Four-step Synthesis of the Antimalarial Cardamom Peroxide via an Oxygen Stitching Strategy

Supporting Information

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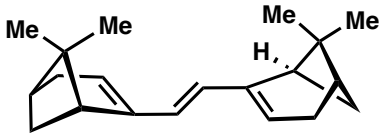
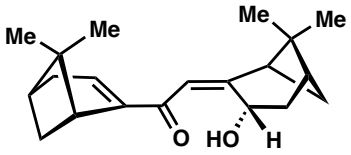
General Procedures

Unless otherwise stated, all reactions were performed in oven-dried or flame-dried glassware under an atmosphere of dry nitrogen or argon. Dry tetrahydrofuran (THF), dichloromethane, toluene, hexane, acetonitrile, and diethyl ether were obtained by passing these previously degassed solvents through activated alumina columns. Amines and alcohols were distilled from calcium hydride prior to use. 1,2-Dimethoxyethane (DME) was distilled from sodium and benzophenone. $TiCl_4$ was distilled prior to use. (1*R*)-(–)-Myrtenal was purchased from Sigma Aldrich and used directly without further purification. Reactions were monitored by thin layer chromatography (TLC) on Silicycle Siliaplate™ G TLC plates (250 μm thickness, 60 Å porosity, F-254 indicator) and visualized by UV irradiation and staining with *p*-anisaldehyde, phosphomolybdic acid, or potassium permanganate developing agents. Volatile solvents were removed under reduced pressure using a rotary evaporator. Flash column chromatography was performed using Silicycle F60 silica gel (60Å, 230-400 mesh, 40-63 μm). Proton nuclear magnetic resonance (^{1}H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on Bruker AVQ-400, AV-500, or AV-600 spectrometers operating respectively at 400, 500, and 600 MHz for ^{1}H, and 100, 125, and 150 MHz for ^{13}C. Chemical shifts are reported in parts per million (ppm) with respect to the residual solvent signal $CDCl_3$ (^{1}H NMR: $\delta = 7.26$; ^{13}C NMR: $\delta = 77.16$). Peak multiplicities are reported as follows: s = singlet, bs = broad singlet, d = doublet, t = triplet, dd = doublet of doublets, td = triplet of doublets, m = multiplet. app = apparent. Melting points were determined using MEl-TEMP™ apparatus and are uncorrected. IR spectra were recorded on a Nicolet 380 FT-IR spectrometer. High-resolution mass spectra (HRMS) were obtained by the qb3 mass spectrometry facility at the University of California, Berkeley. X-ray crystallographic analyses were performed at the UC-Berkeley College of Chemistry X-ray crystallography facility.



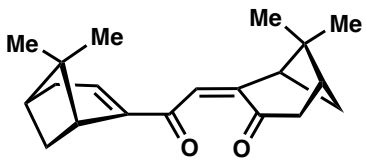
Triene 5. The procedure was adapted from previous conditions reported by McMurry and co-workers (*J. Org. Chem.* **1978**, *43*, 3255.)

A flame-dried 1 L three-necked flask equipped with a large stir bar and reflux condenser was charged with freshly prepared Zn-Cu couple (44 g, 0.67 mol, 40 equiv). The system was evacuated and backfilled with argon three times. DME (600 mL) was added, followed by the dropwise addition of freshly distilled $TiCl_4$ (24 ml, 200 mmol, 10 equiv.) to the rapidly stirring slurry. After 1 hour of sonication, the mixture was heated to reflux for 5 hours, cooled to room temperature, and a solution of myrtenal (3.0 g, 20 mmol, 1 equiv) in 50 mL of degassed DME was added slowly over 12 hours via syringe pump. After 1 hour of additional sonication, the reaction mixture was heated at reflux for 48 hours, cooled to room temperature, and filtered through a pad of Florisil® and eluted with diethyl ether. This filtration was repeated to give a clear solution that was concentrated *in vacuo* and purified by flash column chromatography (200:1 hexanes:Et_3N) affording **5** (1.41 g, 53 % yield) as colorless oil: $[\alpha]^{20}_D$ = +31.70° (c 0.010 g/ml, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.14 (s, 2H), 5.53 (m, 2H), 2.62 (ddd, *J* = 5.7, 5.7, 1.6 Hz, 2H), 2.42 (ddd, *J* = 8.8, 5.7, 5.7 Hz, 2H), 2.39 (ddd, *J* = 19.2, 3.0, 3.0 Hz, 2H), 2.32 (ddd, *J* = 19.2, 2.7, 2.7 Hz, 2H), 2.12 (m, 2H), 1.33 (s, 6H), 1.13 (d, *J* = 8.8 Hz, 2H), 0.81 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 146.9, 126.4, 123.7, 41.3, 41.2, 37.9, 32.2, 31.6, 26.6, 21.0; IR (thin film, cm^{-1}) 2931, 2360, 1705, 1650, 1628, 1369, 1331; HRMS (EI) calcd. for $[C_{20}H_{28}]$: *m/z* 268.2191, found 268.2191.

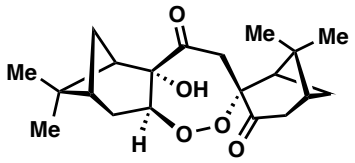


Dienone 7. A flame-dried round bottom flask was charged with triene **5** (1.3 g, 4.84 mmol, 1 equiv) and DCM (145 ml). The solution was cooled to –40 °C and methylene blue (15 mg, 0.05 mmol, 0.01 equiv) in DCM (1 mL) added. Oxygen gas was vigorously bubbled through the solution while irradiated by a 500W halogen lamp. After 1 hour, a second portion of methylene blue (15 mg) in DCM (1 mL) was added and the reaction continued until TLC indicated complete consumption of starting material (~2 hour). Nitrogen was then bubbled through the solution for 30 minutes at which point DBU (3.7 mL, 24 mmol, 5 equiv) was added dropwise at -

40 °C. The resulting mixture was allowed to gradually warm to -20 °C and stirred for an additional 4 hours at this temperature. The reaction mixture was quenched by the additions of 1 N HCl (100 mL) and extracted by DCM (3 X 75 mL). The combined organic layers were washed with sat. $NaHCO_3$, H_2O, and brine, dried over $MgSO_4$, and concentrated *in vacuo*. The crude material was purified by column chromatography (gradient 20:1 → 10:1 hexanes/ethyl acetate) to afford **7** (816 mg, 56 %) as white solid: mp 118.4 − 119.6 °C; $[\alpha]^{20}_D$ = +246.02° (c 0.005 g/ml, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.76 (dddd, *J* = 3.3, 3.3, 1.5, 1.5 Hz, 1H), 6.40 (d, *J* = 0.8 Hz, 1H), 5.11 (d, *J* = 3.0 Hz, 1H, D_2O exchangeable), 4.66 – 4.62 (m, 1H), 2.94 (ddd, *J* = 5.7, 5.7, 1.5 Hz, 1H), 2.56 – 2.50 (m, 2H), 2.49 – 2.40 (m, 3H), 2.38 – 2.31 (m, 1H), 2.16 – 2.11 (m, 1H), 2.07 (dddd, *J* = 6.0, 6.0, 6.0, 2.2 Hz, 1H), 1.95 (dd, *J* = 14.6, 3.6 Hz, 1H), 1.72 (d, *J* = 10.0 Hz, 1H), 1.34 (s, 3H), 1.31 (s, 3H), 1.07 (d, *J* = 9.2 Hz, 1H), 0.75 (s, 3H), 0.69 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.2, 168.3, 150.6, 137.3, 120.4, 62.9, 53.5, 40.8, 40.3, 40.2, 40.0, 37.5, 33.8, 32.7, 31.2, 27.4, 26.0, 25.9, 22.7, 21.0; IR (thin film, cm^{-1}) 3421, 2916, 1636, 1591, 1393, 1366; HRMS (EI) calcd. for $[C_{20}H_{28}O_2]$: *m/z* 300.2089, found 300.2094.



Dienedione 8. Dienone **7** (816 mg, 2.72 mmol, 1.0 equiv) was dissolved in DCM (27 mL) and $NaHCO_3$ (228 mg, 2.72 mmol, 1.0 equiv) and the Dess-Martin periodinane (1.5 g, 3.54 mmol, 1.3 equiv) were added sequentially. The resulting mixture was stirred at room temperature for 1 h (monitored by TLC for complete consumption of **7**). The reaction was quenched by the addition of 1 N NaOH (10 mL) and extracted with DCM (3 X 50 mL). The combined organic phases were washed with water and brine, dried over $MgSO_4$, and purified by flash column chromatography (gradient 20:1 → 10:1 hexanes/ethyl acetate), affording **10** (770 mg, 95 %) as white solid: mp 137.1 − 138.4 °C; $[\alpha]^{20}_D$ +27.20° (c 0.005 g/ml, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.55 (ddd, *J* = 3.4, 1.8, 1.8 Hz, 1H), 6.00 (s, 1H), 3.05 (dd, *J* = 5.5, 5.5 Hz, 1H), 2.77 – 2.68 (m, 2H), 2.64 (ddd, *J* = 19.2, 2.4, 2.4 Hz, 1H), 2.54 – 2.41 (m, 3H), 2.39 (ddd, *J* = 19.9, 2.9, 2.9 Hz, 1H), 2.21 (dddd, *J* = 6.1, 6.1, 2.8, 2.8 Hz, 1H), 2.15 – 2.10 (m, 1H), 1.42 (dd, *J* = 2.9, 2.9 Hz, 1H), 1.40 (s, 3H), 1.35 (s, 3H), 1.08 (d, J = 9.1 Hz, 1H), 0.91 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.4, 195.0, 149.7, 145.0, 138.3, 132.8, 48.9, 42.6, 41.3, 40.8, 39.3, 38.4, 37.9, 32.7, 32.3, 31.2, 26.0, 26.0, 22.0, 21.0; IR (thin film, cm^{-1}) 2931, 1705, 1651, 1628, 1465, 1421, 1368; HRMS (EI) calcd. for $[C_{20}H_{26}O_2]$: *m/z* 298.1933, found 298.1937.



Cardamom Peroxide (**2**): A flame-dried round bottom flask was charged with dienedione **8** (30 mg, 0.1 mmol, 1.0 equiv), $Mn(dpm)_3$ (12 mg, 0.02 mmol, 20 mol %), DCM (1.6 mL), and *i*PrOH (0.4 mL). Oxygen was vigorously bubbled through the solution for 5 minutes, followed by the addition of TBHP (5M in decane, 30 μL, 1.5 equiv). The solution was cooled to -10 °C under an atmosphere of oxygen, and $PhSiH_3$ (30 μL, 0.24 mmol, 2.4 equiv) in DCM (1 mL) was added dropwise over 12 h via syringe pump. After the addition was complete, a solution of triphenylphosphine (56 mg, 0.21 mmol) in DCM was added dropwise at -10 °C to quench intermediate diperoxide **3**. The reaction mixture was diluted with H_2O (5 mL) and extracted with DCM (3 x 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered through Celite®, and concentrated *in vacuo*. The crude mixture was purified by flash column chromatography (DCM) to afford the cardamom peroxide **2** (18.2 mg, 52 %) as a white solid along with hydration product **9** (3.5 mg, 11%), diol **10** (4.4 mg, 13%), and nopinone (9% GC yield using an internal standard of dodecane). [Note: the reaction afforded 48% of **2** on a 150 mg scale].

Cardamom peroxide (2): white solid: mp 154.9 − 156.2 °C; $[\alpha]^{20}_D$ +123.20° (c 0.005 g/ml, hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.28 (s, 1H), 4.22 (d, *J* = 11.3 Hz, 1H), 4.18 (d, *J* = 8.7 Hz, 1H), 2.78 (dd, *J* = 18.9, 2.4 Hz, 1H), 2.69 (ddd, *J* = 18.9, 3.0, 3.0 Hz, 1H), 2.58 (dd, *J* = 15.4, 8.9 Hz, 1H), 2.46 (dddd, *J* = 11.0, 5.8, 2.9, 2.9 Hz, 2H), 2.40 (d, *J* = 11.2 Hz, 1H), 2.41 – 2.35 (m, 1H), 2.30 (dd, *J* = 6.1, 6.1 Hz, 1H), 2.15 – 2.05 (m, 2H), 1.97 – 1.92 (m, 1H), 1.84 – 1.74 (m, 2H), 1.69 (d, *J* = 11.2 Hz, 1H), 1.38 (s, 3H), 1.27 (s, 3H), 1.06 (s, 3H), 0.86 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.8, 204.5, 84.3, 84.2, 83.3, 49.6, 43.9, 43.3, 42.9, 41.0, 40.7, 39.0, 38.3, 30.7, 27.9, 27.5, 26.9, 26.6, 24.1, 22.5; IR (thin film, cm^{-1}) 3447, 3021, 2909, 1722, 1692, 1441, 1406, 1371; HRMS (ESI) calcd. for $[C_{20}H_{28}O_5Na]^+$ $(M+Na)^+$: *m/z* 371.1829, found 371.1837.

Vapor diffusion of an ether solution of **2** with pentane afforded X-ray quality crystals.

Hydration product 9: colorless oil; $[\alpha]^{20}_D$ = -56.7° (c 0.006 g/ml, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (ddd, *J* = 3.4, 1.8, 1.8 Hz, 1H), 3.38 (d, *J* = 15.7 Hz, 1H), 2.99 (ddd, *J* = 5.7, 5.7, 1.6 Hz, 1H), 2.68 (dd, *J* = 19.0, 2.4 Hz, 1H), 2.59 (ddd, *J* = 19.0, 3.2, 3.2 Hz, 1H), 2.53 (dt, *J* = 20.2, 3.2, 3.2 Hz, 1H), 2.50 – 2.41 (m, 3H), 2.39 (d, *J* = 15.8 Hz, 1H), 2.22 (dd, *J* = 6.2, 6.2 Hz, 1H), 2.15 (dddd, *J* = 5.9, 3.0, 3.0, 1.2 Hz, 1H), 2.11 (dddd, *J* = 6.1, 6.1, 3.0, 3.0 Hz, 1H), 1.92 (d,

J = 11.0 Hz, 1H), 1.36 (s, 3H), 1.35 (s, 3H), 1.02 (d, J = 9.1 Hz, 1H), 0.95 (s, 3H), 0.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.6, 200.6, 150.4, 138.8, 79.8, 50.0, 43.3, 40.4, 40.3, 39.6, 39.3, 38.6, 37.6, 32.9, 31.2, 27.9, 27.4, 26.0, 22.9, 21.0; IR (thin film, cm^{-1}) 3359, 2924, 1721, 1640, 1613, 1421, 1370; HRMS (EI) calcd. for $[C_{20}H_{28}O_3]$: *m/z* 316.2038, found 316.2040.

Diol 10: white solid (acid sensitive): mp 132.9 – 134.1 °C; $[\alpha]^{20}_D$ −33.2° (c 0.010 g/ml, CH_2Cl_2); ^{1}H NMR (600 MHz, CD_2Cl_2) δ 5.49 (s, 1H), 3.37 (d, *J* = 15.3 Hz, 1H), 2.78 (ddd, *J* = 15.8, 10.5, 3.5 Hz, 1H), 2.69 – 2.62 (m, 2H), 2.57 (ddd, *J* = 19.1, 3.2, 3.2 Hz, 1H), 2.41 (dddd, *J* = 11.0, 6.2, 6.2, 3.1 Hz, 1H), 2.26 (dddd, *J* = 10.1, 6.0, 6.0, 1.8 Hz, 1H), 2.21 (dd, *J* = 6.1, 4.8 Hz, 1H), 2.18 – 2.14 (m, 2H), 2.08 (dddd, *J* = 6.1, 6.1, 2.9, 2.9 Hz, 1H), 1.99 – 1.90 (m, 2H), 1.85 (d, *J* = 10.9 Hz, 1H), 1.87 – 1.81 (m, 1H), 1.61 (ddd, *J* = 16.0, 11.0, 5.2 Hz, 1H), 1.58 (d, *J* = 10.1 Hz, 1H), 1.35 (s, 3H), 1.22 (s, 3H), 0.90 (s, 3H), 0.72 (s, 3H); ^{13}C NMR (150 MHz, CD_2Cl_2) δ 213.2, 213.1, 82.5, 80.4, 50.7, 49.0, 43.6, 41.7, 41.1, 39.7, 39.0, 38.3, 28.0, 27.5, 27.3, 26.2, 24.6, 24.3, 22.8, 22.0; IR (thin film, cm^{-1}) 3410, 3005, 2925, 2360, 2341, 1823, 1708, 1463, 1408, 1326; HRMS (ESI) calcd. for $[C_{20}H_{30}O_4Na]^+$ $(M+Na)^+$: *m/z* 357.2036, found 357.2035.

Reductive activation of 2:

In a nitrogen-filled glovebox, **2** (12 mg, 0.03 mmol, 1.0 equiv) was dissolved in thoroughly degassed acetonitrile (750 μL) and H_2O (7 μL, 0.4 mmol, 13.0 equiv). To the resulting solution was added $FeCl_2$ (3.5 mg, 0.028 mmol, 0.8 equiv) in one portion and the resulting mixture stirred for 45 minutes. After 45 minutes, the resulting red colored solution was removed from the glovebox, opened to the air, diluted with DCM (15 mL) and washed with brine (2 X 25 mL). The organic phase was concentrated *in vacuo* and the crude material purified by preparative thin layer chromatography (50:6 DCM:MeOH) affording acids **11** (5 mg, yield 42 %), **12** (2.5 mg, yield 20 %), and small amount of **13** (~ 0.5 − 1.0 mg, 5 − 10 %).

Acid 11: white solid: mp 147.5 – 149.8 °C; $[\alpha]^{20}_D$ +77.50° (c 0.002 g/ml, CH_2Cl_2); ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.30 (s, 1H), 4.50 (dd, *J* = 9.7, 4.3 Hz, 1H), 2.95 (dd, *J* = 10.6, 7.7 Hz, 1H), 2.73 (dddd, *J* = 14.8, 9.8, 2.8, 2.8 Hz, 1H), 2.49 (dddd, *J* = 10.3, 7.8, 7.8, 7.7 Hz, 1H), 2.43 (dd, *J* = 15.8, 7.2 Hz, 1H), 2.34 (dd, *J* = 15.8, 8.0 Hz, 1H), 2.30 (ddd, *J* = 11.3, 7.8, 7.8 Hz, 1H), 2.26 (ddddd, *J* = 11.9, 6.2, 3.0, 3.0, 2.3 Hz, 1H), 2.15 (d, *J* = 11.3 Hz, 1H), 2.03 – 1.97 (m, 2H), 1.83 – 1.74 (m, 2H), 1.27 (s, 3H), 1.25 (s, 3H), 1.03 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.0, 192.5, 176.9, 102.8, 96.5, 67.9, 50.0, 44.3, 43.4, 39.9, 39.5, 38.7, 38.6, 34.8,

30.4, 26.8, 25.5, 24.4, 23.6, 17.5; IR (thin film, cm^{-1}) 3849, 3105, 2997, 2924, 2361, 1682, 1587, 1484, 1368; HRMS (ESI) calcd. for $[C_{20}H_{27}O_5]^-$ $(M - H)^-$: *m/z* 347.1864, found 347.1861.

Vapor diffusion of an ether solution of **11** with pentane afforded X-ray quality crystals.

Acid 13: white solid: mp 154.3 – 156.0 °C; ^1H NMR (600 MHz, CD_2Cl_2) δ 5.27 (s, 1H), 4.98 (dd, *J* = 9.1, 3.9 Hz, 1H), 2.77 – 2.71 (m, 1H), 2.71 (dd, *J* = 10.8, 7.6 Hz, 1H), 2.43 – 2.35 (m, 2H), 2.34 – 2.25 (m, 2H), 2.22 (dd, *J* = 5.8, 5.8 Hz, 1H), 2.14 (ddd, *J* = 11.0, 7.5, 7.5 Hz, 1H), 1.99 (dddd, *J* = 5.7, 5.7, 3.3, 3.3 Hz, 1H), 1.90 (ddd, *J* = 14.6, 3.8, 3.8 Hz, 1H), 1.79 (dd, *J* = 10.6, 10.6 Hz, 1H), 1.27 (s, 3H), 1.22 (s, 3H), 1.10 (ddd, *J* = 6.1, 3.8, 3.8 Hz, 1H), 1.05 (s, 3H), 0.95 (s, 3H); IR (thin film, cm^{-1}) 3457, 2997, 2923, 2667, 2365, 1677, 1591, 1367; HRMS (ESI) calcd. for $[C_{20}H_{27}O_5]^-$ $(M - H)^-$: *m/z* 347.1864, found 347.1863.

Vapor diffusion of an ether solution of **13** with pentane afforded X-ray quality crystals.

Acid 12: white foam (unstable over extended periods in $CDCl_3$, converts to **11** and **13**); $[\alpha]^{20}_D$ +48.0° (c 0.001 g/ml, CH_2Cl_2); ^1H NMR (600 MHz, CD_2Cl_2) δ 5.25 (d, *J* = 0.8 Hz, 1H), 4.48 – 4.40 (m, 1H), 2.98 (ddd, *J* = 10.4, 7.6, 0.8 Hz, 1H), 2.91 (d, *J* = 11.0 Hz, 1H), 2.83 (dd, *J* = 18.1, 6.7 Hz, 1H), 2.73 (dd, *J* = 18.1, 7.9 Hz, 1H), 2.73 – 2.68 (m, 1H), 2.50 – 2.43 (m, 1H), 2.26 (ddd, *J* = 11.1, 7.9, 7.9 Hz, 1H), 2.26 – 2.21 (m, 1H), 2.10 (d, *J* = 11.2 Hz, 1H), 2.02 – 1.96 (m, 2H), 1.80 – 1.69 (m, 2H), 1.26 (s, 3H), 1.24 (s, 3H), 1.02 (s, 3H), 0.89 (s, 3H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 206.0, 199.0, 192.8, 102.9, 96.7, 68.2, 50.3, 44.7, 43.9, 40.3, 40.0, 39.0, 38.0, 37.4, 30.6, 26.9, 25.8, 24.7, 23.7, 18.0; IR (thin film, cm^{-1}) 3061, 2926, 2349, 1724, 1598, 1548, 1484, 1379; HRMS (ESI) calcd. for $[C_{20}H_{27}O_5]^-$ $(M - H_2O - H)^-$: *m/z* 347.1864, found 347.1863.

X-Ray crystallographic Analysis of 2

A colorless needle 0.060 x 0.030 x 0.020 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 10 seconds per frame using a scan width of 1.0°. Data collection was 100.0% complete to 67.000° in θ. A total of 25275 reflections were collected covering the indices, -7<=*h*<=7, -15<=*k*<=14, -25<=*l*<=26. 3253 reflections were found to be symmetry independent, with an R_{int} of 0.0218. Indexing and unit cell refinement indicated a primitive, orthorhombic lattice. The space group was found to be P 21 21 21 (No. 19). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by iterative methods (SHELXT) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2013). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2013. Absolute stereochemistry was unambiguously determined to be *R* at C3, C5, C9, C12 and C14, and *S* at C1 and C6, respectively.

Table 1. Crystal data and structure refinement for **2**.

X-ray ID	maimone18	
Sample/notebook ID	XH_NP_1	
Empirical formula	C20 H28 O5	
Formula weight	348.42	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 6.4066(3) Å	α= 90°.
	b = 12.6083(6) Å	β= 90°.
	c = 22.0227(11) Å	γ = 90°.
Volume	1778.91(15) $Å^3$	
Z	4	
Density (calculated)	1.301 Mg/m^3	
Absorption coefficient	0.750 mm^{-1}	
F(000)	752	
Crystal size	0.060 x 0.030 x 0.020 mm^3	
Crystal color/habit	colorless needle	
Theta range for data collection	4.014 to 68.277°.	
Index ranges	-7<=h<=7, -15<=k<=14, -25<=l<=26	
Reflections collected	25275	
Independent reflections	3253 [R(int) = 0.0218]	
Completeness to theta = 67.000°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.929 and 0.863	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3253 / 0 / 231	
Goodness-of-fit on F^2	1.045	
Final R indices [I>2sigma(I)]	R1 = 0.0388, wR2 = 0.1028	
R indices (all data)	R1 = 0.0392, wR2 = 0.1032	
Absolute structure parameter	0.04(4)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.339 and -0.166 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for maimone18. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2027(4)	4887(2)	8864(1)	26(1)
C(2)	1676(4)	5217(2)	8196(1)	30(1)
C(3)	3043(4)	4591(2)	7749(1)	30(1)
C(4)	2565(4)	3383(2)	7776(1)	29(1)
C(5)	4012(4)	3387(2)	8359(1)	25(1)
C(6)	2827(4)	3725(2)	8940(1)	23(1)
C(7)	4297(4)	3577(2)	9489(1)	22(1)
C(8)	6225(4)	4249(2)	9568(1)	23(1)
C(9)	5575(4)	5219(2)	9943(1)	24(1)
C(10)	7098(4)	6169(2)	9846(1)	26(1)
C(11)	7592(4)	6845(2)	10402(1)	31(1)
C(12)	6925(4)	6320(2)	10993(1)	28(1)
C(13)	7424(4)	5106(2)	10979(1)	24(1)
C(14)	5303(4)	4997(2)	10620(1)	23(1)
C(15)	5149(4)	4359(2)	8069(1)	29(1)
C(16)	4584(4)	6030(2)	10942(1)	27(1)
C(17)	3653(5)	2749(2)	7271(1)	37(1)
C(18)	273(4)	3052(2)	7781(1)	32(1)
C(19)	9487(4)	4749(2)	10701(1)	28(1)
C(20)	7215(4)	4590(2)	11606(1)	29(1)
O(1)	3504(3)	5550(1)	9755(1)	26(1)
O(2)	3488(3)	5666(1)	9090(1)	27(1)
O(3)	1088(3)	3054(1)	9027(1)	26(1)
O(4)	3864(3)	2904(1)	9862(1)	26(1)
O(5)	7832(3)	6350(1)	9352(1)	32(1)

Table 3. Bond lengths [Å] and angles [°] for maimone18.

C(1)-O(2)	1.445(3)	C(11)-H(11A)	0.9900
C(1)-C(2)	1.546(3)	C(11)-H(11B)	0.9900
C(1)-C(6)	1.561(3)	C(12)-C(16)	1.548(4)
C(1)-H(1)	1.0000	C(12)-C(13)	1.563(3)
C(2)-C(3)	1.536(4)	C(12)-H(12)	1.0000
C(2)-H(2A)	0.9900	C(13)-C(19)	1.524(3)
C(2)-H(2B)	0.9900	C(13)-C(20)	1.532(4)
C(3)-C(15)	1.550(3)	C(13)-C(14)	1.578(3)
C(3)-C(4)	1.556(4)	C(14)-C(16)	1.553(3)
C(3)-H(3)	1.0000	C(14)-H(14)	1.0000
C(4)-C(18)	1.526(4)	C(15)-H(15A)	0.9900
C(4)-C(17)	1.537(4)	C(15)-H(15B)	0.9900
C(4)-C(5)	1.584(3)	C(16)-H(16A)	0.9900
C(5)-C(6)	1.547(3)	C(16)-H(16B)	0.9900
C(5)-C(15)	1.563(4)	C(17)-H(17A)	0.9800
C(5)-H(5)	1.0000	C(17)-H(17B)	0.9800
C(6)-O(3)	1.411(3)	C(17)-H(17C)	0.9800
C(6)-C(7)	1.544(3)	C(18)-H(18A)	0.9800
C(7)-O(4)	1.213(3)	C(18)-H(18B)	0.9800
C(7)-C(8)	1.508(3)	C(18)-H(18C)	0.9800
C(8)-C(9)	1.533(3)	C(19)-H(19A)	0.9800
C(8)-H(8A)	0.9900	C(19)-H(19B)	0.9800
C(8)-H(8B)	0.9900	C(19)-H(19C)	0.9800
C(9)-O(1)	1.451(3)	C(20)-H(20A)	0.9800
C(9)-C(14)	1.528(3)	C(20)-H(20B)	0.9800
C(9)-C(10)	1.560(3)	C(20)-H(20C)	0.9800
C(10)-O(5)	1.207(3)	O(1)-O(2)	1.472(2)
C(10)-C(11)	1.526(3)	O(3)-H(3A)	0.8400
C(11)-C(12)	1.520(4)		

O(2)-C(1)-C(2)	103.82(19)	C(2)-C(1)-H(1)	108.7
O(2)-C(1)-C(6)	112.87(19)	C(6)-C(1)-H(1)	108.7
C(2)-C(1)-C(6)	113.7(2)	C(3)-C(2)-C(1)	112.9(2)
O(2)-C(1)-H(1)	108.7	C(3)-C(2)-H(2A)	109.0

C(1)-C(2)-H(2A)	109.0	H(8A)-C(8)-H(8B)	108.6
C(3)-C(2)-H(2B)	109.0	O(1)-C(9)-C(14)	103.08(18)
C(1)-C(2)-H(2B)	109.0	O(1)-C(9)-C(8)	109.0(2)
H(2A)-C(2)-H(2B)	107.8	C(14)-C(9)-C(8)	114.3(2)
C(2)-C(3)-C(15)	107.6(2)	O(1)-C(9)-C(10)	108.17(18)
C(2)-C(3)-C(4)	111.5(2)	C(14)-C(9)-C(10)	110.18(19)
C(15)-C(3)-C(4)	88.2(2)	C(8)-C(9)-C(10)	111.7(2)
C(2)-C(3)-H(3)	115.5	O(5)-C(10)-C(11)	122.5(2)
C(15)-C(3)-H(3)	115.5	O(5)-C(10)-C(9)	120.8(2)
C(4)-C(3)-H(3)	115.5	C(11)-C(10)-C(9)	116.7(2)
C(18)-C(4)-C(17)	107.5(2)	C(12)-C(11)-C(10)	112.7(2)
C(18)-C(4)-C(3)	117.2(2)	C(12)-C(11)-H(11A)	109.1
C(17)-C(4)-C(3)	113.1(2)	C(10)-C(11)-H(11A)	109.1
C(18)-C(4)-C(5)	123.9(2)	C(12)-C(11)-H(11B)	109.1
C(17)-C(4)-C(5)	108.8(2)	C(10)-C(11)-H(11B)	109.1
C(3)-C(4)-C(5)	85.02(19)	H(11A)-C(11)-H(11B)	107.8
C(6)-C(5)-C(15)	110.5(2)	C(11)-C(12)-C(16)	108.3(2)
C(6)-C(5)-C(4)	112.56(19)	C(11)-C(12)-C(13)	110.6(2)
C(15)-C(5)-C(4)	86.76(18)	C(16)-C(12)-C(13)	88.05(18)
C(6)-C(5)-H(5)	114.6	C(11)-C(12)-H(12)	115.5
C(15)-C(5)-H(5)	114.6	C(16)-C(12)-H(12)	115.5
C(4)-C(5)-H(5)	114.6	C(13)-C(12)-H(12)	115.5
O(3)-C(6)-C(7)	107.65(18)	C(19)-C(13)-C(20)	108.1(2)
O(3)-C(6)-C(5)	109.53(19)	C(19)-C(13)-C(12)	118.3(2)
C(7)-C(6)-C(5)	108.33(18)	C(20)-C(13)-C(12)	112.4(2)
O(3)-C(6)-C(1)	108.53(19)	C(19)-C(13)-C(14)	121.3(2)
C(7)-C(6)-C(1)	113.40(19)	C(20)-C(13)-C(14)	109.8(2)
C(5)-C(6)-C(1)	109.34(19)	C(12)-C(13)-C(14)	85.36(18)
O(4)-C(7)-C(8)	120.2(2)	C(9)-C(14)-C(16)	109.1(2)
O(4)-C(7)-C(6)	118.3(2)	C(9)-C(14)-C(13)	112.02(19)
C(8)-C(7)-C(6)	121.5(2)	C(16)-C(14)-C(13)	87.34(18)
C(7)-C(8)-C(9)	106.71(19)	C(9)-C(14)-H(14)	115.1
C(7)-C(8)-H(8A)	110.4	C(16)-C(14)-H(14)	115.1
C(9)-C(8)-H(8A)	110.4	C(13)-C(14)-H(14)	115.1
C(7)-C(8)-H(8B)	110.4	C(3)-C(15)-C(5)	85.91(19)
C(9)-C(8)-H(8B)	110.4	C(3)-C(15)-H(15A)	114.3

C(5)-C(15)-H(15A)	114.3	C(4)-C(18)-H(18C)	109.5
C(3)-C(15)-H(15B)	114.3	H(18A)-C(18)-H(18C)	109.5
C(5)-C(15)-H(15B)	114.3	H(18B)-C(18)-H(18C)	109.5
H(15A)-C(15)-H(15B)	111.5	C(13)-C(19)-H(19A)	109.5
C(12)-C(16)-C(14)	86.74(18)	C(13)-C(19)-H(19B)	109.5
C(12)-C(16)-H(16A)	114.2	H(19A)-C(19)-H(19B)	109.5
C(14)-C(16)-H(16A)	114.2	C(13)-C(19)-H(19C)	109.5
C(12)-C(16)-H(16B)	114.2	H(19A)-C(19)-H(19C)	109.5
C(14)-C(16)-H(16B)	114.2	H(19B)-C(19)-H(19C)	109.5
H(16A)-C(16)-H(16B)	111.4	C(13)-C(20)-H(20A)	109.5
C(4)-C(17)-H(17A)	109.5	C(13)-C(20)-H(20B)	109.5
C(4)-C(17)-H(17B)	109.5	H(20A)-C(20)-H(20B)	109.5
H(17A)-C(17)-H(17B)	109.5	C(13)-C(20)-H(20C)	109.5
C(4)-C(17)-H(17C)	109.5	H(20A)-C(20)-H(20C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(20B)-C(20)-H(20C)	109.5
H(17B)-C(17)-H(17C)	109.5	C(9)-O(1)-O(2)	108.54(16)
C(4)-C(18)-H(18A)	109.5	C(1)-O(2)-O(1)	106.23(16)
C(4)-C(18)-H(18B)	109.5	C(6)-O(3)-H(3A)	109.5
H(18A)-C(18)-H(18B)	109.5		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for maimone18. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	21(1)	29(1)	28(1)	1(1)	0(1)	3(1)
C(2)	28(1)	30(1)	31(1)	6(1)	-3(1)	4(1)
C(3)	27(1)	38(1)	23(1)	9(1)	0(1)	3(1)
C(4)	28(1)	35(1)	23(1)	4(1)	-2(1)	7(1)
C(5)	22(1)	29(1)	24(1)	3(1)	-1(1)	5(1)
C(6)	21(1)	26(1)	23(1)	0(1)	1(1)	0(1)
C(7)	23(1)	21(1)	22(1)	-2(1)	3(1)	4(1)
C(8)	23(1)	22(1)	25(1)	0(1)	0(1)	1(1)
C(9)	22(1)	21(1)	28(1)	1(1)	1(1)	-1(1)
C(10)	25(1)	20(1)	32(1)	2(1)	-1(1)	1(1)
C(11)	34(1)	20(1)	38(1)	-2(1)	0(1)	-5(1)
C(12)	30(1)	22(1)	31(1)	-5(1)	0(1)	-2(1)
C(13)	23(1)	22(1)	27(1)	-4(1)	-1(1)	-2(1)
C(14)	20(1)	21(1)	26(1)	-2(1)	1(1)	-1(1)
C(15)	24(1)	38(1)	26(1)	8(1)	3(1)	3(1)
C(16)	26(1)	25(1)	30(1)	-5(1)	0(1)	1(1)
C(17)	41(2)	47(2)	23(1)	0(1)	-2(1)	13(1)
C(18)	32(1)	37(1)	26(1)	2(1)	-8(1)	3(1)
C(19)	22(1)	29(1)	33(1)	-2(1)	-2(1)	-2(1)
C(20)	32(1)	28(1)	27(1)	-2(1)	-3(1)	0(1)
O(1)	26(1)	25(1)	26(1)	0(1)	-1(1)	3(1)
O(2)	30(1)	23(1)	28(1)	4(1)	-1(1)	1(1)
O(3)	25(1)	29(1)	23(1)	2(1)	0(1)	-4(1)
O(4)	30(1)	22(1)	25(1)	2(1)	-3(1)	-2(1)
O(5)	32(1)	27(1)	36(1)	4(1)	7(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for maimone18.

	x	y	z	U(eq)
H(1)	681	4964	9090	31
H(2A)	190	5107	8090	36
H(2B)	1984	5983	8152	36
H(3)	3153	4899	7331	35
H(5)	4902	2740	8407	30
H(8A)	6773	4473	9167	28
H(8B)	7326	3844	9782	28
H(11A)	9112	6983	10416	37
H(11B)	6872	7537	10364	37
H(12)	7340	6700	11372	33
H(14)	4465	4351	10719	27
H(15A)	5585	4909	8364	35
H(15B)	6301	4170	7789	35
H(16A)	3764	6515	10681	33
H(16B)	3886	5912	11337	33
H(17A)	3557	1989	7362	55
H(17B)	5125	2958	7249	55
H(17C)	2973	2895	6882	55
H(18A)	-373	3242	7393	48
H(18B)	-453	3419	8112	48
H(18C)	173	2284	7843	48
H(19A)	10641	4970	10964	42
H(19B)	9653	5072	10299	42
H(19C)	9492	3975	10662	42
H(20A)	7168	3817	11560	43
H(20B)	5927	4836	11800	43
H(20C)	8416	4787	11857	43
H(3A)	1066	2841	9388	39

X-Ray crystallographic Analysis 3

A colorless plate 0.060 x 0.040 x 0.030 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 10 seconds per frame using a scan width of 1.0°. Data collection was 100.0% complete to 67.000° in θ. A total of 27487 reflections were collected covering the indices, -7<=*h*<=7, -16<=*k*<=16, -24<=*l*<=24. 3318 reflections were found to be symmetry independent, with an R_{int} of 0.0290. Indexing and unit cell refinement indicated a primitive, orthorhombic lattice. The space group was found to be P 21 21 21 (No. 19). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by iterative methods (SHELXT) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2013). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2013. Absolute stereochemistry was unambiguously determined to be *R* at C3, C5, C6, C12, and C14, respectively.

Table 1. Crystal data and structure refinement for **3**.

X-ray ID	maimone15	
Sample/notebook ID	XH_54_SPS	
Empirical formula	C20 H28 O6	
Formula weight	364.42	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 6.4231(5) Å	α= 90°.
	b = 13.8311(9) Å	β= 90°.
	c = 20.3077(14) Å	γ = 90°.
Volume	1804.1(2) $Å^3$	
Z	4	
Density (calculated)	1.342 Mg/m^3	
Absorption coefficient	0.807 mm^{-1}	
F(000)	784	
Crystal size	0.060 x 0.040 x 0.030 mm^3	
Crystal color/habit	colorless plate	
Theta range for data collection	3.867 to 68.463°.	
Index ranges	-7<=h<=7, -16<=k<=16, -24<=l<=24	
Reflections collected	27487	
Independent reflections	3318 [R(int) = 0.0290]	
Completeness to theta = 67.000°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.929 and 0.846	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3318 / 0 / 259	
Goodness-of-fit on F^2	1.104	
Final R indices [I>2sigma(I)]	R1 = 0.0714, wR2 = 0.1882	
R indices (all data)	R1 = 0.0725, wR2 = 0.1901	
Absolute structure parameter	0.02(6)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.518 and -0.243 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for maimone15. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	-2499(9)	4845(3)	5749(2)	57(1)
C(2)	-2206(8)	5938(4)	5761(3)	62(2)
C(3)	-406(8)	6277(3)	5369(3)	56(1)
C(4)	-519(8)	5921(3)	4647(2)	46(1)
C(5)	469(6)	4970(3)	4916(2)	35(1)
C(6)	-1109(6)	4295(3)	5241(2)	34(1)
C(7)	60(6)	3430(3)	5561(2)	31(1)
C(8)	-1241(7)	2695(3)	5937(2)	37(1)
C(9)	-1707(7)	2966(3)	6657(2)	34(1)
C(10)	276(7)	3022(3)	7096(2)	40(1)
C(11)	75(8)	2582(4)	7791(2)	54(1)
C(12)	-1894(7)	2019(3)	7890(2)	44(1)
C(13)	-2384(7)	1372(3)	7277(2)	43(1)
C(14)	-3320(6)	2321(3)	6972(2)	39(1)
C(15)	1445(8)	5580(3)	5481(2)	46(1)
C(16)	-3733(7)	2666(4)	7685(2)	47(1)
C(17)	1038(10)	6436(4)	4195(3)	67(2)
C(18)	-2628(8)	5900(3)	4297(2)	50(1)
C(19)	-595(10)	840(4)	6950(3)	62(1)
C(20)	-4119(10)	642(4)	7423(3)	61(1)
O(1)	-2850(10)	3884(5)	6697(3)	38(2)
O(2)	-1556(7)	4647(3)	6426(2)	35(1)
O(1A)	-1930(30)	4011(12)	6621(8)	47(4)
O(2A)	-3725(13)	4104(6)	6188(4)	39(3)
O(3)	-2569(4)	3890(2)	4787(1)	40(1)
O(4)	-1372(5)	3407(2)	4270(2)	49(1)
O(5)	1882(4)	3328(2)	5460(1)	39(1)
O(6)	1836(5)	3400(3)	6910(2)	51(1)

Table 3. Bond lengths [Å] and angles [°] for maimone15.

C(1)-C(2)	1.524(7)	C(11)-H(11A)	0.9900
C(1)-O(2)	1.526(6)	C(11)-H(11B)	0.9900
C(1)-C(6)	1.562(5)	C(12)-C(16)	1.539(6)
C(1)-O(2A)	1.570(10)	C(12)-C(13)	1.565(6)
C(1)-H(1)	1.0000	C(12)-H(12)	1.0000
C(2)-C(3)	1.480(8)	C(13)-C(19)	1.518(7)
C(2)-H(2A)	0.9900	C(13)-C(20)	1.533(6)
C(2)-H(2B)	0.9900	C(13)-C(14)	1.571(6)
C(3)-C(15)	1.547(6)	C(14)-C(16)	1.549(6)
C(3)-C(4)	1.548(6)	C(14)-H(14)	1.0000
C(3)-H(3)	1.0000	C(15)-H(15A)	0.9900
C(4)-C(18)	1.531(6)	C(15)-H(15B)	0.9900
C(4)-C(17)	1.532(8)	C(16)-H(16A)	0.9900
C(4)-C(5)	1.558(5)	C(16)-H(16B)	0.9900
C(5)-C(6)	1.528(6)	C(17)-H(17A)	0.9800
C(5)-C(15)	1.556(5)	C(17)-H(17B)	0.9800
C(5)-H(5)	1.0000	C(17)-H(17C)	0.9800
C(6)-O(3)	1.429(5)	C(18)-H(18A)	0.9800
C(6)-C(7)	1.556(5)	C(18)-H(18B)	0.9800
C(7)-O(5)	1.196(5)	C(18)-H(18C)	0.9800
C(7)-C(8)	1.521(5)	C(19)-H(19A)	0.9800
C(8)-C(9)	1.539(5)	C(19)-H(19B)	0.9800
C(8)-H(8A)	0.9900	C(19)-H(19C)	0.9800
C(8)-H(8B)	0.9900	C(20)-H(20A)	0.9800
C(9)-O(1A)	1.455(17)	C(20)-H(20B)	0.9800
C(9)-O(1)	1.469(8)	C(20)-H(20C)	0.9800
C(9)-C(14)	1.510(6)	O(1)-O(2)	1.452(7)
C(9)-C(10)	1.557(6)	O(1A)-O(2A)	1.454(18)
C(10)-O(6)	1.192(5)	O(3)-O(4)	1.463(4)
C(10)-C(11)	1.542(6)	O(4)-H(4)	0.8400
C(11)-C(12)	1.499(7)		

C(2)-C(1)-O(2)	96.6(4)	O(2)-C(1)-C(6)	106.3(4)
C(2)-C(1)-C(6)	115.0(4)	C(2)-C(1)-O(2A)	134.5(5)

C(6)-C(1)-O(2A)	110.1(4)	C(8)-C(7)-C(6)	117.3(3)
C(2)-C(1)-H(1)	112.6	C(7)-C(8)-C(9)	114.9(3)
O(2)-C(1)-H(1)	112.6	C(7)-C(8)-H(8A)	108.5
C(6)-C(1)-H(1)	112.6	C(9)-C(8)-H(8A)	108.5
C(3)-C(2)-C(1)	113.7(4)	C(7)-C(8)-H(8B)	108.5
C(3)-C(2)-H(2A)	108.8	C(9)-C(8)-H(8B)	108.5
C(1)-C(2)-H(2A)	108.8	H(8A)-C(8)-H(8B)	107.5
C(3)-C(2)-H(2B)	108.8	O(1A)-C(9)-C(14)	122.6(7)
C(1)-C(2)-H(2B)	108.8	O(1)-C(9)-C(14)	98.3(4)
H(2A)-C(2)-H(2B)	107.7	O(1A)-C(9)-C(8)	102.4(7)
C(2)-C(3)-C(15)	108.9(4)	O(1)-C(9)-C(8)	111.1(4)
C(2)-C(3)-C(4)	111.8(4)	C(14)-C(9)-C(8)	113.1(3)
C(15)-C(3)-C(4)	88.7(3)	O(1A)-C(9)-C(10)	93.5(7)
C(2)-C(3)-H(3)	114.9	O(1)-C(9)-C(10)	109.5(4)
C(15)-C(3)-H(3)	114.9	C(14)-C(9)-C(10)	110.4(3)
C(4)-C(3)-H(3)	114.9	C(8)-C(9)-C(10)	113.4(3)
C(18)-C(4)-C(17)	107.9(4)	O(6)-C(10)-C(11)	122.2(4)
C(18)-C(4)-C(3)	119.2(4)	O(6)-C(10)-C(9)	121.9(4)
C(17)-C(4)-C(3)	112.8(4)	C(11)-C(10)-C(9)	115.9(4)
C(18)-C(4)-C(5)	120.4(3)	C(12)-C(11)-C(10)	113.5(4)
C(17)-C(4)-C(5)	109.6(4)	C(12)-C(11)-H(11A)	108.9
C(3)-C(4)-C(5)	85.3(3)	C(10)-C(11)-H(11A)	108.9
C(6)-C(5)-C(15)	106.3(3)	C(12)-C(11)-H(11B)	108.9
C(6)-C(5)-C(4)	113.4(3)	C(10)-C(11)-H(11B)	108.9
C(15)-C(5)-C(4)	88.0(3)	H(11A)-C(11)-H(11B)	107.7
C(6)-C(5)-H(5)	115.3	C(11)-C(12)-C(16)	108.0(4)
C(15)-C(5)-H(5)	115.3	C(11)-C(12)-C(13)	111.1(4)
C(4)-C(5)-H(5)	115.3	C(16)-C(12)-C(13)	87.9(3)
O(3)-C(6)-C(5)	113.3(3)	C(11)-C(12)-H(12)	115.5
O(3)-C(6)-C(7)	106.6(3)	C(16)-C(12)-H(12)	115.5
C(5)-C(6)-C(7)	109.3(3)	C(13)-C(12)-H(12)	115.5
O(3)-C(6)-C(1)	104.0(4)	C(19)-C(13)-C(20)	108.4(4)
C(5)-C(6)-C(1)	111.5(3)	C(19)-C(13)-C(12)	118.3(4)
C(7)-C(6)-C(1)	111.9(3)	C(20)-C(13)-C(12)	111.7(4)
O(5)-C(7)-C(8)	123.0(3)	C(19)-C(13)-C(14)	121.4(3)
O(5)-C(7)-C(6)	119.5(3)	C(20)-C(13)-C(14)	110.4(4)

C(12)-C(13)-C(14)	85.0(3)	C(4)-C(18)-H(18B)	109.5
C(9)-C(14)-C(16)	109.4(4)	H(18A)-C(18)-H(18B)	109.5
C(9)-C(14)-C(13)	113.5(3)	C(4)-C(18)-H(18C)	109.5
C(16)-C(14)-C(13)	87.3(3)	H(18A)-C(18)-H(18C)	109.5
C(9)-C(14)-H(14)	114.6	H(18B)-C(18)-H(18C)	109.5
C(16)-C(14)-H(14)	114.6	C(13)-C(19)-H(19A)	109.5
C(13)-C(14)-H(14)	114.6	C(13)-C(19)-H(19B)	109.5
C(3)-C(15)-C(5)	85.4(3)	H(19A)-C(19)-H(19B)	109.5
C(3)-C(15)-H(15A)	114.4	C(13)-C(19)-H(19C)	109.5
C(5)-C(15)-H(15A)	114.4	H(19A)-C(19)-H(19C)	109.5
C(3)-C(15)-H(15B)	114.4	H(19B)-C(19)-H(19C)	109.5
C(5)-C(15)-H(15B)	114.4	C(13)-C(20)-H(20A)	109.5
H(15A)-C(15)-H(15B)	111.5	C(13)-C(20)-H(20B)	109.5
C(12)-C(16)-C(14)	86.7(3)	H(20A)-C(20)-H(20B)	109.5
C(12)-C(16)-H(16A)	114.2	C(13)-C(20)-H(20C)	109.5
C(14)-C(16)-H(16A)	114.2	H(20A)-C(20)-H(20C)	109.5
C(12)-C(16)-H(16B)	114.2	H(20B)-C(20)-H(20C)	109.5
C(14)-C(16)-H(16B)	114.2	O(2)-O(1)-C(9)	108.8(5)
H(16A)-C(16)-H(16B)	111.4	O(1)-O(2)-C(1)	104.1(4)
C(4)-C(17)-H(17A)	109.5	C(9)-O(1A)-O(2A)	101.3(10)
C(4)-C(17)-H(17B)	109.5	O(1A)-O(2A)-C(1)	90.3(8)
H(17A)-C(17)-H(17B)	109.5	C(6)-O(3)-O(4)	107.3(3)
C(4)-C(17)-H(17C)	109.5	O(3)-O(4)-H(4)	109.5
H(17A)-C(17)-H(17C)	109.5		
H(17B)-C(17)-H(17C)	109.5		
C(4)-C(18)-H(18A)	109.5		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for maimone15. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	77(3)	45(2)	49(2)	12(2)	21(2)	32(2)
C(2)	61(3)	59(3)	66(3)	-37(2)	-27(2)	30(2)
C(3)	64(3)	32(2)	72(3)	-19(2)	-33(3)	13(2)
C(4)	59(3)	25(2)	54(2)	2(2)	-22(2)	-4(2)
C(5)	44(2)	26(2)	35(2)	-2(1)	-6(2)	0(2)
C(6)	41(2)	30(2)	32(2)	0(1)	2(2)	4(2)
C(7)	36(2)	28(2)	30(2)	-5(1)	1(1)	5(1)
C(8)	45(2)	32(2)	33(2)	-2(1)	2(2)	0(2)
C(9)	42(2)	29(2)	31(2)	-1(1)	-2(2)	6(2)
C(10)	44(2)	38(2)	39(2)	0(2)	-4(2)	-1(2)
C(11)	51(3)	73(3)	40(2)	11(2)	-12(2)	-8(2)
C(12)	47(2)	54(2)	32(2)	10(2)	1(2)	6(2)
C(13)	49(2)	41(2)	40(2)	5(2)	7(2)	-1(2)
C(14)	35(2)	50(2)	33(2)	-3(2)	0(2)	5(2)
C(15)	56(3)	35(2)	47(2)	-10(2)	-21(2)	8(2)
C(16)	50(2)	56(2)	34(2)	-2(2)	4(2)	8(2)
C(17)	77(4)	40(2)	84(4)	26(2)	-32(3)	-21(2)
C(18)	60(3)	38(2)	53(2)	6(2)	-25(2)	-1(2)
C(19)	83(4)	40(2)	63(3)	16(2)	24(3)	20(2)
C(20)	74(3)	59(3)	51(3)	3(2)	10(2)	-22(3)
O(1)	51(4)	30(3)	33(2)	3(2)	3(3)	11(3)
O(2)	41(2)	31(3)	33(2)	-4(2)	-6(2)	-1(2)
O(1A)	65(10)	37(7)	38(6)	-5(5)	-8(7)	1(8)
O(2A)	35(5)	39(5)	42(5)	-6(4)	0(4)	2(3)
O(3)	37(1)	42(1)	41(1)	7(1)	-1(1)	-4(1)
O(4)	58(2)	50(2)	38(1)	-6(1)	8(1)	-22(1)
O(5)	40(2)	36(1)	41(1)	3(1)	7(1)	9(1)
O(6)	45(2)	59(2)	48(2)	10(1)	-3(1)	-9(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for maimone15.

	x	y	z	U(eq)
H(1)	-3997	4652	5722	69
H(2A)	-3486	6248	5590	74
H(2B)	-2022	6149	6223	74
H(3)	-65	6978	5422	67
H(5)	1489	4649	4615	42
H(8A)	-503	2067	5931	44
H(8B)	-2579	2607	5702	44
H(11A)	1279	2151	7871	65
H(11B)	133	3110	8120	65
H(12)	-2037	1694	8328	53
H(14)	-4604	2220	6702	47
H(15A)	1447	5257	5916	55
H(15B)	2825	5860	5376	55
H(16A)	-3494	3366	7756	56
H(16B)	-5093	2460	7867	56
H(17A)	1154	6082	3779	100
H(17B)	2403	6461	4410	100
H(17C)	552	7095	4108	100
H(18A)	-3057	6561	4190	76
H(18B)	-3669	5602	4585	76
H(18C)	-2510	5522	3890	76
H(19A)	-128	311	7235	93
H(19B)	561	1290	6875	93
H(19C)	-1061	576	6527	93
H(20A)	-4630	367	7009	92
H(20B)	-5265	968	7651	92
H(20C)	-3572	123	7703	92
H(4)	-1538	2806	4299	73

X-Ray crystallographic Analysis 11

A colorless block 0.040 x 0.030 x 0.030 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 10 seconds per frame using a scan width of 1.0°. Data collection was 98.4% complete to 67.000° in θ. A total of 47377 reflections were collected covering the indices, -8<=*h*<=7, -11<=*k*<=11, -64<=*l*<=65. 6367 reflections were found to be symmetry independent, with an R_{int} of 0.0490. Indexing and unit cell refinement indicated a primitive, orthorhombic lattice. The space group was found to be P 21 21 21 (No. 19). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by iterative methods (SHELXT) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2013). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2013. Absolute stereochemistry was unambiguously determined to be *R* at C2, C4, C13, C15, C22, C24, C33, and C35, and *S* at C1, C6, C21, and C26, respectively.

Table 1. Crystal data and structure refinement for **11**.

X-ray ID	maimone23	
Sample/notebook ID	XH-194-2	
Empirical formula	C20 H28 O5	
Formula weight	348.42	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 6.7606(4) Å	α= 90°.
	b = 10.0156(5) Å	β= 90°.
	c = 54.297(3) Å	γ = 90°.
Volume	3676.5(3) $Å^3$	
Z	8	
Density (calculated)	1.259 Mg/m^3	
Absorption coefficient	0.726 mm^{-1}	
F(000)	1504	
Crystal size	0.040 x 0.030 x 0.030 mm^3	
Crystal color/habit	colorless block	
Theta range for data collection	3.256 to 68.283°.	
Index ranges	-8<=h<=7, -11<=k<=11, -64<=l<=65	
Reflections collected	47377	
Independent reflections	6367 [R(int) = 0.0490]	
Completeness to theta = 67.000°	98.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.929 and 0.774	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6367 / 0 / 463	
Goodness-of-fit on F^2	1.179	
Final R indices [I>2sigma(I)]	R1 = 0.0571, wR2 = 0.1278	
R indices (all data)	R1 = 0.0576, wR2 = 0.1281	
Absolute structure parameter	0.05(7)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.336 and -0.186 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for maimone23. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	-2499(7)	14339(4)	3104(1)	31(1)
C(2)	-1623(6)	13796(5)	2866(1)	33(1)
C(3)	-2844(7)	14162(5)	2632(1)	35(1)
C(4)	-1721(7)	15527(5)	2646(1)	40(1)
C(5)	-2622(8)	16477(5)	2831(1)	44(1)
C(6)	-3159(7)	15834(4)	3080(1)	37(1)
C(7)	-8(7)	14780(5)	2777(1)	44(1)
C(8)	-2197(8)	13311(5)	2414(1)	46(1)
C(9)	-5072(7)	14182(5)	2639(1)	42(1)
C(10)	-1076(7)	14051(4)	3318(1)	31(1)
C(11)	-2065(7)	13108(4)	3473(1)	34(1)
C(12)	-3866(7)	12886(4)	3376(1)	31(1)
C(13)	-5513(7)	12024(4)	3461(1)	33(1)
C(14)	-5177(8)	11053(4)	3676(1)	39(1)
C(15)	-6559(7)	10058(4)	3545(1)	34(1)
C(16)	-6163(7)	10799(4)	3298(1)	32(1)
C(17)	-4450(7)	10218(4)	3151(1)	35(1)
C(18)	-7946(7)	11050(5)	3133(1)	39(1)
C(19)	-6089(7)	8587(4)	3577(1)	39(1)
C(20)	-7514(8)	7655(4)	3454(1)	40(1)
C(21)	355(6)	9649(4)	4413(1)	27(1)
C(22)	-1212(7)	10176(4)	4589(1)	32(1)
C(23)	-713(7)	9868(4)	4865(1)	31(1)
C(24)	658(8)	11111(4)	4850(1)	39(1)
C(25)	2634(7)	10796(5)	4737(1)	39(1)
C(26)	2523(7)	9903(4)	4506(1)	35(1)
C(27)	-695(7)	11655(4)	4644(1)	36(1)
C(28)	-2483(8)	10115(4)	5032(1)	41(1)
C(29)	144(7)	8511(4)	4935(1)	34(1)
C(30)	-50(7)	10109(4)	4149(1)	32(1)
C(31)	-592(7)	8945(4)	4013(1)	33(1)

C(32)	-473(6)	7888(4)	4167(1)	29(1)
C(33)	-922(7)	6460(4)	4130(1)	29(1)
C(34)	-1877(7)	5978(4)	3889(1)	37(1)
C(35)	-3147(7)	5009(4)	4041(1)	31(1)
C(36)	-2733(6)	5861(4)	4276(1)	27(1)
C(37)	-2159(7)	5090(4)	4507(1)	36(1)
C(38)	-4311(6)	6885(4)	4331(1)	28(1)
C(39)	-5238(7)	4790(4)	3954(1)	35(1)
C(40)	-6387(7)	3726(4)	4088(1)	33(1)
O(1)	-2556(6)	16622(3)	3282(1)	46(1)
O(2)	-4233(4)	13555(3)	3167(1)	32(1)
O(3)	594(5)	14519(3)	3338(1)	38(1)
O(4)	-8881(7)	7954(4)	3332(1)	82(2)
O(5)	-7096(6)	6400(3)	3505(1)	61(1)
O(6)	3798(5)	10311(3)	4317(1)	41(1)
O(7)	89(4)	8195(2)	4397(1)	28(1)
O(8)	0(5)	11274(3)	4076(1)	40(1)
O(9)	-5782(5)	3105(3)	4261(1)	48(1)
O(10)	-8156(5)	3535(3)	3987(1)	44(1)

Table 3. Bond lengths [Å] and angles [°] for maimone23.

C(1)-O(2)	1.451(5)	C(13)-H(13)	1.0000
C(1)-C(2)	1.523(6)	C(14)-C(15)	1.541(6)
C(1)-C(10)	1.533(6)	C(14)-H(14A)	0.9900
C(1)-C(6)	1.568(6)	C(14)-H(14B)	0.9900
C(2)-C(7)	1.550(7)	C(15)-C(19)	1.517(6)
C(2)-C(3)	1.559(6)	C(15)-C(16)	1.556(6)
C(2)-H(2)	1.0000	C(15)-H(15)	1.0000
C(3)-C(9)	1.507(7)	C(16)-C(18)	1.522(6)
C(3)-C(8)	1.523(6)	C(16)-C(17)	1.522(6)
C(3)-C(4)	1.565(7)	C(17)-H(17A)	0.9800
C(4)-C(5)	1.515(7)	C(17)-H(17B)	0.9800
C(4)-C(7)	1.551(7)	C(17)-H(17C)	0.9800
C(4)-H(4)	1.0000	C(18)-H(18A)	0.9800
C(5)-C(6)	1.541(6)	C(18)-H(18B)	0.9800
C(5)-H(5A)	0.9900	C(18)-H(18C)	0.9800
C(5)-H(5B)	0.9900	C(19)-C(20)	1.498(7)
C(6)-O(1)	1.411(5)	C(19)-H(19A)	0.9900
C(6)-H(6)	1.0000	C(19)-H(19B)	0.9900
C(7)-H(7A)	0.9900	C(20)-O(4)	1.177(6)
C(7)-H(7B)	0.9900	C(20)-O(5)	1.318(5)
C(8)-H(8A)	0.9800	C(21)-O(7)	1.469(4)
C(8)-H(8B)	0.9800	C(21)-C(22)	1.522(6)
C(8)-H(8C)	0.9800	C(21)-C(30)	1.530(5)
C(9)-H(9A)	0.9800	C(21)-C(26)	1.571(6)
C(9)-H(9B)	0.9800	C(22)-C(27)	1.552(5)
C(9)-H(9C)	0.9800	C(22)-C(23)	1.567(6)
C(10)-O(3)	1.228(5)	C(22)-H(22)	1.0000
C(10)-C(11)	1.430(6)	C(23)-C(28)	1.522(6)
C(11)-C(12)	1.343(6)	C(23)-C(29)	1.526(6)
C(11)-H(11)	0.9500	C(23)-C(24)	1.555(6)
C(12)-O(2)	1.344(5)	C(24)-C(25)	1.503(7)
C(12)-C(13)	1.482(6)	C(24)-C(27)	1.542(6)
C(13)-C(14)	1.538(6)	C(24)-H(24)	1.0000
C(13)-C(16)	1.575(6)	C(25)-C(26)	1.544(6)

C(25)-H(25A)	0.9900	C(34)-H(34A)	0.9900
C(25)-H(25B)	0.9900	C(34)-H(34B)	0.9900
C(26)-O(6)	1.401(5)	C(35)-C(39)	1.508(6)
C(26)-H(26)	1.0000	C(35)-C(36)	1.560(5)
C(27)-H(27A)	0.9900	C(35)-H(35)	1.0000
C(27)-H(27B)	0.9900	C(36)-C(38)	1.509(6)
C(28)-H(28A)	0.9800	C(36)-C(37)	1.522(5)
C(28)-H(28B)	0.9800	C(37)-H(37A)	0.9800
C(28)-H(28C)	0.9800	C(37)-H(37B)	0.9800
C(29)-H(29A)	0.9800	C(37)-H(37C)	0.9800
C(29)-H(29B)	0.9800	C(38)-H(38A)	0.9800
C(29)-H(29C)	0.9800	C(38)-H(38B)	0.9800
C(30)-O(8)	1.232(5)	C(38)-H(38C)	0.9800
C(30)-C(31)	1.428(6)	C(39)-C(40)	1.505(6)
C(31)-C(32)	1.351(6)	C(39)-H(39A)	0.9900
C(31)-H(31)	0.9500	C(39)-H(39B)	0.9900
C(32)-O(7)	1.341(5)	C(40)-O(9)	1.199(5)
C(32)-C(33)	1.476(5)	C(40)-O(10)	1.328(5)
C(33)-C(34)	1.536(6)	O(1)-H(1)	0.8400
C(33)-C(36)	1.578(6)	O(5)-H(5)	0.8400
C(33)-H(33)	1.0000	O(6)-H(6A)	0.8400
C(34)-C(35)	1.536(6)	O(10)-H(10)	0.8400

O(2)-C(1)-C(2)	108.6(3)	C(9)-C(3)-C(2)	120.9(4)
O(2)-C(1)-C(10)	103.2(3)	C(8)-C(3)-C(2)	110.5(4)
C(2)-C(1)-C(10)	109.3(3)	C(9)-C(3)-C(4)	118.2(4)
O(2)-C(1)-C(6)	107.9(3)	C(8)-C(3)-C(4)	112.6(4)
C(2)-C(1)-C(6)	112.4(4)	C(2)-C(3)-C(4)	84.9(3)
C(10)-C(1)-C(6)	114.9(4)	C(5)-C(4)-C(7)	107.3(4)
C(1)-C(2)-C(7)	108.3(4)	C(5)-C(4)-C(3)	112.6(4)
C(1)-C(2)-C(3)	113.7(4)	C(7)-C(4)-C(3)	87.8(4)
C(7)-C(2)-C(3)	88.1(3)	C(5)-C(4)-H(4)	115.3
C(1)-C(2)-H(2)	114.6	C(7)-C(4)-H(4)	115.3
C(7)-C(2)-H(2)	114.6	C(3)-C(4)-H(4)	115.3
C(3)-C(2)-H(2)	114.6	C(4)-C(5)-C(6)	114.6(4)
C(9)-C(3)-C(8)	108.3(4)	C(4)-C(5)-H(5A)	108.6

C(6)-C(5)-H(5A)	108.6	O(2)-C(12)-C(13)	114.5(4)
C(4)-C(5)-H(5B)	108.6	C(12)-C(13)-C(14)	119.5(4)
C(6)-C(5)-H(5B)	108.6	C(12)-C(13)-C(16)	119.4(3)
H(5A)-C(5)-H(5B)	107.6	C(14)-C(13)-C(16)	88.6(3)
O(1)-C(6)-C(5)	112.3(4)	C(12)-C(13)-H(13)	109.2
O(1)-C(6)-C(1)	112.8(4)	C(14)-C(13)-H(13)	109.2
C(5)-C(6)-C(1)	113.9(4)	C(16)-C(13)-H(13)	109.2
O(1)-C(6)-H(6)	105.6	C(13)-C(14)-C(15)	88.1(3)
C(5)-C(6)-H(6)	105.6	C(13)-C(14)-H(14A)	114.0
C(1)-C(6)-H(6)	105.6	C(15)-C(14)-H(14A)	114.0
C(2)-C(7)-C(4)	85.7(3)	C(13)-C(14)-H(14B)	114.0
C(2)-C(7)-H(7A)	114.4	C(15)-C(14)-H(14B)	114.0
C(4)-C(7)-H(7A)	114.4	H(14A)-C(14)-H(14B)	111.2
C(2)-C(7)-H(7B)	114.4	C(19)-C(15)-C(14)	116.7(4)
C(4)-C(7)-H(7B)	114.4	C(19)-C(15)-C(16)	121.6(4)
H(7A)-C(7)-H(7B)	111.5	C(14)-C(15)-C(16)	89.2(3)
C(3)-C(8)-H(8A)	109.5	C(19)-C(15)-H(15)	109.2
C(3)-C(8)-H(8B)	109.5	C(14)-C(15)-H(15)	109.2
H(8A)-C(8)-H(8B)	109.5	C(16)-C(15)-H(15)	109.2
C(3)-C(8)-H(8C)	109.5	C(18)-C(16)-C(17)	111.0(3)
H(8A)-C(8)-H(8C)	109.5	C(18)-C(16)-C(15)	116.7(4)
H(8B)-C(8)-H(8C)	109.5	C(17)-C(16)-C(15)	113.6(4)
C(3)-C(9)-H(9A)	109.5	C(18)-C(16)-C(13)	115.0(4)
C(3)-C(9)-H(9B)	109.5	C(17)-C(16)-C(13)	112.3(4)
H(9A)-C(9)-H(9B)	109.5	C(15)-C(16)-C(13)	86.3(3)
C(3)-C(9)-H(9C)	109.5	C(16)-C(17)-H(17A)	109.5
H(9A)-C(9)-H(9C)	109.5	C(16)-C(17)-H(17B)	109.5
H(9B)-C(9)-H(9C)	109.5	H(17A)-C(17)-H(17B)	109.5
O(3)-C(10)-C(11)	129.1(4)	C(16)-C(17)-H(17C)	109.5
O(3)-C(10)-C(1)	124.8(4)	H(17A)-C(17)-H(17C)	109.5
C(11)-C(10)-C(1)	106.0(4)	H(17B)-C(17)-H(17C)	109.5
C(12)-C(11)-C(10)	107.7(4)	C(16)-C(18)-H(18A)	109.5
C(12)-C(11)-H(11)	126.1	C(16)-C(18)-H(18B)	109.5
C(10)-C(11)-H(11)	126.1	H(18A)-C(18)-H(18B)	109.5
C(11)-C(12)-O(2)	114.5(4)	C(16)-C(18)-H(18C)	109.5
C(11)-C(12)-C(13)	131.1(4)	H(18A)-C(18)-H(18C)	109.5

H(18B)-C(18)-H(18C)	109.5	C(26)-C(25)-H(25A)	108.7
C(20)-C(19)-C(15)	114.9(4)	C(24)-C(25)-H(25B)	108.7
C(20)-C(19)-H(19A)	108.6	C(26)-C(25)-H(25B)	108.7
C(15)-C(19)-H(19A)	108.6	H(25A)-C(25)-H(25B)	107.6
C(20)-C(19)-H(19B)	108.6	O(6)-C(26)-C(25)	113.4(4)
C(15)-C(19)-H(19B)	108.6	O(6)-C(26)-C(21)	112.8(3)
H(19A)-C(19)-H(19B)	107.5	C(25)-C(26)-C(21)	113.6(4)
O(4)-C(20)-O(5)	122.0(5)	O(6)-C(26)-H(26)	105.4
O(4)-C(20)-C(19)	126.7(4)	C(25)-C(26)-H(26)	105.4
O(5)-C(20)-C(19)	111.3(4)	C(21)-C(26)-H(26)	105.4
O(7)-C(21)-C(22)	107.2(3)	C(24)-C(27)-C(22)	86.4(3)
O(7)-C(21)-C(30)	102.7(3)	C(24)-C(27)-H(27A)	114.3
C(22)-C(21)-C(30)	111.1(3)	C(22)-C(27)-H(27A)	114.3
O(7)-C(21)-C(26)	107.1(3)	C(24)-C(27)-H(27B)	114.3
C(22)-C(21)-C(26)	113.1(3)	C(22)-C(27)-H(27B)	114.3
C(30)-C(21)-C(26)	114.7(3)	H(27A)-C(27)-H(27B)	111.4
C(21)-C(22)-C(27)	107.2(4)	C(23)-C(28)-H(28A)	109.5
C(21)-C(22)-C(23)	112.5(3)	C(23)-C(28)-H(28B)	109.5
C(27)-C(22)-C(23)	87.4(3)	H(28A)-C(28)-H(28B)	109.5
C(21)-C(22)-H(22)	115.4	C(23)-C(28)-H(28C)	109.5
C(27)-C(22)-H(22)	115.4	H(28A)-C(28)-H(28C)	109.5
C(23)-C(22)-H(22)	115.4	H(28B)-C(28)-H(28C)	109.5
C(28)-C(23)-C(29)	107.1(3)	C(23)-C(29)-H(29A)	109.5
C(28)-C(23)-C(24)	111.7(3)	C(23)-C(29)-H(29B)	109.5
C(29)-C(23)-C(24)	120.0(4)	H(29A)-C(29)-H(29B)	109.5
C(28)-C(23)-C(22)	111.7(4)	C(23)-C(29)-H(29C)	109.5
C(29)-C(23)-C(22)	119.7(3)	H(29A)-C(29)-H(29C)	109.5
C(24)-C(23)-C(22)	85.4(3)	H(29B)-C(29)-H(29C)	109.5
C(25)-C(24)-C(27)	107.9(4)	O(8)-C(30)-C(31)	128.0(4)
C(25)-C(24)-C(23)	112.6(4)	O(8)-C(30)-C(21)	125.4(4)
C(27)-C(24)-C(23)	88.1(3)	C(31)-C(30)-C(21)	106.6(3)
C(25)-C(24)-H(24)	115.1	C(32)-C(31)-C(30)	107.7(4)
C(27)-C(24)-H(24)	115.1	C(32)-C(31)-H(31)	126.2
C(23)-C(24)-H(24)	115.1	C(30)-C(31)-H(31)	126.2
C(24)-C(25)-C(26)	114.2(4)	O(7)-C(32)-C(31)	114.4(4)
C(24)-C(25)-H(25A)	108.7	O(7)-C(32)-C(33)	113.9(3)

C(31)-C(32)-C(33)	131.6(4)	C(36)-C(37)-H(37B)	109.5
C(32)-C(33)-C(34)	120.4(4)	H(37A)-C(37)-H(37B)	109.5
C(32)-C(33)-C(36)	117.4(3)	C(36)-C(37)-H(37C)	109.5
C(34)-C(33)-C(36)	89.0(3)	H(37A)-C(37)-H(37C)	109.5
C(32)-C(33)-H(33)	109.5	H(37B)-C(37)-H(37C)	109.5
C(34)-C(33)-H(33)	109.5	C(36)-C(38)-H(38A)	109.5
C(36)-C(33)-H(33)	109.5	C(36)-C(38)-H(38B)	109.5
C(33)-C(34)-C(35)	88.6(3)	H(38A)-C(38)-H(38B)	109.5
C(33)-C(34)-H(34A)	113.9	C(36)-C(38)-H(38C)	109.5
C(35)-C(34)-H(34A)	113.9	H(38A)-C(38)-H(38C)	109.5
C(33)-C(34)-H(34B)	113.9	H(38B)-C(38)-H(38C)	109.5
C(35)-C(34)-H(34B)	113.9	C(40)-C(39)-C(35)	115.8(4)
H(34A)-C(34)-H(34B)	111.1	C(40)-C(39)-H(39A)	108.3
C(39)-C(35)-C(34)	116.6(4)	C(35)-C(39)-H(39A)	108.3
C(39)-C(35)-C(36)	120.3(3)	C(40)-C(39)-H(39B)	108.3
C(34)-C(35)-C(36)	89.6(3)	C(35)-C(39)-H(39B)	108.3
C(39)-C(35)-H(35)	109.6	H(39A)-C(39)-H(39B)	107.4
C(34)-C(35)-H(35)	109.6	O(9)-C(40)-O(10)	123.6(4)
C(36)-C(35)-H(35)	109.6	O(9)-C(40)-C(39)	124.7(4)
C(38)-C(36)-C(37)	111.2(3)	O(10)-C(40)-C(39)	111.7(4)
C(38)-C(36)-C(35)	114.0(3)	C(6)-O(1)-H(1)	109.5
C(37)-C(36)-C(35)	116.2(3)	C(12)-O(2)-C(1)	108.5(3)
C(38)-C(36)-C(33)	112.9(3)	C(20)-O(5)-H(5)	109.5
C(37)-C(36)-C(33)	114.1(4)	C(26)-O(6)-H(6A)	109.5
C(35)-C(36)-C(33)	86.3(3)	C(32)-O(7)-C(21)	108.5(3)
C(36)-C(37)-H(37A)	109.5	C(40)-O(10)-H(10)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for maimone23. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	28(2)	31(2)	34(2)	2(2)	2(2)	-3(2)
C(2)	29(2)	37(2)	34(2)	0(2)	0(2)	5(2)
C(3)	29(2)	43(2)	32(2)	4(2)	2(2)	4(2)
C(4)	35(3)	52(3)	34(2)	10(2)	2(2)	-2(2)
C(5)	50(3)	37(2)	45(2)	6(2)	-2(2)	-8(2)
C(6)	30(3)	38(2)	43(2)	1(2)	1(2)	8(2)
C(7)	26(2)	71(3)	36(2)	-5(2)	7(2)	-5(2)
C(8)	42(3)	61(3)	36(2)	-3(2)	1(2)	2(3)
C(9)	37(3)	50(3)	38(2)	0(2)	-7(2)	0(2)
C(10)	31(2)	27(2)	33(2)	-7(2)	3(2)	4(2)
C(11)	42(3)	31(2)	28(2)	-4(2)	-2(2)	4(2)
C(12)	39(3)	25(2)	28(2)	-6(2)	5(2)	7(2)
C(13)	38(3)	31(2)	31(2)	-3(2)	2(2)	2(2)
C(14)	49(3)	39(2)	30(2)	-5(2)	2(2)	-1(2)
C(15)	32(3)	33(2)	37(2)	-1(2)	1(2)	1(2)
C(16)	32(2)	30(2)	33(2)	-2(2)	1(2)	1(2)
C(17)	37(3)	33(2)	34(2)	-3(2)	-2(2)	-1(2)
C(18)	41(3)	39(2)	38(2)	2(2)	-3(2)	0(2)
C(19)	39(3)	33(2)	45(2)	7(2)	-4(2)	2(2)
C(20)	47(3)	32(2)	40(2)	5(2)	0(2)	3(2)
C(21)	27(2)	17(2)	37(2)	3(2)	-4(2)	-5(2)
C(22)	29(2)	18(2)	48(2)	4(2)	-7(2)	1(2)
C(23)	32(2)	24(2)	36(2)	-1(2)	-1(2)	-1(2)
C(24)	55(3)	25(2)	38(2)	-5(2)	-8(2)	-11(2)
C(25)	31(3)	39(2)	47(3)	1(2)	-12(2)	-11(2)
C(26)	32(3)	26(2)	46(2)	10(2)	-4(2)	2(2)
C(27)	37(3)	18(2)	55(3)	-1(2)	1(2)	-1(2)
C(28)	44(3)	29(2)	50(3)	-2(2)	-1(2)	3(2)
C(29)	39(3)	31(2)	32(2)	1(2)	-2(2)	2(2)
C(30)	31(2)	26(2)	40(2)	6(2)	-3(2)	-7(2)

C(31)	35(3)	34(2)	31(2)	5(2)	-2(2)	-13(2)
C(32)	26(2)	31(2)	32(2)	-3(2)	5(2)	-4(2)
C(33)	31(2)	24(2)	32(2)	-2(2)	6(2)	-1(2)
C(34)	45(3)	35(2)	30(2)	-6(2)	9(2)	-11(2)
C(35)	32(2)	28(2)	32(2)	-4(2)	8(2)	-8(2)
C(36)	31(2)	18(2)	31(2)	-1(2)	1(2)	1(2)
C(37)	43(3)	26(2)	39(2)	3(2)	0(2)	-2(2)
C(38)	26(2)	21(2)	36(2)	0(2)	5(2)	-2(2)
C(39)	47(3)	25(2)	32(2)	-2(2)	1(2)	-9(2)
C(40)	33(3)	27(2)	37(2)	-5(2)	1(2)	-4(2)
O(1)	60(2)	35(2)	43(2)	-5(1)	2(2)	12(2)
O(2)	30(2)	34(2)	31(1)	2(1)	0(1)	1(1)
O(3)	34(2)	34(2)	48(2)	-7(1)	-6(1)	2(2)
O(4)	94(3)	36(2)	116(3)	15(2)	-68(3)	-13(2)
O(5)	46(2)	32(2)	105(3)	8(2)	-24(2)	-8(2)
O(6)	31(2)	38(2)	56(2)	8(2)	1(2)	-3(2)
O(7)	32(2)	18(1)	32(1)	1(1)	-2(1)	-1(1)
O(8)	43(2)	29(2)	49(2)	12(1)	-10(2)	-10(2)
O(9)	53(2)	44(2)	46(2)	13(2)	-8(2)	-23(2)
O(10)	34(2)	30(2)	66(2)	11(2)	-1(2)	-7(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for maimone23.

	x	y	z	U(eq)
H(2)	-1191	12842	2875	40
H(4)	-1376	15931	2483	48
H(5A)	-3833	16871	2759	53
H(5B)	-1677	17215	2861	53
H(6)	-4637	15822	3086	44
H(7A)	978	14381	2664	53
H(7B)	637	15296	2910	53
H(8A)	-2630	12387	2440	70
H(8B)	-752	13336	2400	70
H(8C)	-2791	13662	2263	70
H(9A)	-5580	14506	2481	63
H(9B)	-5519	14776	2771	63
H(9C)	-5568	13277	2669	63
H(11)	-1542	12710	3618	41
H(13)	-6695	12592	3497	40
H(14A)	-5690	11379	3836	47
H(14B)	-3794	10738	3692	47
H(15)	-7954	10227	3598	41
H(17A)	-4829	9343	3086	52
H(17B)	-3294	10118	3258	52
H(17C)	-4124	10819	3015	52
H(18A)	-7602	11712	3008	59
H(18B)	-9047	11385	3233	59
H(18C)	-8337	10213	3053	59
H(19A)	-4750	8417	3510	47
H(19B)	-6057	8381	3755	47
H(22)	-2613	10005	4539	38
H(24)	711	11667	5002	47
H(25A)	3465	10344	4862	47
H(25B)	3296	11644	4692	47

H(26)	3025	9009	4559	42
H(27A)	16	12115	4510	44
H(27B)	-1824	12193	4705	44
H(28A)	-3386	9351	5023	62
H(28B)	-3176	10924	4979	62
H(28C)	-2027	10228	5202	62
H(29A)	437	8497	5112	51
H(29B)	1365	8354	4842	51
H(29C)	-817	7809	4896	51
H(31)	-968	8918	3844	40
H(33)	288	5918	4165	35
H(34A)	-943	5531	3776	44
H(34B)	-2650	6674	3803	44
H(35)	-2454	4130	4055	37
H(37A)	-1696	5715	4633	54
H(37B)	-1099	4458	4467	54
H(37C)	-3311	4601	4569	54
H(38A)	-5518	6431	4386	42
H(38B)	-4592	7403	4182	42
H(38C)	-3847	7485	4462	42
H(39A)	-5967	5644	3969	42
H(39B)	-5198	4557	3777	42
H(1)	-1369	16850	3264	69
H(5)	-7893	5897	3431	91
H(6A)	3591	11118	4284	62
H(10)	-8714	2885	4056	65

X-Ray crystallographic Analysis 13

A colorless plate 0.050 x 0.040 x 0.020 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 10 seconds per frame using a scan width of 1.0°. Data collection was 100.0% complete to 67.000° in θ. A total of 28889 reflections were collected covering the indices, -8<=*h*<=8, -10<=*k*<=11, -33<=*l*<=33. 3424 reflections were found to be symmetry independent, with an R_{int} of 0.0362. Indexing and unit cell refinement indicated a primitive, orthorhombic lattice. The space group was found to be P 21 21 21 (No. 19). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by iterative methods (SHELXT) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Absolute stereochemistry was unambiguously determined to be *R* at C3, C5, C13, and C15, and *S* at C1 and C6, respectively.

Table 1. Crystal data and structure refinement for **13**.

X-ray ID	maimone24	
Sample/notebook ID	XH-211-3	
Empirical formula	C20 H28 O5	
Formula weight	348.42	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 6.8100(3) Å	α= 90°.
	b = 9.9666(4) Å	β= 90°.
	c = 27.5837(12) Å	γ = 90°.
Volume	1872.18(14) $Å^3$	
Z	4	
Density (calculated)	1.236 Mg/m^3	
Absorption coefficient	0.713 mm^{-1}	
F(000)	752	
Crystal size	0.050 x 0.040 x 0.020 mm^3	
Crystal color/habit	colorless plate	
Theta range for data collection	3.204 to 68.390°.	
Index ranges	-8<=h<=8, -10<=k<=11, -33<=l<=33	
Reflections collected	28889	
Independent reflections	3424 [R(int) = 0.0362]	
Completeness to theta = 67.000°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.929 and 0.896	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3424 / 0 / 232	
Goodness-of-fit on F^2	1.037	
Final R indices [I>2sigma(I)]	R1 = 0.0364, wR2 = 0.0951	
R indices (all data)	R1 = 0.0377, wR2 = 0.0963	
Absolute structure parameter	0.01(5)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.260 and -0.139 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for maimone24. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	4048(3)	3660(2)	978(1)	26(1)
C(2)	3849(4)	3972(3)	435(1)	33(1)
C(3)	1713(4)	4016(2)	272(1)	30(1)
C(4)	668(4)	5235(2)	520(1)	27(1)
C(5)	417(3)	4259(2)	964(1)	23(1)
C(6)	2259(3)	4144(2)	1285(1)	23(1)
C(7)	1787(3)	3201(2)	1706(1)	25(1)
C(8)	2528(3)	1868(2)	1701(1)	26(1)
C(9)	3746(3)	1433(2)	1348(1)	24(1)
C(10)	453(4)	3079(2)	594(1)	28(1)
C(11)	-1337(4)	5516(3)	290(1)	39(1)
C(12)	1750(4)	6559(2)	555(1)	34(1)
C(13)	4417(3)	20(2)	1289(1)	25(1)
C(14)	3615(3)	-1093(2)	1618(1)	27(1)
C(15)	5643(3)	-1785(2)	1582(1)	23(1)
C(16)	6563(3)	-401(2)	1446(1)	25(1)
C(17)	7293(4)	382(3)	1885(1)	34(1)
C(18)	8084(4)	-389(3)	1044(1)	36(1)
C(19)	6275(4)	-2602(2)	2019(1)	27(1)
C(20)	8049(4)	-3452(2)	1933(1)	26(1)
O(1)	4437(2)	2210(2)	992(1)	29(1)
O(2)	2852(2)	5388(2)	1486(1)	29(1)
O(3)	706(3)	3606(2)	2036(1)	35(1)
O(4)	9351(3)	-3181(2)	1648(1)	35(1)
O(5)	8050(3)	-4559(2)	2198(1)	36(1)

Table 3. Bond lengths [Å] and angles [°] for maimone24.

C(1)-O(1)	1.470(3)	C(11)-H(11C)	0.9800
C(1)-C(2)	1.536(3)	C(12)-H(12A)	0.9800
C(1)-C(6)	1.559(3)	C(12)-H(12B)	0.9800
C(1)-H(1)	1.0000	C(12)-H(12C)	0.9800
C(2)-C(3)	1.523(4)	C(13)-C(14)	1.533(3)
C(2)-H(2A)	0.9900	C(13)-C(16)	1.581(3)
C(2)-H(2B)	0.9900	C(13)-H(13)	1.0000
C(3)-C(10)	1.548(3)	C(14)-C(15)	1.547(3)
C(3)-C(4)	1.564(3)	C(14)-H(14A)	0.9900
C(3)-H(3)	1.0000	C(14)-H(14B)	0.9900
C(4)-C(12)	1.514(3)	C(15)-C(19)	1.518(3)
C(4)-C(11)	1.531(4)	C(15)-C(16)	1.560(3)
C(4)-C(5)	1.574(3)	C(15)-H(15)	1.0000
C(5)-C(6)	1.539(3)	C(16)-C(18)	1.518(3)
C(5)-C(10)	1.558(3)	C(16)-C(17)	1.525(3)
C(5)-H(5)	1.0000	C(17)-H(17A)	0.9800
C(6)-O(2)	1.417(3)	C(17)-H(17B)	0.9800
C(6)-C(7)	1.528(3)	C(17)-H(17C)	0.9800
C(7)-O(3)	1.239(3)	C(18)-H(18A)	0.9800
C(7)-C(8)	1.421(3)	C(18)-H(18B)	0.9800
C(8)-C(9)	1.351(3)	C(18)-H(18C)	0.9800
C(8)-H(8)	0.9500	C(19)-C(20)	1.495(3)
C(9)-O(1)	1.337(3)	C(19)-H(19A)	0.9900
C(9)-C(13)	1.489(3)	C(19)-H(19B)	0.9900
C(10)-H(10A)	0.9900	C(20)-O(4)	1.215(3)
C(10)-H(10B)	0.9900	C(20)-O(5)	1.323(3)
C(11)-H(11A)	0.9800	O(2)-H(2)	0.8400
C(11)-H(11B)	0.9800	O(5)-H(5A)	0.8400

O(1)-C(1)-C(2)	103.80(19)	C(6)-C(1)-H(1)	107.9
O(1)-C(1)-C(6)	115.56(18)	C(3)-C(2)-C(1)	112.18(19)
C(2)-C(1)-C(6)	113.44(19)	C(3)-C(2)-H(2A)	109.2
O(1)-C(1)-H(1)	107.9	C(1)-C(2)-H(2A)	109.2
C(2)-C(1)-H(1)	107.9	C(3)-C(2)-H(2B)	109.2

C(1)-C(2)-H(2B)	109.2	C(3)-C(10)-H(10A)	114.3
H(2A)-C(2)-H(2B)	107.9	C(5)-C(10)-H(10A)	114.3
C(2)-C(3)-C(10)	110.03(19)	C(3)-C(10)-H(10B)	114.3
C(2)-C(3)-C(4)	109.18(19)	C(5)-C(10)-H(10B)	114.3
C(10)-C(3)-C(4)	88.07(18)	H(10A)-C(10)-H(10B)	111.5
C(2)-C(3)-H(3)	115.5	C(4)-C(11)-H(11A)	109.5
C(10)-C(3)-H(3)	115.5	C(4)-C(11)-H(11B)	109.5
C(4)-C(3)-H(3)	115.5	H(11A)-C(11)-H(11B)	109.5
C(12)-C(4)-C(11)	107.5(2)	C(4)-C(11)-H(11C)	109.5
C(12)-C(4)-C(3)	118.9(2)	H(11A)-C(11)-H(11C)	109.5
C(11)-C(4)-C(3)	111.6(2)	H(11B)-C(11)-H(11C)	109.5
C(12)-C(4)-C(5)	122.8(2)	C(4)-C(12)-H(12A)	109.5
C(11)-C(4)-C(5)	109.8(2)	C(4)-C(12)-H(12B)	109.5
C(3)-C(4)-C(5)	84.82(17)	H(12A)-C(12)-H(12B)	109.5
C(6)-C(5)-C(10)	107.90(18)	C(4)-C(12)-H(12C)	109.5
C(6)-C(5)-C(4)	113.91(18)	H(12A)-C(12)-H(12C)	109.5
C(10)-C(5)-C(4)	87.36(16)	H(12B)-C(12)-H(12C)	109.5
C(6)-C(5)-H(5)	114.8	C(9)-C(13)-C(14)	120.74(19)
C(10)-C(5)-H(5)	114.8	C(9)-C(13)-C(16)	120.30(19)
C(4)-C(5)-H(5)	114.8	C(14)-C(13)-C(16)	88.60(16)
O(2)-C(6)-C(7)	107.54(18)	C(9)-C(13)-H(13)	108.5
O(2)-C(6)-C(5)	113.05(17)	C(14)-C(13)-H(13)	108.5
C(7)-C(6)-C(5)	108.13(18)	C(16)-C(13)-H(13)	108.5
O(2)-C(6)-C(1)	105.07(18)	C(13)-C(14)-C(15)	88.07(17)
C(7)-C(6)-C(1)	112.72(18)	C(13)-C(14)-H(14A)	114.0
C(5)-C(6)-C(1)	110.37(17)	C(15)-C(14)-H(14A)	114.0
O(3)-C(7)-C(8)	121.5(2)	C(13)-C(14)-H(14B)	114.0
O(3)-C(7)-C(6)	118.9(2)	C(15)-C(14)-H(14B)	114.0
C(8)-C(7)-C(6)	119.53(19)	H(14A)-C(14)-H(14B)	111.2
C(9)-C(8)-C(7)	121.7(2)	C(19)-C(15)-C(14)	116.15(19)
C(9)-C(8)-H(8)	119.1	C(19)-C(15)-C(16)	123.41(19)
C(7)-C(8)-H(8)	119.1	C(14)-C(15)-C(16)	88.87(16)
O(1)-C(9)-C(8)	124.0(2)	C(19)-C(15)-H(15)	108.9
O(1)-C(9)-C(13)	111.12(19)	C(14)-C(15)-H(15)	108.9
C(8)-C(9)-C(13)	124.8(2)	C(16)-C(15)-H(15)	108.9
C(3)-C(10)-C(5)	85.93(17)	C(18)-C(16)-C(17)	110.8(2)

C(18)-C(16)-C(15)	117.11(19)	H(18A)-C(18)-H(18C)	109.5
C(17)-C(16)-C(15)	113.15(19)	H(18B)-C(18)-H(18C)	109.5
C(18)-C(16)-C(13)	115.4(2)	C(20)-C(19)-C(15)	113.99(19)
C(17)-C(16)-C(13)	112.51(19)	C(20)-C(19)-H(19A)	108.8
C(15)-C(16)-C(13)	85.91(16)	C(15)-C(19)-H(19A)	108.8
C(16)-C(17)-H(17A)	109.5	C(20)-C(19)-H(19B)	108.8
C(16)-C(17)-H(17B)	109.5	C(15)-C(19)-H(19B)	108.8
H(17A)-C(17)-H(17B)	109.5	H(19A)-C(19)-H(19B)	107.6
C(16)-C(17)-H(17C)	109.5	O(4)-C(20)-O(5)	122.8(2)
H(17A)-C(17)-H(17C)	109.5	O(4)-C(20)-C(19)	124.5(2)
H(17B)-C(17)-H(17C)	109.5	O(5)-C(20)-C(19)	112.7(2)
C(16)-C(18)-H(18A)	109.5	C(9)-O(1)-C(1)	121.64(18)
C(16)-C(18)-H(18B)	109.5	C(6)-O(2)-H(2)	109.5
H(18A)-C(18)-H(18B)	109.5	C(20)-O(5)-H(5A)	109.5
C(16)-C(18)-H(18C)	109.5		

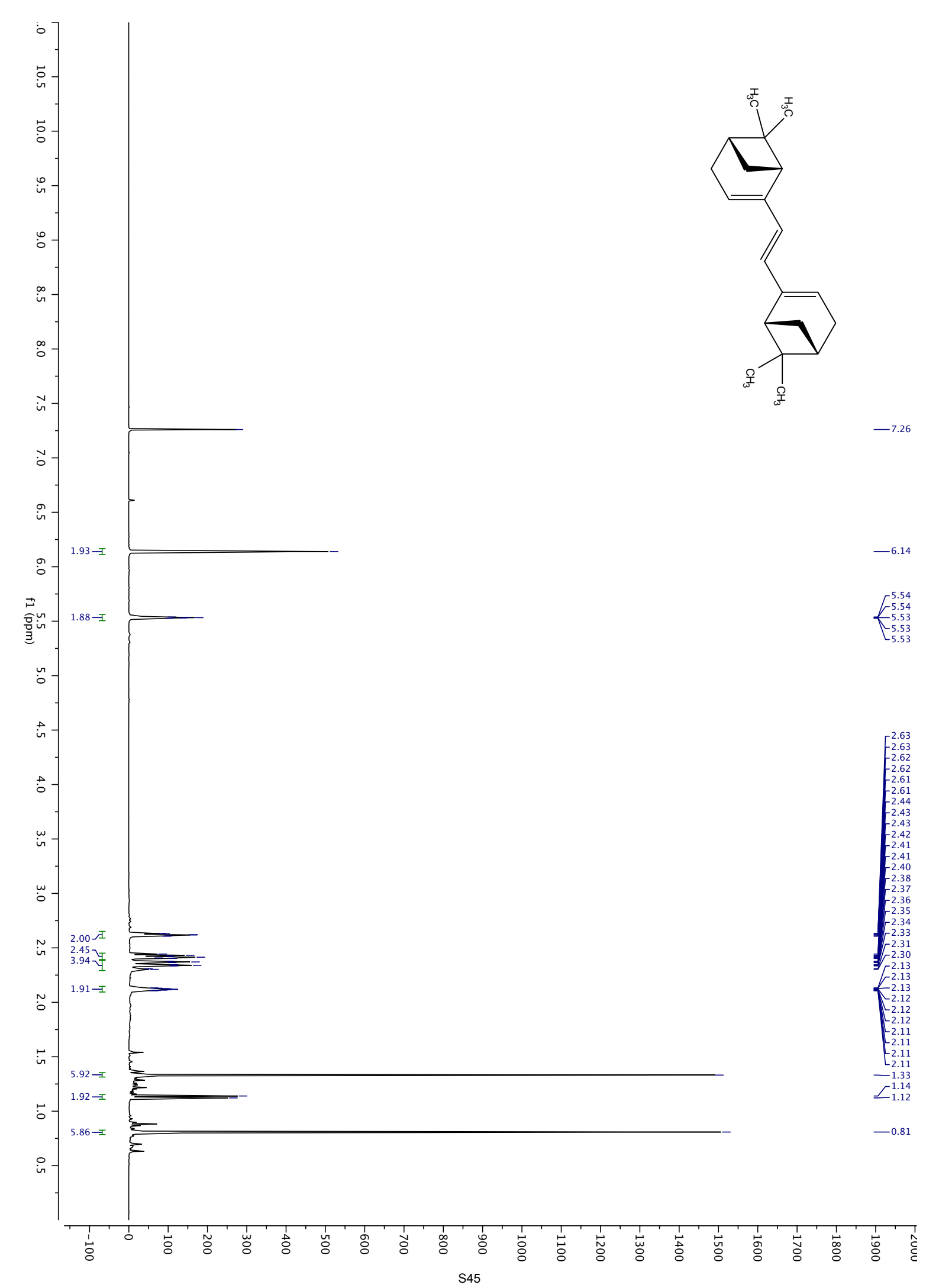
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for maimone24. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	24(1)	18(1)	36(1)	1(1)	3(1)	2(1)
C(2)	39(1)	24(1)	35(1)	2(1)	14(1)	5(1)
C(3)	44(1)	23(1)	22(1)	1(1)	2(1)	-1(1)
C(4)	32(1)	24(1)	25(1)	5(1)	0(1)	3(1)
C(5)	22(1)	21(1)	25(1)	3(1)	2(1)	0(1)
C(6)	23(1)	17(1)	29(1)	-1(1)	-1(1)	3(1)
C(7)	28(1)	26(1)	22(1)	-1(1)	-1(1)	6(1)
C(8)	31(1)	22(1)	25(1)	3(1)	-2(1)	6(1)
C(9)	24(1)	21(1)	27(1)	2(1)	-3(1)	4(1)
C(10)	34(1)	24(1)	27(1)	2(1)	-4(1)	-4(1)
C(11)	41(1)	42(2)	34(1)	10(1)	-7(1)	9(1)
C(12)	47(2)	21(1)	34(1)	6(1)	7(1)	3(1)
C(13)	27(1)	22(1)	26(1)	-3(1)	-2(1)	6(1)
C(14)	26(1)	20(1)	36(1)	0(1)	-2(1)	3(1)
C(15)	27(1)	19(1)	25(1)	-1(1)	-3(1)	4(1)
C(16)	24(1)	21(1)	29(1)	2(1)	-1(1)	6(1)
C(17)	32(1)	24(1)	45(1)	-3(1)	-9(1)	1(1)
C(18)	33(1)	31(1)	44(1)	11(1)	9(1)	10(1)
C(19)	32(1)	22(1)	26(1)	2(1)	1(1)	5(1)
C(20)	31(1)	24(1)	23(1)	-1(1)	-3(1)	5(1)
O(1)	32(1)	21(1)	35(1)	1(1)	8(1)	8(1)
O(2)	30(1)	21(1)	38(1)	-7(1)	-2(1)	3(1)
O(3)	49(1)	30(1)	26(1)	4(1)	7(1)	17(1)
O(4)	34(1)	27(1)	43(1)	6(1)	7(1)	10(1)
O(5)	48(1)	29(1)	31(1)	6(1)	9(1)	17(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for maimone24.

	x	y	z	U(eq)
H(1)	5244	4132	1101	31
H(2A)	4474	4848	366	39
H(2B)	4553	3277	247	39
H(3)	1494	3929	-85	35
H(5)	-845	4363	1146	27
H(8)	2157	1268	1952	31
H(10A)	1150	2268	710	34
H(10B)	-846	2851	456	34
H(11A)	-2121	6072	510	59
H(11B)	-2020	4666	231	59
H(11C)	-1154	5990	-18	59
H(12A)	995	7181	757	51
H(12B)	1910	6940	230	51
H(12C)	3045	6411	701	51
H(13)	4212	-252	944	30
H(14A)	2529	-1616	1472	33
H(14B)	3273	-788	1949	33
H(15)	5657	-2376	1289	28
H(17A)	7606	1304	1788	51
H(17B)	8473	-50	2016	51
H(17C)	6268	397	2135	51
H(18A)	8288	534	932	54
H(18B)	7619	-941	773	54
H(18C)	9325	-754	1166	54
H(19A)	6551	-1982	2291	32
H(19B)	5173	-3188	2118	32
H(2)	1857	5817	1578	44
H(5A)	9021	-5034	2122	54


H3C
H3C
CH3
CH3
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1.88
2.00
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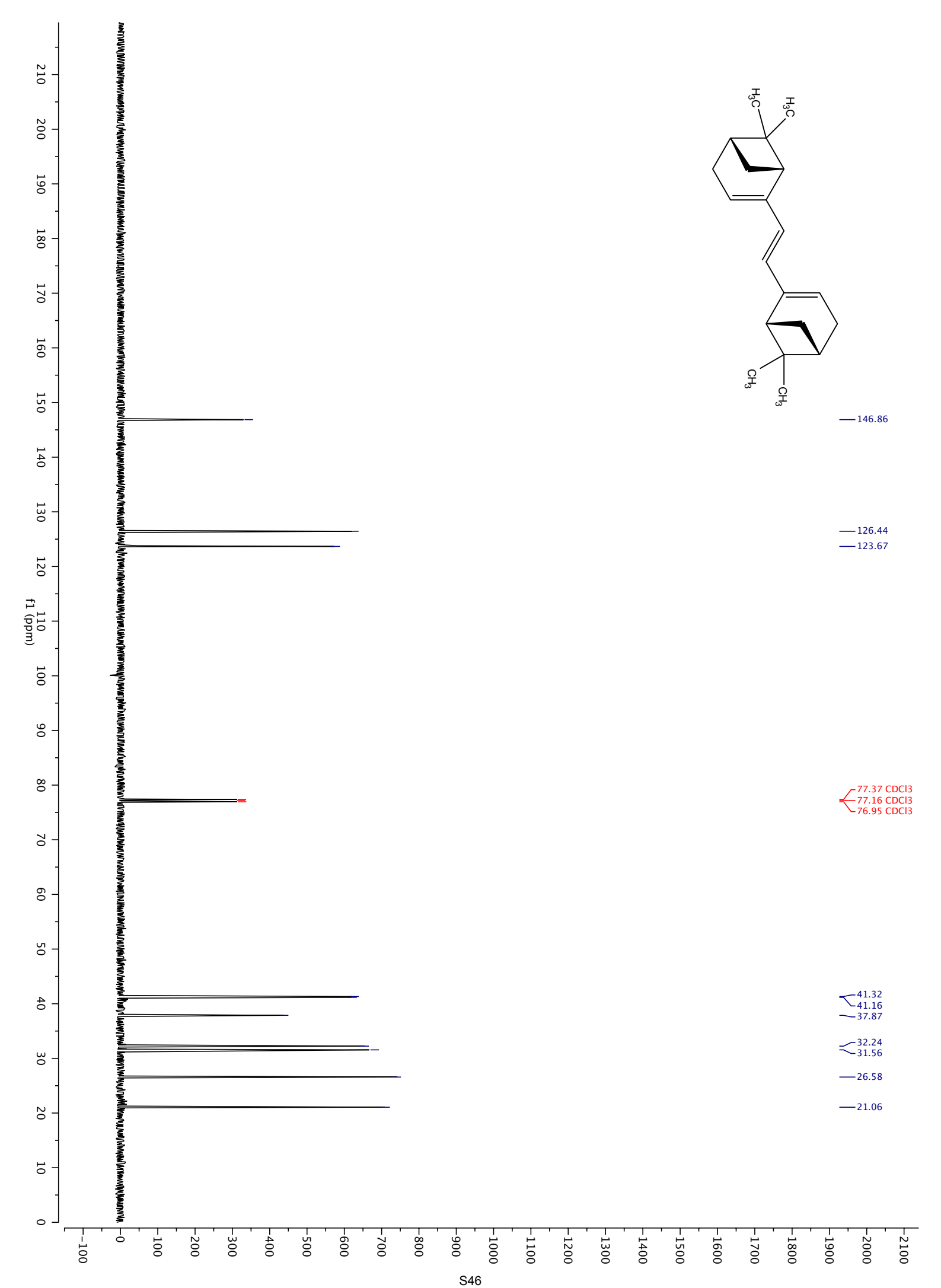
3.94
1.91
5.92
1.92
5.86
7.26
6.14
5.54
5.54
5.53
5.53
5.53
2.63
2.63
2.62
2.62
2.61
2.61
2.44
2.43
2.43
2.42
2.41
2.41
2.40
2.38
2.37
2.36
2.35
2.34
2.33
2.31
2.30
2.13
2.13
2.13
2.12
2.12
2.12
2.11
2.11
2.11
2.11
1.33
1.14
1.12
0.81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77.16 CDCl3
76.95 CDCl3
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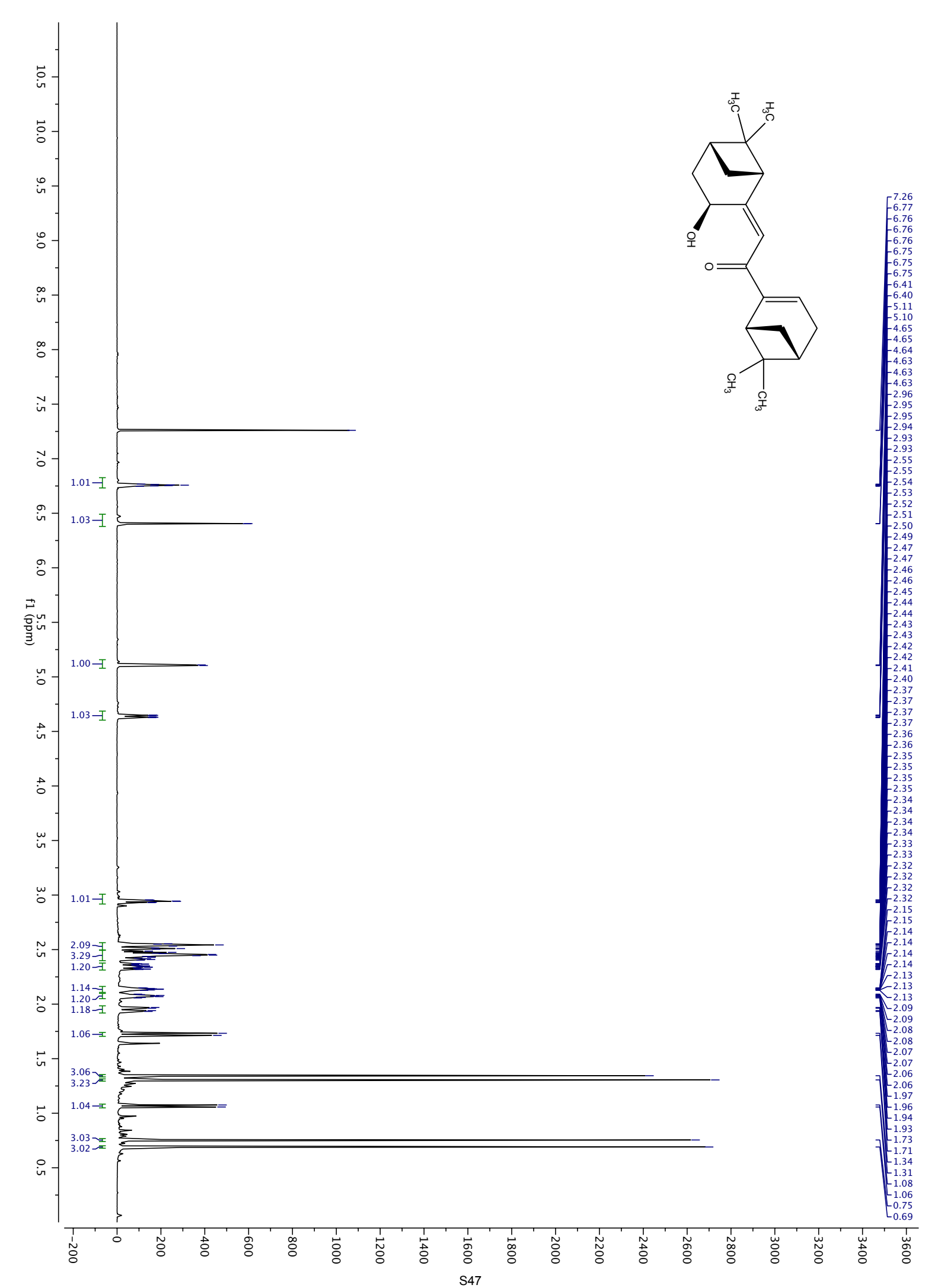
41.16
37.87
32.24
31.56
26.58
21.06
H3C
H3C
CH3
CH3


f1 (ppm)
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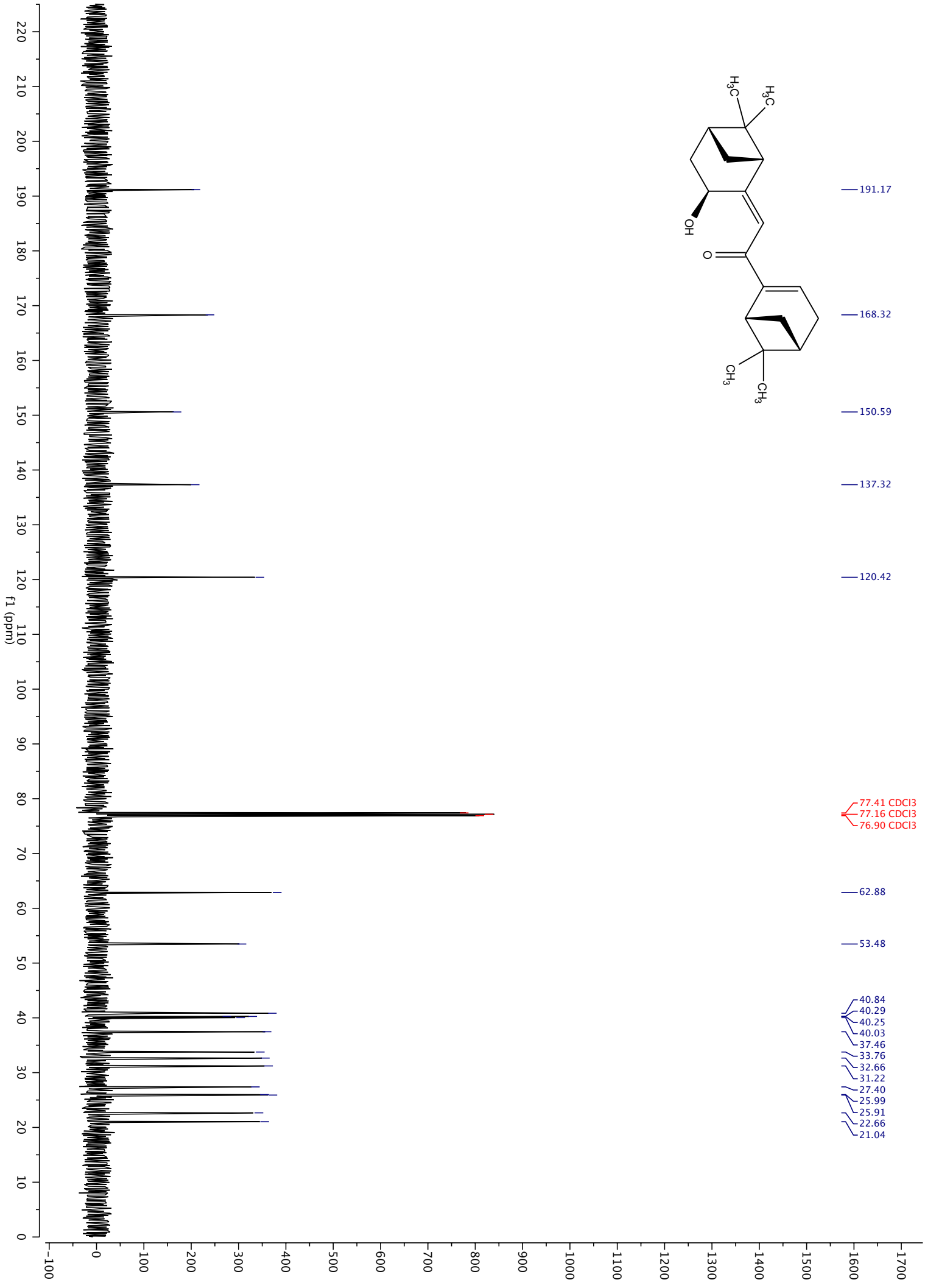
1.00
1.03
1.01
2.09
3.29
1.20
1.14
1.20
1.18
1.06
3.06
3.23
1.04
3.03
3.02
OH
O
CH3
CH3
H3C
H3C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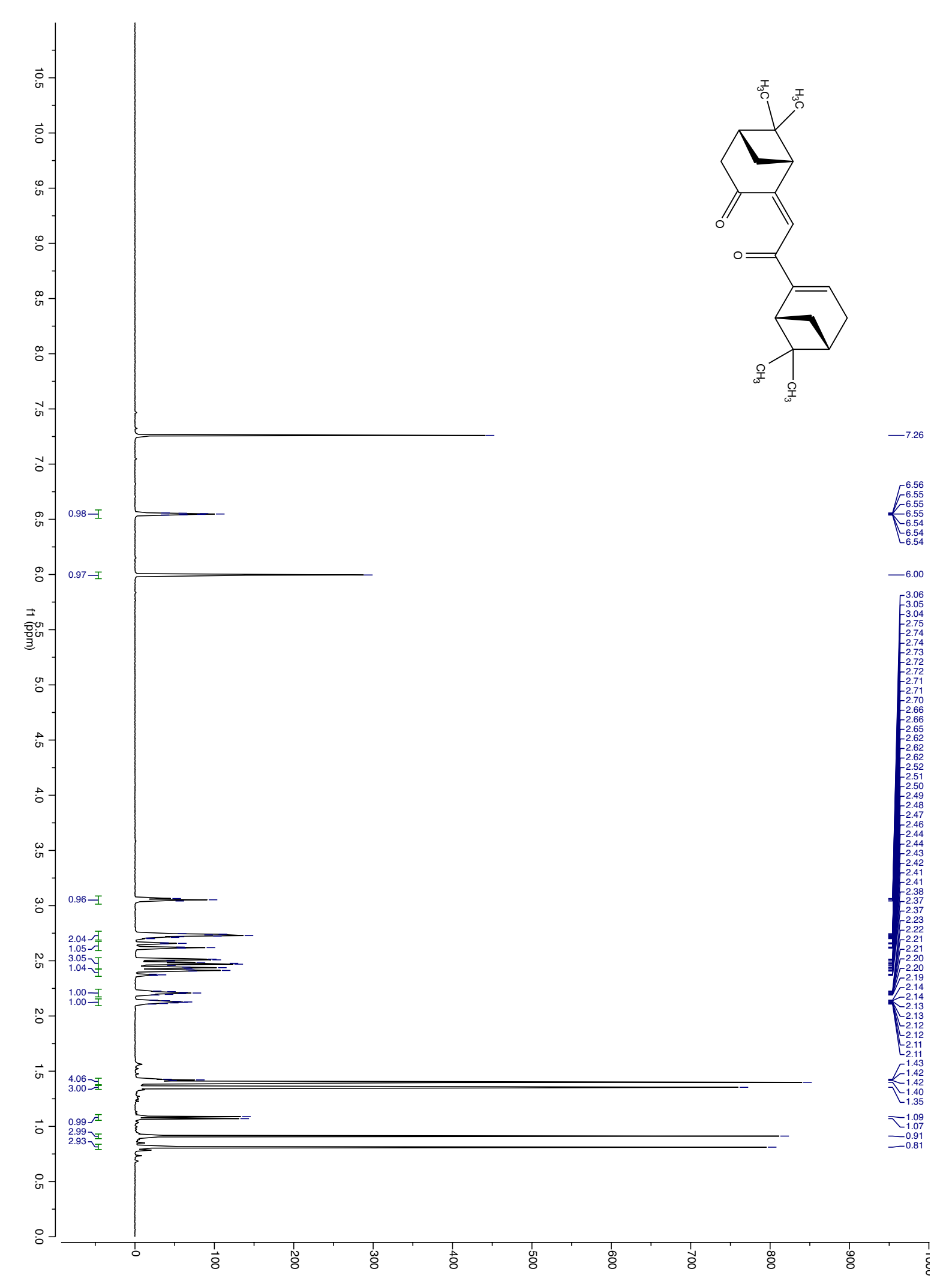
168.32
150.59
137.32
120.42
77.41 CDCl3
77.16 CDCl3
76.90 CDCl3
62.88
53.48
40.84
40.29
40.25
40.03
37.46
33.76
32.66
31.22
27.40
25.99
25.91
22.66
21.04
f1 (ppm)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1.00
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2.99
2.93
7.26
6.56
6.55
6.55
6.55
6.54
6.54
6.54
6.00
3.06
3.05
3.04
2.75
2.74
2.74
2.73
2.72
2.72
2.71
2.71
2.70
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2.66
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2.62
2.62
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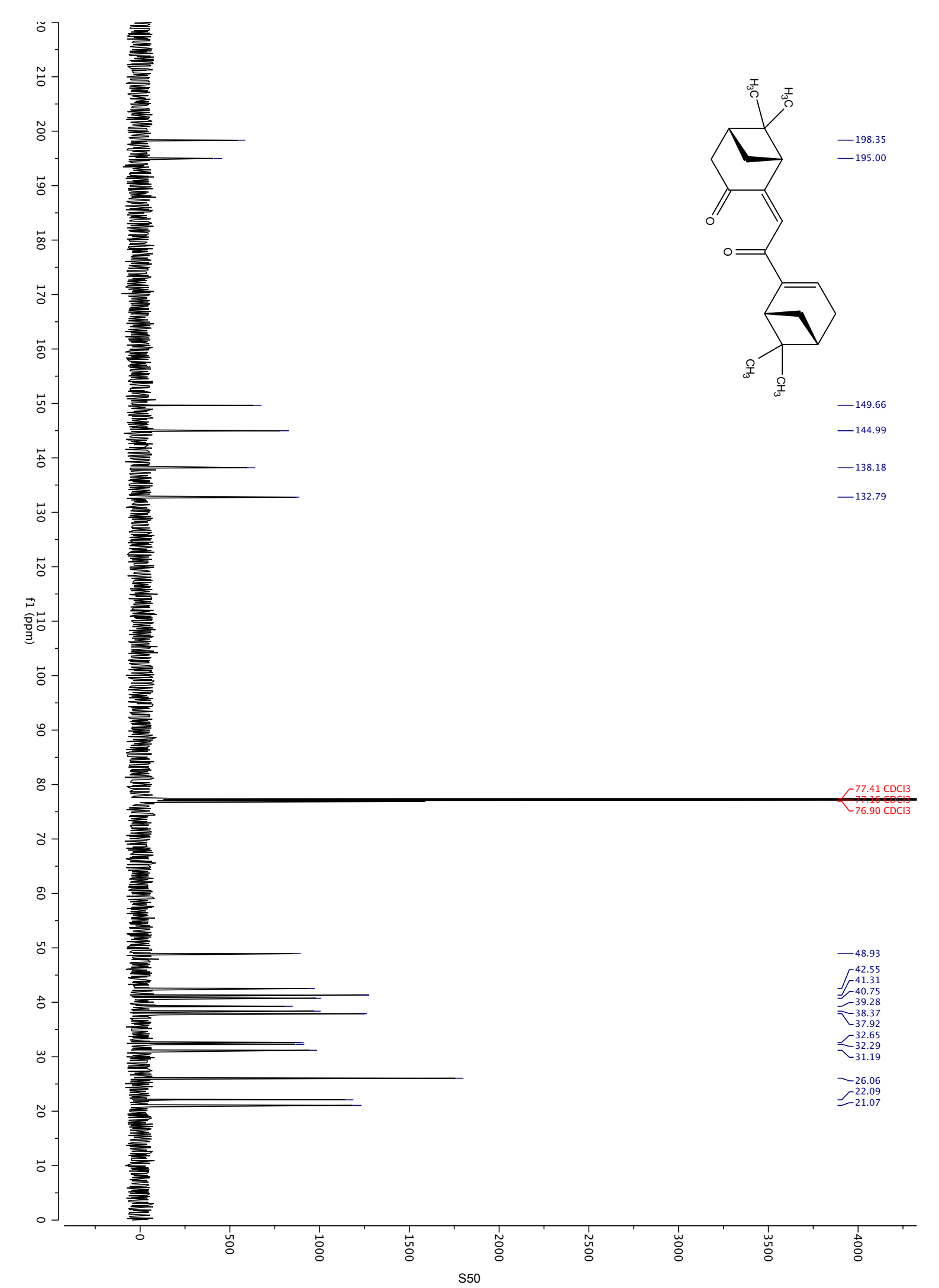
2.43
2.42
2.41
2.41
2.38
2.37
2.37
2.23
2.22
2.21
2.21
2.20
2.20
2.19
2.14
2.14
2.13
2.13
2.12
2.12
2.11
2.11
1.43
1.42
1.42
1.40
1.35
1.09
1.07
0.91
0.81
H3C
H3C
O
O
CH3
CH3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144.99
138.18
132.79
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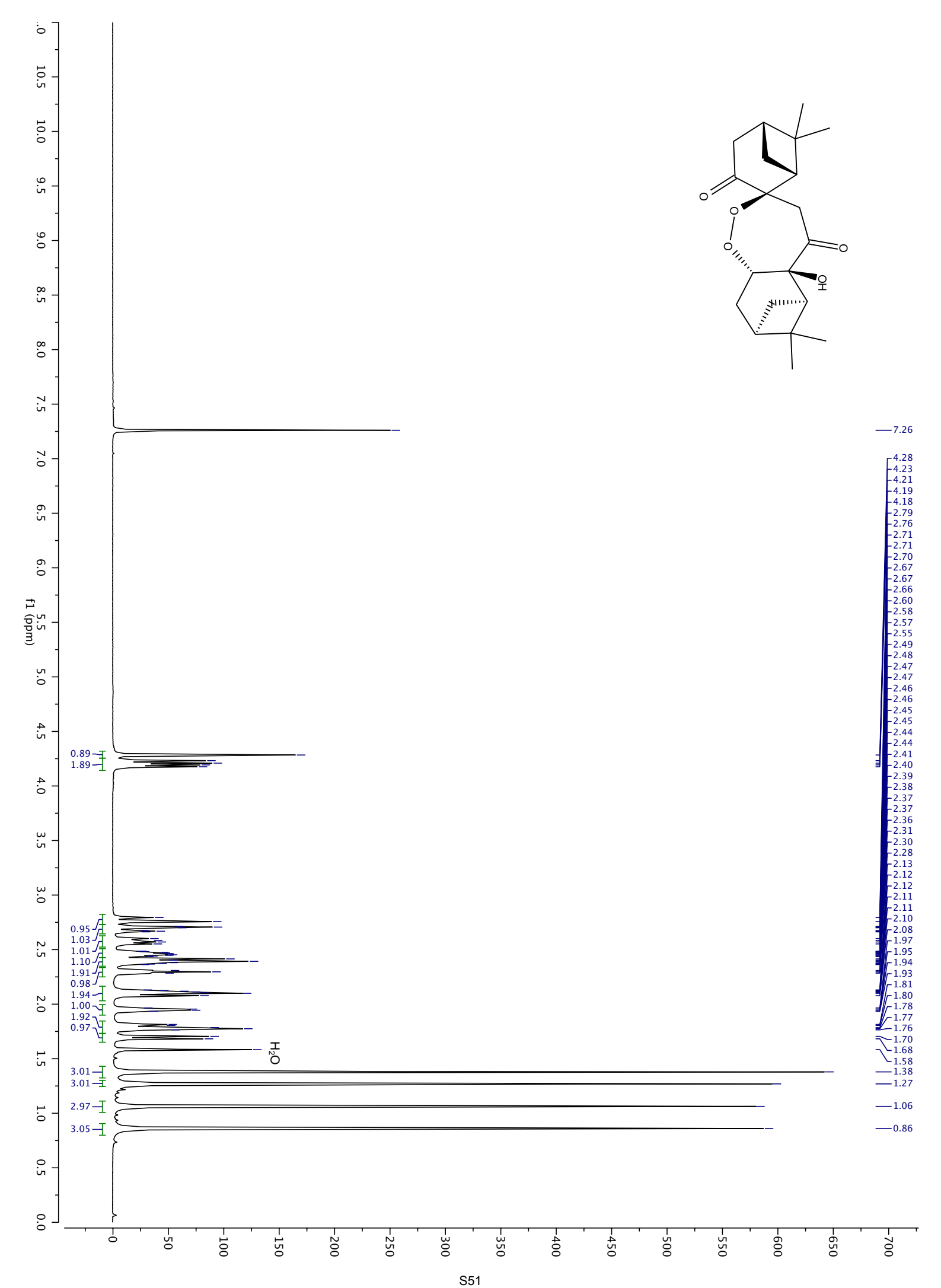
77.16 CDCl3
76.90 CDCl3
48.93
42.55
41.31
40.75
39.28
38.37
37.92
32.65
32.29
31.19
26.06
22.09
21.07
f1 (ppm)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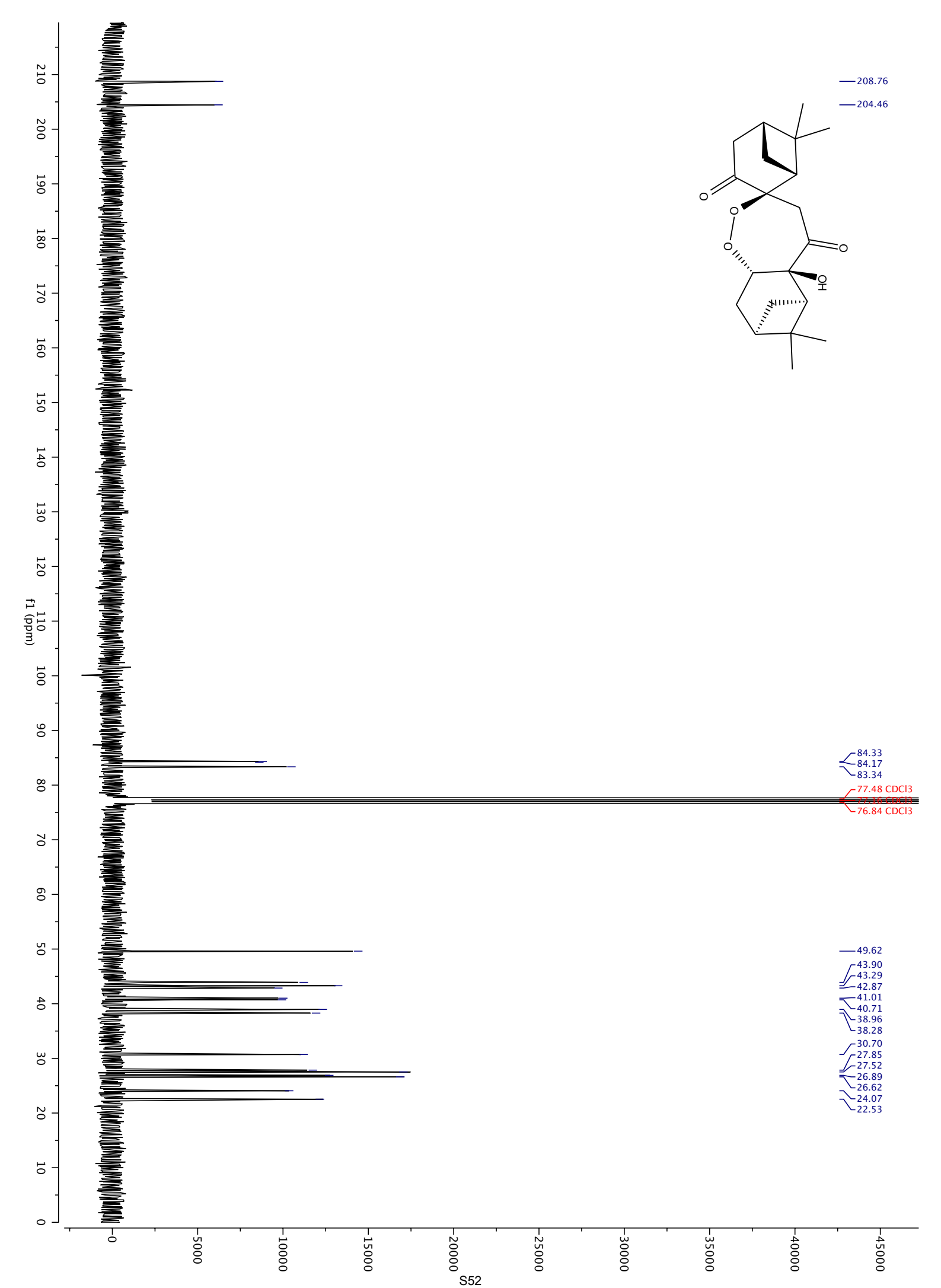
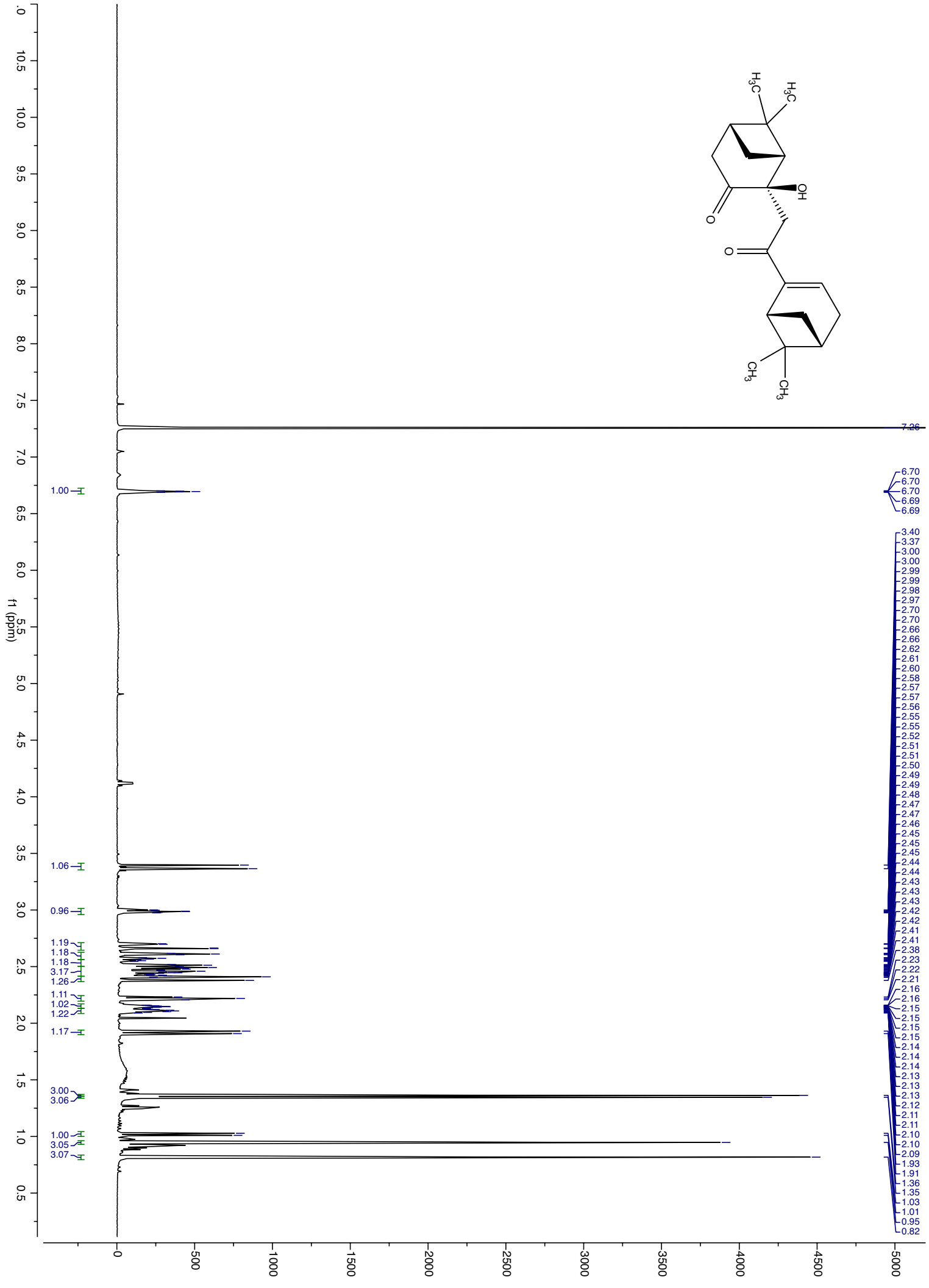
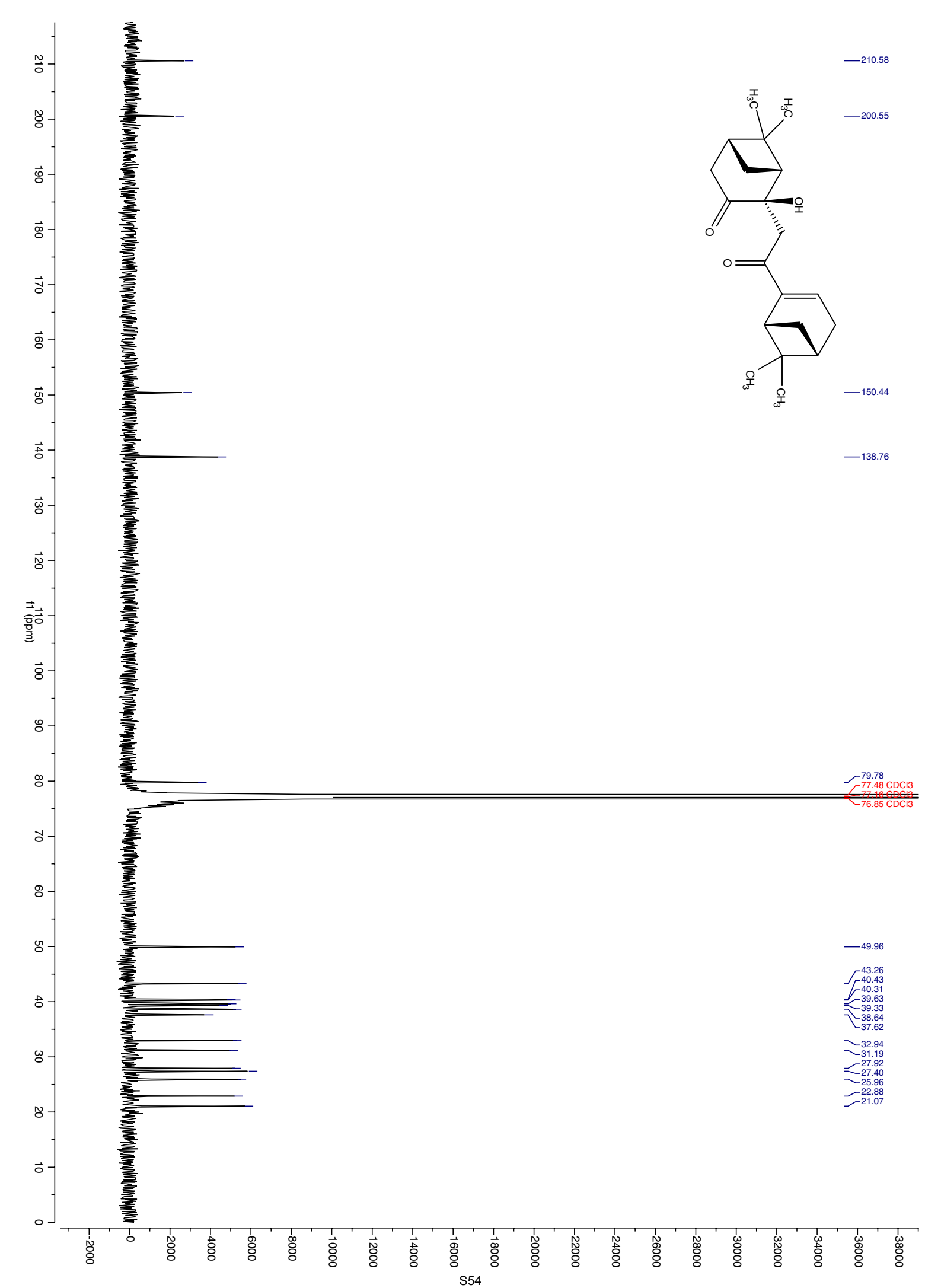
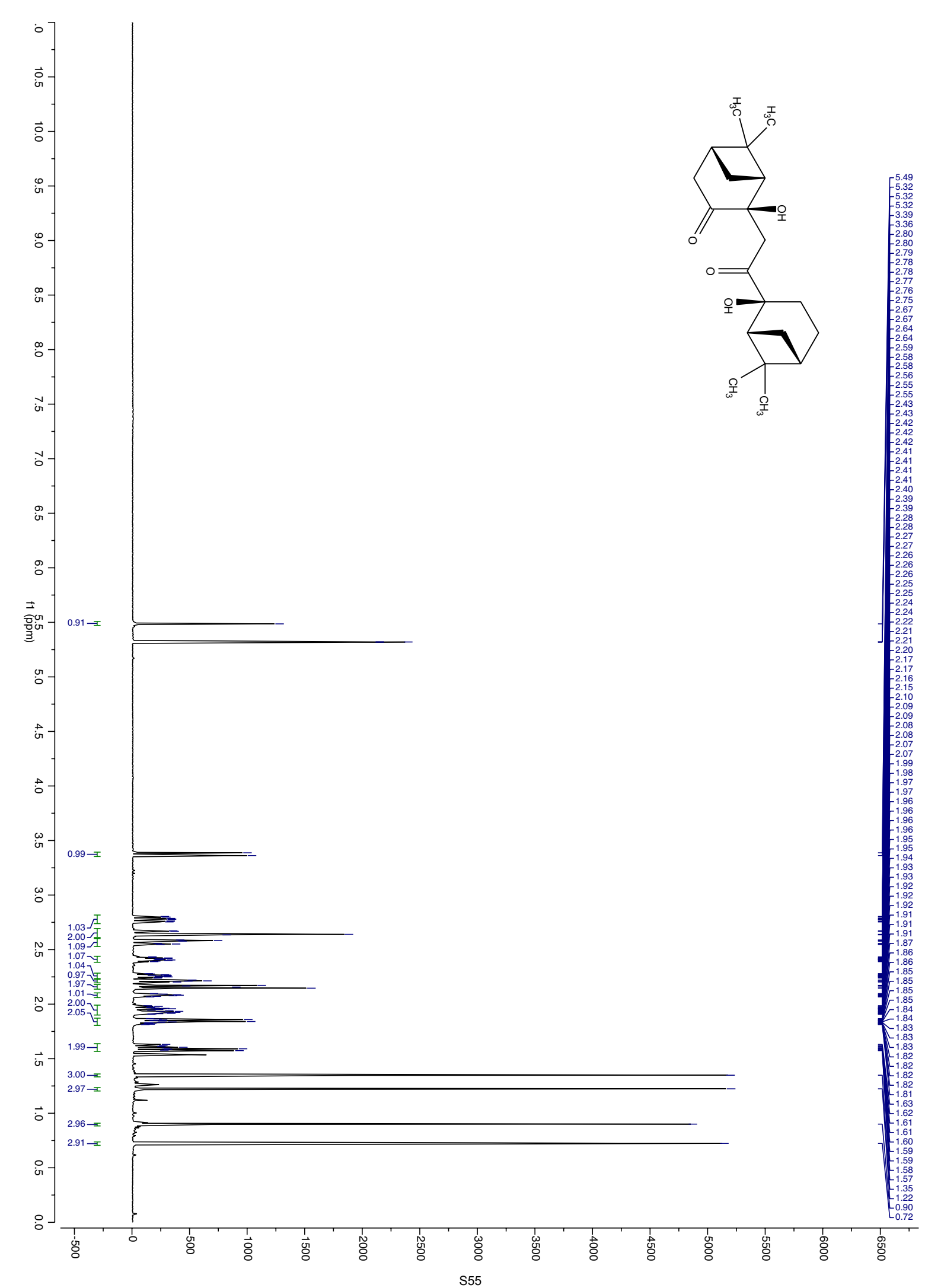
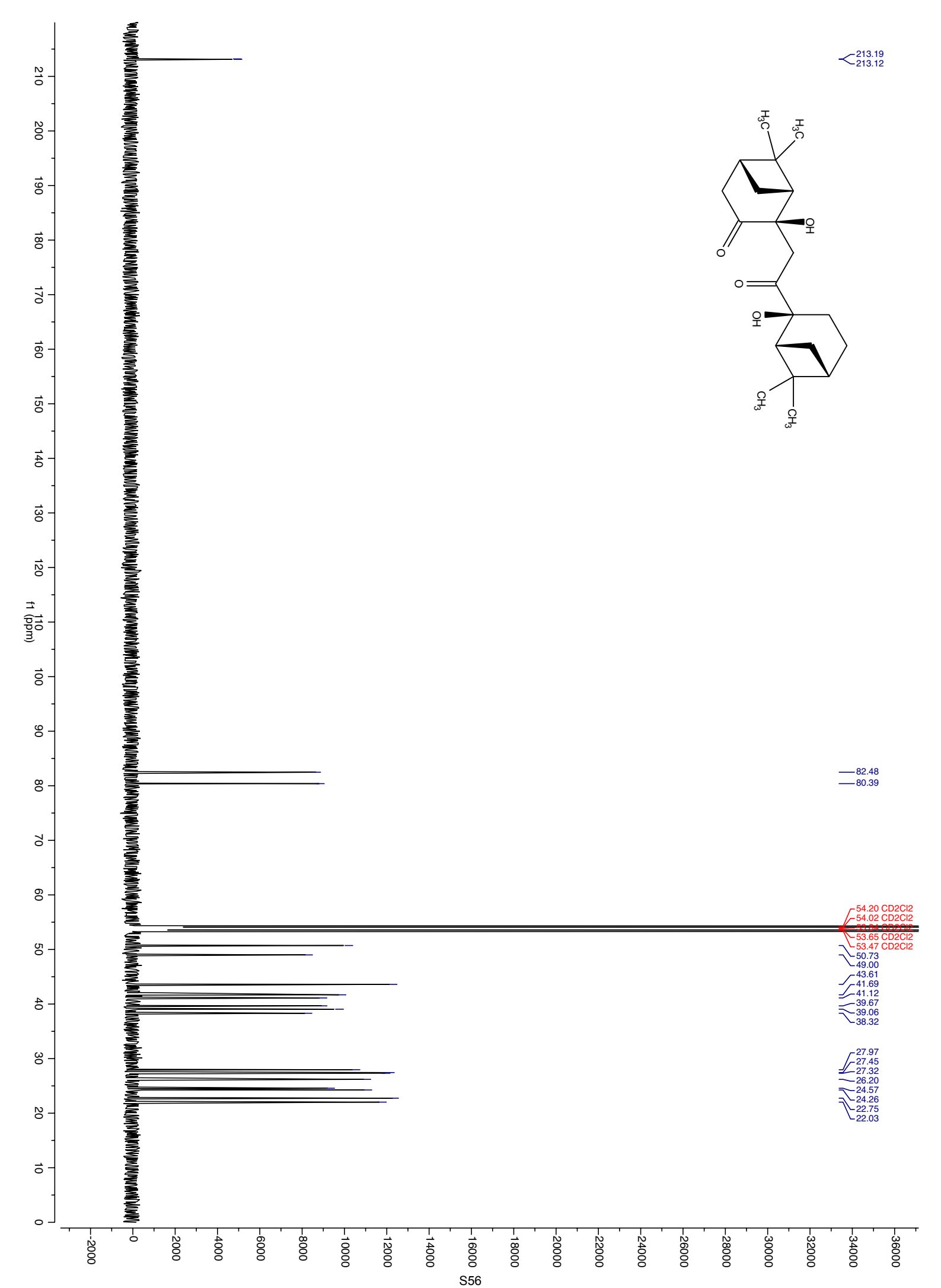
0.95
1.03
1.01
1.10
1.91
0.98
1.94
1.00
1.92
0.97
3.01
3.01
2.97
3.05
7.26
4.28
4.23
4.21
4.19
4.18
2.79
2.76
2.71
2.71
2.70
2.67
2.67
2.66
2.60
2.58
2.57
2.55
2.49
2.48
2.47
2.47
2.46
2.46
2.45
2.45
2.44
2.44
2.41
2.40
2.39
2.38
2.37
2.37
2.36
2.31
2.30
2.28
2.13
2.12
2.12
2.11
2.11
2.10
2.08
1.97
1.95
1.94
1.93
1.81
1.80
1.78
1.77
1.76
1.70
1.68
1.58
1.38
1.27
1.06
0.86
O
O
O
O
OH

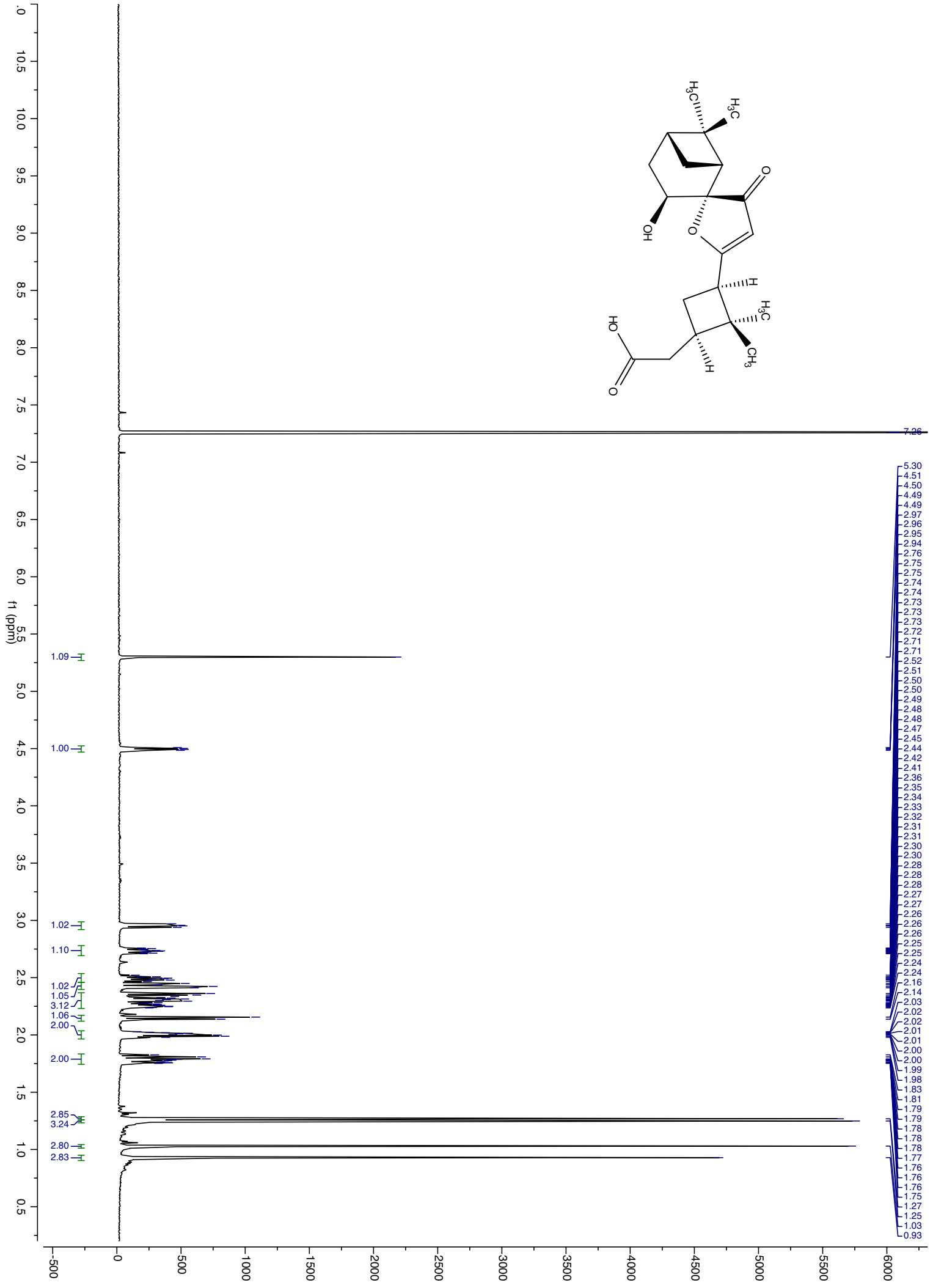

208.76
204.46
84.33
84.17
83.34
77.48 CDCl3
76.84 CDCl3
49.62
43.90
43.29
42.87
41.01
40.71
38.96
38.28
30.70
27.85
27.52
26.89
26.62
24.07
22.53
f1 (ppm)
0
5000
10000
15000
20000
25000
30000
35000
40000
45000

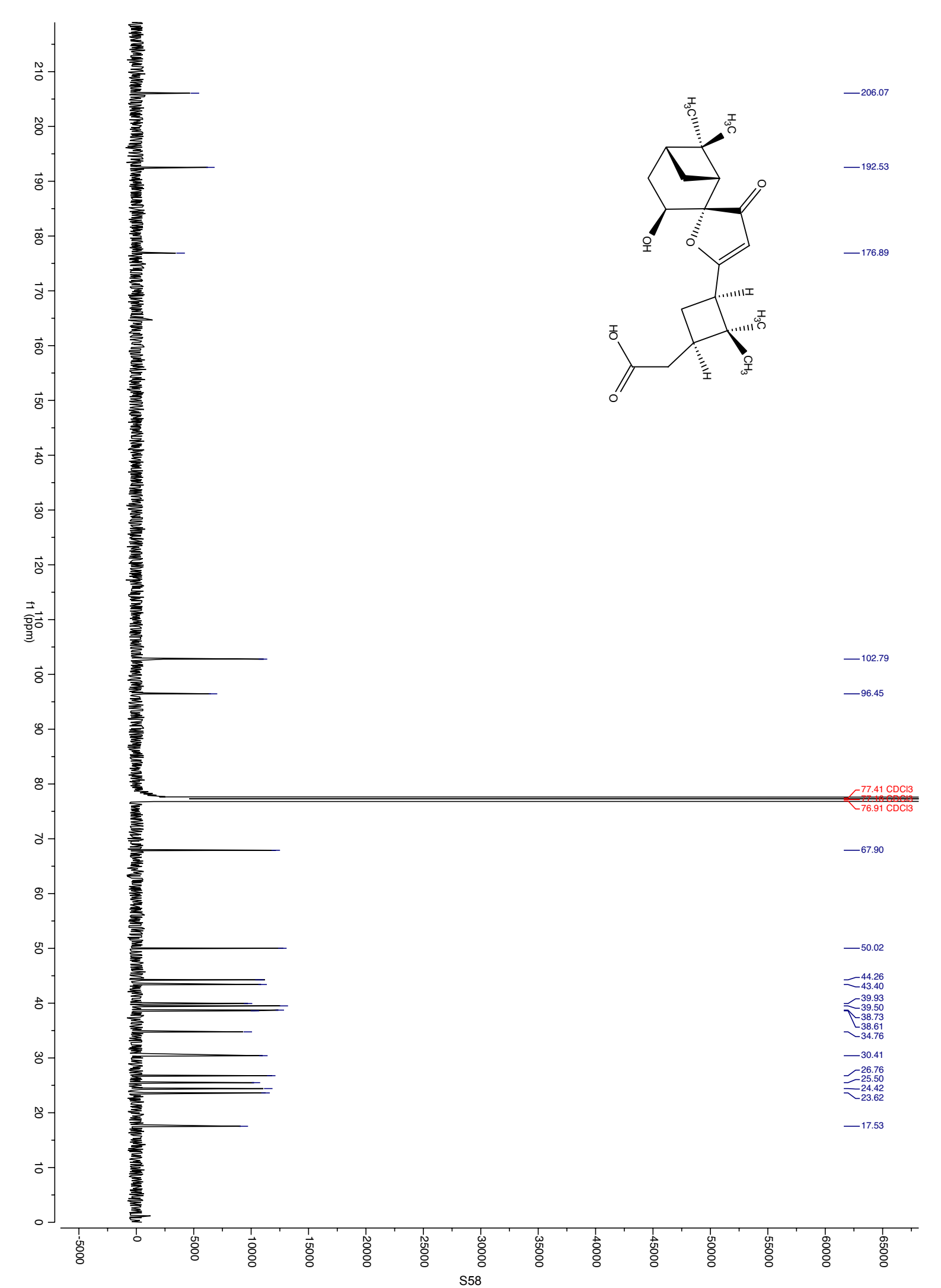

f1 (ppm)
7.26
1.00
1.06
0.96
1.19
1.18
1.18
3.17
1.26
1.11
1.02
1.22
1.17
3.00
3.06
1.00
3.05
3.07
6.70
6.70
6.70
6.69
6.69
3.40
3.37
3.00
3.00
2.99
2.99
2.98
2.97
2.70
2.70
2.66
2.66
2.62
2.61
2.60
2.58
2.57
2.57
2.56
2.55
2.55
2.52
2.51
2.51
2.50
2.49
2.49
2.48
2.47
2.47
2.46
2.45
2.45
2.45
2.44
2.44
2.43
2.43
2.43
2.42
2.42
2.41
2.41
2.38
2.23
2.22
2.21
2.16
2.16
2.15
2.15
2.15
2.15
2.14
2.14
2.14
2.13
2.13
2.13
2.12
2.11
2.11
2.10
2.10
2.09
1.93
1.91
1.36
1.35
1.03
1.01
0.95
0.82
H3C
H3C
OH
O
O
CH3
CH3

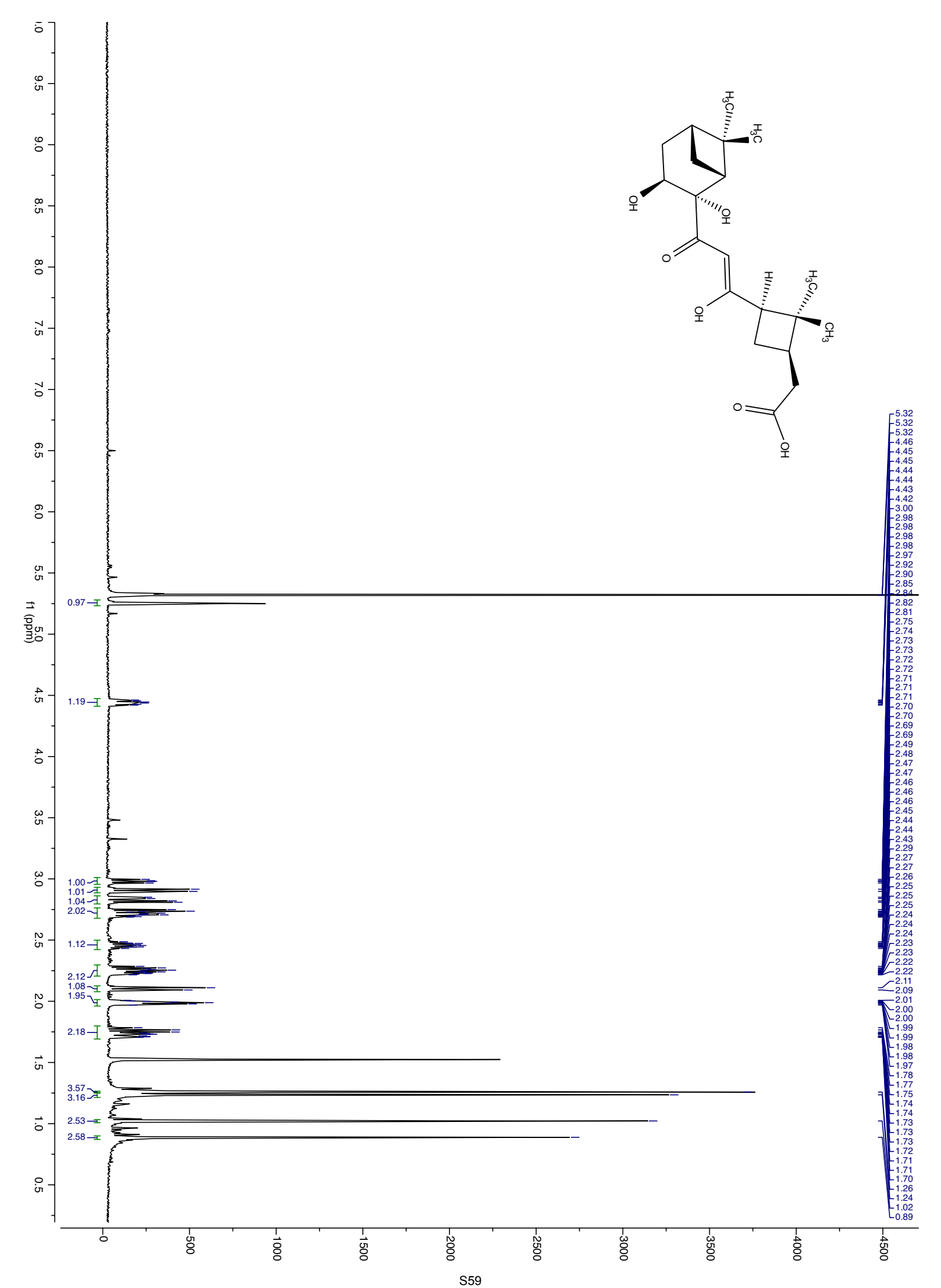

210.58
200.55
150.44
138.76
79.78
77.48 CDCl3
77.16 CDCl3
76.85 CDCl3
49.96
43.26
40.43
40.31
39.63
39.33
38.64
37.62
32.94
31.19
27.92
27.40
25.96
22.88
21.07
H3C
H3C
OH
O
O
CH3
CH3
f1 (ppm)

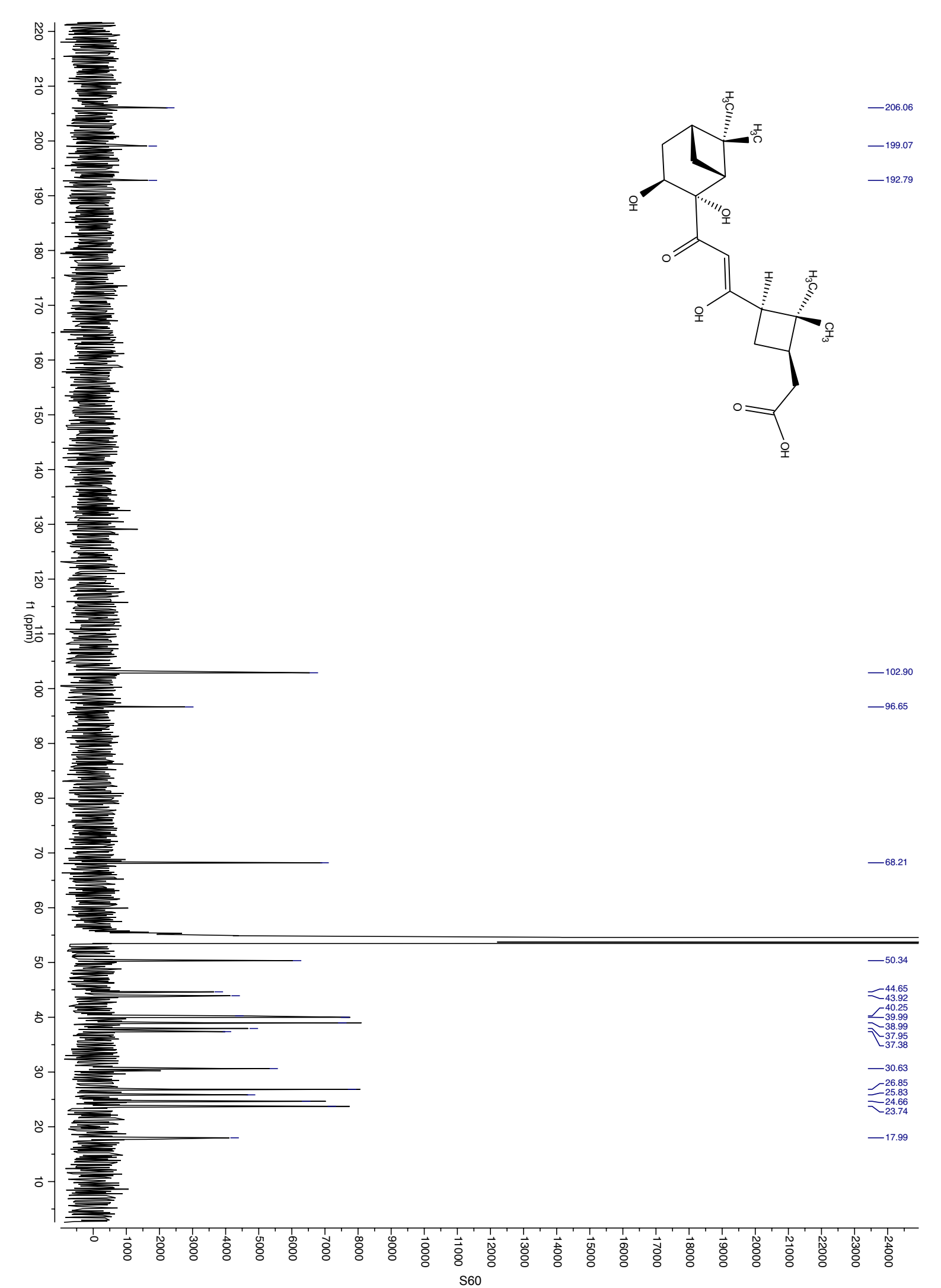

f1 (ppm)
0.91
0.99
1.03
2.00
1.09
1.07
1.04
0.97
1.97
1.01
2.00
2.05
1.99
3.00
2.97
2.96
2.91
H3C
H3C
OH
O
O
OH
CH3
CH3

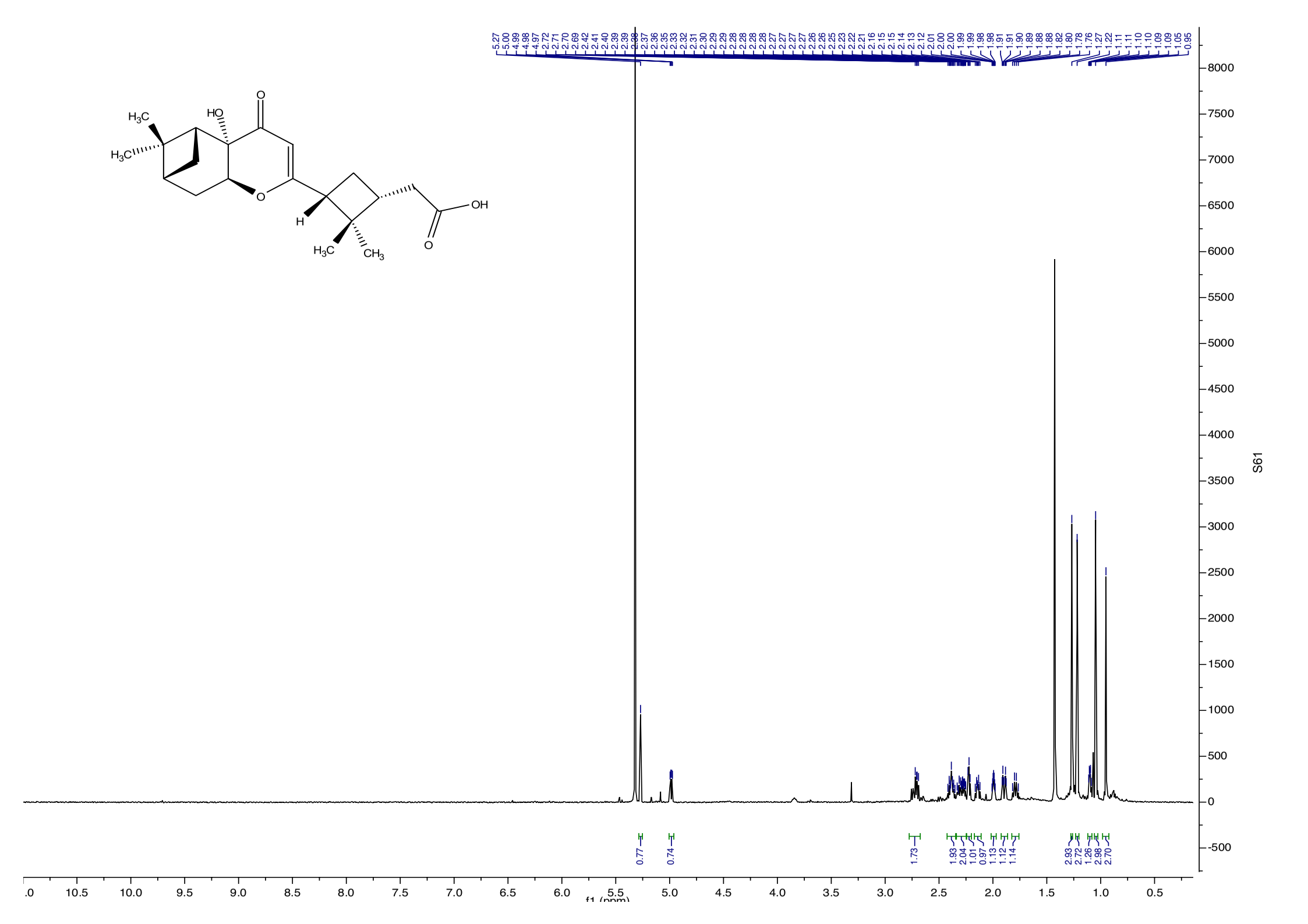

213.19
213.12
82.48
80.39
54.20 CD2Cl2
54.02 CD2Cl2
53.65 CD2Cl2
53.47 CD2Cl2
50.73
49.00
43.61
41.69
41.12
39.67
39.06
38.32
27.97
27.45
27.32
26.20
24.57
24.26
22.75
22.03
f1 (ppm)


f1 (ppm)
7.26
1.09
1.00
1.02
1.10
1.02
1.05
3.12
1.06
2.00
2.00
2.85
3.24
2.80
2.83


206.07
192.53
176.89
102.79
96.45
77.41 CDCl3
76.91 CDCl3
67.90
50.02
44.26
43.40
39.93
39.50
38.73
38.61
34.76
30.41
26.76
25.50
24.42
23.62
17.53
f1 (ppm)
H3C
H3C
O
OH
O
H
H3C
CH3
H
HO
O


f1 (ppm)
0.97
1.19
1.00
1.01
1.04
2.02
1.12
2.12
1.08
1.95
2.18
3.57
3.16
2.53
2.58
OH
O
H3C
CH3
H


206.06
199.07
192.79
102.90
96.65
68.21
50.34
44.65
43.92
40.25
39.99
38.99
37.95
37.38
30.63
26.85
25.83
24.66
23.74
17.99
f1 (ppm)
H3C
H3C
OH
OH
O
OH
H
H3C
CH3
O
OH


H3C
HO
O
H3C
O
H
OH
O
H3C
CH3
5.27 5.00 4.99 4.98 4.97 2.72 2.71 2.70 2.69 2.42 2.41 2.40 2.39 2.39 2.38 2.37 2.36 2.35 2.33 2.32 2.31 2.30 2.29 2.29 2.28 2.28 2.28 2.28 2.27 2.27 2.27 2.26 2.26 2.25 2.23 2.22 2.21 2.16 2.15 2.15 2.14 2.13 2.12 2.01 2.00 2.00 1.99 1.99 1.98 1.98 1.91 1.91 1.90 1.89 1.88 1.88 1.82 1.80 1.78 1.76 1.27 1.22 1.11 1.11 1.10 1.10 1.09 1.09 1.05 0.95
8000 7500 7000 6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500
S61
0.77 0.74 1.73 1.93 2.04 1.01 0.97 1.13 1.12 1.14 2.93 2.72 1.26 2.98 2.70
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)