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Supporting Information

# Total Synthesis of 7,10-Epimer of the Proposed Structure of Amphidinolide N, Part II: Synthesis of C17–C29 Subunit and Completion of the Synthesis

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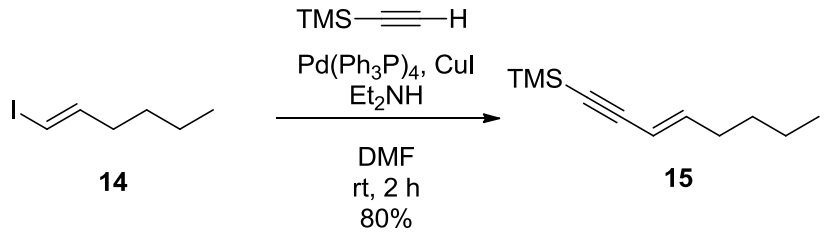
chem_201504675_sm_miscellaneous_information.pdf

# Experimental Section

**General Remark**: All reactions were monitored by thin-layer chromatography using Merck 60 $F_{254}$ precoated silica gel plates (0.25 mm thickness). Specific optical rotations were measured using JASCO P-1020 polarimeter. FT-IR spectra were recorded on a JASCO FT/IR-410 spectrometer or a Perkin–Elmer spectrum 100 spectrometer.

$^{1}H$ and $^{13}C$ NMR spectra were recorded on a Bruker AM-400 or a JEOL JNM-ECA-400 (400 MHz for $^{1}H$ NMR, 100 MHz for $^{13}C$ NMR) instrument. Data for $^{1}H$ NMR are reported as chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, dd = doubledoublet, dt = doubletriplet, m = multiplet, br = broad), coupling constant (Hz), integration, and assignment. Data for $^{13}C$ NMR are reported as chemical shift. High-resolution mass spectral analyses (HRMS) were carried out using a Bruker ESI-TOF MS or a JEOL JMS-SX102X mass spectrometer. Flash chromatography was performed using silica gel 60N of Kanto Chemical CO. Inc., Tokyo, Japan. HPLC analysis was performed on a HITACHI Elite LaChrom Series HPLC, UV detection monitored at appropriate wavelength respectively.

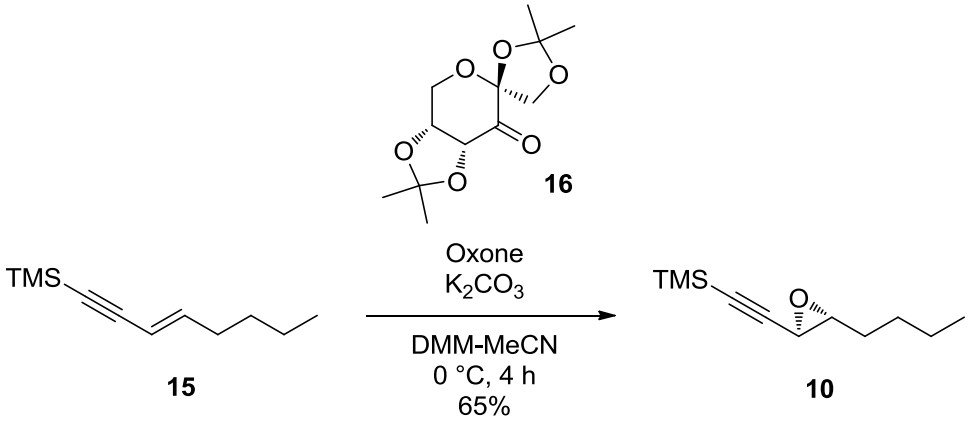
Compound **15**




To a stirred solution of **14**[S1] (6.85 g, 32.6 mmol) in DMF (65 mL) were added $Pd(Ph_3P)_4$ (367 mg, 0.33 mmol), CuI (124 mg, 0.65 mmol), trimethylsilylacetylene (6.76 mL, 48.9 mmol) and diethylamine (16.6 mL, 163 mmol) at room temperature under Ar atmosphere. After stirring for 2 h at the same temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and filtered through a pad of celite. After the filtrate was extracted with $Et_2O$, the organic layer was washed with 1mol/L HCl followed by brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by distillation *in vacuo* (b.p. 67-68 ºC / 5 mmHg) to give **15** (4.69 g, 26.0 mmol, 80%) as a yellow oil.

$^1$H NMR (400Hz, $CDCl_3$) δ 0.18 (9H, s), 0.89 (3H, t, *J* = 6.8 Hz), 1.29-1.41 (4H, m), 2.10 (2H, dt, *J* = 7.2, 7.2 Hz), 5.50 (1H, d, *J* = 16.0 Hz), 6.22 (1H, dt, *J* = 15.6, 7.2 Hz); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 0.37, 14.2, 22.5, 31.1, 33.1, 92.8, 104.6, 110.0, 146.7; IR (ATR) 2959, 2137, 1467, 1249, 1086, 957, 841 cm$^{-1}$; HRMS (EI$^+$) calcd for $C_{11}H_{20}Si$ ([M$^+$]): 180.1337 found: 180.1334.

Coupound **10**[S2]

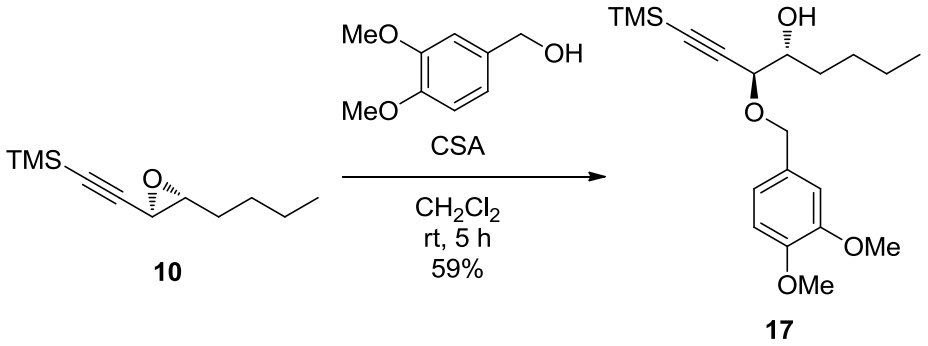



To a stirred solution of **15** (3.40 g, 18.9 mmol) and **16**[S3] (1.70 g, 6.60 mmol) in DMM-MeCN (v/v = 2/1, 300 mL) were added $K_2CO_3$-AcOH buffer (190 mL), which was prepared from AcOH (0.5 mL) and 0.1 mol/L $K_2CO_3$ aq. (100 mL), and *n*-$Bu_4NHSO_4$ (128 mg, 0.378 mmol) at 0 ºC. Oxone (17.4 g, 28.4 mmol) in $4\times10^{-4}$ mol/L $Na_2$(EDTA) aq. (150 mL) and $K_2CO_3$ (15.2 g, 110 mmol) in $H_2O$ (150 mL) were added dropwise separately at the same rate via addition funnel over 3 h. After addition, the reaction mixture was stirred for 1 h at the same temperature. $H_2O$ was added and the resulting mixture was extracted with $Et_2O$. The organic layer was washed with brine, dried

over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 30 : 1) to give **10** (2.39 g, 12.2 mmol, 65%) as a colorless oil. (The enantiomeric excess was determined in the next step.)

$^1$H NMR (400Hz, $CDCl_3$) δ 0.17 (9H, s), 0.91 (3H, t, *J* = 7.2 Hz), 1.33-1.59 (6H, m), 3.06-3.08 (2H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 0.10, 14.3, 22.8, 28.1, 31.9, 45.9, 61.2, 89.5, 102.5; IR (neat) 2960, 2933, 2873, 1467, 1251, 1097, 1050, 846 cm$^{-1}$; $[\alpha]_D^{28}$ +4.6 (*c* = 1.8, $CHCl_3$); HRMS (ESI$^+$) calcd for $C_{11}H_{20}NaOSi$ ($[M+Na]^+$): 219.1176 found: 219.1170.

Compound **17**[S4]

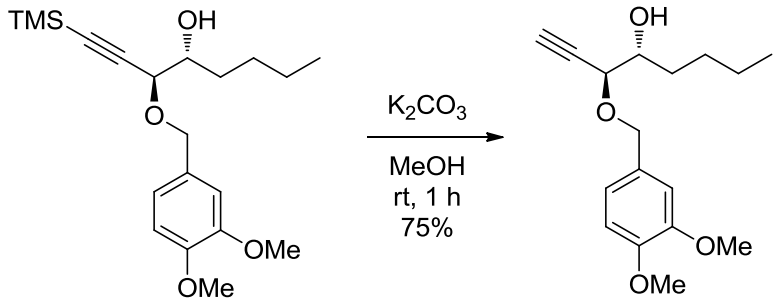



To a stirred solution of **10** (8.74 g, 44.5 mmol) in $CH_2Cl_2$ (45 mL) were added 3,4-dimethoxybenzyl alcohol (37.7 g, 223 mmol) and CSA (2.1 g, 8.9 mmol) at room temperature under Ar atmosphere. After stirring for 5 h at the same temperature, the reaction mixture was quenched with sat. $NaHCO_3$ aq. and extracted with $CHCl_3$. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 4 : 1) to give **17** (9.56 g, 26.2 mmol, 59%) as a colorless oil.

The enantiomeric excess was determined by chiral HPLC (Daicel Chiralcel OD-H column, *n*-hexane : isopropanol = 100 : 1, 1.0 mL/min, 280 nm, $t_{minor}$ = 15.6 min, $t_{major}$ = 28.1 min, 87% ee) .

$^1$H NMR (400Mz, $CDCl_3$) δ 0.21 (9H, s), 0.90 (3H, t, *J* = 3.2 Hz), 1.26-1.61 (6H, m), 2.13 (1H, brs), 3.70-3.71 (1H, m), 3.88 (3H, s), 3.89 (3H, s), 4.04 (1H, d, *J* = 4.0 Hz), 4.49 (1H, d, *J* = 11.2 Hz), 4.77 (1H, d, *J* = 11.2 Hz), 6.84 (1H, d, *J* = 8.4 Hz), 6.85-6.94 (2H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -0.13, 13.9, 22.6, 27.7, 32.2, 55.8, 55.9, 70.7, 72.80, 72.84, 93.3, 101.4, 111.0, 111.6, 120.8, 130.0, 148.8, 149.0; IR (neat) 3522, 2956, 2935, 2861, 1517, 1465, 1264, 1250, 1158, 1139, 1071, 1031, 845 cm$^{-1}$; $[\alpha]_D^{27}$ +111.5 (*c* = 3.2, $CHCl_3$); HRMS (ESI$^+$) calcd for $C_{20}H_{32}NaO_4Si$ ($[M+Na]^+$): 387.1962 found: 387.1960.

Compound **18**

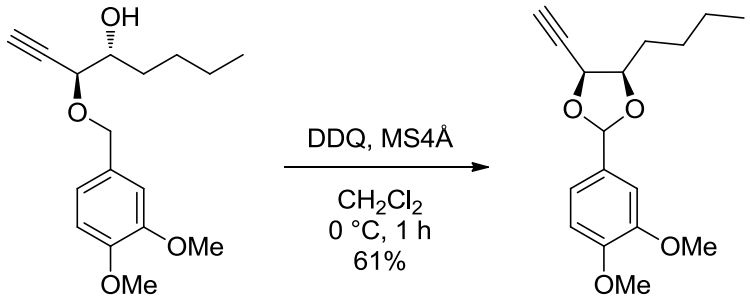



**17** **18**

To a stirred solution of **17** (2.38 g, 6.53 mmol) in MeOH (20 mL) was added $K_2CO_3$ (4.51 g, 32.7 mmol) at room temperature. After stirring for 1 h at the same temperature, the reaction mixture was filtered through a pad of celite. The filtrate was concentrated *in vacuo* and the residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 4 : 1) to give **18** (1.43 g, 4.89 mmol, 75%) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.90 (3H, t, *J* = 7.2 Hz), 1.26-1.61 (6H, m), 2.17 (1H, d, *J* = 4.8 Hz), 2.53 (1H, d, *J* = 2.0 Hz), 3.72-3.78 (1H, m), 3.88 (3H, s), 3.89 (3H, s), 4.05 (1H, dd, *J* = 3.6, 2.0 Hz), 4.49 (1H, d, *J* = 11.6 Hz), 4.79 (1H, d, *J* = 11.6 Hz), 6.85 (1H, d, *J* = 8.8 Hz), 6.90-6.92 (2H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 13.9, 22.6, 27.7, 32.0, 55.8, 55.9, 70.8, 72.2, 72.9, 77.3, 79.7, 111.0, 111.6, 120.9, 129.8, 148.9, 149.0; IR (neat) 3510, 3279, 2954, 2935, 2870, 1607, 1594, 1517, 1465, 1265, 1239, 1158, 1139, 1071, 1029 cm$^{-1}$; $[\alpha]_D^{28}$ +100.0 (*c* = 1.85, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{17}H_{24}NaO_4Si$ ($[M+Na]^+$): 315.1567 found: 315.1560.

Compound **6**



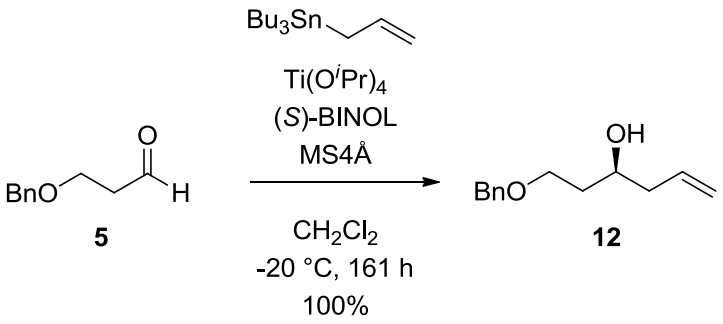

**18** **6**

To a stirred solution of **18** (2.5 g, 8.55 mmol) in $CH_2Cl_2$ (170 mL) were added molecular sieves 4Å (powder, 2.5 g) and DDQ (2.91 g, 12.8 mmol) at 0 ºC under Ar atmosphere. After stirring for 1 h at the same temperature, the reaction mixture was quenched with sat. $NaHCO_3$ aq. and extracted with $CHCl_3$. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 4 : 1) to give **6**

(1.52 mg, 5.23 mmol, 61%, 1 : 1 diastereomixture) as a yellow oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.94 (3H, t, *J* = 7.2 Hz), 1.40-1.52 (4H, m), 1.71-1.97 (2H, m), 2.59 (0.5H, d, *J* = 1.6 Hz), 2.59 (0.5H, d, *J* = 2.0 Hz), 3.88 (3H, s), 3.90 (3H, s), 4.13 (0.5H, dt, *J* = 6.0, 6.0 Hz), 4.21 (0.5H, dt, *J* = 6.0, 6.0 Hz), 4.84 (0.5H, dd, *J* = 5.6, 1.6 Hz), 4.99 (0.5H, dd, *J* = 5.6, 2.0 Hz), 5.78 (0.5H, s), 6.10 (0.5H, s), 6.84 (0.5H, d, *J* = 3.6 Hz), 6.86 (0.5H, d, *J* = 3.6 Hz), 6.99 (0.5H, d, *J* = 1.2 Hz), 7.03 (0.5H, d, *J* = 2.0 Hz), 7.07 (0.5H, d, *J* = 2.0 Hz), 7.16 (0.5H, d, *J* = 1.2 Hz); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 13.91, 13.94, 22.7, 28.1, 28.2, 30.0, 30.3, 55.8, 55.9, 56.0, 69.2, 70.1, 75.6, 76.5, 78.4, 78.6, 79.9, 80.1, 102.5, 104.7, 109.3, 110.1, 110.7, 110.9, 119.1, 120.2, 129.7, 130.6, 149.1, 149.2, 149.8, 150.1; IR (neat) 3270, 2955, 2936, 2872, 2838, 1609, 1597, 1519, 1465, 1265, 1165, 1138, 1086, 1028 $cm^{-1}$; $[\alpha]_D^{20}$ -0.59 (*c* = 3.1, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{17}H_{22}NaO_4$ ($[M+Na]^+$): 313.1410 found: 313.1414.

Compound **12**[S5]



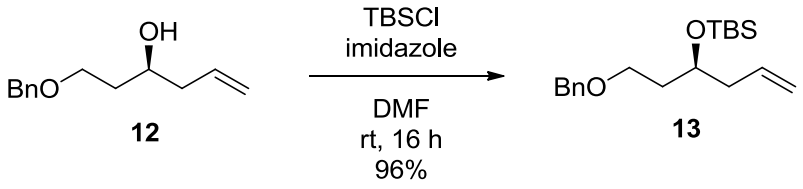

To a stirred suspension of (*S*)-BINOL (888 mg, 3.10 mmol) and molecular sieves 4Å (powder, 5 g) in $CH_2Cl_2$ (25 mL) was added 1 mol/L Ti($O^iPr$)$_4$ in $CH_2Cl_2$ (3.10 mL, 3.10 mmol) at room temperature under Ar atmosphere, and the reaction mixture was stirred for 1 h under reflux conditions. A solution of **5** (5.00 g, 30.5 mmol) in $CH_2Cl_2$ (5 mL) was added to the reaction mixture at room temperature. After stirring for 5 min at the same temperature, the reaction mixture was cooled to -78 ºC and allyltributyltin (11.3 mL, 36.5 mmol) was added. After stirring for 161 h at -20 ºC, the reaction mixture was quenched with sat. $NaHCO_3$ aq. and stirred for 1 h at room temperature. The resulting mixture was filtered through a pad of celite and the filtrate was extracted with $CHCl_3$. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 9 : 1 to 4 : 1) to give **12** (6.27 g, 30.4 mmol, 100%) as a colorless oil.

The enantiomeric excess was determined by chiral HPLC (Daicel Chiralcel OB-H column, *n*-hexane : isopropanol = 20 : 1, 1.0 mL/min, 207 nm, $t_{minor}$ = 24.9 min, $t_{major}$ = 30.3 min, 99% ee) .

$^1$H NMR (400Mz, $CDCl_3$) δ 1.75-1.79 (2H, m), 2.25 (2H, dd, *J* = 6.4, 6.4 Hz), 2.81 (1H, s), 3.62-3.75 (2H, m), 3.88-3.90 (1H, m), 4.53 (2H, s), 5.09 (1H, s), 5.12 (1H, d, *J* = 8.4 Hz), 5.79-5.89 (1H, m), 7.27-7.35 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 35.8, 41.9, 68.9, 70.3, 73.3, 117.5, 127.6,

127.7, 128.4, 134.8, 137.9; IR (neat) 3433, 3067, 3030, 2917, 2862, 1096, 1027, 996 $cm^{-1}$; $[\alpha]_D^{22}$ +4.7 (*c* = 2.26, $CHCl_3$); HRMS ($ESI^+$) calcd for : $C_{13}H_{18}NaO_2$ ($[M+Na]^+$): 229.1199 found: 229.1208.

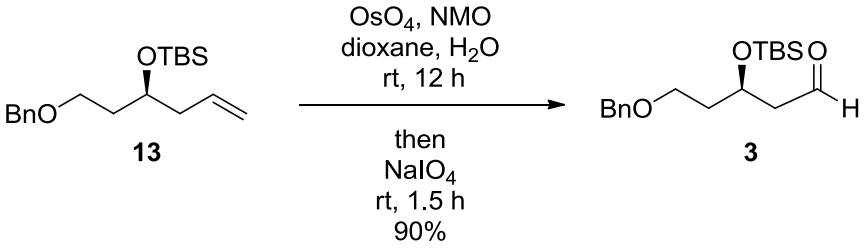
Compound **13**




To a stirred solution of **12** (9.48 g, 46.0 mmol) in DMF (46 mL) were added imidazole (4.07 g, 59.8 mmol) and TBSCl (7.62 g, 50.6 mmol) at 0 ºC. After stirring for 16 h at room temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 100 : 1 to 50 : 1) to give the **13** (14.1 g, 44.0 mmol, 96%) as a colorless oil.

$^1H$ NMR (400Mz, $CDCl_3$) δ -0.03 (3H, s), -0.01 (3H, s), 0.81 (9H, s), 1.61-1.77 (2H, m), 2.10-2.22 (2H, m), 3.46 (2H, t, *J* = 9.6 Hz), 3.80-3.86 (1H, m), 4.39 (1H, d, *J* = 11.6 Hz), 4.44 (1H, d, *J* = 11.6 Hz)4.96 (2H, dd, *J* = 12.8, 1.2 Hz), 5.69-5.80 (1H, m), 7.18-7.23 (5H, m); $^{13}C$ NMR (100 Hz, $CDCl_3$) δ –4.7, -4.4, 18.1, 25.9, 36.7, 42.3, 67.0, 68.9, 72.9, 117.0, 127.5, 127.6, 128.3, 134.9, 138.6; IR (neat) 2954, 2928, 2886, 2856, 1255, 1097, 836, 774 $cm^{-1}$; $[\alpha]_D^{23}$ +20.8 (*c* = 2.45, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{19}H_{32}NaO_2Si$ ($[M+Na]^+$): 343.2064 found: 343.2072.

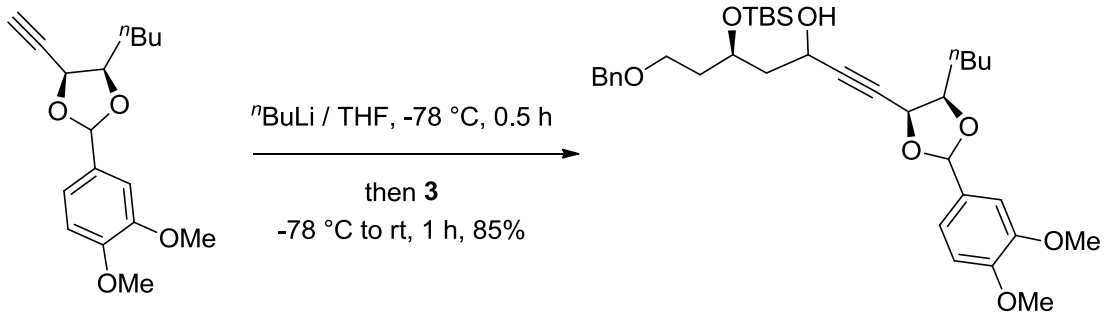
Compound **3**




To a stirred solution of **13** (892 mg, 2.78 mmol) in 1,4-dioxane (12 mL) and $H_2O$ (4 mL) were added 4.83 mol/L NMO in $H_2O$ (0.58 mL, 2.80 mmol) and 0.02 mol/L $OsO_4$ in *t*-BuOH (4.60 mL, 0.092 mmol) at 0 ºC. After stirring for 12 h at the same temperature, $NaIO_4$ (602 mg, 2.82 mmol) was added and the reaction mixture was stirred for 1.5 h at room temperature. The resulting mixture was quenched with sat. $Na_2S_2O_3$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column

chromatography (*n*-hexane : EtOAc = 10 : 1) to give **3** (804 mg, 2.49 mmol, 90%) as a colorless oil. $^1$H NMR (400Mz, $CDCl_3$) δ 0.06 (3H, s), 0.07 (3H, s), 0.87 (9H, s), 1.81-1.91 (2H, m), 2.50-2.62 (2H, m), 3.55 (2H, t, *J* = 6.0 Hz), 4.36-4.42 (1H, m), 4.46 (1H, d, *J* = 12.0 Hz), 4.50 (1H, d, *J* = 12.0 Hz)7.28-7.36 (5H, m), 9.80 (1H, t, *J* = 2.4 Hz); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.73, -4.70, 17.9, 25.7, 37.6, 51.0, 65.6, 66.2, 72.9, 127.5, 127.6, 128.3, 138.2; IR (neat) 2954, 2929, 2886, 2857, 1726, 1255, 1099, 837, 776 cm$^{-1}$; $[\alpha]_D^{22}$ +7.3 (*c* = 2.75, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{18}H_{30}NaO_3Si$ ($[M+Na]^+$): 345.1856 found: 345.1861.

Compound **19**



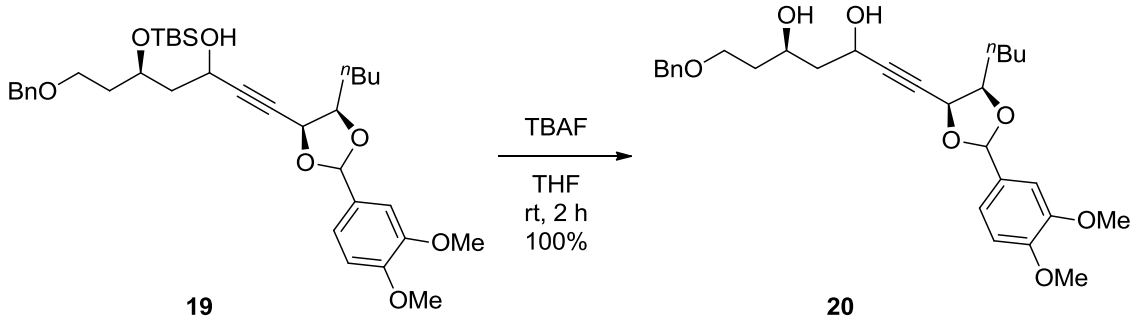

**6** **19**

To a stirred solution of **6** (4.00 g, 13.8 mol) in THF (59 mL) was slowly added 1.56 mol/L *n*-BuLi in *n*-hexane (9.30 mL, 14.5 mmol) at -78 ºC under Ar atmosphere. After stirring for 0.5 h at the same temperature, a solution of **3** (5.30 g, 16.5 mmol) in THF (10 mL) was added to the reaction mixture at -78 ºC and the reaction mixture was allowed to gradually warm to room temperature. After stirring for 1 h, the reaction mixture was quenched with sat. $NH_4Cl$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 3 : 1) to give **19** (5.87 g, 9.58 mmol, 85%, diastereomixture) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.07 (0.75H, s), 0.078 (0.75H, s), 0.082 (0.75H, s), 0.09 (0.75H, s), 0.10 (1.5H, s), 0.14 (0.75H, s), 0.87 (2.25H, s), 0.88 (2.25H, s), 0.886 (2.25H, s), 0.893 (2.25H, s), 0.93 (3H, t, *J* = 6.8 Hz), 1.35-2.04 (10H, m), 2.72 (0.25H, s), 2.77 (0.25H, s), 3.13 (0.25H, d, *J* = 1.2 Hz), 3.25 80.25H, d, *J* = 1.2 Hz), 3.50-3.56 (2H, m), 3.87 (0.75H, s), 3.88 (1.5H, s), 3.888 (0.75H, s), 3.894 (0.75H, s), 3.90 (0.75H, s), 4.05-4.29 (2H, m), 4.44-4.53 (2H, m), 4.60-4.73 (1H, m), 4.87 (0.25H, d, *J* = 1.2 Hz), 4.88 (0.25H, d, *J* = 1.2 Hz), 5.02 (0.25H, d, *J* = 1.2 Hz), 5.04 (0.25H, d, *J* = 1.2 Hz), 5.77 (0.5H, s), 6.065 (0.25H, s), 6.070 (0.25H, s), 6.83-6.86 (1H, m), 6.97-7.12 (2H, m), 7.27-7.36 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.71, -4.66, -4.61, -4.58, -4.43, -4.41, 14.0, 17.89, 17.92, 22.6, 22.7, 25.76, 25.78, 28.09, 28.13, 28.18. 28.21, 30.25, 30.34, 30.4, 30.47, 36.7, 37.3, 37.4, 43.1, 44.3, 44.4, 55.8, 55.9, 59.6, 59.8, 60.65, 60.72, 66.4, 68.0, 68.36, 68.42, 69.5, 70.3, 73.0, 78.5, 78.6, 79.78, 79.80, 102.3, 104.3, 109.1, 109.9, 110.6, 110.7, 119.1, 120.0, 120.1, 127.58,

127.61, 128.4, 129.6, 130.5, 130.6, 138.2, 149.0, 149.7, 150.0; IR (neat) 2954, 2930, 2857, 1520, 1464, 1456, 1263, 1088, 1029, 837 $cm^{-1}$; $[\alpha]_D^{31}$ –4.1 (*c* = 3.06, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{35}H_{52}NaO_7Si$ ($[M+Na]^+$): 635.3375 found: 635.3374.

Compound **20**

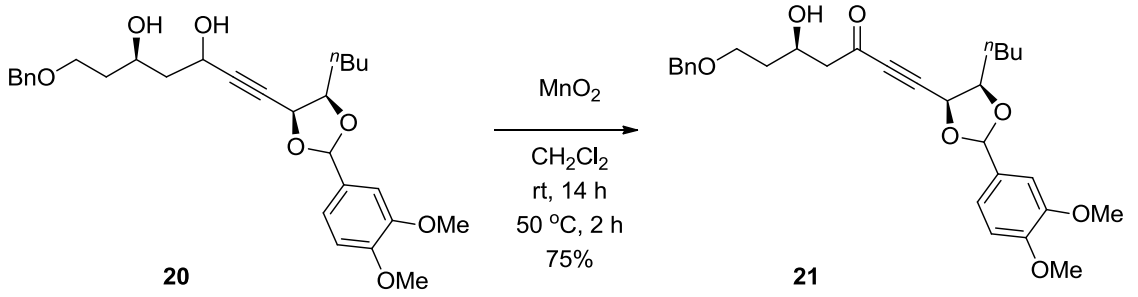



**19** **20**

To a stirred solution of **19** (600 mg, 0.979 mmol) in THF (5 mL) was added 1 mol/L TBAF in THF (1.20 mL, 1.20 mmol) at room temperature. After stirring for 2 h at the same temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 1 : 1) to give **20** (488 mg, 0.979 mmol, diastereomixture) as a colorless oil.

$^1H$ NMR (400Mz, $CDCl_3$) δ 0.93 (3H, t, *J* = 6.8 Hz), 1.38-1.96 (10H, m), 3.58-3.83 (4H, m), 3.865 (0.75H, s), 3.870 (0.75H, s), 3.88 (1.5H, s), 3.89 (1.5H, s), 3.90 (0.75H, s), 3.91 (0.75H, s), 4.10-4.24 (1.5H, m), 4.35-4.43 (0.5H, m), 4.50-4.56 (2H, m), 4.71-4.78 (1H, m), 4.87-4.90 (0.5H, m), 5.02-5.05 (0.5H, m), 5.77 (0.25H, s), 5.78 (0.25H, s), 6.07 (0.25H, s), 6.08 (0.25H, s), 6.83-6.86 (1H, m), 6.97-7.03 (1H, m), 7.05-7.09 (0.5H, m), 7.13 (0.5H, d, *J* = 1.6 Hz), 7.28-7.37 (5H, m); $^{13}C$ NMR (100 Hz, $CDCl_3$) δ 14.0, 22.6, 28.0, 28.08, 28.13, 28.17, 30.2, 30.4, 36.2, 36.5, 42.6, 44.0, 44.1, 55.9, 60.8, 62.0, 62.1, 68.8, 69.2, 69.4, 69.5, 69.8, 70.20, 70.23, 71.2, 73.4, 77.2, 78.5, 78.6, 79.1, 79.3, 79.8, 79.9, 80.4, 80.6, 88.4, 88.6, 89.2, 89.3, 102.3, 104.4, 109.1, 109.9, 110.0, 110.6, 110.7, 119.0, 120.1, 127.63, 127.65, 127.8, 128.5, 129.67, 129.73, 130.50, 130.53, 137.5, 137.6, 148.95, 149.02, 149.7, 150.0; IR (neat) 3446, 2953, 2935, 2870, 1519, 1264, 1088, 1027 $cm^{-1}$; $[\alpha]_D^{21}$ –8.1 (*c* = 2.43, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{29}H_{38}NaO_7$ ($[M+Na]^+$): 521.2510 found: 521.2525.

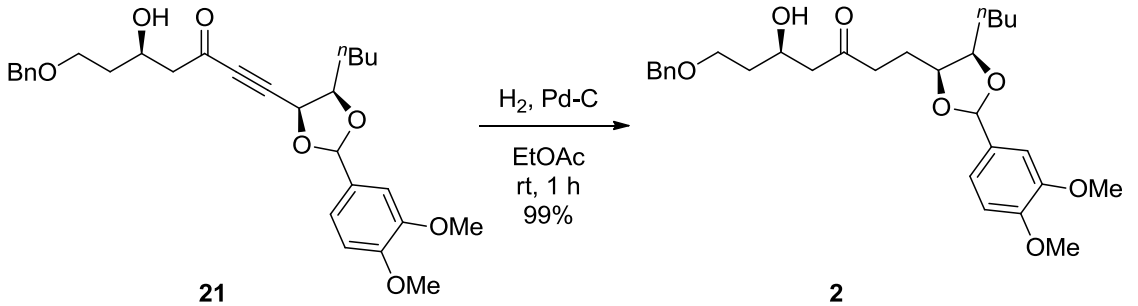
Compound **21**




To a stirred solution of **20** (488 mg, 0.979 mmol) in $CH_2Cl_2$ (5 mL) was added $MnO_2$ (75%, 1.13 g, 9.75 mmol) at room temperature. After stirring for 14 h at room temperature, additional $MnO_2$ (75%, 567 mg, 4.89 mmol) was added and the reaction mixture was stirred for 2 h at 50 ºC. The reaction mixture was filtered through a pad of celite and the filtrate was concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 2 : 1) to give **21** (366 mg, 0.737 mmol, 75%, diastereomixture) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.93 (3H, t, *J* = 6.4 Hz), 1.37-1.42 (4H, m), 1.67-1.95 (4H, m), 2.70-2.89 (2H, m), 3.14 (0.5H, d, *J* = 3.6 Hz), 3.17 (0.5H, d, *J* = 3.6 Hz), 3.61-3.74 (2H, m), 3.875 (1.5H, s), 3.884 (1.5H, s), 3.896 (1.5H, s), 3.902 (1.5H, s), 4.17-4.22 (0.5H, m), 4.26-4.31 (0.5H, m), 4.33-4.44 (1H, m), 4.51 (1H, s), 4.52 (1H, s), 4.96 (0.5H, d, J = 6.0 Hz), 5.11 (0.5H, d, *J* = 6.0 Hz), 5.81 (0.5H, s), 6.09 (0.5H, s), 6.85-6.87 (0.5H, m), 6.97-7.09 (2.5H, m), 7.28-7.38 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 13.81, 13.84, 15.2, 22.47, 22.49, 28.0, 29.1, 29.9, 30.3, 35.9, 52.4, 55.8, 55.9, 65.7, 66.5, 67.9, 69.0, 69.8, 73.21, 73.22, 78.4, 80.1, 85.8, 86.4, 86.7, 87.9, 102.9, 104.9, 109.1, 109.8, 110.6, 110.8, 119.0, 120.0, 127.6, 127.7, 128.4, 129.1, 129.9, 137.8, 149.0, 149.1, 149.9, 150.2, 185.56, 185.58; IR (neat) 3510, 2954, 2935, 2870, 1677, 1519, 1265, 1164, 1089, 1027 cm$^{-1}$; $[\alpha]_D^{20}$ +15.6 (*c* = 1.35, $CHCl_3$); HRMS (ESI$^+$) calcd for $C_{29}H_{36}NaO_7$ ([M+Na]$^+$): 519.2353 found: 519.2377.

Synthesis of **2**

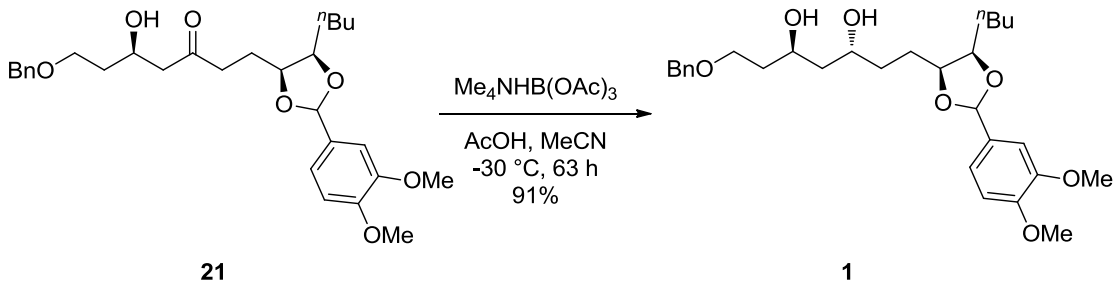



To a stirred solution of **21** (2.61 g, 5.26 mmol) in EtOAc (26 mL) was added 10 wt% Pd-C (261 mg) and the reaction mixture was stirried for 1 h at room temperature under $H_2$ atomosphere (1 atm). The

reaction mixture was filtered through a pad pf celite and the filtrate was concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 2 : 1) to give **2** (2.61 g, 5.21 mmol, 99%, diastereomixture) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.92 (3H, t, *J* = 6.8 Hz), 1.36-1.86 (10H, m), 2.52-2.77 (4H, m), 3.30 (0.5H, brs), 3.34 (0.5H, brs), 3.65-3.69 (2H, m), 3.87 (3H, s), 3.89 (3H, s), 4.05-4.29 (4H, m), 4.498 (1H, s), 4.501 (1H, s), 5.70 (0.5H, s), 5.94 (0.5H, s), 6.84 (0.5H, d, *J* = 8.4 Hz), 6.85 (0.5H, d, *J* = 8.4 Hz), 6.95-7.02 (2H, m), 7.27-7.35 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 13.4, 22.3, 22.7, 24.0, 28.5, 28.6, 29.3, 36.08, 36.11, 40.0, 49.56, 49.60, 55.77, 55,82, 55.9, 66.66, 66.70, 67.9, 73.2, 77.2, 77.6, 77.9, 78.6, 79.4, 101.4, 103.1, 109.0, 109.5, 110.7, 118.7, 119.6, 127.65, 127.69, 128.41, 130.2, 132.1, 138.0, 149.0, 149.5, 149.8, 210.7; IR (neat) 2933, 2860, 1708, 1518, 1264, 1089, 1027 $cm^{-1}$; $[\alpha]_D^{21}$ –22.3 (*c* = 1.35, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{29}H_{40}NaO_7$ ($[M+Na]^+$): 523.2666 found: 523.2654.

Compound **1**[S6]



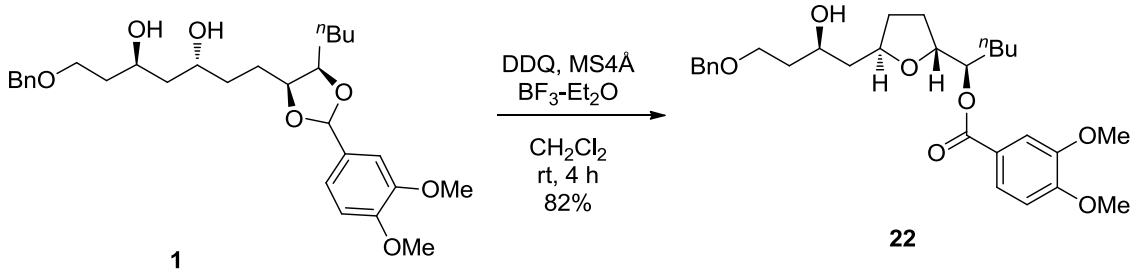

**21** **1**

To a stirred solution of $Me_4NHB(OAc)_3$ (7.10 g, 27.1 mmol) in MeCN (36 mL) was added AcOH (36 mL) at room temperature under Ar atmosphere. After stirring for 0.5 h, a solution of **21** (2.71 g, 5.41 mmol) in MeCN (36 mL) was slowly added to the reaction mixture at -30 ºC. After stirring for 63 h at the same temperature, the reaction mixture was quenched with 0.5 mol/L rochelle's salt aq. and stirred for 0.5 h at room temperature. The reaction mixture was diluted with $CHCl_3$ and washed with sat. $Na_2CO_3$ aq.. The aqueous layer was back extracted several times with $CHCl_3$. The combined extract was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 1 : 2) to give **1** (2.45 g, 4.91 mmol, 91%, diastereomixture) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.90-0.94 (3H, m), 1.37-1.98 (14H, m), 3.06 (0.5H, brs), 3.10 (0.5H, brs), 3.50 (1H, brs), 3.67-3.76 (2H, m), 3.87 (3H, s), 3.88 (1.5H, s), 3.89 (1.5H, s), 3.97 (1H, brs), 4.11-4.20 (3H, m), 4.52 (1H, s), 4.53 (1H, s), 5.73 (0.5H, s), 5.99 (0.5H, s), 6.847 (0.5H, d, *J* = 8.0 Hz), 6.851 (0.5H, d, *J* = 8.0 Hz), 6.98-7.04 (2H, m), 7.29-7.37 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ 14.0, 22.7, 25.3, 26.6, 28.3, 28.5, 29.7, 34.4, 34.5, 36.2, 42.7, 55.7, 55.8, 55.9, 68.9, 69.0, 69.5, 69.6, 73.4, 78.8, 79.1, 79.3, 79.4, 101.3, 103.0, 109.0, 109.6, 110.7, 118.7, 119.6, 127.6, 127.8, 128.5,

130.4, 132.3, 137.6, 148.9, 149.4, 149.7; IR (neat) 3445, 2861, 1518, 1455, 1264, 1089, 1028 $cm^{-1}$; $[\alpha]_D^{23}$ –5.1 (*c* = 2.35, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{29}H_{42}NaO_7$ ($[M+Na]^+$): 525.2823 found: 525.2822.

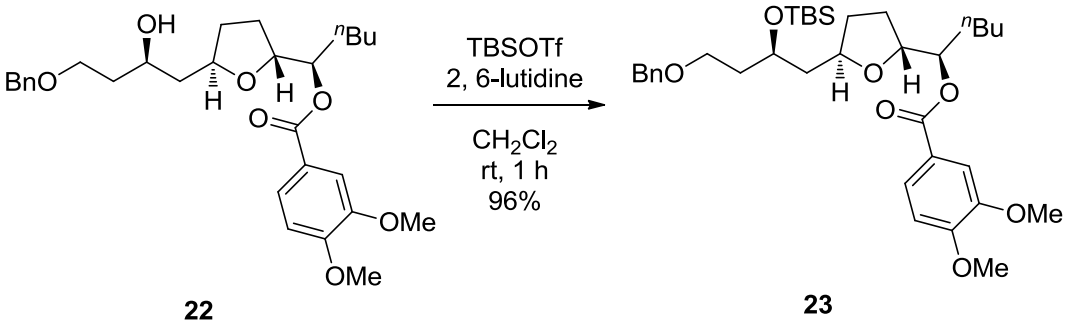
Compound **22**




To a stirred solution of **1** (540 mg, 1.08 mmol) and molecular sieves 4Å (powder, 540 mg) in $CH_2Cl_2$ (11 mL) were added DDQ (294 mg, 1.30 mmol) and a solution of $BF_3$-$Et_2O$ (0.0266 mL, 0.21 mmol) in $CH_2Cl_2$ (0.67 mL) at 0 ºC under Ar atmosphere. After stirring for 4 h at room temperature, the reaction mixture was quenched with sat. $NaHCO_3$ aq. and extracted with $CHCl_3$. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 1 : 1) to give **22** (447 mg, 0.890 mmol, 82%) as a colorless oil.

$^1H$ NMR (400Mz, $CDCl_3$) δ 0.86-0.90 (3H, m), 1.24-1.81 (12H, m), 1.98-2.09 (2H, m), 3.48 (1H, d, *J* = 4.0 Hz), 3.60 (2H, t, *J* = 6.4 Hz), 3.906 (3H, s), 3.913 (3H, s), 3.93-3.94 (1H, m), 4.12 (1H, dt, *J* = 6.8, 6.8 Hz), 4.17-4.23 (1H, m), 4.47 (2H, s), 5.10 (1H, ddd, *J* = 10.0, 6.0, 4.4 Hz), 6.86 (1H, d, *J* = 8.4 Hz), 7.28-7.33 (5H, m), 7.56 (1H, d, *J* = 2.0 Hz), 7.70 (1H, dd, *J* = 8.4, 2.0 Hz); $^{13}C$ NMR (100 Hz, $CDCl_3$) δ 13.9, 22.6, 27.6, 28.0, 30.5, 32.5, 36.8, 41.6, 55.96, 55.97, 67.7, 68.4, 73.1, 75.9, 76.2, 79.6, 110.2, 112.0, 122.9, 123.6, 127.5, 127.6, 128.3, 138.3, 148.6, 153.0, 166.7; IR (neat) 2935, 2862, 1707, 1698, 1513, 1270 $cm^{-1}$; $[\alpha]_D^{32}$ +8.3 (*c* = 1.0, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{29}H_{40}NaO_7$ ($[M+Na]^+$): 523.2666 found: 523.2649.

Compound **23**

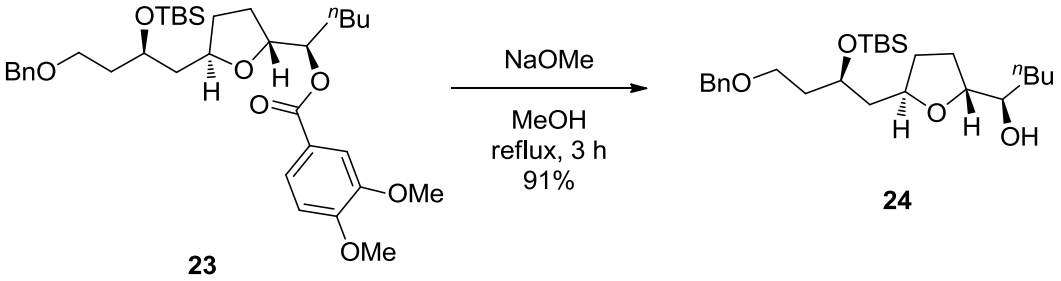



To a stirred solution of **22** (3.33 g, 6.65 mmol) in $CH_2Cl_2$ (33 mL) were added 2, 6-lutidine (1.81

mL, 9.98 mmol) and TBSOTf (1.83 mL, 7.98 mmol) at 0 ºC under Ar atmosphere. After stirring for 3 h at room temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 9 : 1 to 4 : 1) to give **23** (3.91 g, 6.36 mmol, 96%) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.02 (3H, s), 0.06 (3H, s), 0.87 (9H, s), 0.89-0.92 (3H, m), 1.26-1.85 (12H, m), 1.95-2.05 (2H, m), 3.53 (2H, t, *J* = 6.8 Hz), 3.91 (6H, s), 3.94-4.00 (1H, m), 4.07-4.16 (2H, m), 4.46 (2H, dd, *J* = 12.0, 12.0 Hz), 5.09 (1H, dt, *J* = 8.4, 8.4 Hz), 6.85 (1H, d, *J* = 8.4 Hz), 7.28-7.35 (5H, m), 7.57 (1H, d, *J* = 2.0 Hz), 7.70 (1H, dd, *J* = 8.4, 2.0 Hz); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.7, -4.5, 14.0, 18.0, 22.6, 25.9, 27.7, 28.1, 30.8, 32.6, 37.8, 44.0, 56.0, 66.8, 67.5, 72.9, 76.2, 79.0, 110.2, 112.2, 123.1, 123.6, 127.4, 127.5, 128.3, 138.6, 148.6, 152.9, 166.2; IR (neat) 2954, 2932, 2857, 1771, 1601, 1515, 1270, 1223, 1103 cm$^{-1}$; $[\alpha]_D^{24}$ +2.1 (*c* = 1.81, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{35}H_{54}NaO_7Si$ ($[M+Na]^+$): 637.3531 found: 637.3547.

Compound **24**

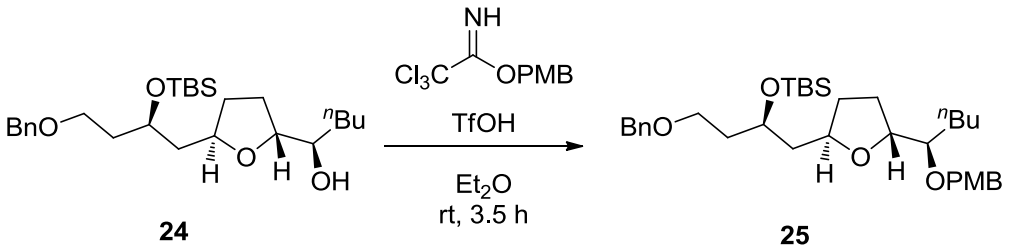



**23** **24**

To a stirred solution of **23** (500 mg, 0.813 mmol) in MeOH (4 mL) was added sodium methoxide (132 mg, 2.44 mmol), and the resulting mixture was stirred for 3 h under reflux conditions. The reaction mixture was quenched with sat. $NH_4Cl$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 9 : 1) to give **24** (333 mg, 0.739 mmol, 91%) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.07 (6H, d, *J* = 7.6 Hz), 0.88 (9H, s), 0.91 (3H, t, *J* = 7.2 Hz), 1.26-1.42 (5H, m), 1.46-1.69 (6H, m), 1.75-1.85 (2H, m), 1.91-2.07 (2H, m), 3.33-3.38 (1H, m), 3.54 (2H, t, *J* = 6.8 Hz), 3.77 (1H, dt, *J* = 7.2, 7.2 Hz), 3.98-4.07 (2H, m), 4.49 (2H, s), 7.26-7.35 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.6, -4.5, 14.0, 18.1, 22.8, 25.9, 27.8, 28.4, 33.0, 33.1, 38.0, 43.7, 66.8, 67.5, 72.9, 74.2, 75.6, 81.9, 127.5, 127.6, 128.3, 138.6; IR (neat) 3481, 2955, 2930, 2857, 1254, 1095, 1006, 856, 775, 734 cm$^{-1}$; $[\alpha]_D^{21}$ –12.7 (*c* = 2.37, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{26}H_{46}NaO_4Si$ ($[M+Na]^+$): 473.3058 found: 473.3055.

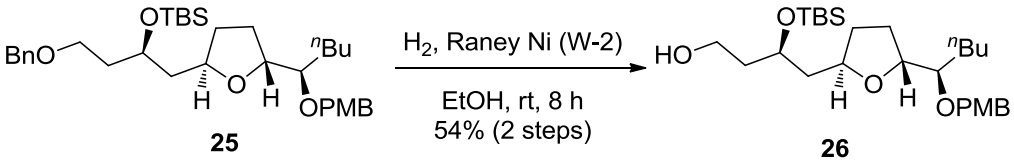
Compound **25**




**24** **25**

To a stirred solution of **25** (280 mg, 0.621 mmol) in $Et_2O$ (3 mL) were added a solution of 4-methoxybenzyl-2,2,2-trichloroacetimidate (351 mg, 1.24 mmol) in $Et_2O$ (3 mL) and trifluoromethanesulfonic acid (5 mmol/L in $Et_2O$, 1.2 mL, 0.006 mmol) at room temperature under Ar atmosphere. After stirring for 3.5 h at the same temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 9 : 1) to give **25** (259 mg, impure) as a colorless oil, which was used in the next step without further purification.

Compound **26**

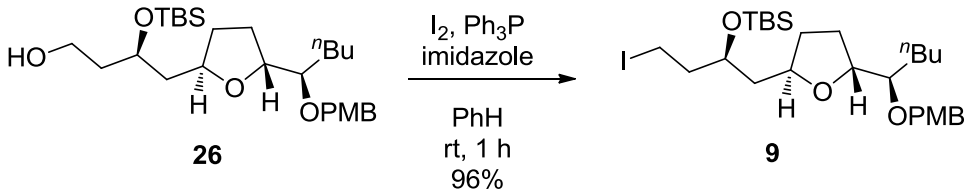



**25** **26**

To a stirred solution of **25** (259 mg, impure) in EtOH (1.2 mL) was added Raney Ni (W-2) in EtOH (excess) at room temperature. After stirring for 8 h at the same temperature under $H_2$ armosphere (1 atm), the reaction mixture was filtered through a pad of celite and the filtrate was concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 4 : 1) to give **26** (161 mg, 0.336 mmol, 54% from **24**) as a colorless oil.

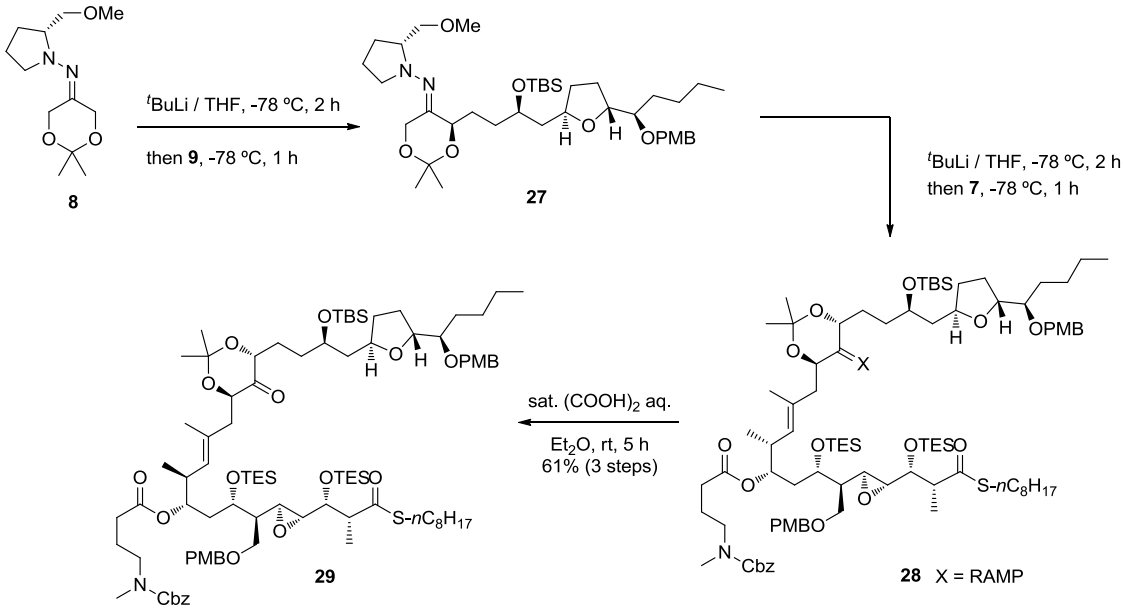
$^1$H NMR (400Mz, $CDCl_3$) δ 0.10 (6H, s), 0.89 (12H, m), 1.25-1.32 (4H, m), 1.42-1.53 (2H, m), 1.59-1.73 (4H, m), 1.88-1.96 (2H, m), 2.00-2.07 (2H, m), 2.12 (1H, brs), 3.26-3.30 (1H, m), 3.72 (1H, dt, *J* = 5.6, 10.8 Hz), 3.80 (3H, s), 3.85-3.89 (1H, m), 3.98-4.07 (2H, m), 4.11 (1H, dt, *J* = 11.2, 4.8 Hz), 4.51 (1H, d, *J* = 11.2 Hz), 4.66 (1H, d, *J* = 11.2 Hz), 6.86 (2H, d, *J* = 8.4 Hz), 7.27 (2H, d, *J* = 8.4 Hz); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.6, -4.5, 14.1, 17.9, 22.8, 25.8, 27.8, 28.6, 30.7, 32.8, 38.7, 43.1, 55.3, 59.8, 69.9, 72.5, 76.0, 81.2, 81.7, 113.6, 129.3, 131.4, 159.0; IR (neat) 3428, 2932, 2857, 1613, 1514, 1464, 1249, 1069, 836, 775 $cm^{-1}$; $[\alpha]_D^{21}$ +6.4 (*c* = 1.13, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{27}H_{48}NaO_5Si$ ($[M+Na]^+$): 503.3163 found: 503.3172.

Compound **9** (C17-C29 fragment)




To a stirred solution of **26** (129 mg, 0.268 mmol) in benzene (1.4 mL) were added imidazole (73 mg, 1.07 mmol), triphenylphosphine (141 mg, 1.07 mmol), and iodine (136 mg, 0.537 mmol) at room temperature. After stirring for 2 h at the same temperature, the reaction mixture was quenched with sat. $Na_2S_2O_3$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 9 : 1) to give **9** (152 mg, 0.257 mmol, 96%) as a colorless oil. $^1$H NMR (400Mz, $CDCl_3$) δ 0.10 (6H, s), 0.89 (3H, t, *J* = 7.3 Hz), 0.90 (9H, s), 1.26-1.32 (3H, m), 1.41-1.51 (4H, m), 1.56-1.70 (2H, m), 1.88-2.14 (4H, m), 3.22 (2H, t, *J* = 6.7 Hz), 3.26-3.30 (1H, m), 3.80 (3H, s), 3.86-3.92 (1H, m), 3.97-4.04 (2H, m), 4.53 (1H, d, *J* = 11.0 Hz), 4.70 (1H, d, *J* = 11.0 Hz), 6.87 (2H, d, *J* = 8.6 Hz), 7.29 (2H, d, *J* = 8.6 Hz); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.41, -4.36, 2.4, 14.1, 18.0, 22.8, 25.9, 27.8, 28.6, 30.7, 32.7, 42.1, 43.3, 55.2, 70.4, 72.5, 75.6, 81.2, 81.6, 113.6, 129.3, 131.4, 158.9; IR (neat) 2954, 2930, 2857, 1613, 1513, 1463, 1247, 1065, 835, 775 cm$^{-1}$; $[\alpha]_D^{24}$ +20.5 (*c* = 0.96, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{27}H_{51}INO_4Si$ ($[M+NH_4]^+$): 608.26320 found: 608.26390.

Compound **29**[S7]



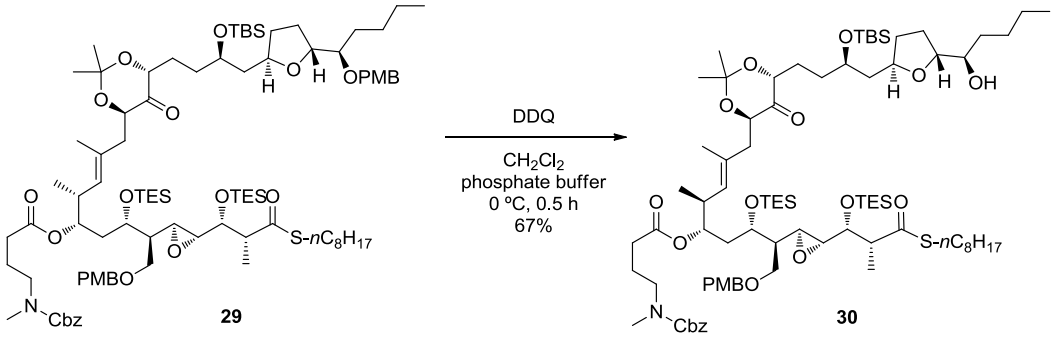
To a stirred solution of **8**[S7] (275 mg, 1.14 mmol) in THF (5 mL) was slowly added *t*-BuLi (1.59 mol/L in *n*-pentane, 0.86 mL, 1.37 mmol) at –78 ºC under Ar atmosphere. After stirring for 2 h at the same temperature, a solution of iodide (639 mg, 1.08 mmol) in THF (5 mL) was slowly added to the reaction mixture at –78 ºC. After stirring for 1 h at the same temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo* The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 5 : 1, 1% triethylamine) to give **27** (696 mg, impure) as a colorless oil, which was used directly in the next step without further purification.

To a stirred solution of **27** (696 mg, impure) in THF (5 mL) was slowly added *t*-BuLi (1.59 mol/L in *n*-pentane, 0.74 mL, 1.18 mmol) at –78 ºC under Ar atmosphere. After stirring for 2 h at the same temperature, a solution of **7** (1.07 g, 0.938 mmol) in THF (5 mL) was slowly added to the reaction mixture at –78 ºC. After stirring for 1 h at the same temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo* to give **28** (1.76 g, crude) as a yellow oil, which was used directly in the next step without purification.

To a stirred solution of **28** (1.76 g, crude) in $Et_2O$ (5 mL) was added sat. oxalic acid aq. (5 mL) at room temperature. After stirring for 5 h at the same temperature, the reaction mixture was diluted with $H_2O$ and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 5 : 1) to give **29** (1.31 g, 0.793 mmol, 73% from **9**) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.06 (3H,s), 0.08 (3H, s), 0.53-0.62 (12H, m), 0.86-0.94 (24H, m), 0.88 (9H, s), 1.16 (3H, d, *J* = 6.7 Hz), 1.18-1.70 (36H, m), 1.70-2.01 (8H, m), 2.23-2.30 (2H, m), 2.58 (1H, dd, *J* = 15.3, 9.8 Hz), 2.66 (1H, brs), 2.72-2.88 (5H, m), 2.91 (3H, s), 3.26-3.31 (3H, m), 3.41 (1H, dd, *J* = 9.2, 7.3 Hz), 3.52 (1H, dd, *J* = 9.2, 5.5 Hz), 3.62 (1H, dd, *J* = 6.7, 4.3 Hz), 3.79 (3H, s), 3.80 (3H, s), 3.87-3.95 (2H, m), 3.96-4.02 (1H, m), 4.05-4.13 (2H, m), 4.24 (1H, d, *J* = 9.2 Hz), 4.34 (1H, d, *J* = 11.6 Hz), 4.37 (1H, d, *J* = 11.6 Hz), 4.51 (1H, d, *J* = 11.6 Hz), 4.70 (1H, d, *J* = 11.6 Hz), 4.84-4.87 (1H, m), 5.02 (1H, d, *J* = 9.2 Hz), 5.12 (2H, s), 6.84-6.87 (4H, m), 7.19 (2H, d, *J* = 8.6 Hz), 7.27-7.35 (7H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.5, -4.4, 4.8, 5.0, 6.7, 6.9, 12.0, 14.0, 16.7, 17.0, 18.0, 22.6, 22.8, 23.3, 23.8, 23.9, 25.9, 27.9, 28.6, 28.8, 28.9, 29.0, 29.1, 29.4, 30.6, 31.4, 31.7, 32.6, 33.4, 34.0, 34.7, 36.6, 36.7, 38.1, 43.4, 48.0, 48.4, 52.7, 54.8, 55.1, 59.7, 66.9, 67.5, 68.5, 69.4, 72.5, 72.9, 73.3, 74.3, 75.2, 75.6, 81.0, 81.7, 100.9, 113.5, 113.6, 127.7, 127.8, 128.4, 129.3, 130.2, 131.5, 132.0, 136.9, 156.2, 158.9, 172.8, 201.3, 210.9; IR (ATR) 2931, 2875, 1732, 1705, 1613, 1514, 1460, 1374, 1302, 1247, 1173, 1069, 1007, 960, 833, 728 cm$^{-1}$; $[\alpha]_D^{25}$ +15.5 (*c* = 0.51, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{91}H_{155}N_2O_{17}SSi_3$ ($[M+NH_4]^+$): 1664.03542 found: 1664.03559.

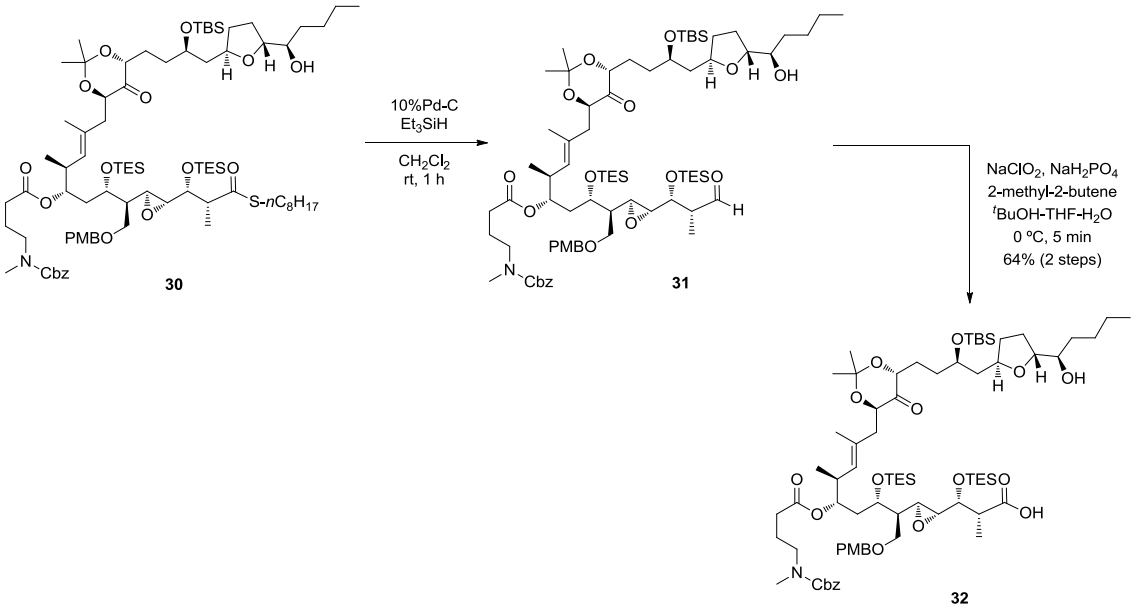
Compound **30**




To a stirred solution of **29** (285 mg, 0.173 mmol) in $CH_2Cl_2$ (2 mL) and pH 7.0 phosphate buffer (0.5 mL) was added DDQ (39.3 mg, 0.173 mmol) at 0 ºC. After stirring for 0.5 h at the same temperature, the reaction mixture was quenched with sat. $NaHCO_3$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography[S8] (*n*-hexane : EtOAc = 6 : 1) to give TM (183 mg, 0.120 mmol) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.06 (3H,s), 0.07 (3H, s), 0.53-0.62 (12H, m), 0.86-0.99 (24H, m), 0.89 (9H, s), 1.16 (3H, d, *J* = 7.3 Hz), 1.26-1.70 (36H, m), 1.70-2.05 (8H, m), 2.10 (1H, dd, *J* = 15.3, 9.8 Hz), 2.27-2.30 (2H, m), 2.43 (1H, d, *J* = 3.7 Hz), 2.58 (1H, d, *J* = 15.3 Hz), 2.65 (1H, brs), 2.72-2.87 (5H, m), 2.91 (3H, s), 3.27-3.35 (3H, m), 3.41 (1H, dd, *J* = 9.2, 9.2 Hz), 3.52 (1H, dd, *J* = 9.2, 6.1 Hz), 3.62 (1H, dd, *J* = 6.7, 4.3 Hz), 3.79 (3H, s), 3.85-3.88 (2H, m), 3.90-4.10 (1H, m), 4.11-4.13 (1H, m), 4.25 (1H, d, *J* = 8.6 Hz), 4.34 (1H, d, *J* = 11.0 Hz), 4.37 (1H, d, *J* = 11.0 Hz), 4.83-4.86 (1H, m), 5.02 (1H, d, *J* = 9.8 Hz), 5.12 (2H, s), 6.85 (2H, d, *J* = 8.6 Hz), 7.19 (2H, d, *J* = 8.6 Hz), 7.26-7.34 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.7, -4.4, 4.7, 5.0, 6.7, 6.9, 11.9, 13.9, 14.0, 16.7, 17.0, 18.0, 22.5, 22.7, 23.6, 23.7, 23.8, 25.8, 27.7, 28.2, 28.99, 29.04, 29.4, 31.4, 31.7, 32.8, 33.0, 33.2, 33.4, 33.9, 34.6, 36.5, 36.6, 38.0, 42.7, 48.0, 48.4, 52.7, 54.7, 55.1, 59.7, 66.9, 67.4, 68.4, 69.2, 72.8, 73.3, 74.1, 74.3, 75.2, 75.5, 81.9, 100.8, 113.6, 127.7, 127.8, 128.3, 129.2, 130.1, 132.0, 136.8, 156.1, 159.1, 172.8, 201.2, 210.9; IR (ATR) 2930, 2875, 1731, 1705, 1613, 1514, 1459, 1374, 1302, 1248, 1173, 1069, 1006, 960, 834, 730 cm$^{-1}$; $[\alpha]_D^{23}$ +12.2 (*c* = 0.63, $CHCl_3$); HRMS (ESI$^+$) calcd for $C_{83}H_{147}N_2O_{16}SSi_3$ ($[M+NH_4]^+$): 1543.97791 found: 1543.97818.

Compound **32**[S9,S10]



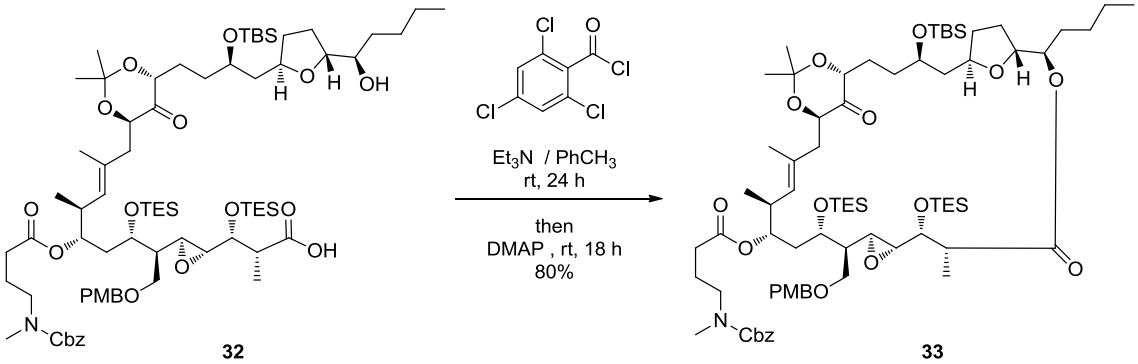

To a stirred solution of **30** (180 mg, 0.118 mmol) in $CH_2Cl_2$ (0.3 mL) were added 10% Pd-C (18 mg) and triethylsilane (0.094 mL, 0.589 mmol) at room temperature. After stirring for 0.5 h, the reaction mixture was diluted with $CH_2Cl_2$ and filtered through a pad of celite. The filtrate was concentrated *in vacuo.* The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 5 : 1) to give **31** (131 mg, impure) as a colorless oil, which was used directly in the next step without further purification.

To a stirred solution of **31** (131 mg, impure) and 2-methyl-2-butene (0.96 mL, 0.903 mmol) in *t*-BuOH (1.5 mL) and THF (1.5 mL) was slowly added a solution of $NaClO_2$ (80%, 30.6 mg, 0.271 mmol) and $NaH_2PO_4 2H_2O$ (70.4 mg, 0.451 mmol) in $H_2O$ (0.5 mL) at 0 ºC. After stirring for 10 min at the same temperature, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 3 : 1) to give **32** (105 mg, 0.075 mmol, 64% from **30**) as a colorless oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.06 (3H,s), 0.07 (3H, s), 0.52-0.67 (12H, m), 0.85-0.97 (24H, m), 0.89 (9H, s), 1.16 (3H, d, *J* = 6.7 Hz), 1.21-2.17 (28H, m), 1.39 (6H, s), 2.29-2.35 (2H, m), 2.57-2.69 (3H, m), 2.88-2.92 (2H, m), 2.92 (3H, s), 3.30-3.37 (3H, m), 3.42-3.48 (1H, m), 3.54-3.57 (1H, m), 3.74-3.79 (1H, m), 3.79 (3H, s), 3.84-3.88 (2H, m), 3.98-4.03 (1H, m), 4.11-4.14 (1H, m), 4.25 (1H, d, *J* = 10.3 Hz), 4.36 (1H, d, *J* = 10.9 Hz), 4.41 (1H, d, *J* = 10.9 Hz), 4.80-4.82 (1H, m), 5.02 (1H, d, *J* = 9.2 Hz), 5.12 (2H, s), 6.86 (2H, d, *J* = 8.6 Hz), 7.22 (2H, d, *J* = 8.6 Hz), 7.26-7.34 (5H, m); $^{13}$C NMR (100 Hz, $C_6D_6$) δ 4.4, -4.2, 5.3, 5.5, 7.1, 7.3, 11.5, 11.6, 13.6, 14.3, 17.17, 17.22, 18.3, 19.7, 20.3, 22.9, 23.2, 24.0, 24.1, 24.3, 26.1, 26.237, 26.240, 28.4, 30.9, 31.5, 33.3, 33.5, 33.7, 33.8, 34.6, 37.3, 38.6, 43.3, 44.5, 48.1, 48.6, 48.9, 49.0, 54.8, 55.6, 60.2, 60.5, 63.7, 67.3, 67.6, 69.1, 69.2, 70.0,

73.3, 74.1, 74.4, 74.7, 75.6, 75.7, 75.9, 82.4, 101.1, 114.1, 127.8, 128.5, 128.6, 129.6, 129.7, 130.5, 132.8, 137.5, 156.4, 159.8, 172.8, 172.9, 178.1, 210.1; IR (ATR) 2954, 2876, 1732, 1707, 1613, 1459, 1374, 1248, 1174, 1069, 1006, 835, 743 $cm^{-1}$; $[\alpha]_D^{23}$ +9.0 (*c* = 0.5, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{75}H_{131}N_2O_{17}Si_3$ ($[M+NH_4]^+$): 1415.87555 found: 1415.87464.

Compound **33**[S11]



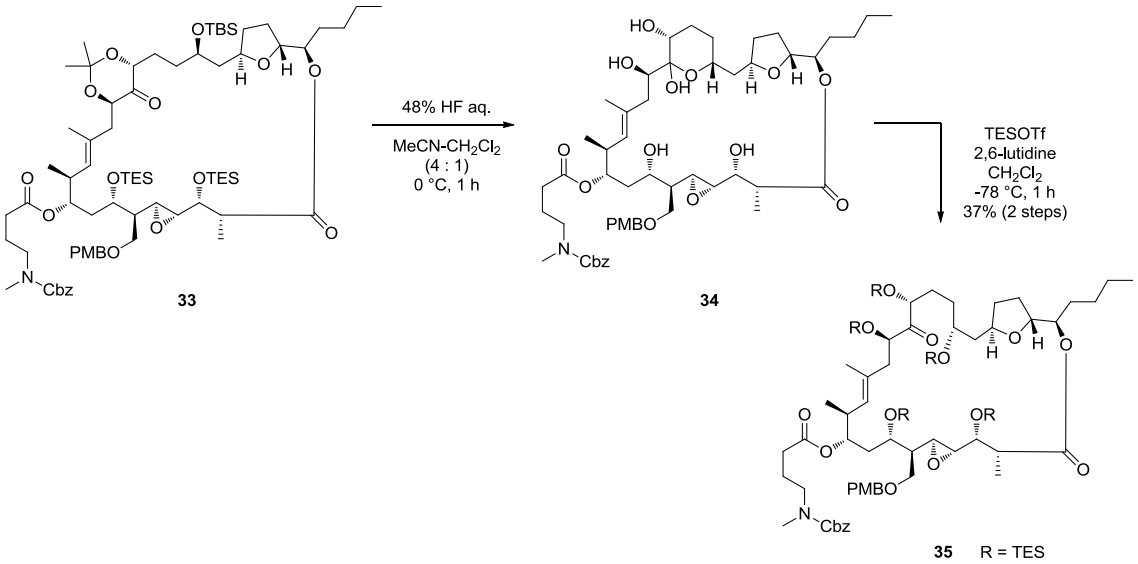

**32** **33**

To a stirred solution of **32** (400 mg, 0.286 mmol) in toluene (100 mL) were added triethylamine (1.6 mL, 11.5 mmol) and 2,4,6-trichlorobenzoyl chloride (1.3 mL, 8.32 mmol) at 0 ºC under Ar atmosphere. After stirring for 24 h at room temperature, toluene (100 mL) was added. The resulting mixture was slowly added to a solution of DMAP (1.02 g, 8.32 mmol) in toluene (400 mL) over 9 h using syringe pump at room temperature, and the reaction mixture was stirred for 9 h. The resulting suspension was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane : EtOAc = 6 : 1) to give **33** (315 mg, 0.228 mmol, 80%) as a light yellow oil.

$^1$H NMR (400Mz, $CDCl_3$) δ 0.038 (3H,s), 0.042 (3H, s), 0.51-0.67 (12H, m), 0.88-0.95 (21H, m), 0.88 (9H, s), 0.928 (3H, s), 0.934 (3H, s), 1.12 (3H, d, *J* = 7.3 Hz), 1.30-1.71 (12H, m), 1.41 (6H, s), 1.81-1.93 (4H, m), 2.02 (1H, dd, *J* = 15.3, 9.2 Hz), 2.26 (2H, brs), 2.57-2.64 (2H, m), 2.73 (1H, d, *J* = 8.6 Hz), 2.78 (1H, d, *J* = 5.5 Hz), 2.92 (4H, brs), 3.26-3.29 (2H, m), 3.43 (1H, dd, *J* = 8.6, 8.6 Hz), 3.60 (1H, dd, *J* = 8.6, 4.3 Hz), 3.70-3.73 (1H, m), 3.79 (3H, s), 3.81-3.98 (4H, m), 4.05-4.18 (1H, m), 4.27-4.30 (1H, m), 4.34 (1H, d, *J* = 11.0 Hz), 4.38 (1H, d, *J* = 11.0 Hz), 4.84-4.95 (2H, m), 5.08 (1H, d, *J* = 9.2 Hz), 5.12 (2H, s), 6.85 (2H, d, *J* = 8.6 Hz), 7.20 (2H, d, *J* = 8.6 Hz), 7.31-7.35 (5H, m); $^{13}$C NMR (100 Hz, $CDCl_3$) δ -4.5, -4.4, 5.0, 6.85, 6.88, 10.3, 13.8, 13.9, 14.1, 17.5, 17.7, 18.0, 18.35, 18.4, 22.6, 22.7, 23.2, 23.9, 24.1, 24.2, 25.8, 25.9, 27.5, 28.4, 30.8, 31.5, 32.6, 32.8, 32.9, 33.0, 34.0, 34.7, 35.0, 37.1, 42.9, 43.7, 45.6, 48.0, 48.4, 49.2, 49.8, 55.1, 55.2, 61.0, 67.0, 67.1, 67.9, 69.6, 73.2, 74.0, 74.3, 74.4, 74.6, 76.0, 79.0, 100.8, 113.7, 125.5, 127.8, 127.9, 128.4, 129.2, 129.5, 130.1, 133.4, 133.5, 136.9, 156.2, 159.2, 172.7, 172.9, 173.2, 210.6; IR (ATR) 2953, 2875, 1730, 1706, 1613, 1514, 1459, 1375, 1247, 1174, 1068, 1006, 835, 742 $cm^{-1}$; $[\alpha]_D^{24}$ +27.7 (*c* = 2.15, $CHCl_3$); HRMS

(ESI$^+$) calcd for $C_{75}H_{129}N_2O_{16}Si_3$ ([M+$NH_4$]$^+$): 1397.86499 found: 1397.86432.

Compound **35**

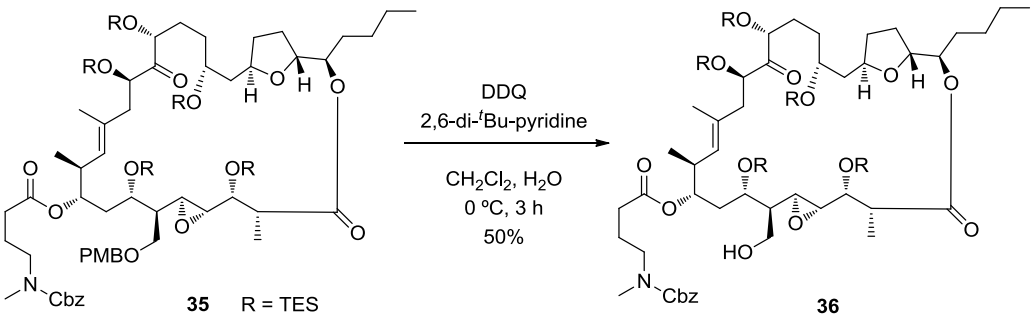



To a stirred solution of **33** (310 mg, 0.224 mmol) in MeCN (36 mL) and $CH_2Cl_2$ (9 mL) was added 48% HF aq. (4.4 mL) at 0 ºC. After stirring for 1 h at the same temperature, the reaction mixture was poured into sat. $NaHCO_3$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo* to give **34** (240 mg, crude) as a white solid, which was directly used in the next step.

To a stirred solution of **34** (240 mg, crude) in $CH_2Cl_2$ (5 mL) were added 2,6-lutidine (0.5 mL, 2.75 mmol) and TESOTf (0.5 mL, 2.21 mmol) at –78 ºC under Ar atmosphere. After stirring for 2 h, the reaction mixture was quenched with pH 7.0 phosphate buffer and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by preparative thin layer chromatography (toluene : EtOAc = 19 : 1) to give **35** (131 mg, 0.0834 mmol, 37% from **33**) as a colorless oil.

$^1$H NMR (400Mz, $C_6D_6$) δ 0.64-0.96 (36H, m), 1.03-1.15 (49H, m), 1.17-1.51 (10H, m), 1.50 (3H, d, *J* = 7.3 Hz), 1.62-1.93 (15H, m), 2.16 (1H, brs), 2.30-2.36 (2H, m), 2.64 (2H, brs), 2.76-2.90 (4H, m), 3.14-3.16 (2H, m), 3.25-3.36 (1H, m), 3.44 (3H, s), 3.55 (1H, dd, *J* = 8.6, 8.6 Hz), 3.72 (1H, dd, *J* = 8.6, 4.3 Hz), 3.94-4.05 (3H, m), 4.18-4.20 (1H, m), 4.26-4.35 (3H, m), 4.64 (1H, dd, *J* = 5.5, 5.5 Hz), 4.76 (1H, dd, *J* = 10.4, 2.5 Hz), 5.11 (1H, dt, *J* = 6.7, 6.7 Hz), 5.18 (2H, s), 5.29 (1H, brs), 5.31 (1H, d, *J* = 9.8 Hz), 6.93 (2H, d, *J* = 8.6 Hz), 7.08-7.38 (5H, m), 7.29 (2H, d, *J* = 8.6 Hz); IR (ATR) 2954, 2876, 1728, 1708, 1613, 1514, 1458, 1414, 1245, 1180, 1080, 1005, 822, 727 cm$^{-1}$; $[\alpha]_D^{22}$ -23.0 (*c* = 0.77, $CHCl_3$); HRMS (ESI$^+$) calcd for $C_{84}H_{153}N_2O_{16}Si_5$ ([M+$NH_4$]$^+$): 1586.00664 found: 1586.00663.

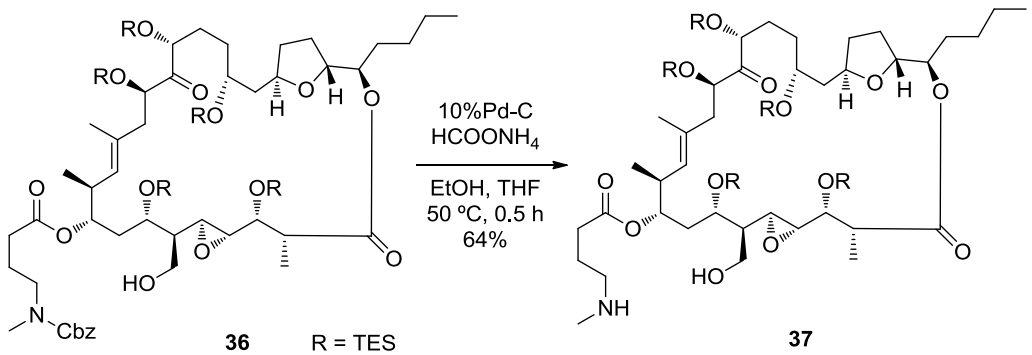
Compound **36**




To a stirred solution of **35** (46 mg, 0.0293 mmol) in $CH_2Cl_2$ (2 mL) and $H_2O$ (0.5 mL) were added DDQ (33.3 mg, 0.147 mmol) and 2,6-di-*t*-Bu-pyridine (0.097 mL, 0.441 mmol) at 0 ºC. After stirring for 4 h at the same temperature, the reaction mixture was quenched with sat. $NaHCO_3$ aq. and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (toluene : $Et_2O$ = 9 : 1) to give **36** (21.4 mg, 0.0147 mmol, 50%) as a colorless oil.

$^1$H NMR (400Mz, $C_6D_6$) δ 0.62-0.92 (36H, m), 1.02-1.20 (49H, m), 1.22-1.39 (8H, m), 1.46 (3H, d, *J* = 7.3 Hz), 1.58 (2H, brs), 1.72 (3H, s), 1.76-1.90 (4H, m), 2.02 (3H, brs), 2.16-2.20 (3H, m), 2.31-2.36 (2H, m), 2.64-2.67 (2H, m), 2.72 (1H, dd, *J* = 13.5, 5.5 Hz), 2.83 (1H, brs), 2.89-2.96 (2H, m), 3.11-3.18 (2H, m), 3.40 (1H, brs), 3.73-3.84 (3H, m), 3.93-3.95 (2H, m), 4.07 (1H, brs), 4.36 (1H, brs), 4.66 (1H, dd, *J* = 7.3, 6.1 Hz), 4.79 (1H, dd, *J* = 5.5, 5.5 Hz), 4.99 (1H, brs), 5.16-5.24 (4H, m), 5.16-5.24 (4H, m), 7.09-7.33 (5H, m); $^{13}$C NMR (100 Hz, $C_6D_6$) δ 5.3, 5.38, 5.43, 5.6, 5.9, 7.17, 7.24, 7.3, 7.4, 12.5, 13.8, 14.1, 14.5, 16.2, 17.7, 23.0, 23.2, 23.7, 27.2, 29.3, 30.3, 31.1, 31.6, 32.7, 33.0, 33.8, 34.7, 36.1, 37.3, 39.2, 42.5, 44.0, 44.9, 46.3, 48.1, 48.6, 50.9, 57.3, 57.5, 60.0, 60.4, 67.1, 69.8, 70.3, 75.1, 75.9, 76.0, 76.5, 77.2, 79.6, 130.6, 132.2, 137.8, 156.2, 172.6, 172.7, 174.1, 211.1; IR (ATR) 3456, 2954, 2876, 1730, 1708, 1458, 1379, 1238, 1174, 1079, 1004, 726 cm$^{-1}$; $[\alpha]_D^{22}$ -15.4 (*c* = 0.19, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{76}H_{145}N_2O_{15}Si_5$ ($[M+NH_4]^+$): 1465.94912 found: 1465.94822.

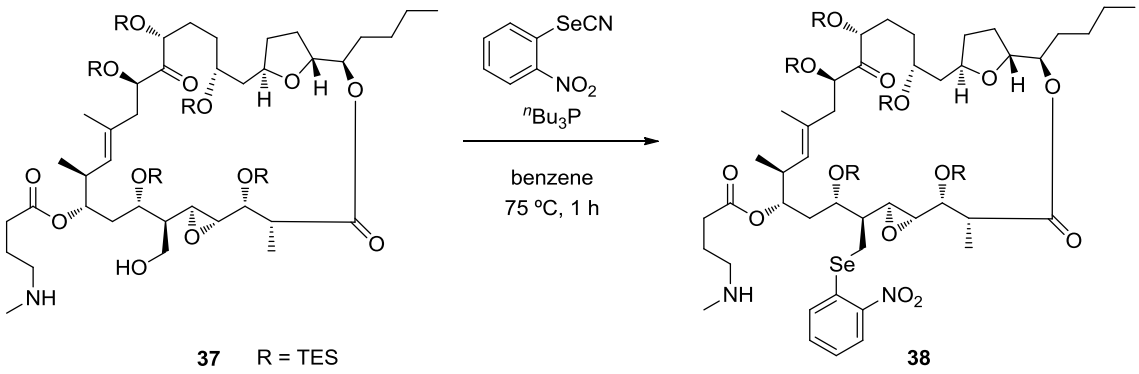
Compound **37**



To a stirred solution of **36** (13 mg, 0.0089 mmol) in EtOH (0.4 mL) and THF (0.2 mL) were added 10% Pd-C (2 mg) and ammonium formate (6 mg, 0.0951 mmol) at room temperature under Ar atmosphere. After stirring for 1 h at 50 ºC, the reaction mixture was filtered through a pad of celite. The filtrate was diluted with $H_2O$ and extracted with EtOAc. The organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by preparative thin layer chromatography ($CHCl_3$ : MeOH = 9 : 1) to give **37** (7.5 mg, 0.0057 mmol, 64%) as a colorless oil.

$^1$H NMR (400Mz, $C_6D_6$) δ 0.34-0.64 (36H, m), 0.74-0.88 (49H, m), 0.96-1.12 (5H, m), 1.17 (3H, d, *J* = 7.3 Hz), 1.33-1.41 (2H, m), 1.46 (3H, s), 1.52-1.87 (10H, m), 1.95 (3H, s), 2.05-2.10 (1H, m), 2.12 (2H, t, *J* = 7.3 Hz), 2.25 (2H, t, *J* = 7.3 Hz), 2.44 (1H, dd, *J* = 13.5, 5.8 Hz), 2.60-2.68 (2H, m), 2.88 (1H, t, *J* = 7.3 Hz), 3.48 (1H, dd, *J* = 9.7, 7.3 Hz), 3.58-3.71 (4H, m), 3.77-3.85 (1H, m), 4.04-4.08 (1H, m), 4.40 (1H, dd, *J* = 7.9, 5.5 Hz), 4.50 (1H, dd, *J* = 4.9, 4.9 Hz), 4.71-4.75 (1H, m), 4.91-4.99 (2H, m); IR (ATR) 3448, 2954, 2876, 1731, 1459, 1415, 1380, 1239, 1187, 1080, 1007, 740, 728 $cm^{-1}$; $[\alpha]_D^{23}$ -18.4 (*c* = 1.09, $CHCl_3$); HRMS ($ESI^+$) calcd for $C_{68}H_{136}NO_{13}Si_5$ ($[M+H]^+$): 1314.88580 found: 1314.88634.

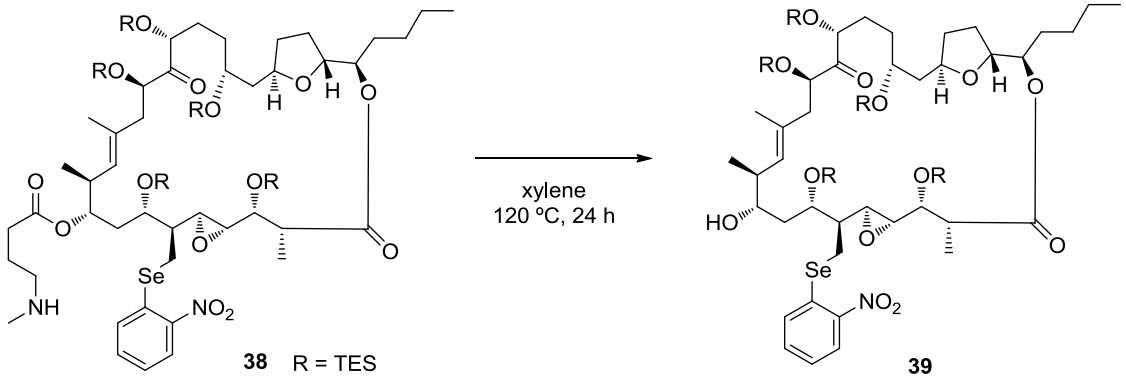
Compound **38**[S12]




**37** R = TES **38**

To a stirred solution of **37** (20 mg, 0.0152 mmol) in benzene (0.05 mL) were added 2-nitrophenylseleno cyanate (17.2 mg, 0.0757 mmol) and tri *n*-butylphosphine (1 mol/L in benzene, 0.1 mL, 0.1 mmol) at 75 ºC under Ar atmosphere. After stirring for 0.5 h at the same temperature, the reaction mixture was concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$ : MeOH = 9 : 1) to give **38** (15.8 mg, impure) as a brown oil, which was used in the next step without further purification.

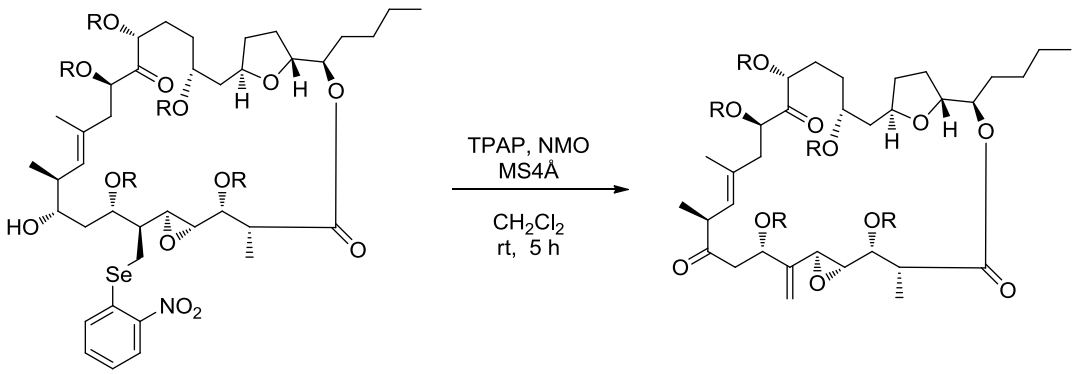
Compound **39**




**38** R = TES **39**

A solution of **38** (7 mg, impure) in xylene (2 mL) was stirred at 120 ºC under Ar atmosphere. After stirring for 24 h, the reaction mixture was cooled to rt and purified by silica gel column chromatography (*n*-hexane : EtOAc = 6 : 1) to give **39** (4 mg, impure) as a yellow oil, which was used in the next step without further purification.

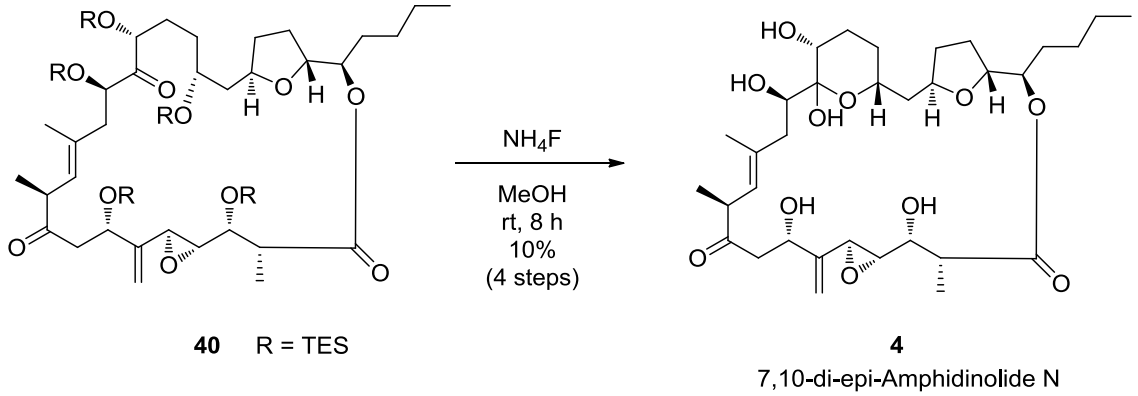
Compound **40**




**39** R = TES **40**

To a stirred solution of **39** (2 mg, impure) in $CH_2Cl_2$ (0.2 mL) were added molecular sieves 4Å (powder, 2 mg), NMO (2 mg, 0.017 mmol), and TPAP (2 mg, 0.0056 mmol) at room temperature under Ar atmosphere. After stirring for 0.5 h, the reaction mixture was diluted with $CH_2Cl_2$ and filtered through a pad of celite. The filtrate was concentrated *in vacuo* and the residue was purified by preparative thin layer chromatography (*n*-hexane : EtOAc = 6 : 1) to give **40** (1.1 mg, impure) as a colorless oil, which was used in the next step without further purification.

Compound **4** (7,10-di-epi-Amphidinolide N)




**40** R = TES

**4**
7,10-di-epi-Amphidinolide N

To a stirred solution of **40** (1.1 mg, impure) in MeOH (0.2 mL) was added ammonium fluoride (2 mg, 0.054 mmol) at room temperature under Ar atmosphere. After stirring for 8 h, the reaction mixture was concentrated *in vacuo*. The residue was purified by preparative thin layer chromatography (*n*-hexane : EtOAc = 1 : 2) to give **4** (0.2 mg, 0.0003 mmol, 10% from **37**).

$^1$H NMR (600Mz, $C_6D_6$) δ 0.92 (3H, t, *J* = 7.3 Hz), 1.04 (3H, t, *J* = 6.8 Hz), 1.11 (overlap, 1H), 1.12 (3H, t, *J* = 7.2 Hz), 1.13 (overlap, 2H), 1.16 (overlap, 2H), 1.38 (overlap, 7H), 1.49 (overlap, 1H), 1.74 (overlap, 1H), 1.89 (3H, s), 1.94 (1H, m), 2.26 (1H, m), 2.47 (1H, dd, *J* = 13.3, 9.0 Hz), 2.59 (1H, dd, *J* = 16.7, 9.3 Hz), 2.68 (1H, dd, *J* = 7.2, 3.9 Hz), 2.70 (1H, dd, *J* = 16.7, 2.7 Hz), 2.90 (1H, dd, *J* = 13.8, 3.6 Hz), 3.00 (1H, dd, *J* = 3.6, 2.2), 3.23 (1H, dd, *J* = 6.8, 9.1 Hz), 3.42 (1H, d, *J* = 2.1 Hz), 3.70 (overlap, 1H), 3.72 (overlap, 1H), 3.89 (1H, dd, *J* = 9.0, 3.5 Hz), 4.15 (1H, dd, *J* = 2.8, 2.8 Hz), 4.26 (overlap, 1H), 4.29 (overlap, 1H), 4.54 (1H, dd, *J* = 9.2, 2.5 Hz), 4.81 (overlap, 1H), 5.12 (1H, d, *J* = 9.0 Hz), 5.33 (1H, s), 5.49 (1H, s); HRMS (ESI$^-$) calcd for $C_{33}H_{51}O_{11}$ ([M-H]$^+$): 623.34314 found: 623.34334.

References

[S1] J. K. Stille, J. H. Simpson, *J. Am. Chem. Soc.* **1987**, *109*, 2138.

[S2] Z.-X. Wang, G.-A. Cao, Y. Shi, *J. Org. Chem*. **1999**, *64*, 7646.

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[S5] a) G. E. Keck, K. H. Tarbet, L. S. Geraci, *J. Am. Chem. Soc.* **1993**, *115*, 8467; b) G. E. Keck, D. Krishnamurthy, M. C. Grier, *J. Org. Chem*. **1993**, *58*, 6543; c) G. E. Keck, L. S. Geraci, *Tetrahedron Lett.* **1993**, *34*, 7827.

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[S7] a) K. C. Nicolaou, W. E. Brenzovich, P. G. Bulger, T. M. Francis, *Org. Biomol. Chem.* **2006**, *4*, 2119; b) K. C. Nicolaou, P. G. Bulger, W. E. Brenzovich, *Org. Biomol. Chem.* **2006**, *4*, 2158.

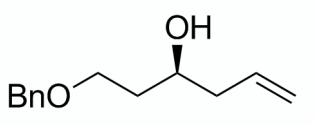
[S8] Chromatorex® NH DM2035 (200–350 mesh; Fuji Silysia Chemical, Ltd., Aichi, Japan) was used for frash column chromatography.

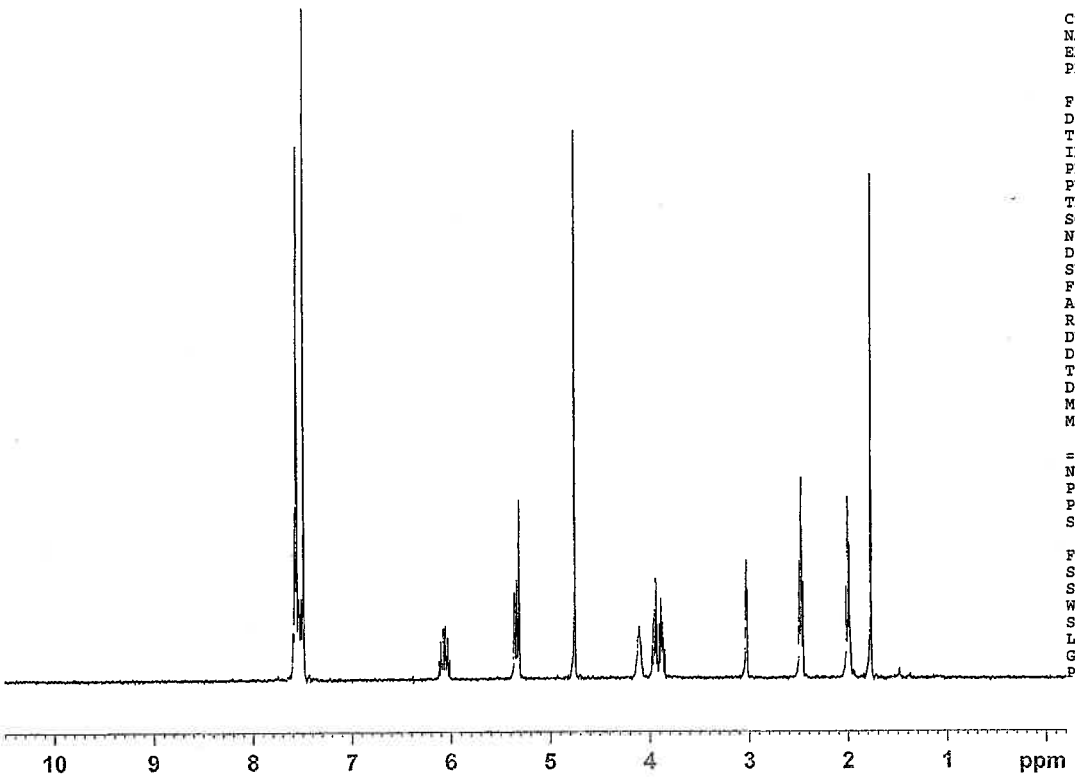
[S9] T. Fukuyama, S.-C. Lin, L. Li, *J. Am. Chem. Soc.* **1990**, *112*, 7050.

[S10] a) G. A. Kraus, B. Roth, *J. Org. Chem.* **1980**, *45*, 4825; b) G. A. Kraus, M. J. Taschner, *J. Org. Chem.* **1980**, *45*, 1175; c) B. S. Bal, W. E. Jr. Childers, H. W. Pinnick, *Tetrahedron* **1981**, *37*, 2091.

[S11] J. Inanaga, K. Hirata, H. Saeki, T. Katsuki, M. Yamaguchi, *Bull. Chem. Soc. Jpn.* **1979**, *52*, 1989.

[S12] T. W. Lee, E. J. Corey, *J. Am. Chem. Soc*. **2001**, *123*, 1872.




BRUKER

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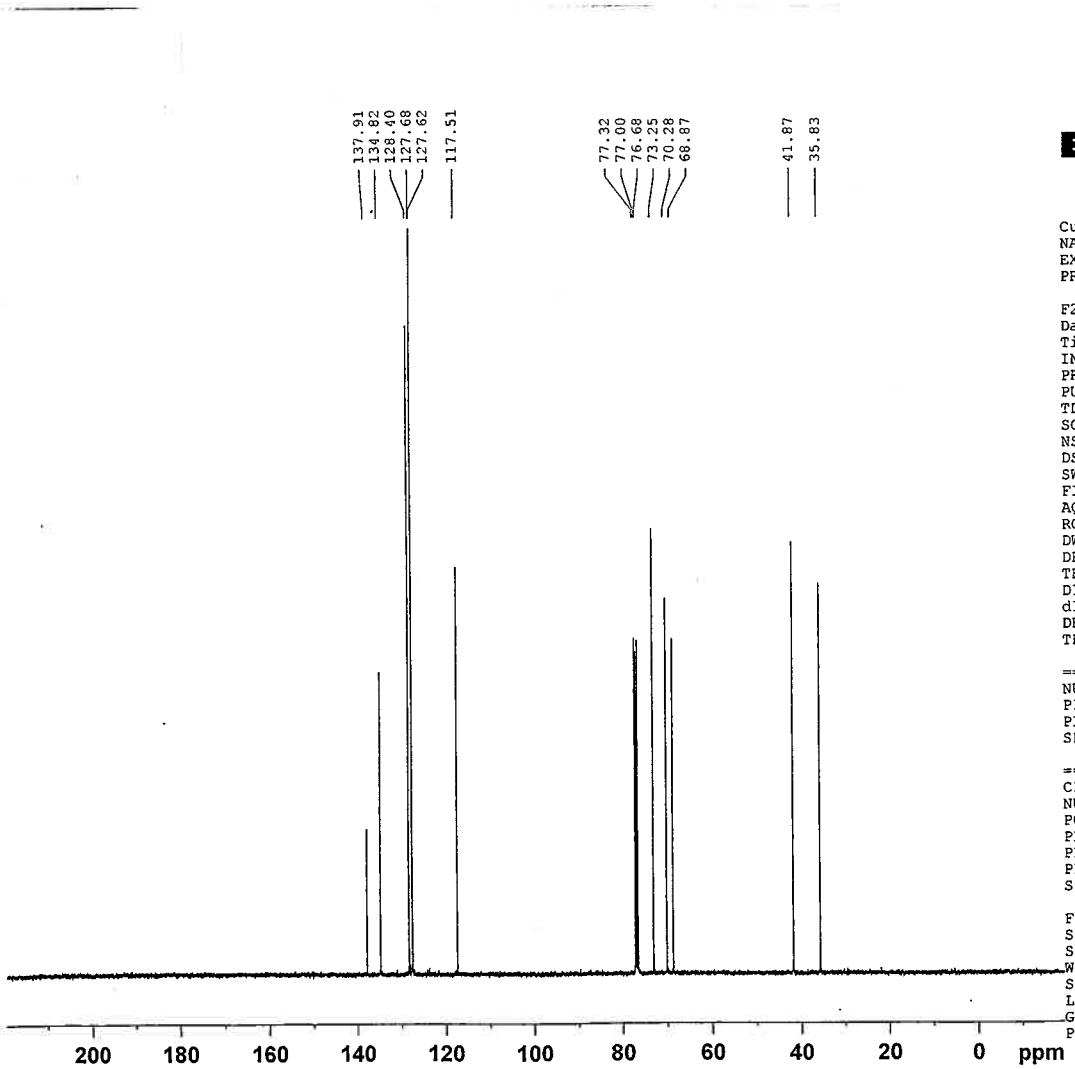


BRUKER

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PL1               -4.00 dB
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PCPD2             80.00 usec
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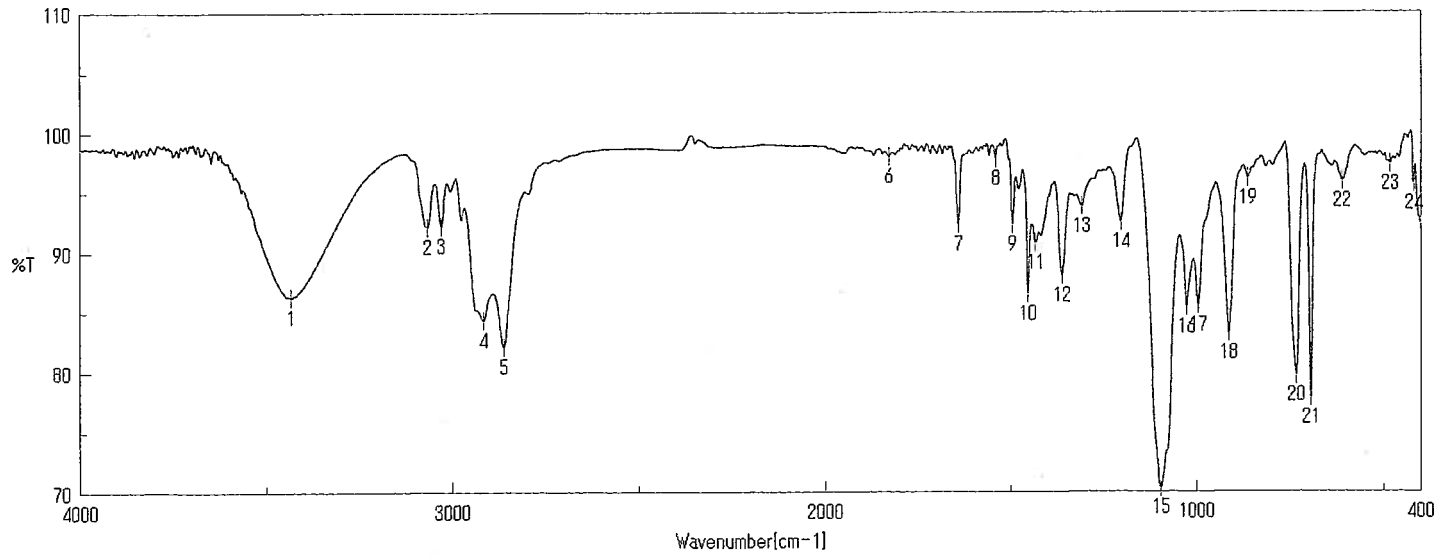


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| 測定者 | | | |
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| コメント | | | |

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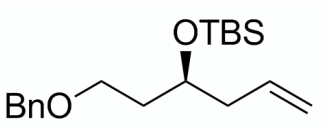





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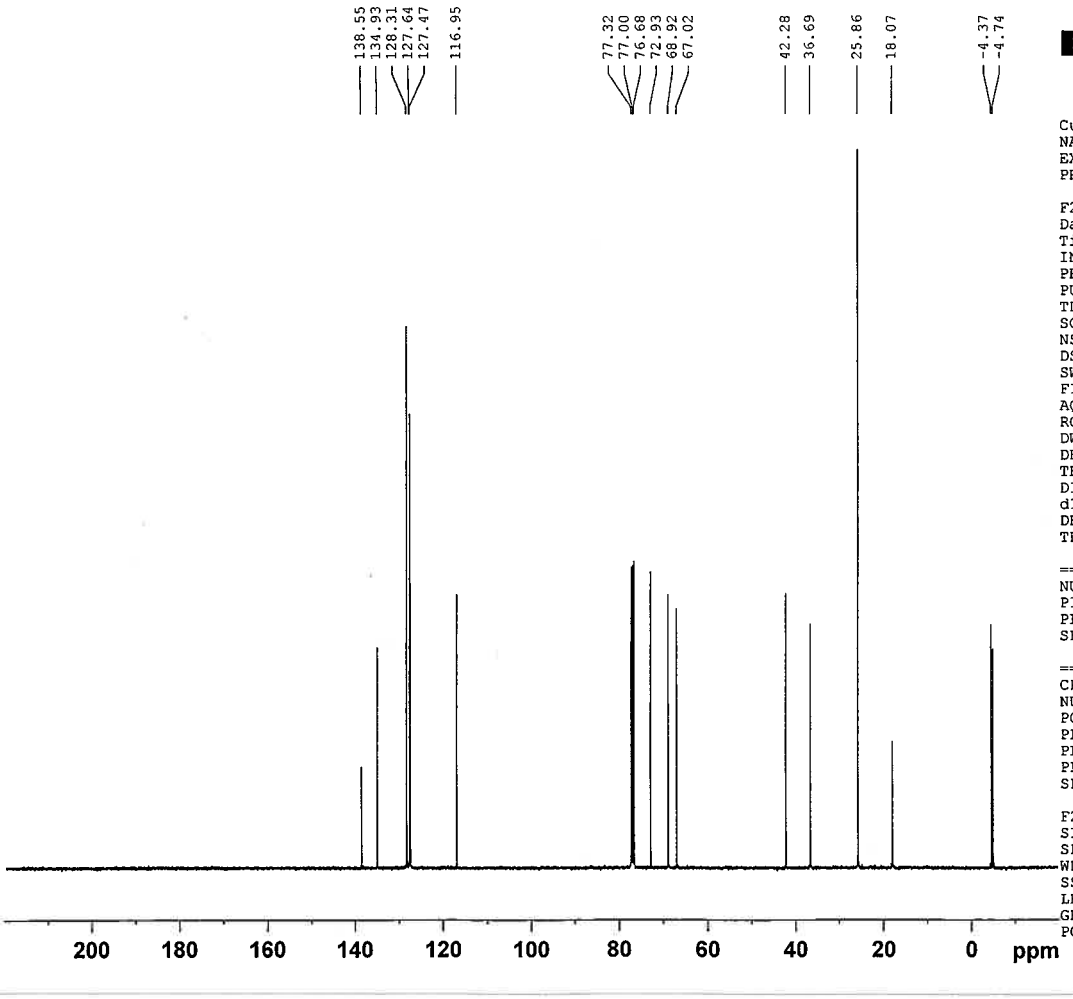


BRUKER

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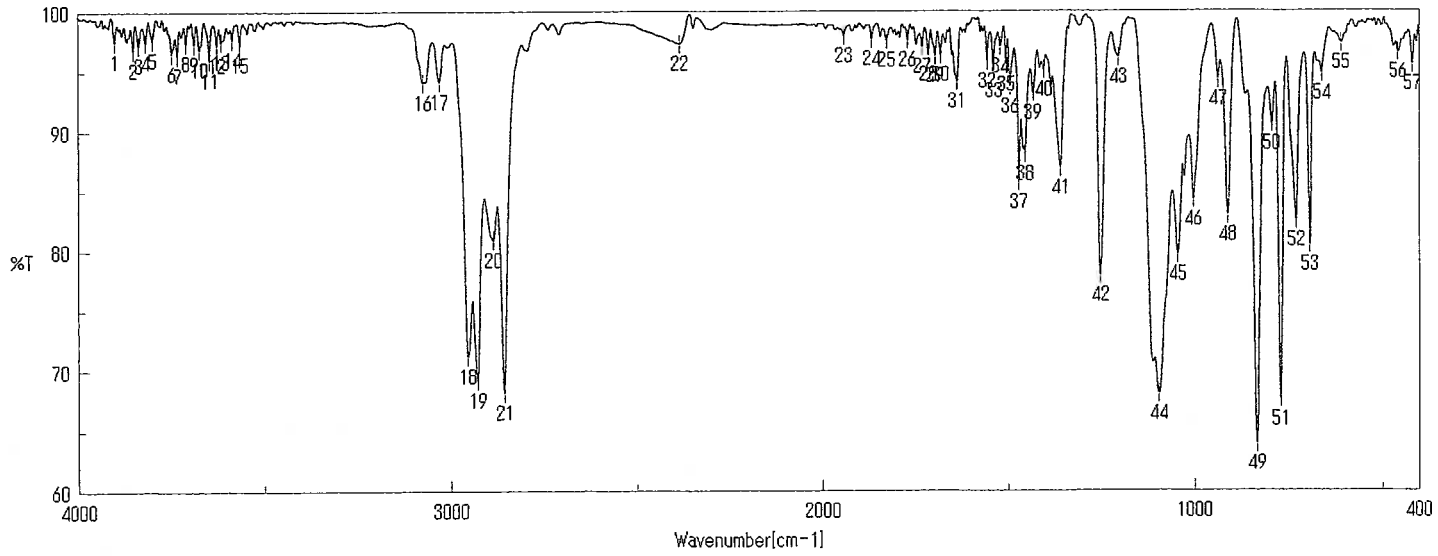


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| 13: | 3617.80, | 97.5011 | 14: | 3586.95, | 97.6806 | 15: | 3566.70, | 97.2521 | 16: | 3074.94, | 94.0272 |
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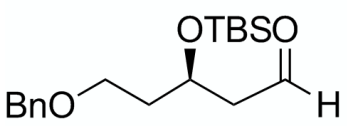





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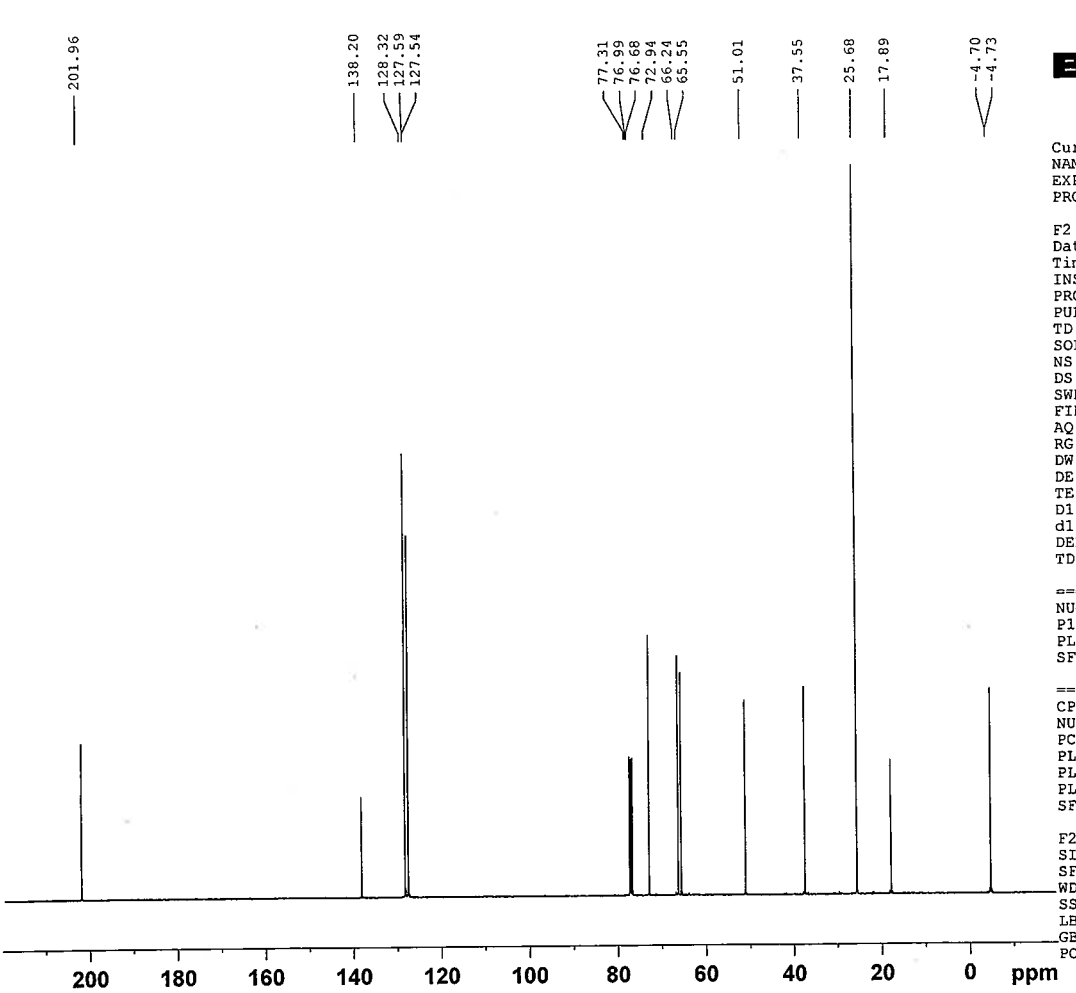


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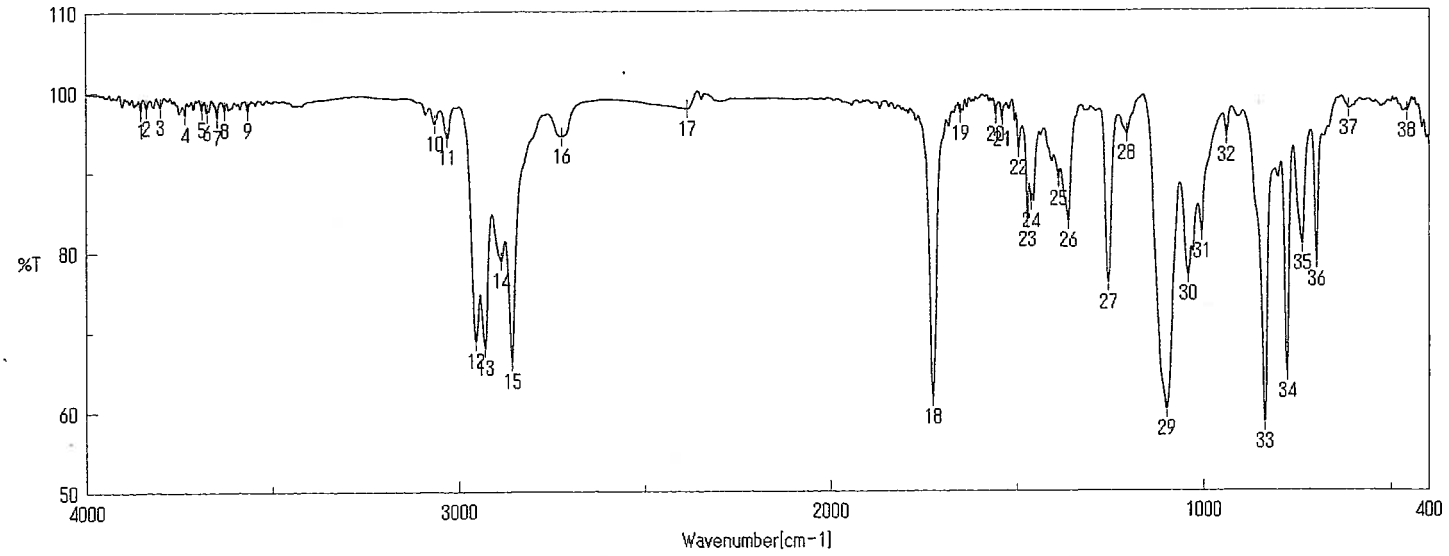


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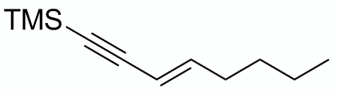






BRUKER

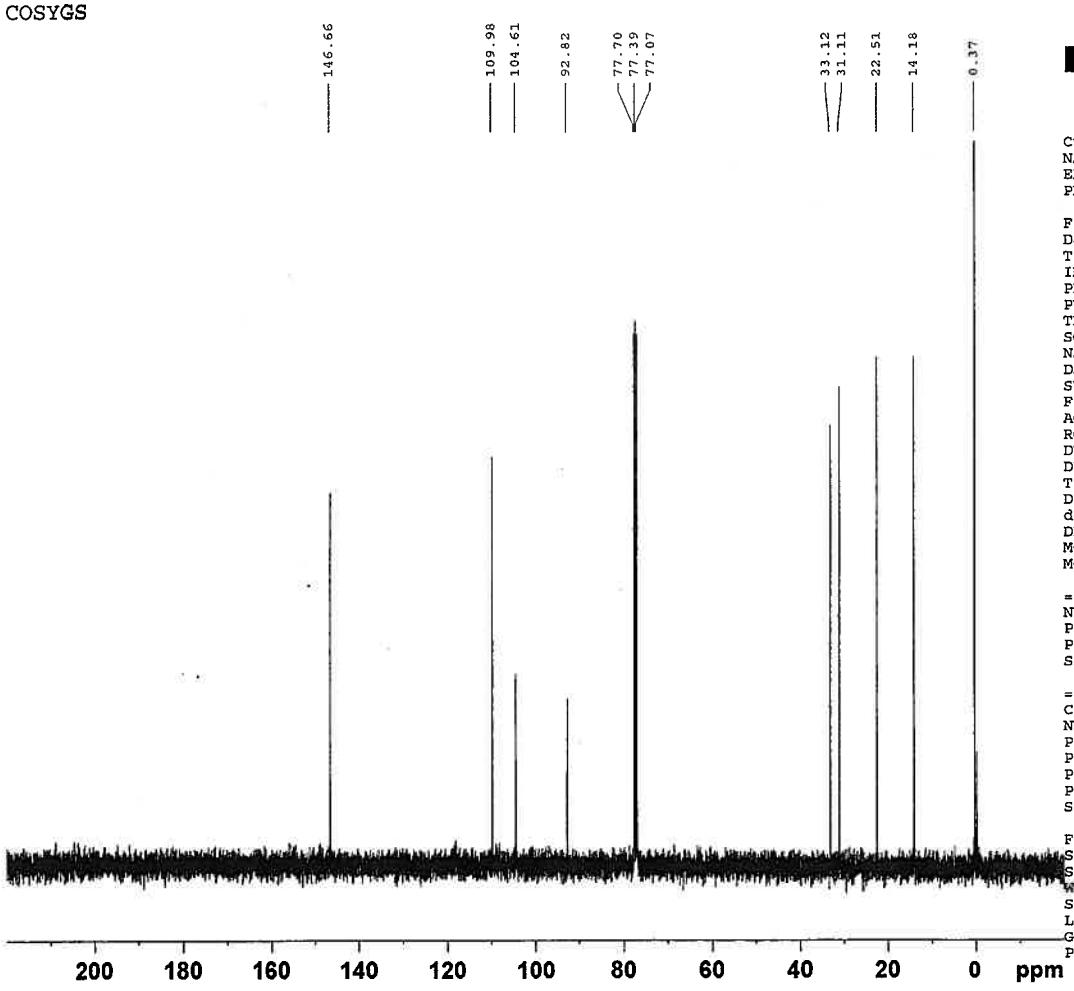
Current Data Parameters
NAME Oct27-2006
EXPNO 45
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061027
Time 13.49
INSTRUM dpx400
PROBHD 5 mm BBO 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 456.1
DW 60.800 usec
DE 6.00 usec
TE 303.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

COSYGS




BRUKER

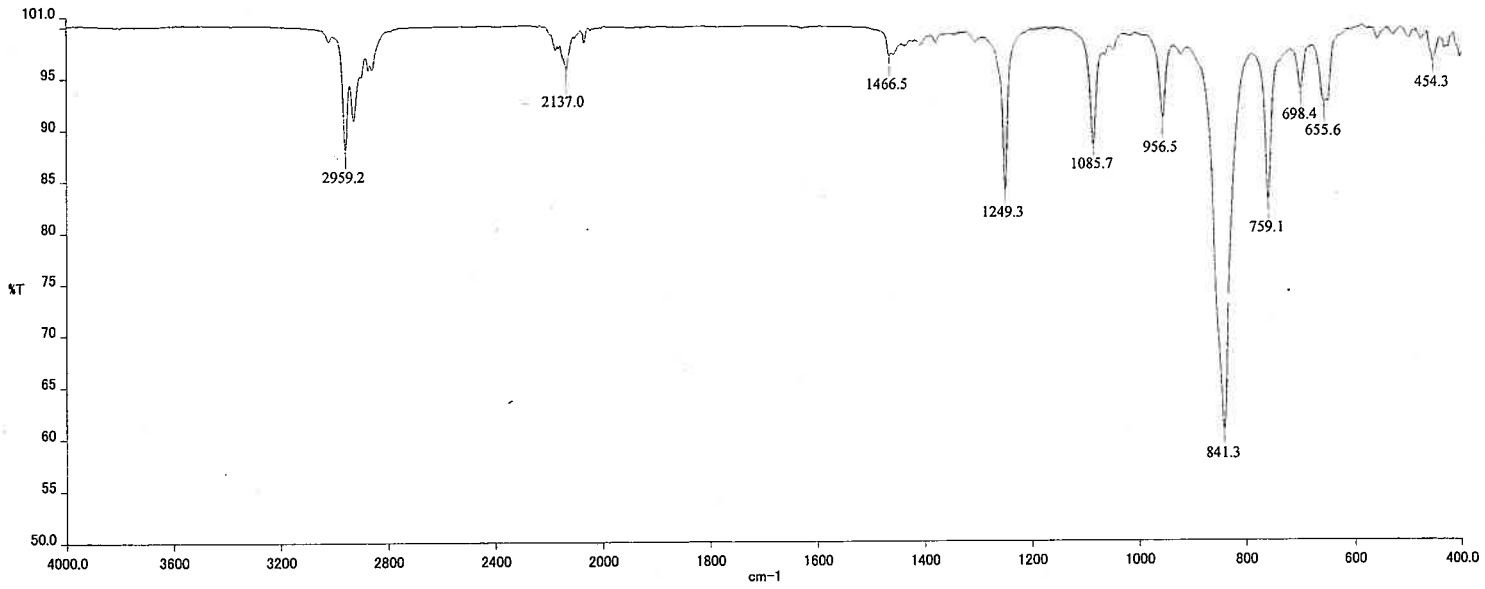
Current Data Parameters
NAME Oct27-2006
EXPNO 52
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061027
Time 14.25
INSTRUM dpx400
PROBHD 5 mm BBO 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 82
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 3251
DW 15.700 usec
DE 6.00 usec
TE 303.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 3.00 dB
SFO1 100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



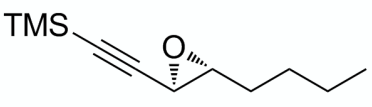

——— 6-015SM.sp

6-015SM.pk

6-015SM.sp 3601 4000.0 400.0 60.7 100.1 4.0 %T 16 1.2

REF 4000 100.0 2000 99.9 600
2959.2 88.0 2929.2 90.9 2137.0 95.9 2069.3 98.3 1466.5 96.9
1249.3 84.0 1085.7 88.4 956.5 91.0 841.3 60.7 759.1 83.0
698.4 93.6 655.6 92.4 454.3 96.3
END 13 PEAK(S) FOUND

分光器型式: Spectrum 100
測定日時: 2009年1月20日 16:00 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 6-015SM.sp
スキャン回数: 16
分解能:4.0cm-1
測定方法:ATR(ダイヤモンド/KRS-5)



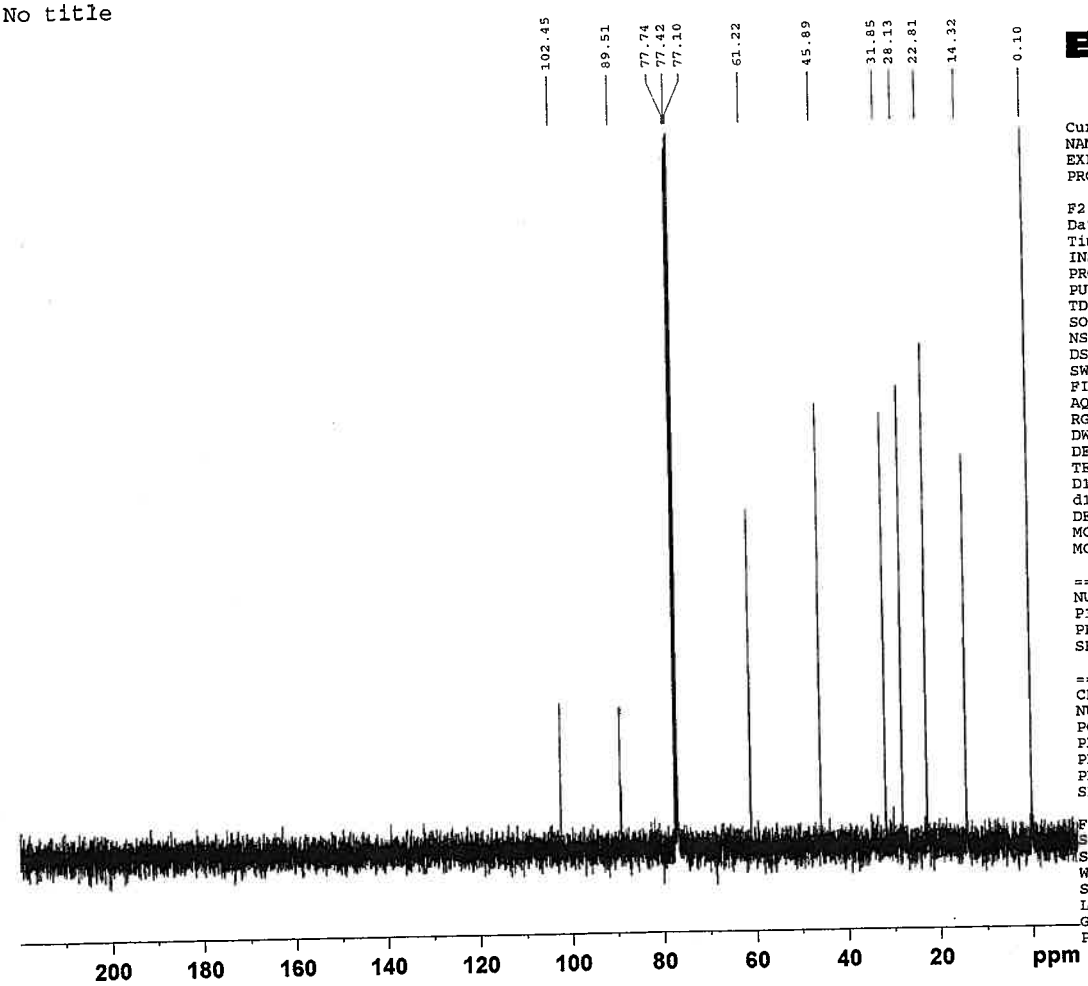




BRUKER

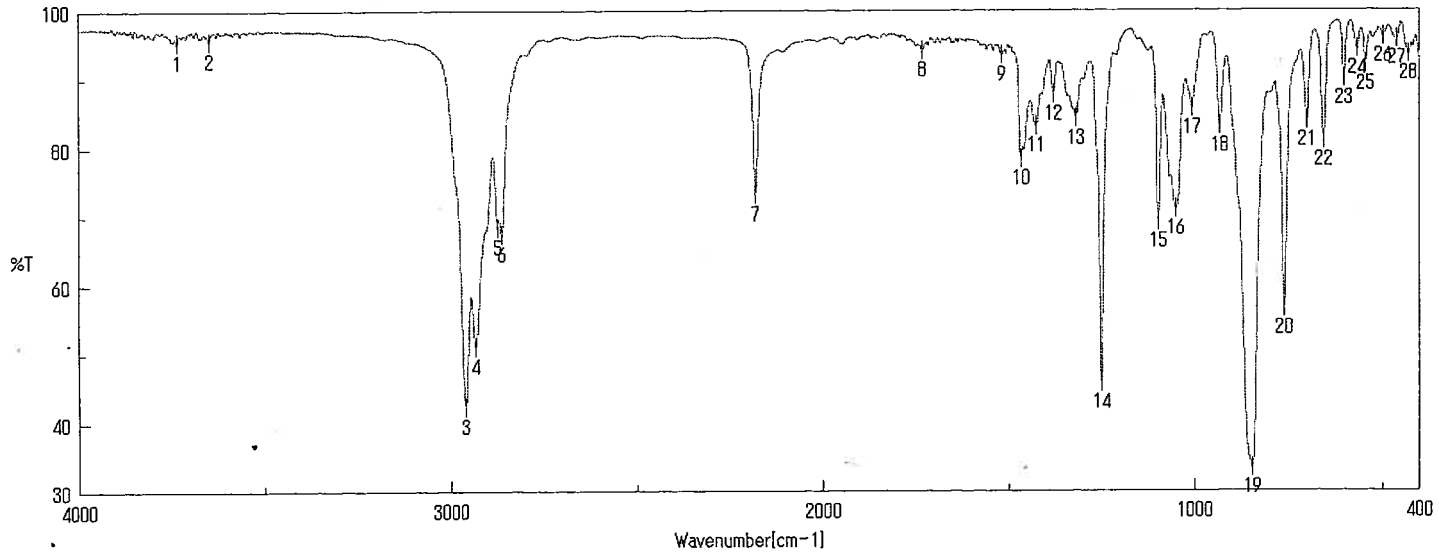
```
Current Data Parameters
NAME                 Oct31-2006
EXPNO                        12
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20061031
Time                      10.03
INSTRUM                 dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          21
DS                           0
SWH                   8223.685 Hz
FIDRES                0.250967 Hz
AQ                    1.9923444 sec
RG                         512
DW                      60.800 usec
DE                       6.00 usec
TE                      303.2 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                       8.10 usec
PL1                      1.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                       16384
SF                  400.1300092 MHz
WDW                         EM
SSB                          0
LB                       0.30 Hz
GB                           0
PC                       1.00
```

No title



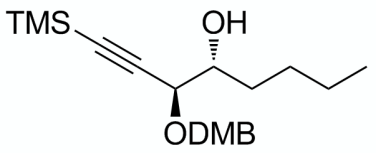

BRUKER

```
Current Data Parameters
NAME                 Nov02-2006
EXPNO                        76
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20061102
Time                      15.21
INSTRUM                 dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          84
DS                           2
SWH                  31847.133 Hz
FIDRES                0.485949 Hz
AQ                    1.0289652 sec
RG                      14596.5
DW                      15.700 usec
DE                       6.00 usec
TE                      297.2 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                       9.30 usec
PL1                      3.00 dB
SFO1                100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                      3.00 dB
PL12                    22.00 dB
PL13                    22.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                  100.6127290 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/01/29 16:16 | | |
| 測定者 | | | |
| ファイル名 | Memory#5 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3735.44, | 95.4356 | 2: | 3648.66, | 95.6694 | 3: | 2960.20, | 42.2945 | 4: | 2933.20, | 51.1333 |
| 5: | 2873.42, | 68.4602 | 6: | 2861.84, | 67.4590 | 7: | 2181.10, | 73.2184 | 8: | 1732.73, | 94.2457 |
| 9: | 1518.67, | 93.5245 | 10: | 1466.60, | 78.4011 | 11: | 1427.07, | 83.2652 | 12: | 1379.82, | 87.8053 |
| 13: | 1319.07, | 84.3277 | 14: | 1250.61, | 45.8486 | 15: | 1097.30, | 69.2646 | 16: | 1050.05, | 71.1756 |
| 17: | 1006.66, | 85.8207 | 18: | 931.45, | 83.1871 | 19: | 845.63, | 33.4332 | 20: | 760.78, | 56.6930 |

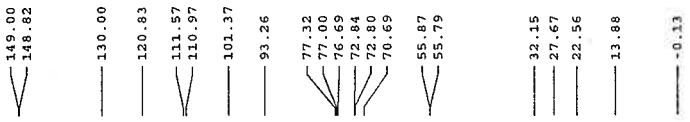


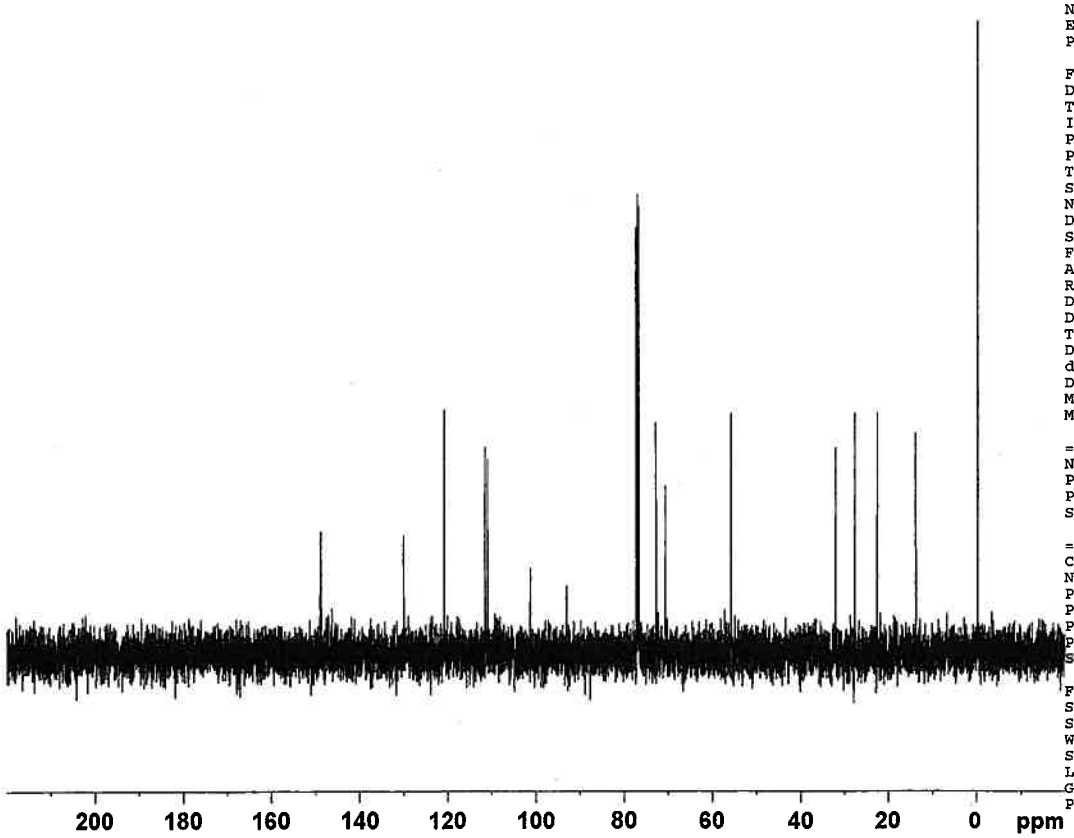




BRUKER

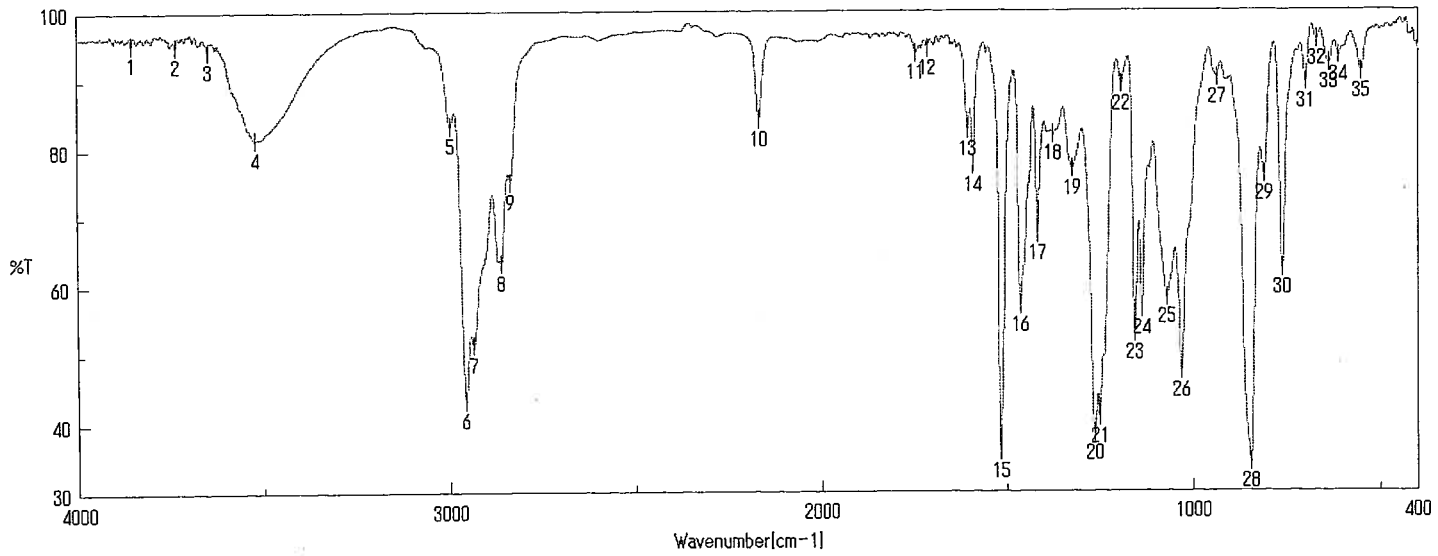
```
Current Data Parameters
NAME                  Oct16-2006
EXPNO                         12
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20061016
Time                       10.00
INSTRUM                   dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                             8
DS                             0
SWH                     8223.685 Hz
FIDRES                  0.250967 Hz
AQ                     1.9923444 sec
RG                           512
DW                        60.800 usec
DE                         6.00 usec
TE                        303.2 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                         8.10 usec
PL1                        1.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        16384
SF                  400.1300087 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



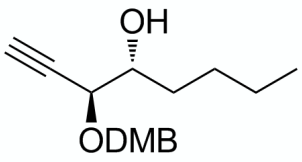

BRUKER

```
Current Data Parameters
NAME                  Jan22-2007
EXPNO                         22
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20070122
Time                       14.21
INSTRUM                   dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                            39
DS                             2
SWH                    31847.133 Hz
FIDRES                  0.485949 Hz
AQ                     1.0289652 sec
RG                        32768
DW                        15.700 usec
DE                         6.00 usec
TE                        303.2 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                          13C
P1                        10.00 usec
PL1                        2.20 dB
SFO1                100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                        3.00 dB
PL12                      20.00 dB
PL13                      22.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127740 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/01/29 16:23 | | |
| 測定者 | | | |
| ファイル名 | Memory#8 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3854.04, | 95.4786 | 2: | 3735.44, | 95.1067 | 3: | 3648.66, | 94.4123 | 4: | 3522.34, | 81.4481 |
| 5: | 2998.77, | 83.3702 | 6: | 2956.34, | 43.3017 | 7: | 2935.13, | 51.3554 | 8: | 2861.84, | 63.2805 |
| 9: | 2836.77, | 75.1143 | 10: | 2169.53, | 84.2131 | 11: | 1748.16, | 93.8342 | 12: | 1716.34, | 94.5248 |
| 13: | 1607.38, | 82.6871 | 14: | 1593.88, | 77.4567 | 15: | 1516.74, | 35.8192 | 16: | 1464.67, | 57.0787 |
| 17: | 1419.35, | 67.5171 | 18: | 1378.85, | 82.0107 | 19: | 1326.79, | 76.9550 | 20: | 1264.11, | 38.4080 |



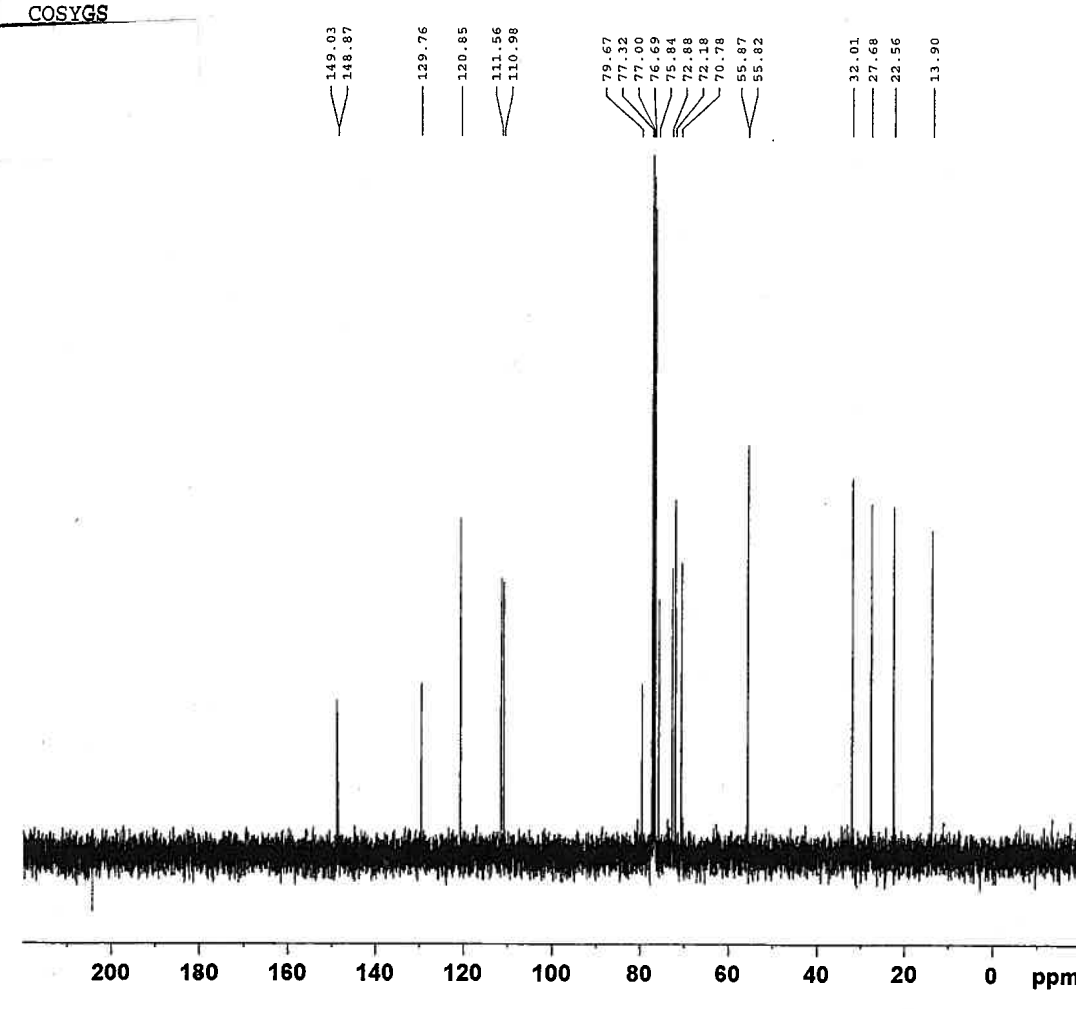




BRUKER

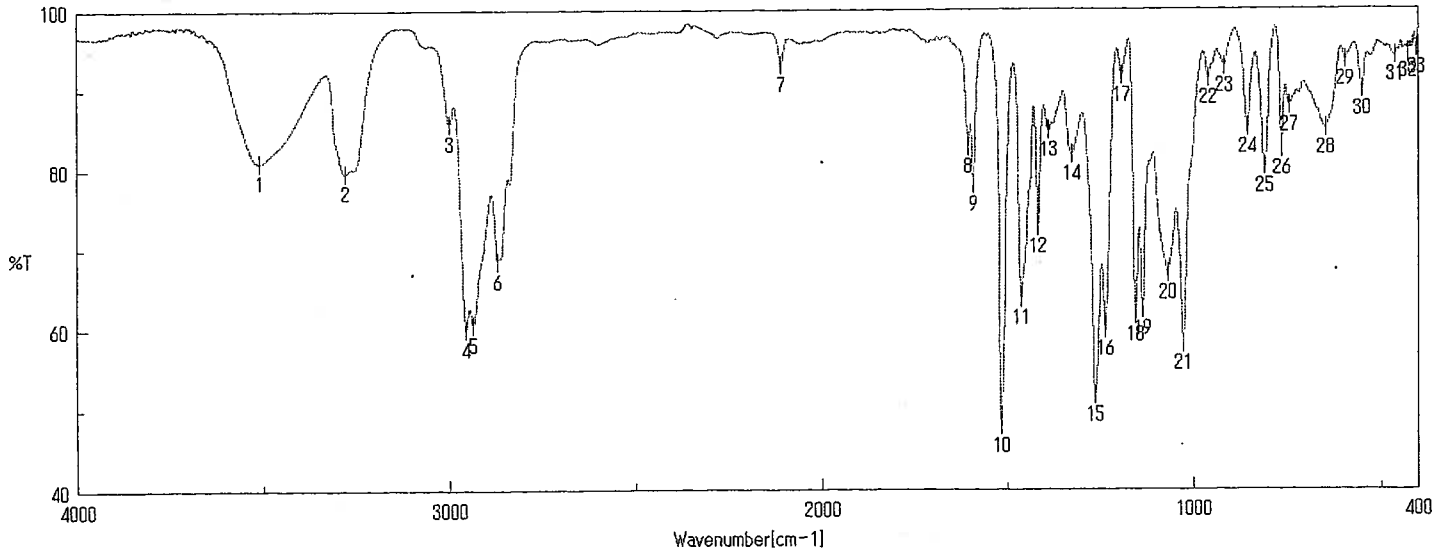
```
Current Data Parameters
NAME                  Jan06-2007
EXPNO                        151
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20070106
Time                       21.23
INSTRUM                   dpx400
PROBHD     5 mm BBO 13C-1
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                             8
DS                             0
SWH                     8223.685 Hz
FIDRES                  0.250967 Hz
AQ                    1.9923444 sec
RG                        645.1
DW                       60.800 usec
DE                        6.00 usec
TE                       303.2 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                        8.10 usec
PL1                       1.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                         16384
SF                  400.1300087 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                         1.00
```

COSYGS



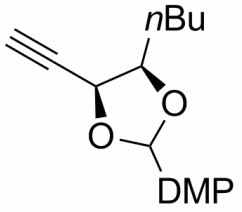

BRUKER

```
Current Data Parameters
NAME                  Jan06-2007
EXPNO                         36
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20070106
Time                       12.21
INSTRUM                   dpx400
PROBHD     5 mm BBO 13C-1
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           160
DS                             2
SWH                    31847.133 Hz
FIDRES                  0.485949 Hz
AQ                    1.0289652 sec
RG                        7298.2
DW                       15.700 usec
DE                        6.00 usec
TE                       303.2 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                         13C
P1                        9.30 usec
PL1                       3.00 dB
SFO1                100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       3.00 dB
PL12                     22.00 dB
PL13                     22.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127747 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                         1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/01/29 16:08 | | |
| 測定者 | | | |
| ファイル名 | Memory#3 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3509.81, | 80.9714 | 2: | 3279.36, | 79.6292 | 3: | 2999.73, | 85.8188 | 4: | 2954.41, | 59.9481 |
| 5: | 2935.13, | 60.5462 | 6: | 2869.56, | 68.5015 | 7: | 2109.74, | 93.0949 | 8: | 1607.38, | 82.7852 |
| 9: | 1593.88, | 78.0120 | 10: | 1516.74, | 47.9457 | 11: | 1464.67, | 63.8107 | 12: | 1420.32, | 72.7210 |
| 13: | 1392.35, | 84.7740 | 14: | 1329.68, | 81.8133 | 15: | 1265.07, | 51.7665 | 16: | 1239.04, | 59.9354 |
| 17: | 1195.65, | 91.6601 | 18: | 1158.04, | 61.7008 | 19: | 1138.76, | 62.4574 | 20: | 1071.26, | 66.9197 |







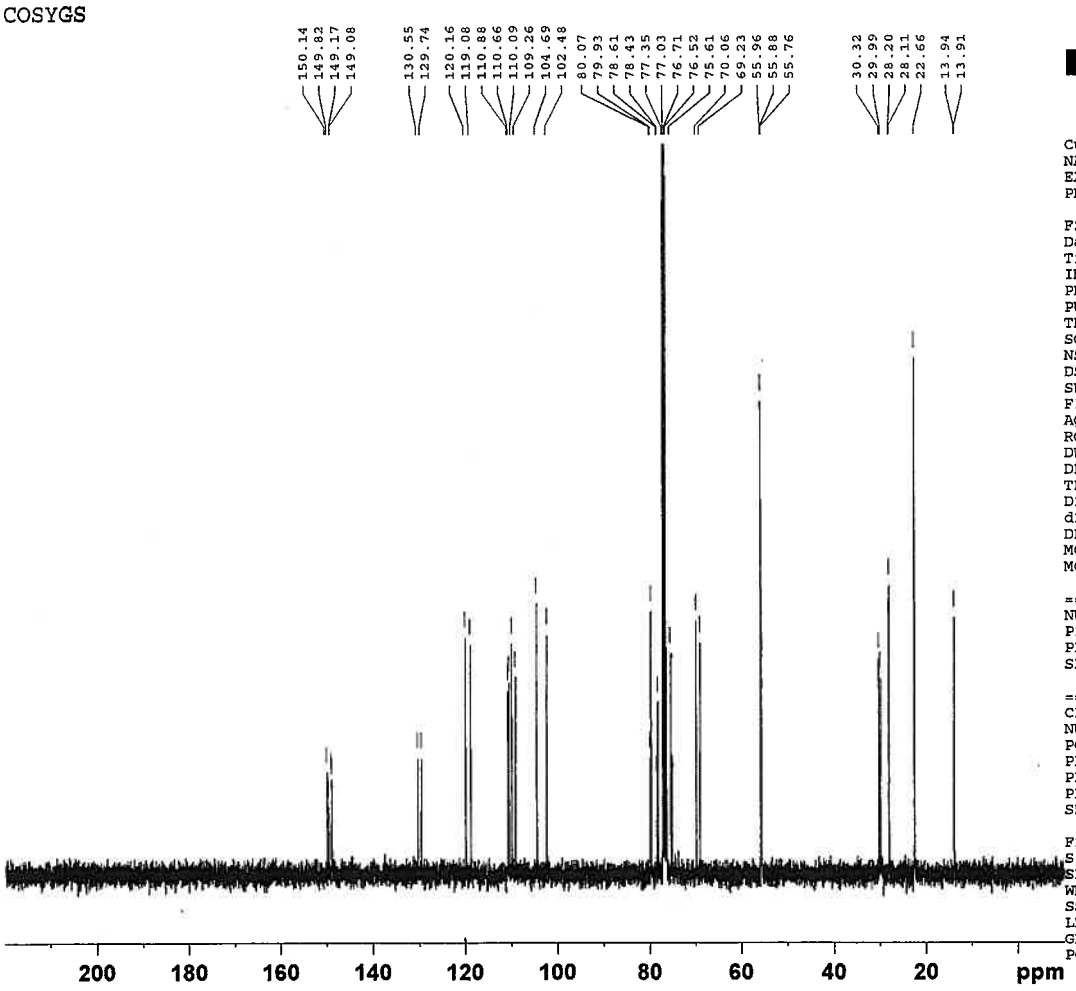
Current Data Parameters
NAME Jan26-2007
EXPNO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070126
Time 14.13
INSTRUM dpx400
PROBHD 5 mm BBO 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 812.7
DW 60.800 usec
DE 6.00 usec
TE 303.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

COSYGS



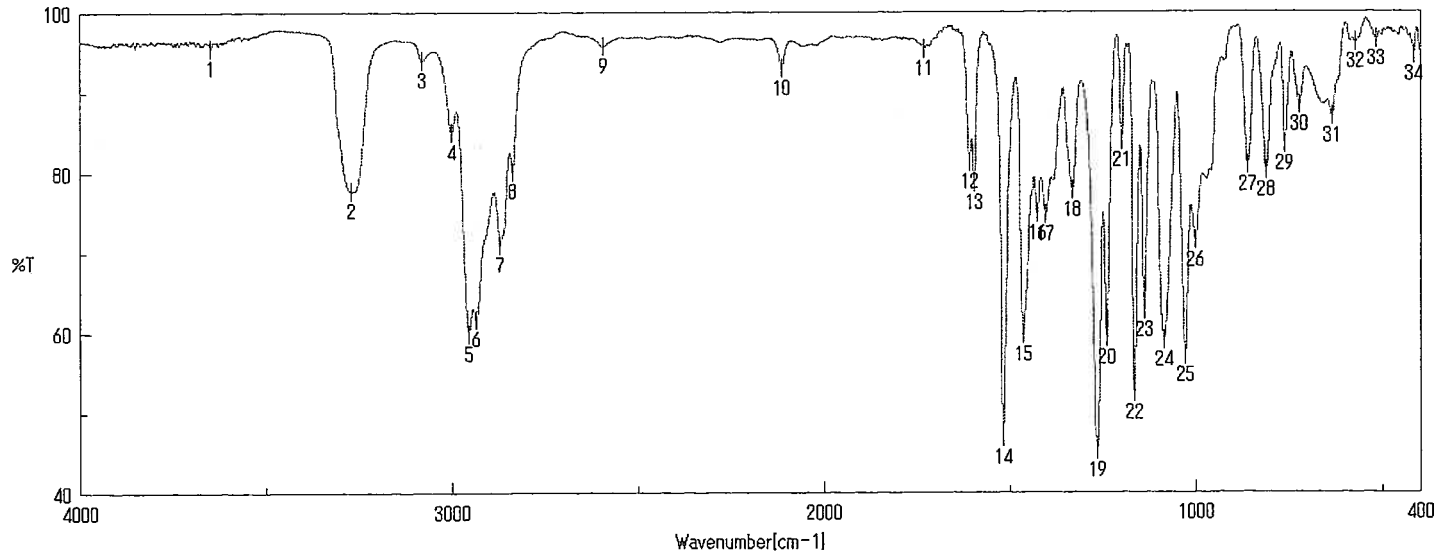

Current Data Parameters
NAME Nov18-2006
EXPNO 49
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061118
Time 14.33
INSTRUM dpx400
PROBHD 5 mm BBO 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 221
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 3649.1
DW 15.700 usec
DE 6.00 usec
TE 303.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 3.00 dB
SFO1 100.6254358 MHz

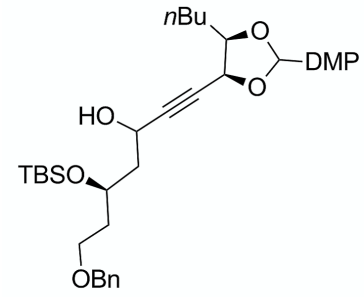
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/01/29 15:42 | | |
| 測定者 | | | |
| ファイル名 | Memory#4 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1: | 3647.70, | 95.5825 | 2: | 3269.72, | 77.7557 | 3: | 3079.76, | 94.0105 | 4: | 3000.69, | 85.0728 |
| 5: | 2955.38, | 59.8116 | 6: | 2936.09, | 61.7417 | 7: | 2871.49, | 70.9889 | 8: | 2837.74, | 80.1689 |
| 9: | 2593.79, | 95.8121 | 10: | 2114.56, | 93.0170 | 11: | 1731.76, | 95.4584 | 12: | 1609.31, | 81.3072 |
| 13: | 1596.77, | 78.7174 | 14: | 1518.67, | 46.8763 | 15: | 1464.67, | 59.7451 | 16: | 1427.07, | 74.8979 |
| 17: | 1404.89, | 74.7521 | 18: | 1331.61, | 77.7911 | 19: | 1265.07, | 45.2871 | 20: | 1239.04, | 59.3724 |

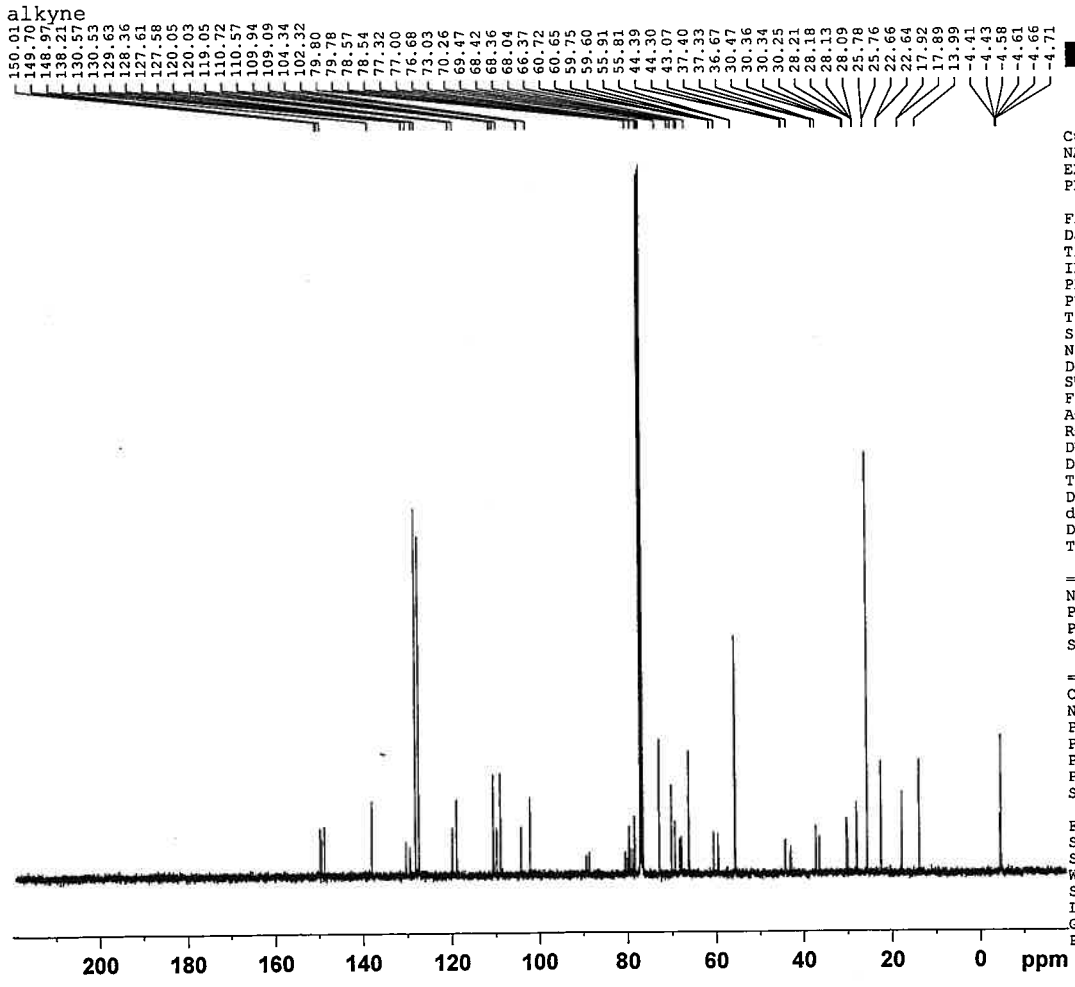






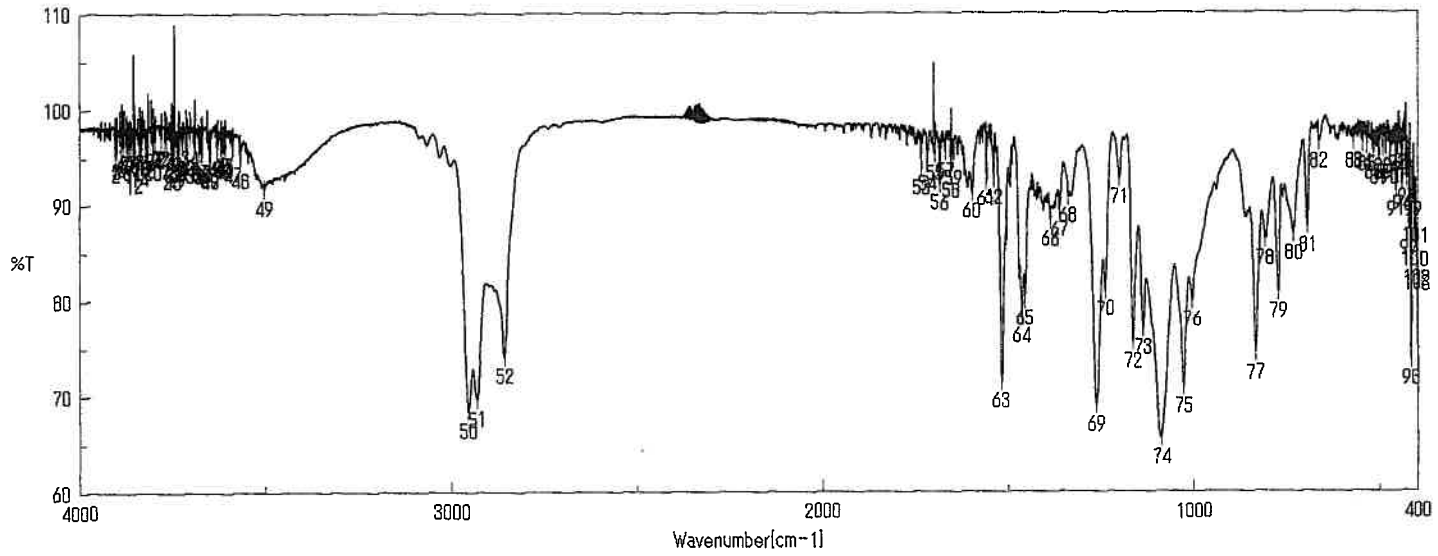
BRUKER

```
Current Data Parameters
NAME      May30-2007-hayashi
EXPNO                     50
PROCNO                     1

F2 - Acquisition Parameters
Date_              20070531
Time                   1.40
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        0
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       57
DW                   60.800 usec
DE                    6.00 usec
TE                   295.8 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     12.00 usec
PL1                    -4.00 dB
SFO1           400.1824713 MHz

F2 - Processing parameters
SI                     32768
SF             400.1800075 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



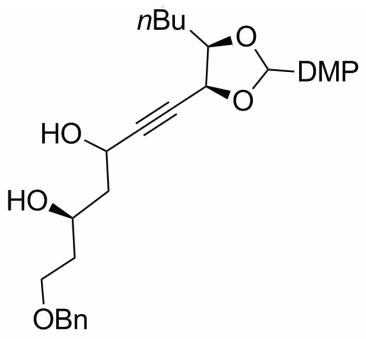

BRUKER

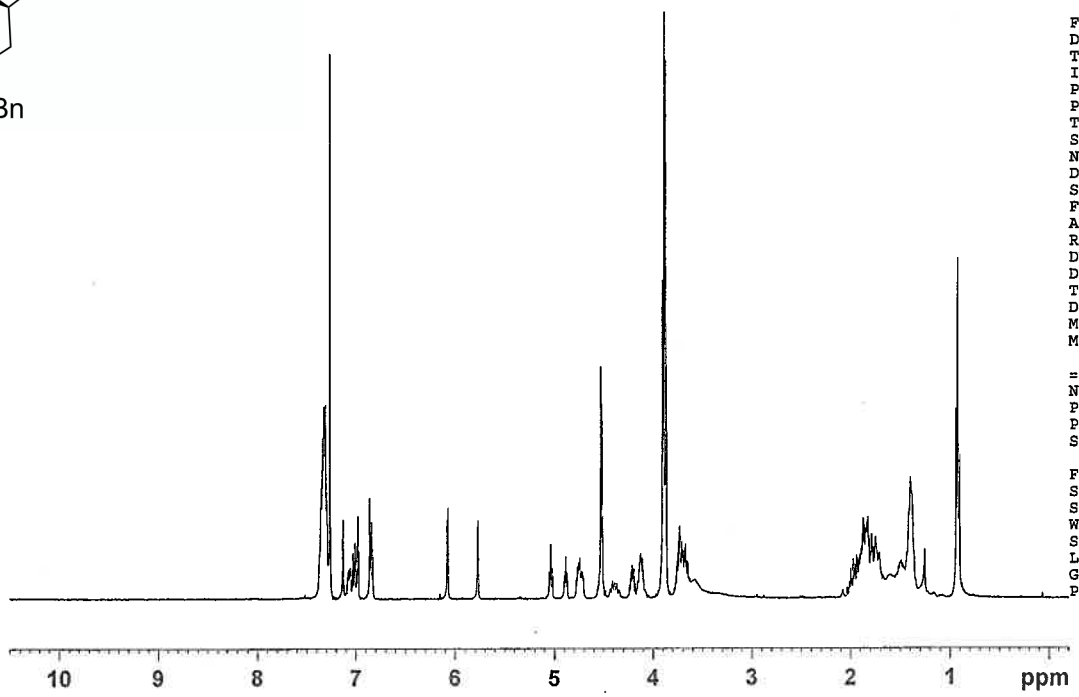
```
Current Data Parameters
NAME      May30-2007-hayashi
EXPNO                     51
PROCNO                     1

F2 - Acquisition Parameters
Date_              20070531
Time                   2.40
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      40.3
DW                   20.800 usec
DE                    6.00 usec
TE                   297.1 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     7.20 usec
PL1                    -4.00 dB
SFO1           100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    -4.00 dB
PL12                   15.00 dB
PL13                   15.00 dB
SFO2           400.1816007 MHz

F2 - Processing parameters
SI                     32768
SF             100.6253456 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 16 | 分解 | 1 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/06/12 20:59 | | |
| 測定者 | | | |
| ファイル名 | Memory#7 | | |
| サンプル名 | buckground | | |
| コメント | | | |

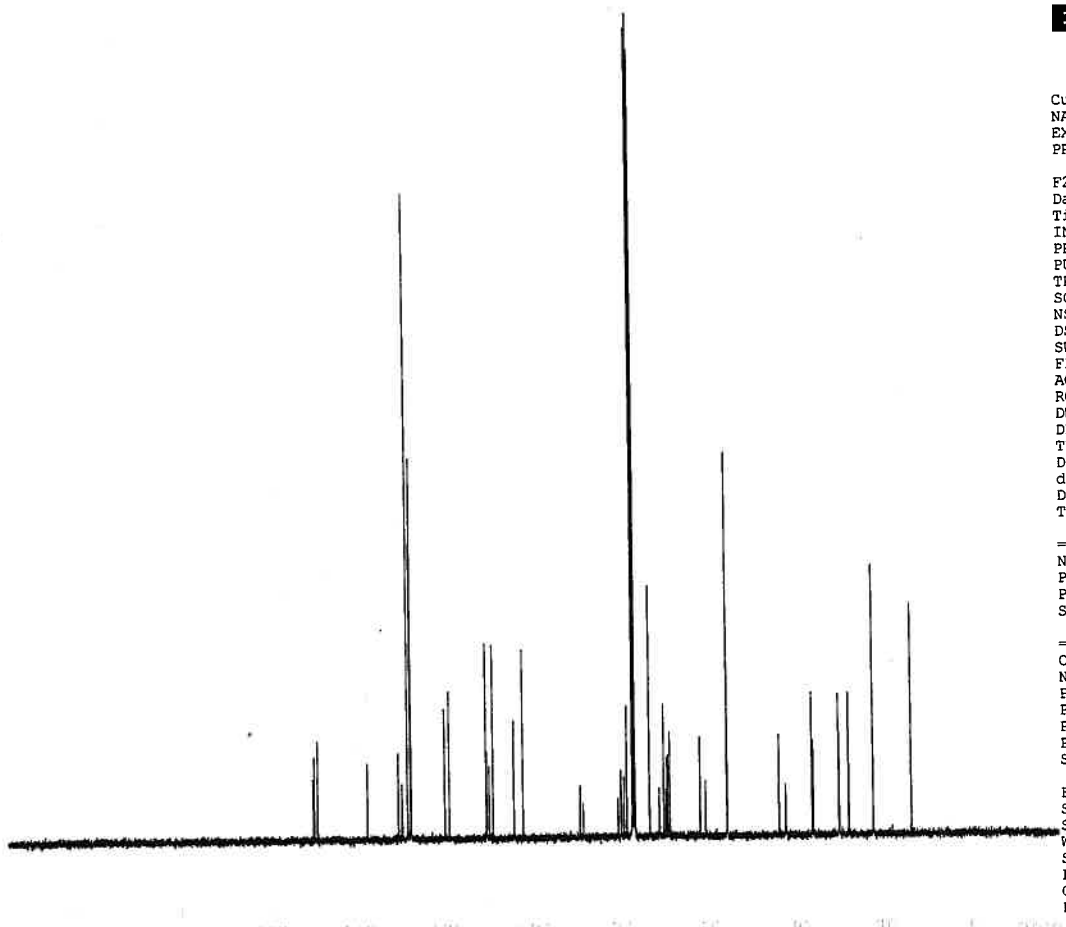
| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1: | 3904.42, | 95.7357 | 2: | 3902.25, | 95.3493 | 3: | 3899.60, | 95.6767 | 4: | 3891.65, | 96.4808 |
| 5: | 3886.10, | 95.9304 | 6: | 3880.80, | 96.3094 | 7: | 3874.53, | 96.5233 | 8: | 3870.43, | 95.5831 |
| 9: | 3865.37, | 96.1507 | 10: | 3862.00, | 96.3076 | 11: | 3854.76, | 95.2605 | 12: | 3852.35, | 94.1288 |
| 13: | 3849.94, | 96.5814 | 14: | 3838.13, | 94.9181 | 15: | 3835.24, | 95.8700 | 16: | 3832.10, | 96.6105 |
| 17: | 3821.98, | 96.0316 | 18: | 3816.44, | 95.7531 | 19: | 3807.28, | 96.7789 | 20: | 3801.73, | 95.5190 |



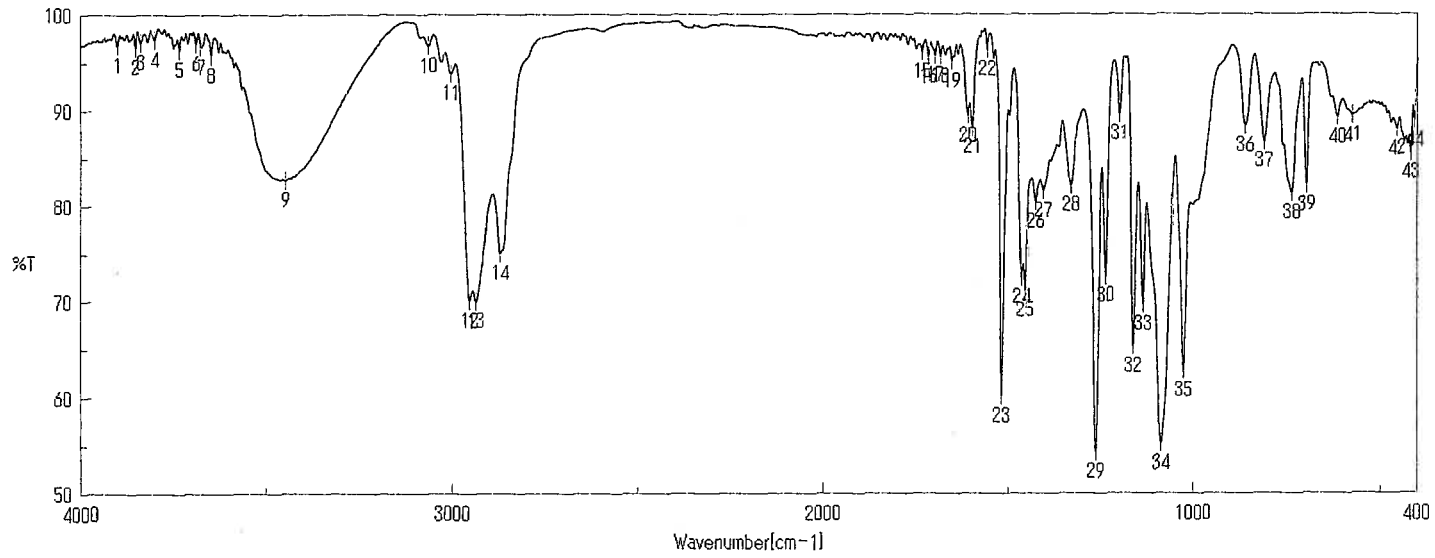

BRUKER

```
Current Data Parameters
NAME          Feb13-2007
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070213
Time              11.38
INSTRUM           dpx400
PROBHD   5 mm BBO 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ             1.9923444 sec
RG                1149.4
DW                60.800 usec
DE                 6.00 usec
TE                303.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.90 usec
PL1                3.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300087 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



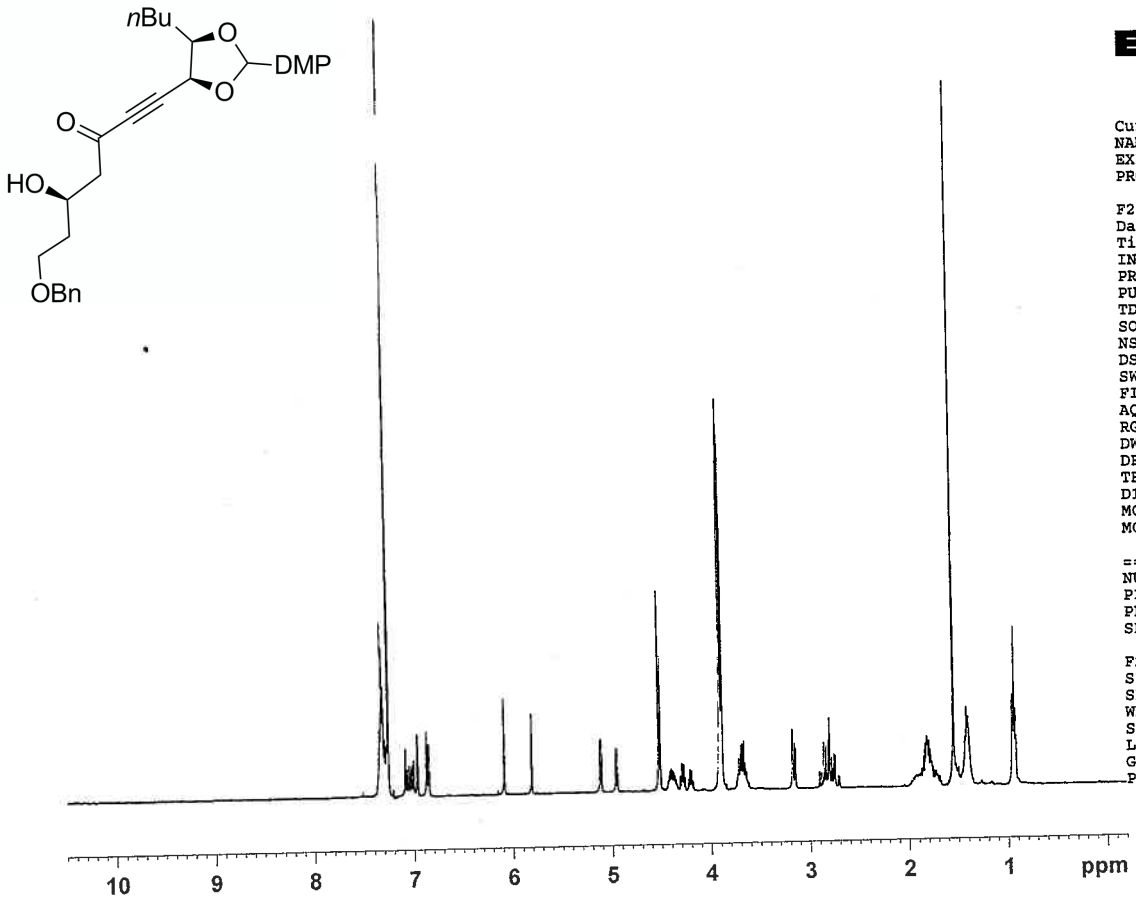
BRUKER

```
Current Data Parameters
NAME      Apr02-2007-hayashi
EXPNO                   80
PROCNO                   1

F2 - Acquisition Parameters
Date_           20070403
Time              14.46
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES           0.366798 Hz
AQ             1.3631988 sec
RG                   114
DW                20.800 usec
DE                 6.00 usec
TE                297.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.20 usec
PL1               -4.00 dB
SFO1         100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2         400.1816007 MHz

F2 - Processing parameters
SI                 32768
SF          100.6253476 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/04/04 20:34 | | |
| 測定者 | | | |
| ファイル名 | Memory#7 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1: | 3901.29, | 96.8622 | 2: | 3852.11, | 96.6631 | 3: | 3837.65, | 97.0935 | 4: | 3800.04, | 97.4233 |
| 5: | 3734.48, | 96.3919 | 6: | 3688.19, | 97.0297 | 7: | 3674.69, | 96.6342 | 8: | 3647.70, | 95.7723 |
| 9: | 3446.17, | 82.7460 | 10: | 3063.37, | 96.7845 | 11: | 3002.62, | 93.8954 | 12: | 2952.48, | 70.1243 |
| 13: | 2935.13, | 70.0416 | 14: | 2869.56, | 75.1254 | 15: | 1732.73, | 96.1171 | 16: | 1716.34, | 95.6138 |
| 17: | 1698.02, | 95.7512 | 18: | 1683.55, | 95.6526 | 19: | 1652.70, | 95.1716 | 20: | 1609.31, | 89.4769 |

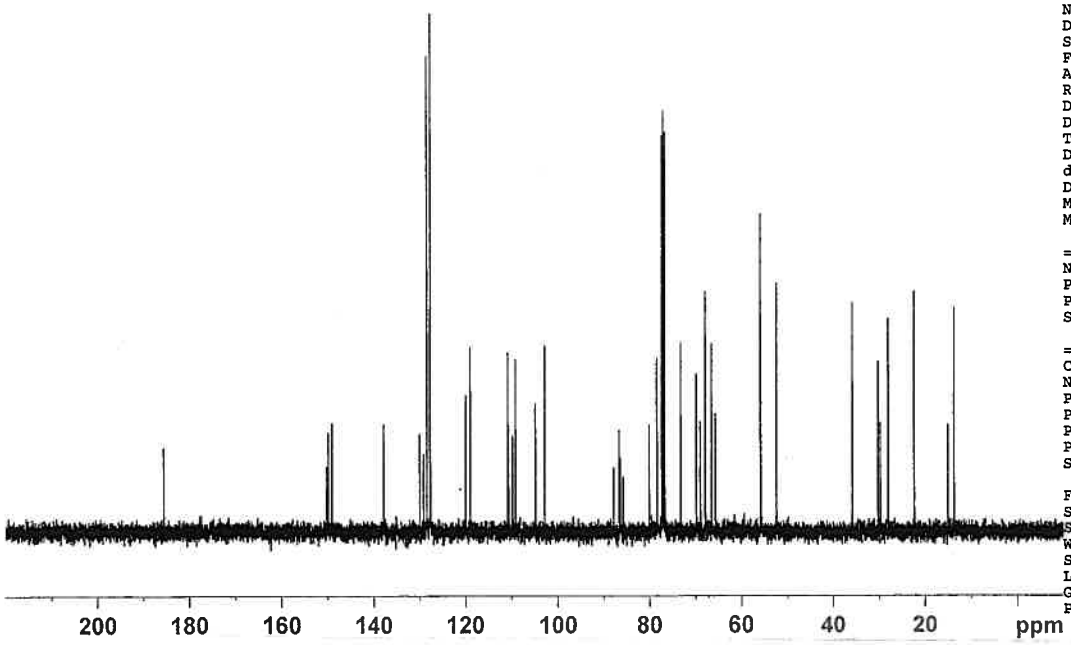



BRUKER

```
Current Data Parameters
NAME                  Apr02-2007
EXPNO                         92
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20070402
Time                      21.46
INSTRUM                 dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                             8
DS                             0
SWH                 8223.685 Hz
FIDRES              0.250967 Hz
AQ                  1.9923444 sec
RG                          1024
DW                     60.800 usec
DE                      6.00 usec
TE                      303.2 K
D1                  1.00000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                       8.10 usec
PL1                      1.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                         16384
SF                  400.1300092 MHz
WDW                           EM
SSB                            0
LB                       0.30 Hz
GB                             0
PC                          1.00
```

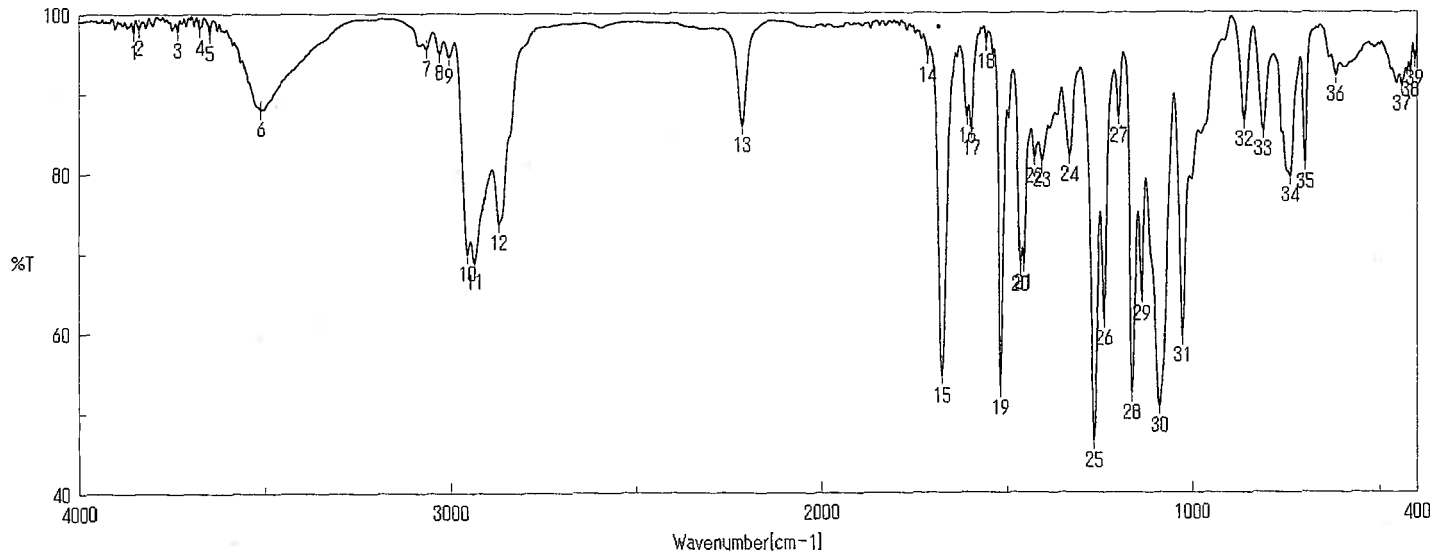
COSYGS

185.58
185.56
150.20
149.85
149.14
149.02
137.80
129.92
129.08
128.36
127.66
127.56
119.96
118.98
110.82
110.64
109.81
109.14
104.89
102.85
87.88
86.67
86.43
85.80
80.07
78.38
77.32
77.00
76.68
73.22
73.21
69.83
68.98
67.90
66.52
65.70
55.85
55.80
52.43
35.85
30.26
29.85
28.12
28.04
22.49
22.47
15.15
13.84
13.81



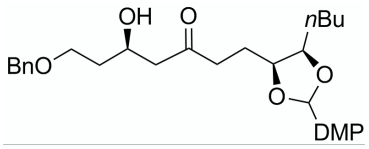

BRUKER

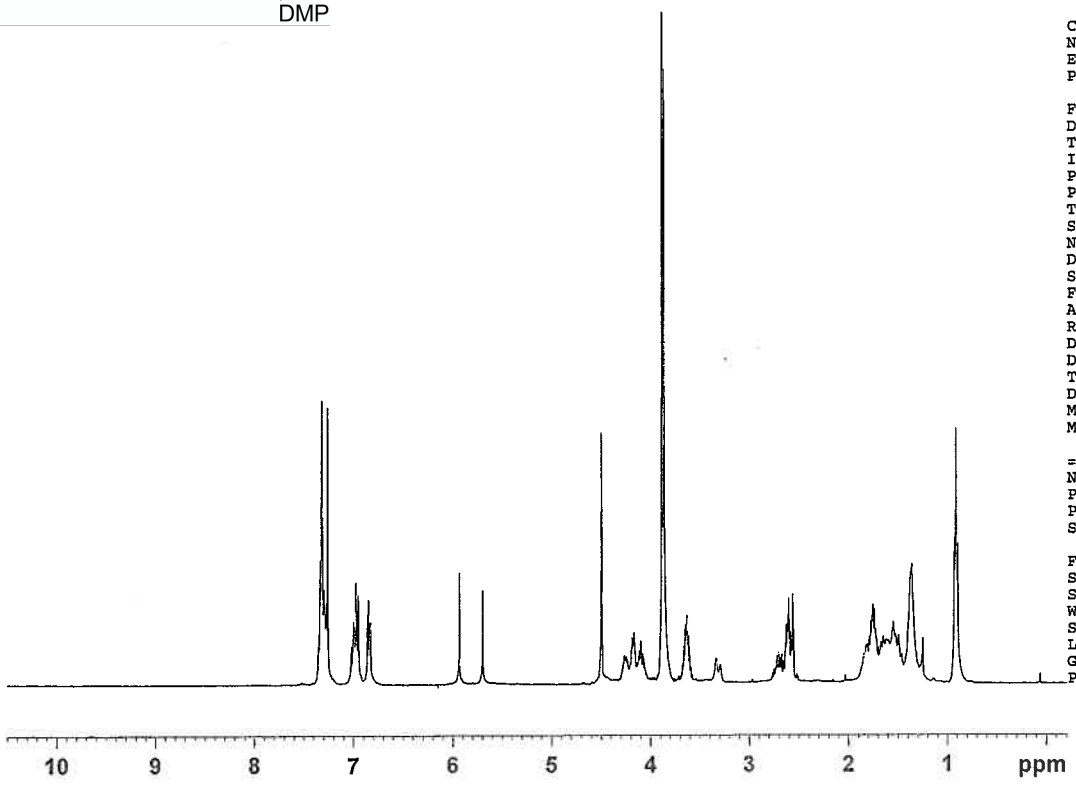
```
Current Data Parameters
NAME                  Mar31-2007
EXPNO                        106
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20070331
Time                      20.30
INSTRUM                 dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                            72
DS                             2
SWH                31847.133 Hz
FIDRES              0.485949 Hz
AQ                  1.0289652 sec
RG                      10321.3
DW                     15.700 usec
DE                      6.00 usec
TE                      303.2 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                        13C
P1                       9.30 usec
PL1                      3.00 dB
SFO1               100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                         1H
PCPD2                   80.00 usec
PL2                      3.00 dB
PL12                    22.00 dB
PL13                    22.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                 100.6127795 MHz
WDW                           EM
SSB                            0
LB                       1.00 Hz
GB                             0
PC                          1.00
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/04/04 20:18 | | |
| 測定者 | | | |
| ファイル名 | Memory#5 | | |
| サンプル名 | buckground | | |
| コメント | | | |

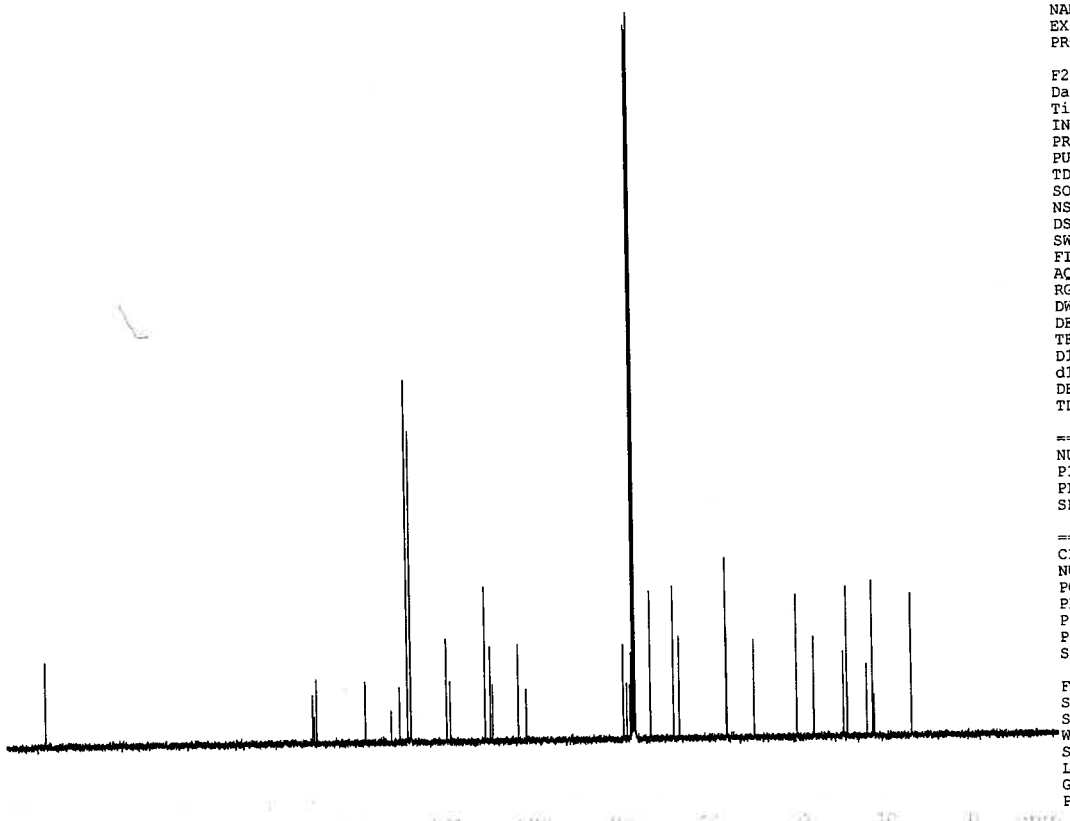
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3852.11, | 97.8664 | 2: | 3837.65, | 98.2421 | 3: | 3734.48, | 98.0097 | 4: | 3674.69, | 98.3380 |
| 5: | 3648.66, | 97.5930 | 6: | 3509.81, | 88.0843 | 7: | 3064.33, | 95.6304 | 8: | 3029.62, | 95.0783 |
| 9: | 3003.59, | 94.6301 | 10: | 2954.41, | 69.9048 | 11: | 2935.13, | 68.8814 | 12: | 2869.56, | 73.7164 |
| 13: | 2213.88, | 85.9454 | 14: | 1716.34, | 94.6865 | 15: | 1676.80, | 54.5614 | 16: | 1608.34, | 86.9969 |
| 17: | 1596.77, | 85.4599 | 18: | 1558.20, | 96.3382 | 19: | 1518.67, | 53.0061 | 20: | 1464.67, | 68.3940 |



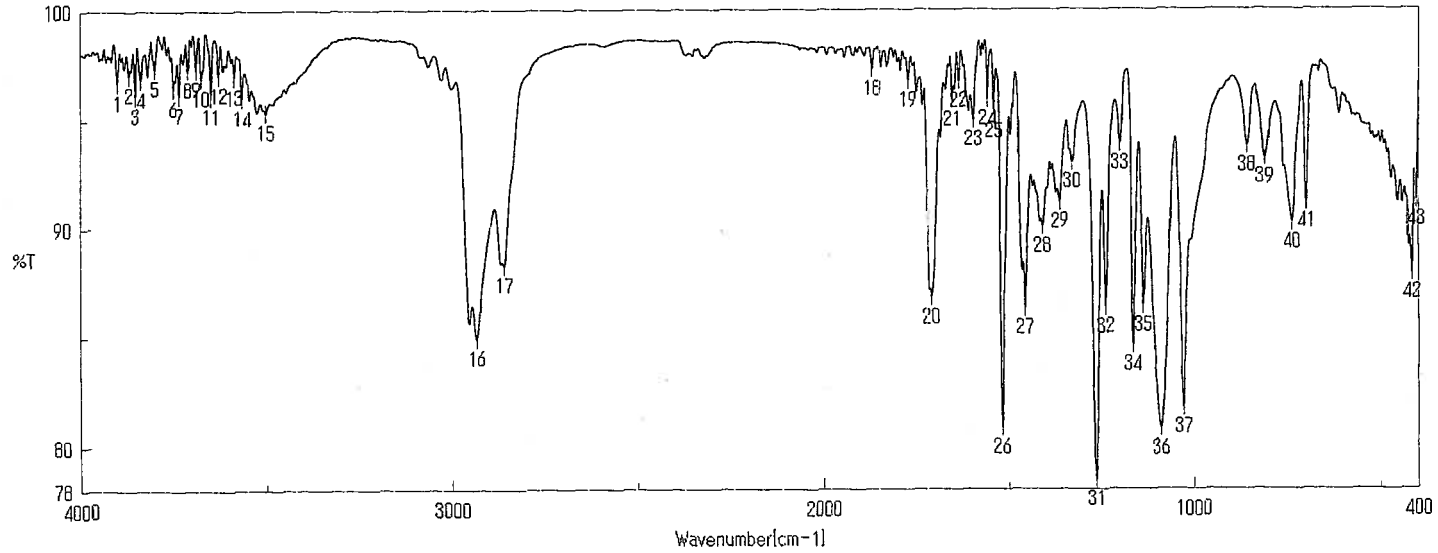

BRUKER

```
Current Data Parameters
NAME          Feb21-2007
EXPNO                 54
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070221
Time              13.41
INSTRUM           dpx400
PROBHD   5 mm BBO 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ             1.9923444 sec
RG                 574.7
DW                60.800 usec
DE                 6.00 usec
TE                303.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  8.10 usec
PL1                 1.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300087 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



BRUKER

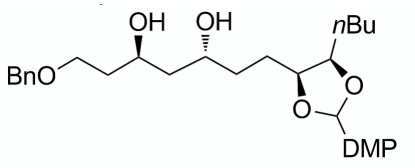
```
Current Data Parameters
NAME      Apr03-2007-hayashi
EXPNO                  30
PROCNO                  1

F2 - Acquisition Parameters
Date_           20070404
Time              1.10
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 40.3
DW                20.800 usec
DE                 6.00 usec
TE                297.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 7.20 usec
PL1               -4.00 dB
SFO1        100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1816007 MHz

F2 - Processing parameters
SI                 32768
SF          100.6253454 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

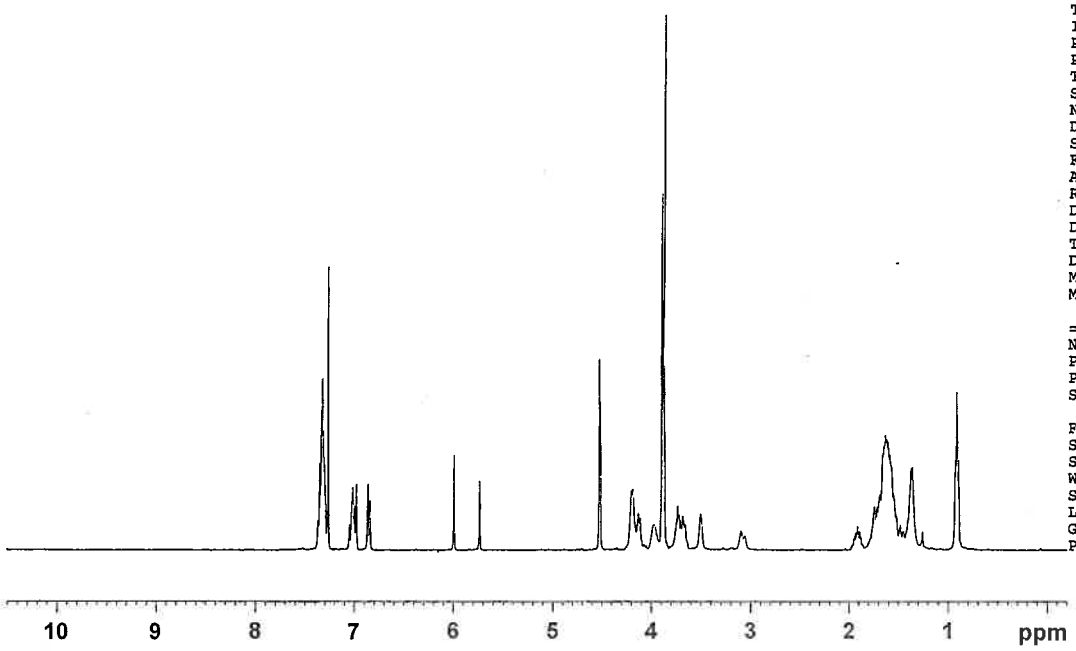



| | |
|---|---|
| 積算回数 | 32 |
| ゼロフィリング | ON |
| ゲイン | 1 |
| 日時 | 107/04/04 20:53 |
| 測定者 | |
| ファイル名 | Memory#9 |
| サンプル名 | buckground |
| コメント | |

| | |
|---|---|
| 分解 | 4 cm-1 |
| アポダイゼーション | Cosine |
| スキャンスピード | 2 mm/sec |

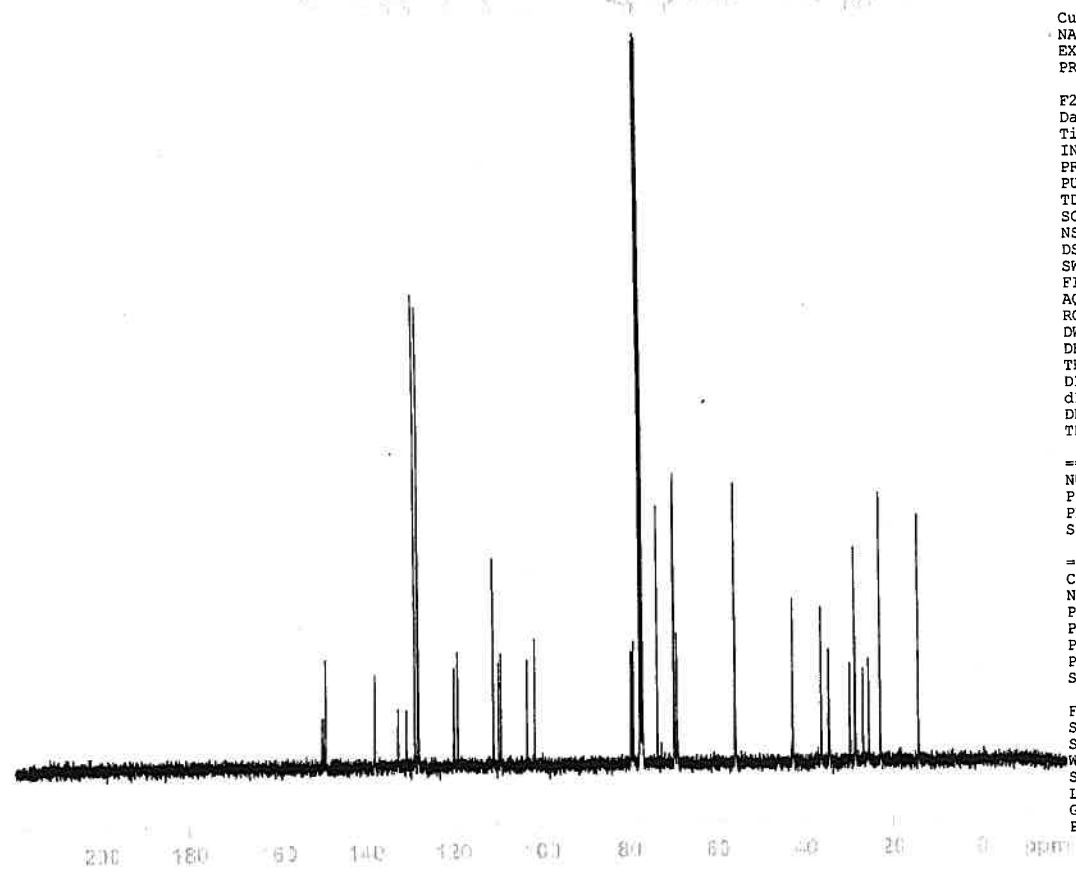
| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1: | 3901.29, | 96.7092 | 2: | 3869.47, | 97.0636 | 3: | 3852.11, | 96.1054 | 4: | 3837.65, | 96.8196 |
| 5: | 3800.04, | 97.3422 | 6: | 3748.94, | 96.4355 | 7: | 3734.48, | 96.1300 | 8: | 3710.37, | 97.2770 |
| 9: | 3688.19, | 97.3335 | 10: | 3674.69, | 96.9004 | 11: | 3647.70, | 96.0800 | 12: | 3627.45, | 97.0468 |
| 13: | 3586.95, | 96.9356 | 14: | 3565.74, | 95.9738 | 15: | 3502.10, | 95.3231 | 16: | 2933.20, | 84.9136 |
| 17: | 2859.92, | 88.2674 | 18: | 1867.72, | 97.2778 | 19: | 1771.30, | 96.7747 | 20: | 1707.66, | 86.7825 |



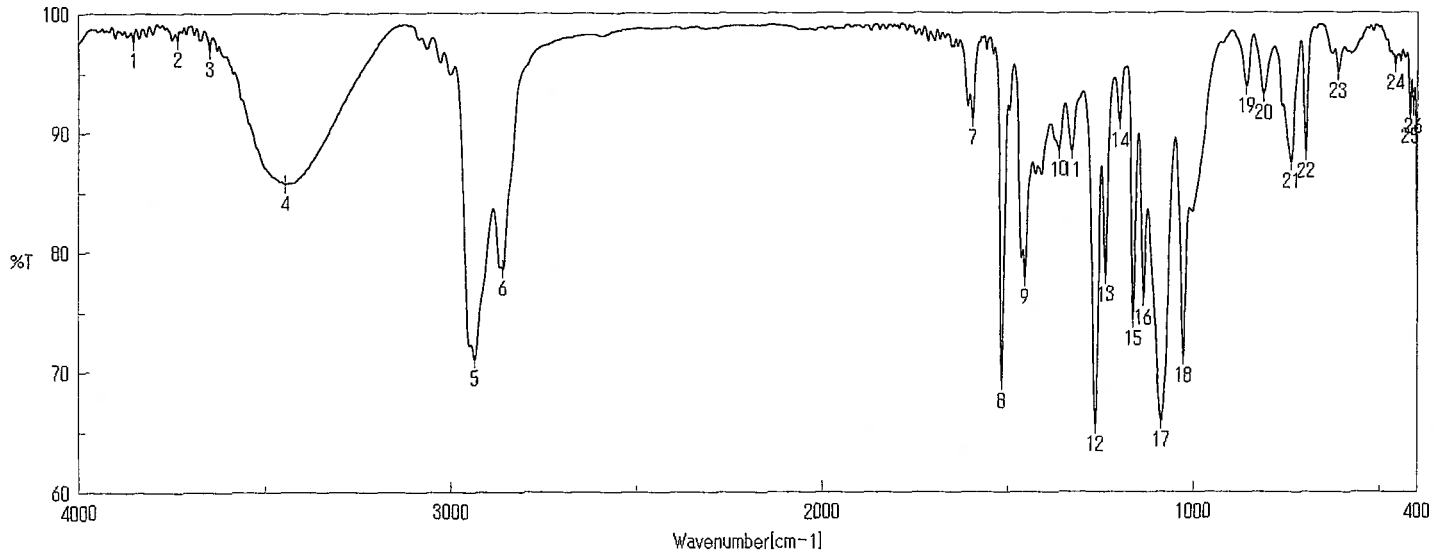

BRUKER

```
Current Data Parameters
NAME          Feb24-2007
EXPNO                 74
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070224
Time               18.54
INSTRUM          dpx400
PROBHD   5 mm BBO 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                  512
DW               60.800 usec
DE                6.00 usec
TE               303.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.10 usec
PL1                1.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                16384
SF          400.1300087 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



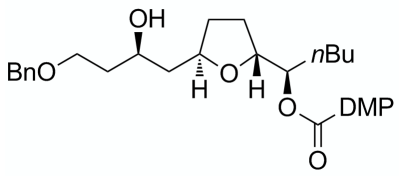

BRUKER

```
Current Data Parameters
NAME      Apr03-2007-hayashi
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070404
Time               2.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  114
DW               20.800 usec
DE                6.00 usec
TE               297.7 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                7.20 usec
PL1               -4.00 dB
SFO1        100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1816007 MHz

F2 - Processing parameters
SI                32768
SF          100.6253481 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/04/09 16:03 | | |
| 測定者 | | | |
| ファイル名 | 2007.4.9 Evans.JWS | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3853.08, | 97.7869 | 2: | 3734.48, | 97.8429 | 3: | 3648.66, | 96.9956 | 4: | 3445.21, | 85.7182 |
| 5: | 2936.09, | 71.0794 | 6: | 2860.88, | 78.5801 | 7: | 1596.77, | 91.1494 | 8: | 1517.70, | 69.1875 |
| 9: | 1455.03, | 77.8000 | 10: | 1363.43, | 88.5372 | 11: | 1328.71, | 88.4543 | 12: | 1264.11, | 65.6226 |
| 13: | 1238.08, | 78.0219 | 14: | 1199.51, | 90.9309 | 15: | 1163.83, | 74.3802 | 16: | 1135.87, | 76.1257 |
| 17: | 1088.62, | 65.9289 | 18: | 1027.87, | 71.2892 | 19: | 858.17, | 93.8641 | 20: | 810.92, | 93.2285 |







BRUKER

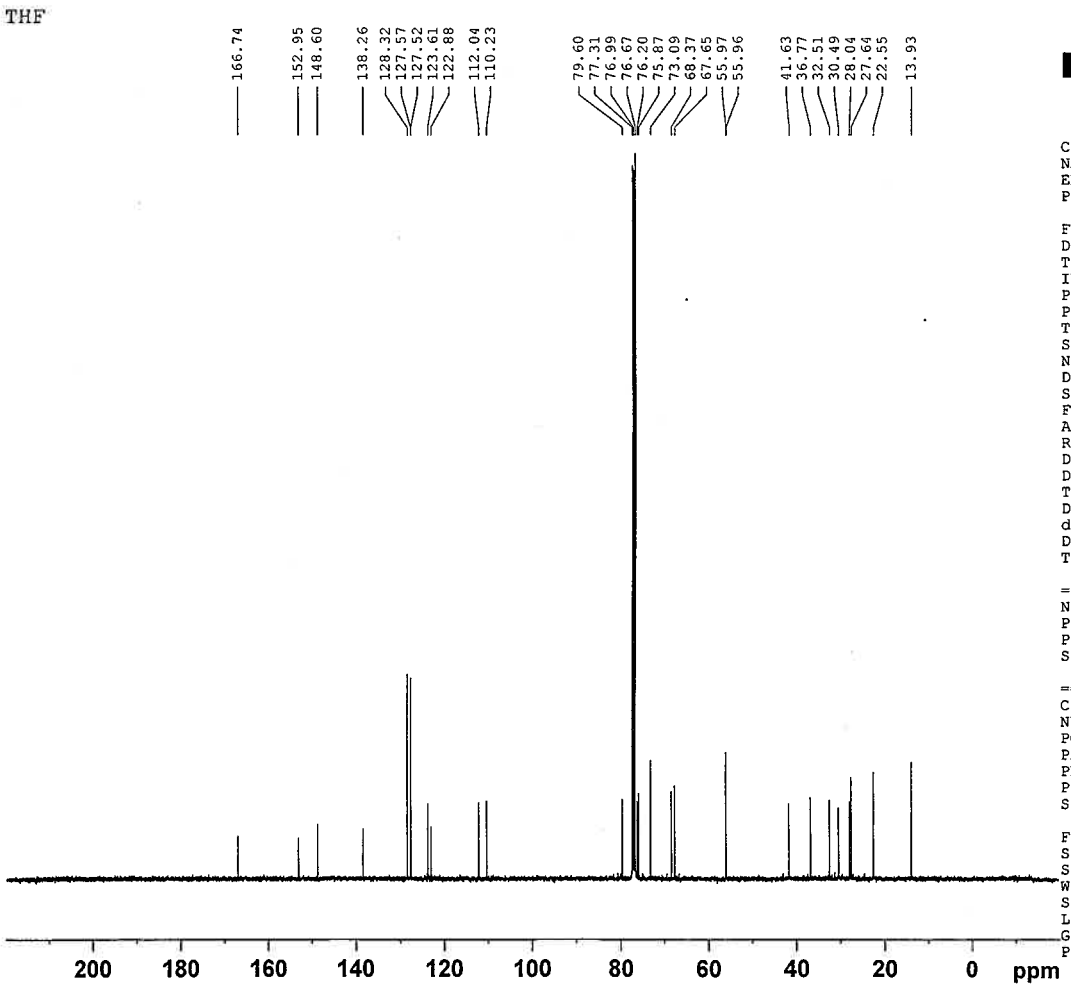
Current Data Parameters
NAME Jun12-2007-hayashi
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070612
Time 3.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 400.1824713 MHz

F2 - Processing parameters
SI 32768
SF 400.1800077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

THF




BRUKER

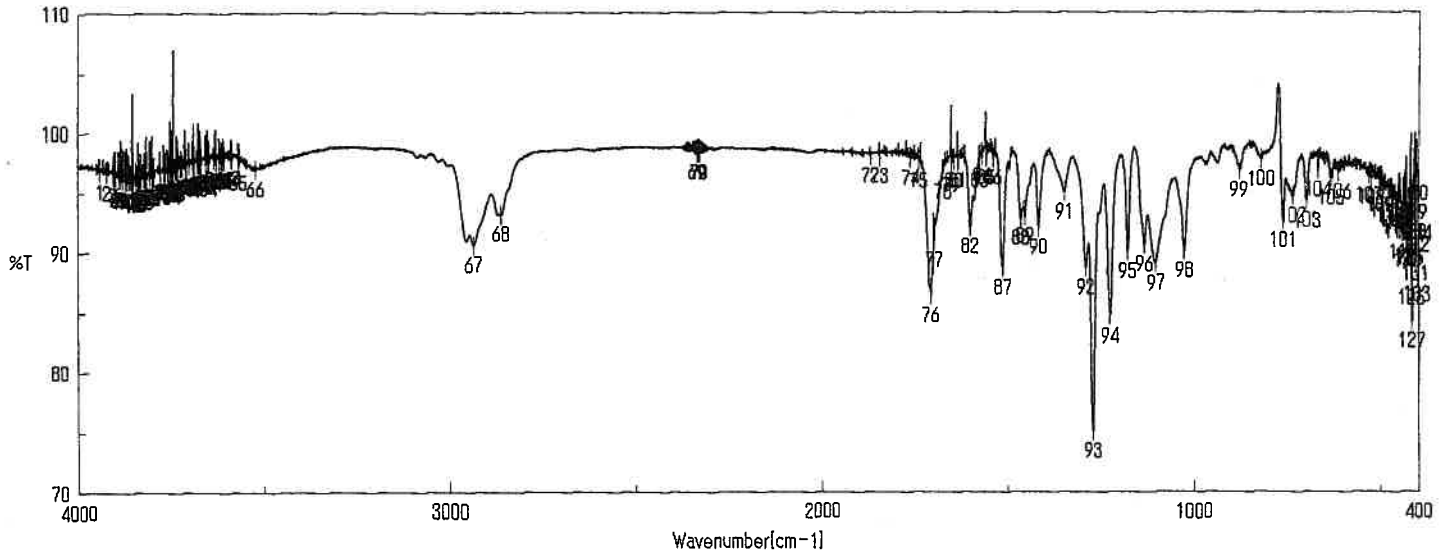
Current Data Parameters
NAME Jun12-2007-hayashi
EXPNO 51
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070612
Time 5.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 80.6
DW 20.800 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 -4.00 dB
SFO1 100.6354036 MHz

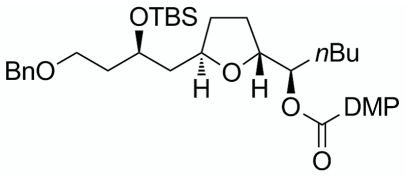
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1816007 MHz

F2 - Processing parameters
SI 32768
SF 100.6253453 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




| | | | |
|---|---|---|---|
| 積算回数 | 16 | 分解 | 1 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 107/06/12 20:31 | | |
| 測定者 | | | |
| ファイル名 | Memory#3 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3941.06, | 96.7123 | 2: | 3922.02, | 96.6176 | 3: | 3904.66, | 96.4678 | 4: | 3902.49, | 95.9946 |
| 5: | 3900.08, | 96.0522 | 6: | 3897.91, | 96.4494 | 7: | 3889.72, | 96.1525 | 8: | 3884.17, | 96.1153 |
| 9: | 3878.63, | 96.0841 | 10: | 3872.36, | 95.9867 | 11: | 3867.78, | 95.9995 | 12: | 3863.20, | 96.0194 |
| 13: | 3860.31, | 96.0812 | 14: | 3855.49, | 95.9451 | 15: | 3852.35, | 95.6835 | 16: | 3850.43, | 95.7734 |
| 17: | 3842.23, | 95.9677 | 18: | 3838.61, | 95.6634 | 19: | 3835.96, | 95.7404 | 20: | 3833.55, | 95.8967 |

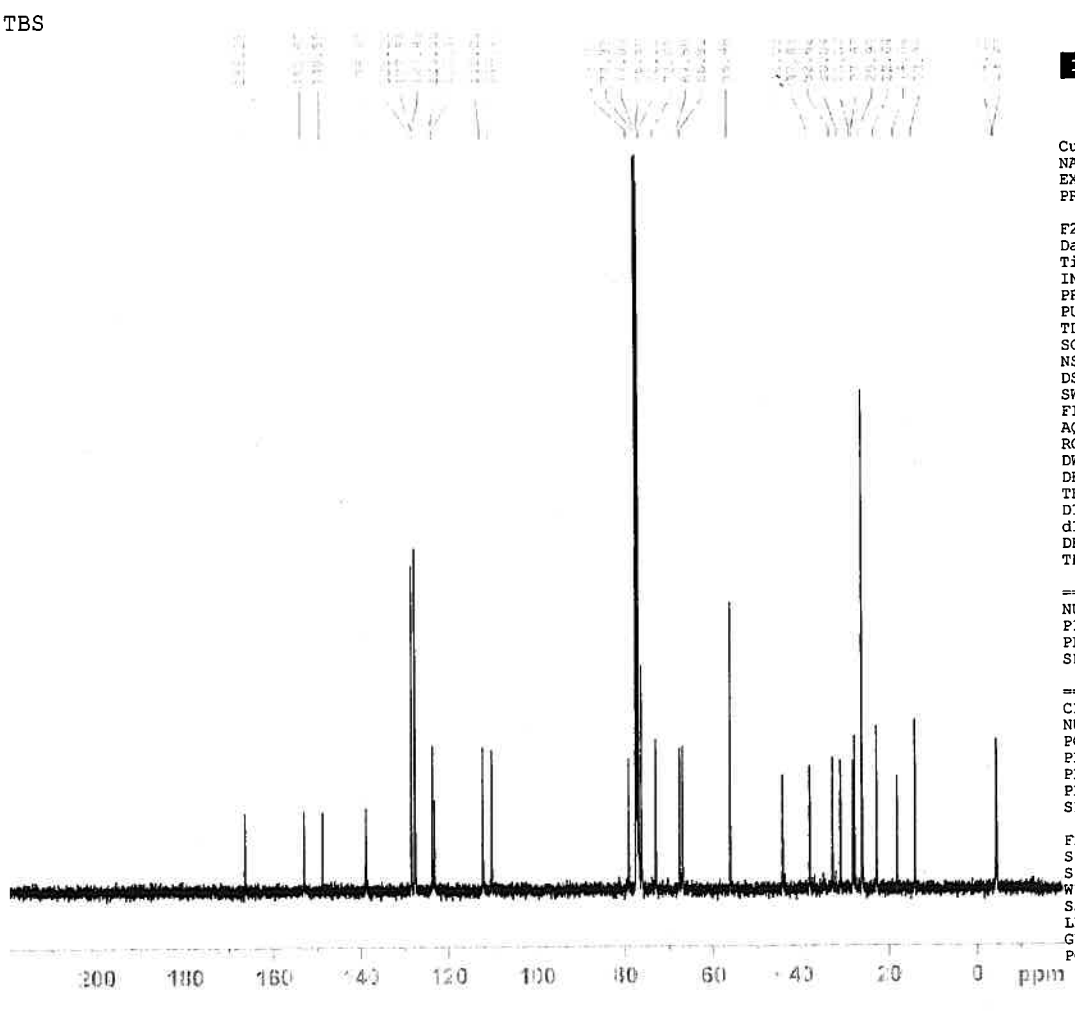




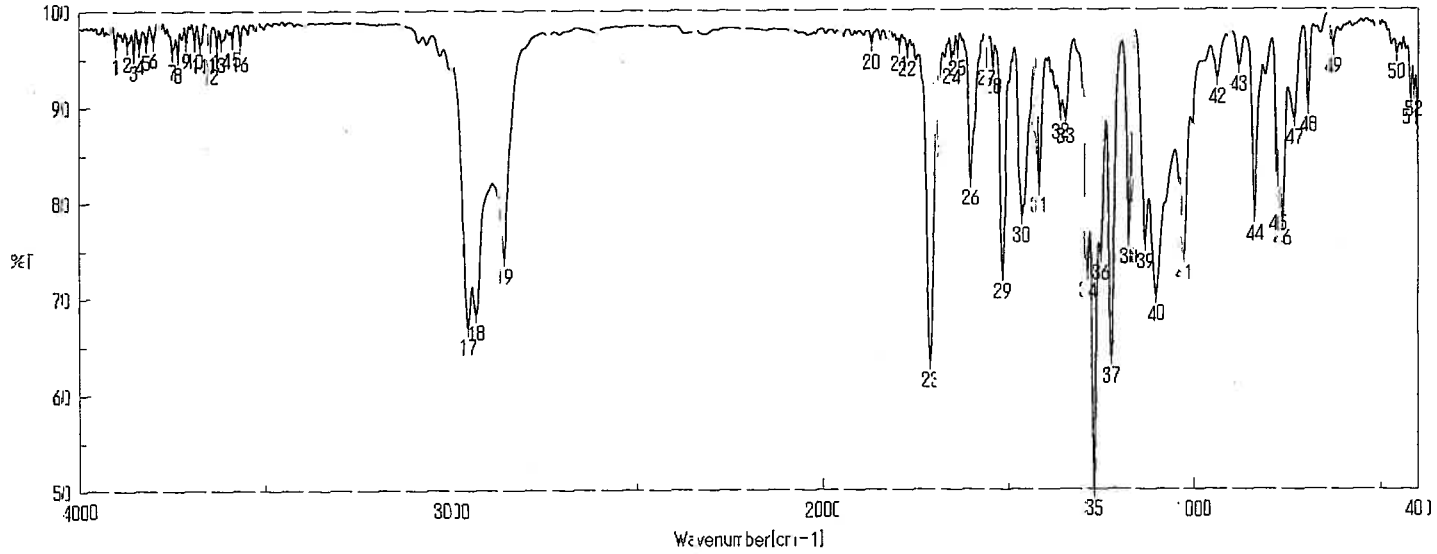


```
Current Data Parameters
NAME                  Apr06-2007
EXPNO                        105
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20070406
Time                       22.26
INSTRUM                  dpx400
PROBHD    5 mm BBO 13C-1
PULPROG                     zg30
TD                         32768
SOLVENT                    CDCl3
NS                             8
DS                             0
SWH                     8223.685 Hz
FIDRES                  0.250967 Hz
AQ                     1.9923444 sec
RG                        1824.6
DW                        60.800 usec
DE                         6.00 usec
TE                        303.2 K
D1                   1.00000000 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                         8.10 usec
PL1                        1.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        16384
SF                  400.1300089 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

TBS

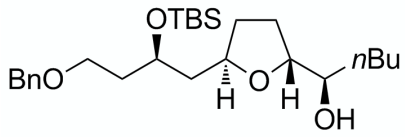



```
Current Data Parameters
NAME            Apr06-2007-hayashi
EXPNO                          50
PROCNO                          1

F2 - Acquisition Parameters
Date_                   20070407
Time                       1.27
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          2048
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                     1.3631988 sec
RG                          90.5
DW                        20.800 usec
DE                         6.00 usec
TE                        297.5 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                         7.20 usec
PL1                       -4.00 dB
SFO1               100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                       -4.00 dB
PL12                      15.00 dB
PL13                      15.00 dB
SFO2               400.1816007 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6253453 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 32 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 07/04/09 16:38 | | |
| 測定者 | | | |
| ファイル名 | 2007.4.9 TBS.JWS | | |
| サンプル名 | Background | | |
| コメント | | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1: | 3902.25, | 96.3163 | 2: | 3870.43, | 96.6095 | 3: | 3853.08, | 95.6243 | 4 | 3838.61, | 96.2707 |
| 5: | 3820.29, | 96.7002 | 6: | 3801.01 | 96.9504 | 7: | 3749.90, | 95.7649 | 8 | 3734.48, | 95.4430 |
| 9: | 3711.33, | 96.8453 | 10: | 3639.16 | 96.8567 | 11: | 3674.69, | 96.3758 | 12 | 3648.66, | 95.2945 |
| 13: | 3628.41, | 96.5042 | 14: | 3617.80 | 96.9040 | 15: | 3586.95, | 97.0250 | 16 | 3566.70, | 96.5394 |
| 17: | 2954.41, | 67.0162 | 18: | 2932.23, | 68.3959 | 19: | 2857.02, | 74.2734 | 20 | 1868.68, | 96.5639 |

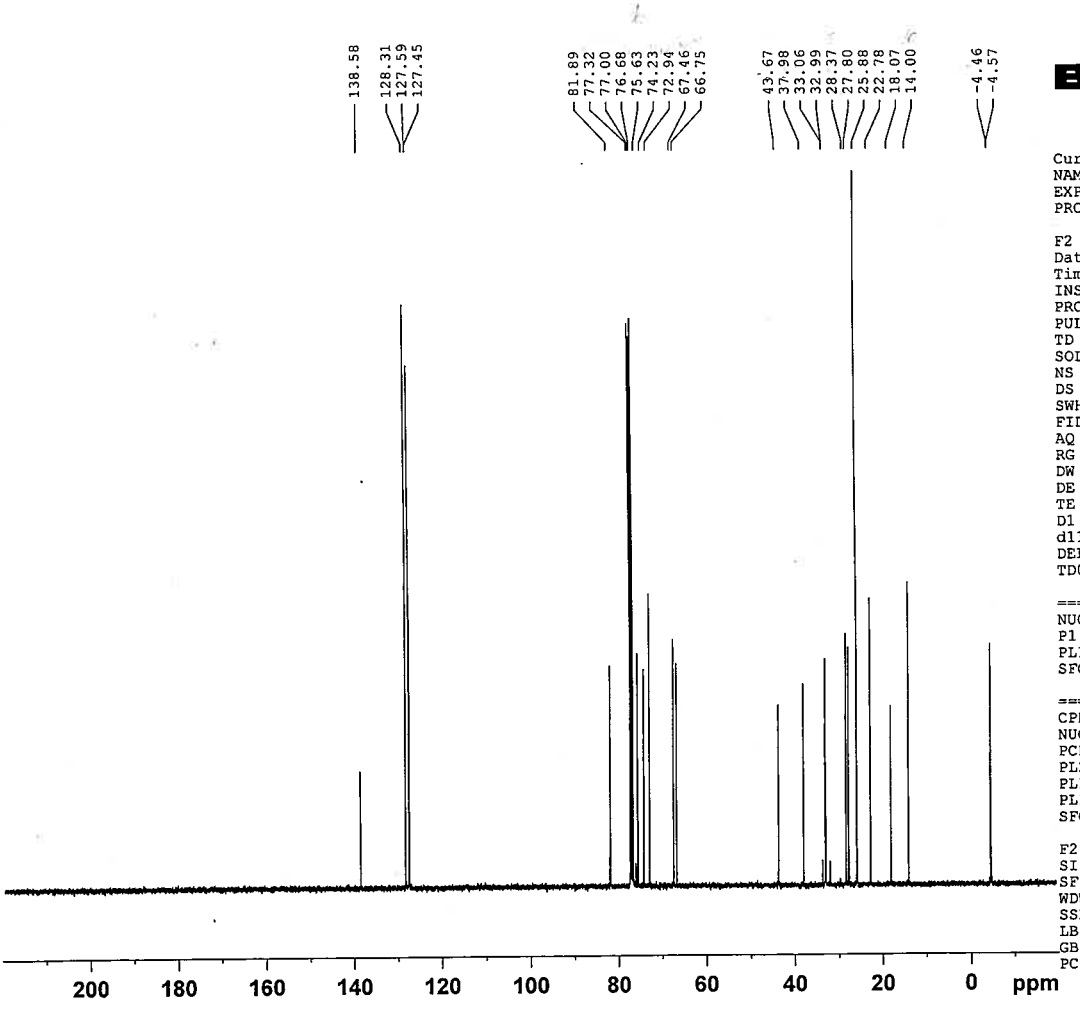






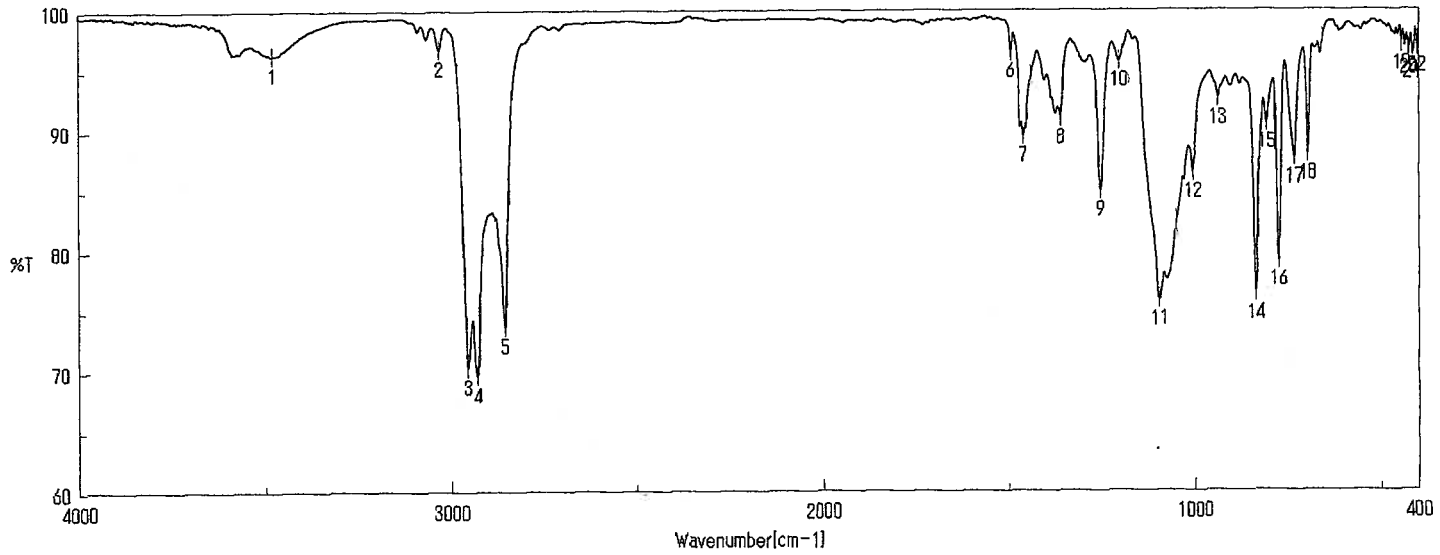
BRUKER

```
Current Data Parameters
NAME      Mar21-2008-hayashi
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080322
Time                   0.46
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     80.6
DW                  60.800 usec
DE                   6.00 usec
TE                   303.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                   12.00 usec
PL1                  -4.00 dB
SFO1          400.1824713 MHz

F2 - Processing parameters
SI                        32768
SF              400.1800074 MHz
WDW                          EM
SSB                           0
LB                     0.30 Hz
GB                            0
PC                         1.00
```



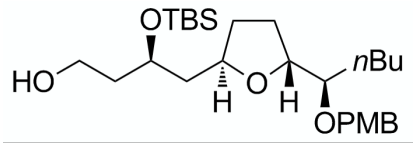

BRUKER

```
Current Data Parameters
NAME      Mar21-2008-hayashi
EXPNO                     11
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080322
Time                   1.46
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                      114
DW                  20.800 usec
DE                   6.00 usec
TE                   302.9 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                    7.20 usec
PL1                  -4.00 dB
SFO1          100.6354036 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2          400.1816007 MHz

F2 - Processing parameters
SI                        32768
SF              100.6253427 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40
```




| | | | |
|---|---|---|---|
| 積算回数 | 16 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 108/03/22 19:46 | | |
| 測定者 | | | |
| ファイル名 | Memory#3 | | |
| サンプル名 | | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3480.88, | 96.3774 | 2: | 3030.59, | 96.8638 | 3: | 2955.38, | 70.3633 | 4: | 2930.31, | 69.6795 |
| 5: | 2857.02, | 73.7201 | 6: | 1496.49, | 96.6006 | 7: | 1462.74, | 89.5422 | 8: | 1361.50, | 91.0526 |
| 9: | 1254.47, | 85.0325 | 10: | 1204.33, | 95.8087 | 11: | 1095.37, | 75.9310 | 12: | 1005.70, | 86.5649 |
| 13: | 938.20, | 92.7177 | 14: | 835.99, | 76.4312 | 15: | 807.06, | 90.6799 | 16: | 775.24, | 79.1928 |
| 17: | 733.78, | 87.7646 | 18: | 697.14, | 88.0590 | 19: | 445.48, | 97.1969 | 20: | 426.19, | 96.8051 |







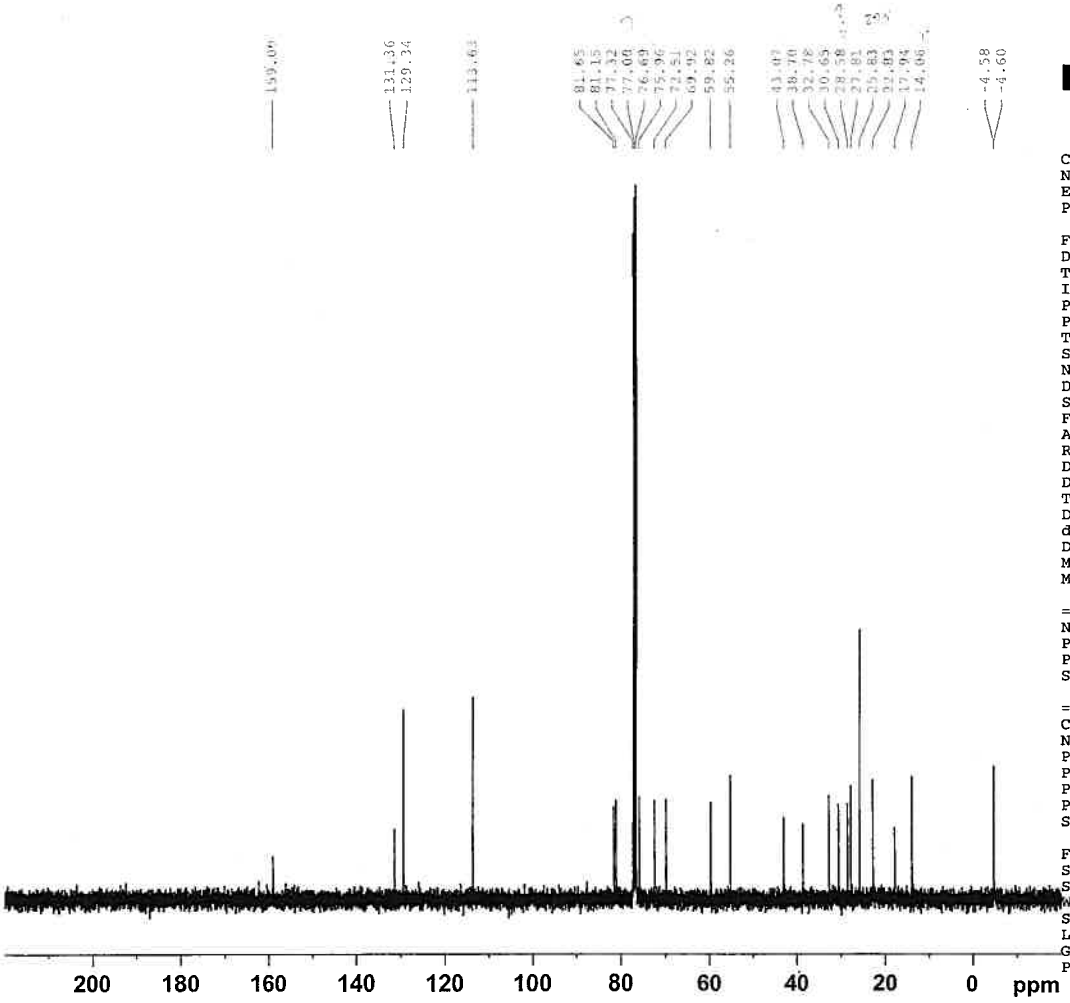
BRUKER

Current Data Parameters
NAME Sep09-2008
EXPNO 121
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080909
Time 19.43
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 1024
DW 60.800 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.70 usec
PL1 4.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300196 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




BRUKER

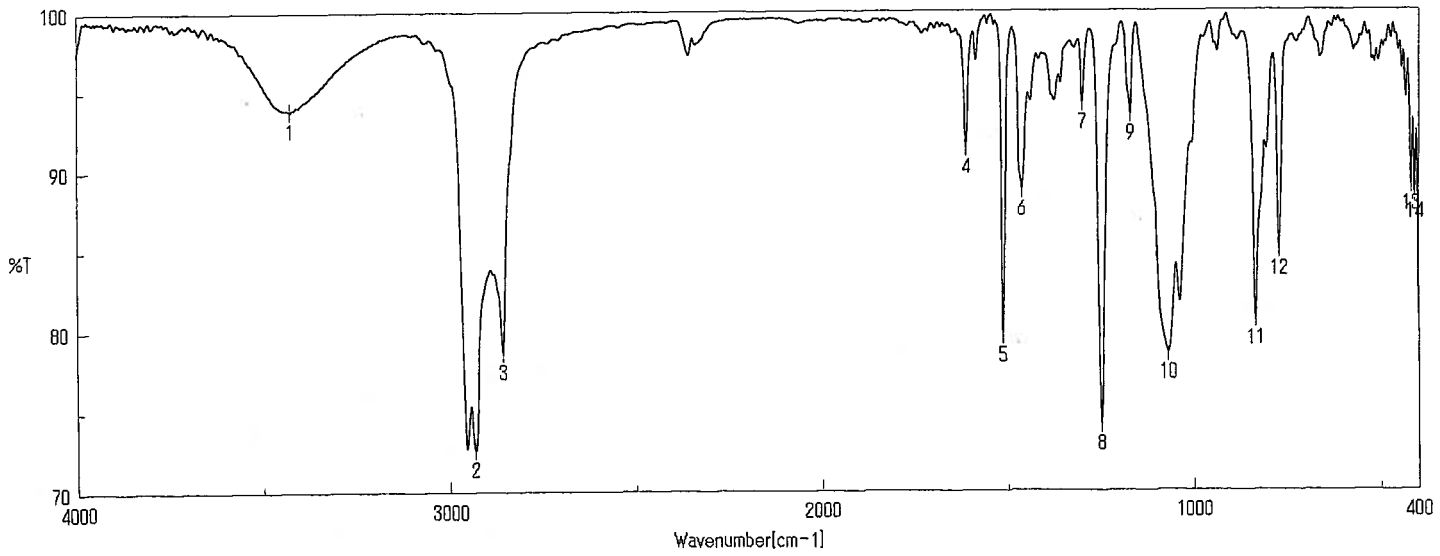
Current Data Parameters
NAME Sep09-2008
EXPNO 122
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080909
Time 19.55
INSTRUM dpx400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 291
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 46341
DW 15.700 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 2.00 dB
SFO1 100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 21.47 dB
PL13 21.47 dB
SFO2 400.1316005 MHz

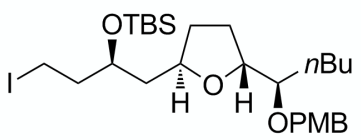
F2 - Processing parameters
SI 32768
SF 100.6127721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




| | | | |
|---|---|---|---|
| 積算回数 | 16 | 分解 | 4 cm-1 |
| ゼロフィリング | ON | アポダイゼーション | Cosine |
| ゲイン | 1 | スキャンスピード | 2 mm/sec |
| 日時 | 108/09/09 21:24 | | |
| 測定者 | | | |
| ファイル名 | Memory#3 | | |
| サンプル名 | buckground | | |
| コメント | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1: | 3427.85, | 93.8146 | 2: | 2932.23, | 72.6525 | 3: | 2857.02, | 78.7324 | 4: | 1613.16, | 91.4240 |
| 5: | 1513.85, | 79.6526 | 6: | 1463.71, | 88.7850 | 7: | 1301.72, | 94.2011 | 8: | 1248.68, | 74.1527 |
| 9: | 1172.51, | 93.4748 | 10: | 1069.33, | 78.5694 | 11: | 835.99, | 80.6734 | 12: | 775.24, | 85.0072 |
| 13: | 419.44, | 89.0080 | 14: | 409.80, | 88.5106 | | | | | | |

8-105re



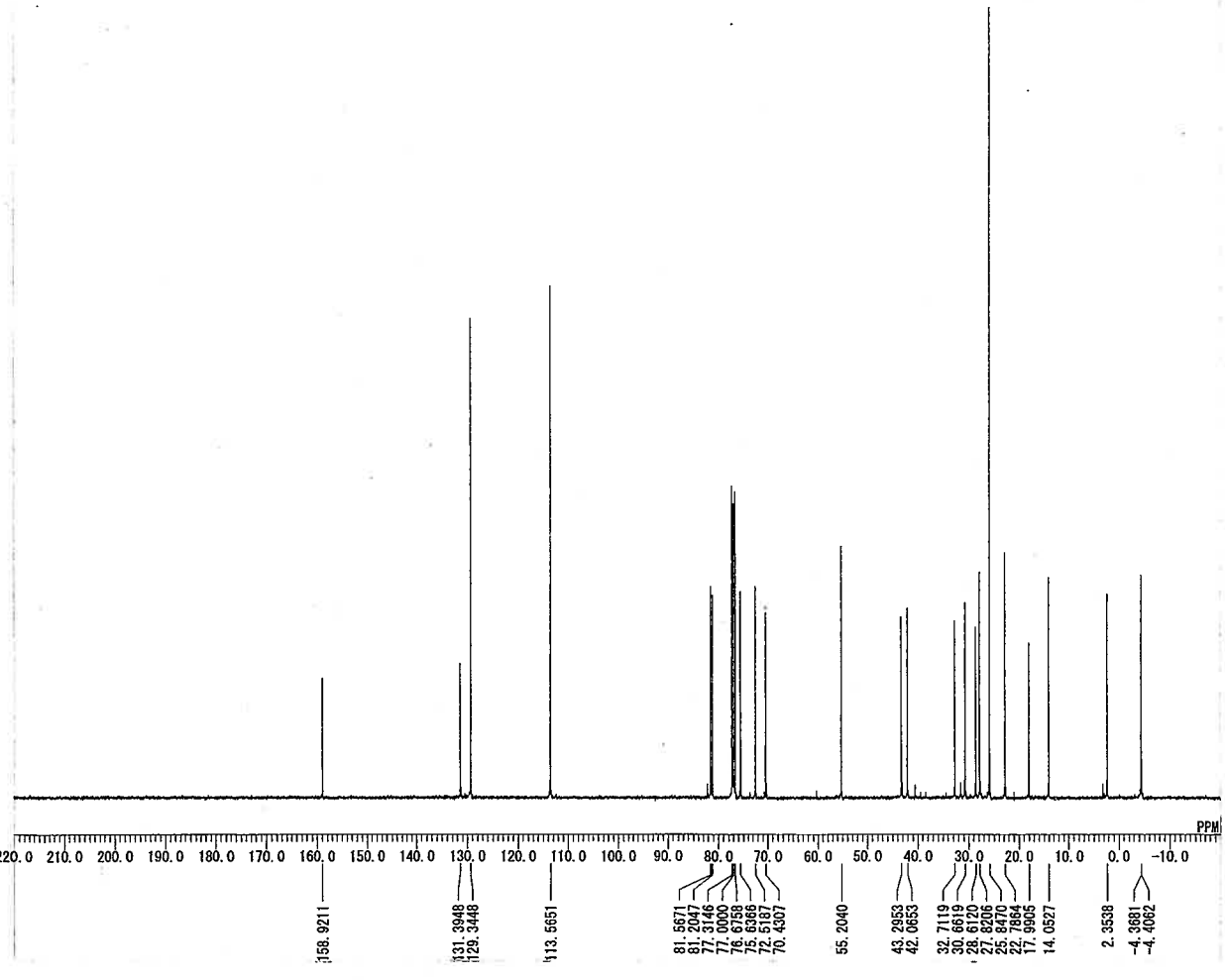

C17-C29 fragment

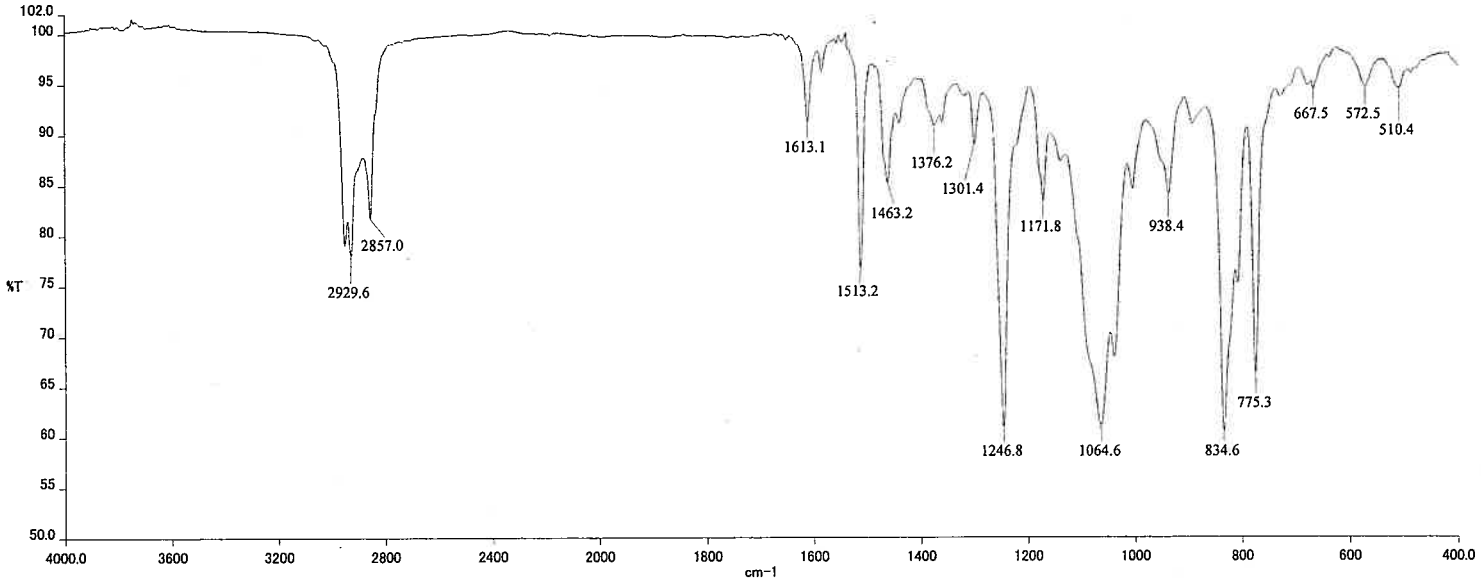
```
DFILE  C:¥ALICE¥DATA¥ochiai8-105re_
COMNT  8-105re
DATIM  20-08-2008 20:20:47
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        399.78 MHz
OBSET          4.19 KHz
OBFIN          7.29 Hz
POINT         32768
FREQU      20032.05 Hz
SCANS            16
ACQTM        1.6358 sec
PD           5.0000 sec
PW1            2.50 usec
IRNUC  1H
CTEMP         22.4 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            44
```




8-105

```
DFILE  C:¥ALICE¥DATA¥ochiai8-105_CA
COMNT  8-105
DATIM  12-09-2008 00:09:51
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        100.53 MHz
OBSET          5.35 KHz
OBFIN          5.86 Hz
POINT         32768
FREQU      31407.03 Hz
SCANS          1000
ACQTM        1.0433 sec
PD           2.0000 sec
PW1            3.00 usec
IRNUC  1H
CTEMP         22.7 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            58
```






——— 8-105.sp

8-105.pk

8-105.sp 3601 4000.0 400.0 60.6 101.5 4.0 %T 16 0.6

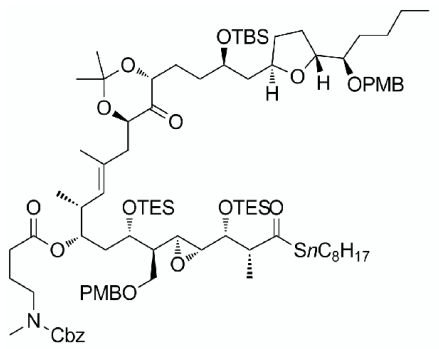
REF 4000 100.2 2000 99.6 600

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 2953.5 | 79.0 | 2929.6 | 78.0 | 2857.0 | 81.7 | 1613.1 | 91.4 | 1586.7 | 96.2 |
| 1513.2 | 76.7 | 1463.2 | 85.2 | 1441.8 | 91.0 | 1376.2 | 90.9 | 1301.4 | 88.9 |
| 1246.8 | 60.9 | 1171.8 | 83.4 | 1139.9 | 87.3 | 1064.6 | 61.1 | 1039.4 | 67.9 |
| 1005.4 | 84.5 | 938.4 | 84.0 | 894.1 | 91.0 | 834.6 | 60.5 | 809.4 | 75.2 |
| 775.3 | 66.2 | 730.4 | 93.6 | 667.5 | 94.3 | 572.5 | 94.5 | 510.4 | 94.5 |

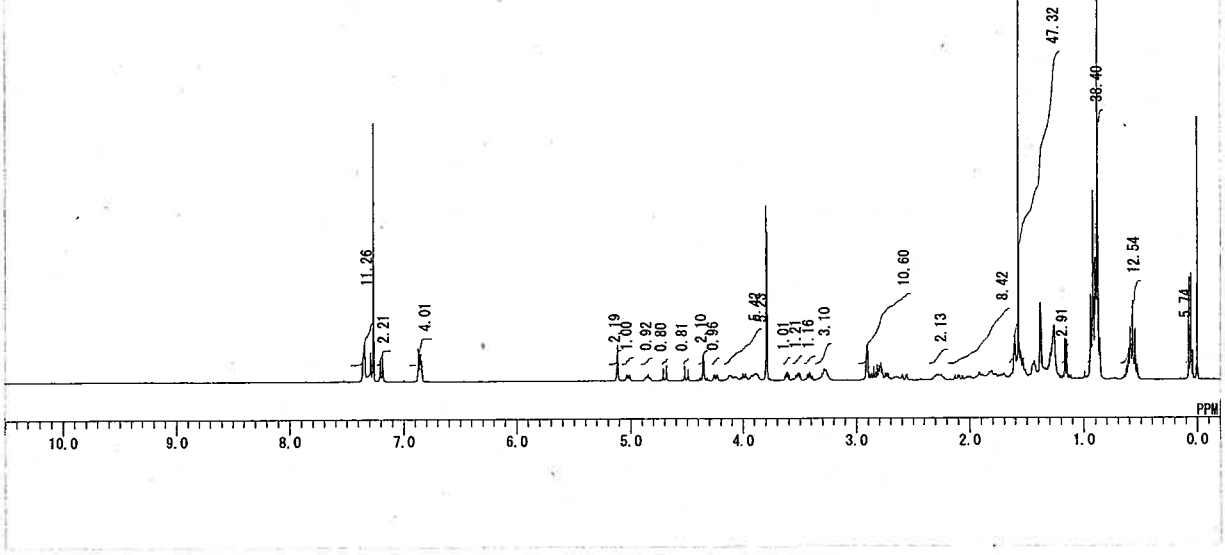
END 25 PEAK(S) FOUND

分光器型式: Spectrum 100
測定日時: 2008年8月20日 16:30 東京 (標準時)
光源: MIR　検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 8-105.sp
スキャン回数: 16
分解能:4.0cm-1
測定方法:ATR(ダイヤモンド/KRS-5)

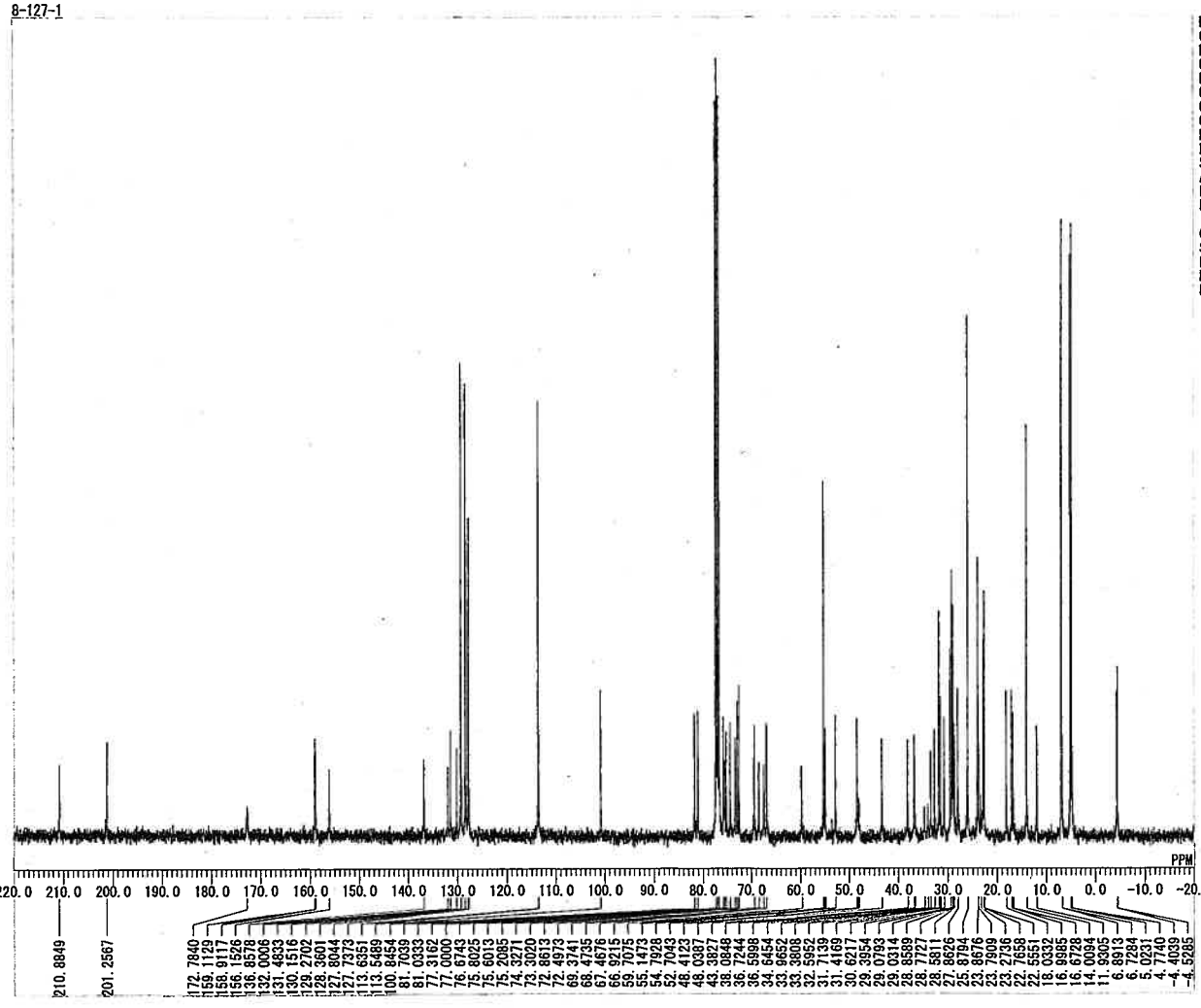
8-127-1re



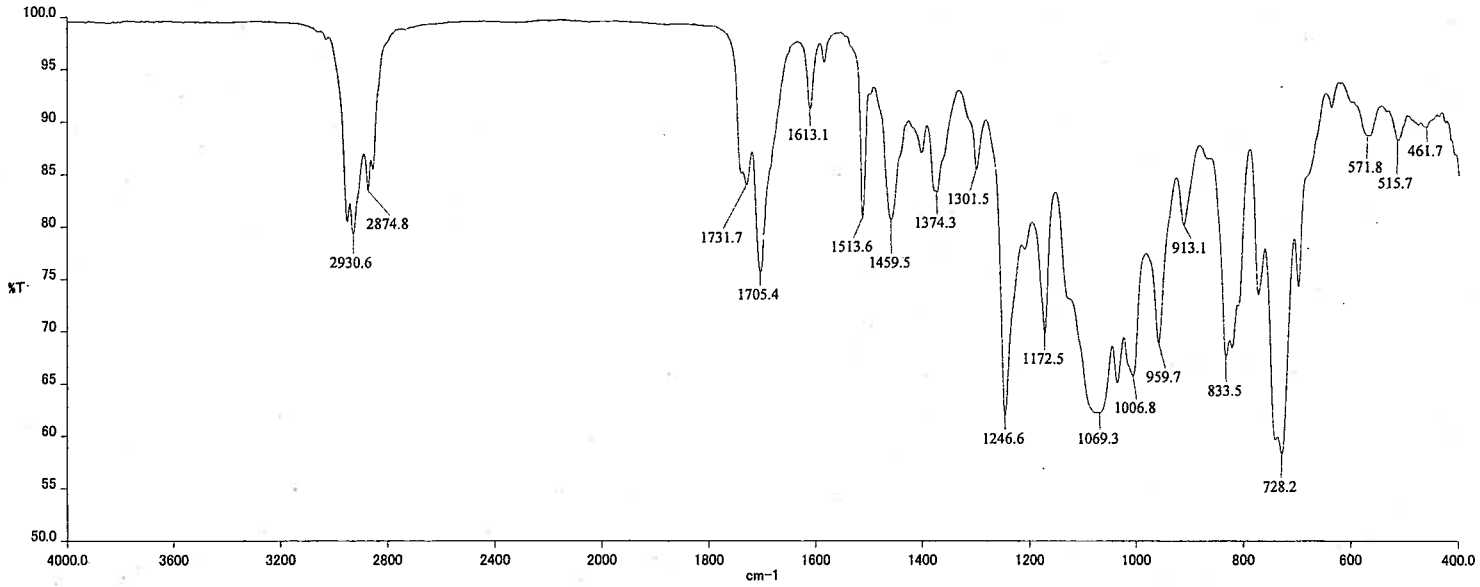

```
DFILE  C:¥ALICE¥DATA¥ochiai8-127-1r
COMNT  8-127-1re
DATIM  05-09-2008 12:50:42
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        399.78 MHz
OBSET          4.19 KHz
OBFIN          7.29 Hz
POINT         32768
FREQU      20032.05 Hz
SCANS            16
ACQTM          1.6358 sec
PD             5.0000 sec
PW1            2.50 usec
IRNUC  1H
CTEMP         21.9 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            44
```




8-127-1




```
DFILE  C:¥ALICE¥DATA¥ochiai8-127-1_
COMNT  8-127-1
DATIM  10-09-2008 12:48:08
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ         99.55 MHz
OBSET          5.13 KHz
OBFIN          0.98 Hz
POINT         32768
FREQU      31250.00 Hz
SCANS          1000
ACQTM          1.0486 sec
PD             2.0000 sec
PW1            3.08 usec
IRNUC  1H
CTEMP         25.4 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            50
```




——— 8-116.sp

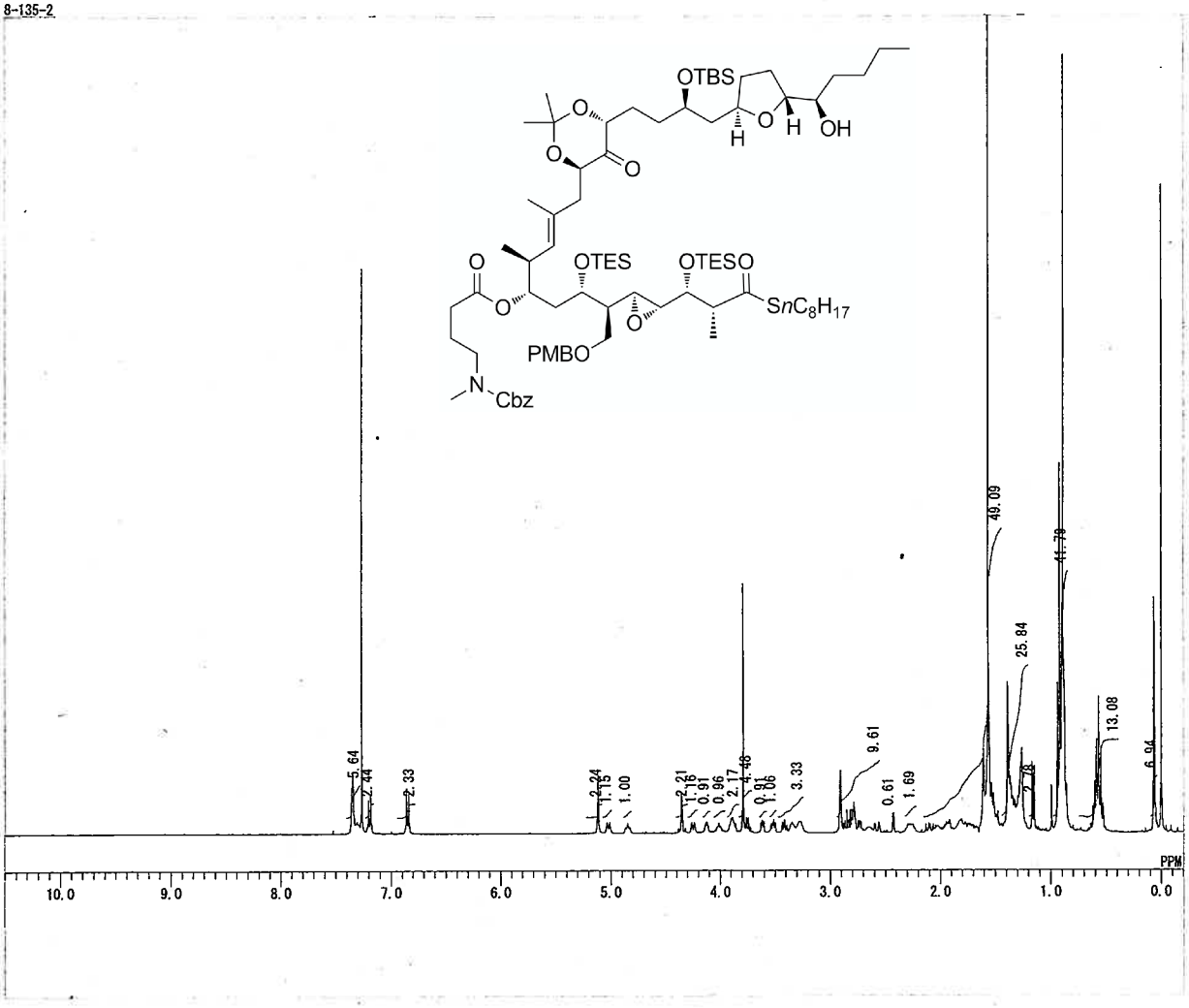
8-116.pk

8-116.sp 3601 4000.0 400.0 58.2 99.7 4.0 %T 16 0.6

REF 4000 99.5 2000 99.5 600
2954.0 80.4 2930.6 79.3 2874.8 83.5 2857.5 85.5 1731.7 83.9
1705.4 75.6 1613.1 91.2 1586.8 95.6 1513.6 80.8 1459.5 80.6
1403.8 87.0 1374.3 83.3 1301.5 85.4 1246.6 61.9 1172.5 69.8
1069.3 62.2 1036.8 65.0 1006.8 65.7 959.7 68.8 913.1 80.1
833.5 67.5 822.1 68.4 773.5 73.5 728.2 58.2 697.7 74.2
637.4 91.3 571.8 88.7 515.7 88.3 461.7 89.5
END 29 PEAK(S) FOUND

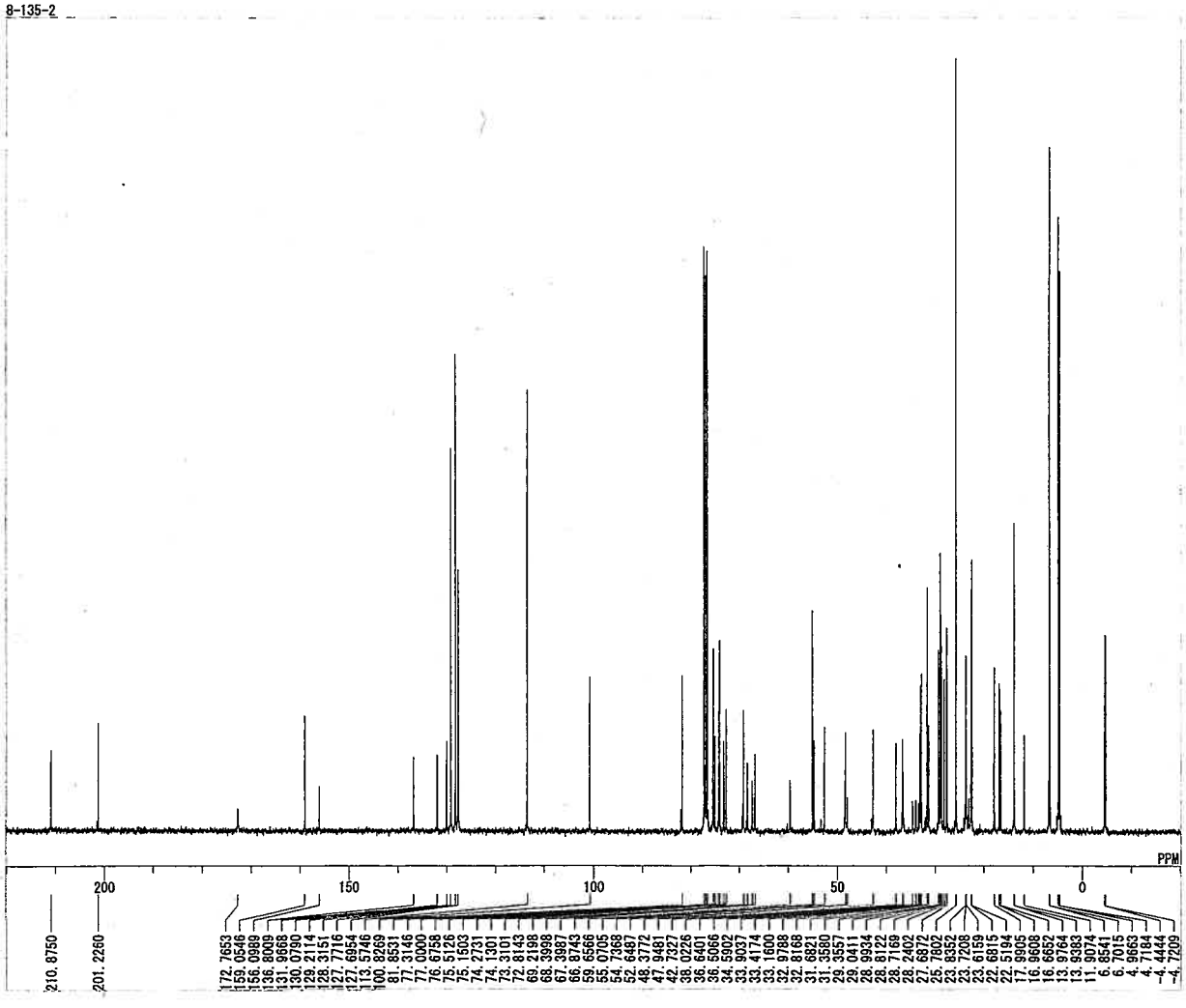
分光器型式: Spectrum 100
測定日時: 2008年8月27日 10:44 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 8-116.sp
スキャン回数: 16
分解能: 4.0cm-1
測定方法: ATR(ダイヤモンド/KRS-5)

8-135-2

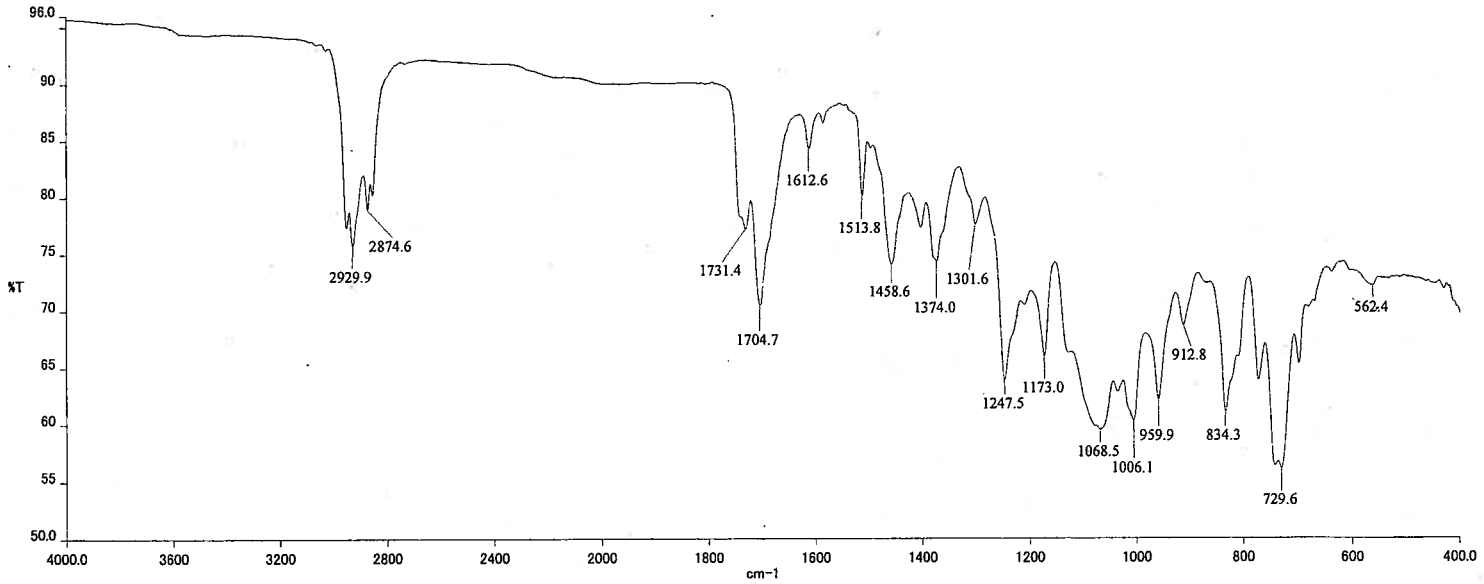



```
DFILE  C:¥ALICE¥DATA¥ochiai8-135-2_
COMNT  8-135-2
DATIM  10-09-2008 19:31:02
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        399.78 MHz
OBSET          4.19 KHz
OBFIN          7.29 Hz
POINT         32768
FREQU      20032.05 Hz
SCANS            16
ACQTM        1.6358 sec
PD           5.0000 sec
PW1          2.50 usec
IRNUC  1H
CTEMP         23.1 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.12 Hz
RGAIN           48
```

8-135-2




```
DFILE  C:¥ALICE¥DATA¥ochiai8-135-2_
COMNT  8-135-2
DATIM  16-09-2008 14:58:18
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        100.53 MHz
OBSET          5.35 KHz
OBFIN          5.86 Hz
POINT         32768
FREQU      31407.03 Hz
SCANS          1000
ACQTM        1.0433 sec
PD           2.0000 sec
PW1          3.00 usec
IRNUC  1H
CTEMP         22.2 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF            1.20 Hz
RGAIN           58
```




——— 8-135②.sp

8-135②.pk

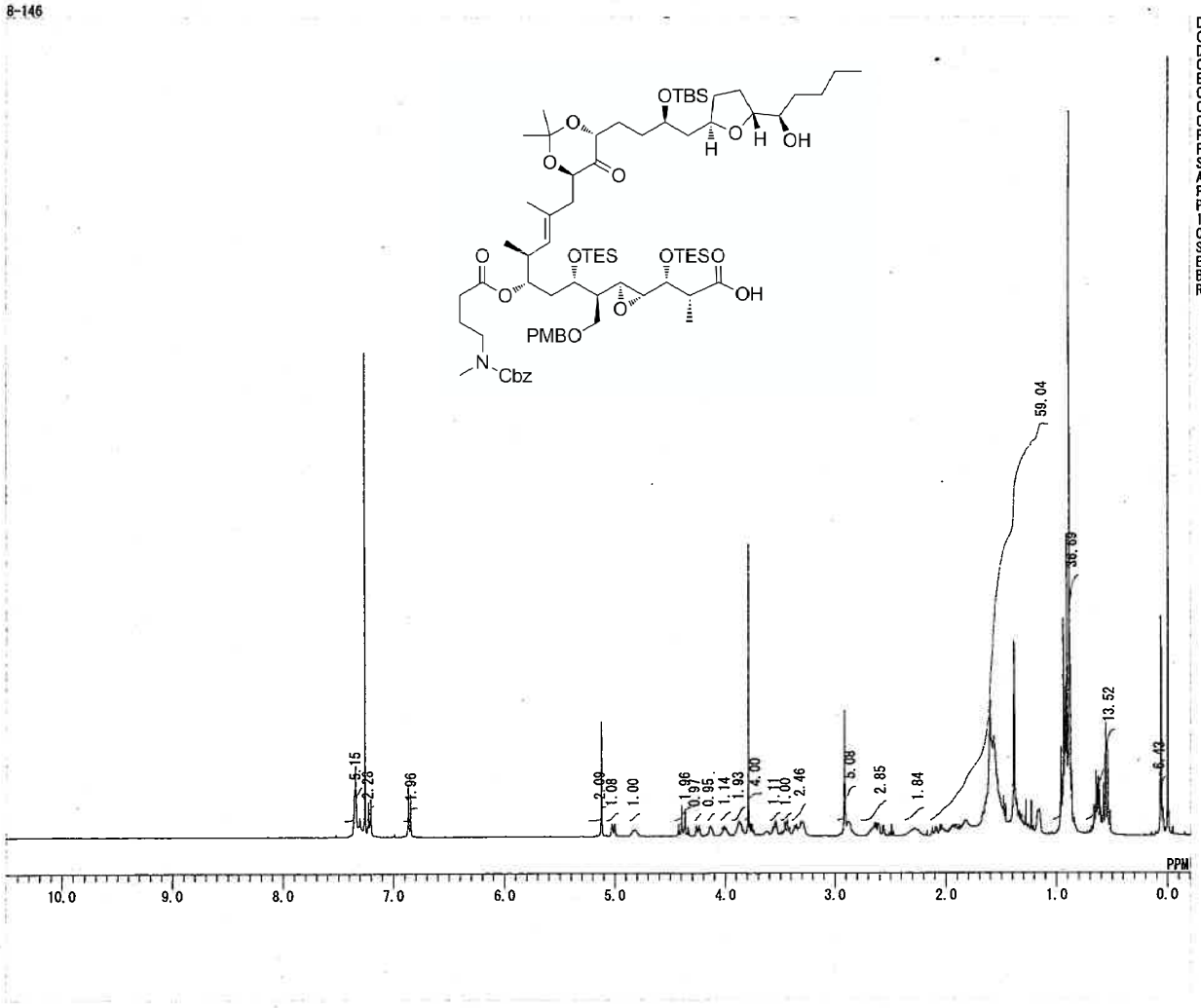
8-135②.sp 3601 4000.0 400.0 56.1 95.7 4.0 %T 16 0.6

REF 4000 95.7 2000 89.9 600
2954.3 77.3 2929.9 75.7 2874.6 78.9 2856.8 80.2 1731.4 77.1
1704.7 70.5 1612.6 84.2 1586.6 86.5 1513.8 80.1 1458.6 74.0
1403.6 77.3 1374.0 74.2 1301.6 77.5 1247.5 63.8 1173.0 65.9
1068.5 59.5 1036.7 62.8 1006.1 60.3 959.9 62.1 912.8 68.6
834.3 61.0 773.4 63.8 729.6 56.1 697.9 65.2 562.4 72.1

END 25 PEAK(S) FOUND

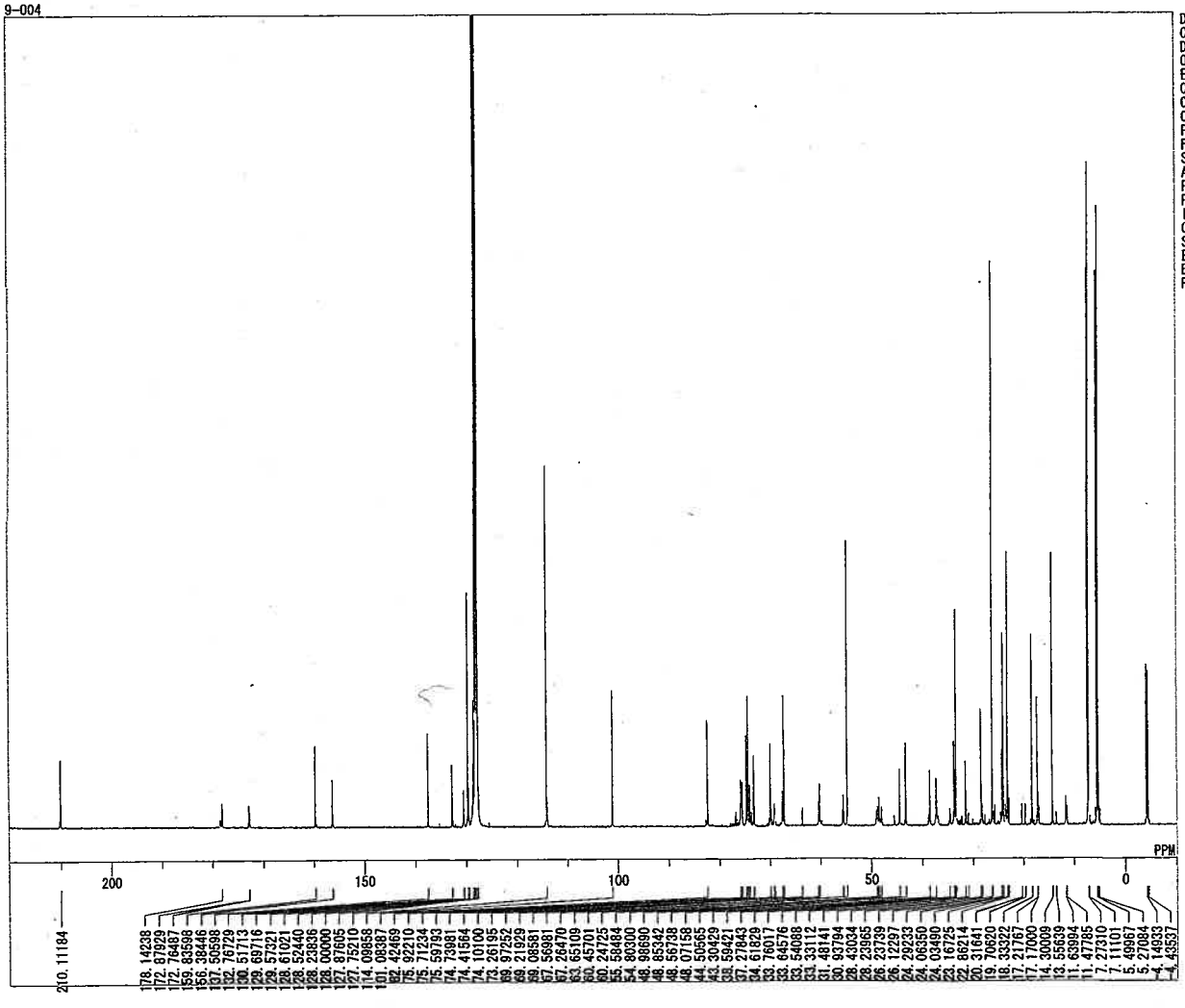
分光器型式: Spectrum 100
測定日時: 2008年9月11日 13:06 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 8-135②.sp
スキャン回数: 16
分解能:4.0cm-1
測定方法:ATR(ダイヤモンド/KRS-5)

8-146

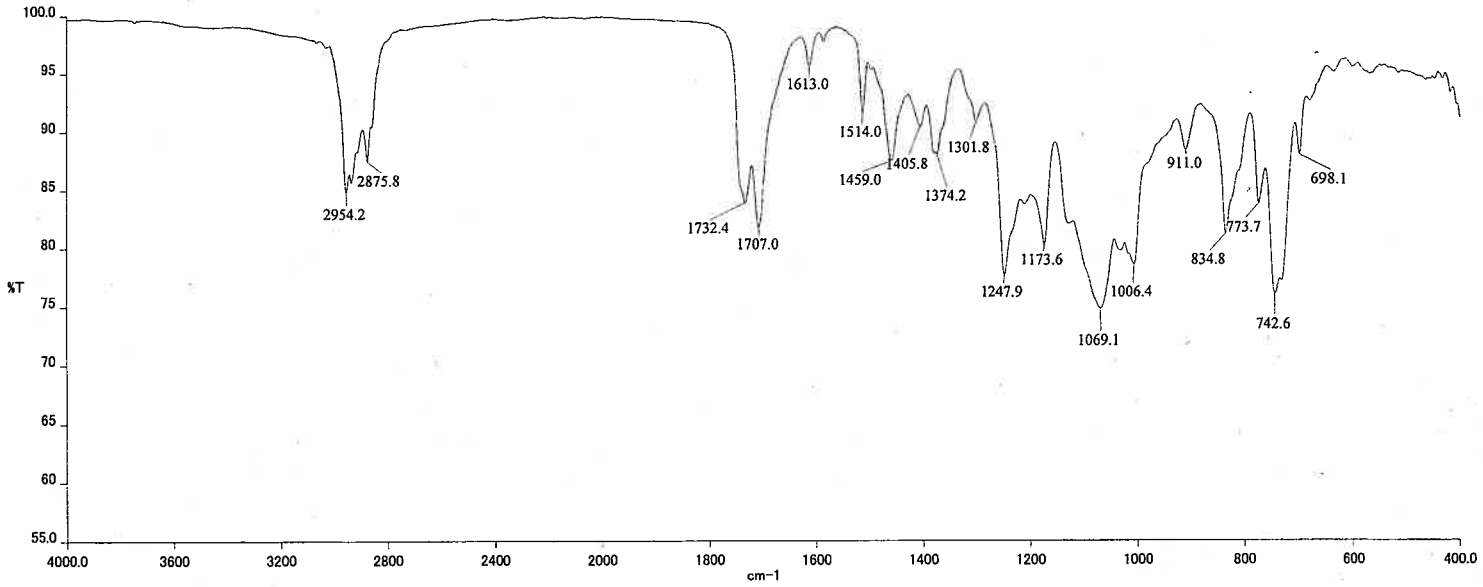



DFILE C:¥ALICE¥DATA¥ochiai8-146_PR
COMNT 8-146
DATIM 17-09-2008 20:10:29
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 32768
FREQU 19841.27 Hz
SCANS 64
ACQTM 1.6515 sec
PD 5.0000 sec
PW1 4.56 usec
IRNUC 1H
CTEMP 25.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

9-004




DFILE C:¥ALICE¥DATA¥ochiai9-004_CA
COMNT 9-004
DATIM 09-11-2008 19:41:40
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32768
FREQU 31407.03 Hz
SCANS 10000
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.00 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT C6D6
EXREF 128.00 ppm
BF 1.20 Hz
RGAIN 52




——— 9-004.sp

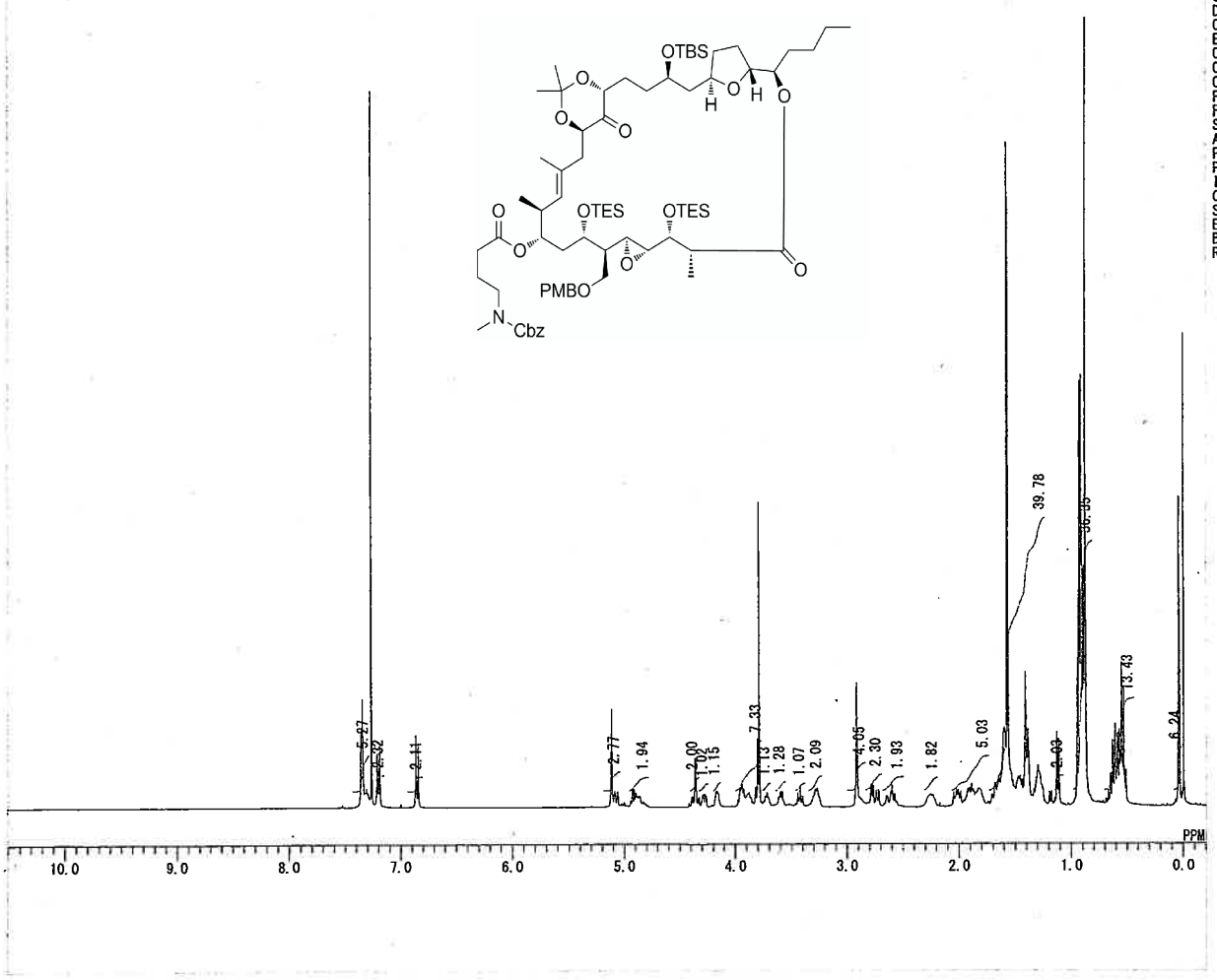
9-004.pk

9-004.sp 3601 4000.0 400.0 74.7 99.8 4.0 %T 16 1.2

REF 4000 99.6 2000 99.8 600
2954.2 84.8 2875.8 87.4 1732.4 83.9 1707.0 81.8 1613.0 95.7
1514.0 91.5 1459.0 87.4 1405.8 90.4 1374.2 88.0 1301.8 90.7
1247.9 77.5 1173.6 80.1 1069.1 74.7 1006.4 78.5 911.0 88.3
834.8 81.1 773.7 83.7 742.6 75.9 698.1 87.9
END 19 PEAK(S) FOUND

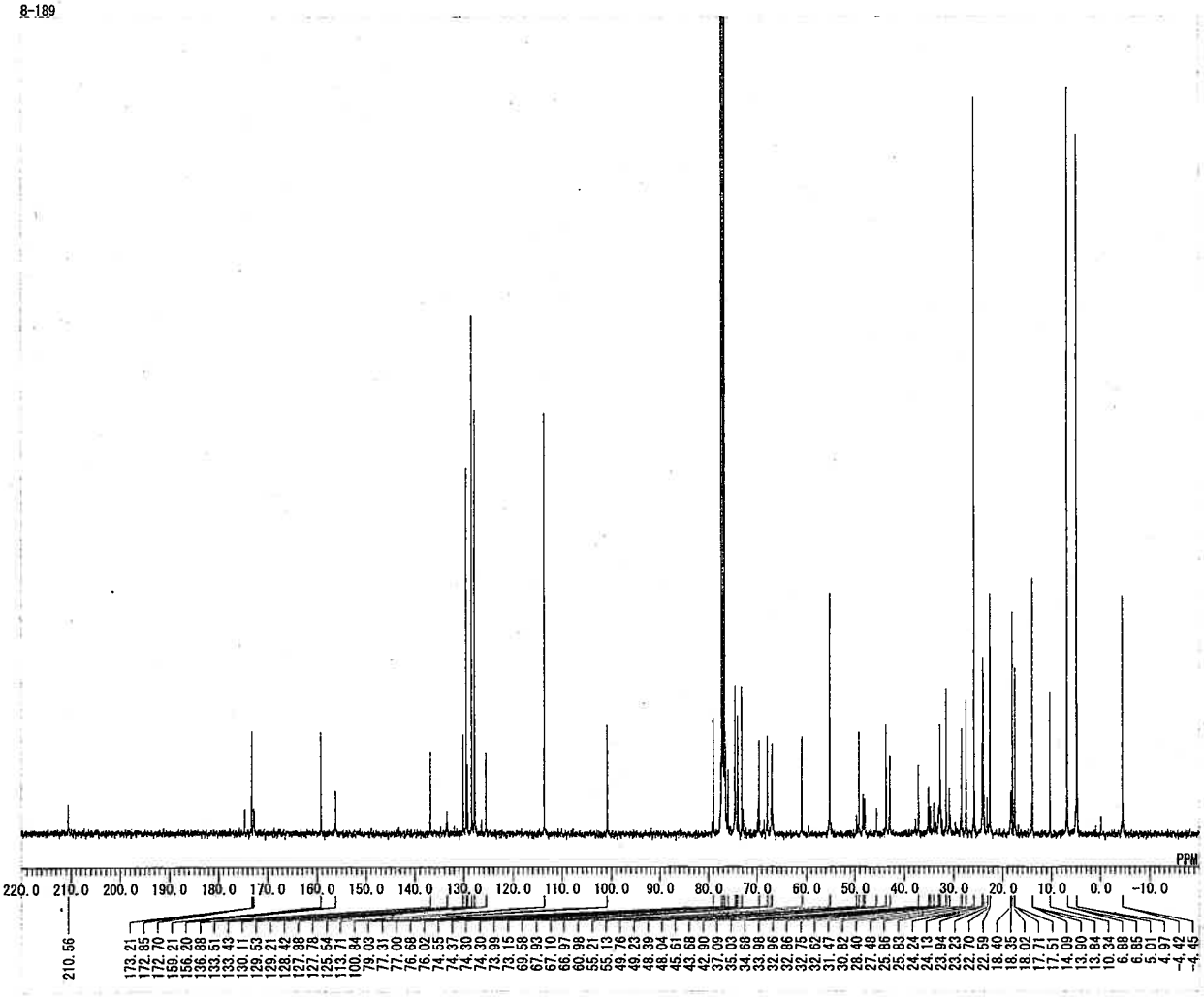
分光器型式: Spectrum 100
測定日時: 2008年11月6日 16:11 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 9-004.sp
スキャン回数: 16
分解能: 4.0cm-1
測定方法: ATR(ダイヤモンド/KRS-5)

8-189re

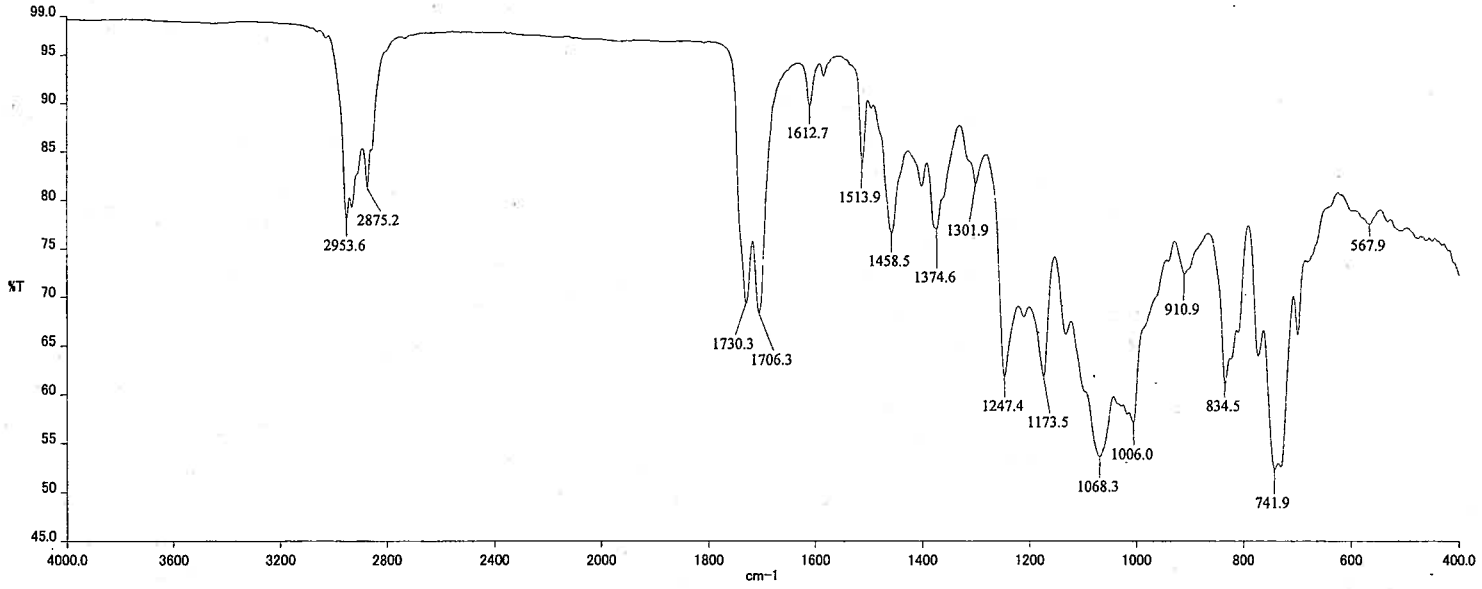



```
DFILE  C:¥ALICE¥DATA¥ochiai8-189re_
COMNT  8-189re
DATIM  17-10-2008 17:24:37
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        399.78 MHz
OBSET          4.19 KHz
OBFIN          7.29 Hz
POINT         32768
FREQU      20032.05 Hz
SCANS            64
ACQTM         1.6358 sec
PD            5.0000 sec
PW1           5.65 usec
IRNUC  1H
CTEMP         23.4 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.12 Hz
RGAIN           38
```

8-189




```
DFILE  C:¥ALICE¥DATA¥ochiai8-189_CA
COMNT  8-189
DATIM  21-10-2008 07:24:35
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        100.53 MHz
OBSET          5.35 KHz
OBFIN          5.86 Hz
POINT         32768
FREQU      31407.03 Hz
SCANS           12000
ACQTM         1.0433 sec
PD            2.0000 sec
PW1           3.00 usec
IRNUC  1H
CTEMP         23.5 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           58
```




——— 8-189.sp

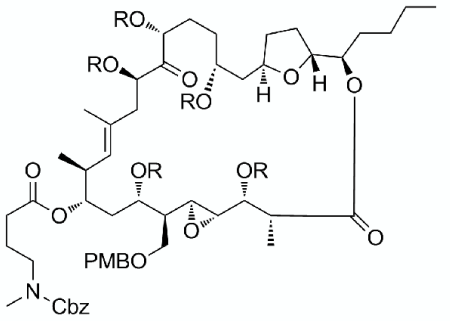
8-189.pk

8-189.sp 3601 4000.0 400.0 52.3 98.6 4.0 %T 16 0.6

REF 4000 98.6 2000 96.5 600
2953.6 78.1 2934.6 79.2 2875.2 81.2 1730.3 69.3 1706.3 68.3
1612.7 89.7 1586.7 92.7 1513.9 83.7 1458.5 76.6 1403.1 81.4
1374.6 77.0 1301.9 81.6 1247.4 61.8 1211.0 67.9 1173.5 61.8
1132.1 66.1 1068.3 53.6 1006.0 57.1 910.9 72.3 834.5 61.1
772.1 63.9 741.9 52.3 698.0 66.1 567.9 77.5
END 24 PEAK(S) FOUND

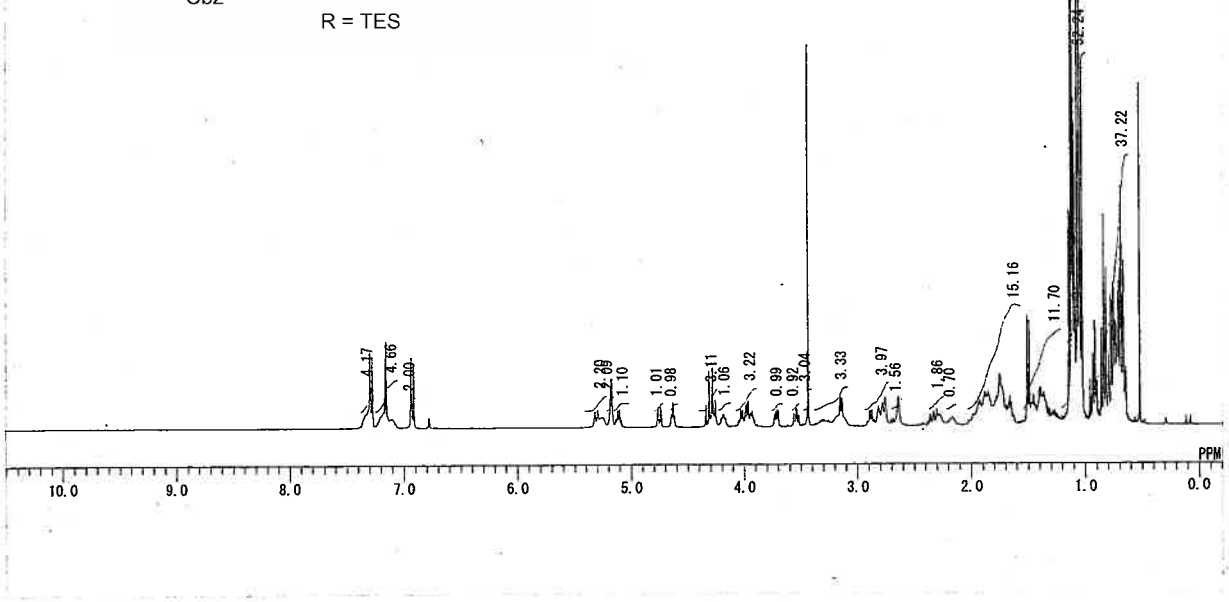
分光器型式: Spectrum 100
測定日時: 2008年10月20日 10:15 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 8-189.sp
スキャン回数: 16
分解能: 4.0cm-1
測定方法: ATR(ダイヤモンド/KRS-5)

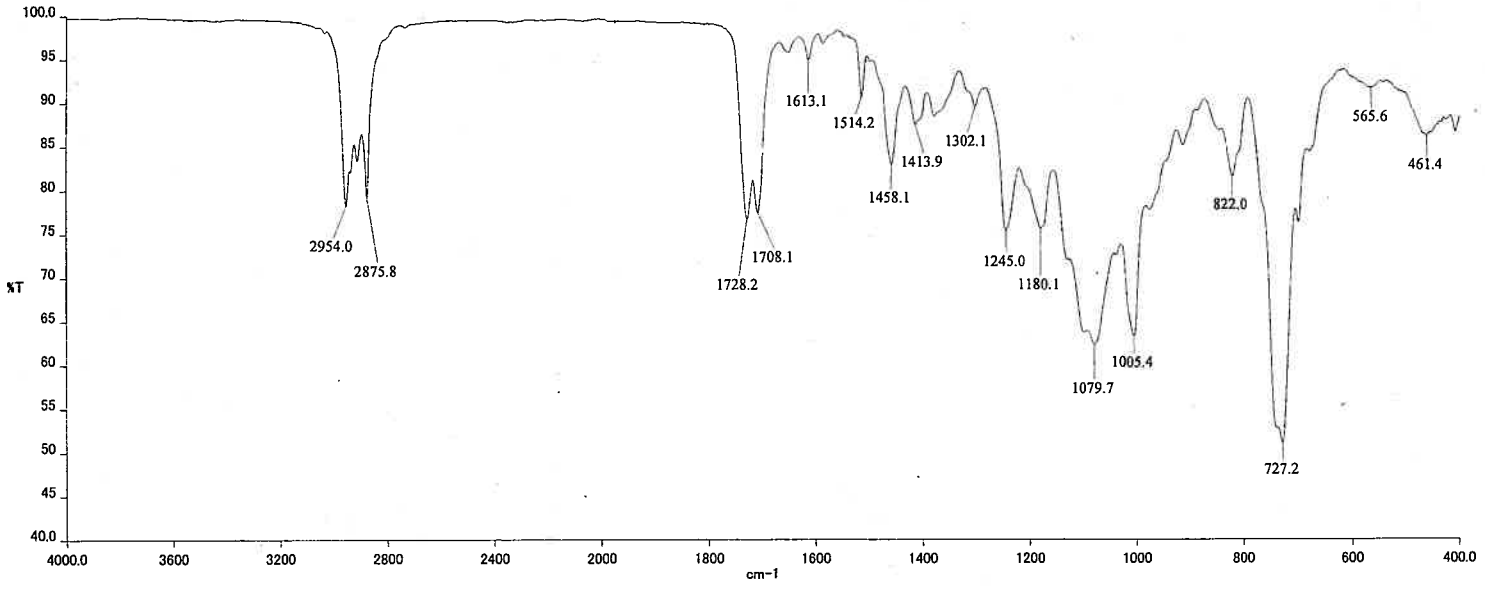
8-206-1




R = TES

```
DFILE  C:¥ALICE¥DATA¥ochiai8-206-1_
COMNT  8-206-1
DATIM  30-10-2008 18:12:25
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        399.78 MHz
OBSET          4.19 KHz
OBFIN          7.29 Hz
POINT         32768
FREQU      20032.05 Hz
SCANS            64
ACQTM        1.6358 sec
PD           5.0000 sec
PW1             5.65 usec
IRNUC  1H
CTEMP          23.3 c
SLVNT  C6D6
EXREF          7.16 ppm
BF             0.12 Hz
RGAIN            36
```




——— 8-206①.sp

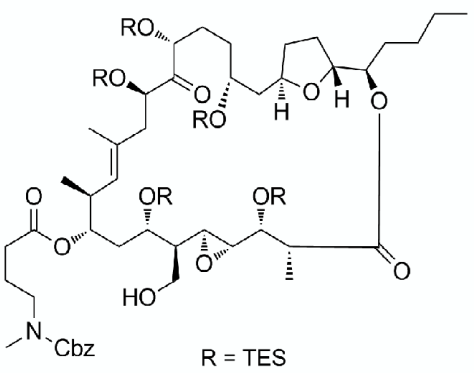
8-206①.pk

8-206①.sp 3601 4000.0 400.0 51.0 99.8 4.0 %T 16 0.8

REF 4000 99.7 2000 99.5 600
2954.0 78.2 2911.6 83.4 2875.8 79.2 1728.2 76.7 1708.1 77.4
1650.0 95.8 1613.1 95.0 1586.6 96.8 1514.2 90.7 1458.1 83.1
1413.9 87.6 1378.0 88.5 1302.1 89.6 1245.0 75.5 1180.1 75.7
1079.7 62.4 1005.4 63.3 913.6 85.2 822.0 81.6 727.2 51.0
698.0 76.4 565.6 91.7 461.4 86.2 407.5 86.7
END 24 PEAK(S) FOUND

分光器型式: Spectrum 100
測定日時: 2008年11月4日 10:47 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 8-206①.sp
スキャン回数: 16
分解能:4.0cm-1
測定方法:ATR(ダイヤモンド/KRS-5)

8-206-2



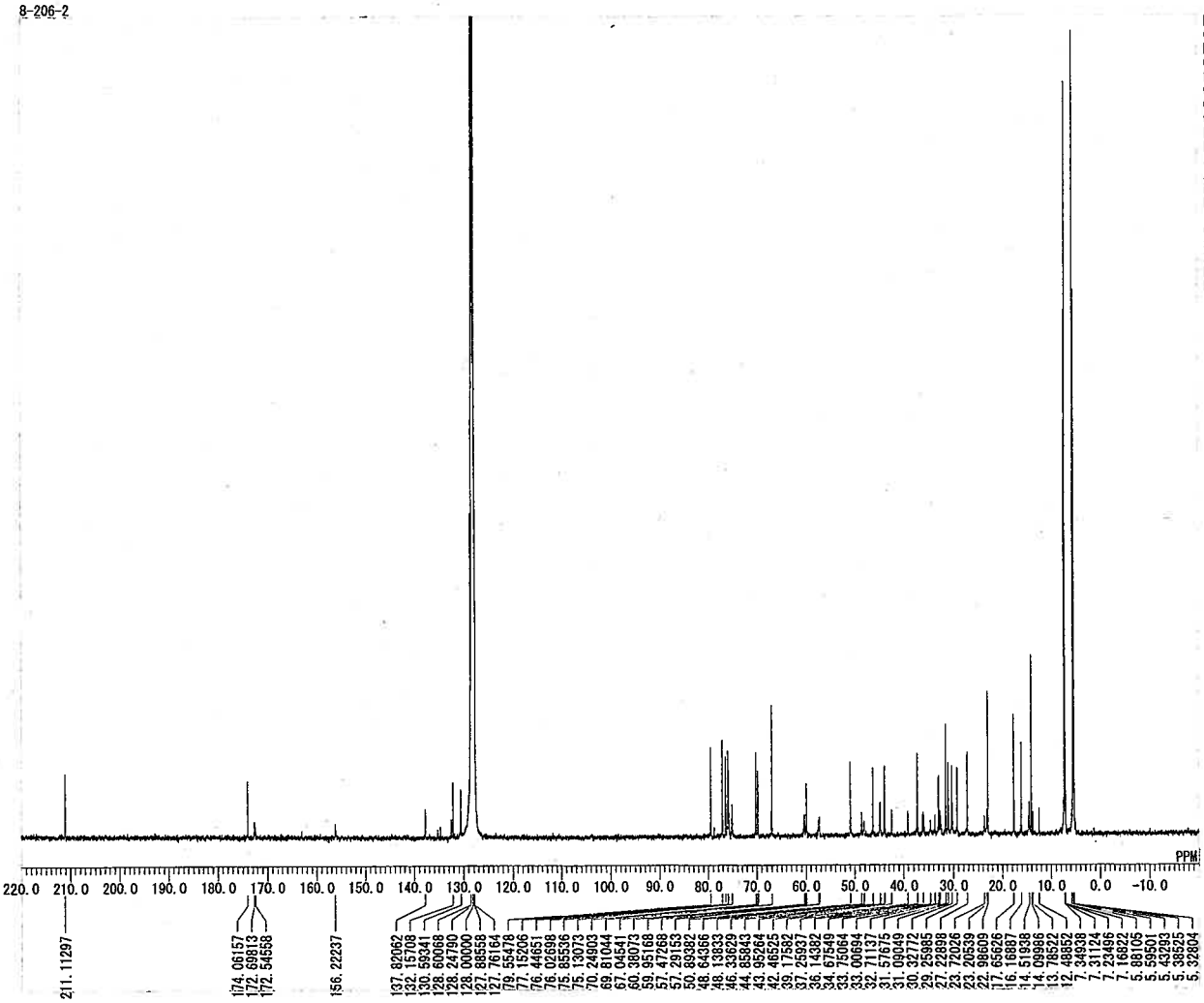

R = TES

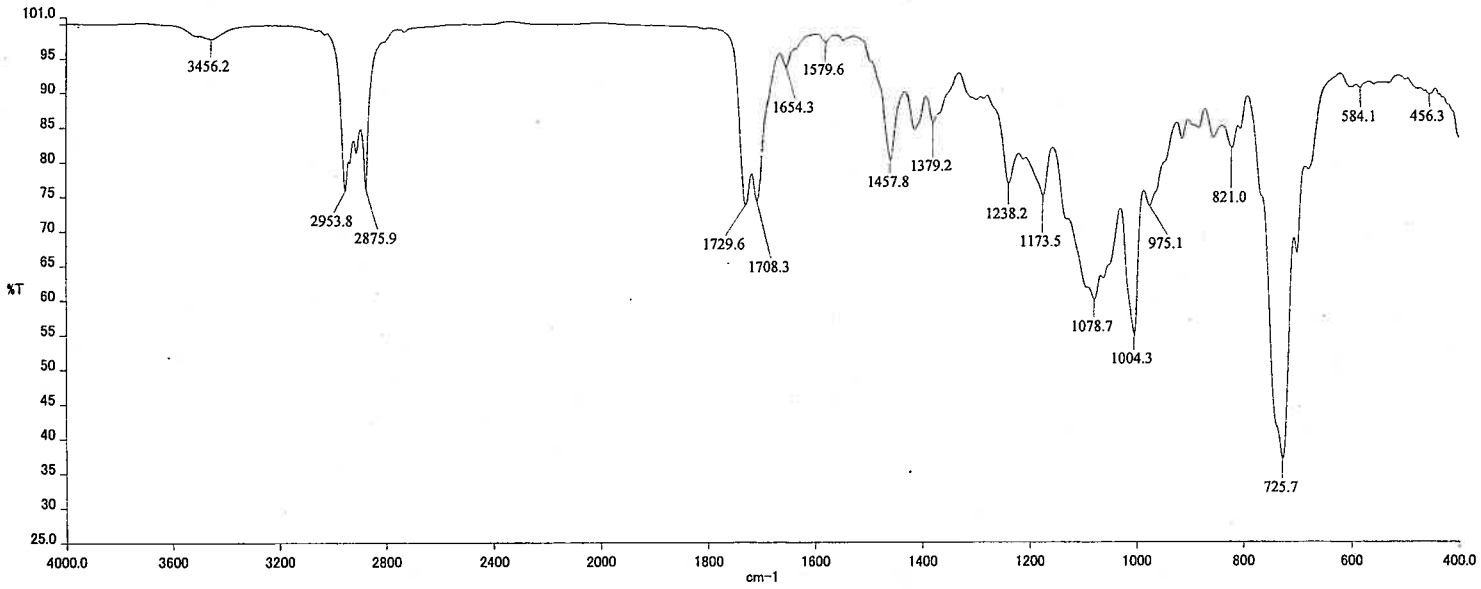
```
DFILE  C:¥ALICE¥DATA¥ochiai8-206-2_
COMNT  8-206-2
DATIM  30-10-2008 17:58:26
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        399.78 MHz
OBSET          4.19 KHz
OBFIN          7.29 Hz
POINT         32768
FREQU      20032.05 Hz
SCANS            32
ACQTM          1.6358 sec
PD             5.0000 sec
PW1            5.65 usec
IRNUC  1H
CTEMP         23.5 c
SLVNT  C6D6
EXREF          7.16 ppm
BF             0.12 Hz
RGAIN            30
```




8-206-2

```
DFILE  C:¥ALICE¥DATA¥ochiai8-206-2_
COMNT  8-206-2
DATIM  31-10-2008 07:59:56
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        100.53 MHz
OBSET          5.35 KHz
OBFIN          5.86 Hz
POINT         32768
FREQU      31407.03 Hz
SCANS         12000
ACQTM          1.0433 sec
PD             2.0000 sec
PW1            3.00 usec
IRNUC  1H
CTEMP         23.4 c
SLVNT  C6D6
EXREF        128.00 ppm
BF             1.20 Hz
RGAIN            58
```






——— 8-206②.sp

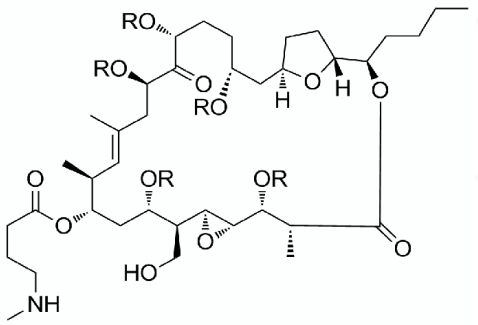
8-206②.pk

8-206②.sp 3601 4000.0 400.0 37.0 100.3 4.0 %T 16 0.8

REF 4000 100.0 2000 100.1 600
3456.2 97.7 2953.8 75.8 2911.9 81.3 2875.9 76.1 1729.6 73.7
1708.3 74.2 1654.3 93.7 1579.6 97.3 1457.8 80.2 1413.8 84.6
1379.2 85.7 1238.2 76.7 1173.5 75.0 1078.7 59.9 1004.3 55.0
975.1 73.5 914.9 83.1 883.2 84.7 855.4 83.3 821.0 81.8
725.7 37.0 697.8 66.6 584.1 90.4 456.3 89.5
END 24 PEAK(S) FOUND

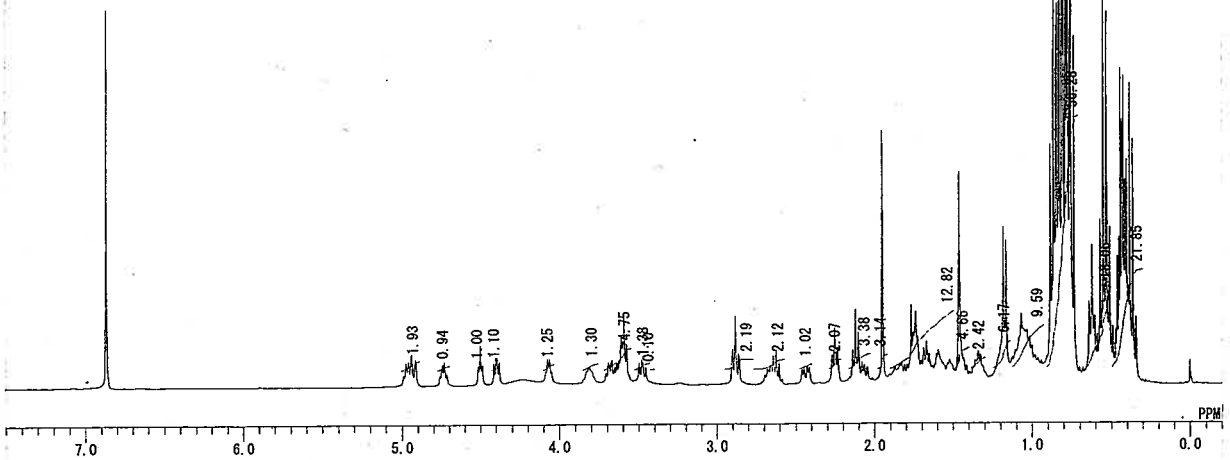
分光器型式: Spectrum 100
測定日時: 2008年10月31日 11:08 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 8-206②.sp
スキャン回数: 16
分解能:4.0cm-1
測定方法:ATR(ダイヤモンド/KRS-5)

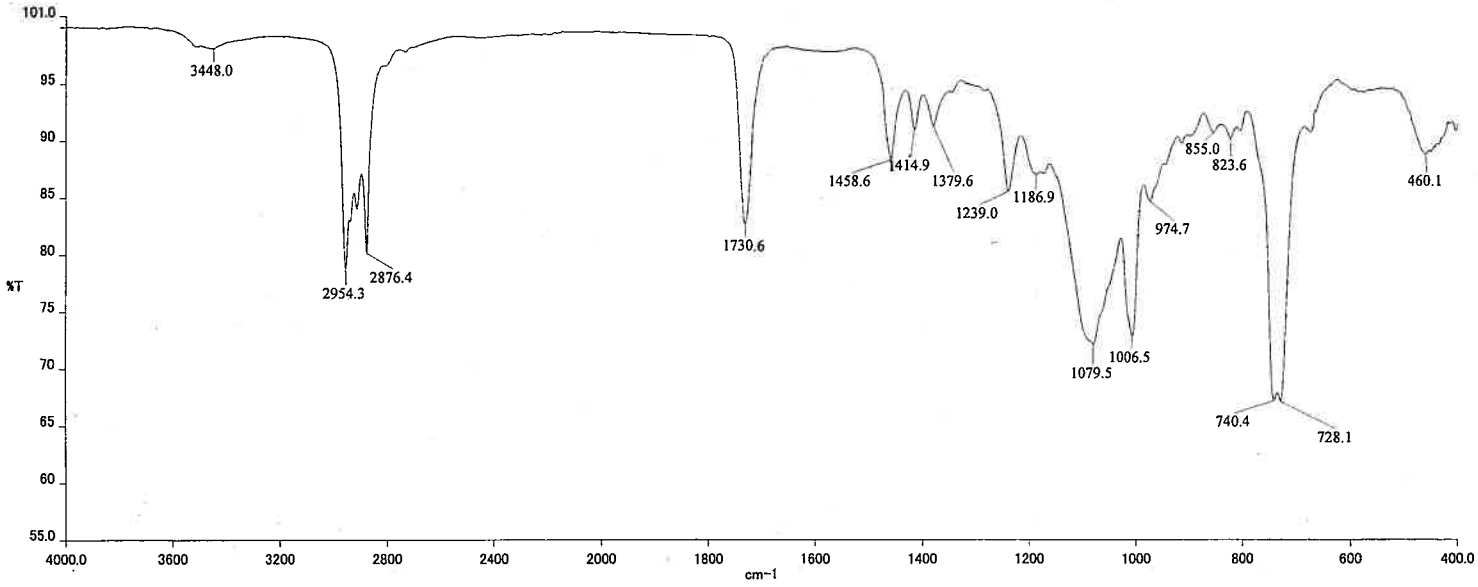
9-005




R = TES

```
DFILE  C:¥ALICE¥DATA¥ochiai9-005_PR
COMNT  9-005
DATIM  04-11-2008 20:08:36
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        395.88 MHz
OBSET          6.28 KHz
OBFIN          0.87 Hz
POINT         32768
FREQU      19841.27 Hz
SCANS           128
ACQTM        1.6515 sec
PD           5.0000 sec
PW1            4.56 usec
IRNUC  1H
CTEMP         25.5 c
SLVNT  C6D6
EXREF         0.00 ppm
BF            0.12 Hz
RGAIN           34
```



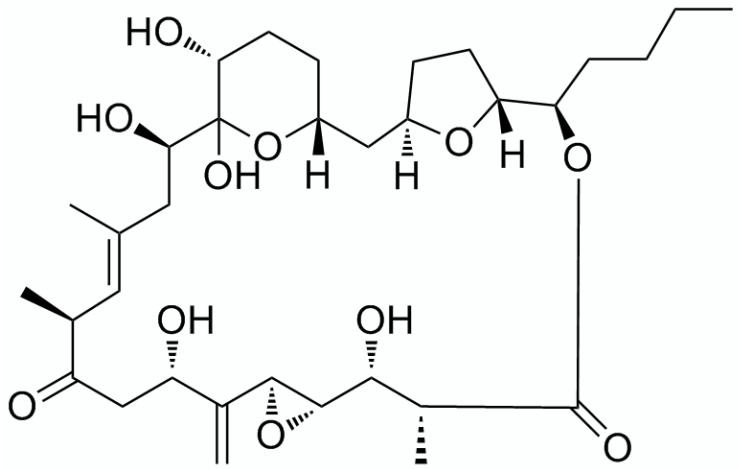

——— 9-005.sp

9-005.pk

9-005.sp 3601 4000.0 400.0 67.2 100.0 4.0 %T 16 0.5

REF 4000 100.0 2000 99.5 600
3448.0 98.1 2954.3 78.8 2912.4 84.0 2876.4 80.1 1730.6 82.8
1458.6 88.4 1414.9 91.1 1379.6 91.4 1239.0 85.6 1186.9 87.1
1079.5 72.1 1006.5 72.9 974.7 84.8 914.7 89.8 855.0 90.8
823.6 90.3 740.4 67.2 728.1 67.2 460.1 88.8
END 19 PEAK(S) FOUND

分光器型式: Spectrum 100
測定日時: 2008年11月5日 17:00 東京 (標準時)
光源: MIR 検出器: LiTa03
ｽﾍﾟｸﾄﾙﾌｧｲﾙ名: 9-005.sp
スキャン回数: 16
分解能:4.0cm-1
測定方法:ATR(ダイヤモンド/KRS-5)




7,10-di-epi-Amphidinolide N

Current Data Parameters
NAME yakusou
EXPNO 1001
PROCNO 1

F2 – Acquisition Parameters
Date_ 500000
Time 14.58
INSTRUM amx600
PROBHD 5 mm Multinu
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 128
DS 2
SWH 5434.783 Hz
FIDRES 0.331713 Hz
AQ 1.5073780 sec
RG 4096
DW 92.000 usec
DE 115.00 usec
TE 300.0 K

F2 – Processing parameters
SI 32768
SF 600.1364521 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40