Total Synthesis of (–)-Chromodorolide B

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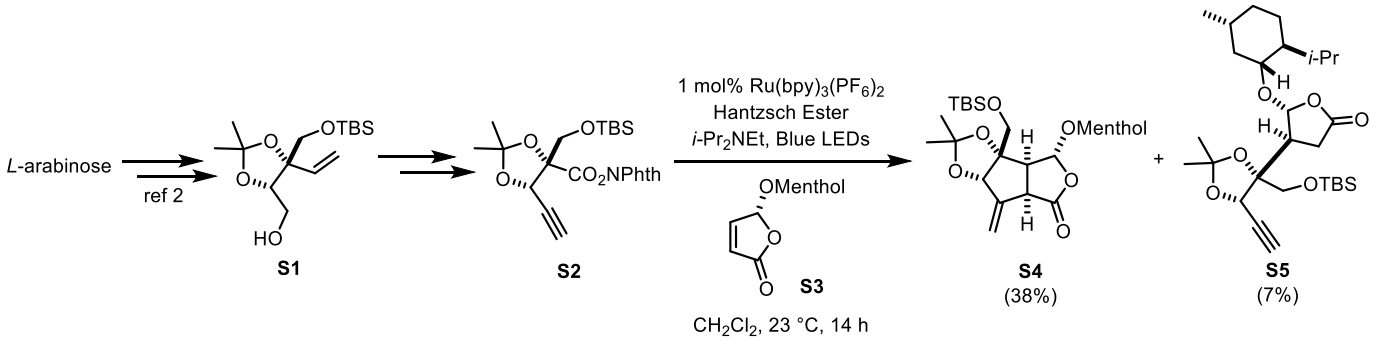
Materials and Methods

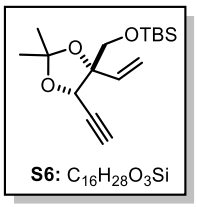
Unless stated otherwise, reactions were conducted in oven-dried glassware under an atmosphere of nitrogen or argon. Tetrahydrofuran (THF), diethyl ether, toluene, benzene, dichloromethane, methanol (MeOH), pyridine, DIPEA, and triethylamine were dried by passage through activated alumina. Benzyloxymethyl chloride (BOM-Cl) distilled under Ar from CaH directly before use. 1,1,3,3-Tetramethylguanidine was distilled under Ar from barium oxide directly before use. Thionyl chloride was distilled from quinoline under Ar. Tributylphosphine was distilled under reduced pressure and stored in a Schlenk flask. All other commercial reagents were used as received unless otherwise noted. Hantzsch ester[1a] (**HE**) and its 4-dideutero derivative[1b] were prepared according to literature procedures. Reaction temperatures were controlled using a temperature modulator, and unless stated otherwise, reactions were performed at room temperature (rt, approximately 23 °C). Thin-layer chromatography (TLC) was conducted with silica gel 60 F254 pre-coated plates, (0.25 mm) and visualized by exposure to UV light (254 nm) or by *p*-anisaldehyde, ceric ammonium molybdate, and potassium permanganate staining. Silica gel 60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded at 500 or 600 MHz and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), and integration. For late-stage intermediates, integrations for the upfield protons (δ2.00–0.50) of the hydrindane fragment were assigned based on 2D NMR techniques and chemical intuition. ^{13}C NMR spectra were recorded at 125 or 150 MHz. Data for ^{13}C NMR spectra are reported in terms of chemical shift. IR spectra were recorded on a FT-IR spectrometer and are reported in terms of frequency of absorption (cm^{-1}). High-resolution mass spectra were obtained with a LCT spectrometer. Blue LEDs (30 cm, 1 watt) were purchased from http://www.creativelightings.com (product code CL-FRS5050-12WP-12V) and powered by 8 AA

batteries. See JOC Standard Abbreviations and Acronyms for abbreviations (available at http://pubs.acs.org/userim ages/ContentEditor/1218717864819/joceah _abbreviations.pdf).

Model System Study and Synthetic Procedures



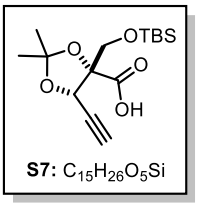
(+)-Tert-butyl(((4*S*,5*S*)-5-ethynyl-2,2-dimethyl-4-vinyl-1,3-dioxolan-4-yl)methoxy)dimethylsilane (S6): To a suspension of known alcohol **S1**[2] (0.732 g, 2.42 mmol) and solid $NaHCO_3$ (1.01 g, 12.1 mmol) in CH_2Cl_2 (4 mL) was added Dess-Martin periodinane (1.23 g, 2.90 mmol). The reaction was vigorously stirred for 2 h, at which point the suspension was filtered through Celite and concentrated *in vacuo*. The residue was then washed with pentanes (4 x 8 mL), and the combined organic washes were filtered through Celite and concentrated *in vacuo* to afford the crude aldehyde as a yellow oil which was carried forward immediately.



The crude aldehyde and dimethyl (1-azoacetonyl)phosphonate (0.558 g, 2.90 mmol) were dissolved in MeOH (9 mL). Solid K_2CO_3 (0.669 g, 4.84 mmol) was then added, and the suspension was vigorously stirred for 2 h. Celite (~5 g) was added to the reaction vessel, and the reaction was concentrated *in vacuo.* Purification by flash column chromatography (5% EtOAc in hexanes to 7% EtOAc in hexanes) afforded alkyne **S6** (0.550 g, 1.86 mmol, 77% yield) as a clear oil, which solidified upon standing. R_f 0.90 (20% EtOAc in hexanes; visualized with $KMnO_4$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.12 (dd, *J* = 17.2, 10.9 Hz, 1H), 5.53 (dd, *J* = 17.5, 1.6 Hz, 1H), 5.30 (dd, *J* = 10.9, 1.6 Hz, 1H), 4.99 (d, *J* = 2.1 Hz, 1H), 3.58 (d, *J* = 10.6 Hz, 1H), 3.55 (d, *J* = 10.7 Hz, 1H),

2.60 (d, J = 2.2 Hz, 1H), 1.54 (s, 3H), 1.43 (s, 3H), 0.89 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 136.46, 116.73, 110.27, 85.75, 79.25, 77.10, 69.70, 65.41, 27.87, 27.01, 26.00, 18.44, –5.23, –5.49; IR (thin film) 3312, 2988, 2955, 2858, 1741, 1378, 1253 cm^{-1}; $[\alpha]^{25}_{D}$: +0.79 (c = 2.5, CH_2Cl_2); HRMS (ESI) calculated for $C_{16}H_{29}O_3Si$ (M+H) 297.1887, observed 297.1890; mp 39–41 ºC.

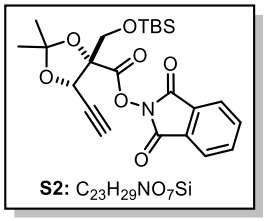
(–)-(4*S*,5*S*)-4-(((tert-butyldimethylsilyl)oxy)methyl)-5-ethynyl-2,2-dimethyl-1,3-dioxolane-4-carboxylic acid (S7): A solution of alkyne **S6** (0.553 g, 1.87 mmol) in methanol (8 mL) was cooled to –78 °C. Ozone from an ozone generator was bubbled through the solution until a pale blue color was observed (~5 min). The solution was then sparged with oxygen until the pale blue color disappeared. Dimethyl sulfide (0.31 mL, 4.3 mmol) was added to the solution, which was maintained at –78 °C for 1 h. The reaction vessel was allowed to warm to 23 °C and concentrated *in vacuo*. The residue was then redissolved in a 3:1 solution of *t*-BuOH/H_2O (8 mL). A solution of 2-methyl-2-butene (2.0 mL, 19 mmol) was added to the mixture, followed by NaH_2PO_4 (1.80 g, 15.0 mmol) and $NaClO_2$ (0.845 g, 9.35 mmol). The reaction was maintained at 23 °C for 2 h, at which point H_2O (4 mL) was added. This mixture was washed with EtOAc (3 x 10 mL), and the combined organic layers were washed with aq. NaOH (5 mL of 0.5 M soln). The aqueous layer was then acidified with aq. HCl (7 mL of 0.5 M soln). The aqueous layer was then washed with EtOAc (3 x 10 mL), and the combined organic layers were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo* to provide acid **S7** as a colorless oil (0.450 g, 1.43 mmol, 76% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.97 (d, J = 2.3 Hz, 1H), 3.94 (d, J = 11.0 Hz, 1H), 3.92 (d, J = 11.0 Hz, 1H), 2.63 (d, J = 2.2 Hz, 1H), 1.67 (s, 3H), 1.47 (s, 3H), 0.90 (s, 9H), 0.09 (s, 3H), 0.08 (s, 3H); ^{13}C NMR (500 MHz,



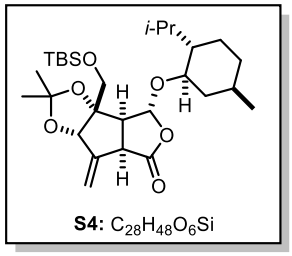
S7: $C_{15}H_{26}O_5Si$

$CDCl_3$) δ 173.25, 113.33, 88.23, 77.69, 69.04, 63.64, 26.99, 26.97, 25.95, 18.48, 14.32, –5.26, –5.48; IR (thin film) 3505, 3277, 2990, 2931, 2858, 1731, 1379 cm^{-1}; $[\alpha]^{25}_D$: –30.0 (c = 2.1, CH_2Cl_2); HRMS (ESI) calculated for $C_{15}H_{25}O_5Si$ (M–H) 313.1471, observed 313.1467.

(+)-1,3-dioxoisoindolin-2-yl(4*S*,5*S*)-4-(((tert-butyldimethylsilyl)oxy)methyl)-5-ethynyl-2,2-dimethyl-1,3-dioxolane-4-carboxylate (S2): Acid **S7** (0.453 g, 1.44 mmol) was charged into a flask with THF (8 mL). *N*-hydroxyphthalimide (0.399 g, 2.45 mmol), *N*,*N*'-dicycylohexylcarbodiimide (0.446 g, 2.16 mmol), and 4-dimethylaminopyridine (9 mg, 0.07 mmol) were added to the reaction vessel, which was maintained at 23 °C for 20 h. Hexanes (5 mL) was added to the reaction, and the resulting suspension was filtered through Celite. The yellow filtrate was concentrated *in vacuo* and then purified by flash column chromatography (10% EtOAc in hexanes to 15% EtOAc in hexanes) to provide *N*-acyloxyphthalimide **S2** (0.539 g, 1.18 mmol, 82% yield) as a colorless crystalline solid. R_f 0.25 (20% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (m, 2H), 7.78 (m, 2H), 5.14 (d, *J* = 2.2 Hz, 1H), 4.10 (d, *J* = 11.5 Hz, 1H), 4.07 (d, *J* = 11.3 Hz, 1H), 2.78 (d, *J* = 2.2 Hz, 1H), 1.71 (s, 3H), 1.50 (s, 3H), 0.93 (s, 9H), 0.13 (s, 3H), 0.12 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 166.77, 161.37, 134.87, 129.09, 124.07, 113.53, 88.06, 78.80, 76.25, 68.81, 62.80, 26.7, 26.64, 26.00, 18.54, –5.18, –5.49; IR (thin film) 3283, 2930, 2855, 2360, 2340, 2118, 1789, 1748 cm^{-1}; $[\alpha]^{25}_D$: +38.3 (c = 2.0, CH_2Cl_2); HRMS (ESI) calculated for $C_{23}H_{29}NO_7SiNa$(M+Na) 482.1611, observed 482.1612; mp 105–109 ºC.

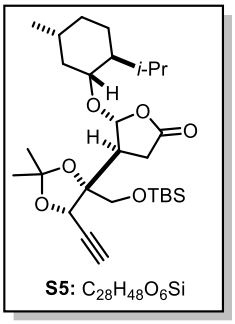


(–)-(3a*S*,3b*S*,4*R*,6a*R*,7a*S*)-3a-(((tert-butyldimethylsilyl)oxy)methyl)-4-(((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)oxy)-2,2-dimethyl-7-methylenehexahydro-6H-furo[3',4':3,4]cyclopenta[1,2-d][1,3]dioxol-6-one (S4): To a vial charged with *N*-acyloxyphthalimide **S2** (100 mg, 0.218 mmol) was added CH_2Cl_2 (2 mL) that had been separately sparged with argon. Butenolide **S3**[3] (78 mg, 0.33 mmol), Hantzsch ester (82 mg, 0.63 mmol), $Ru(bpy)_3(PF_6)_2$ (2 mg, 0.02 mmol), and Hünig's base (80 μL, 0.48 mmol) were then added to the reaction. The vial was then vigorously stirred while being irradiated by a single strip of blue LED lights (450 nm) at 23 ºC. After 6 h, the reaction mixture was diluted with hexanes (2 mL) and filtered through Celite. The resulting solution was then concentrated *in vacuo* and separated by flash column chromatography (3% EtOAc in hexanes to 5% EtOAc in hexanes) to provide lactone **S4** (42 mg, 0.083 mmol, 38% yield) as a colorless, crystalline solid and addition product **S5** (7.5 mg, 0.015 mmol, 7% yield) as an oil. A single crystal X-ray structure of lactone **S4** was obtained after recrystallization in MeOH/hexanes to confirm structural assignment.[4] R_f for **S4**: 0.65 (10% EtOAc in hexanes; visualized with ceric ammonium molybdate). R_f for **S5**: 0.60 (10% EtOAc in hexanes; visualized with ceric ammonium molybdate).



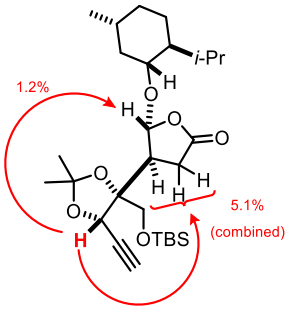
S4 for ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.90 (d, *J* = 2.6 Hz, 1H), 5.62 (dd, *J* = 2.3, 1.0 Hz, 1H), 5.50 (dd, *J* = 2.7, 0.8 Hz, 1H), 4.73 (app s, 1H), 3.96–3.92 (m, 1H), 3.86 (d, *J* = 10.7 Hz, 1H), 3.77 (d, *J* = 10.8 Hz, 1H), 3.51 (dt, *J* = 10.6, 4.3 Hz, 1H), 3.07 (dd, *J* = 10.3, 2.3 Hz, 1H), 2.14–2.03 (m, 2H), 1.69–1.60 (m, 2H), 1.47 (s, 3H), 1.40–1.32 (m, 4H), 1.27–1.16 (m, 2H), 1.04–0.93 (m, 1H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.90–0.80 (m, 13H), 0.77 (d, *J* = 6.9 Hz, 3H), 0.09 (s, 3H), 0.08 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 175.05, 143.32, 116.61, 112.69, 99.71, 90.76, 86.88, 77.22, 64.36, 57.42, 48.22, 47.87, 40.01, 34.42, 31.51, 28.37, 27.04, 25.93, 25.51, 23.20, 22.39, 21.03, 18.41,

15.81, –5.45, –5.50; IR (thin film) 2953, 2929, 2858, 1779, 1461 cm^{-1}; $[\alpha]^{25}_D$: –133 (c = 1.9, CH_2Cl_2); HRMS (ESI) calculated for $C_{28}H_{48}O_6SiNa$ (M+Na) 531.3118, observed 531.3126; mp 136–142 ºC.



S5: $C_{28}H_{48}O_6Si$

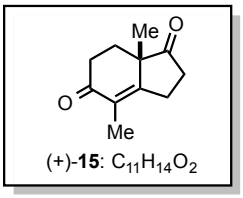
(–)-S5 for ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.82 (d, *J* = 2.2 Hz, 1H), 4.59 (d, *J* = 2.2 Hz, 1H), 3.98 (d, *J* = 10.7 Hz, 1H), 3.75 (d, *J* = 10.7 Hz, 1H), 3.52 (dt, *J* = 10.9, 4.4 Hz, 1H), 2.82–2.65 (m, 3H), 2.58 (d, *J* = 2.3 Hz, 1H), 2.17–2.04 (m, 2H), 1.69–1.59 (m, 2H), 1.50 (s, 3H), 1.38 (s, 3H), 1.37–1.32 (m, 1H), 1.27–1.17 (m, 2H), 0.99 (app qd, *J* = 12.5, 3.3 Hz, 1H), 0.91 (d, *J* = 6.9 Hz, 3H), 0.90 (s, 9H), 0.87 (d, *J* = 7.0 Hz, 3H), 0.86–0.81 (m, 1H), 0.77 (d, *J* = 7.0 Hz, 3H), 0.09 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 175.88, 110.04, 100.76, 83.81, 78.81, 77.21, 76.74, 71.62, 64.38, 47.88, 46.89, 39.88, 34.46, 31.50, 29.82, 28.24, 27.07, 26.04, 25.56, 23.22, 22.42, 21.02, 18.36, 15.80, –5.44, –5.46; IR (thin film) 3311, 3262, 2955, 2929, 2858, 1791, 1462, 1374, 1252 cm^{-1}; $[\alpha]^{25}_D$: –93.7 (c = 1.2, CH_2Cl_2); HRMS (ESI) calculated for $C_{28}H_{48}O_6SiNa$ (M+Na) 531.3118, observed 531.3131.



NOE Correlations

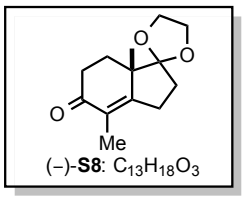
Synthetic Procedures for Total Synthesis of (–)-Chromodorolide B

(+)-(*S*)-4,7a-dimethyl-2,3,7,7a-tetrahydro-1*H*-indene-1,5(6*H*)-dione (15): (*S*)-enone was prepared according to a literature procedure.[5] A 100 mL round-bottom flask was charged with 2-methyl-2-(3-oxopentyl)cyclopetane-1,3-dione (39.6 g, 202 mmol), followed by the addition of *L*-phenylalanine (10 g, 61 mmol), PPTS (25.3 g, 101 mmol), and DMSO (14 mL, 200 mmol). The heterogeneous reaction mixture was then sonicated for 36 h at 50 ºC. The mixture was transferred into a separatory funnel with EtOAc (500 mL), followed by the addition of H_2O (500 mL). The two layers were separated and the aqueous phase was extracted with EtOAc (3 x 500 mL). The combined organic extracts were washed sequentially with aq. HCl (1 x 500 mL of 1 M soln), sat. aq. $NaHCO_3$ (1 x 500 mL), and brine (1 x 500 mL). The organic phase was dried over $MgSO_4$ and concentrated *in vacuo* to yield a viscous red oil. The resulting oil was filtered through a silica gel plug (100 g SiO_2) with 30% EtOAc in hexanes (1 L) to afford 32.7 g of crude (+)-**15** as an orange oil. R_f 0.23 (30% EtOAc in hexanes; visualized with *p*-anisaldehyde). The resulting oil was crystallized from Et_2O (23 mL) at –20 ºC utilizing a seed crystal (obtained via crystallization from Et_2O/benzene at –20 ºC) to provide (+)-**15** as an off-white crystalline solid (21.3 g, 120 mmol, 59% yield, 99% *ee*). Spectral data were consistent with reported values.[5] The enantiomeric excess was determined by chiral stationary-phase HPLC analysis (Chiracel OB-H column; flow: 2.0 mL/min, 20% isopropanol:*n*-hexane; λ = 254 nm; minor enantiomer t_R = 5.86 min, major enantiomer t_R = 7.25 min).



(+)-**15**: $C_{11}H_{14}O_2$

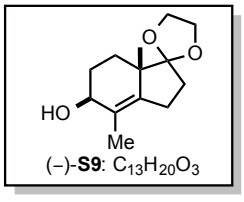
(–)-(*S*)-4,7a-dimethyl-2,3,7,7a-tetrahydrospiro[indene-1,2-[1,3]dioxolan]-5(6*H*)-one (S8): A



(–)-**S8**: $C_{13}H_{18}O_3$

2 L round-bottom flask was charged with enone (+)-**15** (7.7 g, 43 mmol), followed by the addition of benzene (860 mL), ethylene glycol (2.9 mL, 52 mmol), and *p*-TsOH•H_2O (1.6 g, 8.6 mmol). A Dean-Stark apparatus was fitted to the flask, and the homogenous reaction mixture was maintained at reflux overnight. Upon completion of the reaction, as indicated by TLC analysis (40% EtOAc in hexanes; visualized with *p*-anisaldehyde), the mixture was cooled to 0 ºC and sat. aq. $NaHCO_3$ (300 mL) was added. The resulting biphasic mixture was separated and the aqueous layer was extracted with Et_2O (3 x 200 mL). The combined organic layers were washed with brine (1 x 500 mL), dried over $MgSO_4$, and concentrated *in vacuo* to yield a yellow oil. The crude residue was purified by flash column chromatography (20% EtOAc in hexanes) to yield **S8** as a yellow oil (9.6 g, 43 mmol, 100% yield). R_f 0.45 (40% EtOAc in hexanes; visualized with *p*-anisaldehyde). ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.04–3.91 (m, 4H), 2.58–2.51 (m, 2H), 2.43 (dd, *J* = 5.4, 3.6 Hz, 1H), 2.28 (td, *J* = 13.2, 5.4 Hz, 1H), 2.20–2.16 (m, 1H), 1.95–1.91 (m, 1H), 1.68 (s, 3H), 1.61–1.58 (m, 1H), 1.22 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.63, 167.10, 128.96, 118.04, 65.94, 65.05, 47.68, 33.23, 31.92, 26.80, 25.99, 20.49, 10.83; IR (thin film) 2953, 2881, 1660, 1451, 1153 cm^{-1}; $[\alpha]^{21}_D$: –7.79 (c = 1.6, CH_2Cl_2); HRMS (ESI) calculated for $[C_{13}H_{18}O_3Na]^+$ (M+Na) 245.1154, observed 245.1163.

(–)-(5*S*,7a*S*)-4,7a-dimethyl-2,3,5,6,7,7a-hexahydrospiro[indene-1,2'-[1,3]dioxolan]-5-ol (S9):

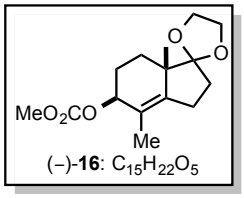


(–)-**S9**: $C_{13}H_{20}O_3$

A 1 L round-bottom flask was charged with enone **S8** (9.6 g, 43 mmol), followed by the addition of Et_2O (310 mL). The solution was cooled to –78 ºC. A solution of $LiAlH_4$ (65 mL of 1 M in Et_2O, 65 mmol) was added dropwise. The homogenous solution was warmed to 0 ºC and maintained at that temperature until TLC

analysis (40% EtOAc in hexanes; visualized with *p*-anisaldehyde) indicated complete consumption of the starting material, typically 10–20 min. Upon completion of the reaction, sat. aq. Rochelle's salt (150 mL) was slowly added. The biphasic mixture was stirred vigorously for 30 min at 0 ºC. The solution was then transferred to a separatory funnel and extracted with Et_2O (3 x 150 mL). Combined organic layers were washed with brine (1 x 500 mL), dried over $MgSO_4$, and concentrated *in vacuo* to yield a colorless solid. The crude residue was purified by flash column chromatography (30% EtOAc in hexanes) to yield **S9** as a colorless, crystalline solid (9.5 g, 42 mmol, 98% yield). R_f 0.25 (40% EtOAc in hexanes; visualized with *p*-anisaldehyde). ^1H NMR (500 MHz, $CDCl_3$) δ 4.14–4.12 (m, 1H), 3.96–3.86 (m, 4H), 2.33–2.30 (m, 2H), 2.13–2.06 (m, 2H), 1.87–1.78 (m, 2H), 1.71–1.62 (m, 4H), 1.35–1.31 (m, 2H), 1.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.44, 127.91, 118.50, 71.73, 65.73, 64.94, 46.72, 32.07, 30.05, 26.64, 23.98, 22.51, 14.57; IR (thin film) 3411, 2947, 2873, 1642, 1149 cm^{-1}; $[\alpha]^{21}_D$: –32.0 (c = 1.8, CH_2Cl_2); HRMS (ESI) calculated for $[C_{13}H_{20}O_3]$ (M) 224.1413, observed 224.1406; mp 86–88 ºC.

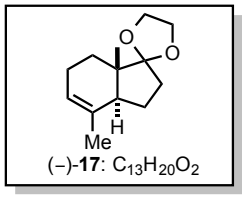
(–)-(5*S*,7a*S*)-4,7a-dimethyl-2,3,5,6,7,7a-hexahydrospiro[indene-1,2'-[1,3]dioxolan]-5-yl



methyl carbonate (16): A 1 L round-bottom flask was charged with allylic alcohol **S9** (9.5 g, 42 mmol), followed by the addition of DMAP (15.0 g, 126 mmol), CH_2Cl_2 (420 mL), and methyl chloroformate (13 mL, 170 mmol). The resulting homogenous solution was maintained at 35 ºC until TLC analysis (30% EtOAc in hexanes; visualized with *p*-anisaldehyde) indicated complete consumption of the starting material, typically 30–90 min. Upon completion of the reaction, the mixture was cooled to room temperature, followed by the addition of sat. aq. NH_4Cl (200 mL). Next, the solution was transferred to a separatory funnel and extracted with CH_2Cl_2 (3 x 200 mL). The combined organic

layers were washed with brine (1 x 500 mL), dried over $MgSO_4$, and concentrated *in vacuo* to yield a colorless solid. The crude residue was purified by flash column chromatography (10% EtOAc in hexanes) to yield **16** as a colorless, crystalline solid (11.4 g, 40.4 mmol, 96% yield). R_f 0.35 (20% EtOAc in hexanes; visualized with *p*-anisaldehyde). ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.19 (app t, *J* = 7.8 Hz, 1H), 3.96–3.87 (m, 4H), 3.79 (s, 3H), 2.34 (t, *J* = 7.8 Hz, 2H), 2.24–2.20 (m, 1H), 2.12–2.07 (m, 1H), 1.89 (td, *J* = 14.4, 3.0 Hz, 1H), 1.84–1.77 (m, 2H), 1.57 (s, 3H), 1.36 (dt, *J* = 12.7, 3.8 Hz, 1H), 1.15 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.27, 144.24, 123.87, 118.39, 78.66, 65.76, 64.93, 54.80, 46.47, 31.90, 26.34, 25.76, 24.11, 22.15, 14.69; IR (thin film) 2954, 2878, 1742, 1442, 1260 cm^{-1}; $[\alpha]^{22}_D$: –41.32 (c = 1.1, CH_2Cl_2); HRMS (ESI) calculated for $[C_{15}H_{22}O_5Na]^+$ (M+Na) 305.1365, observed 305.1370; mp 56–58 ºC.

(–)-(3a*S*,7a*S*)-4,7a-dimethyl-2,3,3a,6,7,7a-hexahydrospiro[indene-1,2'-[1,3]dioxolane] (17):

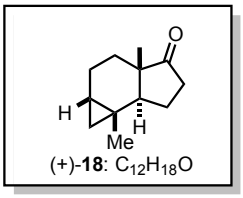


(–)-**17**: $C_{13}H_{20}O_2$

A 200 mL round-bottom flask was charged with $Pd(acac)_2$ (490 mg, 1.6 mmol), followed by the addition of benzene (21 mL), PBu_3 (0.4 mL, 1.6 mmol). The homogenous mixture was maintained at room temperature for 5 min. Ammonium formate (4.2 g, 67 mmol) was finely crushed with a mortar and pestle and added to the reaction mixture in one portion. The resulting heterogeneous solution was stirred vigorously for 10 min. Next, a solution of carbonate **16** (3.0 g, 10.6 mmol) in benzene (32 mL) was added dropwise. The heterogeneous mixture was stirred vigorously overnight at room temperature. Upon completion of the reaction, as indicated by TLC analysis (10% EtOAc in hexanes; visualized with *p*-anisaldehyde), the mixture was filtered through a silica gel plug (10%EtOAc in hexanes) to provide a brown oil. The crude residue was purified by flash column chromatography (350 g SiO_2, 1 L 100% hexanes, 1 L 0.5% EtOAc in hexanes, 5 L 1% EtOAc in hexanes) to yield (–)-**17** (1.72

g, 8.26 mmol, 78% yield) as a yellow oil that contained ~1% tributylphosphine oxide as an impurity. R_f 0.2 (2% EtOAc in hexanes, visualized with *p*-anisaldehyde). *Note*: It is helpful to develop the TLC plate 2 times to visualize two more polar impurities with similar R_f values. If desired, the yellow oil can be purified further by Kugelrohr short-pass distillation (135 ºC, 0.6 torr) to yield **17** (1.69 g, 8.11 mmol, 77% yield) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.20 (s 1H), 3.94–3.84 (m, 4H), 2.46 (br s, 1H), 2.10–2.05 (m, 3H), 1.91–1.84 (m, 1H), 1.78–1.69 (m, 2H), 1.62 (s, 3H), 1.46–1.40 (m, 2H), 0.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.88, 119.96, 119.36, 65.44, 64.73, 46.38, 45.69, 35.21, 28.47, 24.08, 22.04, 20.45, 13.84; IR (thin film) 2944, 2878, 1436, 1376, 1044 cm^{-1}; $[\alpha]^{21}_D$: –62.3 (c = 1.0, CH_2Cl_2); HRMS (ESI) calculated for [$C_{13}H_{20}O_2$] (M) 208.1463, observed 208.1460.

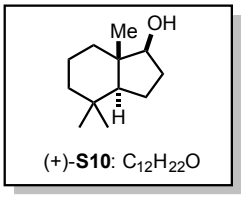
(+)-(1a*S*,3a*S*,6a*S*,6b*R*)-3a,6b-dimethyloctahydrocyclopropa[*e*]inden-4(1*H*)-one (18): A 500 mL round-bottom was charged with ketal **17** (6.2 g, 30 mmol), followed by the addition of CH_2Cl_2 (150 mL). The solution was cooled to 0 ºC, and a solution of Et_2Zn (60 mL of 1 M in hexanes, 60 mmol) was added dropwise. After 10 min at 0 ºC, chloroidomethane (8.7 mL, 120 mmol) was added. After 2 h at 0 ºC, the heterogeneous mixture was allowed to warm to room temperature and stirred overnight while shielded from light. Upon cooling the reaction mixture to 0 ºC, conc. HCl (7.6 mL) in MeOH (115 mL) was added dropwise. Upon complete deprotection of the ketal (typically 10–30 min), as indicated by TLC analysis (5% EtOAc in hexanes, visualized with *p*-anisaldehyde), the mixture was transferred to a separatory funnel. H_2O (150 mL) was added, and the resulting biphasic mixture was extracted with CH_2Cl_2 (3 x 200 mL). The combined organic layers were washed with brine (1 x 500 mL), dried over $MgSO_4$, and concentrated *in vacuo* to yield a yellow oil. The crude



(+)-**18**: $C_{12}H_{18}O$

residue was purified by flash column chromatography (0% to 2% EtOAc in hexanes) to yield cyclopropane **18** as a colorless oil (4.9 g, 28 mmol, 92% yield). R_f 0.15 (5% EtOAc in hexanes; visualized with *p*-anisaldehyde). Alternatively, cyclopropane **18** can be purified by Kugelrohr short-pass distillation (100 ºC, 0.4 torr). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.50–2.45 (m, 1H), 2.10–2.04 (m, 2H), 2.01–1.97 (m, 1H), 1.91–1.85 (m, 1H), 1.81 (dd, *J* = 14.4, 6.9 Hz, 1H), 1.66 (dd, *J* = 13.4, 7.8 Hz, 1H), 1.37 (dd, *J* = 13.3, 6.0 Hz, 1H), 1.10 (s, 3H), 0.90–0.85 (m, 4H), 0.67–0.64 (m, 1H), 0.56 (dd, *J* = 9.5, 4.3 Hz, 1H), 0.01 (app t, *J* = 5.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 221.17, 50.66, 47.34, 36.62, 27.99, 23.29, 22.96, 22.21, 19.73, 17.84, 16.30, 13.33; IR (thin film) 3051, 2945, 2864, 1737, 1445, 1024 cm^{-1}; $[\alpha]^{22}_D$: +118 (c = 1.0, CH_2Cl_2); HRMS (ESI) calculated for [$C_{12}H_{18}O$] (M) 178.1358, observed 178.1358.

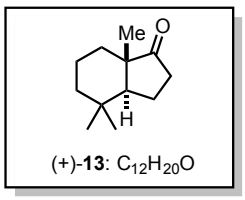
(+)-(1*S*,3a*S*,7a*S*)-4,4,7a-trimethyloctahydro-1*H*-inden-1-ol (S10): A 20 mL vial was charged with ketone **18** (1.1 g, 6.0 mmol), followed by the addition of AcOH (6.0 mL), PtO_2•H_2O (270 mg, 1.2 mmol). The flask was then placed in a Parr high pressure vessel that was subsequently filled with H_2 (10 atm). The vessel was placed on top of an IKA magnetic plate and stirred overnight. The reaction mixture was filtered through Celite into a separatory funnel, followed by the addition of EtOAc (50 mL). The resulting solution was washed with H_2O (3 x 50 mL), sat. aq. $NaHCO_3$ (1 x 50 mL), and brine (1 x 50 mL). The organic layer was then dried over $MgSO_4$ and concentrated *in vacuo* to yield a colorless solid. The crude residue was purified by flash column chromatography (2% to 6% EtOAc in hexanes) to yield **S10** as a colorless solid (1.02 g, 5.59 mmol, 93% yield) that contained ~5% of an impurity. R_f 0.20 (10% EtOAc in hexanes; visualized with *p*-anisaldehyde). Recrystallization from hot *n*-



(+)-**S10**: $C_{12}H_{22}O$

hexanes (50 mL) yielded **S10** as colorless needles (820 mg, 4.5 mmol, 81% recovery). Spectral data were consistent with reported values.[6]

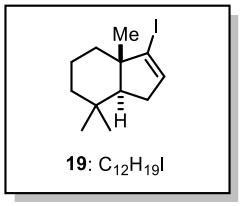
(+)-(3a*S*,7a*S*)-4,4,7a-trimethyloctahydro-1H-inden-1-one (13): A 100 mL round-bottom flask



(+)-**13**: $C_{12}H_{20}O$

was charged with alcohol **S10** (0.80 g, 4.4 mmol), followed by the addition of PCC (2.0 g, 9.4 mmol), Celite (2.0 g), and CH_2Cl_2 (22 mL). The resulting heterogenous solution was stirred vigorously at room temperature, until TLC analysis (10% EtOAc in hexanes, visualized with *p*-anisaldehyde) indicated complete consumption of the starting material (typically 60–90 min). Hexanes (22 mL) was added to the reaction mixture, which was subsequently gravity filtered. The reaction vessel and filtrate were washed with 10% EtOAc in hexanes (4 **x** 25 mL). The combined organic washes were concentrated *in vacuo* to yield an orange oil. The crude residue was purified by flash column chromatography (0% to 2% EtOAc in hexanes) to provide **13** (0.75 g, 4.2 mmol, 95% yield, 98.5% *ee*) as a colorless oil, which solidified upon standing. R_f 0.29 (5% EtOAc in hexanes, visualized with *p*-anisaldehyde). Alternatively, ketone **13** can be purified by Kugelrohr short-pass distillation (130 ºC, 0.8 torr). Spectral data were consistent with reported values.[6] The enantiomeric excess of the corresponding trisyl hydrazone[7] was determined by chiral stationary-phase HPLC analysis (Chiracel OD-H column; flow: 1.0 mL/min, 1% isopropanol:*n*-hexane; λ = 254 nm; minor enantiomer t_R = 13.65 min, major enantiomer t_R = 21.34 min). *Note*: The reaction is readily scalable. In a separate experiment, crude alcohol **S10** (4.46 g, 24.5 mmol) was oxidized according to the above procedure to yield ketone **13** (4.33 g, 24.0 mmol, 96% yield) as a colorless oil. The material contained ~5% impurity that was carried through from the previous step. Therefore, it is important to recrystallize alcohol **S10** prior to oxidation to obtain pure ketone **13**.

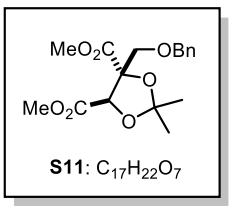
(–)-(3a*S*,7a*S*)-3-iodo-3a,7,7-trimethyl-3a,4,5,6,7,7a-hexahydro-1H-indene (19): Hydrazine hydrate (20 mL) and NEt_3 (16.3 mL, 118 mmol) were added to a solution of ketone **13** (1.06 g, 5.88 mmol) in EtOH (45 mL). The reaction was heated to reflux for 20 h; upon cooling to 23 °C, CH_2Cl_2 (100 mL) and H_2O (150 mL) were added. The aqueous layer was washed with CH_2Cl_2 (3 x 100 mL), and the combined organic layers were dried over Mg_2SO_4, filtered, and concentrated *in vacuo*. The remaining white solid (excess hydrazine) was removed by filtration using hexanes. Concentration *in vacuo* provided the crude hydrazone as a yellow oil, which was carried forward without further purification.



19: $C_{12}H_{19}I$

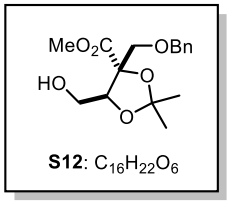
A solution of 1,1,3,3-Tetramethylguanidine (5.15 mL, 41.2 mmol) in THF (30 mL) was added dropwise over 10 min to a solution of I_2 (3.28 g, 12.9 mmol) in THF (30 mL). The hydrazone (5.88 mmol) in THF (6 mL) was then added dropwise over 10 min, and the reaction was maintained for 30 min. The dark red solution was then concentrated *in vacuo*, and the resulting red oil was heated neat at 90 °C for 5 h with a reflux condenser attached. The reaction was then cooled to 23 °C, diluted with Et_2O (60 mL), and concentrated *in vacuo* over silica gel (~10 g). Purification by flash column chromatography (100% hexanes) provided light-sensitive vinyl iodide **19** (1.33 g, 4.58 mmol, 78%) as a colorless, crystalline solid. Spectral data were consistent with reported values.[6,7]

(–)-Dimethyl (4*R*,5*R*)-4-((benzyloxy)methyl)-2,2-dimethyl-1,3-dioxolane-4,5-dicarboxylate (S11): The procedure for the preparation of diester **S11** was a slight modification from the literature procedure.[8] Dimethyl 2,3,-*O*-isopropylidene-*L*-tartrate (3.83 g, 17.6 mmol) was dissolved in THF (67 mL) and cooled to –78 °C. HMPA (13 mL) was added, followed by BOM-Cl (5.6 mL, 40 mmol). Freshly prepared



S11: $C_{17}H_{22}O_7$

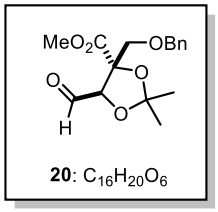
LDA (17.7 mmol) in THF (50 mL) was then added to the reaction flask via cannula over ~30 min. The reaction was maintained for 5 h at –78 °C, before warming to 0 °C. After 3 h, the reaction was quenched with sat. aq. NH_4Cl solution (50 mL). The organic layer was washed with H_2O (3 x 40 mL) and brine (1 x 40 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Unreacted dimethyl 2,3,-*O*-isopropylidene-*L*-tartrate was distilled from the crude product (120 °C, 0.3 torr). The remaining oil was purified by flash column chromatography (8% EtOAc in hexanes to 15% EtOAc in hexanes) to provide diester **S11** (2.75 g, 8.14 mmol, 46%) as a light yellow oil. This reaction could be run on larger scale (~5x) with similar yields (41–43%). R_f 0.80 (40% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.23 (m, 5H), 5.12 (s, 1H), 4.52 (d, *J* = 12.2 Hz, 1H), 4.47 (d, *J* = 12.2 Hz, 1H), 3.82 (s, 3H), 3.73 (d, *J* = 9.8 Hz, 1H), 3.70 (d, *J* = 9.8 Hz, 1H), 3.63 (s, 3H), 1.59 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 170.80, 168.80, 137.60, 128.43, 127.77, 127.53, 112.71, 85.27, 77.56, 73.71, 70.11, 53.17, 52.40, 27.44, 25.95; IR (thin film) 2989, 2950, 1743, 1442, 1436, 1391, 1382, 1256, 1211 cm^{-1}; $[\alpha]^{25}_D$: –33.8 (c = 1.7, CH_2Cl_2); HRMS (ESI) calculated for $C_{17}H_{22}O_7Na$ (M+Na) 361.1263, observed 361.1271.



(–)-Methyl (4*R*,5*S*)-4-((benzyloxy)methyl)-5-(hydroxymethyl)-2,2-dimethyl-1,3-dioxolane-4-carboxylate (S12): The procedure for the preparation of alcohol **S12** was a slight modification from the literature procedure.[8] Diester **S11** (17.7 g, 52.3 mmol) was dissolved in THF (450 mL) and cooled to –78 °C. DIBAL-H (14 mL, 79 mmol) was added dropwise to the reaction. After 5 min, the reaction was warmed to 0 °C. After 1 h, a saturated solution of Rochelle's salt (250 mL) and EtOAc (100 mL) were added. The reaction was allowed to warm to 23 °C, and the heterogeneous mixture was

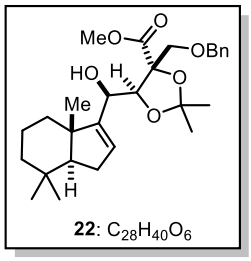
extracted with EtOAc (4 x 150 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was then purified by flash column chromatography (20% EtOAc in hexanes to 50% EtOAc in hexanes) to provide recovered diester **17** (6.34 g, 18.6 mmol, 36%) as a light yellow oil and alcohol **S12** (7.44 g, 23.9 mmol, 46%) as a clear oil. R_f 0.35 (40% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.25 (m, 5H), 4.56–4.51 (m 3H), 3.91 (dd, *J* = 12.1, 5.3 Hz, 1H), 3.85 (dd, *J* = 12.2, 5.5 Hz, 1H), 3.80 (s, 3H), 3.72 (d, *J* = 9.4 Hz, 1H), 3.65 (d, *J* = 9.4 Hz, 1H), 2.37 (bs, 1H), 1.47 (s, 3H), 1.40 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 171.80, 137.24, 128.70, 128.07, 127.89, 110.16, 83.83, 73.94, 70.51, 60.65, 52.91, 27.75, 25.34; IR (thin film) 3500, 2989, 2937, 2871, 1743, 1454, 1380 cm^{-1}; $[\alpha]^{25}_D$: –2.17 (c = 1.2); HRMS (ESI) calculated for $C_{16}H_{22}O_6NH_4$ ($M+NH_4$) 328.1760, observed 328.1754.

(–)-Methyl (4*R*,5*R*)-4-((benzyloxy)methyl)-5-formyl-2,2-dimethyl-1,3-dioxolane-4-carboxylate (20): To a stirring suspension of alcohol **S12** (4.80 g, 15.5 mmol) and $NaHCO_3$ (6.50 g, 77.4 mmol) in CH_2Cl_2 (40 mL) was added Dess-Martin periodinane (7.87 g, 18.6 mmol) in two portions over 5 min. After 2 h, the reaction mixture was diluted with Et_2O (40 mL) and filtered through a cotton plug to remove solid $NaHCO_3$. The filtrate was concentrated *in vacuo*, resulting in a white solid. The solid was then washed with hexanes (6 x 30 mL), and the combined hexane washes were filtered through Celite. Upon concentration, aldehyde **20** (4.33 g, 14.0 mmol, 91%) was obtained as a colorless oil. Notes: 1) Aldehyde **20** was found to decompose within 14 h upon its formation (at room temperature or in the freezer), possibly due to self-aldol polymerization. Therefore, it was always carried forward *immediately* into the next reaction. 2) Aldehyde **20** did not appear unstable to column



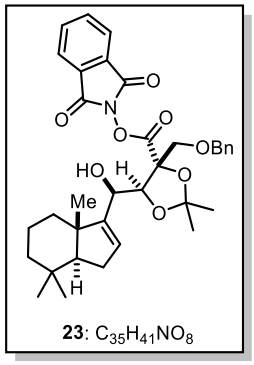
20: $C_{16}H_{20}O_6$

chromatography, but it could not be purified in that manner. 3) Aqueous washes diminished the yields, possibly from hydrate formation. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.69 (s, 1H); 7.36–7.24 (m, 5H), 4.89 (s, 1H), 4.47 (d, *J* = 12.1 Hz, 1H), 4.44 (d, *J* = 12.1 Hz, 1H), 3.82 (s, 3H), 3.66 (d, *J* = 10.0 Hz, 1H), 3.63 (d, *J* = 10.0 Hz, 1H), 1.59 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 197.23, 170.67, 137.15, 128.54, 127.84, 127.68, 112.75, 86.30, 82.95, 73.47, 69.28, 53.27, 27.30, 25.77; IR (thin film) 2991, 2937, 2868, 1740, 1454, 1374 cm^{-1}; $[\alpha]^{25}_D$: –2.37 (c = 2.5, CH_2Cl_2); HRMS (ESI) calculated for $[C_{16}H_{20}O_6NH_4]^+$ (M+NH_4) 326.1604, observed 326.1612.



(–)-Methyl (4*R*,5*S*)-4-((benzyloxy)methyl)-5-((*R*)-hydroxy((3a*S*,7a*S*)-3a,7,7-trimethyl-3a,4,5,6,7,7a-hexahydro-1H-inden-3-yl)methyl)-2,2-dimethyl-1,3-dioxolane-4-carboxylate (22): *L*-Oxazoline **21**[9] (2.86 g, 9.65 mmol) and $CrCl_2$ (1.19 g, 9.65 mmol) were dissolved in THF (20 mL) in the glove box, and NEt_3 (1.34 mL, 9.65 mmol) was then added. The suspension was vigorously stirred for 6 h, and then $NiCl_2$ (36 mg, 0.28 mmol) was added, followed by a solution of vinyl iodide **19** (0.80 g, 2.8 mmol) and aldehyde **20** (1.30 g, 4.21 mmol) in THF (10 mL). Vigorous stirring was maintained for 20 h before removing the flask from the glovebox and cooling the solution to 0 ºC. Ethylene diamine (2 mL) was added to quench the reaction. After stirring for 30 min, H_2O (40 mL) and Et_2O (40 mL) were added. The aqueous layer was extracted with EtOAc (4 x 20 mL), and the combined organic layers were washed with sat. aq. $NaHCO_3$ solution (40 mL) and brine (1 x 40 mL), dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by flash column chromatography (5% EtOAc in hexanes to 11% EtOAc in hexanes) provided a single diastereomer, alcohol **22** (0.860 g, 1.82 mmol, 66%) as a clear oil. *L*-Oxazoline **21** was recovered during flash column chromatography (60–80% recovery) and recrystallized from Et_2O/hexanes for reuse. R_f 0.50 for **22** (20% EtOAc in hexanes; visualized with ceric ammonium molybdate). Diagnostic

peaks for ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.24 (m, 5H), 5.78–5.74 (m, 1H), 4.61 (d, *J* = 12.6 Hz, 1H), 4.55 (d, *J* = 12.6 Hz, 1H), 4.44 (d, *J* = 9.0 Hz, 1H), 4.37 (s, 1H), 3.97 (d, *J* = 9.8 Hz, 1H), 3.80 (d, *J* = 9.8 Hz, 1H), 3.77 (s, 3H), 2.58 (d, *J* = 9.0 Hz, 1H), 2.12–1.99 (m, 2H), 1.74–1.70 (m, 2H), 1.58–1.56 (m, 1H), 1.54 (s, 3H), 1.5–1.43 (m, 2H), 1.42 (s, 3H), 1.25–1.19 (m, 1H), 1.11–1.01 (m, 1H), 0.98 (s, 3H), 0.95 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 172.59, 155.05, 137.89, 128.42, 127.76, 127.72, 126.03, 110.24, 80.41, 73.71, 71.94, 65.50, 59.97, 52.69, 41.51, 35.53, 33.29, 32.93, 28.76, 27.61, 25.46, 21.45, 20.15, 18.15; IR (thin film) 3527, 2989, 2926, 2848, 1741, 1454, 1380 cm^{-1}; $[\alpha]^{25}_D$: –4.85 (c = 1.5, CH_2Cl_2); HRMS (ESI) calculated for $C_{28}H_{40}O_6NH_4$ ($M+NH_4$) 490.3169, observed 490.3165.



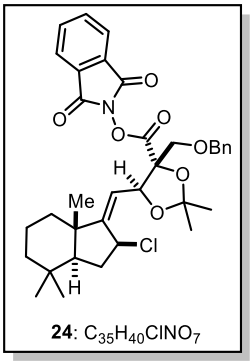
23: $C_{35}H_{41}NO_8$

(–)-1,3-Dioxoisoindolin-2-yl (4*S*,5*R*)-4-((benzyloxy)methyl)-5-((*R*)-hydroxy((3a*S*,7a*S*)-3a,7,7-trimethyl-3a,4,5,6,7,7a-hexahydro-1H-inden-3-yl)methyl)-2,2-dimethyl-1,3-dioxolane-4-carboxylate (23): Alcohol **22** (0.850 g, 1.80 mmol) was dissolved in a mixture of MeOH (10 mL) and H_2O (10 mL). Potassium hydroxide pellets (0.807 g, 14.4 mmol) were then added, and the reaction was warmed to 50 ºC. After 3 h, TLC analysis confirmed starting material was consumed; and the reaction was cooled to 23 ºC. Aqueous HCl (18 mL of 1 M soln) was added to the flask, and the heterogeneous mixture was extracted with EtOAc (5 x 15 mL). The combined organic layers were washed with brine (1 x 20 mL), dried over $MgSO_4$, filtered, and concentrated *in vacuo* to provide the crude acid as a clear oil which was carried forward without further purification.

The crude acid was dissolved in THF (20 mL) to which *N*-hydroxyphthalimide (0.881 g, 5.40 mmol), DCC (0.483 g, 2.34 mmol), and DMAP (11 mg, 0.090 mmol) were added. The

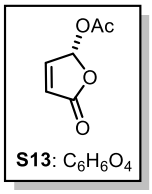
reaction was maintained for 3 h at 23 ºC, at which point Celite (~2 g) was added. The reaction mixture was concentrated *in vacuo*, and the resulting residue was purified by flash column chromatography using pH 7 silica gel (10% EtOAc in hexanes to 20% EtOAc in hexanes) to provide *N*-acyloxyphthalimide **23** as a colorless solid. Recrystallization from acetone/hexanes afforded *N*-acyloxyphthalimide **23** (0.750 g, 1.24 mmol, 69%) as colorless needles. R_f 0.25 (20% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91–7.88 (2H, m), 7.81–7.78 (2H, m), 7.42–7.26 (m, 5H), 5.80 (s, 1H), 4.75 (d, *J* = 12.4 Hz, 1H), 4.71 (d, *J* = 12.4 Hz, 1H), 4.69 (s, 1H), 4.49 (d, *J* = 9.7 Hz, 1H), 4.14 (d, *J* = 9.9 Hz, 1H), 3.97 (d, *J* = 10.1 Hz, 1H), 2.47 (d, *J* = 9.6 Hz, 1H), 2.11–2.00 (m, 2H), 1.74–1.64 (m, 2H), 1.59 (s, 3H), 1.56-1.51 (m, 2H), 1.54 (s, 3H), 1.43 (app d, *J* = 13.3 Hz, 1H), 1.27 (app td, *J* = 12.5, 3.7 Hz, 1H), 1.13 (app td, *J* = 13.5, 4.3 Hz, 1H), 0.98 (s, 3H), 0.95 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 169.29, 161.58, 154.82, 137.69, 134.94, 129.11, 128.46, 128.03, 127.75, 126.32, 124.17, 111.30, 85.13, 80.61, 74.20, 71.82, 65.56, 59.87, 47.34, 41.49, 35.42, 33.28, 32.93, 28.79, 27.39, 25.05, 21.48, 20.13, 18.18; IR (thin film) 3524, 2989, 2928, 2862, 1813, 1788, 1747, 1454, 1373 cm^{-1}; $[\alpha]^{25}_D$: –7.60 (c = 1.6, CH_2Cl_2); HRMS (ESI) calculated for $C_{35}H_{41}NO_8Na$ (M+Na) 626.2730, observed 626.2712; mp 139–141 ºC.

(+)-1,3-dioxoisoindolin-2-yl (4*R*,5*S*)-4-((benzyloxy)methyl)-5-(((2*S*,3a*S*,7a*S*,*Z*)-2-chloro-4,4,7a-trimethyloctahydro-1H-inden-1-ylidene)methyl)-2,2-dimethyl-1,3-dioxolane-4-carboxylate (24): *N*-acyloxyphthalimide **23** (0.223 g, 0.369 mmol) was dissolved in a 10:1 mixture of Et_2O/pyridine (3.5 mL) and cooled to –45 ºC. A solution of $SOCl_2$ (54 µL, 0.74 mmol) in a 10:1 mixture of Et_2O/pyridine (0.5 mL) was then added dropwise to the reaction over 5

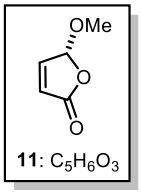


24: $C_{35}H_{40}ClNO_7$

min. The reaction was maintained at –45 ºC until full conversion of starting material was observed by TLC analysis (~45 min). Saturated aq. $NaHCO_3$ solution (2 mL) was added, and the reaction was allowed to warm to 23 ºC. The mixture was then diluted with H_2O (2 mL) and washed with EtOAc (3 x 3 mL). The combined organic layers were washed with brine (1 x 2 mL), dried over $MgSO_4$, filtered, and concentrated *in vacuo* onto Celite (~1 g). Purification by flash column chromatography using pH 7 silica gel (5% EtOAc in hexanes to 11% EtOAc in hexanes) provided allylic chloride **24** as a colorless solid. Recrystallization from acetone/hexanes afforded allylic chloride **24** (0.143 g, 0.229 mmol, 62%) as colorless needles. R_f 0.40 (20% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95-7.88 (m, 2H), 7.83-7.78 (m, 2H), 7.40–7.26 (m, 5H), 5.59 (d, *J* = 9.6 Hz, 1H), 5.18 (d, *J* = 9.6 Hz, 1H), 4.96 (app t, *J* = 7.6 Hz, 1H), 4.70 (d, *J* = 12.3 Hz, 1H), 4.64 (d, *J* = 12.3 Hz, 1H), 3.78 (d, *J* = 10.0 Hz, 1H), 3.71 (d, *J* = 10.0 Hz, 1H), 2.32 (app quint, *J* = 6.4 Hz, 1H), 1.84 (td, *J* = 13.7 Hz, 7.5 Hz, 1H), 1.74 (app d, *J* = 12.6 H, 1H), 1.67–1.59 (m, 1H), 1.59 (s, 6H), 1.55–1.48 (m, 1H), 1.41 (app d, *J* = 13.6 Hz, 1H), 1.13 (s, 3H), 0.99–0.84 (m, 3H), 0.87 (s, 3H), 0.77 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 169.07, 161.68, 161.30, 137.64, 134.99, 129.09, 128.45, 127.95, 127.80, 124.25, 114.32, 111.61, 84.96, 76.44, 74.04, 71.07, 54.60, 54.10, 45.10, 41.19, 37.03, 34.08, 32.24, 32.81, 27.55, 24.78, 21.25, 21.11, 19.49; IR (thin film) 2986, 2928, 2866, 2350, 2336, 1813, 1787, 1747, 1459 cm^{-1}; $[\alpha]^{25}_D$: +83.2 (c = 1.8, CH_2Cl_2); HRMS (ESI) calculated for $C_{35}H_{40}ClNO_7Na$ (M+Na) 644.2391, observed 644.2383; mp 154–158 ºC.

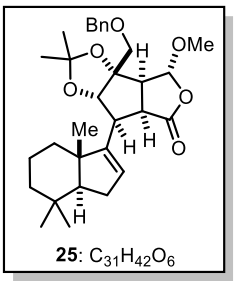


(–)-(*R*)-5-acetoxyfuran-2(5H)-one (S13): The procedure for preparation of acetoxy butenolide **S13** was a slight modification from the literature procedure.[10] 5-Hydroxyfuran-2(5H)-one[3] (2.90 g, 28.9 mmol) was dissolved in vinyl acetate (30 mL). Amano lipase AK (2.00 g) was then added, and the suspension was stirred for 8 days at 23 ºC. The suspension was then filtered through Celite, and the filtrate was concentrated *in vacuo.* Purification of the residue by flash column chromatography (40% EtOAc in hexanes) provided (*R*)-5-acetoxyfuran-2(5H)-one **S13** (3.58 g, 25.3 mmol, 87% yield) as a yellow oil. R_f 0.35 (40% EtOAc in hexanes; visualized with $KMnO_4$). Spectral data were consistent with reported values.[10] The enantiomeric excess was determined to be 92% *ee* by known methods.[10]



(–)-(*R*)-5-methoxyfuran-2(5H)-one (11): Acetoxy butenolide **S13** (1.23 g, 8.65 mmol) was dissolved in MeOH (35 mL), and $Pd(PPh_3)_4$ (0.500 g, 0.433 mmol) was added to the solution. The solution, which turned a deep red, was maintained at 23 ºC for 50 min. Upon TLC analysis confirming consumption of starting material (TLC, 10% acetone in hexanes and running the TLC plate 3x), the reaction solution was directly filtered through a silica gel plug (250 mL of 40% acetone in hexanes). The eluent was concentrated *in vacuo*, and the residue was distilled (0.8 torr, 110 ºC) to provide methoxy butenolide **11** and a trace amount of AcOH. Removal of AcOH upon further concentration *in vacuo* afforded methoxy butenolide **11** (0.705 g, 6.18 mmol, 71% yield) as a clear oil. Spectral data were consistent with reported values.[11] HLPC analysis was used to determine the enantiomeric ratio to be 92:8 (Chiracel AS column; flow: 2.0 mL/min, 10% isopropanol:*n*-hexane; $\lambda = 210$ nm; major enantiomer $t_R = 8.70$ min, minor enantiomer $t_R = 11.60$ min); $[\alpha]^{25}_D$: –124 (c = 1.2, CH_2Cl_2).

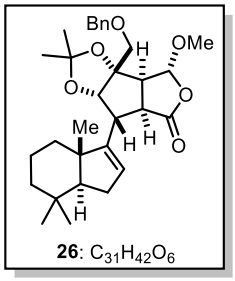
(–)-(3a*S*,3b*S*,4*R*,6a*S*,7a*S*)-3a-((benzyloxy)methyl)-4-methoxy-2,2-dimethyl-7-((3a*S*,7a*S*)-



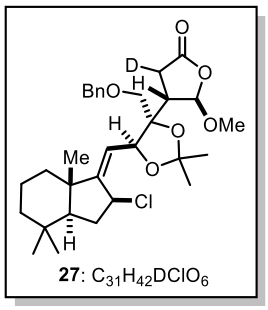
3a,7,7-trimethyl-3a,4,5,6,7,7a-hexahydro-1H-inden-3-yl)hexahydro-6H-furo[3',4':3,4]cyclopenta[1,2-d][1,3]dioxol-6-one (25): Allylic chloride **24** (70 mg, 0.11 mmol), methoxy butenolide **11** (51 mg, 0.45 mmol), D_2-Hantzsch ester (43 mg, 0.17 mmol), and $[Ru(bpy)_3](PF_6)_2$ (1 mg, 0.001 mmol) were charged into a vial. Acetonitrile (1.1 mL) was added, and the solution was sparged with Ar. The vial was then vigorously stirred while being irradiated by a single strip of blue LED lights (450 nm) at 23 ºC. After 6 h, the reaction mixture was concentrated *in vacuo*, and the residue was dissolved in EtOAc (1 mL) and washed with aq. HCl (4 x 2 mL of 4 M soln) followed by H_2O (2 x 2 mL). The organic layer was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. ^{1}H NMR analysis of the crude residue using an internal standard (dimethoxybenzene) showed 28% yield of **25**, 37% yield of **26**, 8% yield of **27**, and 13% yield of **28**. Purification of the crude residue by flash column chromatography (0% acetone in hexanes to 5% acetone in hexanes) provided **25** (15 mg, 0.030 mmol, 27%) as a clear oil. R_f for **25**: 0.55 (20% acetone in hexanes; visualized with ceric ammonium molybdate). Flash column chromatography under separate conditions of the remaining mixed fractions from the first purification (4% EtOAc in hexanes to 10% EtOAc in hexanes) provided epimeric product **26** (20 mg, 0.039 mmol, 35%) as a clear oil. R_f for **26**: 0.45 (20% acetone in hexanes; visualized with ceric ammonium molybdate).

Desired ACF product **25** for ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.28 (m, 5H), 5.48 (app s, 1H), 5.38 (app s, 1H), 4.62 (d, *J* = 12.0 Hz, 1H), 4.59 (d, *J* = 12.1 Hz, 1H), 4.41 (d, *J* = 7.5 Hz, 1H), 3.64 (d, *J* = 10.5 Hz, 1H), 3.50 (d, *J* = 10.3 Hz, 1H), 3.43 (app t, *J* = 8.7 Hz, 1H), 3.38 (s, 3H), 3.07 (app d, *J* = 8.7 Hz, 1H), 3.00 (app t, *J* = 8.2 Hz, 1H), 2.10 (ddd, *J* = 14.9, 6.3, 3.0 Hz, 1H), 2.02 (app t, *J* = 13.3 Hz, 1H), 1.76 (dd, *J* = 11.7, 6.3 Hz, 1H), 1.60–1.50 (m, 2H), 1.58 (s,

3H), 1.50 (s, 3H), 1.45–1.39 (m, 1H), 1.19 (td, *J* = 13.2, 3.4 Hz, 1H), 0.95 (s, 3H), 0.92–0.82 (m, 2H), 0.89 (s, 3H), 0.87 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 174.65, 150.69, 137.18, 128.71, 128.38, 128.30, 124.70, 113.17, 103.64, 89.95, 86.77, 73.97, 70.81, 58.24, 56.76, 55.12, 47.84, 45.94, 43.72, 41.46, 34.73, 33.05, 32.90, 20.22, 29.38, 29.16, 21.45, 20.18, 17.70; $[\alpha]^{25}_D$: –84.9 (c = 1.0, CH_2Cl_2); IR (thin film) 2993, 2934, 2862, 1785, 1636, 1455, 1371, 1234, 1215 cm^{-1}; HRMS (ESI) calculated for $C_{31}H_{42}O_6NH_4$ (M+NH_4) 528.3325, observed 528.3331.

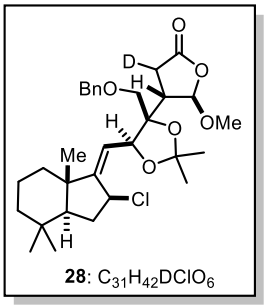


Epimer **(–)-26** for ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.29 (m, 5H), 5.75 (app s, 1H), 5.53 (d, *J* = 4.8 Hz, 1H), 4.57 (d, *J* = 12.3 Hz, 1H), 4.55 (d, *J* = 12.3 Hz, 1H), 4.47 (d, *J* = 3.7 Hz, 1H), 3.82 (d, *J* = 9.6 Hz, 1H), 3.61 (d, *J* = 9.7 Hz, 1H), 3.50 (app t, *J* = 10.8 Hz, 1H), 3.41 (s, 3H), 2.88 (dd, *J* = 9.7, 4.8 Hz, 1H), 2.72 (app d, *J* = 11.3 Hz, 1H), 2.13–2.06 (m, 2H), 1.76–1.65 (m, 1H), 1.61–1.52 (m, 2H), 1.43 (s, 3H), 1.36–1.27 (m, 1H), 1.28 (s, 3H), 1.15 (td, *J* = 13.6, 4.1 Hz, 1H), 0.96 (s, 3H), 0.92–0.81 (m, 2H), 0.89 (s, 3H), 0.83 (s, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 175.80, 148.52, 137.12, 128.69, 128.26, 128.05, 127.25, 111.58, 105.02, 89.60, 87.36, 73.83, 72.05, 59.28, 58.09, 57.57, 47.71, 47.54, 45.77, 41.61, 35.68, 33.09, 32.97, 29.17, 27.80, 26.00, 21.47, 20.22, 17.36; $[\alpha]^{25}_D$: –88.2 (c = 2.0, CH_2Cl_2); IR (thin film) 2988, 2929, 2861, 1775, 1454, 1373, 1246 cm^{-1}; HRMS (ESI) calculated for $C_{31}H_{42}O_6Na$ (M+Na) 533.2879, observed 533.2897.

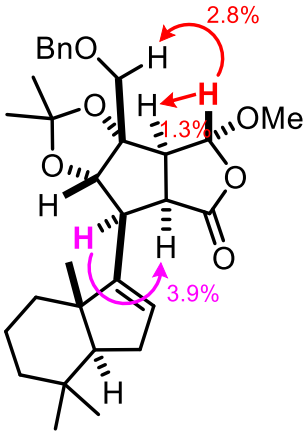


An analytical sample of clean product **(+)-27** was obtained from flash column chromatography (0% acetone in hexanes to 4% acetone in hexanes). R_f: 0.60 (20% acetone in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36–7.27 (m, 5H), 5.61 (s, 1H),

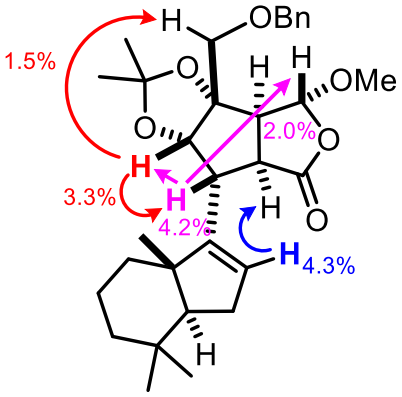
5.18 (d, J = 9.6, 1H), 5.09 (dd, J = 9.6, 1.6 Hz, 1H), 4.57 (td, J = 8.0 Hz, 1.6 Hz, 1H), 4.46 (d, J = 10.4 Hz, 1H), 4.41 (d. J = 10.4 Hz, 1H), 3.63 (d, J = 9.6 Hz, 1H), 3.53 (d, J = 9.6 Hz, 1H), 3.51 (s, 3H), 2.68 (d, J = 2.1 Hz, 1H), 2.50 (d, J = 2.1 Hz, 1H), 1.93–1.86 (m, 1H), 1.76–1.51 (m, 4H), 1.48 (s, 3H), 1.42 (s, 3H), 1.09 (s, 3H), 1.04–0.95 (m, 2H), 0.91–0.83 (m, 1H), 0.85 (s, 3H), 0.74 (s, 3H), 0.60 (dd, J = 14.4, 6.0 Hz, 1H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 176.81, 159.67, 137.02, 128.73, 128.34, 128.26, 115.56, 109.03, 106.81, 82.60, 78.24, 74.46, 74.39, 56.90, 54.57, 54.53, 45.17, 44.93, 41.17, 36.72, 33.93, 33.15, 32.96, 29.85, 27.58, 26.19, 21.49, 21.15, 19.49; $[\alpha]^{25}_{D}$: +109.1 (c = 0.57, CH_2Cl_2); IR (thin film) 2986, 2931, 2864, 2359, 2342, 1787, 1455, 1370, 1252 cm^{-1}; HRMS (ESI) calculated for $C_{31}H_{42}DClO_6Na$ (M+Na) 570.2709, observed 570.2702.



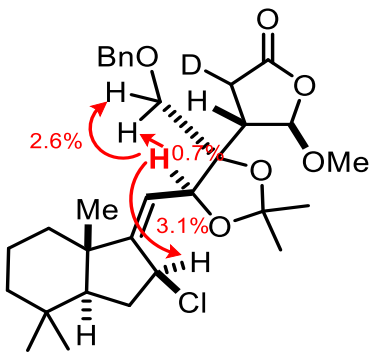
Diagnostic peaks of addition product **28** for ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.43 (d, J = 2.9 Hz, 1H), 5.21 (d, J = 9.6, 1.7 Hz, 1H), 4.75 (d, J = 9.6 Hz, 1H), 4.59 (app t, J = 7.0 Hz, 1H).



desired product (**25**)

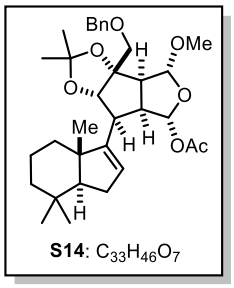


epimeric product (**26**)



prematurely quenched product (**27**)

(–)-(3a*S*,3b*S*,4*R*,6*R*,6a*S*,7*S*,7a*S*)-3a-((benzyloxy)methyl)-4-methoxy-2,2-dimethyl-7-((3a*S*,7a*S*)-3a,7,7-trimethyl-3a,4,5,6,7,7a-hexahydro-1H-inden-3-yl)hexahydro-4H-furo[3',4':3,4]cyclopenta[1,2-d][1,3]dioxol-6-yl acetate (S14): Product **25** (40 mg, 0.078 mmol)

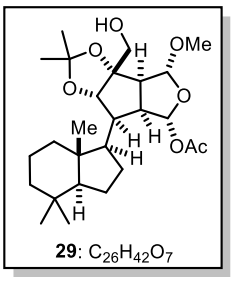


S14: $C_{33}H_{46}O_7$

was charged into a flask with toluene (1.4 mL) and then cooled to –78 ºC. A solution of DIBAL-H (18 μL, 0.10 mmol) in toluene (0.2 mL) was added dropwise to the reaction vessel, keeping the temperature near –78 ºC. After 45 min, TLC analysis showed some remaining starting material, and an additional solution of DIBAL-H (5 μL, 0.03 mmol) in toluene (0.05 mL) was added. After 45 min, a solution of DMAP (19 mg, 0.16 mmol), pyridine (20 μL, 0.23 mmol), and CH_2Cl_2 (0.2 mL) was added, followed by Ac_2O (44 μL, 0.47 mmol). The reaction was maintained at –78 ºC for 12 h, at which point it was allowed to warm to 23 ºC. An aqueous solution saturated with Rochelle's salt (3 mL) was added, and the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were washed with brine (1 x 5 mL), dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification of the residue by flash column chromatography (6% EtOAc in hexanes to 10% EtOAc in hexanes) provided a single diastereomer, diacetal **S14** (36 mg, 0.065 mmol, 83%), as a colorless oil. R_f 0.35 (20% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.27 (m, 5H), 5.92 (d, J = 4.1 Hz, 1H), 5.60 (app s, 1H), 5.17 (s, 1H), 4.61 (d, J = 12.3 Hz, 1H), 4.58 (d, J = 12.3 Hz, 1H), 4.30 (d, J = 8.8 Hz, 1H), 3.60 (d, J = 10.4 Hz, 1H), 3.54 (d, J = 10.5 Hz, 1H), 3.27 (s, 3H), 3.20 (app td, J = 7.8, 4.0 Hz, 1H), 3.03 (d, J = 8.0 Hz, 1H), 2.92 (app t, J = 8.1 Hz, 1H), 2.05 (s, 3H), 2.06–2.00 (m, 2H), 1.72–1.51 (m, 2H), 1.51 (s, 3H), 1.46 (app d, J = 13.6 Hz, 1H), 1.35 (s, 3H), 1.28–1.24 (m, 1H), 1.20 (td, J = 12.7, 3.9 Hz, 1H), 0.98 (td, J = 13.7, 4.4 Hz, 1H), 0.93 (s, 3H), 0.90–0.85 (m, 1H), 0.84 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 170.46, 151.77, 137.54, 128.61, 128.36, 128.13, 125.03, 113.25, 107.80, 99.65, 90.22, 85.91,

73.84, 70.54, 50.88, 47.64, 43.33, 42.11, 36.75, 35.58, 33.29, 32.95, 31.72, 30.64, 29.72, 29.01, 24.81, 22.79, 21.25, 21.17, 20.09, 17.24, 14.27; $[\alpha]^{25}_{D}$: –46.3 (c = 2.1, CH_2Cl_2); IR (thin film) 2991, 2930, 2861, 1748, 1455, 1367 cm^{-1}; HRMS (ESI) calculated for $C_{33}H_{46}O_7Na$ (M+Na) 577.3141, observed 577.3127.

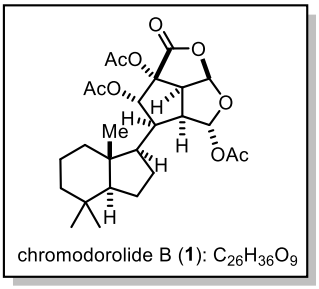
(–)-(3a*S*,3b*S*,4*R*,6*R*,6a*S*,7*R*,7a*S*)-3a-(hydroxymethyl)-4-methoxy-2,2-dimethyl-7-((1*R*,3a*S*,7a*R*)-4,4,7a-trimethyloctahydro-1H-inden-1-yl)hexahydro-4H-furo[3',4':3,4]cyclopenta[1,2-d][1,3]dioxol-6-yl acetate (29): Diacetal **S14** (28 mg, 0.050 mmol) and 10% Pd/C (28 mg) were charged into a flask with MeOH (1.0 mL). The reaction vessel was then evacuated and refilled with Ar (3x). Formic acid (50 μL) was then added dropwise to the vigorously stirring suspension. After 2 h, TLC analysis showed full consumption of starting material. The reaction mixture was diluted with MeOH (1 mL), filtered through Celite, and concentrated *in vacuo* to provide the crude alcohol, which was carried forward to the subsequent step.



29: $C_{26}H_{42}O_7$

To a flask containing the crude alcohol (0.050 mmol) was added PtO_2 (12 mg, 0.050 mmol) and EtOAc (1.0 mL). The reaction vessel was then evacuated and refilled with H_2 (3x, 1 atm H_2). The reaction was maintained under 1 atm of H_2 for 12 h at 23 ºC, at which point the reaction vessel was refilled first with Ar and then air. Filtration of the suspension through Celite, concentration of the filtrate *in vacuo*, and purification of the residue by flash column chromatography (30% EtOAc in hexanes) provided alcohol **29** (20 mg, 0.043 mmol, 86%) as a colorless oil. R_f 0.25 (30% EtOAc in hexanes; visualized with ceric ammonium molybdate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.13 (d, *J* = 3.5 Hz, 1H), 5.31 (s, 1H), 3.86 (d, *J* = 9.6 Hz, 1H), 3.65 (bs, 2H), 3.31 (s, 3H), 3.19 (app td, *J* = 7.4, 3.6 Hz, 1H), 2.87 (app d, *J* = 7.6 Hz, 1H), 2.30 (app dt, *J* = 10.1, 7.3 Hz, 1H), 2.16 (bs, 1H),

2.05 (s, 3H), 1.80–1.66 (m, 2H), 1.63–1.56 (m, 2H), 1.53 (s, 3H), 1.43 (s, 6H), 1.36–1.28 (m, 1H), 1.11–0.93 (m, 2H), 0.90–0.86 (m, 1H), 0.85 (s, 3H), 0.83 (s, 3H), 0.76 (s, 3H), 0.76–0.69 (m, 1H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 170.84, 112.73, 106.84, 97.67, 90.98, 88.07, 63.62, 57.74, 56.04, 54.85, 52.29, 50.68, 44.98, 42.90, 41.39, 40.00, 33.60, 33.26, 30.71, 30.49, 29.84, 25.75, 21.20, 20.99, 20.94, 20.22, 13.86; $[\alpha]^{25}_D$: –17.6 (c = 1.7, CH_2Cl_2); IR (thin film) 3490, 2951, 2931, 2873, 1745, 1459, 1368 cm^{-1}; HRMS (ESI) calculated for $C_{26}H_{42}O_7Na$ (M+Na) 489.2828, observed 489.2813.



chromodorolide B (**1**): $C_{26}H_{36}O_9$

(–)-Chromodorolide B (1): Alcohol **29** (9.0 mg, 0.019 mmol) and Dess-Martin periodinane (12 mg, 0.029 mmol) were charged into a flask with CH_2Cl_2 (0.3 mL). The reaction mixture was maintained at 23 ºC for 5 h, at which point it was diluted with hexanes (0.5 mL), filtered through Celite, and concentrated *in vacuo*. The residue was dissolved in hexanes (1 mL) and filtered through Celite. The filtrate was then concentrated *in vacuo* to afford the crude aldehyde which was carried forward into the next step.

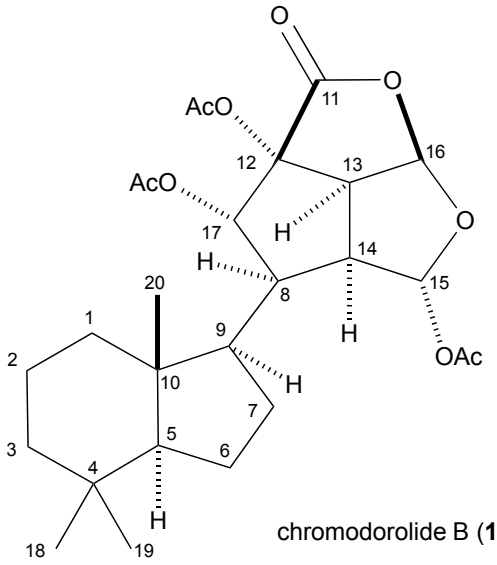
To a solution of crude aldehyde in THF (0.1 mL) was added *t*-BuOH (0.1 mL), H_2O (0.1 mL), 2-methyl-2-butene (50 μL), NaH_2PO_4 (25 mg, 0.21 mmol), and $NaClO_2$ (14 mg, 0.15 mmol). The reaction was maintained at 23ºC for 12 h and then diluted with H_2O (1 mL). The solution was washed with EtOAc (3 x 1 mL); and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo* to provide crude carboxylic acid **30**.

This crude acid **30** was then dissolved in a solution of THF (0.3 mL) and aq. HCl (0.3 mL of 4 M soln), which was maintained at 23 ºC for 72 h. The reaction was then diluted with H_2O (1 mL), and the solution was washed with EtOAc (3 x 1 mL). The combined organic layers were

washed with brine (1 x 1 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo* to afford crude lactol **31**.

Crude lactol **31** was then dissolved in CH_2Cl_2 (0.3 mL). Next, DMAP (2 mg, 0.019 mmol) and pyridine (31 μL, 0.38 mmol) were added, followed by Ac_2O (28 μL, 0.29 mmol). The reaction was maintained at 23 ºC for 24 h, at which point it was diluted with H_2O (2 mL), and the heterogeneous solution was washed with EtOAc (3 x 2 mL). The combined organic layers were washed with brine (1 x 3 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Purification by flash column chromatography (20% EtOAc in hexanes to 30% EtOAc in hexanes) provided **1** (4.7 mg, 0.010 mmol, 49% over 4 steps) as a colorless solid. Recrystallization of the solid from acetone/hexanes afforded colorless needles. The NMR data matched that of the isolation data.[12] $[\alpha]^{25}_D$: –66.8 (c = 0.12, CH_2Cl_2) compared to isolation sample $[\alpha]^{25}_D$: –95 (c = 0.10, CH_2Cl_2)[12]; IR (thin film) 2948, 2876, 1813, 1752, 1370, 1214, 1093, 1000, 964 cm^{-1}; HRMS (ESI) calculated for $C_{26}H_{36}O_9Na$ (M+Na) 515.2257, observed 515.2260; mp 236–238 ºC (decomp).

Table S1. Synthetic and natural (−)-Chromodorolide B (**1**) in $CDCl_3$.



chromodorolide B (**1**)

Pos.	Literature (400 MHz,$CDCl_3$) δH (multiplicity, *J* / Hz)	δC	Synthetic (500 MHz, $CDCl_3$) δH (multiplicity, *J* / Hz)	δC
1	1.03 (dt, *J* = 12.3, 3.6)	40.9	1.03 (dt, *J* = 12.3, 3.6)	41.06
1a	1.38 (m)		1.39 (m)	
2	1.55 (m, 2H)	21.1	1.56 (m, 2H)	21.27
3	0.95 (m)	39.1	0.95 (dt, *J* = 12.6, 3.6)	39.26
3a	1.51 (m)		1.52 (m)	
4		33.1		33.31
5	1.09 (dd, *J* = 13.1, 6.6)	57.0	1.10 (dd, *J* = 13.7, 3.7)	57.10
6	1.40 (m)	19.9	1.42 (m)	20.08
6a	1.56 (m)		1.57 (m)	
7	1.48 (m)	25.2	1.50 (m)	25.36
7a	1.58 (m)		1.61 (m)	
8	2.57 (ddd, *J* = 12.1, 11.6, 7.9)	48.0	2.58 (ddd, *J* = 12.2, 11.1, 7.6)	48.13
9	1.69 (bdd, *J* = 12.0, 9.9)	50.3	1.71 (q, *J* = 10.0)	50.43
10		43.9		44.00
11		169.1		169.24
12		81.4		81.48
13	3.79 (dd, *J* = 8.9, 6.1)	50.4	3.80 (dd, *J* = 9.0, 6.0)	50.56
14	2.93 (bt, *J* = 8.2)	45.6	2.94 (bt, *J* = 8.2)	45.74
15	6.50 (bs)	97.8	6.51 (bs)	97.88
16	6.08 (d, *J* = 6.1)	103.4	6.09 (d, *J* = 6.0)	103.51
17	5.30 (d, *J* = 11.6)	73.9	5.31 (d, *J* = 12.4)	74.06
18	0.79 (s, 3H)	33.4	0.79 (s, 3H)	33.55
19	0.83 (s, 3H)	21.0	0.84 (s, 3H)	21.11
20	0.84 (s, 3H)	13.7	0.85 (s, 3H)	13.82
OAc	2.04 (s, 3H)	20.8	2.05 (s, 3H)	20.96
		169.2		169.32
OAc	2.11 (s, 3H)	20.8	2.12 (s, 3H)	20.98
		170.0		170.13
OAc	2.19 (s, 3H)	20.9	2.21 (s, 3H)	21.05
		170.2		170.38

X-ray image of (–)-Chromodorolide B (CCDC1446027)

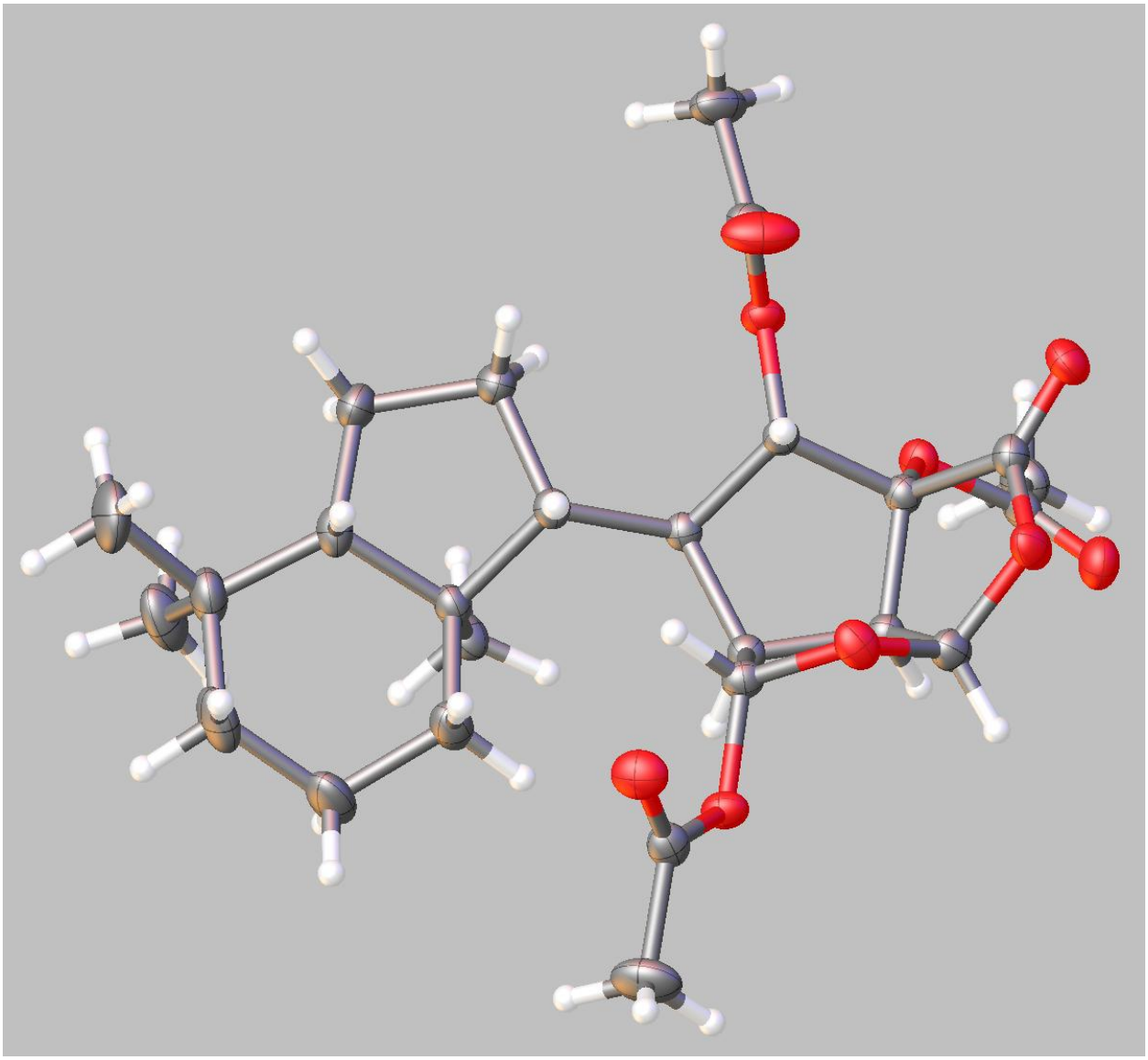
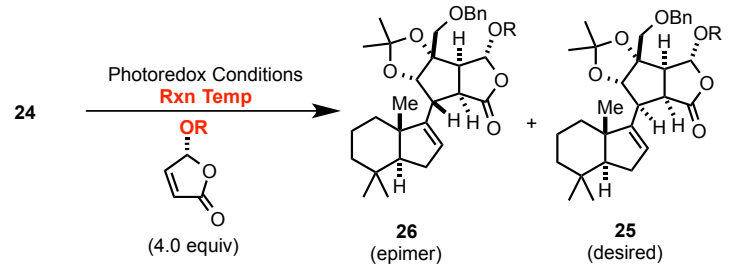
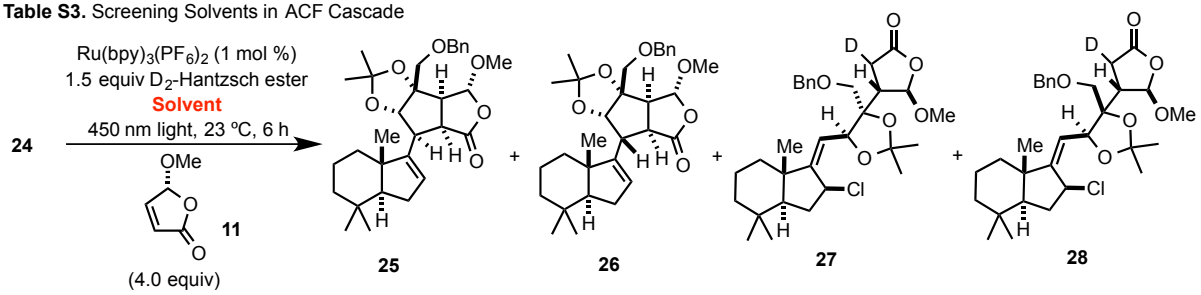


Table S2. Effects of Butenolide Substitution and Temperature on Diastereoselectivity

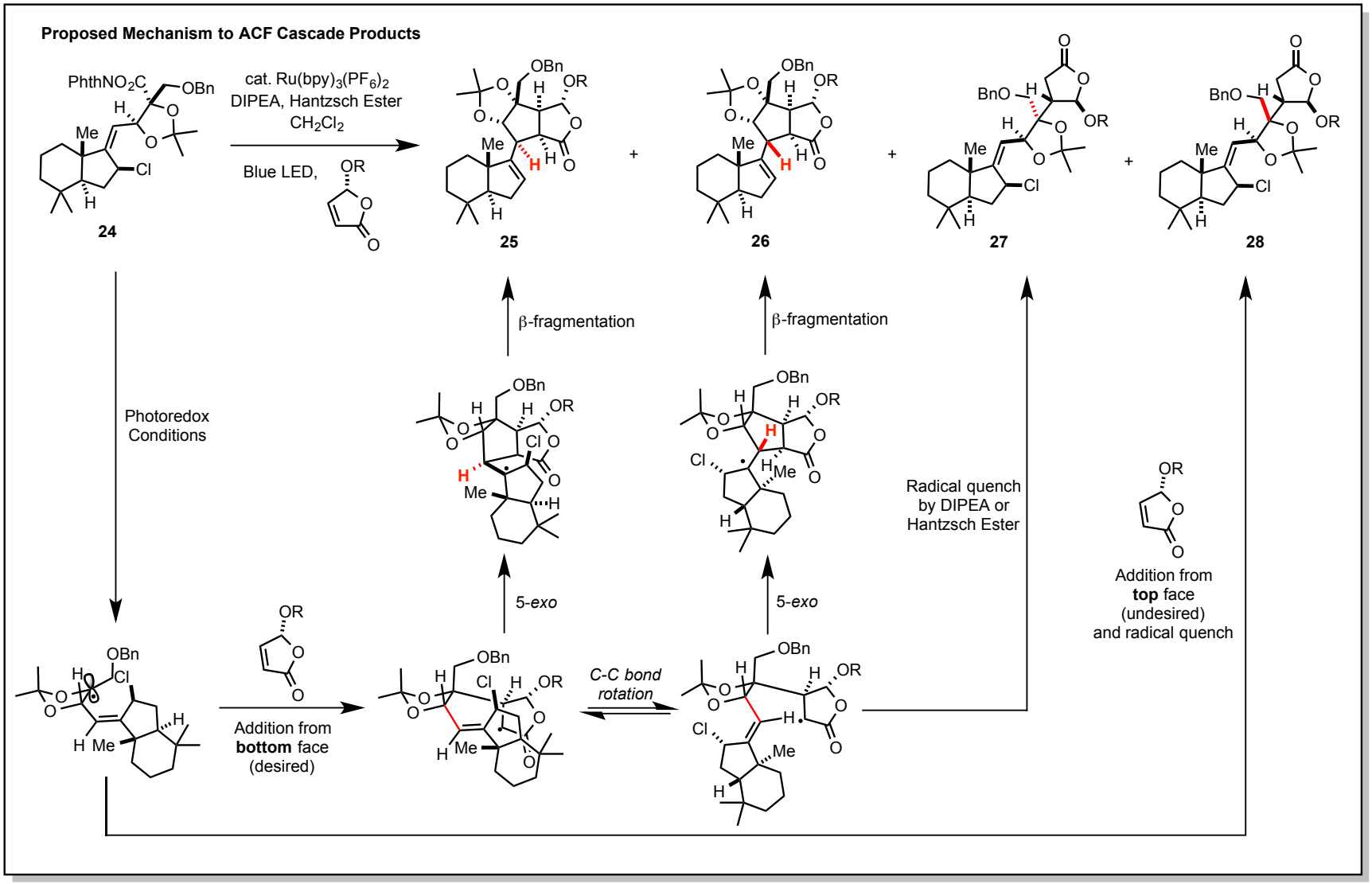


Entry	R	Rxn Temp	Diastereoselectivity (26:25)
1	Menthol	23 ºC	3.1 : 1.0
2	Ac	23 ºC	2.7 : 1.0
3	Me	23 ºC	1.8 : 1.0
4	Me	38 ºC	1.8 : 1.0
5	Me	0 ºC	1.5 : 1.0

Table S3. Screening Solvents in ACF Cascade



Entry	Solvent	Respective Yields by ^{1}H NMR (with internal standard)	dr 26:25
1	CH_2Cl_2	25% :45% : 6% : 10%	1.8 : 1
2	DME	23% : 36% : 4% : 9%	1.6 : 1
3	THF	21% : 34% : 5% : 8%	1.6 : 1
4	MeCN	28% : 37% : 8% : 13%	1.3 : 1
5	MeOH	12% : 24% : 15% : 12%	2.0 : 1
6	DMSO	17% : 20% : 17% : 15%	1.2 : 1


Proposed Mechanism to ACF Cascade Products
cat. Ru(bpy)3(PF6)2
DIPEA, Hantzsch Ester
CH2Cl2
Blue LED,
24
25
26
27
28
β-fragmentation
β-fragmentation
Photoredox Conditions
5-exo
5-exo
C-C bond rotation
Addition from bottom face (desired)
Radical quench by DIPEA or Hantzsch Ester
Addition from top face (undesired) and radical quench

References:

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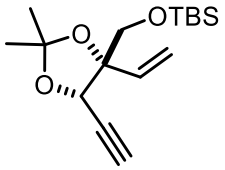
^{1}H NMR ($CDCl_3$, 500 MHz)



S6: $C_{16}H_{28}O_3Si$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PROCNO                         1

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PULPROG                   zg30
TD                       81728
SOLVENT                  CDCl3
NS                             8
DS                             2
SWH                 8012.820 Hz
FIDRES              0.098043 Hz
AQ                  5.0998273 sec
RG                             8
DW                     62.400 usec
DE                      6.00 usec
TE                      298.0 K
D1                  0.10000000 sec
MCREST     0 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
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P1                      7.50 usec
PL1                     1.60 dB
SFO1               500.2235015 MHz

F2 - Processing parameters
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WDW                           no
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GB           0
PC                        4.00
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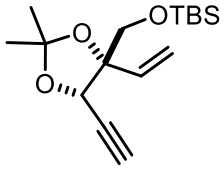
^{13}C NMR ($CDCl_3$, 126 MHz)



S6: $C_{16}H_{28}O_3Si$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PROCNO                   1

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d17           0.00019600 sec
MCREST   0 sec
MCWRK         0.01500000 sec
P2                31.00 usec

======== CHANNEL f1 ========
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P11              500.00 usec
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SP1                3.20 dB
SP2                3.20 dB
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SPOFF2   0 Hz

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PCPD2             100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
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GPNAM[2]        SINE.100
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GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1              30.00 %
GPZ2              50.00 %
p15              500.00 usec
p16             1000.00 usec

F2 - Processing parameters
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WDW                  EM
SSB       0
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GB        0
PC                2.00
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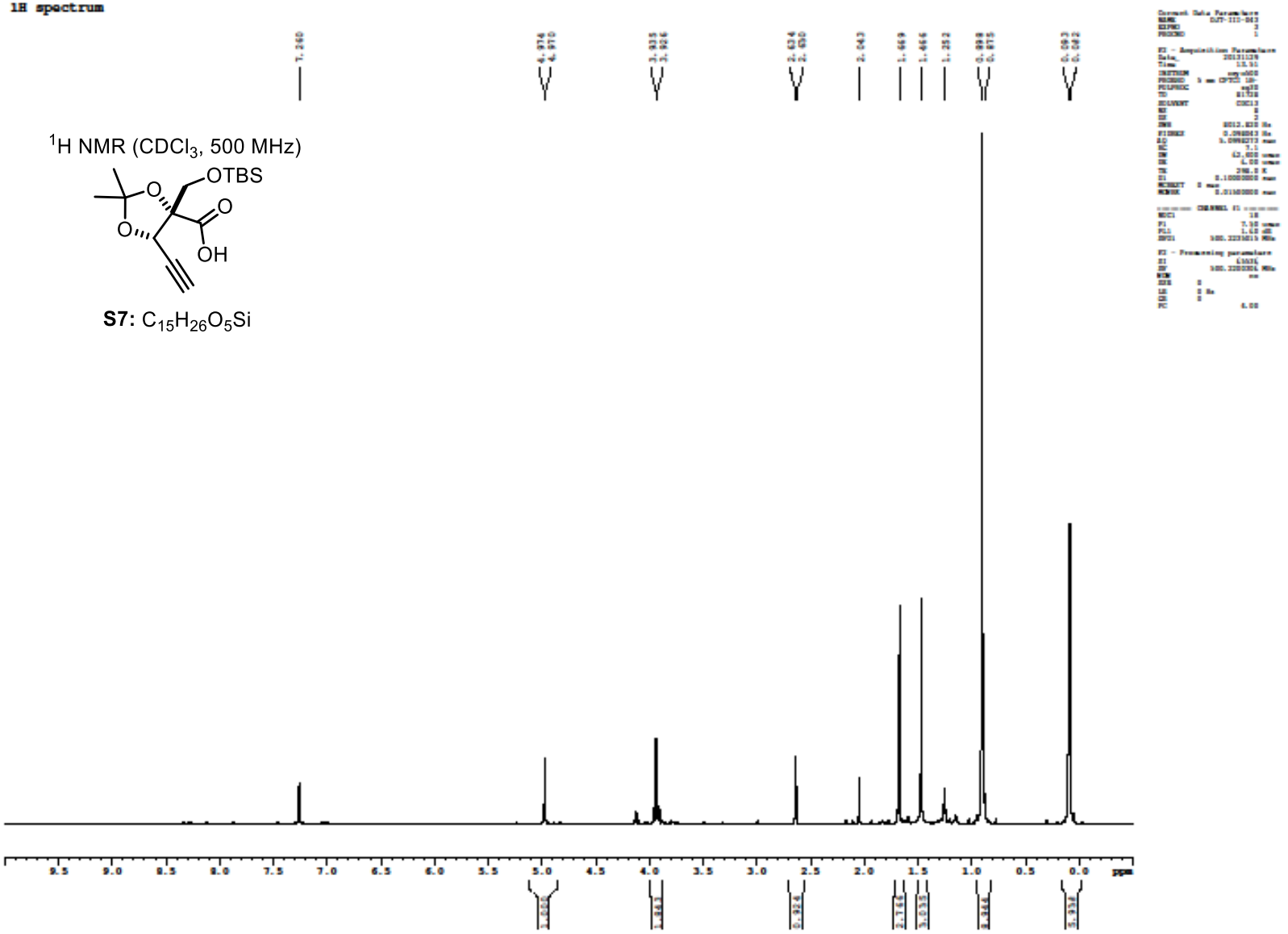
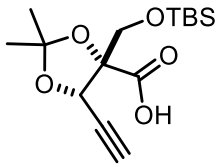
1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

S7: $C_{15}H_{26}O_5Si$

^{13}C NMR ($CDCl_3$, 126 MHz)



S7: $C_{15}H_{26}O_5Si$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PROCNO                 1

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FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                 13004
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST   0 sec
MCWRK         0.01500000 sec
P2                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
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SPNAM[2]    Crp60comp.4
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SPOFF2   0 Hz

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PCPD2             100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== GRADIENT CHANNEL =====
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GPNAM[2]         SINE.100
GPX1     0 %
GPX2     0 %
GPY1     0 %
GPY2     0 %
GPZ1              30.00 %
GPZ2              50.00 %
p15              500.00 usec
p16             1000.00 usec

F2 - Processing parameters
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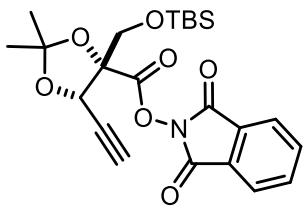
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 2.00
```

^{1}H NMR ($CDCl_3$, 500 MHz)



S2: $C_{23}H_{29}NO_7Si$

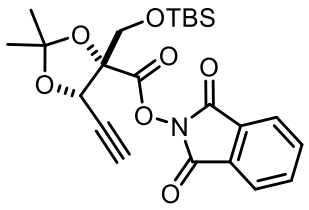
Current Data Parameters
NAME DJT-III-081
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131206
Time 13.53
INSTRUM qnn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998272 sec
RG 114
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -5.00 dB
SFO1 499.2934950 MHz

F2 - Processing parameters
SI 65536
SF 499.2900294 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR ($CDCl_3$, 126 MHz)



S2: $C_{23}H_{29}NO_7Si$


```
Current Data Parameters
NAME         DJT-III-081
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131207
Time               11.22
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   338
DS                    16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG               7298.2
DW              16.500 usec
DE               6.00 usec
TE              298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
D16          0.00020000 sec
d17          0.00019600 sec
MCREST   0 sec
MCWRK        0.01500000 sec
P2              31.00 usec

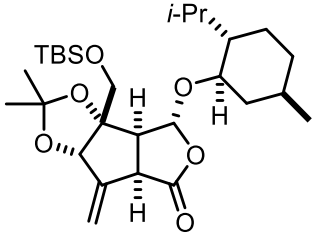
======== CHANNEL f1 ========
NUC1                 13C
P1              15.50 usec
P11            500.00 usec
P12           2000.00 usec
PL0            120.00 dB
PL1             -1.00 dB
SFO1       125.7942548 MHz
SP1              3.20 dB
SP2              3.20 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1   0 Hz
SPOFF2   0 Hz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2          100.00 usec
PL2              1.60 dB
PL12            24.60 dB
SFO2       500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPX1     0 %
GPX2     0 %
GPY1     0 %
GPY2     0 %
GPZ1            30.00 %
GPZ2            50.00 %
p15            500.00 usec
p16           1000.00 usec

F2 - Processing parameters
SI                 65536
SF         125.7804080 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                2.00
```

^{1}H NMR ($CDCl_3$, 500 MHz)



S4: $C_{28}H_{48}O_6Si$

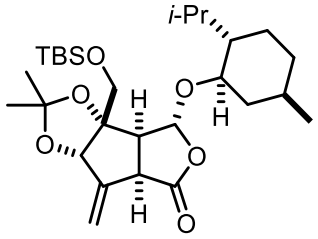

```
Current Data Parameters
NAME            DJT-III-088
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_           20131209
Time                14.43
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ              5.0998273 sec
RG                     4
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST    0 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 7.50 usec
PL1                1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF            500.2200302 MHz
WDW                   EM
SSB          0
LB                 0.30 Hz
GB           0
PC                 4.00
```

^{13}C NMR ($CDCl_3$, 126 MHz)



S4: $C_{28}H_{48}O_6Si$


```
Current Data Parameters
NAME         DJT-III-088
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131209
Time               14.48
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   143
DS                    16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                 8192
DW               16.500 usec
DE                6.00 usec
TE                298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
D16          0.00020000 sec
d17          0.00019600 sec
MCREST   0 sec
MCWRK        0.01500000 sec
P2                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1   0 Hz
SPOFF2   0 Hz

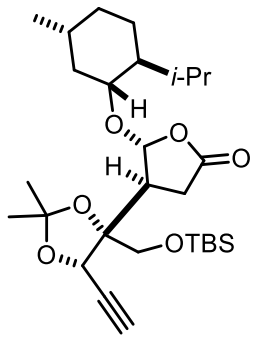
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1               30.00 %
GPZ2               50.00 %
p15               500.00 usec
p16              1000.00 usec

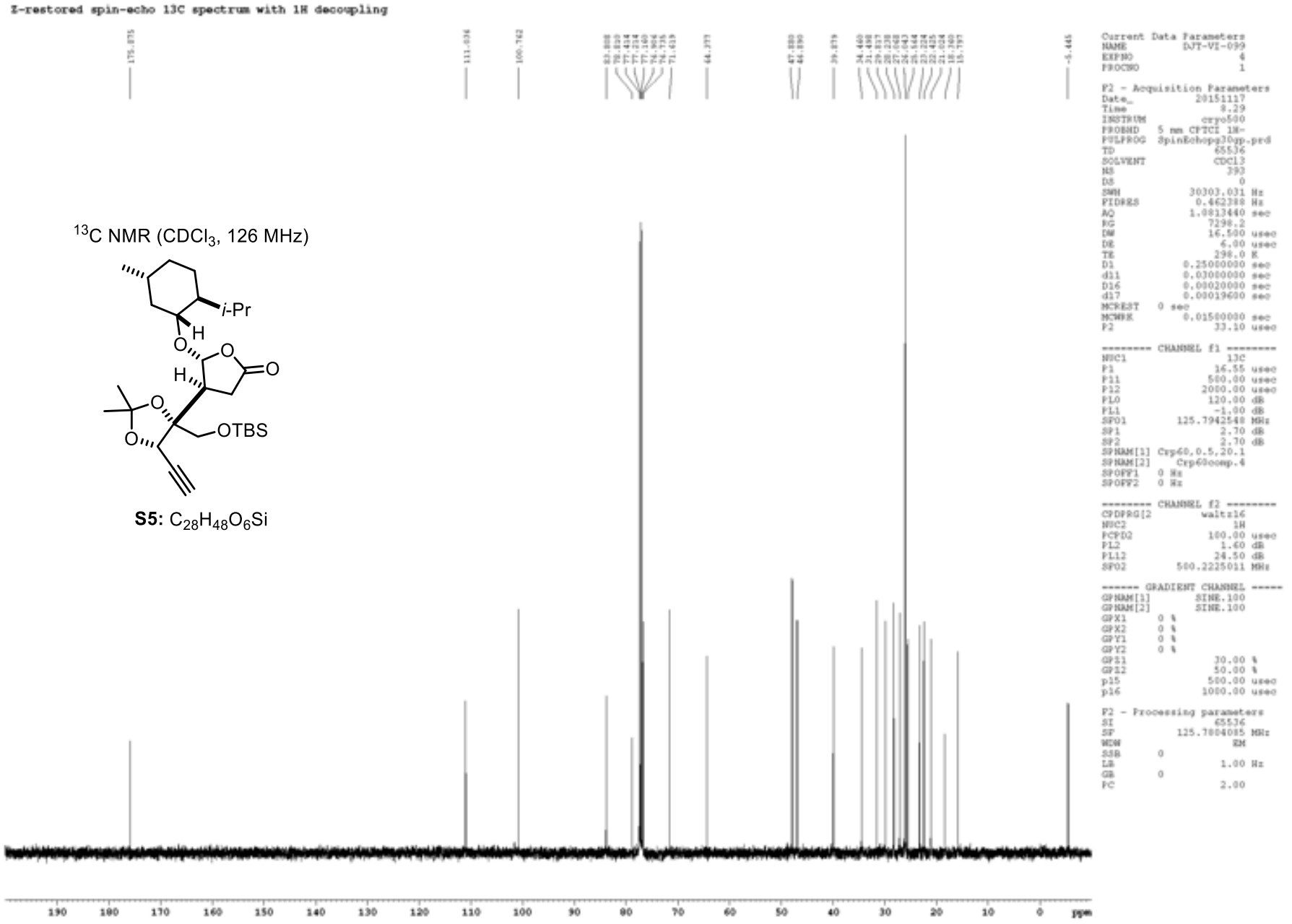
F2 - Processing parameters
SI                 65536
SF           125.7804090 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 2.00
```

1H spectrum

^{1}H NMR ($CDCl_3$, 500 MHz)

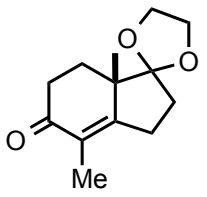


S5: $C_{28}H_{48}O_6Si$


Z-restored spin-echo 13C spectrum with 1H decoupling
175.875
111.036
100.762
83.808
78.820
77.434
77.234
77.160
76.904
76.735
71.619
64.377
47.880
41.390
39.879
34.460
31.498
29.837
28.230
27.668
26.643
25.564
23.234
22.405
21.024
18.790
15.797
-5.445
13C NMR (CDCl3, 126 MHz)
i-Pr
H
O
O
O
H
O
O
OTBS
S5: C28H48O6Si
Current Data Parameters
NAME DJT-VI-099
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 8.29
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG 3pinEchopg30pp.prd
TD 65536
SOLVENT CDCl3
NS 393
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0 sec
MCWRK 0.01500000 sec
P2 33.10 usec
-------- CHANNEL f1 --------
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2] Crp60comp.4
SPOFF1 0 Hz
SPOFF2 0 Hz
-------- CHANNEL f2 --------
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.40 dB
PL12 24.50 dB
SFO2 500.2225011 MHz
------ GRADIENT CHANNEL -----
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804085 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

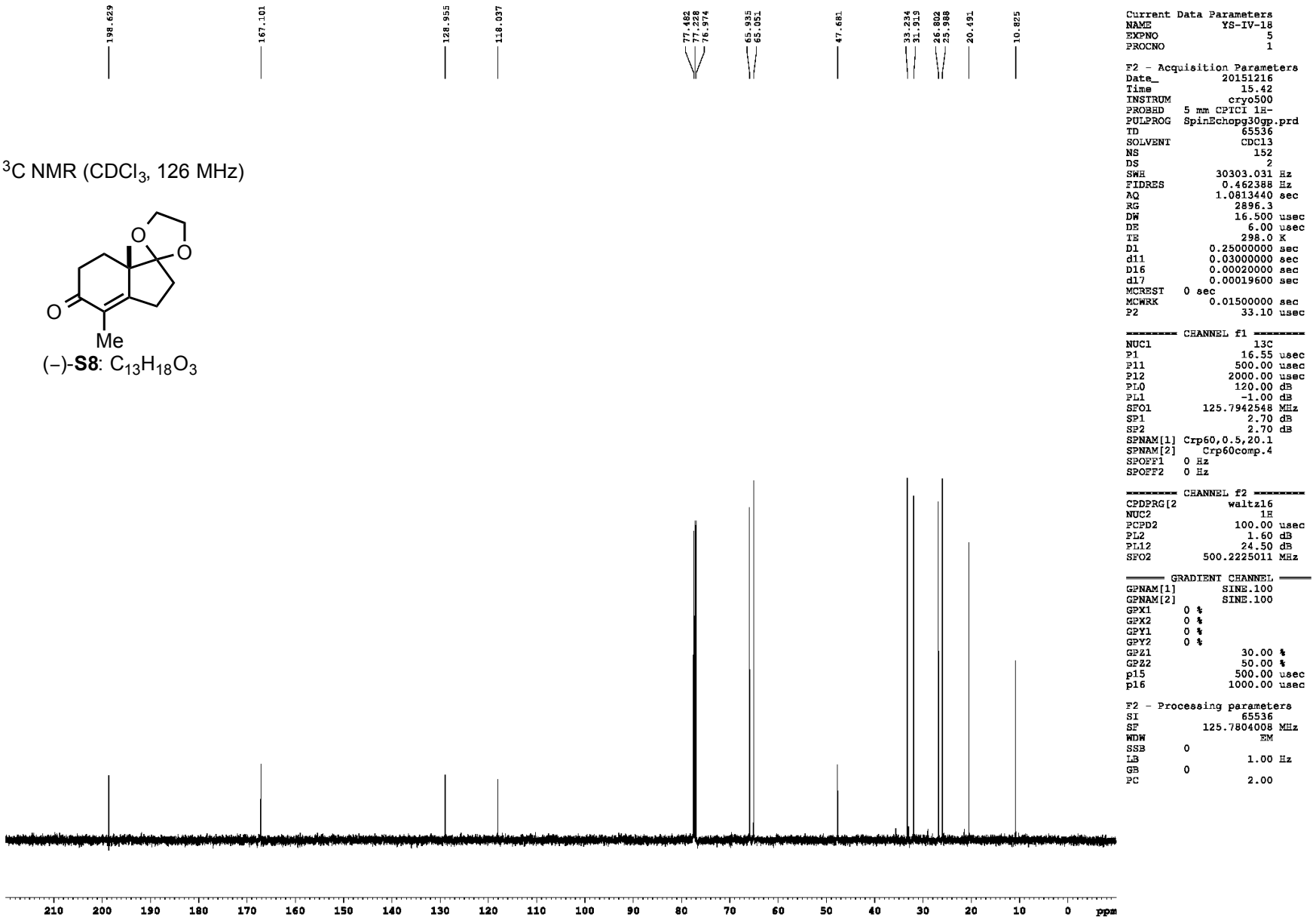
YS-IV-18

^{1}H NMR ($CDCl_3$, 600 MHz)



(–)-**S8**: $C_{13}H_{18}O_3$

YS-IV-18

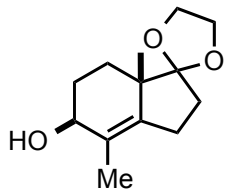


^{13}C NMR ($CDCl_3$, 126 MHz)

(–)-**S8**: $C_{13}H_{18}O_3$

YS-III-292

^{1}H NMR ($CDCl_3$, 500 MHz)



(–)-**S9**: $C_{13}H_{20}O_3$


```
Current Data Parameters
NAME              YS-III-292
EXPNO                      3
PROCNO                     1

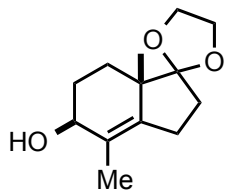
F2 - Acquisition Parameters
Date_            20151117
Time                 8.52
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG              zg30
TD                  32048
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8012.820 Hz
FIDRES           0.250026 Hz
AQ               1.9997952 sec
RG                    7.1
DW                 62.400 usec
DE                 6.00 usec
TE                 298.0 K
D1               0.10000000 sec
MCREST    0 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1                1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                    65536
SF           500.2200324 MHz
WDW                     EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    4.00
```

YS-IV-20

^{13}C NMR ($CDCl_3$, 126 MHz)



(–)-**S9**: $C_{13}H_{20}O_3$


```
Current Data Parameters
NAME                  YS-IV-20
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_              20151209
Time                    17.41
INSTRUM              cryo500
PROBHD    5 mm CPICI 1H-
PULPROG   SpinEchopg30gp.prd
TD                       65536
SOLVENT                  CDCl3
NS                         640
DS                           2
SWH                30303.031 Hz
FIDRES             0.462388 Hz
AQ                 1.0813440 sec
RG                    7298.2
DW                      16.500 usec
DE                       6.00 usec
TE                     298.0 K
D1                 0.25000000 sec
d11                0.03000000 sec
D16                0.00020000 sec
d17                0.00019600 sec
MCREST    0 sec
MCWRK              0.01500000 sec
P2                      33.10 usec

======== CHANNEL f1 ========
NUC1                       13C
P1                       16.55 usec
P11                     500.00 usec
P12                    2000.00 usec
PL0                     120.00 dB
PL1                      -1.00 dB
SFO1              125.7942548 MHz
SP1                       2.70 dB
SP2                       2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

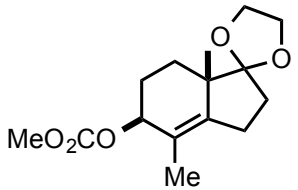
======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                       1H
PCPD2                  100.00 usec
PL2                      1.60 dB
PL12                    24.50 dB
SFO2              500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                    30.00 %
GPZ2                    50.00 %
p15                    500.00 usec
p16                   1000.00 usec

F2 - Processing parameters
SI                       65536
SF                125.7804059 MHz
WDW                         EM
SSB       0
LB                       1.00 Hz
GB        0
PC                       2.00
```

YS-III-293

^{1}H NMR ($CDCl_3$, 600 MHz)



(–)-**16**: $C_{15}H_{22}O_5$


```
Current Data Parameters
NAME                  YS-III-293
EXPNO                          2
PROCNO                         1

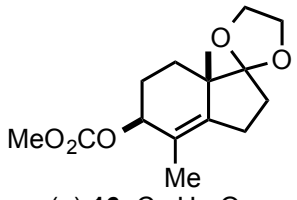
F2 - Acquisition Parameters
Date_               20151117
Time                      17.10
INSTRUM                  av600
PROBHD     5 mm TBI 1H/13
PULPROG                  zg30
TD                      38460
SOLVENT                 CDCl3
NS                             8
DS                             2
SWH                 9615.385 Hz
FIDRES              0.250010 Hz
AQ                  1.9999200 sec
RG                           161
DW                    52.000 usec
DE                    14.54 usec
TE                    298.0 K
D1                  0.10000000 sec
TD0                            1

====== CHANNEL f1 ======
SFO1               600.1342009 MHz
NUC1                         1H
P1                        8.00 usec
PLW1               24.00000000 W

F2 - Processing parameters
SI                         65536
SF                600.1300296 MHz
WDW                           EM
SSB                 0
LB                          0.30 Hz
GB                  0
PC                          1.00
```

YS-III-293

^{13}C NMR ($CDCl_3$, 126 MHz)



(–)-**16**: $C_{15}H_{22}O_5$


```
Current Data Parameters
NAME                  YS-III-293
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151117
Time                       17.20
INSTRUM                  cryo500
PROBHD    5 mm CPICI 1H-
PULPROG   SpinEchopg30gp.prd
TD                         65536
SOLVENT                   CDCl3
NS                           424
DS                            16
SWH                   30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                       5792.6
DW                       16.500 usec
DE                        6.00 usec
TE                       298.0 K
D1                    0.25000000 sec
d11                   0.03000000 sec
D16                   0.00020000 sec
d17                   0.00019600 sec
MCREST    0 sec
MCWRK                 0.01500000 sec
P2                       33.10 usec

======== CHANNEL f1 ========
NUC1                         13C
P1                         16.55 usec
P11                       500.00 usec
P12                      2000.00 usec
PL0                       120.00 dB
PL1                        -1.00 dB
SFO1                125.7942548 MHz
SP1                        2.70 dB
SP2                        2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

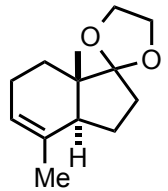
======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                    100.00 usec
PL2                        1.60 dB
PL12                      24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]               SINE.100
GPNAM[2]               SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                      30.00 %
GPZ2                      50.00 %
p15                      500.00 usec
p16                     1000.00 usec

F2 - Processing parameters
SI                         65536
SF                  125.7804002 MHz
WDW                           EM
SSB       0
LB                         1.00 Hz
GB        0
PC                         2.00
```

YS-III-296

^{1}H NMR ($CDCl_3$, 500 MHz)



(–)-**17**: $C_{13}H_{20}O_2$


```
Current Data Parameters
NAME              YS-III-296
EXPNO                      4
PROCNO                     1

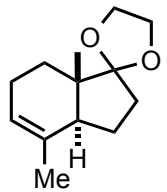
F2 - Acquisition Parameters
Date_            20151119
Time                 16.51
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG              zg30
TD                 128202
SOLVENT             CDCl3
NS                         8
DS                         2
SWH              8012.820 Hz
FIDRES           0.062502 Hz
AQ               7.9958050 sec
RG                       6.3
DW                  62.400 usec
DE                   6.00 usec
TE                  298.0 K
D1               0.10000000 sec
MCREST    0 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   7.50 usec
PL1                  1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                     65536
SF          500.2200323 MHz
WDW                       EM
SSB                    0
LB                   0.30 Hz
GB                     0
PC                   4.00
```

YS-III-296

^{13}C NMR ($CDCl_3$, 126 MHz)



(–)-**17**: $C_{13}H_{20}O_2$


```
Current Data Parameters
NAME                  YS-III-296
EXPNO                          5
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151119
Time                       16.57
INSTRUM                  cryo500
PROBHD     5 mm CPTCI 1H-
PULPROG    SpinEchopg30gp.prd
TD                         65536
SOLVENT                   CDCl3
NS                           504
DS                            16
SWH                   30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                         6502
DW                       16.500 usec
DE                        6.00 usec
TE                       298.0 K
D1                    0.25000000 sec
d11                   0.03000000 sec
D16                   0.00020000 sec
d17                   0.00019600 sec
MCREST     0 sec
MCWRK                 0.01500000 sec
P2                       33.10 usec

======== CHANNEL f1 ========
NUC1                         13C
P1                         16.55 usec
P11                       500.00 usec
P12                      2000.00 usec
PL0                       120.00 dB
PL1                        -1.00 dB
SFO1                125.7942548 MHz
SP1                         2.70 dB
SP2                         2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1     0 Hz
SPOFF2     0 Hz

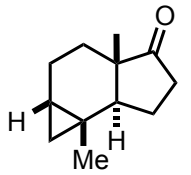
======== CHANNEL f2 ========
CPDPRG[2                 waltz16
NUC2                         1H
PCPD2                     100.00 usec
PL2                         1.60 dB
PL12                       24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]                SINE.100
GPNAM[2]                SINE.100
GPX1       0 %
GPX2       0 %
GPY1       0 %
GPY2       0 %
GPZ1                       30.00 %
GPZ2                       50.00 %
p15                       500.00 usec
p16                      1000.00 usec

F2 - Processing parameters
SI                         65536
SF                  125.7803998 MHz
WDW                           EM
SSB        0
LB                          1.00 Hz
GB         0
PC                          2.00
```

YS-IV-29

^{1}H NMR ($CDCl_3$, 600 MHz)



(+)-**18**: $C_{12}H_{18}O$

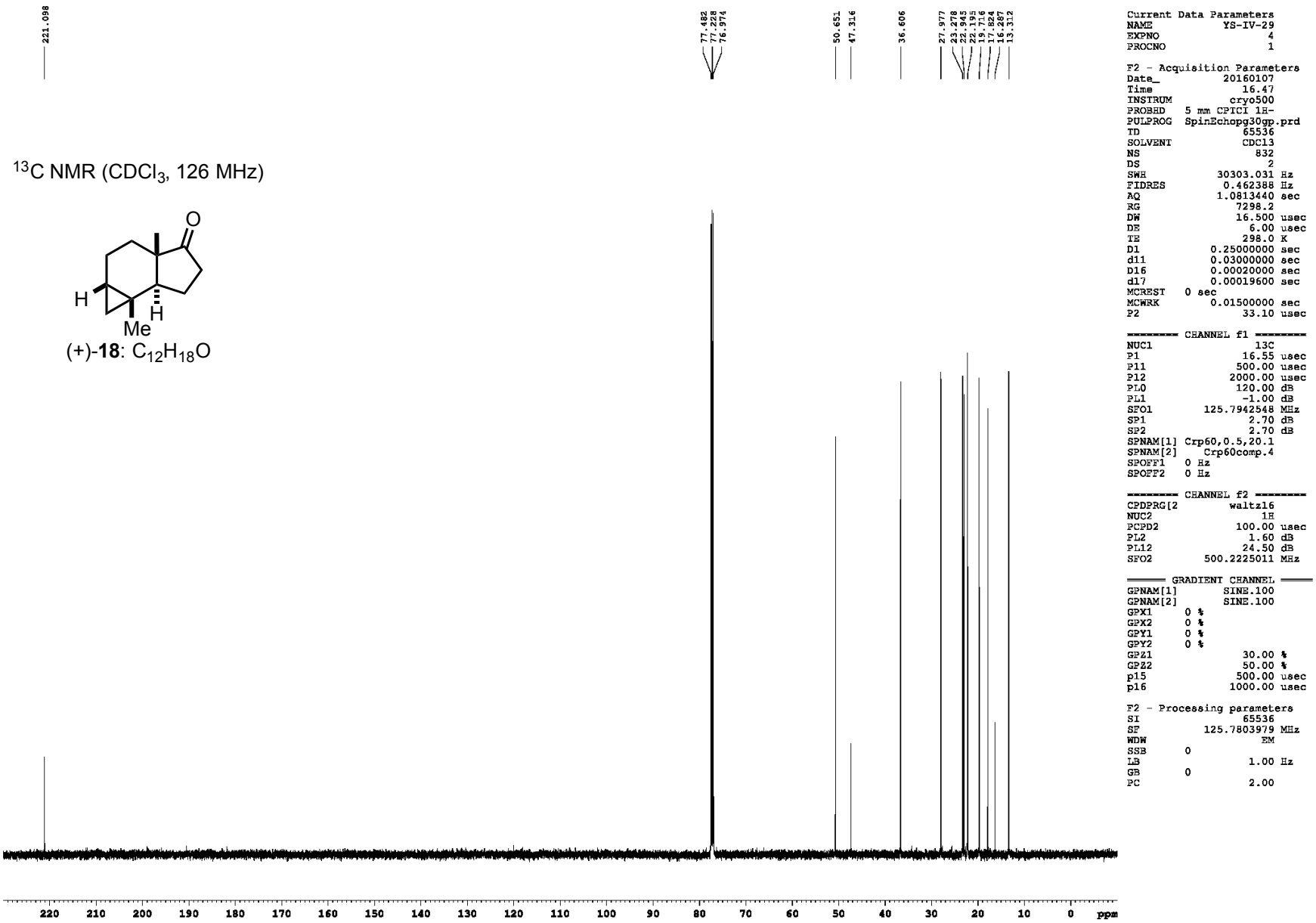

```
Current Data Parameters
NAME                YS-IV-29
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160107
Time                  16.39
INSTRUM                av600
PROBHD   5 mm TBI 1H/13
PULPROG                zg30
TD                    98074
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                9615.385 Hz
FIDRES            0.098042 Hz
AQ                5.0998478 sec
RG                      362
DW                 52.000 usec
DE                 14.54 usec
TE                 298.1 K
D1              0.10000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          600.1342009 MHz
NUC1                     1H
P1                 8.00 usec
PLW1          24.00000000 W

F2 - Processing parameters
SI                    65536
SF            600.1300354 MHz
WDW                      EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

YS-IV-29



^{13}C NMR ($CDCl_3$, 126 MHz)

(+)-**18**: $C_{12}H_{18}O$

```
Current Data Parameters
NAME                  YS-IV-29
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_              20160107
Time                   16.47
INSTRUM              cryo500
PROBHD    5 mm CPICI 1H-
PULPROG   SpinEchopg30gp.prd
TD                     65536
SOLVENT                CDCl3
NS                       832
DS                         2
SWH                30303.031 Hz
FIDRES             0.462388 Hz
AQ                 1.0813440 sec
RG                    7298.2
DW                    16.500 usec
DE                     6.00 usec
TE                    298.0 K
D1                 0.25000000 sec
d11                0.03000000 sec
D16                0.00020000 sec
d17                0.00019600 sec
MCREST    0 sec
MCWRK              0.01500000 sec
P2                    33.10 usec

======== CHANNEL f1 ========
NUC1                      13C
P1                     16.55 usec
P11                   500.00 usec
P12                  2000.00 usec
PL0                   120.00 dB
PL1                    -1.00 dB
SFO1             125.7942548 MHz
SP1                    2.70 dB
SP2                    2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

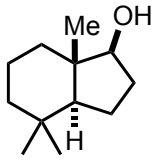
======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                    1.60 dB
PL12                  24.50 dB
SFO2             500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                  30.00 %
GPZ2                  50.00 %
p15                  500.00 usec
p16                 1000.00 usec

F2 - Processing parameters
SI                     65536
SF               125.7803979 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     2.00
```

YS-IV-24

^{1}H NMR ($CDCl_3$, 500 MHz)



(+)-**S10**: $C_{12}H_{22}O$


```
Current Data Parameters
NAME                  YS-IV-24
EXPNO                        5
PROCNO                       1

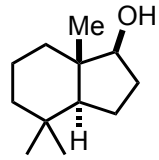
F2 - Acquisition Parameters
Date_              20151216
Time                    15.49
INSTRUM                cryo500
PROBHD     5 mm CPTCI 1H-
PULPROG                 zg30
TD                      81728
SOLVENT                 CDCl3
NS                          8
DS                          2
SWH                  8012.820 Hz
FIDRES               0.098043 Hz
AQ                   5.0958273 sec
RG                          5
DW                     62.400 usec
DE                      6.00 usec
TE                     298.0 K
D1                  0.10000000 sec
MCREST       0 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      7.50 usec
PL1                     1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                       65536
SF            500.2200314 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      4.00
```

YS-IV-24

^{13}C NMR ($CDCl_3$, 126 MHz)



(+)-**S10**: $C_{12}H_{22}O$


```
Current Data Parameters
NAME                  YS-IV-24
EXPNO                        6
PROCNO                       1

F2 - Acquisition Parameters
Date_              20151216
Time                   15.50
INSTRUM              cryo500
PROBHD    5 mm CPICI 1H-
PULPROG   SpinEchopg30gp.prd
TD                    65536
SOLVENT               CDCl3
NS                      248
DS                        2
SWH               30303.031 Hz
FIDRES            0.462388 Hz
AQ                1.0813440 sec
RG                   7298.2
DW                   16.500 usec
DE                    6.00 usec
TE                   298.0 K
D1                0.25000000 sec
d11               0.03000000 sec
D16               0.00020000 sec
d17               0.00019600 sec
MCREST    0 sec
MCWRK             0.01500000 sec
P2                   33.10 usec

======== CHANNEL f1 ========
NUC1                    13C
P1                    16.55 usec
P11                  500.00 usec
P12                 2000.00 usec
PL0                  120.00 dB
PL1                   -1.00 dB
SFO1           125.7942548 MHz
SP1                   2.70 dB
SP2                   2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

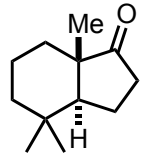
======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                    1.60 dB
PL12                  24.50 dB
SFO2           500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                  30.00 %
GPZ2                  50.00 %
p15                  500.00 usec
p16                 1000.00 usec

F2 - Processing parameters
SI                    65536
SF             125.7803692 MHz
WDW                      EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     2.00
```

YS-IV-25

^{1}H NMR ($CDCl_3$, 600 MHz)



(+)-**13**: $C_{12}H_{20}O$


```
Current Data Parameters
NAME                  YS-IV-25
EXPNO                        1
PROCNO                       1

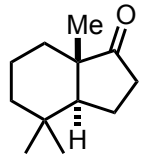
F2 - Acquisition Parameters
Date_              20151217
Time                    10.44
INSTRUM                av600
PROBHD     5 mm TBI 1H/13
PULPROG                 zg30
TD                     98074
SOLVENT                CDCl3
NS                           8
DS                           2
SWH                 9615.385 Hz
FIDRES              0.098042 Hz
AQ                  5.0958478 sec
RG                         228
DW                    52.000 usec
DE                    14.54 usec
TE                    293.0 K
D1                  0.10000000 sec
TD0                          1

====== CHANNEL f1 ======
SFO1             600.1342009 MHz
NUC1                       1H
P1                    8.00 usec
PLW1            24.00000000 W

F2 - Processing parameters
SI                       65536
SF               600.1300296 MHz
WDW                        EM
SSB                 0
LB                         0.30 Hz
GB                  0
PC                         1.00
```

YS-IV-25

^{13}C NMR ($CDCl_3$, 126 MHz)



(+)-**13**: $C_{12}H_{20}O$


```
Current Data Parameters
NAME                  YS-IV-25
EXPNO                        2
PROCNO                       1

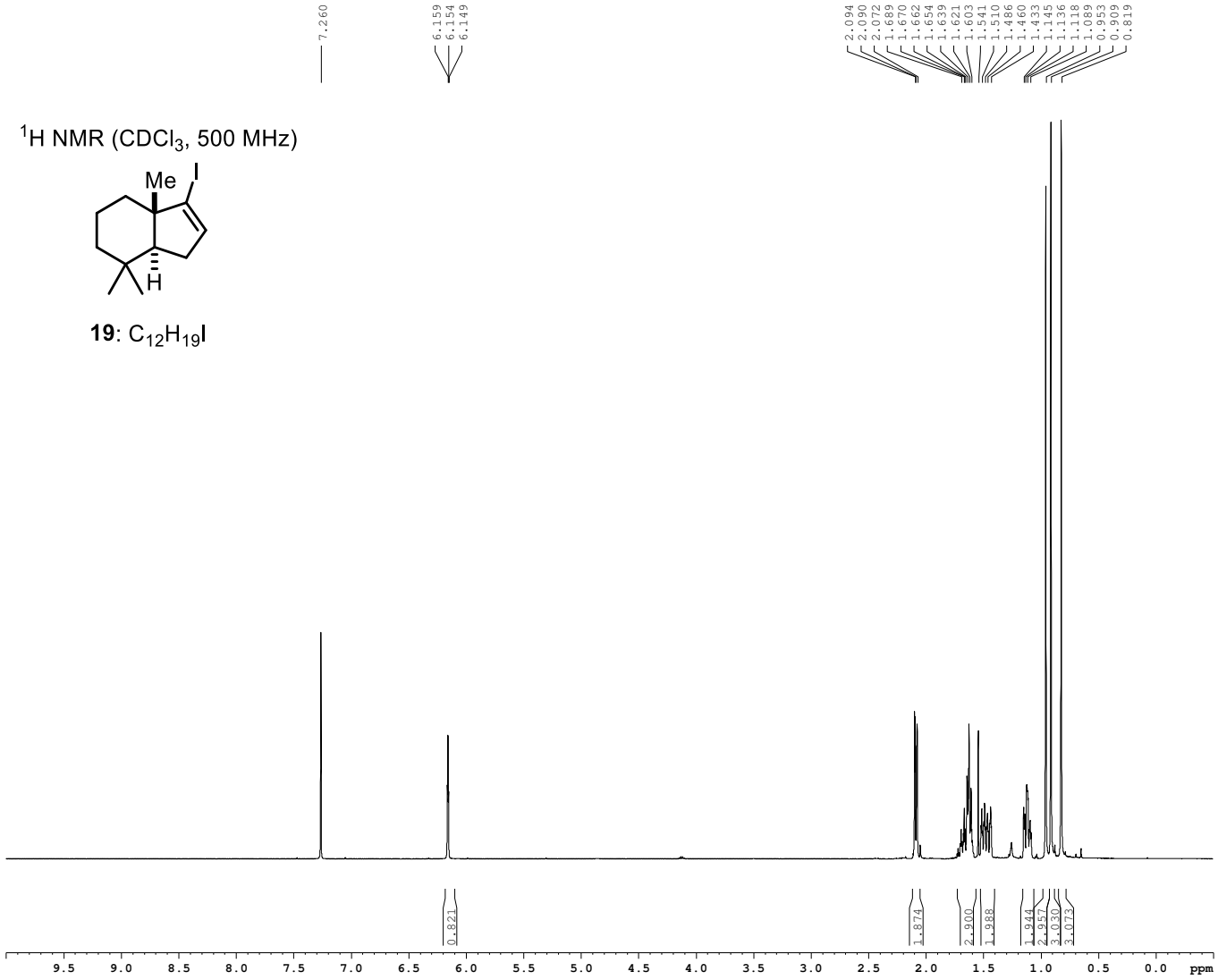
F2 - Acquisition Parameters
Date_              20151217
Time                   10.53
INSTRUM                gn500
PROBHD   5 mm broadband
PULPROG              zgdc30
TD                    65536
SOLVENT               CDCl3
NS                      800
DS                        2
SWH               30303.031 Hz
FIDRES            0.462388 Hz
AQ                1.0813440 sec
RG                   18390.4
DW                  16.500 usec
DE                   4.50 usec
TE                   298.0 K
D1                0.25000000 sec
d11               0.03000000 sec
MCREST    0 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.00 usec
PL1                  -0.60 dB
SFO1          125.5327181 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 12.80 dB
SFO2          499.1824959 MHz

F2 - Processing parameters
SI                    65536
SF            125.5188906 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    2.00
```

1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

19: $C_{12}H_{19}I$

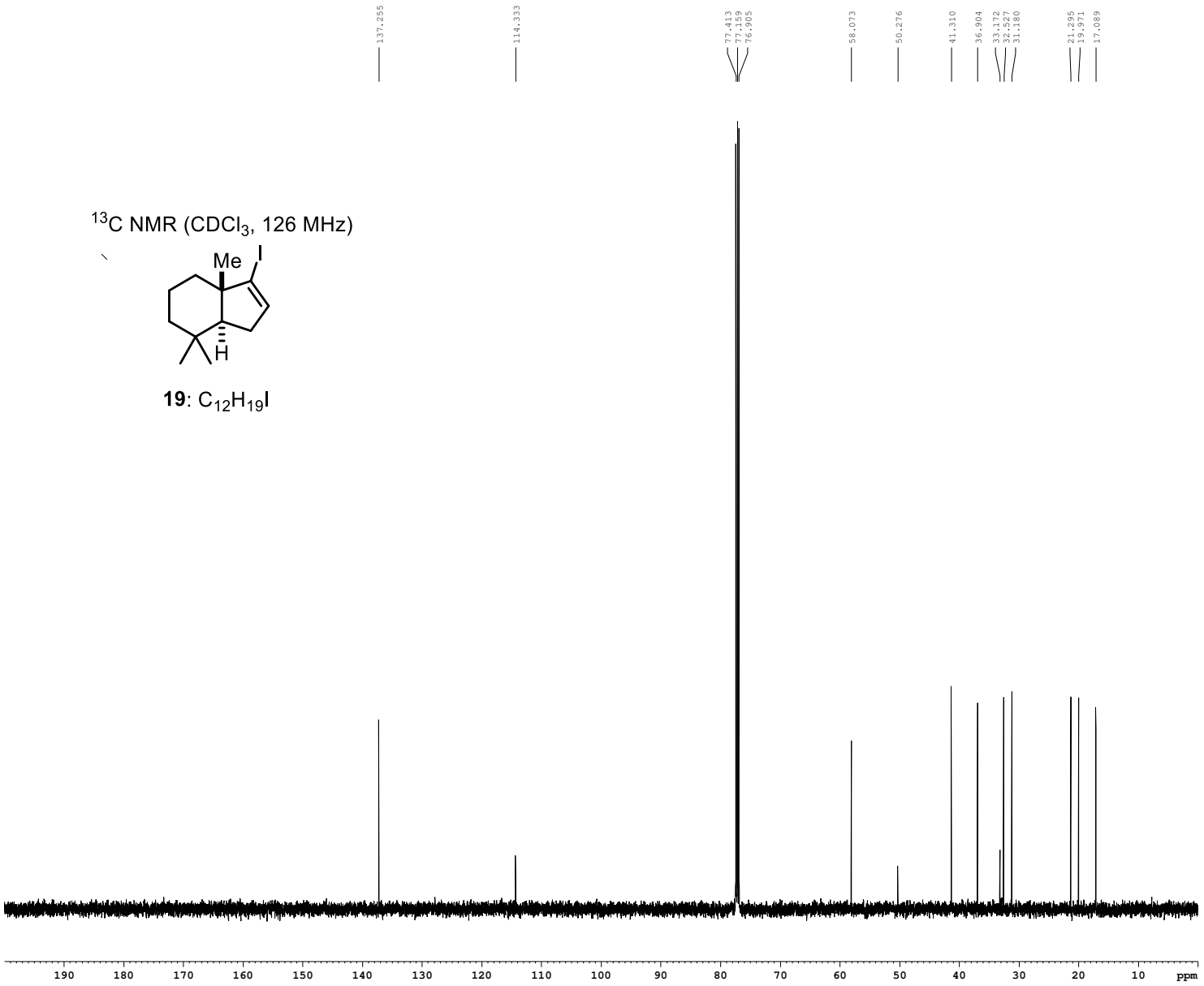
```
Current Data Parameters
NAME                  DJT-V-036
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150203
Time                      18.19
INSTRUM                cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG                    zg30
TD                        16022
SOLVENT                   CDCl3
NS                           60
DS                            0
SWH                    8012.820 Hz
FIDRES                 0.500114 Hz
AQ                     0.9997728 sec
RG                           5.7
DW                        62.400 usec
DE                         6.00 usec
TE                        298.0 K
D1                  0.10000000 sec
MCREST   0 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                         7.50 usec
PL1                        1.60 dB
SFO1                500.2235015 MHz

F2 - Processing parameters
SI                        65536
SF                  500.2200311 MHz
WDW                          no
SSB       0
LB        0 Hz
GB        0
PC                         4.00
```

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

19: $C_{12}H_{19}I$

```
Current Data Parameters
NAME                  DJT-V-036
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150203
Time                      18.24
INSTRUM                cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                         65536
SOLVENT                    CDCl3
NS                           391
DS                             0
SWH                  30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                        13004
DW                       16.500 usec
DE                        6.00 usec
TE                       298.0 K
D1                    0.25000000 sec
d11                   0.03000000 sec
D16                   0.00020000 sec
d17                   0.00019600 sec
MCREST    0 sec
MCWRK                 0.01500000 sec
P2                       33.10 usec

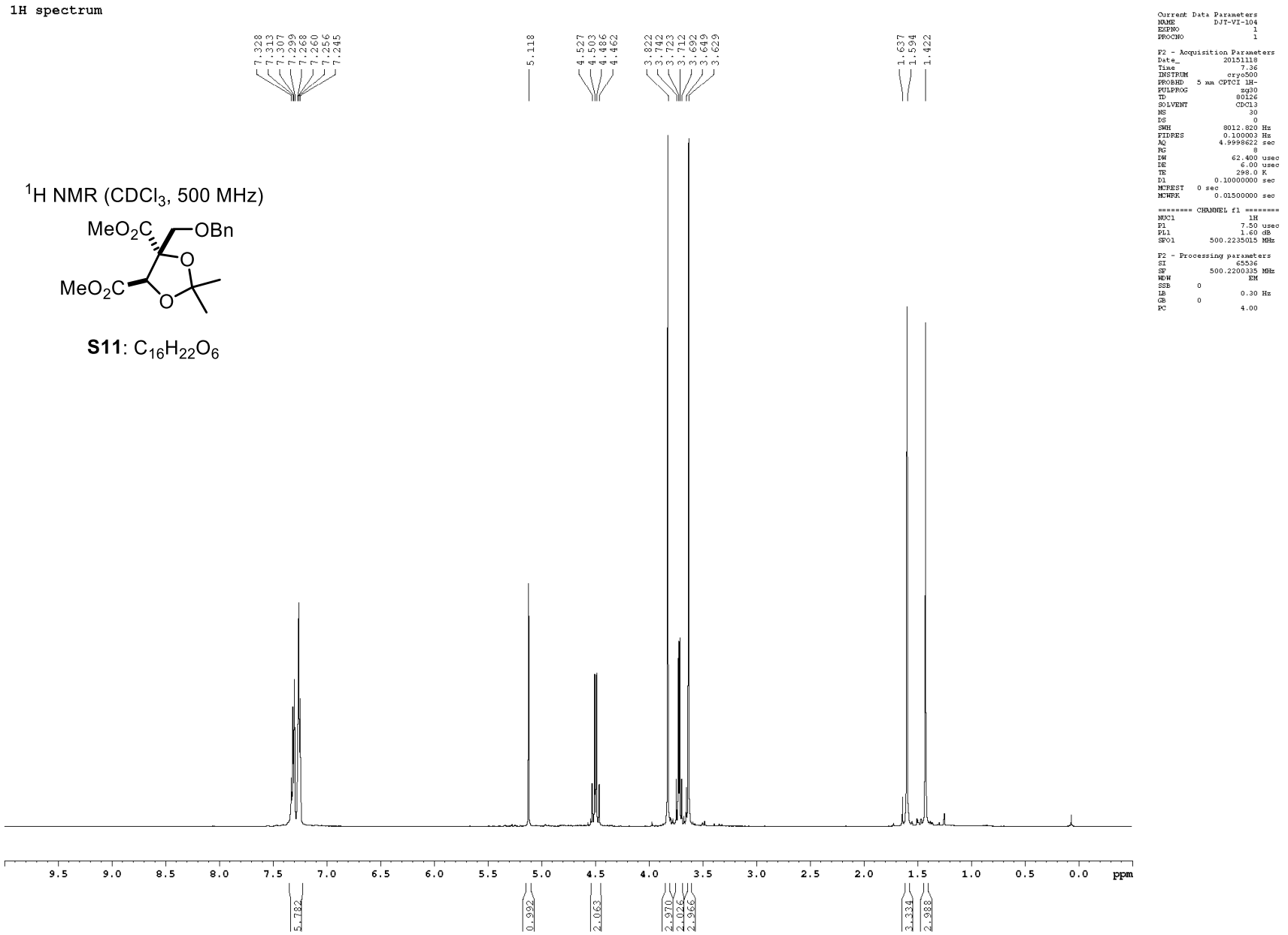
======== CHANNEL f1 ========
NUC1                        13C
P1                       16.55 usec
P11                     500.00 usec
P12                    2000.00 usec
PL0                     120.00 dB
PL1                      -1.00 dB
SFO1                125.7942548 MHz
SP1                       2.70 dB
SP2                       2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                   100.00 usec
PL2                       1.60 dB
PL12                     24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]               SINE.100
GPNAM[2]               SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                     30.00 %
GPZ2                     50.00 %
p15                     500.00 usec
p16                    1000.00 usec

F2 - Processing parameters
SI                         65536
SF                  125.7804074 MHz
WDW                           EM
SSB       0
LB                        1.00 Hz
GB        0
PC                        2.00
```

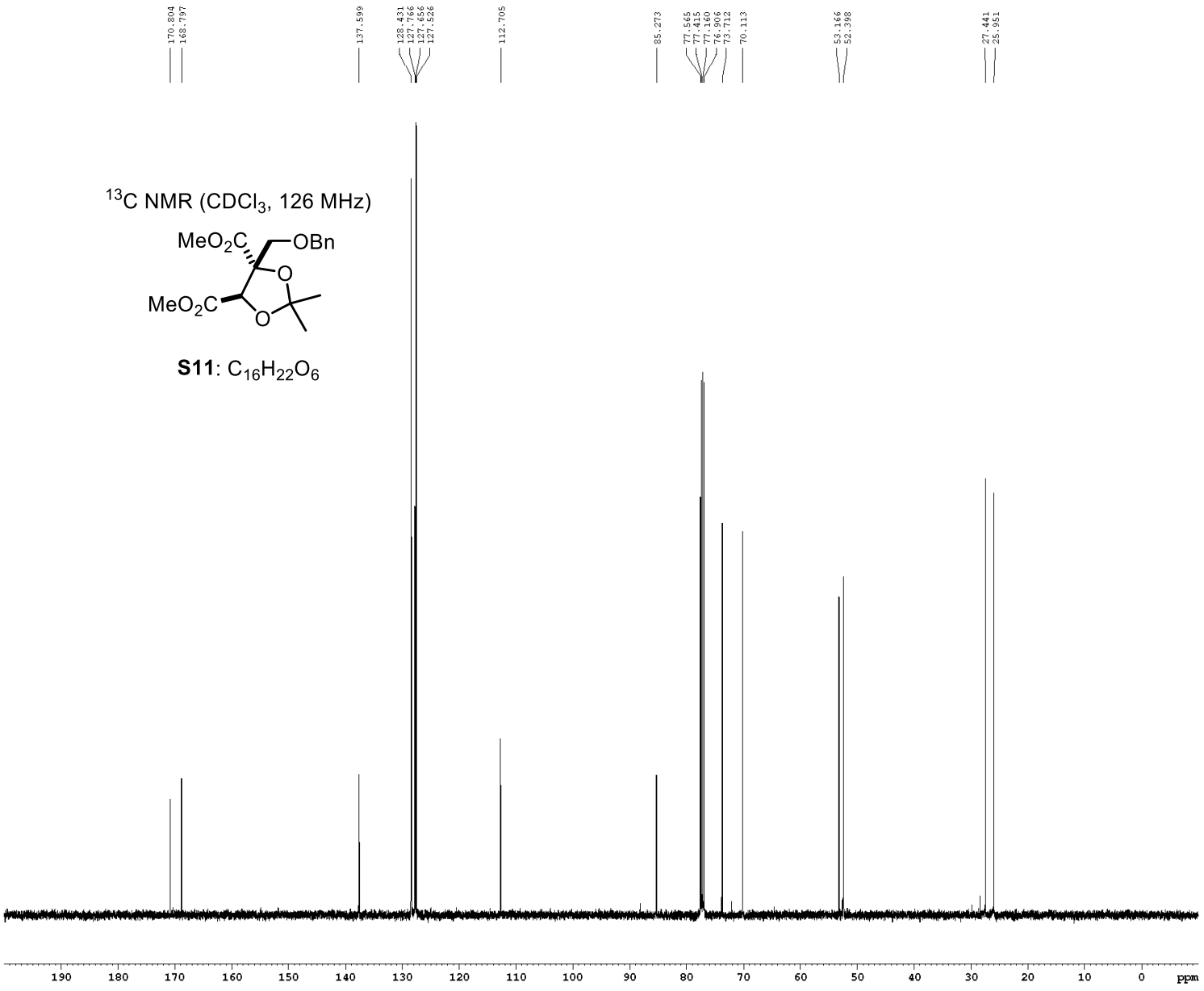
1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

S11: $C_{16}H_{22}O_6$

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

S11: $C_{16}H_{22}O_6$

```
Current Data Parameters
NAME          DJT-VI-104
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151118
Time                7.42
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   374
DS                     0
SWH            30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                  6502
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
D16          0.00020000 sec
d17          0.00019600 sec
MCREST    0 sec
MCWRK        0.01500000 sec
P2                33.10 usec

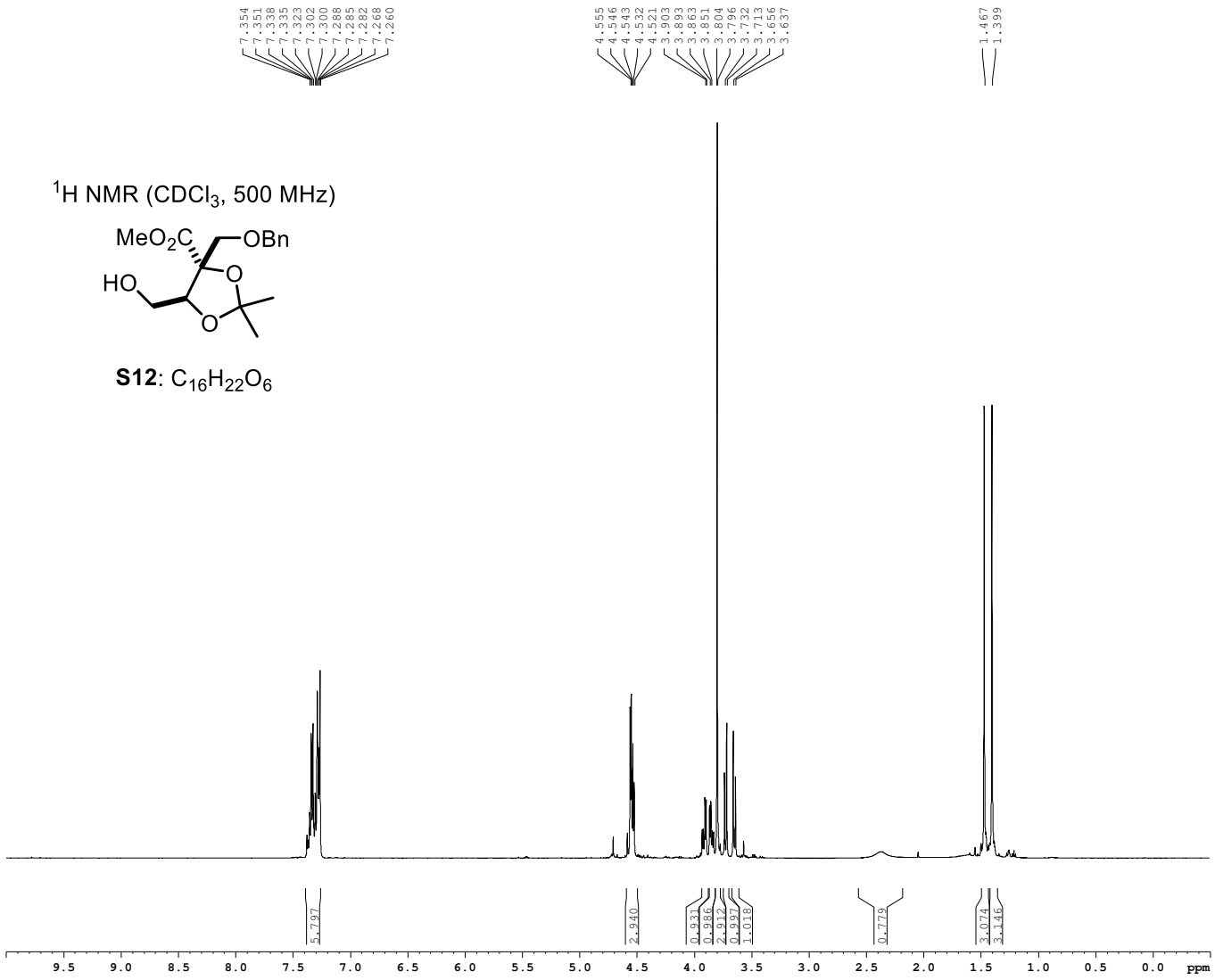
======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1        125.7942548 MHz
SP1                 2.70 dB
SP2                 2.70 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              24.50 dB
SFO2        500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1               30.00 %
GPZ2               50.00 %
p15               500.00 usec
p16              1000.00 usec

F2 - Processing parameters
SI                 65536
SF          125.7804103 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 2.00
```

1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

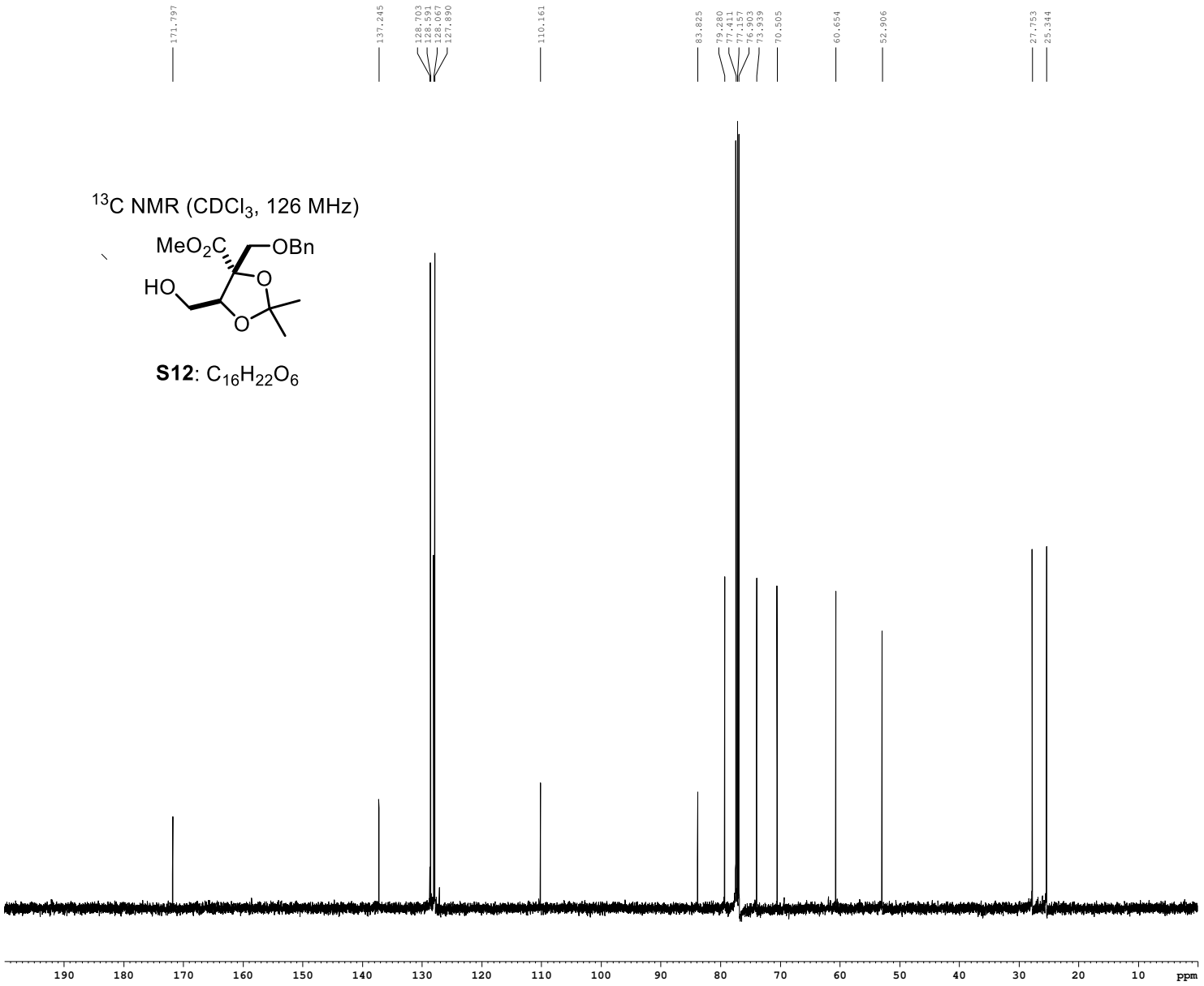
S12: $C_{16}H_{22}O_6$

```
Current Data Parameters
NAME          DJT-IV-110
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140813
Time               14.46
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES             0.098043 Hz
AQ              5.0998273 sec
RG                   7.1
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1              0.10000000 sec
MCREST   0 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.50 usec
PL1                 1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF            500.2200307 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  4.00
```



^{13}C NMR ($CDCl_3$, 126 MHz)

S12: $C_{16}H_{22}O_6$

```
Current Data Parameters
NAME          DJT-IV-110
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140814
Time               11.08
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   562
DS                    16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG               7298.2
DW              16.500 usec
DE               6.00 usec
TE              298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
D16          0.00020000 sec
d17          0.00019600 sec
MCREST    0 sec
MCWRK        0.01500000 sec
P2              31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]       SINE.100
GPNAM[2]       SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1              30.00 %
GPZ2              50.00 %
p15              500.00 usec
p16             1000.00 usec

F2 - Processing parameters
SI                 65536
SF           125.7804089 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 2.00
```

YS-III-303

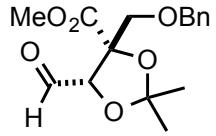
Current Data Parameters
NAME YS-III-303
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151128
Time 13.45
INSTRUM av600
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 38460
SOLVENT CDCl3
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.250010 Hz
AQ 1.9999200 sec
RG 1620
DW 52.000 usec
DE 14.54 usec
TE 298.0 K
D1 0.10000000 sec
TD0 1

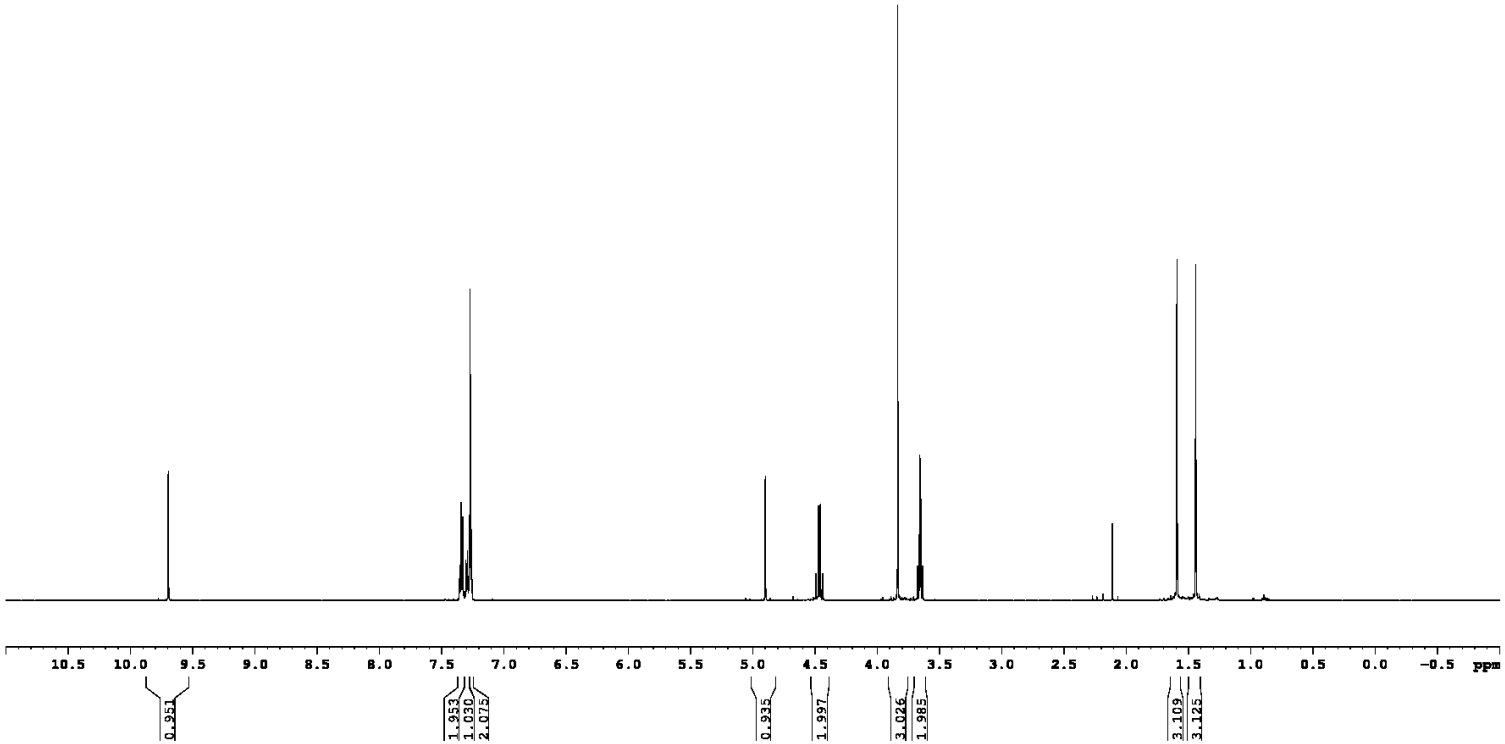
======== CHANNEL f1 ========
SFO1 600.1342009 MHz
NUC1 1H
P1 8.00 usec
PLW1 24.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1300297 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

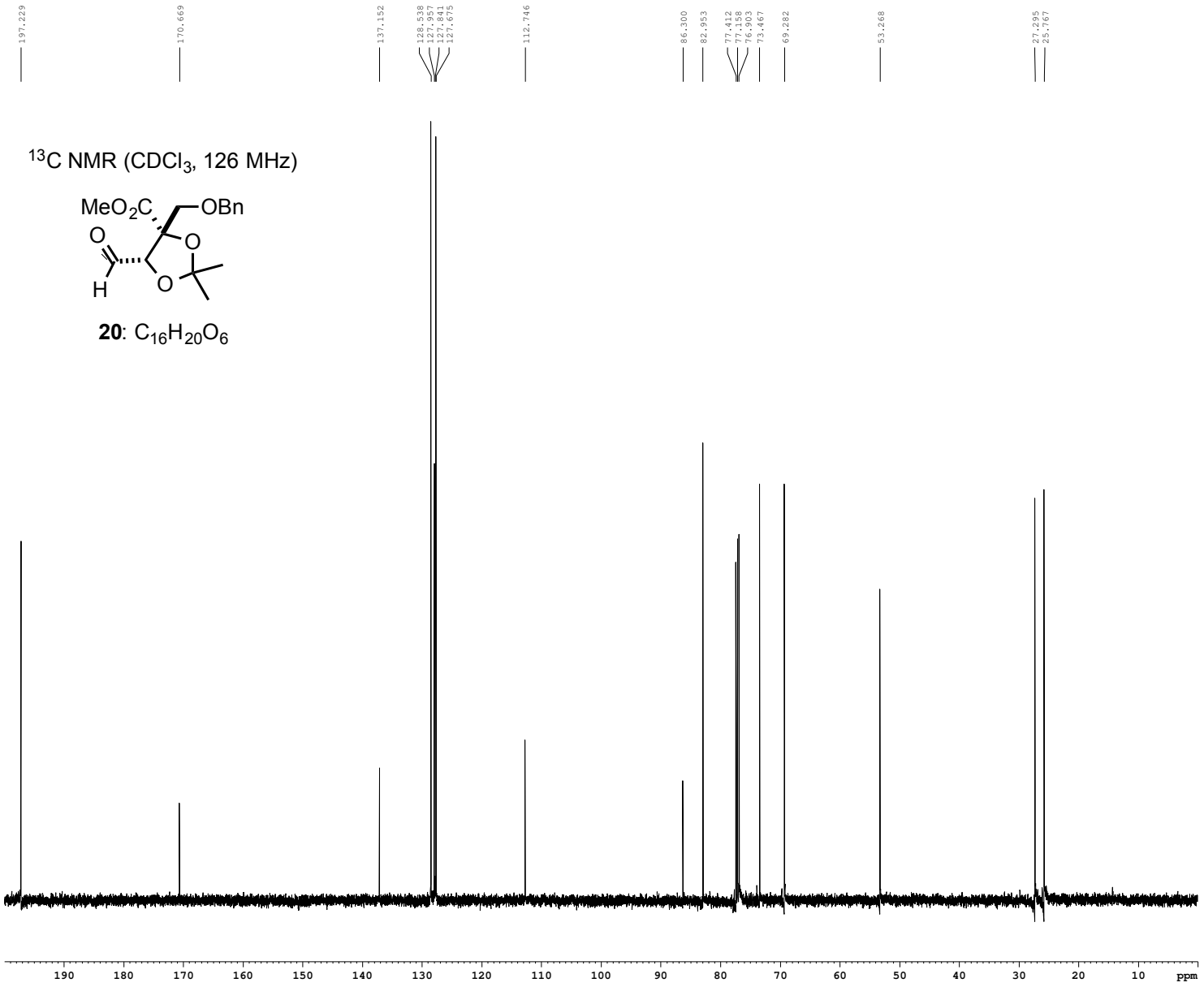
^{1}H NMR ($CDCl_3$, 600 MHz)



20: $C_{16}H_{20}O_6$



Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

20: $C_{16}H_{20}O_6$

```
Current Data Parameters
NAME          DJT-IV-040
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140616
Time               16.32
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   101
DS                     0
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813440 sec
RG                  6502
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST    0 sec
MCWRK         0.01500000 sec
P2                31.00 usec

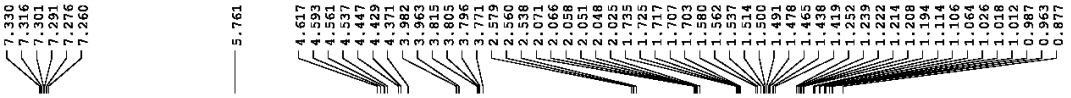
======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

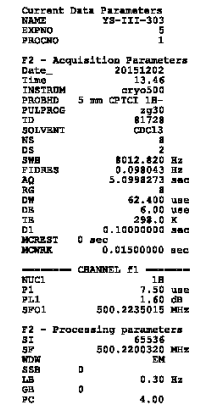
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]       SINE.100
GPNAM[2]       SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1              30.00 %
GPZ2              50.00 %
p15              500.00 usec
p16             1000.00 usec

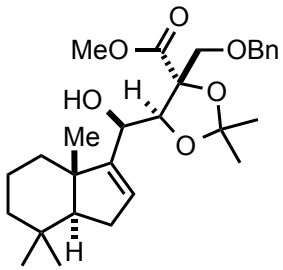
F2 - Processing parameters
SI                 65536
SF           125.7804122 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 2.00
```

YS-III-303

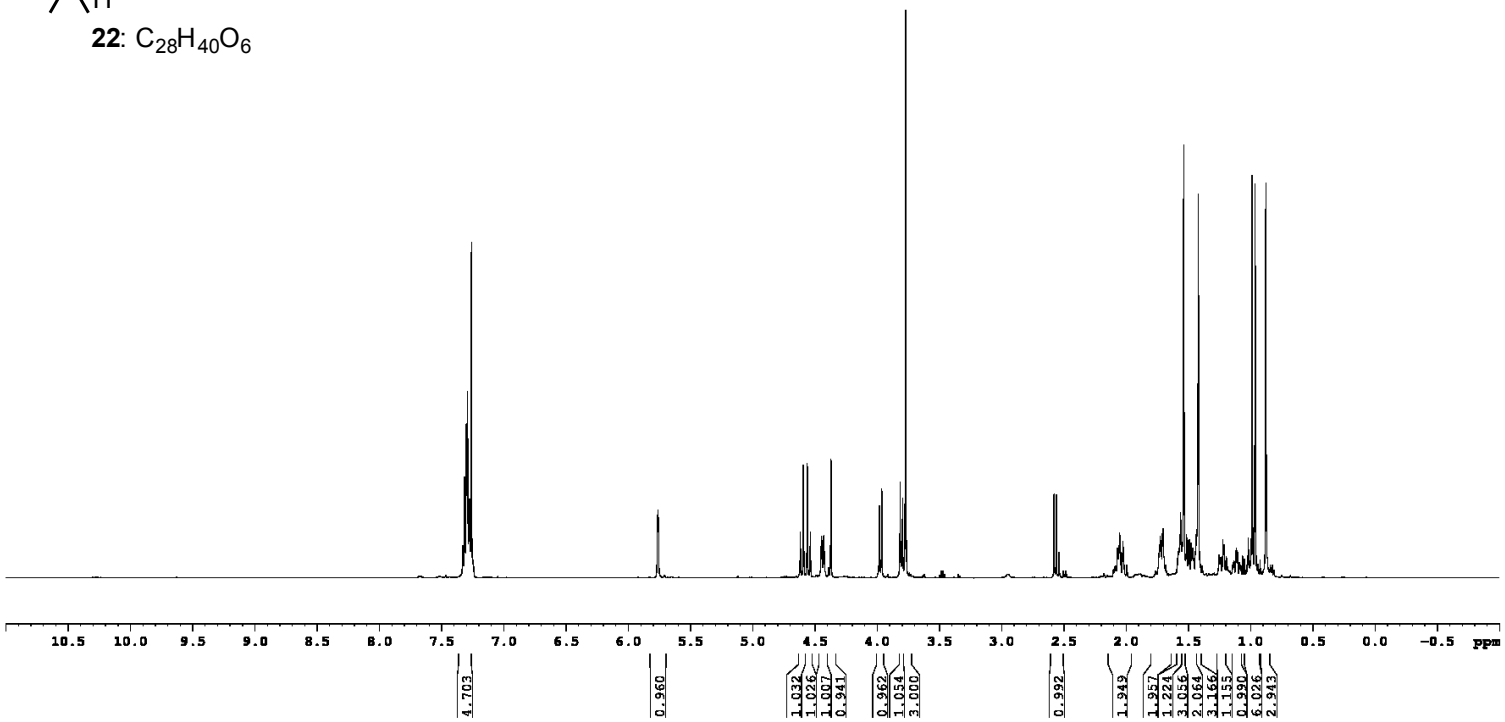




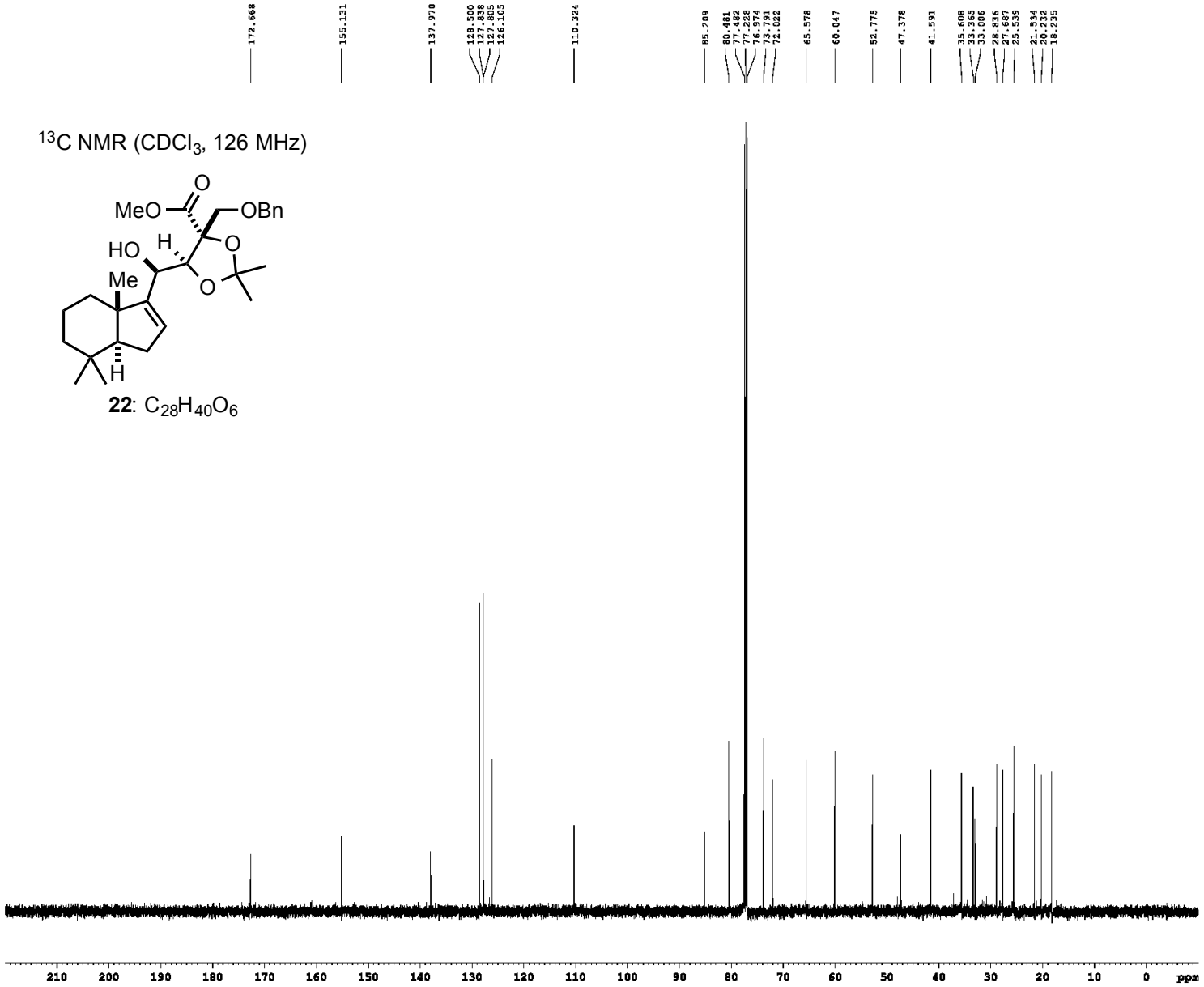
^{1}H NMR ($CDCl_3$, 500 MHz)



22: $C_{28}H_{40}O_6$



YS-III-303



^{13}C NMR ($CDCl_3$, 126 MHz)

22: $C_{28}H_{40}O_6$

```
Current Data Parameters
NAME                  YS-III-303
EXPNO                          6
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151202
Time                       13.57
INSTRUM                  cryo500
PROBHD     5 mm CPICI 1H-
PULPROG    SpinEchopg30gp.prd
TD                          65536
SOLVENT                   CDCl3
NS                            496
DS                              2
SWH                   30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                       2580.3
DW                       16.500 usec
DE                        6.00 usec
TE                       298.0 K
D1                    0.25000000 sec
d11                   0.03000000 sec
D16                   0.00020000 sec
d17                   0.00019600 sec
MCREST     0 sec
MCWRK                 0.01500000 sec
P2                       33.10 usec

======== CHANNEL f1 ========
NUC1                        13C
P1                       16.55 usec
P11                     500.00 usec
P12                    2000.00 usec
PL0                     120.00 dB
PL1                      -1.00 dB
SFO1                125.7942548 MHz
SP1                        2.70 dB
SP2                        2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1     0 Hz
SPOFF2     0 Hz

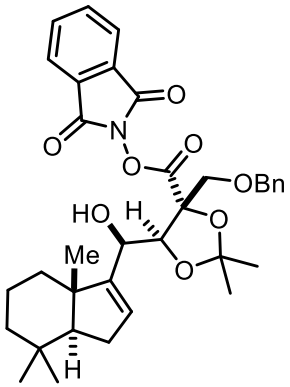
======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                      1.60 dB
PL12                    24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]            SINE.100
GPNAM[2]            SINE.100
GPX1       0 %
GPX2       0 %
GPY1       0 %
GPY2       0 %
GPZ1                    30.00 %
GPZ2                    50.00 %
p15                    500.00 usec
p16                   1000.00 usec

F2 - Processing parameters
SI                          65536
SF                 125.7803996 MHz
WDW                           EM
SSB        0
LB                       1.00 Hz
GB         0
PC                       2.00
```

1H spectrum

^{1}H NMR ($CDCl_3$, 600 MHz)



23: $C_{35}H_{41}NO_8$

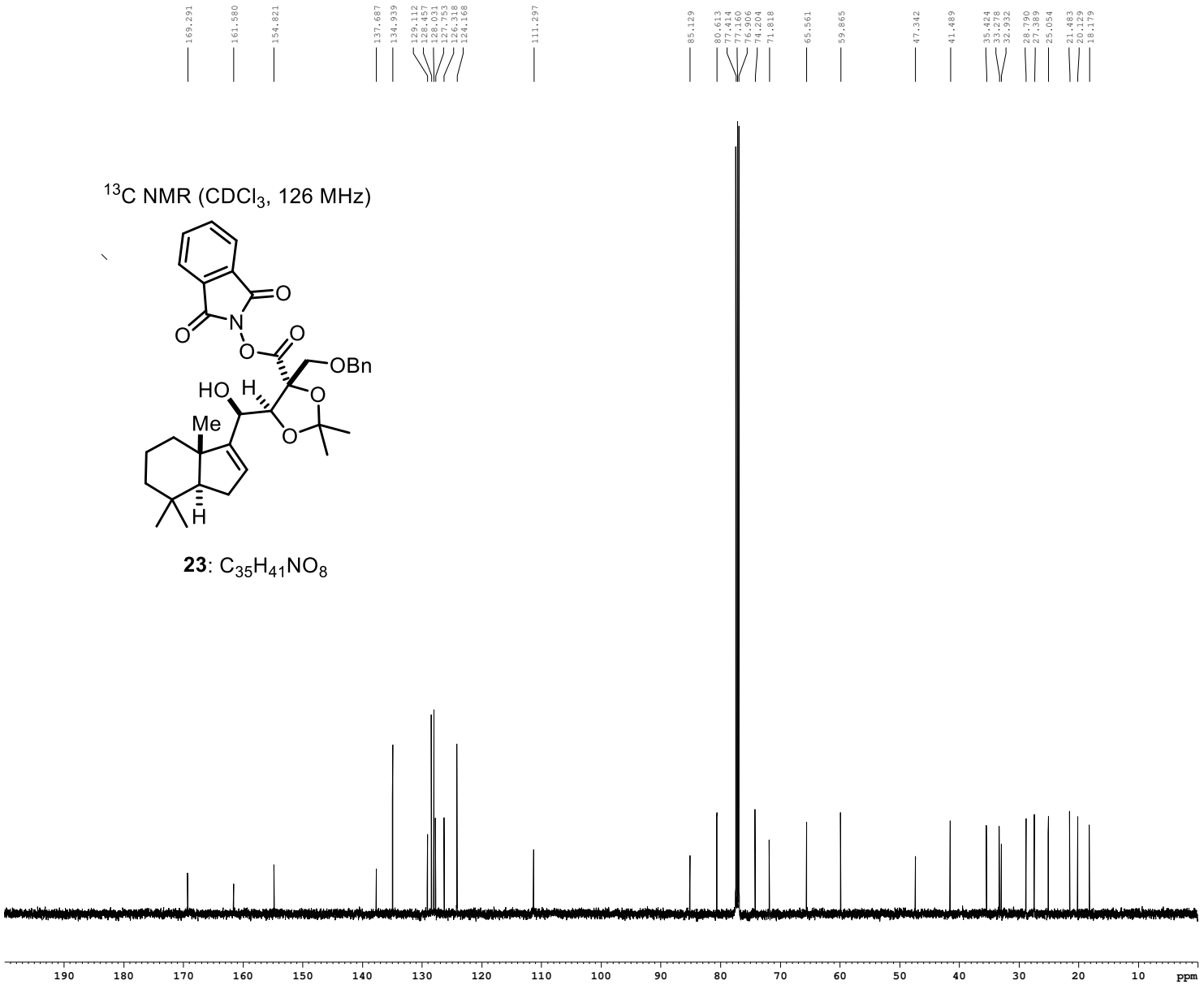

```
Current Data Parameters
NAME              DJT-IV-203
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20141013
Time                  16.31
INSTRUM               av600
PROBHD   5 mm TBI 1H/13
PULPROG                zg30
TD                    98074
SOLVENT               CDCl3
NS                       12
DS                        0
SWH                9615.385 Hz
FIDRES            0.098042 Hz
AQ                5.0998478 sec
RG                      322
DW                   52.000 usec
DE                   14.54 usec
TE                   298.0 K
D1                0.10000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           600.1342009 MHz
NUC1                     1H
P1                    8.00 usec
PLW1          23.01441956 W

F2 - Processing parameters
SI                    65536
SF             600.1300343 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

23: $C_{35}H_{41}NO_8$

```
Current Data Parameters
NAME          DJT-IV-203
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150414
Time                8.55
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   857
DS                     0
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813440 sec
RG                7298.2
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
D16            0.00020000 sec
d17            0.00019600 sec
MCREST    0 sec
MCWRK          0.01500000 sec
P2                33.10 usec

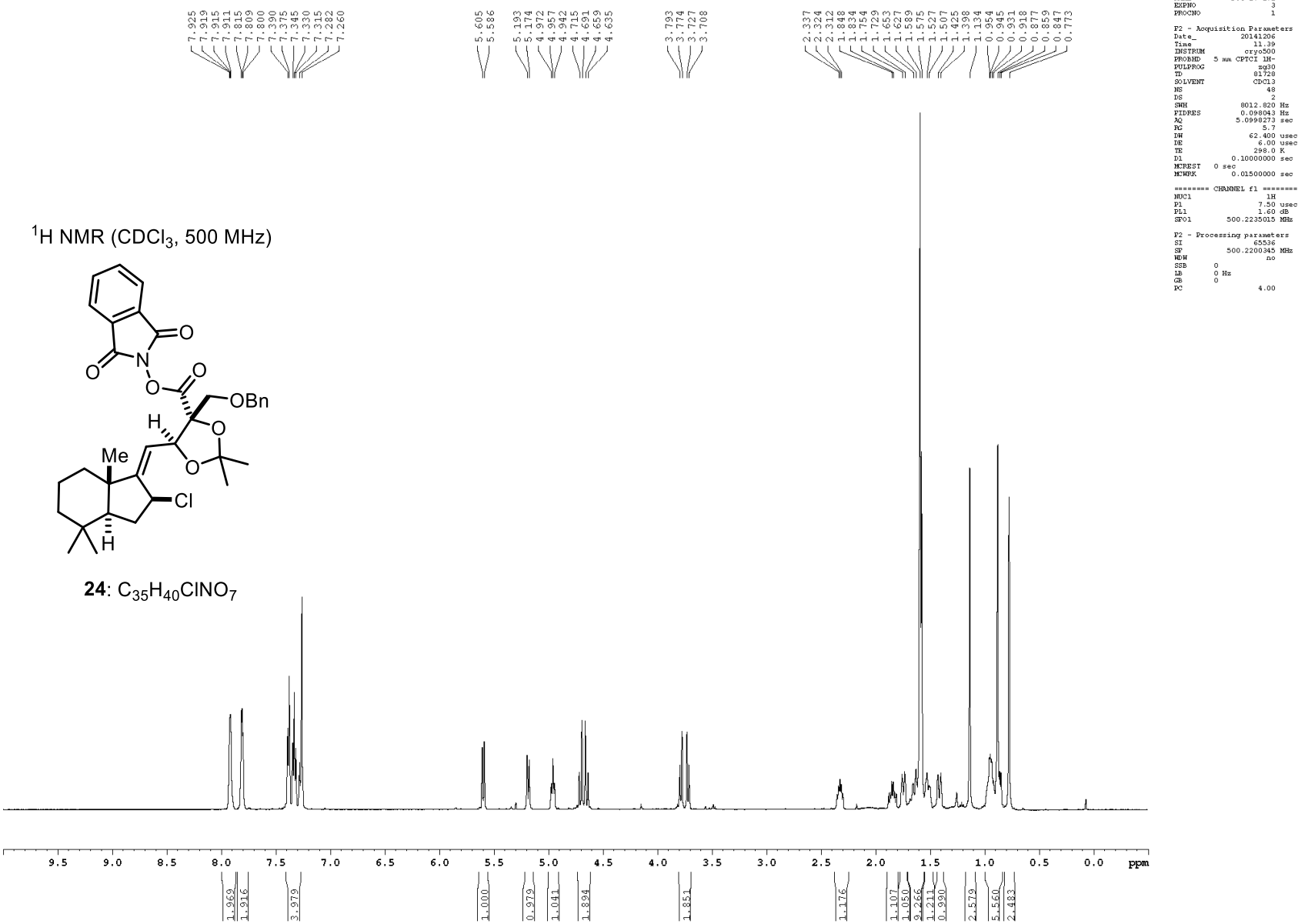
======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              24.50 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1              30.00 %
GPZ2              50.00 %
p15              500.00 usec
p16             1000.00 usec

F2 - Processing parameters
SI                 65536
SF           125.7804076 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 2.00
```

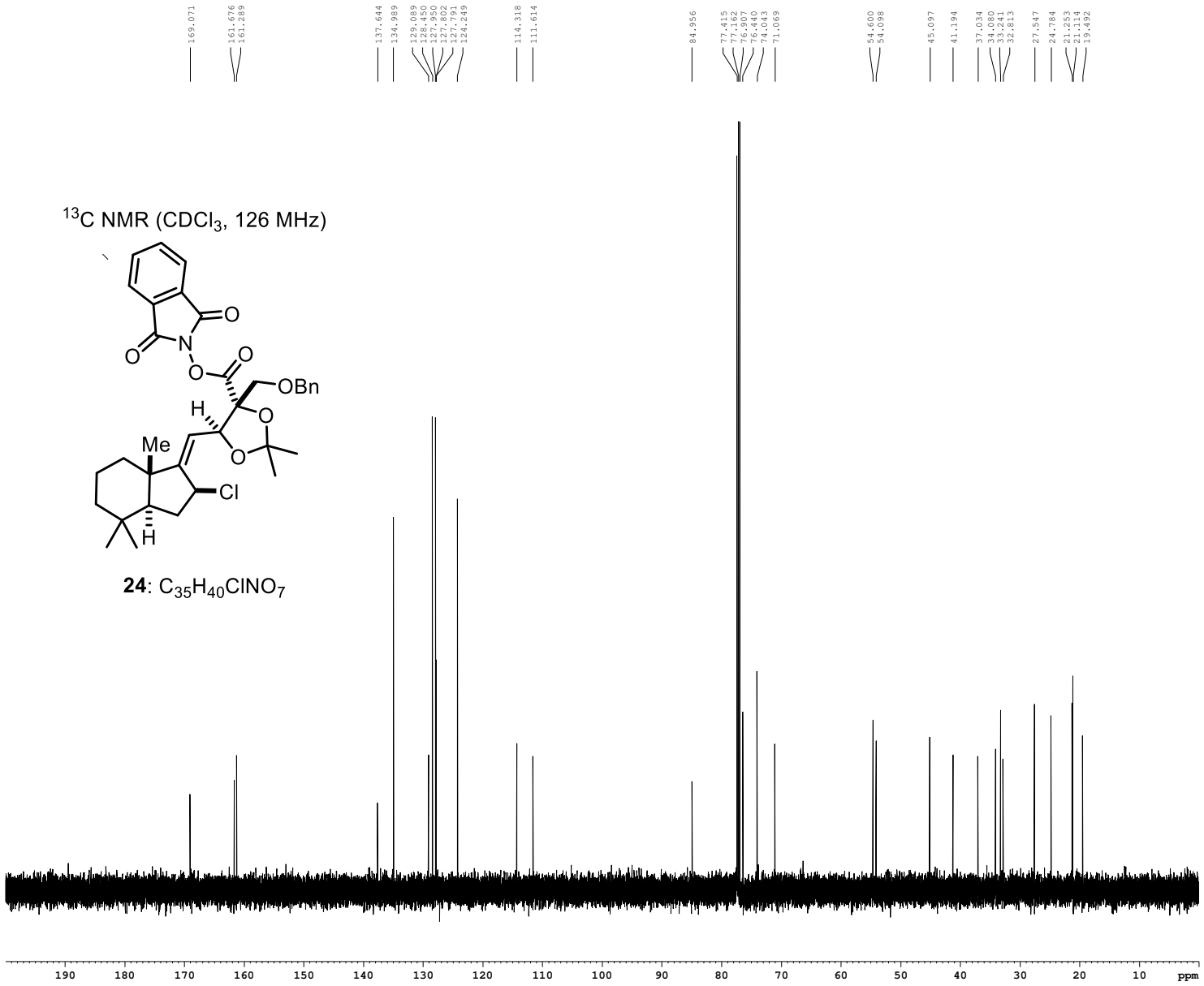
1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

24: $C_{35}H_{40}ClNO_7$

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

24: $C_{35}H_{40}ClNO_7$

```
Current Data Parameters
NAME            DJT-IV-271
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141206
Time                 11.49
INSTRUM              cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                    65536
SOLVENT               CDCl3
NS                      269
DS                       16
SWH              30303.031 Hz
FIDRES            0.462388 Hz
AQ               1.0813440 sec
RG                  7298.2
DW                 16.500 usec
DE                  6.00 usec
TE                  298.0 K
D1              0.25000000 sec
d11             0.03000000 sec
D16             0.00020000 sec
d17             0.00019600 sec
MCREST    0 sec
MCWRK           0.01500000 sec
P2                 33.10 usec

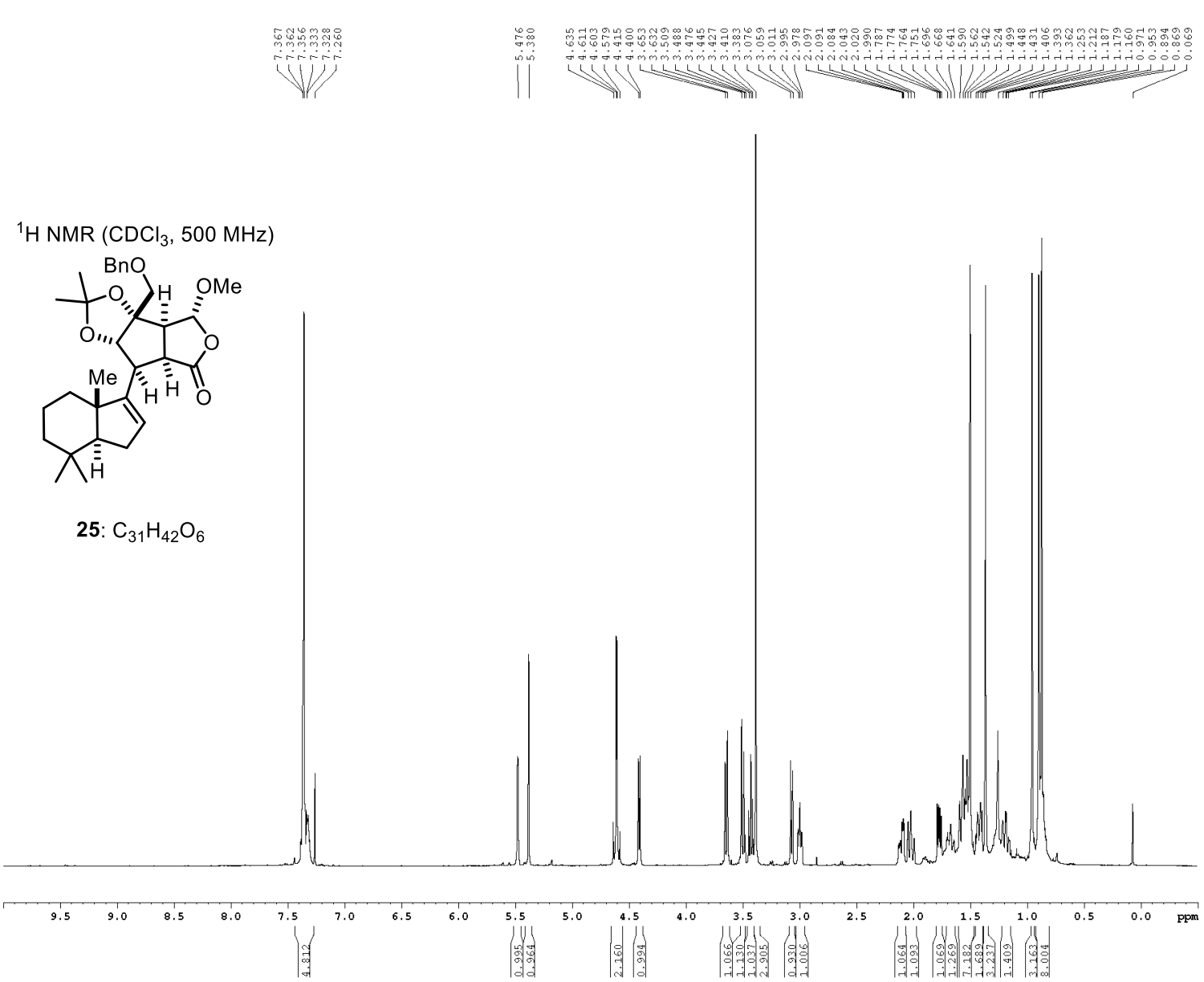
======== CHANNEL f1 ========
NUC1                  13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 2.70 dB
SP2                 2.70 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.50 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1               30.00 %
GPZ2               50.00 %
p15               500.00 usec
p16              1000.00 usec

F2 - Processing parameters
SI                    65536
SF           125.7804099 MHz
WDW                     no
SSB       0
LB        0 Hz
GB        0
PC                  2.00
```

1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

25: $C_{31}H_{42}O_6$

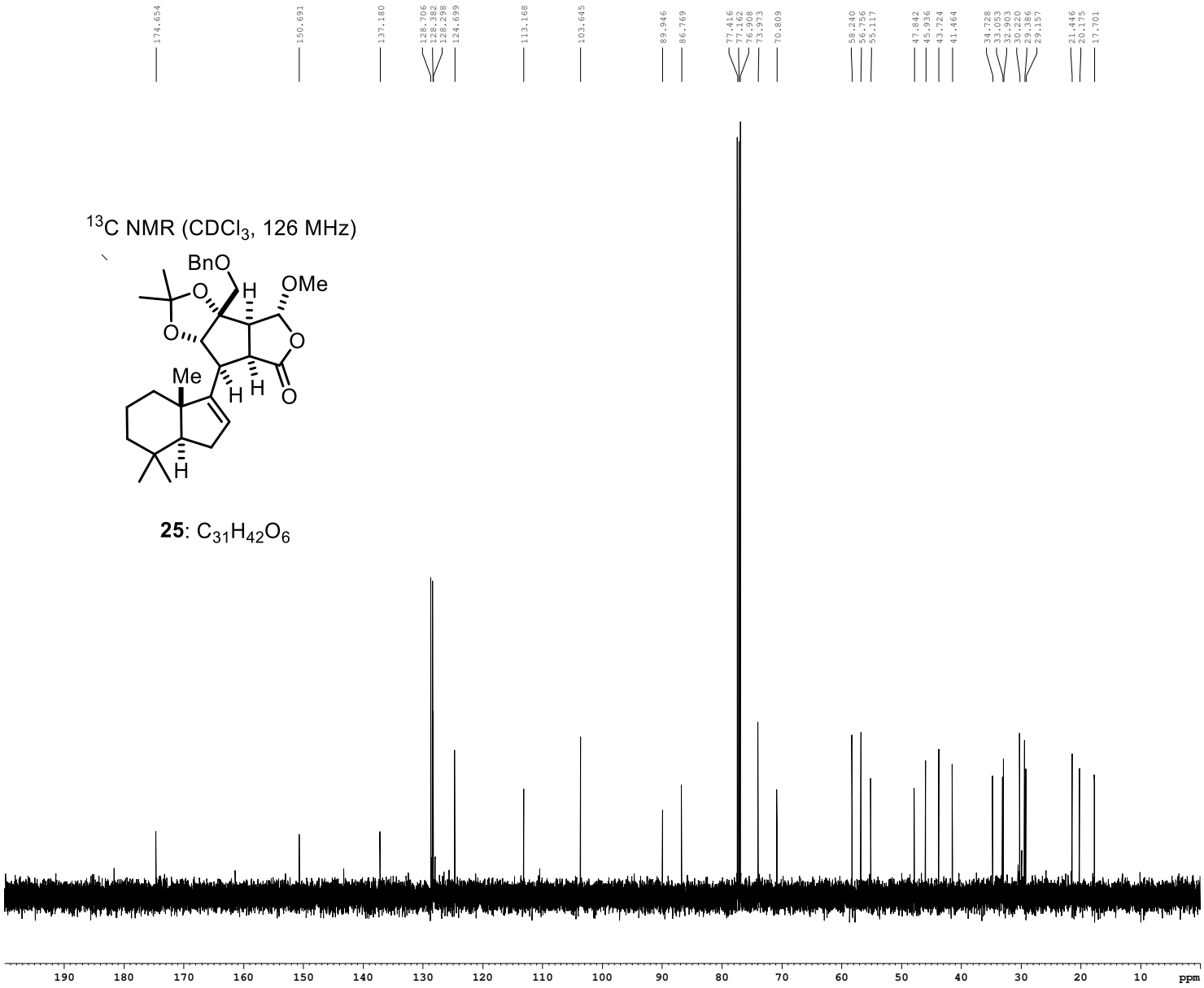
Current Data Parameters
NAME DJT-V-103
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150328
Time 14.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 16022
SOLVENT CDCl3
NS 30
DS 0
SWH 8012.820 Hz
FIDRES 0.500114 Hz
AQ 0.9997728 sec
RG 3.6
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200308 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

25: $C_{31}H_{42}O_6$

```
Current Data Parameters
NAME                 DJT-V-027
EXPNO                        6
PROCNO                       1

F2 - Acquisition Parameters
Date_               20150128
Time                     8.37
INSTRUM                cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                       65536
SOLVENT                  CDCl3
NS                         260
DS                          16
SWH                 30303.031 Hz
FIDRES                0.462388 Hz
AQ                  1.0813440 sec
RG                        6502
DW                      16.500 usec
DE                       6.00 usec
TE                      298.0 K
D1                  0.25000000 sec
d11                 0.03000000 sec
D16                 0.00020000 sec
d17                 0.00019600 sec
MCREST    0 sec
MCWRK               0.01500000 sec
P2                      33.10 usec

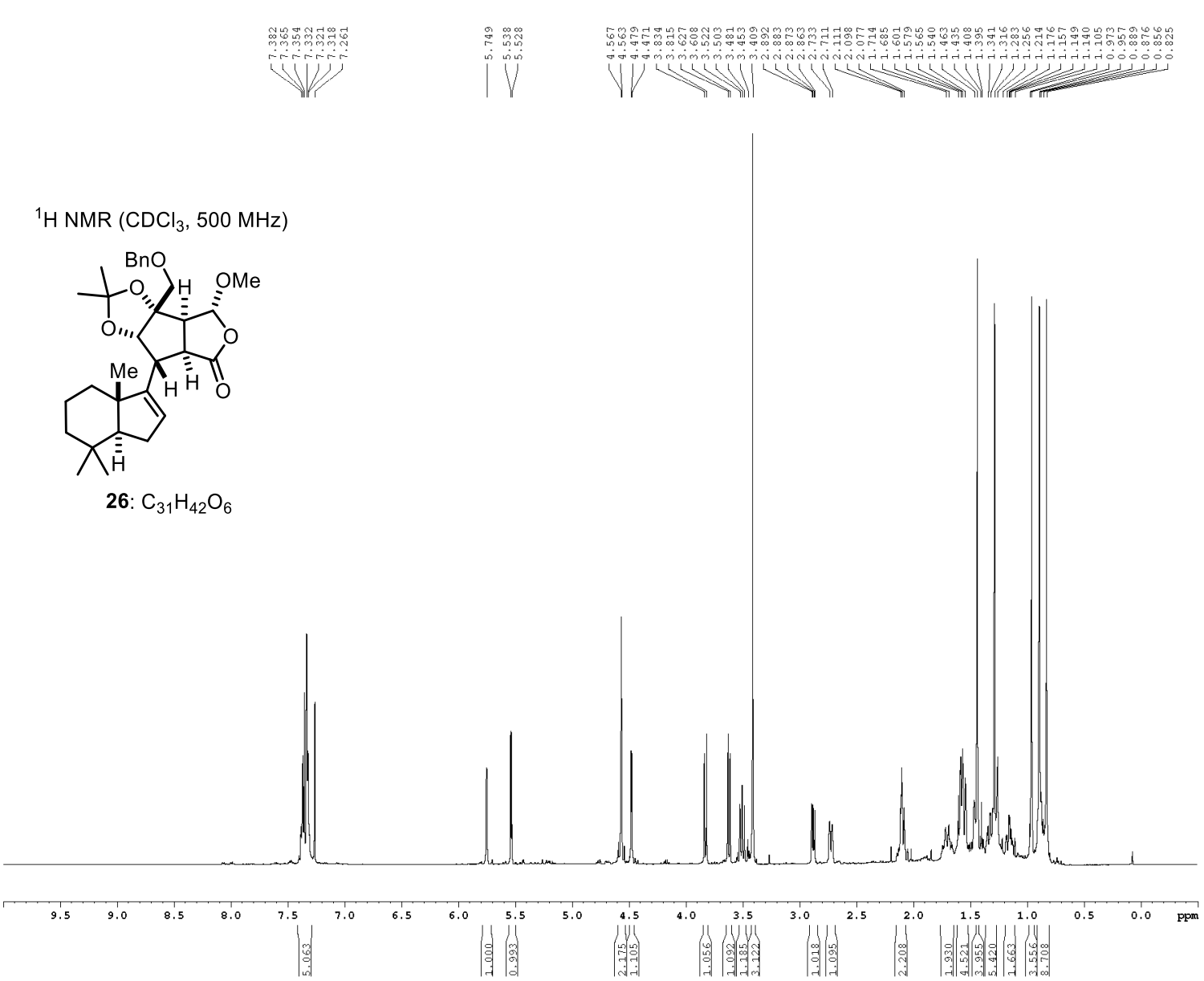
======== CHANNEL f1 ========
NUC1                       13C
P1                      16.55 usec
P11                    500.00 usec
P12                   2000.00 usec
PL0                    120.00 dB
PL1                     -1.00 dB
SFO1               125.7942548 MHz
SP1                      2.70 dB
SP2                      2.70 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                     1.60 dB
PL12                   24.50 dB
SFO2               500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                   30.00 %
GPZ2                   50.00 %
p15                   500.00 usec
p16                  1000.00 usec

F2 - Processing parameters
SI                       65536
SF                 125.7804076 MHz
WDW                         no
SSB       0
LB        0 Hz
GB        0
PC                        2.00
```

1H spectrum



^{1}H NMR ($CDCl_3$, 500 MHz)

26: $C_{31}H_{42}O_6$

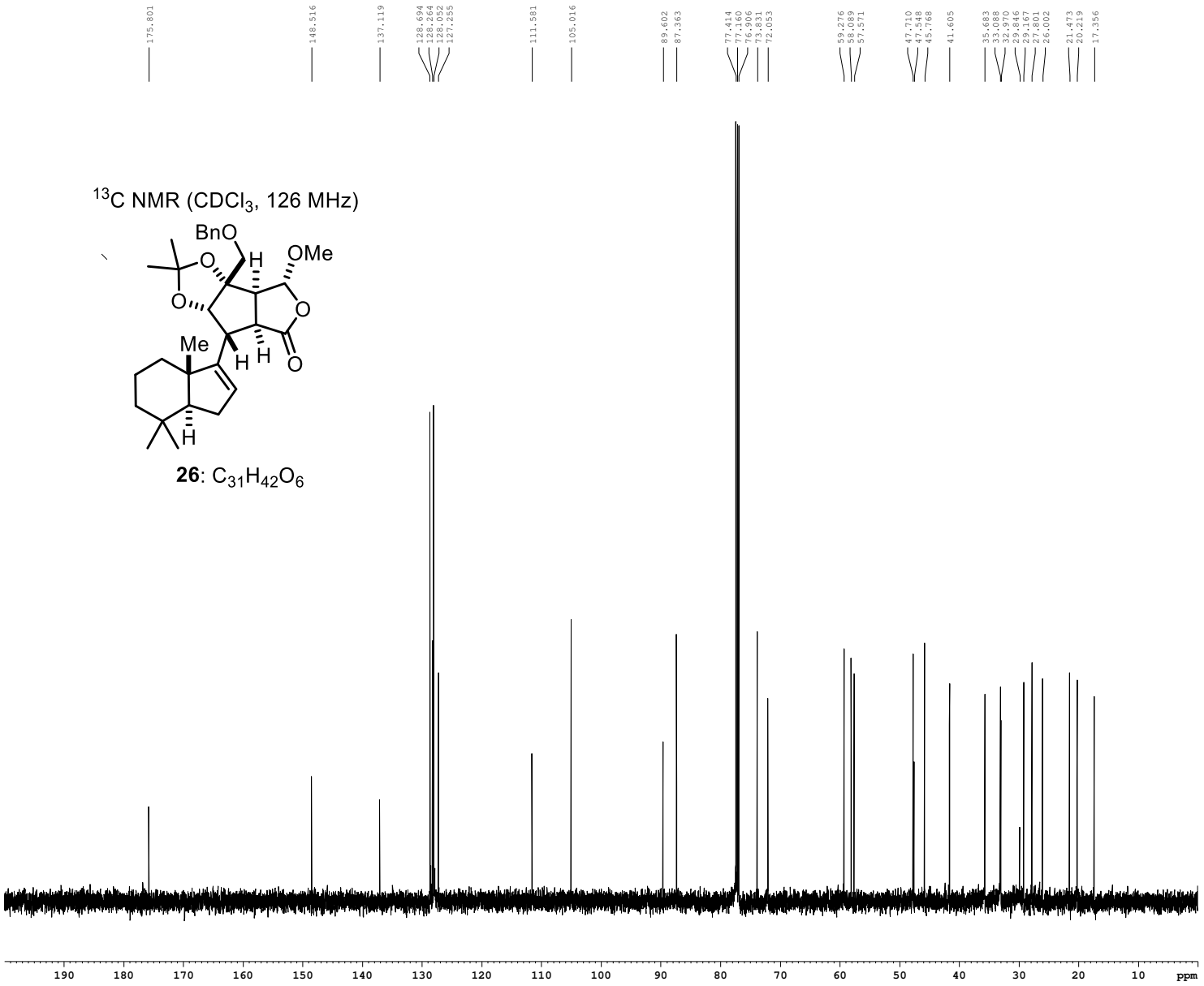
```
Current Data Parameters
NAME              DJT-V-103
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150328
Time                 14.19
INSTRUM              cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG                zg30
TD                    16022
SOLVENT               CDCl3
NS                       60
DS                        0
SWH                8012.820 Hz
FIDRES             0.500114 Hz
AQ                 0.9997728 sec
RG                        5
DW                  62.400 usec
DE                   6.00 usec
TE                  298.0 K
D1                 0.10000000 sec
MCREST    0 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    7.50 usec
PL1                   1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                       65536
SF            500.2200304 MHz
WDW                        EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    4.00
```

Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
NAME                  DJT-V-056
EXPNO                         6
PROCNO                        1

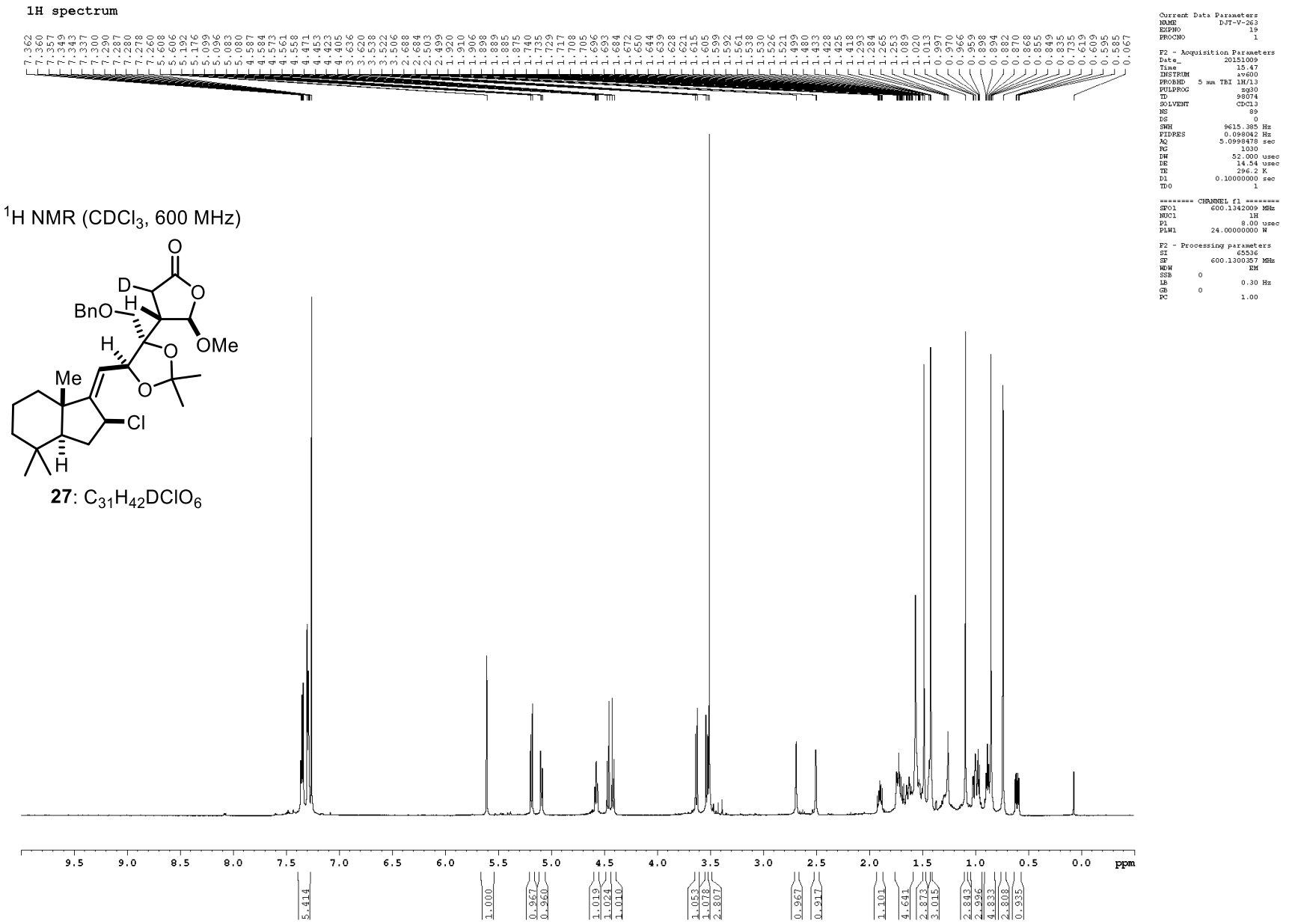
F2 - Acquisition Parameters
Date_                  20150225
Time                      16.33
INSTRUM                cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                         65536
SOLVENT                    CDCl3
NS                           135
DS                             0
SWH                  30303.031 Hz
FIDRES                0.462388 Hz
AQ                   1.0813440 sec
RG                          3251
DW                        16.500 usec
DE                         6.00 usec
TE                        298.0 K
D1                   0.25000000 sec
d11                  0.03000000 sec
D16                  0.00020000 sec
d17                  0.00019600 sec
MCREST    0 sec
MCWRK                0.01500000 sec
P2                        33.10 usec

======== CHANNEL f1 ========
NUC1                         13C
P1                        16.55 usec
P11                      500.00 usec
P12                     2000.00 usec
PL0                      120.00 dB
PL1                       -1.00 dB
SFO1                125.7942548 MHz
SP1                        2.70 dB
SP2                        2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

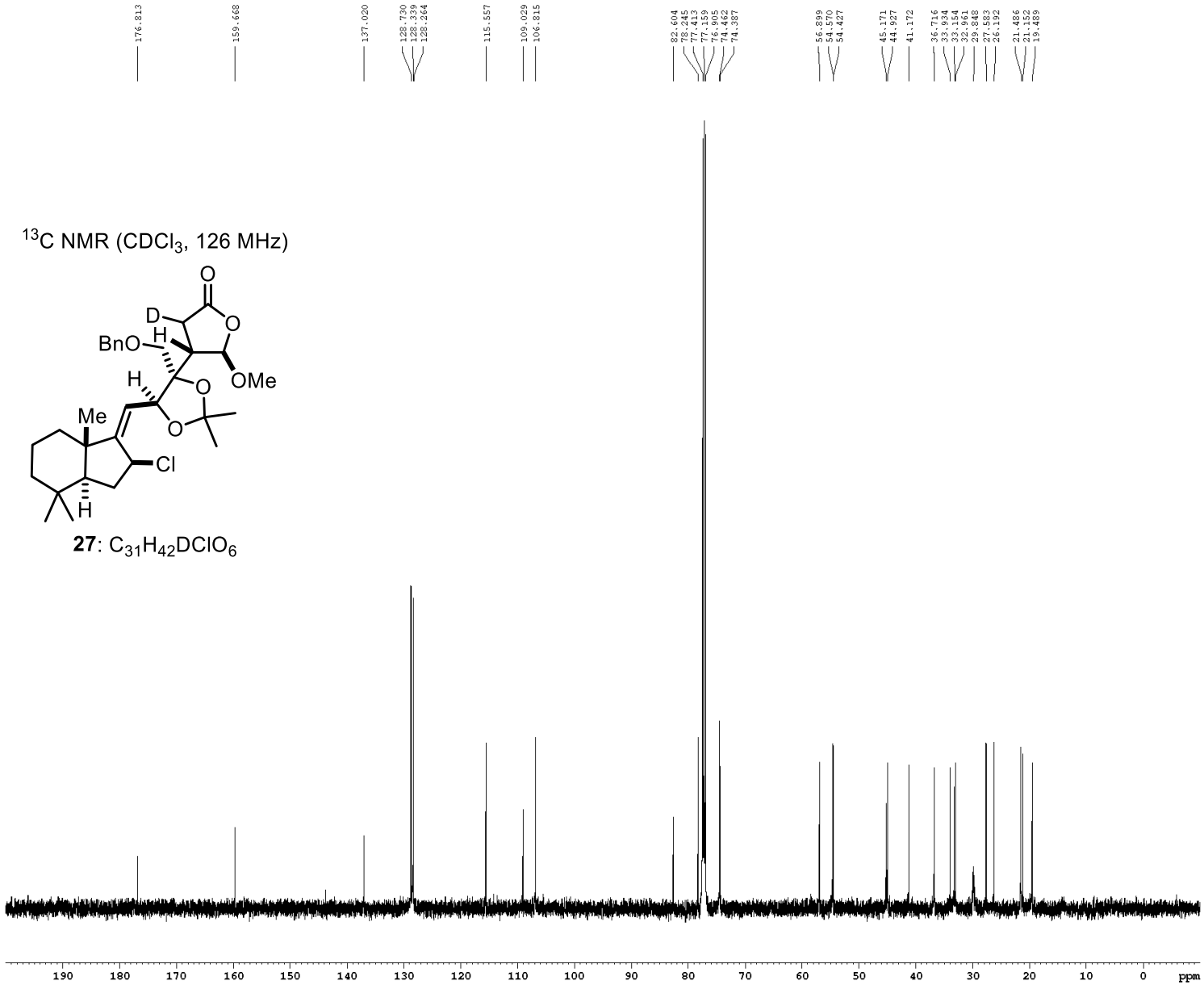
======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                         1H
PCPD2                   100.00 usec
PL2                       1.60 dB
PL12                     24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                     30.00 %
GPZ2                     50.00 %
p15                     500.00 usec
p16                    1000.00 usec

F2 - Processing parameters
SI                         65536
SF                  125.7804085 MHz
WDW                           EM
SSB       0
LB                        1.00 Hz
GB        0
PC                        2.00
```


1H spectrum
1H NMR (CDCl3, 600 MHz)
D
H
BnO
H
O
OMe
Me
O
Cl
H
27: C31H42DClO6
Current Data Parameters
NAME DJT-V-263
EXPNO 19
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151009
Time 15.47
INSTRUM av600
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 98074
SOLVENT CDCl3
NS 89
DS 0
SWH 9615.385 Hz
FIDRES 0.098042 Hz
AQ 5.0998478 sec
RG 1030
DW 52.000 usec
DE 14.54 usec
TE 296.2 K
D1 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1342009 MHz
NUC1 1H
P1 8.00 usec
PLW1 24.00000000 W
F2 - Processing parameters
SI 65536
SF 600.1300357 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

27: $C_{31}H_{42}DClO_6$

```
Current Data Parameters
NAME                  DJT-V-263
EXPNO                        21
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20151010
Time                       9.52
INSTRUM                 cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                         65536
SOLVENT                  CDCl3
NS                           842
DS                             0
SWH                  30303.031 Hz
FIDRES               0.462388 Hz
AQ                   1.0813440 sec
RG                      5792.6
DW                     16.500 usec
DE                      6.00 usec
TE                      298.0 K
D1                 0.25000000 sec
d11                0.03000000 sec
D16                0.00020000 sec
d17                0.00019600 sec
MCREST    0 sec
MCWRK              0.01500000 sec
P2                     33.10 usec

======== CHANNEL f1 ========
NUC1                      13C
P1                     16.55 usec
P11                   500.00 usec
P12                  2000.00 usec
PL0                   120.00 dB
PL1                    -1.00 dB
SFO1             125.7942548 MHz
SP1                     2.70 dB
SP2                     2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

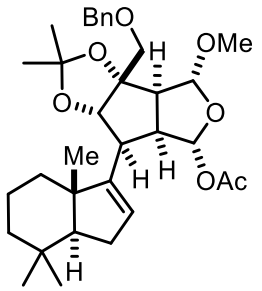
======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    1.60 dB
PL12                  24.50 dB
SFO2             500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                  30.00 %
GPZ2                  50.00 %
p15                  500.00 usec
p16                 1000.00 usec

F2 - Processing parameters
SI                         65536
SF               125.7804080 MHz
WDW                           EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     2.00
```

1H spectrum

^{1}H NMR ($CDCl_3$, 500 MHz)



S14: $C_{33}H_{46}O_7$

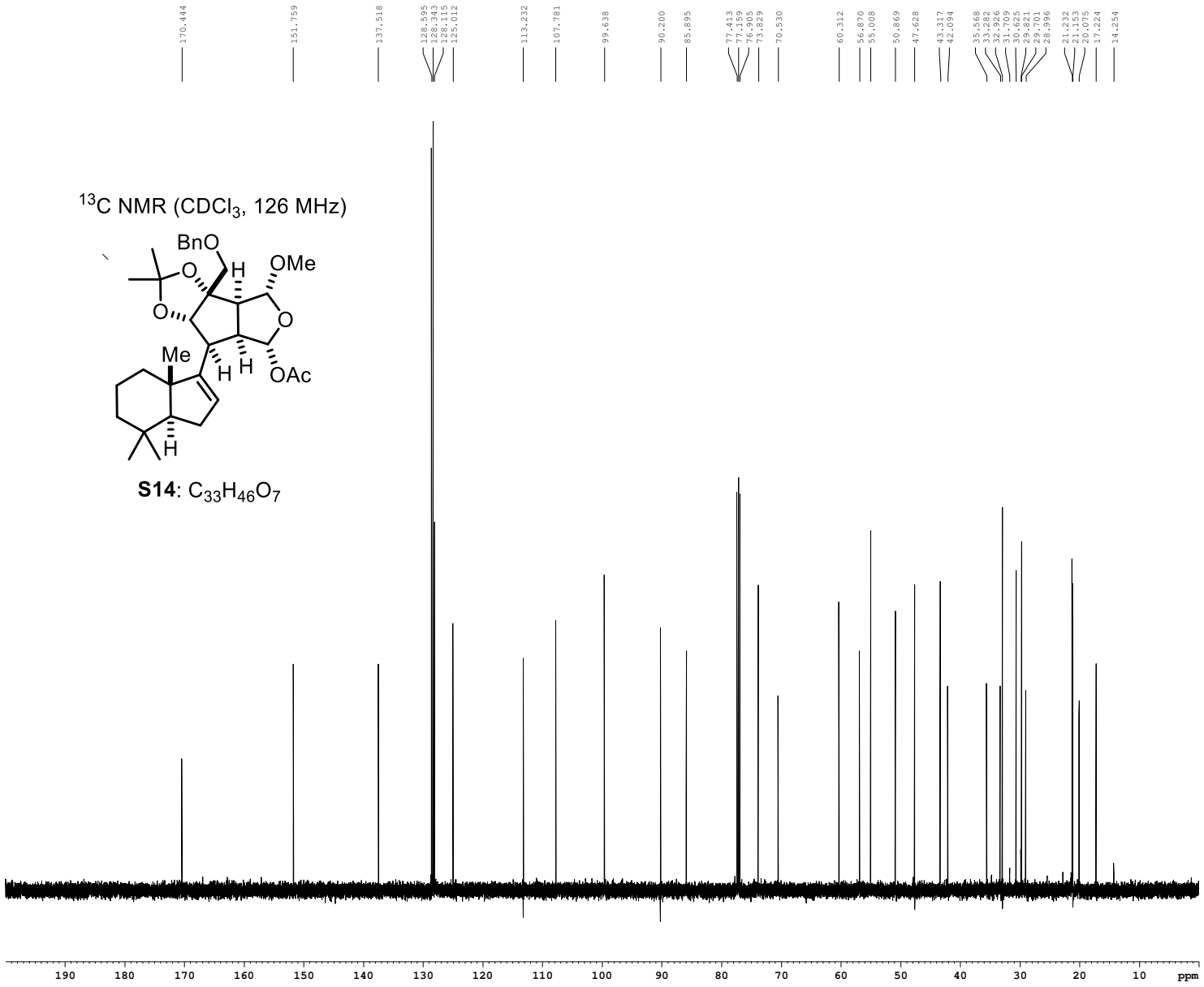
Current Data Parameters
NAME DJT-V-268
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150821
Time 11.02
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 20
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998273 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200313 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

S14: $C_{33}H_{46}O_7$

```
Current Data Parameters
NAME                  DJT-V-268
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150821
Time                      11.07
INSTRUM                cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                         65536
SOLVENT                   CDCl3
NS                          171
DS                            0
SWH                  30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                       5792.6
DW                       16.500 usec
DE                        6.00 usec
TE                       298.0 K
D1                    0.25000000 sec
d11                   0.03000000 sec
D16                   0.00020000 sec
d17                   0.00019600 sec
MCREST    0 sec
MCWRK                 0.01500000 sec
P2                       33.10 usec

======== CHANNEL f1 ========
NUC1                        13C
P1                       16.55 usec
P11                     500.00 usec
P12                    2000.00 usec
PL0                     120.00 dB
PL1                      -1.00 dB
SFO1                125.7942548 MHz
SP1                       2.70 dB
SP2                       2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

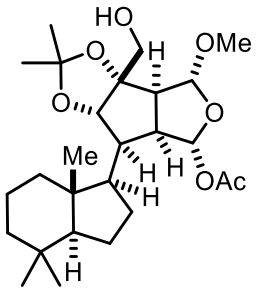
======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                      1.60 dB
PL12                    24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]             SINE.100
GPNAM[2]             SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                    30.00 %
GPZ2                    50.00 %
p15                    500.00 usec
p16                   1000.00 usec

F2 - Processing parameters
SI                         65536
SF                  125.7804108 MHz
WDW                           no
SSB       0
LB        0 Hz
GB        0
PC                        2.00
```

1H spectrum

^{1}H NMR ($CDCl_3$, 500 MHz)



29: $C_{26}H_{42}O_7$

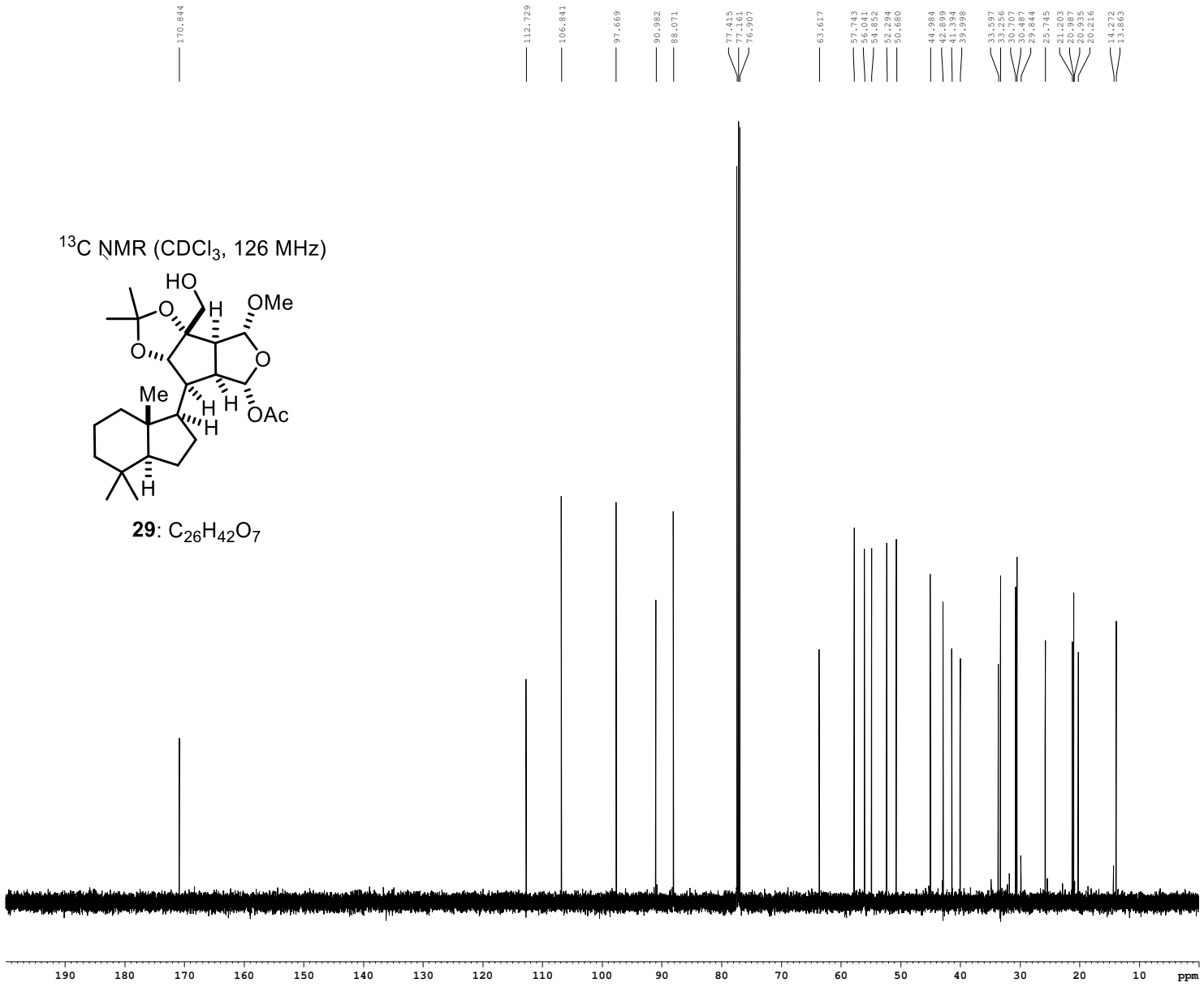

```
Current Data Parameters
NAME              DJT-V-270
EXPNO                     5
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150824
Time                  13.07
INSTRUM             cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG                zg30
TD                    81728
SOLVENT               CDCl3
NS                       20
DS                        0
SWH                8012.820 Hz
FIDRES             0.098043 Hz
AQ                 5.0998273 sec
RG                        5
DW                   62.400 usec
DE                    6.00 usec
TE                   298.0 K
D1                0.10000000 sec
MCREST    0 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    7.50 usec
PL1                   1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                        65536
SF            500.2200312 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    4.00
```

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

29: $C_{26}H_{42}O_7$

```
Current Data Parameters
NAME                  DJT-V-270
EXPNO                         6
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150824
Time                      13.11
INSTRUM                cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                         65536
SOLVENT                    CDCl3
NS                           498
DS                             0
SWH                  30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                         4096
DW                       16.500 usec
DE                        6.00 usec
TE                       298.0 K
D1                  0.25000000 sec
d11                 0.03000000 sec
D16                 0.00020000 sec
d17                 0.00019600 sec
MCREST    0 sec
MCWRK               0.01500000 sec
P2                       33.10 usec

======== CHANNEL f1 ========
NUC1                        13C
P1                       16.55 usec
P11                     500.00 usec
P12                    2000.00 usec
PL0                     120.00 dB
PL1                      -1.00 dB
SFO1                125.7942548 MHz
SP1                       2.70 dB
SP2                       2.70 dB
SPNAM[1]  Crp60,0.5,20.1
SPNAM[2]     Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

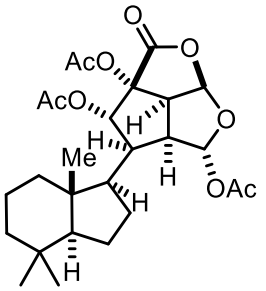
======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                   100.00 usec
PL2                       1.60 dB
PL12                     24.50 dB
SFO2                500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]               SINE.100
GPNAM[2]               SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                     30.00 %
GPZ2                     50.00 %
p15                     500.00 usec
p16                    1000.00 usec

F2 - Processing parameters
SI                         65536
SF                  125.7804080 MHz
WDW                           no
SSB       0
LB        0 Hz
GB        0
PC                        2.00
```

1H spectrum

^{1}H NMR ($CDCl_3$, 500 MHz)



chromodorolide B (**1**): $C_{26}H_{36}O_9$

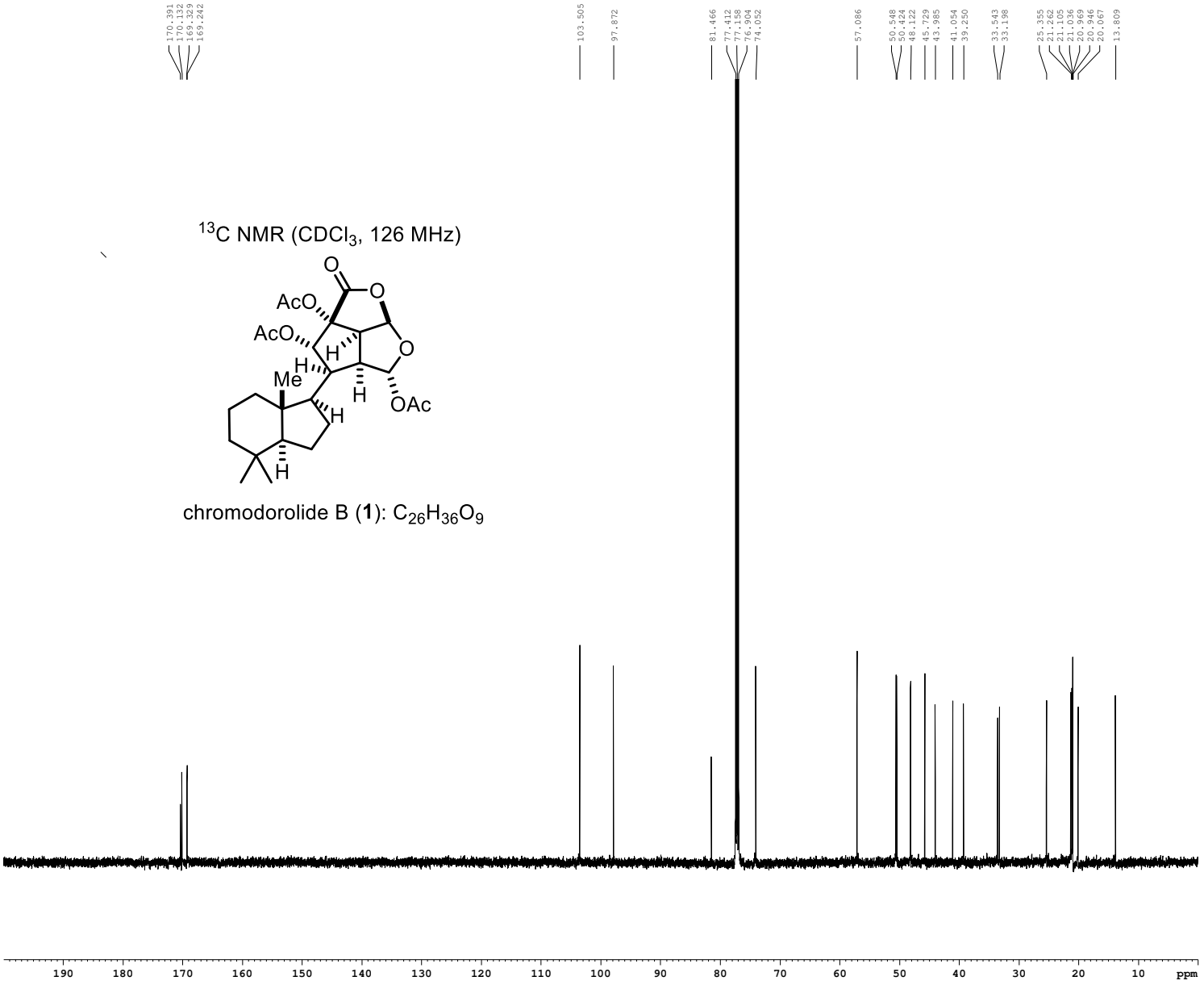

```
Current Data Parameters
NAME           DJT-V-272
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150907
Time                9.43
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                   140
DS                     0
SWH             8012.820 Hz
FIDRES             0.098043 Hz
AQ              5.0998273 sec
RG                     8
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1          0.10000000 sec
MCREST   0 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1                1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF           500.2200312 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 4.00
```

Z-restored spin-echo 13C spectrum with 1H decoupling



^{13}C NMR ($CDCl_3$, 126 MHz)

chromodorolide B (**1**): $C_{26}H_{36}O_9$

```
Current Data Parameters
NAME                 DJT-V-272
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_              20151012
Time                    7.21
INSTRUM              cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                      65536
SOLVENT                 CDCl3
NS                       2116
DS                          0
SWH                30303.031 Hz
FIDRES             0.462388 Hz
AQ                 1.0813440 sec
RG                    2298.8
DW                    16.500 usec
DE                     6.00 usec
TE                    298.0 K
D1                 0.25000000 sec
d11                0.03000000 sec
D16                0.00020000 sec
d17                0.00019600 sec
MCREST    0 sec
MCWRK              0.01500000 sec
P2                    33.10 usec

======== CHANNEL f1 ========
NUC1                      13C
P1                     16.55 usec
P11                   500.00 usec
P12                  2000.00 usec
PL0                   120.00 dB
PL1                    -1.00 dB
SFO1             125.7942548 MHz
SP1                    2.70 dB
SP2                    2.70 dB
SPNAM[1] Crp60,0.5,20.1
SPNAM[2]    Crp60comp.4
SPOFF1    0 Hz
SPOFF2    0 Hz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    1.60 dB
PL12                  24.50 dB
SFO2             500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPX1      0 %
GPX2      0 %
GPY1      0 %
GPY2      0 %
GPZ1                  30.00 %
GPZ2                  50.00 %
p15                  500.00 usec
p16                 1000.00 usec

F2 - Processing parameters
SI                      65536
SF             125.7804080 MHz
WDW                        EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     2.00
```