

## Supporting Information

Biomimetic Total Synthesis of Plakortone Q *via* Acid-mediated Tandem Cyclization

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## Experimental

### General experimental procedures

Optical rotations were measured with a JASCO P-1030 polarimeter. IR spectra were recorded with a JASCO FT-IR/620 spectrometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker Biospin AVANCE III HD 400 (400 MHz for  $^1\text{H}$ , 100 MHz for  $^{13}\text{C}$ ) and a Bruker Biospin AVANCE III HD 500 (500 MHz for  $^1\text{H}$ , 125 MHz for  $^{13}\text{C}$ ). The reported chemical shifts ( $\delta$ ) in parts per million (ppm) were relative to the internal  $\text{CHCl}_3$  (7.26 ppm for  $^1\text{H}$  and 77.0 ppm for  $^{13}\text{C}$ ); the coupling constant ( $J$ ) values were measured in hertz. The coupling patterns are denoted as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), and br (broad). HR-ESI-MS spectra were obtained using a Micromass LCT spectrometer with a time-of-flight (TOF) analyzer. Precoated silica gel plates with a fluorescent indicator (Merck 60 F254) were used for analytical and preparative thin-layer chromatography (TLC). Flash column chromatography was performed using Kanto Chemical silica gel 60N (spherical, natural) 40–50  $\mu\text{m}$ . All reagents and solvents were of commercial quality and were used as received.

**(S)-5-Methylnonan-3-one (5a).** To a stirred solution of (S)-3-methylheptan-1-ol (**3a**) (5.58 g, 42.8 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (214 mL) were added DMSO (30.4 mL, 428 mmol), DIPEA (37.3 mL, 214 mmol), and SO<sub>3</sub>·Py (27.2 g, 171 mmol) at 0 °C. After stirring for 30 min at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NaHCO<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in THF (214 mL) was added ethylmagnesium bromide (64.0 mL, 64.6 mmol, 1.01 M in THF) at -78 °C. After stirring for 20 min at 0 °C, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NH<sub>4</sub>Cl solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 8:1) and then concentrated *in vacuo* to give a crude product.

To a stirred solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (154 mL) were added DMSO (21.9 mL, 308 mmol), DIPEA (19.9 mL, 114 mmol), and SO<sub>3</sub>·Py (19.6 g, 123 mmol) at 0 °C. After stirring for 30 min at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O,

washed with saturated aqueous NaHCO<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 25:1) to give ketone **5a** (3.81 g, 57% yield, 3 steps) as a colorless oil.; *R*<sub>f</sub> 0.50 (hexane/AcOEt 6:1); [α]<sup>25</sup><sub>D</sub> -5.0 (*c* 1.75, CHCl<sub>3</sub>); IR (neat)  $\nu_{\max}$  = 2958, 2928, 2873, 2859, 1715, 1460, 1413, 1377, 1106 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz) δ 2.41 (1H, q, *J* = 7.4 Hz), 2.40 (1H, q, *J* = 7.4 Hz), 2.38 (1H, dd, *J* = 5.7, 15.3 Hz), 2.20 (1H, dd, *J* = 8.0, 15.6 Hz), 1.99 (1H, m), 1.31–1.14 (6H, m), 1.04 (3H, t, *J* = 7.3 Hz), 0.88 (3H, t, *J* = 6.5 Hz), 0.88 (3H, dd, *J* = 6.6 Hz); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz) δ 211.8 (C), 50.0 (CH<sub>2</sub>), 36.6 (CH<sub>2</sub>), 36.5 (CH<sub>2</sub>), 29.3 (CH), 29.2 (CH<sub>2</sub>), 22.8 (CH<sub>2</sub>), 19.9 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 7.8 (CH<sub>3</sub>); HRMS (ESI-TOF) *m/z*: [M+Na]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>20</sub>ONa 179.1412, Found 179.1426.

**(*S,E*)-3-Ethyl-5-methylnon-2-en-1-ol and (*S,Z*)-3-ethyl-5-methylnon-2-en-1-ol (6a).**

To a suspension of NaH (7.45 g, 171 mmol, 55% in oil) in THF (230 mL) was slowly added ethyl 2-(diethoxyphosphoryl)acetate (43.7 g, 195 mmol) at 0 °C, and the mixture

was stirred for 30 min. A solution of ketone **5a** (3.81 g, 24.4 mmol) in THF (14.0 mL) at the same temperature was slowly added, and the mixture was then refluxed and stirred for 17 hr. The mixture was quenched with saturated aqueous NH<sub>4</sub>Cl and diluted with Et<sub>2</sub>O. The organic layer was washed with H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 30:1) and then concentrated *in vacuo* to give a crude product (*E/Z* = 2:1).

To a solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (220 mL) was slowly added DIBAH (47.2 mL, 48.2 mmol, 1.02 M in hexane) at -78 °C under Ar, and the mixture was stirred at the same temperature for 30 min. The mixture was diluted with Et<sub>2</sub>O, and Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O was added; the mixture stirred at rt for 12 hr. MgSO<sub>4</sub> was added to the suspension, and the mixture stirred for 15 min. The suspension was filtered through anhydrous Na<sub>2</sub>SO<sub>4</sub> and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 5:1) to give allylic alcohol **6a** (3.69 g, 82% yield, 2 steps, *E/Z* = 2:1) as a colorless oil.; *R*<sub>f</sub> 0.10 (hexane/AcOEt 10:1); IR (neat)

$\nu_{\max}$  = 3325, 2962, 2925, 2872, 1661, 1460, 1377, 1008  $\text{cm}^{-1}$ ;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  5.46 (0.33H, t,  $J$  = 7.0 Hz), 5.34 (0.67H, t,  $J$  = 7.0 Hz), 4.17–4.16 (2H, m), 2.12–1.99 (3H, m), 1.91 (0.33H, dd,  $J$  = 8.8, 13.4 Hz), 1.75 (0.67H, dd,  $J$  = 8.3, 13.7 Hz), 1.56 (1H, m), 1.33–1.20 (6H, m), 1.02 (1H, t,  $J$  = 7.5 Hz), 0.98 (2H, t,  $J$  = 7.6 Hz), 0.91–0.87 (3H, m), 0.82 (3H, t,  $J$  = 6.5 Hz);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  144.6 (C), 144.4 (C), 124.3 (CH), 123.3 (CH), 59.3 ( $\text{CH}_2$ ), 59.1 ( $\text{CH}_2$ ), 44.5 ( $\text{CH}_2$ ), 37.9 ( $\text{CH}_2$ ), 36.8 ( $\text{CH}_2$ ), 36.7 ( $\text{CH}_2$ ), 31.5 (CH), 30.7 (CH), 29.4 ( $\text{CH}_2$ ), 29.3 ( $\text{CH}_2$ ), 23.1 ( $\text{CH}_2$ ), 22.93 ( $\text{CH}_2$ ), 22.91 ( $\text{CH}_2$ ), 19.53 ( $\text{CH}_3$ ), 19.47 ( $\text{CH}_3$ ), 14.12 ( $\text{CH}_3$ ), 14.09 ( $\text{CH}_3$ ), 13.6 ( $\text{CH}_3$ ), 12.5 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{12}\text{H}_{24}\text{ONa}$  207.1725, Found 207.1721.

**3-Ethyl-3-((*S*)-2-methylhexyl)oxirane-2-carbaldehyde (7a).** To a cold ( $-20\text{ }^\circ\text{C}$ ) suspension of 4Å molecular sieves (3.42 g) in  $\text{CH}_2\text{Cl}_2$  (600 mL) were added D-(–)-DIPT (652 mg, 2.79 mmol),  $\text{Ti}(\text{O}i\text{Pr})_4$  (0.710 mL, 2.41 mmol), and TBHP (9.80 mL, 55.7 mmol, 5.69 M in  $\text{CH}_2\text{Cl}_2$ ). After stirring for 30 min at the same temperature, a solution of allylic alcohol **6a** (3.42 g, 18.6 mmol) in  $\text{CH}_2\text{Cl}_2$  (325 mL) was added over 6 hr. After stirring at



-20 °C for 1 hr, NaOH (1.63 mL, 30% in brine) was added. The mixture was diluted with Et<sub>2</sub>O, warmed to room temperature, and stirred for 30 min. MgSO<sub>4</sub> (1.45 g) was then added, and after stirring for 15 min the mixture was passed through a pad of celite and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 3:1) and then concentrated *in vacuo* to give a crude product.

To a stirred solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (176 mL) were added DMSO (12.5 mL, 176 mmol), DIPEA (15.3 mL, 88.1 mmol), and SO<sub>3</sub>·Py (11.2 g, 70.5 mmol) at 0 °C.

After stirring for 30 min at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NaHCO<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 12:1) to give epoxyaldehyde **7a** (3.03 g,

82% yield, 2 steps, dr 1.0:11.0:4.5:1.4) as a colorless oil.; *R*<sub>f</sub> 0.60 (hexane/AcOEt 3:1);

IR (neat)  $\nu_{\max}$  = 2959, 2930, 2873, 2859, 1723, 1462, 1051 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)

$\delta$  9.52–9.49 (1H, m), 3.17 (0.05H, d, *J* = 5.0 Hz), 3.15 (0.57H, d, *J* = 4.9 Hz), 3.12 (0.29H,

d, *J* = 5.0 Hz), 3.10 (0.09H, d, *J* = 4.9 Hz), 2.17–2.16 (0.14H, m), 1.96–1.43 (4.86H, m),

1.37–1.15 (6H, m), 1.06–0.82 (9H, m);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  199.9 (C), 199.7 (C), 67.51 (C), 67.48 (C), 64.6 (CH), 63.6 (CH), 63.3 (CH), 62.5 (CH), 41.5 ( $\text{CH}_2$ ), 37.6 ( $\text{CH}_2$ ), 36.95 ( $\text{CH}_2$ ), 36.92 ( $\text{CH}_2$ ), 36.80 ( $\text{CH}_2$ ), 36.76 ( $\text{CH}_2$ ), 30.05 (CH), 30.01 (CH), 29.5 (CH), 29.25 (CH), 29.23 ( $\text{CH}_2$ ), 29.13 ( $\text{CH}_2$ ), 29.07 ( $\text{CH}_2$ ), 27.9 ( $\text{CH}_2$ ), 27.8 ( $\text{CH}_2$ ), 23.8 ( $\text{CH}_2$ ), 23.7 ( $\text{CH}_2$ ), 22.84 ( $\text{CH}_2$ ), 22.82 ( $\text{CH}_2$ ), 22.76 ( $\text{CH}_2$ ), 20.0 ( $\text{CH}_3$ ), 19.9 ( $\text{CH}_3$ ), 19.8 ( $\text{CH}_3$ ), 19.6 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 14.0 ( $\text{CH}_3$ ), 9.8 ( $\text{CH}_3$ ), 9.5 ( $\text{CH}_3$ ), 8.8 ( $\text{CH}_3$ ), 8.4 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}-\text{H}]^+$  Calcd for  $\text{C}_{12}\text{H}_{21}\text{O}_2$  197.1542, Found 197.1545.

**1-((2S,3R)-3-Ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)propan-1-one and 1-((2R,3S)-3-ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)propan-1-one (8a) and 1-((2S,3S)-3-ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)propan-1-one and 1-((2R,3R)-3-ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)propan-1-one (9a).** To a solution of epoxyaldehyde **7a** (2.96 g, 14.9 mmol) in THF (149 mL) was added ethylmagnesium bromide (37.7 mL, 37.3 mmol, 1.01 M in THF) at  $-78\text{ }^\circ\text{C}$ . After stirring for 1 hr at  $0\text{ }^\circ\text{C}$ , the reaction mixture was diluted with  $\text{Et}_2\text{O}$ , washed with saturated aqueous  $\text{NH}_4\text{Cl}$  solution,  $\text{H}_2\text{O}$  and brine, dried

over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 7:1) and then concentrated *in vacuo* to give a crude product.

To a stirred solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (135 mL) were added DMSO (9.60 mL, 135 mmol), DIPEA (11.8 mL, 67.7 mmol), and SO<sub>3</sub>·Py (8.61 g, 54.1 mmol) at 0 °C.

After stirring for 1 hr at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NaHCO<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous

Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column

chromatography on silica gel (hexane/AcOEt = 15:1) to give epoxyketone **8a** (1.61 g, 48% yield, 2 steps, α-epoxide/β-epoxide = 6:1) as a colorless oil and epoxyketone **9a** (814 mg, 24% yield, 2 steps, α-epoxide/β-epoxide = 1:4) as a colorless oil.; Epoxyketone **8a**:

*R<sub>f</sub>* 0.55 (hexane/AcOEt 4:1); IR (neat)  $\nu_{\text{max}}$  = 2959, 2929, 2873, 2859, 1725, 1460, 1416,

1379, 1111, 979 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)  $\delta$  3.38 (0.86H, s), 3.34 (0.14H, s), 2.54 (2H,

ddq, *J* = 14.6, 17.8, 7.3 Hz), 1.94 (0.86H, dd, *J* = 5.4, 14.1 Hz), 1.71 (0.14H, dd, *J* = 6.1,

14.0 Hz), 1.62–1.50 (2H, m), 1.45–1.13 (8H, m), 1.10 (0.42H, t, *J* = 7.3 Hz), 1.10 (2.58H,

t,  $J = 7.3$  Hz), 0.99–0.88 (9H, m);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  206.73 (C), 206.68 (C), 66.5 (C), 66.2 (C), 65.5 (CH), 64.4 (CH), 41.4 ( $\text{CH}_2$ ), 41.3 ( $\text{CH}_2$ ), 37.0 ( $\text{CH}_2$ ), 36.7 ( $\text{CH}_2$ ), 34.2 ( $\text{CH}_2$ ), 34.1 ( $\text{CH}_2$ ), 29.6 (CH), 29.5 (CH), 29.0 ( $\text{CH}_2$ ), 22.80 ( $\text{CH}_2$ ), 22.77 ( $\text{CH}_2$ ), 22.5 ( $\text{CH}_2$ ), 22.3 ( $\text{CH}_2$ ), 19.9 ( $\text{CH}_3$ ), 19.8 ( $\text{CH}_3$ ), 14.0 ( $\text{CH}_3$ ), 9.5 ( $\text{CH}_3$ ), 9.1 ( $\text{CH}_3$ ), 7.14 ( $\text{CH}_3$ ), 7.06 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{14}\text{H}_{26}\text{O}_2\text{Na}$  249.1830, Found 249.1825. Epoxyketone **9a**:  $R_f$  0.50 (hexane/AcOEt 4:1); IR (neat)  $\nu_{\text{max}} = 2931, 2873, 1724, 1460, 1413, 1379, 1111, 924$   $\text{cm}^{-1}$ ;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  3.38 (0.2H, s), 3.34 (0.8H, s), 2.54 (2H, ddq,  $J = 14.8, 17.9, 7.4$  Hz), 1.83 (0.8H, dq,  $J = 14.9, 7.5$  Hz), 1.76–1.49 (4.2H, m), 1.33–1.13 (6H, m), 1.09 (3H, t,  $J = 7.3$  Hz), 0.98–0.77 (9H, m);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  206.9 (C), 206.8 (C), 66.4 (C), 66.3 (C), 63.7 (CH), 62.9 (CH), 36.9 ( $\text{CH}_2$ ), 36.6 ( $\text{CH}_2$ ), 36.0 ( $\text{CH}_2$ ), 35.6 ( $\text{CH}_2$ ), 34.4 ( $\text{CH}_2$ ), 34.2 ( $\text{CH}_2$ ), 29.6 (CH), 29.2 ( $\text{CH}_2$ ), 29.0 ( $\text{CH}_2$ ), 27.8 ( $\text{CH}_2$ ), 27.7 ( $\text{CH}_2$ ), 22.7 ( $\text{CH}_3$ ), 19.7 ( $\text{CH}_3$ ), 19.5 ( $\text{CH}_3$ ), 13.93 ( $\text{CH}_3$ ), 13.90 ( $\text{CH}_3$ ), 9.0 ( $\text{CH}_3$ ), 8.6 ( $\text{CH}_3$ ), 7.0 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{14}\text{H}_{26}\text{O}_2\text{Na}$  249.1830, Found 249.1827.

**(Z)-3-((2R,3R)-3-Ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol** and **(Z)-3-((2S,3S)-3-ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol (10a)** and **(E)-3-((2R,3R)-3-ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol** and **(E)-3-((2S,3S)-3-ethyl-3-((S)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol (11a)**. To a suspension of NaH (925 mg, 21.2 mmol, 55% in oil) in THF (90.0 mL) was slowly added ethyl 2-(diethoxyphosphoryl)acetate (7.13 g, 31.8 mmol) at 0 °C, and the mixture was stirred for 30 min. A solution of epoxyketone **8a** (1.20 g, 5.30 mmol, dr 6:1) in THF (16.0 mL) at the same temperature was slowly added, and the mixture was warmed to room temperature and stirred for 4 hr. The mixture was quenched with saturated aqueous NH<sub>4</sub>Cl and diluted with Et<sub>2</sub>O. The organic layer was washed with H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 30:1) and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in THF (106 mL) was slowly added LiAlH<sub>4</sub> (476 mg, 12.5 mmol) at 0 °C under Ar, and the mixture was stirred at the same temperature for 30

min. The mixture was diluted with Et<sub>2</sub>O, and Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O was added; the mixture stirred at rt for 15 min. MgSO<sub>4</sub> was added to the suspension, and the mixture stirred for 15 min. The suspension was filtered through anhydrous Na<sub>2</sub>SO<sub>4</sub> and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 4:1) to give allylic alcohol **10a** (1.10 g, 82% yield, 2 steps, dr 6:1) as a colorless oil and allylic alcohol **11a** (85.0 mg, 6% yield, 2 steps, dr 6:1) as a colorless oil.;

Allylic alcohol **10a**: *R*<sub>f</sub> 0.30 (hexane/AcOEt 4:1); IR (neat)  $\nu_{\text{max}}$  = 3390, 2964, 2928, 2873, 2858, 1660, 1461, 1378, 1059, 932 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)  $\delta$  5.65–5.60 (1H, m), 4.30 (1H, ddd, *J* = 5.6, 6.3, 12.3 Hz), 4.12 (1H, ddd, *J* = 6.4, 6.7, 13.0 Hz), 3.36 (0.86H, s), 3.32 (0.14H, s), 2.12 (1H, dd, *J* = 5.8, 6.8 Hz), 2.04 (2H, q, *J* = 7.3 Hz), 1.98 (1H, dd, *J* = 4.8, 14.0 Hz), 1.63–1.42 (4H, m), 1.40–1.10 (6H, m), 1.07 (3H, t, *J* = 7.4 Hz), 0.99–0.88 (9H, m); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz)  $\delta$  139.33 (C), 139.26 (C), 126.2 (CH), 126.1 (CH), 65.0 (CH), 64.1 (CH), 63.9 (C), 63.8 (C), 58.93 (CH<sub>2</sub>), 58.87 (CH<sub>2</sub>), 41.4 (CH<sub>2</sub>), 41.3 (CH<sub>2</sub>), 37.5 (CH<sub>2</sub>), 36.9 (CH<sub>2</sub>), 29.7 (CH), 29.6 (CH), 29.23 (CH<sub>2</sub>), 29.20 (CH<sub>2</sub>), 27.3 (CH<sub>2</sub>), 27.1 (CH<sub>2</sub>), 22.93 (CH<sub>2</sub>), 22.89 (CH<sub>2</sub>), 22.87 (CH<sub>2</sub>), 22.5 (CH<sub>2</sub>), 20.4 (CH<sub>3</sub>), 20.1

(CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 12.5 (CH<sub>3</sub>), 12.4 (CH<sub>3</sub>), 9.2 (CH<sub>3</sub>), 8.9 (CH<sub>3</sub>); HRMS (ESI-TOF) *m/z*:

[M+Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>30</sub>O<sub>2</sub>Na 277.2143, Found 277.2141. Allylic alcohol **11a**: *R<sub>f</sub>* 0.20

(hexane/AcOEt 4:1); IR (neat)  $\nu_{\text{max}}$  = 3369, 2964, 2928, 2873, 1666, 1462, 1378, 1012,

932 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)  $\delta$  5.54 (1H, t, *J* = 7.0 Hz), 4.26–4.21 (2H, m), 3.29

(0.86H, s), 3.18 (0.14H, s), 2.21–2.11 (2H, m), 2.01 (1H, dd, *J* = 4.8, 14.0 Hz), 1.56–1.05

(10H, m), 1.05–0.99 (3H, m), 0.97–0.88 (9H, m); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz)  $\delta$  138.7 (C),

138.5 (C), 124.8 (CH), 124.7 (CH), 66.3 (C), 66.1 (C), 65.6 (CH), 64.8 (CH), 58.5 (CH<sub>2</sub>),

42.0 (CH<sub>2</sub>), 41.8 (CH<sub>2</sub>), 37.5 (CH<sub>2</sub>), 36.7 (CH<sub>2</sub>), 29.8 (CH), 29.7 (CH), 29.21 (CH<sub>2</sub>), 29.18

(CH<sub>2</sub>), 22.94 (CH<sub>2</sub>), 22.88 (CH<sub>2</sub>), 22.4 (CH<sub>2</sub>), 22.3 (CH<sub>2</sub>), 21.2 (CH<sub>2</sub>), 20.9 (CH<sub>2</sub>), 20.4

(CH<sub>3</sub>), 19.9 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 13.7 (CH<sub>3</sub>), 9.4 (CH<sub>3</sub>), 9.1 (CH<sub>3</sub>); HRMS (ESI-TOF) *m/z*:

[M+Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>30</sub>O<sub>2</sub>Na 277.2143, Found 277.2142.

**((2*S*,2'*S*,3*S*,3'*R*)-2,3'-Diethyl-3'-((*S*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)methanol**

**and ((2*R*,2'*R*,3*R*,3'*S*)-2,3'-diethyl-3'-((*S*)-2-methylhexyl)-[2,2'-bioxiran]-3-**

**yl)methanol (12a) and ((2*R*,2'*S*,3*R*,3'*R*)-2,3'-diethyl-3'-((*S*)-2-methylhexyl)-[2,2'-**

**bioxiran]-3-yl)methanol and ((2*S*,2'*R*,3*S*,3'*S*)-2,3'-diethyl-3'-((*S*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)methanol (13a).** To a cold (−20 °C) suspension of 4Å molecular sieves (721 mg) in CH<sub>2</sub>Cl<sub>2</sub> (15.4 mL) were added L-(+)-DIPT (732 mg, 3.12 mmol), Ti(O*i*Pr)<sub>4</sub> (0.859 mL, 2.84 mmol), and TBHP (1.58 mL, 8.51 mmol, 5.37 M in CH<sub>2</sub>Cl<sub>2</sub>). After stirring for 30 min at the same temperature and then cooled to −40 °C. To a stirred solution was added a solution of allylic alcohol **10a** (721 mg, 2.84 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10.0 mL) was added over 2 hr. After stirring at −13 °C for 16 hr, NaOH (30% solution in brine, 1.90 mL) was added. The mixture was diluted with Et<sub>2</sub>O, warmed to room temperature, and stirred for 30 min. MgSO<sub>4</sub> (1.90 g) was then added, and after stirring for 15 min the mixture was passed through a pad of celite and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 5:2) to give *syn*-diepoxide **12a** (551 mg, 72% yield, dr >20:1) as a colorless oil and *anti*-diepoxide **13a** (205 mg, 27% yield, dr 3:2) as a colorless oil.; *syn*-Diepoxide **12a**: *R*<sub>f</sub> 0.40 (hexane/AcOEt 2:1); [α]<sup>25</sup><sub>D</sub> +10.4 (*c* 0.59, CHCl<sub>3</sub>); IR (neat) ν<sub>max</sub> = 3433, 2961, 2928, 2873, 1462, 1379, 1029, 936 cm<sup>−1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz) δ 3.91 (1H, m), 3.89 (1H, dd,



$J = 1.2, 4.5$  Hz), 3.05 (1H, dd,  $J = 4.8, 5.9$  Hz), 3.00 (1H, s), 2.01 (1H, dd,  $J = 4.5, 14.1$  Hz), 1.93 (1H, dd,  $J = 5.8, 6.7$  Hz), 1.85 (1H, dq,  $J = 15.0, 7.5$  Hz), 1.76 (1H, dq,  $J = 15.1, 7.4$  Hz), 1.65 (1H, dq,  $J = 15.0, 7.5$  Hz), 1.62 (1H, dq,  $J = 15.3, 7.6$  Hz), 1.54 (1H, m), 1.34–1.15 (7H, m), 1.03 (3H, t,  $J = 7.5$  Hz), 0.98 (3H, t,  $J = 7.6$  Hz), 0.93 (3H, d,  $J = 6.6$  Hz), 0.89 (3H, t,  $J = 6.5$  Hz);  $^{13}\text{C}$  (CDCl<sub>3</sub>, 100 MHz)  $\delta$  63.7 (C), 61.7 (CH), 60.8 (C), 60.6 (CH<sub>2</sub>), 59.7 (CH), 41.5 (CH<sub>2</sub>), 37.6 (CH<sub>2</sub>), 29.6 (CH), 29.2 (CH<sub>2</sub>), 26.5 (CH<sub>2</sub>), 22.9 (CH<sub>2</sub>), 22.2 (CH<sub>2</sub>), 20.0 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.1 (CH<sub>3</sub>), 8.7 (CH<sub>3</sub>); HRMS (ESI-TOF)  $m/z$ : [M+Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>30</sub>O<sub>3</sub>Na 293.2093, Found 293.2092. *anti*-Diepoxide **13a**:  $R_f$  0.50 (hexane/AcOEt 2:1); IR (neat)  $\nu_{\text{max}} = 3430, 2962, 2929, 2874, 2858, 1463, 1379, 1028, 933, 908$  cm<sup>-1</sup>;  $^1\text{H}$  (CDCl<sub>3</sub>, 400 MHz)  $\delta$  3.97–3.89 (1H, m), 3.80–3.74 (1H, m), 3.14 (0.58H, s), 3.01 (0.42H, s), 3.08–3.04 (1H, m), 2.12–2.05 (1H, m), 1.96 (0.58H, dd,  $J = 4.8, 14.1$  Hz), 1.83–1.64 (3H, m), 1.62–1.38 (2.42H, m), 1.35–1.08 (6H, m), 1.05–0.88 (12H, m);  $^{13}\text{C}$  (CDCl<sub>3</sub>, 100 MHz)  $\delta$  64.7 (C), 64.4 (C), 63.4 (CH), 63.2 (C), 63.0 (C), 62.6 (CH), 62.3 (CH), 62.2 (CH), 62.05 (CH<sub>2</sub>), 61.99 (CH<sub>2</sub>), 41.4 (CH<sub>2</sub>), 41.2 (CH<sub>2</sub>), 37.4 (CH<sub>2</sub>), 36.9 (CH<sub>2</sub>), 29.6 (CH), 29.5 (CH), 29.2 (CH<sub>2</sub>), 27.1 (CH<sub>2</sub>), 26.8 (CH<sub>2</sub>), 24.3 (CH<sub>2</sub>),

23.9 (CH<sub>2</sub>), 22.92 (CH<sub>2</sub>), 22.86 (CH<sub>2</sub>), 20.4 (CH<sub>3</sub>), 20.2 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.4 (CH<sub>3</sub>), 9.1 (CH<sub>3</sub>), 8.4 (CH<sub>3</sub>), 8.2 (CH<sub>3</sub>); HRMS (ESI-TOF) *m/z*: [M+Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>30</sub>O<sub>3</sub>Na 293.2093, Found 293.2093.

**2-((2*S*,2'*S*,3*S*,3'*R*)-2,3'-Diethyl-3'-((*S*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)ethan-1-**

**ol (14a).** To a stirred solution of *syn*-diepoxide **12a** (200 mg, 0.740 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (14.8 mL) were added NaHCO<sub>3</sub> (249 mg, 2.96 mmol) and Dess–Martin periodinane (628 mg, 1.48 mmol) at room temperature. After stirring for 45 min, NaHCO<sub>3</sub> (125 mg, 1.48 mmol) and Dess–Martin periodinane (314 mg, 0.740 mmol) were added. After stirring for 30 min, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 9:1) and then concentrated *in vacuo* to give a crude product.

To a stirred suspension of methyltriphenylphosphonium bromide (424 mg, 1.19 mmol) in THF (7.20 mL) were added BuLi (0.677 mL, 1.06 mmol, 1.56 M in hexane) dropwise

at 0 °C and the resulting mixture was stirred for 30 min at same temperature. The mixture was cooled to -78 °C. A solution of the crude product in THF (6.00 mL) was then added to the mixture. After stirring for 30 min at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NH<sub>4</sub>Cl solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 20:1) and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in THF (6.00 mL) was added 9-BBN (1.80 mL, 0.900 mmol, 0.50 M in THF) dropwise at 0 °C. After stirring for 2 hr at room temperature, NaBO<sub>3</sub>·4H<sub>2</sub>O (138 mg, 0.897 mmol) and H<sub>2</sub>O were added to the reaction mixture. After stirring for 24 hr, NaBO<sub>3</sub>·4H<sub>2</sub>O (46.0 mg, 0.299 mmol) was added and then stirred for 24hr, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NH<sub>4</sub>Cl solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 2:1) to give alcohol **14a** (50.0 mg, 24% yield, 3 steps) as a colorless oil.; *R*<sub>f</sub> 0.50

(hexane/AcOEt 1:2);  $[\alpha]_D^{25} +4.6$  ( $c$  0.86,  $\text{CHCl}_3$ ); IR (neat)  $\nu_{\text{max}} = 3433, 2961, 2928, 2875,$   
1461, 1379, 1057, 939, 904  $\text{cm}^{-1}$ ;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  3.93–3.78 (2H, m), 3.02 (1H,  
s), 2.97 (1H, dd,  $J = 5.4, 7.1$  Hz), 2.04–1.91 (2H, m), 2.01 (1H, dd,  $J = 4.5, 14.1$  Hz), 1.83  
(1H, dq,  $J = 14.9, 7.5$  Hz), 1.75 (1H, dq,  $J = 15.0, 7.4$  Hz), 1.68–1.43 (4H, m), 1.34–1.13  
(6H, m), 1.03 (3H, t,  $J = 7.5$  Hz), 0.98 (3H, t,  $J = 7.6$  Hz), 0.94 (3H, d,  $J = 6.5$  Hz), 0.93–  
0.87 (3H, m);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  62.7 (C), 62.0 (CH), 60.6 ( $\text{CH}_2$ ), 60.4 (C), 58.0  
(CH), 41.5 ( $\text{CH}_2$ ), 37.6 ( $\text{CH}_2$ ), 30.9 ( $\text{CH}_2$ ), 29.6 (CH), 29.2 ( $\text{CH}_2$ ), 26.7 ( $\text{CH}_2$ ), 22.9 ( $\text{CH}_2$ ),  
22.3 ( $\text{CH}_2$ ), 20.0 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 9.1 ( $\text{CH}_3$ ), 8.7 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  
[ $\text{M}+\text{Na}$ ] $^+$  Calcd for  $\text{C}_{17}\text{H}_{32}\text{O}_3\text{Na}$  307.2249, Found 307.2248.

**Methyl 2-((2*S*,2'*S*,3*S*,3'*R*)-2,3'-diethyl-3'-((*S*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)acetate (2a), plakdiepoxide.** To a stirred solution of alcohol **14a** (45.2 mg, 0.159 mmol) in pH 6.8 phosphate buffer (0.530 mL, 1.0 M in  $\text{H}_2\text{O}$ ) were added a solution of 1-Me-AZADO (2.7 mg, 0.0159 mmol) in  $\text{CH}_3\text{CN}$  (0.795 mL),  $\text{NaClO}_2$  (0.024 mL, 0.0159 mmol, 5% in  $\text{H}_2\text{O}$ ), and  $\text{NaClO}$  (54.6 mg, 0.477 mmol) at room temperature. After stirring

for 30 min at room temperature, the reaction mixture was quenched with pH 2.3 phosphate buffer (1.0 M in H<sub>2</sub>O), diluted with CHCl<sub>3</sub>, washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in benzene/MeOH (2:1, 3.20 mL) was added TMSCHN<sub>2</sub> (0.400 mL, 0.240 mmol, 0.60 M in hexane) dropwise at room temperature.

After stirring for 30 min at same temperature, the reaction mixture was quenched with acetic acid and diluted with Et<sub>2</sub>O. After stirring for 15 min, the resultant mixture was concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 10:1) to give diepoxyester **2a** (40.4 mg, 81% yield, 2 steps) as a colorless oil.; *R*<sub>f</sub> 0.60 (hexane/AcOEt 5:1); [α]<sup>25</sup><sub>D</sub> +32.3 (*c* 0.80, CHCl<sub>3</sub>); IR (neat)  $\nu_{\text{max}}$  = 2960, 2928, 2874, 2858, 1742, 1461, 1436, 1198, 1174 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 3.72 (3H, s), 3.14 (1H, dd, *J* = 6.1, 6.5 Hz), 3.03 (1H, s), 2.89 (1H, dd, *J* = 6.0, 17.3 Hz), 2.79 (1H, dd, *J* = 6.6, 17.3 Hz), 2.03 (1H, dd, *J* = 4.4, 14.0 Hz), 1.89 (1H, dq, *J* = 14.4, 7.4 Hz), 1.75 (1H, dq, *J* = 14.3, 7.5 Hz), 1.64 (1H, dq, *J* = 14.6, 7.4 Hz), 1.58 (1H, dq, *J* = 14.3, 7.6 Hz), 1.55 (1H, m), 1.36–1.12 (6H, m), 1.02 (3H, t, *J* = 7.5 Hz),

1.01 (3H, t,  $J = 7.6$  Hz), 0.94 (3H, d,  $J = 6.6$  Hz), 0.90 (3H, t,  $J = 6.9$  Hz), 0.88 (1H, m);

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  171.5 (C), 62.7 (C), 61.6 (CH), 60.2 (C), 55.2 (CH), 51.8 (CH<sub>3</sub>), 41.6 (CH<sub>2</sub>), 37.6 (CH<sub>2</sub>), 33.5 (CH<sub>2</sub>), 29.6 (CH), 29.2 (CH<sub>2</sub>), 26.6 (CH<sub>2</sub>), 22.9 (CH<sub>2</sub>), 22.1 (CH<sub>2</sub>), 20.0 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.1 (CH<sub>3</sub>), 8.7 (CH<sub>3</sub>); HRMS (ESI-TOF)  $m/z$ :

$[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{18}\text{H}_{32}\text{O}_4\text{Na}$  335.2198, Found 335.2194.

**(3a*S*,5*S*,6*S*,6a*S*)-5,6a-Diethyl-6-hydroxy-5-((*S*)-2-methylhexyl)tetrahydrofuro[3,2-**

**b]furan-2(3*H*)-one (1a), plakortone Q.** A solution of 10-camphorsulfonic acid (1.48 mL,

0.0637 mmol, 1% in  $\text{CH}_2\text{Cl}_2$ ) and  $\text{H}_2\text{O}$  (0.0013 mL, 0.0721 mmol) were added to

diepoxyester **2a** (23.1 mg, 0.0739 mmol) at room temperature. After stirring for 10 hr at

same temperature, the reaction mixture was concentrated *in vacuo*. The residue was

purified with flash column chromatography on silica gel (hexane/acetone = 7:1) to give

tetrahydrofuran- $\gamma$ -lactone **1a** (18.1 mg, 82% yield) as a colorless oil.;  $R_f$  0.40

(hexane/acetone 3:1);  $[\alpha]_D^{25} -19.8$  ( $c$  0.81,  $\text{CHCl}_3$ ); IR (neat)  $\nu_{\text{max}} = 3468, 2928, 1783,$

$1462, 1219, 1127$   $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  4.33 (1H, dd,  $J = 0.6, 5.5$  Hz),

3.87 (1H, d,  $J = 10.3$  Hz), 2.77 (1H, dd,  $J = 5.5, 18.5$  Hz), 2.67 (1H, dd,  $J = 0.6, 18.5$  Hz),  
2.25 (1H, d,  $J = 10.3$  Hz), 1.92 (1H, dq,  $J = 14.5, 7.5$  Hz), 1.79 (1H, dq,  $J = 14.5, 7.4$  Hz),  
1.62 (1H, dq,  $J = 14.2, 7.4$  Hz), 1.57–1.45 (3H, m), 1.36 (1H, dd,  $J = 7.0, 14.5$  Hz), 1.36–  
1.20 (5H, m), 1.16 (1H, m), 1.04 (3H, t,  $J = 7.4$  Hz), 0.95 (3H, d,  $J = 6.7$  Hz), 0.91 (3H,  
t,  $J = 7.4$  Hz), 0.89 (3H, t,  $J = 7.0$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  174.7 (C), 94.9  
(C), 88.3 (C), 81.6 (CH), 77.3 (CH), 42.4 ( $\text{CH}_2$ ), 38.3 ( $\text{CH}_2$ ), 38.0 ( $\text{CH}_2$ ), 29.2 ( $\text{CH}_2$ ), 29.1  
( $\text{CH}_2$ ), 28.5 (CH), 26.0 ( $\text{CH}_2$ ), 22.9 ( $\text{CH}_2$ ), 21.1 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 8.1 ( $\text{CH}_3$ ), 7.9 ( $\text{CH}_3$ );  
HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{17}\text{H}_{30}\text{O}_4\text{Na}$  321.2042, Found 321.2041.

**(R)-5-Methylnonan-3-one (5b).** To a stirred solution of (*R*)-3-methylheptan-1-ol (**3b**)  
(3.22 g, 24.7 mmol) in  $\text{CH}_2\text{Cl}_2$  (247 mL) were added DMSO (17.6 mL, 248 mmol),  
DIPEA (21.5 mL, 123 mmol), and  $\text{SO}_3\cdot\text{Py}$  (15.7 g, 98.9 mmol) at 0 °C. After stirring for  
30 min at room temperature, the reaction mixture was diluted with  $\text{Et}_2\text{O}$ , washed with  
saturated aqueous  $\text{NaHCO}_3$  solution,  $\text{H}_2\text{O}$  and brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and  
then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in THF (247 mL) was added ethylmagnesium bromide (36.7 mL, 37.1 mmol, 1.01 M in THF) at  $-78\text{ }^{\circ}\text{C}$ . After stirring for 20 min at  $0\text{ }^{\circ}\text{C}$ , the reaction mixture was diluted with  $\text{Et}_2\text{O}$ , washed with saturated aqueous  $\text{NH}_4\text{Cl}$  solution,  $\text{H}_2\text{O}$  and brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 8:1) and then concentrated *in vacuo* to give a crude product.

To a stirred solution of the crude product in  $\text{CH}_2\text{Cl}_2$  (195 mL) were added DMSO (13.8 mL, 194 mmol), DIPEA (16.9 mL, 97.0 mmol), and  $\text{SO}_3\cdot\text{Py}$  (12.4 g, 77.8 mmol) at  $0\text{ }^{\circ}\text{C}$ . After stirring for 30 min at room temperature, the reaction mixture was diluted with  $\text{Et}_2\text{O}$ , washed with saturated aqueous  $\text{NaHCO}_3$  solution,  $\text{H}_2\text{O}$  and brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 25:1) to give ketone **5b** (2.41 g, 62% yield, 3 steps) as a colorless oil.;  $[\alpha]_{\text{D}}^{25} +5.4$  ( $c$  1.84,  $\text{CHCl}_3$ ). The spectra data were consistent with the literature<sup>1</sup> and our synthesized compound **5a**.



**(*R,E*)-3-Ethyl-5-methylnon-2-en-1-ol and (*R,Z*)-3-ethyl-5-methylnon-2-en-1-ol (6b).**

To a suspension of NaH (854 mg, 19.6 mmol, 55% in oil) in THF (25.0 mL) was slowly added ethyl 2-(diethoxyphosphoryl)acetate (6.58 g, 29.3 mmol) at 0 °C, and the mixture was stirred for 30 min. A solution of ketone **5b** (504 mg, 3.23 mmol) in THF (7.60 mL) at the same temperature was slowly added, and the mixture was then refluxed and stirred for 20 hr. The mixture was quenched with saturated aqueous NH<sub>4</sub>Cl and diluted with Et<sub>2</sub>O. The organic layer was washed with H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 30:1) and then concentrated *in vacuo* to give a crude product (*E/Z* = 2:1).

To a solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (28.7 mL) was slowly added DIBAH (6.18 mL, 6.31 mmol, 1.02 M in hexane) at -78 °C under Ar, and the mixture was stirred at the same temperature for 30 min. The mixture was diluted with Et<sub>2</sub>O, and Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O was added; the mixture stirred at rt for 12 hr. MgSO<sub>4</sub> was added to the suspension, and the mixture stirred for 15 min. The suspension was filtered through anhydrous Na<sub>2</sub>SO<sub>4</sub>

and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 5:1) to give allylic alcohol **6b** (495 mg, 82% yield, 2 steps, *E/Z* = 2:1) as a colorless oil.; HRMS (ESI-TOF) *m/z*: [M+Na]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>24</sub>ONa 207.1725, Found 207.1721. The spectra data were consistent with our synthesized compound **6a**.

**3-Ethyl-3-((R)-2-methylhexyl)oxirane-2-carbaldehyde (7b)**. To a cold (−20 °C) suspension of 4Å molecular sieves (1.94 g) in CH<sub>2</sub>Cl<sub>2</sub> (300 mL) were added D-(−)-DIPT (370 mg, 1.58 mmol), Ti(O*i*Pr)<sub>4</sub> (0.404 mL, 1.37 mmol), and TBHP (5.88 mL, 31.6 mmol, 5.37 M in CH<sub>2</sub>Cl<sub>2</sub>). After stirring for 30 min at the same temperature, a solution of allylic alcohol **6b** (1.94 g, 10.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (200 mL) was added over 2 hr. After stirring at −20 °C for 4 hr, NaOH (0.920 mL, 30% in brine) was added. The mixture was diluted with Et<sub>2</sub>O, warmed to room temperature, and stirred for 30 min. MgSO<sub>4</sub> (0.920 g) was then added, and after stirring for 15 min the mixture was passed through a pad of celite and then concentrated *in vacuo*. The residue was passed through a pad of silica gel

(hexane/AcOEt = 3:1) and then concentrated *in vacuo* to give a crude product.

To a stirred solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (105 mL) were added DMSO (7.50 mL, 106 mmol), DIPEA (9.20 mL, 52.8 mmol), and SO<sub>3</sub>·Py (6.70 g, 42.1 mmol) at 0 °C.

After stirring for 30 min at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O,

washed with saturated aqueous NaHCO<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous

Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column

chromatography on silica gel (hexane/AcOEt = 12:1) to give epoxyaldehyde **7b** (1.95 g,

93% yield, 2 steps, dr 6.3:2.1:1.0:10.7) as a colorless oil.; *R*<sub>f</sub> 0.60 (hexane/AcOEt 3:1);

IR (neat)  $\nu_{\max}$  = 2958, 2929, 2873, 2858, 1723, 1461 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.52–

9.49 (1H, m), 3.17 (0.30H, d, *J* = 4.9 Hz), 3.15 (0.10H, d, *J* = 4.9 Hz), 3.12 (0.05H, d, *J*

= 5.1 Hz), 3.10 (0.55H, d, *J* = 4.9 Hz), 2.17–2.16 (0.09H, m), 1.96–1.84 (0.91H, m), 1.82–

1.53 (3H, m), 1.38–1.11 (7H, m), 1.06–0.82 (9H, m); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz)  $\delta$  199.9 (C),

199.7 (C), 67.8 (C), 67.49 (C), 67.46 (C), 67.2 (C), 64.6 (CH), 63.6 (CH), 63.3 (CH), 62.5

(CH), 41.5 (CH<sub>2</sub>), 37.6 (CH<sub>2</sub>), 37.2 (CH<sub>2</sub>), 36.9 (CH<sub>2</sub>), 36.8 (CH<sub>2</sub>), 36.7 (CH<sub>2</sub>), 30.04

(CH), 30.00 (CH), 29.5 (CH), 29.2 (CH), 29.13 (CH<sub>2</sub>), 29.07 (CH<sub>2</sub>), 27.9 (CH<sub>2</sub>), 27.8

(CH<sub>2</sub>), 23.8 (CH<sub>2</sub>), 23.7 (CH<sub>2</sub>), 22.84 (CH<sub>2</sub>), 22.81 (CH<sub>2</sub>), 20.0 (CH<sub>3</sub>), 19.9 (CH<sub>3</sub>), 19.8 (CH<sub>3</sub>), 19.6 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 14.0 (CH<sub>3</sub>), 9.8 (CH<sub>3</sub>), 9.5 (CH<sub>3</sub>), 8.8 (CH<sub>3</sub>), 8.4 (CH<sub>3</sub>);

HRMS (ESI-TOF) *m/z*: [M-H]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>21</sub>O<sub>2</sub> 197.1542, Found 197.1560.

**1-((2*S*,3*R*)-3-Ethyl-3-((*R*)-2-methylhexyl)oxiran-2-yl)propan-1-one and 1-((2*R*,3*S*)-3-ethyl-3-((*R*)-2-methylhexyl)oxiran-2-yl)propan-1-one (8b) and 1-((2*S*,3*S*)-3-ethyl-3-((*R*)-2-methylhexyl)oxiran-2-yl)propan-1-one and 1-((2*R*,3*R*)-3-ethyl-3-((*R*)-2-methylhexyl)oxiran-2-yl)propan-1-one (9b).** To a solution of epoxyaldehyde **7b** (1.90 g, 9.58 mmol) in THF (95.8 mL) was added ethylmagnesium bromide (23.7 mL, 24.0 mmol, 1.01 M in THF) at -78 °C. After stirring for 1 hr at 0 °C, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NH<sub>4</sub>Cl solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 7:1) and then concentrated *in vacuo* to give a crude product.

To a stirred solution of the crude product in CH<sub>2</sub>Cl<sub>2</sub> (96.0 mL) were added DMSO (6.80

mL, 95.8 mmol), DIPEA (8.30 mL, 47.7 mmol), and SO<sub>3</sub>·Py (6.10 g, 38.3 mmol) at 0 °C.

After stirring for 1 hr at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NaHCO<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 15:1) to give epoxyketone **8b** (1.04 g, 48% yield, 2 steps, α-epoxide/β-epoxide = 5:1) as a colorless oil and epoxyketone **9b** (535 mg, 25% yield, 2 steps, α-epoxide/β-epoxide = 1:4) as a colorless oil.; Epoxyketone **8b**: *R*<sub>f</sub> 0.55 (hexane/AcOEt 4:1); IR (neat)  $\nu_{\max}$  = 2961, 2931, 2873, 1724, 1460, 1412, 1379, 1112, 937 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)  $\delta$  3.38 (0.17H, s), 3.34 (0.83H, s), 2.65–2.44 (2H, m), 1.94 (0.17H, dd, *J* = 5.4, 14.0 Hz), 1.71 (0.83H, dd, *J* = 6.2, 14.0 Hz), 1.64–1.49 (2H, m), 1.47–1.13 (8H, m), 1.10 (2.49H, t, *J* = 7.3 Hz), 1.10 (0.51H, t, *J* = 7.4 Hz), 0.99–0.88 (9H, m); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz)  $\delta$  206.91 (C), 206.86 (C), 66.6 (C), 66.3 (C), 65.6 (CH), 64.6 (CH), 41.5 (CH<sub>2</sub>), 41.4 (CH<sub>2</sub>), 37.1 (CH<sub>2</sub>), 36.8 (CH<sub>2</sub>), 34.3 (CH<sub>2</sub>), 34.2 (CH<sub>2</sub>), 29.7 (CH), 29.6 (CH), 29.1 (CH<sub>2</sub>), 22.90 (CH<sub>2</sub>), 22.87 (CH<sub>2</sub>), 22.6 (CH<sub>2</sub>), 22.4 (CH<sub>2</sub>), 20.1 (CH<sub>3</sub>), 19.9 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.6 (CH<sub>3</sub>), 9.3 (CH<sub>3</sub>), 7.3 (CH<sub>3</sub>), 7.2 (CH<sub>3</sub>); HRMS (ESI-

TOF)  $m/z$ :  $[M+Na]^+$  Calcd for  $C_{14}H_{26}O_2Na$  249.1830, Found 249.1839. Epoxyketone **9b**:  
 $R_f$  0.50 (hexane/AcOEt 4:1); IR (neat)  $\nu_{max}$  = 2959, 2931, 2873, 2859, 1723, 1460, 1413, 1379, 1111, 930  $cm^{-1}$ ;  $^1H$  ( $CDCl_3$ , 400 MHz)  $\delta$  3.38 (0.8H, s), 3.34 (0.2H, s), 2.63–2.47 (2H, m), 1.83 (0.2H, dq,  $J$  = 15.0, 7.6 Hz), 1.72 (0.8H, dq,  $J$  = 14.4, 7.6 Hz), 1.69–1.51 (4H, m), 1.40–1.15 (6H, m), 1.09 (3H, t,  $J$  = 7.3 Hz), 0.97 (3H, t,  $J$  = 7.5 Hz), 0.90–0.86 (3H, m), 0.78 (3H, d,  $J$  = 6.6 Hz);  $^{13}C$  ( $CDCl_3$ , 100 MHz)  $\delta$  206.9 (C), 66.5 (C), 66.4 (C), 63.8 (CH), 63.0 (CH), 36.9 (CH<sub>2</sub>), 36.7 (CH<sub>2</sub>), 36.0 (CH<sub>2</sub>), 35.6 (CH<sub>2</sub>), 34.4 (CH<sub>2</sub>), 34.3 (CH<sub>2</sub>), 29.6 (CH), 29.2 (CH<sub>2</sub>), 29.0 (CH<sub>2</sub>), 27.83 (CH<sub>2</sub>), 27.76 (CH<sub>2</sub>), 22.8 (CH<sub>2</sub>), 19.7 (CH<sub>3</sub>), 19.6 (CH<sub>3</sub>), 14.00 (CH<sub>3</sub>), 13.96 (CH<sub>3</sub>), 9.1 (CH<sub>3</sub>), 8.7 (CH<sub>3</sub>), 7.1 (CH<sub>3</sub>); HRMS (ESI-TOF)  $m/z$ :  $[M+Na]^+$  Calcd for  $C_{14}H_{26}O_2Na$  249.1830, Found 249.1838.

**(Z)-3-((2R,3R)-3-Ethyl-3-((R)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol** and **(Z)-3-((2S,3S)-3-ethyl-3-((R)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol (10b)** and **(E)-3-((2R,3R)-3-ethyl-3-((R)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol** and **(E)-3-((2S,3S)-3-ethyl-3-((R)-2-methylhexyl)oxiran-2-yl)pent-2-en-1-ol (11b)**. To a

suspension of NaH (623 mg, 14.3 mmol, 55% in oil) in THF (60.0 mL) was slowly added ethyl 2-(diethoxyphosphoryl)acetate (4.80 g, 21.4 mmol) at 0 °C, and the mixture was stirred for 30 min. A solution of epoxyketone **8b** (809 mg, 3.57 mmol, dr 6:1) in THF (11.5 mL) at the same temperature was slowly added, and the mixture was warmed to room temperature and stirred for 4 hr. The mixture was quenched with saturated aqueous NH<sub>4</sub>Cl and diluted with Et<sub>2</sub>O. The organic layer was washed with H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 30:1) and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in THF (71.5 mL) was slowly added LiAlH<sub>4</sub> (339 mg, 8.93 mmol) at 0 °C under Ar, and the mixture was stirred at the same temperature for 40 min. The mixture was diluted with Et<sub>2</sub>O, and Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O was added; the mixture stirred at rt for 15 min. MgSO<sub>4</sub> was added to the suspension, and the mixture stirred for 15 min. The suspension was filtered through anhydrous Na<sub>2</sub>SO<sub>4</sub> and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel

(hexane/AcOEt = 4:1) to give allylic alcohol **10b** (776 mg, 86% yield, 2 steps, dr 5:1) as a colorless oil and allylic alcohol **11b** (75.0 mg, 8% yield, 2 steps, dr 5:1) as a colorless oil.; Allylic alcohol **10b**:  $R_f$  0.30 (hexane/AcOEt 4:1); IR (neat)  $\nu_{\max}$  = 3365, 2964, 2928, 2873, 2858, 1661, 1462, 1379, 1041  $\text{cm}^{-1}$ ;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  5.64–5.59 (1H, m), 4.33–4.26 (1H, m), 4.16–4.10 (1H, m), 3.36 (0.17H, s), 3.32 (0.83H, s), 2.11 (0.17H, dd,  $J$  = 5.5, 7.1 Hz), 2.08–2.02 (2.83H, m), 1.98 (0.17H, dd,  $J$  = 4.8, 14.1 Hz), 1.62–1.55 (1H, m), 1.52–1.42 (3.83H, m), 1.38–1.13 (6H, m), 1.07 (3H, t,  $J$  = 7.4 Hz), 0.99–0.88 (9H, m);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  139.45 (C), 139.37 (C), 126.2 (CH), 126.0 (CH), 65.1 (CH), 64.1 (CH), 63.9 (C), 63.7 (C), 59.0 ( $\text{CH}_2$ ), 58.9 ( $\text{CH}_2$ ), 41.5 ( $\text{CH}_2$ ), 41.3 ( $\text{CH}_2$ ), 37.5 ( $\text{CH}_2$ ), 36.9 ( $\text{CH}_2$ ), 29.70 (CH), 29.66 (CH), 29.24 ( $\text{CH}_2$ ), 29.21 ( $\text{CH}_2$ ), 27.3 ( $\text{CH}_2$ ), 27.1 ( $\text{CH}_2$ ), 22.95 ( $\text{CH}_2$ ), 22.89 ( $\text{CH}_2$ ), 22.5 ( $\text{CH}_2$ ), 20.5 ( $\text{CH}_3$ ), 20.1 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 12.5 ( $\text{CH}_3$ ), 12.4 ( $\text{CH}_3$ ), 9.2 ( $\text{CH}_3$ ), 8.9 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{16}\text{H}_{30}\text{O}_2\text{Na}$  277.2143, Found 277.2144. Allylic alcohol **11b**:  $R_f$  0.20 (hexane/AcOEt 4:1); IR (neat)  $\nu_{\max}$  = 3365, 2964, 2928, 2872, 2858, 1668, 1463, 1378, 1012  $\text{cm}^{-1}$ ;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  5.56–5.52 (1H, m), 4.26–4.22 (2H, m), 3.22 (0.17H, s), 3.18 (0.83H, s), 2.24–2.09 (2H,



m), 2.02 (0.17H, dd,  $J = 4.8, 14.0$  Hz), 1.64–1.41 (4.83H, m), 1.35–1.08 (6H, m), 1.04 (3H, t,  $J = 7.6$  Hz), 0.97–0.88 (9H, m);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  138.8 (C), 138.6 (C), 124.7 (CH), 124.6 (CH), 66.2 (C), 66.1 (C), 65.6 (CH), 64.8 (CH), 58.6 ( $\text{CH}_2$ ), 42.0 ( $\text{CH}_2$ ), 41.8 ( $\text{CH}_2$ ), 37.5 ( $\text{CH}_2$ ), 36.7 ( $\text{CH}_2$ ), 29.8 (CH), 29.7 (CH), 29.23 ( $\text{CH}_2$ ), 29.20 ( $\text{CH}_2$ ), 23.0 ( $\text{CH}_2$ ), 22.9 ( $\text{CH}_2$ ), 22.4 ( $\text{CH}_2$ ), 22.3 ( $\text{CH}_2$ ), 21.2 ( $\text{CH}_2$ ), 20.9 ( $\text{CH}_2$ ), 20.4 ( $\text{CH}_3$ ), 19.9 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 13.8 ( $\text{CH}_3$ ), 9.4 ( $\text{CH}_3$ ), 9.1 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$   
Calcd for  $\text{C}_{16}\text{H}_{30}\text{O}_2\text{Na}$  277.2143, Found 277.2146.

**((2*S*,2'*S*,3*S*,3'*R*)-2,3'-Diethyl-3'-((*R*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)methanol**  
and **((2*R*,2'*R*,3*R*,3'*S*)-2,3'-diethyl-3'-((*R*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)methanol (12b)** and **((2*R*,2'*S*,3*R*,3'*R*)-2,3'-diethyl-3'-((*R*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)methanol** and **((2*S*,2'*R*,3*S*,3'*S*)-2,3'-diethyl-3'-((*R*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)methanol (13b)**. To a cold ( $-20$  °C) suspension of 4Å molecular sieves (358 mg) in  $\text{CH}_2\text{Cl}_2$  (8.10 mL) were added L-(+)-DIPT (363 mg, 1.55 mmol),  $\text{Ti}(\text{O}i\text{Pr})_4$  (0.416 mL, 1.38 mmol), and TBHP (0.790 mL, 4.24 mmol, 5.37 M in  $\text{CH}_2\text{Cl}_2$ ).

After stirring for 30 min at the same temperature and then cooled to  $-40\text{ }^{\circ}\text{C}$ . To a stirred solution was added a solution of allylic alcohol **10b** (358 mg, 1.41 mmol) in  $\text{CH}_2\text{Cl}_2$  (4.00 mL) was added over 1 hr. After stirring at  $-13\text{ }^{\circ}\text{C}$  for 16 hr, NaOH (30% solution in brine, 0.940 mL) was added. The mixture was diluted with  $\text{Et}_2\text{O}$ , warmed to room temperature, and stirred for 30 min.  $\text{MgSO}_4$  (0.940 g) was then added, and after stirring for 15 min the mixture was passed through a pad of celite and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 5:2) to give *syn*-diepoxide **12b** (255 mg, 67% yield, dr >20:1) as a colorless oil and *anti*-diepoxide **13b** (82.0 mg, 22% yield, dr 3:2) as a colorless oil.; *syn*-Diepoxide **12b**:  $R_f$  0.40 (hexane/AcOEt 2:1);  $[\alpha]_D^{25} +9.5$  ( $c$  1.38,  $\text{CHCl}_3$ ); IR (neat)  $\nu_{\text{max}} = 3433, 2961, 2928, 2873, 1462, 1379, 1028, 938, 914\text{ cm}^{-1}$ ;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  3.92–3.88 (2H, m), 3.05 (1H, t,  $J = 5.4$  Hz), 2.96 (1H, s), 1.87 (1H, t,  $J = 6.2$  Hz), 1.82 (1H, dd,  $J = 7.4, 14.9$  Hz), 1.72 (1H, q,  $J = 7.6$  Hz), 1.72 (1H, q,  $J = 7.5$  Hz), 1.63 (1H, dd,  $J = 7.6, 14.4$  Hz), 1.59–1.54 (2H, m), 1.40–1.19 (6H, m), 1.10 (1H, m), 1.03 (3H, t,  $J = 7.5$  Hz), 0.99 (3H, t,  $J = 7.6$  Hz), 0.94 (3H, d,  $J = 6.2$  Hz), 0.90 (3H, t,  $J = 7.0$  Hz);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  63.6 (C),

61.1 (CH), 60.7 (C), 60.5 (CH<sub>2</sub>), 60.0 (CH), 41.7 (CH<sub>2</sub>), 36.7 (CH<sub>2</sub>), 29.5 (CH), 29.2 (CH<sub>2</sub>), 26.7 (CH<sub>2</sub>), 22.9 (CH<sub>2</sub>), 22.5 (CH<sub>2</sub>), 20.4 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.4 (CH<sub>3</sub>), 8.8 (CH<sub>3</sub>); HRMS (ESI-TOF) *m/z*: [M+Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>30</sub>O<sub>3</sub>Na 293.2093, Found 293.2092. *anti*-Diepoxide **13b**: *R<sub>f</sub>* 0.50 (hexane/AcOEt 2:1); IR (neat)  $\nu_{\max}$  = 3434, 2962, 2929, 2873, 2858, 1462, 1379, 1028, 933, 908 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz)  $\delta$  3.98–3.89 (1H, m), 3.80–3.74 (1H, m), 3.15 (0.58H, s), 3.10 (0.42H, s), 3.09–3.05 (1H, m), 2.07 (0.58H, dd, *J* = 6.7, 6.8 Hz), 2.03 (0.42H, dd, *J* = 6.7, 6.8 Hz), 1.97 (0.58H, dd, *J* = 4.8, 14.1 Hz), 1.82–1.67 (2.42H, m), 1.60–1.40 (3H, m), 1.31–1.09 (6H, m), 1.06–0.88 (12H, m); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz)  $\delta$  64.7 (C), 64.4 (C), 63.4 (CH), 63.2 (C), 63.0 (C), 62.6 (CH), 62.3 (CH), 62.2 (CH), 62.1 (CH<sub>2</sub>), 62.0 (CH<sub>2</sub>), 41.4 (CH<sub>2</sub>), 41.2 (CH<sub>2</sub>), 37.4 (CH<sub>2</sub>), 36.9 (CH<sub>2</sub>), 29.6 (CH), 29.5 (CH), 29.2 (CH<sub>2</sub>), 27.1 (CH<sub>2</sub>), 26.8 (CH<sub>2</sub>), 24.3 (CH<sub>2</sub>), 23.9 (CH<sub>2</sub>), 22.93 (CH<sub>2</sub>), 22.87 (CH<sub>2</sub>), 20.4 (CH<sub>3</sub>), 20.2 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.4 (CH<sub>3</sub>), 9.1 (CH<sub>3</sub>), 8.4 (CH<sub>3</sub>), 8.3 (CH<sub>3</sub>); HRMS (ESI-TOF) *m/z*: [M+Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>30</sub>O<sub>3</sub>Na 293.2093, Found 293.2094.

**2-((2*S*,2'*S*,3*S*,3'*R*)-2,3'-Diethyl-3'-((*R*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)ethan-1-**

**ol (14b).** To a stirred solution of *syn*-diepoxide **12b** (107 mg, 0.396 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (8.00 mL) were added NaHCO<sub>3</sub> (200 mg, 2.38 mmol) and Dess–Martin periodinane (503 mg, 1.19 mmol) at room temperature. After stirring for 35 min, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through a pad of silica gel (hexane/AcOEt = 9:1) and then concentrated *in vacuo* to give a crude product.

To a stirred suspension of methyltriphenylphosphonium bromide (198 mg, 0.554 mmol) in THF (3.40 mL) were added BuLi (0.310 mL, 0.487 mmol, 1.57 M in hexane) dropwise at 0 °C and the resulting mixture was stirred for 30 min at same temperature. The mixture was cooled to –78 °C. A solution of the crude product in THF (4.00 mL) was then added to the mixture. After stirring for 30 min at room temperature, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NH<sub>4</sub>Cl solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was passed through

a pad of silica gel (hexane/AcOEt = 30:1) and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in THF (2.60 mL) was added 9-BBN (1.56 mL, 0.780 mmol, 0.50 M in THF) dropwise at 0 °C. After stirring for 2 hr at room temperature, NaBO<sub>3</sub>·4H<sub>2</sub>O (80.0 mg, 0.520 mmol) and H<sub>2</sub>O were added to the reaction mixture. After stirring for 24 hr, the reaction mixture was diluted with Et<sub>2</sub>O, washed with saturated aqueous NH<sub>4</sub>Cl solution, H<sub>2</sub>O and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and then concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 2:1) to give alcohol **14b** (50.0 mg, 35% yield, 3 steps) as a colorless oil.; *R*<sub>f</sub> 0.50 (hexane/AcOEt 1:2); [α]<sup>25</sup><sub>D</sub> +2.4 (*c* 0.49, CHCl<sub>3</sub>); IR (neat) ν<sub>max</sub> = 3446, 2961, 2928, 2875, 1461, 1379, 1057, 939, 904 cm<sup>-1</sup>; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz) δ 3.88 (1H, ddd, *J* = 5.4, 6.2, 10.6 Hz), 3.84 (1H, ddd, *J* = 5.0, 7.4, 10.6 Hz), 2.98 (1H, s), 2.97 (1H, dd, *J* = 5.0, 7.4 Hz), 2.00 (1H, dddd, *J* = 5.0, 5.4, 7.4, 14.4 Hz), 1.93 (1H, dddd, *J* = 5.0, 6.2, 7.4, 14.4 Hz), 1.81 (1H, dq, *J* = 14.3, 7.5 Hz), 1.72 (1H, ddd, *J* = 6.6, 7.4, 14.2 Hz), 1.70 (1H, ddd, *J* = 7.0, 7.4, 14.2 Hz), 1.64–1.54 (3H, m), 1.44–1.08 (7H, m), 1.03

(3H, t,  $J = 7.5$  Hz), 0.99 (3H, t,  $J = 7.6$  Hz), 0.94 (3H, d,  $J = 6.2$  Hz), 0.90 (3H, t,  $J = 7.0$  Hz);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  62.7 (C), 61.5 (CH), 60.7 ( $\text{CH}_2$ ), 60.4 (C), 58.3 (CH), 41.9 ( $\text{CH}_2$ ), 36.8 ( $\text{CH}_2$ ), 31.0 ( $\text{CH}_2$ ), 29.6 (CH), 29.3 ( $\text{CH}_2$ ), 27.0 ( $\text{CH}_2$ ), 23.0 ( $\text{CH}_2$ ), 22.6 ( $\text{CH}_2$ ), 20.5 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 9.5 ( $\text{CH}_3$ ), 9.0 ( $\text{CH}_3$ ). NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  3.82 (1H, dd,  $J = 4.2, 12.1$  Hz); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{17}\text{H}_{32}\text{O}_3\text{Na}$  307.2249, Found 307.2248.

**Methyl 2-((2*S*,2'*S*,3*S*,3'*R*)-2,3'-diethyl-3'-((*R*)-2-methylhexyl)-[2,2'-bioxiran]-3-yl)acetate (2b).** To a stirred solution of alcohol **14b** (21.0 mg, 0.0738 mmol) in pH 6.8 phosphate buffer (0.250 mL, 1.0 M in  $\text{H}_2\text{O}$ ) were added a solution of 1-Me-AZADO (1.2 mg, 0.00722 mmol) in  $\text{CH}_3\text{CN}$  (0.370 mL),  $\text{NaClO}_2$  (0.011 mL, 0.00738 mmol, 5% in  $\text{H}_2\text{O}$ ), and  $\text{NaClO}$  (25.0 mg, 0.218 mmol) at room temperature. After stirring for 30 min at room temperature, the reaction mixture was quenched with pH 2.3 phosphate buffer (1.0 M in  $\text{H}_2\text{O}$ ), diluted with  $\text{CHCl}_3$ , washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and then concentrated *in vacuo* to give a crude product.

To a solution of the crude product in benzene/MeOH (2:1, 1.50 mL) was added TMSCHN<sub>2</sub> (0.180 mL, 0.108 mmol, 0.60 M in hexane) dropwise at room temperature. After stirring for 30 min at same temperature, the reaction mixture was quenched with acetic acid and diluted with Et<sub>2</sub>O. After stirring for 15 min, the resultant mixture was concentrated *in vacuo*. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 10:1) to give diepoxyester **2b** (20.2 mg, 88% yield, 2 steps) as a colorless oil.; *R<sub>f</sub>* 0.60 (hexane/AcOEt 5:1); [ $\alpha$ ]<sup>25</sup><sub>D</sub> +33.6 (*c* 0.42, CHCl<sub>3</sub>); IR (neat)  $\nu_{\text{max}}$  = 2957, 2928, 2873, 1742, 1461, 1436, 1341, 1197, 1174 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  3.72 (3H, s), 3.13 (1H, dd, *J* = 6.0, 6.3 Hz), 2.99 (1H, s), 2.88 (1H, dd, *J* = 5.9, 17.2 Hz), 2.79 (1H, dd, *J* = 6.4, 17.2 Hz), 1.87 (1H, dq, *J* = 14.3, 7.5 Hz), 1.713 (1H, q, *J* = 7.5 Hz), 1.706 (1H, q, *J* = 7.5 Hz), 1.63–1.55 (3H, m), 1.44–1.18 (6H, m), 1.09 (1H, m), 1.024 (3H, t, *J* = 7.5 Hz), 1.017 (3H, t, *J* = 7.6 Hz), 0.94 (3H, d, *J* = 6.4 Hz), 0.90 (3H, t, *J* = 7.0 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  171.5 (C), 62.8 (C), 60.9 (CH), 60.2 (C), 55.5 (CH), 51.8 (CH<sub>3</sub>), 41.9 (CH<sub>2</sub>), 36.7 (CH<sub>2</sub>), 33.6 (CH<sub>2</sub>), 29.7 (CH), 29.3 (CH<sub>2</sub>), 26.9 (CH<sub>2</sub>), 23.0 (CH<sub>2</sub>), 22.4 (CH<sub>2</sub>), 20.5 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 9.4 (CH<sub>3</sub>), 8.9 (CH<sub>3</sub>); HRMS

(ESI-TOF)  $m/z$ :  $[M+Na]^+$  Calcd for  $C_{18}H_{32}O_4Na$  335.2198, Found 335.2197.

**(3a*S*,5*S*,6*S*,6a*S*)-5,6a-Diethyl-6-hydroxy-5-((*R*)-2-methylhexyl)tetrahydrofuro[3,2-**

**b]furan-2(3*H*)-one (1b).** A solution of 10-camphorsulfonic acid (0.560 mL, 0.0241

mmol, 1% in  $CH_2Cl_2$ ) and  $H_2O$  (0.00051 mL, 0.0283 mmol) were added to diepoxyester

**2b** (8.8 mg, 0.0283 mmol) at room temperature. After stirring for 9 hr at same temperature,

the reaction mixture was concentrated *in vacuo*. The residue was purified with flash

column chromatography on silica gel (hexane/acetone = 7:1) to give tetrahydrofuran- $\gamma$ -

lactone **1b** (7.4 mg, 88% yield) as a colorless oil.;  $R_f$  0.40 (hexane/acetone 3:1);  $[\alpha]_D^{25} -$

14.7 (*c* 0.29,  $CHCl_3$ ); IR (neat)  $\nu_{max} = 3468, 2928, 1783, 1462, 1218, 1129, 1055\text{ cm}^{-1}$ ;

$^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  4.35 (1H, dd,  $J = 0.6, 5.5$  Hz), 3.93 (1H, d,  $J = 10.9$  Hz),

2.77 (1H, dd,  $J = 5.5, 18.5$  Hz), 2.67 (1H, dd,  $J = 0.6, 18.5$  Hz), 2.23 (1H, d,  $J = 10.9$  Hz),

1.93 (1H, dq,  $J = 14.6, 7.5$  Hz), 1.80 (1H, dq,  $J = 14.6, 7.5$  Hz), 1.60 (1H, dd,  $J = 4.0,$

14.2 Hz), 1.58 (1H, dd,  $J = 7.5, 14.2$  Hz), 1.50 (1H, m), 1.47 (1H, dd,  $J = 14.2, 7.5$  Hz),

1.38–1.21 (6H, m), 1.17 (1H, m), 1.03 (3H, t,  $J = 7.5$  Hz), 0.96 (3H, d,  $J = 6.6$  Hz), 0.91



(3H, t,  $J = 7.5$  Hz), 0.89 (3H, t,  $J = 6.8$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  174.7 (C), 95.0 (C), 87.8 (C), 81.1 (CH), 77.3 (CH), 43.1 ( $\text{CH}_2$ ), 38.33 ( $\text{CH}_2$ ), 38.27 ( $\text{CH}_2$ ), 29.2 ( $\text{CH}_2$ ), 29.0 ( $\text{CH}_2$ ), 28.7 (CH), 26.2 ( $\text{CH}_2$ ), 22.9 ( $\text{CH}_2$ ), 21.1 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 8.0 ( $\text{CH}_3$ ), 7.8 ( $\text{CH}_3$ ); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{Na}]^+$  Calcd for  $\text{C}_{17}\text{H}_{30}\text{O}_4\text{Na}$  321.2042, Found 321.2043.

## Reference

1) Ahlbrecht, H.; Schmidt, R.; Beyer, U. Asymmetric synthesis of  $\beta$ -methylated aliphatic ketones via lithiated 3-[(*S*)-2-(methoxymethyl)pyrrolidino]hex-3-ene, *Eur. J. Org. Chem.* **1998**, 1371.

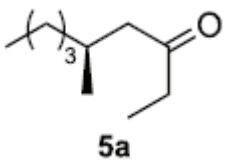
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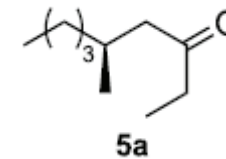
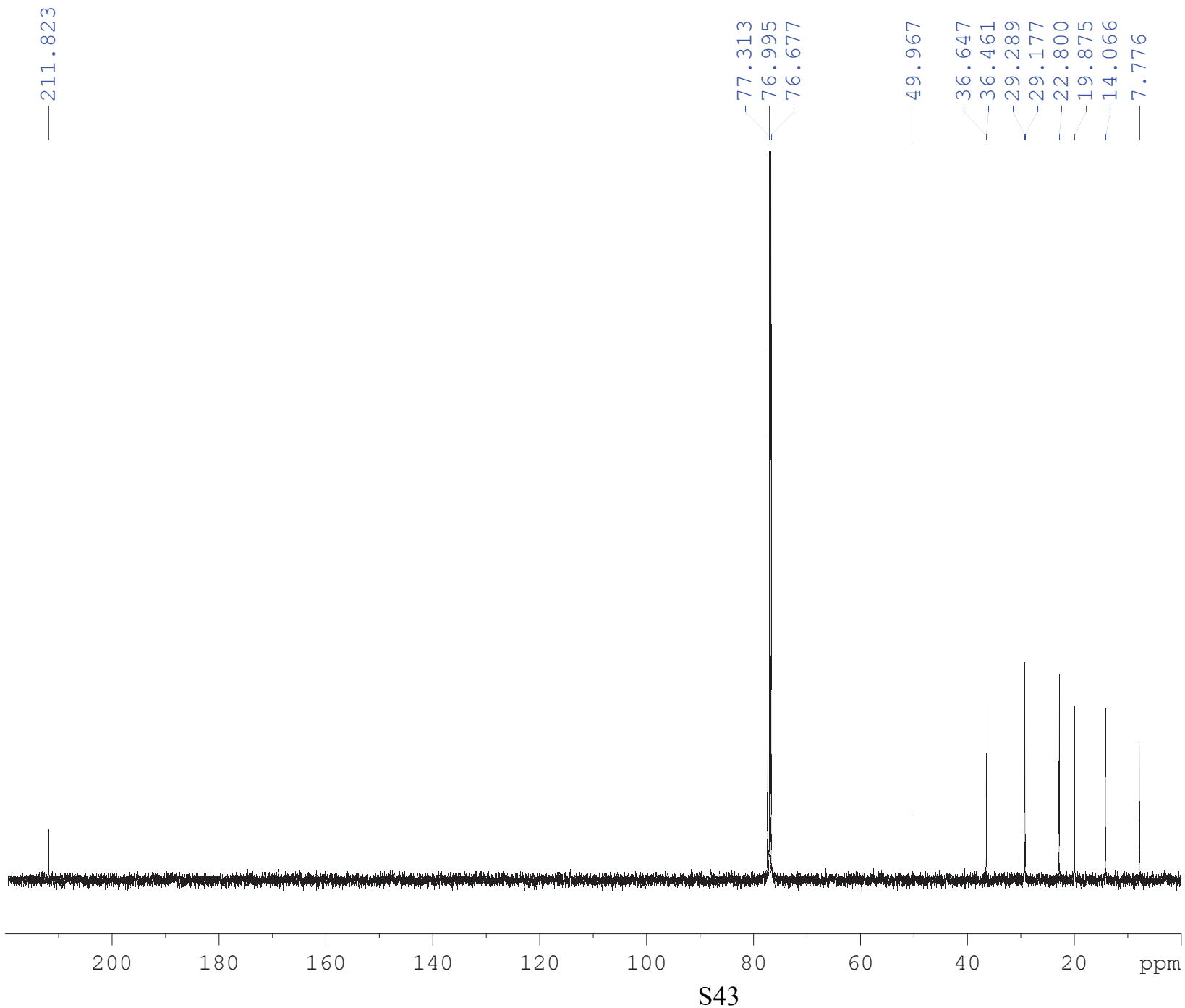
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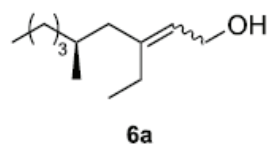
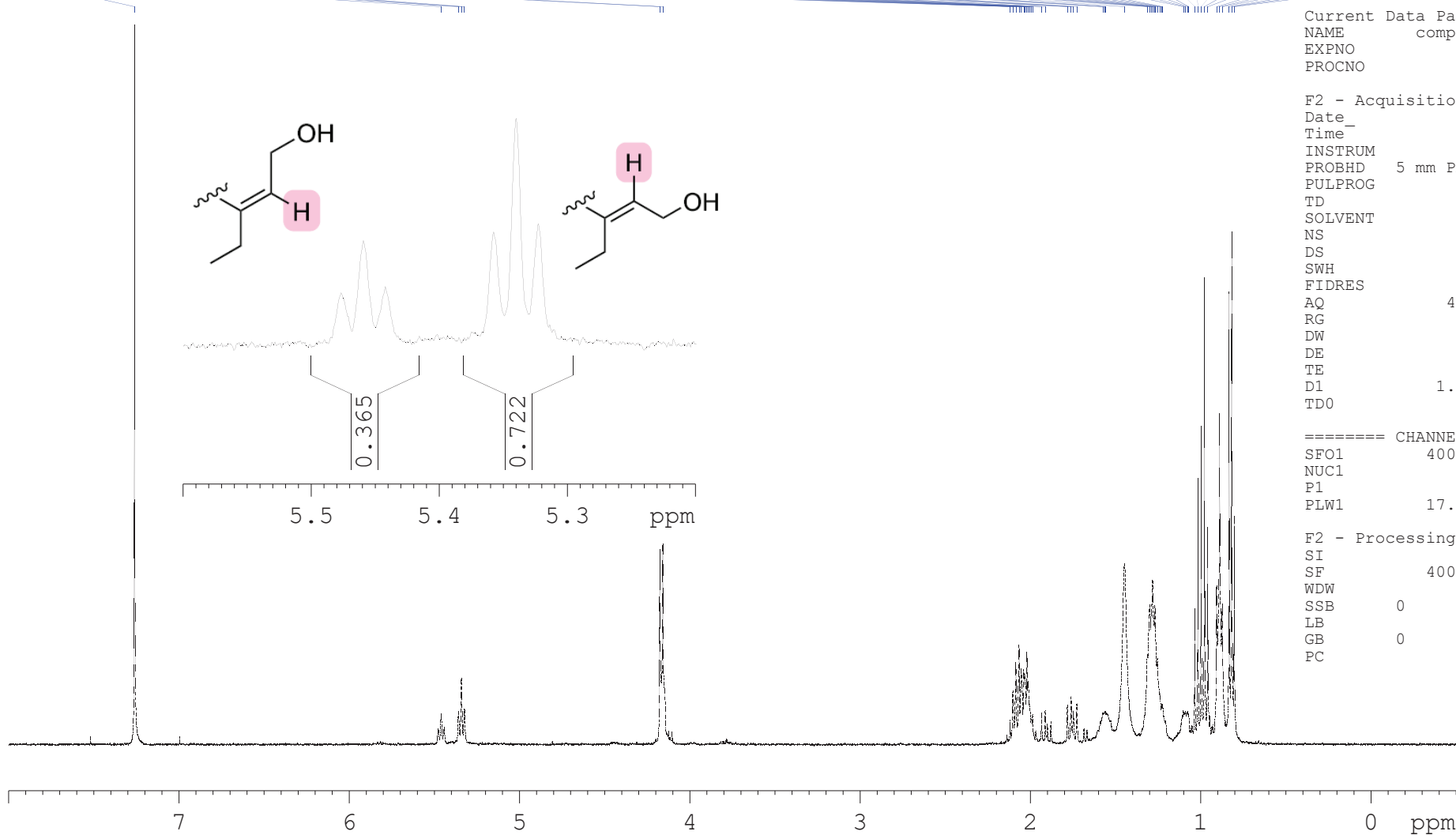
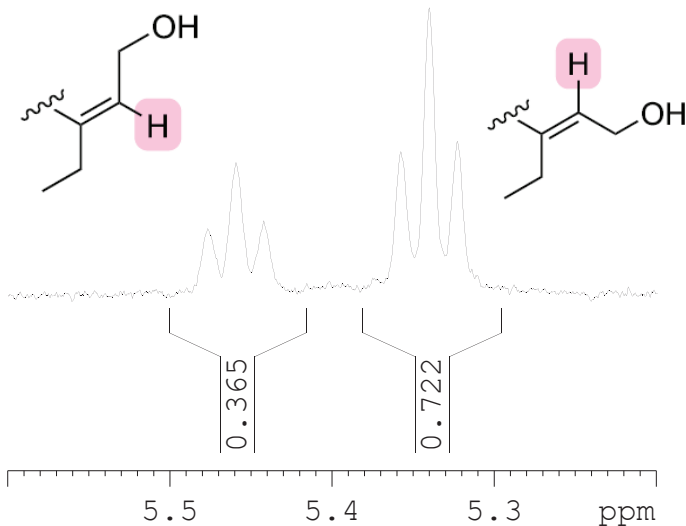
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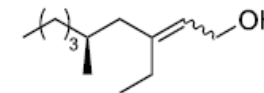
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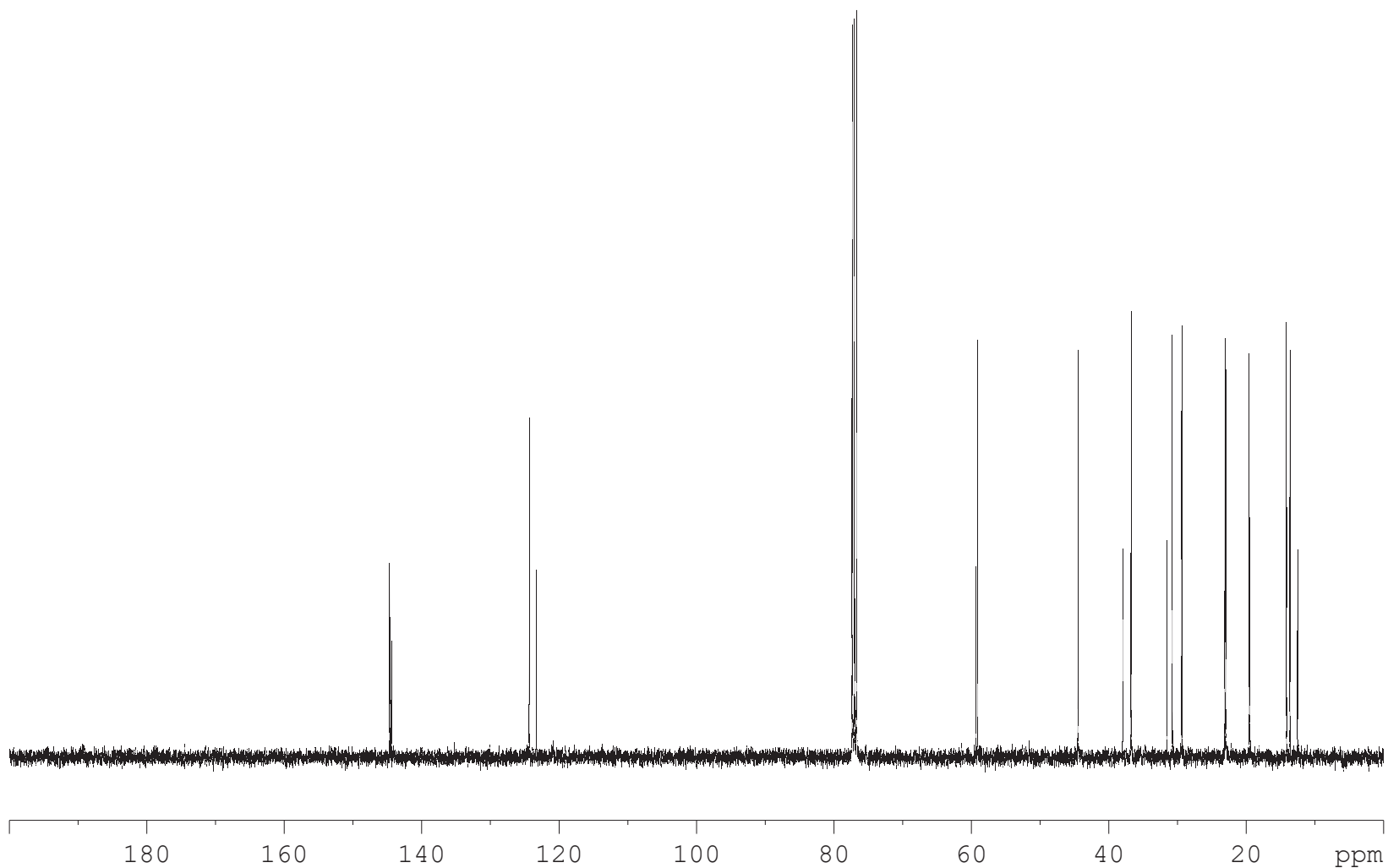
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19.528  
19.468



6a



S45

Current Data Parameters  
NAME compound 6a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210702  
Time\_ 15.45  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 298.8 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

=====  
CHANNEL f1  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

=====  
CHANNEL f2  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.20987999 W  
PLW13 0.10557000 W

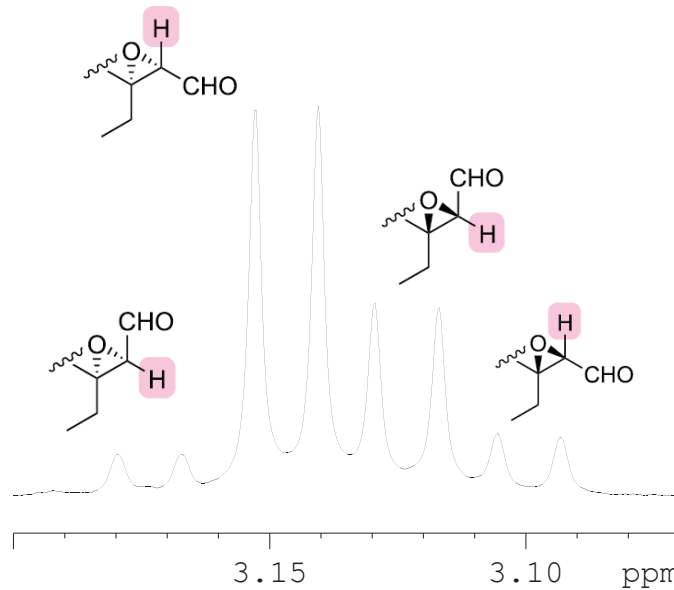
9.522  
9.510  
9.505  
9.493  
7.260  
3.153  
3.141  
3.130  
3.117  
1.894  
1.880  
1.859  
1.845  
1.739  
1.720  
1.701  
1.682  
1.332  
1.318  
1.310  
1.305  
1.301  
1.298  
1.293  
1.287  
1.279  
1.275  
1.272  
1.258  
1.242  
1.239  
1.235  
1.208  
1.186  
1.173  
1.150  
1.056  
1.037  
1.018  
1.010  
1.003  
0.997  
0.991  
0.986  
0.972  
0.966  
0.955  
0.947  
0.930  
0.914  
0.908  
0.897  
0.881  
0.864

Current Data Parameters  
NAME compound 7a  
EXPNO 1  
PROCNO 1

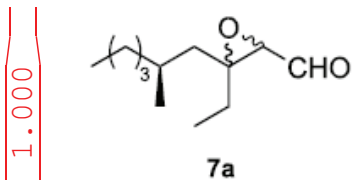
F2 - Acquisition Parameters  
Date\_ 20210712  
Time 20.45  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 114  
DW 62.400 usec  
DE 6.50 usec  
TE 297.3 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 10.00 usec  
PLW1 17.00000000 W

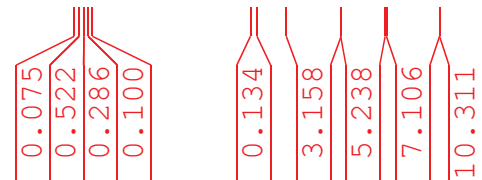
F2 - Processing parameters  
SI 65536  
SF 400.1800099 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



9  
8  
7  
6  
5  
4  
3  
2  
1  
0 ppm

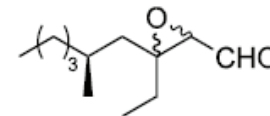


S46



199.916  
199.654

77.316  
76.998  
76.681  
67.502  
64.564  
62.467  
41.509  
37.641  
36.942  
36.799  
36.758  
30.006  
29.483  
29.221  
29.127  
29.063  
27.866  
23.797  
23.745  
22.842  
22.815



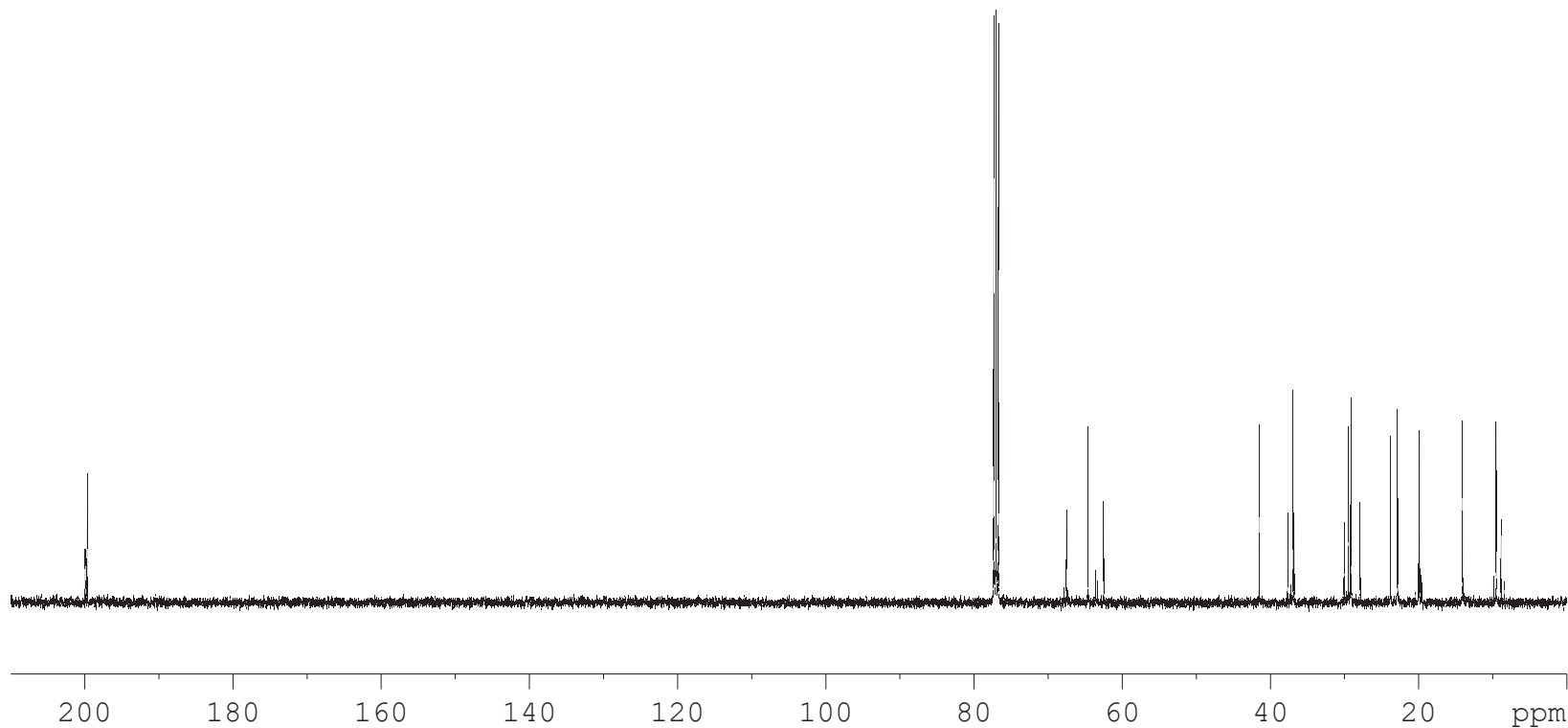
7a

Current Data Parameters  
NAME compound 7a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210712  
Time\_ 21.03  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 299.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

=====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

=====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.20987999 W  
PLW13 0.10557000 W



S47

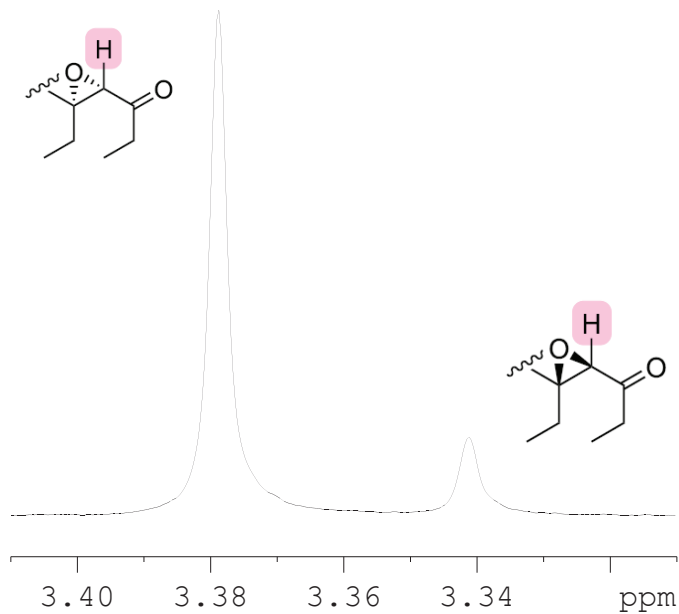
7.260  
3.379  
3.341  
2.631  
2.613  
2.586  
2.568  
2.518  
2.500  
2.473  
2.455  
1.965  
1.952  
1.930  
1.917  
1.588  
1.569  
1.533  
1.514  
1.416  
1.398  
1.380  
1.362  
1.339  
1.328  
1.323  
1.309  
1.300  
1.295  
1.290  
1.286  
1.278  
1.261  
1.244  
1.169  
1.146  
1.134  
1.120  
1.115  
1.111  
1.102  
1.097  
1.083  
1.079  
0.994  
0.975  
0.969  
0.956  
0.952  
0.928  
0.915  
0.909  
0.897  
0.880

Current Data Parameters  
NAME compound 8a  
EXPNO 1  
PROCNO 1

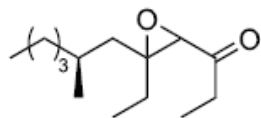
F2 - Acquisition Parameters  
Date\_ 20210811  
Time 13.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 128  
DW 62.400 usec  
DE 6.50 usec  
TE 297.6 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 10.00 usec  
PLW1 17.00000000 W

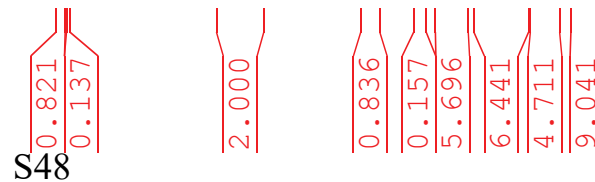
F2 - Processing parameters  
SI 65536  
SF 400.1800099 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



7 6 5 4 3 2 1 0 ppm

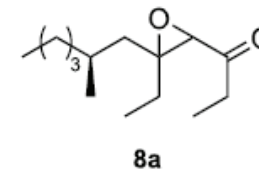
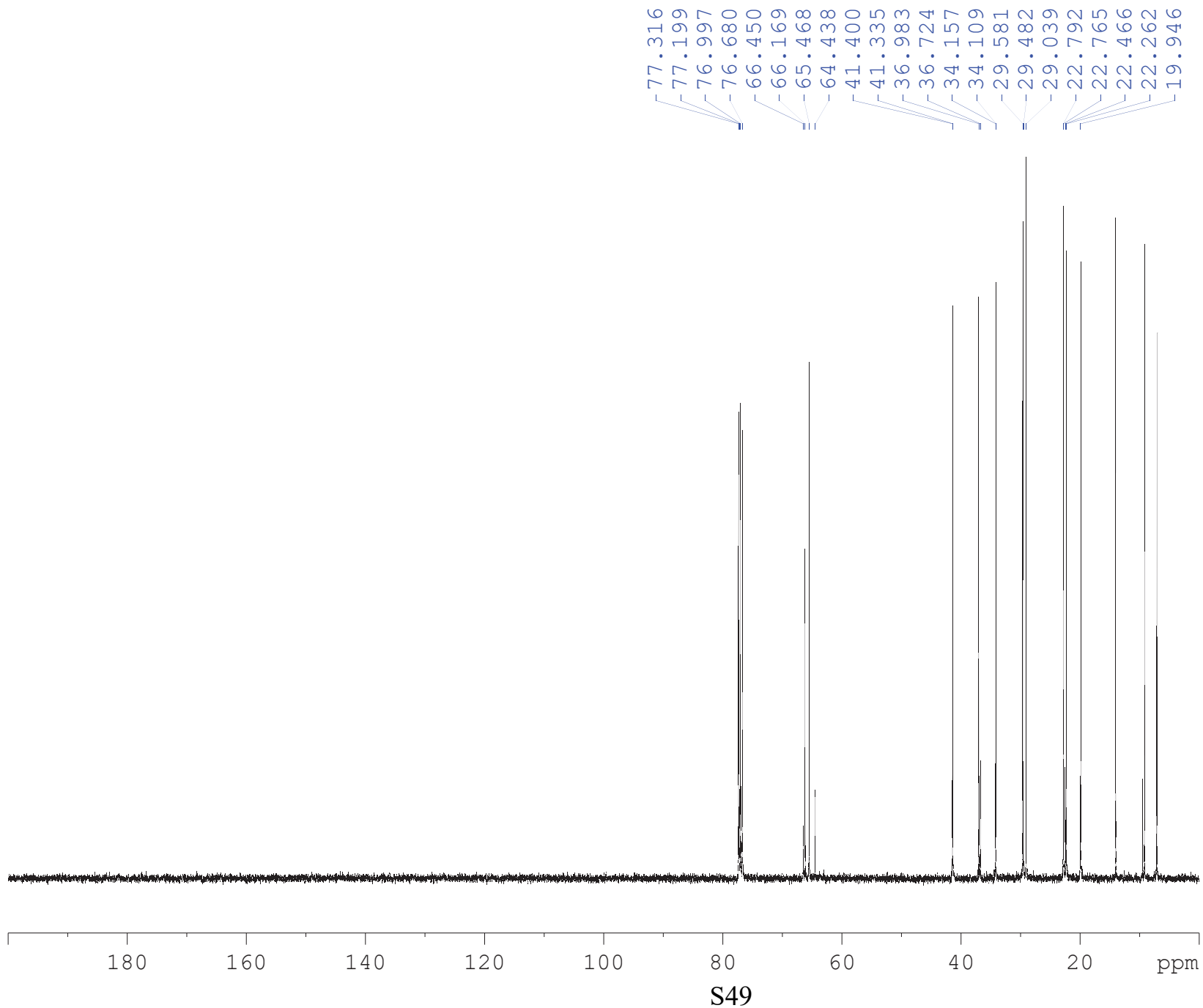


8a



S48





Current Data Parameters  
 NAME compound 8a  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20210811  
 Time\_ 14.04  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 297.9 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

==== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.20987999 W  
 PLW13 0.10557000 W

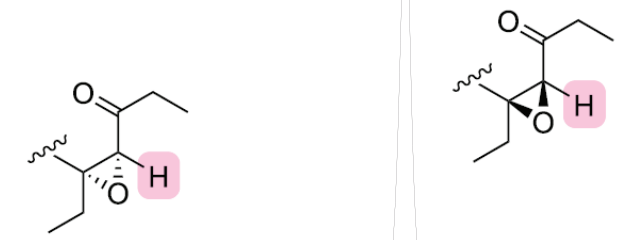
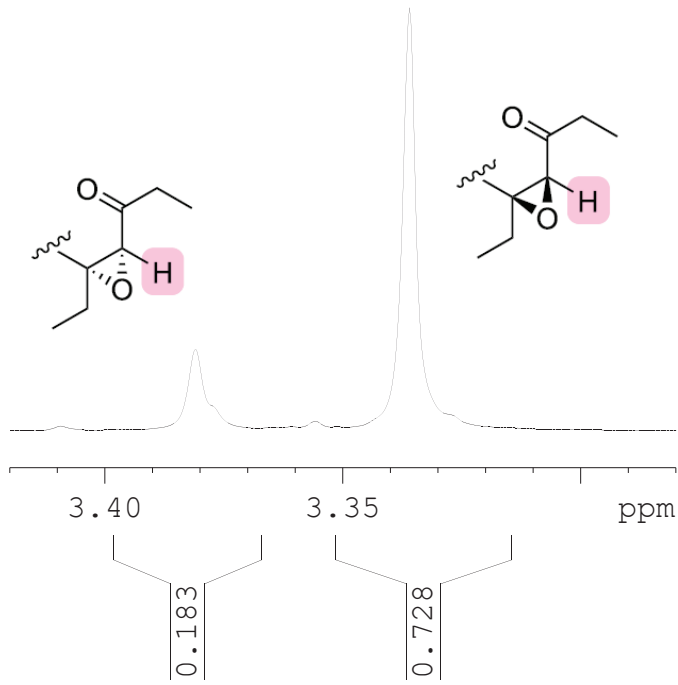
7.260  
3.381  
3.336  
2.565  
2.547  
2.529  
2.511  
1.859  
1.841  
1.824  
1.805  
1.652  
1.635  
1.620  
1.613  
1.599  
1.578  
1.569  
1.564  
1.543  
1.525  
1.507  
1.287  
1.281  
1.279  
1.271  
1.262  
1.257  
1.246  
1.231  
1.219  
1.212  
1.205  
1.202  
1.196  
1.190  
1.169  
1.155  
1.134  
1.112  
1.093  
1.075  
0.982  
0.972  
0.963  
0.948  
0.945  
0.896  
0.884  
0.878  
0.867  
0.861  
0.787  
0.771

Current Data Parameters  
 NAME compound 9a  
 EXPNO 1  
 PROCNO 1

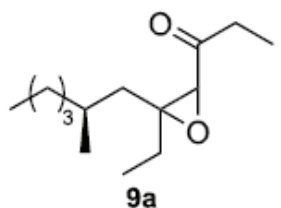
F2 - Acquisition Parameters  
 Date\_ 20210804  
 Time 18.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 101  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 297.2 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 =====  
 SFO1 400.1824713 MHz  
 NUC1 1H  
 P1 10.00 usec  
 PLW1 17.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1800099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



7  
6  
5  
4  
3  
2  
1  
0 ppm



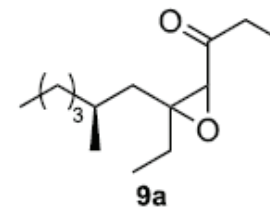
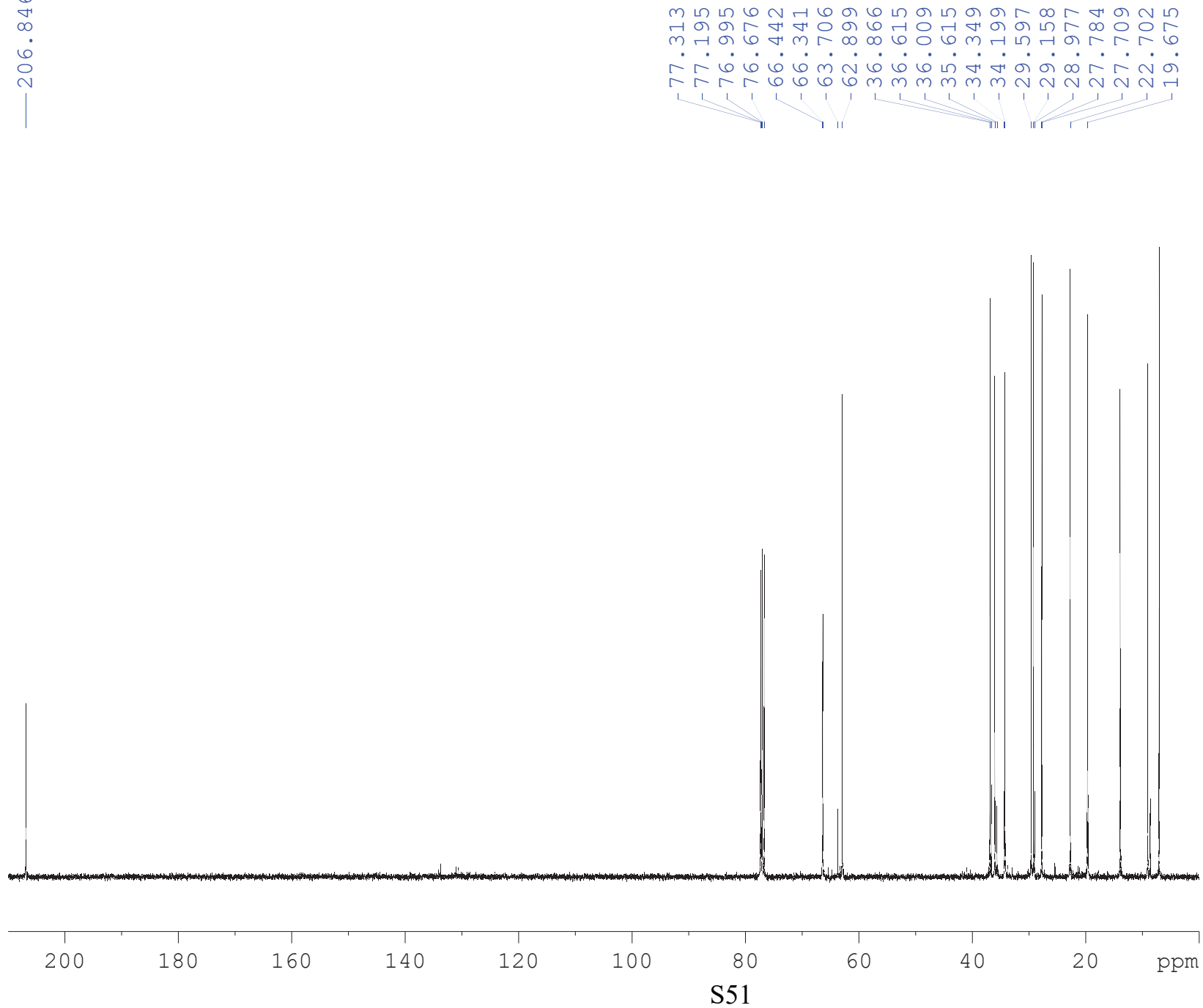
S50

0.183  
0.728

2.000

0.864  
4.287  
5.852  
3.078  
0.877  
5.555  
2.848  
0.491

— 206.846



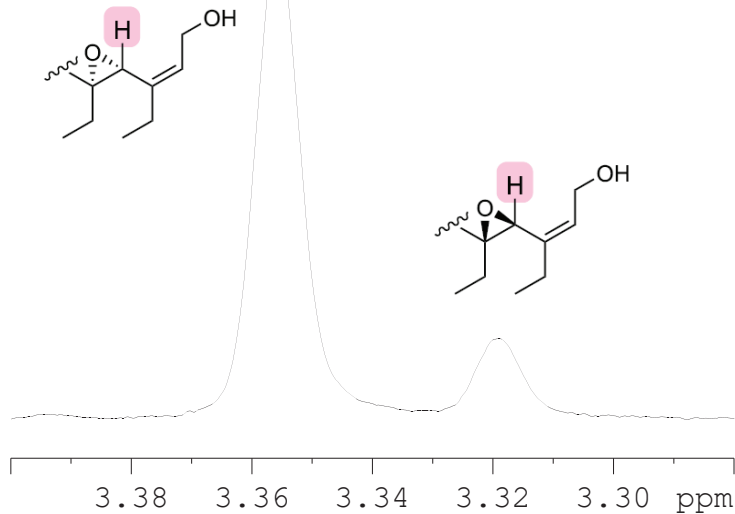
Current Data Parameters  
NAME Compound 9a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210804  
Time\_ 16.36  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 298.6 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.20987999 W  
PLW13 0.10557000 W

7.260  
5.626  
5.622  
5.609  
4.139  
3.356  
2.136  
2.122  
2.119  
2.105  
2.051  
2.032  
2.014  
2.002  
1.990  
1.966  
1.955  
1.597  
1.578  
1.543  
1.524  
1.517  
1.345  
1.326  
1.308  
1.299  
1.295  
1.291  
1.278  
1.273  
1.252  
1.087  
1.068  
1.049  
1.031  
1.020  
0.993  
0.974  
0.966  
0.956  
0.950  
0.945  
0.937  
0.929  
0.916  
0.909  
0.899  
0.882

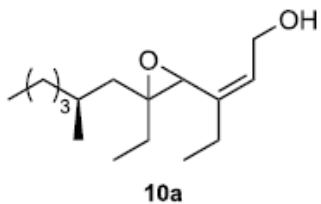


Current Data Parameters  
NAME compound 10a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210812  
Time\_ 14.12  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 128  
DW 62.400 usec  
DE 6.50 usec  
TE 297.8 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 10.00 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800099 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



1.000

1.038

1.045

0.824

0.138

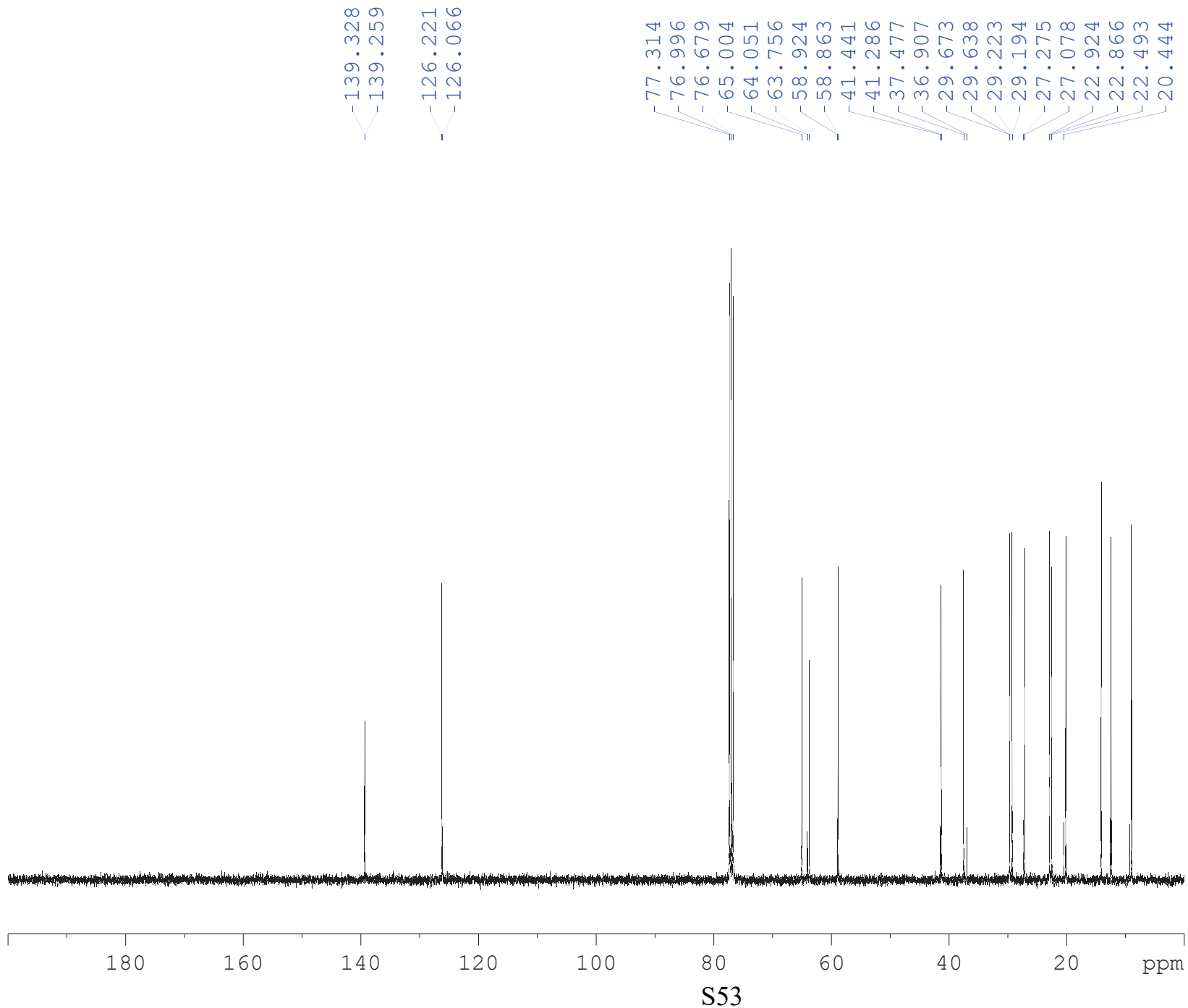
S52

4.027

10.463

7.384

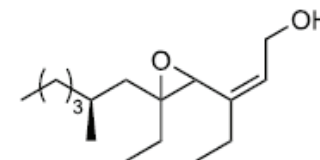
13.679



139.328  
139.259

126.221  
126.066

77.314  
76.996  
76.679  
65.004  
64.051  
63.756  
58.924  
58.863  
41.441  
41.286  
37.477  
36.907  
29.673  
29.638  
29.223  
29.194  
27.275  
27.078  
22.924  
22.866  
22.493  
20.444



10a

Current Data Parameters  
NAME compound 10a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210812  
Time\_ 14.30  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 298.2 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.20987999 W  
PLW13 0.10557000 W

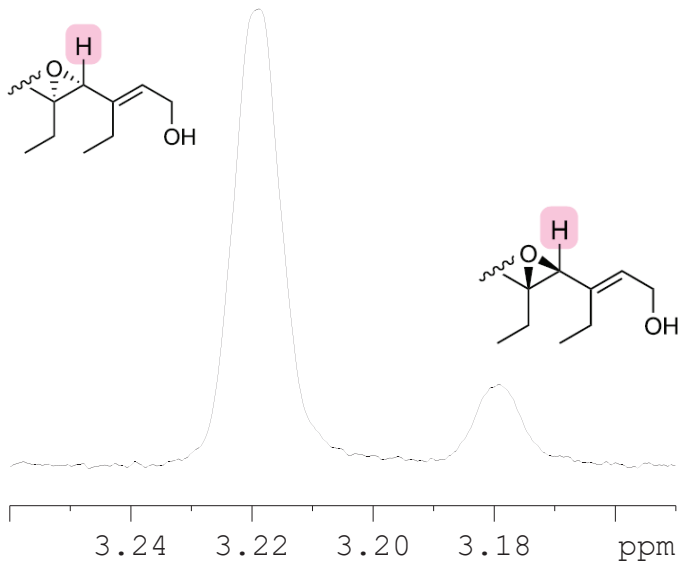
7.260  
5.562  
5.544  
5.527  
4.240  
4.222  
3.219  
2.176  
2.171  
2.163  
2.157  
2.145  
2.138  
2.036  
2.024  
2.002  
1.990  
1.589  
1.531  
1.512  
1.494  
1.477  
1.458  
1.445  
1.440  
1.427  
1.363  
1.345  
1.327  
1.308  
1.301  
1.280  
1.255  
1.236  
1.207  
1.192  
1.179  
1.053  
1.034  
1.028  
1.015  
1.010  
0.992  
0.970  
0.965  
0.951  
0.940  
0.932  
0.923  
0.915  
0.904  
0.899  
0.889  
0.881

Current Data Parameters  
NAME compound 11a  
EXPNO 1  
PROCNO 1

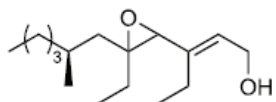
F2 - Acquisition Parameters  
Date\_ 20210819  
Time 15.19  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 161  
DW 62.400 usec  
DE 6.50 usec  
TE 297.2 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 10.00 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



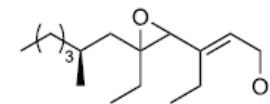
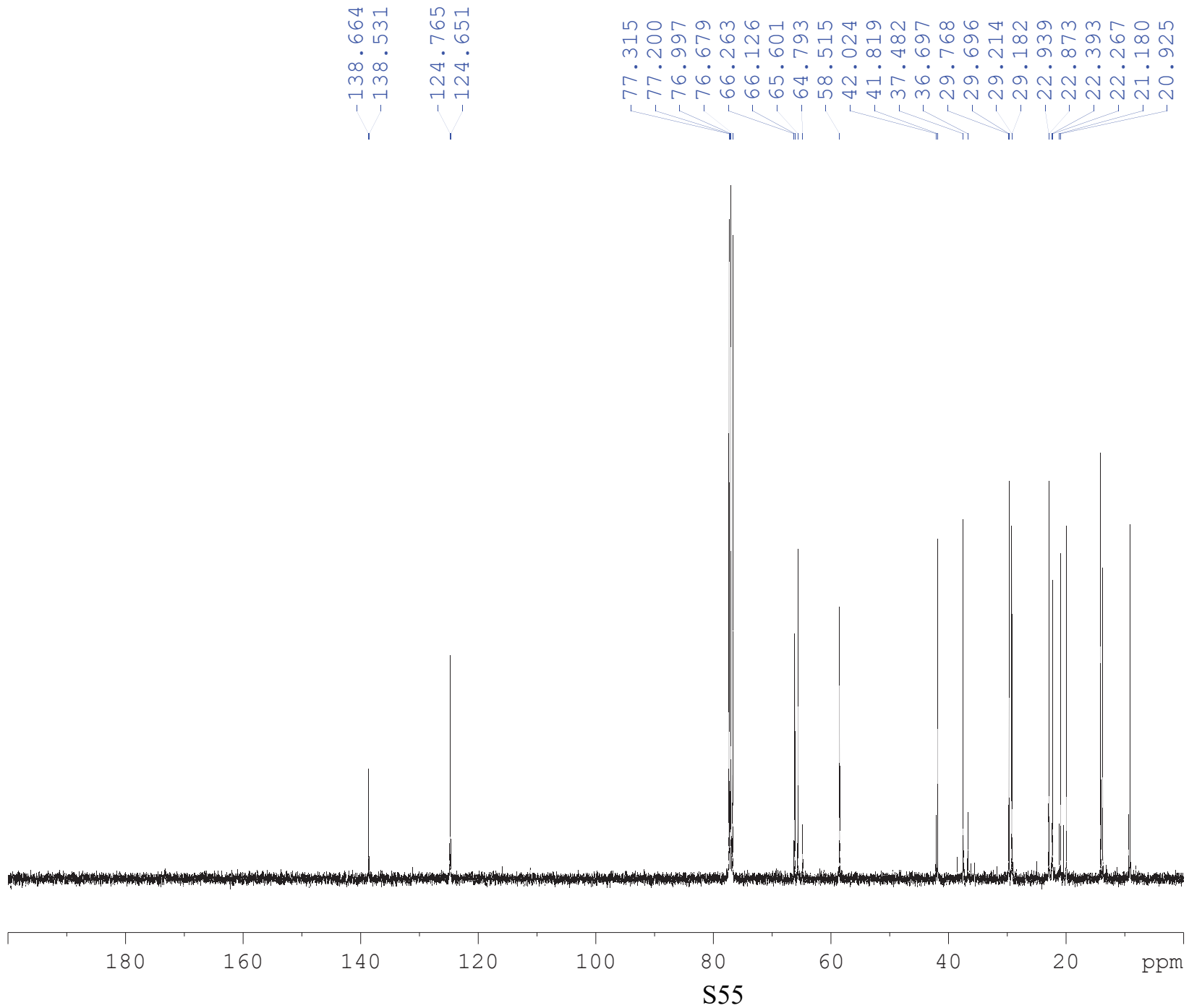
7  
6  
5  
4  
3  
2  
1  
0 ppm



11a



S54



11a

Current Data Parameters  
 NAME compound 11a  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20210819  
 Time\_ 15.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 297.3 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

==== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.20987999 W  
 PLW13 0.10557000 W

7.260  
3.919  
3.917  
3.905  
3.899  
3.890  
3.887  
3.062  
3.049  
3.047  
3.035  
2.996  
2.030  
2.019  
1.995  
1.984  
1.945  
1.928  
1.914  
1.873  
1.854  
1.837  
1.818  
1.784  
1.765  
1.748  
1.729  
1.673  
1.654  
1.647  
1.636  
1.628  
1.619  
1.611  
1.303  
1.294  
1.285  
1.268  
1.257  
1.244  
1.224  
1.206  
1.048  
1.029  
1.011  
0.996  
0.958  
0.936  
0.920  
0.910  
0.906  
0.894  
0.878  
0.872

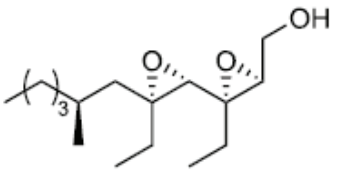
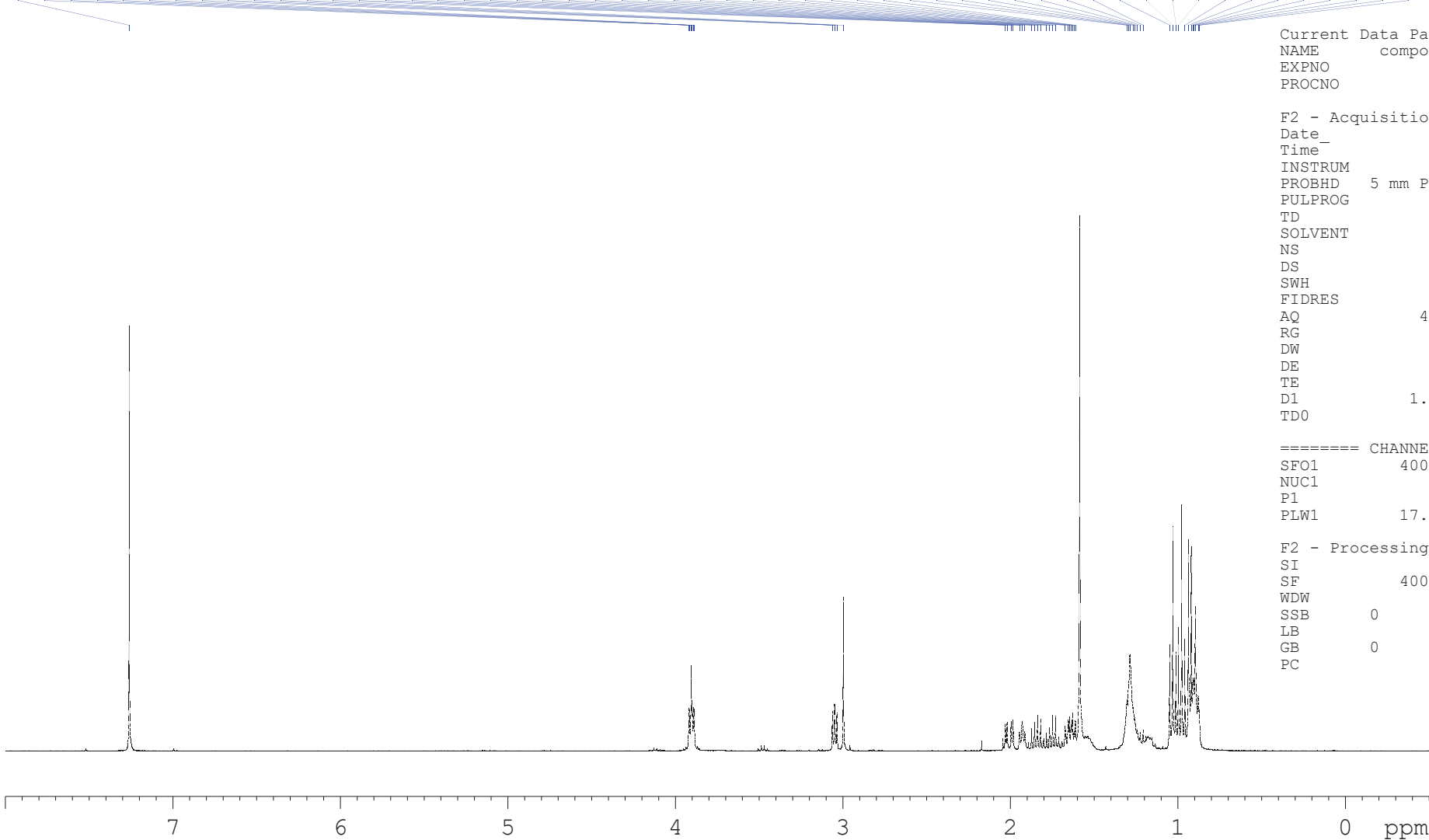
```

Current Data Parameters
NAME      compound 12a
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20210803
Time     16.32
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894465 sec
RG       144
DW       62.400 usec
DE       6.50 usec
TE       297.2 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
SFO1     400.1824713 MHz
NUC1     1H
P1       10.00 usec
PLW1     17.00000000 W

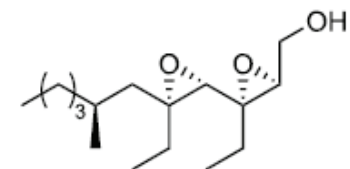
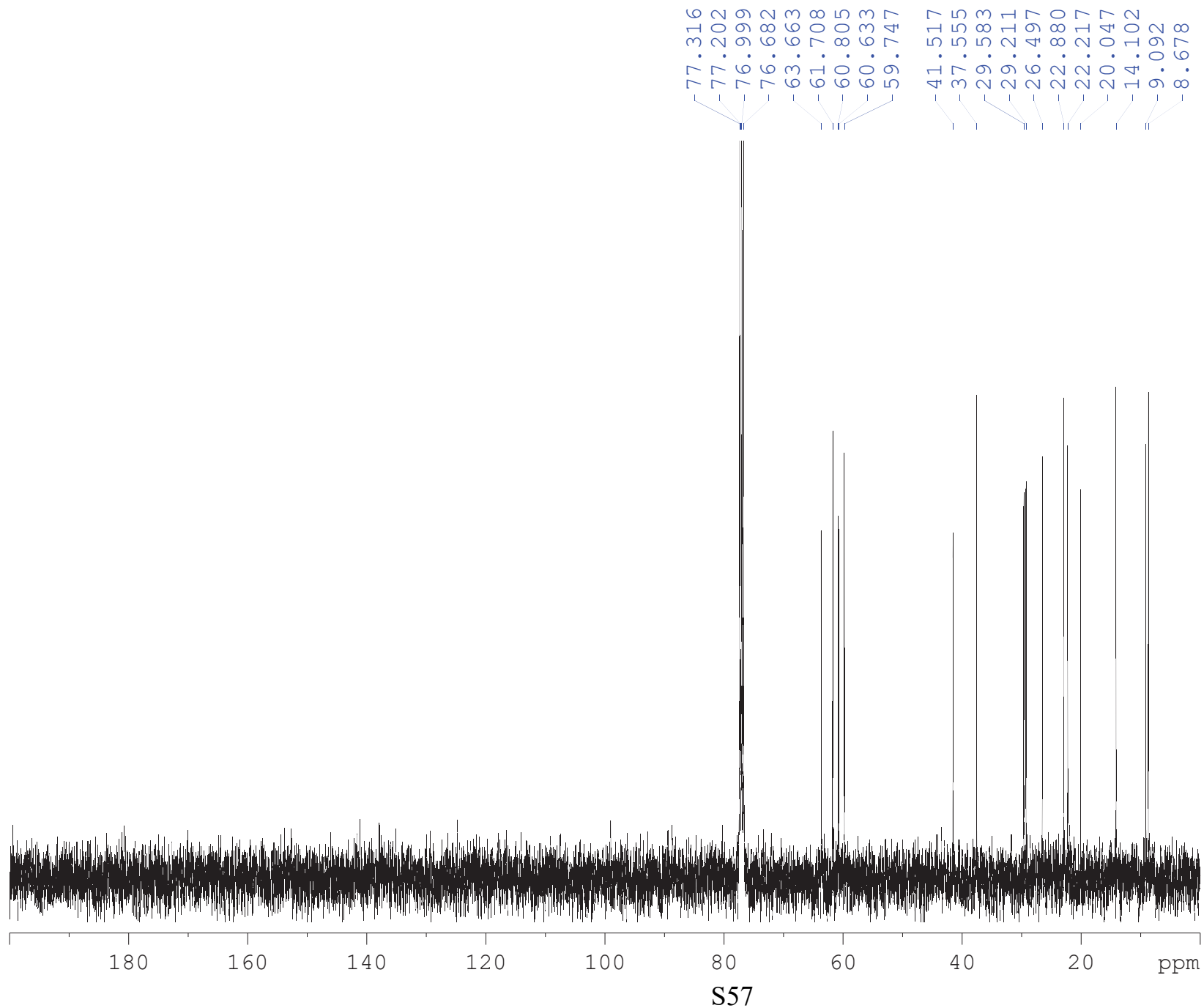
F2 - Processing parameters
SI       65536
SF       400.1800098 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



12a

2.074  
S56  
1.059  
1.000  
1.075  
1.005  
2.288  
7.823  
6.918  
14.327





12a

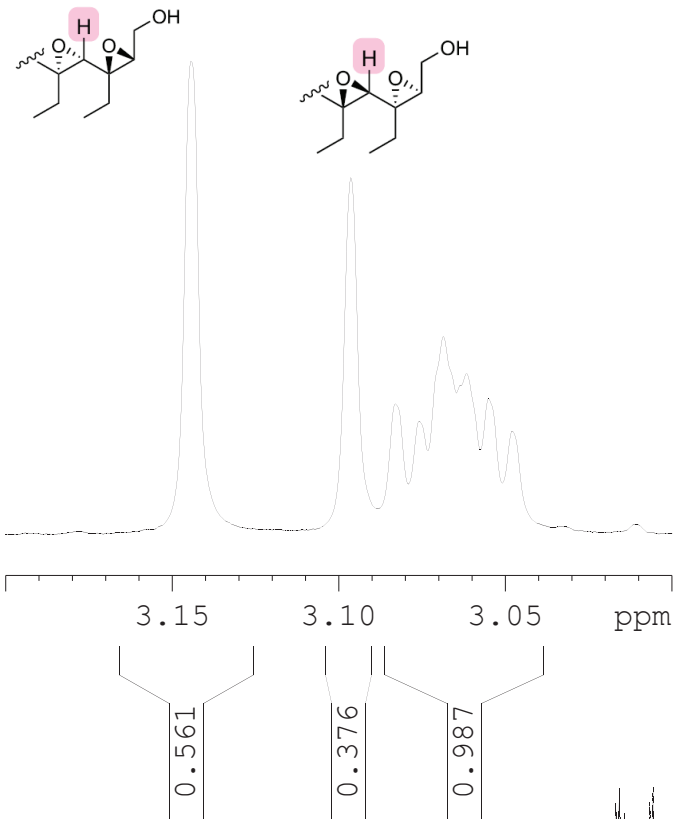
Current Data Parameters  
NAME compound 12a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210803  
Time\_ 19.14  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 298.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

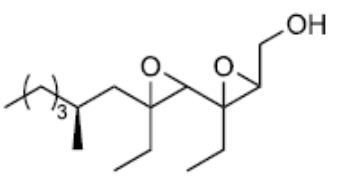
==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.20987999 W  
PLW13 0.10557000 W

7.260  
3.941  
3.925  
3.784  
3.767  
3.144  
3.096  
3.083  
3.069  
3.062  
3.055  
2.100  
1.939  
1.776  
1.758  
1.730  
1.726  
1.716  
1.711  
1.707  
1.697  
1.693  
1.688  
1.680  
1.674  
1.661  
1.562  
1.526  
1.491  
1.473  
1.441  
1.437  
1.425  
1.328  
1.319  
1.306  
1.295  
1.286  
1.283  
1.264  
1.054  
1.036  
1.033  
1.017  
1.007  
0.988  
0.969  
0.950  
0.947  
0.931  
0.928  
0.912  
0.894  
0.877

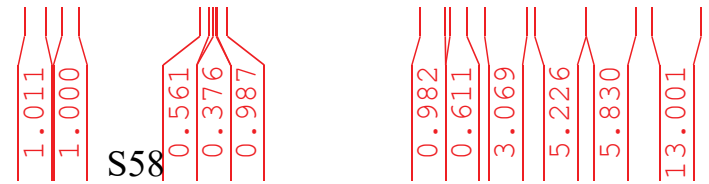


Current Data Parameters  
 NAME compound 13a  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20210819  
 Time\_ 16.02  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 161  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 297.9 K  
 D1 1.00000000 sec  
 TD0 1  
 ===== CHANNEL f1 =====  
 SFO1 400.1824713 MHz  
 NUC1 1H  
 P1 10.00 usec  
 PLW1 17.00000000 W  
 F2 - Processing parameters  
 SI 65536  
 SF 400.1800099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

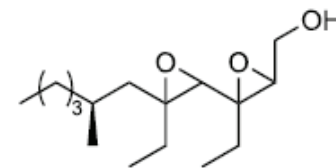
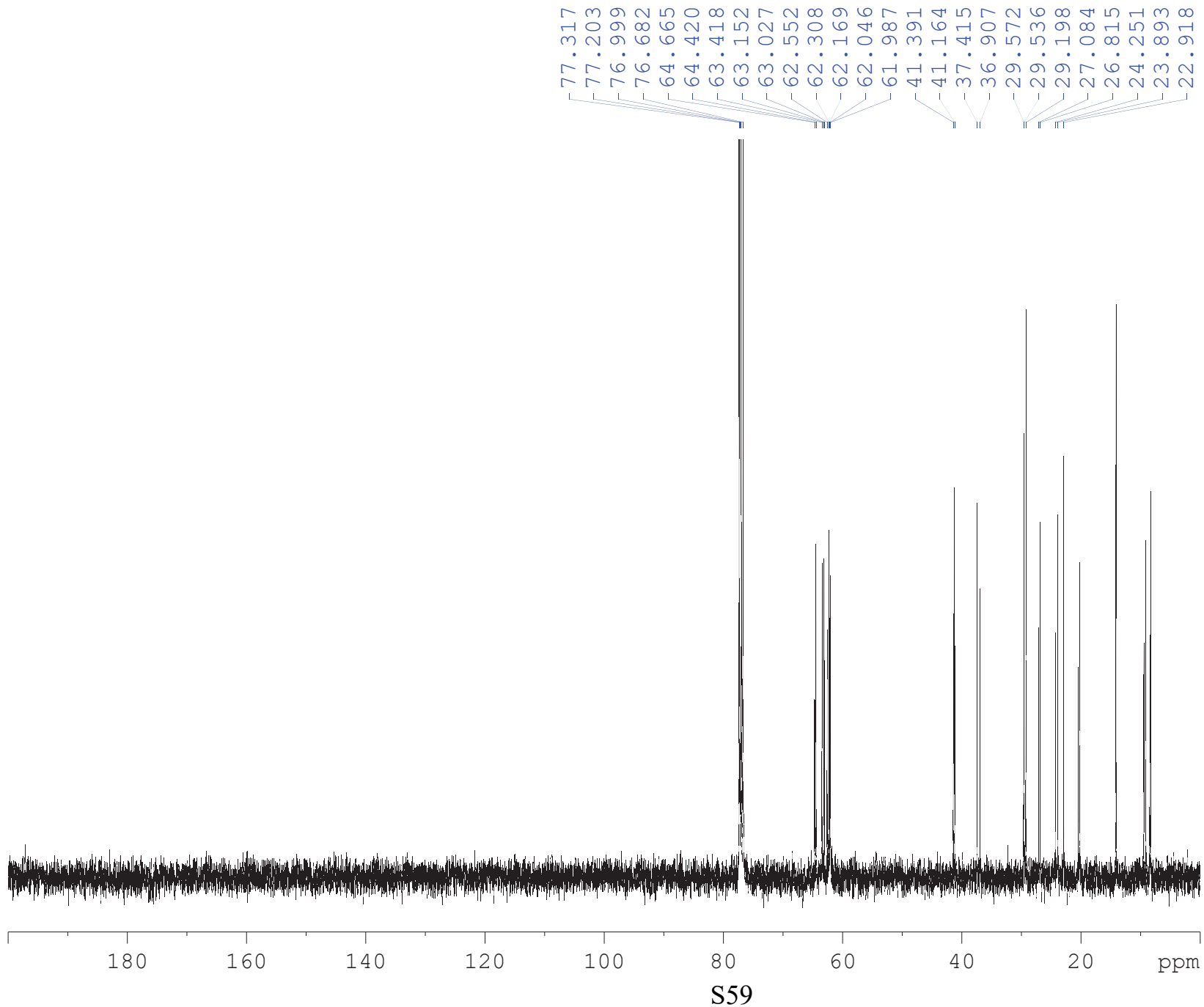
7  
6  
5  
4  
3  
2  
1  
0 ppm



13a



S58



**13a**

Current Data Parameters  
 NAME compound 13a  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20210819  
 Time\_ 16.19  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 297.7 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

==== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.20987999 W  
 PLW13 0.10557000 W

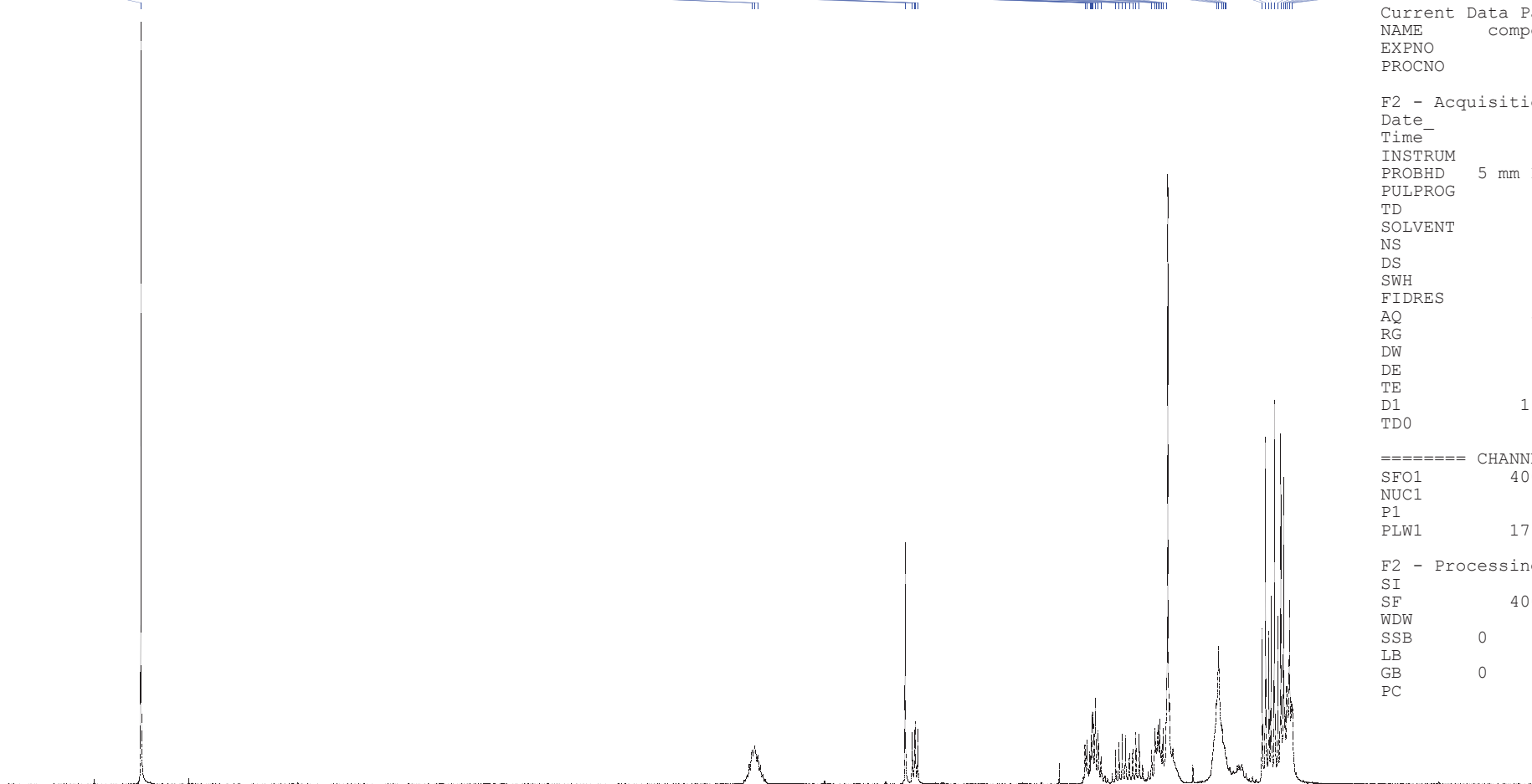
7.260  
3.873  
3.859  
3.840  
3.024  
2.986  
2.972  
2.968  
2.955  
2.029  
2.018  
2.001  
1.994  
1.988  
1.983  
1.970  
1.956  
1.940  
1.858  
1.839  
1.822  
1.804  
1.782  
1.763  
1.746  
1.728  
1.659  
1.641  
1.634  
1.623  
1.614  
1.606  
1.595  
1.578  
1.299  
1.287  
1.273  
1.268  
1.260  
1.252  
1.246  
1.046  
1.028  
1.009  
0.997  
0.978  
0.959  
0.944  
0.927  
0.919  
0.910  
0.894  
0.883  
0.878

Current Data Parameters  
NAME compound 14a  
EXPNO 1  
PROCNO 1

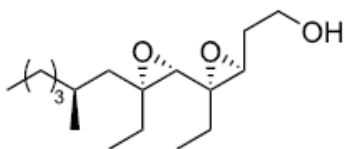
F2 - Acquisition Parameters  
Date\_ 20210928  
Time 12.06  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 144  
DW 62.400 usec  
DE 6.50 usec  
TE 297.6 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 10.00 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

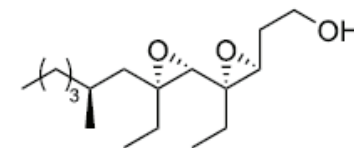
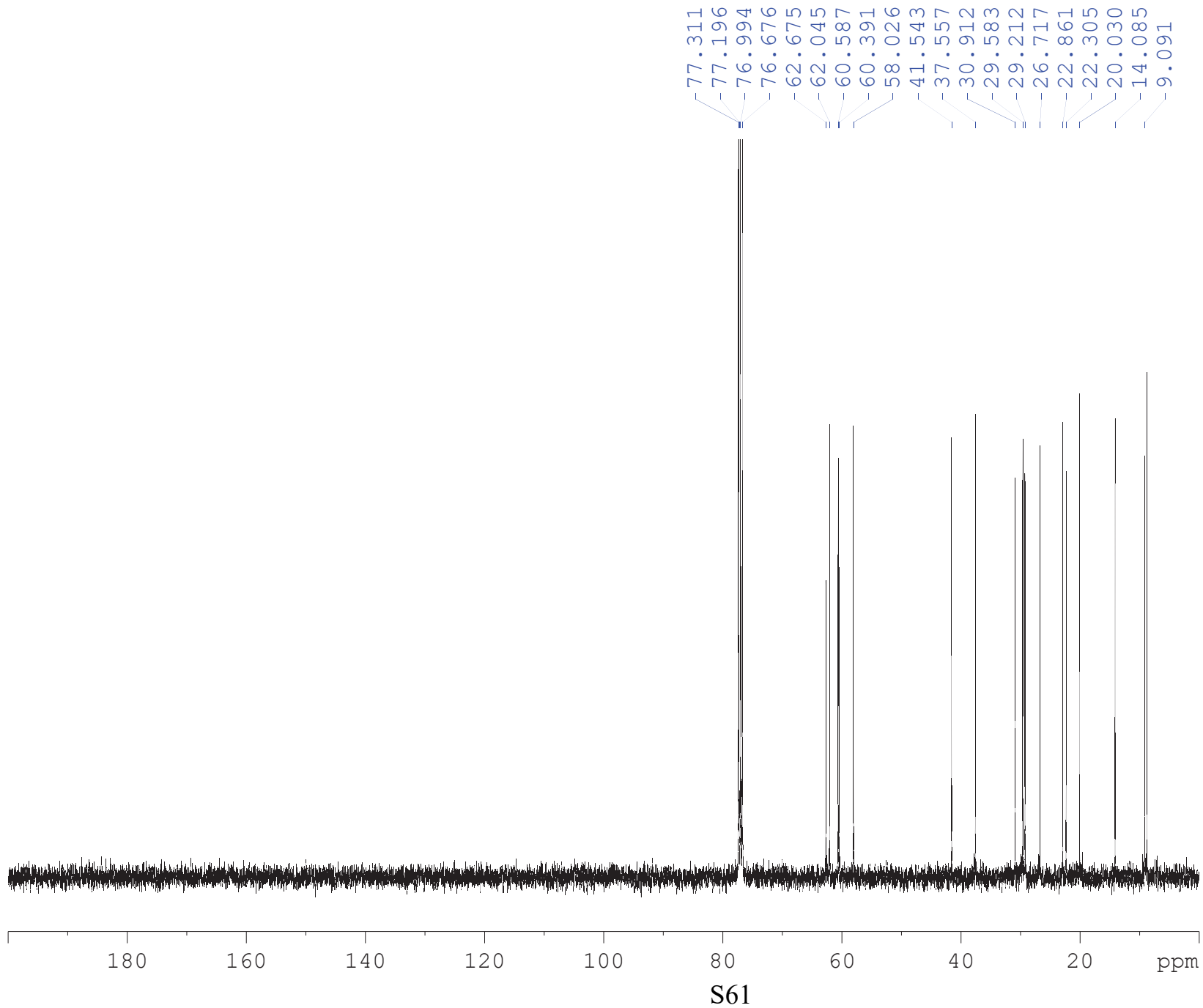


7  
6  
5  
4  
3  
2  
1  
0 ppm



14a

1.980  
S60  
0.871  
1.000  
2.867  
2.134  
7.769  
6.273  
12.948



**14a**

Current Data Parameters  
 NAME compound 14a  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20210826  
 Time\_ 16.26  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 297.7 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

==== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.20987999 W  
 PLW13 0.10557000 W

7.260  
3.717  
3.150  
3.137  
3.125  
3.033  
2.914  
2.902  
2.880  
2.868  
2.818  
2.805  
2.784  
2.771  
2.046  
2.037  
2.018  
2.009  
1.915  
1.901  
1.887  
1.872  
1.771  
1.756  
1.743  
1.728  
1.665  
1.651  
1.636  
1.621  
1.604  
1.589  
1.575  
1.560  
1.310  
1.304  
1.297  
1.291  
1.288  
1.272  
1.265  
1.256  
1.182  
1.036  
1.021  
1.006  
0.991  
0.945  
0.932  
0.909  
0.900  
0.896  
0.882  
0.873

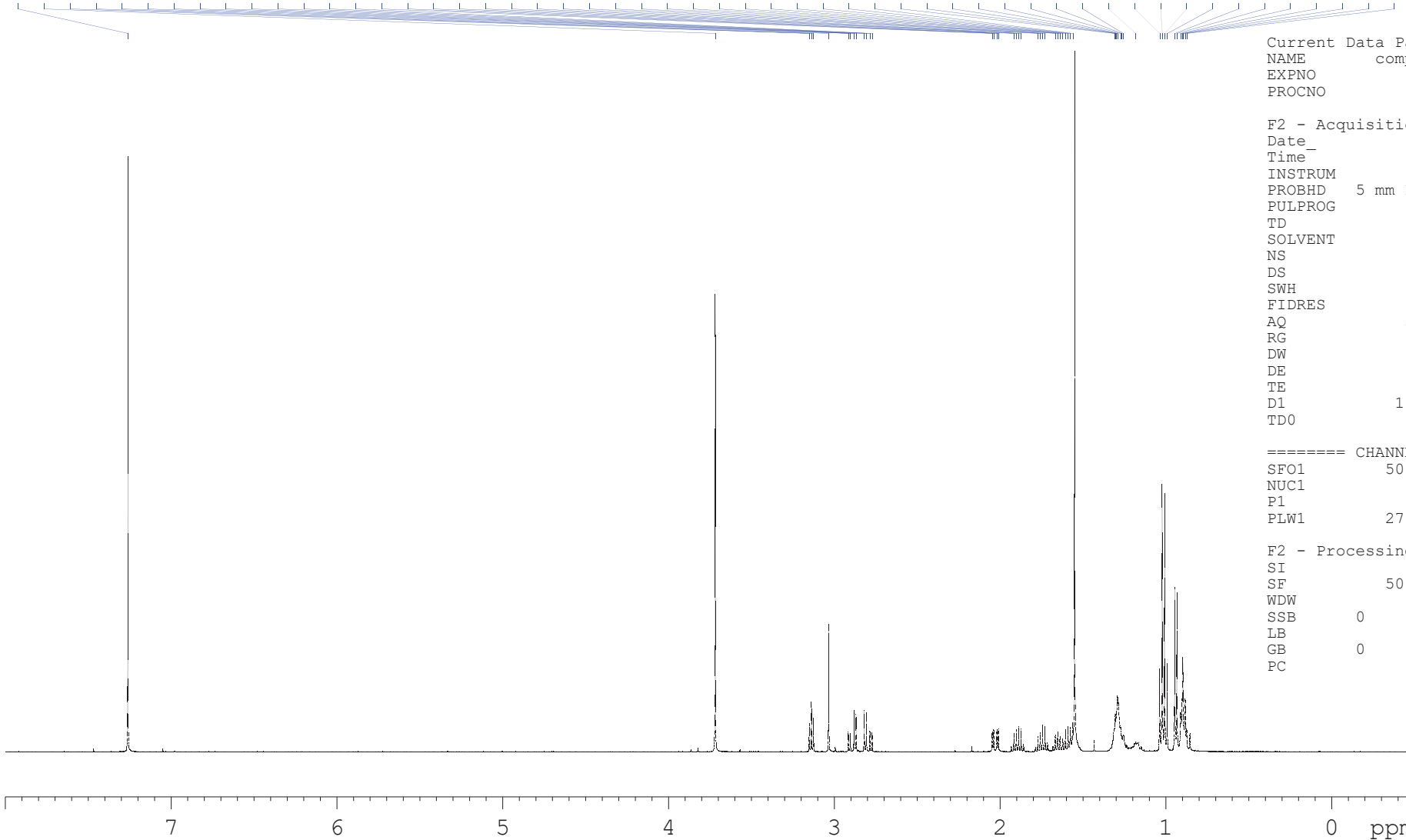
```

Current Data Parameters
NAME      compound 2a
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20211012
Time      14.39
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        4
SWH       10000.000 Hz
FIDRES    0.152588 Hz
AQ        3.2767999 sec
RG        190.86
DW        50.000 usec
DE        6.50 usec
TE        300.0 K
D1        1.00000000 sec
TD0       1

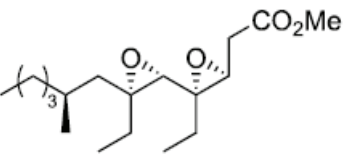
===== CHANNEL f1 =====
SFO1      500.2330891 MHz
NUC1      1H
P1        11.00 usec
PLW1      27.00000000 W

F2 - Processing parameters
SI        65536
SF        500.2300124 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



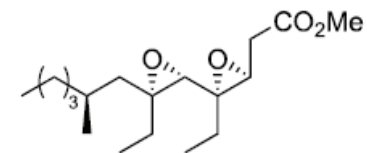
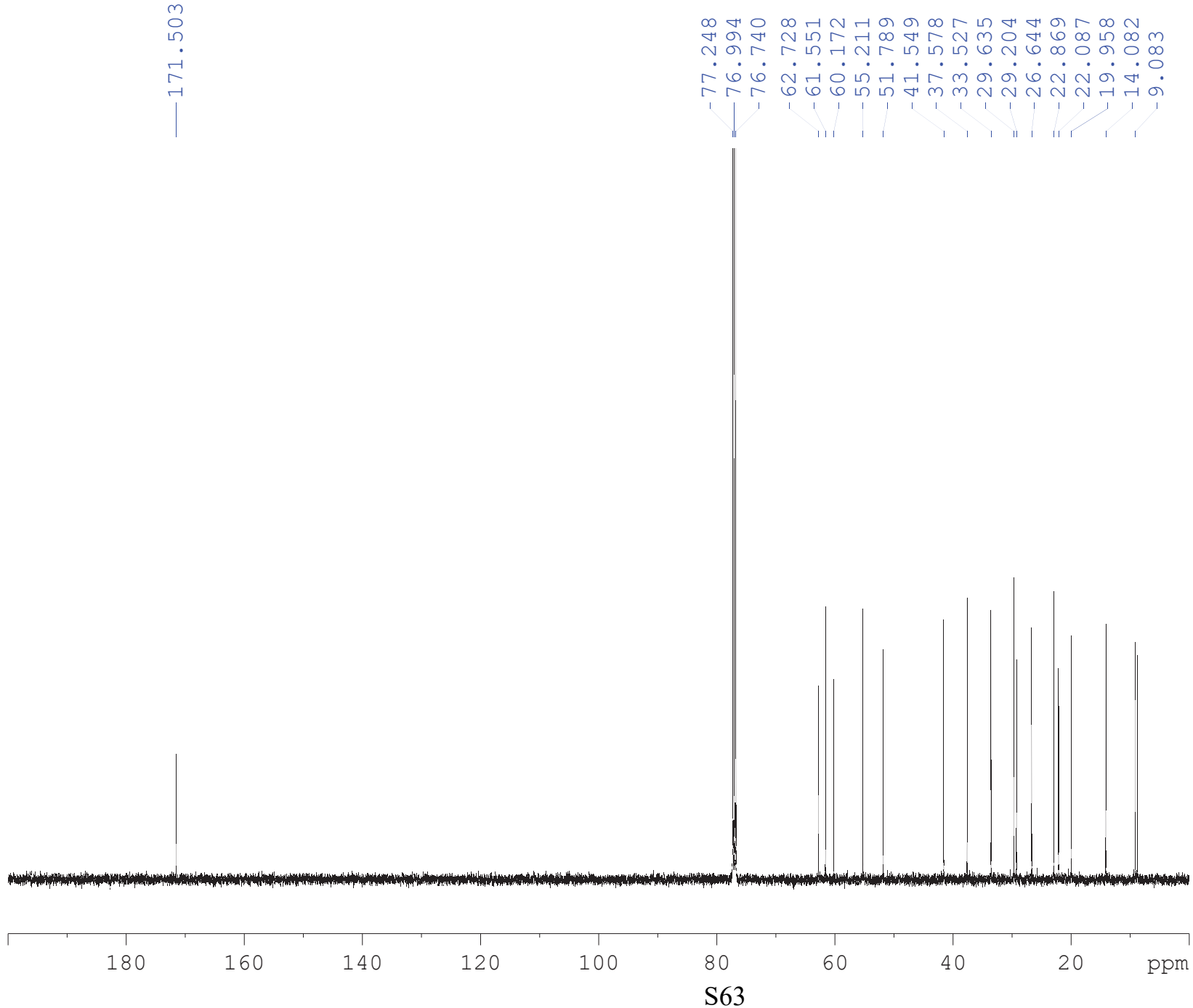
3.027  
1.000  
0.972  
1.007  
1.040  
1.016  
1.046  
1.079  
2.426  
6.815  
6.259  
3.181  
4.039

S62



plakdiepoxide (2a)

—171.503



**plakdiepoxide (2a)**

Current Data Parameters  
NAME compound 2a  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20211012  
Time\_ 16.02  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010048 sec  
RG 190.86  
DW 16.800 usec  
DE 6.50 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 125.7955112 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 88.00000000 W

==== CHANNEL f2 =====  
SFO2 500.2320009 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 27.00000000 W  
PLW12 0.51046997 W  
PLW13 0.32670000 W

7.260  
4.340  
4.339  
4.329  
4.328  
3.878  
3.858  
2.791  
2.780  
2.754  
2.743  
2.693  
2.692  
2.656  
2.655  
2.262  
2.241  
1.927  
1.913  
1.898  
1.816  
1.801  
1.787  
1.614  
1.599  
1.526  
1.504  
1.496  
1.475  
1.467  
1.383  
1.369  
1.354  
1.340  
1.306  
1.304  
1.294  
1.285  
1.279  
1.274  
1.264  
1.262  
1.255  
1.050  
1.035  
1.020  
0.953  
0.940  
0.929  
0.914  
0.909  
0.899  
0.895  
0.881

```

Current Data Parameters
NAME      compound 1a
EXPNO    10
PROCNO   1

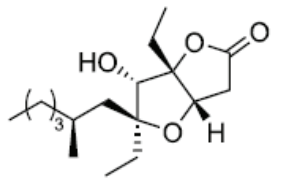
F2 - Acquisition Parameters
Date_    20211012
Time     14.32
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       4
SWH      10000.000 Hz
FIDRES   0.152588 Hz
AQ       3.2767999 sec
RG       190.86
DW       50.000 usec
DE       6.50 usec
TE       300.0 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
SFO1     500.2330891 MHz
NUC1     1H
P1       11.00 usec
PLW1     27.00000000 W

F2 - Processing parameters
SI       65536
SF       500.2300126 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



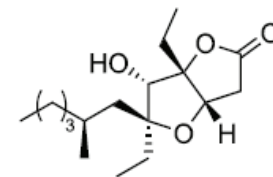
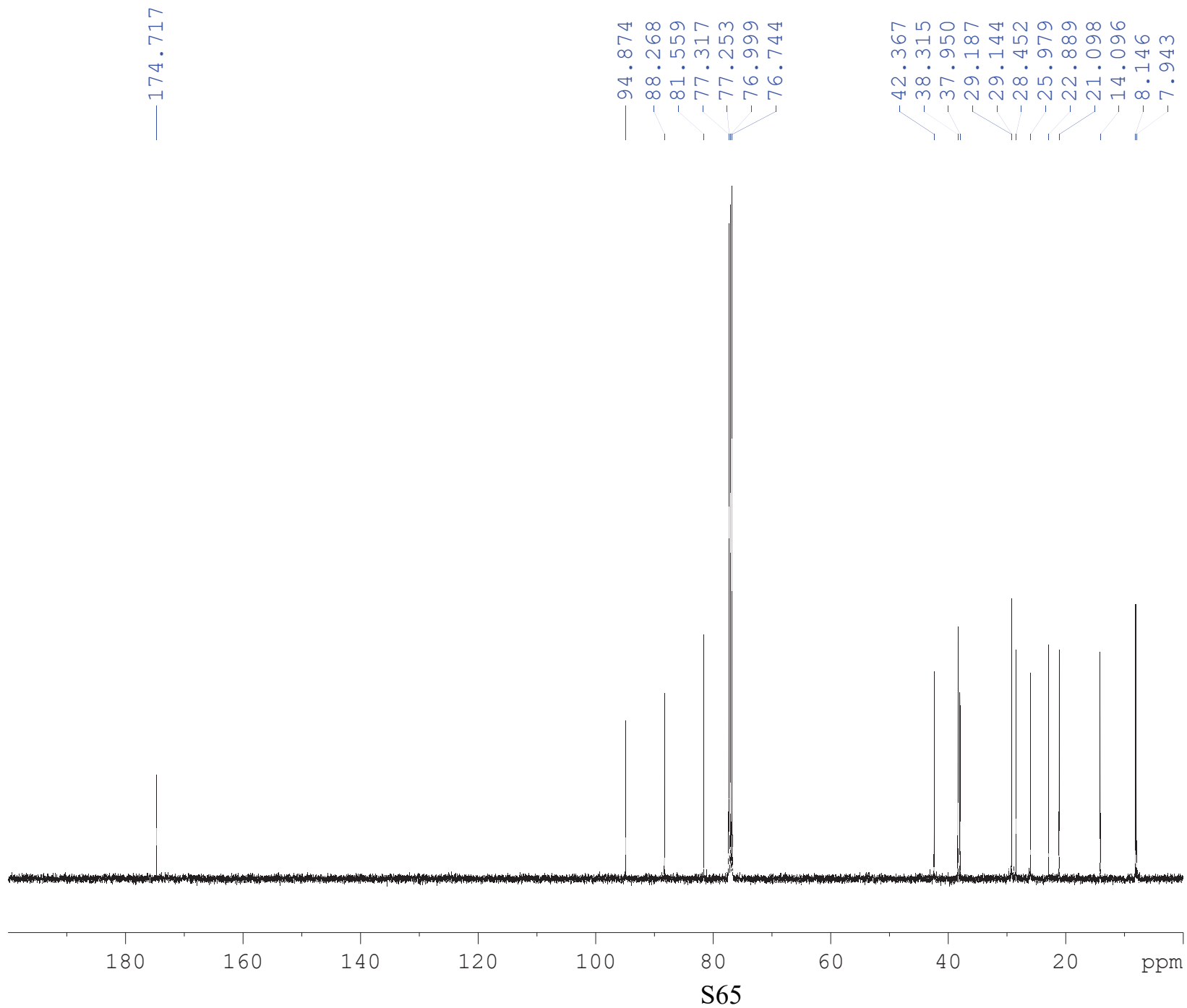
7 6 5 4 3 2 1 0 ppm



plakortone Q (1a)

1.000  
0.976  
S64  
1.010  
1.020  
0.962  
1.020  
1.022  
16.404  
6.825  
1.145  
3.131  
9.436





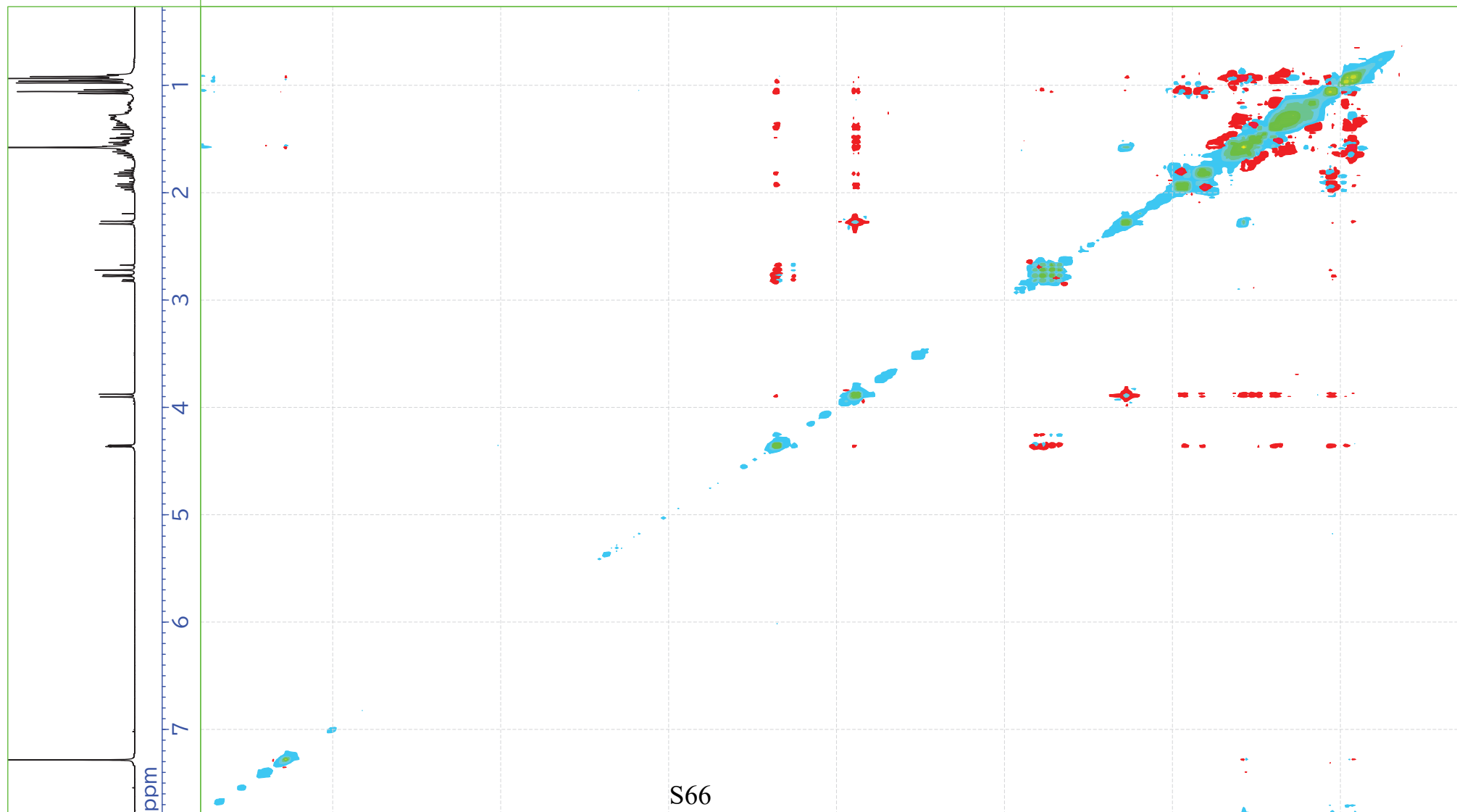
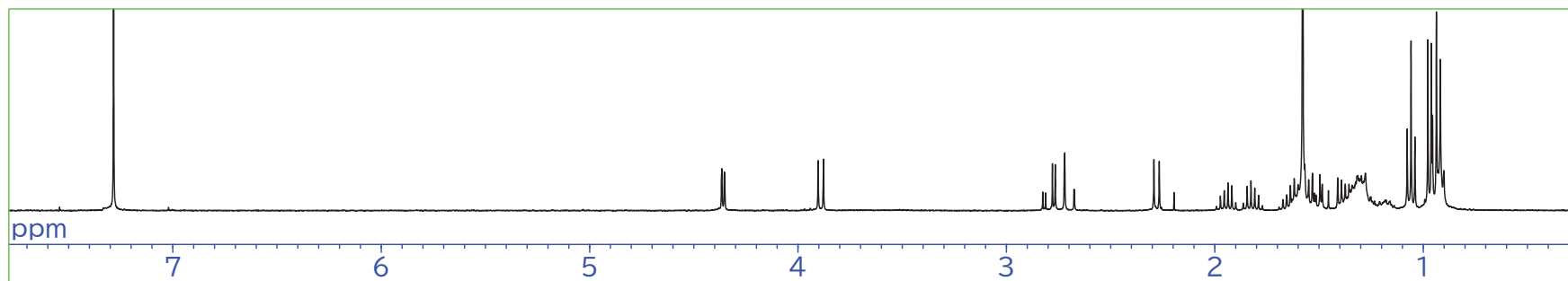
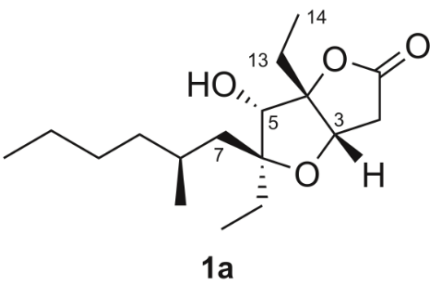
**plakortone Q (1a)**

Current Data Parameters  
 NAME compound 1a  
 EXPNO 10  
 PROCNO 1

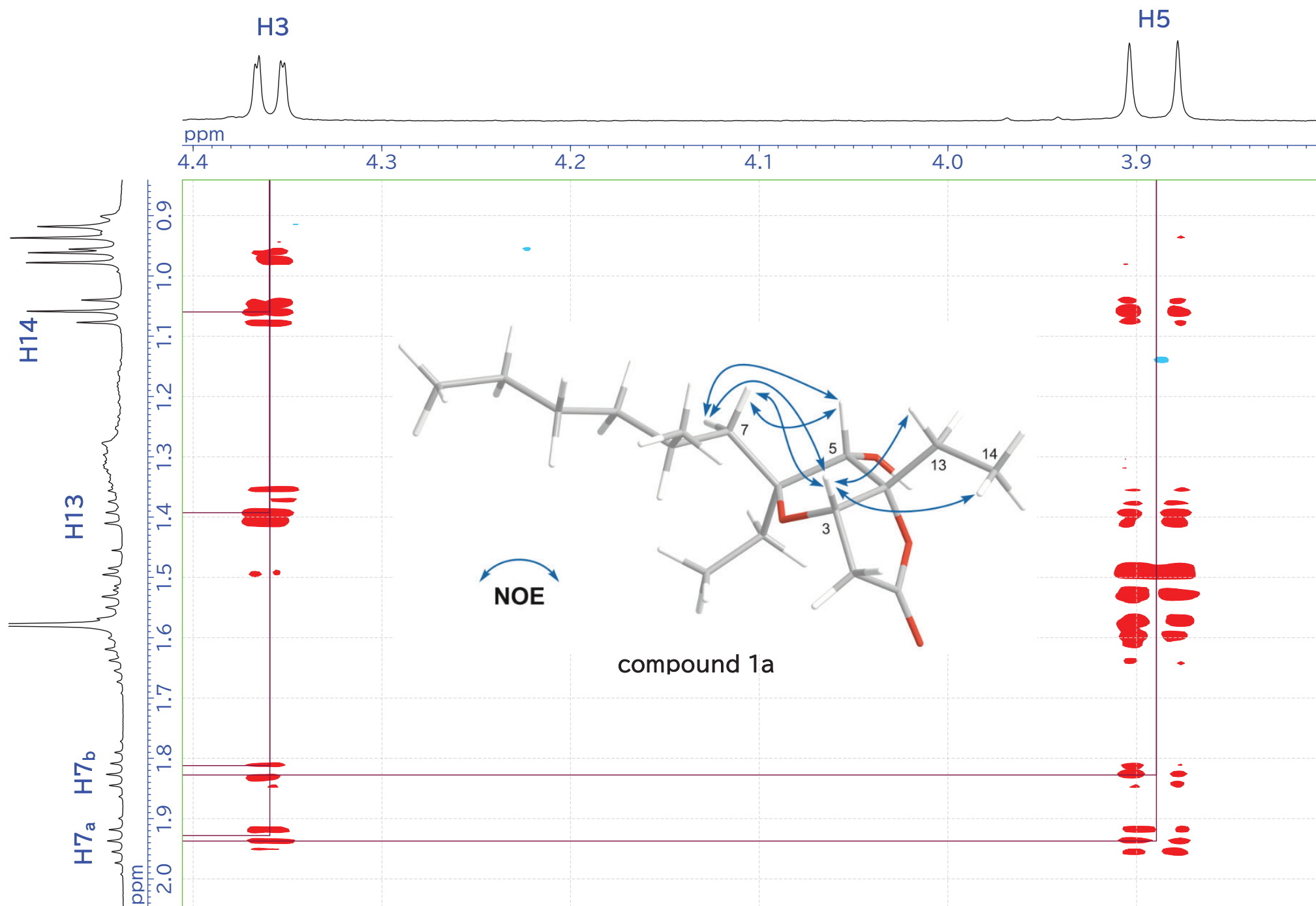
F2 - Acquisition Parameters  
 Date\_ 20211012  
 Time\_ 15.42  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 29761.904 Hz  
 FIDRES 0.454131 Hz  
 AQ 1.1010048 sec  
 RG 190.86  
 DW 16.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 125.7955112 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 88.00000000 W

==== CHANNEL f2 =====  
 SFO2 500.2320009 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 80.00 usec  
 PLW2 27.00000000 W  
 PLW12 0.51046997 W  
 PLW13 0.32670000 W



S66



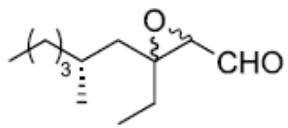
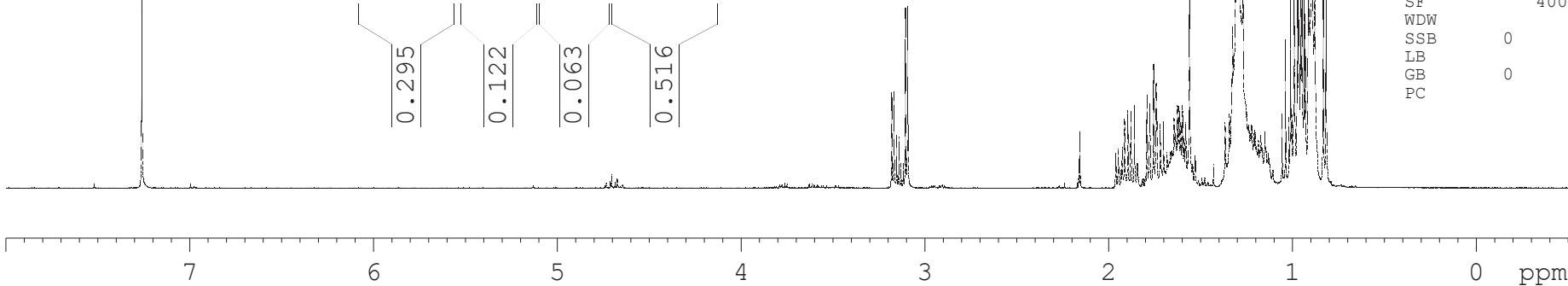
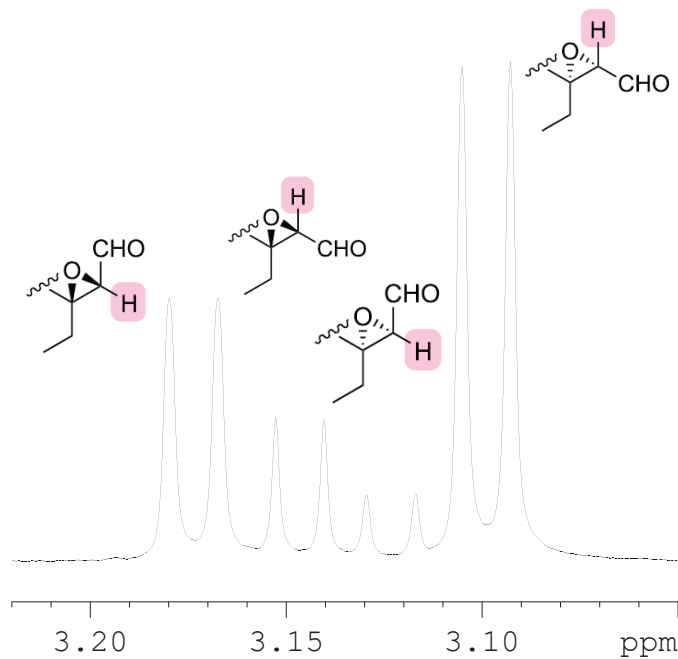
7.260  
3.180  
3.167  
3.105  
3.093  
1.913  
1.895  
1.878  
1.859  
1.791  
1.776  
1.755  
1.741  
1.736  
1.701  
1.644  
1.625  
1.617  
1.607  
1.598  
1.342  
1.330  
1.325  
1.310  
1.305  
1.300  
1.289  
1.281  
1.277  
1.275  
1.272  
1.268  
1.259  
1.247  
1.056  
1.037  
1.018  
1.010  
1.003  
0.991  
0.972  
0.964  
0.955  
0.948  
0.936  
0.930  
0.913  
0.910  
0.906  
0.896  
0.893  
0.878  
0.832  
0.816

Current Data Parameters  
NAME compound 7b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20211220  
Time\_ 19.14  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 101  
DW 62.400 usec  
DE 6.50 usec  
TE 295.9 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 9.22 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800099 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

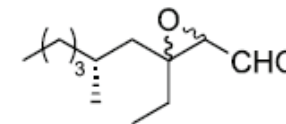


7b

S68

199.892  
199.654

77.314  
76.996  
76.679  
67.772  
67.489  
67.234  
64.558  
63.560  
63.303  
62.458  
41.510  
37.626  
37.147  
36.914  
36.790  
36.746  
30.039  
29.996  
29.476  
29.238  
29.122  
29.068  
27.853  
27.811



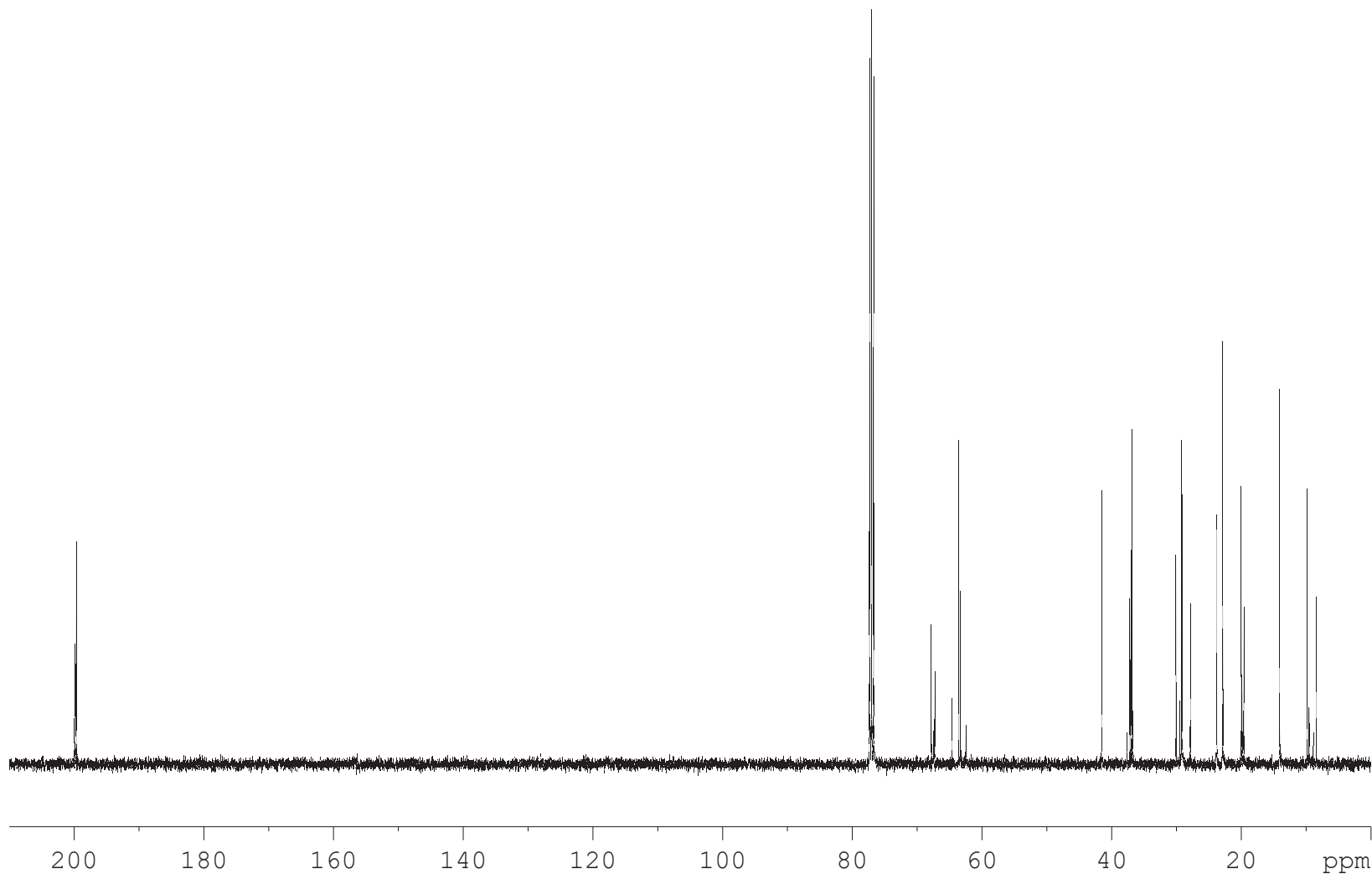
7b

Current Data Parameters  
NAME compound 7b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20211223  
Time\_ 18.39  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 297.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.17840999 W  
PLW13 0.08974000 W



S69

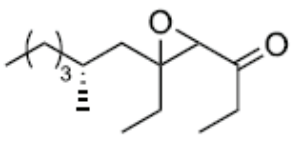
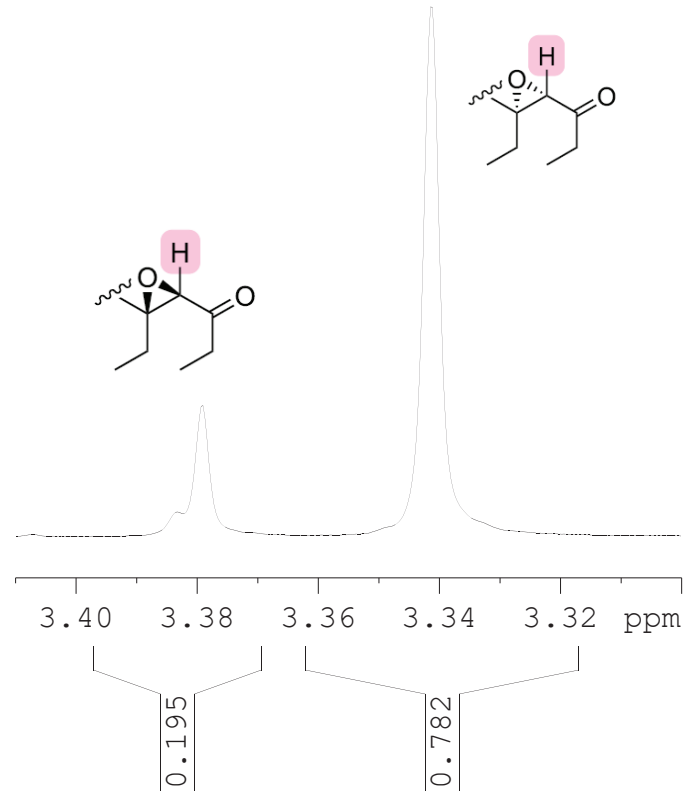
7.260  
3.379  
3.341  
2.611  
2.584  
2.566  
2.548  
2.534  
2.515  
2.489  
2.471  
1.735  
1.719  
1.700  
1.684  
1.624  
1.605  
1.588  
1.569  
1.549  
1.446  
1.427  
1.422  
1.410  
1.402  
1.392  
1.387  
1.367  
1.338  
1.334  
1.327  
1.320  
1.314  
1.307  
1.298  
1.292  
1.287  
1.277  
1.258  
1.119  
1.114  
1.101  
1.096  
1.082  
0.974  
0.965  
0.949  
0.945  
0.926  
0.918  
0.914  
0.907  
0.900  
0.883

Current Data Parameters  
 NAME compound 8b  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20211223  
 Time 19.48  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 114  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 295.7 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 =====  
 SFO1 400.1824713 MHz  
 NUC1 1H  
 P1 9.22 usec  
 PLW1 17.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1800099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



S70

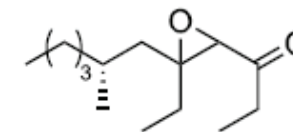
0.195  
0.782

2.000

0.172  
0.829  
2.686  
8.115  
3.220  
9.030

206.911  
206.857

77.316  
77.202  
76.999  
76.682  
66.592  
66.311  
65.608  
64.567  
41.503  
41.440  
37.084  
36.819  
34.273  
34.224  
29.686  
29.583  
29.143  
22.900  
22.867  
22.570



8b

Current Data Parameters  
NAME compound 8b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20211224  
Time\_ 11.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 296.7 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.17840999 W  
PLW13 0.08974000 W



S71

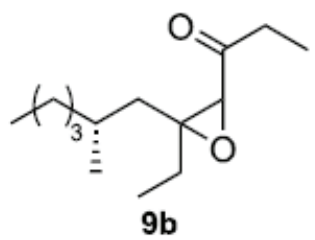
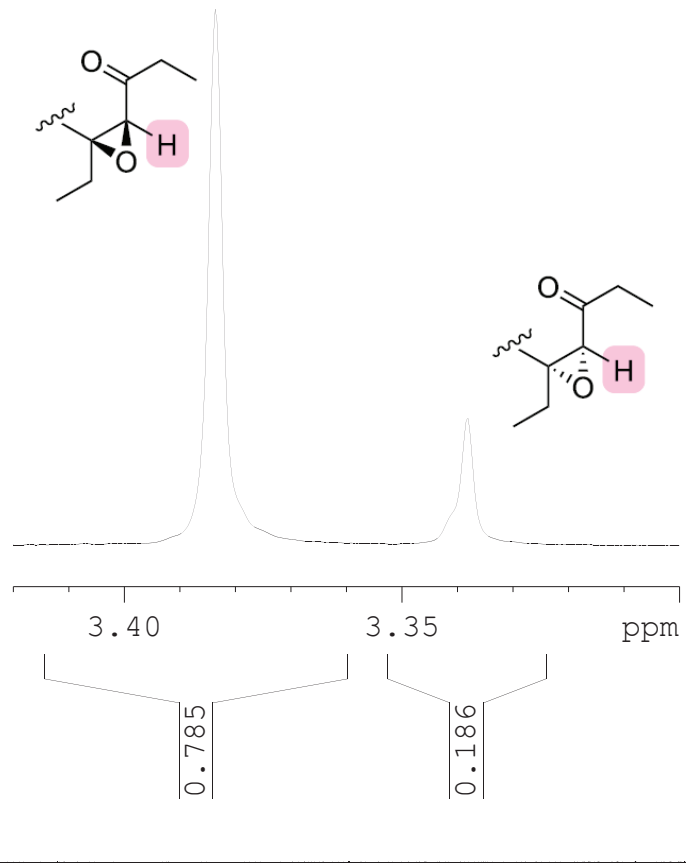
7.260  
3.384  
3.338  
2.587  
2.569  
2.550  
2.548  
2.529  
2.511  
1.742  
1.724  
1.705  
1.687  
1.670  
1.665  
1.651  
1.633  
1.622  
1.616  
1.554  
1.311  
1.307  
1.301  
1.296  
1.286  
1.283  
1.279  
1.272  
1.267  
1.261  
1.253  
1.251  
1.246  
1.238  
1.233  
1.222  
1.218  
1.212  
1.205  
1.202  
1.193  
1.111  
1.093  
1.075  
0.986  
0.967  
0.948  
0.902  
0.896  
0.885  
0.879  
0.867  
0.787  
0.771

Current Data Parameters  
 NAME compound 9b  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20211224  
 Time 12.35  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 114  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 295.6 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 400.1824713 MHz  
 NUC1 1H  
 P1 9.22 usec  
 PLW1 17.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1800099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



S72

0.785  
0.186

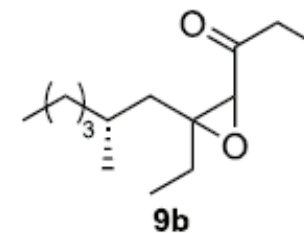
2.000

4.538  
6.693  
3.313  
3.593  
3.051  
2.412



— 206.928

77.313  
77.197  
76.995  
76.677  
66.531  
66.427  
63.783  
62.970  
36.907  
36.665  
36.038  
35.642  
34.416  
34.266  
29.630  
29.213  
29.038  
27.831  
27.759  
22.748

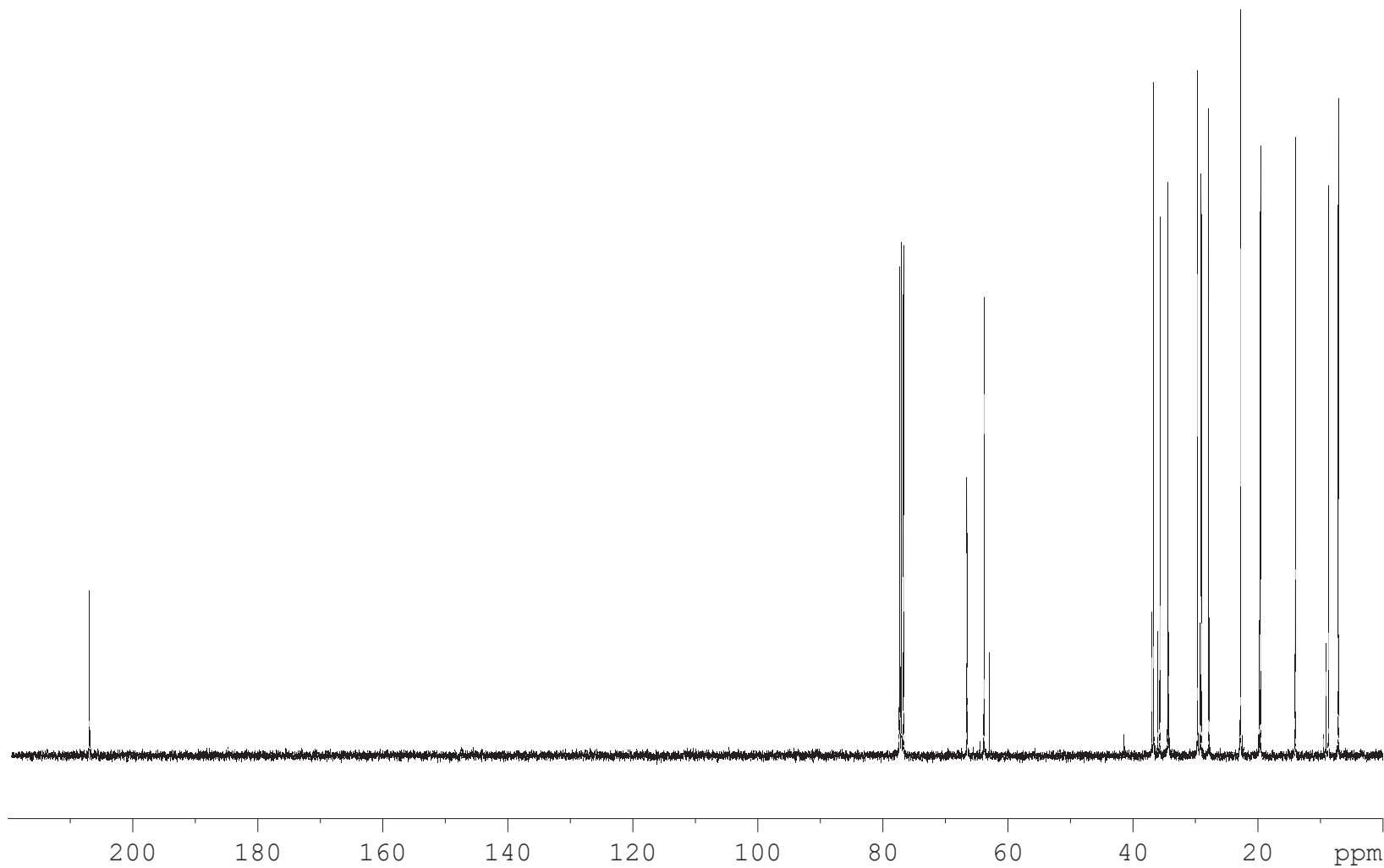


Current Data Parameters  
NAME compound 9b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20211224  
Time\_ 14.21  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 296.2 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.17840999 W  
PLW13 0.08974000 W



S73

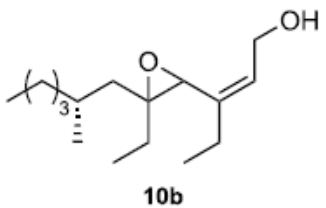
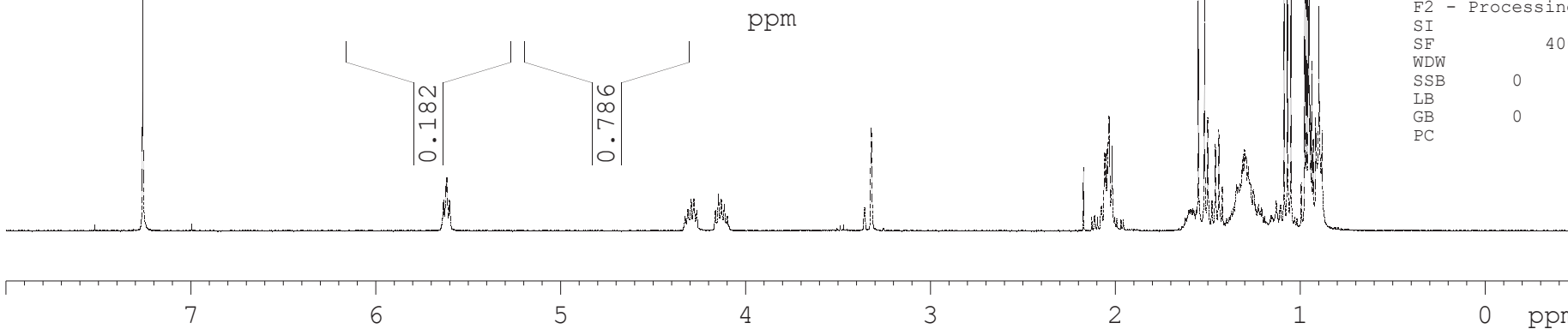
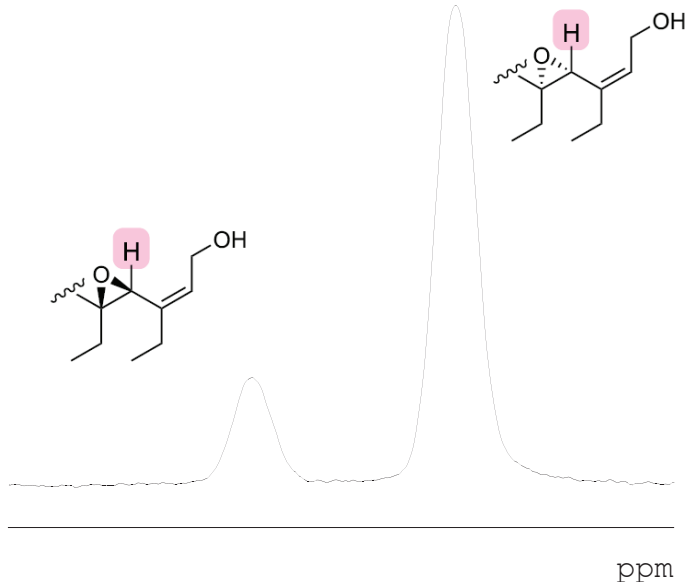
7.260  
5.632  
5.618  
5.615  
5.601  
4.295  
4.294  
4.279  
4.147  
4.145  
4.131  
3.319  
2.172  
2.056  
2.047  
2.037  
2.033  
2.030  
2.016  
1.551  
1.517  
1.500  
1.459  
1.456  
1.440  
1.438  
1.420  
1.342  
1.335  
1.330  
1.324  
1.315  
1.308  
1.300  
1.295  
1.291  
1.283  
1.279  
1.268  
1.252  
1.087  
1.069  
1.050  
0.994  
0.975  
0.967  
0.956  
0.950  
0.945  
0.937  
0.929  
0.916  
0.898  
0.881

Current Data Parameters  
NAME compound 10b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220217  
Time 15.29  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 181  
DW 62.400 usec  
DE 6.50 usec  
TE 296.3 K  
D1 1.00000000 sec  
TD0 1

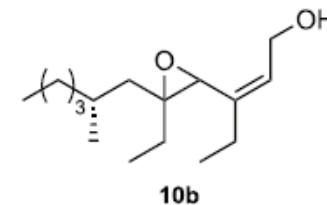
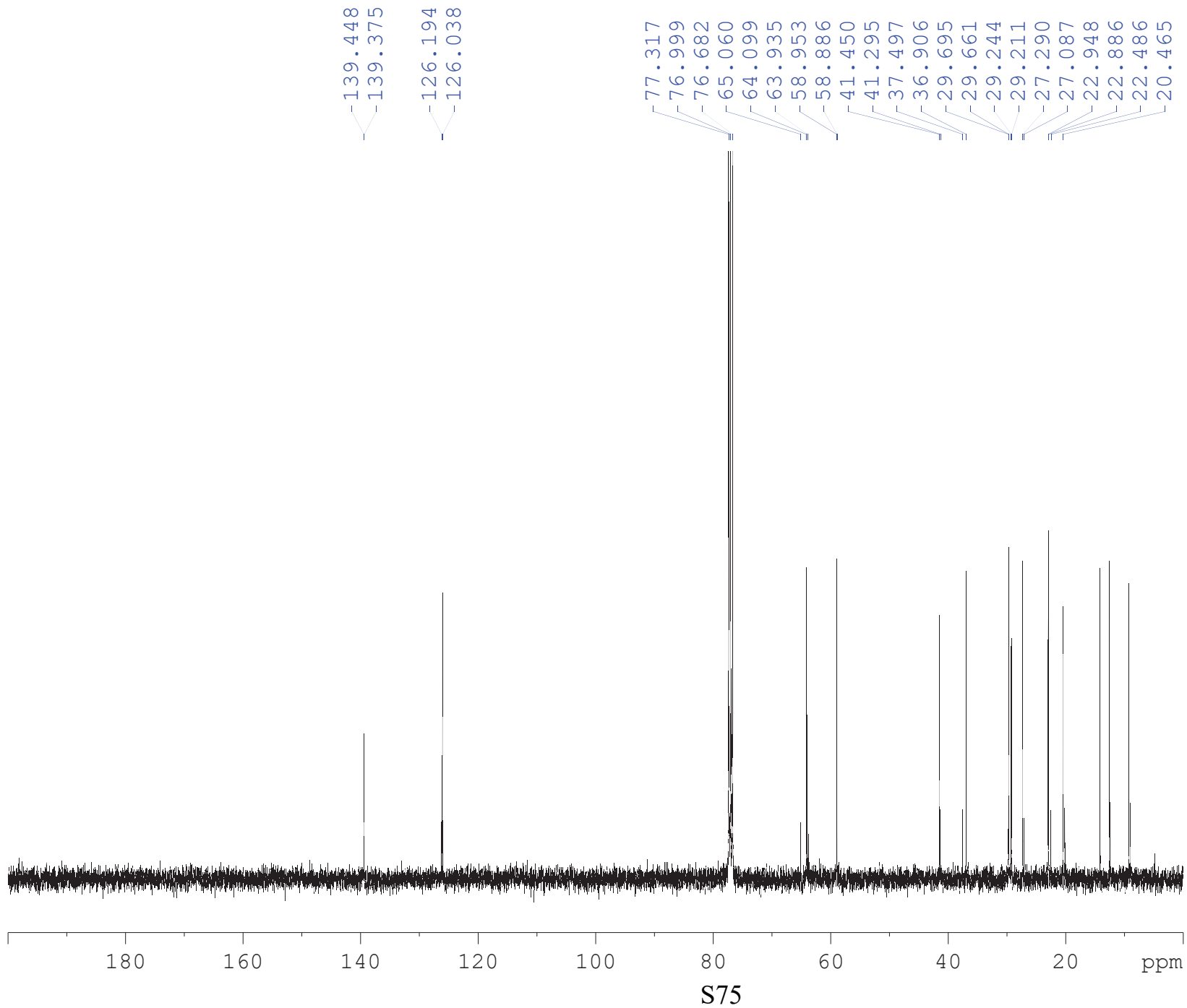
==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 9.22 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



1.000  
1.023  
1.032  
0.182  
0.786  
3.183  
2.269  
1.747  
1.717  
5.820  
3.299  
9.386

S74



Current Data Parameters  
 NAME compound 10b  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20220217  
 Time\_ 16.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 295.9 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

==== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.17840999 W  
 PLW13 0.08974000 W

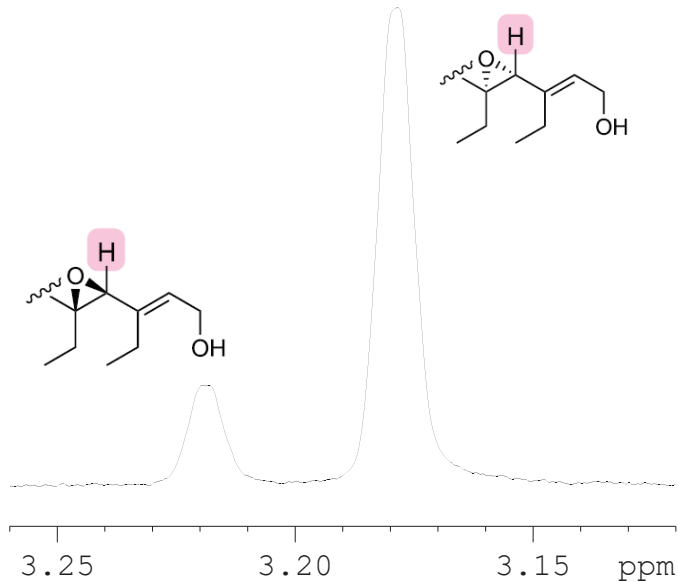
7.260  
5.542  
5.541  
5.539  
4.258  
4.254  
4.238  
4.227  
4.222  
3.178  
2.188  
2.172  
2.168  
2.149  
1.589  
1.538  
1.509  
1.492  
1.475  
1.462  
1.458  
1.446  
1.444  
1.426  
1.407  
1.356  
1.350  
1.344  
1.338  
1.324  
1.315  
1.307  
1.301  
1.295  
1.289  
1.278  
1.265  
1.251  
1.208  
1.170  
1.156  
1.142  
1.057  
1.038  
1.034  
1.018  
0.965  
0.949  
0.942  
0.923  
0.914  
0.904  
0.897  
0.880

Current Data Parameters  
NAME compound 11b  
EXPNO 1  
PROCNO 1

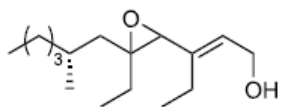
F2 - Acquisition Parameters  
Date\_ 20220225  
Time 11.58  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 128  
DW 62.400 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 9.22 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



7 6 5 4 3 2 1 0 ppm



11b

1.000

2.068

S76

0.173

0.834

2.087

0.198

3.869

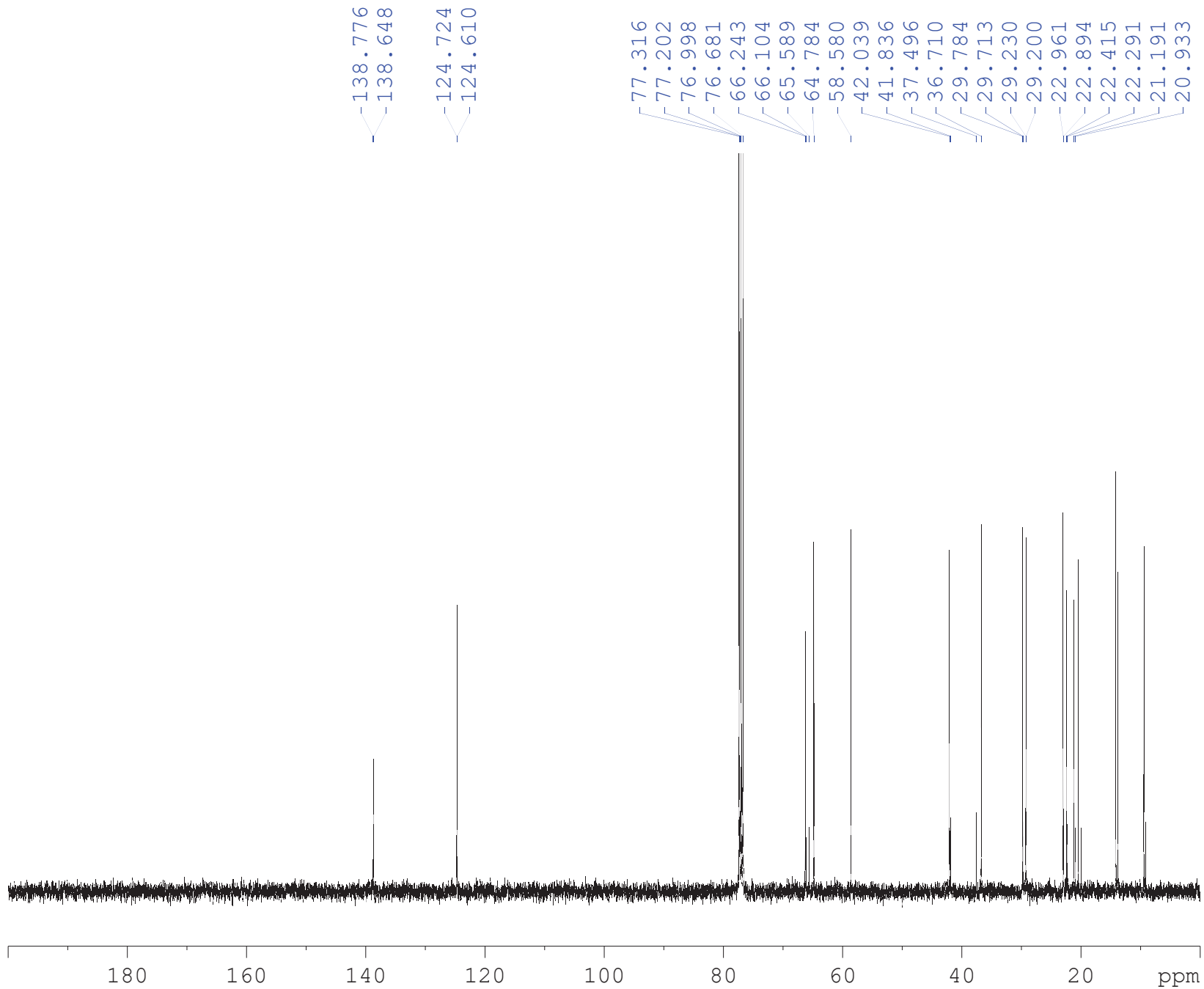
0.752

2.293

8.330

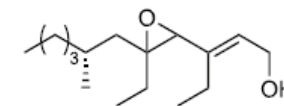
3.281

10.134



138.776  
138.648  
124.724  
124.610

77.316  
77.202  
76.998  
76.681  
66.243  
66.104  
65.589  
64.784  
58.580  
42.039  
41.836  
37.496  
36.710  
29.784  
29.713  
29.230  
29.200  
22.961  
22.894  
22.415  
22.291  
21.191  
20.933



**11b**

Current Data Parameters  
NAME compound 11b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220225  
Time\_ 12.16  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 296.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.00000000 W  
PLW12 0.17840999 W  
PLW13 0.08974000 W

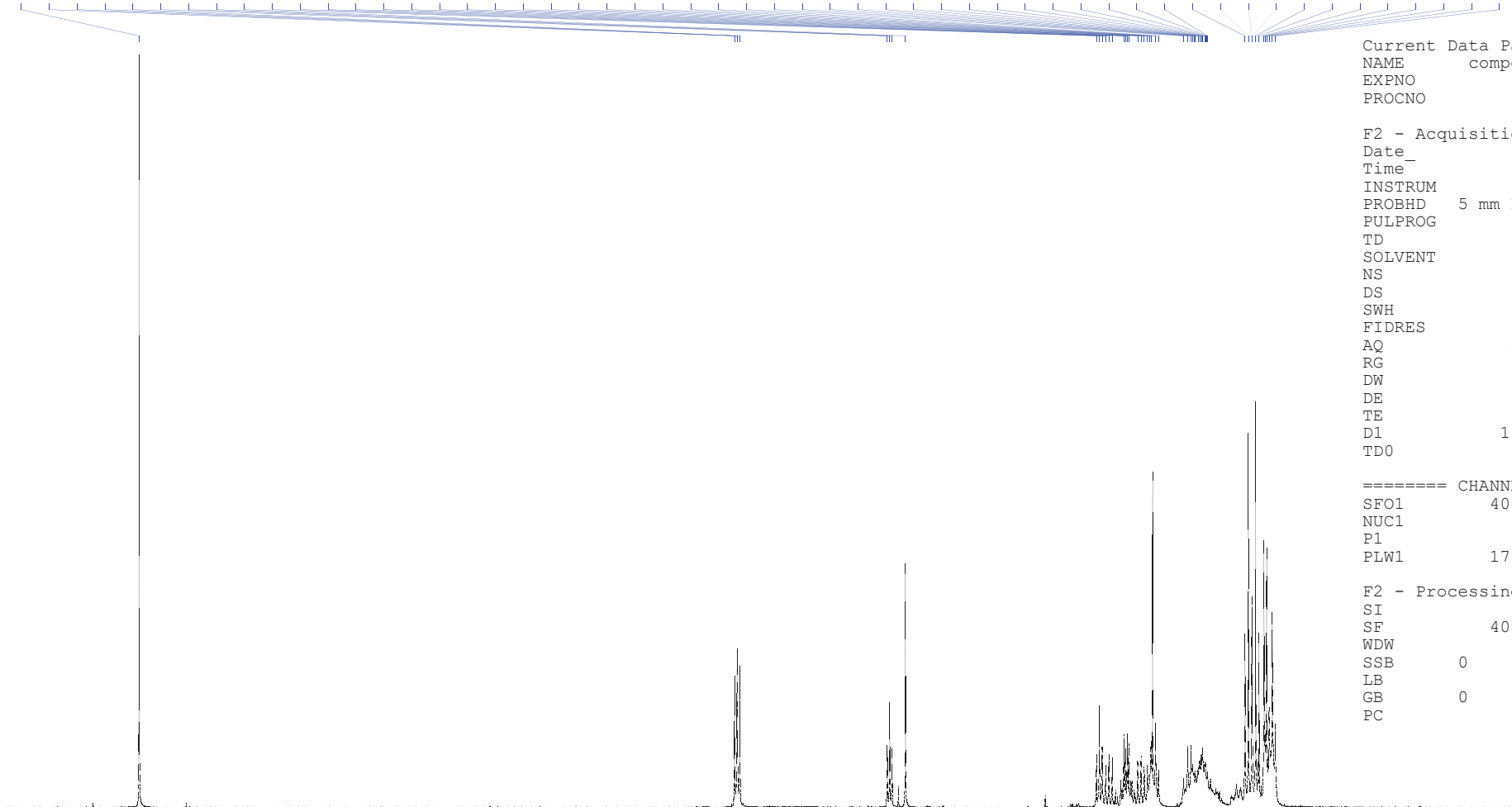
7.260  
3.916  
3.902  
3.899  
3.887  
3.059  
3.045  
3.032  
2.958  
1.884  
1.868  
1.852  
1.831  
1.814  
1.796  
1.730  
1.721  
1.711  
1.703  
1.653  
1.634  
1.617  
1.598  
1.585  
1.577  
1.553  
1.536  
1.395  
1.372  
1.354  
1.343  
1.337  
1.330  
1.314  
1.310  
1.304  
1.295  
1.290  
1.277  
1.273  
1.264  
1.261  
1.051  
1.032  
1.013  
1.009  
0.990  
0.971  
0.944  
0.937  
0.928  
0.915  
0.897  
0.880

Current Data Parameters  
NAME compound 12b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220118  
Time 18.04  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 144  
DW 62.400 usec  
DE 6.50 usec  
TE 295.8 K  
D1 1.00000000 sec  
TD0 1

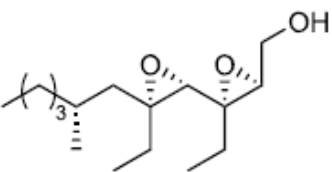
==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 9.22 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

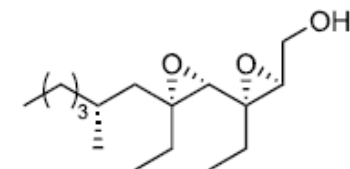
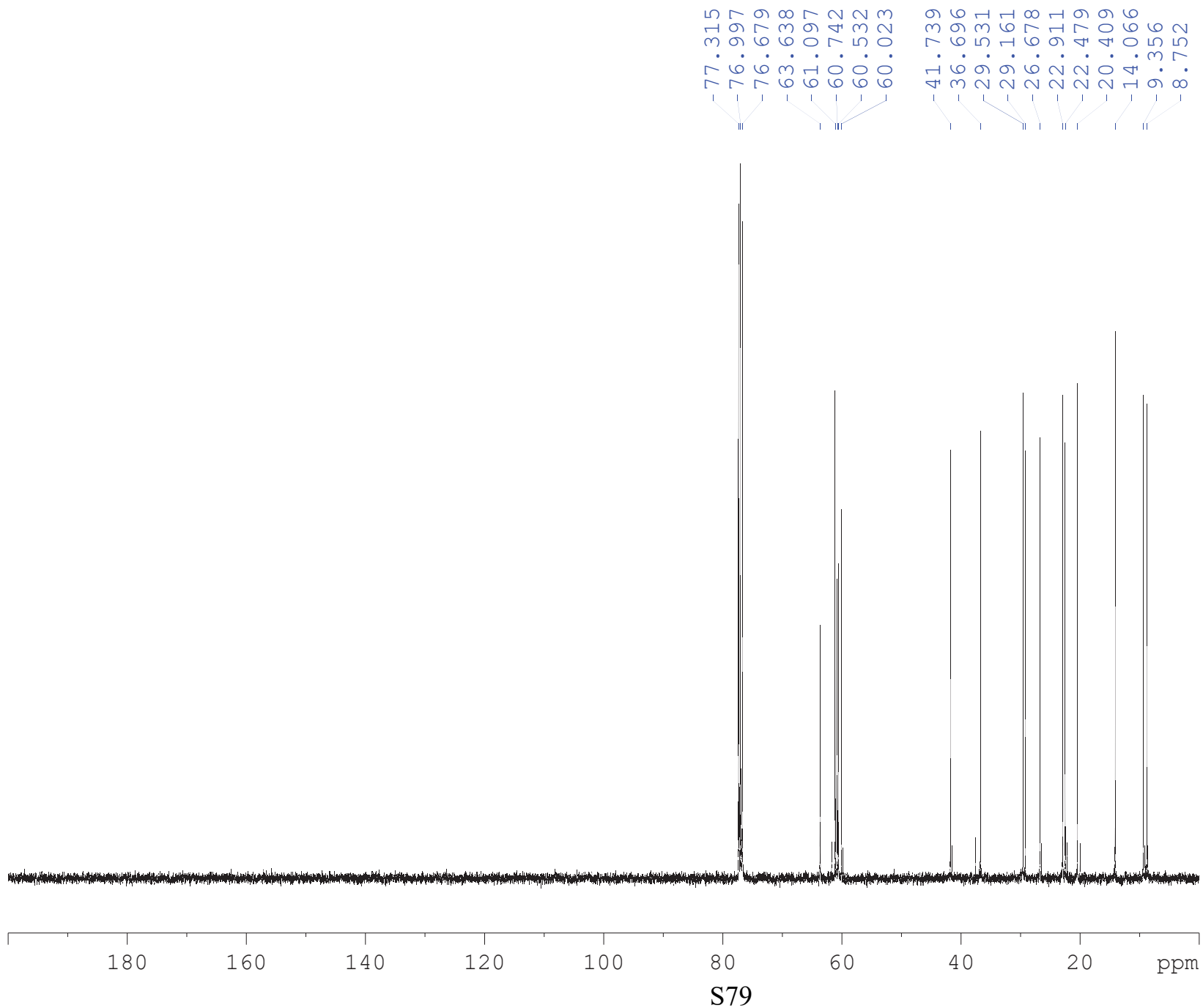


7  
6  
5  
4  
3  
2  
1  
0 ppm

2.092  
S78  
1.000  
0.926  
1.184  
0.771  
1.877  
4.190  
6.418  
12.778



12b



**12b**

Current Data Parameters  
 NAME compound 12b  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20220118  
 Time\_ 18.32  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 296.3 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

===== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.17840999 W  
 PLW13 0.08974000 W

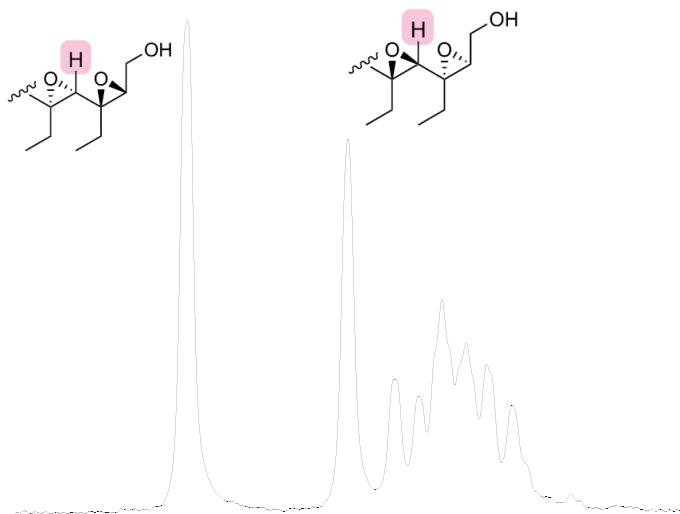
7.260  
3.962  
3.946  
3.930  
3.922  
3.907  
3.785  
3.780  
3.770  
3.767  
3.148  
3.100  
3.072  
3.064  
3.058  
2.085  
2.068  
2.051  
2.048  
2.031  
1.780  
1.731  
1.713  
1.694  
1.683  
1.679  
1.676  
1.530  
1.495  
1.477  
1.321  
1.307  
1.296  
1.287  
1.283  
1.273  
1.265  
1.255  
1.056  
1.054  
1.038  
1.035  
1.019  
1.017  
1.009  
0.990  
0.970  
0.951  
0.949  
0.932  
0.930  
0.913  
0.896  
0.879

Current Data Parameters  
NAME compound 13b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220301  
Time 15.00  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 128  
DW 62.400 usec  
DE 6.50 usec  
TE 295.5 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 9.22 usec  
PLW1 17.00000000 W

F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



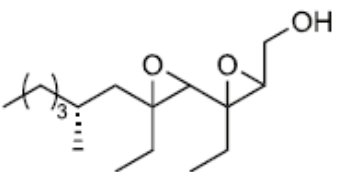
3.15 3.10 3.05 ppm

0.514

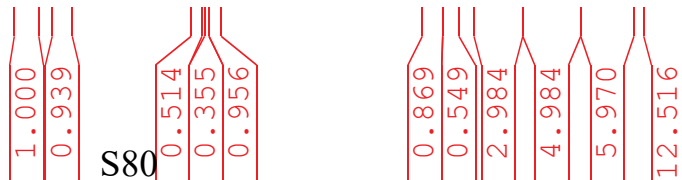
0.355

0.956

7 6 5 4 3 2 1 0 ppm



13b



S80

1.000

0.939

0.514

0.355

0.956

0.869

0.549

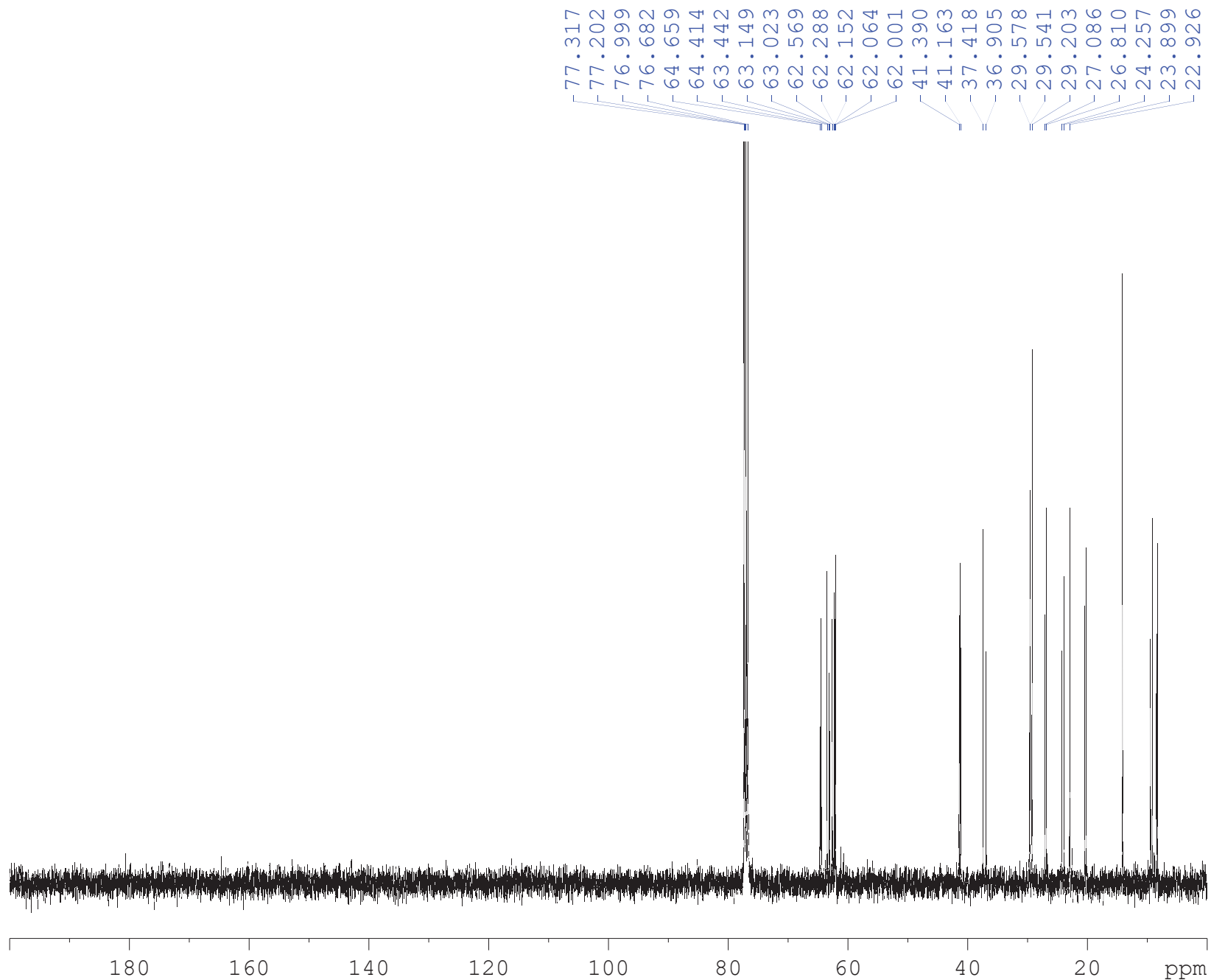
2.984

4.984

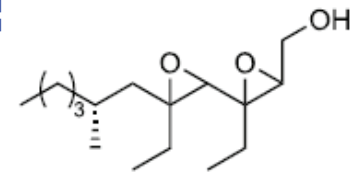
5.970

12.516





77.317  
77.202  
76.999  
76.682  
64.659  
64.414  
63.442  
63.149  
63.023  
62.569  
62.288  
62.152  
62.064  
62.001  
41.390  
41.163  
37.418  
36.905  
29.578  
29.541  
29.203  
27.086  
26.810  
24.257  
23.899  
22.926



**13b**

Current Data Parameters  
NAME compound 13b  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220301  
Time\_ 15.53  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 434  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 296.2 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6354031 MHz  
NUC1 13C  
P1 10.00 usec  
PLW1 70.0000000 W

==== CHANNEL f2 =====  
SFO2 400.1816007 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 17.0000000 W  
PLW12 0.17840999 W  
PLW13 0.08974000 W

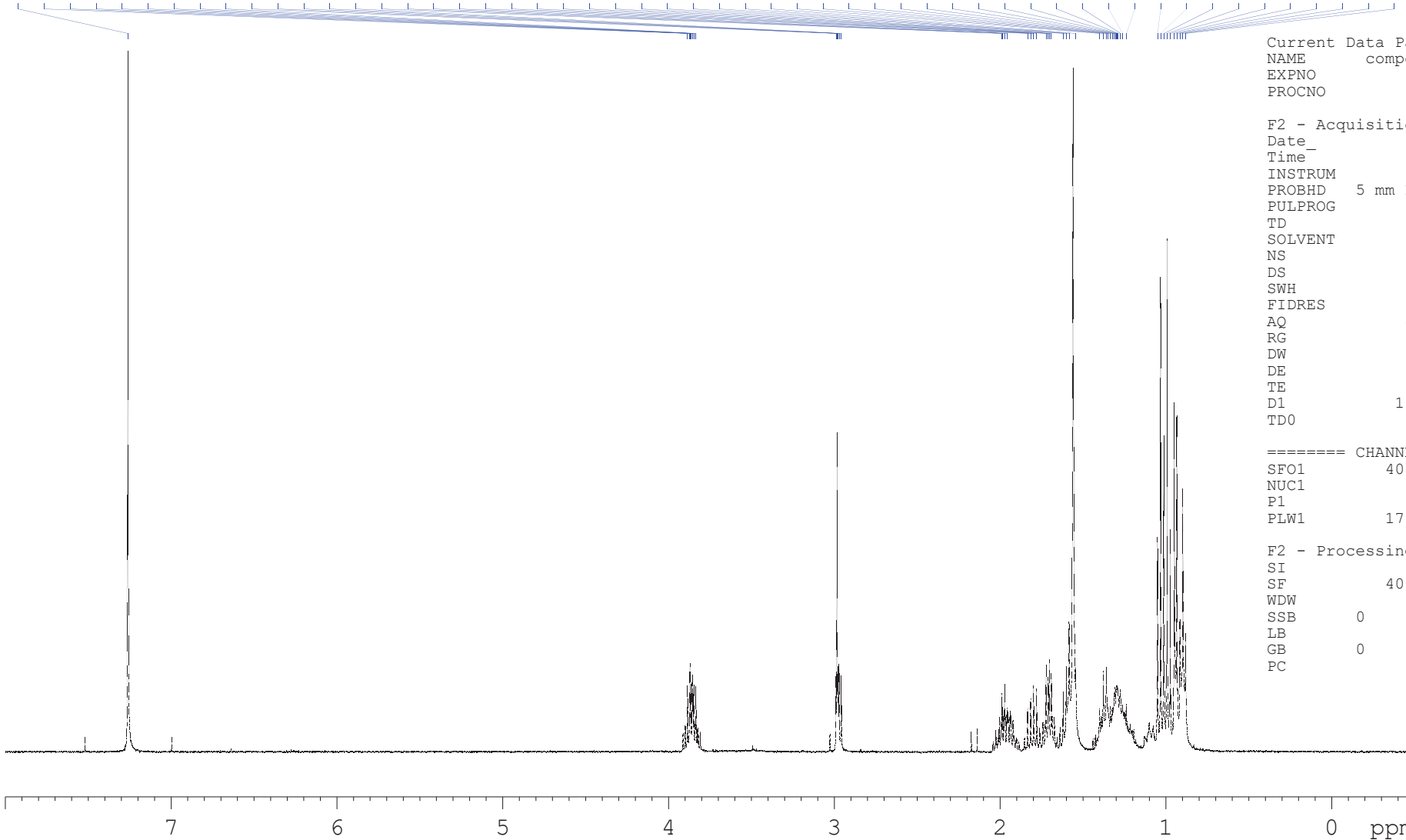
7.260  
3.885  
3.872  
3.870  
3.865  
3.856  
3.853  
3.847  
3.834  
2.989  
2.983  
2.977  
2.971  
2.958  
1.991  
1.985  
1.972  
1.956  
1.833  
1.815  
1.798  
1.779  
1.722  
1.709  
1.703  
1.690  
1.618  
1.599  
1.583  
1.543  
1.399  
1.376  
1.358  
1.348  
1.335  
1.323  
1.310  
1.305  
1.296  
1.292  
1.287  
1.274  
1.261  
1.237  
1.050  
1.031  
1.011  
0.991  
0.973  
0.949  
0.933  
0.915  
0.897  
0.880

Current Data Parameters  
NAME compound 14b  
EXPNO 1  
PROCNO 1

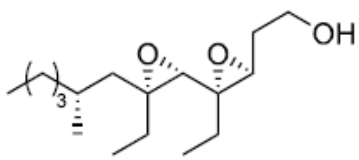
F2 - Acquisition Parameters  
Date\_ 20220216  
Time 13.28  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894465 sec  
RG 144  
DW 62.400 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 400.1824713 MHz  
NUC1 1H  
P1 9.22 usec  
PLW1 17.00000000 W

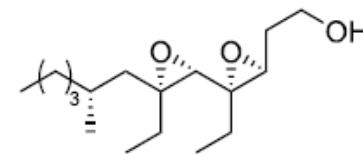
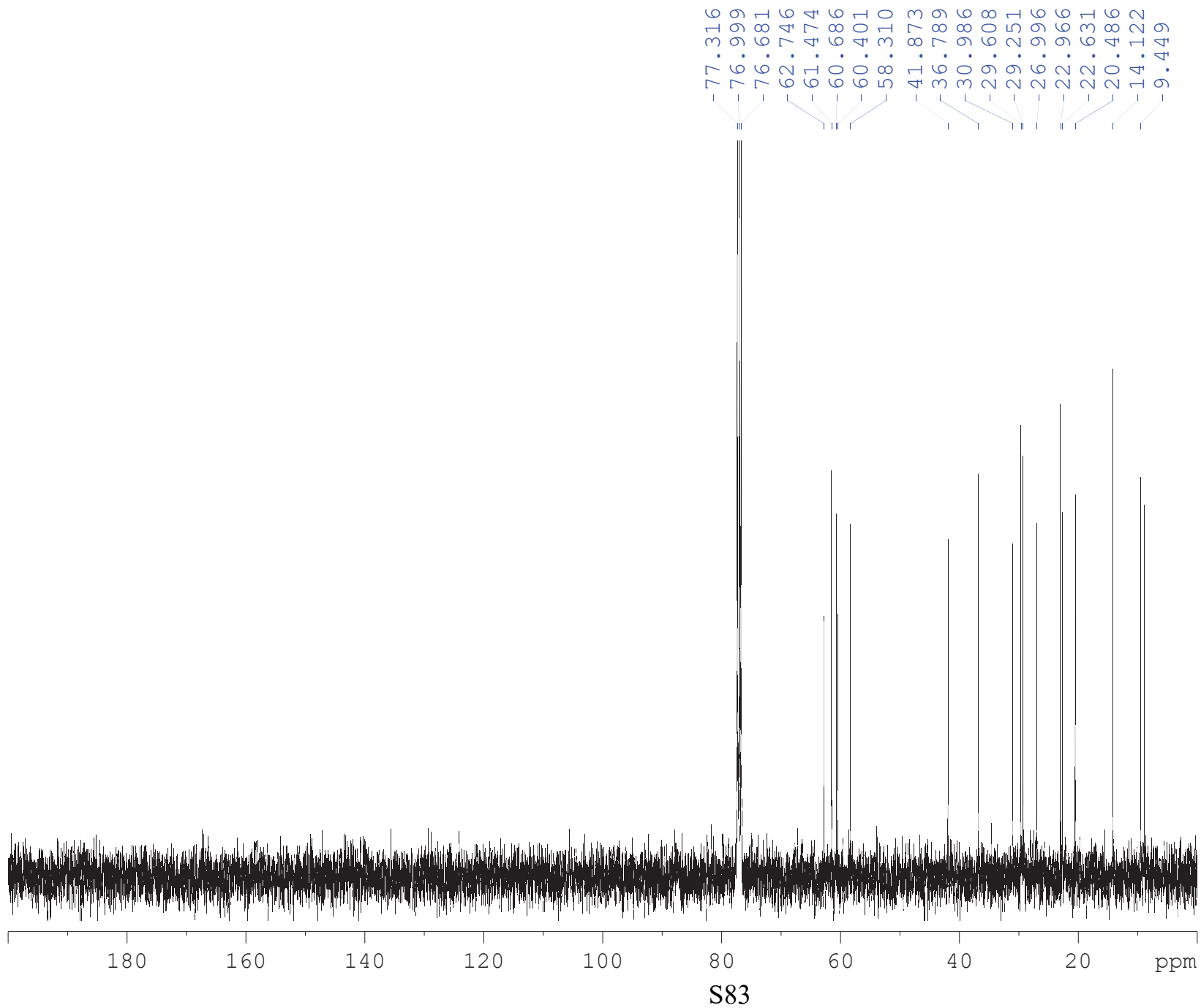
F2 - Processing parameters  
SI 65536  
SF 400.1800098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



2.000  
S82  
1.868  
2.026  
1.091  
1.793  
7.551  
6.548  
0.934  
12.314



14b



**14b**

Current Data Parameters  
 NAME compound 14b  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20220204  
 Time\_ 18.30  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 295.9 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 100.6354031 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 70.00000000 W

==== CHANNEL f2 =====  
 SFO2 400.1816007 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 17.00000000 W  
 PLW12 0.17840999 W  
 PLW13 0.08974000 W

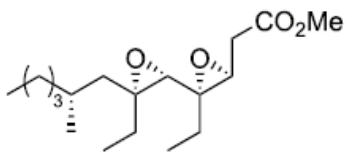
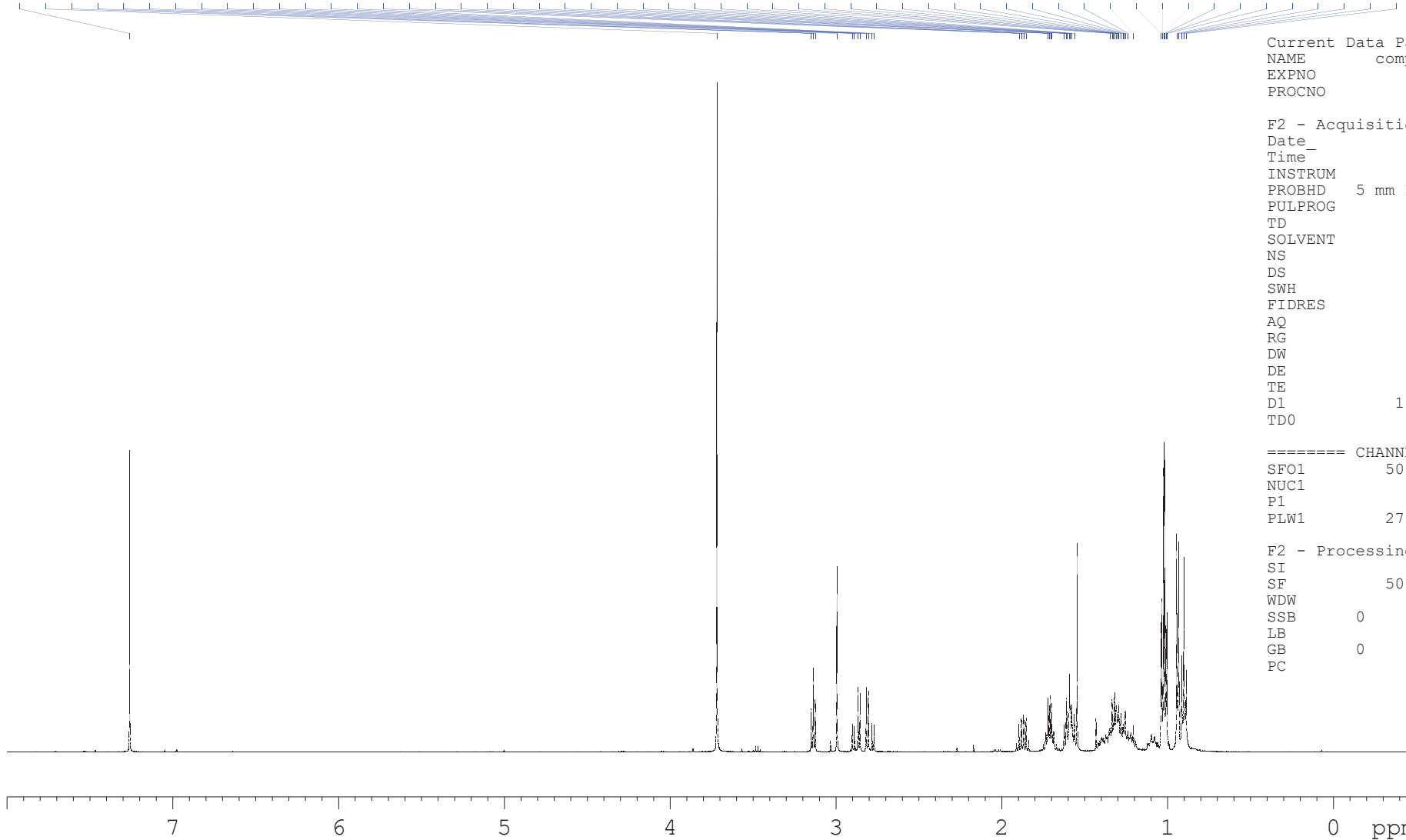
7.260  
3.717  
3.149  
3.136  
3.124  
2.993  
2.900  
2.888  
2.866  
2.854  
2.817  
2.804  
2.782  
2.769  
1.895  
1.881  
1.867  
1.852  
1.720  
1.713  
1.705  
1.698  
1.624  
1.610  
1.605  
1.590  
1.585  
1.577  
1.562  
1.346  
1.333  
1.326  
1.319  
1.308  
1.300  
1.297  
1.293  
1.279  
1.267  
1.265  
1.254  
1.237  
1.207  
1.038  
1.032  
1.023  
1.017  
1.008  
1.002  
0.944  
0.931  
0.914  
0.900  
0.886

Current Data Parameters  
 NAME compound 2b  
 EXPNO 10  
 PROCNO 1

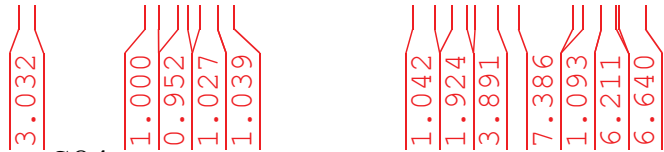
F2 - Acquisition Parameters  
 Date\_ 20220228  
 Time 12.36  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 190.86  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 500.2330891 MHz  
 NUC1 1H  
 P1 11.00 usec  
 PLW1 27.00000000 W

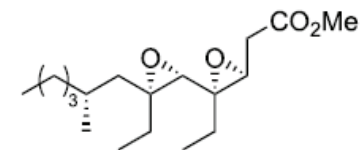
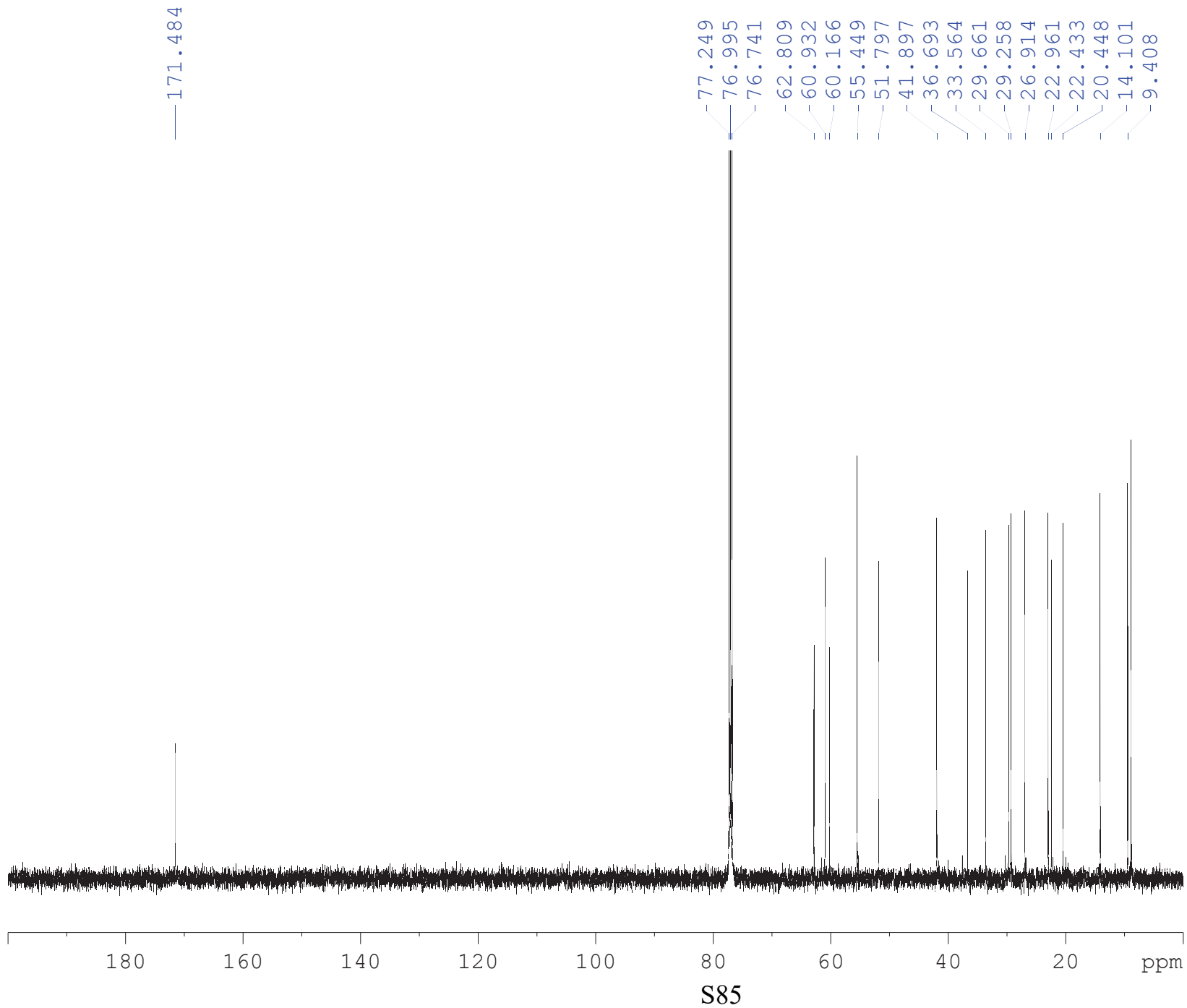
F2 - Processing parameters  
 SI 65536  
 SF 500.2300120 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



2b



S84



**2b**

Current Data Parameters  
 NAME compound 2b  
 EXPNO 10  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20220228  
 Time\_ 13.37  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 4  
 SWH 29761.904 Hz  
 FIDRES 0.454131 Hz  
 AQ 1.1010048 sec  
 RG 190.86  
 DW 16.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 125.7955112 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 88.00000000 W

==== CHANNEL f2 =====  
 SFO2 500.2320009 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 80.00 usec  
 PLW2 27.00000000 W  
 PLW12 0.51046997 W  
 PLW13 0.32670000 W

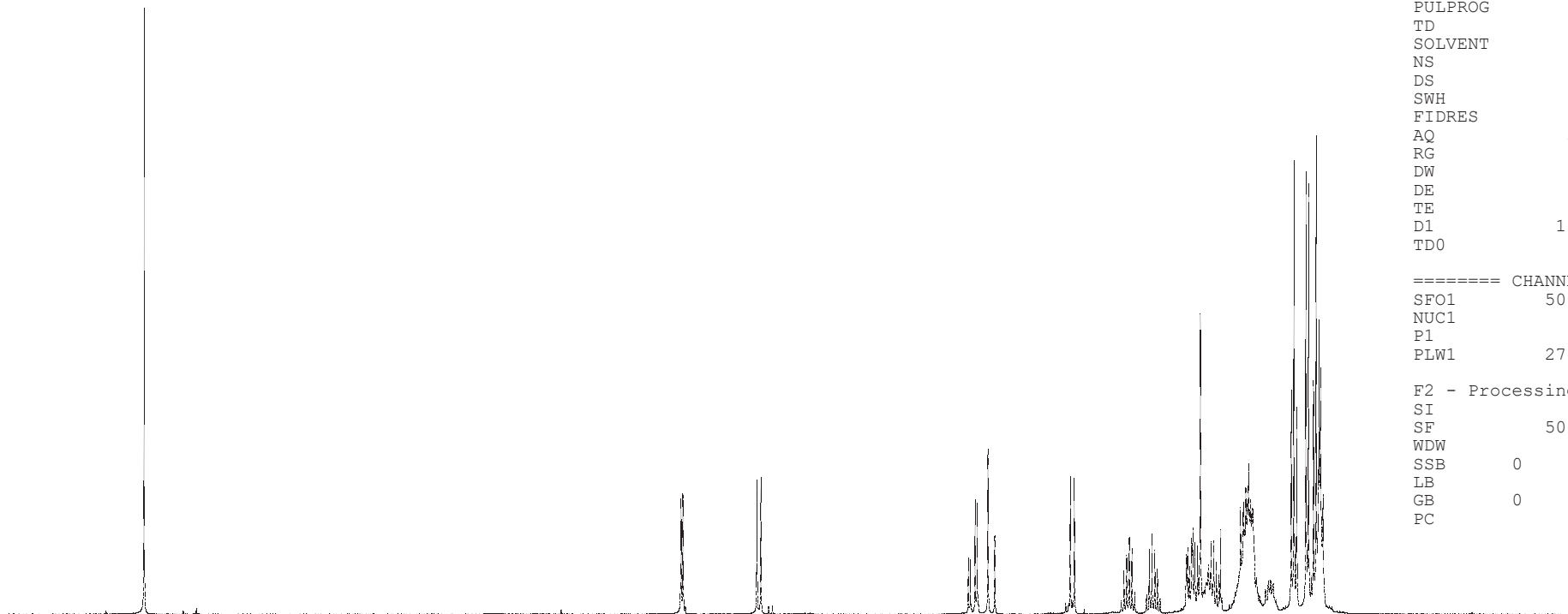
7.260  
4.353  
4.343  
3.942  
3.920  
2.797  
2.786  
2.760  
2.749  
2.691  
2.654  
2.245  
2.223  
1.940  
1.926  
1.911  
1.817  
1.802  
1.788  
1.617  
1.610  
1.589  
1.585  
1.581  
1.571  
1.556  
1.541  
1.483  
1.468  
1.454  
1.432  
1.323  
1.308  
1.300  
1.295  
1.289  
1.285  
1.279  
1.274  
1.268  
1.262  
1.258  
1.249  
1.049  
1.034  
1.019  
0.969  
0.955  
0.928  
0.913  
0.903  
0.899  
0.889  
0.876

Current Data Parameters  
NAME compound 1b  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220228  
Time 14.05  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 4  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 190.86  
DW 50.000 usec  
DE 6.50 usec  
TE 300.0 K  
D1 1.00000000 sec  
TD0 1

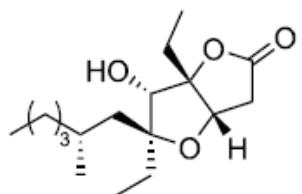
==== CHANNEL f1 =====  
SFO1 500.2330891 MHz  
NUC1 1H  
P1 11.00 usec  
PLW1 27.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.2300121 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

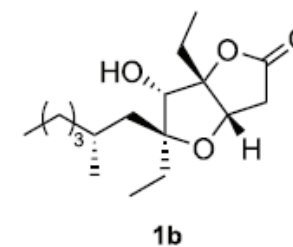
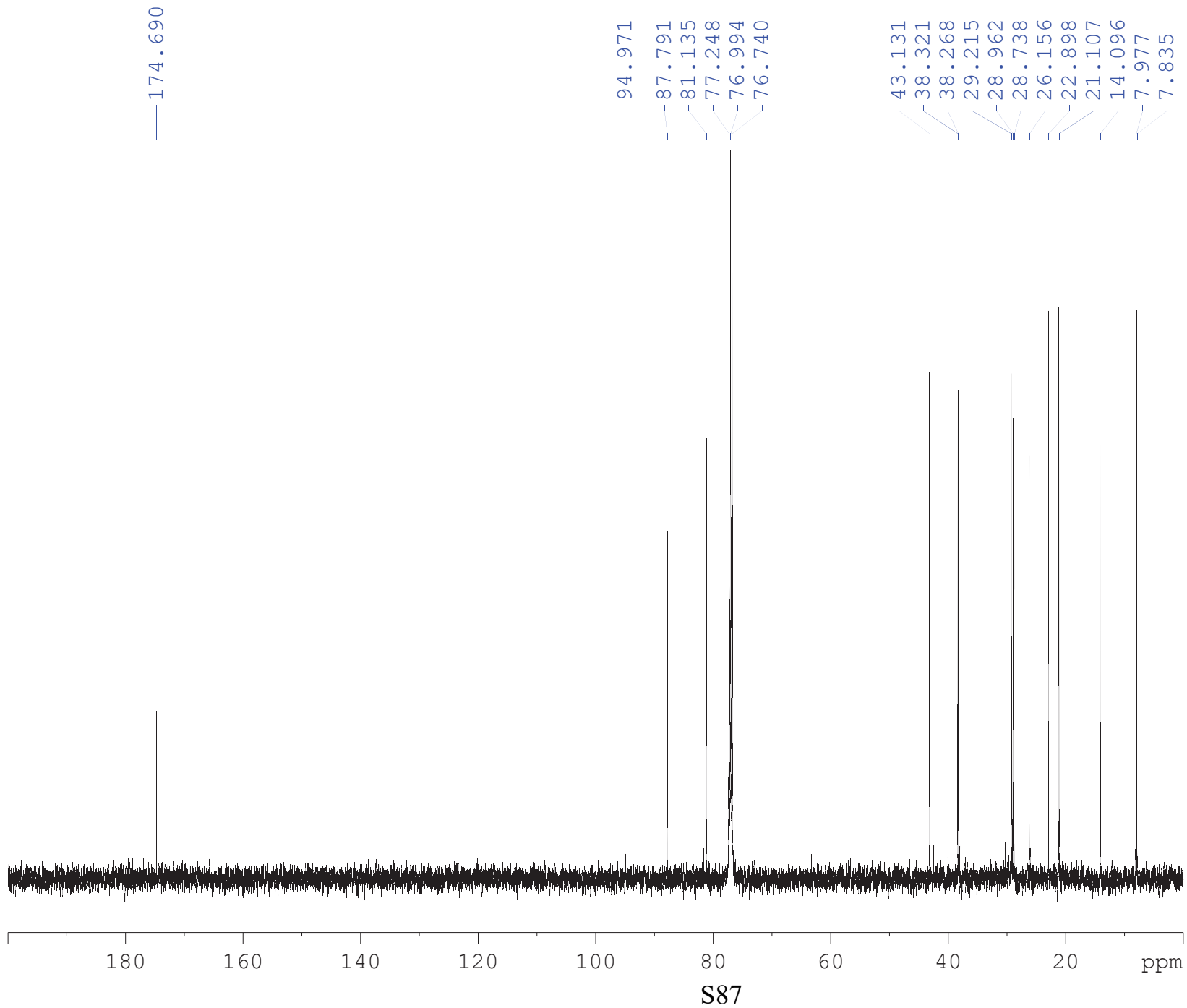


1.000  
0.998  
1.096  
1.048  
0.991  
1.112  
1.124  
6.241  
9.227  
3.218  
3.015  
6.410

S86



1b



Current Data Parameters  
 NAME compound 1b  
 EXPNO 10  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20220228  
 Time\_ 16.11  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1024  
 DS 4  
 SWH 29761.904 Hz  
 FIDRES 0.454131 Hz  
 AQ 1.1010048 sec  
 RG 190.86  
 DW 16.800 usec  
 DE 6.50 usec  
 TE 299.8 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 SFO1 125.7955112 MHz  
 NUC1 13C  
 P1 10.00 usec  
 PLW1 88.00000000 W

==== CHANNEL f2 =====  
 SFO2 500.2320009 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 80.00 usec  
 PLW2 27.00000000 W  
 PLW12 0.51046997 W  
 PLW13 0.32670000 W

