This journal is © The Royal Society of Chemistry 2012

Supplementary Information for

Total Synthesis of (±)-Pallambins C and D

Xue-Song Xu, Zhen-Wu Li, Yi-Jun Zhang, Xiao-Shui Peng, and Henry N. C. Wong*

Table of contents

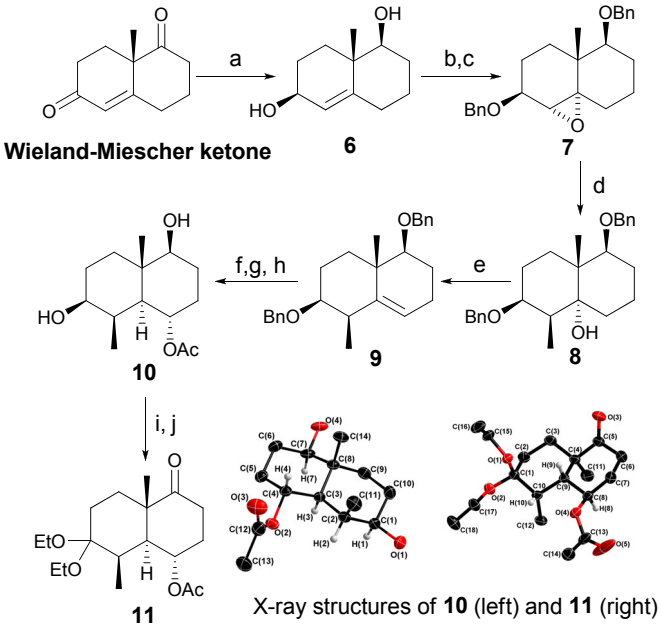
General information……S2
Synthesis of compound 6……S3
Synthesis of compound 6a……S4
Synthesis of compound 7……S4
Synthesis of compound 8……S5
Synthesis of compound 9……S5
Synthesis of compound 9a……S6
Synthesis of compound 9b……S6
Synthesis of compound 10……S7
Synthesis of compound 10a……S7
Synthesis of compound 11……S8
Synthesis of compound 12……S9
Synthesis of compound 12a……S9
Synthesis of compound 13……S10
Synthesis of compound 13a……S10
Synthesis of compound 14……S11
Synthesis of compound 14a……S11
Synthesis of compound 15……S12
Synthesis of compound 15b……S13
Synthesis of compound 16……S13
Synthesis of compound 16a……S14
Synthesis of compound 4……S14
Synthesis of compound 17……S15
Synthesis of compound 17a……S15
Synthesis of compound 18……S16
Synthesis of compound 19……S16
Synthesis of compound 20……S17
Synthesis of compound 21……S17
Synthesis of compound 22……S18
Synthesis of compound 23……S18
Synthesis of compound 24……S19
Synthesis of compound 25……S19
Synthesis of compound 26……S20
Synthesis of compound 27……S20
Synthesis of compound 26a……S21
Synthesis of pallambin C (1)……S22
Synthesis of compound 27a……S22
Synthesis of pallambin D (2)……S23
NMR spectra and basic crystal data……S24-S70

General Information

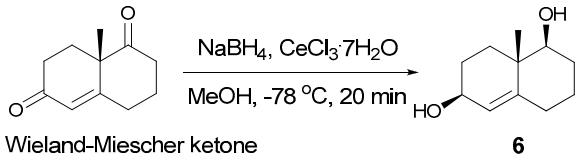
Unless stated otherwise, all reactions were carried out under an argon atmosphere under anhydrous conditions and all reagents were purchased from commercial suppliers without further purification. Solvent drying by standard methods were employed when necessary. The plates used for thin-layer chromatography (TLC) were E. Merck silica gel $60F_{254}$ (0.24 nm thickness) precoated on aluminum plates, and then visualized under UV light (365 nm and 254 nm) or through staining with a 5% of dodecamolybdophosphoric acid in ethanol and subsequent heating. Column chromatography was performed using E. Merck silica gel (230-400 mesh). Melting points were measured on an Electrothermal 9100 apparatus without correction. NMR spectra were recorded on a Bruker Advance III 400 spectrometer (400.00 MHz for ^{1}H and 100 MHz for ^{13}C) and a Bruker 400 MHz NMR AV400Q (400.00 MHz for ^{1}H and 100 MHz for ^{13}C) at room temperature. Solvent signal was used as reference for ^{1}H, ^{13}C NMR ($CDCl_3$, 7.26, 77.16 ppm; C_6D_6, 7.16, 128.06 ppm). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, brs = broad singlet, dd = double doublet, td = triple doublet, dt = double triplet, dq = double quartet, m = multiplet. Mass spectra (ESI, EI and FAB) were obtained with a ThermoFinnigan MAT 95 XL spectrometer and determined at an ionized voltage of 70 eV unless otherwise mentioned. Elemental analyses were performed at Shanghai Institute of Organic Chemistry, The Chinese Academy of Sciences, China. Infrared spectra (IR) were recorded on a Perkin Elmer (Spectrum One) FT-IR spectrometer.

Experimental details and characteristic data for compounds

From Wieland-Miescher ketone to 11

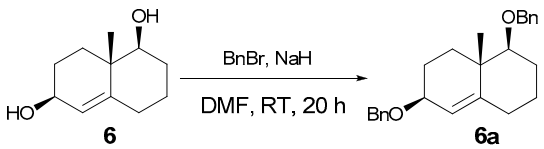


Synthesis of compound 6



To a stirred solution of (±)-Wieland-Miescher ketone (12.0 g, 67.4 mmol) and $CeCl_3$•$7H_2O$ (37.6 g, 101 mmol) in MeOH (250 mL) at -78 °C was added $NaBH_4$ (5.1 g, 13.5 mmol) in one portion. The reaction was stirred for 20 min at -78 °C and warmed up to rt, filtered through Celite. The filtered cake was washed with MeOH (200 mL x 2). After evaporation, the residue was partitioned between EA (500 mL) and saturated NH_4Cl (500 mL), and the aqueous phase was extracted with EA (300 mL x 3). The combined organic phase was dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silical gel, Hex/EA = 2:1) to afford **6** (10.6 g, 86%) as a white solid. mp 151.0-152.0 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 5.41 (s, 1H), 4.18 (brs, 1H), 3.29-3.24 (m, 1H), 2.18-2.10 (m, 1H), 2.01-1.93 (m, 2H), 1.86-1.75 (m, 3H), 1.61-1.54 (m, 1H), 1.48-1.43 (m, 4H), 1.34-1.23 (m, 1H), 1.07 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 144.92, 126.06, 79.22, 67.79, 40.76, 33.64, 31.46, 30.85, 29.00, 24.71, 16.92 ppm; **IR** (neat) 2934, 2860, 1658, 1445, 1050, 931 cm^{-1}; **HRMS** (EI) *m/z* Calcd for $C_{11}H_{18}O_2$ $[M]^+$: 182.1301, found 182.1306.

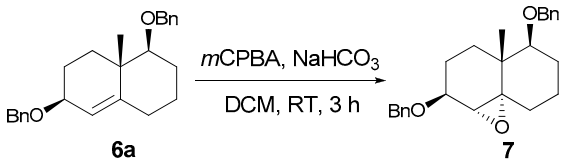
Synthesis of compound 6a



To a stirred solution of compound **6** (14.2 g, 78 mmol) in DMF (160 mL) at was

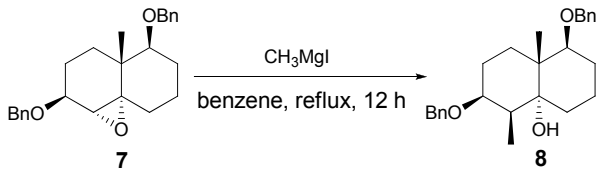
added NaH (~60%, 12.5 g, 312 mmol) in portions 0 °C. The formed mixture was stirred for 30 min at the same temperature, and then BnBr (28 mL, 234 mmol) was added dropwise during 15 min. The reaction was allowed to warm to rt and stirred for 20 h. The reaction mixture was pre-cooled in ice bath and quenched with water (500 mL), extracted with EA (300 mL x 2), washed with brine (300 mL), dried over Na_2SO_4, concentrated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 100:1-50:1) to afford **6a** (26.8 g, 95%) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.38-7.27 (m, 10H), 5.49 (s, 1H), 4.66 (d, *J* = 11.9 Hz, 1H), 4.62 (d, *J* = 11.9 Hz, 1H), 4.56 (d, *J* = 12.0 Hz, 1H), 4.43 (d, *J* = 11.9 Hz, 1H), 3.97-3.94 (m, 1H), 2.98 (dd, *J* = 4.2, 11.6 Hz, 1H), 2.21-2.15 (m, 1H), 2.04-1.96 (m, 4H), 1.82-1.79 (m, 1H), 1.60-1.54 (m, 2H), 1.38-1.31 (m, 1H), 1.26-1.17 (m, 1H), 1.15 (s, 3H); **^{13}C NMR** δ = (100 MHz, $CDCl_3$) δ = 145.59, 139.40, 139.14, 128.45, 128.35, 127.81, 127.56, 127.54, 127.45, 123.39, 86.46, 74.48, 71.44, 70.05, 41.13, 34.07, 31.78, 26.74, 25.35, 24.63, 17.99 ppm;. **IR** (neat) 2937, 2860, 1658, 1453, 1358, 1094, 1070, 734, 696 cm^{-1}; **HRMS** (EI) *m/z* Calcd for $C_{25}H_{30}O_2$ $[M]^+$: 362.2240, found 362.2254.

Synthesis of compound 7



To a well stirred suspension of compound **6a** (19.7 g, 54.4 mmol) and $NaHCO_3$ (20 g, 238.1 mmol) in DCM (500 mL) was added *m*CPBA (~70%, 20 g, 81.4 mmol). The reaction was stirred for 3 h at rt, and then was partitioned with water (500 mL). The aqueous phase was extracted with DCM (300 mL), and the combined organic phase was washed with saturated $NaHCO_3$ (300 mL) and brine (300 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 40:1-20:1) to afford **7** (17.7 g, 86%) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.38-7.27 (m, 10H), 4.65 (d, *J* = 11.1 Hz, 1 H), 4.63 (s, 2 H), 4.44 (d, J = 11.8 Hz, 1 H), 3.72 (dd, *J* = 7.8, 8.1 Hz, 1 H), 3.37-3.33 (m, 1H), 3.04 (s, 1H), 2.04-1.98 (m, 2H), 1.94-1.89 (m, 1H), 1.76-1.73 (m, 1H), 1.65-1.56 (m, 3H), 1.53-1.43 (m, 1H),1.32-1.24 (m, 1H), 1.19 (s, 3H), 0.96 (d, *J* = 14.4 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 139.31,138.33, 128.58, 128.35, 127.86, 127.53, 127.46, 83.69, 72.70, 71.68, 71.32, 66.62, 61.53, 38.88, 29.14, 27.56, 26.48, 24.07, 20.74, 16.25 ppm; **IR** (neat) 2948, 2868, 1605, 1497, 1454, 1360, 1093, 1073, 738, 702 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{25}H_{30}O_3Na$ $[M+Na]^+$: 401.2087, found 401.2100; **Anal.** Calcd for $C_{25}H_{30}O_3$: C, 79.33; H, 7.99; found C, 79.28; H, 8.15.

Synthesis of compound 8

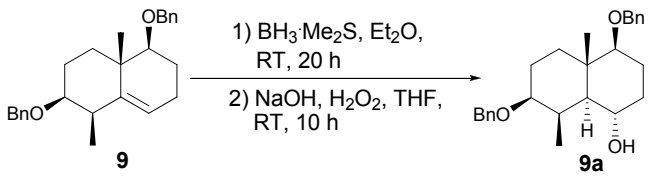


To a stirred solution of MeMgI in minimized amount of Et_2O [prepared from Mg turnings (5.5 g, 230 mmol) and MeI (13.7 mL, 220 mmol), and distilled off most of the solvent Et_2O] was added a solution of the epoxide above (16.8 g, 44.4 mmol) in benzene (300 mL), and the reaction was stirred under reflux for 12 h. After being cooled to 0 °C, the reaction was quenched with 5% HCl (500 mL), extracted with EA (300 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (300 mL) and brine (300 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 20:1-10:1) to afford **8** (15.6 g, 89%) as a colorless oil. **^{1}HNMR** (400 MHz, $CDCl_3$) δ = 7.37-7.27 (m, 10H), 4.61 (d, *J* = 11.8 Hz, 1H), 4.55 (d, *J* = 11.9 Hz, 1H), 4.49 (d, *J* = 11.9 Hz, 1H), 4.44 (d, *J* = 11.8 Hz, 1H), 4.09-4.04 (m, 1H), 3.56 (dd, *J* = 4.5, 11.5 Hz, 1H), 2.12-1.96 (m, 2H), 1.89-1.60 (m, 8H), 1.52-1.42 (m, 1H), 1.09 (s, 3H), 1.05 (d, *J* = 7.6 Hz, 3H), 0.74 (s, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 139.72, 139.32, 128.45, 128.31, 127.55, 127.47, 127.45, 127.32, 82.12, 78.91, 76.60, 71.89, 69.96, 45.71, 42.42, 32.81, 30.35, 26.26, 23.52, 19.05, 15.26, 10.33 ppm; **IR** (neat) 2939, 2867, 1496, 1453, 1386, 1364, 1095, 1071, 953, 735, 698 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{26}H_{34}O_3Na$ $[M+Na]^+$: 417.2400, found 417.2390; **Anal.** Calcd for $C_{26}H_{34}O_3$: C, 79.15; H, 8.69; found C, 78.96; H, 8.92.

Synthesis of compound 9

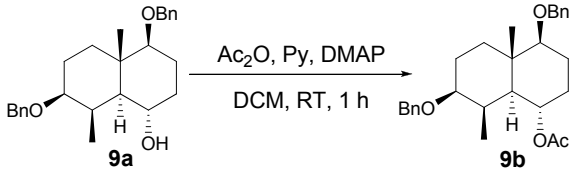
To a stirred solution of compound **8** (18.3 g, 46.4 mmol) and pyridine (100 mL) in DCM (250 mL) at 0 °C was added a solution of $SOCl_2$ (10 mL, 138 mmol) in DCM (50 mL) dropwise during 10 min. The reaction was stirred for 30 min at the same temperature and quenched with ice water (500 mL). The aqueous phase was extracted with DCM (300 mL), and the combined organic phase was washed with 5% HCl (300 mL) and brine (300 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 100:1-50:1) to afford **9** (16.1 g, 92%) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.42-7.32 (m, 10H), 5.42 (brs, 1H), 4.75 (d, *J* = 11.9 Hz, 1H), 4.65 (d, *J* = 11.9 Hz, 1H), 4.54 (d, *J* = 12.1 Hz, 1H), 4.50 (d, *J* = 12.0 Hz, 1H), 3.50-3.45 (m, 1H), 3.19 (dd, *J* = 3.1, 12.2 Hz, 1H), 2.91-2.84 (m, 1H), 2.25-2.10 (m, 3H), 2.00-1.68 (m, 4H), 1.28 (s, 3H), 1.26 (d, *J* = 7.5 Hz, 3H), 1.15 (dt, *J* = 3.9, 13.5 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 145.67, 139.42, 139.19, 128.39, 128.28, 127.47, 127.40, 127.34, 122.34, 85.61, 79.91, 71.20, 69.66, 41.55, 39.29, 36.53, 25.17, 23.19, 22.72, 20.64, 16.55 ppm; **IR** (neat) 2940, 2870, 1605, 1496, 1454, 1363, 1099, 1074, 1028, 734, 696 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{26}H_{32}O_2Na$ $[M+Na]^+$: 399.2295, found 399.2286.

Synthesis of compound 9a



To a stirred solution of compound **9** (14.7 g, 39.1 mmol) in Et_2O (400 mL) was added BH_3•Me_2S (neat, 8 mL, 80 mmol) dropwise during 10 min. The reaction was stirred for 20 h at rt, and then was added water/THF (1:1,100 mL) slowly at -10 °C. NaOH (3M, 100 mL) and 30% H_2O_2 (50 mL) were added successively, and the formed mixture was stirred for 10 h at rt. Cooled to 0 °C, saturated $Na_2S_2O_3$ was added dropwise during 30 min, and the stirring continued for 30 min. The aqueous phase was extracted with Et_2O (300 mL), and the combined organic phase was washed with saturated NH_4Cl (300 mL) and brine (300 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 5:1) to afford **9a** (12.6 g, 82%) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.38-7.28 (m, 10H), 4.63 (d, *J* = 11.8 Hz, 1H), 4.61 (d, *J* = 11.8 Hz, 1H), 4.48 (d, *J* = 11.9 Hz, 1H), 4.41 (d, *J* = 11.8 Hz, 1H), 3.82 (dt, *J* = 4.1, 10.4 Hz, 1H), 3.42-3.37 (m, 1H), 2.89 (dd, *J* = 4.0, 11.4 Hz, 1H), 2.69-2.65 (m, 1H), 2.13-2.09 (m, 1H), 2.04-2.00 (m, 1H), 1.95-1.91 (m, 1H), 1.74-1.68 (m, 2H), 1.57-1.47 (m, 2H), 1.33-1.23 (m, 1H), 1.07-1.04 (m, 2H), 1.03 (d, *J* = 7.4 Hz, 3H), 1.01 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 139.25, 139.17, 12844, 128.33, 127.58, 127.46, 86.88, 80.59, 71.49, 69.61, 66.96, 51.45, 39.43, 37.01, 34.23, 30.28, 24.97, 23.27, 14.72, 9.14 ppm; **IR** (neat) 2936, 2856, 1637, 1453, 1384, 1363, 1073, 1027, 735, 697 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{26}H_{34}O_3Na$ $[M + Na]^+$: 417.2400, found 417.2391; **Anal.** Calcd for $C_{26}H_{34}O_3$: C, 79.15; H, 8.69; found C, 78.61; H, 9.07.

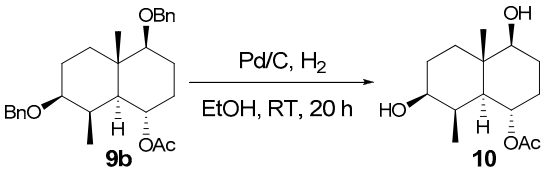
Synthesis of compound 9b



To a stirred solution of compound **9a** (12.6 g, 32 mmol), pyridine (3.5 mL) and DMAP (0.5 g) in DCM (50 mL) was added Ac_2O (3.6 mL, 35 mmol) dropwise during 5 min at 0 °C. The reaction was stirred for 1 h at rt, and then was quenched with ice water (100 mL). The aqueous phase was extracted with DCM (100 mL), and the combined organic phase was washed with 5% HCl (100 mL x 2) and brine (100 mL x 2), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 10:1) to afford **9b** (13.8 g, 99%) as a white solid. mp 80.5-81.8 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.36-7.27 (m, 10H), 5.01 (dt, *J* = 4.8, 11.1 Hz, 1H), 4.61 (d, *J* = 12.2 Hz, 1H), 4.58 (d, *J* = 13.0 Hz, 1H), 4.51 (d, *J* = 11.9 Hz, 1H), 4.41 (d, *J* = 11.8 Hz, 1H), 3.41-3.36 (m, 1H), 2.90 (dd, *J* = 4.0 , 11.5 Hz, 1H), 2.44-2.40 (m, 1H), 2.21-2.16 (m, 1H), 2.08-2.03 (m, 1H), 2.05 (s, 3H), 1.95-1.91 (m, 1H), 1.76-1.66 (m, 2H), 1.62-1.51 (m, 1H), 1.35-1.21 (m, 2H), 1.09-1.01 (m, 1H),1.04 (s, 3H), 0.91 (d, *J* = 7.4 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 170.70,

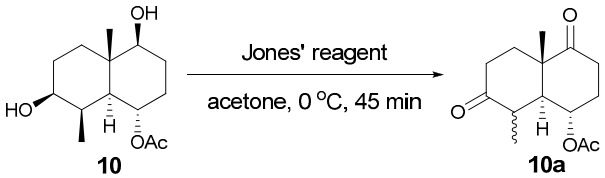
139.21, 139.15, 128.45, 128.36, 127.54, 127.50, 86.46, 80.59, 71.49, 70.10, 69.86, 48.88, 39.75, 36.90, 30.84, 30.31, 24.69, 22.97, 21.35, 14.59, 9.13 ppm; **IR** (neat) 2940, 2861, 1735, 1454, 1365, 1244, 1098, 1075, 1027, 735, 698 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{28}H_{36}O_4Na$ $[M + Na]^+$ 459.2506, found 459.2496; **Anal.** Calcd for $C_{28}H_{36}O_4$: C, 77.03; H, 8.31; found C, 76.72; H, 8.14.

Synthesis of compound 10



To a solution of compound **9b** (18.5 g, 42.4 mmol) in EtOH (500 mL) was added Pd/C (10%, 2 g), and the reaction was stirred for 20 h at rt under an atmosphere of H_2. The reaction mixture was filtered through Celite and washed with MeOH (500 mL), then concentrated and purified by a flash column (silica gel, Hex/EA = 2:1) to afford **10** (10.3 g, 95%) as a white solid. mp 168.7- 170.1 °C. The structure of **10** was confirmed by X-ray crystallographic results. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 4.97 (dt, *J* = 4.8 Hz, 11.2 Hz, 1H), 3.71-3.66 (m, 1H), 3.21 (dd, *J* = 3.6, 11.3 Hz, 1H), 2.29-2.25 (m, 1H), 2.15-2.11 (m, 1H), 2.02 (s, 3H), 1.83 (td, *J* = 3.2, 13.2 Hz, 1H), 1.74-1.70 (m, 1H), 1.66-1.63 (m, 2H), 1.45-1.28 (m, 4H), 1.18-1.10 (m, 1H), 0.94 (s, 3H), 0.87 (d, *J* = 7.4 Hz, 3H) ppm; **^{13}C NMR** (100MHz, $CDCl_3$) δ = 170.75, 79.26, 73.69, 69.79, 48.57, 38.93, 36.53, 33.64, 30.32, 28.62, 25.69, 21.28, 13.45, 8.59 ppm; **IR** (neat) 2931, 2869, 1735, 1712, 1639, 1242, 1057, 1015 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{14}H_{24}O_4Na$ $[M + Na]^+$ 279.1567, found 279.1569; **Anal.** Calcd for $C_{14}H_{24}O_4$: C, 65.60; H, 9.44; found C, 65.39; H, 9.30.

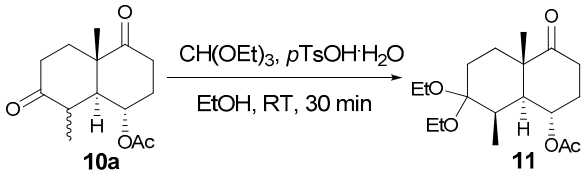
Synthesis of compound 10a



To a stirred solution of compound **10** (9.6 g, 37.5 mmol) in acetone (400 mL) at 0 °C was added Jones' reagent dropwise [prepared from CrO_3 (27 g) and conc. H_2SO_4 (23 mL), and diluted to 100 mL with water] until TLC indicated the starting material was consumed completely. The reaction was quenched with *i*PrOH (10 mL) and filtered through Celite. The filtered cake was washed with acetone (400 mL). After concentration, the residue was partitioned between DCM (500 mL) and water (300 mL). The aqueous phase was extracted with DCM (200 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (200 mL) and brine (200 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 6:1-3:1) to afford **10a** (8.7 g, 92%) as a colorless oil, and revealed by ^{1}H NMR a mixture of the C-4 methyl diastereomers with a α/β ratio of 7:3. mp 61.4-65.2 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 5.36 (dt, *J* = 4.5, 11.0 Hz,

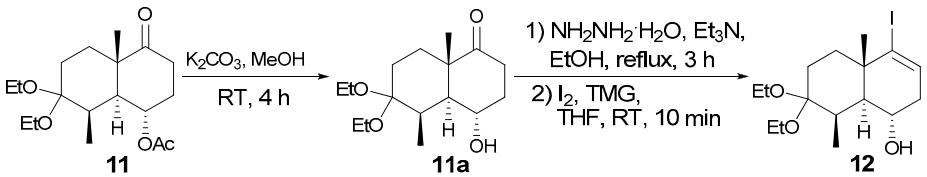
0.7H), 5.18 (td, J = 5.6, 9.4 Hz, 0.3H), 2.83-2.33 (m, 6H), 2.19-1.74 (m, 6H), 1.65-1.58 (m, 1H), 1.40 (s, 1.93 H), 1.31 (s, 0.85H), 1.16 (d, J = 7.8 Hz, 1.93 H), 1.09 (d, J = 6.8 Hz, 0.85 H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 213.16, 211.24, 170.60, 71.80, 67.99, 50.68, 48.68, 46.61, 46.28, 45.02, 44.28, 36.02, 35.18, 34.52, 34.05, 32.49, 30.79, 29.39, 21.57, 21.12, 19.20, 17.84, 14.07 ppm; **IR** (neat) 2954, 1736, 1711, 1458, 1429, 1379, 1241, 1062, 1030 cm^{-1}; **HRMS** (EI) *m/z* Calcd for $C_{14}H_{20}O_4$ $[M]^+$ 252.1356, found 252.1345; **Anal.** Calcd for $C_{14}H_{20}O_4$: C, 66.65; H, 7.99; found C, 66.23; H, 7.81.

Synthesis of compound 11



To a stirred solution of compound **10a** (7.8 g, 31 mmol) in EtOH (150 mL) was added $CH(OEt)_3$ (3.5 mL, 21 mmol) and pTsOH•H_2O (150 mg, 0.8 mmol). The reaction was stirred for 30 min at rt, and then quenched with saturated $NaHCO_3$ (150 mL), extracted with DCM (300 mL x 2). The combined organic phase was washed with brine (200 mL x 2), dried over Na_2SO_4, and evaporated to give a residue, which was purified by flash column (silica gel, Hex/EA = 10:1-5:1) to afford **11** (6.2 g, 61%, 90% brsm) as a white solid, and recovered dione (2.5 g, 32%). mp 83.5-85.0 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 5.27 (dt, J = 4.7, 11.1 Hz, 1H), 3.44-3.32 (m, 4H), 2.67 (dt, J = 6.7, 14.6, 1H), 2.35-2.19 (m, 3H), 2.08 (dd, J = 4.5, 11.9, 1H), 2.04 (s, 3H), 1.86-1.42 (m, 5H), 1.17 (s, 3H), 1.12 (t, J = 6.6 Hz, 6H), 0.92 (d, J = 7.5 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 212.79, 170.71, 101.82, 68.76, 54.92, 54.70, 47.04, 45.75, 35.35, 35.25, 30.93, 29.53, 23.91, 21.23, 19.68, 15.39, 15.29, 11.48 ppm; **IR** (neat) 2974, 1738, 1714, 1639, 1378, 1238, 1097, 1048, 1033 cm^{-1}; **HRMS** (EI) *m/z* Calcd for $C_{18}H_{30}O_5$ $[M]^+$ 326.2088, found 326.2090; **Anal.** Calcd for $C_{18}H_{30}O_5$: C, 66.23; H, 9.26; found C, 66.29; H, 9.39.

Synthesis of compound 12

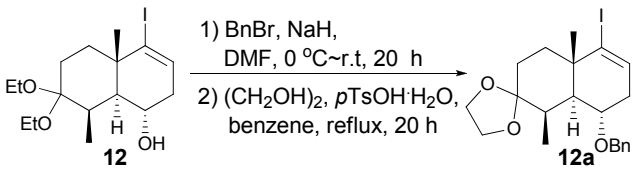


To a solution of compound **11** (7.9 g, 24.2 mmol) in MeOH (200 mL) was added K_2CO_3 (2 g), and the formed mixture was stirred for 4 h at rt. The reaction was quenched with saturated NH_4Cl (100 mL), and extracted with DCM (250 mL x 3). The combined organic phase was washed with brine (100 mL), dried over Na_2SO_4, and evaporated under vacuum to give **11a** as white solid, which was dissolved in EtOH (30 mL), and then was added dropwise to a stirred solution of hydrazine monohydrate (20 mL) and Et_3N (20 mL) in EtOH (10 mL). The solution was stirred under reflux for 3 h. After evaporation, the residue was partitioned between EA (200

mL) and water (50 mL), and the aqueous phase was extracted with EA (100 mL x 3). The combined organic phase was washed with brine (50 mL), dried over Na_2SO_4, evaporated and dried under vacuum to give the hydrazone as a white solid, which was then used directly for the next process.

To a stirred solution of I_2 (19 g, 75 mmol) in THF (80 mL) was added a solution of TMG (1,1,3,3-tetramethylguanidine) (20 mL, 160 mmol) in THF (80 mL) dropwise during 30 min at rt, and then a solution of the above hydrazone in THF (80 mL) was added dropwise during 10 min. The reaction was stirred at rt for 10 min, cooled and quenched with saturated $Na_2S_2O_3$ (100 mL), partitioned between EA (300 mL) and water (100 mL). The aqueous phase was extracted with EA (200 mL), and the combined organic phase was washed with brine (100 mL x 2), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA/Et_3N = 20:1:0.01) to afford **12** (8.0 g, 84%) as a pale yellow syrup. **^{1}H NMR** (400 MHz, C_6D_6) δ = 5.89 (dd, *J* = 2.8, 5.1 Hz, 1H), 3.58 (brs, 1H), 3.43-3.35 (m, 3H), 3.21-3.17 (m, 1H), 2.57-2.54 (m, 1H), 2.19 (dd, *J* = 4.4, 11.6, 1H), 2.01-1.93 (m, 1H), 1.74-1.71 (m, 1H), 1.63-1.55 (m, 3H), 1.51-1.47 (m, 1H), 1.11 (t, *J* = 7.1 Hz, 3H), 1.05 (t, *J* = 7.0 Hz, 3H), 0.94-0.92 (m, 6H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 134.71, 117.85, 102.88, 64.39, 55.05, 54.95, 48.15, 42.99, 40.98, 39.90, 34.68, 25.66, 20.80, 15.57, 15.52, 11.52 ppm; **IR** (neat) 2971, 2926, 1645, 1634, 1449, 1385, 1086, 1049 cm^{-1}; **HRMS** (EI) *m/z* Calcd for $C_{16}H_{27}IO_3$ $[M]^+$ 394.0999, found 394.0986.

Synthesis of compound 12a

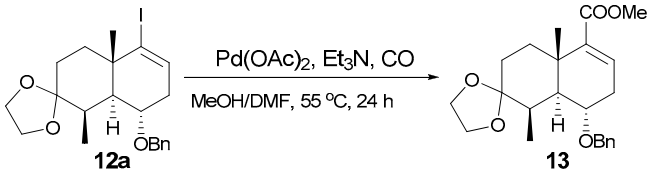


To a stirred solution of compound **12** (4 g, 10 mmol) in DMF (40 mL) at 0 °C was added NaH (~60%, 2 g, 50 mmol) in portions. After stirring for 30 min, BnBr (3.5 mL, 29 mmol) was added dropwise during 5 min. The reaction was warmed up to rt and stirred for 20 h. Cooled to 0 °C, the reaction was quenched with water (40 mL), extracted with EA (100 mL x 2), washed with saturated NH_4Cl (100 mL) and brine (100 mL), dried over Na_2SO_4, evaporated and dried under vacuum to give a residue, which was used directly for the next process.

The residue above was dissolved in benzene (100 mL), followed by addition of ethylene glycol (4 mL) and *p*TsOH•H_2O (400 mg, 2.1 mmol). The reaction was stirred under reflux for 20 h with the Dean-Stark apparatus to remove water formed, and then cooled to rt, partitioned between EA (100 mL) and water (100 mL). The organic phase was washed with saturated $NaHCO_3$ (100 mL) and brine (100 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 50:1-30:1) to afford **12a** (4.1 g, 89%) as a pale yellow semi-solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.37-7.27 (m, 5H), 6.17 (dd, *J* = 2.8, 5.2 Hz, 1H), 4.64 (d, *J* = 11.4 Hz, 1H), 4.42 (d, *J* = 11.4 Hz, 1H), 3.97-3.88 (m, 4H), 3.77-3.70 (m, 1H), 2.66 (td, *J* = 5.6, 17.2 Hz, 1H), 2.32-2.26 (m, 2H), 2.12 (dq, *J* = 2.8, 8.8 Hz, 1H), 1.89-1.81 (m, 1H), 1.65-1.61 (m, 1H), 1.57-1.49 (m, 2H), 1.03 (s,

3H), 0.97 (d, J = 7.3 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 138.54, 134.05, 128.49, 127.86, 127.77, 117.25, 111.57, 71.36, 71.02, 64.48, 64.23, 47.58, 42.34, 40.20, 37.40, 36.15, 27.62, 20.72, 12.01 ppm; **IR** (neat) 2945, 2881, 1453, 1373, 1355, 1099, 736, 698 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{21}H_{28}IO_3$ $[M+H]^+$ 455.1078, found 455.1085.

Synthesis of compound 13



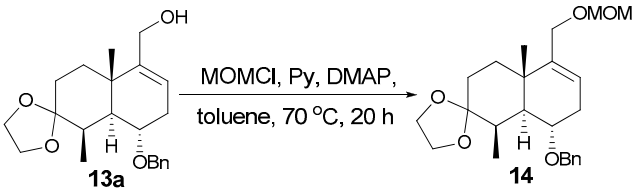
A suspension of compound **12a** (4.5 g, 10 mmol), $Pd(OAc)_2$ (225 mg , 1 mmol) and Et_3N (5.5 mL, 40 mmol) in MeOH/DMF (64 mL, 7/1) was degassed and charged with CO (balloon), stirred at 55 °C for 24 h. The reaction mixture was cooled to rt, diluted with EA (100 mL) and filtered through Cellite. The filtrate was washed with water (50 mL), saturated NH_4Cl (50 mL) and brine (50 mL). The organic phase was collected, dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 50:1-20:1) to afford **13** (3.1 g, 81%) as a white solid. mp 99.5-100.2 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.35-7.27 (m, 5H), 6.59 (dd, J = 3.1, 4.6 Hz, 1H), 4.67 (d, J = 11.4 Hz, 1H), 4.45 (d, J = 11.4 Hz, 1H), 3.96-3.87 (m, 4H), 3.79-3.73 (m, 1H), 3.68 (s, 3H), 2.85 (td, J = 5.4, 18.9 Hz, 1H), 2.42-2.36 (m, 1H), 2.34-2.29 (m, 1H), 2.19 (dq, J = 3.0, 8.5 Hz, 1H), 2.03 (dd, J = 4.6, 11.5 Hz, 1H), 1.93 (ddd, J = 3.1, 4.1, 5.2 Hz, 1H), 1.52-1.44 (m, 2H), 1.27 (s, 3H), 1.02 (d, J = 7.5 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 167.28, 140.62, 138.67, 135.19, 128.50, 127.86, 127.75, 111.37, 71.41, 71.04, 64.39, 64.17, 51.38, 47.56, 38.05, 36.09, 33.89, 33.09, 27.05, 21.20, 12.18 ppm; **IR** (neat) 2981, 2950, 2882, 1714, 1435, 1359, 1239, 1101, 1077, 737, 699 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{23}H_{31}O_5$ $[M+H]^+$ 387.2166, found 387.2163. **Anal.** Calcd for $C_{23}H_{30}O_5$: C, 71.48; H, 7.82; found C, 71.49; H, 7.84.

Synthesis of compound 13a

To a solution of compound **13** (3 g, 7.8 mmol) in DCM (30 mL) at -78 °C was added DIBAL-H solution (15 mL, 1 M in toluene) dropwise during 5 min, and the reaction was stirred at the same temperature for 20 min. The reaction was quenched with saturated NH_4Cl (20 mL), diluted with DCM (50 mL) and water (50 mL). The organic phase was washed with brine (50 mL x 2), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 2:1) to afford **13a** (2.7 g, 97%) as a white solid. mp 109.7-110.7 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.35-7.27 (m, 5H), 5.54-5.52 (m, 1H), 4.67 (d, J = 11.4 Hz, 1H), 4.43 (d, J = 11.4

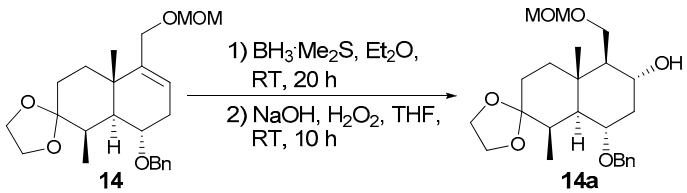
Hz, 1H), 4.13-4.06 (m, 2H), 3.97-3.88 (m, 4H), 3.81-3.74 (m, 1H), 2.81-2.74 (m, 1H), 2.40-2.33 (m, 1H), 2.13-2.05 (m, 2H), 1.92 (dt, *J* = 5.0, 13.3 Hz, 1H), 1.73-1.66 (m, 2H), 1.55-1.49 (m, 1H), 1.22 (brs, 1H), 1.13 (s, 3H), 1.01 (d, *J* = 7.5 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 146.33, 138.87, 128.45, 127.86, 127.64, 119.91, 111.49, 72.17, 70.75, 64.36, 64.18, 62.72, 47.19, 37.98, 36.15, 33.46, 33.25, 26.98, 21.93, 12.14 ppm; **IR** (neat) 2929, 2883, 1454, 1383, 1355, 1092, 988 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{22}H_{31}O_4$ $[M+H]^+$ 359.2217, found 359.2245. **Anal.** Calcd for $C_{22}H_{30}O_4$: C, 73.71; H, 8.44; found C, 73.36; H, 8.96.

Synthesis of compound 14



To a stirred solution of compound **13a** (2.7 g, 7.5 mmol), DIPEA (6 mL, 36 mmol) and DMAP (10 mg) in toluene (50 mL) was added MOMCl (2.3 mL, 30 mmol) dropwise during 5 min at rt. The reaction was stirred at 70 °C for 20 h and then allowed to rt. The reaction was quenched with water (50 mL), extracted with EA (100 mL x 2), washed with saturated $NaHCO_3$ (50 mL) and brine (50 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 20:1-10:1) to afford **14** (2.9 g, 96%) as a pale yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.35-7.28 (m, 5H), 5.55 (brs, 1H), 4.67 (d, *J* = 11.4 Hz, 1H), 4.61 (s, 2H), 4.42 (d, *J* = 11.4 Hz, 1H), 4.03 (d, *J* = 12.3 Hz, 1H), 3.96-3.88 (m, 5H), 3.81-3.75 (m, 1H), 3.36 (s, 3H), 2.77 (td, *J* = 5.5, 17.5 Hz, 1H), 2.39-2.33 (m, 1H), 2.12-2.05 (m, 2H), 1.92 (dt, *J* = 5.2, 13.0 Hz, 1H), 1.71-1.65 (m, 2H), 1.52 (dd, *J* = 1.7, 13.5 Hz, 1H), 1.10 (s, 3H), 1.01 (d, *J* = 7.5 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 142.70, 138.89, 128.43, 127.87, 127.62, 122.09, 111.51, 95.65, 72.11, 70.71, 67.20, 64.35, 64.18, 55.50, 47.15, 37.95, 36.13, 33.47, 33.36, 27.00, 21.69, 12.15 ppm; **IR** (neat) 2923, 2883, 1725, 1454, 1382, 1358, 1098, 698 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{24}H_{34}O_5Na$ $[M+Na]^+$ 425.2298, found 425.2294.

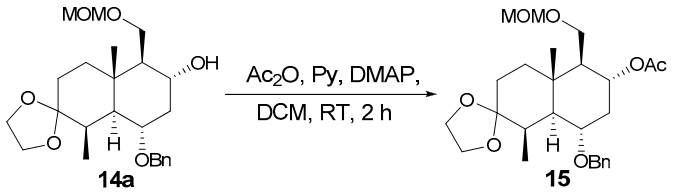
Synthesis of compound 14a



To a stirred solution of compound **14** (2.6 g, 6.5 mmol) in Et_2O (60 mL) was added BH_3•Me_2S (neat, 1.3 mL, 13 mmol) dropwise during 10 min. The reaction was stirred for 20 h at rt, and then was added water/THF (1:1, 20 mL) slowly at -10 °C. NaOH (3M, 20 mL) and 30% H_2O_2 (10 mL) were added successively, and the resultant mixture was stirred for 10 h at rt. Cooled to 0 °C, saturated $Na_2S_2O_3$ was added dropwise during 30 min, and the stirring continued for additional 30 min. The aqueous phase was extracted with Et_2O (100 mL), and the combined organic phase

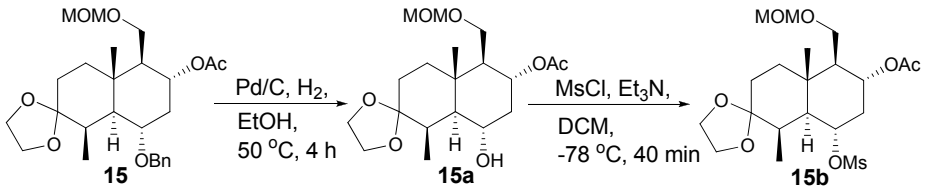
was washed with saturated NH_4Cl (100 mL) and brine (100 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 10:1-2:1) to afford **14a** (2.1 g, 77%) as a colorless oil. **^{1}H NMR** (400 MHz, C_6D_6) δ = 7.32-7.30 (m, 2H), 7.18-7.15 (m, 2H), 7.10-7.06 (m, 1H), 4.53 (d, *J* = 11.4 Hz, 1H), 4.29 (s, 2H), 4.16 (d, *J* = 11.5 Hz, 1H), 3.87-3.80 (m, 1H), 3.70 (dd, *J* = 3.2, 9.9 Hz, 1H), 3.57-3.36 (m, 6H), 3.34 (d, *J* = 2.7 Hz, 1H), 3.09 (s, 3H), 2.77 (td, *J* = 4.6, 11.8 Hz, 1H), 2.68-2.61 (m, 1H), 2.05 (dd, *J* = 4.8, 11.2 Hz, 1H), 1.89 (ddd, *J* = 4.6, 4.1, 3.6 Hz, 1H), 1.65-1.46 (m, 4H), 1.39-1.34 (m, 1H), 1.12 (d, *J* = 7.6 Hz, 3H), 0.67 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 139.63, 128.54, 128.00, 127.59, 111.39, 96.65, 73.08, 70.53, 69.80, 67.78, 64.25, 64.08, 56.00, 55.18, 50.77, 41.60, 36.72, 36.39, 36.22, 27.14, 16.65, 12.55 ppm; **IR** (neat) 2934, 2884, 1453, 1386, 1148, 1101, 1030, 924, 698 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{24}H_{36}O_6Na$ $[M+Na]^+$ 443.2404, found 443.2424.

Synthesis of compound 15



To a stirred solution of compound **14a** (2 g, 4.8 mmol), pyridine (1 mL) and DMAP (100 mg) in DCM (20 mL) was added Ac_2O (0.7 mL, 7.1 mmol) at 0 °C. The reaction was warmed up to rt and stirred for 2 h, and then quenched with water (20 mL). The aqueous phase was extracted with DCM (50 mL), and the combined organic phase was washed with 5% HCl (50 mL) and brine (50 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 7:1-5:1) to afford **15** (2.1 g, 95%) as a white solid. mp 92.7-94.0 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ = 7.30-7.28 (m, 2H), 7.18-7.14 (m, 2H), 7.09-7.06 (m, 1H), 5.27 (dt, *J* = 5.0, 11.5 Hz, 1H), 4.45-4.39 (m, 3H), 4.05 (d, *J* = 11.5 Hz, 1H), 3.58 (d, *J* = 3.5 Hz, 2H), 3.52-3.39 (m, 5H), 3.17 (s, 3H), 2.83 (td, *J* = 4.8, 11.3 Hz, 1H), 2.64-2.58 (m, 1H), 2.03 (dd, *J* = 4.8, 11.2 Hz, 1H), 1.95 (dt, *J* = 5.8, 12.6 Hz, 1H), 1.80 (s, 3H), 1.70-1.66 (m, 2H), 1.55-1.38 (m, 3H), 1.10 (d, *J* = 7.6 Hz, 3H), 0.90 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 169.94, 139.53, 128.55, 127.59, 111.41, 96.81, 73.07, 70.80, 69.61, 64.49, 64.24, 64.08, 55.03, 54.43, 50.74, 38.20, 36.78, 36.62, 36.52, 27.10, 20.94, 16.83, 12.36 ppm; **IR** (neat) 2946, 2885, 1733, 1369, 1245, 1149, 1101, 1029, 698 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{26}H_{38}O_7Na$ $[M+Na]^+$ 485.2510, found 485.2517.

Synthesis of compound 15b

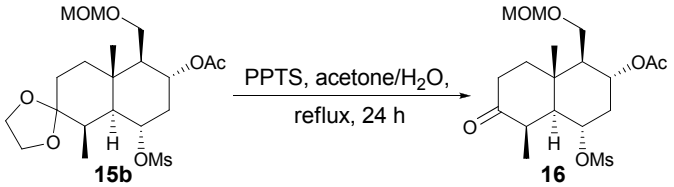


To a solution of compound **15** (2 g, 4.3 mmol) in EtOH (50 mL) was added Pd/C (200

mg, 10%). The reaction was degassed and charged with H_2 (balloon), stirred at 50 °C for 4 h. Cooled to rt, the reaction mixture was diluted with EA (200 mL), filtered through Celite. The filtrate was concentrated and the residue was dried under vacuum to give **15a** as a colorless oil, which was used directly for the next process.

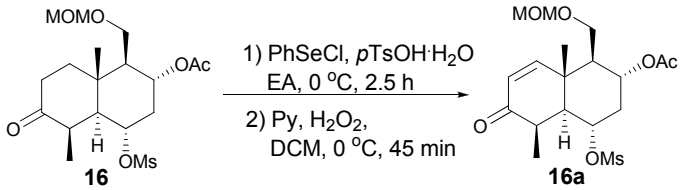
To a stirred solution of compound **15a** and Et_3N (3 mL) in DCM (40 mL) was added MsCl (0.6 mL, 7.5 mmol) dropwise during 10 min at -78 °C. The reaction was stirred at the same temperature for 40 min, and then quenched with water (20 mL), extracted with DCM (30 mL x 2). The combined organic phase was washed with brine (40 mL x 2), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 7:1-5:1) to afford **15b** (1.8 g, 92%) as a white solid. mp. 109.5-110.7 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ = 5.15 (dt, *J* = 5.0, 11.5 Hz, 1H), 5.15 (dt, *J* = 5.1, 11.3 Hz, 1H), 4.37 (d, *J* = 6.4 Hz, 1H), 4.33 (d, *J* = 6.5 Hz, 1H), 3.50-3.44 (m, 6H), 3.12 (s, 3H), 2.99-2.94 (m, 1H), 2.32-2.30 (m, 1H), 2.27 (s, 3H), 2.01 (dd, *J* = 4.6, 11.6 Hz, 1H), 1.85-1.77 (m, 1H), 1.75 (s, 3H), 1.66-1.53 (m, 3H), 1.43-1.29 (m, 2H), 1.20 (d, *J* = 7.6 Hz, 3H), 0.65 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 169.69, 110.93, 96.68, 76.88, 68.60, 64.29, 64.17, 55.03, 53.99, 49.58, 39.50, 38.87, 36.87, 36.71, 36.38, 26.59, 20.76, 16.10, 12.06 ppm; **IR** (neat) 2944, 2879, 1735, 1451, 1342, 1244, 1174, 1150, 1100, 1032, 931, 903 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{20}H_{34}O_9SNa$ $[M+Na]^+$ 473.1816, found 473.1810.

Synthesis of compound 16



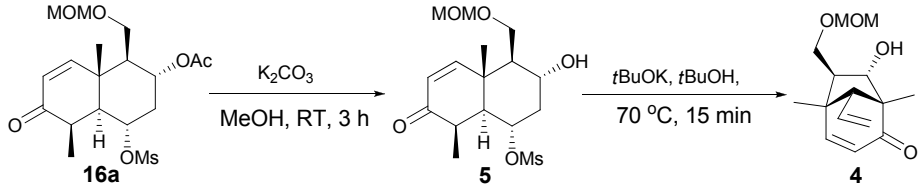
A solution of compound **15b** (1.7 g, 3.8 mmol) and PPTS (100 mg, 0.4 mmol) in acetone/water (30 mL, 9/1) was stirred under reflux for 24 h. Cooled to rt, the reaction mixture was partitioned between EA (100mL) and water (50 mL), washed with saturated $NaHCO_3$ (50 mL) and brine (50 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:2) to afford **16** (1.3 g, 85%) as a white solid. mp. 118.5-121.2 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ = 5.07 (dt, *J* = 5.0, 11.5 Hz, 1H), 4.80 (dt, *J* = 5.1, 11.2 Hz, 1H), 4.34 (d, *J* = 6.4 Hz, 1H), 4.30 (d, *J* = 6.5 Hz, 1H), 3.38-3.31 (m, 2H), 3.11 (s, 3H), 3.05-2.99 (m, 1H), 2.76-2.69 (m, 1H), 2.20-2.11 (m, 1H), 2.15 (s, 3H), 2.03-1.99 (m, 1H), 1.72 (s, 3H), 1.60-1.53 (m, 2H), 1.34 (dd, *J* = 5.8, 11.4 Hz, 1H), 1.06 (d, *J* = 7.7 Hz, 3H), 1.03-0.95 (m, 2H), 0.50 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 210.87, 169.47, 96.72, 74.97, 68.07, 64.10, 55.11, 53.31, 50.75, 43.70, 39.32, 38.62, 37.96, 36.54, 33.80, 20.66, 15.50, 13.44 ppm; **IR** (neat) 2936, 2891, 1732, 1709, 1336, 1242, 1172, 1151, 1045, 931, 905 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{18}H_{30}O_8SNa$ $[M+Na]^+$ 429.1554, found 429.1541; **Anal.** Calcd for $C_{18}H_{30}O_8S$: C, 53.19; H, 7.44; found C, 53.55; H, 7.20.

Synthesis of compound 16a



To a stirred solution of compound **16** (1.3 g, 3.2 mmol) in EA (35 mL) at 0 °C was added PhSeCl (0.6 g, 4.8 mmol) and *p*TsOH•H_2O (67 mg, 0.35 mmol). The reaction was stirred at the same temperature for 2.5 h, and then diluted with DCM (35 mL), followed by addition of pyridine (1.5 mL) and H_2O_2 (30%, 1.5 mL). The stirring continued at 0 °C for 45 min, and aqueous $CuSO_4$ (10%, 15 mL) was added. The resultant mixture was stirred for additional 10 min, diluted with water (50 mL), extracted with DCM (50 mL x 2). The combined organic phase was washed with saturated $Na_2S_2O_3$ (50 mL), 5% HCl (50 mL) and brine (50 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:2) to afford **16a** (0.92 g, 71%) as a white solid. mp 93.8-95.4 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.77 (d, *J* = 10.4 Hz, 1H), 5.86 (d, *J* = 10.4 Hz, 1H), 4.97 (dt, *J* = 5.2, 11.4 Hz, 1H), 4.84 (dt, *J* = 5.1, 11.2 Hz, 1H), 4.33 (d, *J* = 6.5 Hz, 1H), 4.28 (d, *J* = 6.5 Hz, 1H), 3.41 (d, *J* = 4.2 Hz, 2H), 3.09 (s, 3H), 3.03-2.97 (m, 1H), 2.82-2.74 (m, 1H), 2.20 (s, 3H), 1.76-1.72 (m, 1H), 1.69 (s, 3H), 1.54-1.46 (m, 1H), 1.40-1.35 (m, 1H), 1.21 (d, *J* = 7.8 Hz, 3H), 0.57 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 199.81, 169.49, 154.48, 126.65, 96.60, 74.15, 67.79, 63.93, 55.20, 49.75, 48.92, 39.76, 39.19, 39.12, 38.47, 20.59, 17.64, 13.83 ppm; **IR** (neat) 2938, 2890, 1736, 1676, 1338, 1240, 1176, 1031, 936 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{18}H_{29}O_8S$ $[M+H]^+$ 405.1578, found 405.1611; **Anal.** Calcd for $C_{18}H_{28}O_8S$: C, 53.45; H, 6.98; found C, 53.74; H, 6.88.

Synthesis of compound 4

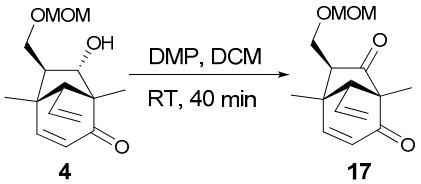


To a solution of compound **16a** (800 mg, 2 mmol) in MeOH (20 mL) was added K_2CO_3 (200 mg). The reaction was stirred for 3 h at rt, and then partitioned between EA (50 mL) and water (20 mL), extracted with EA (20 mL x 2). The combined organic phase was washed with brine (20 mL), dried over Na_2SO_4, evaporated and dried under vacuum to give **5** as a white solid (700 mg, 98%), which was used directly for the next process.

To a stirred solution of compound **5** in *t*BuOH (20 mL) was added *t*BuOK (2.4 g, 21 mmol) rapidly. The reaction was stirred at 70 °C for 15 min, and then cooled at an ice bath, quenched with saturated NH_4Cl (20 mL), partitioned between EA (50 mL) and water (20 mL). The aqueous phase was extracted with EA (50 mL), and the combined organic phase was washed with brine (20 mL x 2), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:1-2:1) to afford **4** (0.32 g, 61%) as a white semi-solid. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.33 (dd,

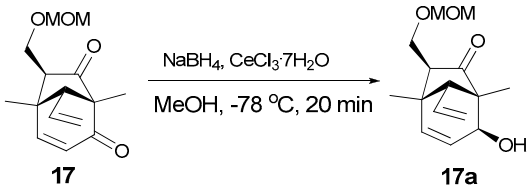
J = 1.8, 9.6 Hz, 1H), 6.04 (d, *J* = 9.6 Hz, 1H), 5.58 (td, *J* = 10.2, 16.7 Hz, 1H), 4.98-4.92 (m, 2H), 4.43 (s, 2H), 4.06 (brs, 1H), 3.58 (dd, *J* = 5.4, 10.0 Hz, 1H), 3.48-3.44 (m, *2*H), 3.16 (s, 3H), 2.37 (d, *J* = 9.6 Hz, 1H), 2.06 (dd, *J* = 5.0, 9.1 Hz, 1H), 1.38 (s, 3H), 0.88 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 200.78, 158.44, 134.07, 128.34, 120.16, 96.93, 81.94, 67.80, 64.26, 60.89, 55.36, 54.86, 46.09, 18.67, 18.45 ppm; **IR** (neat) 2964, 2930, 1668, 1455, 1382, 1150, 1110, 1061, 919 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{15}H_{22}O_4Na$ $[M+Na]^+$ 289.1410, found 289.1429.

Synthesis of compound 17



To a solution of compound **4** (300 mg, 1.1 mmol) in DCM (15 mL) was added DMP (700 mg, 1.7 mmol). The reaction was stirred at rt for 40 min, and then was quenched with saturated $Na_2S_2O_3$ (10 mL), extracted with DCM (20 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (20 mL) and brine (20 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 4:1) to afford **17** (268 mg, 90%) as a colorless oil. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.03 (dd, *J* = 2.2, 9.6 Hz, 1H), 5.72-5.62 (m, 2H), 5.05-4.99 (m, 2H), 4.21 (s, 2H), 3.80 (dd, *J* = 4.1, 10.1 Hz, 1H), 3.31 (dd, *J* = 2.4, 10.2 Hz, 1H), 3.11 (dd, *J* = 2.0, 9.4 Hz, 1H), 3.04 (s, 3H), 1.92 (dd, *J* = 2.4, 4.0 Hz, 1H), 1.45 (s, 3H), 0.94 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 210.67, 192.45, 157.85, 132.70, 127.06, 121.43, 96.51, 69.47, 65.25, 64.52, 55.28, 54.97, 44.01, 17.85, 13.79 ppm; **IR** (neat) 2932, 2884, 1747, 1681, 1450, 1373, 1151, 1112, 1033, 920 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{15}H_{20}O_4Na$ $[M+Na]^+$ 287.1254, found 287.1254.

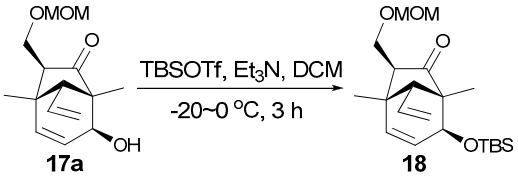
Synthesis of compound 17a



To a stirred solution of compound **17** (400 mg, 1.5 mmol) and $CeCl_3$•$7H_2O$ (840 mg, 2.3 mmol) in MeOH (15 mL) at -78 °C was added $NaBH_4$ (60 mg, 1.5 mmol) in portions during 15 min, and the stirring continued for 5 min at the same temperature. The reaction was quenched with saturated NH_4Cl (10 mL), extracted with EA (30mL x 2). The combined organic phase was washed with brine (20 mL x 2), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 4:1-1:1) to afford **17a** (181 mg, 45%) as a colorless oil. Other generated isomers were mixed and oxidized to the starting material diketone, which was used again for this reduction. After three recycles, the desired alcohol **17a** (314 mg, total 78%) was obtained. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.17 (td, *J* = 9.9 , 17.1 Hz, 1H), 5.58 (dd, *J* = 3.7, 9.6 Hz, 1H), 5.46 (d, *J* = 9.5 Hz, 1H), 5.13 (dd, *J* = 2.2,

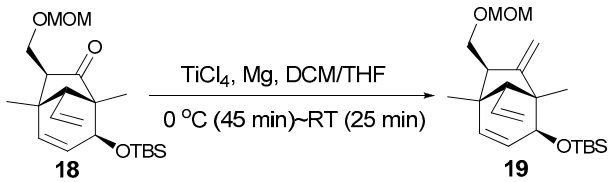
17.1 Hz, 1H), 5.06 (dd, *J* = 2.4 , 10.2 Hz, 1H), 4.25 (s, 2H), 3.74 (dd, *J* = 4.6, 10.0 Hz, 1H), 3.66-3.64 (m, 1H), 3.34 (dd, *J* = 2.5, 10.0 Hz, 1H), 3.08 (s, 3H), 2.82 (d, *J* = 9.7 Hz, 1H), 2.01 (dd, *J* = 2.5, 4.6 Hz, 1H), 1.41 (d, *J* = 7.2 Hz, 1H), 1.29 (s, 3H), 1.04 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 219.71, 140.82, 135.90, 127.82, 118.55, 96.49, 70.63, 65.68, 58.94, 57.10, 56.24, 55.18, 42.72, 18.37, 16.46 ppm; **IR** (neat) 2962, 2929, 2874, 1738, 1451, 1373, 1150, 1106, 1049, 1013, 917 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{15}H_{22}O_4Na$ $[M+Na]^+$ 289.1410, found 289.1405.

Synthesis of compound 18



To a stirred solution of compound **17a** (330 mg, 1.2 mmol) and Et_3N (0.7 mL, 5.0 mmol) in DCM (10 mL) at ice-salt bath was added TBSOTf (0.8 mL, 3.5 mmol) dropwise during 5 min. The reaction was allowed to warm to 0 °C and stirred under argon atmosphere for 3 h, and then quenched with saturated $NaHCO_3$ (5 mL).The aqueous phase was extracted with DCM (20 mL), and the combined organic phase was washed with brine (20 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 40:1) to afford **18** (424 mg, 90%) as a colorless oil. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.40 (td, *J* = 10.3 , 16.6 Hz, 1H), 5.51 (s, 2H), 5.15 (dd, *J* = 2.3, 8.5 Hz, 1H), 5.12 (s, 1H), 4.28 (s, 2H), 3.83 (s, 1H), 3.78 (dd, *J* = 4.8, 10.0 Hz, 1H), 3.42 (dd, *J* = 2.4, 10.0 Hz, 1H), 3.11 (s, 3H), 2.85 (d, *J* = 10.0 Hz, 1H), 2.10 (dd, *J* = 2.4, 4.7 Hz, 1H), 1.34 (s, 3H), 1.11 (s, 3H), 0.94 (s, 9H), -0.01(s, 3H), -0.04 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 219.83, 140.49, 136.89, 127.82, 117.06, 96.56, 70.80, 65.75, 59.29, 57.18, 55.20, 42.87, 25.92, 18.48, 18.23, 17.34, 1.41, -4.35, -5.00 ppm; **IR** (neat) 2957, 2930, 2885, 2858, 1742, 1691, 1473, 1463, 1253, 1151, 1066, 883, 777 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{21}H_{36}O_4SiNa$ $[M+Na]^+$ 403.2275, found 403.2297.

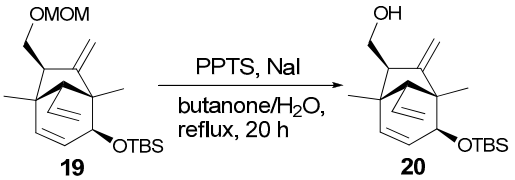
Synthesis of compound 19



To a stirred mixture of Mg (192 mg, 8 mmol) and $TiCl_4$ (0.21 mL, 2 mmol) in DCM (4 mL) at 0 °C was added a solution of compound **18** (380 mg, 1 mmol) in DCM/THF (3 mL/2 mL) dropwise during 2 min. The reaction was stirred for 45 min at 0 °C under argon atmosphere, and the resulting green-black mixture was stirred for additional 25 min at rt. The reaction mixture was re-cooled to 0 °C, and then quenched with saturated K_2CO_3 (2 mL), diluted with EA (50 mL), filtered through Celite. The filtrate was washed with saturated $NaHCO_3$ (20 mL) and brine (20 mL), dried over Na_2SO_4, evaporated to give a residue, which was purified by a flash

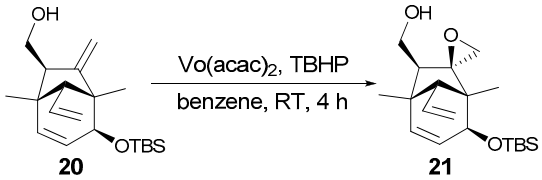
column (silica gel, Hex/EA = 40:1-20:1) to afford **19** (291 mg, 77%) as a colorless oil. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.37 (td, *J* = 10.1, 17.0 Hz, 1H), 5.58 (s, 2H), 5.16-5.06 (m, 4H), 4.45 (s, 2H), 3.70 (s, 1H), 3.58 (dd, *J* = 6.8, 10.0 Hz, 1H), 3.43 (dd, *J* = 4.7, 10.2 Hz, 1H), 3.18 (s, 3H), 2.61-2.59 (m, 1H), 2.29 (d, *J* = 10.0 Hz, 1H), 1.29 (s, 3H), 1.14 (s, 3H), 0.99 (s, 9H), 0.06(s, 3H), 0.03 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 159.58, 139.47, 138.20, 127.82, 116.19, 110.00, 96.81, 75.73, 70.10, 58.28, 55.15, 54.76, 51.11, 44.54, 26.03, 21.40, 19.48, 18.29, -4.07, -4.88 ppm; **IR** (neat) 2956, 2929, 2858, 1635, 1251, 1151, 1112, 1064, 1042, 886, 836, 774 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{22}H_{38}O_3SiNa$ $[M+Na]^+$ 401.2482, found 401.2501.

Synthesis of compound 20



To a solution of compound **19** (240 mg, 0.63 mmol) in butanone/water (5mL/0.5 mL) was added NaI (286 mg, 1.9 mmol) and PPTS (240 mg, 0.95 mmol). The reaction was stirred under reflux for 20 h, and then cooled to rt, partitioned between EA (20 mL) and water (20 mL), extracted with EA (10 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (10 mL) and brine (10 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA =20:1-10:1) to afford **20** (119 mg, 56%) as a colorless oil. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.33 (td, *J* = 10.1, 17.0 Hz, 1H), 5.57-5.50 (m, 2H), 5.13-5.01 (m, 4H), 3.66 (d, *J* = 3.1 Hz, 1H), 3.51-3.45 (m, 1H), 3.33-3.28 (m, 1H), 2.35-2.32 (m, 1H), 2.21 (d, *J* = 10.0 Hz, 1H), 1.23 (s, 3H), 1.03 (s, 3H), 0.99 (s, 9H), 0.05(s, 3H), 0.02 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 159.48, 139.45, 138.04, 127.63, 116.23, 109.83, 75.78, 63.92, 58.26, 51.21, 44.45, 26.01, 21.26, 19.14, 18.28, -4.08, -4.90 ppm; **IR** (neat) 2957, 2929, 2857, 1635, 1251, 1060, 885, 836, 774 cm^{-1}; **HRMS** (EI) *m/z* Calcd for $C_{20}H_{34}O_2Si$ $[M]^+$ 334.2323, found 334.2324.

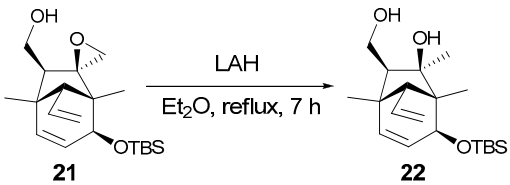
Synthesis of compound 21



To a stirred solution of compound **20** (100 mg, 0.3 mmol) and $VO(acac)_2$ (1.6 mg, 0.006 mmol) in benzene (3 mL) was added TBHP (5.5 M in decane, 66 μL, 0.36 mmol) at rt. The reaction was stirred for 4 h, and then diluted with EA (20 mL). The solution was washed with saturated $NaHCO_3$ (10 mL) and brine (10 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA =15:1) to afford **21** (90 mg, 86%) as a colorless oil. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.20 (td, *J* = 10.1, 17.0 Hz, 1H), 5.50-5.43 (m, 2H), 5.08-5.00 (m, 2H), 3.71-3.65 (m, 1H), 3.53 (d, *J* = 3.4 Hz, 1H), 3.50-3.44 (m, 1H), 2.46 (dd, *J* = 5.6,

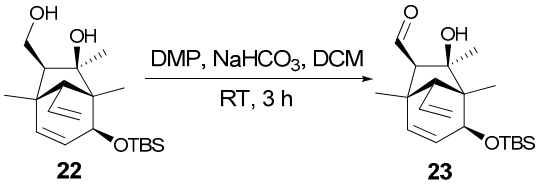
8.3 Hz, 1H), 2.41 (d, J = 10.0 Hz, 1H), 2.33 (d, J = 4.5 Hz, 1H), 2.28 (d, J = 4.5 Hz, 1H), 1.82 (dd, J = 3.9, 7.5 Hz, 1H), 0.95 (s, 9H), 0.94 (s, 3H), 0.93 (s, 3H), 0.01(s, 3H), -0.02 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 139.59, 137.32, 126.69, 116.77, 72.84, 69.17, 61.46, 58.10, 53.26, 48.67, 48.52, 44.15, 25.95, 19.09, 18.24, 16.09, -4.03, -4.97 ppm; **IR** (neat) 2958, 2929, 1635, 1256, 1063, 837, 774 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{20}H_{34}O_3SiNa$ $[M+Na]^+$ 373.2169, found 373.2170.

Synthesis of compound 22



To a stirred solution of compound **21** (80 mg, 0.23 mmol) in Et_2O (8 mL) was added LAH (43 mg, 1.1 mmol) at rt. The reaction was stirred under reflux for 7 h. After being cooled to 0 °C, the reaction was quenched with Na_2SO_4•10 H_2O (5 g) in portions and stirred for additional 2 h at rt, filtered. The filtered cake was washed with EA (10 mL x 3), and the combined filtrate was evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA =10:1-5:1) to afford **22** (72 mg, 90%) as a white solid. mp 91.0-92.0 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.27 (td, J = 10.2, 16.9 Hz, 1H), 5.50 (dd, J = 3.6, 9.4 Hz, 1H), 5.45 (dd, J = 1.2, 9.6 Hz, 1H), 5.10-5.04 (m, 2H), 3.79 (d, J = 3.5 Hz, 1H), 3.64-3.58 (m, 1H), 3.55-3.50 (m, 1H), 2.48 (d, J = 10.1 Hz, 1H), 2.12 (s, 1H), 1.88 (t, J = 5.3 Hz, 1H), 1.62 (dd, J = 4.3, 9.0 Hz, 1H), 1.14 (s, 3H), 1.07 (s, 3H), 1.00 (s, 9H), 0.89 (s, 3H), 0.07 (s, 3H), 0.06 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 140.63, 138.50, 126.06, 115.91, 81.76, 70.47, 61.84, 59.31, 57.59, 53.59, 43.76, 42.48, 26.05, 19.27, 18.31, 16.14, -3.97, -4.88 ppm; **IR** (neat) 2958, 2929, 2857, 2886, 1462, 1374, 1253, 1048, 1022, 836, 774 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{20}H_{36}O_3SiNa$ $[M+Na]^+$ 375.2326, found 375.2337.

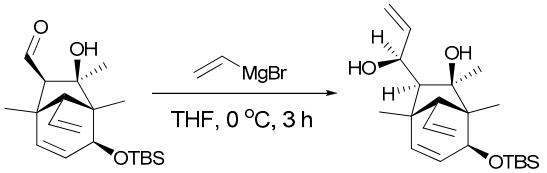
Synthesis of compound 23



To a solution of compound **22** (60 mg, 0.17 mmol) in DCM (9 mL) was added DMP (144 mg, 0.34 mmol) and $NaHCO_3$ (71 mg, 0.85 mmol). The reaction was stirred at rt for 3 h, and then quenched with saturated $NaHCO_3$ (10 mL), extracted with DCM (20 mL x 2). The combined organic phase was washed with $NaHCO_3$ (20 mL) and brine (20 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 8:1) to afford **23** (53 mg, 88%) as a white solid. mp 108.5-109.7 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ = 9.70 (d, J = 5.3 Hz, 1H), 6.18 (td, J = 10.1, 17.0 Hz, 1H), 5.39 (dd, J = 3.9, 9.4 Hz, 1H), 5.25(d, J = 9.4 Hz, 1H), 5.06 (dd, J = 2.4, 10.2 Hz, 1H), 5.02 (dd, J = 2.4, 17.1 Hz, 1H), 3.63 (d, J = 3.8 Hz, 1H), 2.69 (d, J = 10.0 Hz, 1H), 2.25 (d, J = 5.3 Hz, 1H), 1.07 (s, 1H), 0.96 (s, 12H), 0.89 (s,

3H), 0.86 (s, 3H), 0.01 (s, 3H), 0.00 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 201.08, 138.66, 137.20, 127.87, 116.59, 82.76, 70.05, 69.84, 58.40, 53.80, 44.50, 26.38, 25.97, 18.69, 18.24, 15.91, -4.06, -4.99 ppm; **IR** (neat) 2958, 2929, 2855, 1710, 1374, 1251, 1055, 881,772 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{20}H_{34}O_3SiNa$ $[M+Na]^+$ 373.2169, found 373.2193.

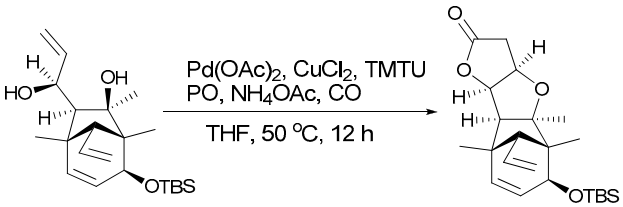
Synthesis of compound 24



23 **24**

To a stirred solution of compound **23** (25 mg, 0.071 mmol) in THF (5 mL) at 0 °C was added vinyl magnesium bromide (0.18 mL, 1M; freshly prepared following typical method) dropwise during 5 min. The reaction was stirred for 3 h at 0 °C under argon atmosphere, and then quenched with saturated NH_4Cl (5 mL), extracted with EA (10 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (10 mL) and brine (10 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 10:1) to afford **24** (13 mg, 48%) as a white semi-solid. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.31 (td, *J* = 10.1, 17.0 Hz, 1H), 6.14-6.05 (m, 1H), 5.50 (s, 2H), 5.20-5.11 (m, 3H), 5.00 (td, *J* = 1.5, 10.4 Hz, 1H), 4.27-4.23 (m, 1H), 3.80 (d, *J* = 1.8 Hz, 1H), 2.96 (d, *J* = 8.2 Hz, 1H), 2.75 (d, *J* = 10.1 Hz, 1H), 2.22 (s, 1H), 1.62 (d, *J* = 3.3 Hz, 1H), 1.23 (s, 3H), 1.13 (s, 3H), 1.07 (s, 3H), 1.00 (s, 9H), 0.07 (s, 6H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 143.20, 141.50, 138.74, 125.95, 84.35, 74.19, 70.44, 62.27, 58.11, 53.90, 44.91, 30.09, 27.20, 26.05, 25.98, 20.25, 18.31, 16.28, -3.96, -4.86 ppm; **IR** (neat) 2956, 2929, 2857, 1636, 1462, 1256, 1050, 836, 773 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{22}H_{38}O_3SiNa$ $[M+Na]^+$ 401.2482, found 401.2492.

Synthesis of compound 25

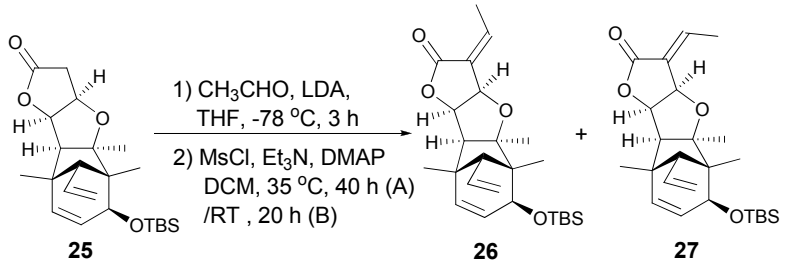


24 **25**

To a stirred mixture of $Pd(OAc)_2$ (1 mg, 0.005 mmol) and $CuCl_2$ (17 mg, 0.13 mmol) in THF (0.5 mL) was added 1,1,3,3-tetramethylthiourea (TMTU) (0.7 mg, 0.005 mmol) at rt, and the formed mixture was stirred for 30 min under argon atmosphere. To this solution was added propene oxide (PO) (0.018 mL, 0.25 mmol) and NH_4OAc (4 mg, 0.005 mmol), and the mixture was purged with CO, followed by dropwise addition of enediol **24** (19 mg, 0.05 mmol) in THF (0.1 mL) during 2 min, and the reaction was stirred at 50 °C under CO atmosphere for 12 h. After being cooled to rt, the reaction mixture was diluted with EA (20 mL), filtered through Celite. The filtrate was washed with saturated NH_4Cl (10 mL) and brine (10 mL), dried over Na_2SO_4,

and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 10:1-5:1) to afford **25** (16 mg, 78%) as a white semi-solid. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.22 (td, *J* = 10.0, 17.1 Hz, 1H), 5.53 (dd, *J* = 4.0, 9.5 Hz, 1H), 5.40 (d, *J* = 9.5 Hz, 1H), 5.22 (dd, *J* = 2.2, 17.1 Hz, 1H), 5.08 (dd, *J* = 2.4, 10.0 Hz, 1H), 3.90 (dd, *J* = 3.0, 6.6 Hz, 1H), 3.78 (d, *J* = 3.9 Hz, 1H), 3.65 (dd, *J* = 3.2, 4.4 Hz, 1H), 2.62 (d, *J* = 10.1 Hz, 1H), 2.28 (d, *J* = 17.6 Hz, 1H), 1.74 (dd, *J* = 4.5, 17.6 Hz, 1H), 1.59 (d, *J* = 6.6 Hz, 1H), 1.30 (s, 3H), 1.26 (s, 3H), 0.97 (s, 9H), 0.87 (s, 3H), 0.05 (s, 3H), 0.04 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 173.87, 139.66, 137.14, 126.86, 116.86, 94.71, 85.29, 78.10, 70.09, 62.78, 57.78, 51.87, 44.98, 34.51, 25.98, 20.64, 19.89, 18.29, 16.16, -3.98, -4.93 ppm; **IR** (neat) 2957, 2928, 2857, 1783, 1635, 1258, 1059, 774 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{23}H_{36}O_4SiNa$ $[M+Na]^+$ 427.2275, found 427.2271.

Synthesis of compound 26 and 27



To a solution of compound **25** (10 mg, 0.025 mmol) in THF (5 mL) was added LDA (0.15 mL, 1 M in THF, 0.15 mmol, freshly prepared following typical method) at -78 °C. The resultant mixture was stirred at the same temperature for 20 min, and acetaldehyde (10 μL, 0.17 mmol) was added. The reaction was stirred at -78 °C for 3 h, and then quenched with saturated NH_4Cl (5 mL), extracted with EA (10 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (10 mL) and brine (10 mL), dried over Na_2SO_4, and evaporated to give a residue, which was subjected to the next process without further purification.

Procedure A:

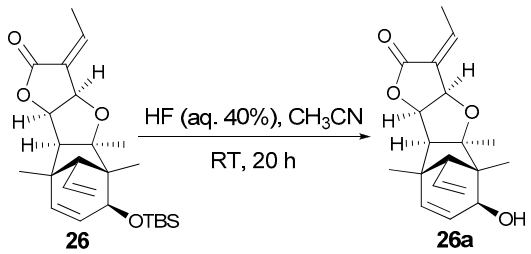
To a solution of the above crude product in DCM (5 mL) was added Et_3N (60 μL, 0.43 mmol), DMAP (1 mg) and MsCl (10 μL, 0.12 mmol). The resultant mixture was stirred at 35 °C for 40 h, and then quenched with saturated NH_4Cl (5 mL), extracted with DCM (10 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (5 mL) and brine (5 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 8:1) to afford **26** (2.7 mg, 25% two steps) and **27** (6.5 mg, 61% two steps), both as a white semi-solid.

Procedure B:

To a solution of the above crude product in DCM (0.5 mL) was added Et_3N (60 μL, 0.43 mmol), DMAP (1 mg) and MsCl (10 μL, 0.12 mmol). The resultant mixture was stirred at rt for 20 h, and then quenched with saturated NH_4Cl (5 mL), extracted with DCM (10 mL x 2). The combined organic phase was washed with saturated $NaHCO_3$ (5 mL) and brine (5 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 8:1) to afford **26** (5.5 mg, 52% two steps) and **27** (3.2 mg, 30% two steps).

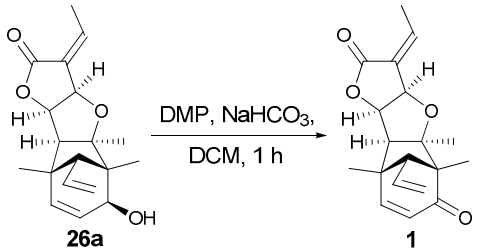
Compound **26**: **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.23 (td, *J* = 10.1, 17.1 Hz, 1H), 5.96 (q, *J* = 7.2 Hz, 1H), 5.54 (dd, *J* = 3.9, 9.5 Hz, 1H), 5.42 (d, *J* = 9.5 Hz, 1H), 5.19 (dd, *J* = 2.1, 17.0 Hz, 1H), 5.06 (dd, *J* = 2.3, 10.1 Hz, 1H), 4.14 (d, *J* = 3.2 Hz, 1H), 4.06 (dd, *J* = 3.4, 6.8 Hz, 1H), 3.80 (d, *J* = 3.8 Hz, 1H), 2.71 (d, *J* = 10.1 Hz, 1H), 1.97 (d, *J* = 7.3 Hz, 3H), 1.71 (d, *J* = 6.8 Hz, 1H), 1.36 (s, 3H), 1.30 (s, 3H), 0.97 (s, 3H), 0.95 (s, 9H), 0.05 (s, 3H), 0.02 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 167.84, 142.69, 139.78, 137.17, 127.16, 126.78, 116.80, 95.33, 83.55, 81.26, 70.14, 62.91, 57.84, 52.01, 45.16, 25.98, 21.09, 20.09, 18.28, 16.25, 14.14, -3.99, -4.93 ppm; **IR** (neat) 2957, 2929, 2857, 1761, 1686, 1258, 1130, 1117, 1053, 869, 837 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{25}H_{38}O_4SiNa$ $[M+Na]^+$ 453.2432, found 453.2430.
Compound **27**: **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.72 (dq, *J* = 0.9, 7.2 Hz, 1H), 6.22 (td, *J* = 10.0, 17.1 Hz, 1H), 5.54 (dd, *J* = 4.0, 9.5 Hz, 1H), 5.43 (d, *J* = 9.4 Hz, 1H), 5.16 (dd, *J* = 2.2, 17.0 Hz, 1H), 5.05 (dd, *J* = 2.4, 10.0 Hz, 1H), 4.39 (d, *J* = 3.0 Hz, 1H), 4.08 (dd, *J* = 3.7, 7.1 Hz, 1H), 3.79 (d, *J* = 3.9 Hz, 1H), 2.64 (d, *J* = 10.2 Hz, 1H), 1.75 (d, *J* = 7.1 Hz, 1H), 1.41 (d, *J* = 7.2 Hz, 3H), 1.38 (s, 3H), 1.26 (s, 3H), 0.96 (s, 3H), 0.95 (s, 9H), 0.05 (s, 3H), 0.02 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 168.66, 139.78, 139.74, 137.09, 128.71, 126.77, 116.83, 95.79, 84.02, 76.69, 70.08, 62.75, 57.90, 51.99, 45.18, 25.97, 21.23, 20.18, 18.27, 16.29, 15.39, -4.01, -4.95 ppm; **IR** (neat) 2957, 2929, 2857, 1766, 1693, 1056, 773 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{25}H_{38}O_4SiNa$ $[M+Na]^+$ 453.2432, found 453.2430.

Synthesis of compound 26a



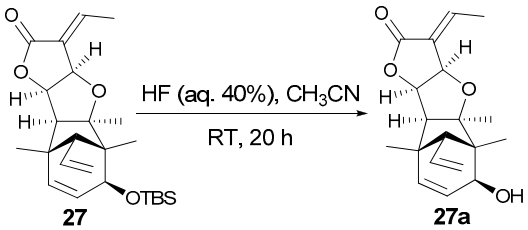
To a solution of compound **26** (5 mg, 0.012 mmol) in CH_3CN (1 mL) was added aqueous HF (aq. 40%, 0.2 mL). The reaction was stirred at rt for 20 h, and then quenched with saturated $NaHCO_3$ (2 mL), extracted with EA (5 mL x 2). The combined organic phase was washed with brine (5 mL x 2), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:1) to afford **26a** (3.3 mg, 90%) as a white semi-solid. **^{1}H NMR** (400 MHz, C_6D_6) δ = 5.97-5.82 (m, 2H), 5.59 (dd, *J* = 4.0, 9.5 Hz, 1H), 5.35 (d, *J* = 9.4 Hz, 1H), 5.12 (dd, *J* = 1.4, 17.1 Hz, 1H), 4.96 (dd, *J* = 2.0, 10.3 Hz, 1H), 4.07 (d, *J* = 3.3 Hz, 1H), 4.00 (dd, *J* = 3.4, 6.8 Hz, 1H), 3.58-3.55 (m, 1H), 2.66 (d, *J* = 9.5 Hz, 1H), 1.98 (d, *J* = 7.3 Hz, 3H), 1.64 (d, *J* = 6.7 Hz, 1H), 1.28 (s, 3H), 1.26 (s, 3H), 1.15 (d, *J* = 8.3 Hz, 1H), 0.84 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 167.82, 142.68, 139.74, 135.62, 127.37, 127.14, 119.13, 95.30, 83.53, 81.15, 69.90, 62.74, 56.30, 51.96, 45.01, 20.85, 19.98, 15.43, 14.13 ppm; **IR** (neat) 2961, 2925, 1755, 1684, 1374, 1215, 1131, 1031, 1011, 965 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{19}H_{24}O_4Na$ $[M+Na]^+$ 339.1567, found 339.1568.

Synthesis of pallambin C (1)



To a solution of compound **26a** (3 mg, 0.0095 mmol) in DCM (0.5 mL) was added DMP (8 mg, 0.019 mmol) and $NaHCO_3$ (4 mg, 0.048 mmol). The reaction was stirred at rt for 1 h, and then quenched with saturated $Na_2S_2O_3$ (2 mL), extracted with DCM (5 mL x 2). The combined organic phase was washed with aqueous $NaHCO_3$ (5 mL) and brine (5 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:1) to afford **1** (2.8 mg, 95%) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 6.71-6.66 (m, 2H), 5.89 (d, *J* = 9.6 Hz, 1H), 5.54 (td, *J* = 9.8, 16.9 Hz, 1H), 5.22-5.15 (m, 2H), 4.88 (dd, *J* = 3.2, 6.7 Hz, 1H), 4.84 (d, *J* = 3.2 Hz, 1H), 2.83 (dd, *J* = 1.6, 9.5 Hz, 1H), 2.53 (d, *J* = 6.7 Hz, 1H), 2.25 (d, *J* = 7.3 Hz, 3H), 1.42 (s, 3H), 1.14 (s, 3H), 1.04 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 201.56, 168.38, 157.11, 144.82, 132.39, 127.01, 126.10, 121.78, 94.17, 83.13, 82.34, 63.98, 62.79, 60.83, 47.02, 22.43, 19.68, 14.60, 12.88 ppm; **IR** (neat) 2967, 2929, 1759, 1681, 1375, 1118, 916 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{19}H_{23}O_4$ $[M+H]^+$ 315.1591, found 315.1606.

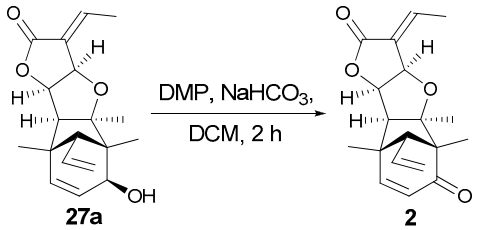
Synthesis of compound 27a



To a solution of compound **27** (5 mg, 0.012 mmol) in CH_3CN (1 mL) was added aqueous HF (aq. 40%, 0.2 mL). The reaction was stirred at rt for 20 h, and then quenched with saturated $NaHCO_3$ (2 mL), extracted with EA (5 mL x 2). The combined organic phase was washed with brine (5 mL x 2), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:1) to afford **27a** (3.3 mg, 90%) as a white semi-solid. **^{1}H NMR** (400 MHz, C_6D_6) δ = 6.72 (q, *J* = 7.2 Hz, 1H), 5.86 (td, *J* = 9.9, 17.2 Hz, 1H), 5.59 (dd, *J* = 4.0, 9.4 Hz, 1H), 5.35 (d, *J* = 9.4 Hz, 1H), 5.09 (dd, *J* = 1.4, 17.2 Hz, 1H), 4.95 (dd, *J* = 1.9, 10.2 Hz, 1H), 4.32 (d, *J* = 3.1 Hz, 1H), 4.02 (dd, *J* = 3.6, 7.0 Hz, 1H), 3.55-3.52 (m, 1H), 2.61 (d, *J* = 9.4 Hz, 1H), 1.67 (d, *J* = 7.0 Hz, 1H), 1.39 (d, *J* = 7.2 Hz, 3H), 1.31 (s, 3H), 1.23 (s, 3H), 1.15 (d, *J* = 8.3 Hz, 1H), 0.83 (s, 3H) ppm; **^{13}C NMR** (100 MHz, C_6D_6) δ = 168.64, 139.75, 135.56, 128.68, 127.33, 119.11, 95.78, 84.00, 76.58, 69.84, 62.57, 56.37, 51.94, 45.02, 20.97, 20.07, 15.48, 15.40 ppm; **IR** (neat) 2956, 2927, 1756, 1693, 1375, 1217, 1143, 1074, 1031, 738 cm^{-1}; **HRMS** (ESI) *m/z* Calcd

for $C_{19}H_{24}O_4Na$ $[M+Na]^+$ 339.1567, found 339.1568.

Synthesis of pallambin D (2)

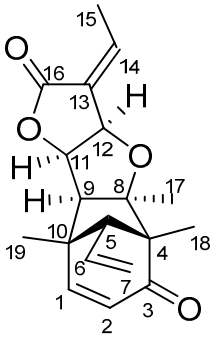


To a solution of compound **27a** (3 mg, 0.0095 mmol) in DCM (0.5 mL) was added DMP (8 mg, 0.019 mmol) and (4 mg, 0.048 mmol). The reaction was stirred at rt for 2 h, and then quenched with saturated $Na_2S_2O_3$ (2 mL), extracted with DCM (5 mL x 2). The combined organic phase was washed with aqueous $NaHCO_3$ (5 mL) and brine (5 mL), dried over Na_2SO_4, and evaporated to give a residue, which was purified by a flash column (silica gel, Hex/EA = 3:1) to afford **2** (2.6 mg, 89%) as a white solid. mp 184.5-185.7 °C (lit.[1] mp 191 °C). **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.00 (q, *J* = 7.3 Hz, 1H), 6.69 (dd, *J* = 1.9, 9.5 Hz, 1H), 5.89 (d, *J* = 9.6 Hz, 1H), 5.53 (td, *J* = 9.8, 16.9 Hz, 1H), 5.20-5.15 (m, 2H), 5.09 (d, *J* = 3.2 Hz, 1H), 4.90 (dd, *J* = 3.6, 7.0 Hz, 1H), 2.76 (dd, *J* = 1.8, 9.4 Hz, 1H), 2.56 (d, *J* = 7.0 Hz, 1H), 2.03 (d, *J* = 7.2 Hz, 3H), 1.42 (s, 3H), 1.16 (s, 3H), 1.01 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 201.61, 169.53, 157.12, 141.89, 132.37, 127.68, 126.97, 121.73, 94.51, 83.80, 77.76, 64.03, 62.79, 60.62, 47.03, 22.50, 19.78, 16.18, 12.84 ppm; **IR** (neat) 2967, 2931, 1764, 1682, 1376, 1209, 1121, 1072, 973 cm^{-1}; **HRMS** (ESI) *m/z* Calcd for $C_{19}H_{23}O_4$ $[M+H]^+$ 315.1591, found 315.1594.

[1] M. Toyota, T. Sato, Y. Asakawa, *Chem. Pharm. Bull.* **1998**, *46*, 178 – 180.

Spectral comparison between natural and synthetic

Pallambin C (1):

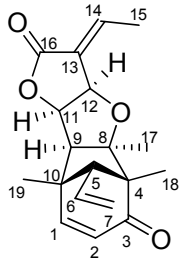


H	**Natural** (600M, $CDCl_3$, solvent signal 7.28, δ ppm)	**Synthetic** (400M, $CDCl_3$, solvent signal 7.26, δ ppm)
1	6.69 (m)	6.68 (m)
2	5.89 (d, 9.6)	5.89 (d, 9.6)
5	2.84 (dd, 9.9, 2.0)	2.83 (dd, 9.5, 1.6)
6	5.54 (dt, 17.0, 9.9)	5.54 (dt, 16.9, 9.8)
7	5.19 (2H, m)	5.18 (2H, m)
9	2.54 (d, 6.7)	2.53 (d, 6.7)
11	4.89 (dd, 6.8, 3.3)	4.88 (dd, 6.7, 3.2)
12	4.85 (d, 3.3)	4.84 (d, 3.2)
14	6.69 (1H, m)	6.67 (1H, m)
15	2.26 (3H, d, 7.3)	2.25 (3H, d, 7.3)
17	1.15 (3H, S)	1.14 (3H, S)
18	1.04 (3H, S)	1.04 (3H, S)
19	1.42 (3H, S)	1.42 (3H, S)

C	**Natural** (150M, $CDCl_3$, solvent signal 77.02, δ ppm)	**Synthetic** (100M, $CDCl_3$, solvent signal 77.16, δ ppm)
1	156.9	157.1
2	126.8	127.0
3	201.3	201.6
4	62.6	62.8
5	63.8	64.0
6	132.2	132.4
7	121.5	121.8
8	94.0	94.2
9	60.6	60.8
10	46.8	47.0
11	82.9	83.1

12	82.1	82.3
13	125.9	126.1
14	144.6	144.8
15	14.4	14.6
16	168.1	168.4
17	22.2	22.4
18	12.7	12.9
19	19.5	19.7

Pallambin D (2):



H	**Natural** (600M, $CDCl_3$, solvent signal 7.28, δ ppm)	**Synthetic** (400M, $CDCl_3$, solvent signal 7.26, δ ppm)
1	6.70 (dd, 9.6, 2.1)	6.69 (dd, 9.5, 1.9)
2	5.91 (d, 9.6)	5.89 (d, 9.6)
5	2.78 (dd, 9.9, 2.0)	2.76 (dd, 9.4, 1.8)
6	5.55 (dt, 16.9, 9.9)	5.53 (dt, 16.9, 9.8)
7	5.19 (2H, m)	5.17 (2H, m)
9	2.58 (d, 7.0)	2.56 (d, 7.0)
11	4.92 (dd, 7.0, 3.6)	4.90 (dd, 7.0, 3.6)
12	5.11 (dd, 3.5, 0.8)	5.09 (d, 3.2)
14	7.07 (qd, 7.2, 0.8)	7.00 (q, 7.3)
15	2.06 (3H, d, 7.2)	2.03 (3H, d, 7.2)
17	1.18 (3H, S)	1.16 (3H, S)
18	1.03 (3H, S)	1.01 (3H, S)
19	1.44 (3H, S)	1.42 (3H, S)

C	**Natural** (150M, $CDCl_3$, solvent signal 77.00, δ ppm)	**Synthetic** (100M, $CDCl_3$, solvent signal 77.16, δ ppm)
1	157.0	157.1
2	126.8	127.0
3	201.3	201.6
4	62.7	62.8
5	63.9	64.0
6	132.2	132.4

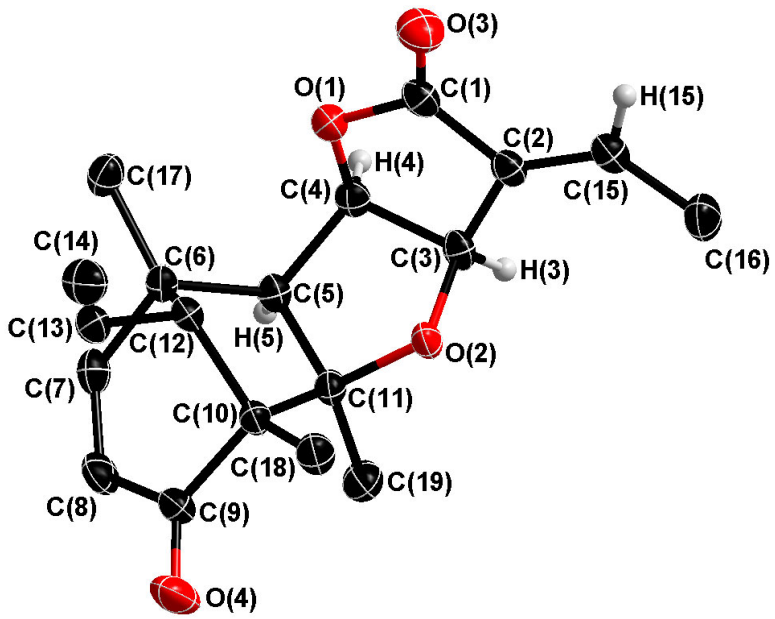
7	121.5	121.7
8	94.4	94.5
9	60.3	60.6
10	46.7	47.0
11	83.6	83.8
12	77.6	77.8
13	127.5	127.7
14	141.7	141.9
15	16.0	16.2
16	169.3	169.5
17	22.3	22.5
18	12.7	12.8
19	19.6	19.8

Basic crystal data for compounds 2, 10, 11, 16 and 22

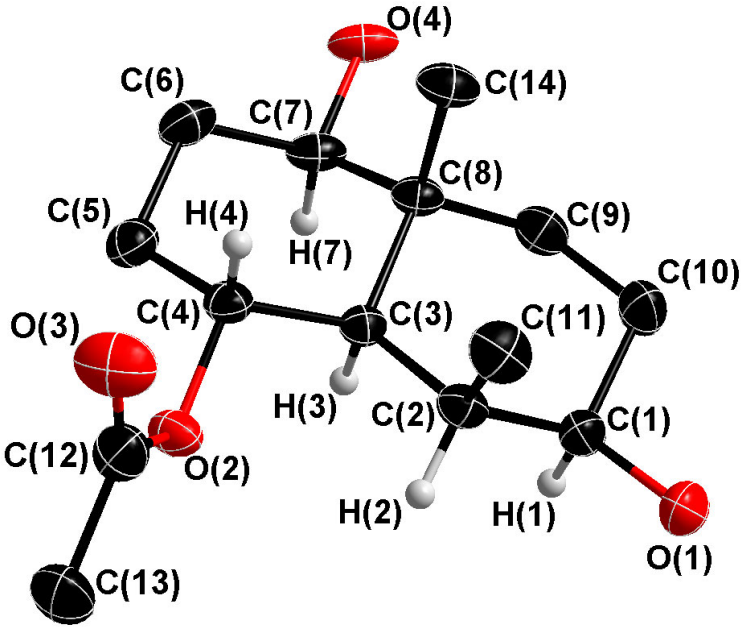
CCDC 879025 (**2**), CCDC 879026 (**10**), CCDC 879027 (**11**), CCDC 879028 (**16**) and CCDC 879029 (**22**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Chem. Commun.
Deposition Number(s) 879025 879026 879027 879028 879029
CIF files for 5 structures are attached to this message.

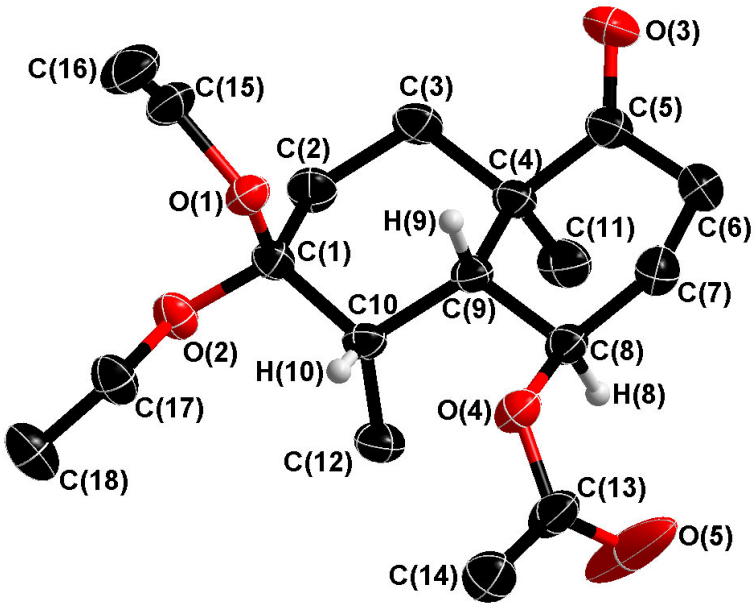
CCDC No	Acell	Bcell	Ccell	Space Gp.
879025	7.3712	8.0916	13.8262	P-1
879026	14.4194	14.4194	6.0541	P32
879027	8.3310	8.7830	13.7240	P-1
879028	14.7240	6.7900	22.3940	P21/n
879029	11.6292	7.7435	12.2967	P21



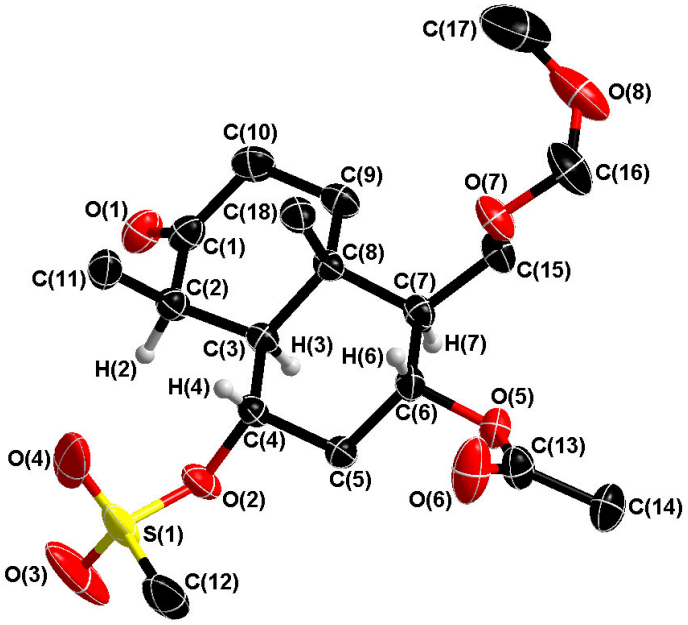
ORTEP diagram of compound **2**. Thermal ellipsoids were plotted at 30 % probability level. Hydrogen atoms are omitted for clarity.



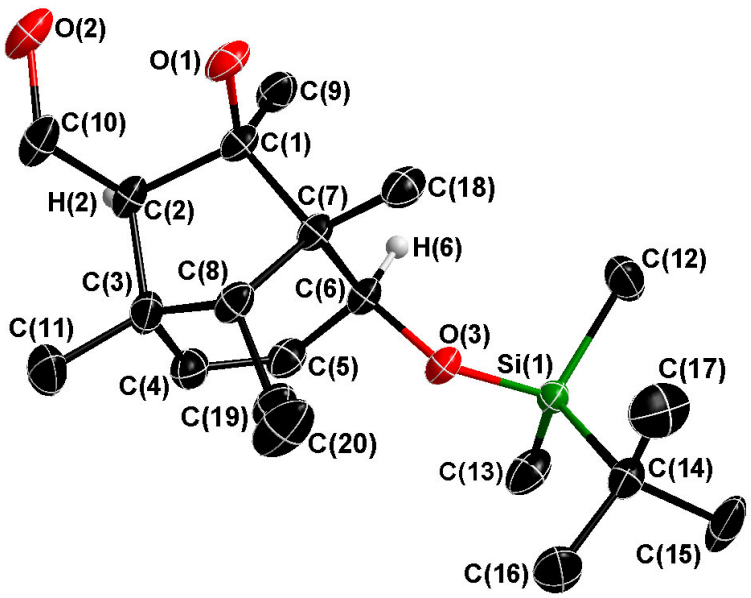
ORTEP diagram of compound **10**. Thermal ellipsoids were plotted at 30 % probability level. Hydrogen atoms are omitted for clarity.



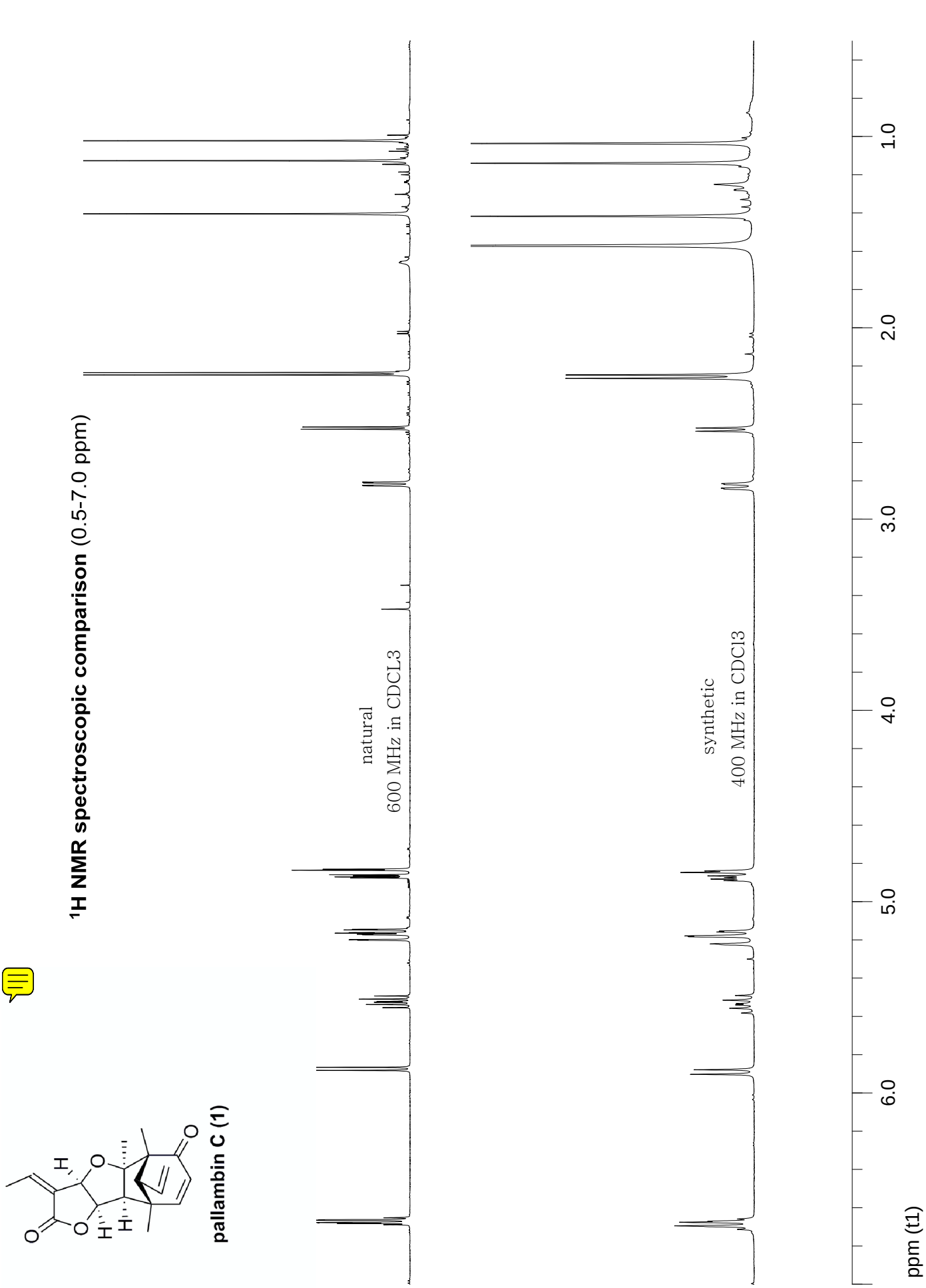
ORTEP diagram of compound **11**. Thermal ellipsoids were plotted at 30 % probability level. Hydrogen atoms are omitted for clarity.



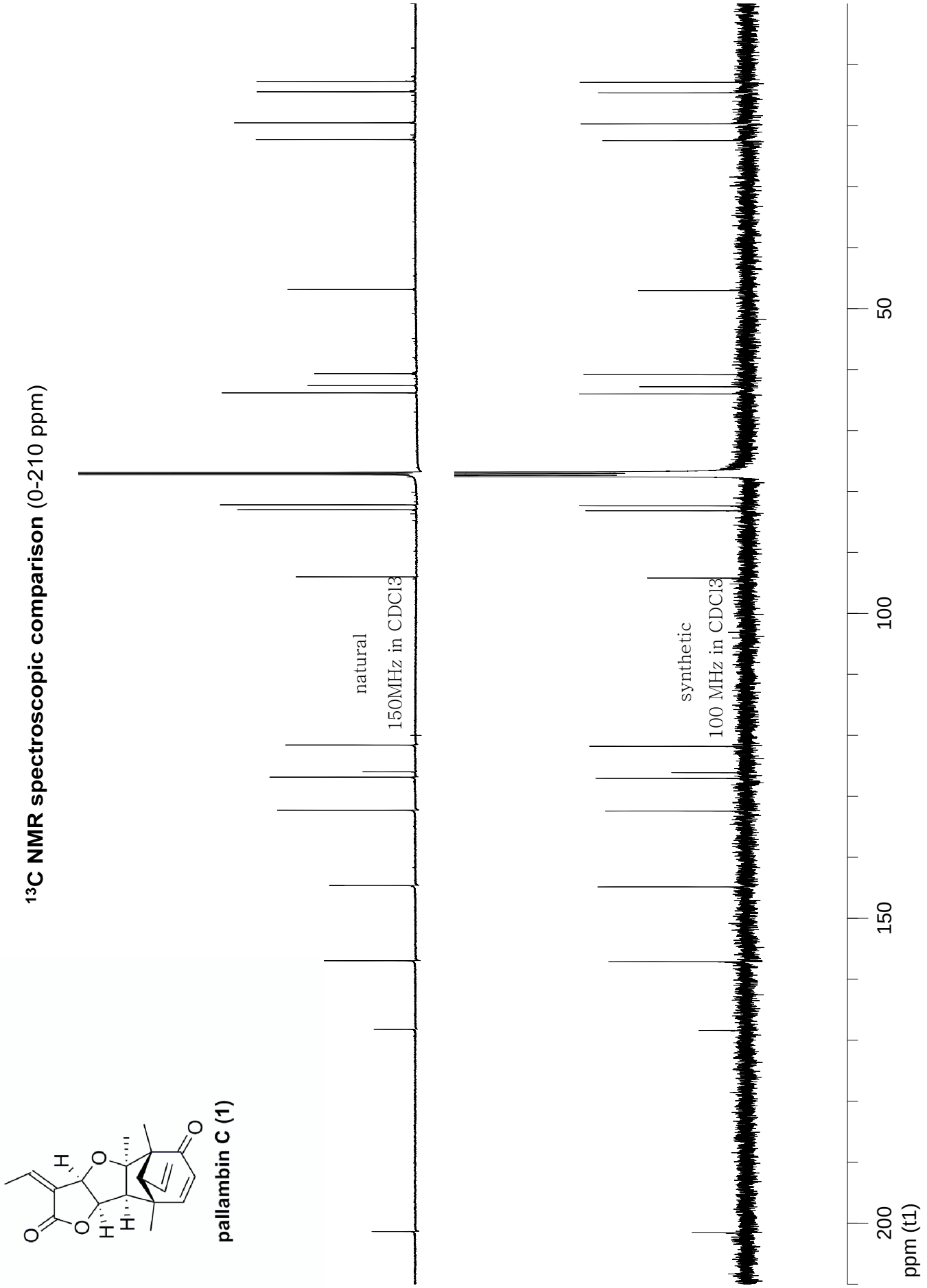
ORTEP diagram of compound **16**. Thermal ellipsoids were plotted at 30 % probability level. Hydrogen atoms are omitted for clarity.



ORTEP diagram of compound **22.** Thermal ellipsoids were plotted at 30 % probability level. Hydrogen atoms are omitted for clarity.

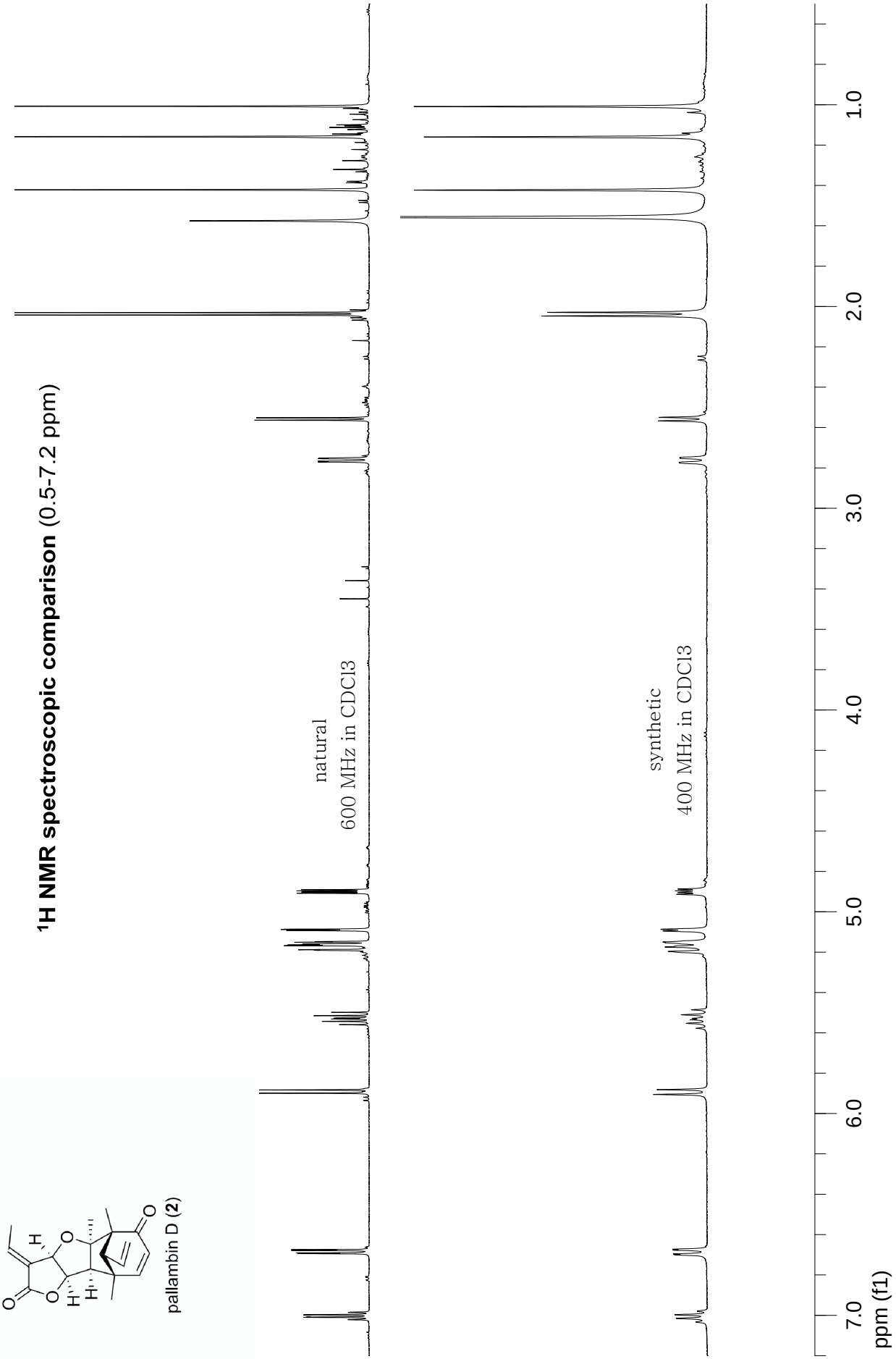

pallambin C (1)
¹H NMR spectroscopic comparison (0.5-7.0 ppm)
natural
600 MHz in CDCL3
synthetic
400 MHz in CDCl3
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

^{13}C NMR spectroscopic comparison (0-210 ppm)



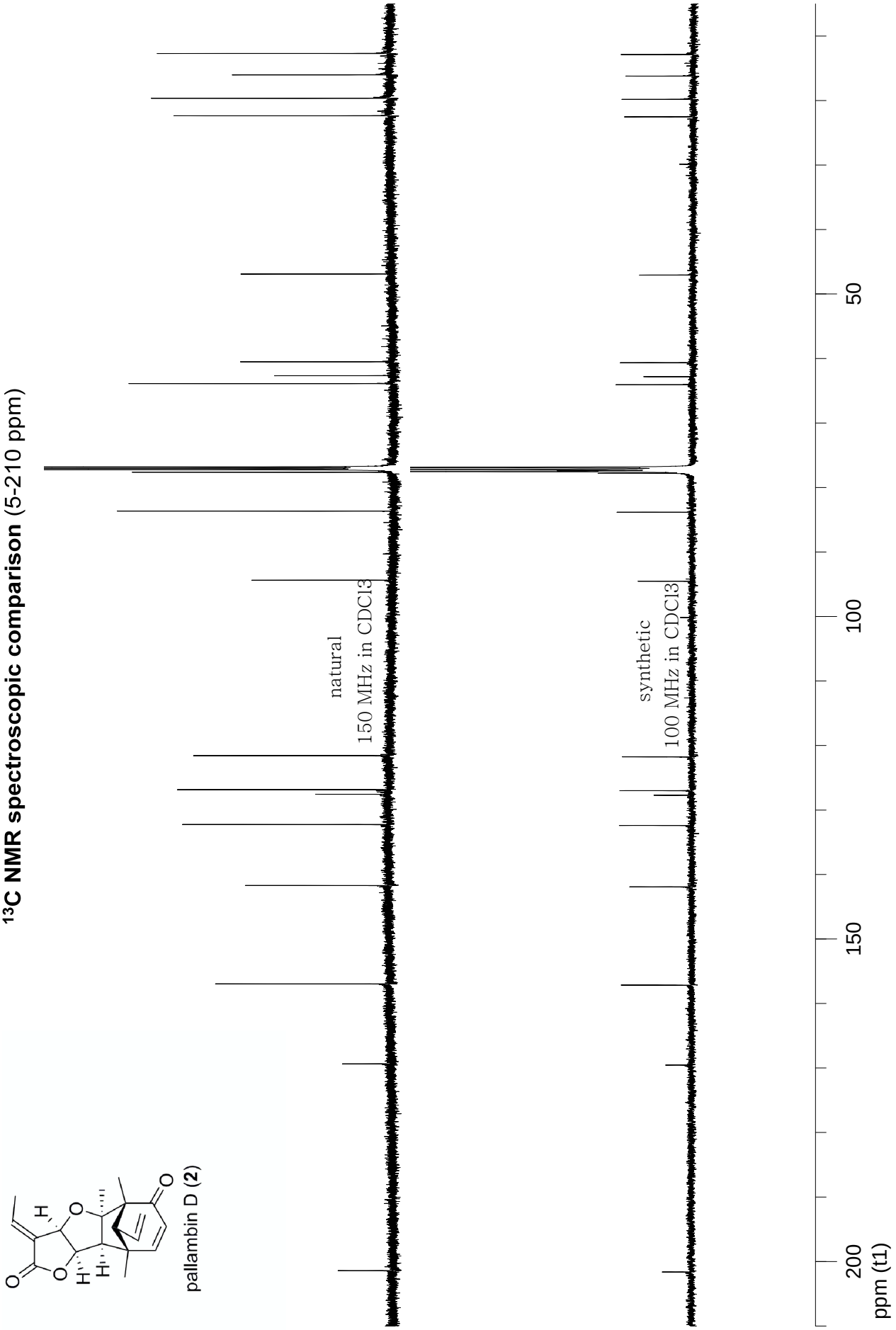
pallambin C (1)

^{1}H NMR spectroscopic comparison (0.5-7.2 ppm)

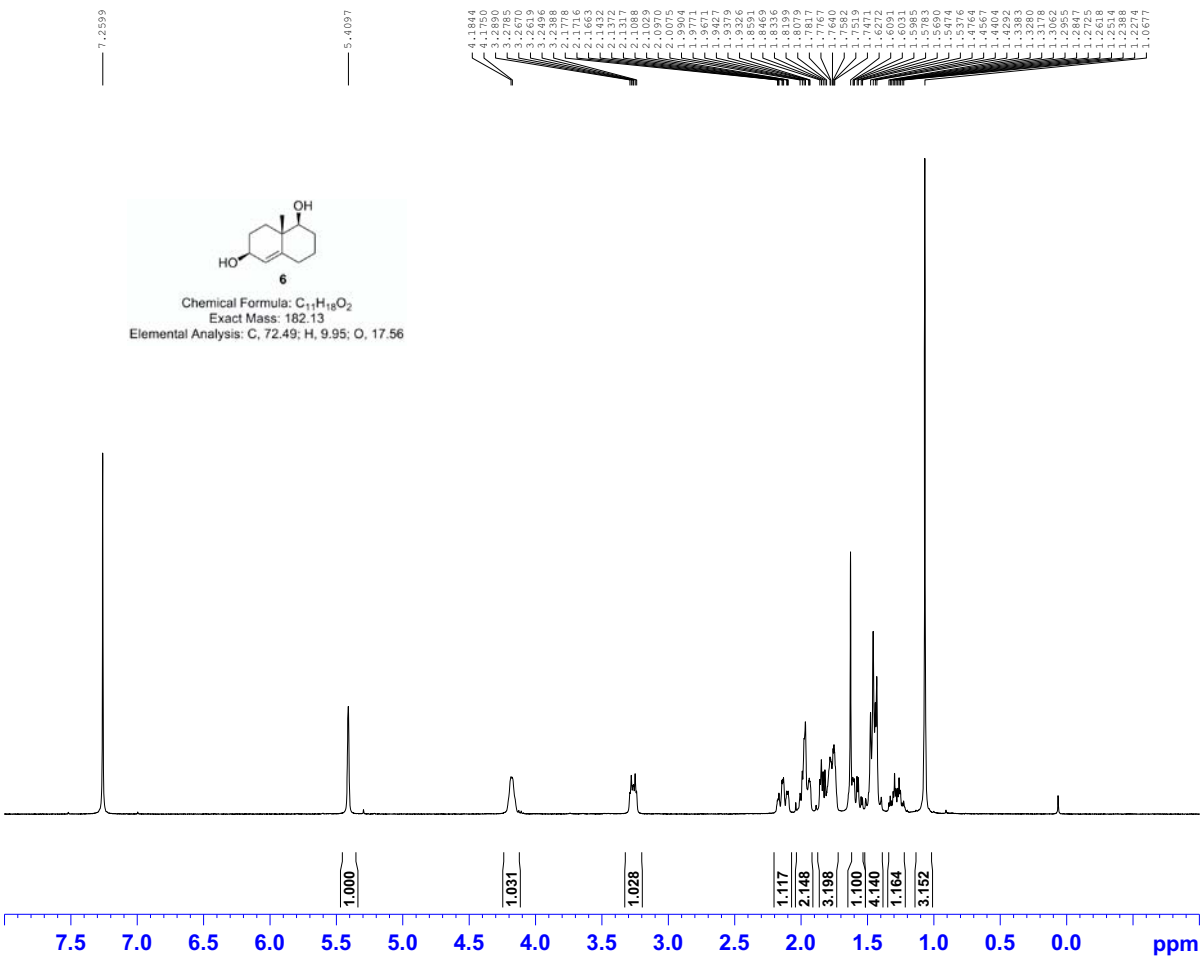


pallambin D (**2**)

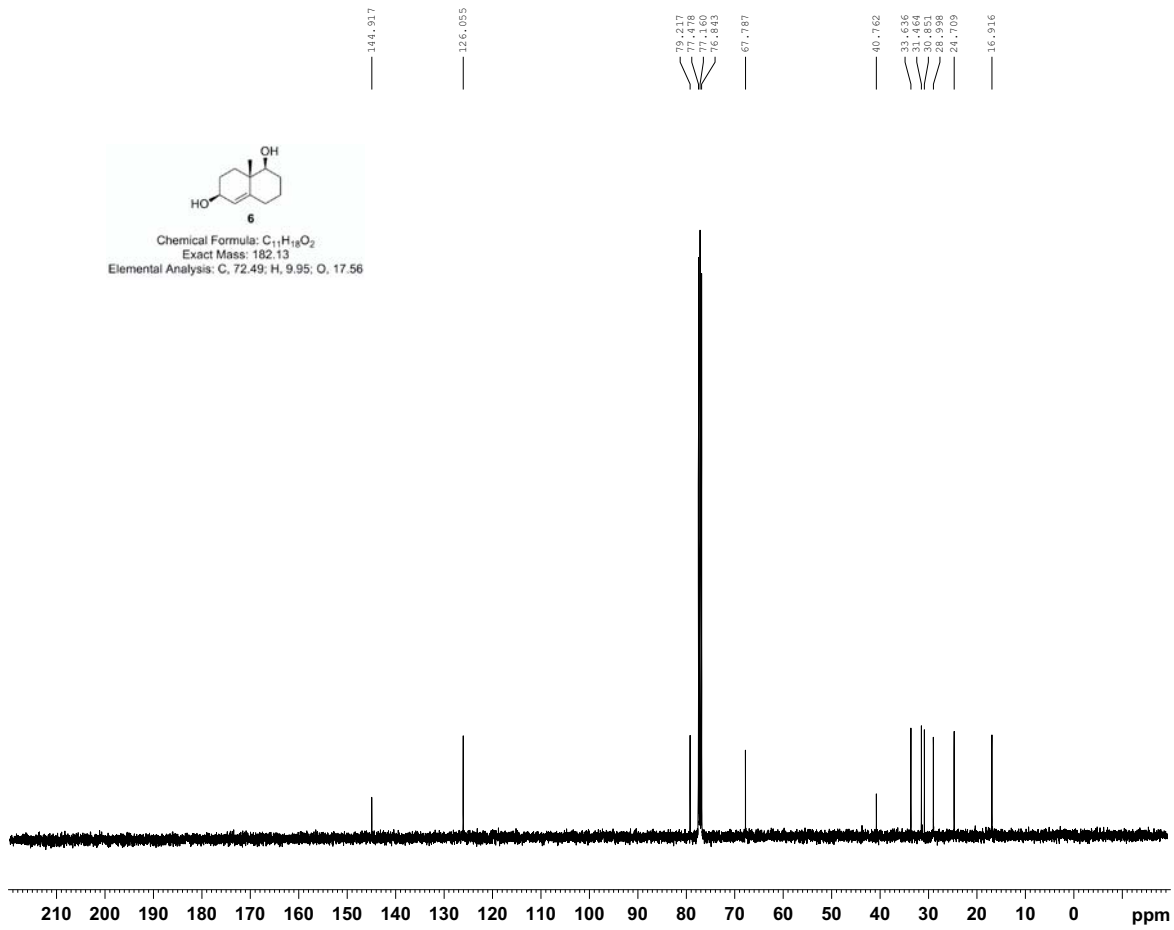
^{13}C NMR spectroscopic comparison (5-210 ppm)



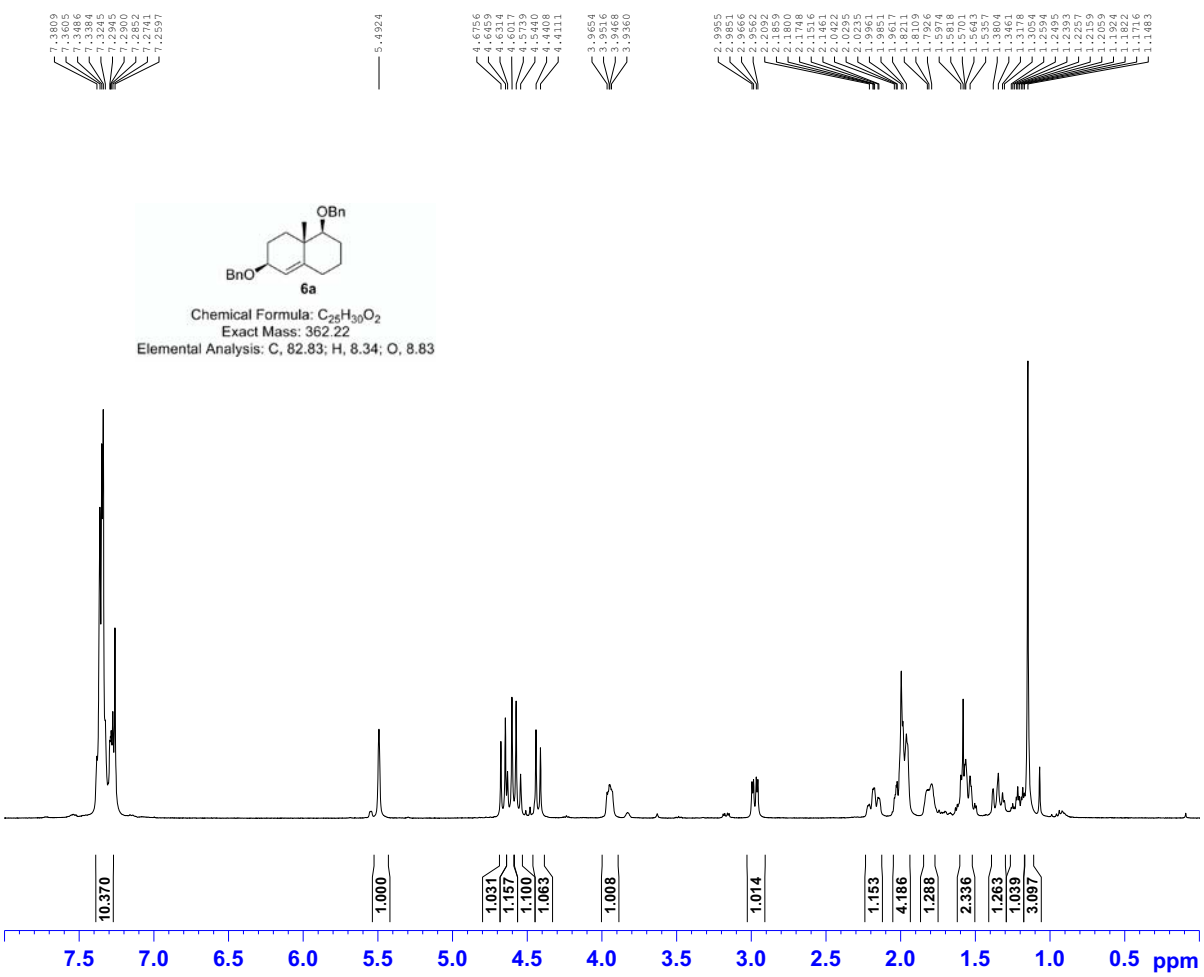
pallambin D (**2**)



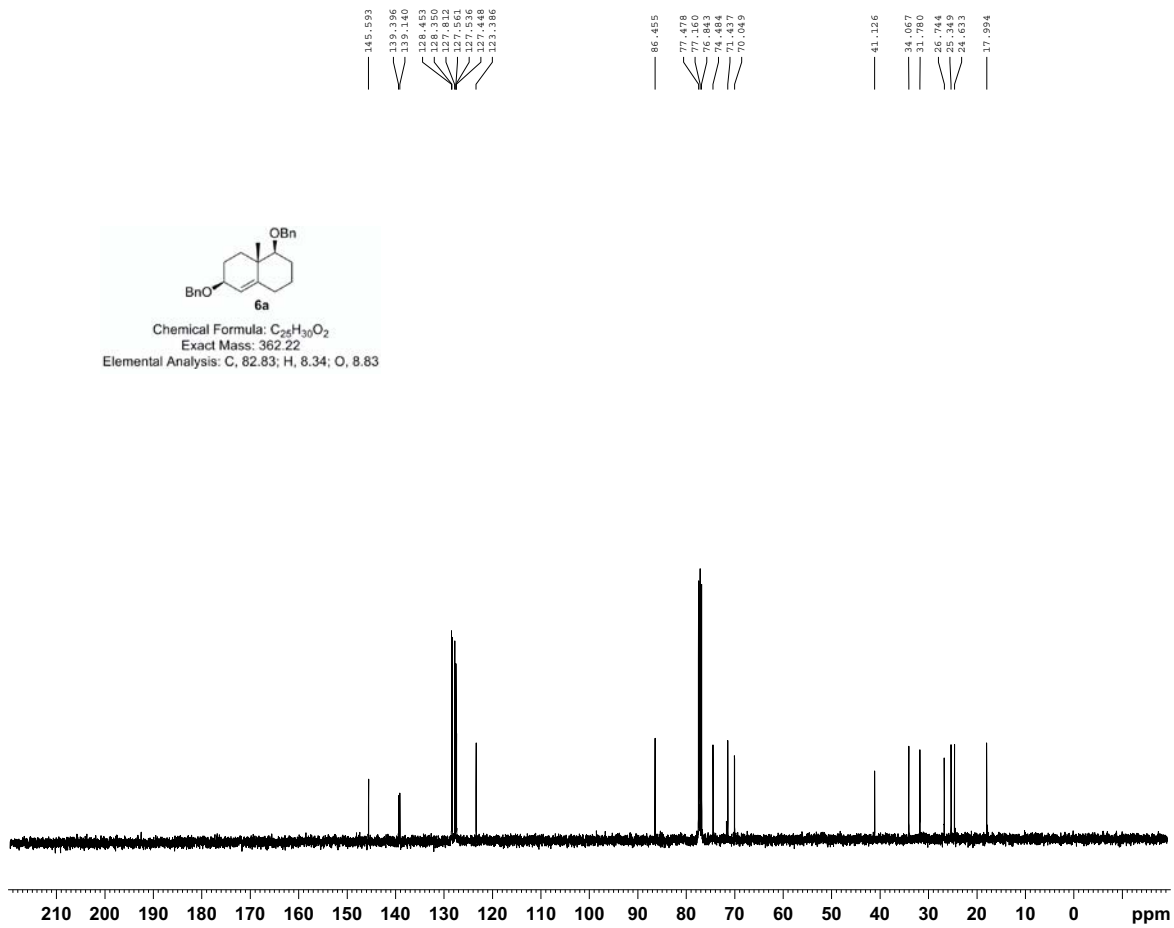

```
Bruker Advance III 400

NAME          XS-1-7-A1
EXPNO                 1
PROCNO                1
Date_          20110919
Time              18.50
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                  101
DW               60.800 usec
DE                 6.50 usec
TE                298.9 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                32768
SF          400.1900141 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



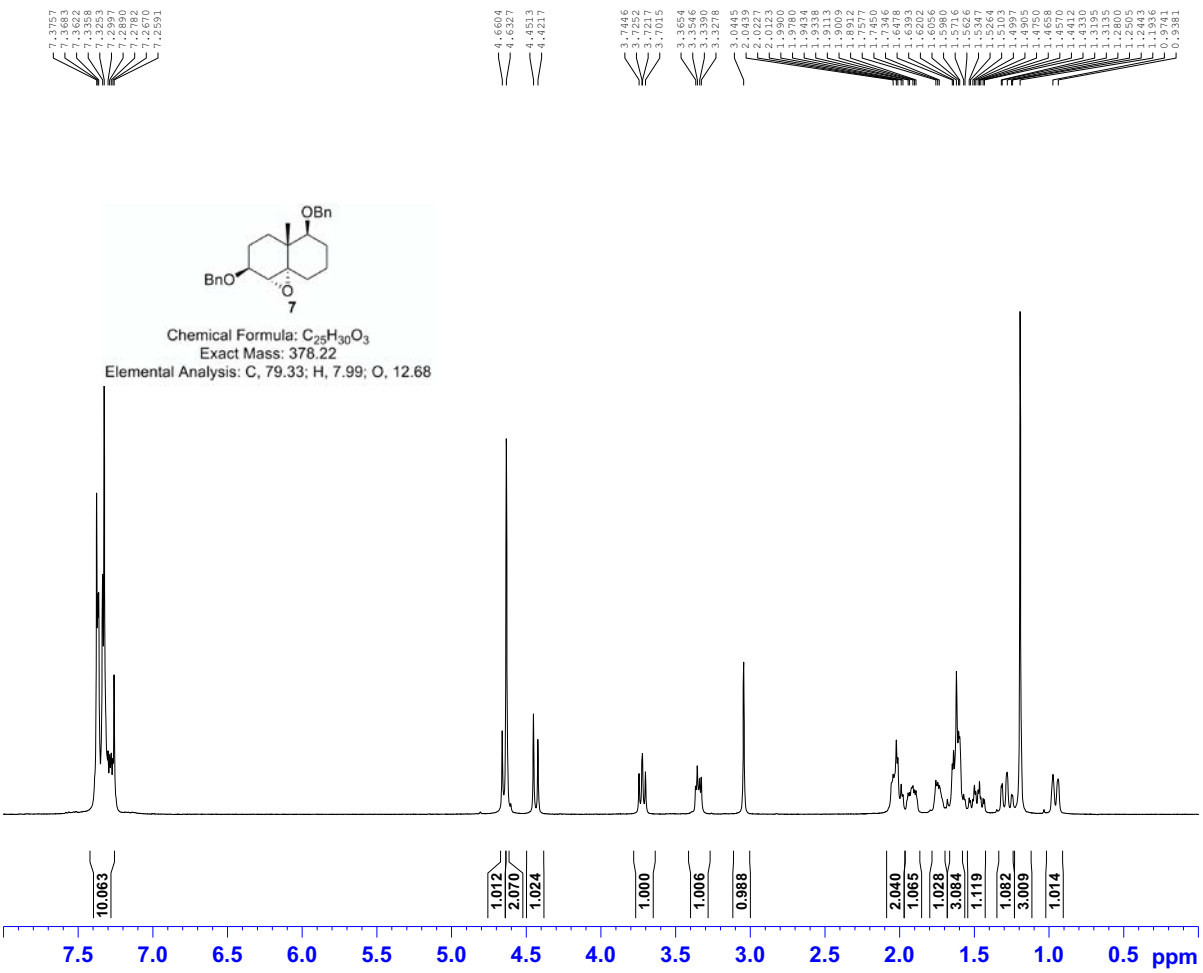

```
Bruker Advance III 400

NAME          XS-1-7-A1
EXPNO                 2
PROCNO                1
Date_          20110919
Time              18.57
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  176
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  203
DW               20.800 usec
DE                 6.50 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                32768
SF          100.6278427 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



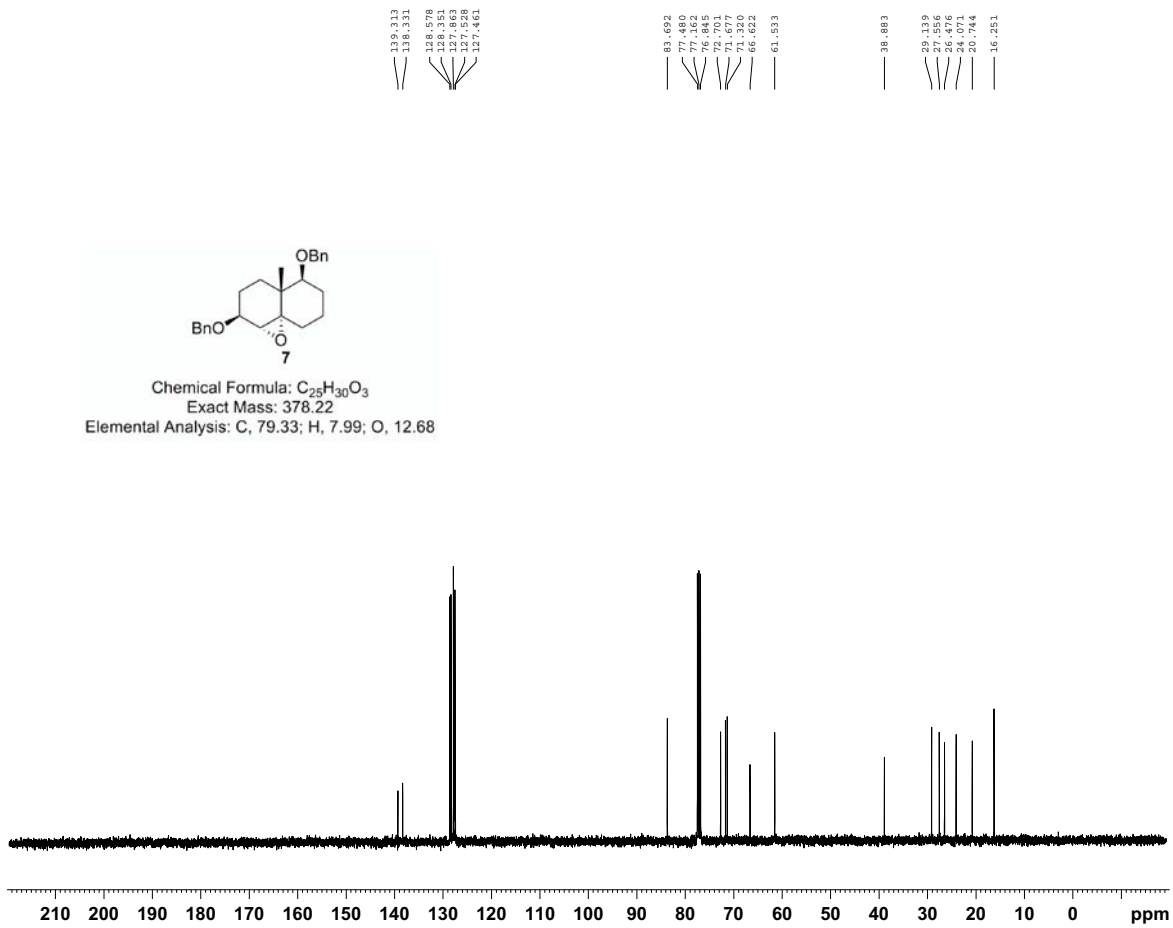

```
Bruker Advance III 400

NAME          XS-1-5-A1
EXPNO                 2
PROCNO                1
Date_          20110923
Time               17.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     5
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.98446387 sec
RG                  50.8
DW                60.800 usec
DE                  6.50 usec
TE                299.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900141 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



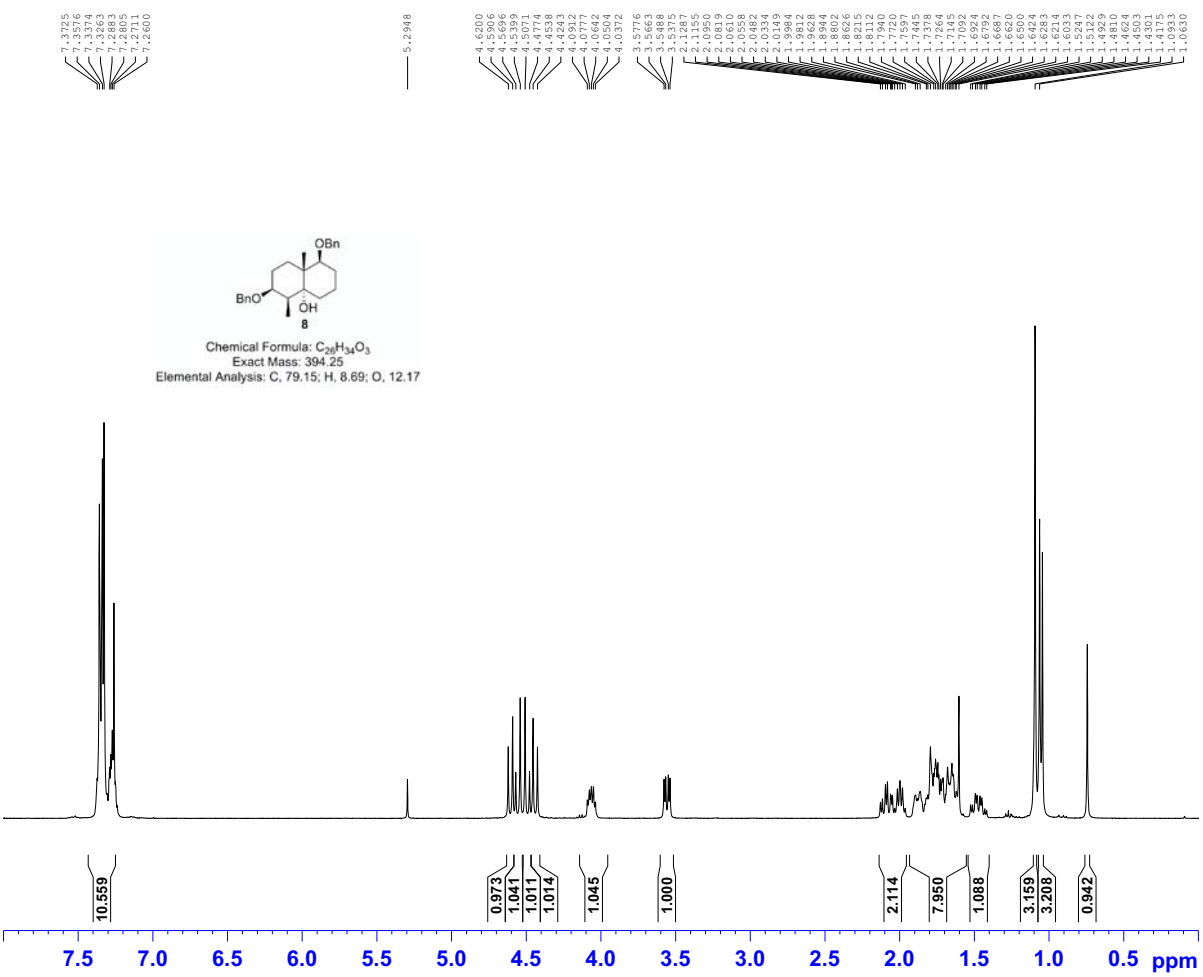

```
Bruker Advance III 400

NAME          XS-1-5-A1
EXPNO                 3
PROCNO                1
Date_          20110923
Time               17.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    79
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                299.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.90 usec
PL1                -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                 32768
SF          100.6278456 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
Bruker Advance III 400

NAME          XS-1-9-A1
EXPNO                 1
PROCNO                1
Date_          20110924
Time              15.14
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                   36
DW               60.800 usec
DE                 6.50 usec
TE                298.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                32768
SF          400.1900144 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

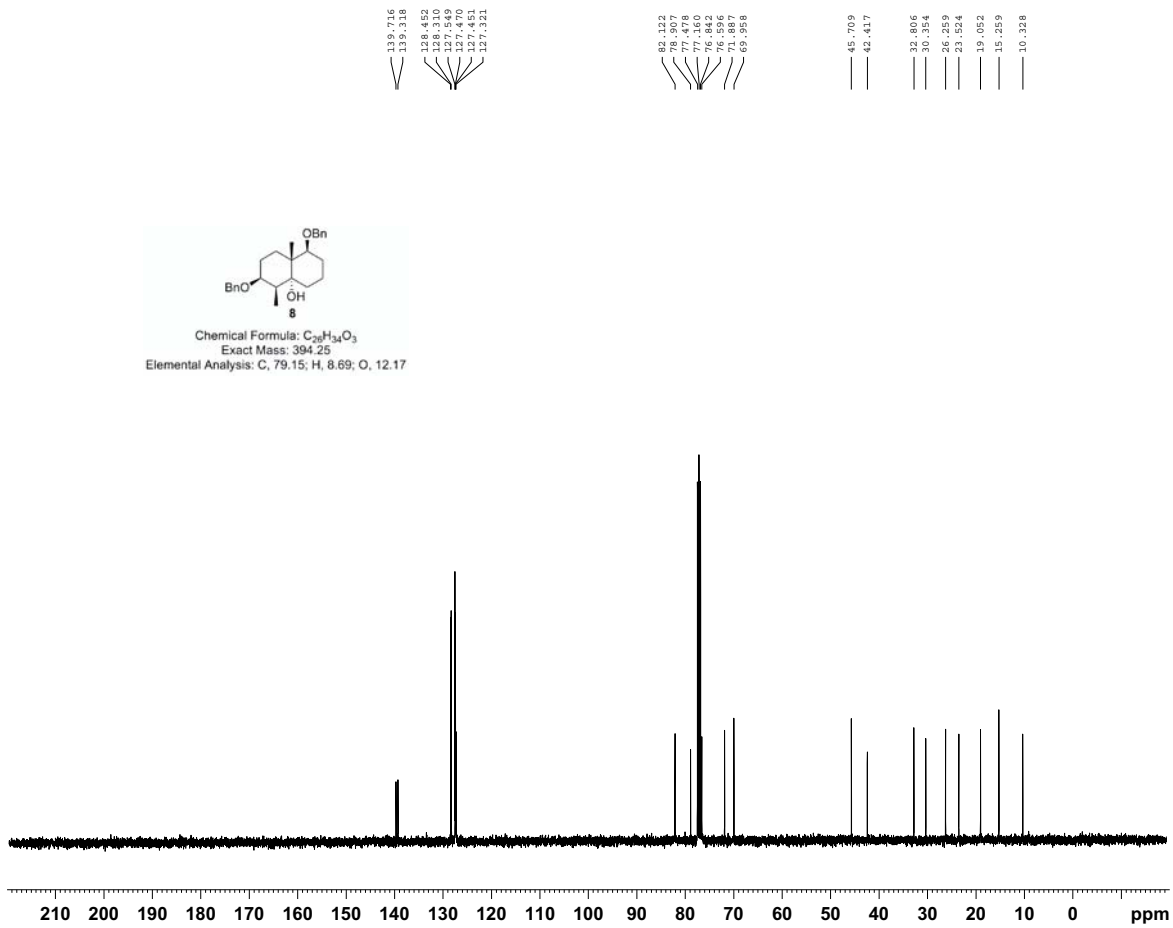



```
Bruker Advance III 400

NAME          XS-1-9-A1
EXPNO                 2
PROCNO                1
Date_          20110924
Time              15.19
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   56
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  203
DW               20.800 usec
DE                 6.50 usec
TE                299.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                32768
SF          100.6278471 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



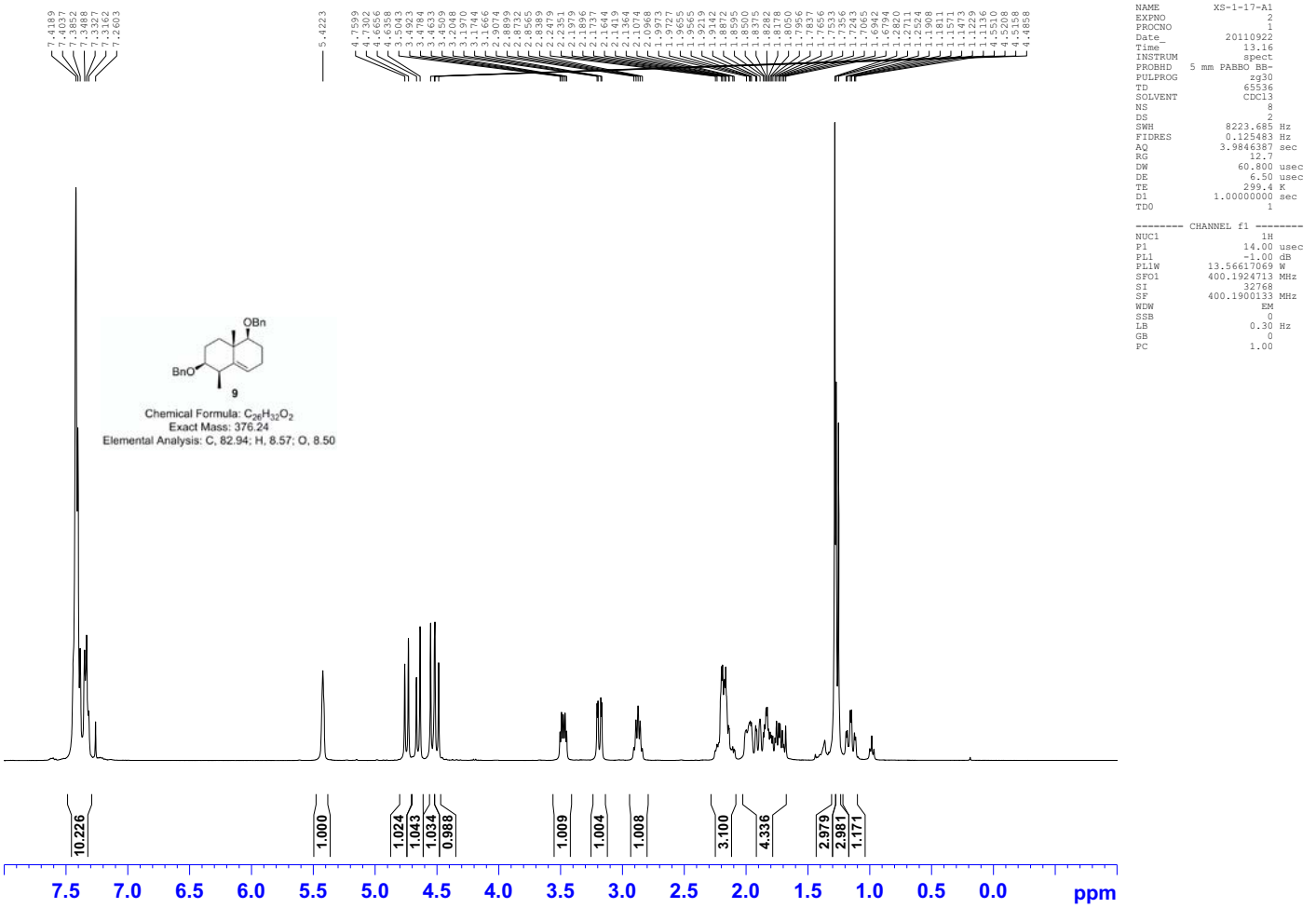
Bruker Advance III 400

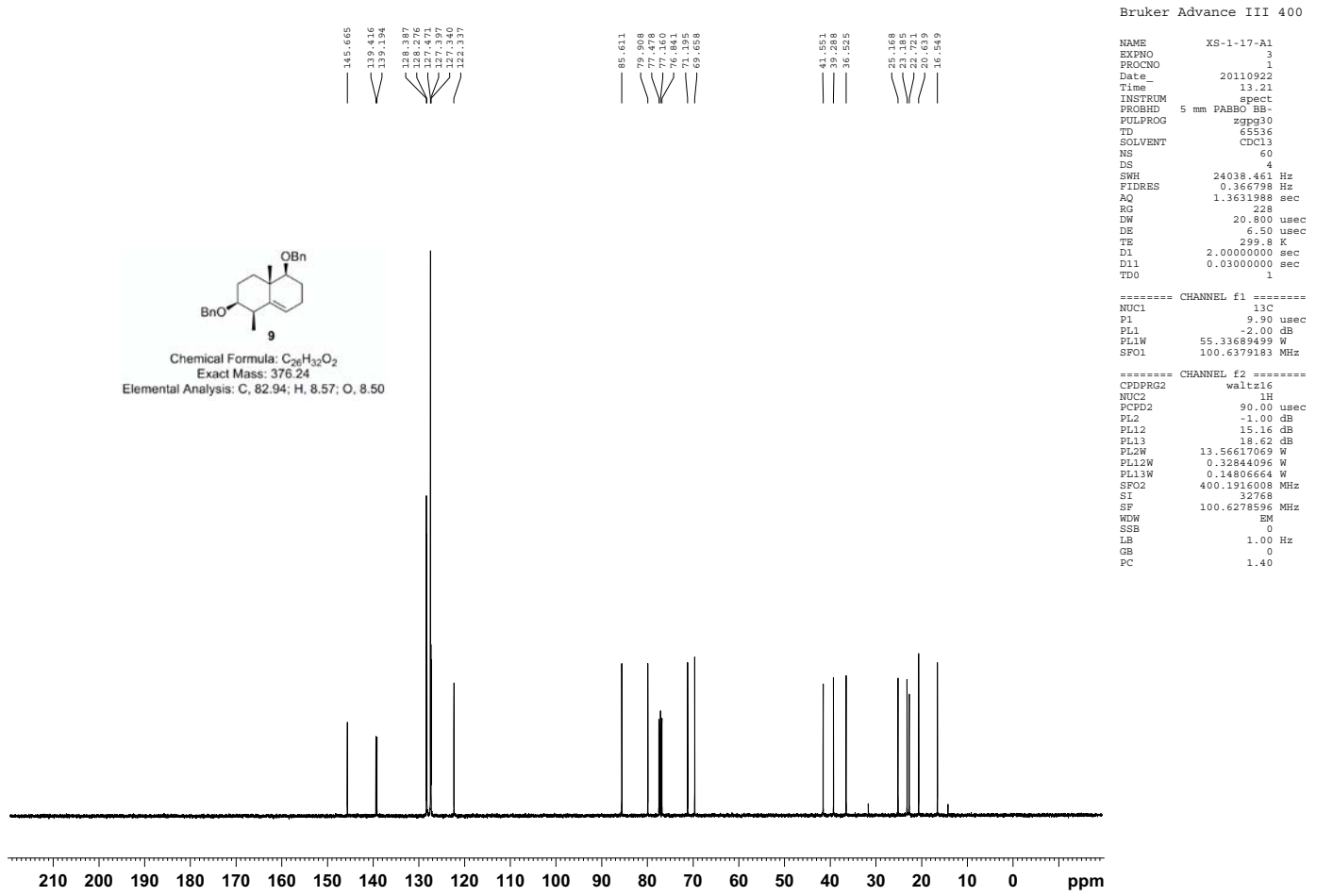
```
NAME          XS-1-16-A1
EXPNO                  1
PROCNO                 1
Date_           20110926
Time_              15.13
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.98466387 sec
RG                  40.3
DW                60.800 usec
DE                  6.50 usec
TE                298.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900138 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

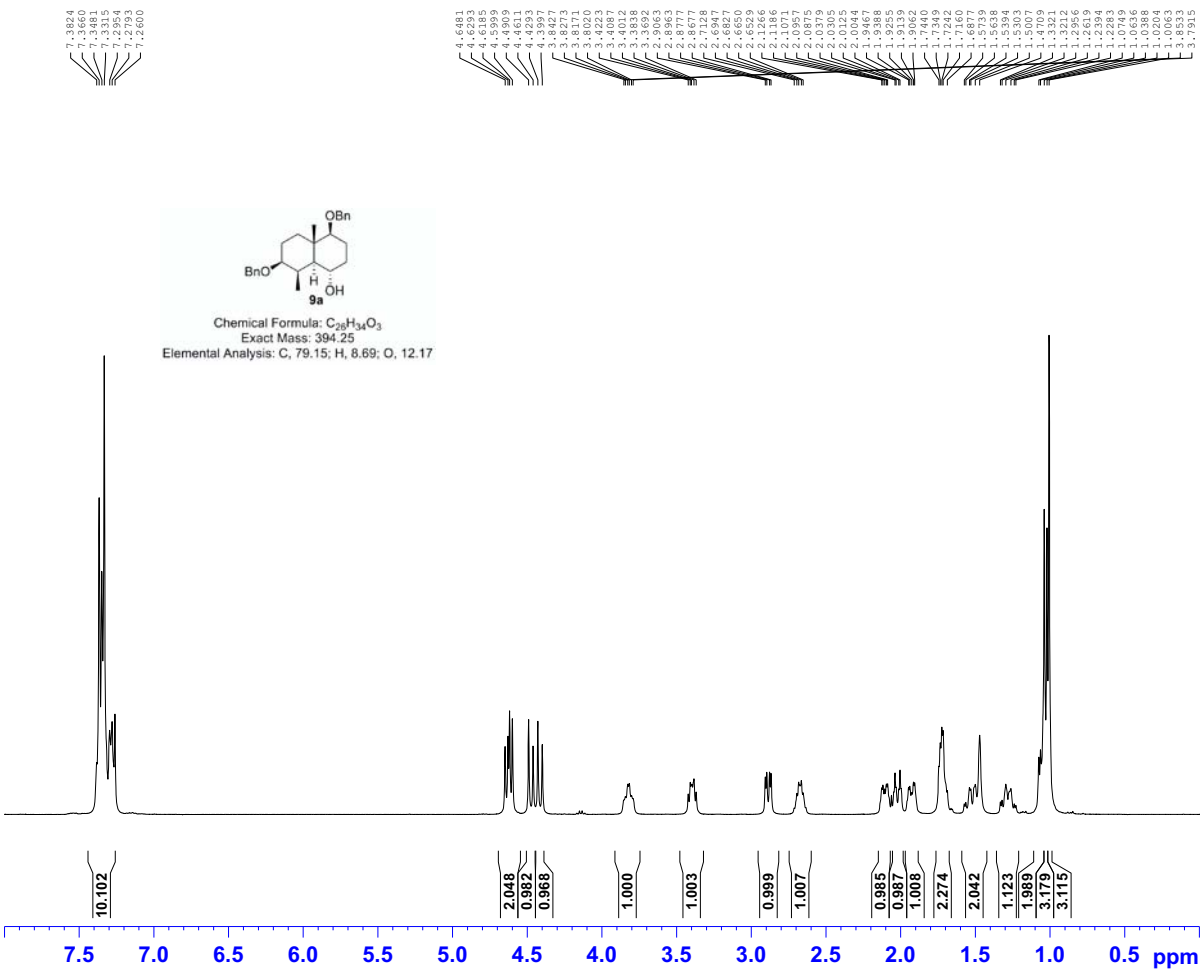


Bruker Advance III 400

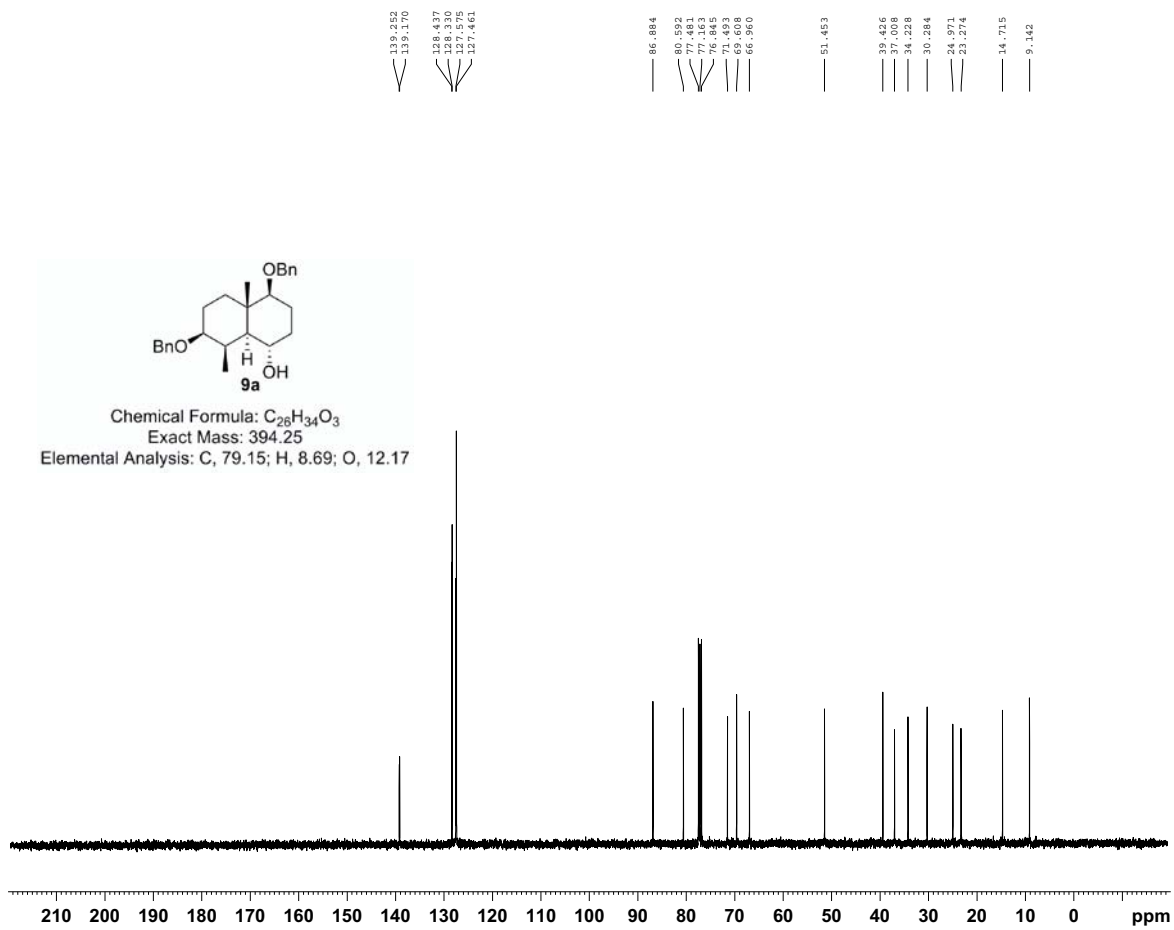
```
NAME          XS-1-16-A1
EXPNO                  2
PROCNO                 1
Date_           20110926
Time_              15.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    64
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   181
DW                20.800 usec
DE                  6.50 usec
TE                299.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL13               18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                 32768
SF          100.6278464 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


Bruker Advance III 400
NAME XS-1-17-A1
EXPNO 2
PROCNO 1
Date_ 20110922
Time 13.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.98466387 sec
RG 12.7
DW 60.800 usec
DE 6.50 usec
TE 299.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OBn
BnO
9
Chemical Formula: C26H32O2
Exact Mass: 376.24
Elemental Analysis: C, 82.94; H, 8.57; O, 8.50
10.226
1.000
1.024
1.043
1.034
0.988
1.009
1.004
1.008
3.100
4.336
2.979
2.981
1.171
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

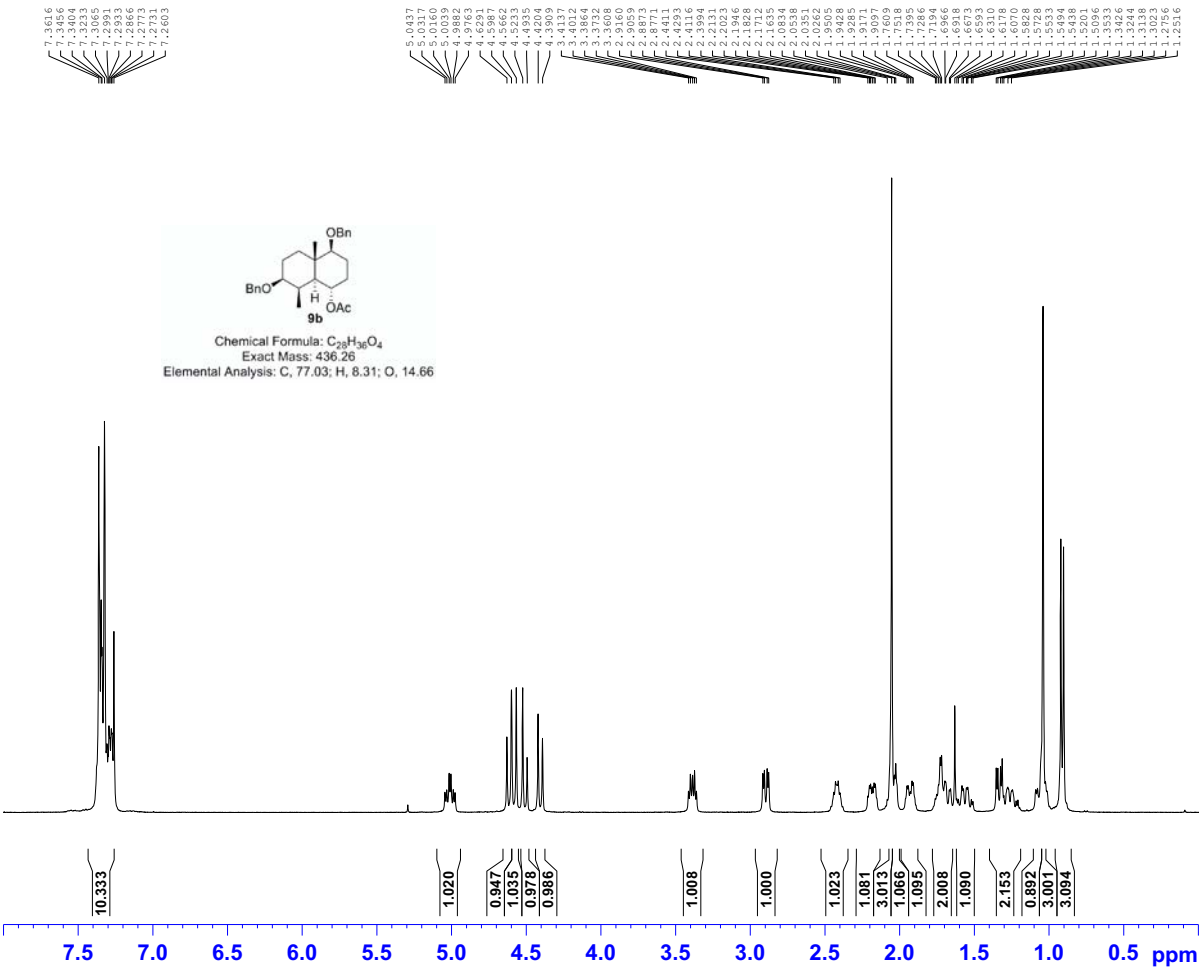

Bruker Advance III 400
NAME XS-1-17-A1
EXPNO 3
PROCNO 1
Date_ 20110922
Time 13.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 60
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 228
DW 20.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6379183 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 18.62 dB
PL2W 13.56617069 W
PL12W 0.32844096 W
PL13W 0.14806664 W
SFO2 400.1916008 MHz
SI 32768
SF 100.6278596 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
145.665
139.416
139.194
128.387
128.276
127.471
127.397
127.340
122.337
85.611
79.908
77.478
77.160
76.841
73.195
69.458
41.551
39.288
36.525
25.168
23.185
22.721
20.639
16.549
OBn
BnO
9
Chemical Formula: C26H32O2
Exact Mass: 376.24
Elemental Analysis: C, 82.94; H, 8.57; O, 8.50
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



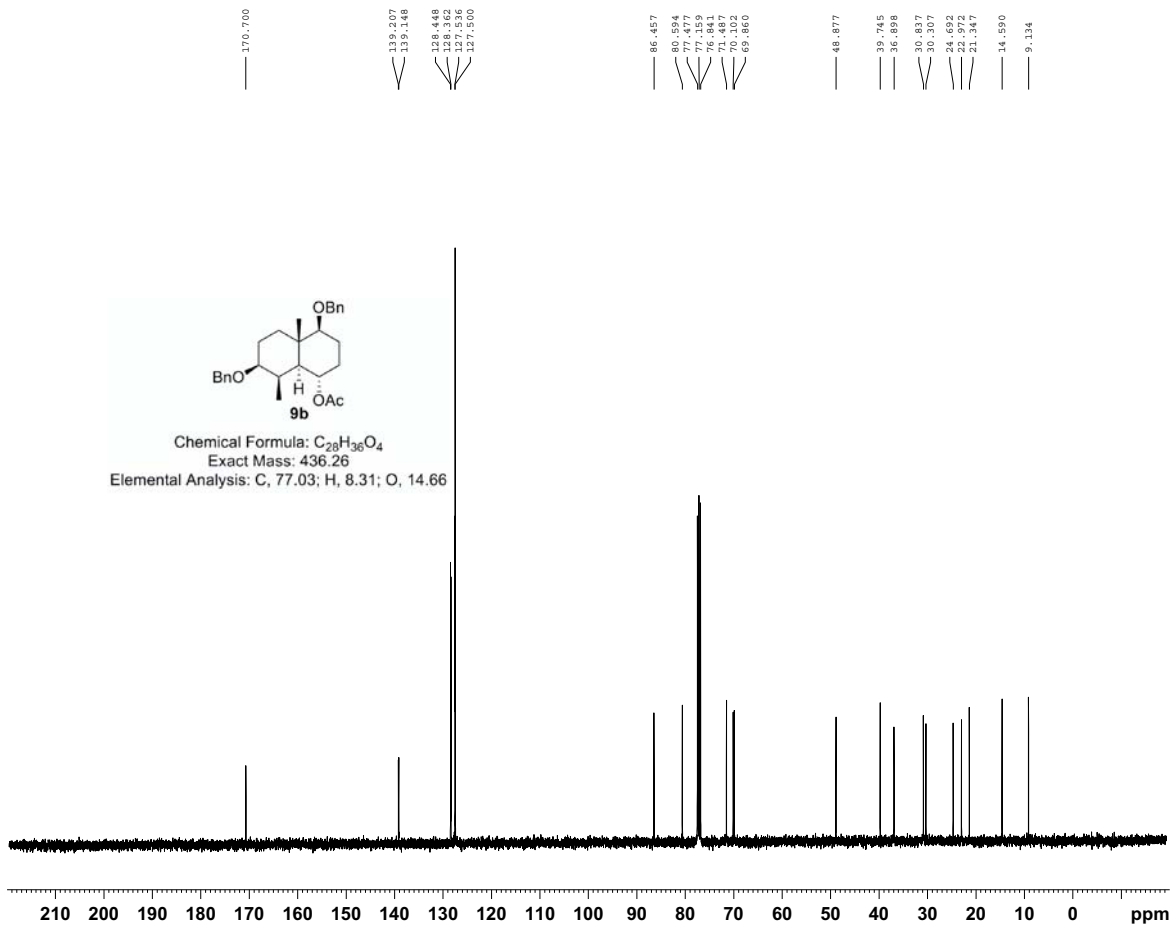

```
Bruker Advance III 400

NAME          XS-1-20-A1
EXPNO                  2
PROCNO                 1
Date_           20110927
Time                12.30
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ             3.98446387 sec
RG                   28.5
DW                 60.800 usec
DE                   6.50 usec
TE                  299.3 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  -1.00 dB
PL1W          13.56617069 W
SFO1          400.1924713 MHz
SI                   32768
SF            400.1900141 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



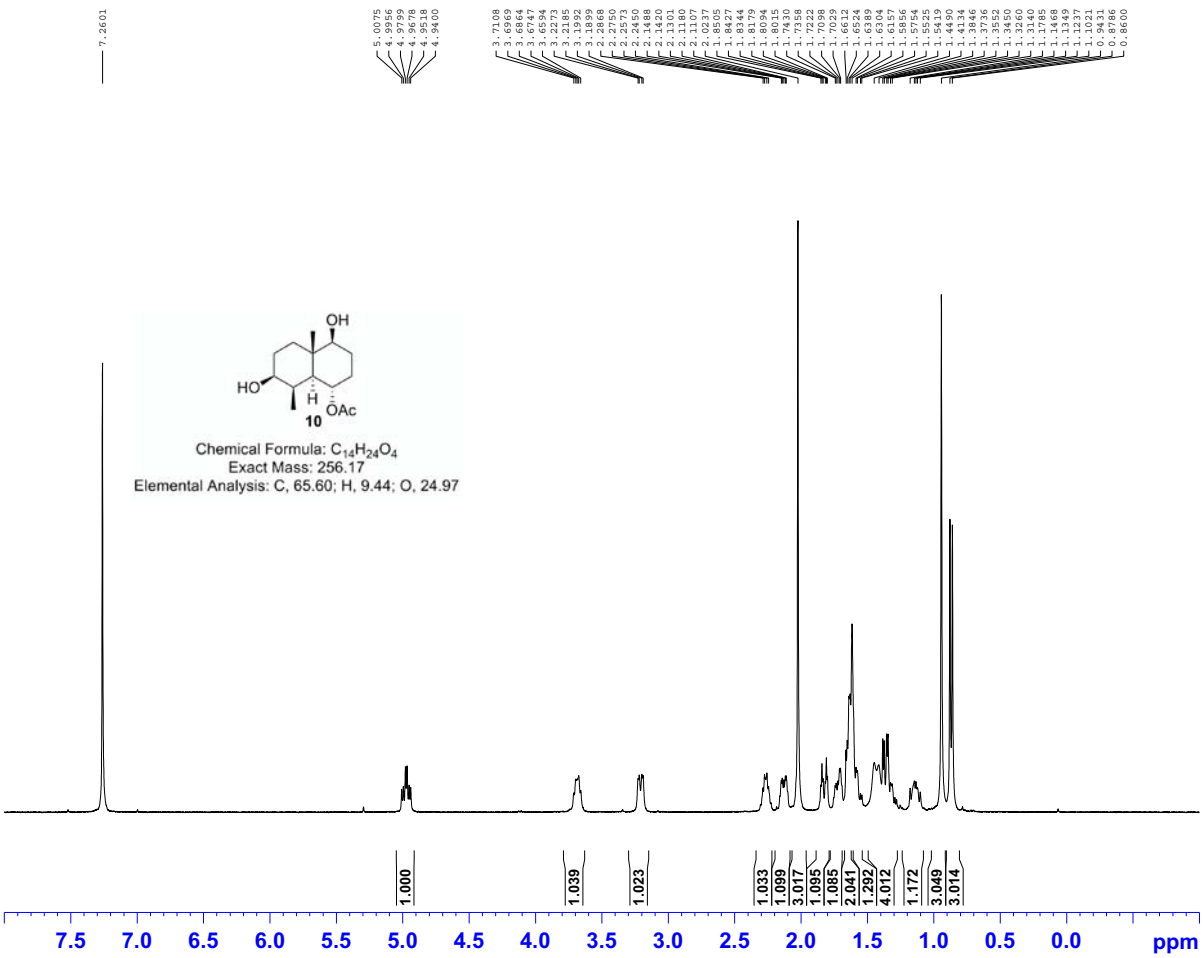

```
Bruker Advance III 400

NAME          XS-1-20-A1
EXPNO                  3
PROCNO                 1
Date_           20110927
Time                21.14
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     41
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ             1.3631988 sec
RG                    181
DW                 20.800 usec
DE                   6.50 usec
TE                  299.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                  -2.00 dB
PL1W          55.33689499 W
SFO1          100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL13                 18.62 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
PL13W          0.14806664 W
SFO2          400.1916008 MHz
SI                   32768
SF            100.6278508 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



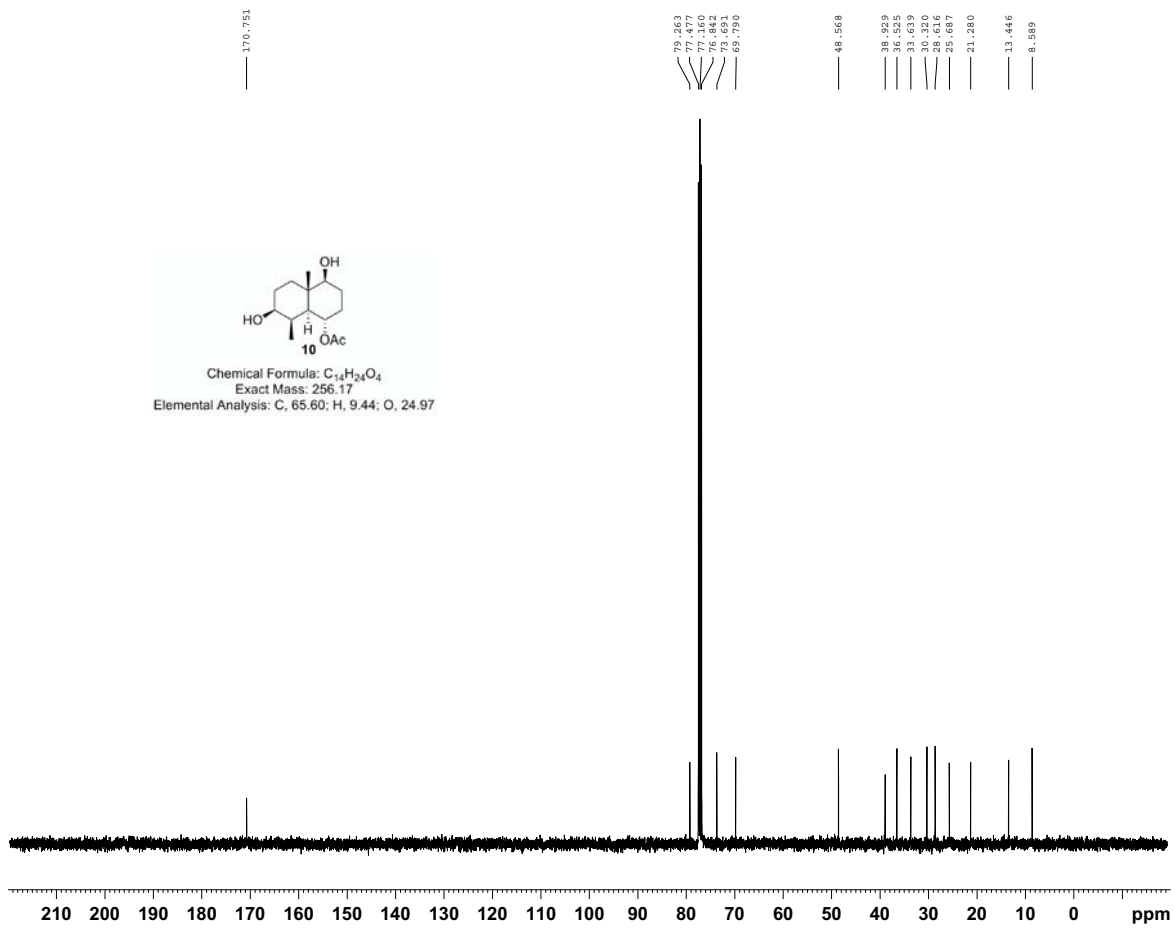

```
Bruker Advance III 400

NAME          XS-1-21-A1
EXPNO                  1
PROCNO                 1
Date_           20110927
Time_             21.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     2
DS                     2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.98446387 sec
RG                  40.3
DW               60.800 usec
DE                 6.50 usec
TE                298.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900138 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



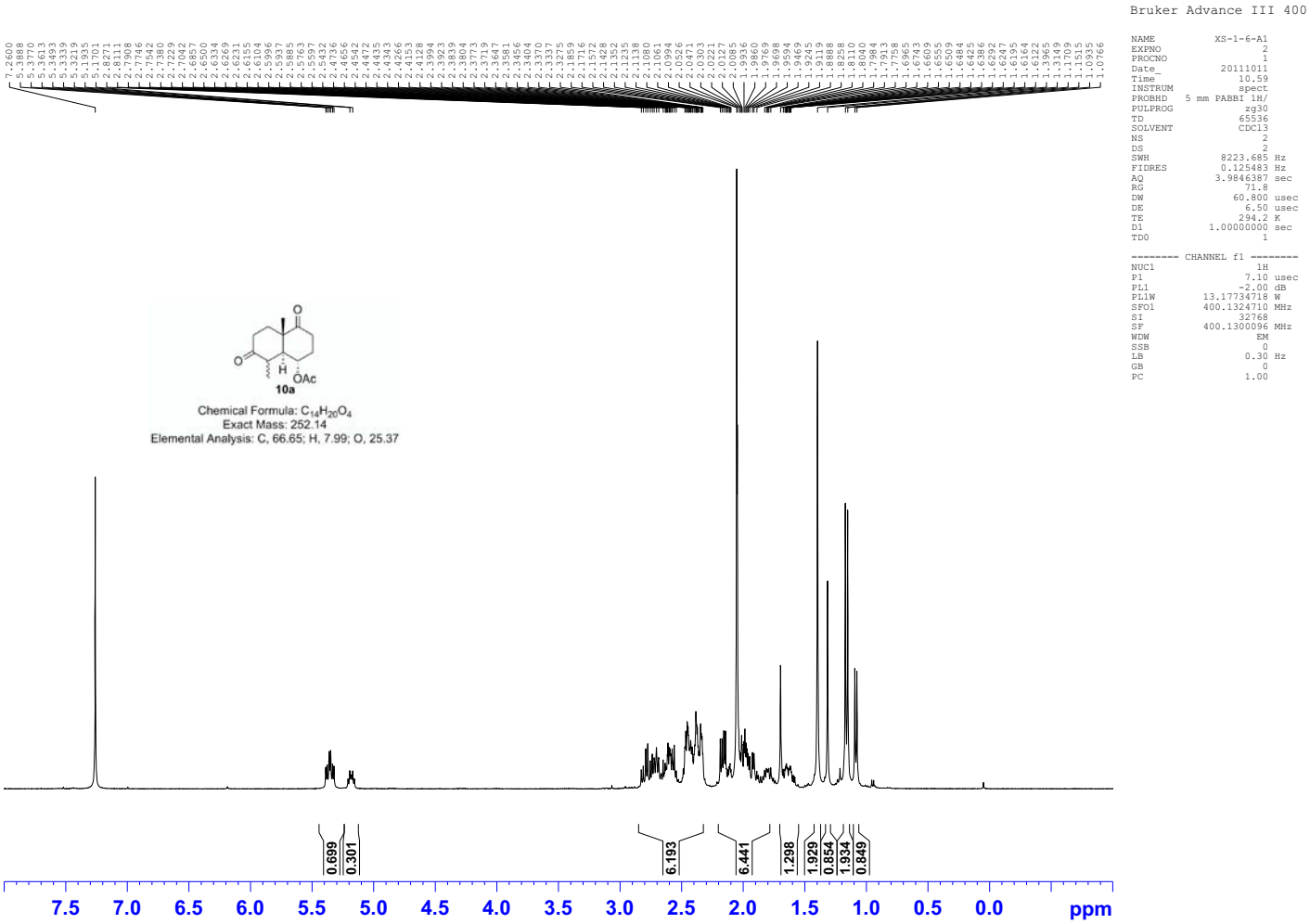

```
Bruker Advance III 400

NAME          XS-1-21-A1
EXPNO                  2
PROCNO                 1
Date_           20110927
Time_             21.25
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    72
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                   203
DW               20.800 usec
DE                 6.50 usec
TE                299.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.90 usec
PL1               -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                 32768
SF          100.6278471 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

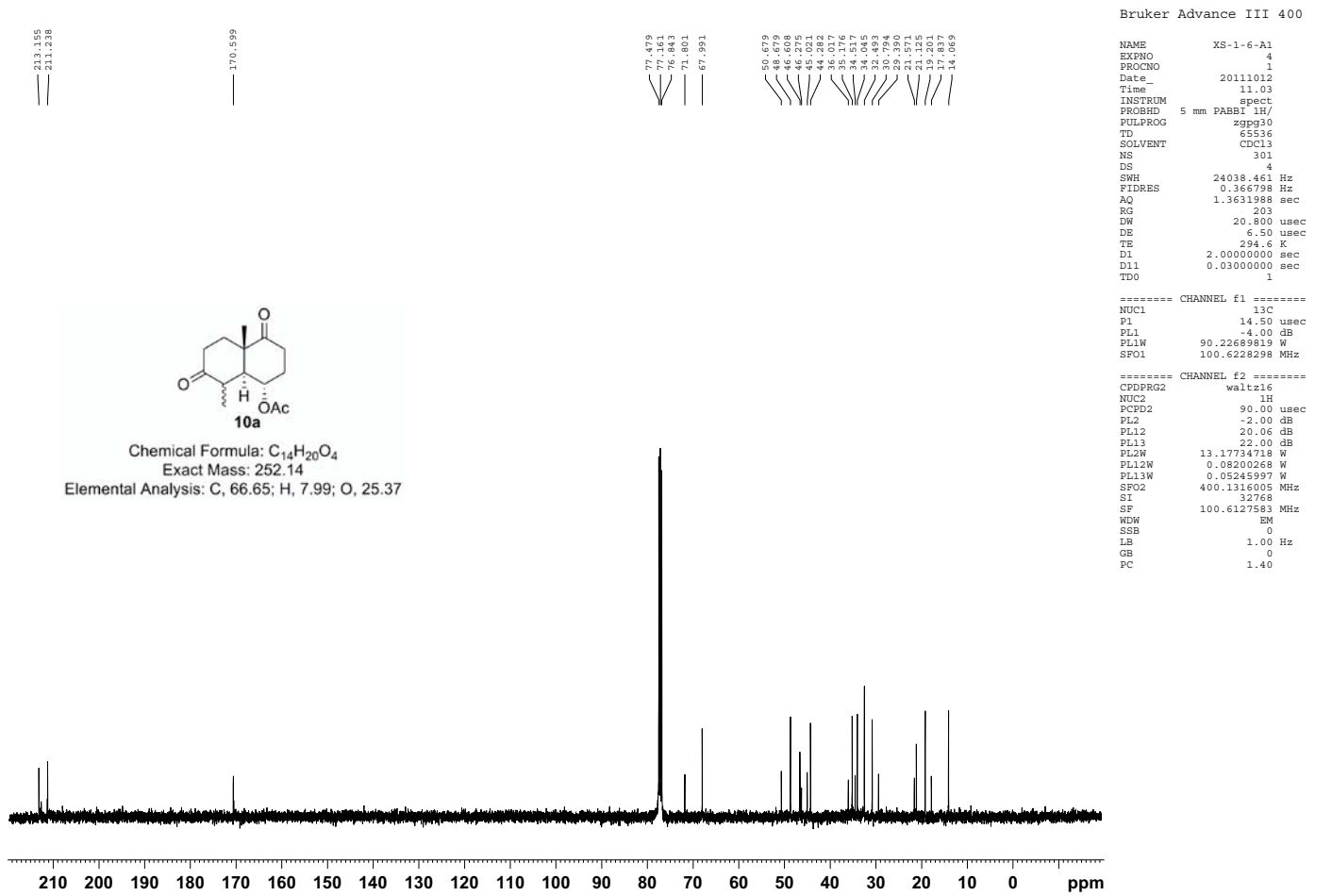


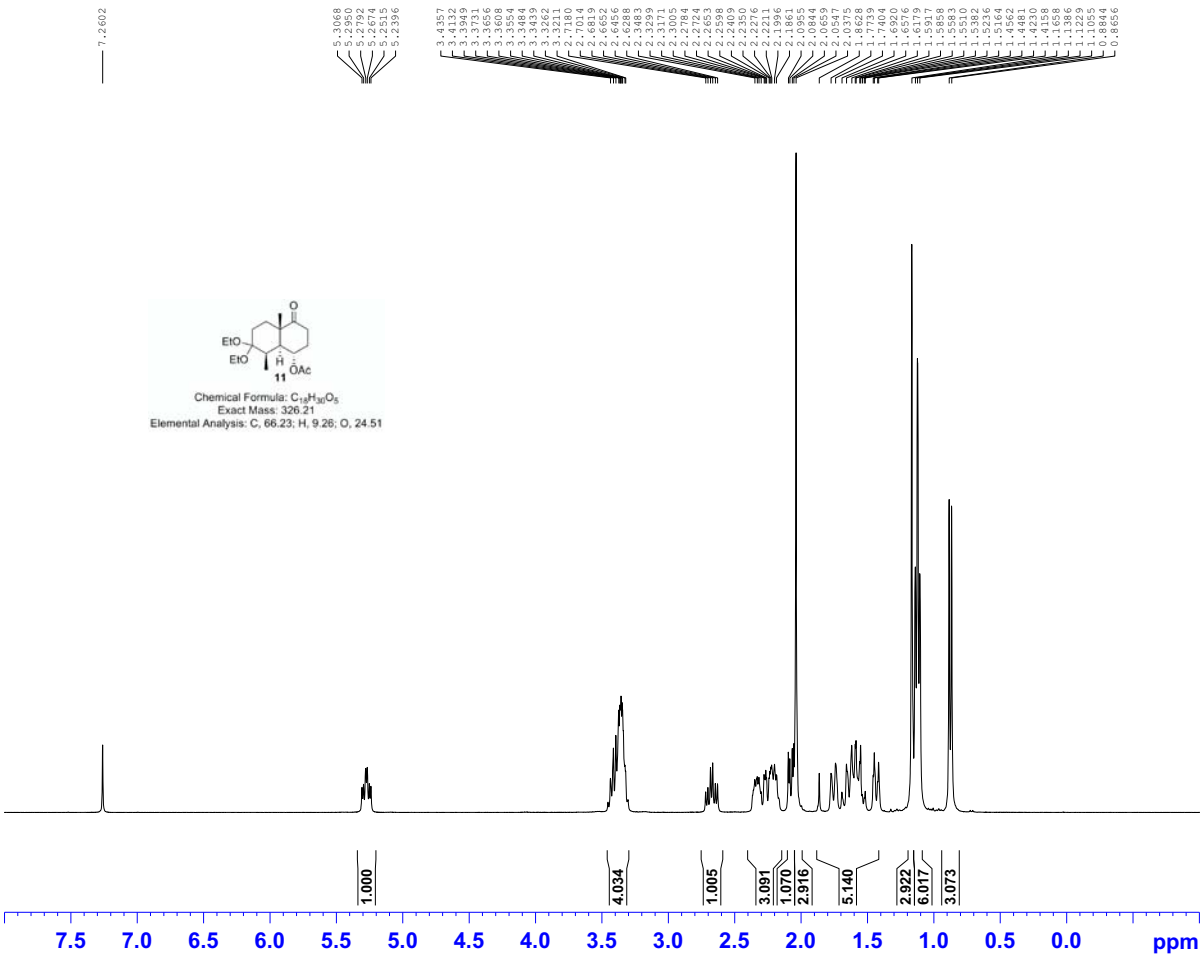

```
Bruker Advance III 400

NAME          XS-1-2-A1
EXPNO                 1
PROCNO                1
Date_          20110928
Time_             14.29
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    2
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                  101
DW               60.800 usec
DE                6.50 usec
TE                298.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                32768
SF          400.1900138 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



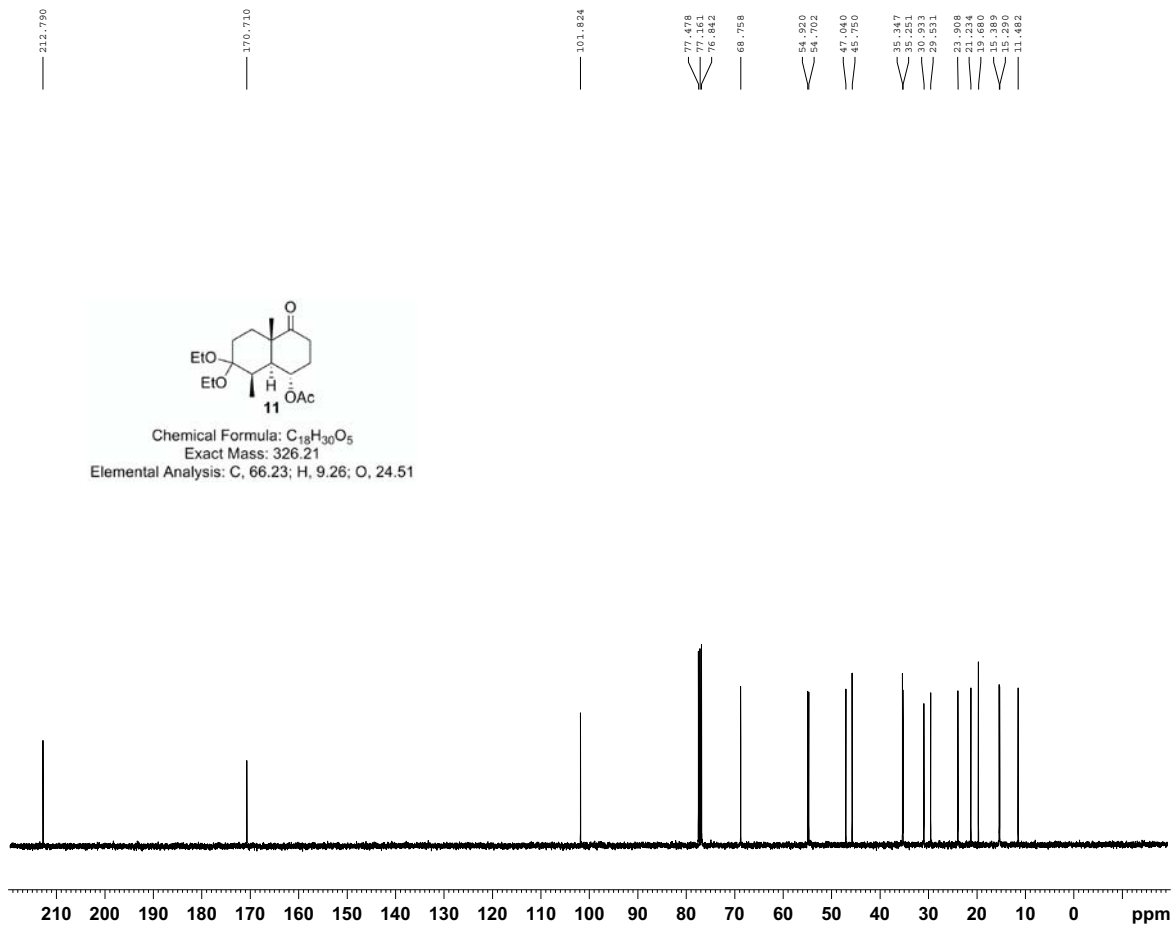

```
Bruker Advance III 400

NAME          XS-1-2-A1
EXPNO                 2
PROCNO                1
Date_          20110927
Time_             21.34
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  200
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ           1.3631988 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                32768
SF          100.6278427 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


Bruker Advance III 400
NAME XS-1-6-A1
EXPNO 2
PROCNO 1
Date_ 20111011
Time 10.59
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.98466387 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.10 usec
PL1 -2.00 dB
PL1W 13.17734718 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10a
Chemical Formula: C14H20O4
Exact Mass: 252.14
Elemental Analysis: C, 66.65; H, 7.99; O, 25.37
0.699
0.301
6.193
6.441
1.298
1.929
0.854
1.934
0.849
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

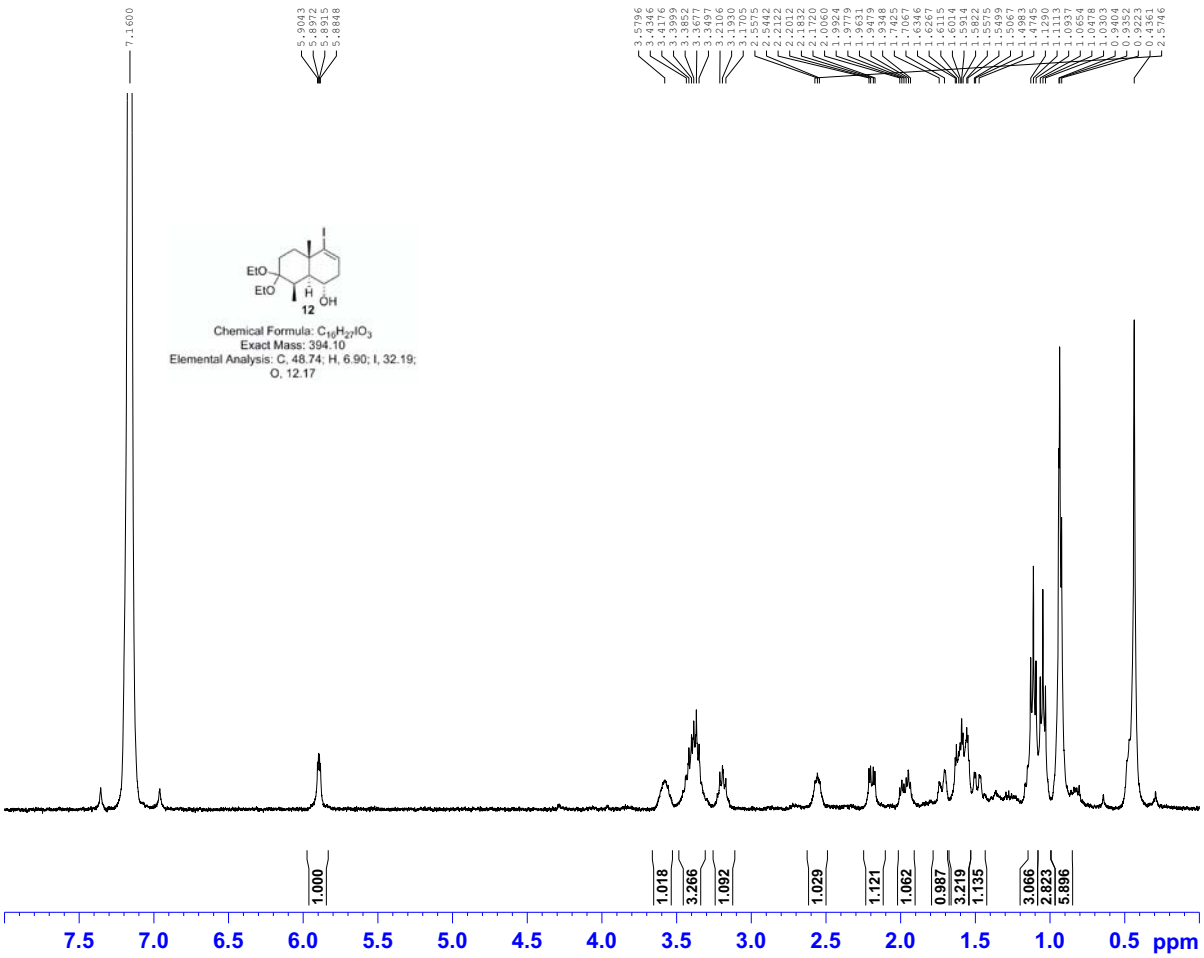

Bruker Advance III 400
NAME XS-1-6-A1
EXPNO 4
PROCNO 1
Date_ 20111012
Time 11.03
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 301
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 20.06 dB
PL13 22.00 dB
PL2W 13.17734718 W
PL12W 0.08200268 W
PL13W 0.05245997 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127583 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10a
Chemical Formula: C14H20O4
Exact Mass: 252.14
Elemental Analysis: C, 66.65; H, 7.99; O, 25.37
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm




```
Bruker Advance III 400

NAME          XS-1-10-A1
EXPNO                  1
PROCNO                 1
Date_           20111009
Time                15.23
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     2
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                  20.2
DW                60.800 usec
DE                  6.50 usec
TE                299.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900136 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

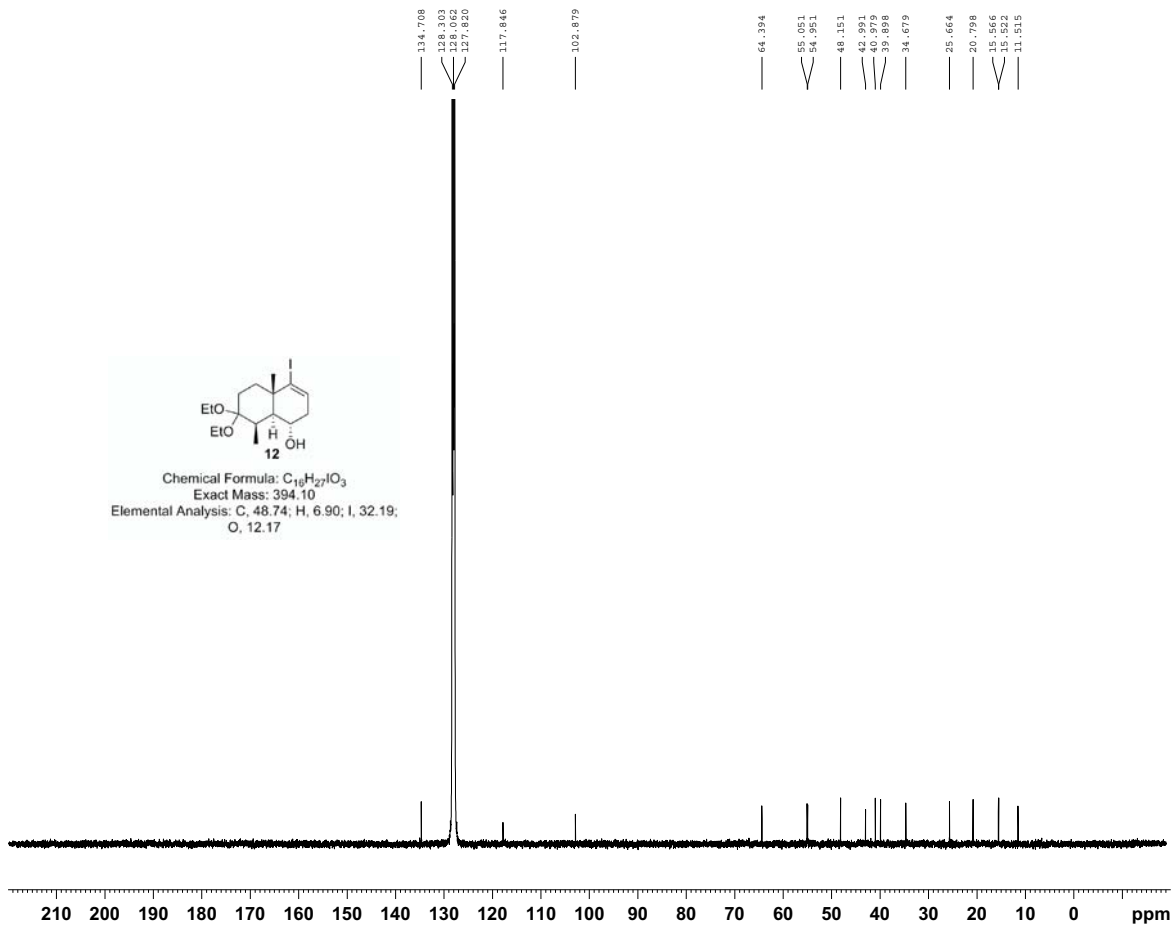



```
Bruker Advance III 400

NAME          XS-1-10-A1
EXPNO                  2
PROCNO                 1
Date_           20111009
Time                15.28
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     53
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                    203
DW                20.800 usec
DE                  6.50 usec
TE                299.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL13               18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                 32768
SF          100.6278475 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



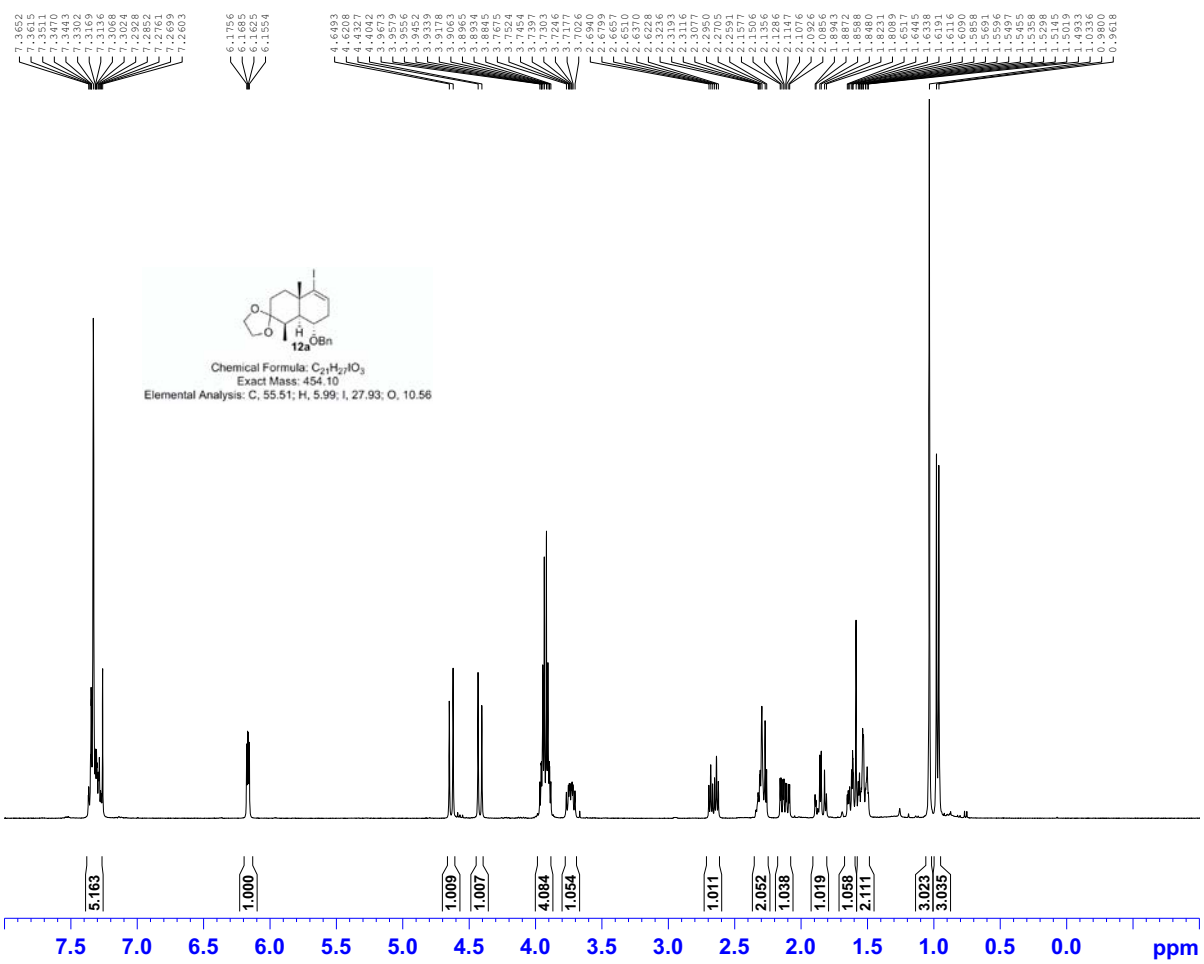
Bruker Advance III 400

```
NAME          XS-1-31-A1
EXPNO                 11
PROCNO                 1
Date_           20111101
Time               16.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                     4
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   128
DW                60.800 usec
DE                 6.50 usec
TE                302.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900016 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

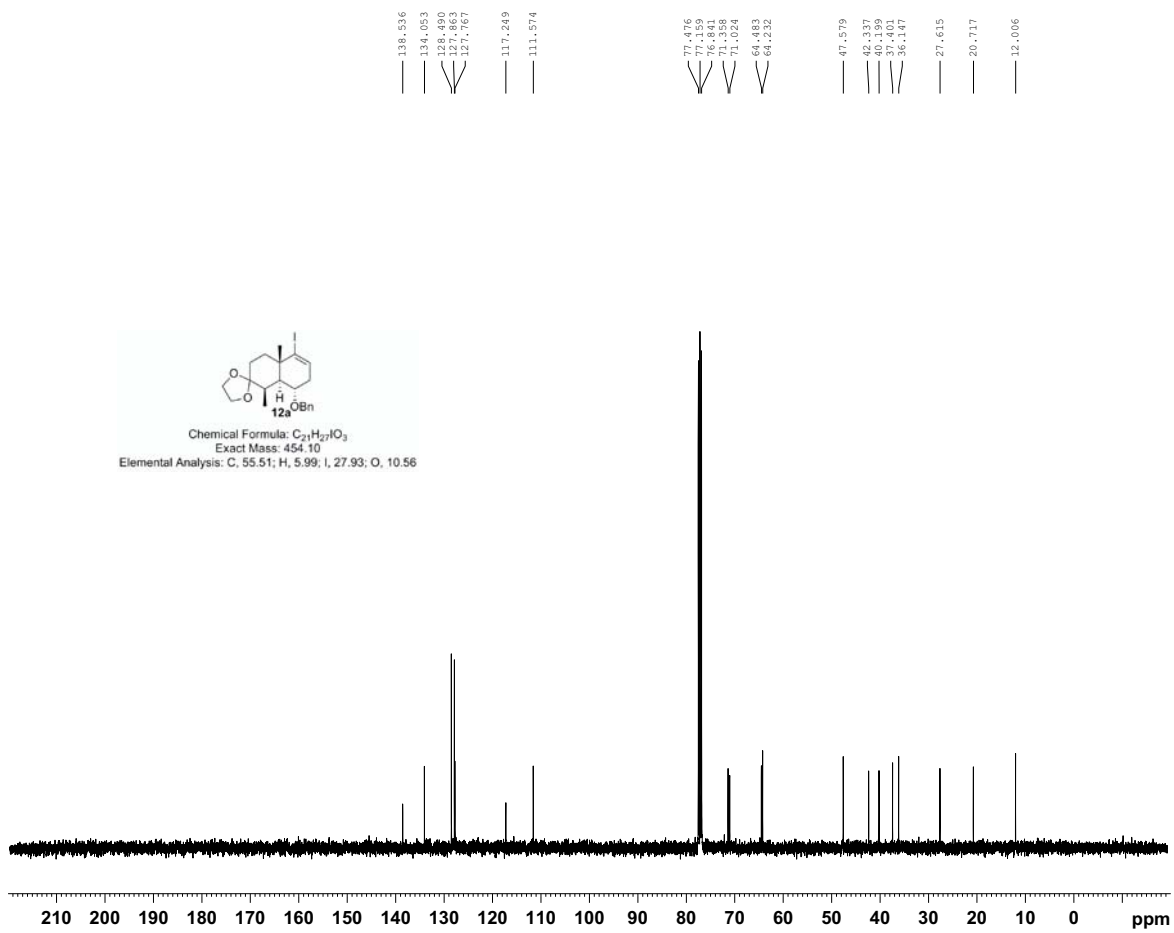


Bruker Advance III 400

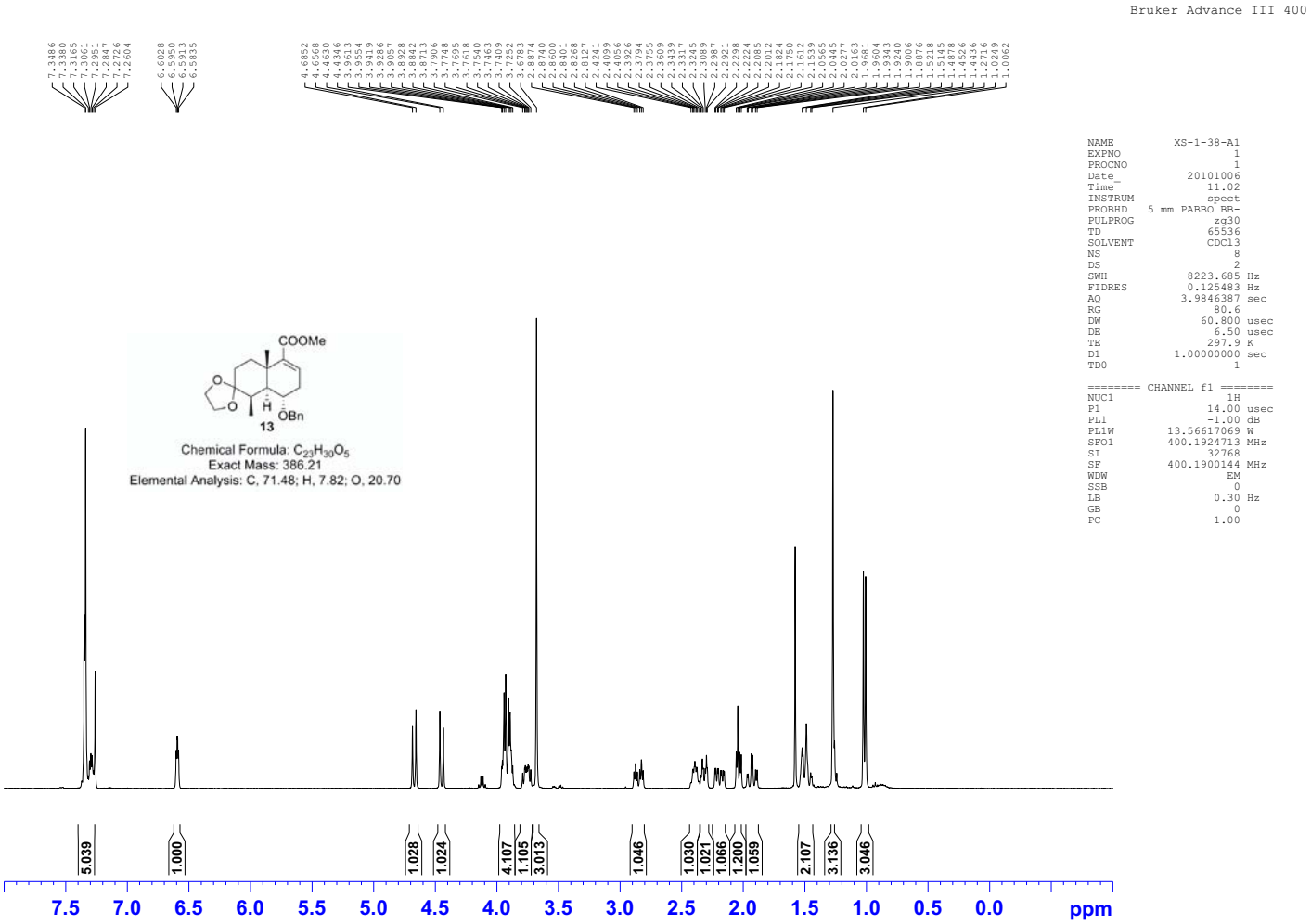
```
NAME          XS-1-31-A1
EXPNO                 14
PROCNO                 1
Date_           20111103
Time               21.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT             C6D6
NS                  6000
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   228
DW                20.800 usec
DE                 6.50 usec
TE                300.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL13               18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                 32768
SF          100.6278185 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

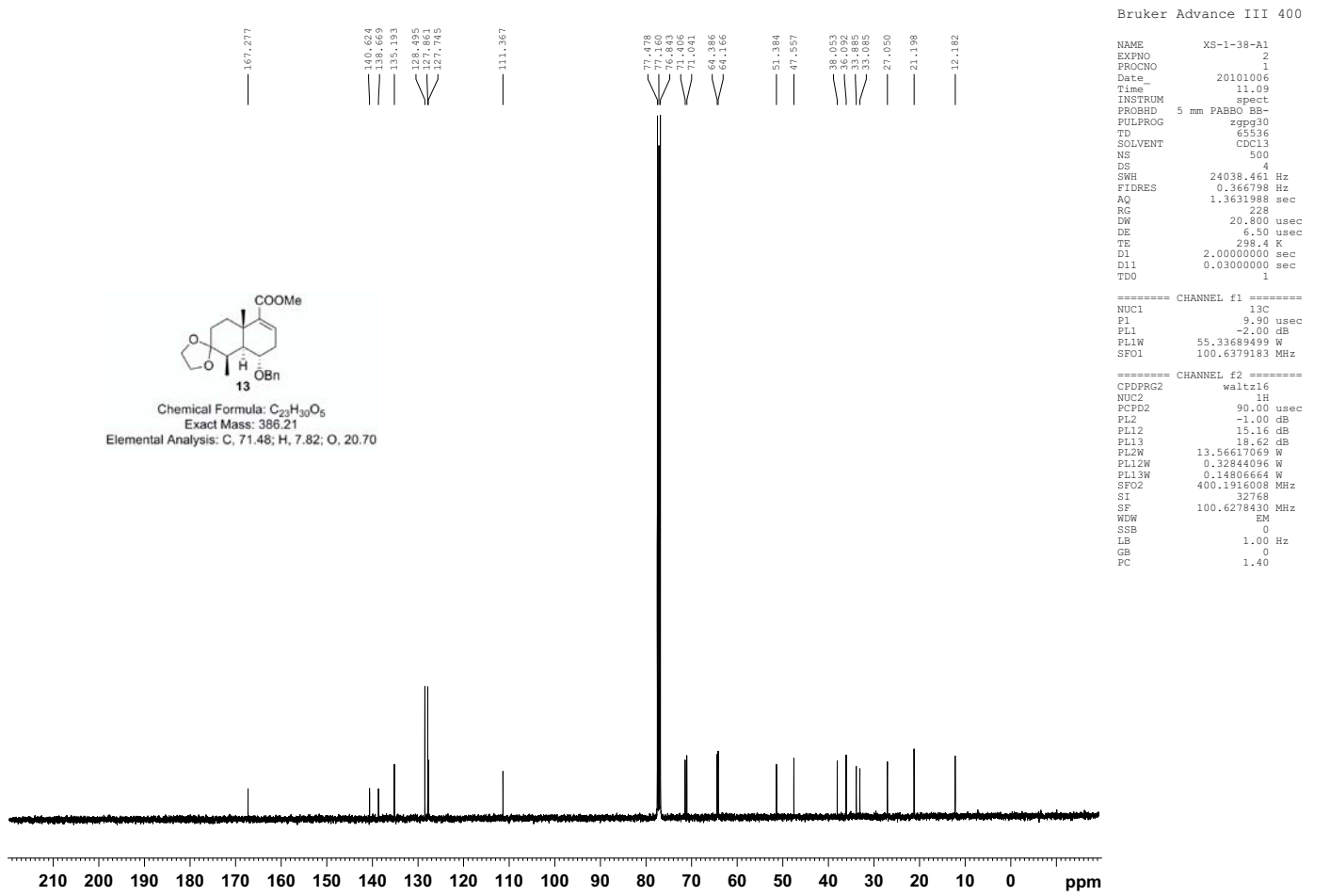


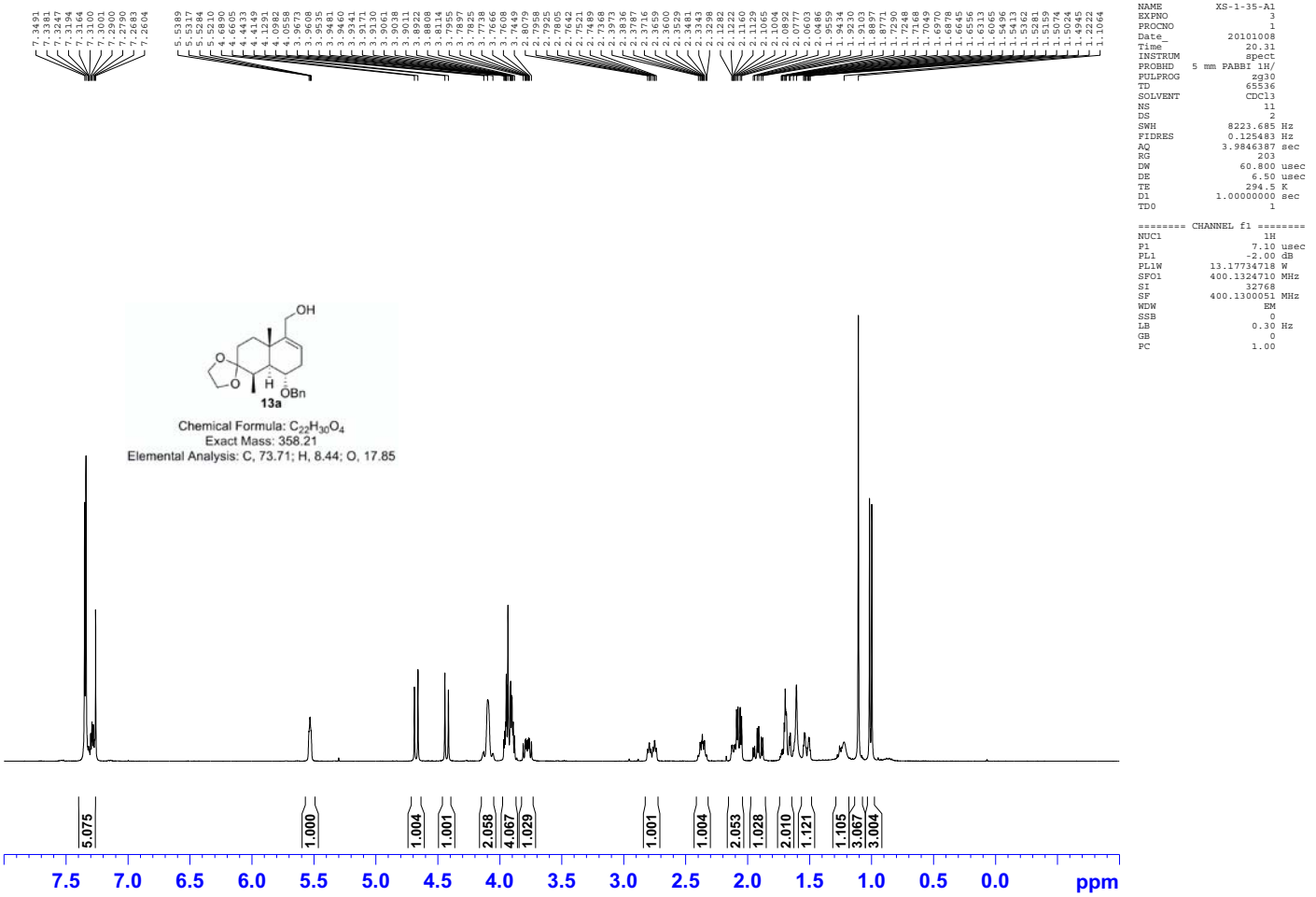

```
Bruker Advance III 400

NAME            XS-33-A1
EXPNO                  1
PROCNO                 1
Date_           20101004
Time                17.02
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                  90.5
DW                60.800 usec
DE                  6.50 usec
TE                294.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.10 usec
PL1                -2.00 dB
PL1W        13.17734718 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300051 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

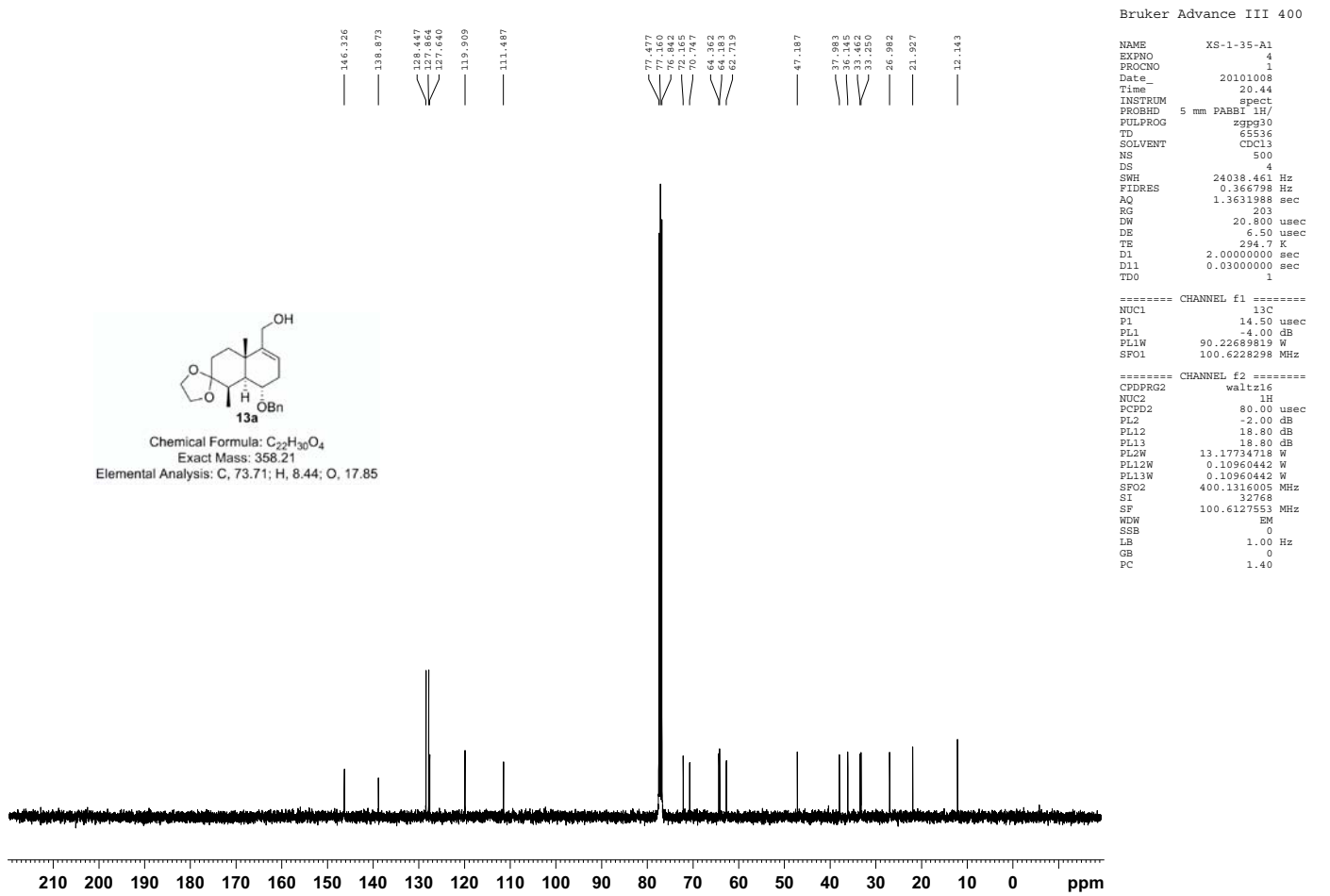


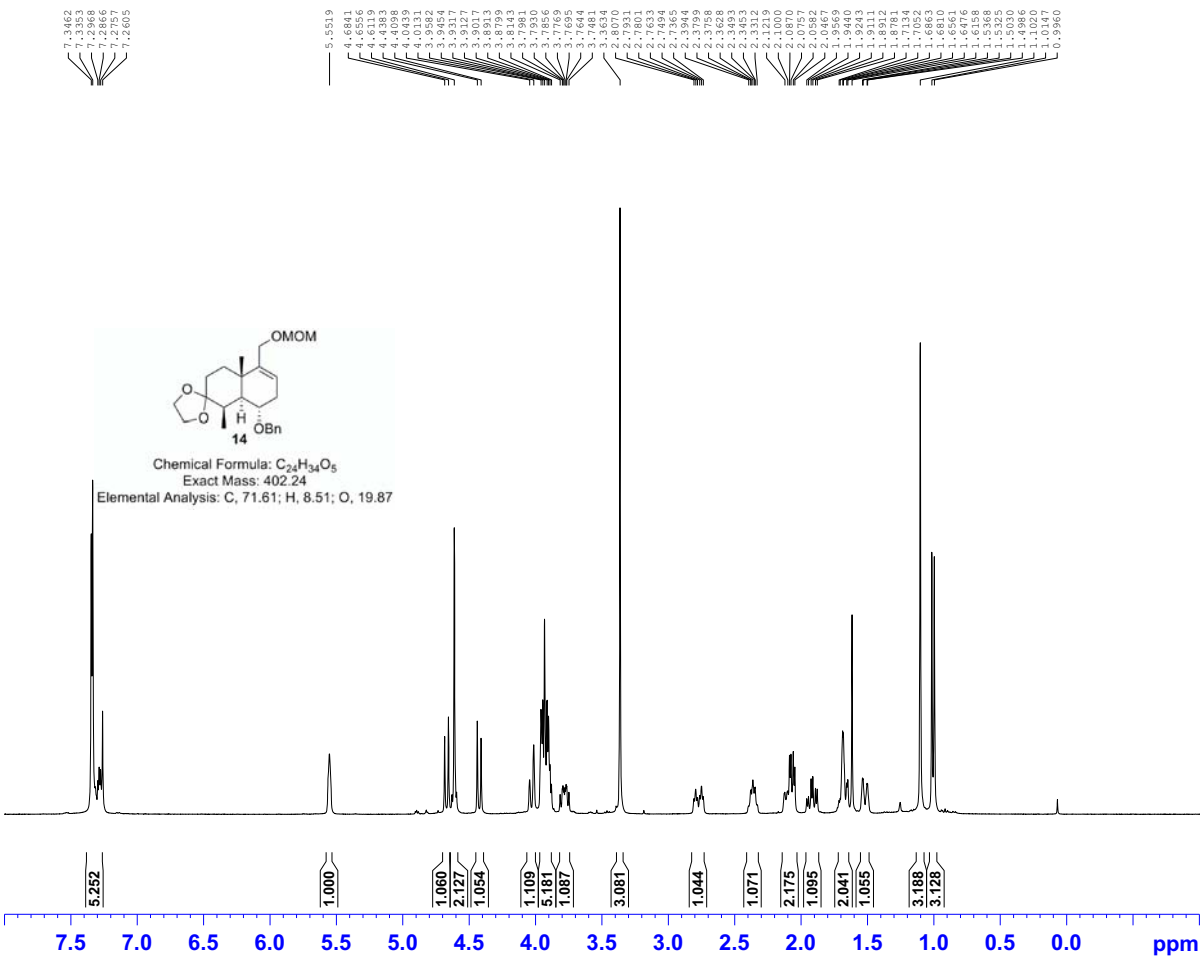

```
Bruker Advance III 400

NAME            XS-33-A1
EXPNO                  2
PROCNO                 1
Date_           20101004
Time                17.08
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   200
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                294.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.50 usec
PL1                -4.00 dB
PL1W        90.22689819 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.80 dB
PL13               18.80 dB
PL2W        13.17734718 W
PL12W        0.10960442 W
PL13W        0.10960442 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127561 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


Bruker Advance III 400
NAME XS-1-38-A1
EXPNO 1
PROCNO 1
Date_ 20101006
Time 11.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
COOMe
OBn
13
Chemical Formula: C23H30O5
Exact Mass: 386.21
Elemental Analysis: C, 71.48; H, 7.82; O, 20.70
5.039
1.000
1.028
1.024
4.107
1.105
3.013
1.046
1.030
1.021
1.066
1.200
1.059
2.107
3.136
3.046
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


Bruker Advance III 400
NAME XS-1-38-A1
EXPNO 2
PROCNO 1
Date_ 20101006
Time 11.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 228
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6379183 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 18.62 dB
PL2W 13.56617069 W
PL12W 0.32844096 W
PL13W 0.14806664 W
SFO2 400.1916008 MHz
SI 32768
SF 100.6278430 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
167.277
140.624
138.669
135.193
128.495
127.861
127.745
111.367
77.478
77.160
76.843
71.406
71.041
64.386
64.166
51.384
47.557
38.053
36.092
33.845
33.085
27.050
21.198
12.182
COOMe
OBn
13
Chemical Formula: C23H30O5
Exact Mass: 386.21
Elemental Analysis: C, 71.48; H, 7.82; O, 20.70
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

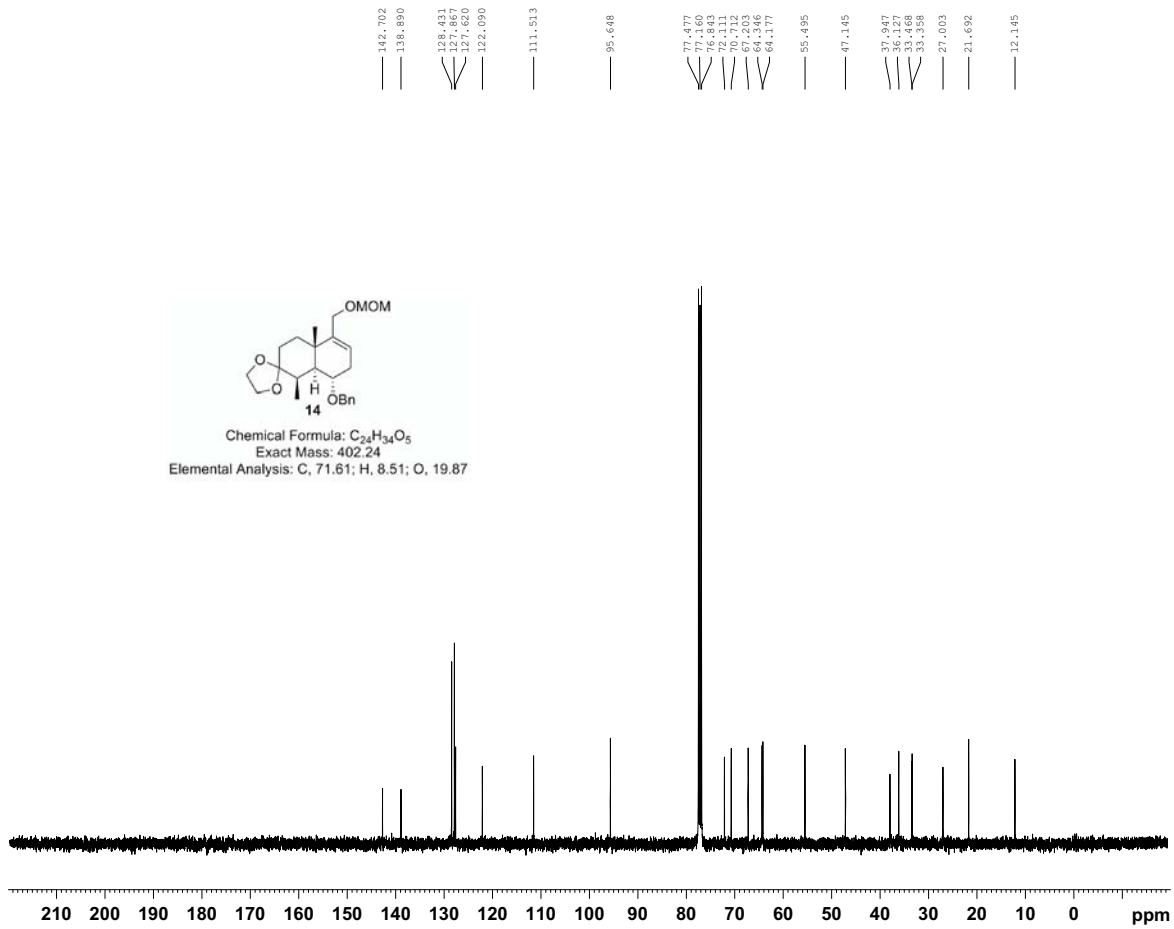

Bruker Advance III 400
NAME XS-1-35-A1
EXPNO 3
PROCNO 1
Date_ 20101008
Time 20.31
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.10 usec
PL1 -2.00 dB
PL1W 13.17734718 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OBn
13a
Chemical Formula: C22H30O4
Exact Mass: 358.21
Elemental Analysis: C, 73.71; H, 8.44; O, 17.85
5.075
1.000
1.004
1.001
2.058
4.067
1.029
1.001
1.004
2.053
1.028
2.010
1.121
1.105
3.067
3.004
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


Bruker Advance III 400
NAME XS-1-35-A1
EXPNO 4
PROCNO 1
Date_ 20101008
Time 20.44
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
146.326
138.873
128.447
127.864
127.640
119.909
111.487
77.477
77.160
76.842
72.165
70.747
64.362
64.183
62.719
47.187
37.983
36.145
33.462
33.290
26.982
21.927
12.143
OH
OBn
13a
Chemical Formula: C22H30O4
Exact Mass: 358.21
Elemental Analysis: C, 73.71; H, 8.44; O, 17.85
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



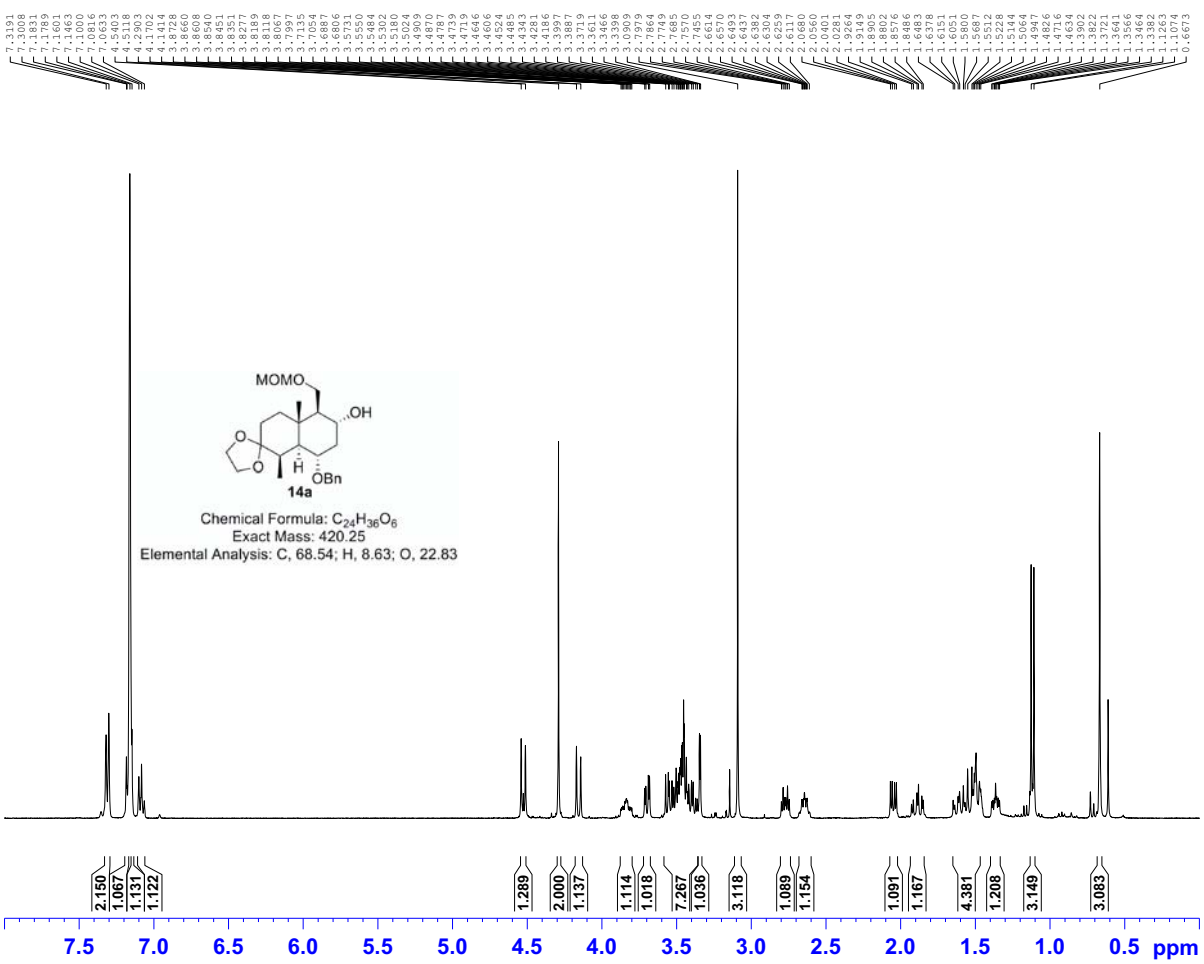
Bruker Advance III 400

```
NAME            XS-36-A1
EXPNO                  1
PROCNO                 1
Date_           20101004
Time                17.20
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     14
DS                      2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                  90.5
DW               60.800 usec
DE                 6.50 usec
TE                294.4 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.10 usec
PL1                -2.00 dB
PL1W        13.17734718 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300051 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

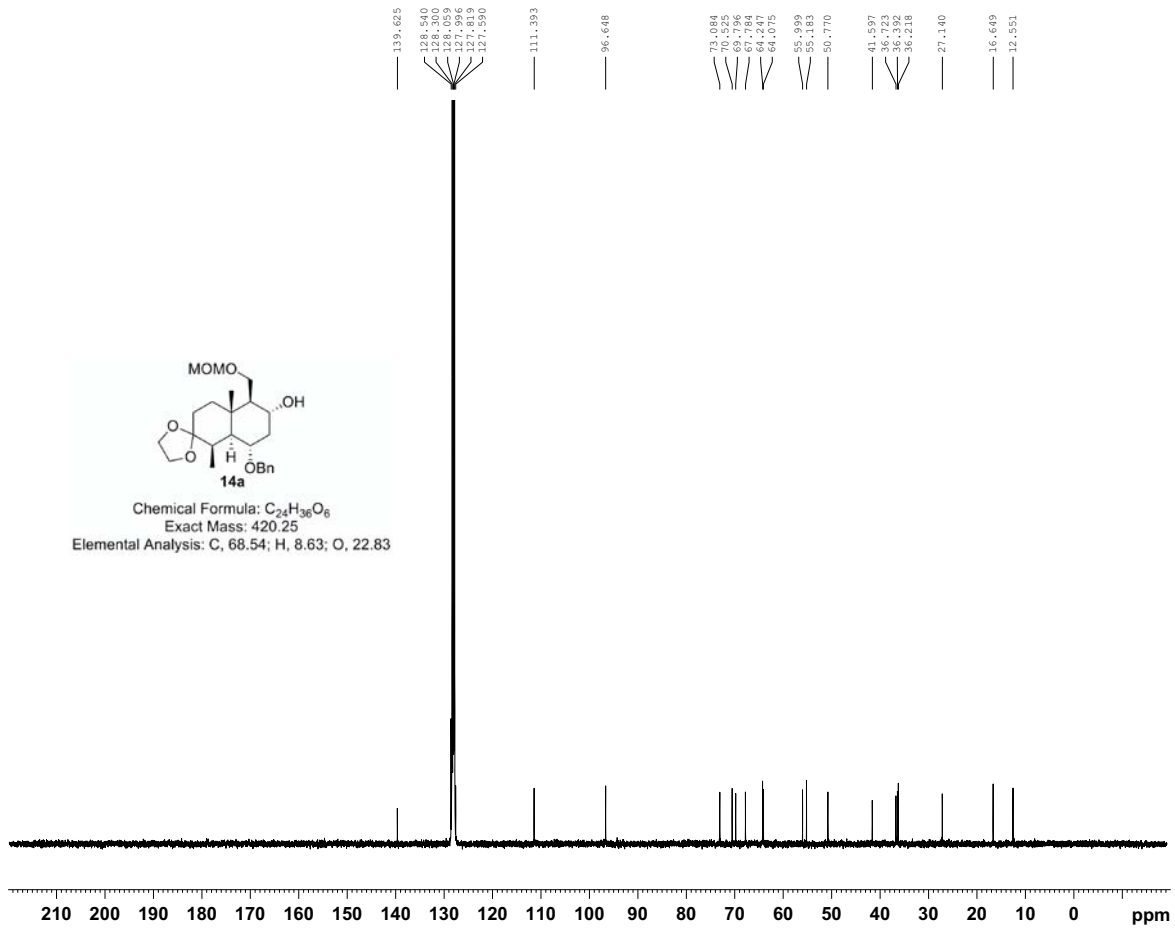


Bruker Advance III 400

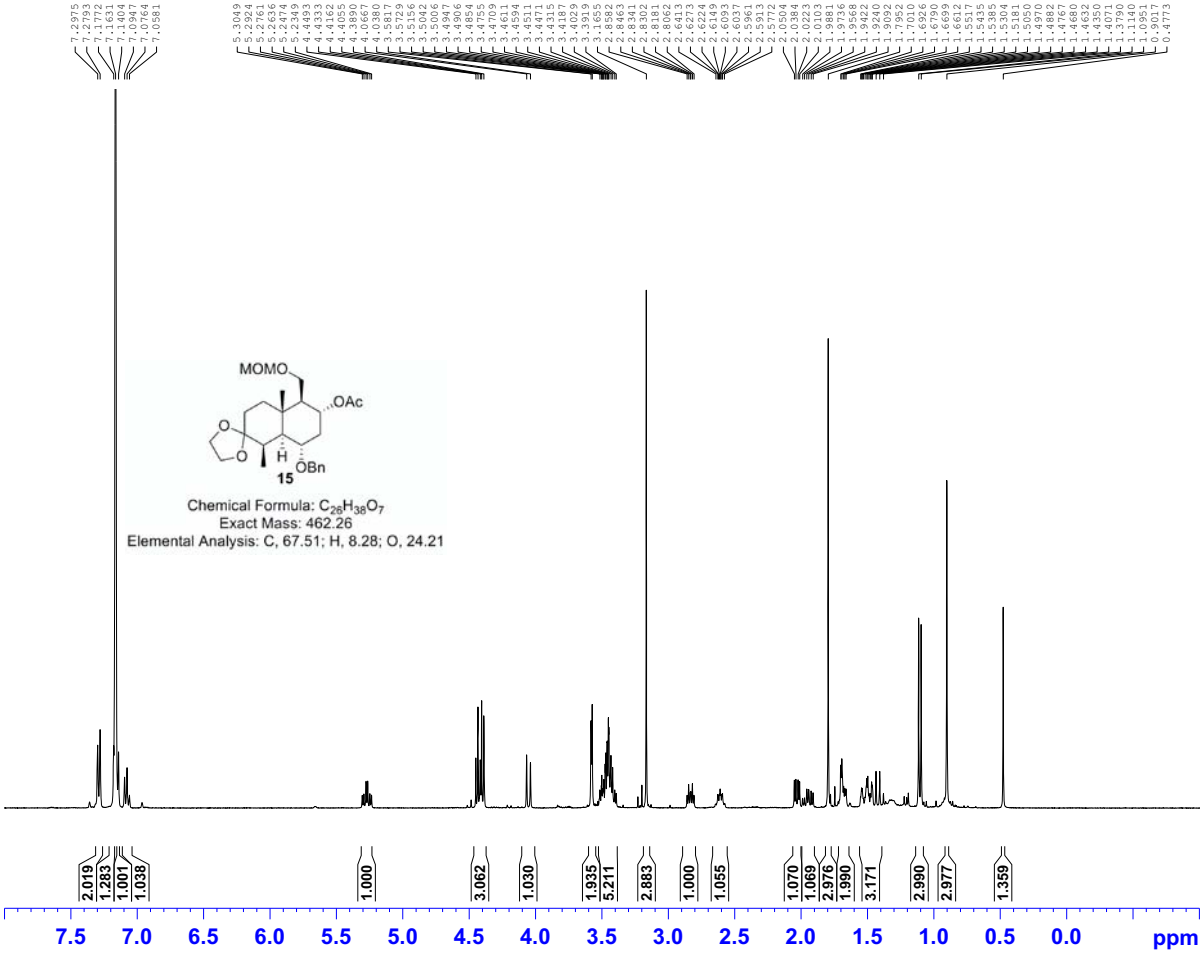
```
NAME            XS-36-A1
EXPNO                  2
PROCNO                 1
Date_           20101004
Time                17.28
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                    203
DW               20.800 usec
DE                 6.50 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.50 usec
PL1                -4.00 dB
PL1W        90.22689819 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.80 dB
PL13               18.80 dB
PL2W        13.17734718 W
PL12W        0.10960442 W
PL13W        0.10960442 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127557 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



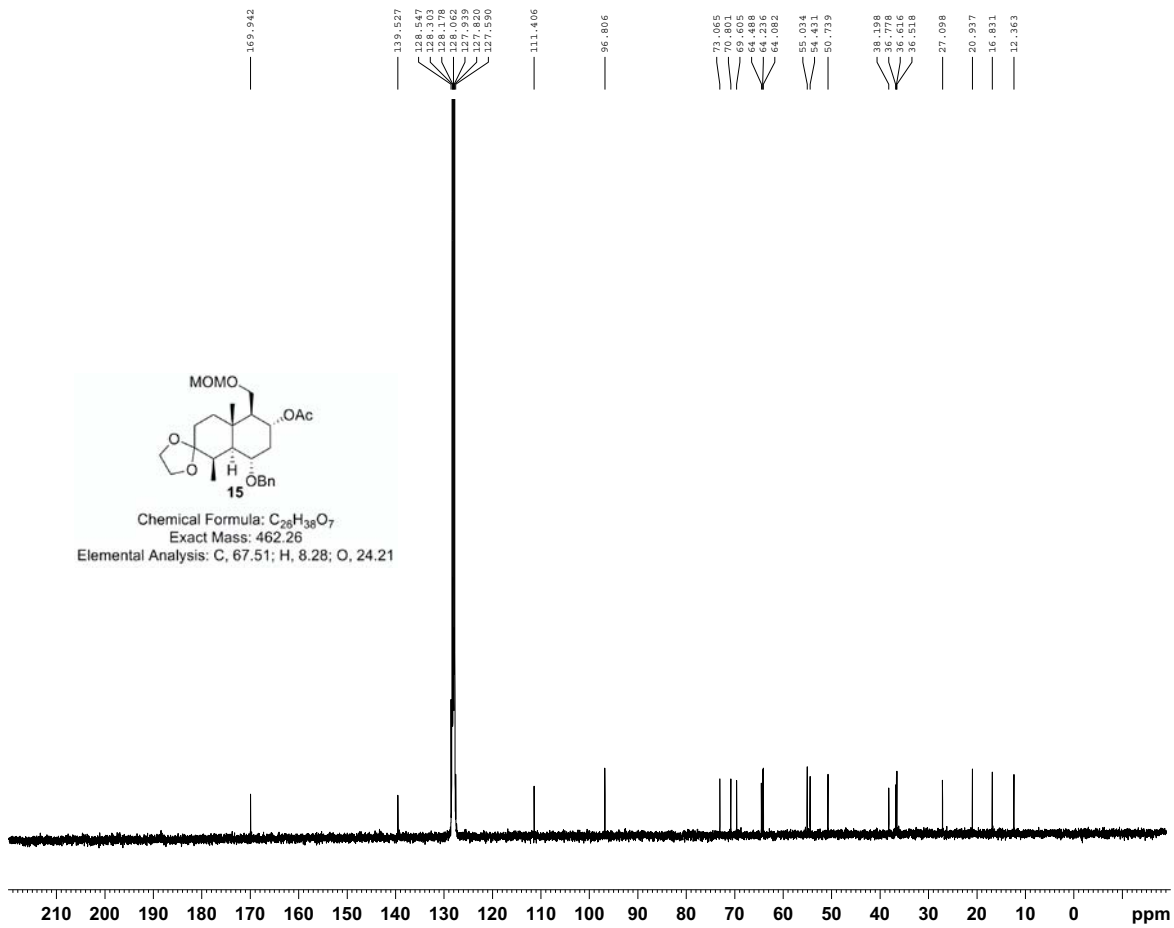

```
Bruker Advance III 400

NAME          XS-1-37-A1
EXPNO                  3
PROCNO                 1
Date_           20101027
Time                17.38
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT              C6D6
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   203
DW                60.800 usec
DE                  6.50 usec
TE                 295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.83 usec
PL1                 0.00 dB
PL1W          8.31434441 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300444 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



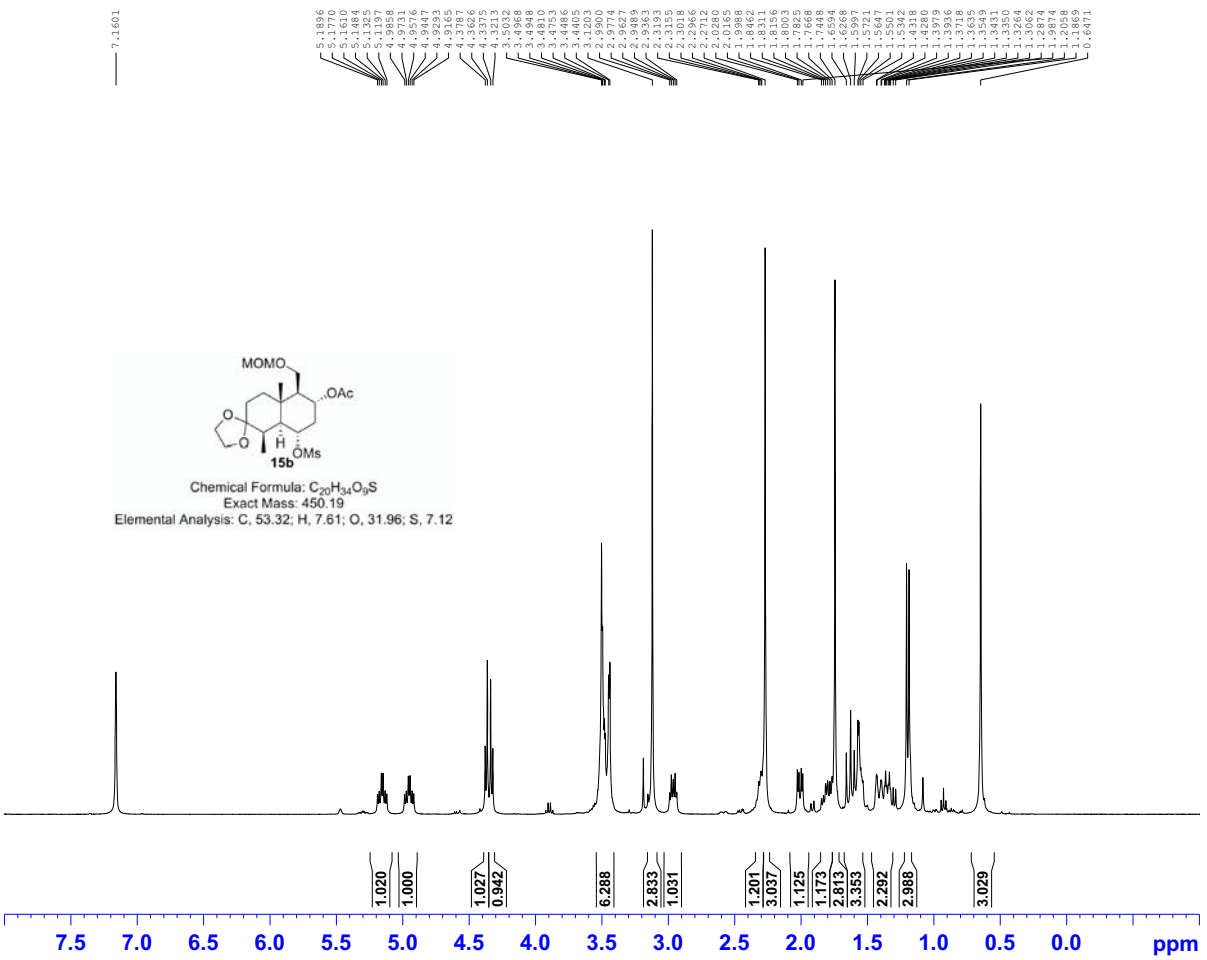

```
Bruker Advance III 400

NAME          XS-1-37-A1
EXPNO                  4
PROCNO                 1
Date_           20101027
Time                18.08
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                  65536
SOLVENT              C6D6
NS                    500
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                 295.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.68 usec
PL1                -0.60 dB
PL1W         41.24164963 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 0.00 dB
PL12               15.66 dB
PL13               15.92 dB
PL2W          8.31434441 W
PL12W         0.22585411 W
PL13W         0.21272963 W
SFO2         400.1316005 MHz
SI                 32768
SF          100.6127436 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



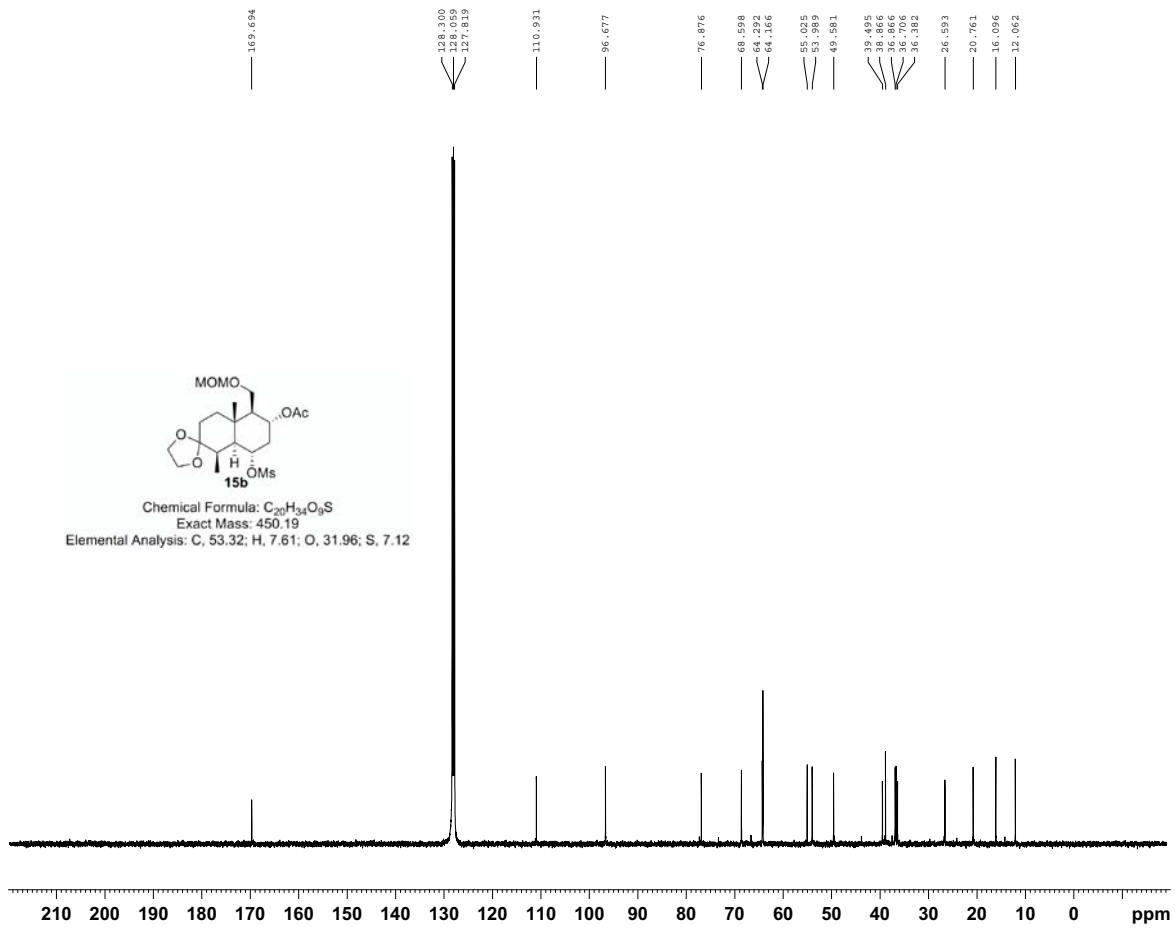

```
Bruker Advance III 400

NAME          XS-1-39-A1
EXPNO                  7
PROCNO                 1
Date_           20101025
Time_             21.19
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            C6D6
NS                    16
DS                     2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                   101
DW              60.800 usec
DE                6.50 usec
TE                297.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                32768
SF          400.1900000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



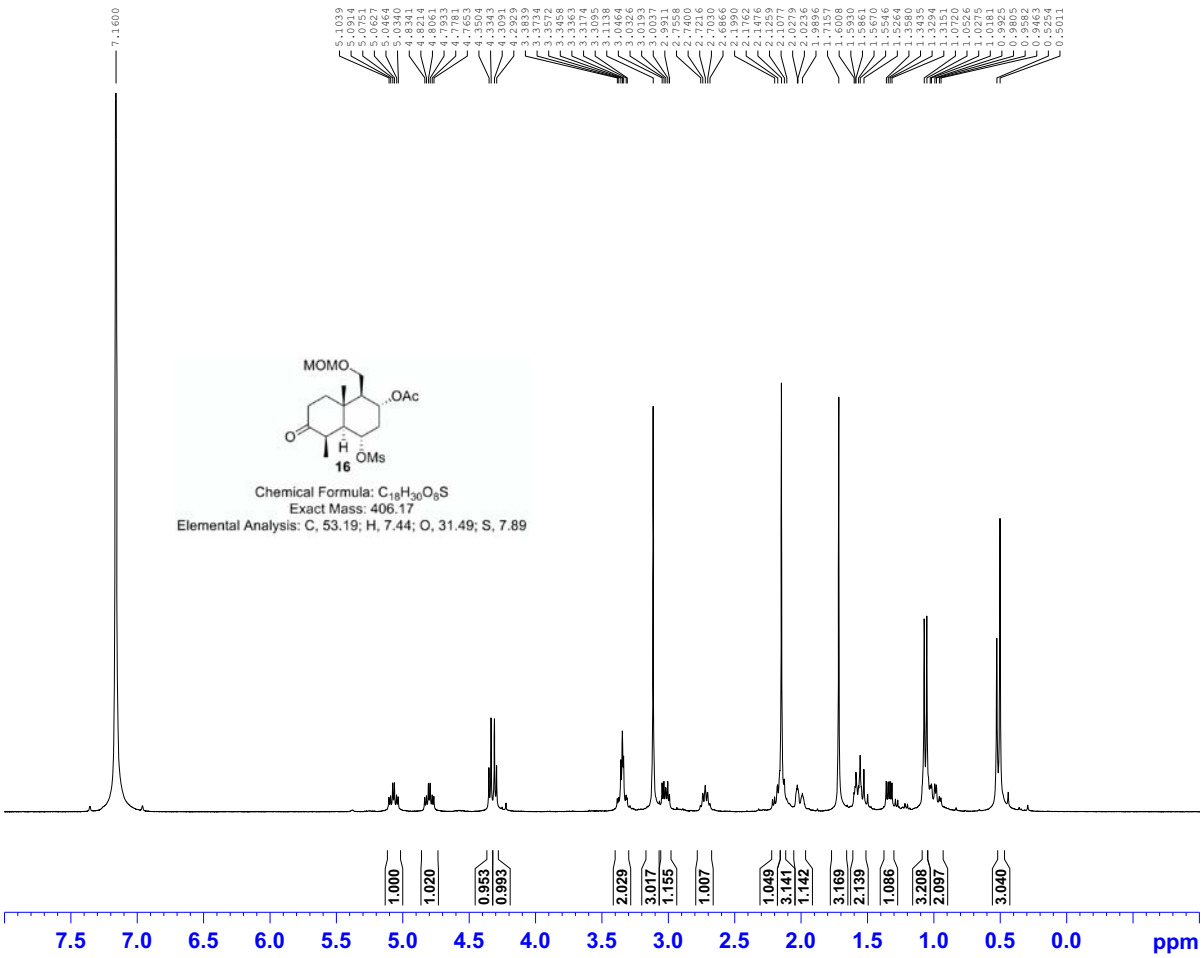

```
Bruker Advance III 400

NAME          XS-1-39-A1
EXPNO                  8
PROCNO                 1
Date_           20101025
Time_             21.32
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            C6D6
NS                   615
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ           1.3631988 sec
RG                   203
DW              20.800 usec
DE                6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.90 usec
PL1               -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                32768
SF          100.6278192 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



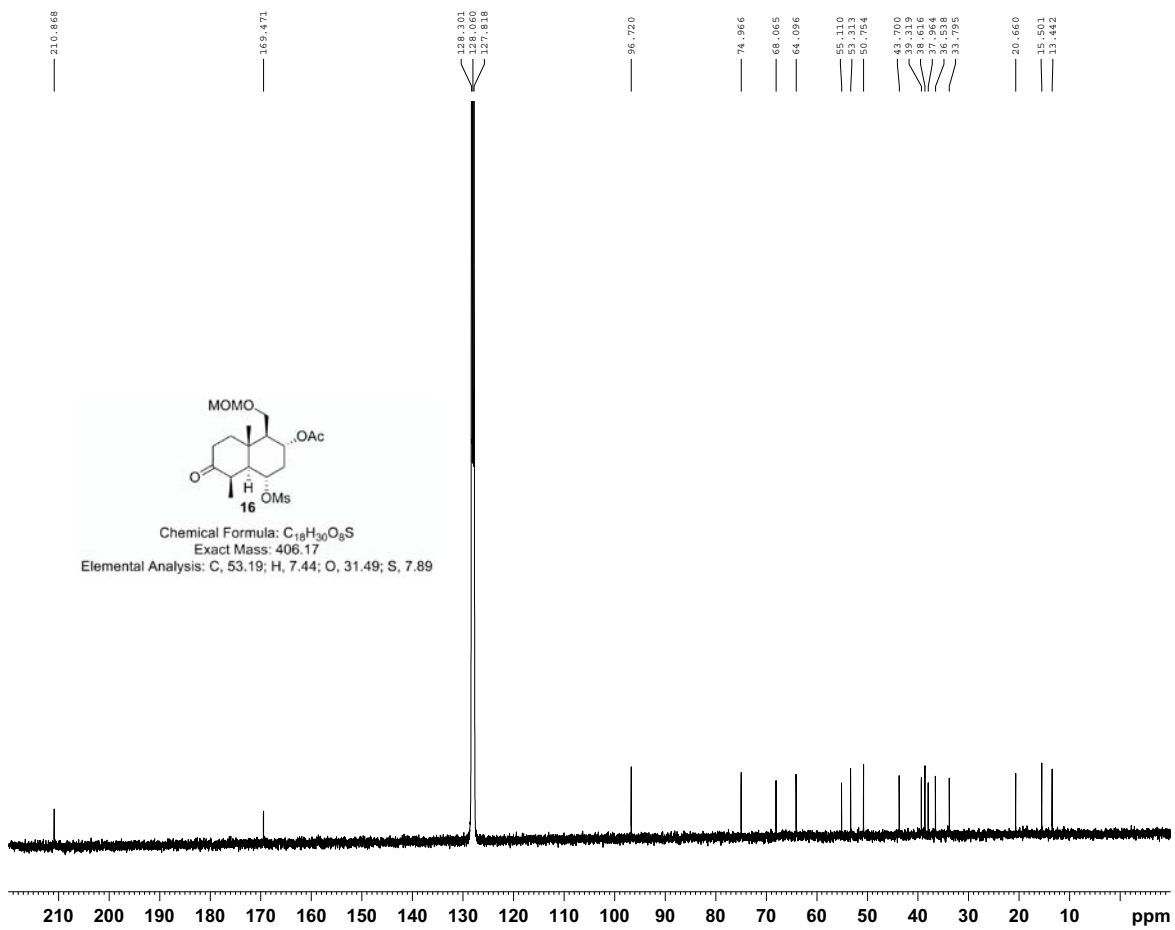

```
Bruker Advance III 400

NAME          XS-1-43-A1
EXPNO                  2
PROCNO                 1
Date_           20111129
Time                20.15
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT              C6D6
NS                     4
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.98466387 sec
RG                  25.4
DW                60.800 usec
DE                  6.50 usec
TE                294.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.10 usec
PL1                -2.00 dB
PL1W          13.17734718 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300438 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



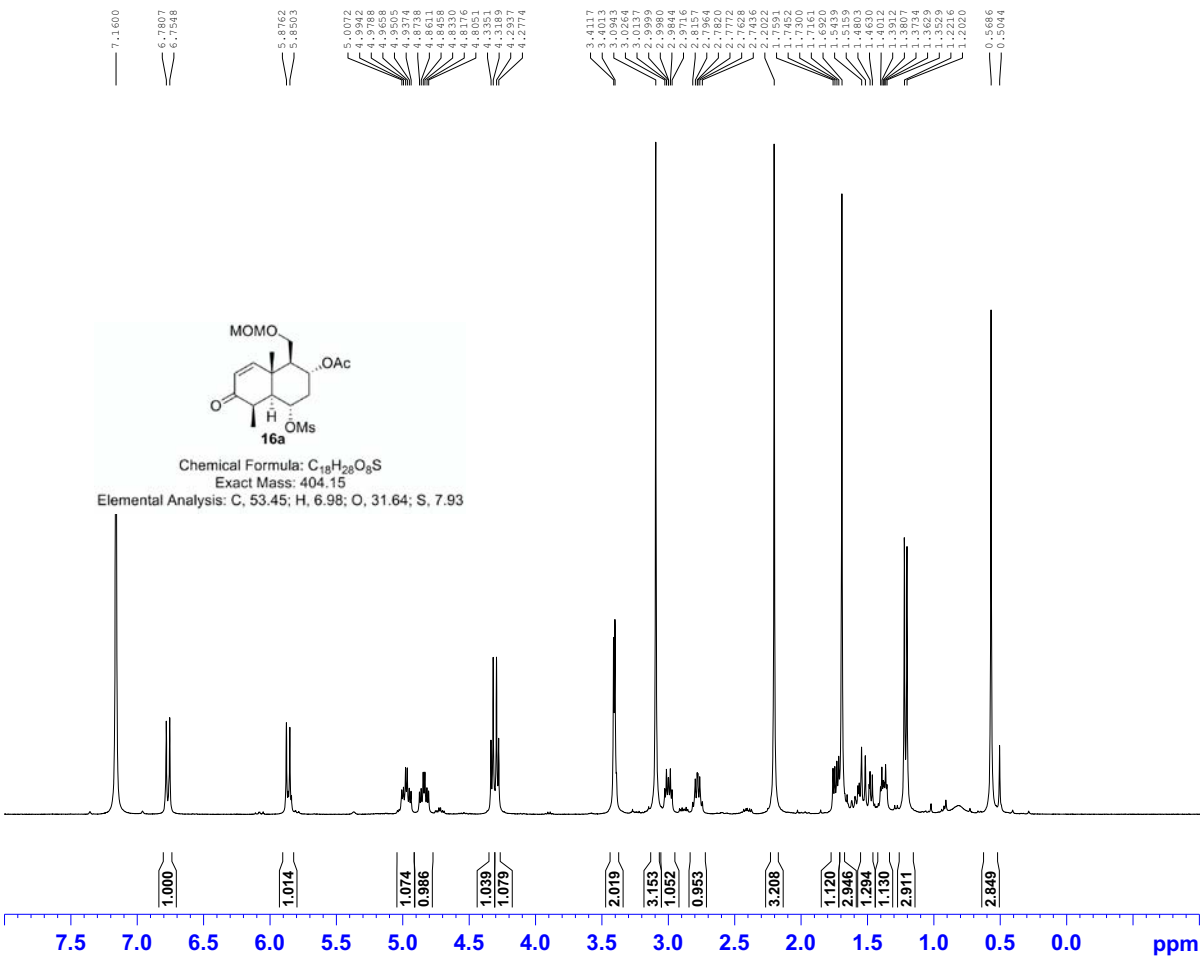

```
Bruker Advance III 400

NAME          XS-1-43-A1
EXPNO                  3
PROCNO                 1
Date_           20111129
Time                20.22
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT              C6D6
NS                    202
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                294.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.50 usec
PL1                -4.00 dB
PL1W          90.22689819 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.00 dB
PL12               20.06 dB
PL13               22.00 dB
PL2W          13.17734718 W
PL12W          0.08200268 W
PL13W          0.05245997 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127444 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



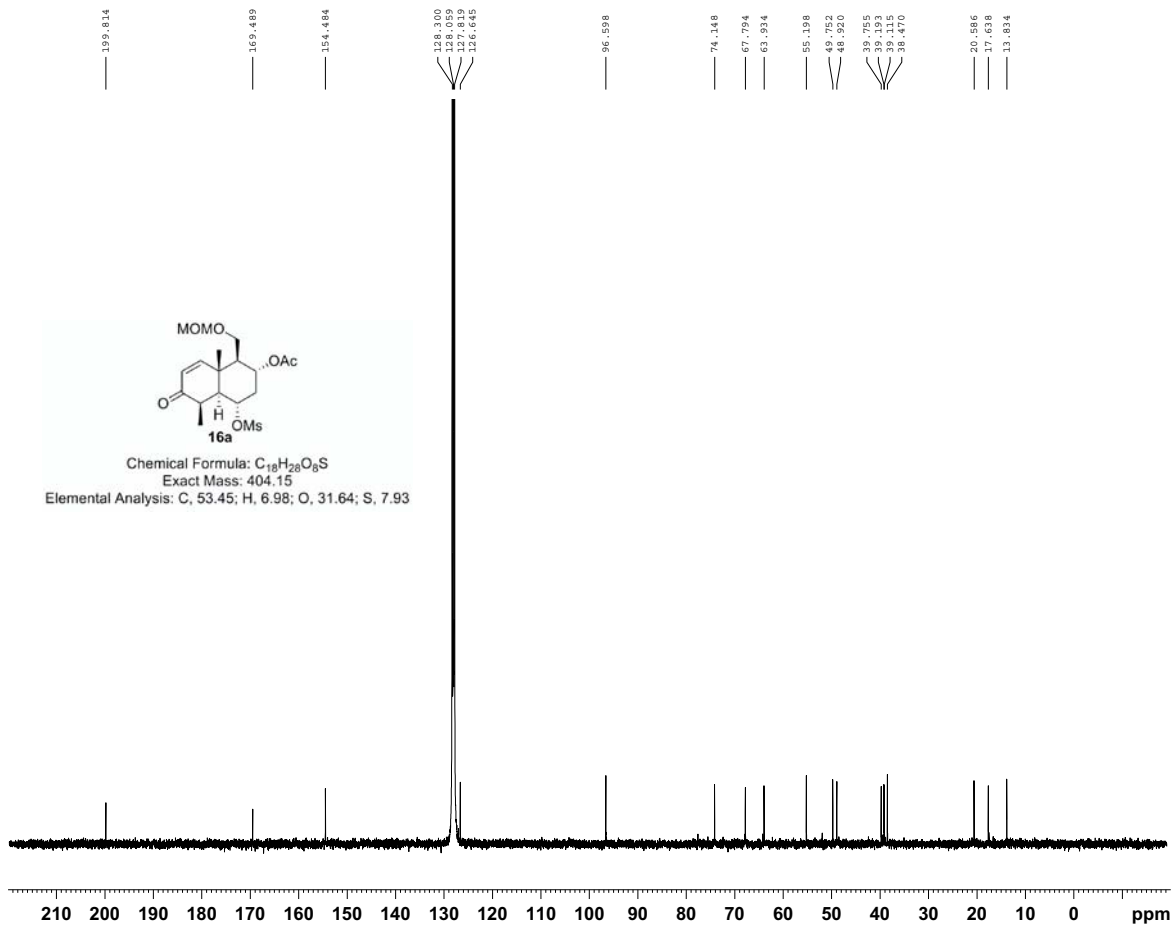

```
Bruker Advance III 400

NAME          XS-1-44-A1
EXPNO                 11
PROCNO                 1
Date_           20120303
Time                10.24
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              C6D6
NS                     16
DS                      2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                    114
DW                 60.800 usec
DE                  6.50 usec
TE                 298.9 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                 -1.00 dB
PL1W          13.56617069 W
SFO1          400.1924713 MHz
SI                  32768
SF            400.1900013 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



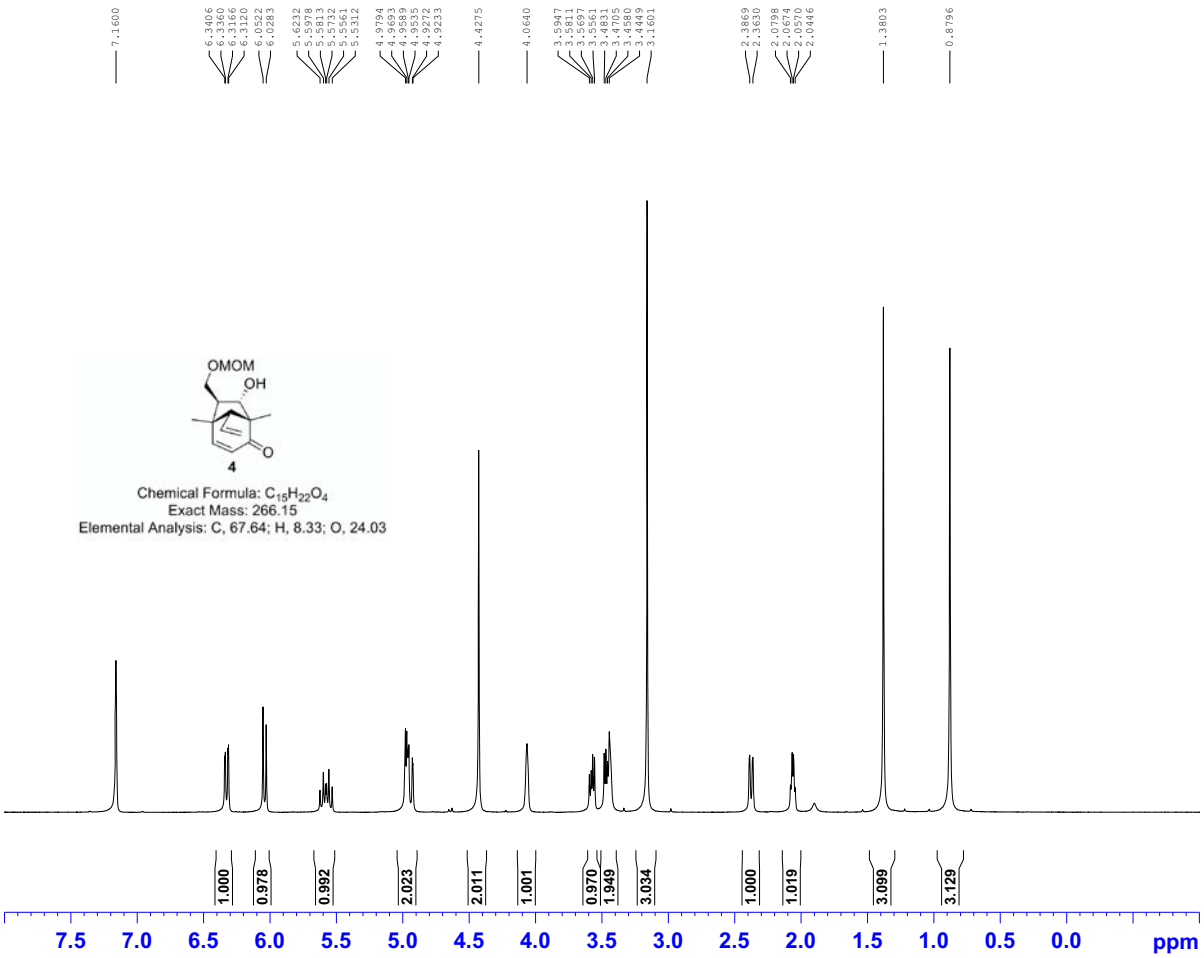

```
Bruker Advance III 400

NAME          XS-1-44-A1
EXPNO                 12
PROCNO                 1
Date_           20120303
Time                10.40
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT              C6D6
NS                    871
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                    228
DW                 20.800 usec
DE                  6.50 usec
TE                 299.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                 -2.00 dB
PL1W          55.33689499 W
SFO1          100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL13                18.62 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
PL13W          0.14806664 W
SFO2          400.1916008 MHz
SI                  32768
SF            100.6278192 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



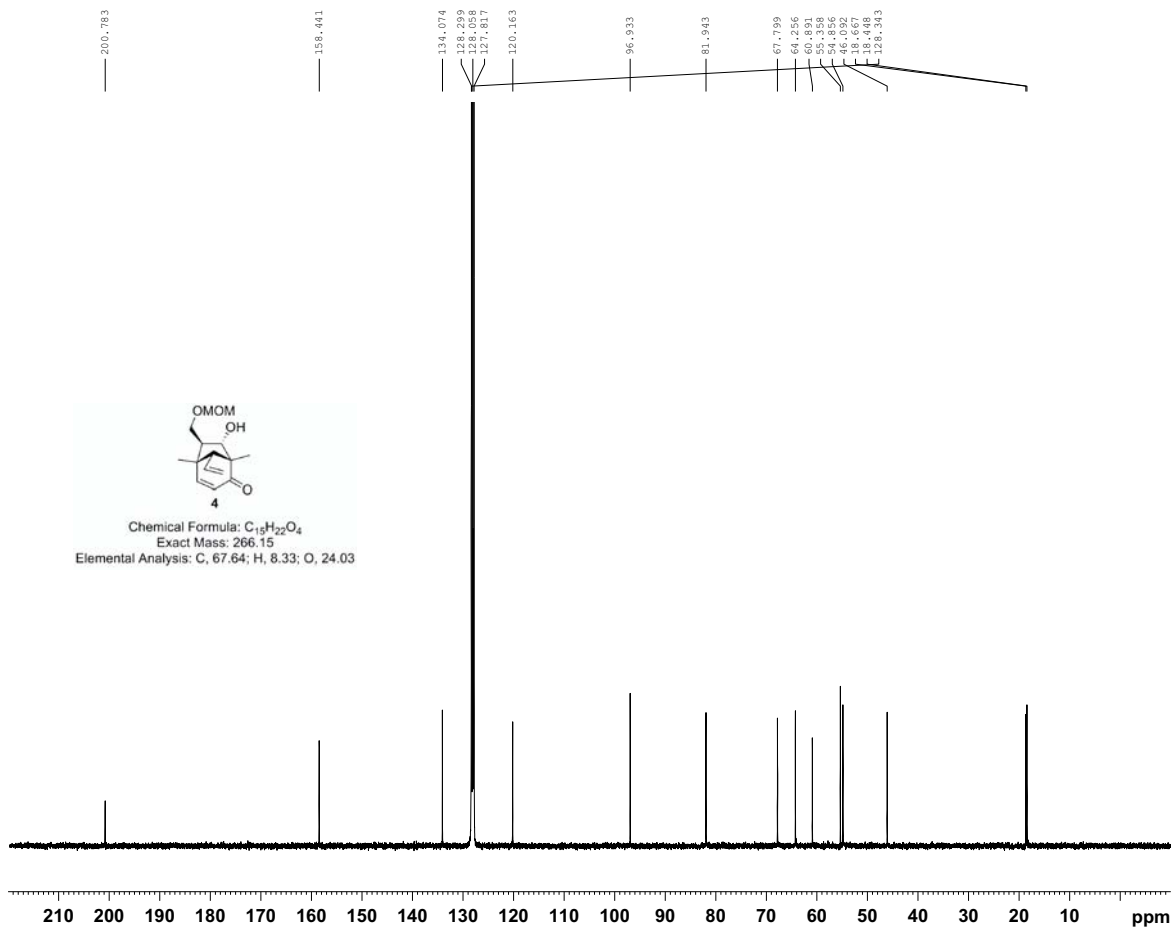

```
Bruker Advance III 400

NAME          XS-1-46-A1
EXPNO                  5
PROCNO                 1
Date_           20111205
Time                15.39
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                     4
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.98446387 sec
RG                  71.8
DW                60.800 usec
DE                  6.50 usec
TE                 295.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.10 usec
PL1                -2.00 dB
PL1W        13.17734718 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300440 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



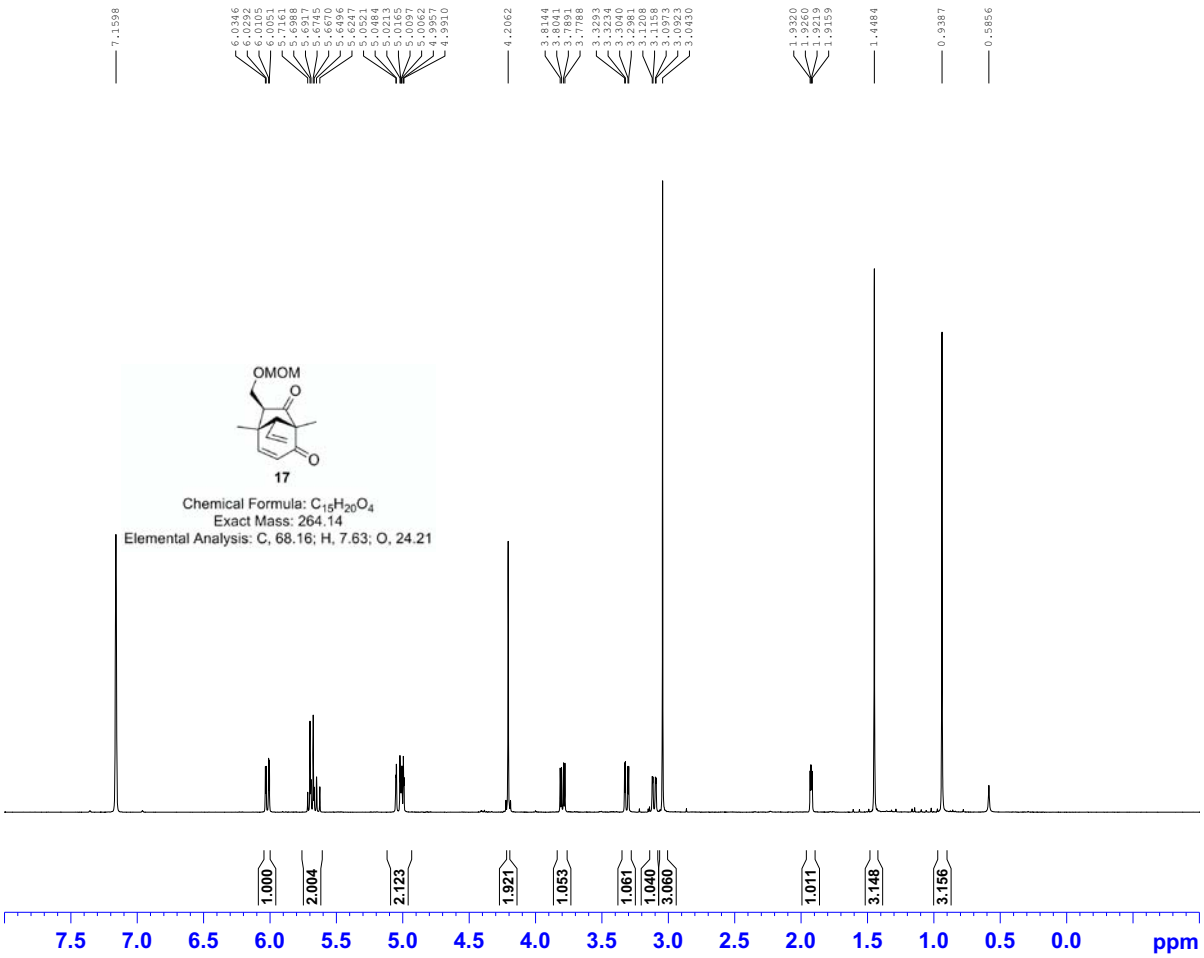

```
Bruker Advance III 400

NAME          XS-1-46-A1
EXPNO                  6
PROCNO                 1
Date_           20111205
Time                15.48
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT             C6D6
NS                   312
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                 295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.50 usec
PL1                -4.00 dB
PL1W        90.22689819 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.00 dB
PL12               20.06 dB
PL13               22.00 dB
PL2W        13.17734718 W
PL12W        0.08200268 W
PL13W        0.05245997 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127444 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



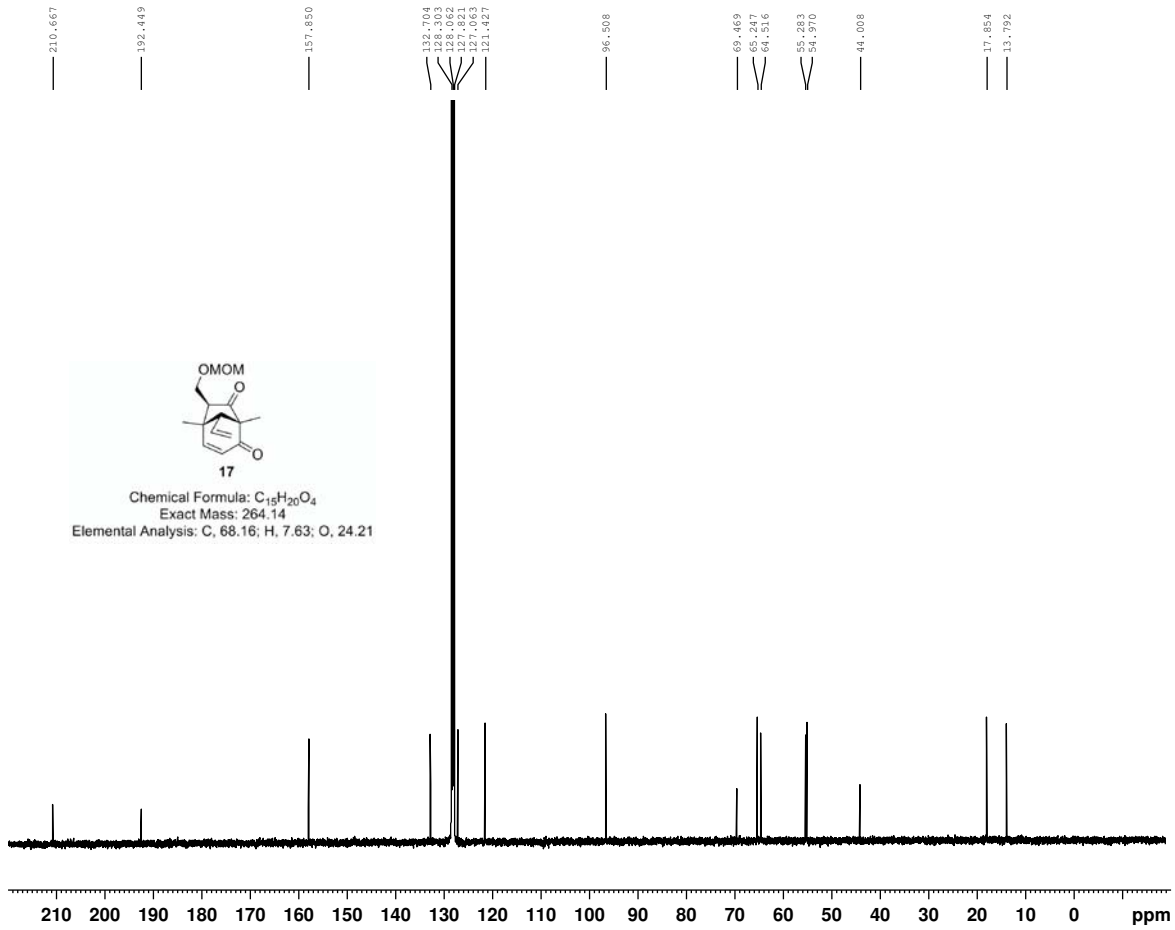

```
Bruker Advance III 400

NAME          XS-1-91-A1
EXPNO                  1
PROCNO                 1
Date_           20110409
Time                10.48
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT              C6D6
NS                      8
DS                      2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.98446387 sec
RG                   80.6
DW                60.800 usec
DE                  6.50 usec
TE                 294.1 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.83 usec
PL1                  0.00 dB
PL1W          8.31434441 W
SFO1        400.1324710 MHz
SI                  32768
SF          400.1300438 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



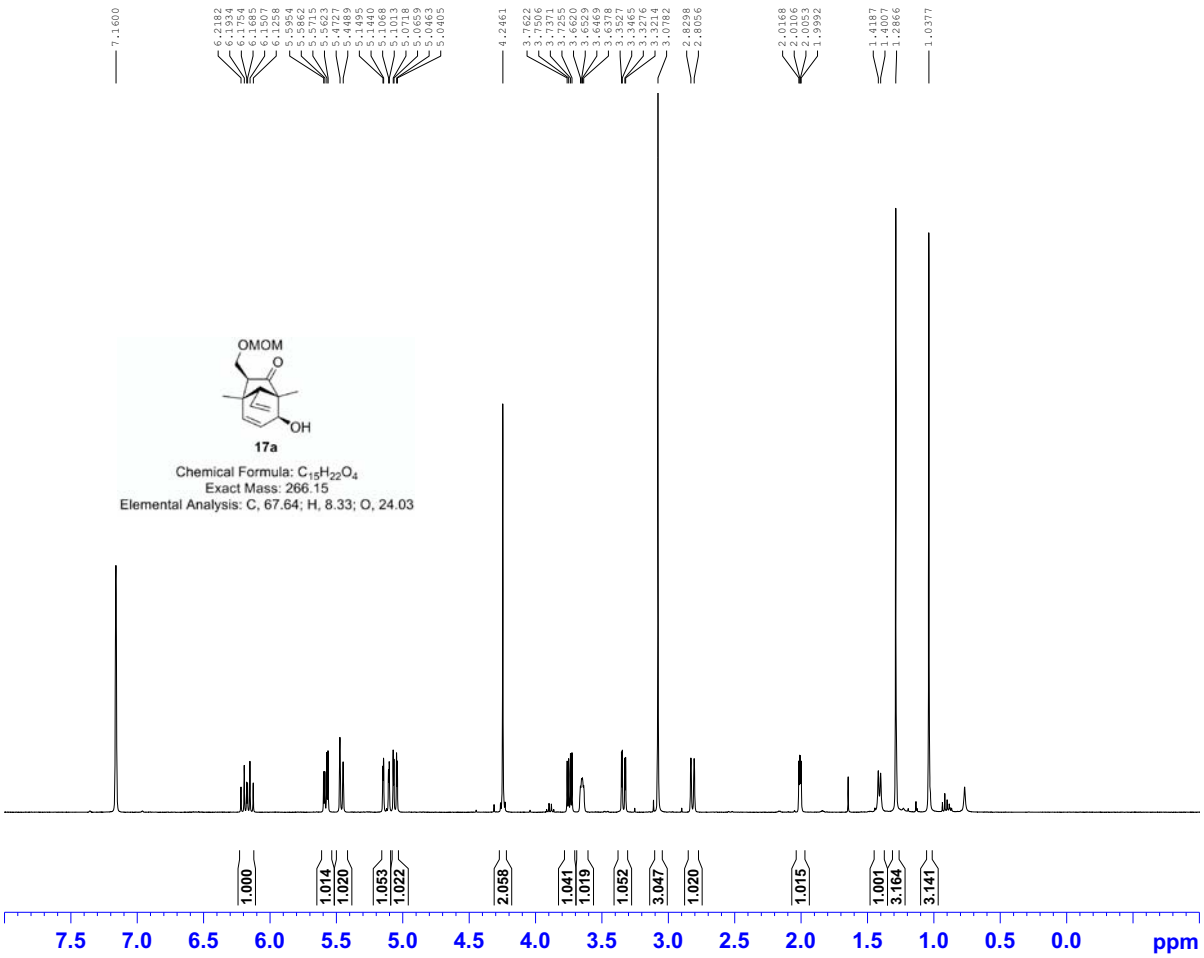

```
Bruker Advance III 400

NAME          XS-1-91-A1
EXPNO                  2
PROCNO                 1
Date_           20110409
Time                10.56
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                  65536
SOLVENT              C6D6
NS                    202
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                    203
DW                20.800 usec
DE                  6.50 usec
TE                 294.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.68 usec
PL1                 -0.60 dB
PL1W         41.24164963 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                  0.00 dB
PL12                15.66 dB
PL13                15.92 dB
PL2W          8.31434441 W
PL12W         0.22585411 W
PL13W         0.21272963 W
SFO2        400.1316005 MHz
SI                  32768
SF          100.6127444 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



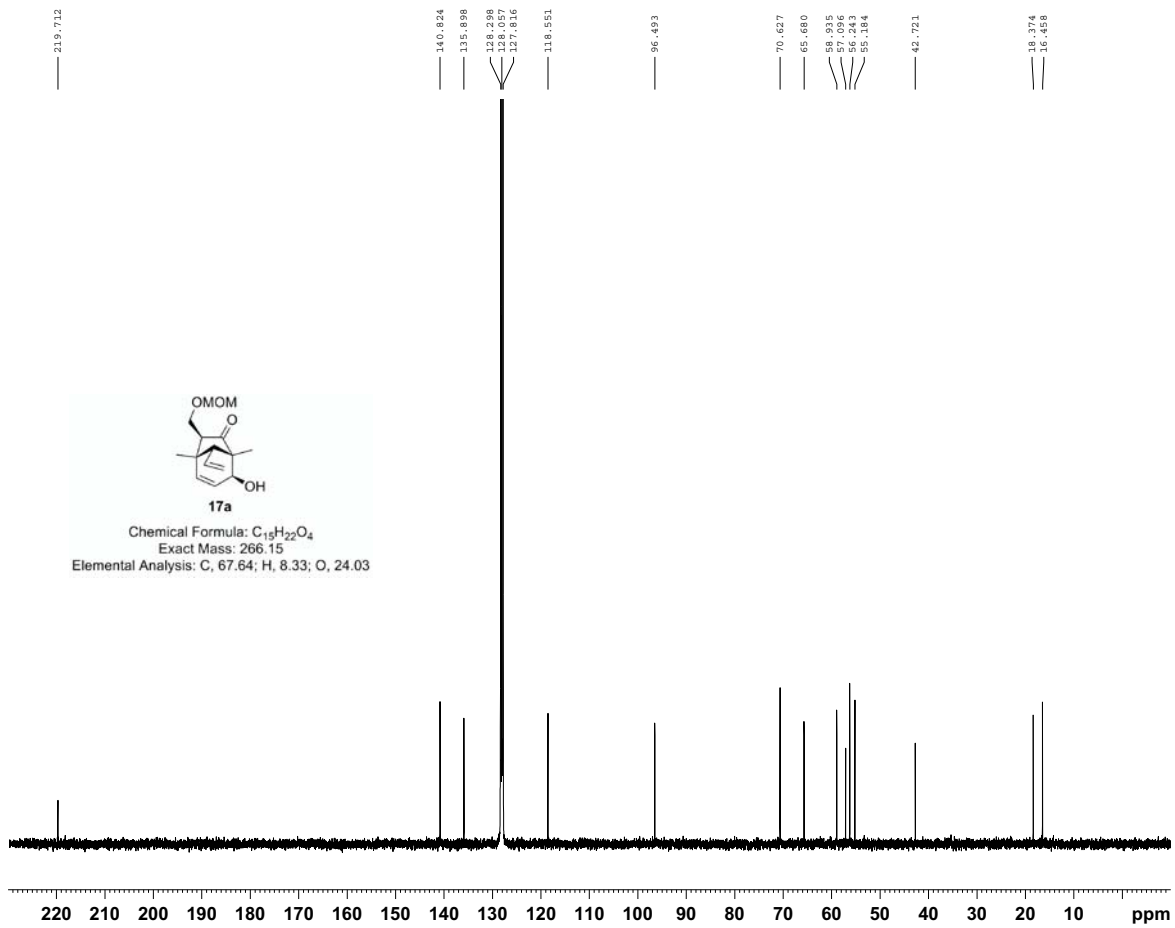

```
Bruker Advance III 400

NAME          XS-1-51-A1
EXPNO                  1
PROCNO                 1
Date_           20101203
Time               18.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                  71.8
DW                60.800 usec
DE                  6.50 usec
TE                 297.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900014 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



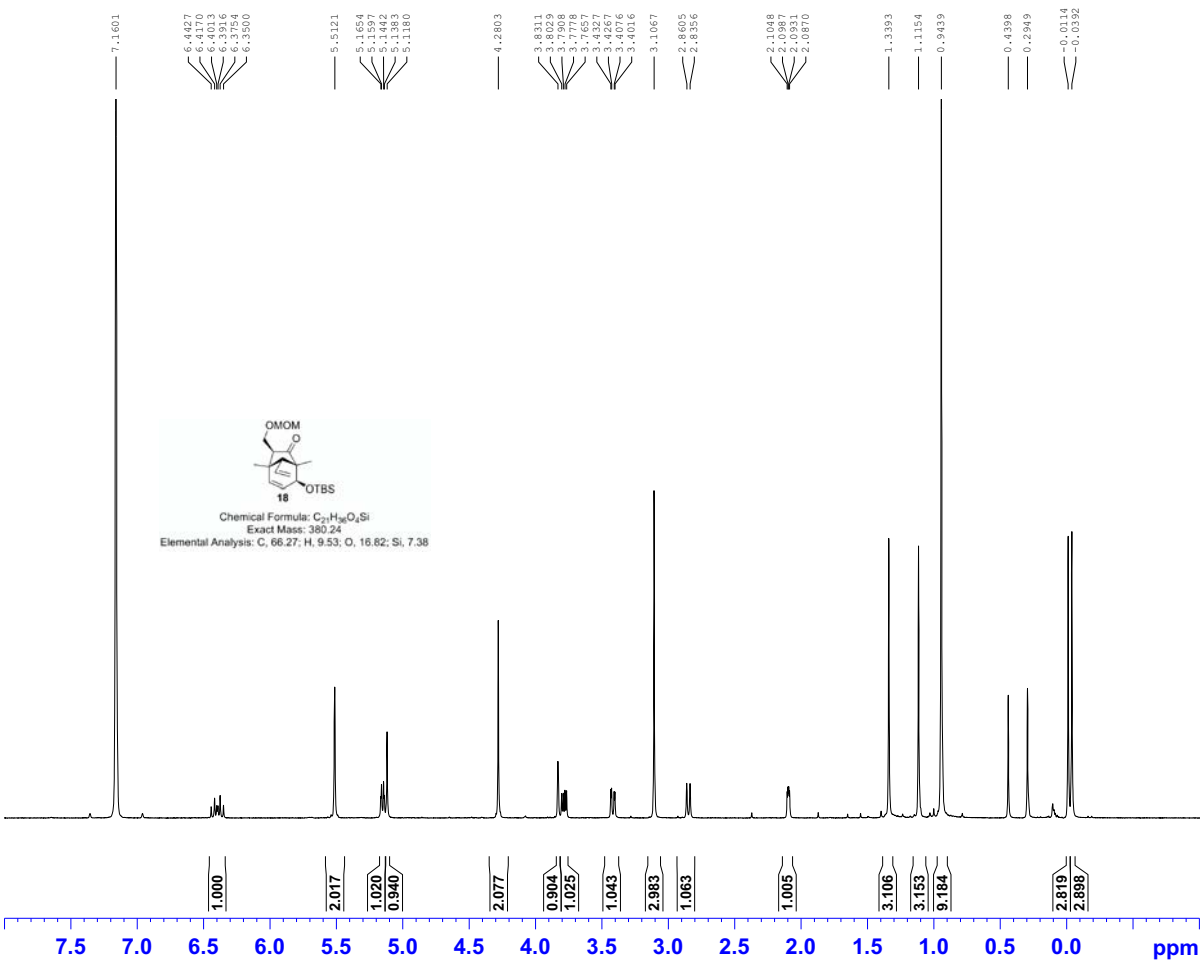

```
Bruker Advance III 400

NAME          XS-1-51-A1
EXPNO                  2
PROCNO                 1
Date_           20101203
Time               19.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT             C6D6
NS                   203
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ            1.3631988 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL13               18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                 32768
SF          100.6278192 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



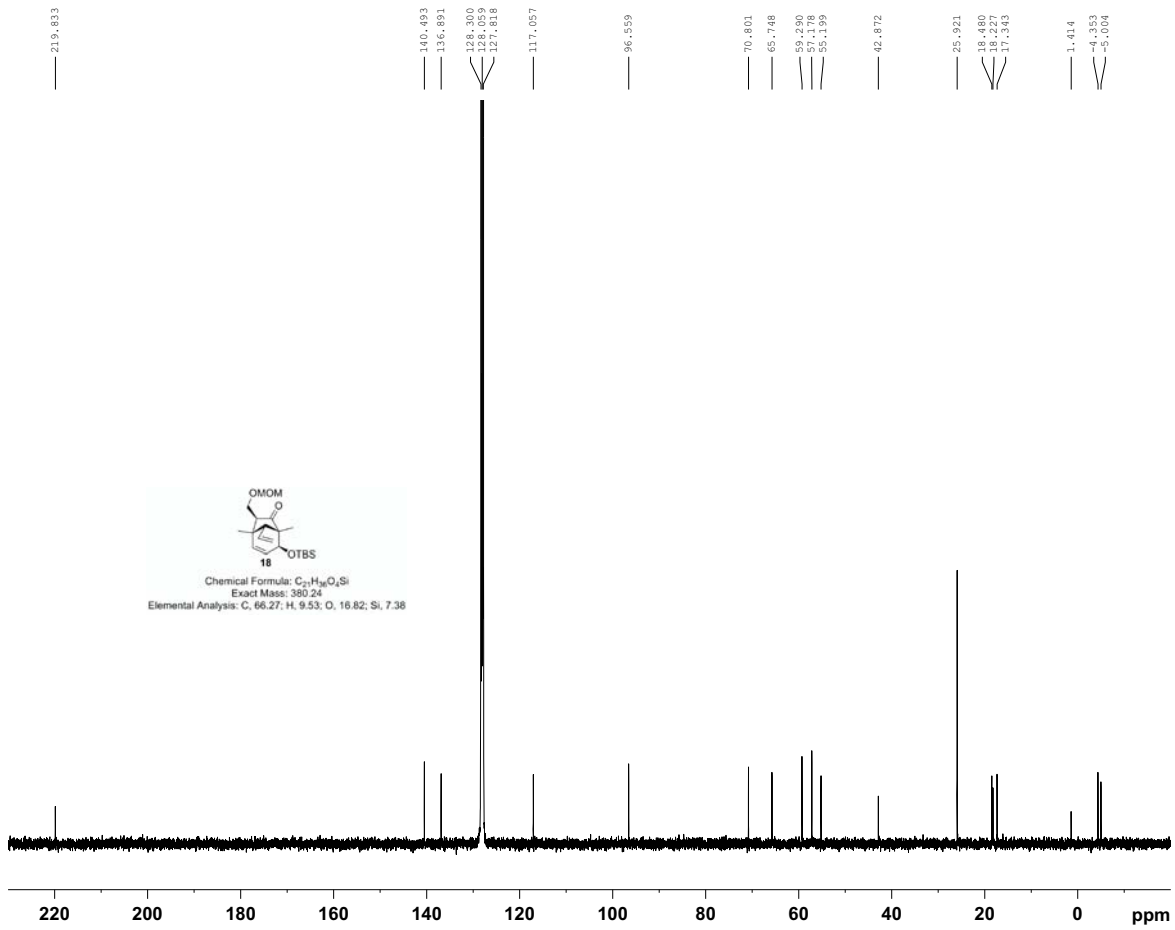

```
Bruker Advance III 400

NAME          XS-1-105-A1
EXPNO                   1
PROCNO                  1
Date_            20110426
Time_               19.34
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                   65536
SOLVENT               C6D6
NS                       8
DS                       2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                     161
DW                60.800 usec
DE                  6.50 usec
TE                 294.5 K
D1            1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.83 usec
PL1                   0.00 dB
PL1W          8.31434441 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300441 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



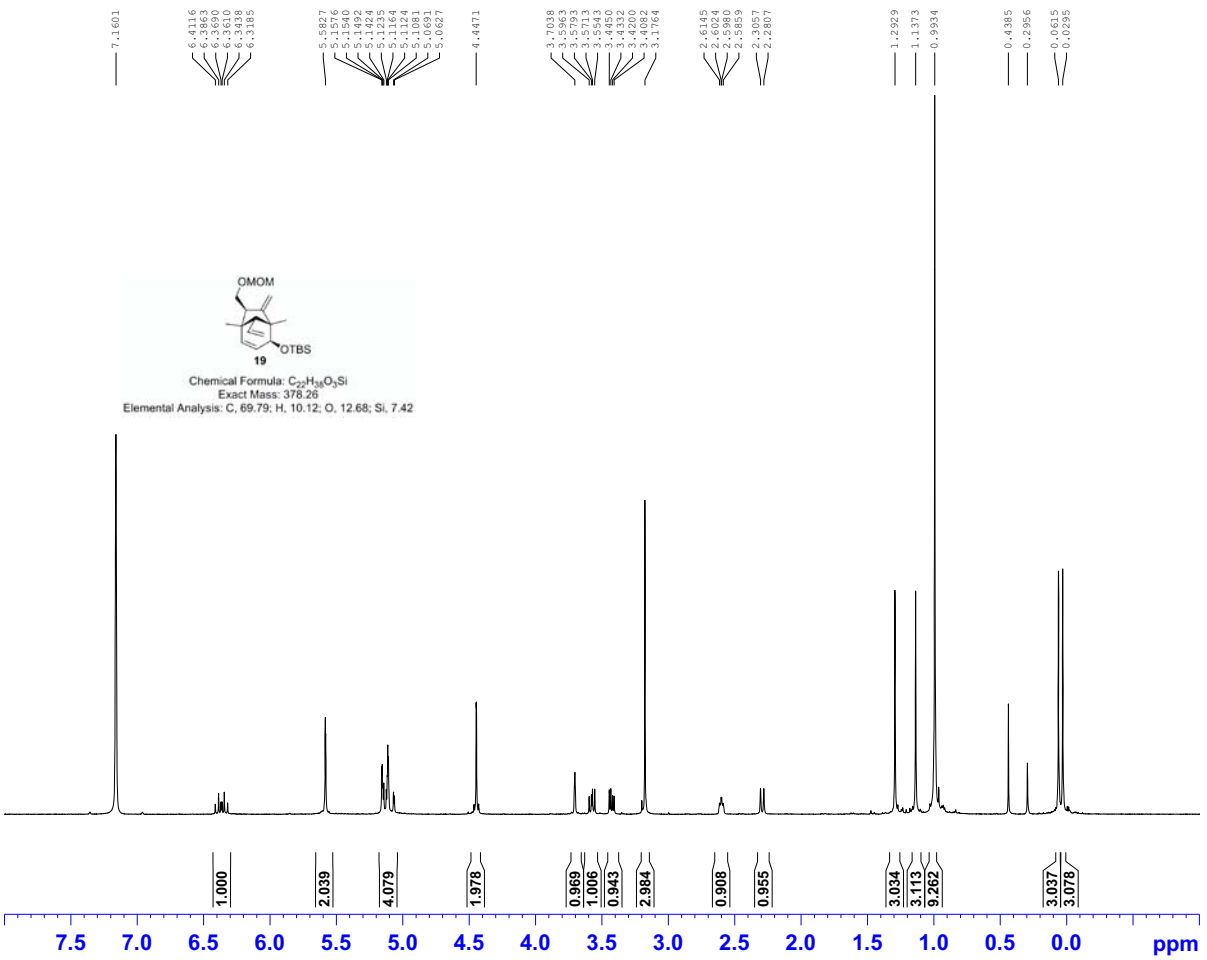

```
Bruker Advance III 400

NAME          XS-1-105-A1
EXPNO                   3
PROCNO                  1
Date_            20110426
Time_               20.29
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG           zgpg30
TD                   65536
SOLVENT               C6D6
NS                     178
DS                       4
SWH            26041.666 Hz
FIDRES          0.397364 Hz
AQ            1.2583412 sec
RG                     203
DW                19.200 usec
DE                  6.50 usec
TE                 295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.68 usec
PL1                  -0.60 dB
PL1W         41.24164963 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   0.00 dB
PL12                 15.66 dB
PL13                 15.92 dB
PL2W          8.31434441 W
PL12W         0.22585411 W
PL13W         0.21272963 W
SFO2         400.1316005 MHz
SI                   32768
SF           100.6127439 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



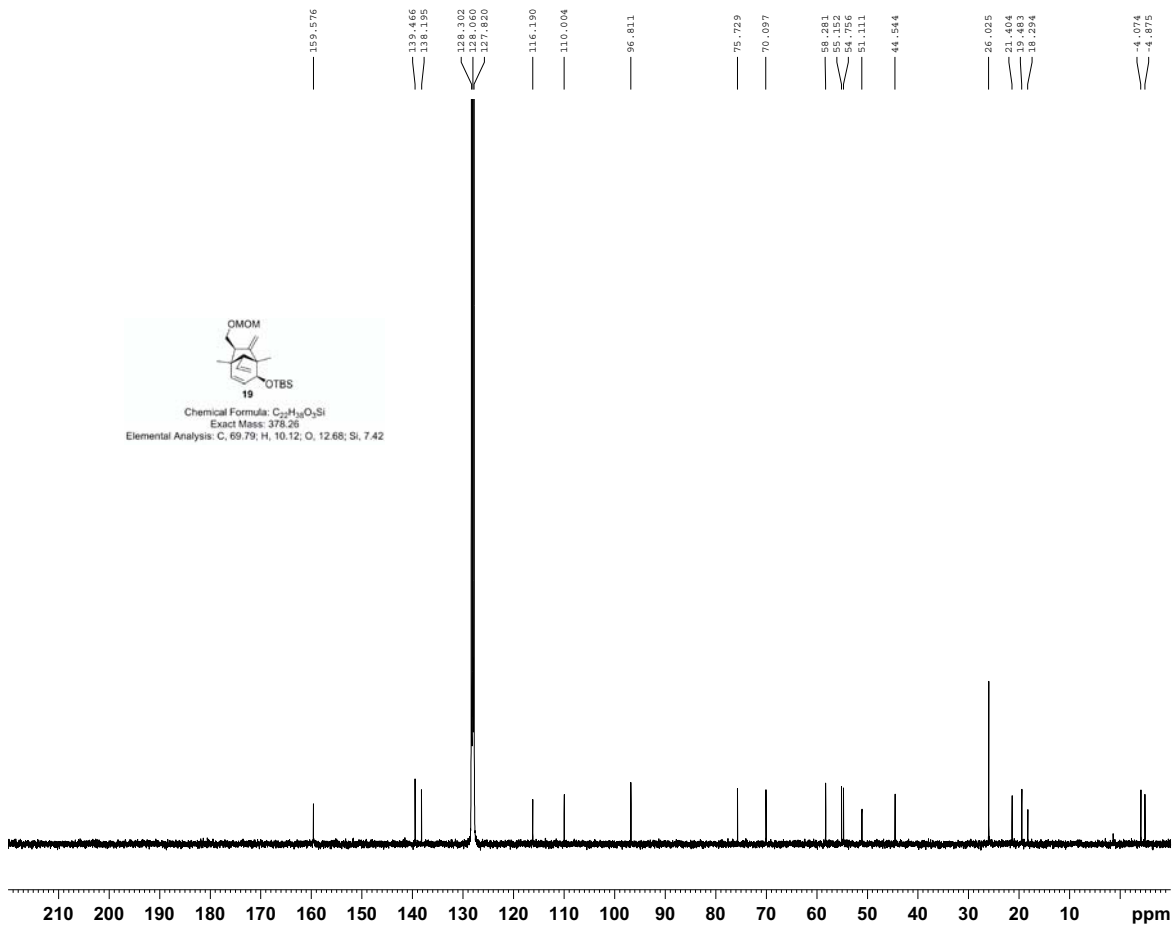

```
Bruker Advance III 400

NAME          XS-1-54-A1
EXPNO                  1
PROCNO                 1
Date_           20101218
Time                16.38
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   101
DW                60.800 usec
DE                 6.50 usec
TE                297.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                -1.00 dB
PL1W          13.56617069 W
SFO1          400.1924713 MHz
SI                 32768
SF            400.1900014 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



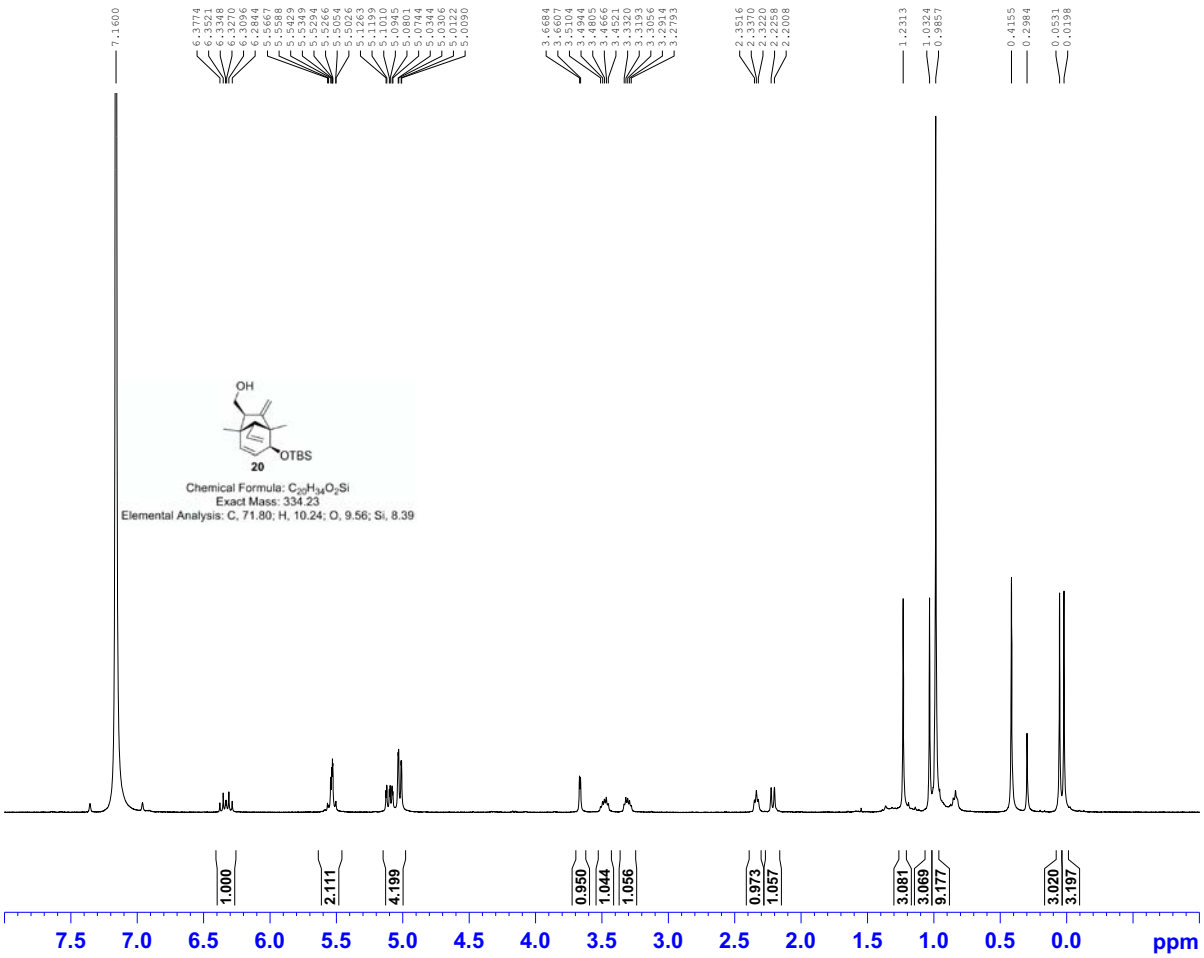

```
Bruker Advance III 400

NAME          XS-1-54-A1
EXPNO                  3
PROCNO                 1
Date_           20101218
Time                17.26
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             C6D6
NS                   841
DS                     4
SWH            28409.092 Hz
FIDRES          0.433488 Hz
AQ             1.1534836 sec
RG                   181
DW                17.600 usec
DE                 6.50 usec
TE                297.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -2.00 dB
PL1W          55.33689499 W
SFO1          100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL13               18.62 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
PL13W          0.14806664 W
SFO2          400.1916008 MHz
SI                 32768
SF            100.6278197 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



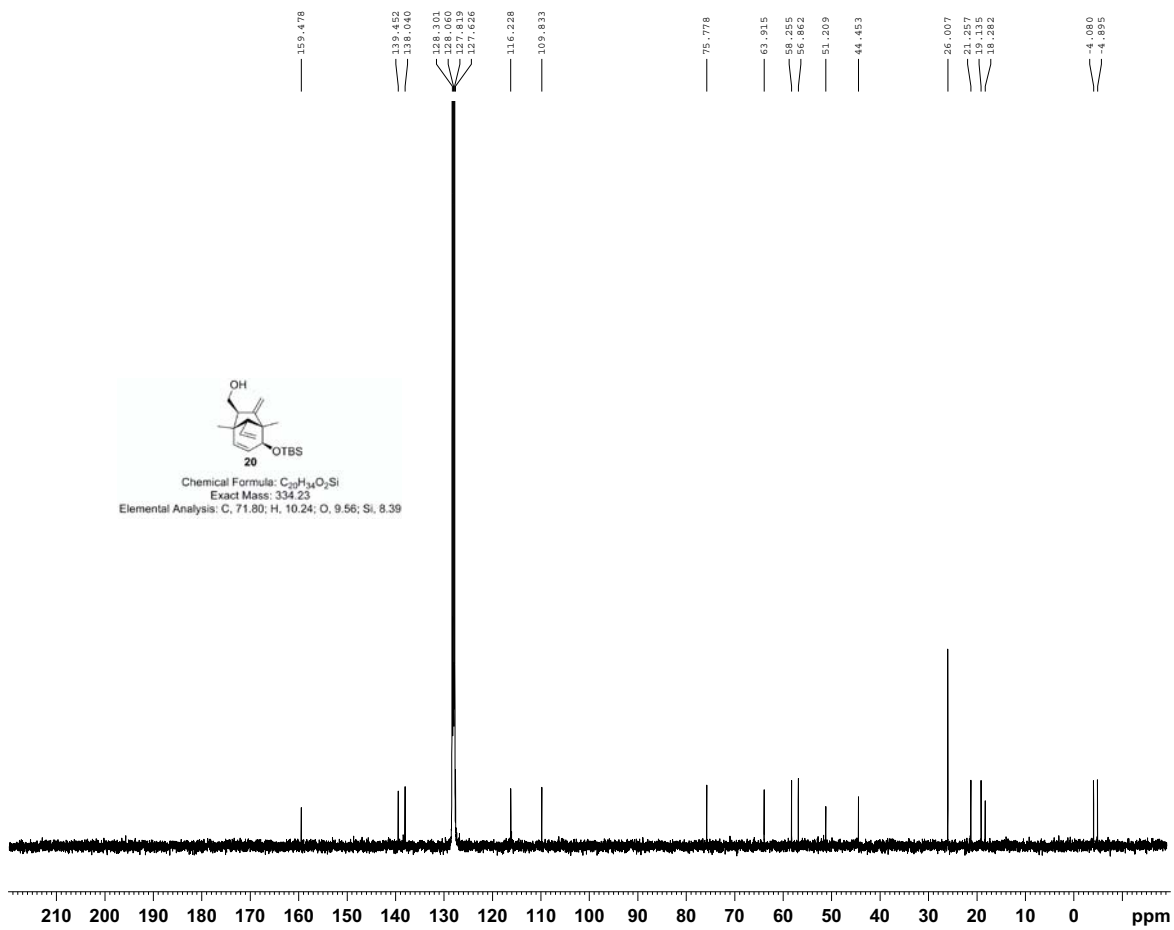

```
Bruker Advance III 400

NAME          XS-1-124-A1
EXPNO                   1
PROCNO                  1
Date_            20110617
Time                11.04
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT               C6D6
NS                       7
DS                       2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.98446387 sec
RG                     161
DW                60.800 usec
DE                  6.50 usec
TE                 294.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.83 usec
PL1                   0.00 dB
PL1W           8.31434441 W
SFO1          400.1324710 MHz
SI                    32768
SF            400.1300441 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



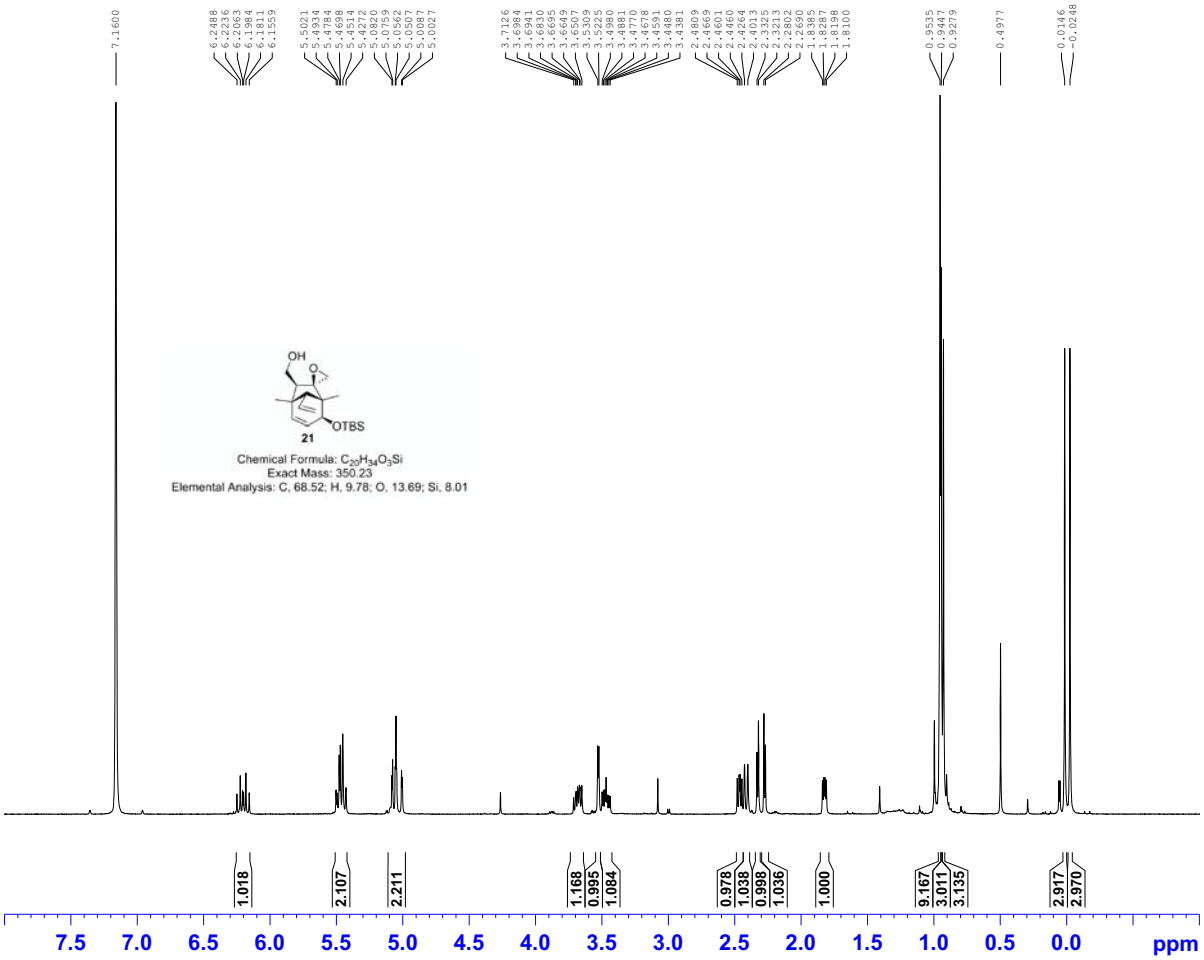

```
Bruker Advance III 400

NAME          XS-1-124-A1
EXPNO                   3
PROCNO                  1
Date_            20110617
Time                11.25
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG            zgpg30
TD                   65536
SOLVENT               C6D6
NS                      713
DS                       4
SWH            26041.666 Hz
FIDRES          0.397364 Hz
AQ             1.2583412 sec
RG                     203
DW                19.200 usec
DE                  6.50 usec
TE                 294.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.68 usec
PL1                  -0.60 dB
PL1W          41.24164963 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   0.00 dB
PL12                 15.66 dB
PL13                 15.92 dB
PL2W           8.31434441 W
PL12W          0.22585411 W
PL13W          0.21272963 W
SFO2         400.1316005 MHz
SI                    32768
SF           100.6127432 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```




```
Bruker Advance III 400

NAME        XS-1-127-A1(H1)
EXPNO                   1
PROCNO                  1
Date_            20120110
Time_               19.44
INSTRUM             spect
PROBHD    5 mm PABBI 1H/
PULPROG               zg30
TD                   65536
SOLVENT               C6D6
NS                        6
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                      203
DW                   60.800 usec
DE                    6.50 usec
TE                   295.4 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                     7.10 usec
PL1                   -2.00 dB
PL1W            13.17734718 W
SFO1            400.1324710 MHz
SI                    32768
SF              400.1300442 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

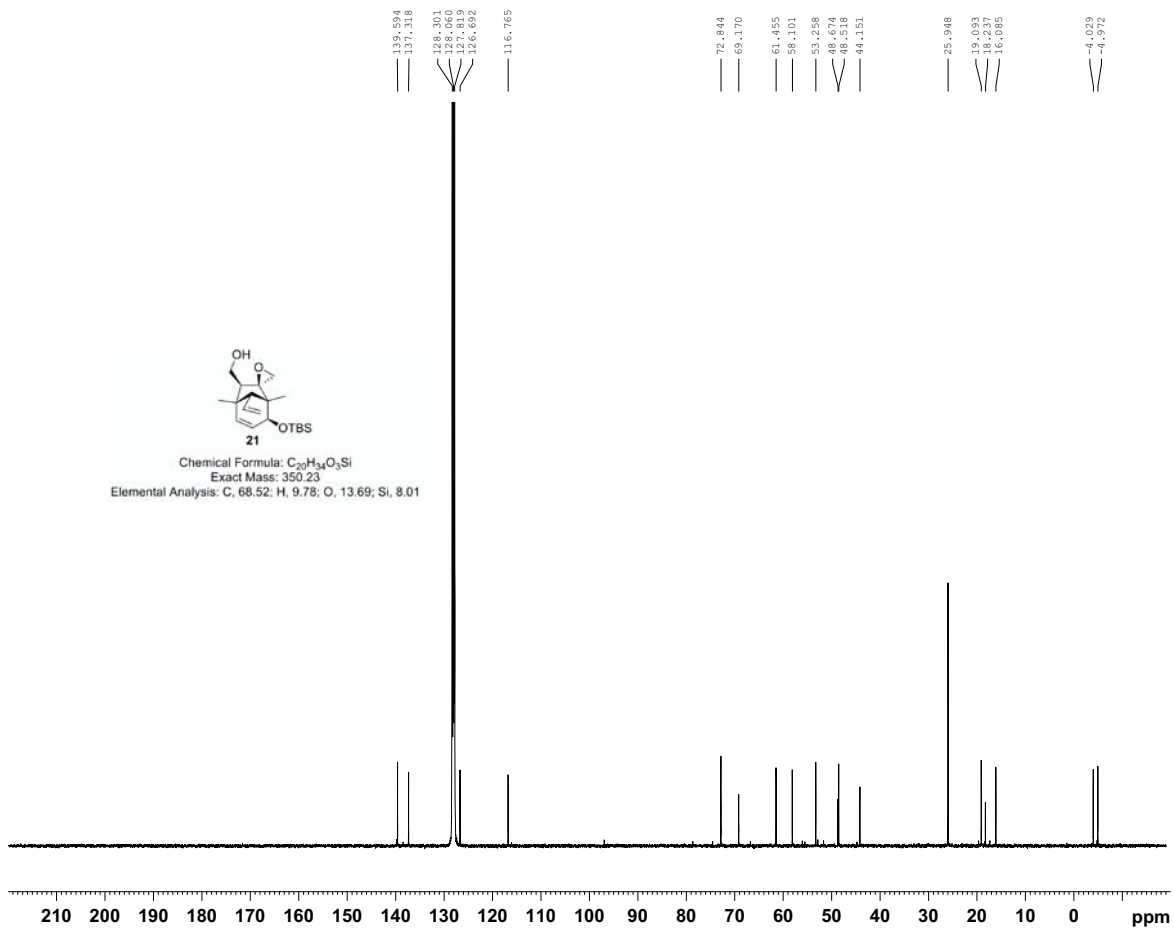



```
Bruker Advance III 400

NAME          XS-1-127-A1
EXPNO                   2
PROCNO                  1
Date_            20110908
Time_               19.56
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG            zgpg30
TD                   65536
SOLVENT               C6D6
NS                      349
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                      203
DW                   20.800 usec
DE                    6.50 usec
TE                   295.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.68 usec
PL1                   -0.60 dB
PL1W            41.24164963 W
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                     1H
PCPD2                 90.00 usec
PL2                    0.00 dB
PL12                  15.66 dB
PL13                  15.92 dB
PL2W             8.31434441 W
PL12W            0.22585411 W
PL13W            0.21272963 W
SFO2            400.1316005 MHz
SI                    32768
SF              100.6127436 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



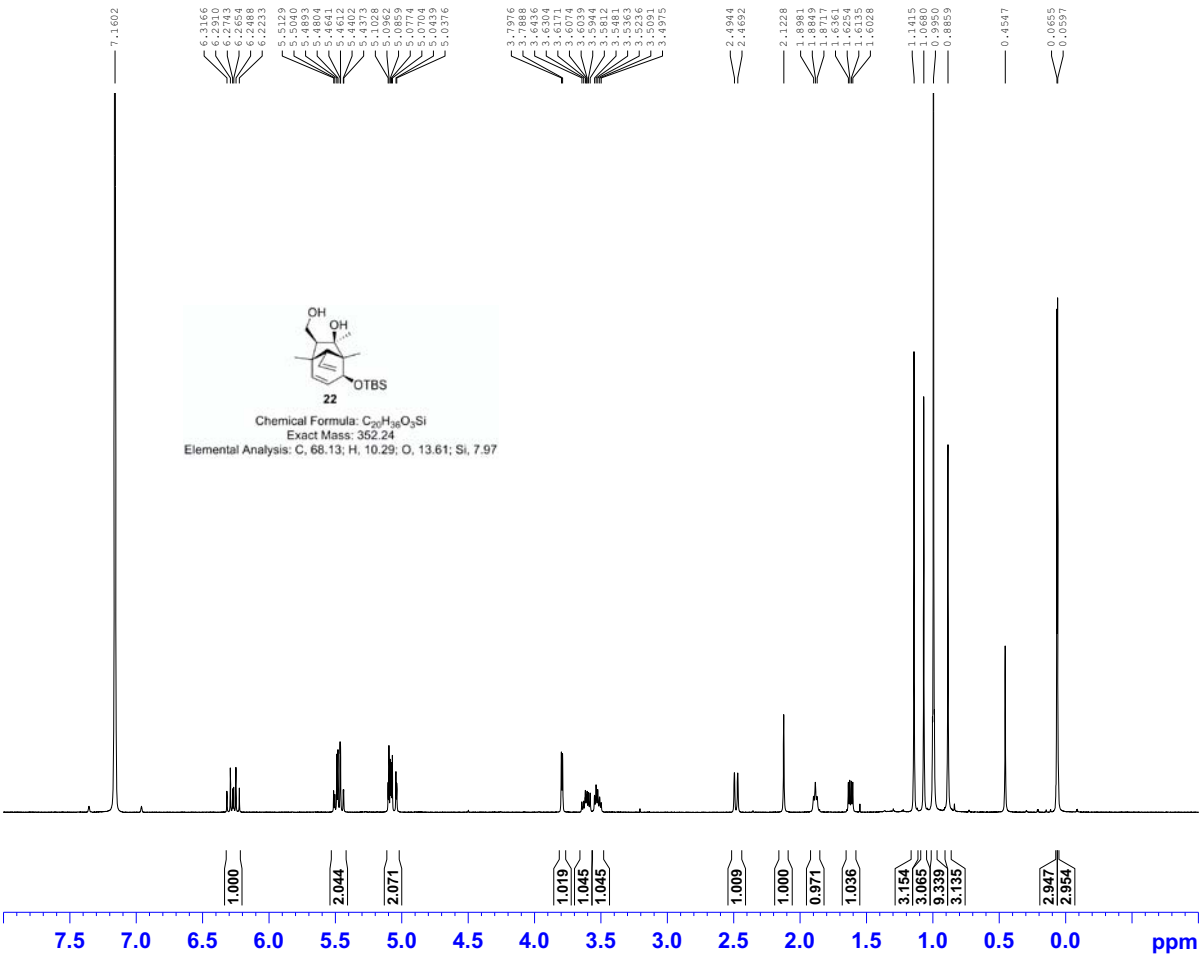
Bruker Advance III 400

```
NAME        XS-1-130-A1
EXPNO                 1
PROCNO                1
Date_          20110629
Time_            22.41
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           C6D6
NS                   16
DS                    2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ          3.9846387 sec
RG                90.5
DW              60.800 usec
DE                6.50 usec
TE               297.7 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                32768
SF          400.1900016 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

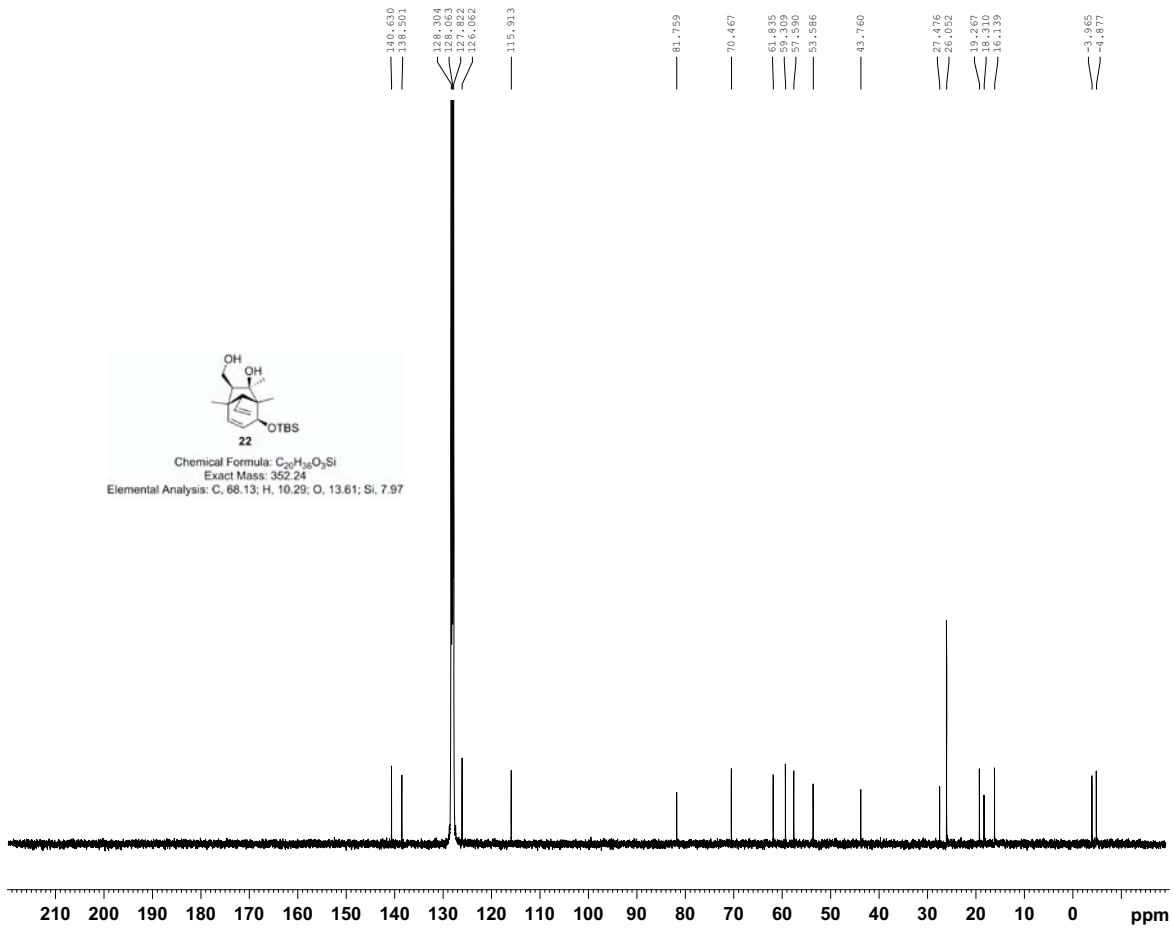


Bruker Advance III 400

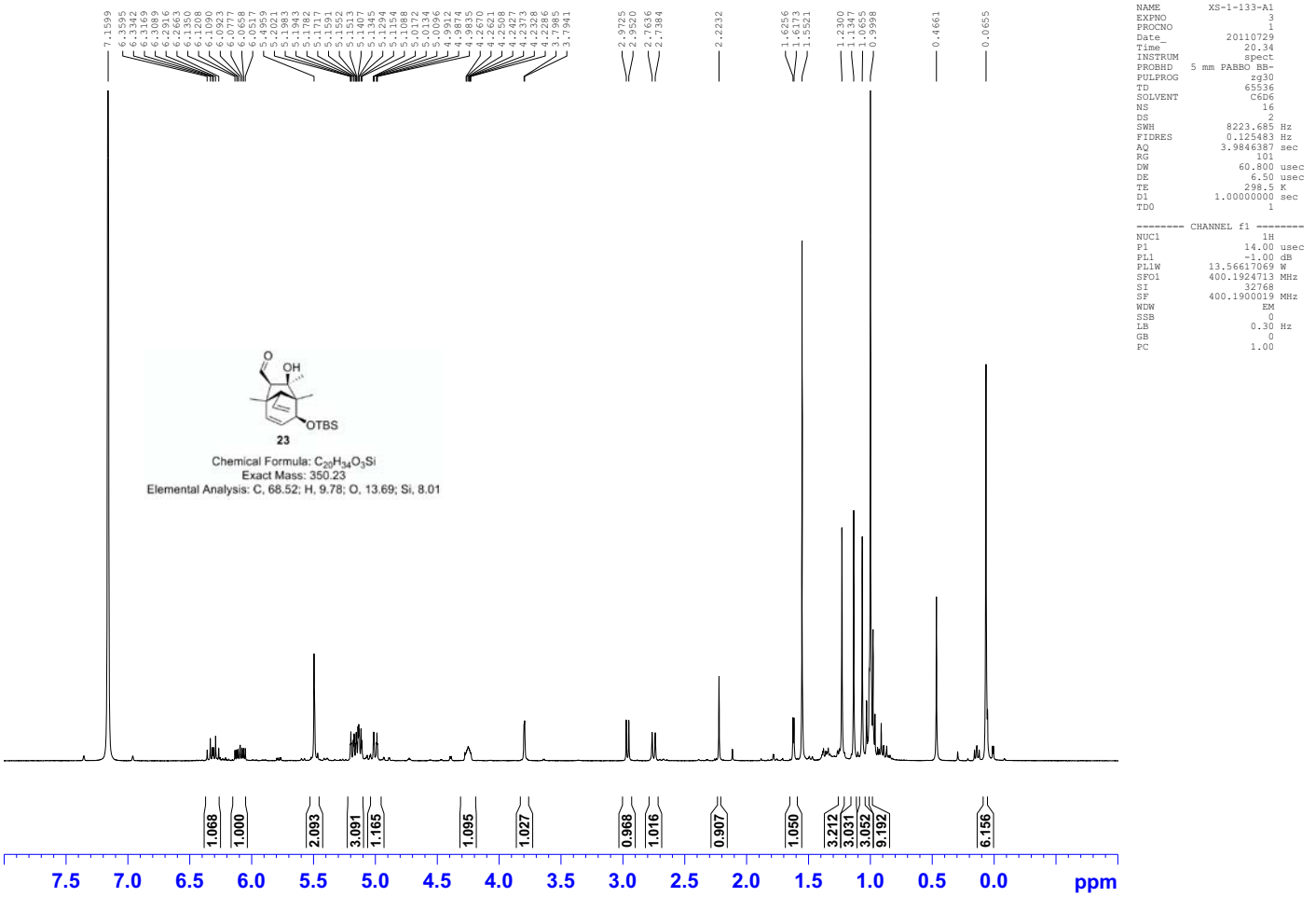
```
NAME        XS-1-130-A1
EXPNO                 2
PROCNO                1
Date_          20110630
Time_            3.27
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           C6D6
NS                 5000
DS                    4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ          1.3631988 sec
RG                  161
DW              20.800 usec
DE                6.50 usec
TE               299.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.90 usec
PL1              -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            90.00 usec
PL2              -1.00 dB
PL12             15.16 dB
PL13             18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                32768
SF          100.6278192 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

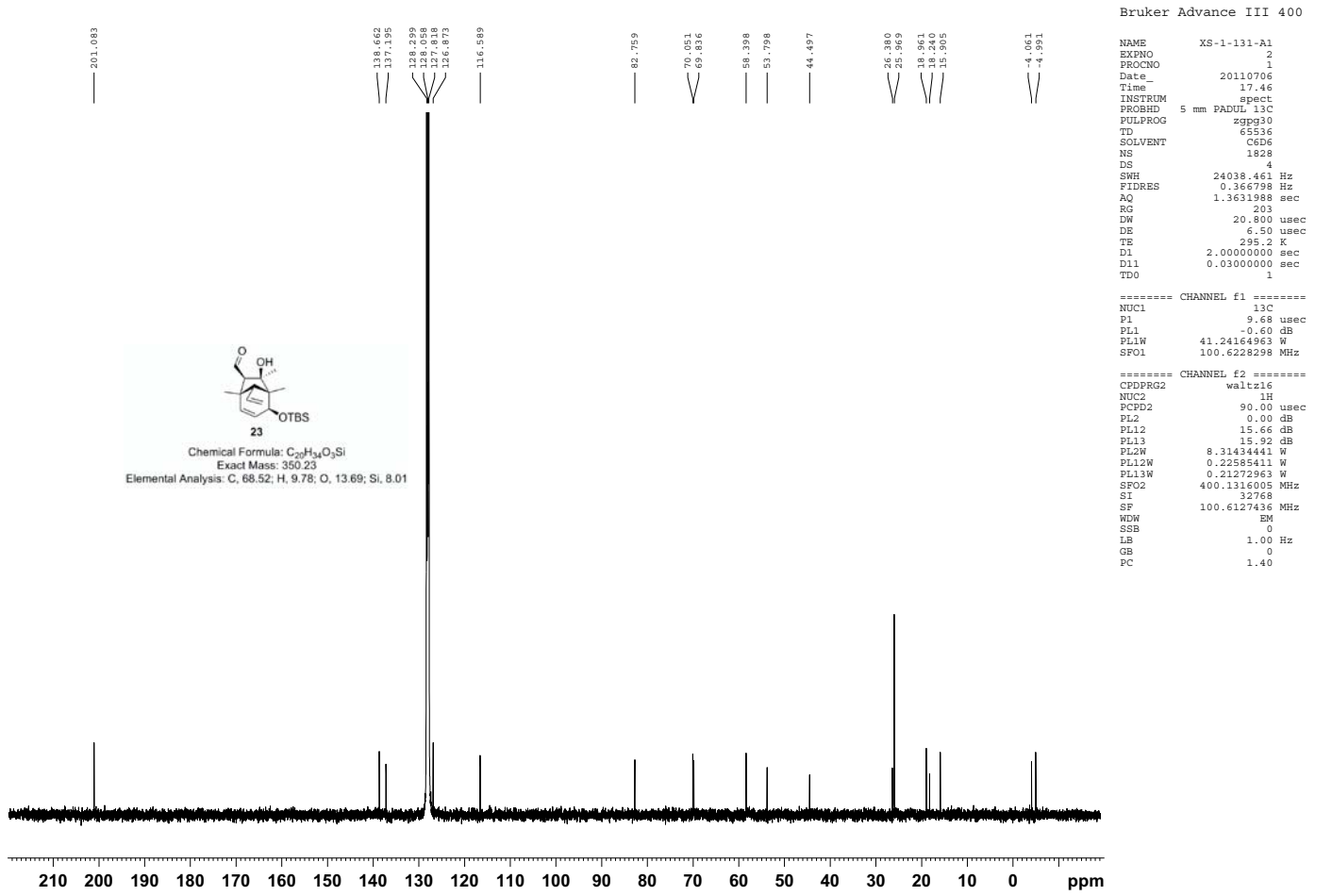


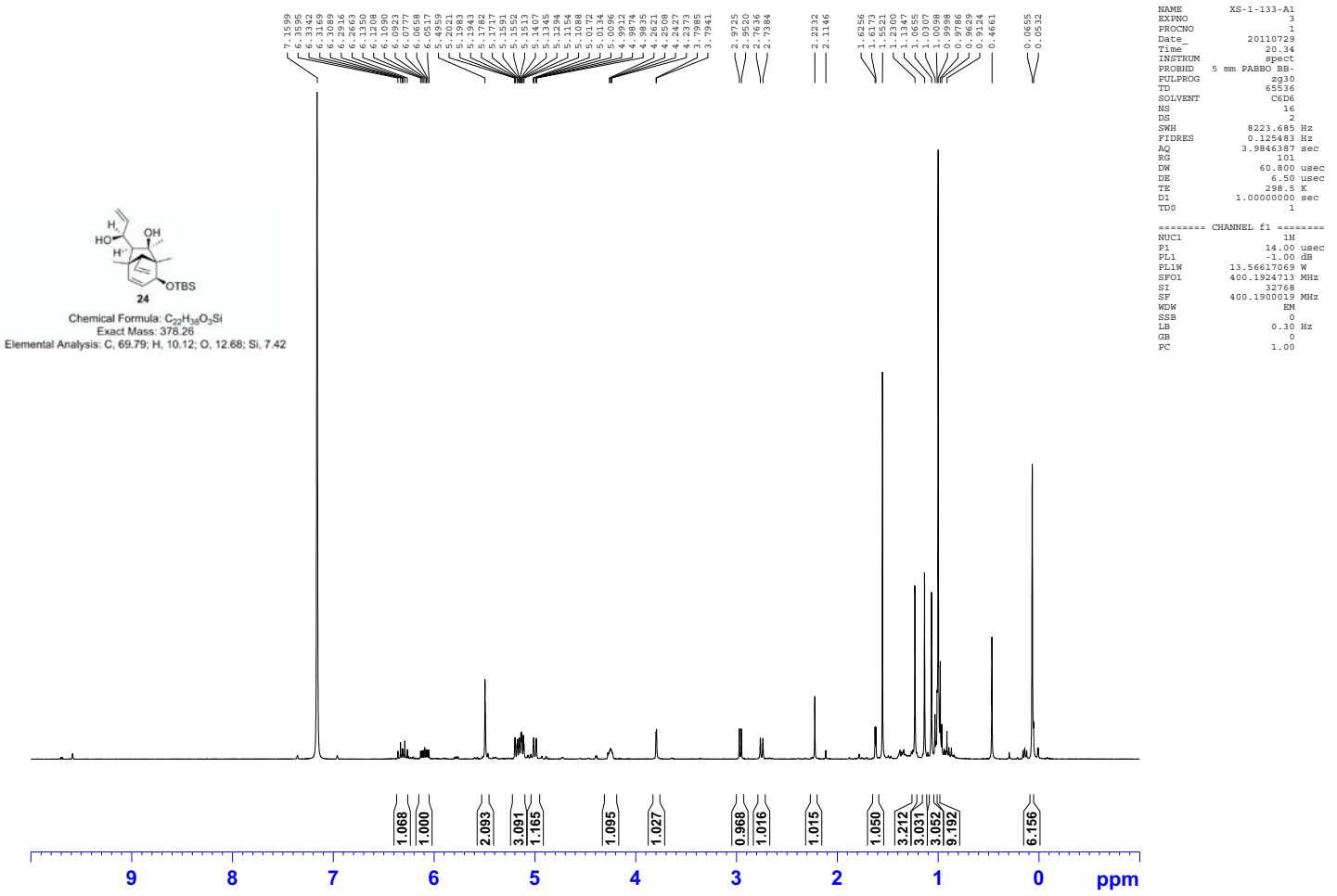

```
Bruker Advance III 400

NAME          XS-1-128-A1
EXPNO                  12
PROCNO                  1
Date_            20110701
Time                11.36
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG              zg30
TD                   65536
SOLVENT               C6D6
NS                        8
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                      203
DW                   60.800 usec
DE                     6.50 usec
TE                    296.2 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.83 usec
PL1                    0.00 dB
PL1W            8.31434441 W
SFO1           400.1324710 MHz
SI                     32768
SF             400.1300447 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

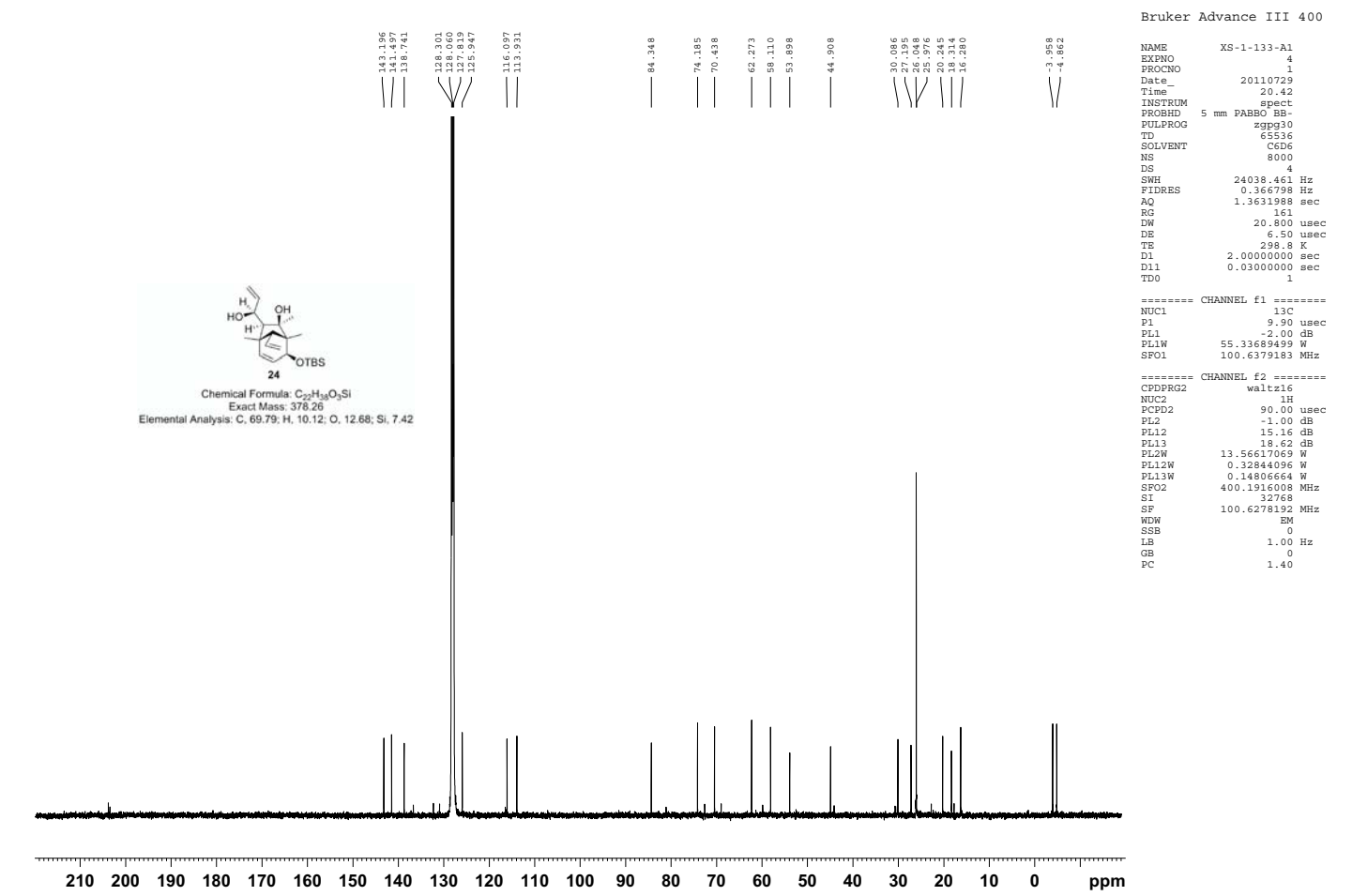


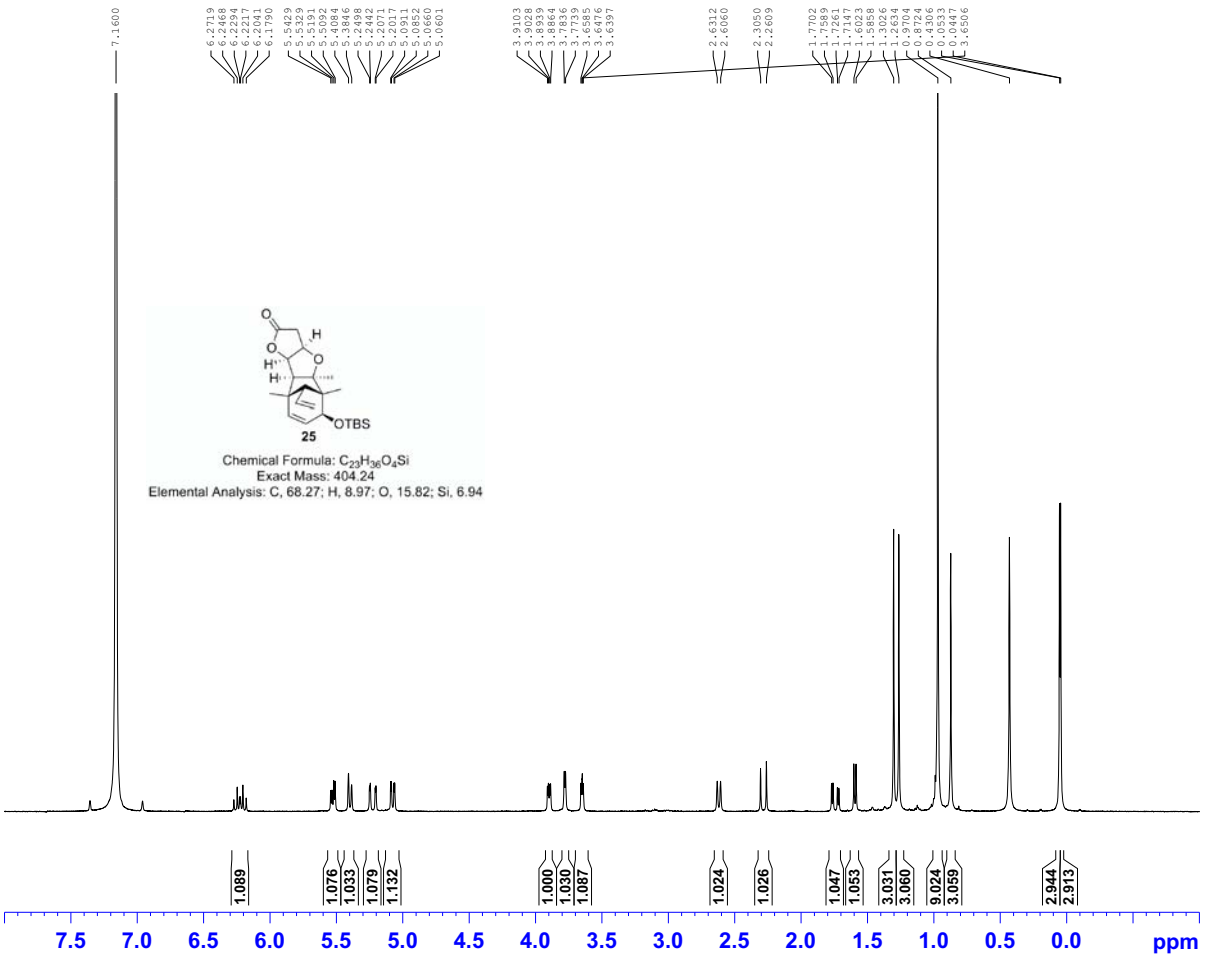

```
Bruker Advance III 400

NAME          XS-1-128-A1
EXPNO                  13
PROCNO                  1
Date_            20110701
Time                11.41
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                   65536
SOLVENT               C6D6
NS                     2525
DS                        4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                      203
DW                   20.800 usec
DE                     6.50 usec
TE                    296.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.68 usec
PL1                   -0.60 dB
PL1W           41.24164963 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                     1H
PCPD2                90.00 usec
PL2                    0.00 dB
PL12                  15.66 dB
PL13                  15.92 dB
PL2W            8.31434441 W
PL12W           0.22585411 W
PL13W           0.21272963 W
SFO2          400.1316005 MHz
SI                     32768
SF            100.6127429 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```


Bruker Advance III 400
NAME XS-1-133-A1
EXPNO 3
PROCNO 1
Date_ 20110729
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.98446387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900019 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OTBS
23
Chemical Formula: C20H34O3Si
Exact Mass: 350.23
Elemental Analysis: C, 68.52; H, 9.78; O, 13.69; Si, 8.01
1.068
1.000
2.093
3.091
1.165
1.095
1.027
0.968
1.016
0.907
1.050
3.212
3.031
3.052
9.192
6.156
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

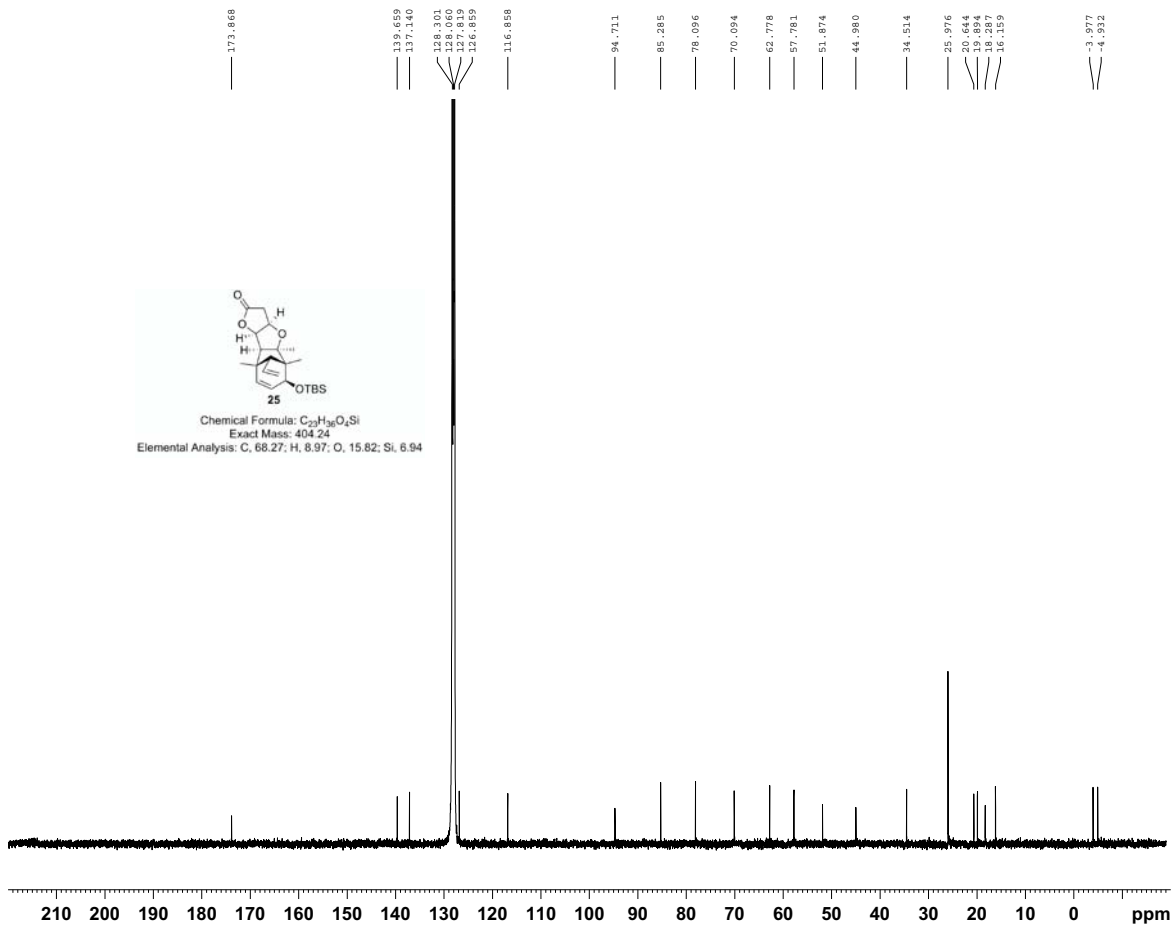

Bruker Advance III 400
NAME XS-1-131-A1
EXPNO 2
PROCNO 1
Date_ 20110706
Time 17.46
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1828
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.68 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 15.66 dB
PL13 15.92 dB
PL2W 8.31434441 W
PL12W 0.22585411 W
PL13W 0.21272963 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127436 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OTBS
23
Chemical Formula: C20H34O3Si
Exact Mass: 350.23
Elemental Analysis: C, 68.52; H, 9.78; O, 13.69; Si, 8.01
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


24
Chemical Formula: $C_{22}H_{38}O_3Si$
Exact Mass: 378.26
Elemental Analysis: C, 69.79; H, 10.12; O, 12.68; Si, 7.42
Bruker Advance III 400
NAME XS-1-133-A1
EXPNO 3
PROCNO 1
Date_ 20110729
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900019 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

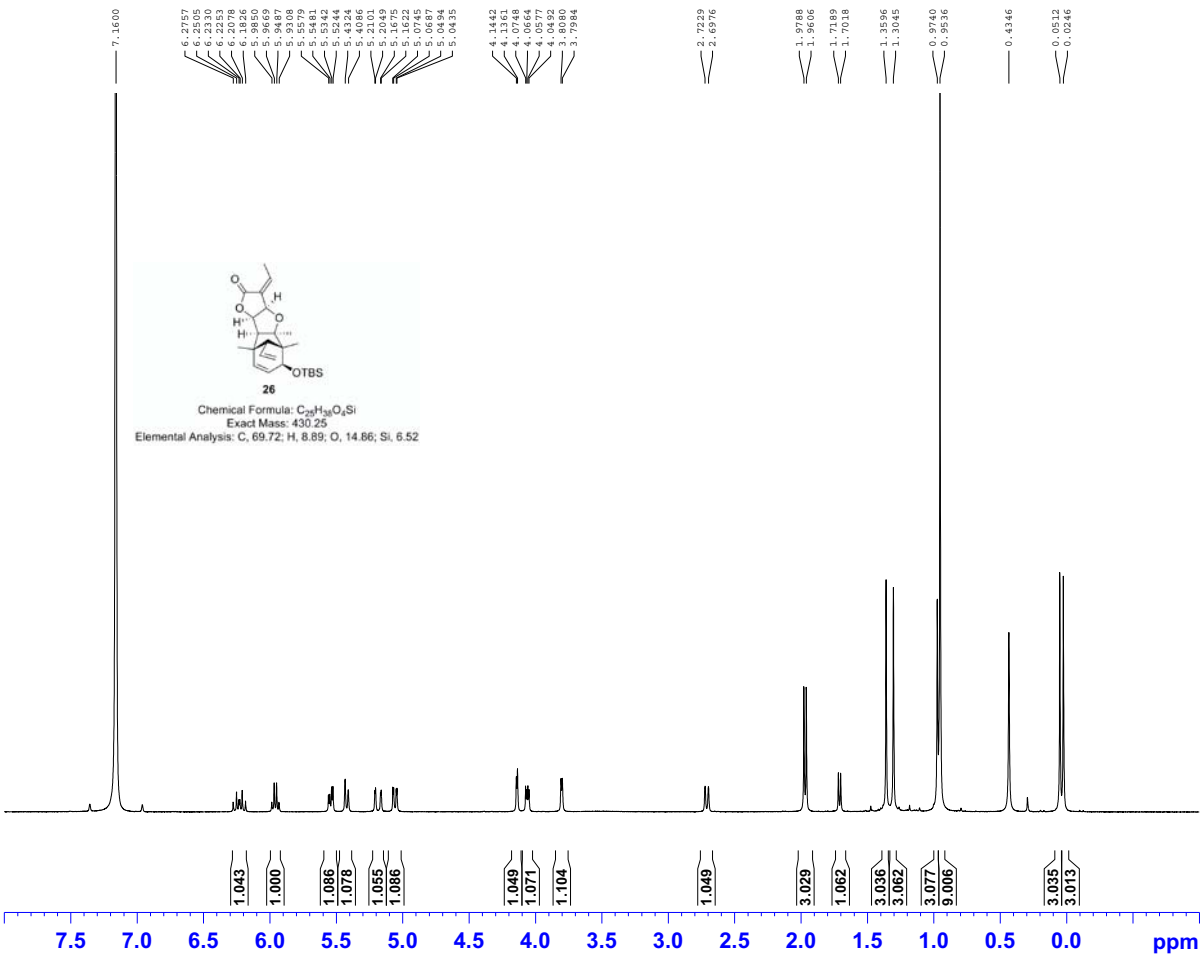

24
Chemical Formula: $C_{22}H_{38}O_3Si$
Exact Mass: 378.26
Elemental Analysis: C, 69.79; H, 10.12; O, 12.68; Si, 7.42
Bruker Advance III 400
NAME XS-1-133-A1
EXPNO 4
PROCNO 1
Date_ 20110729
Time 20.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 8000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6379183 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.05 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 18.62 dB
PL2W 13.56617069 W
PL12W 0.32844096 W
PL13W 0.14806664 W
SFO2 400.1916008 MHz
SI 32768
SF 100.6278192 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm



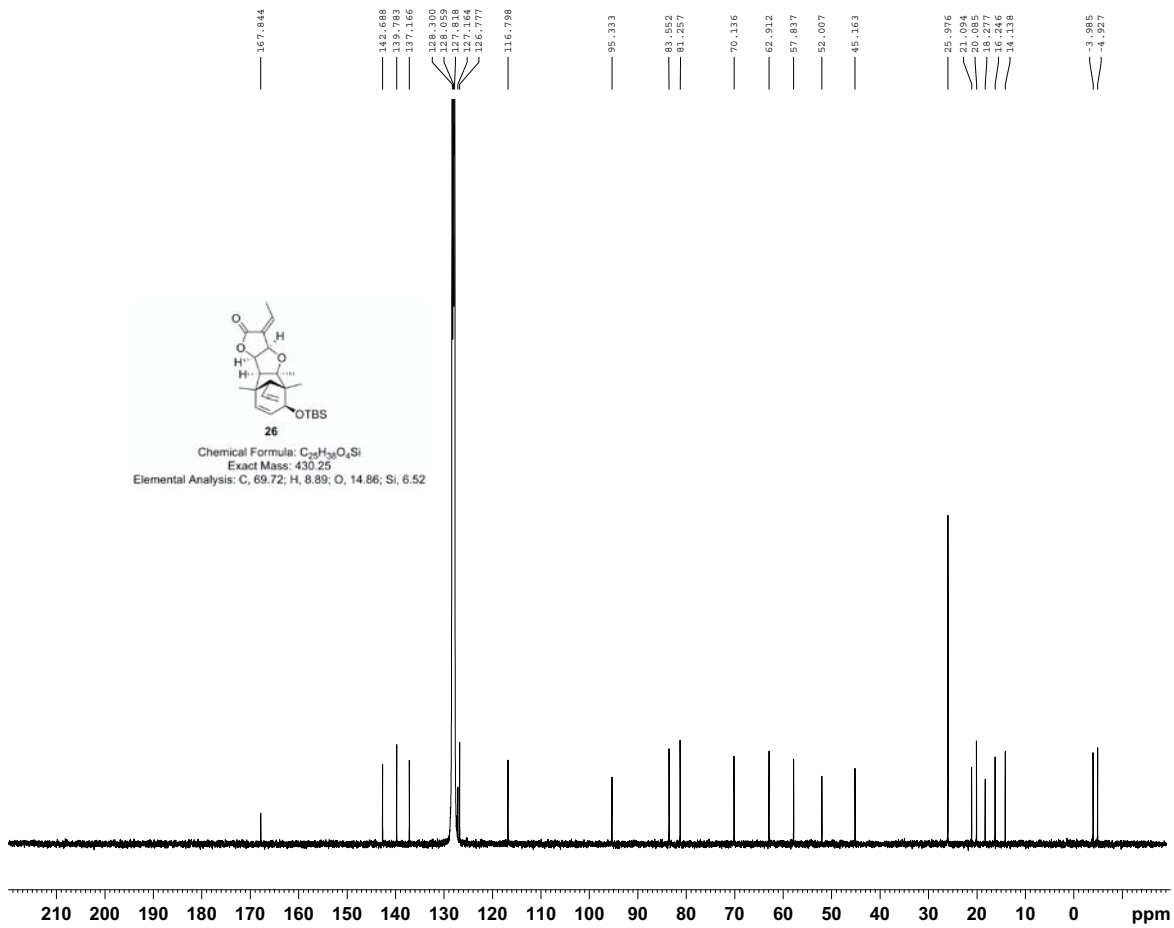

```
Bruker Advance III 400

NAME          XS-1-134-A1
EXPNO                  11
PROCNO                  1
Date_            20110928
Time                 14.36
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT               C6D6
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                     128
DW                  60.800 usec
DE                    6.50 usec
TE                  298.8 K
D1               1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  -1.00 dB
PL1W            13.56617069 W
SFO1            400.1924713 MHz
SI                   32768
SF              400.1900012 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



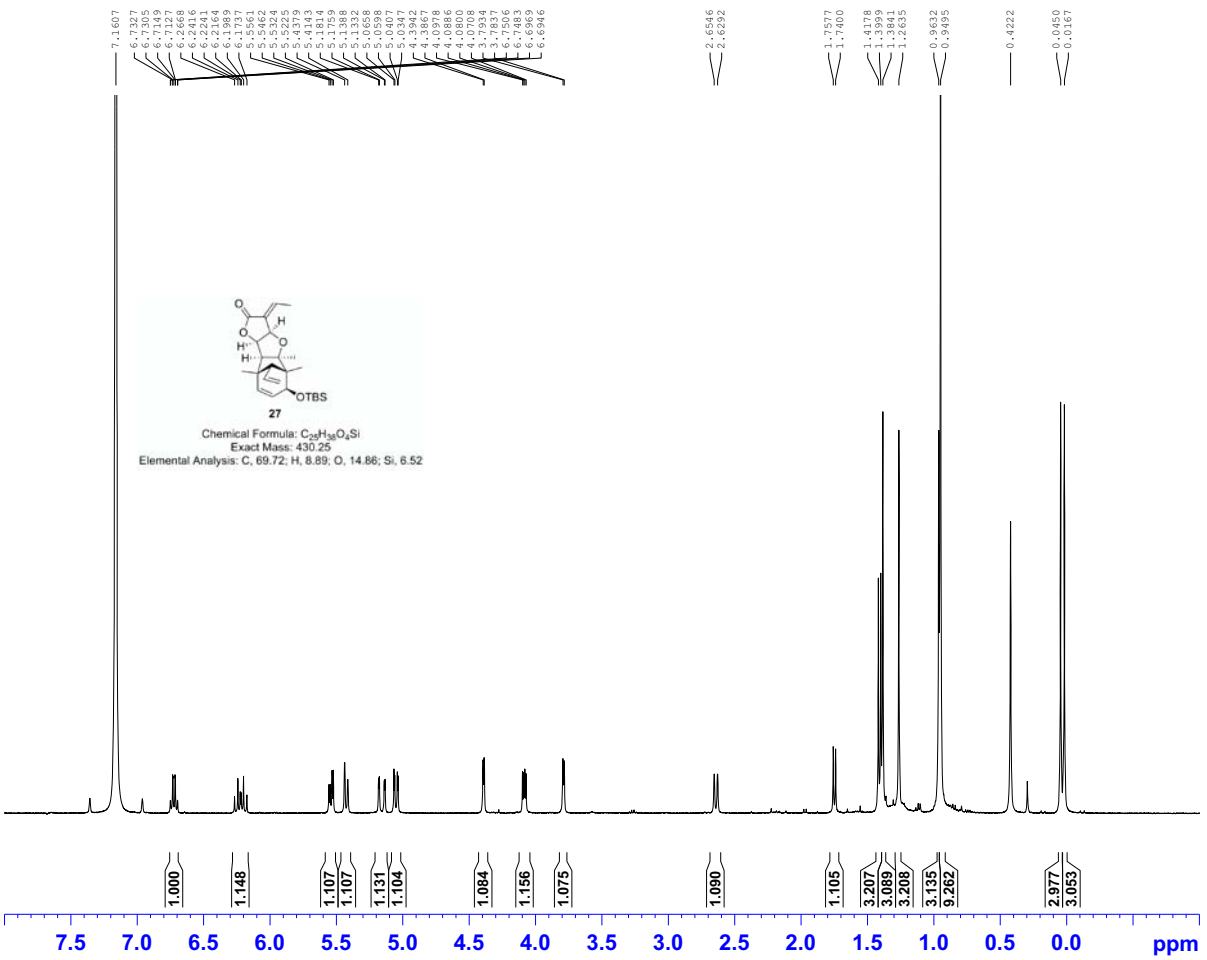

```
Bruker Advance III 400

NAME          XS-1-134-A1
EXPNO                  14
PROCNO                  1
Date_            20111001
Time                  2.58
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               C6D6
NS                     5000
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                    71.8
DW                  20.800 usec
DE                    6.50 usec
TE                   300.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                  -2.00 dB
PL1W            55.33689499 W
SFO1            100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL13                 18.62 dB
PL2W            13.56617069 W
PL12W            0.32844096 W
PL13W            0.14806664 W
SFO2            400.1916008 MHz
SI                   32768
SF              100.6278189 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



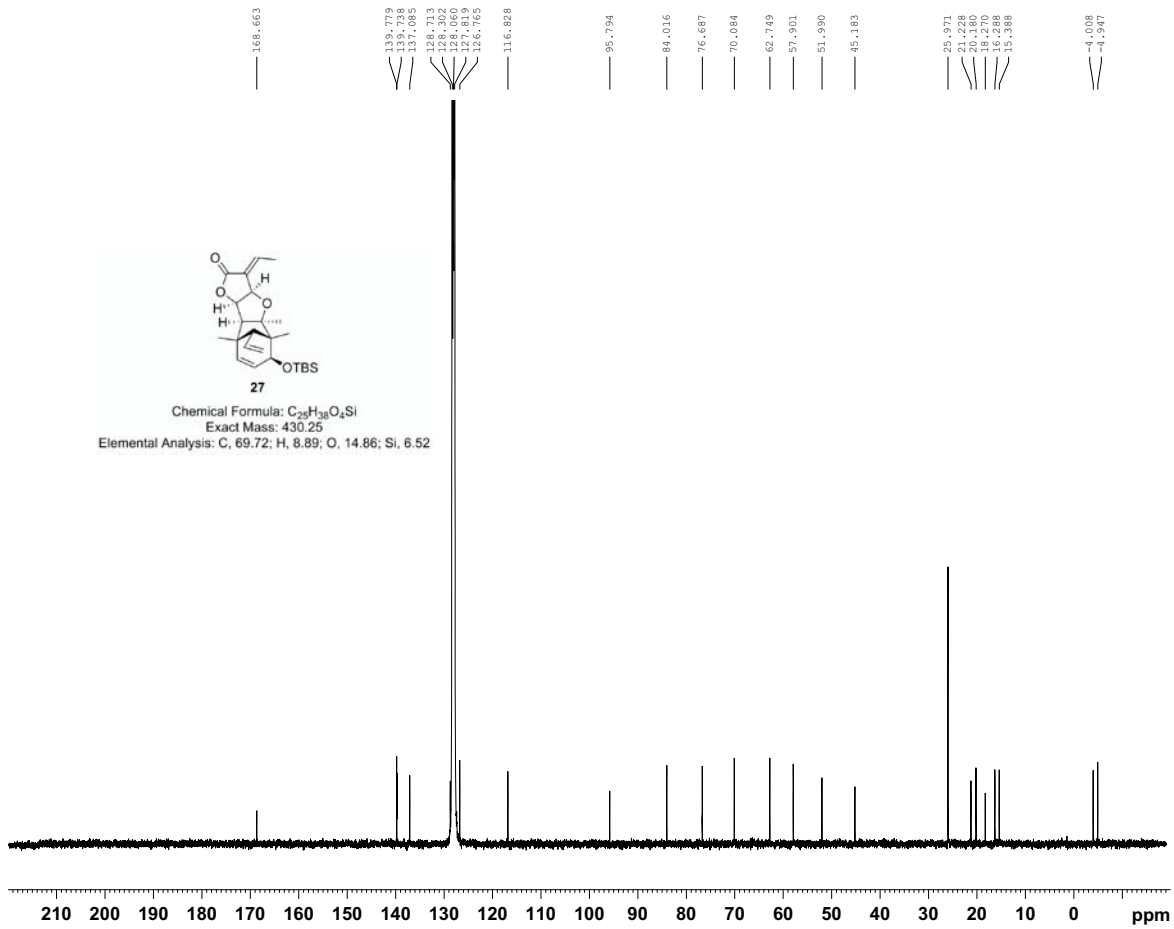

```
Bruker Advance III 400

NAME          XS-1-142-A1
EXPNO                   1
PROCNO                  1
Date_            20120403
Time_               11.02
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              C6D6
NS                     16
DS                      2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                    114
DW               60.800 usec
DE                6.50 usec
TE                298.9 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                 -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                  32768
SF          400.1900013 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



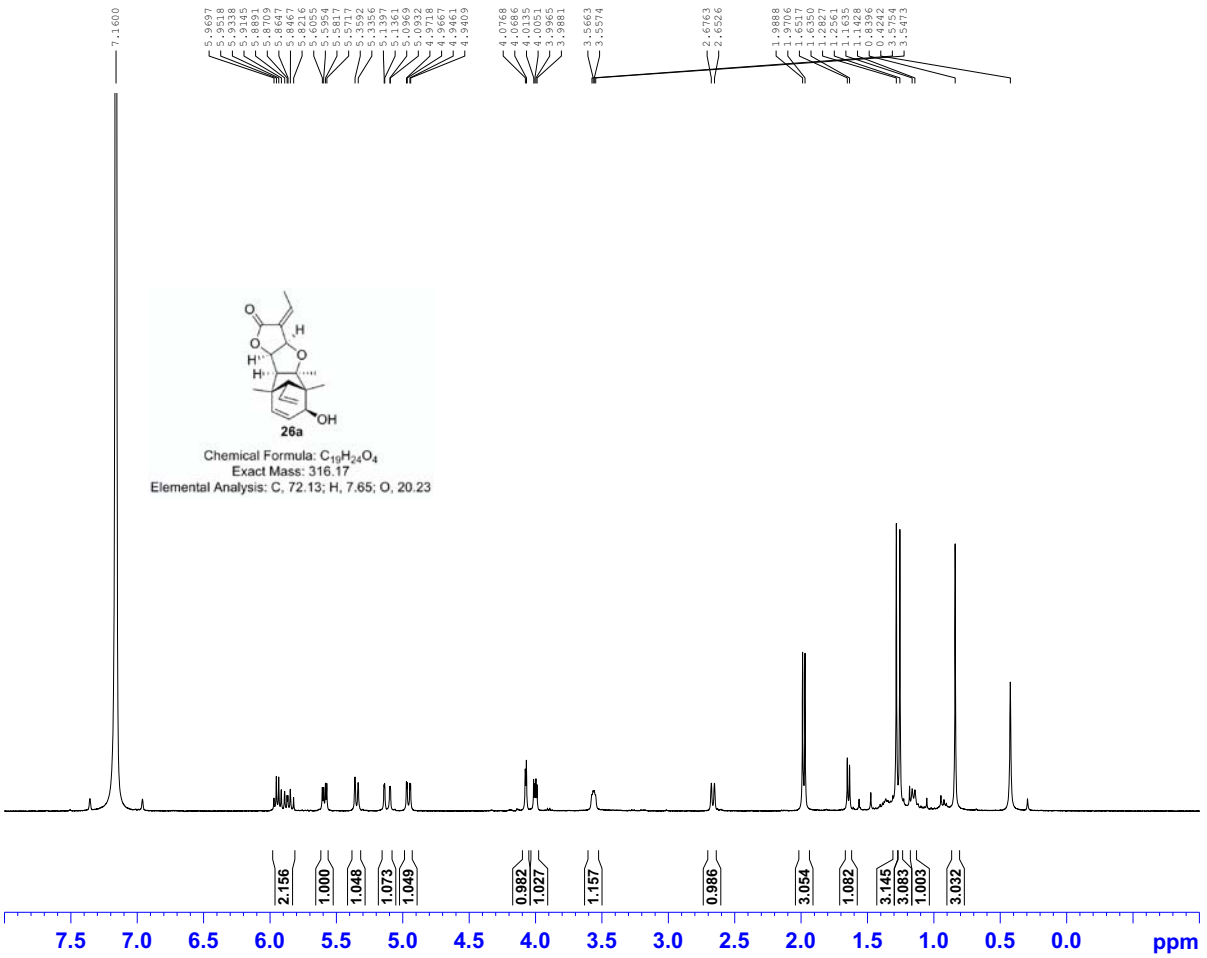

```
Bruker Advance III 400

NAME          XS-1-142-A1
EXPNO                   2
PROCNO                  1
Date_            20120403
Time_               21.16
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT              C6D6
NS                   12488
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ           1.3631988 sec
RG                    203
DW               20.800 usec
DE                6.50 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.90 usec
PL1                 -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL13                18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                  32768
SF          100.6278192 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```




```
Bruker Advance III 400

NAME          XS-1-135-A1
EXPNO                  10
PROCNO                  1
Date_            20111208
Time                 21.25
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                    65536
SOLVENT               C6D6
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                     144
DW                  60.800 usec
DE                   6.50 usec
TE                  298.9 K
D1               1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  -1.00 dB
PL1W           13.56617069 W
SFO1           400.1924713 MHz
SI                   32768
SF             400.1900009 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

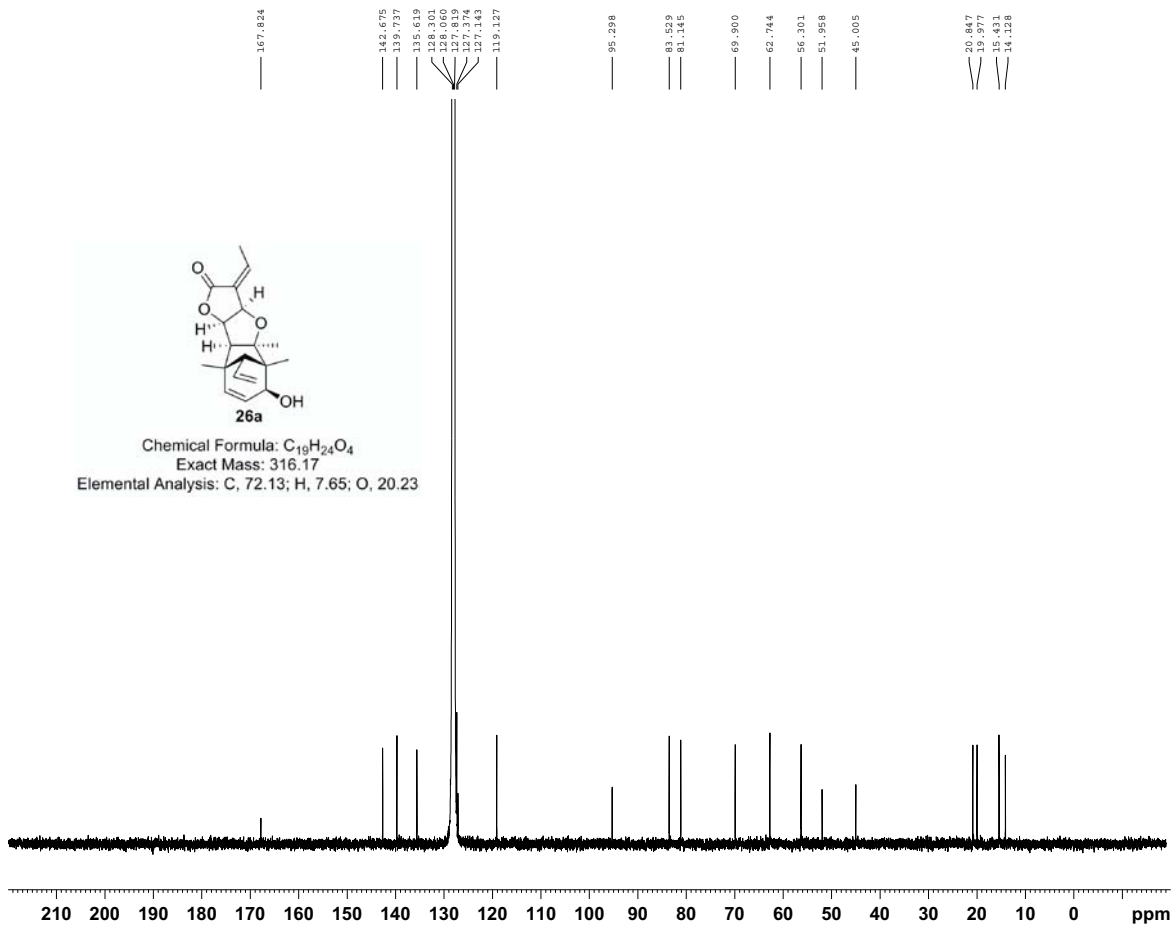



```
Bruker Advance III 400

NAME          XS-1-135-A1
EXPNO                  11
PROCNO                  1
Date_            20111208
Time                 21.31
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                    65536
SOLVENT               C6D6
NS                    12045
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                     203
DW                  20.800 usec
DE                   6.50 usec
TE                  299.6 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                 -2.00 dB
PL1W           55.33689499 W
SFO1           100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL13                18.62 dB
PL2W           13.56617069 W
PL12W           0.32844096 W
PL13W           0.14806664 W
SFO2           400.1916008 MHz
SI                   32768
SF             100.6278189 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



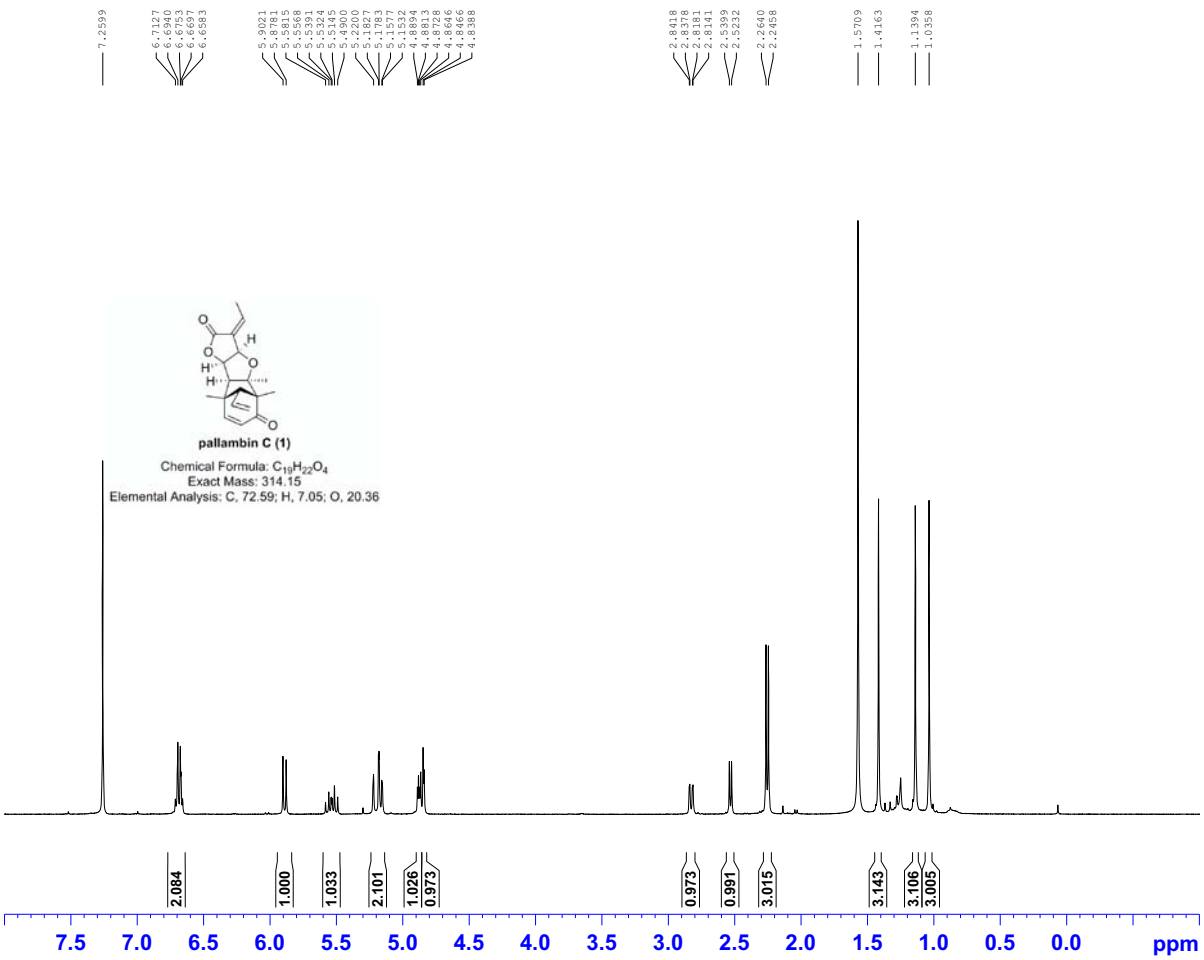
Bruker Advance III 400

```
NAME          XS-1-143-A1
EXPNO                   1
PROCNO                  1
Date_            20120416
Time_               21.24
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              C6D6
NS                     16
DS                      2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG                    128
DW              60.800 usec
DE                6.50 usec
TE                299.6 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                 -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                  32768
SF          400.1900016 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



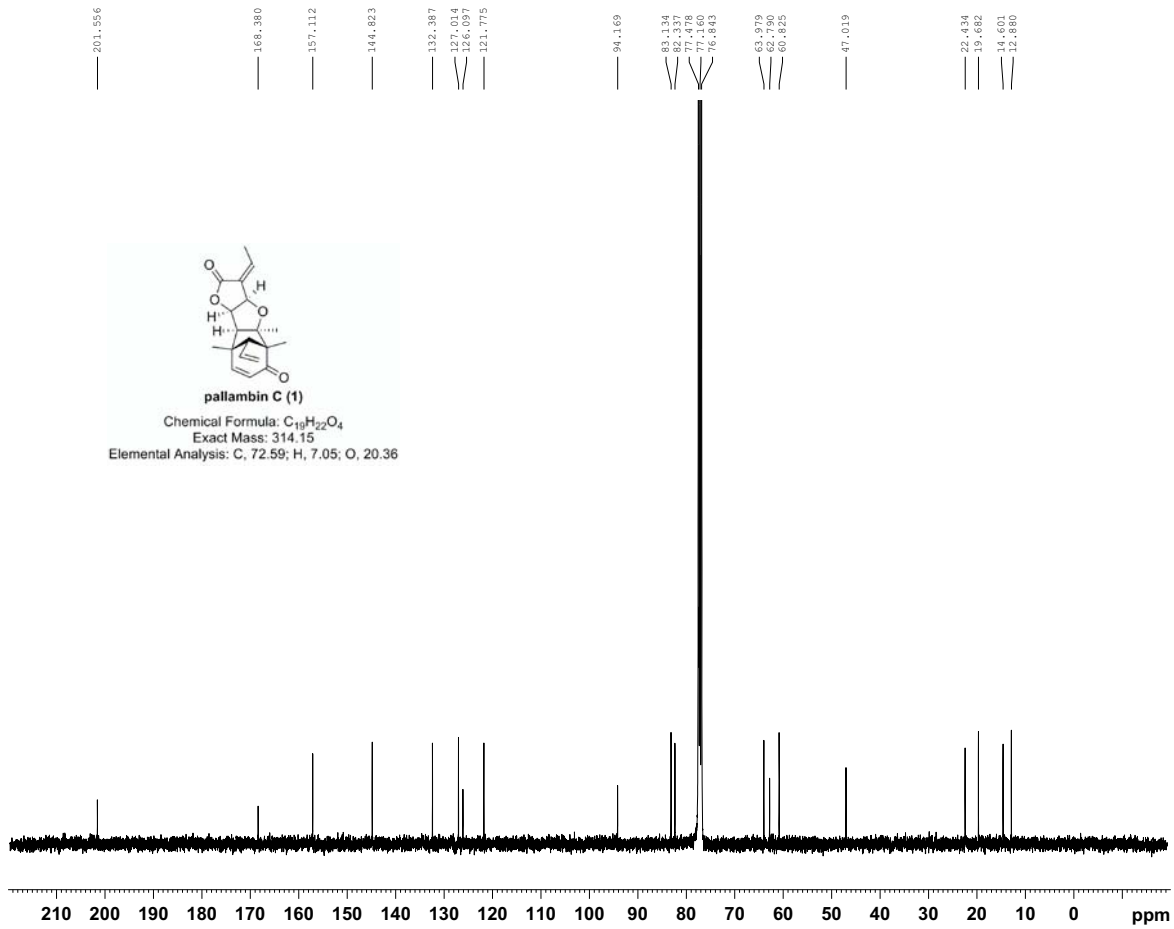
Bruker Advance III 400

```
NAME          XS-1-143-A1
EXPNO                   2
PROCNO                  1
Date_            20120416
Time_               22.36
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT              C6D6
NS                   11006
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ           1.3631988 sec
RG                    181
DW              20.800 usec
DE                6.50 usec
TE                299.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.90 usec
PL1                 -2.00 dB
PL1W        55.33689499 W
SFO1        100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL13                18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2        400.1916008 MHz
SI                  32768
SF          100.6278188 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



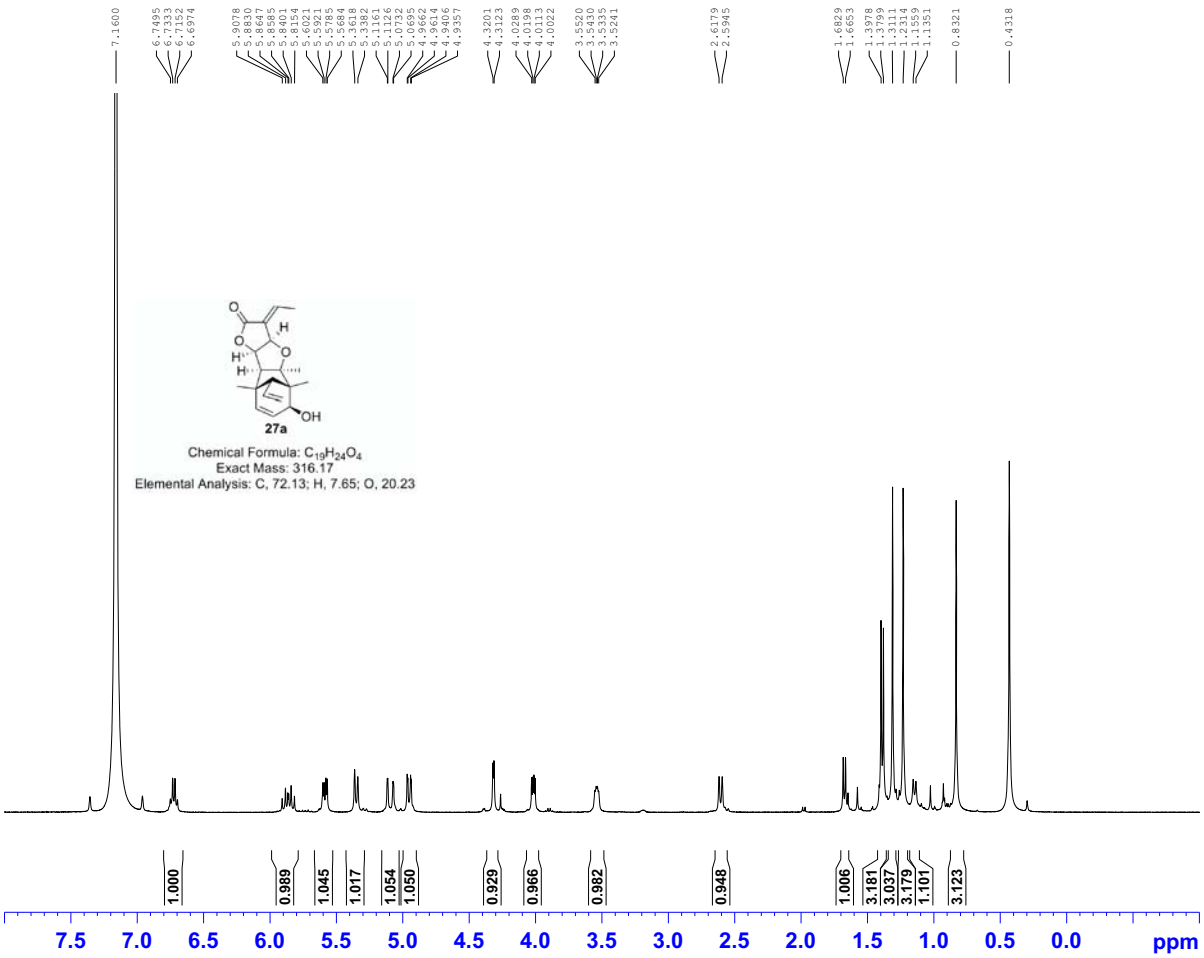
Bruker Advance III 400

```
NAME          XS-1-144-A1
EXPNO                  11
PROCNO                  1
Date_            20120504
Time                 19.26
INSTRUM              spect
PROBHD   5 mm PABBI 1H/
PULPROG              zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                     203
DW                  60.800 usec
DE                    6.50 usec
TE                   294.2 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    7.10 usec
PL1                  -2.00 dB
PL1W            13.17734718 W
SFO1            400.1324710 MHz
SI                    32768
SF              400.1300099 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

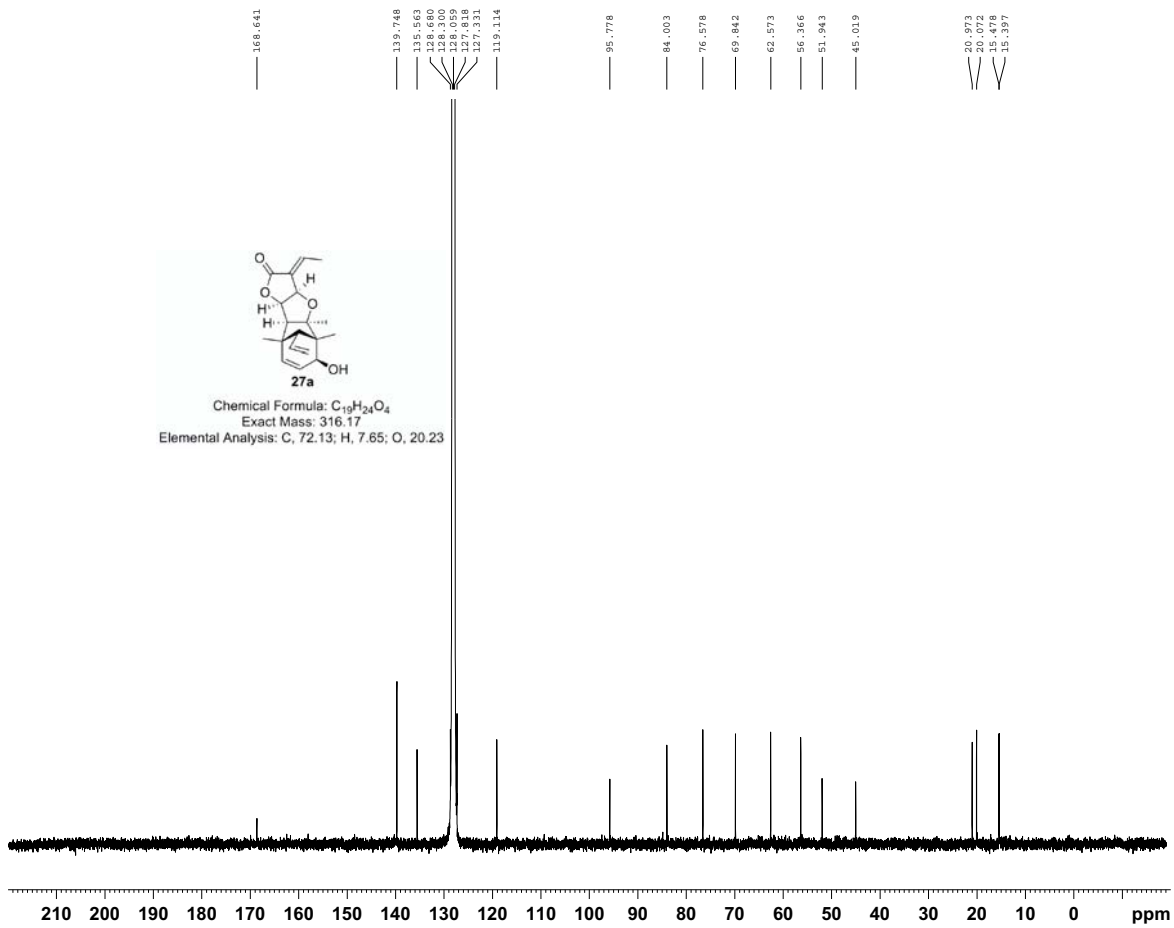


Bruker Advance III 400

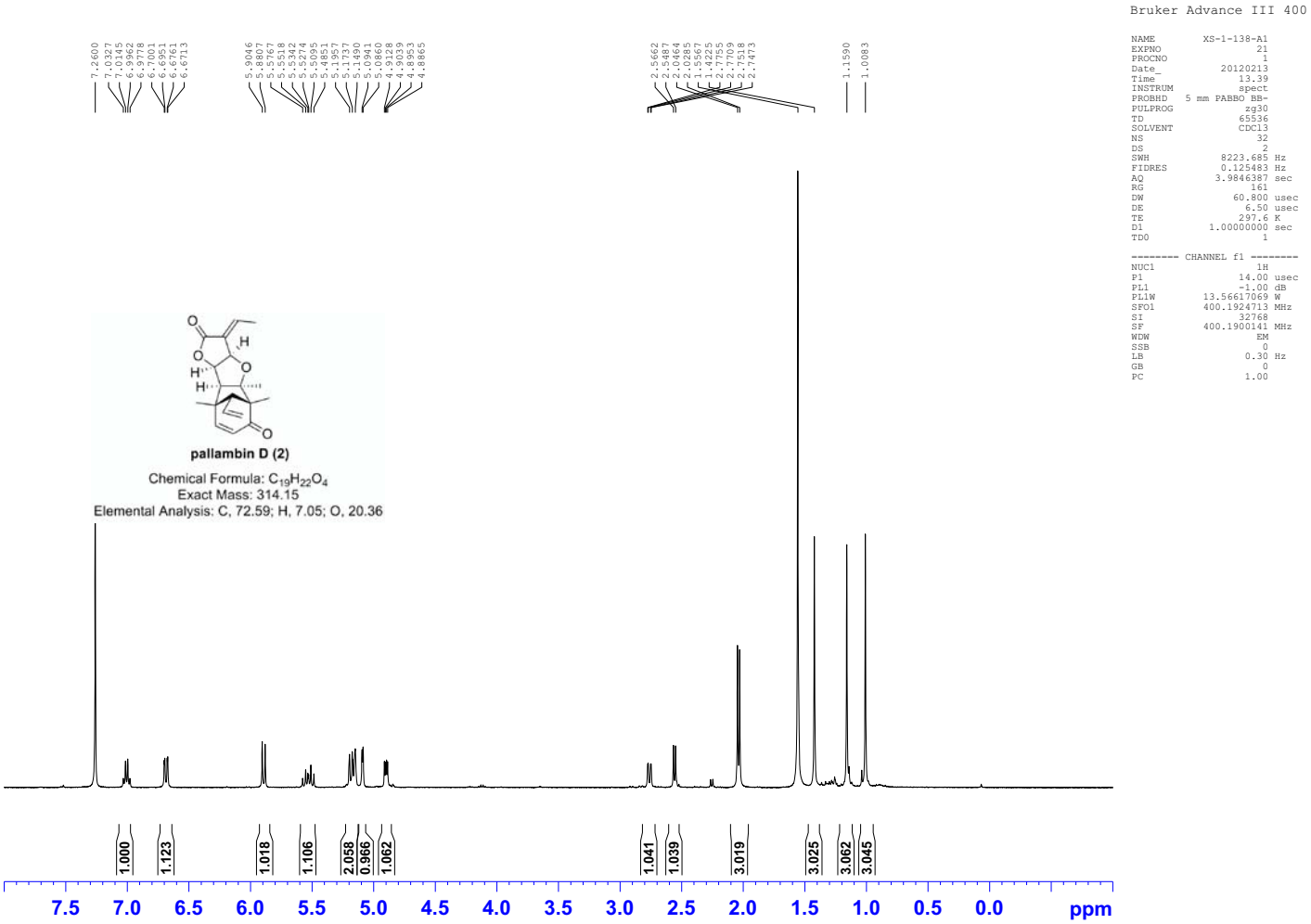
```
NAME          XS-1-144-A1
EXPNO                   3
PROCNO                  1
Date_            20120417
Time                 22.06
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                   11820
DS                       4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631988 sec
RG                     228
DW                  20.800 usec
DE                    6.50 usec
TE                   300.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.90 usec
PL1                  -2.00 dB
PL1W            55.33689499 W
SFO1            100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL13                 18.62 dB
PL2W            13.56617069 W
PL12W            0.32844096 W
PL13W            0.14806664 W
SFO2            400.1916008 MHz
SI                    32768
SF              100.6278416 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

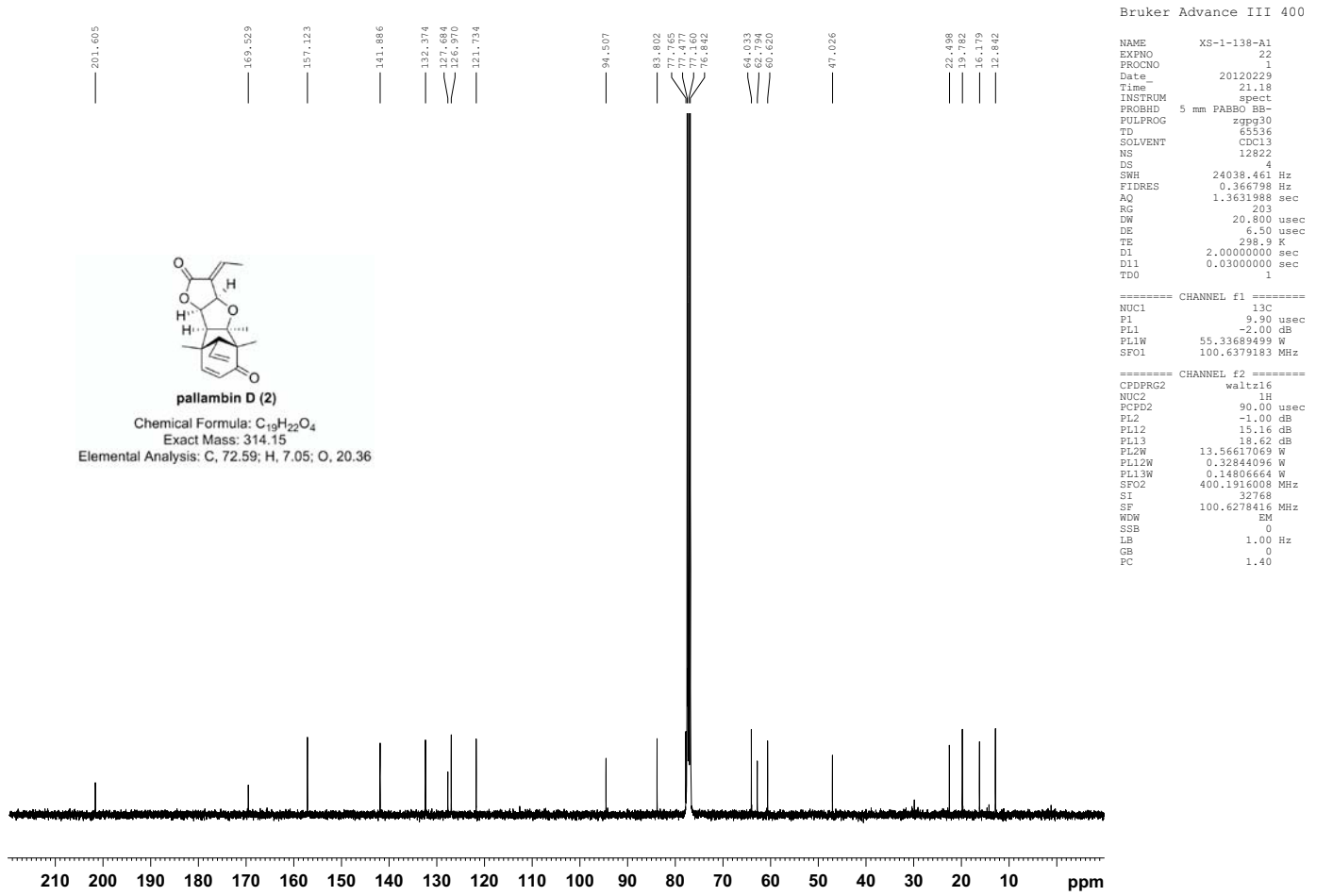



```
Bruker Advance III 400

NAME          XS-1-136-A1
EXPNO                   1
PROCNO                  1
Date_            20111217
Time                 14.58
INSTRUM              spect
PROBHD   5 mm PABBI 1H/
PULPROG              zg30
TD                   65536
SOLVENT              C6D6
NS                      32
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ             3.9846387 sec
RG                     203
DW                60.800 usec
DE                  6.50 usec
TE                 294.9 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.10 usec
PL1                 -2.00 dB
PL1W          13.17734718 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300441 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
Bruker Advance III 400

NAME          XS-1-136-A1
EXPNO                   4
PROCNO                  1
Date_            20111223
Time                 9.32
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                   65536
SOLVENT              C6D6
NS                   12972
DS                       4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ             1.3631988 sec
RG                     181
DW                20.800 usec
DE                  6.50 usec
TE                 299.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                 -2.00 dB
PL1W          55.33689499 W
SFO1          100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL13                18.62 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
PL13W          0.14806664 W
SFO2          400.1916008 MHz
SI                   32768
SF            100.6278192 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


Bruker Advance III 400
NAME XS-1-138-A1
EXPNO 21
PROCNO 1
Date_ 20120213
Time 13.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.2600 7.0327 7.0145 6.9962 6.9778 6.7001 6.6951 6.6761 6.6713
5.9046 5.8807 5.5577 5.5518 5.5342 5.5274 5.5095 5.4851 5.4757 5.1737 5.1490 5.0941 5.0860 4.9128 4.9039 4.8953 4.8865
2.5662 2.5487 2.0464 2.0265 2.0257 1.4225 2.7755 2.7709 2.7518 2.7473
1.1590 1.0083
pallambin D (2)
Chemical Formula: C19H22O4
Exact Mass: 314.15
Elemental Analysis: C, 72.59; H, 7.05; O, 20.36
1.000 1.123 1.018 1.106 2.058 0.966 1.062 1.041 1.039 3.019 3.025 3.062 3.045
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


Bruker Advance III 400
NAME XS-1-138-A1
EXPNO 22
PROCNO 1
Date_ 20120229
Time 21.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12822
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6379183 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 18.62 dB
PL2W 13.56617069 W
PL12W 0.32844096 W
PL13W 0.14806664 W
SFO2 400.1916008 MHz
SI 32768
SF 100.6278416 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
201.605 169.529 157.123 141.886 132.374 127.684 126.970 121.734 94.507 83.802 77.765 77.477 77.160 76.842 64.033 62.794 60.620 47.026 22.498 19.782 16.179 12.842
pallambin D (2)
Chemical Formula: C19H22O4
Exact Mass: 314.15
Elemental Analysis: C, 72.59; H, 7.05; O, 20.36
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm