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An Enantioselective Total Synthesis of (+)-Aigialospirol.

authored by

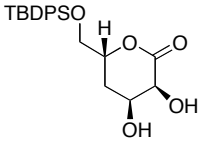
Ruth Figueroa, Richard P. Hsung*, and Christle C. Guevarra

Division of Pharmaceutical Sciences and Department of Chemistry
7111 Rennebohm Hall, 777 Highland Avenue
University of Wisconsin at Madison, Madison, WI 53705-2222

EXPERIMENTAL SECTION

All moisture sensitive reactions were performed on glassware flame dried under vacuo and in a N_2 atmosphere. All reactions were performed in flame-dried glassware under a nitrogen atmosphere. Solvents were distilled prior to use. Reagents were used as purchased (Aldrich, Acros), except where noted. Chromatographic separations were performed using Bodman 60 Å SiO_2. ^1H and ^{13}C NMR spectra were obtained on Varian VI-300, VI-400, and VI-500 spectrometers using $CDCl_3$ (except where noted) with TMS or residual $CHCl_3$ in the solvent as standard. Melting points were determined using a Laboratory Devices MEL-TEMP and are uncorrected/calibrated. Infrared spectra were obtained using NaCl plates on a Bruker Equinox 55/S FT–IR Spectrophotometer, and relative intensities are expressed qualitatively as s (strong), m (medium), and w (weak). TLC analysis was performed using Aldrich 254 nm polyester-backed plates (60 Å, 250 μm) and visualized using UV and a suitable chemical stain. Low-resolution mass spectra were obtained using an Agilent-1100-HPLC/MSD and can be either APCI or ESI, or an IonSpec HiRes-MALDI FT-Mass Spectrometer. High-resolution mass spectral analyses were performed at University of Wisconsin Mass Spectrometry Laboratories. All spectral data obtained for new compounds are reported here. X-Ray analysis was performed at University of Minnesota Department of Chemistry X-Ray facility.

Experimental Procedures and Characterizations.



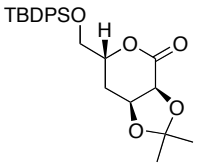
S1

To a solution of dihydro-γ-pyrone **6** (4.00 g, 10.9 mmol) in a 4:1 mixture of acetone-H_2O (70 mL) were added 4-methylmorpholine oxide (3.20 g, 27.3 mmol) and OsO_4 (176.0 mg, 0.69 mmol). The resulting mixture was stirred at rt for 1 h before 5% aq $NaHSO_3$ (60 mL) was added. The mixture was stirred for an additional 5 min and extracted with three portions of equal volume of EtOAc. The combined organic extracts were dried ($MgSO_4$) and concentrated *in vacuo* to give a crude residue that was purified via silica gel flash column chromatography (eluted with 5% MeOH in CH_2Cl_2) to afford diol **S1** in 87% yield (3.80 g) as a colorless thick oil.

R_f = 0.35 [5% MeOH in CH_2Cl_2]; $[\alpha]_D^{20}$ = - 19.1 (*c* 1.13, $CHCl_3$);

^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.05 (s, 9H), 2.18 (dt, *J* = 10.3, 4.3 Hz, 1H), 2.29 (m, 1H), 2.88 (s, 1H), 3.57 (s, 1H), 3.70 (dd, *J* = 11.3, 3.1 Hz, 1H), 3.94 (dd, *J* = 11.4, 3.6 Hz, 1H), 4.13 (dd, *J* = 2.5, 0.8 Hz, 1H), 4.41 (br m, 1H), 4.81 (dq, *J* = 11.3, 3.7 Hz, 1H), 7.37-7.47 (m, 6H), 7.63-7.66 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.5, 27.0, 29.8, 65.2, 66.4, 70.7, 78.4, 128.1, 130.2, 132.9, 133.2, 135.8, 135.9,

174.2; IR (neat) cm^{-1} 3399m, 2930w, 2856w, 1725m, 1426m, 1391m, 1233m, 1189m, 1102s, 1045m, 996m, 937m, 822m, 740m, 699s; HRMS (MALDI) calcd for $C_{22}H_{28}O_5NaSi$ 423.1598, found 423.1581.



7

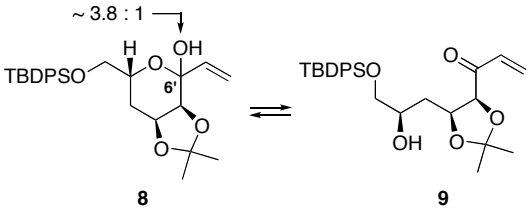
To a solution of diol **S1** (1.50 g, 3.74 mmol) in acetone (23 mL) and 2,2-dimethoxypropane (7 mL) was added *p*-TsOH (71.2 mg, 0.374 mmol, practical grade). The resulting mixture was stirred at rt for 3 h before it was concentrated *in vacuo* and diluted with CH_2Cl_2. The resulting solution was washed with sat aq $NaHCO_3$, and the aqueous layer was extracted with two portions of equal volume of CH_2Cl_2. The combined organic layers were dried ($MgSO_4$) and concentrated *in vacuo* to afford a crude oil that was purified via silica gel flash column chromatography (eluted with 10% EtOAc in hexane) to yield the acetonide protected □-lactone **7** in 78% yield (1.28 g) as a colorless oil.

R_f = 0.11 [10% EtOAc in hexanes];

On occasions, □-lactone **7** solidifies into a white solid upon standing: mp 71-72 °C;

$[\square]_D^{20}$ = □ 27.6 (*c* 0.71, $CHCl_3$);

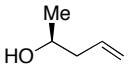
^{1}H NMR (400 MHz, $CDCl_3$) □ 1.06 (s, 9H), 1.38 (s, 3H), 1.49 (s, 3H), 2.03 (ddd, *J* = 15.0, 10.5, 3.5 Hz, 1H), 2.11 (dt, *J* = 15.0, 2.5 Hz, 1H), 3.77 (dd, *J* = 11.1, 3.9 Hz, 1H), 3.86 (dd, *J* = 11.3, 4.5 Hz, 1H), 4.59 (d, *J* = 6.8 Hz, 1H), 4.63-4.71 (m, 2H), 7.37-7.46 (m, 6H), 7.64-7.68 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) □ 19.5, 24.3, 26.3, 27.0, 30.6, 65.2, 71.9, 73.1, 75.4, 110.9, 128.0, 130.1, 132.9, 133.2, 135.7, 135.8, 168.3; IR (neat) cm^{-1} 2932w, 2857w, 1749m, 1472w, 1427m, 1376m, 1260m, 1209m, 1139 m, 1110m, 1068m, 1041m, 1005m, 945m, 871w, 822m, 802m, 737m, 701s; HRMS (MALDI) calcd for $C_{25}H_{32}O_5NaSi$ 463.1911, found 463.1906.



8 **9**

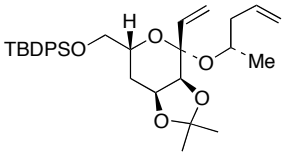
To a solution of □-lactone **7** (534.0 mg, 1.21 mmol) in Et_2O (9 mL) was added vinyl magnesium bromide (1.0 *M* in THF: 1.30 mL, 1.30 mmol, 1.07 equiv) carefully dropwise at –78 °C. The pale yellow solution was stirred at □78 °C for 1 h before it was quenched with sat aq NH_4Cl at that temperature. H_2O

was then added until all precipitate was dissolved. The resulting mixture was allowed to reach rt and was diluted with Et_2O. The aqueous phase was separated and extracted with two portions of Et_2O. The combined organic extracts were dried ($MgSO_4$) and concentrated *in vacuo* to give a cloudy crude oil that was purified via silica gel flash column chromatography (eluted with 10% EtOAc/90% hexane) to afford a mixture of lactol **8** and vinyl ketone **9** in 88% combined yield (500.0 mg) as a colorless oil.
R_f = 0.20 [10% EtOAc in hexanes]; $[\alpha]_D^{20}$ = − 9.82 (*c* 2.50, $CHCl_3$);
^{1}H NMR (400 MHz, C_6D_6) δ 1.07 (s, 3H), 1.14 (s, 9H), 1.16 (s, 12H), 1.169 (s, 9H), 1.171 (s, 3H), 1.41 (s, 3H), 1.45 (s, 3H), 1.49 (s, 3H), 1.65-1.78 (m, 3H), 1.87 (dt, *J* = 14.2, 3.9 Hz, 1H), 2.07 (dt, *J* = 14.6, 2.7, Hz, 1H), 2.54 (dt, *J* = 14.5, 11.9, 3.0 Hz, 1H), 2.77-2.78 (m, 1H), 2.91-2.92 (m, 1H), 3.28-3.29 (m, 1H), 3.41 (dd, *J* = 10.6, 0.9 Hz, 1H), 3.59-3.78 (m, 5H), 3.93-4.00 (m, 1H), 4.11 (d, *J* = 8.0 Hz, 2H), 4.26-4.35 (m, 4H), 4.39 (d, *J* = 7.6 Hz, 1H), 4.42 (dt, *J* = 7.8, 2.7 Hz, 1H), 5.09 (dd, *J* = 10.5, 1.7 Hz, 1H), 5.17 (dd, *J* = 10.7, 1.8 Hz, 1H), 5.19 (dd, *J* = 10.3, 1.9 Hz, 1H), 5.64 (dd, *J* = 17.3, 1.7 Hz, 1H), 5.66 (dd, *J* = 17.2, 1.8 Hz, 1H), 6.00 (dd, *J* = 17.2, 10.5 Hz, 1H), 6.24 (dd, *J* = 17.3, 1.9 Hz, 1H), 6.40 (dd, *J* = 17.3, 10.6 Hz, 1H), 6.73 (dd, *J* = 17.3, 10.5 Hz, 1H), 7.27-7.18 (m, 18H), 7.74-7.83 (m, 12H); IR (neat) cm^{-1} 3441w, 2931w, 2857w, 1753w, 1697w, 1472w, 1427w, 1373w, 1262m, 1210m, 1110s, 1065m, 1041m, 701s; HRMS (MALDI) calcd for $C_{27}H_{36}O_5NaSi$ 491.2224, found 491.2229.



10

To a slurry of CuI (836 mg, 4.39 mmol) in THF (200 mL) was added vinyl magnesium chloride (1.6 *M* in THF, 27.4 mL, 43.9 mmol, 1.70 equiv) carefully dropwise via syringe at –78 °C. The resulting mixture was stirred for 15 min and *(S)*-propylene oxide (1.50 g, 1.8 mL, 25.8 mmol) was added in one shot. The yellow mixture was stirred for 30 min. The cooling bath was replaced with an acetonitrile/CO_2 bath and the reaction was kept at -35 °C to -25 °C for 1 h. The black mixture was then stirred for 1 h at 0 °C (ice water bath) before it was quenched with sat aq NH_4Cl (50 mL) and H_2O (50 mL). The organic phase was separated, and the aqueous layer was extracted with Et_2O (2 x 50 mL). The combined organic layers were dried ($MgSO_4$) and concentrated by atmospheric distillation. The residue was distilled at atmospheric pressure to afford alcohol **10** contaminated with THF (fractions collected between 100 °C to 120 °C). The distillate was further flushed with N_2 to afford alcohol **10** in 70% yield (1.56 g) as a colorless liquid.[1] NMR data match the one reported in the literature.[2]
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.21 (d, *J* = 6.2 Hz, 3H), 1.64 (s, 1H), 2.13-2.21 (m, 1H), 2.24-2.31 (m, 1H), 3.82-3.89 (m, 1H), 5.11-5.17 (m, 2H), 5.78-5.88 (m, 1H).



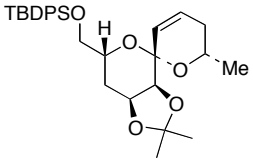
11

Note: Two batch of (each batch 573.6 mg of the mixture **8** and **9**) the same amount were done and combined during the purification process. A round-bottomed flask was charged with the mixture of **8** and **9** (573.6 mg, 1.22 mmol), CH_2Cl_2 (13 mL), activated pulverized 4Å molecular sieves (1.10 g) and homo-allylic alcohol **10** (1.05 g, 12.24 mmol). The mixture was cooled to −78 °C and a solution of Tf_2NH (344.1 mg, 1.22 mmol) in CH_2Cl_2 (13 mL) was added via syringe (NOTE: This solution was freshly made prior to be used). The reaction mixture was stirred for 1 h before it was quenched with Et_3N (10 mL). The cooling bath was removed and the mixture was allowed to reach rt. After filtration through a pad of Celite™, the filtrate was concentrated *in vacuo* to give a crude yellow oil. At this point, the two identical batches were combined and purified via silica gel flash column chromatography (eluted with 5% EtOAc in hexanes) to afford ketal **11** in 76 % yield (1.01 g) as a colorless oil.

R_f = 0.23 [5% EtOAc in hexanes]; $[\alpha]_D^{20}$ = + 3.85 (*c* 1.82, CH_2Cl_2);

^{1}H NMR (500 MHz, C_6D_6) δ 1.08 (d, *J* = 6.3 Hz, 3H), 1.16 (s, 3H), 1.18 (s, 9H), 1.43 (s, 3H), 1.87 (dt, *J* = 12.4, 1.5 Hz, 1H), 2.13-2.24 (m, 3H), 3.74 (dd, *J* = 10.5, 5.6 Hz, 1H), 3.94 (dd, *J* = 10.4, 5.6 Hz, 1H), 4.06-4.12 (m, 1H), 4.24 (d, *J* = 7.6 Hz, 1H), 4.49 (dd, *J* = 7.4, 2.6 Hz, 1H), 4.56 (dtd, *J* = 10.7, 5.6, 2.0 Hz, 1H), 4.91-4.99 (m, 2H), 5.28 (dd, *J* = 8.8, 3.9 Hz, 1H), 5.68 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.77-5.86 (m, 2H), 7.22-7.24 (m, 6H), 7.80-7.85 (m, 4H); ^{13}C NMR (125 MHz) δ In C_6D_6: 19.5, 21.5, 24.0, 26.6, 27.1, 31.0, 43.0, 66.9, 68.1, 69.6, 70.9, 76.6, 98.5, 108.3, 117.1, 118.1, 128.1, 130.0, 133.9, 134.1, 135.3, 136.07, 136.15, 138.3 [missing two aryl carbons due to overlap]; in $CDCl_3$: 19.6, 21.5, 24.2, 26.5, 27.1, 30.8, 42.7, 66.9, 67.7, 69.3, 70.8, 76.3, 98.0, 108.5, 117.3, 118.5, 127.9, 128.0, 129.9, 133.8, 133.9, 135.0, 135.9, 136.0, 137.7 [missing one aryl carbon due to overlapping *para*-carbons of the TBDPS group at 129.9 ppm];

IR (neat) cm^{-1} 3073w, 2930m, 2859w, 1375w, 1263w, 1212m, 1107s, 1053s, 998s, 824m, 703s; HRMS (MALDI) calcd for $C_{32}H_{44}O_5NaSi$ 559.2850, found 559.2821.



12

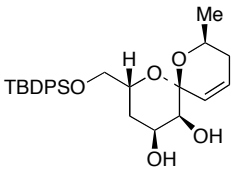
To a purple solution of the Grubb's first generation catalyst (129.0 mg, 0.157 mmol) in toluene (270 mL) was added a solution of cyclic ketal **11** (676.0 mg, 1.26 mmol) in toluene (60 mL). The mixture was stirred at rt for 3 h. Subsequently, it was concentrated *in vacuo* to give a crude brown oil

that was purified via silica gel flash column chromatography (eluted with 5% EtOAc in hexanes) to yield the pure spiroketal **12** in 86% yield (554.0 mg) as a colorless oil.

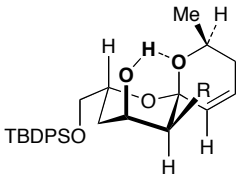
R_f = 0.20 [5% EtOAc in hexanes]; $[\alpha]_D^{20}$ = + 20.9 (*c* 0.77, $CHCl_3$);

^{1}H NMR (500 MHz, C_6D_6) δ 1.03 (d, *J* = 6.3 Hz, 3H), 1.17 (s, 9H), 1.23 (s, 3H), 1.52 (s, 3H), 1.58-1.68 (m, 3H), 2.06 (ddd, *J* = 14.1, 3.4, 2.2 Hz, 1H), 2.32 (ddd, *J* = 14.4, 11.7, 3.2 Hz, 1H), 3.64-3.70 (m, 1H), 3.83 (dd, *J* = 10.1, 6.2 Hz, 1H), 3.92 (dd, *J* = 10.0, 4.9 Hz, 1H), 4.42 (dt, *J* = 7.6, 2.7 Hz, 1H), 4.48-4.53 (m, 1H), 5.68 (ddd, *J* = 10.3, 4.6, 3.7 Hz, 1H), 6.18 (dt, *J* = 10.3, 2.0 Hz, 1H), 7.15-7.21 (m, 6H), 7.79-7.83 (m, 4H); ^{13}C NMR (125MHz) δ In C_6D_6: 19.6, 21.9, 24.3, 26.6, 27.2, 28.8, 31.1, 68.0, 68.6, 69.5, 71.1, 75.3, 95.3, 108.9, 126.3, 129.2, 129.82, 129.84, 134.3, 134.4, 136.1, 136.2 [missing two aryl carbons due to overlap]; in $CDCl_3$: 19.6, 22.0, 24.5, 26.5, 27.2, 28.6, 31.2, 68.0, 68.2, 69.2, 70.9, 75.1, 95.1, 109.1, 127.7, 127.8, 127.9, 128.0, 129.7, 129.8, 134.1, 135.9, 136.0 [missing one aryl carbon due to overlapping Si-substituted *ipso*-carbons of the TBDPS group at 129.9 ppm];

IR (neat) cm^{-1} 3071w, 3050w, 2931m, 1428w, 1371m, 1262w, 1210m, 1166m, 1139m, 1111s, 1032s, 823m, 700s; HRMS (MALDI) calcd for $C_{30}H_{40}O_5NaSi$ 531.2537, found 531.2550.



13



13: R = OH

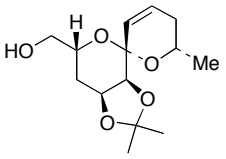
To a solution of spiroketal **12** (9.00 mg, 0.018 mmol) in MeOH (1 mL) was added *p*-TsOH (0.50 mg, 0.0027 mmol, practical grade). The mixture was stirred at rt for 1 h until TLC showed completion consumption of **12**. (NOTE: The reaction time varied, and thus, it should be carefully monitored via TLC analysis.) The reaction mixture was concentrated *in vacuo* and diluted with CH_2Cl_2 (10 mL). The resulting solution was washed with sat aq $NaHCO_3$ (1 mL). The aqueous layer was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layers were dried ($MgSO_4$) and concentrated *in vacuo* to afford a crude residue that was purified via silica gel flash column chromatography (eluted with 2% MeOH in CH_2Cl_2) to afford diol **13** in 81% yield (6.70 mg) as a white solid.

R_f = 0.16 [2% MeOH in CH_2Cl_2]; mp 43-45 °C; $[\alpha]_D^{20}$ = + 51.8 (*c* 0.62, CH_2Cl_2);

^{1}H NMR (500MHz, C_6D_6) δ 0.90 (d, *J* = 6.3 Hz, 3H), 1.17 (s, 9H), 1.39-1.48 (m, 2H), 1.62 (ddt, *J* = 17.6, 11.0, 2.4 Hz, 1H), 1.91 (ddd, *J* = 13.9, 3.4, 2.4 Hz, 1H), 2.61 (d, *J* = 10.9 Hz, 1H), 3.30 (dd, *J* = 10.4, 3.4 Hz, 1H), 3.49 (d, *J* = 10.6 Hz, 1H), 3.65 (dd, *J* = 10.6, 4.3 Hz, 1H), 3.74 (dd, *J* = 10.6, 5.7 Hz, 1H), 3.98 (dqd, *J* = 10.9, 6.3, 3.3 Hz, 1H), 4.05-4.09 (m, 1H), 4.14-4.19 (m, 1H), 5.56 (ddd, *J* = 10.0, 2.7, 1.3 Hz, 1H), 5.72 (ddd, *J* = 10.0, 5.9, 1.9 Hz, 1H), 7.22-7.24 (m, 6H), 7.80-7.84 (m, 4H);

^{13}C NMR (125 MHz) δ In C_6D_6: 19.6, 20.8, 27.1, 31.7, 35.2, 64.5, 65.5, 67.2, 69.3, 71.4, 99.3, 128.4, 129.1, 129.99, 130.03, 134.10, 134.14, 136.16, 136.18; in $CDCl_3$: 19.6, 21.4, 27.1, 31.9, 35.3, 64.8, 65.3, 66.9, 71.3, 99.1, 127.5, 127.8, 127.9, 129.9, 130.0, 130.3, 133.8, 133.9, 135.9, 136.0;

IR (CH_2Cl_2 film) cm^{-1} 3497br, 3047w, 2930m, 2857m, 1589w, 1427m, 1111s, 1066s, 1012s, 822m, 740m, 702s; HRMS (MALDI) calcd for $C_{27}H_{36}O_5NaSi$ 491.2224, found 491.2269.



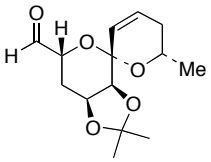
S2

To a solution of spiroketal **12** (138.5 mg, 0.27 mmol) in THF (10 mL) was added tetra-n-butylammonium fluoride (1.0 *M* in THF: 0.41 mL, 0.41 mmol). The resulting yellow solution was stirred at rt for 22 h. The mixture was concentrated *in vacuo* to give a crude residue that was purified via silica gel flash column chromatography (eluted with 35% EtOAc in hexanes) to afford the desired alcohol **S2** in 98% yield (72.3 mg) as a white solid.

R_f = 0.18 [35% EtOAc in hexanes];

mp 131-134 °C; $[\alpha]_D^{20}$ = + 108.9 (*c* 0.52, $CHCl_3$);

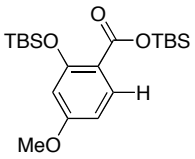
^{1}H NMR (400 MHz, C_6D_6) δ 0.97 (d, *J* = 6.1 Hz, 3H), 1.23 (d, *J* = 0.4 Hz, 3H), 1.44 (dddd, *J* = 17.1, 5.5, 3.8, 1.4 Hz, 1H), 1.52 (d, *J* = 0.4 Hz, 3H), 1.60 (ddt, *J* = 17.4, 9.1, 2.6 Hz, 1H), 1.64 (ddd, *J* = 14.4, 4.2, 2.6 Hz, 1H), 2.47 (ddd, *J* = 14.6, 12.0, 2.8 Hz, 1H), 2.83 (dd, *J* = 8.9, 2.6 Hz, 1H), 3.37 (ddd, *J* = 11.5, 8.8, 2.9 Hz, 1H), 3.55 (dqd, *J* = 9.3, 6.2, 3.6 Hz, 1H), 3.81 (dt, *J* = 11.4, 2.6 Hz, 1H), 3.97 (d, *J* = 7.7 Hz, 1H), 4.27-4.33 (m, 2H), 5.65 (ddd, *J* = 10.3, 5.7, 2.6 Hz, 1H), 6.13 (ddd, *J* = 10.3, 2.6, 1.4 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6) δ 21.5, 24.4, 25.4, 26.5, 31.1, 65.5, 68.4, 70.2, 70.8, 74.3, 95.5, 108.9, 126.8, 128.5; IR (neat) cm^{-1} 3447brw, 3050w, 2979w, 2921w, 1451w, 1431w, 1382m, 1370m, 1209m, 1168m, 1035s, 1016s; mass spectrum (APCI): m/e (% relative intensity) 271 (10) $(M + H)^+$, 253 (7) $([M+H]-H_2O)^+$, 227 (5), 213 (100), 195 (37), 167 (80); HRMS (ESI) calcd for $C_{14}H_{22}O_5NaSi$ 293.1365, found 293.1353.



14

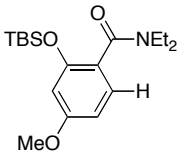
To a solution of alcohol **S2** (117.1 mg, 0.43 mmol) in CH_2Cl_2 (4.6 mL) were added iodobenzene diacetate (153.5 mg, 0.48 mmol) and then 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) (8.50 mg, 0.054 mmol). The resulting orange solution was stirred at rt for 21 h before it was quenched with sat aq $Na_2S_2O_3$ (5.0 mL). The mixture was extracted with CH_2Cl_2 (3 x 10 mL), and the combined organic extracts were dried (Na_2SO_4) and concentrated *in vacuo* to give a yellow/orange oil that was purified via

silica gel flash column chromatography (eluted with 15% EtOAc in hexanes to remove impurities and 30% EtOAc in hexanes) to afford aldehyde **14** in 78% yield (90.6 mg) as a white solid.
R_f = 0.66 [20% EtOAc in hexanes]; mp 62-63 °C; $[\alpha]_D^{20}$ = + 136.7 (*c* 0.73, $CHCl_3$);
^{1}H NMR (500 MHz, C_6D_6) δ 0.96 (d, *J* = 6.4 Hz, 3H), 1.18 (s, 3H), 1.46 (s, 3H), 1.56-1.59 (m, 2H), 2.04 (ddd, *J* = 14.4, 5.7, 2.8 Hz, 1H), 2.21 (ddd, *J* = 14.4, 11.1, 3.1 Hz, 1H), 3.52-3.58 (m, 1H), 3.84 (d, *J* = 7.4 Hz, 1H), 4.17 (dt, *J* = 7.6, 2.9 Hz, 1H), 4.34 (dd, *J* = 11.1, 5.7 Hz, 1H), 5.67 (ddd, *J* = 10.3, 4.7, 3.6 Hz, 1H), 6.14 (dt, *J* = 10.4, 2.0 Hz, 1H), 9.81 (s, 1H); ^{13}C NMR (125 MHz, C_6D_6) δ 21.5, 24.7, 25.3, 26.7, 30.9, 68.2, 70.3, 73.8, 75.1, 95.6, 109.3, 127.2, 127.8, 203.2; IR (neat) cm^{-1} 2984w, 2936w, 1735s, 1381s; mass spectrum (APCI): m/e (% relative intensity) 269 (15) $(M + H)^+$.



S3

To a solution of 4-methoxysalicylic acid (2.52 g, 15.0 mmol) in DMF (12 mL) was added diisopropylethylamine (9.40 mL, 54.0 mmol) and *tert*-butyldimethylsilyl chloride (5.65 g, 37.5 mL). The mixture was vigorously stirred at rt for 3 h. The reaction mixture was poured over H_2O (30 mL) and extracted with Et_2O (4 x 50 mL). The combined organic extracts were washed with sat aq NaCl (2 x 25 mL), dried over $MgSO_4$, and concentrated *in vacuo* to yield a yellow oil that was purified via silica gel flash column chromatography (eluted with 2% EtOAc in hexanes) to afford the desired silyl ester **S3** in 74% yield (4.40 g) as a colorless oil.[3]
R_f = 0.21 [2% EtOAc in hexanes];
^{1}H NMR (400MHz, $CDCl_3$) δ 0.19 (s, 6H), 0.32 (s, 6H), 0.97 (s, 9H), 0.99 (s, 9H), 3.78 (s, 3H), 6.37 (d, *J* = 2.5 Hz, 1H), 6.49 (dd, *J* = 8.8, 2.5 Hz, 1H), 7.75 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.4, -4.2, 18.0, 18.7, 26.0, 26.1, 55.5, 106.9, 107.5, 116.6, 133.7, 158.4, 163.7, 164.8; IR (neat) cm^{-1} 2954w, 2930w, 2858w, 1704m, 1606m, 1493w, 1463m, 1443m, 1337w, 1251s, 1169m, 1149m, 1134m, 1082s, 834s, 782s; HRMS (ESI) calcd for $C_{20}H_{37}O_4Si_2$ 397.2230, found 397.2221.



15

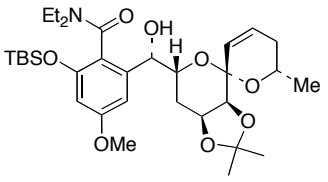
To a solution of silyl ester **S3** (250.0 mg, 0.630 mmol) in CH_2Cl_2 (0.6 mL) was added a drop of DMF. After cooling to 0 °C in an ice-water bath, oxalyl chloride (88.0 mg, 0.060 mL, 0.693 mmol) was added dropwise at a rate to maintain the control of the reaction. The resulting yellow solution was stirred 0 °C for 1.5 h before the ice bath was removed and the mixture was further stirred overnight.

After which, the reaction mixture was concentrated *in vacuo*, re-diluted with fresh CH_2Cl_2, and re-concentrated *in vacuo* to afford the crude acyl chloride.

To a solution of the crude acyl chloride prepared above in CH_2Cl_2 (0.6 mL) was added diethylamine (93.0 mg, 0.13 mL, 1.26 mmol) at rt dropwise at a rate to maintain the control of the reaction. The resulting mixture was stirred for 1 h before being concentrated *in vacuo* to afford a crude residue that was purified via silica gel flash column chromatography (eluted with 20% EtOAc in hexanes) to yield the desired amide **15** in 56% yield (119.0 mg) as a colorless oil.[3]

R_f = 0.41 [20% EtOAc in hexanes];

^{1}H NMR (400MHz, $CDCl_3$) □ 0.22 (s, 6H), 0.96 (s, 9H), 1.01 (t, *J* = 7.1 Hz, 3H), 1.23 (t, *J* = 7.2 Hz, 3H), 3.10-3.34 (broad m, 2H), 3.34-3.70 (broad m, 2H), 3.78 (s, 3H), 6.36 (d, *J* = 2.3 Hz, 1H), 6.53 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.12 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) □ 13.5, 14.4, 18.3, 25.8, 39.5, 43.1, 55.5, 106.0, 106.5, 123.0, 128.8, 152.5, 160.9, 169.3 [missing two Me carbon resonances from the TBS group due to rotameric line broadening]; IR (neat) cm^{-1} 2933w, 2898w, 2860w, 1632brm, 1608brm, 1463brm, 1428br, 1288m, 1255m, 1166m, 839s, 783s; mass spectrum (APCI): m/e (% relative intensity) 338 (100) $(M + H)^+$; HRMS (MALDI) calcd for $C_{18}H_{32}O_3NSi$ 338.2146, found 338.2131.



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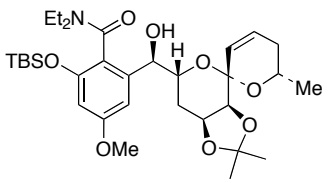


17

To a round-bottomed flask charged with anhyd THF (1.2 mL) were added tetramethylethylenediamine (19.8 mg, □ 0.030 mL, 0.17 mmol) and *s*-BuLi (1.4 *M* in cyclohexane: 0.12 mL, 0.17 mmol) at –78 °C via syringe. The deep yellow solution was stirred for 2 min before a solution of amide **15** (57.6 mg, 0.17 mmol) in THF (0.3 mL) was added dropwise also via syringe. The pale yellow solution was stirred for 45 min at –78 °C before a solution of aldehyde **14** (41.6 mg, 0.155 mmol) in THF (0.3 mL) was added. Additional THF (0.2 mL) was used to ensure complete transferred of the aldehyde. The mixture was stirred for 1 h at –78 °C and 2.5 h at rt, and it was quenched with H_2O (3 mL) and extracted with Et_2O (3 x 5 mL). The combined organic layers were dried (Na_2SO_4) and concentrated *in vacuo* to give a crude mixture that was purified via silica gel flash column chromatography (eluted with 10-60% EtOAc in hexanes) to afford secondary alcohols **16** (18.5 mg) and **17** (28.8 mg) in a combined 50% yield.

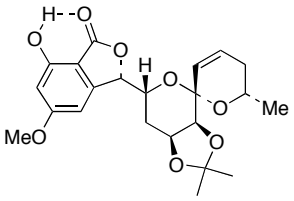
16: colorless thick oil; R_f = 0.34 [35% EtOAc in hexanes];

^{1}H NMR (400 MHz, C_6D_6) □ 0.15 (s, 3H), 0.21 (s, 3H), 0.79 (t, *J* = 7.1 Hz, 3H), 0.98 (s, 9H), 0.99 (d, *J* = 6.2 Hz, 3H), 1.09 (t, *J* = 7.1 Hz, 3H), 1.19 (s, 3H), 1.34-1.40 (m, 1H), 1.55 (s, 3H), 1.62 (ddt, *J* = 13.6,

9.8, 2.3 Hz, 1H), 1.86-1.90 (m, 1H), 2.52-2.58 (m, 1H), 2.94 (q, J = 7.1 Hz, 2H), 3.10 (sextet, J = 7.2 Hz, 1H), 3.39 (s, 3H), 3.49-3.60 (m, 2H), 3.97 (d, J = 7.6 Hz, 1H), 4.32 (dt, J = 7.8, 2.7 Hz, 1H), 4.58 (s, 1H), 5.24-5.27 (m, 2H), 5.63 (ddd, J = 10.3, 5.8, 2.0 Hz, 1H), 6.16 (dd, J = 10.3, 1.8 Hz, 1H), 6.58 (d, J = 2.5 Hz, 1H), 7.39 (d, J = 2.5 Hz, 1H); mass spectrum (APCI): m/e (% relative intensity) 606 (100) (M + H)$^+$, 588 (33) ([M+H]$^+$-H_2O), 570 (20), 548 (8) ([M+H]$^+$-tBu), 530 (15), 512 (10), 475 (15).

17: colorless thick oil; R_f = 0.20 [35% EtOAc in hexane];
^{1}H NMR (400 MHz, C_6D_6) □ 0.15 (s, 3H), 0.22 (s, 3H), 0.99 (d, J = 6.2 Hz, 3H), 1.01 (s, 9H), 1.07 (t, J = 7.1 Hz, 3H), 1.21 (s, 3H), 1.28 (t, J = 7.0 Hz, 3H), 1.30-1.40 (m, 1H), 1.52 (s, 3H), 1.52-1.62 (m, 1H), 1.93 (ddd, J = 15.0, 4.3, 3.1 Hz, 1H), 2.62 (ddd, J = 15.0, 12.0, 2.7 Hz, 1H), 2.96-3.05 (m, 1H), 3.26 (qd, J = 7.7, 1.1 Hz, 2H), 3.32 (s, 3H), 3.48-3.53 (m, 1H), 3.94 (d, J = 7.6 Hz, 1H), 4.00-4.09 (m, 1H), 4.28 (dt, J = 7.8, 2.5 Hz, 1H), 4.40 (s, 1H), 4.69 (dt, J = 12.1, 4.1 Hz, 1H), 5.60-5.64 (m, 2H), 6.06 (dd, J = 10.4, 1.9 Hz, 1H), 6.55 (d, J = 2.3 Hz, 1H), 7.20 (d, J = 2.3 Hz, 1H); mass spectrum (APCI): m/e (% relative intensity) 606 (100) (M + H)$^+$, 588 (25) ([M+H]$^+$-H_2O), 570 (15), 548 (7) ([M+H]$^+$-tBu), 530 (10), 512 (8), 475 (15).

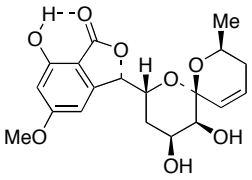
Data as mixture **16** + **17**: [□]$_D^{20}$ = + 24.3 (c 0.070, C_6H_6); IR (neat) cm^{-1} 3430brm, 3242brm, 2936brm, 1767m, 1675m, 1606s, 1464s, 1257m, 1160s, 1023s, 840s; HRMS (ESI) calcd for $C_{32}H_{51}NO_8NaSi$ 628.3282, found 628.3287.



18

To a solution of alcohol **16** (7.20 mg, 0.0119 mmol) in a ternary solvent mixture consisting of MeOH (0.6 mL), THF (0.2 mL), and H_2O (0.06 mL) was added KOH (6.70 mg, 0.119 mmol). The resulting mixture was stirred at rt overnight before being partitioned between H_2O (5 mL) and CH_2Cl_2 (5 mL). The mixture was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated *in vacuo* to give a crude oil that was purified via preparative TLC (eluted with 5% MeOH in CH_2Cl_2) to afford lactone **18** in 71% yield (2.60 mg) as a thick oil along with the recovered but desilylated starting material (1.70 mg, 29%).
R_f = 0.75 [5% MeOH in CH_2Cl_2];
[□]$_D^{20}$ = - 37.9 (c 0.68, $CHCl_3$);
^{1}H NMR (400 MHz, $CDCl_3$) □ 1.30 (d, J = 6.2 Hz, 3H), 1.33 (s, 3H), 1.44 (s, 3H), 1.99 (ddt, J = 17.3, 7.6, 3.0 Hz, 1H), 2.09-2.18 (m, 2H), 2.40 (ddd, J = 14.5, 11.3, 3.1 Hz, 1H), 3.83 (s, 3H), 3.99-4.06 (m,

2H), 4.14 (d, J = 7.6 Hz, 1H), 4.60 (dt, J = 7.6, 2.7 Hz, 1H), 5.34 (d, J = 8.0 Hz, 1H), 5.88 (dt, J = 10.3, 2.0 Hz, 1H), 6.07 (ddd, J = 10.2, 4.8, 3.3 Hz, 1H), 6.43 (d, J =1.7 Hz, 1H), 6.69 (d, J = 0.9 Hz, 1H), 7.65 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) □21.9, 24.5, 26.5, 27.5, 31.1, 56.1, 68.3, 70.2, 70.4, 74.9, 84.2, 95.5, 101.2, 103.0, 104.5, 109.4, 127.4, 127.8, 150.5, 157.7, 167.2, 171.9; IR (neat) cm^{-1} 2928w, 1733s, 1612s, 1480m, 1372m, 1320m, 1253m, 1213m, 1153s, 1032s, 849m; mass spectrum (APCI): m/e (% relative intensity) 419 (3) $(M + H)^+$, 401 (7), 361 (100), 343 (25), 317 (20), 167 (5); HRMS (ESI) calcd for $C_{22}H_{26}O_8Na$ 441.1525, found 441.1529.



(+)-**1**: aigialospirol

To a solution of lactone **18** (28.0 mg, 0.067 mmol) in MeOH (4 mL) was added *p*-TsOH (2.70 mg, 0.014 mmol, practical grade). The mixture was stirred at rt for 4 h before it was concentrated *in vacuo* and diluted with CH_2Cl_2 (10 mL). The solution was washed with sat aq $NaHCO_3$ (10 mL). The aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL), and the combined organic layers were dried (Na_2SO_4) and concentrated *in vacuo*. The resulting crude residue was purified via preparative TLC (eluted with 5% MeOH in CH_2Cl_2) to afford (+)-**1**-aigialospiol in 53% yield (13.4 mg) as a white solid.

R_f = 0.49 [5% MeOH in CH_2Cl_2];

mp 90-94 °C; lit.[ref] 85-89 °C

$[□]_D^{20}$ = + 79.2 (*c* 0.67, $CHCl_3$); lit.[ref] $[□]_D^{25}$ = + 47 (*c* 0.50, $CHCl_3$)

^{1}H NMR (500 MHz, $CDCl_3$) □1.27 (d, J = 6.1 Hz, 3H), 1.89 (ddd, J = 14.2, 11.9, 2.6 Hz, 1H), 2.03-2.08 (m, 3H), 2.54 (d, J = 10.5 Hz, 1H), 3.44 (d, J = 10.5 Hz, 1H), 3.52 (dd, J = 9.9, 3.3 Hz, 1H), 3.86 (s, 3H), 4.01 (m, 1H), 4.12-4.18 (m, 2H), 5.36 (d, J = 5.9 Hz, 1H), 5.67 (dt, J = 10.0, 2.0 Hz, 1H), 6.17 (dt, J = 10.0, 4.0 Hz, 1H), 6.48 (d, J = 1.7 Hz, 1H), 6.60 (s, 1H), 7.68 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) □21.1, 31.4, 33.6, 56.0, 65.0, 65.8, 68.3, 70.7, 82.3, 99.1, 101.2, 101.6, 104.3, 126.5, 130.5, 149.1, 157.7, 167.3, 171.3; IR ($CHCl_3$ film) cm^{-1} 3452brm, 3013w, 2970w, 2930w, 1736s, 1612s, 1444m, 1381m, 1316m, 1216m, 1154s, 1063s, 997s, 843w; mass spectrum (APCI): m/e (% relative intensity) 379 (15) $(M + H)^+$, 361 (100) $([M+H]-H_2O)^+$, 343 (25), 317 (12), 307 (10), 253 (37), 235 (16), 219 (7), 146 (98), 127 (10), 116 (7), 101 (30); HRMS (ESI) calcd for $C_{19}H_{22}O_8Na$ 401.1212, found 401.1208.

Reference: Vongvilai, P.; Isaka, M.; Kittakoop, P. Prasert Srikitikulchai, P.; Kongsaeree, P.; Thebtaranonth, Y. *J. Nat. Prod.* **2004**, *67*, 457.

NMR Data for the Synthetic Aigialospirol ^{1}H NMR with internal reference $CDCl_3$ set at δ = 7.28 ppm; ^{13}C NMR with internal reference $CDCl_3$ set at δ = 77.0 ppm.		**Reported Data for (+)-Aigialospirol*** ** ^{1}H NMR with internal reference $CDCl_3$ set at δ = 7.28 ppm; ^{13}C NMR with internal reference $CDCl_3$ set at δ = 77.0 ppm.	
^{1}H NMR, δ ($\Delta\delta$ with lit. data)	^{13}C NMR, δ ($\Delta\delta$ with lit. data)	^{1}H NMR, δ	^{13}C NMR, δ
1.27 (0.01)	21.1 (0.0)	1.26	21.1
1.89 (0.01)	31.4 (0.0)	1.88	31.4
2.03-2.08	33.6 (0.0)	2.03-2.05	33.6
2.54	56.0 (0.0)		56.0
3.44	65.0 (0.0)		65.0
3.52 (0.00)	65.8 (0.0)	3.52	65.8
3.86 (0.00)	68.3 (0.0)	3.86	68.3
4.01 (0.00)	70.7 (0.0)	4.01	70.7
4.12-4.18	82.3 (0.0)	4.13	82.3
	99.1 (0.0)	4.15	99.1
5.36 (0.00)	101.2 (0.0)	5.36	101.2
5.67 (0.01)	101.6 (0.0)	5.66	101.6
6.17 (0.01)	104.3 (0.0)	6.16	104.3
6.48 (0.01)	126.5 (0.0)	6.47	126.5
6.60 (0.01)	130.5 (0.0)	6.59	130.5
7.68	149.1 (0.0)		149.1
	157.7 (0.0)		157.7
	167.3 (0.0)		167.3
	171.3 (0.0)		171.3

***We again thank Professor Masahiko Isaka for kindly providing the original spectra data of (+)-aigialospirol.

References

1 Marshall, J. A.; Sabatini, J. J. *Org. Lett.* **2005**, *7*, 4819.

2 Kumar, P.; Gupta, P.; Naidu, S. V *Chem. Eur. J.* **2006**, *12*, 1397.

3 Compounds prepared by following Sellès' conditions: (a) Sellès, P.; Lett, R. *Tetrahedron Lett.* **2002**, *43*, 4621. Also see: (b) Wissner, A.; Grudzinskas, C. *J. Org. Chem.* **1978**, *43*, 3972.

SUPPORTING INFORMATION

PROTON NMR AND SELECTED CARBON NMR SPECTRA

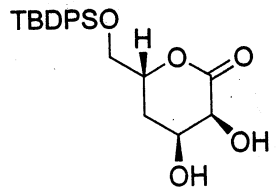
for the
communication
entitled

A Concise Total Synthesis of (+)-Aigialospirol via a Cyclic Ketal-Tethered Ring-Closing Metathesis.

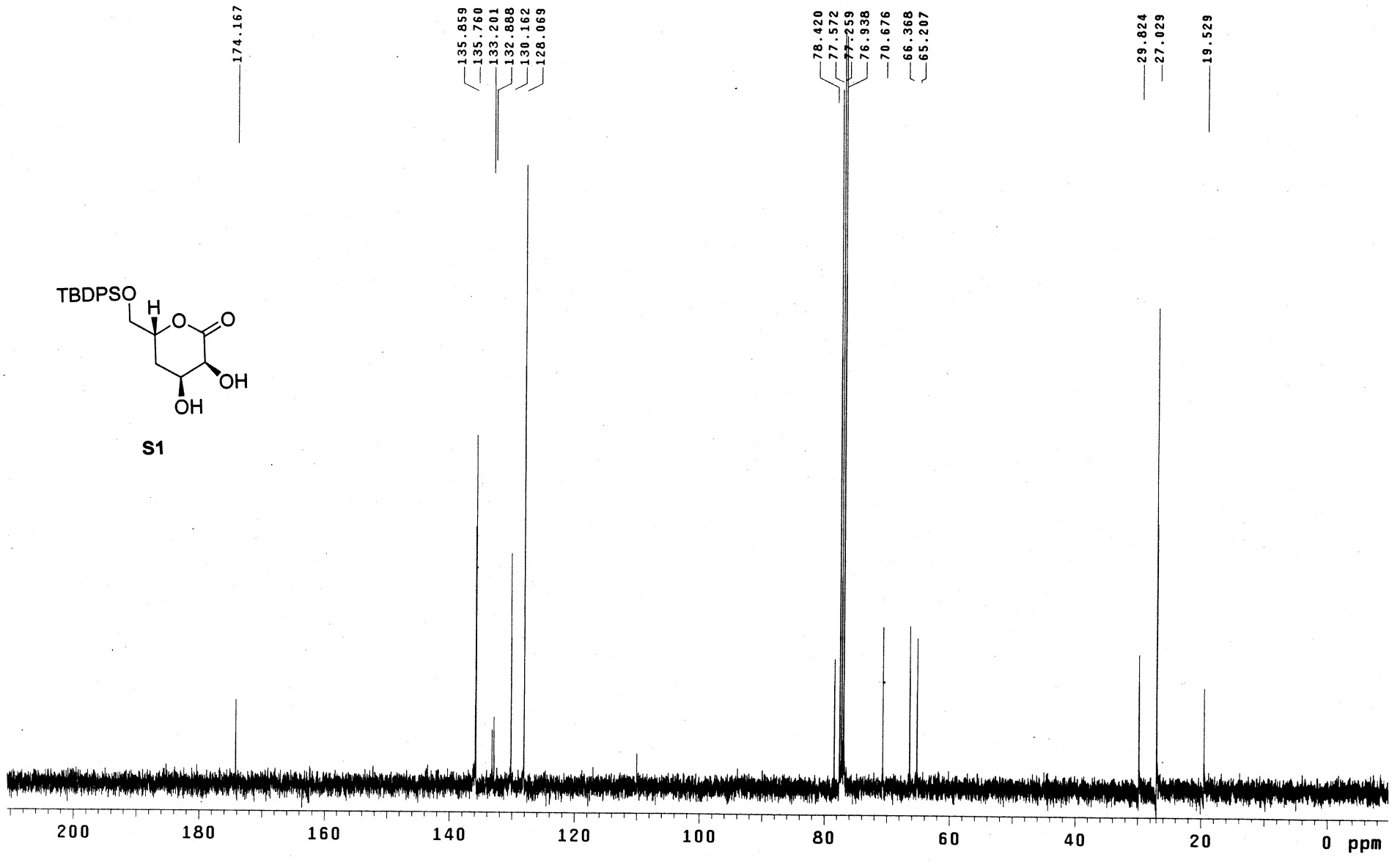
authored by

Ruth Figueroa, Richard P. Hsung*, and Christle C. Guevarra

Division of Pharmaceutical Sciences and Department of Chemistry, 7111 Rennebohm Hall, 777 Highland Avenue
University of Wisconsin at Madison, Madison, WI 53705-2222



S1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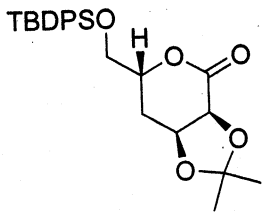

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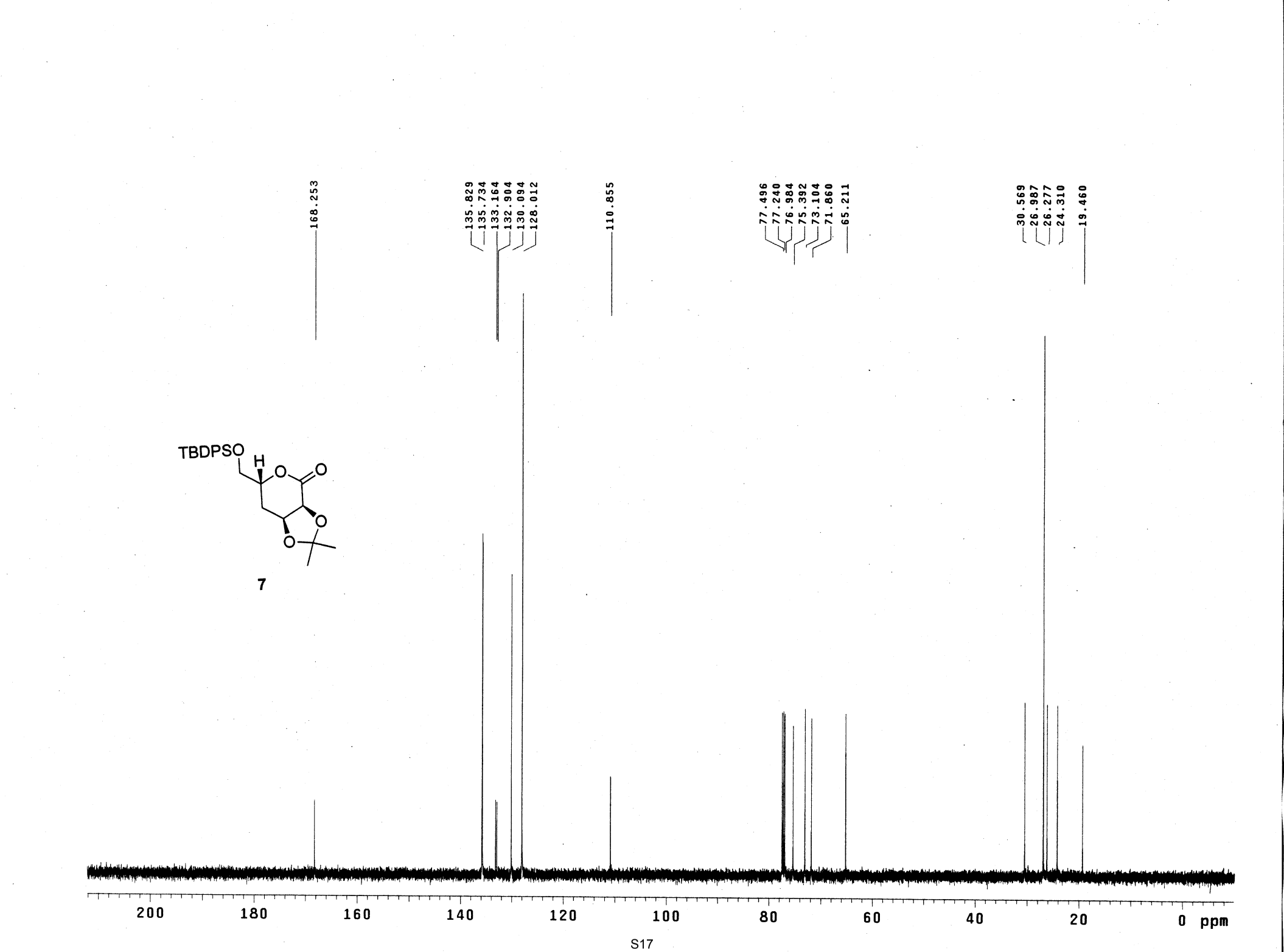
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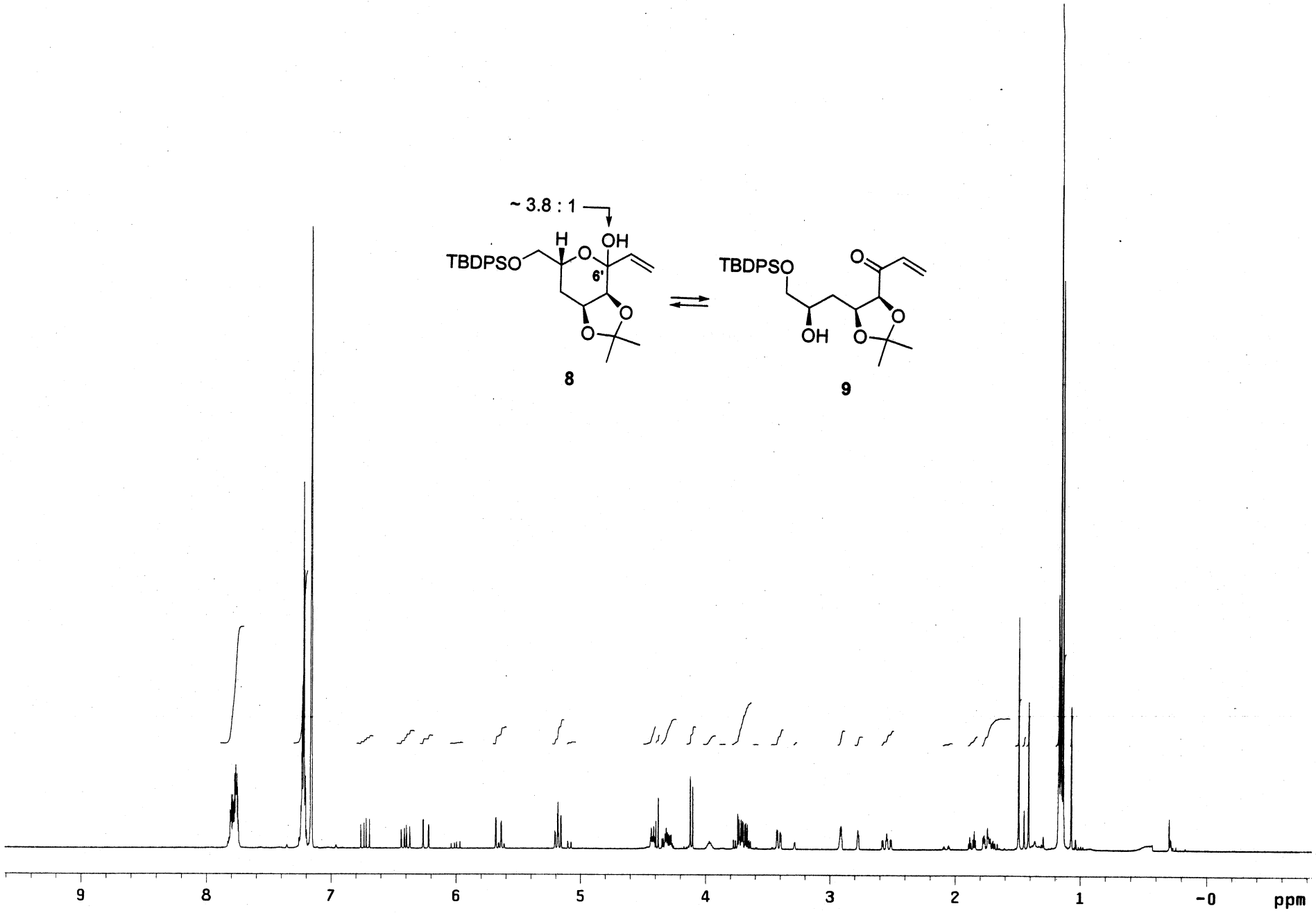
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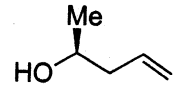

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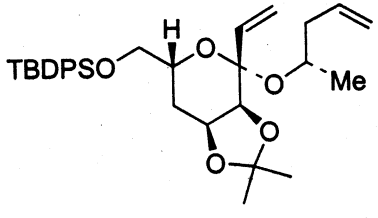
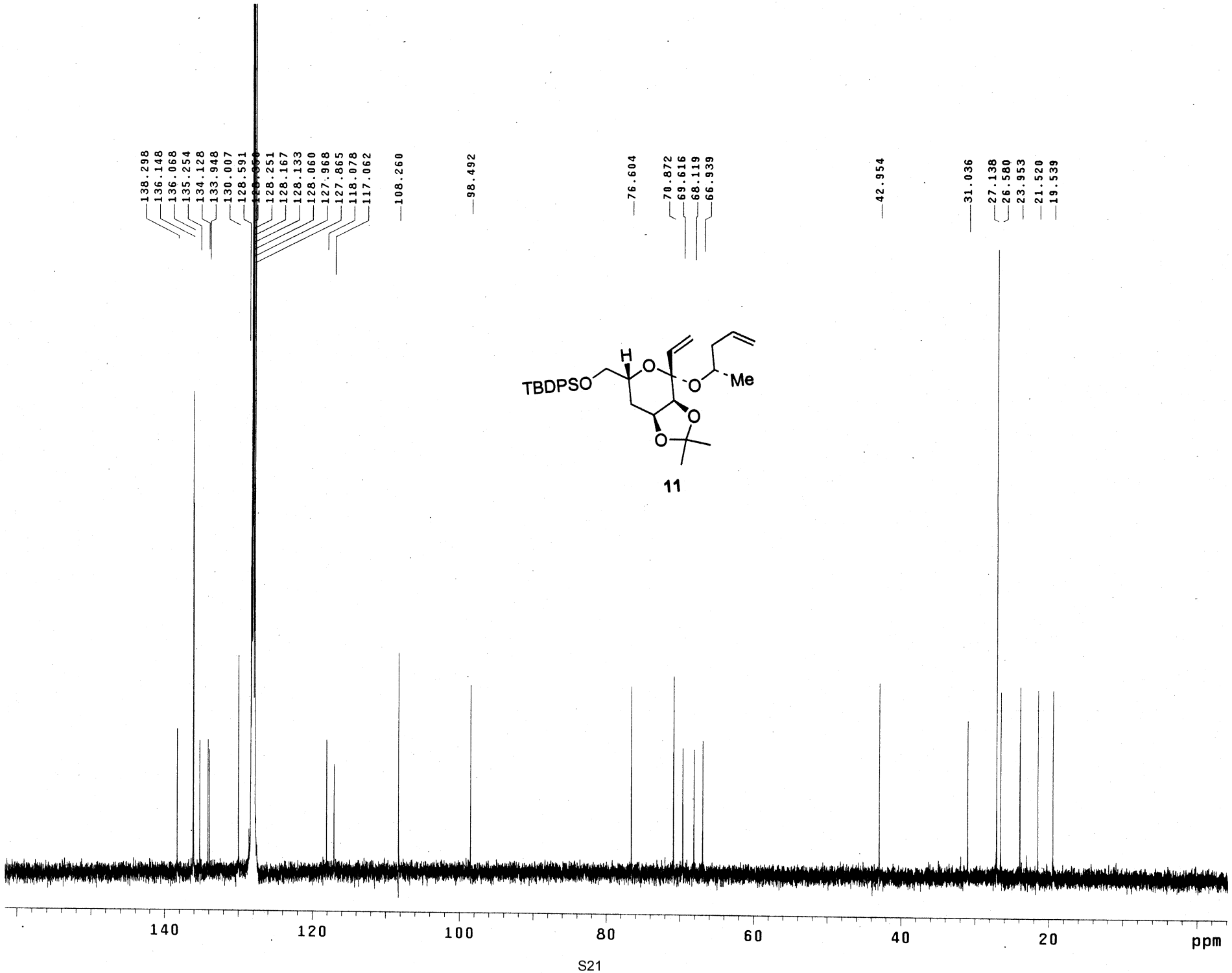


11


138.298
136.148
136.068
135.254
134.128
133.948
130.007
128.591
128.251
128.167
128.133
128.060
127.968
127.865
118.078
117.062
108.260
98.492
76.604
70.872
69.616
68.119
66.939
42.954
31.036
27.138
26.580
23.953
21.520
19.539
TBDPSO
H
O
O
Me
O
O
11
140
120
100
80
60
40
20
ppm

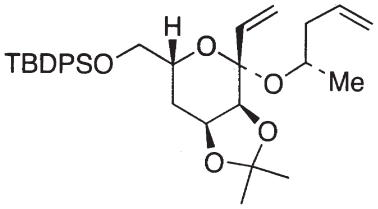
RF-I-103A_10Jul2006

Archive directory: /export/home/ruth/vnmrsys/data
Sample directory: RF-I-103A_10Jul2006

Pulse Sequence: gCOSY

Solvent: C6D6
Temp. 25.0 C / 298.1 K
File: gCOSY
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Acq. time 0.128 sec
Width 8000.0 Hz
2D Width 8000.0 Hz
Single scan
128 increments
OBSERVE H1, 499.7289905 MHz
DATA PROCESSING
Sq. sine bell 0.064 sec
F1 DATA PROCESSING
Sq. sine bell 0.016 sec
FT size 2048 x 2048
Total time 0 min, -1 sec



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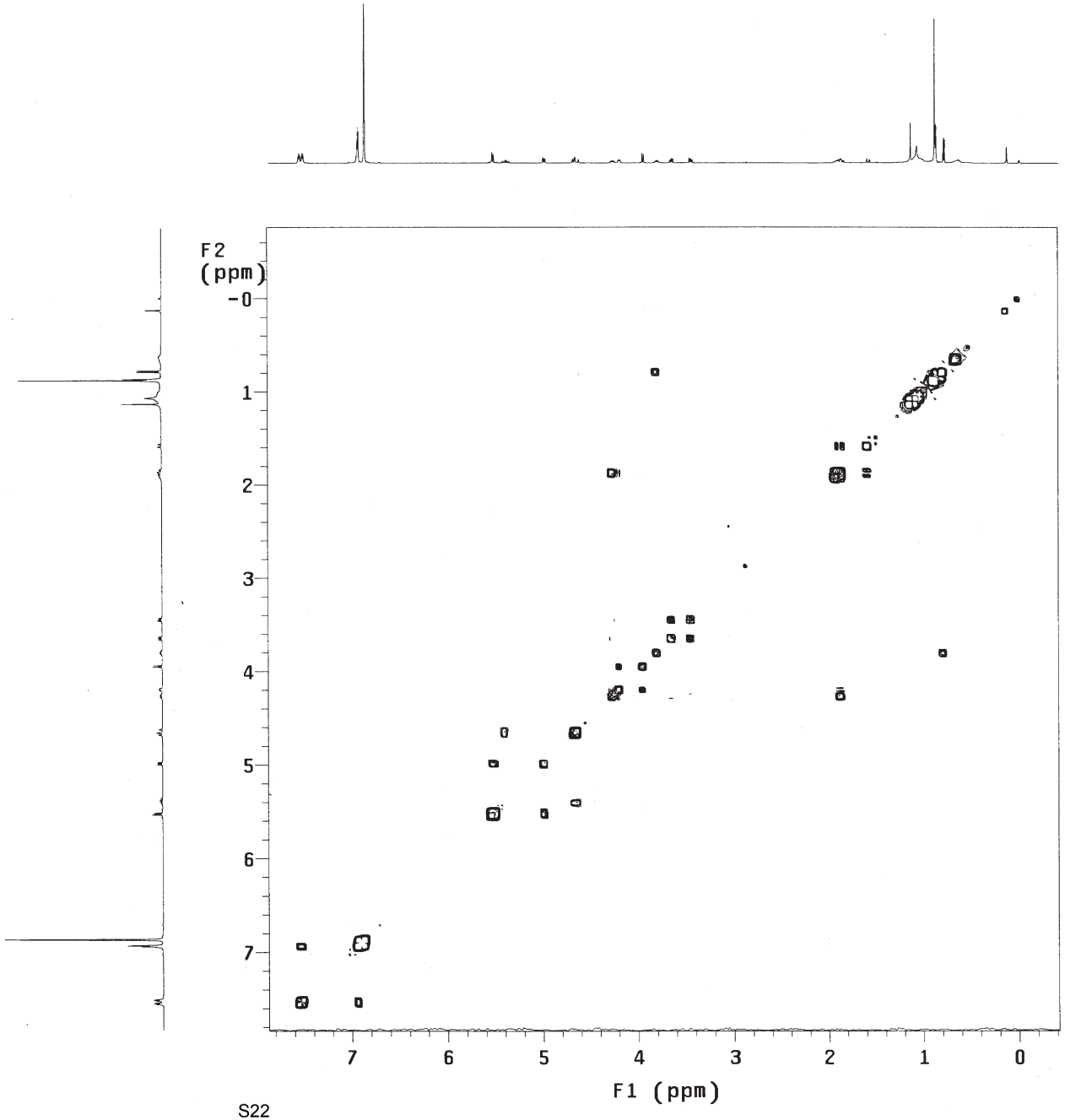

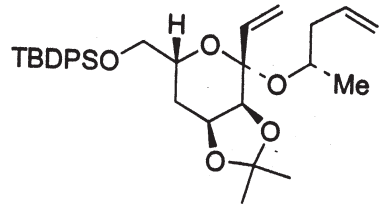

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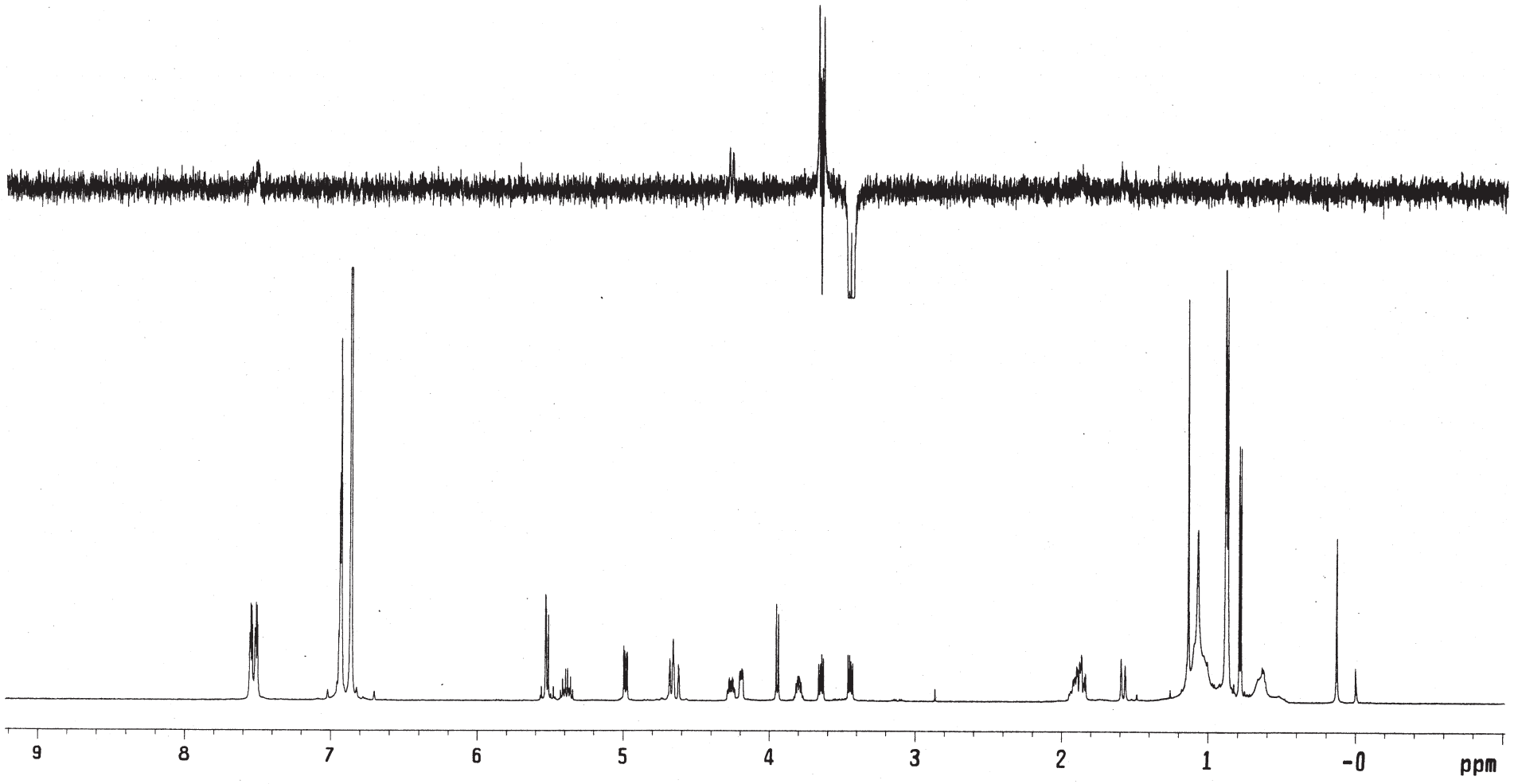

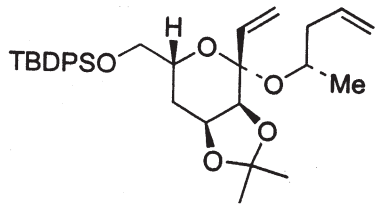

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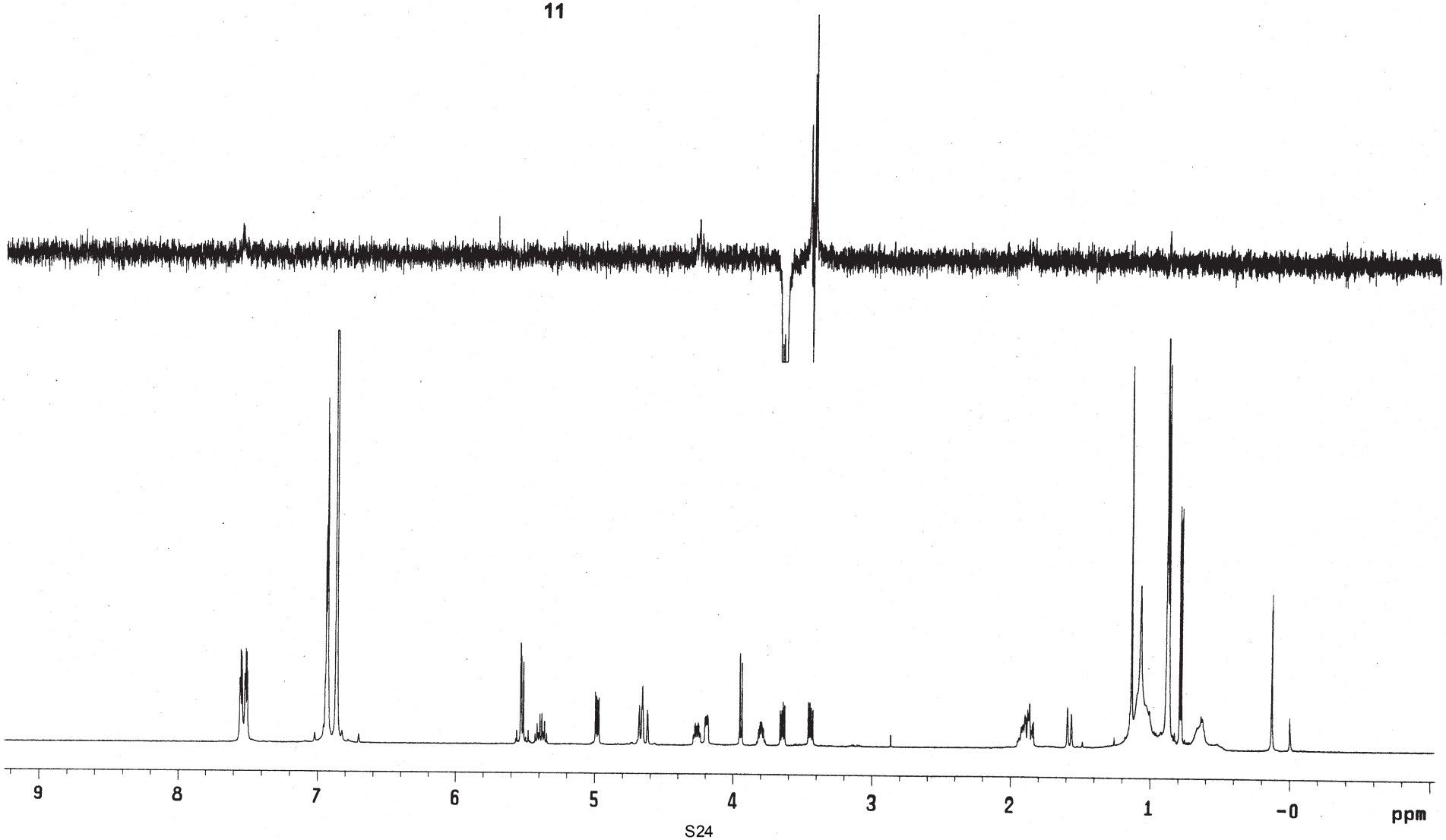

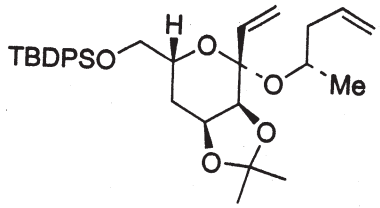

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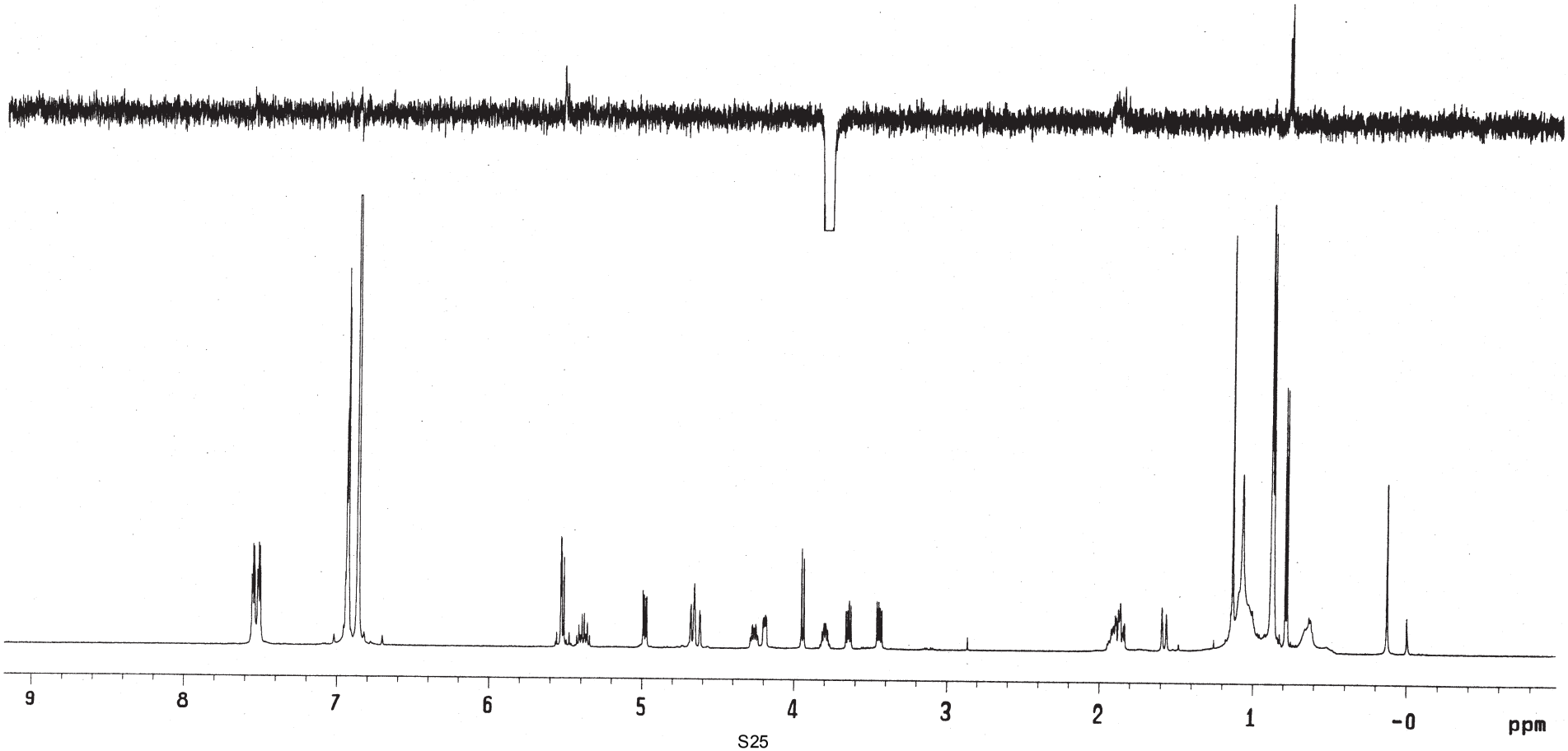

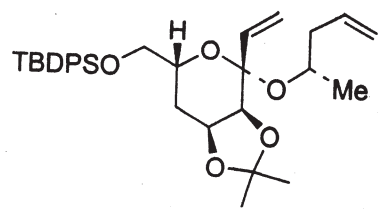

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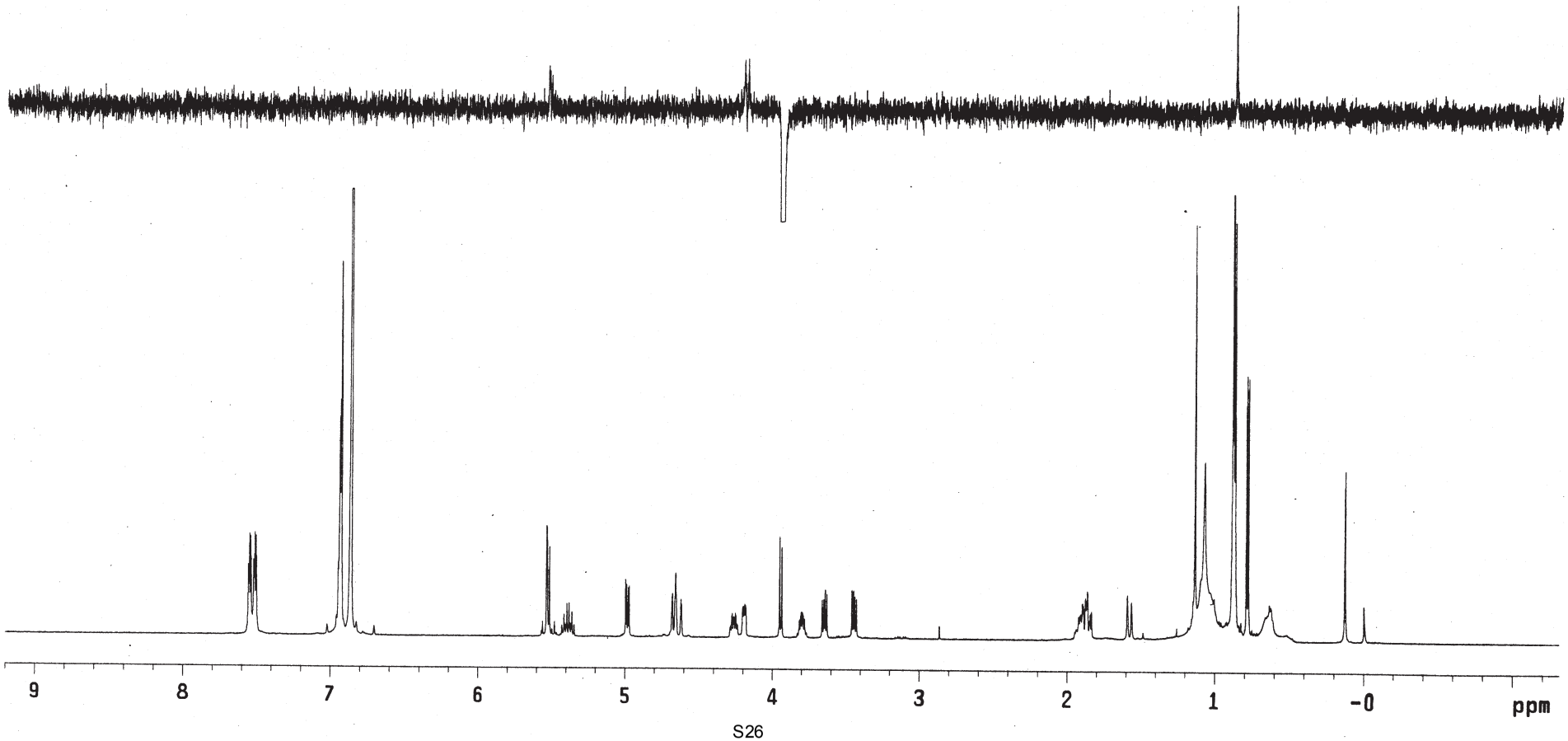

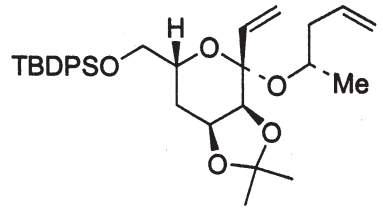

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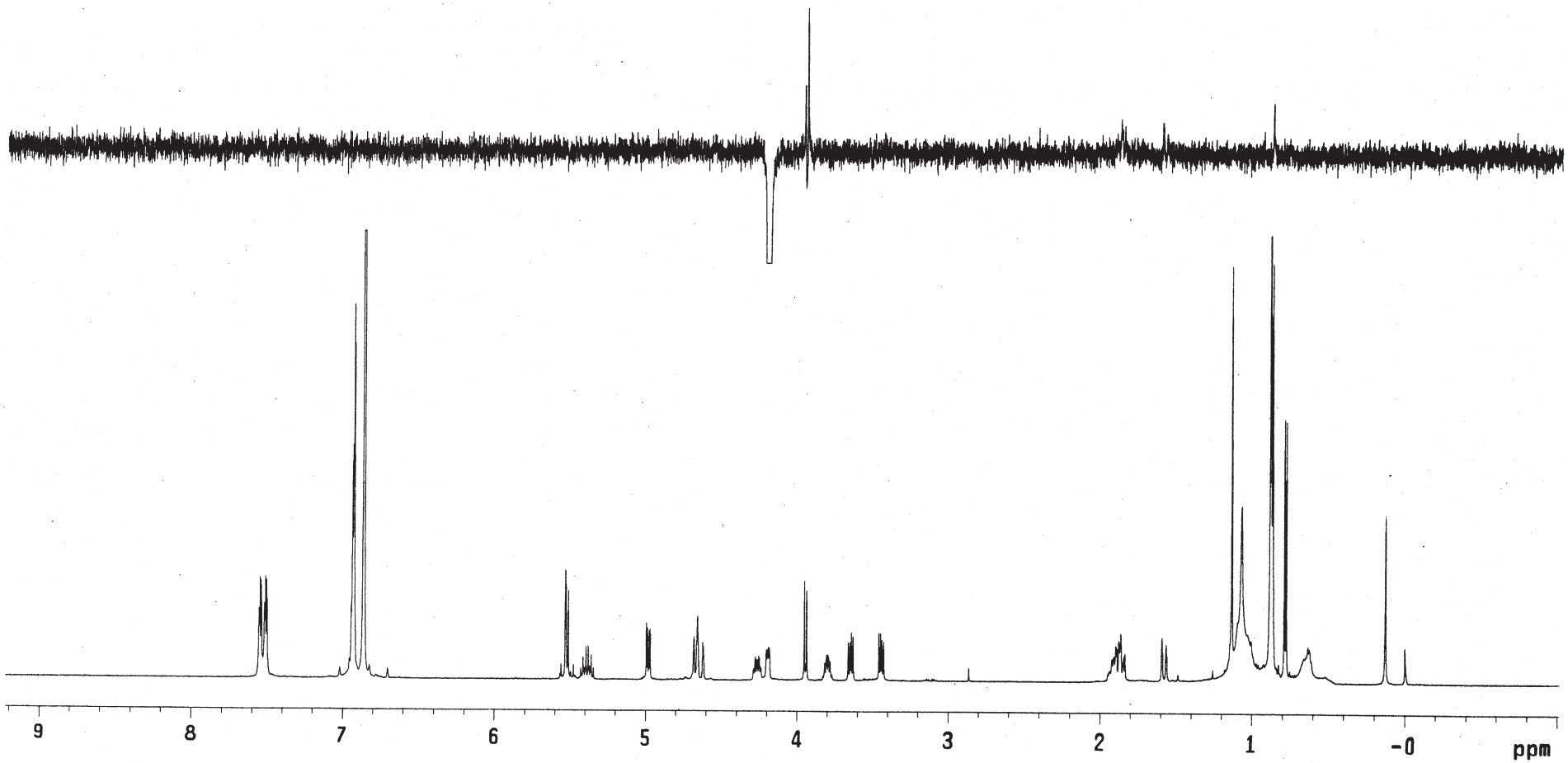

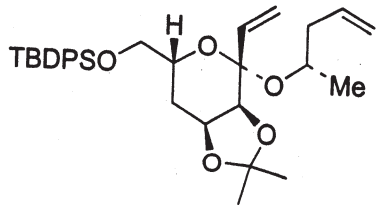

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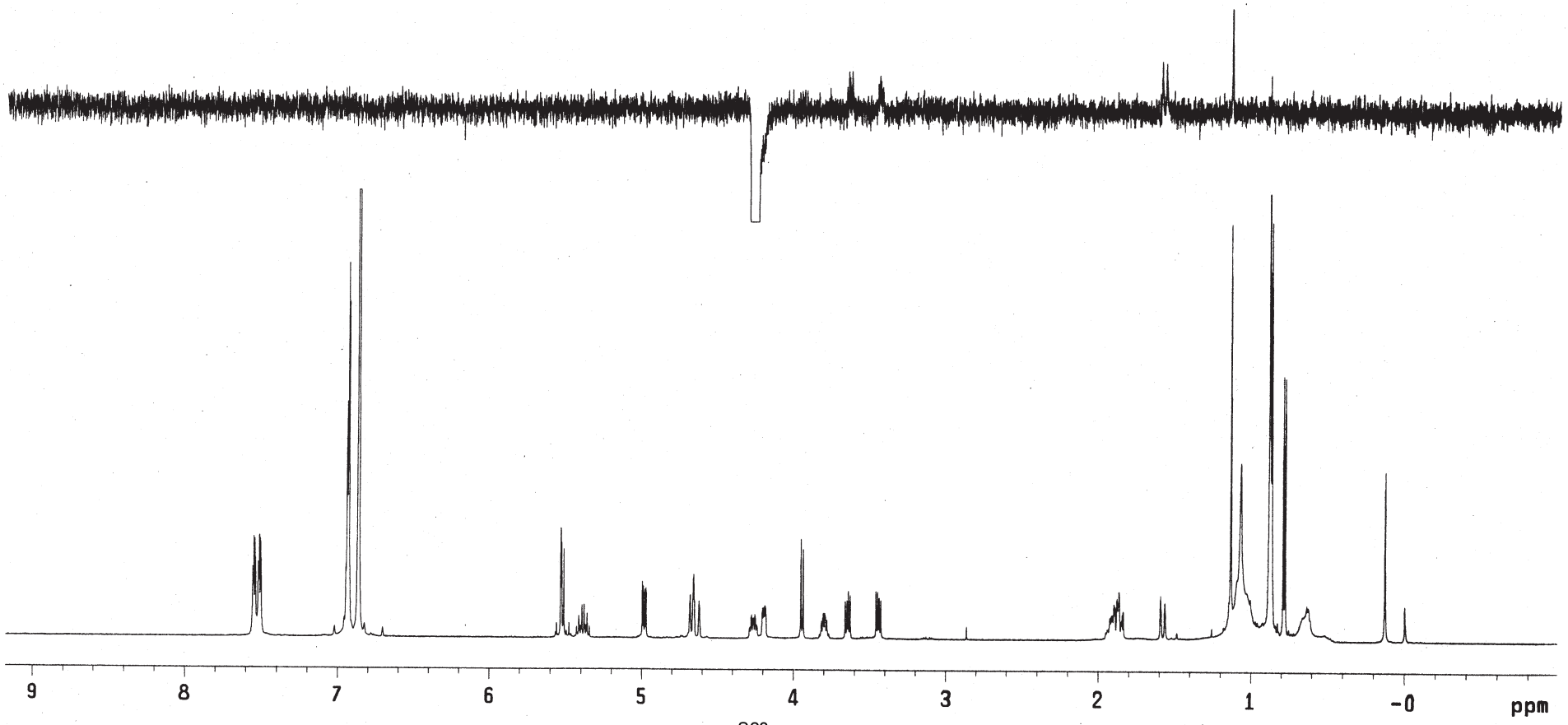

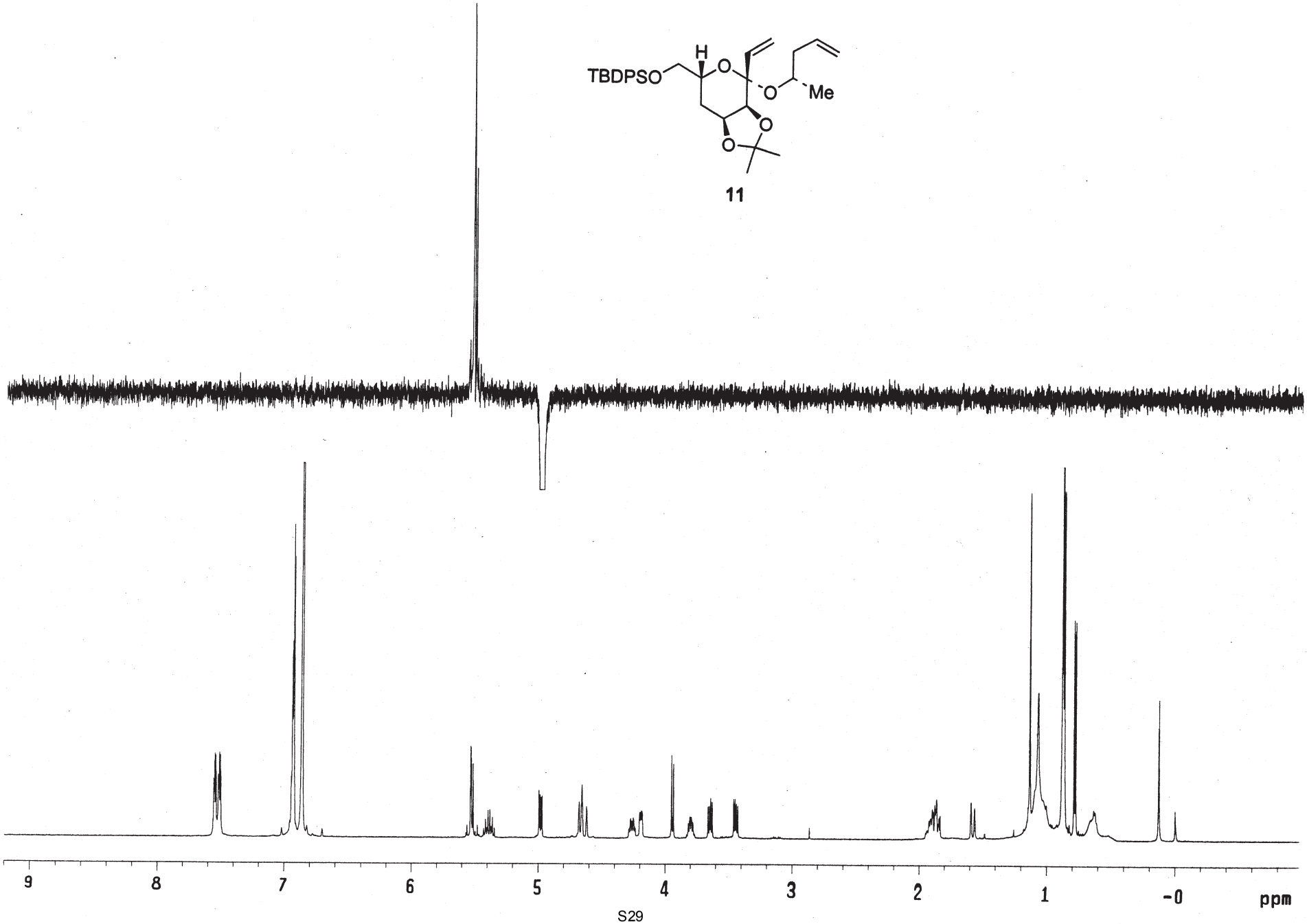

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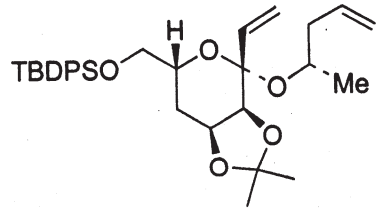

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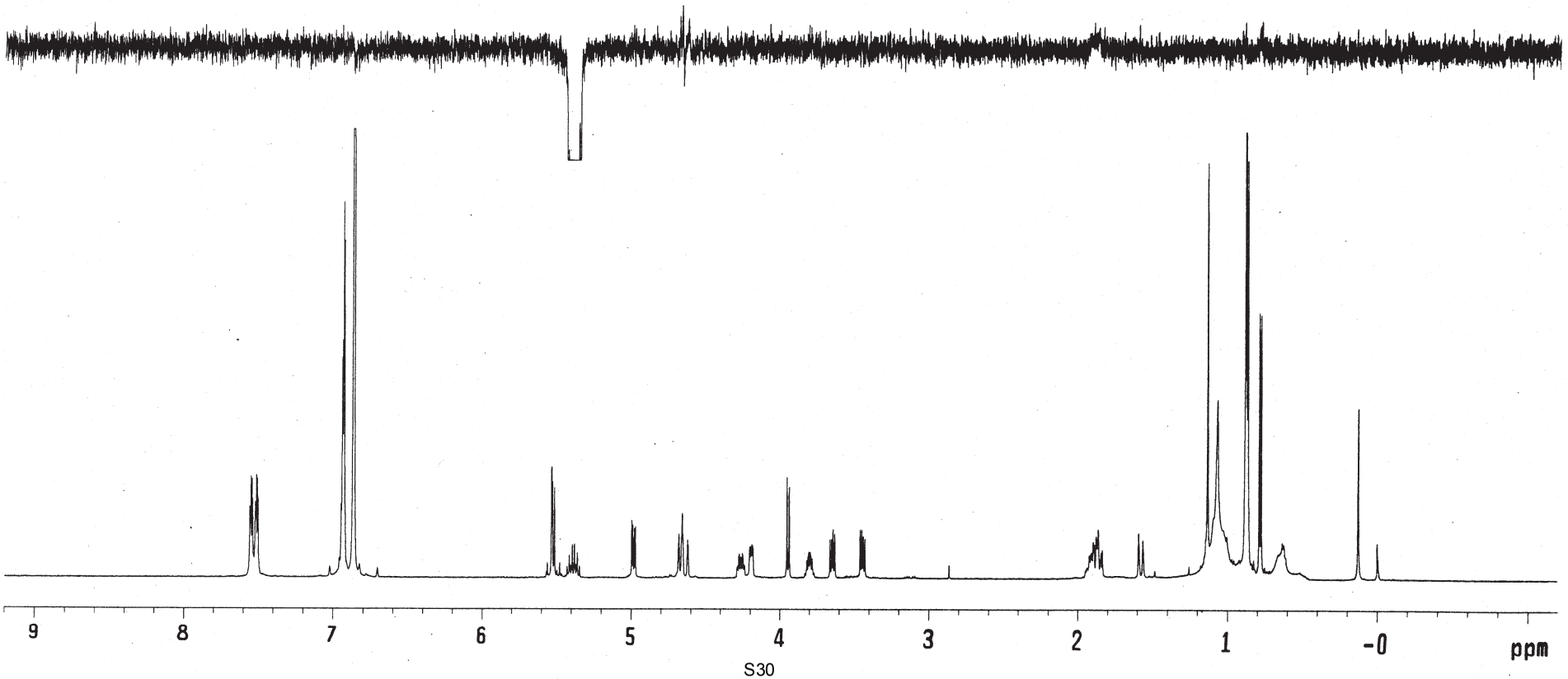
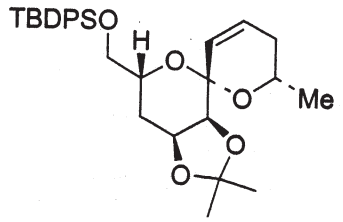
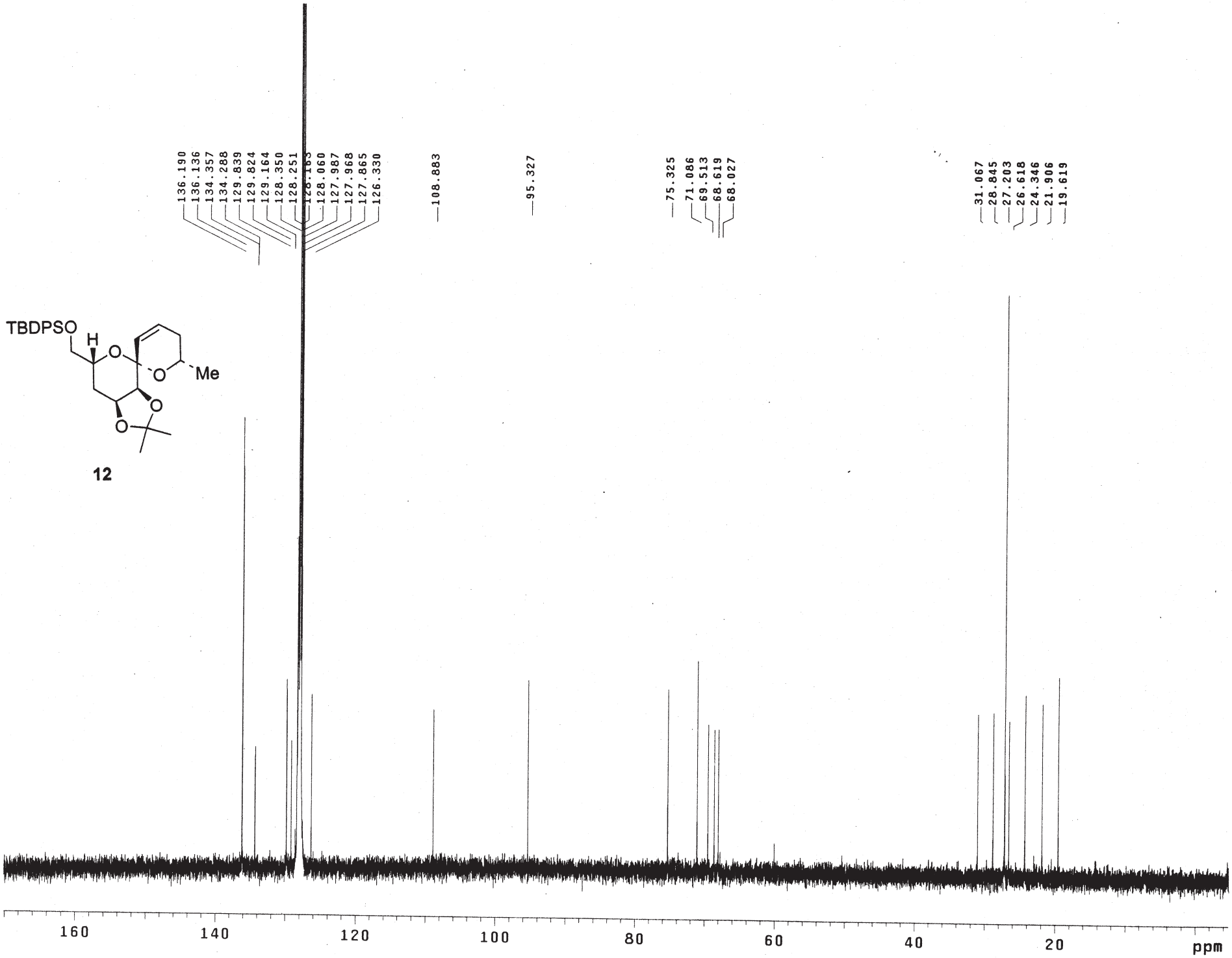
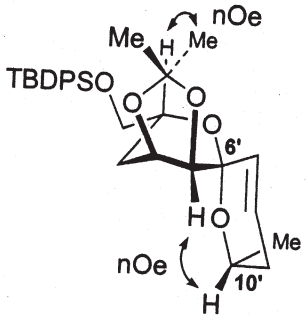




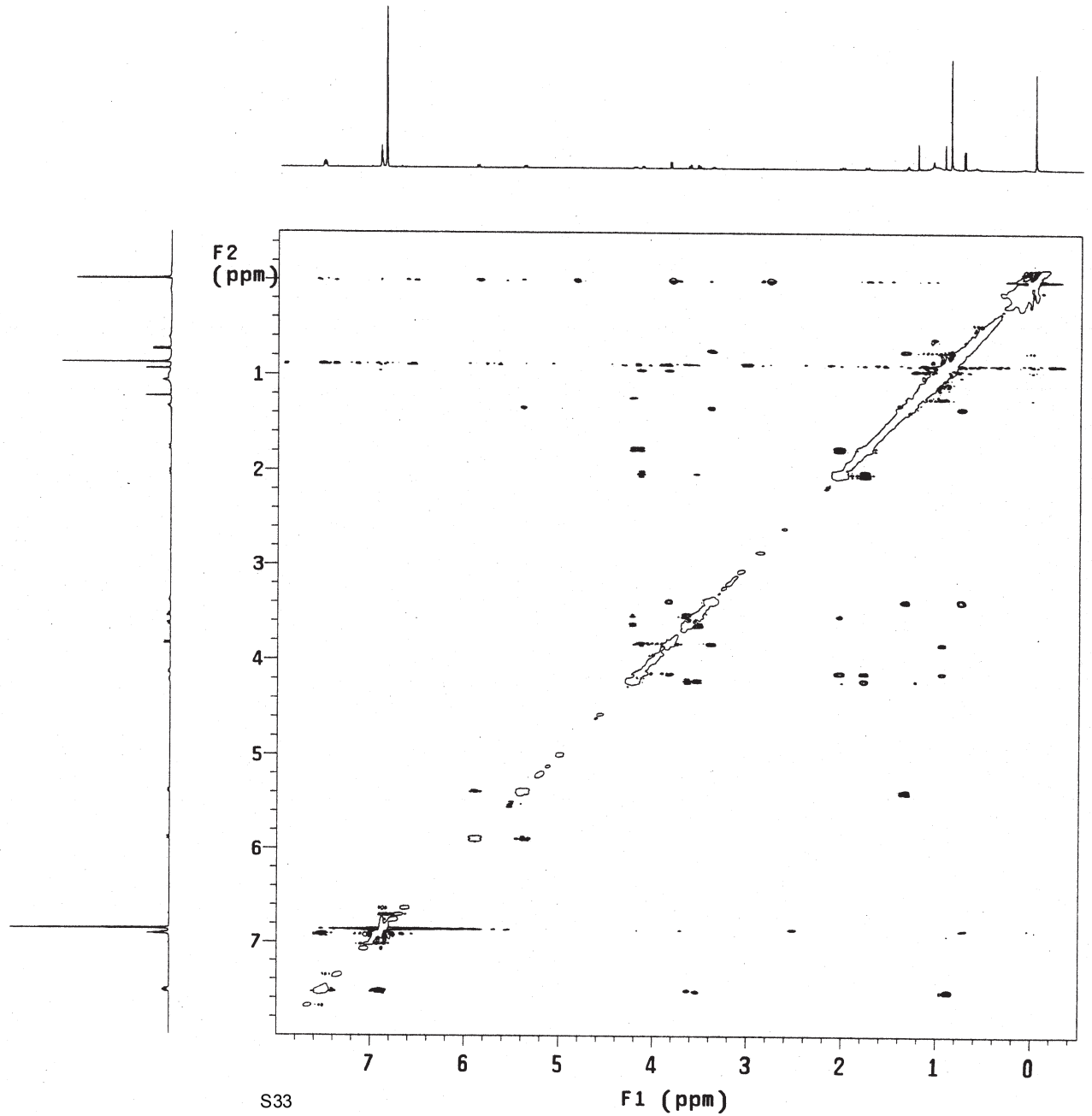
12

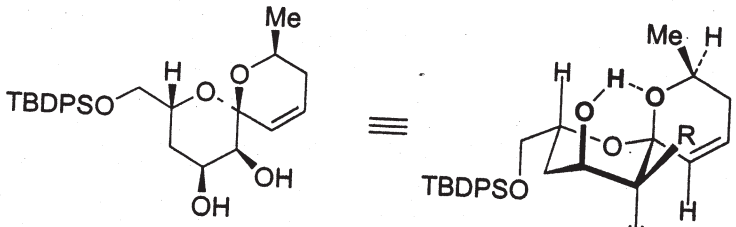

136.190
136.136
134.357
134.288
129.839
129.824
129.164
128.350
128.251
128.163
128.060
127.987
127.968
127.865
126.330
108.883
95.327
75.325
71.086
69.513
68.619
68.027
31.067
28.845
27.203
26.618
24.346
21.906
19.619
TBDPSO
H
O
O
Me
O
O
160
140
120
100
80
60
40
20
ppm

12


nOe
Me
H
Me
TBDPSO
O
O
O
6'
H
O
Me
nOe
10'
H

12

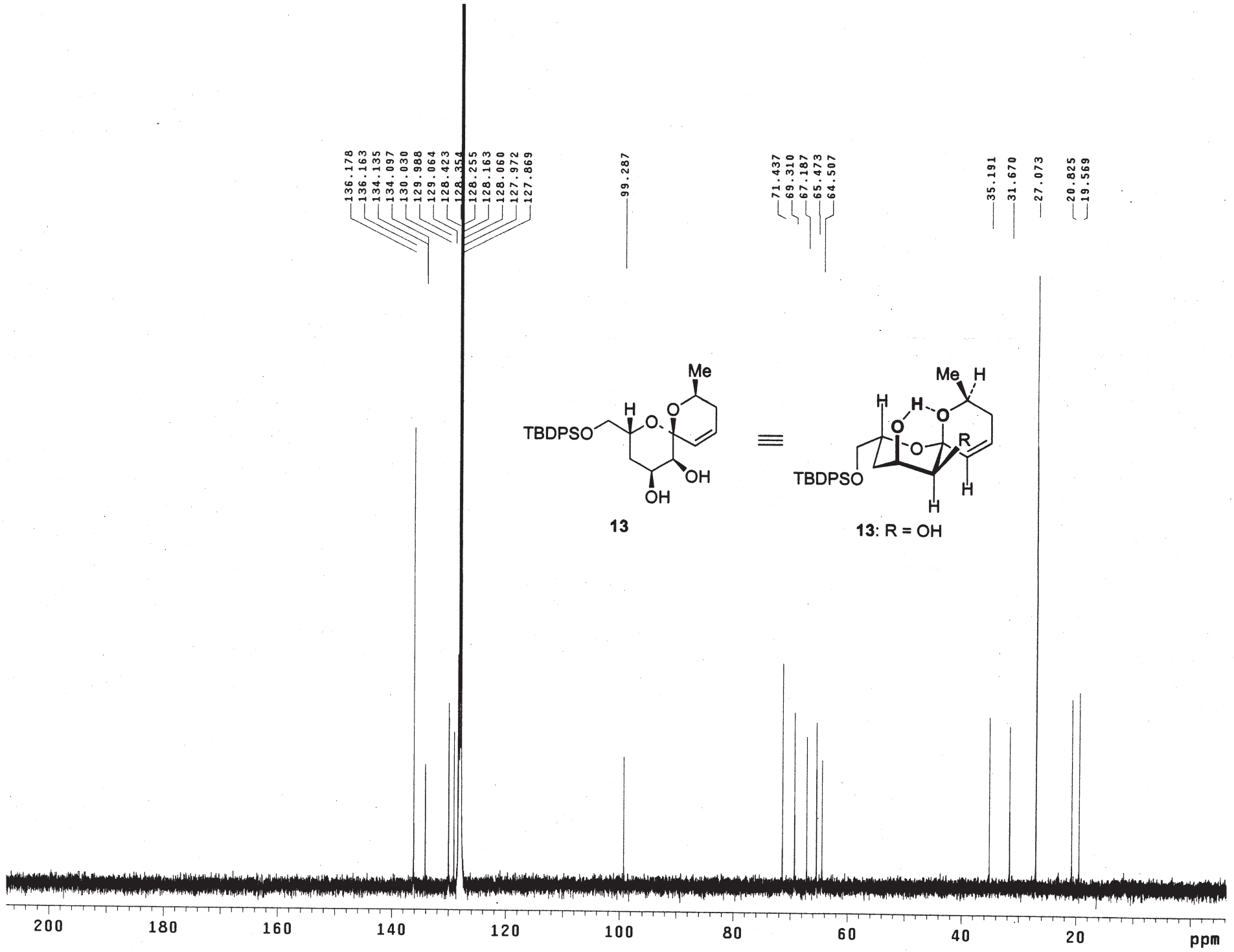

F2
(ppm)
1
2
3
4
5
6
7
7
6
5
4
3
2
1
0
F1 (ppm)

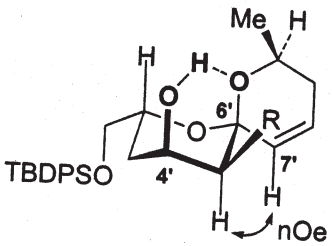


13

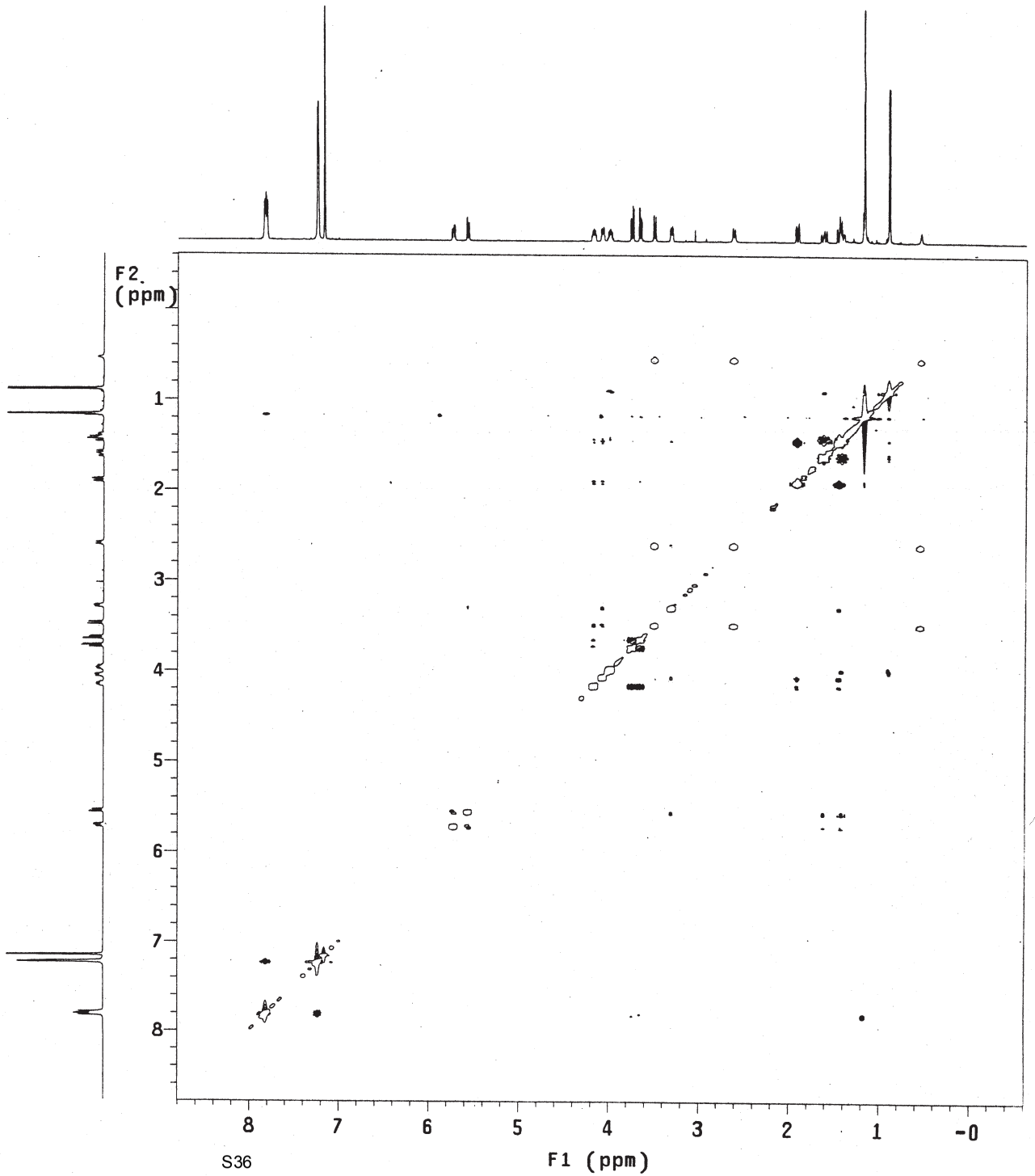
≡

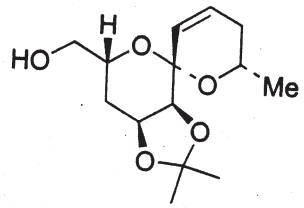
13: R = OH


136.178
136.163
134.135
134.097
130.030
129.988
129.064
128.423
128.354
128.255
128.163
128.060
127.972
127.869
99.287
71.437
69.310
67.187
65.473
64.507
35.191
31.670
27.073
20.825
19.569
Me
H
O
O
TBDPSO
OH
OH
13
≡
Me
H
H
H
O
O
O
O
R
TBDPSO
H
H
13: R = OH
200
180
160
140
120
100
80
60
40
20
ppm

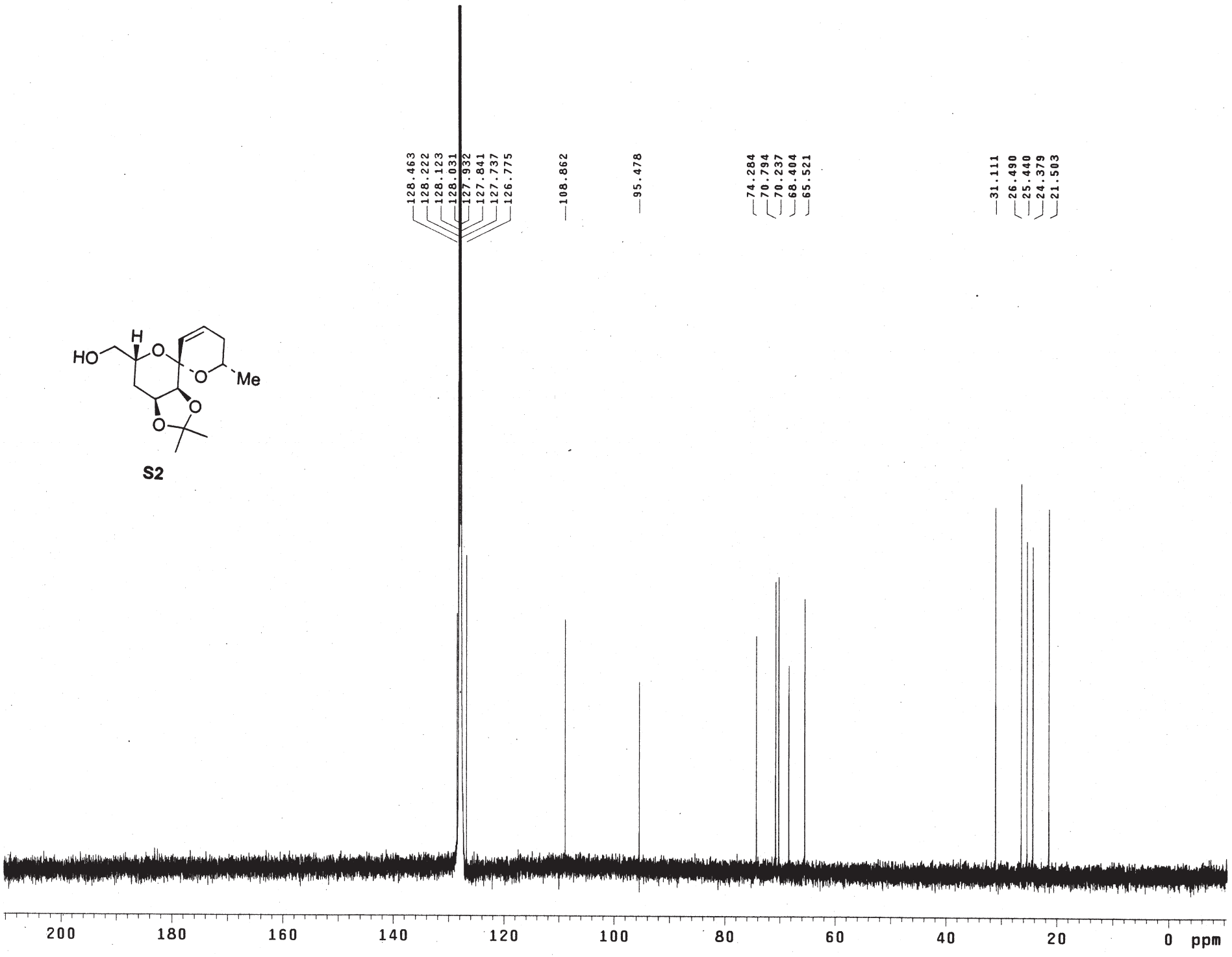

Me
H
H
H
O
O
6'
O
R
TBDPSO
4'
7'
H
H
nOe

13: R = OH

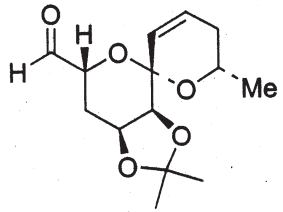

F2 (ppm)
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
-0
F1 (ppm)



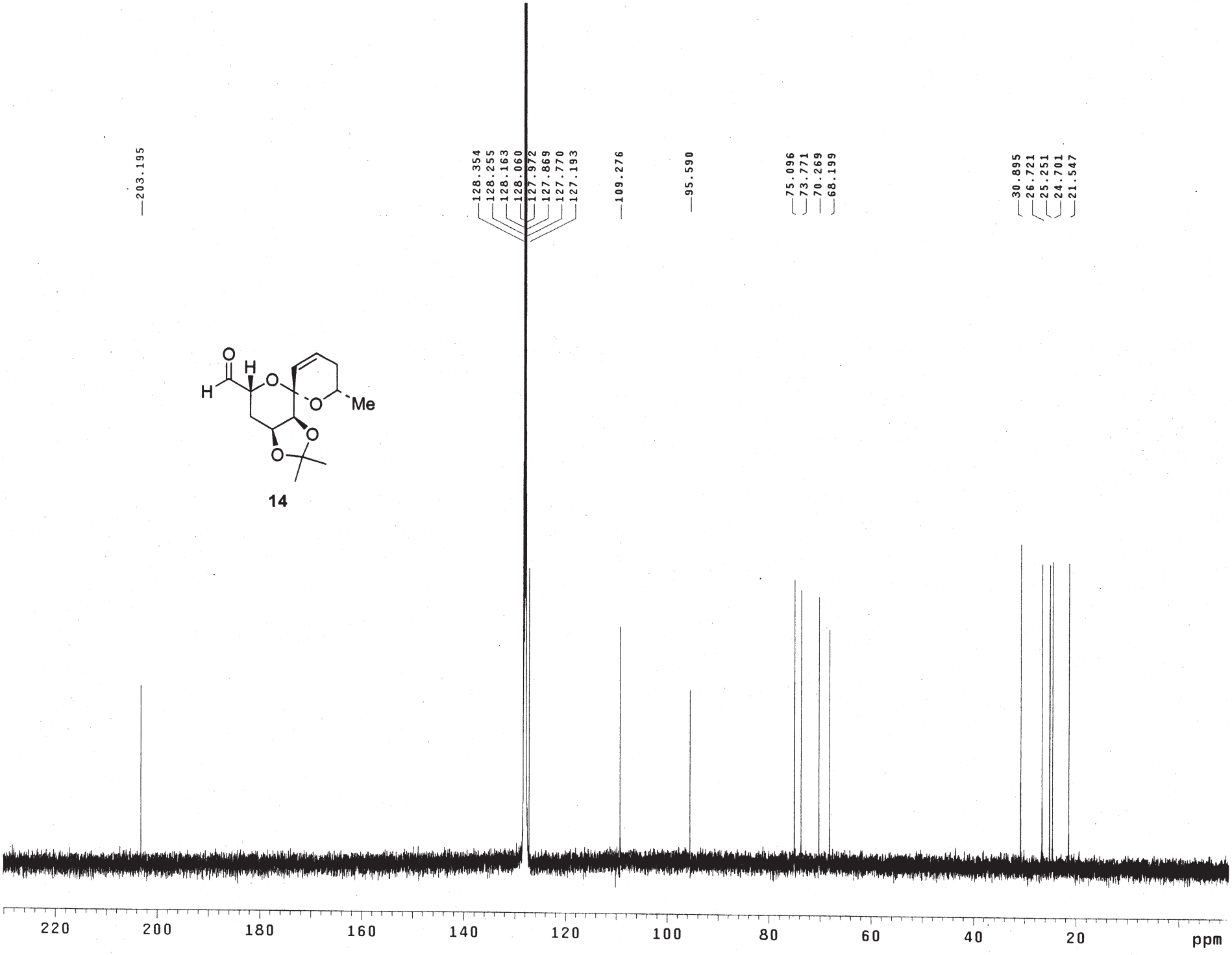
S2


128.463
128.222
128.123
128.031
127.932
127.841
127.737
126.775
108.862
95.478
74.284
70.794
70.237
68.404
65.521
31.111
26.490
25.440
24.379
21.503
HO
H
O
O
Me
O
O
200
180
160
140
120
100
80
60
40
20
0
ppm

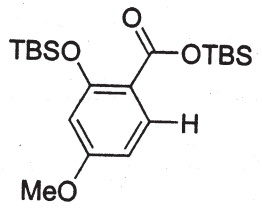
S2



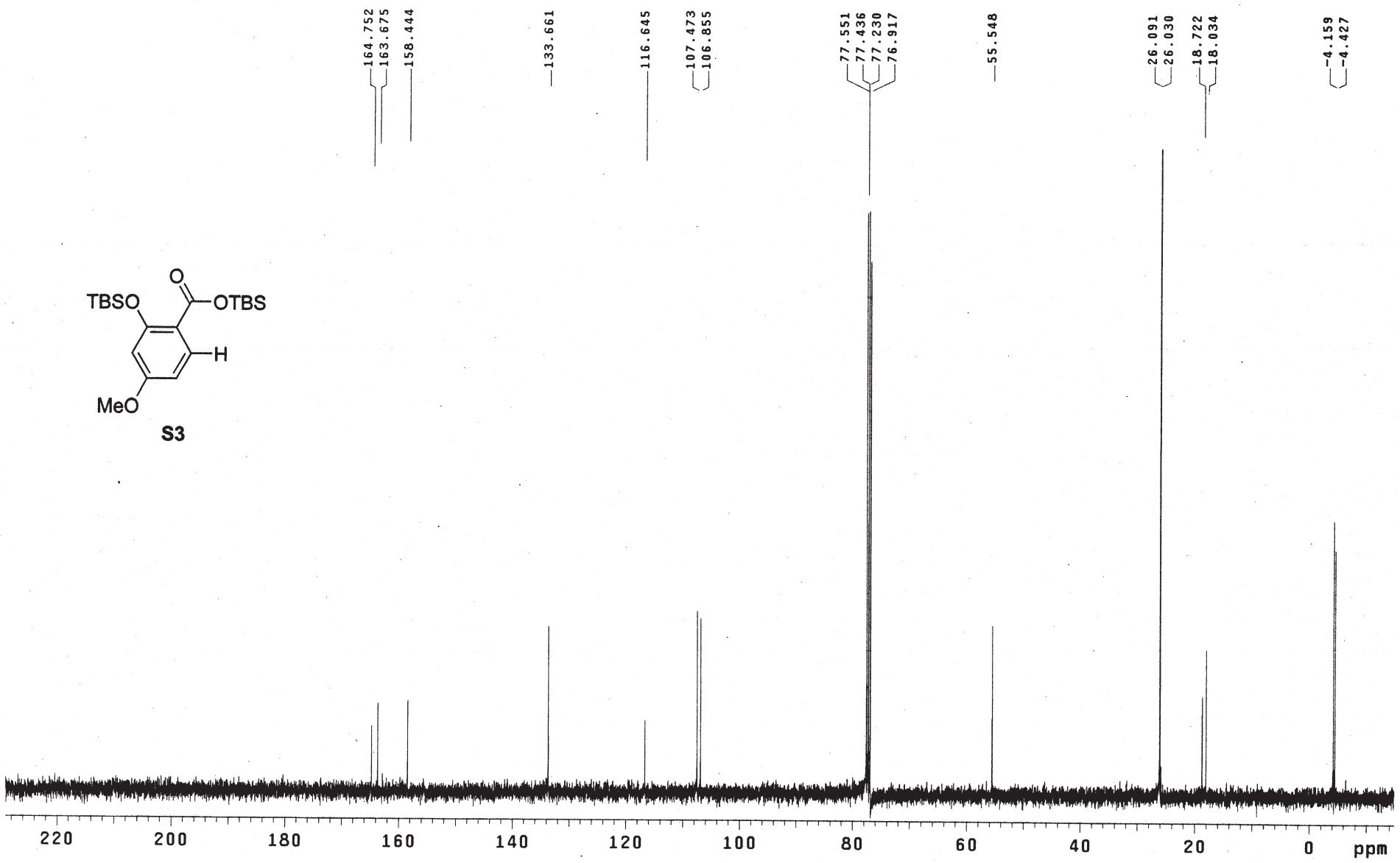
14

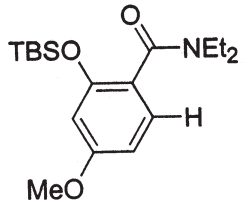

203.195
128.354
128.255
128.163
128.060
127.972
127.869
127.770
127.193
109.276
95.590
75.096
73.771
70.269
68.199
30.895
26.721
25.251
24.701
21.547
O
H
H
O
O
O
Me
O
O
220
200
180
160
140
120
100
80
60
40
20
ppm

14

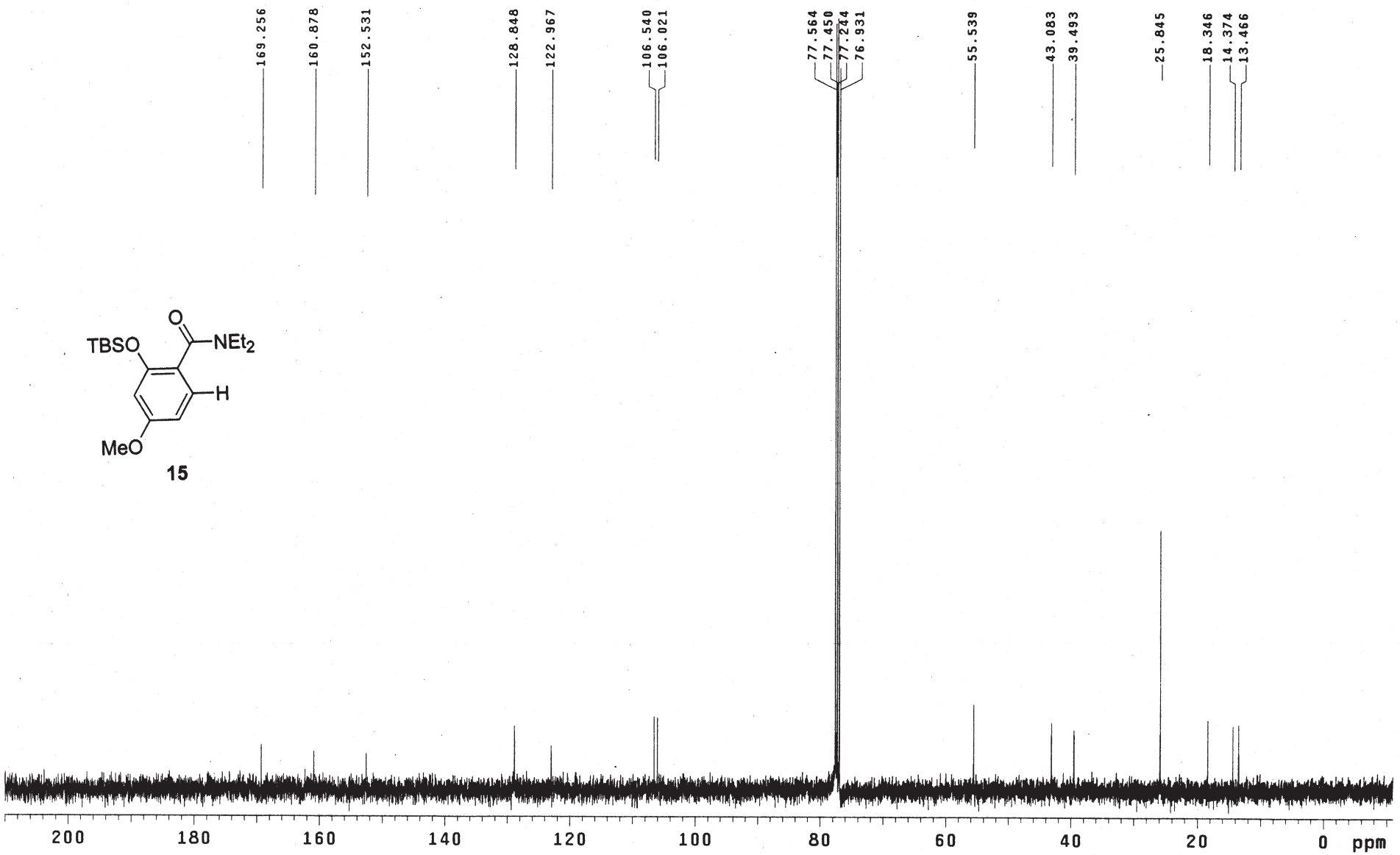


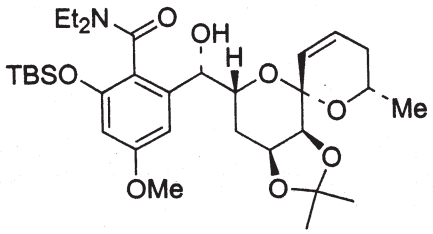
S3


164.752
163.675
158.444
133.661
116.645
107.473
106.855
77.551
77.436
77.230
76.917
55.548
26.091
26.030
18.722
18.034
-4.159
-4.427
TBSO
OTBS
O
H
MeO
S3
220
200
180
160
140
120
100
80
60
40
20
0
ppm

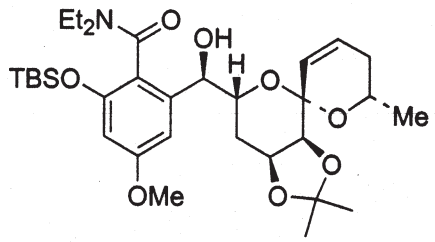


15

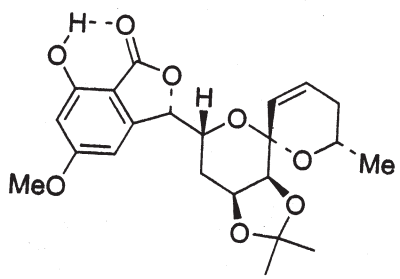

169.256
160.878
152.531
128.848
122.967
106.540
106.021
77.564
77.450
77.244
76.931
55.539
43.083
39.493
25.845
18.346
14.374
13.466
TBSO
O
NEt2
H
MeO
15
200
180
160
140
120
100
80
60
40
20
0
ppm



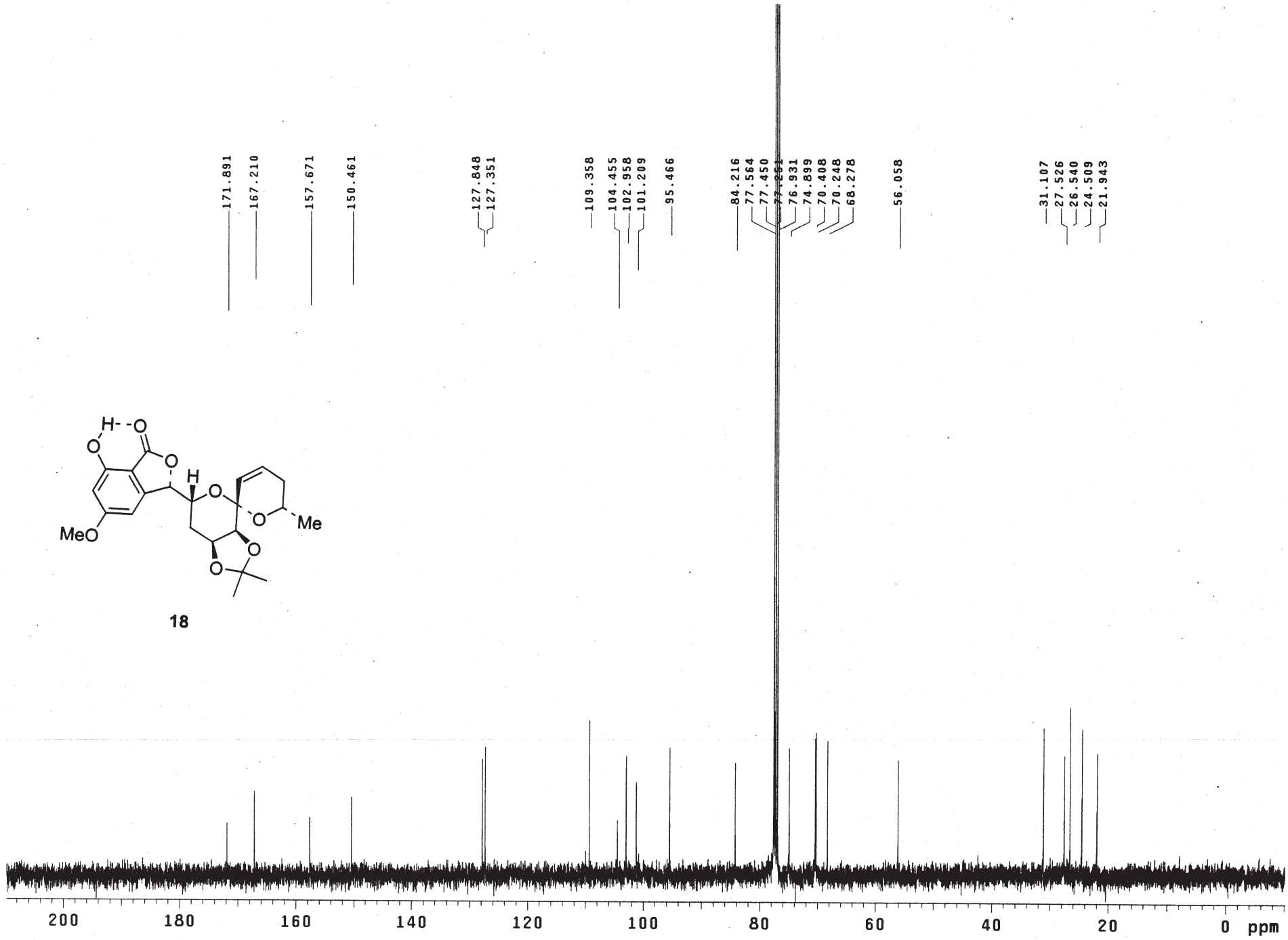
16



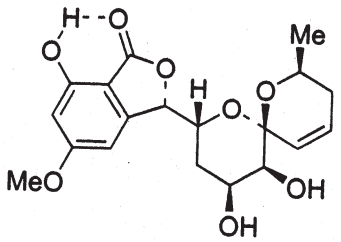
17



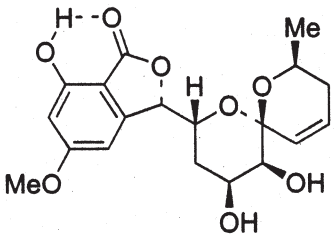
18


171.891
167.210
157.671
150.461
127.848
127.351
109.358
104.455
102.958
101.209
95.466
84.216
77.564
77.450
76.931
74.899
70.408
70.248
68.278
56.058
31.107
27.526
26.540
24.509
21.943
H
O
O
O
H
O
O
Me
MeO
O
O
200
180
160
140
120
100
80
60
40
20
0 ppm

18



(+)-**1**: aigialospirol



(+)-**1**: aigialospirol

171.289
167.279
157.702
149.064
130.521
126.519
104.272
101.615
101.233
99.117
82.346
77.313
77.199
76.679
70.699
68.278
65.827
65.017
55.982
33.583
31.437
21.104

200 180 160 140 120 100 80 60 40 20 0 ppm

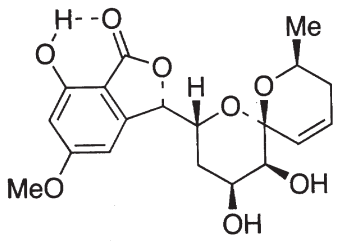
RF-II-21B_16Feb2007

Archive directory: /export/home/ruth/vnmrsys/data
Sample directory: RF-II-21B_16Feb2007

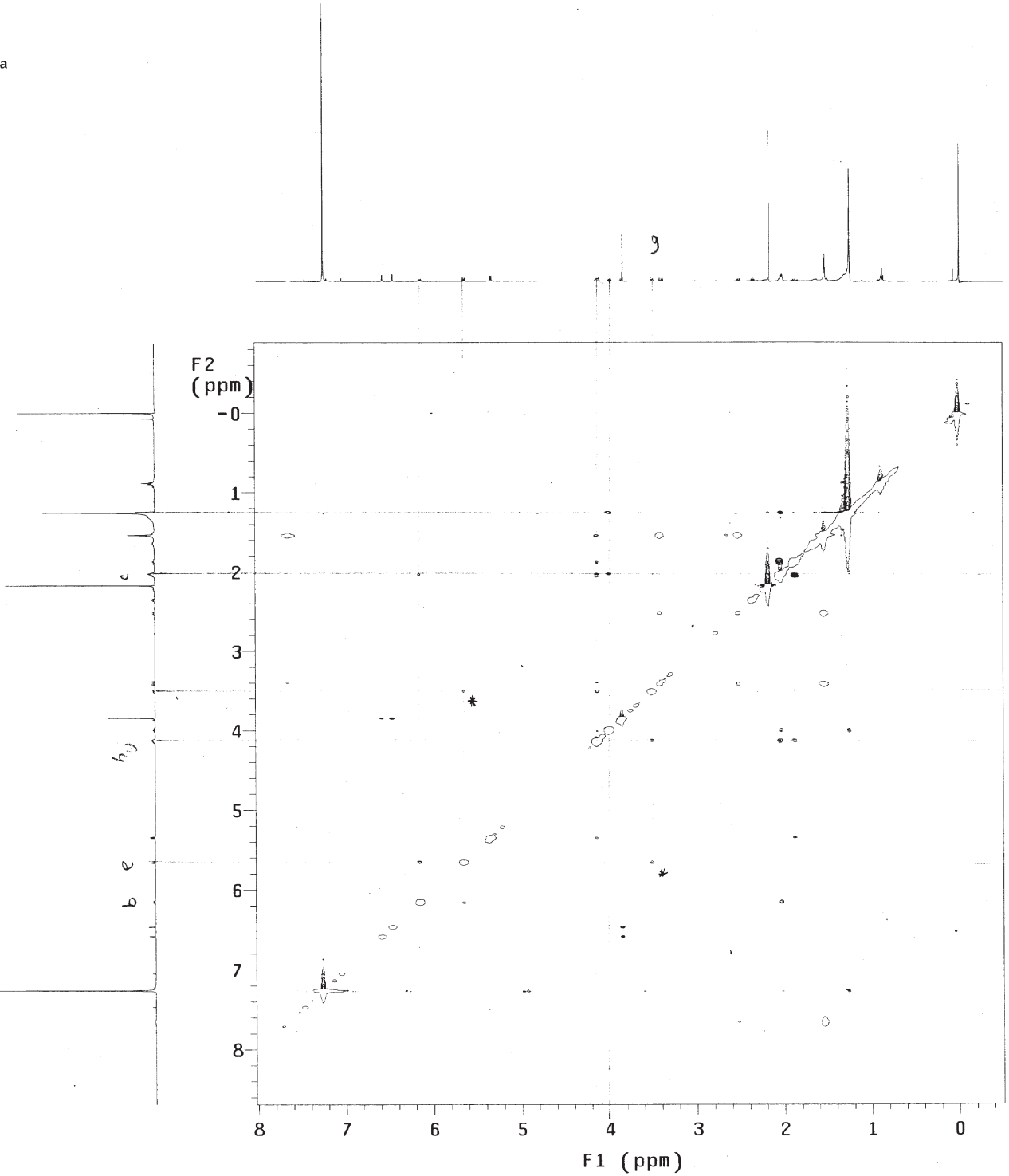
Pulse Sequence: NOESY

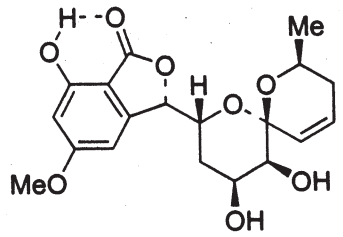
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: NOESY
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Mixing 1.000 sec
Acq. time 0.171 sec
Width 6000.6 Hz
2D Width 6000.6 Hz
16 repetitions
2 x 200 increments
OBSERVE H1, 499.7288181 MHz
DATA PROCESSING
Gauss apodization 0.079 sec
F1 DATA PROCESSING
Gauss apodization 0.031 sec
FT size 2048 x 2048
Total time 3 hr, 55 min, 56 sec



(+)-**1**: aigialospirol





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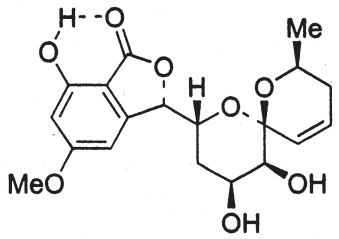

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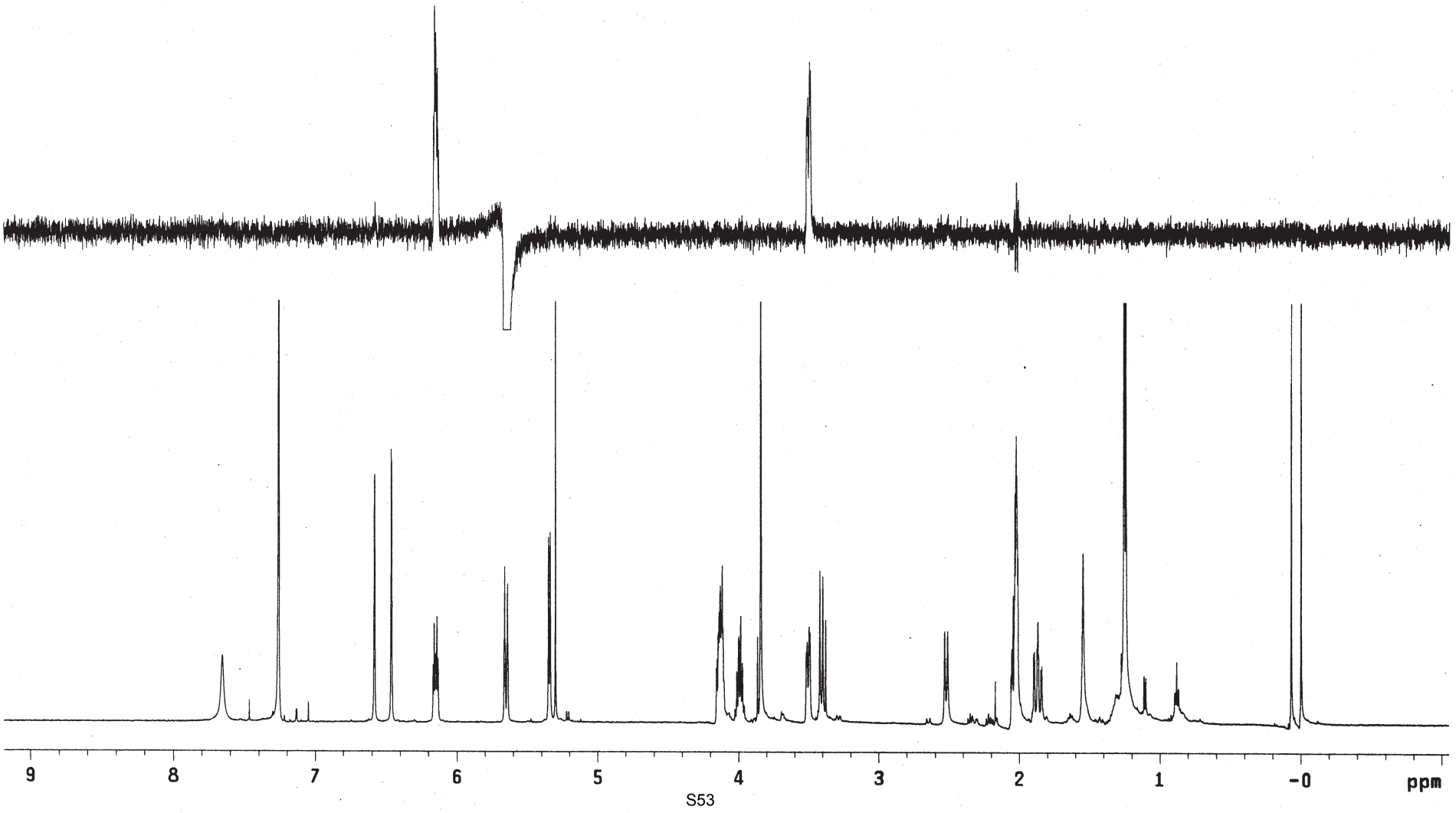

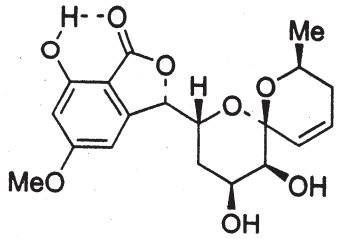

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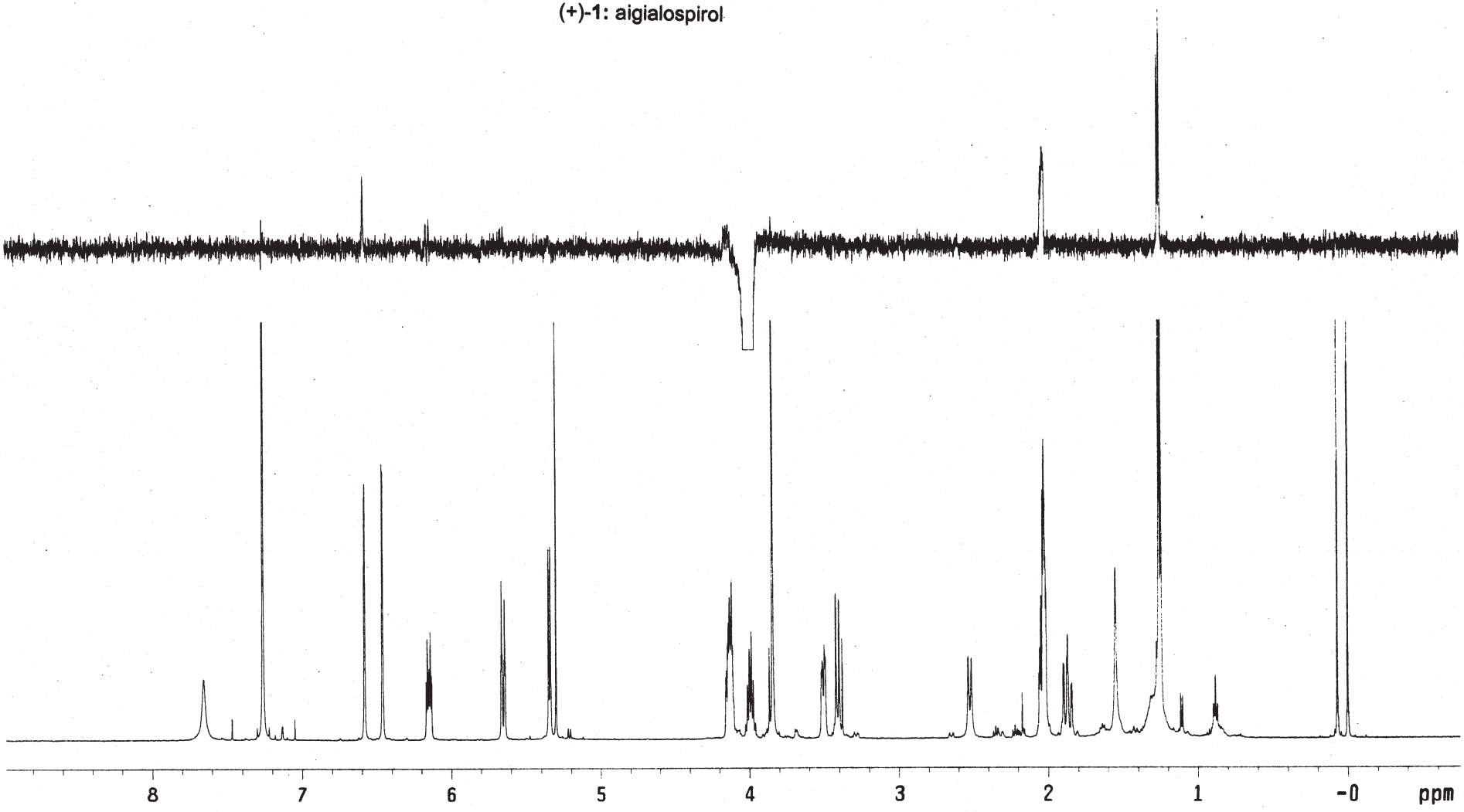

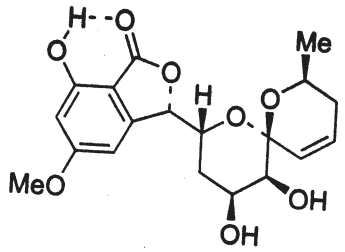

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