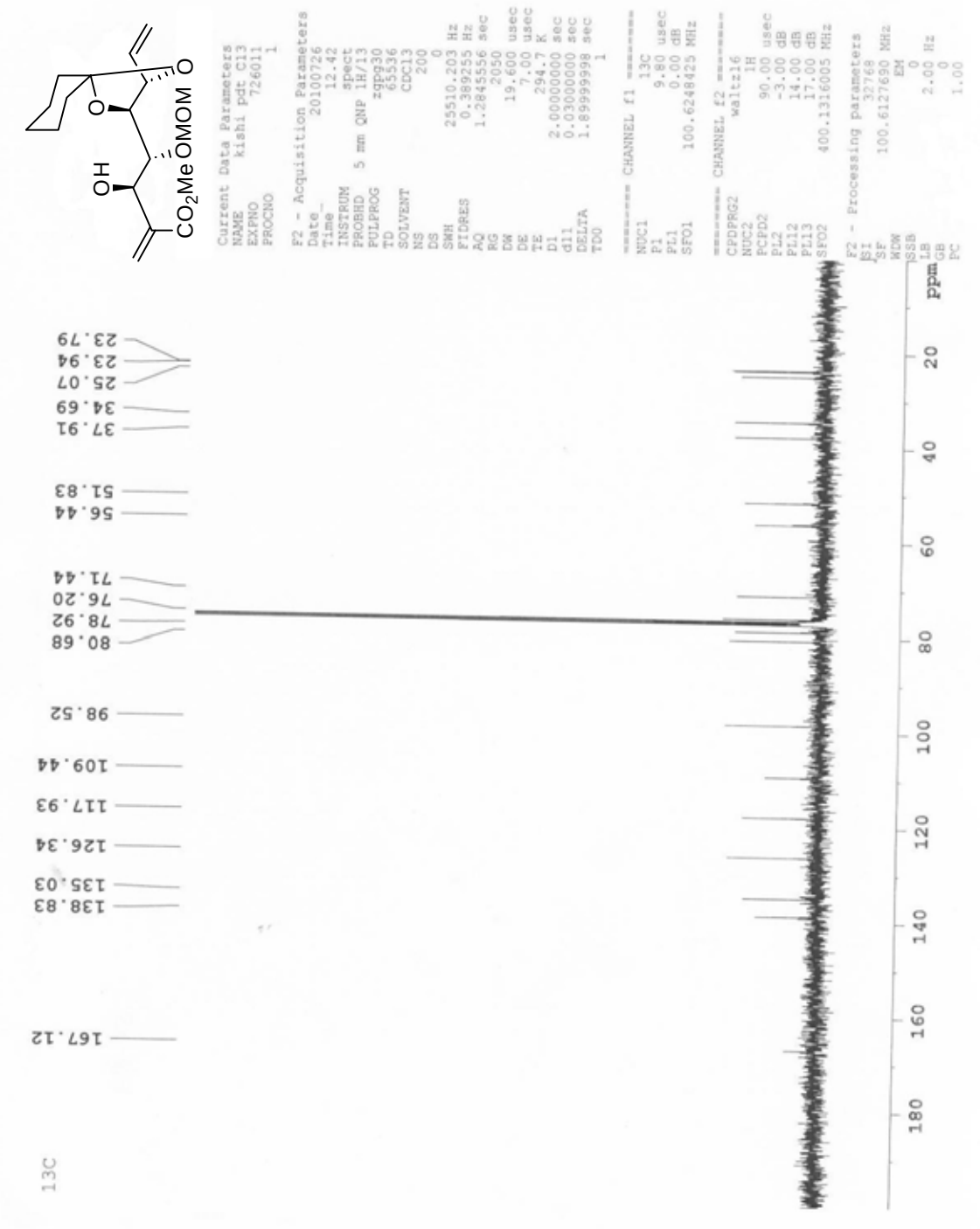
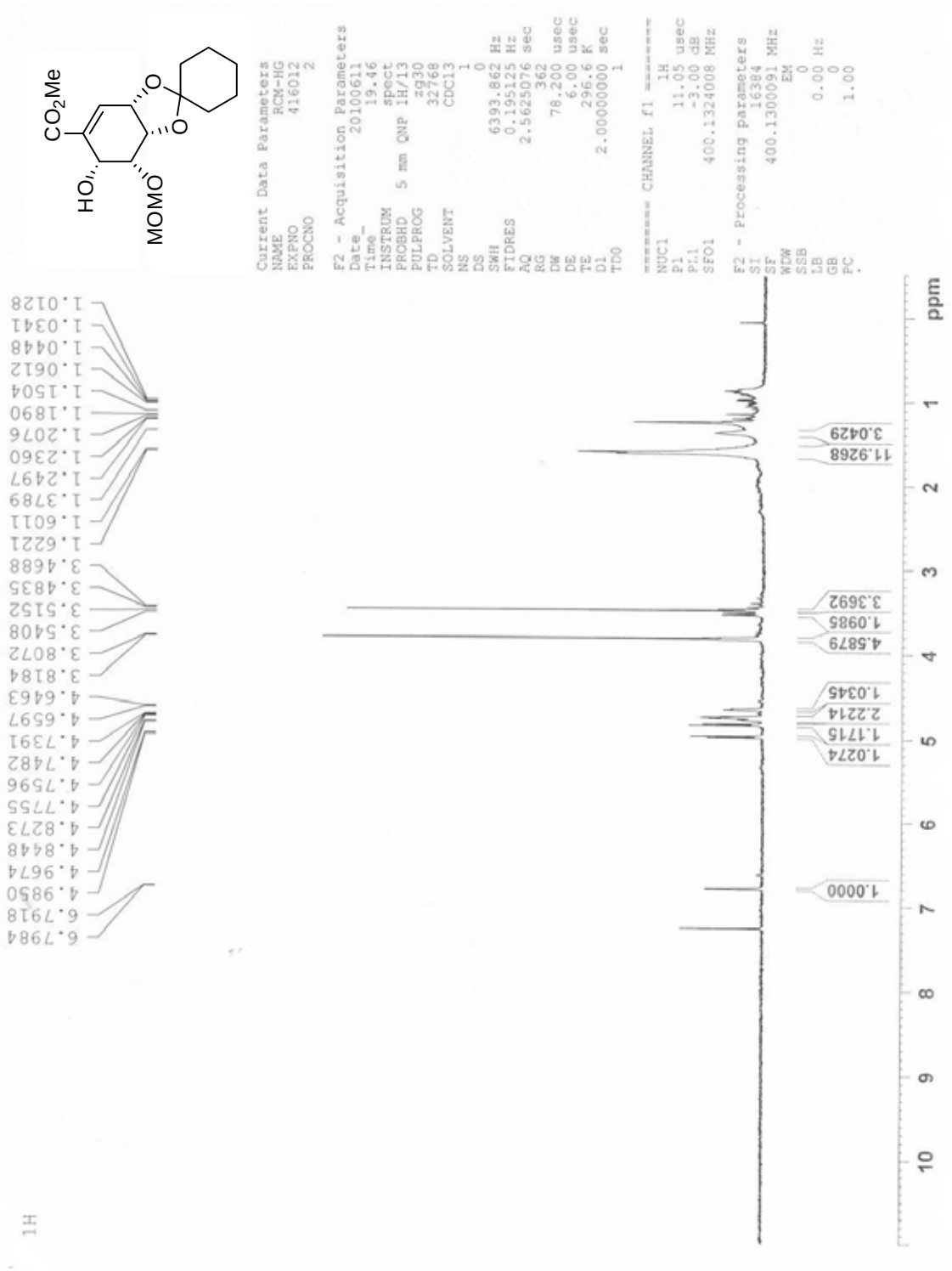
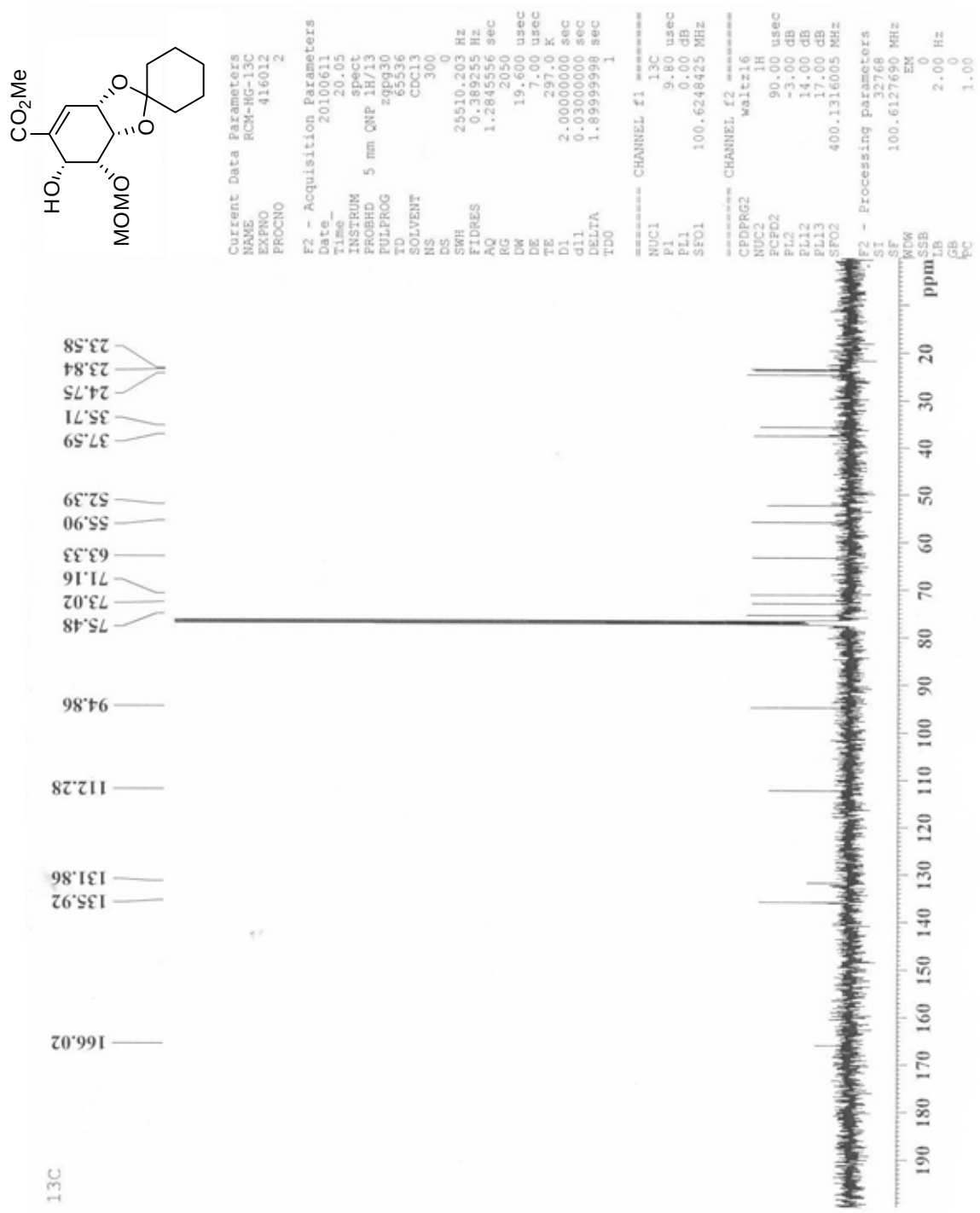
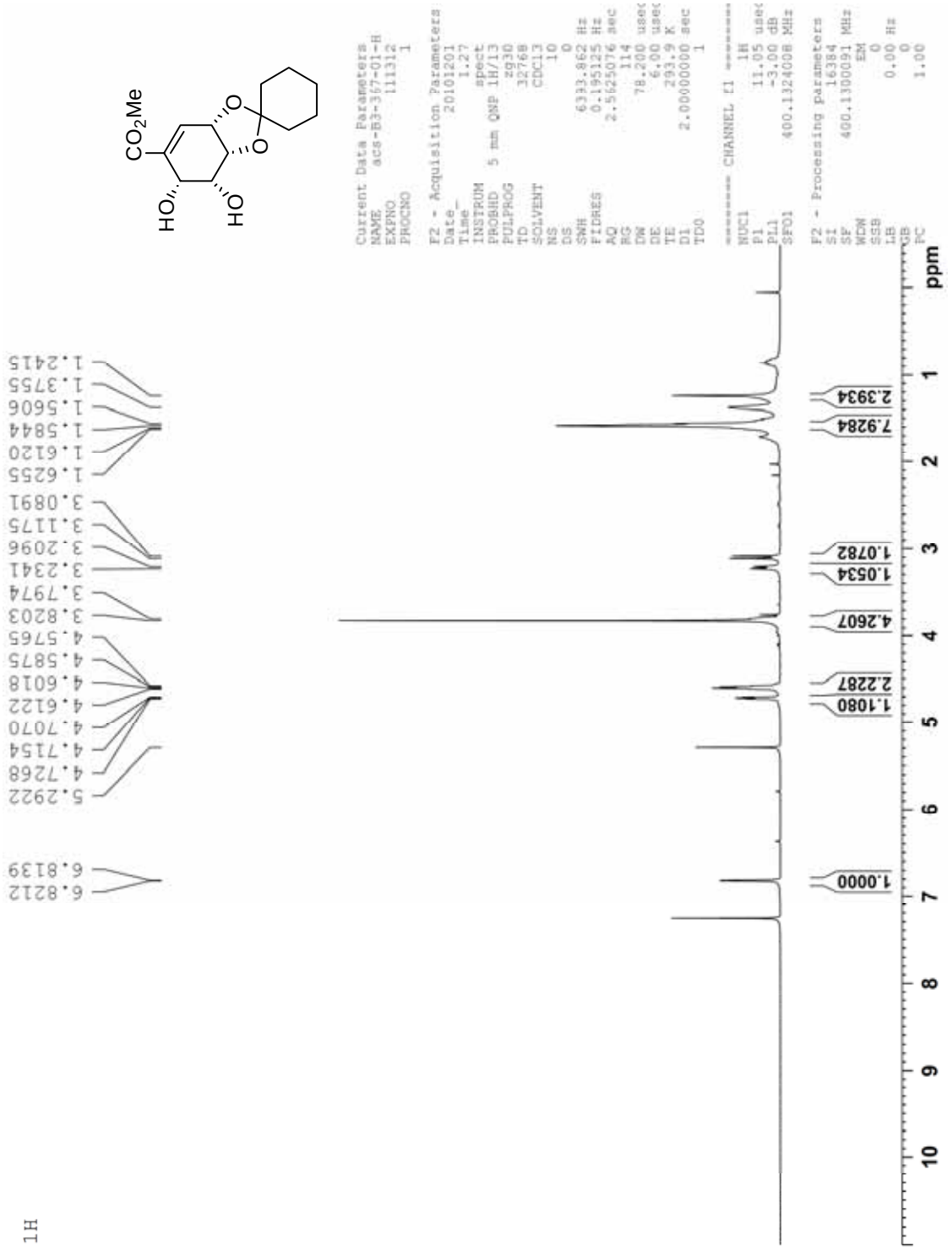
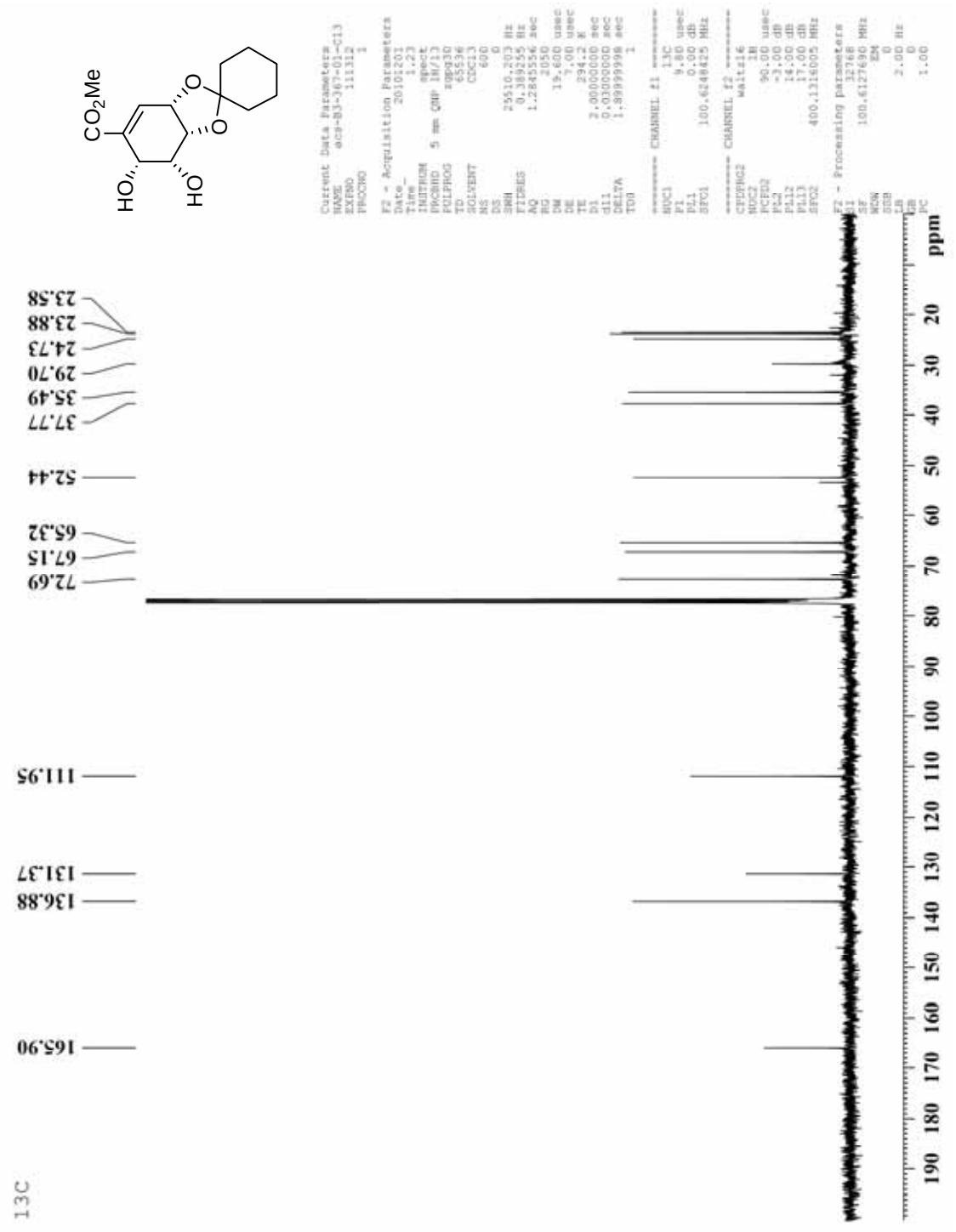
3.6702
1.3410
1.1434
1.2988
2.3677
1.0000
ppm

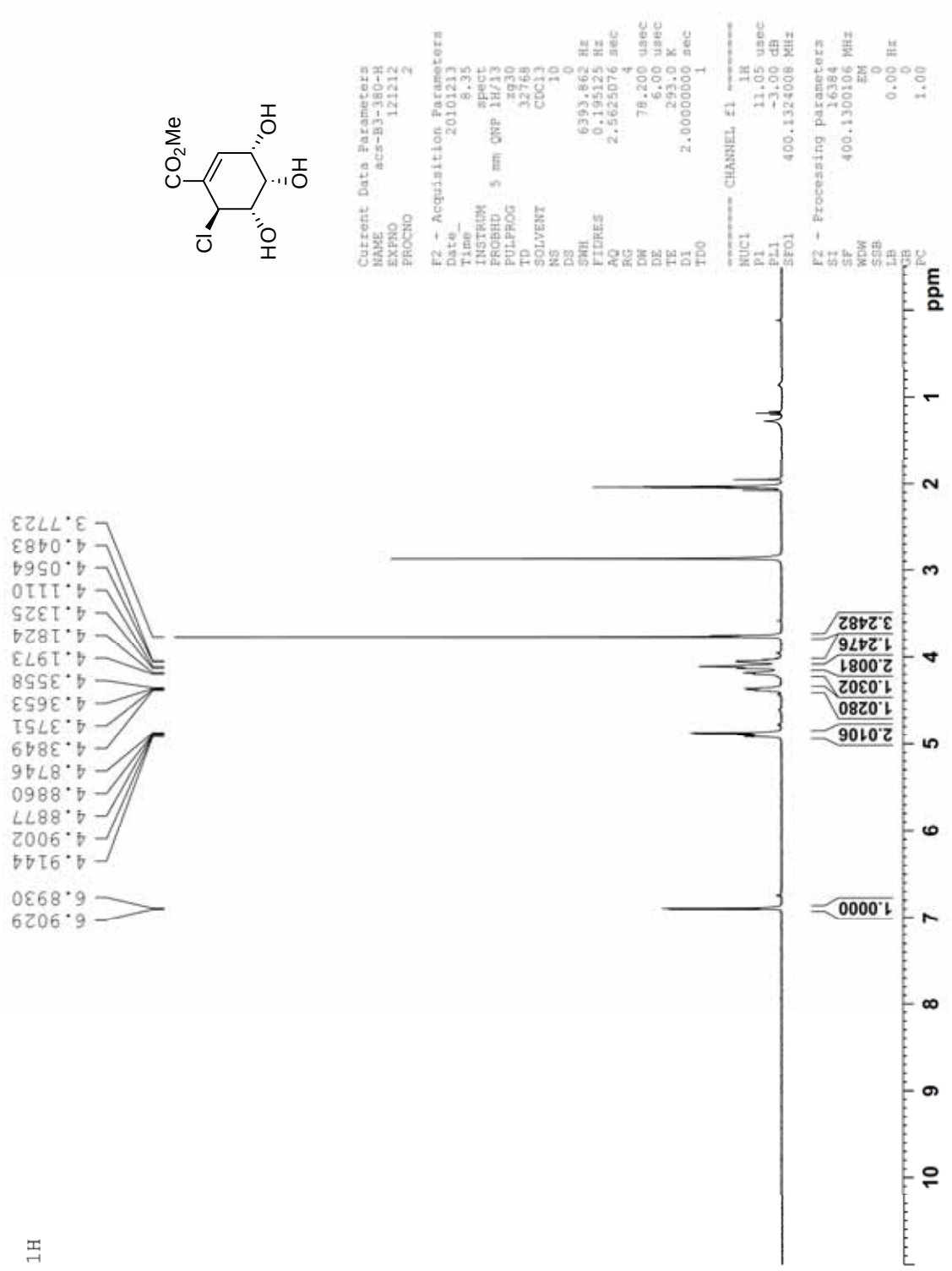

OH
O
CO2Me
OMOM
O
23.79
23.94
25.07
34.69
37.91
51.83
56.44
71.44
76.20
78.92
80.68
98.52
109.44
117.93
126.34
135.03
138.83
167.12
13C
ppm
20
40
60
80
100
120
140
160
180

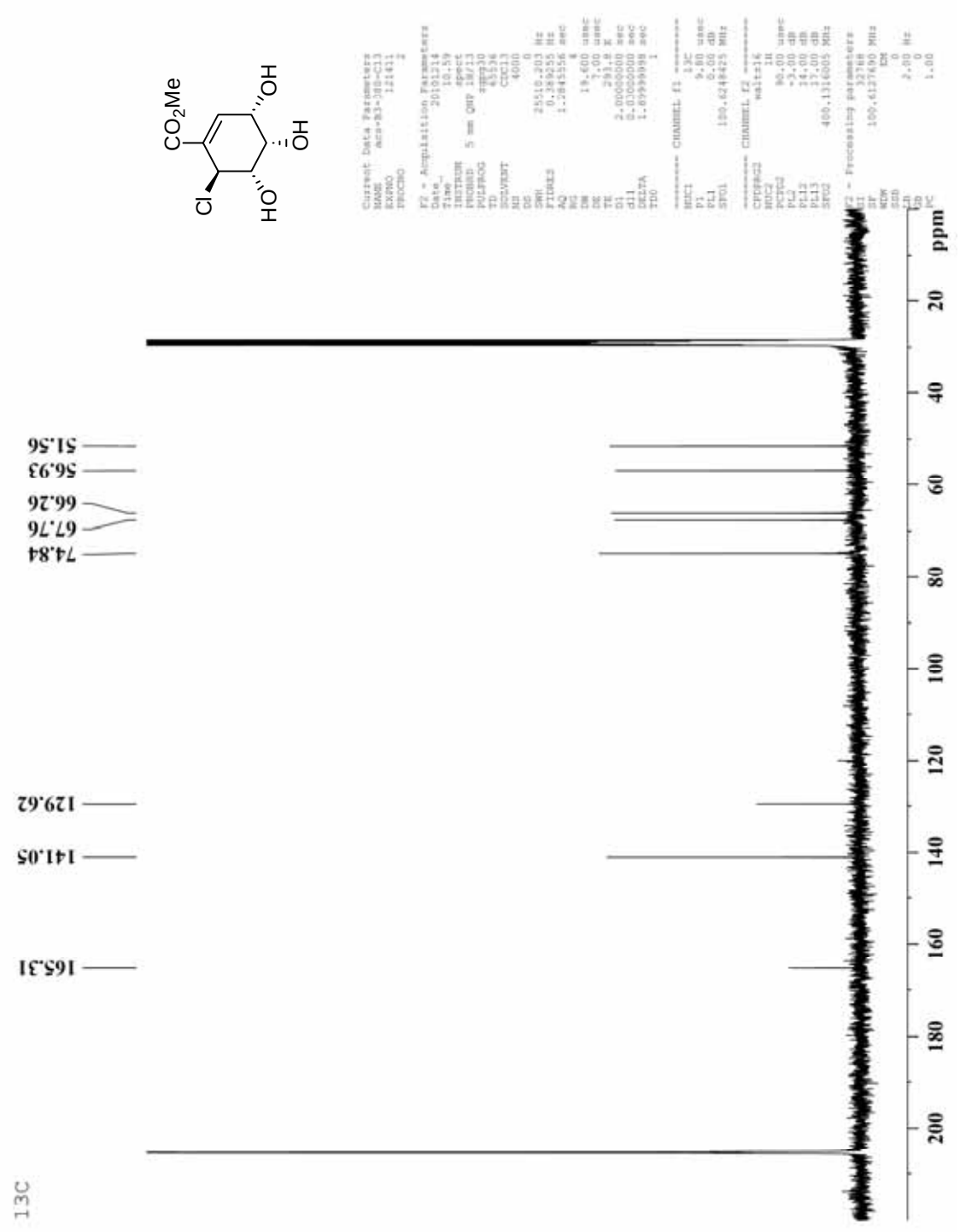

CO2Me
HO
MOMO
O
O
Current Data Parameters
NAME RCM-HG
EXPNO 416012
PROCNO 2
F2 - Acquisition Parameters
Date_ 20100611
Time_ 19.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 1
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 362
DW 78.200 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.0128
1.0341
1.0448
1.0612
1.1504
1.1890
1.2076
1.2360
1.2497
1.3789
1.6011
1.6221
3.4688
3.4835
3.5152
3.5408
3.8072
3.8184
4.6463
4.6597
4.7391
4.7482
4.7596
4.7755
4.8273
4.8448
4.9674
4.9850
6.7918
6.7984
3.0429
11.9268
3.3692
1.0985
4.5879
1.0345
2.2214
1.1715
1.0274
1.0000
ppm
1H

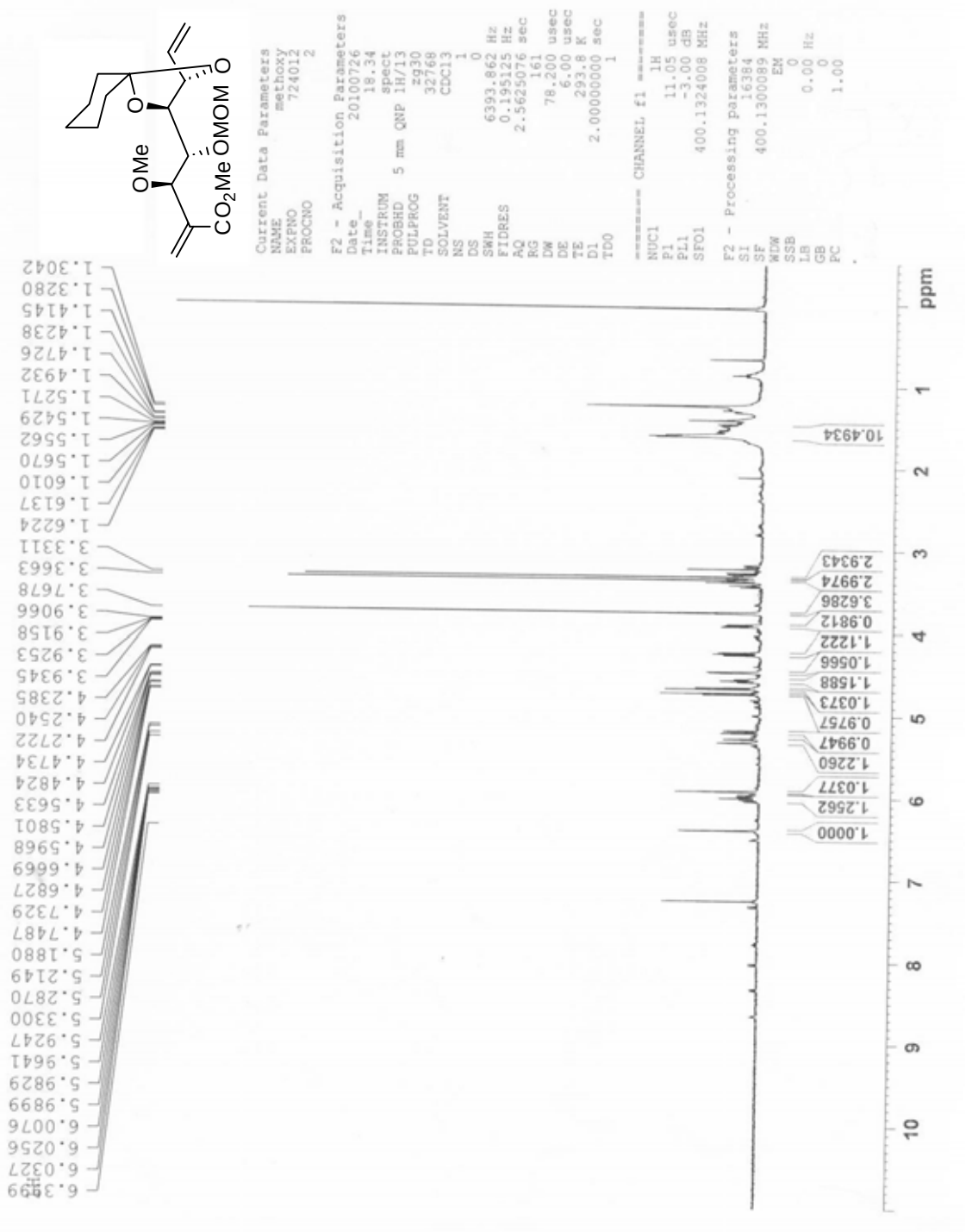

CO2Me
HO
MOMO
O
O
Current Data Parameters
NAME RCM-HG-13C
EXPNO 416012
PROCNO 2
F2 - Acquisition Parameters
Date_ 20100611
Time_ 20.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
ppm
23.58
23.84
24.75
35.71
37.59
52.39
55.90
63.33
71.16
73.02
75.48
94.86
112.28
131.86
135.92
166.02
13C

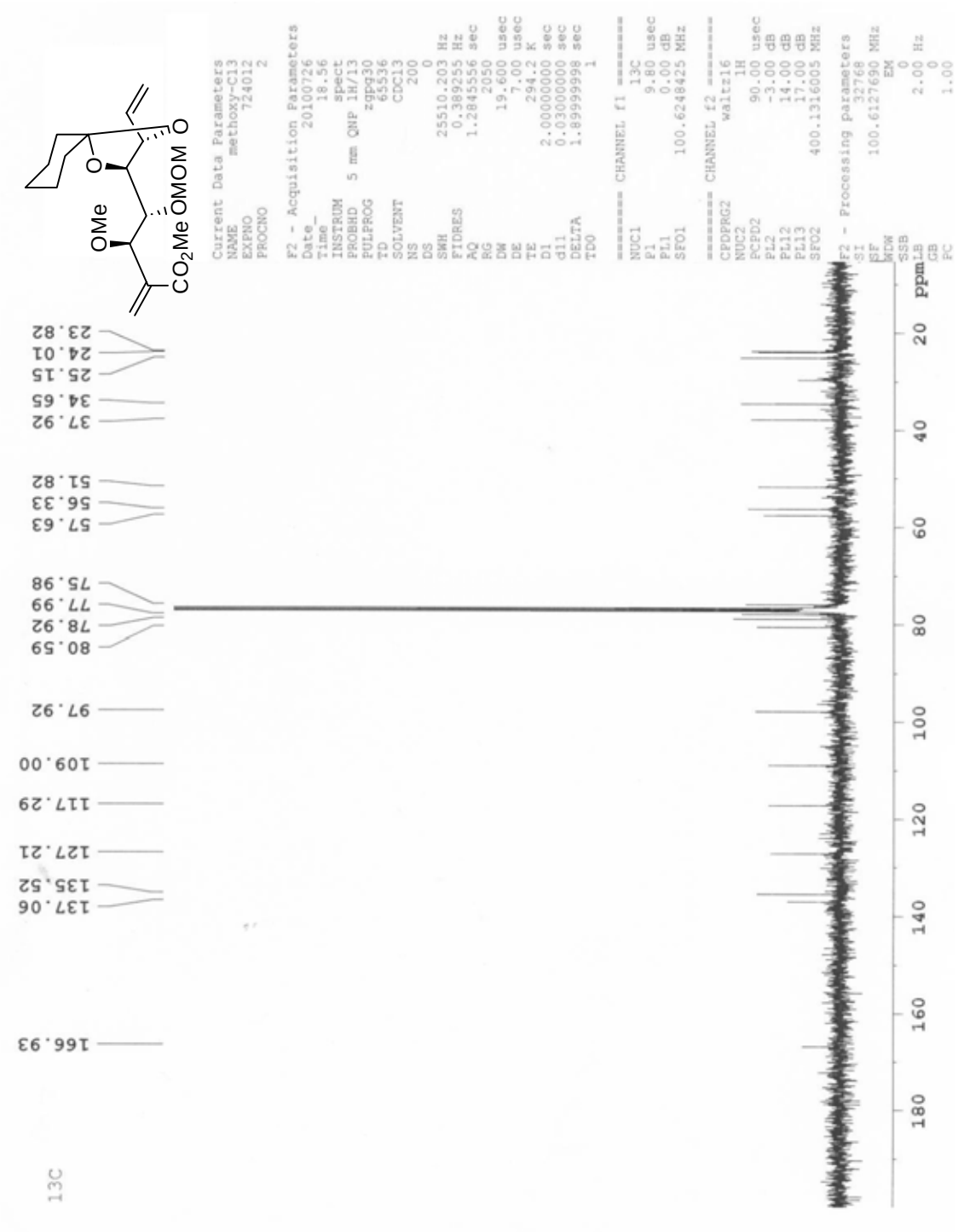

CO2Me
HO
HO
O
O
1H
ppm
1.0000
6.8212
6.8139
5.2922
4.7268
4.7154
4.7070
4.6122
4.6018
4.5875
4.5765
3.8203
3.7974
3.2341
3.2096
3.1175
3.0891
1.6255
1.6120
1.5844
1.5606
1.3755
1.2415
2.3934
7.9284
1.0782
1.0534
4.2607
2.2287
1.1080

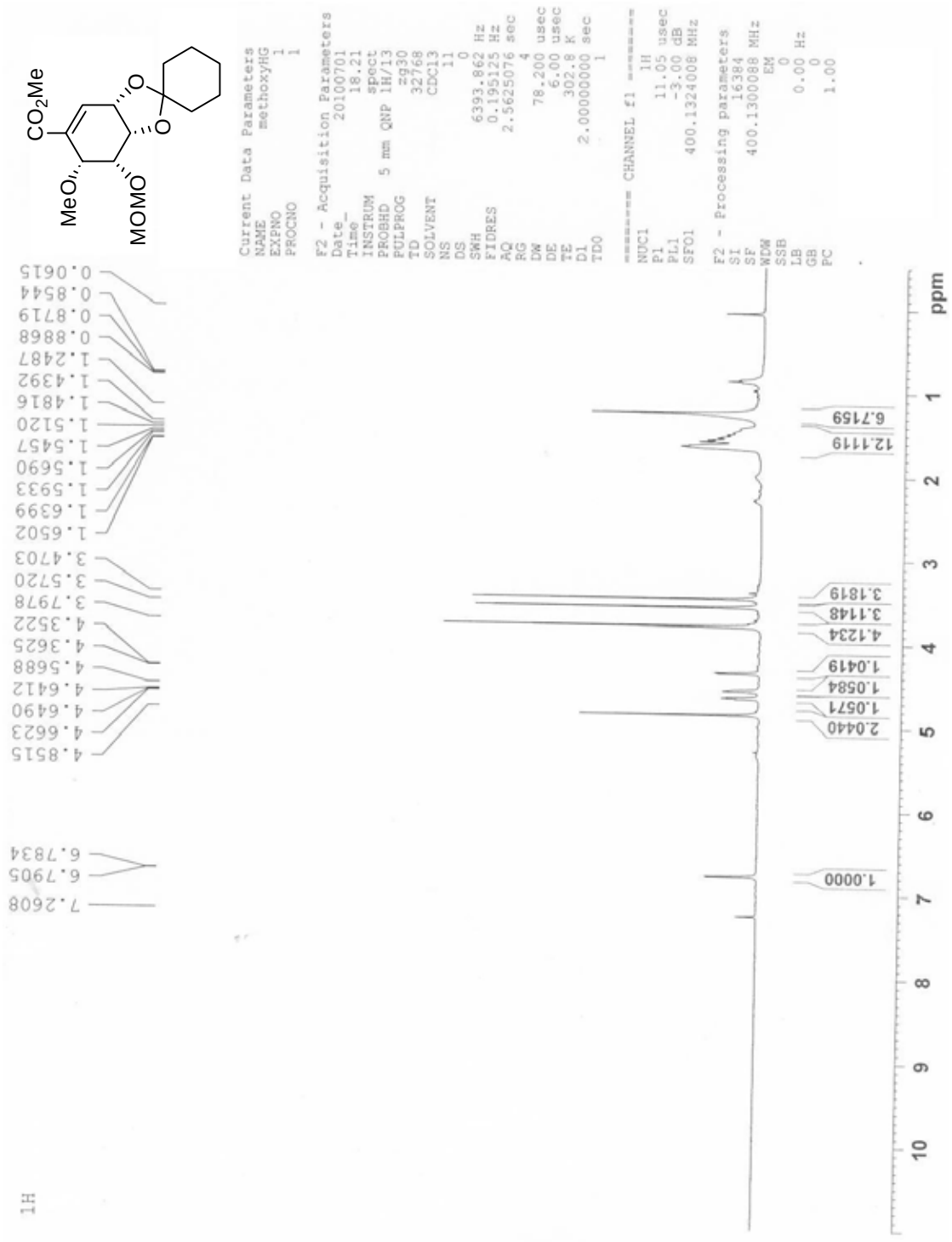


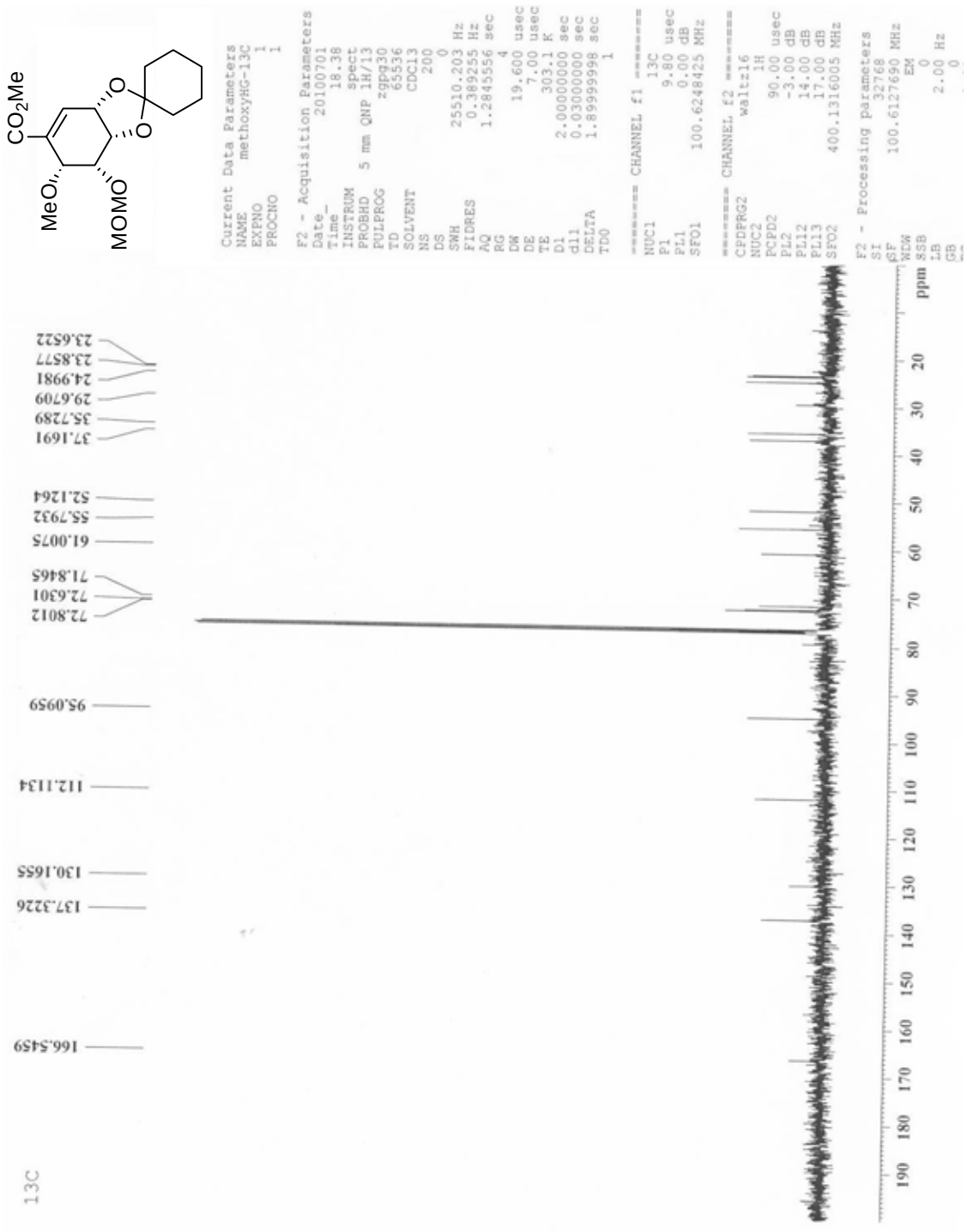

CO2Me
OH
OH
Cl
HO
1H
ppm
3.7723
4.0483
4.0564
4.1110
4.1325
4.1824
4.1973
4.3558
4.3653
4.3751
4.3849
4.8746
4.8860
4.8877
4.9002
4.9144
6.8930
6.9029
3.2482
1.2476
2.0081
1.0302
1.0280
2.0106
1.0000

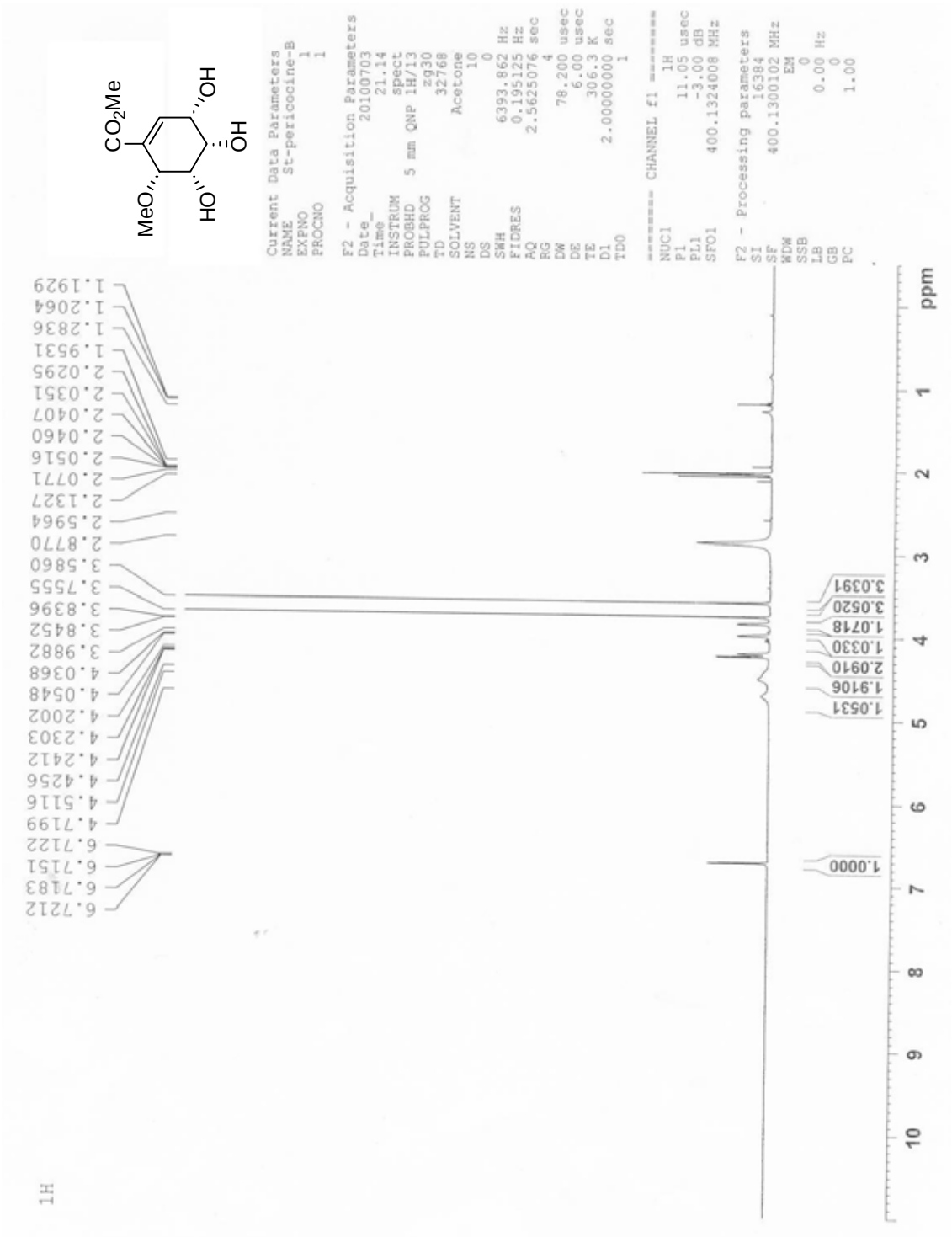


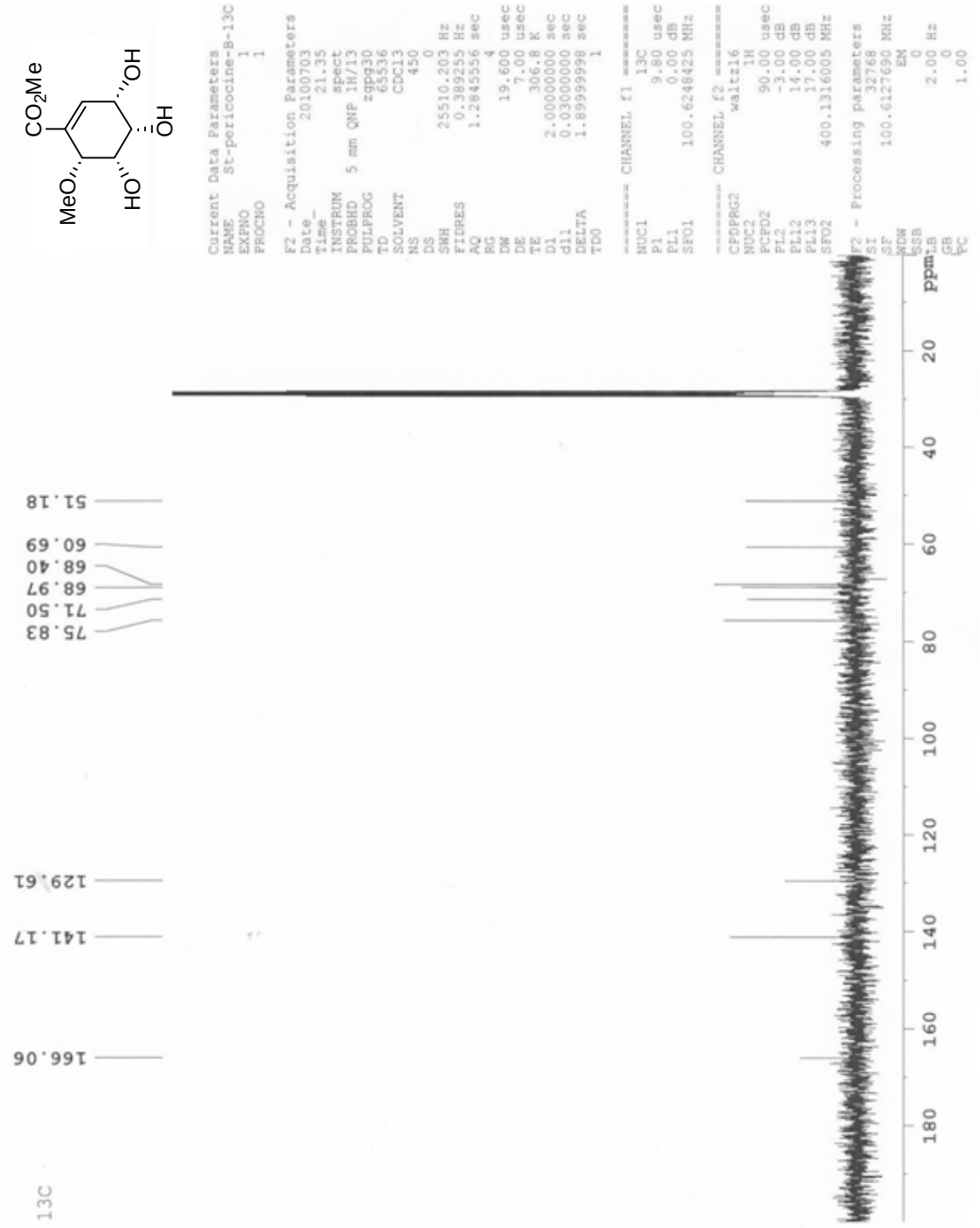

OMe
CO2Me
OMOM
O
Current Data Parameters
NAME methoxy
EXPNO 724012
PROCNO 2
F2 - Acquisition Parameters
Date_ 20100726
Time 18.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 161
DW 78.200 usec
DE 6.00 usec
TE 293.8 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10.4934
2.9343
2.9974
3.6286
0.9812
1.1222
1.0566
1.1588
1.0373
0.9757
0.9947
1.2260
1.0377
1.2562
1.0000
ppm


OMe
OMOM
O
O
CO2Me
13C
23.82
24.01
25.15
34.65
37.92
51.82
56.33
57.63
75.98
77.99
78.92
80.59
97.92
109.00
117.29
127.21
135.52
137.06
166.93
ppm


CO2Me
MeO
MOMO
Current Data Parameters
NAME methoxyHG
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100701
Time 18.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 11
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 4
DW 78.200 usec
DE 6.00 usec
TE 302.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0.0615
0.8544
0.8719
0.8868
1.2487
1.4392
1.4816
1.5120
1.5457
1.5690
1.5933
1.6399
1.6502
3.4703
3.5720
3.7978
4.3522
4.3625
4.5688
4.6412
4.6490
4.6623
4.8515
6.7834
6.7905
7.2608
6.7159
12.1119
3.1819
3.1148
4.1234
1.0419
1.0584
1.0571
2.0440
1.0000
ppm
1H




CO2Me
MeO
HO
OH
OH
Current Data Parameters
NAME St-pericocine-B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100703
Time_ 21.14
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT Acetone
NS 10
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 4
DW 78.200 usec
DE 6.00 usec
TE 306.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.1929
1.2064
1.2836
1.9531
2.0295
2.0351
2.0407
2.0460
2.0516
2.0771
2.1327
2.5964
2.8770
3.5860
3.7555
3.8396
3.8452
3.9882
4.0368
4.0548
4.2002
4.2303
4.2412
4.4256
4.5116
4.7199
6.7122
6.7151
6.7183
6.7212
1.0000
1.0531
1.9106
2.0910
1.0330
1.0718
3.0520
3.0391
1H
ppm


CO2Me
OH
HO
MeO
HO
Current Data Parameters
NAME St-pericocine-B-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100703
Time_ 21.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 450
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 4
DW 19.600 usec
DE 7.00 usec
TE 306.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
ppm
51.18
60.69
68.40
68.97
71.50
75.83
129.61
141.17
166.06
13C