Supporting information

Toward the Stereochemical Assignment of Euvesperins A and B: Total Synthesis of the Possible Structures of the Natural Products

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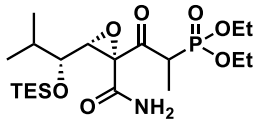
Materials and Methods: ^{1}H NMR spectra were measured at 300 MHz (JNM-AL300, JEOL), 400 MHz (JNM-AL400, ECZ400S, JEOL), or 400 MHz (JNM-ECZL400S, JEOL). Chemical shifts are expressed in ppm relative to tetramethylsilane (δ = 0) as an internal standard ($CDCl_3$ solution) or solvent resonance (C_5H_5N: δ = 8.73). Splitting patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; sept, septet; m, multiplet; br, broad peak. ^{13}C NMR spectra were measured at 100 MHz (JNM-AL400, JEOL), 100 MHz (JNM-ECZL400S, JEOL), or 125 HMz (JNM-ECA500, JEOL). ^{13}C spectra were calibrated from solvent resonance ($CDCl_3$: δ = 77.00, C_5H_5N: δ = 150.07). Infrared spectra (IR) were measured on an IR spectrometer (VALOR-III, JASCO) and are reported in wavenumbers (cm^{-1}). High-resolution mass spectra (HRMS) were obtained using a mass spectrometer (JMS 700, JEOL) with a direct inlet system. Optical rotations were measured on a polarimeter (P-2200, JASCO) using a 100 mm pathlength cell. Column chromatography was carried out on silica gel (40−100 mesh). Analytical thin-layer chromatography (TLC) was performed using 0.25 mm silica gel 60-F plates.

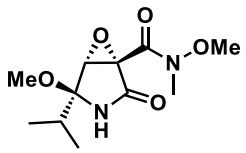
Diethyl[1-((2*R*,3*S*)-2-carbamoyl-3-{(*R*)-2-methyl-1-[(triethylsilyl)oxy]propyl}oxiran-2-yl)-1-oxopropan-2-yl]phosphonate (**19**)



To a stirred solution of diethyl ethylphosphonate (0.29 mL, 1.8 mmol) in THF (3.0 mL) was added *n*-BuLi (2.6 M solution in hexane, 0.84 mL, 1.7 mmol) dropwise at −45 °C. After stirring at −45 °C for 20 min, amide **20** (100 mg, 0.277 mmol) in THF (1.5 mL) was added. After stirring for 14 h, the reaction was quenched by addition of saturated aqueous NH_4Cl and the mixture was extracted with EtOAc (three times). The combined organic layers were washed with brine, dried over $MgSO_4$, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (EtOAc) to afford phosphonate **19** (95 mg, 74% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (6.85)* (br s, 1H), 5.55 (5.67)* (br s, 1H), 4.26-4.06 (m, 4H), 3.78 (4.06)* (dq, *J* = 12.0, 6.7 Hz, 1H), 3.62 (3.02)* (d, *J* = 8.0 Hz, 1H), 3.43 (3.49)* (dd, *J* = 8.0, 4.0 Hz, 1H), 1.86-1.69 (m, 1H), 1.42-1.23 (m, 9H), 0.98 (t, *J* = 8.0 Hz, 9H), 0.92 (d, *J* = 6.8 Hz, 3H), 0.89 (d, *J* = 6.8 Hz, 3H), 0.76-0.57 (m, 6H) (*The δ value of the minor isomer); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.2, 164.4 (166.4)*, 74.33 (74.27)*, 65.7 (65.8)*, 64.0 (64.5)*, 63.56 (63.62)*, 63.04 (63.09)*, 62.8 (62.7)*, 40.6 (41.1)*, 32.3 (32.2)*, 18.8 (18.9)*, 17.1 (16.7)*, 16.33 (16.38)*, 16.32 (16.26)*, 6.8 (3C), 4.96 (5.02)* (3C) (*The δ value of the minor isomer); IR ($CHCl_3$) 3439, 3320, 3195, 2958, 2876, 1713, 1697, 1389, 1243, 1055, 1021 cm^{-1}; $[\alpha]_D^{22}$ +7.1 (*c* 0.59, $CHCl_3$); FAB-HRMS *m/z* 466.2393 ($[M+H]^+$ calcd for $C_{20}H_{41}NO_7PSi$: 466.2390).

(1*S*,4*S*,5*R*)-4-Isopropyl-*N*,4-dimethoxy-*N*-methyl-2-oxo-6-oxa-3-azabicyclo[3.1.0]hexane-1-carboxamide (**22**)

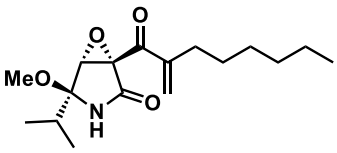


Dess–Martin periodinane (84.8 mg, 0.200 mmol) was added to a stirred solution of alcohol **21** (31.5 mg, 0.128 mmol) in CH_2Cl_2 (2.5 mL). After stirring for 1 h, MeOH (2.5 mL) was added to the reaction mixture and CH_2Cl_2 was removed under heating at 75 °C. Then, $CH(OMe)_3$ (0.24 mL, 2.2 mmol) and pyridinium *p*-toluenesulfonate (32.7 mg, 0.130 mmol) was added to the resultant mixture. After refluxing for 16 h, the reaction was quenched with Et_3N (0.08 mL, 0.6 mmol), and the mixture was concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 1:2) to afford amide **22** (30.5 mg, 92%) as a colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.61 (br s, 1H), 3.99 (d, *J* = 2.6 Hz, 1H), 3.78 (s, 3H), 3.34 (s, 3H), 3.28 (br s, 3H), 2.19-1.97 (m, 1H), 1.09 (d, *J* = 7.0 Hz, 3H), 0.99 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 162.4, 90.9, 61.9, 61.2, 59.8, 50.0, 33.0, 32.2, 17.3, 15.9; IR ($CHCl_3$) 3267, 2972,

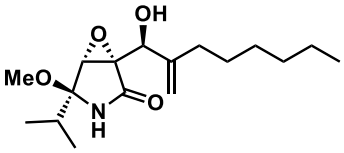
2943, 1720, 1672, 1460, 1427, 1389, 1097, 1065, 942 cm^{-1}; $[\alpha]_D^{24}$ –41.1 (*c* 0.61, $CHCl_3$); FAB-HRMS *m/z* 259.1293 ($[M+H]^+$ calcd for $C_{11}H_{19}N_2O_5$: 259.1294).

(1*R*,4*S*,5*R*)-4-Methoxy-1-(2-methylideneoctanoyl)-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**26**)



t-BuLi (1.65 M in pentane, 0.59 mL, 0.98 mmol) was added dropwise to a stirred solution of allyl iodide **25** (119 mg, 0.50 mmol) in THF (1.7 mL) at −78 °C. After stirring for 30 min, amide **22** (26.8 mg, 0.104 mmol) in THF (1.0 mL) was added. After stirring for 30 min, the reaction was quenched with saturated aqueous NH_4Cl. The mixture was extracted with EtOAc (3 times). The combined organic layers were washed with water and brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 4:1) to afford ketone **26** (28.1 mg, 88%) as a pale yellow oil.
^1H NMR (400 MHz, $CDCl_3$) δ 6.38 (s, 1H), 6.09 (s, 1H), 5.76 (br s, 1H), 3.89 (d, *J* = 2.2 Hz, 1H), 3.34 (s, 3H), 2.39-2.20 (m, 2H), 2.09 (sept, *J* = 6.8 Hz, 1H), 1.47-1.21 (m, 8H), 1.08 (d, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.8 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.7, 169.6, 146.9, 130.5, 90.6, 61.8, 61.7, 50.0, 33.0, 31.6, 30.4, 28.9, 28.0, 22.5, 17.3, 16.1, 14.0; IR ($CHCl_3$) 3230, 2958, 2930, 2872, 2858, 1724, 1667, 1468, 1416, 1389, 1368, 1317, 1098, 1069, 1039, 950 cm^{-1}; $[\alpha]_D^{23}$ –137.9 (*c* 0.27, $CHCl_3$); FAB-HRMS *m/z* 332.1840 ($[M+Na]^+$ calcd for $C_{17}H_{27}NO_4Na$: 332.1838).

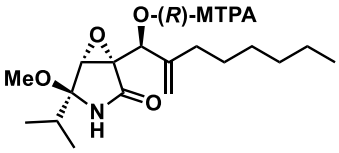
(1*S*,4*S*,5*R*)-1-{(*S*)-1-Hydroxy-2-methylideneoctanoyl}-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(6*S*)-**28**}



$NaBH_4$ (9.8 mg, 0.26 mmol) was added a stirred solution of ketone **26** (48.9 mg, 0.158 mmol) in MeOH (4.0 mL) at −78 °C. After stirring for 13 h, the reaction mixture was concentrated *in vacuo*. The residue was diluted with EtOAc and washed with water. The organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 3:2) to afford alcohol (6*S*)-**28** (37.2 mg, 76%) as a colorless oil.
^1H NMR (400 MHz, $CDCl_3$) δ 5.61 (br s, 1H), 5.22 (s, 1H), 5.04 (s, 1H), 4.82 (d, *J* = 2.9 Hz, 1H), 3.72 (d, *J* = 2.9 Hz, 1H), 3.22 (s, 3H), 2.79 (d, *J* = 2.9 Hz, 1H), 2.24-2.13 (m, 1H), 2.12-2.00 (m, 2H), 1.53-1.43 (m, 2H), 1.39-1.22 (m, 6H), 1.07 (d, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.7, 146.2, 112.8, 90.3, 68.6, 61.8, 60.2, 49.7, 33.0, 32.3, 31.7,

29.1, 27.8, 22.6, 17.4, 15.9, 14.1; IR ($CHCl_3$) 3238, 2958, 2929, 2873, 2858, 1716, 1652, 1468, 1457, 1418, 1388, 1366, 1098, 1069, 1026, 951 cm^{-1}; $[\alpha]_D^{25}$ –36.1 (*c* 0.43, $CHCl_3$); FAB-HRMS *m/z* 312.2176 ($[M+H]^+$ calcd for $C_{17}H_{30}NO_4$: 312.2175).

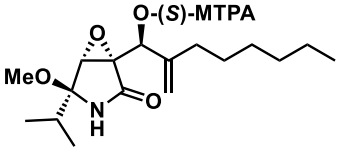
Mosher's ester (**S1**)



(*S*)-MTPACl (2 drops) and 4-DMAP (cat.) were added to a stirred solution of alcohol (6*S*)-**28** (2.0 mg, 6.4 μmol) in pyridine (0.08 mL) at room temperature. After stirring for 22 h, the mixture was diluted with EtOAc and washed with saturated aqueous NH_4Cl (2 times). The organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 4:1) to afford ester **S1** (3.0 mg, 88%) as a colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57-7.50 (m, 2H), 7.43-7.35 (m, 3H), 6.11 (s, 1H), 5.49 (br s, 1H), 5.33 (s, 1H), 5.17 (br s, 1H), 3.55 (s, 3H), 3.54 (d, *J* = 1.7 Hz, 1H), 3.15 (s, 3H), 2.21-2.12 (m, 2H), 2.03 (sept, *J* = 7.0 Hz, 1H), 1.36-1.19 (m, 8H), 1.00 (d, *J* = 7.0 Hz, 3H), 0.93 (d, *J* = 7.0 Hz, 3H), 0.88 (t, *J* = 6.5 Hz, 3H).

Mosher's ester (**S2**)



S2 (1.4 mg, 82% yield, colorless oil) was prepared from (6*S*)-**28** (1.0 mg, 3.2 μmol) and (*R*)-MTPACl by a procedure similar to that for **S1**.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60-7.50 (m, 2H), 7.44-7.34 (m, 3H), 6.04 (s, 1H), 5.59 (br s, 1H), 5.11 (s, 1H), 5.05 (s, 1H), 3.67 (d, *J* = 2.7 Hz, 1H), 3.58 (br s, 3H), 3.17 (s, 3H), 2.17-1.93 (m, 2H), 2.07 (sept, *J* = 6.9 Hz, 1H), 1.51-1.16 (m, 8H), 1.04 (d, *J* = 6.9 Hz, 3H), 0.96 (d, *J* = 6.9 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H).

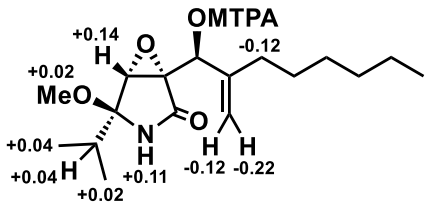
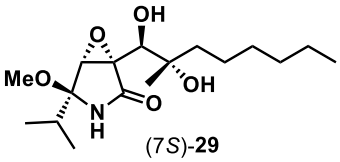
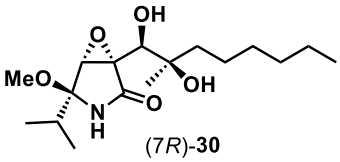


Fig. S1 Modified Mosher's method: differences of the chemical shifts ($\Delta\delta = \delta_S - \delta_R$, $CDCl_3$) between (*R*)- and (*S*)-MTPA esters of (6*S*)-**28**.

(1*S*,4*S*,5*R*)-1-((1*S*,2*S*)-1,2-Dihydroxy-2-methyloctyl)-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(7*S*)-**29**)} and (1*S*,4*S*,5*R*)-1-((1*S*,2*R*)-1,2-dihydroxy-2-methyloctyl))-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(7*R*)-**30**)}



(7*S*)-**29**



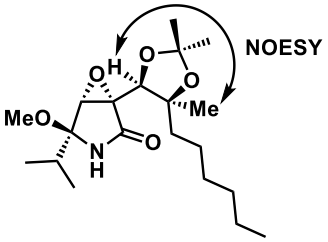
(7*R*)-**30**

$Co(acac)_2$ (20 mg, 0.077 mmol) and $PhSiH_3$ (0.19 mL, 1.6 mmol) were added to a stirred solution of alcohol (6*S*)-**28** (120 mg, 0.385 mmol) in 2,2,2-trifluoroethanol (8.0 mL). After stirring for 1 h under an O_2 atmosphere, the mixture was diluted with EtOAc and washed with saturated aqueous $Na_2S_2O_3$, water, and brine. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 3:1) to afford diol (7*S*)-**29** (35.1 mg, 28%) and (7*R*)-**30** (45.2 mg, 36%) as a colorless oil.

(7*S*)-**29**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.74 (br s, 1H), 4.90 (s, 1H), 3.68 (d, *J* = 2.8 Hz, 1H), 3.37 (br d, *J* = 9.2 Hz, 1H), 3.36 (s, 3H), 3.26 (br d, *J* = 9.2 Hz, 1H), 2.05 (sept, *J* = 7.0 Hz, 1H), 1.73-1.18 (m, 10H), 1.23 (s, 3H), 1.09 (d, *J* = 6.8 Hz, 3H), 0.96 (d, *J* = 6.8 Hz, 3H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 90.7, 77.2, 73.8, 61.1, 60.6, 50.1, 38.4, 33.3, 31.8, 29.9, 23.3, 22.7, 22.6, 17.3, 15.7, 14.1; IR ($CHCl_3$) 3255, 2937, 2872, 2858, 1693, 1467, 1458, 1430, 1389, 1376, 1134, 1095, 1064, 938, 852 cm^{-1}; $[\alpha]_D^{23}$ –149.3 (*c* 0.27, $CHCl_3$); FAB-HRMS *m/z* 330.2282 ($[M+H]^+$ calcd for $C_{17}H_{32}NO_5$: 330.2280).

(7*R*)-**30**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.80 (br s, 1H), 5.11 (s, 1H), 3.70 (d, *J* = 2.8 Hz, 1H), 3.36 (s, 3H), 3.32 (d, *J* = 9.2 Hz, 1H), 3.29 (d, *J* = 9.2 Hz, 1H), 2.05 (sept, *J* = 6.8 Hz, 1H), 1.39-1.20 (m, 10H), 1.34 (s, 3H), 1.08 (d, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.8 H, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 90.7, 74.7, 73.2, 61.2, 60.8, 50.1, 38.7, 33.3, 31.8, 29.7, 24.0, 22.9, 22.6, 17.3, 15.8, 14.1; IR ($CHCl_3$) 3249, 2931, 2858, 2176, 1693, 1468, 1430, 1384, 1132, 1094, 1064, 934, 849 cm^{-1}; $[\alpha]_D^{25}$ –124.3 (*c* 0.07, $CHCl_3$); FAB-HRMS *m/z* 330.2284 ($[M+H]^+$ calcd for $C_{17}H_{32}NO_5$: 330.2280).

(1*S*,4*S*,5*R*)-1-((5*S*)-5-Hexyl-2,2,5-trimethyl-1,3-dioxolan-4-yl)-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**S3**)

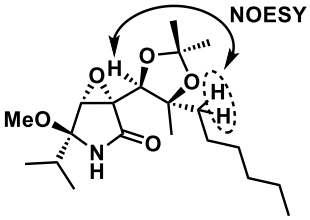


2,2-Dimethoxy propane (0.06 mL, 0.2 mmol) and *p*-TsOH (catalytic amount) were added to a stirred solution of (7*S*)-**29** (3.8 mg, 0.012 mmol) in CH_2Cl_2 (0.2 mL) at room temperature. After stirring for

22 h, the mixture was diluted with Et_2O and washed with saturated aqueous $NaHCO_3$, water, and brine. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 4:1) to afford acetonide **S3** (1.4 mg, 33 %) as a yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.68 (br s, 1H), 4.51 (s, 1H), 4.08 (d, *J* = 2.7 Hz, 1H), 3.28 (s, 3H), 2.16 (sept, *J* = 6.6 Hz, 1H), 1.54-1.16 (m, 10H), 1.49 (s, 3H), 1.42 (s, 3H), 1.39 (s, 3H), 1.07 (d, *J* = 6.6 Hz, 3H), 1.06 (d, *J* = 6.6 Hz, 3H), 0.87 (t, *J* = 6.7 Hz, 3H).

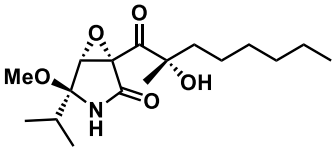
(1*S*,4*S*,5*R*)-1-((5*R*)-5-Hexyl-2,2,5-trimethyl-1,3-dioxolan-4-yl)-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**S4**)



S4 (1.5 mg, 68% yield, yellow oil) was prepared from (7*R*)-**30** (2.0 mg, 6.1 μmol) by a procedure similar to that for **S3**.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.78 (br s, 1H), 4.47 (s, 1H), 4.04 (d, *J* = 2.7 Hz, 1H), 3.28 (s, 3H), 2.15 (sept, *J* = 6.9 Hz, 1H), 1.91-1.61 (m, 2H), 1.51-1.17 (m, 8H), 1.45 (s, 3H), 1.37 (s, 3H), 1.25 (s, 3H), 1.06 (d, *J* = 6.9 Hz, 3H), 1.05 (d, *J* = 6.9 Hz, 3H), 0.88 (t, *J* = 6.4 Hz, 3H).

(1*R*,4*S*,5*R*)-1-((*S*)-2-Hydroxy-2-methyloctanoyl)-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(7*S*)-**31**}

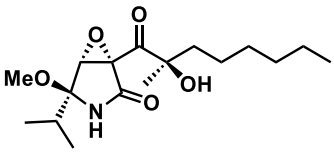


Saturated aqueous $NaHCO_3$ (0.17 mL), AZADOL (0.4 mg, 0.003 mmol), KBr (3.2 mg, 0.027 mmol), and 5% NaClO aqueous solution (0.01 mL, 0.1 mmol) were added to a stirred solution of diol (7*S*)-**29** (9.0 mg, 0.027 mmol) in CH_2Cl_2 (0.34 mL) at 0 °C. After vigorously stirring for 30 min, the reaction was quenched with saturated aqueous $Na_2S_2O_3$. The mixture was extracted with EtOAc (3 times). The combined organic layers were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 3:1) to afford ketone (7*S*)-**31** (8.3 mg, 93%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.75 (br s, 1H), 3.98 (d, *J* = 2.4 Hz, 1H), 3.58 (br s, 1H), 3.32 (s, 3H), 2.09 (sept, *J* = 6.8 Hz, 1H), 2.00-1.88 (m, 1H), 1.81-1.69 (m, 1H), 1.50-1.18 (m, 8H),1.32 (s, 3H), 1.09 (d, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.8 Hz, 3H), 0.87 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 169.6, 90.6, 80.0, 63.4, 61.9, 50.3, 38.6, 33.6, 31.6, 29.5, 24.1, 23.1, 22.6, 17.4, 16.1, 14.1; IR

($CHCl_3$) 3244, 2958, 2931, 2872, 2857, 2359, 2342, 1731, 1714, 1673, 1457, 1430, 1419, 1395, 1363, 1098, 1068, 1036 cm^{-1}; $[\alpha]_D^{25}$ –49.2 (*c* 1.39, $CHCl_3$); FAB-HRMS *m/z* 328.2130 ($[M+H]^+$ calcd for $C_{17}H_{30}NO_5$: 328.2124).

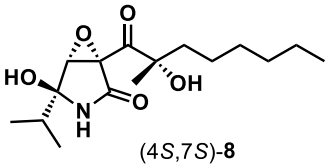
(1*R*,4*S*,5*R*)-1-((*R*)-2-Hydroxy-2-methyloctanoyl)-4-methoxy-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(7*R*)-**S5**}



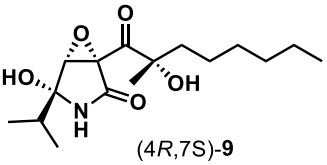
(7*R*)-**S5** (9.1 mg, 95% yield, colorless oil) was prepared from (7*R*)-**30** (9.7 mg, 0.029 μmol) by a procedure similar to that for (7*S*)-**31**.

^1H NMR (400 MHz, $CDCl_3$) δ 5.78 (br s, 1H), 3.94 (s, 1H), 3.34 (s, 3H), 3.25 (br s, 1H), 2.09 (sept, *J* = 7.0 Hz, 1H), 1.87-1.73 (m, 1H), 1.67-1.45 (m, 1H), 1.51 (s, 3H), 1.45-1.17 (m, 8H), 1.09 (d, *J* = 7.0 Hz, 3H), 0.99 (d, *J* = 7.0 Hz, 3H), 0.87 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 170.0, 90.8, 80.2, 63.7, 62.8, 50.3, 39.3, 33.4, 31.6, 29.4, 24.6, 23.5, 22.5, 17.2, 16.0, 14.0; IR ($CHCl_3$) 3256, 2957, 2931, 2871, 2857, 1729, 1467, 1430, 1390, 1368, 1132, 1097, 1072, 1036, 850 cm^{-1}; $[\alpha]_D^{21}$ –84.6 (*c* 035, $CHCl_3$); FAB-HRMS *m/z* 328.2130 ($[M+H]^+$ calcd for $C_{17}H_{30}NO_5$: 328.2124).

(1*R*,4*S*,5*R*)-4-Hydroxy-1-((*S*)-2-hydroxy-2-methyloctanoyl)-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(4*S*,7*S*)-**8**} and (1*R*,4*R*,5*R*)-4-hydroxy-1-((*S*)-2-hydroxy-2-methyloctanoyl)-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(4*R*,7*S*)-**9**}



(4*S*,7*S*)-**8**



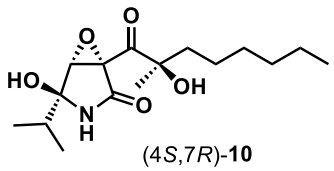
(4*R*,7S)-**9**

Aminal (7*S*)-**31** (16.1 mg, 0.049 mmol) was dissolved in water/TFA (1:1 v/v, 0.78 mL), and the solution was stirred for 10 min at room temperature. The reaction was quenched with saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (3 times). The combined organic layers were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 6:1) to afford hemiaminal (4*S*,7*S*)-**8** (6.2 mg, 40%) and (4*R*,7*S*)-**9** (0.8 mg, 5%) as a pale yellow oil.

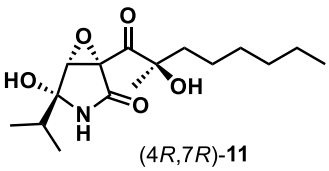
(4*S*,7*S*)-**8**: ^1H NMR (300 MHz, C_5D_5N) δ 10.17 (br s, 1H), 8.23 (br s, 1H), 7.02 (br s, 1H), 4.74 (d, *J* = 2.1 Hz, 1H), 2.36 (sept, *J* = 6.9 Hz, 1H), 2.43-2.26 (m, 1H), 2.18-2.01 (m, 1H), 1.78-1.63 (m, 2H), 1.59 (s, 3H), 1.35 (d, *J* = 6.9 Hz. 3H), 1.32 (d, *J* = 6.9 Hz, 3H), 1.29-1.12 (m, 6H), 0.76 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, C_5D_5N) δ 206.2, 170.8, 88.1, 80.1, 65.4, 63.7, 39.3, 34.7, 32.1, 30.2, 25.2, 23.9, 23.0, 18.2, 16.9, 14.3; IR ($CHCl_3$) 3309, 2957, 2929, 2858, 1732, 1716, 1685, 1558, 1541, 1521, 1507, 1472, 1457 cm^{-1}; $[\alpha]_D^{22}$ –71.3 (*c* 0.41, $CHCl_3$); FAB-HRMS *m/z* 314.1973 ($[M+H]^+$ calcd for $C_{16}H_{28}NO_5$: 314.1967).

(4*R*,7*S*)-**9**: ^{1}H NMR (300 MHz, C_5D_5N) δ 9.92 (br s, 1H), 8.47 (br s, 1H), 7.08 (br s, 1H), 4.72 (d, *J* = 2.4 Hz, 1H), 2.52-2.28 (m, 2H), 2.25-2.03 (m, 1H), 1.85-1.64 (m, 2H), 1.62 (s, 3H), 1.42-1.06 (m, 6H), 1.32 (d, *J* = 6.9 Hz, 3H), 1.29 (d, *J* = 6.9 Hz, 3H), 0.76 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, C_5D_5N) δ 206.7, 169.6, 88.4, 80.0, 71.0, 64.0, 39.4, 36.0, 32.1, 30.2, 25.5, 23.9, 23.0, 17.00, 16.97, 14.3; IR ($CHCl_3$) 3545, 3019, 2931, 2858, 2369, 2322, 1716, 1698, 1684, 1558, 1541, 1533, 1521, 1507, 1214 cm^{-1}; $[\alpha]_D^{22}$ +23.2 (*c* 0.04, $CHCl_3$); FAB-HRMS *m/z* 314.1973 ($[M+H]^+$ calcd for $C_{16}H_{28}NO_5$: 314.1967).

(1*R*,4*S*,5*R*)-4-Hydroxy-1-((*R*)-2-hydroxy-2-methyloctanoyl)-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(4*S*,7*R*)-**10**} and (1*R*,4*R*,5*R*)-4-hydroxy-1-((*R*)-2-hydroxy-2-methyloctanoyl)-4-(propan-2-yl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one {(4*R*,7*R*)-**11**}



(4*S*,7*R*)-**10**



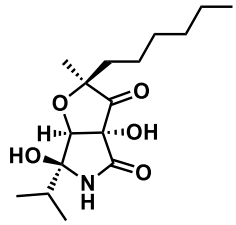
(4*R*,7*R*)-**11**

(4*S*,7*R*)-**10** (4.7 mg, 43%, pale yellow oil) and (4*R*,7*R*)-**11** (0.9 mg, 8%, pale yellow oil) were prepared from (7*R*)-**S5** (11.5 mg, 0.035 mmol) by a procedure similar to that for (4*S*,7*S*)-**8** and (4*R*,7*S*)-**9**.

(4*S*,7*R*)-**10**: ^{1}H NMR (300 MHz, C_5D_5N) δ 10.13 (br s, 1H), 7.79 (br s, 1H), 7.34 (br s, 1H), 4.71 (d, *J* = 2.4 Hz, 1H), 2.37 (sept, *J* = 7.2 Hz, 1H), 2.14-1.97 (m, 1H), 1.93-1.76 (m, 1H), 1.83 (s, 3H), 1.79-1.58 (m, 1H), 1.45-1.01 (m, 7H), 1.34 (d, *J* = 6.9 Hz, 3H), 1.31 (d, *J* = 7.2 Hz, 3H), 0.78 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (125 MHz, C_5D_5N) δ 206.6, 171.1, 88.4, 80.3, 65.1, 64.7, 40.8, 34.6, 32.0, 30.1, 25.0, 24.1, 23.0, 18.2, 16.9, 14.4; IR ($CHCl_3$) 3335, 2958, 2930, 2859, 1733, 1717, 1558, 1541, 1507, 1472, 1457 cm^{-1}; $[\alpha]_D^{20}$ +202.4 (*c* 0.15, $CHCl_3$); FAB-HRMS *m/z* 314.1968 ($[M+H]^+$ calcd for $C_{16}H_{28}NO_5$: 314.1967).

(4*R*,7*R*)-**11**: ^{1}H NMR (400 MHz, C_5D_5N) δ 9.88 (br s, 1H), 8.46 (br s, 1H), 7.38 (br s, 1H), 4.69 (d, *J* = 2.4 Hz, 1H), 2.52 (sept, *J* = 6.8 Hz, 1H), 2.17-2.00 (m, 1H), 1.93-1.63 (m 2H), 1.88 (s, 3H), 1.45-1.10 (m, 7H), 1.37 (d, *J* = 6.8 Hz, 3H), 1.36 (d, *J* = 6.8 Hz, 3H), 0.80 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, C_5D_5N) δ 207.5, 169.8, 88.7, 80.0, 67.3, 63.8, 41.4, 36.0, 32.1, 30.1, 25.5, 24.2, 23.0, 17.1, 17.0, 14.4; IR ($CHCl_3$) 3360, 2958, 2929, 2859, 2359, 2342, 2331, 1732, 1716, 1699, 1558, 1541, 1507, 1472, 1457 cm^{-1}; $[\alpha]_D^{21}$ +289.8 (*c* 0.14, $CHCl_3$); FAB-HRMS *m/z* 314.1968 ($[M+H]^+$ calcd for $C_{16}H_{28}NO_5$: 314.1967).

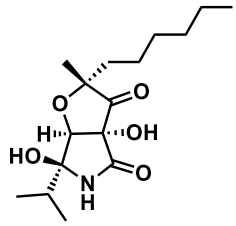
(2*S*,3a*R*,6*S*,6a*S*)-2-Hexyl-3a,6-dihydroxy-6-(propan-2-yl)-2-methyltetrahydro-2*H*-furo[2,3-*c*]pyrrole-3,4-dione {(4*S,*7*S*)-**12**}



A solution of NaOMe (2.6 mg, 0.048 mmol) in MeOH (0.26 mL) was added to a mixture of (4*S*,7*S*)-**8** and (4*R*,7*S*)-**9** (5.6:1 ratio, 4.9 mg, 0.016 mmol) in MeOH (0.54 mL). After stirring for 1 h, the reaction mixture was concentrated *in vacuo.* The residue was diluted with EtOAc and washed with water. The organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 1:1) to afford alcohol (4*S,*7*S*)-**12** (4.1 mg, 84%) as a pale yellow oil.

^{1}H NMR (400 MHz, C_5D_5N) δ 10.37 (br s, 1H), 9.11 (br s, 1H), 8.71 (br s, 1H), 5.08 (s, 1H), 2.66 (sept, *J* = 7.2 Hz, 1H), 2.06-1.92 (m, 1H), 1.85 (s, 3H), 1.82-1.71 (m, 1H), 1.70-1.60 (m, 1H), 1.55-1.45 (m, 1H), 1.43 (d, *J* = 6.8 Hz, 3H), 1.19 (d, *J* = 6.8 Hz, 3H), 1.12-0.85 (m, 6H), 0.71 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, C_5D_5N) δ 209.0, 171.9, 91.6, 88.2, 84.9, 77.9, 42.4, 32.5, 32.0, 29.9, 24.3, 23.7, 23.0, 17.8, 17.3,14.3; IR ($CHCl_3$) 3313, 2952, 2931, 2871, 2858, 1744, 1715, 1470, 1458, 1369, 1175, 1114, 1074 cm^{-1}; $[\alpha]_D^{23}$ +84.4 (*c* 0.33, $CHCl_3$); FAB-HRMS *m/z* 314.1972 ($[M+H]^+$ calcd for $C_{16}H_{28}NO_5$: 314.1967).

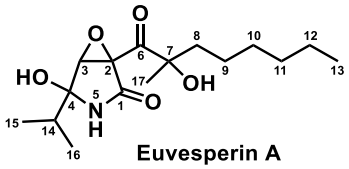
(2*R*,3a*R*,6*S*,6a*S*)-2-Hexyl-3a,6-dihydroxy-6-(propan-2-yl)-2-methyltetrahydro-2*H*-furo[2,3-*c*]pyrrole-3,4-dione { (4*S,*7*R*)-**14**}

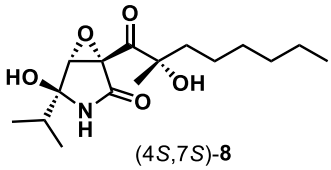


(4*S,*7*R*)-**14** (7.0 mg, 81%, pale yellow oil) was prepared from a mixture of (4*S,*7*R*)-**10** and (4*R,*7*R*)-**11** (5.6:1 ratio, 8.6 mg, 0.027 mmol) by a procedure similar to that for the (4*S,*7*S*)-**12**.

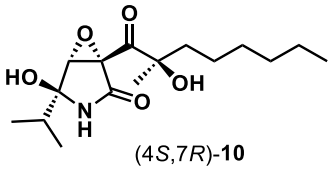
^{1}H NMR (300 MHz, C_5D_5N) δ 10.30 (br s, 1H), 9.11 (br s, 1H), 8.63 (br s, 1H), 5.07 (s, 1H), 2.63 (sept, *J* = 6.9 Hz, 1H), 2.45 (dt, *J* = 4.5, 12.3 Hz, 1H), 2.16-2.02 (m, 1H), 1.79-1.62 (m, 1H), 1.60 (s, 3H), 1.49-1.33 (m, 1H), 1.37 (d, *J* = 6.9 Hz, 3H), 1.31-1.08 (m, 6H), 1.24 (d, *J* = 6.9 Hz, 3H), 0.79 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, C_5D_5N) δ 209.3, 172.1, 91.4, 88.3, 84.2, 77.6, 39.7, 32.7, 32.3, 30.3, 28.2, 23.8, 23.1, 17.9, 17.4, 14.4; IR ($CHCl_3$) 3333, 2958, 2928, 2872, 2857, 1743, 1716, 1457, 1417, 1373, 1338, 1113, 1074, 1023 cm^{-1}; $[\alpha]_D^{23}$ +37.1 (*c* 0.48, $CHCl_3$); FAB-HRMS *m/z* 314.1959 ($[M+H]^+$ calcd for $C_{16}H_{28}NO_5$: 314.1967).

Table S1 ^{1}H and ^{13}C NMR data of natural euvesperin A, and synthetic (4*S*,7*S*)-**8** and (4*S*,7*R*)-**10**





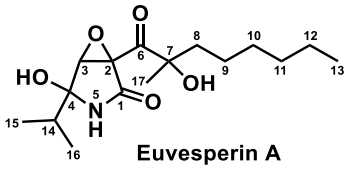
(4*S*,7*S*)-**8**

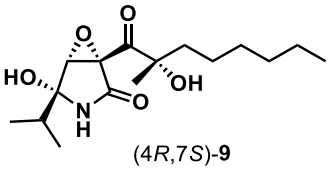


(4*S*,7*R*)-**10**

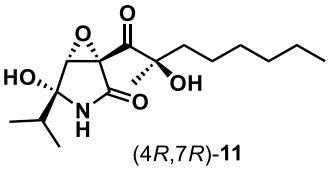
Position	Natural euvesperin A (isomer-1)		(4*S*,7*S*)-**8**		Natural euvesperin A (isomer-2)		(4*S*,7*R*)-**10**	
	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C
1		170.7		170.8		171.1		171.1
2		63.7		63.7		64.7		64.7
3	4.75 (d, 2.2)	65.4	4.74 (d, 2.1)	65.4	4.71 (d, 1.8)	65.1	4.71 (d, 2.4)	65.1
4		88.1		88.1		88.4		88.4
5	10.17 (d, 2.2)		10.17 (br s)		10.14 (d, 1.8)		10.13 (br s)	
6		206.2		206.2		206.7		206.6
7	-	80.0		80.1	-	80.2	-	80.3
8	2.08, 2.36 (br s)	39.3	2.18-2.01 2.43-2.26 (m)	39.3	1.83, 2.06 (br s)	40.7	1.93-1.76 2.14-1.97 (m)	40.8
9	1.23, 1.28 (m)	30.2	1.29-1.12 (m)	30.2	1.23, 1.28 (m)	30.2	1.12-1.28 (m)	30.1
10	1.67, 1.72 (m)	23.9	1.78-1.63 (m)	23.9	1.48, 1.80 (m)	23.7	1.45-1.01 1.79-1.58 (m)	24.1
11	1.02, 1.14 (m)	32.1	1.29-1.12 (m)	32.1	1.16, 1.24 (m)	32.1	1.45-1.01 (m)	32.0
12	1.16, 1.22 (m)	23.0	1.29-1.12 (m)	23.0	1.16, 1.22 (m)	23.0	1.45-1.01 (m)	23.0
13	0.76 (t, 6.0)	14.3	0.76 (t, 6.9)	14.3	0.78 (t, 6.0)	14.3	0.78 (t, 6.6)	14.4
14	2.37 (dq, 7.3, 7.3)	34.6	2.36 (sept, 6.9)	34.7	2.35 (dq, 7.3, 7.3)	34.6	2.37 (sept, 7.2)	34.6
15	1.35 (d, 7.3)	16.9	1.35 (d, 6.9)	16.9	1.31 (d, 7.3)	18.1	1.31 (d, 7.2)	18.2
16	1.32 (d, 7.3)	18.1	1.32 (d, 6.9)	18.2	1.34 (d, 7.3)	16.9	1.34 (d, 6.9)	16.9
17	1.59 (s)	28.0	1.59 (s)	25.2	1.83 (s)	24.9	1.83 (s)	25.0

Table S2 ^{1}H and ^{13}C NMR data of natural euvesperin A, and synthetic (4*R*,7*S*)-**9** and (4*R*,7*R*)-**11**





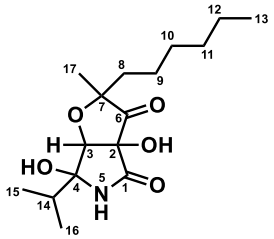
(4*R*,7*S*)-**9**



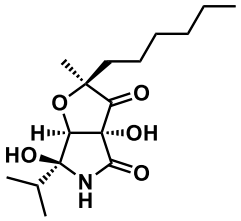
(4*R*,7*R*)-**11**

Position	Natural euvesperin A (isomer-1)		(4*R*,7*S*)-**9**		Natural euvesperin A (isomer-2)		(4*R*,7*R*)-**11**	
	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C
1		170.7		169.6		171.1		169.8
2		63.7		64.0		64.7		63.8
3	4.75 (d, 2.2)	65.4	4.72 (d, 2.4)	71.0	4.71 (d, 1.8)	65.1	4.69 (d, 2.4)	67.3
4		88.1		88.4		88.4		88.7
5	10.17 (d, 2.2)		9.92 (br s)		10.14 (d, 1.8)		9.88 (br s)	
6		206.2		206.7		206.7		207.5
7	-	80.0		80.0	-	80.2	-	80.0
8	2.08, 2.36 (br s)	39.3	2.25-2.03 2.52-2.28 (m)	39.4	1.83, 2.06 (br s)	40.7	1.93-1.63 2.17-2.00 (m)	41.4
9	1.23, 1.28 (m)	30.2	1.42-1.06 (m)	30.2	1.23, 1.28 (m)	30.2	1.45-1.10 (m)	30.1
10	1.67, 1.72 (m)	23.9	1.85-1.64 (m)	23.9	1.48, 1.80 (m)	23.7	1.45-1.10 1.93-1.63 (m)	24.2
11	1.02, 1.14 (m)	32.1	1.42-1.06 (m)	32.1	1.16, 1.24 (m)	32.1	1.45-1.10 (m)	32.1
12	1.16, 1.22 (m)	23.0	1.42-1.06 (m)	23.0	1.16, 1.22 (m)	23.0	1.45-1.10 (m)	23.0
13	0.76 (t, 6.0)	14.3	0.76 (t, 7.2)	14.3	0.78 (t, 6.0)	14.3	0.80 (t, 6.9)	14.4
14	2.37 (dq, 7.3, 7.3)	34.6	2.52-2.28 (m)	36.0	2.35 (dq, 7.3, 7.3)	34.6	2.52 (sept, 6.8)	36.0
15	1.35 (d, 7.3)	16.9	1.32 (d, 6.9)	16.97	1.31 (d, 7.3)	18.1	1.36 (d, 6.8)	17.1
16	1.32 (d, 7.3)	18.1	1.29 (d, 6.9)	17.00	1.34 (d, 7.3)	16.9	1.37 (d, 6.8)	17.0
17	1.59 (s)	28.0	1.62 (s)	25.5	1.83 (s)	24.9	1.88 (s)	25.5

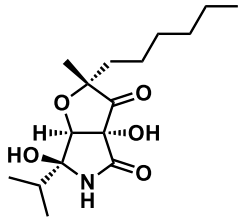
Table S3 ^{1}H and ^{13}C NMR data of natural euvesperin B, and synthetic (4*S*,7*S*)-**12** and (4*S*,7*R*)-**14**



Euvesperin B



(4*S*,7*S*)-**12**

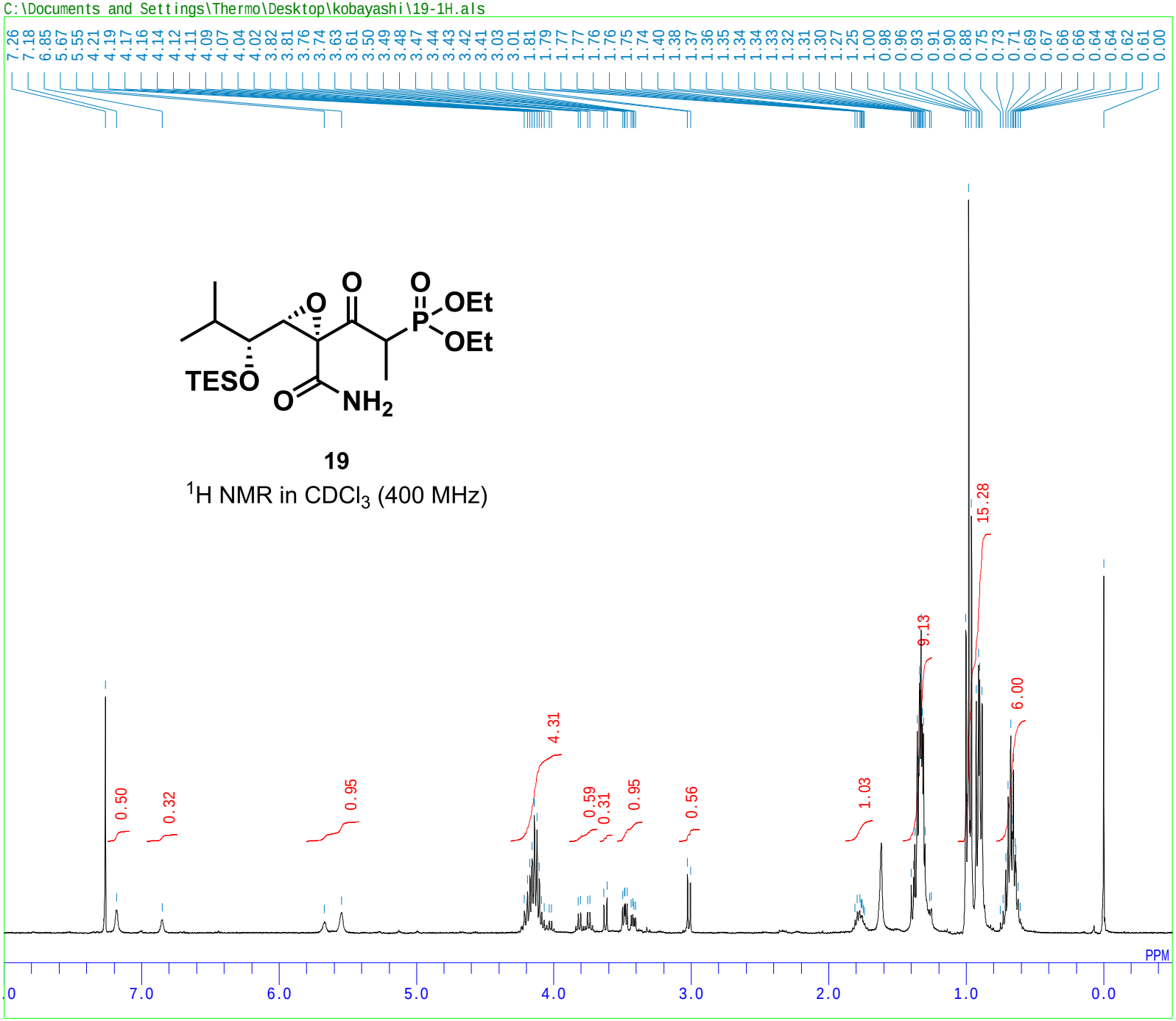


(4*S*,7*R*)-**14**

Position	Natural euvesperin B (isomer-1)		(4*S*,7*S*)-**12**		Natural euvesperin B (isomer-2)		(4*S*,7*R*)-**14**	
	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C
1		171.9		171.9		172.1		172.1
2		88.1		88.2		88.2		88.3
3	5.09 (s)	77.8	5.08 (s)	77.9	5.07 (s)	77.6	5.07 (s)	77.6
4		91.6		91.6		91.4		91.4
5	10.38 (s)		10.37 (br s)		10.31 (br s)		10.30 (br s)	
6		209.0		209.0		209.3		209.3
7	-	84.8		84.9	-	84.1	-	84.2
8	1.76, 2.00 (br s)	42.3	1.70-1.60 2.06-1.92 (m)	42.4	2.06, 2.45 (br s)	39.6	2.16-2.02 (m) 2.45 (dt, 4.5, 12.3)	39.7
9	0.93, 0.99 (m)	29.8	1.12-0.85 (m)	29.9	1.18, 1.28 (m)	30.0	1.31-1.08 (m)	30.3
10	1.48, 1.80 (m)	23.7	1.55-1.45 1.82-1.71 (m)	23.7	1.40, 1.68 (m)	24.0	1.49-1.33 1.79-1.62 (m)	23.8
11	1.02, 1.14 (m)	32.0	1.12-0.85 (m)	32.0	1.20, 1.24 (m)	32.2	1.31-1.08 (m)	32.3
12	1.05, 1.09 (m)	22.9	1.12-0.85 (m)	23.0	1.16, 1.22 (m)	23.0	1.31-1.08 (m)	23.1
13	0.71 (t, 8.0)	14.2	0.71 (t, 6.8)	14.3	0.79 (t, 8.0)	14.3	0.79 (t, 7.2)	14.4
14	2.67 (dq, 7.4, 7.4)	32.4	2.66 (sept, 6.8)	32.5	2.63 (dq, 7.6, 7.6)	32.7	2.63 (sept, 6.9)	32.7
15	1.19 (d, 7.4)	17.8	1.19 (d, 7.2)	17.8	1.24 (t, 7.6)	17.8	1.24 (d, 6.9)	17.9
16	1.43 (d, 7.4)	17.3	1.43 (d, 6.8)	17.3	1.38 (t, 7.6)	17.3	1.37 (d, 6.9)	17.4
17	1.85 (s)	24.2	1.85 (s)	24.3	1.61 (s)	28.2	1.60 (s)	28.2

19-1H

C:\Documents and Settings\Thermo\Desktop\kobayashi\19-1H.als

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COMNT  19-1H
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OBNUC  1H
EXMOD  NON
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OBSET        124.00 KHz
OBFIN      10500.00 Hz
POINT        16384
FREQU       7992.01 Hz
SCANS            8
ACQTM        2.0500 sec
PD           4.9500 sec
PW1             5.80 usec
IRNUC  1H
CTEMP          22.2 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            17
```

19-13C

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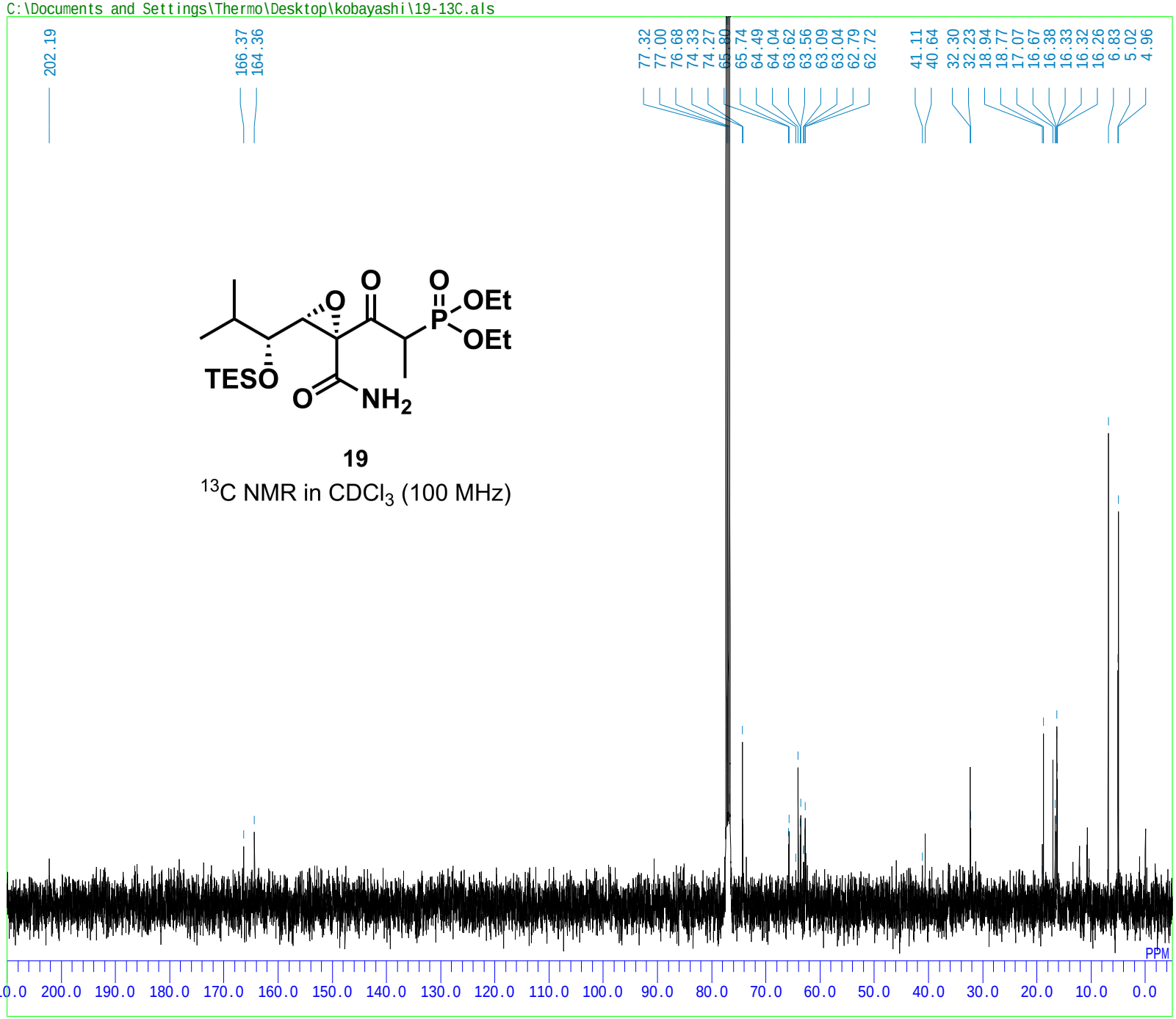


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DATIM  Fri Mar 01 18:56:37 2019
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EXMOD  BCM
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OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         1000
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP         22.8 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           24
```
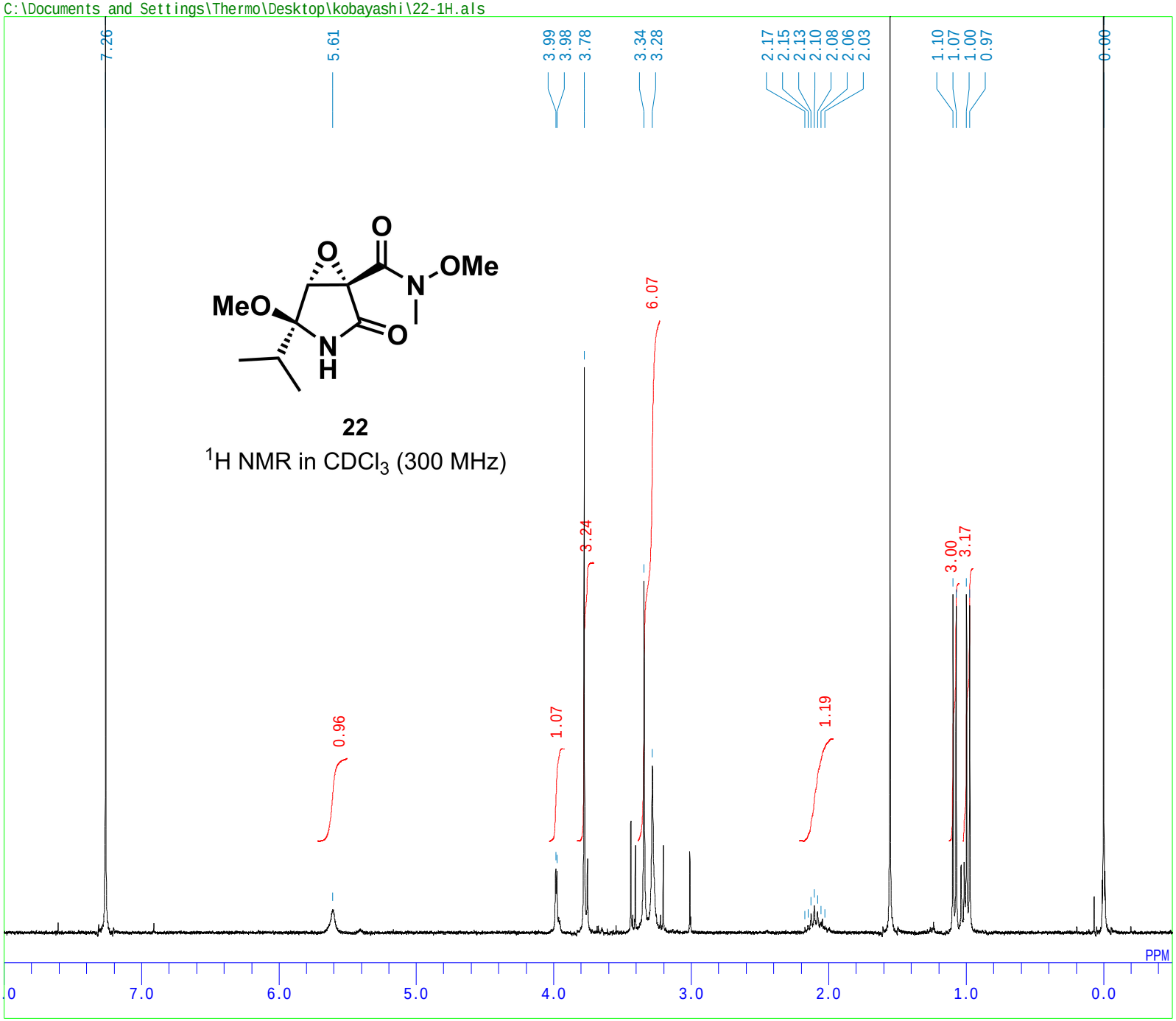

22-1H

C:\Documents and Settings\Thermo\Desktop\kobayashi\22-1H.als



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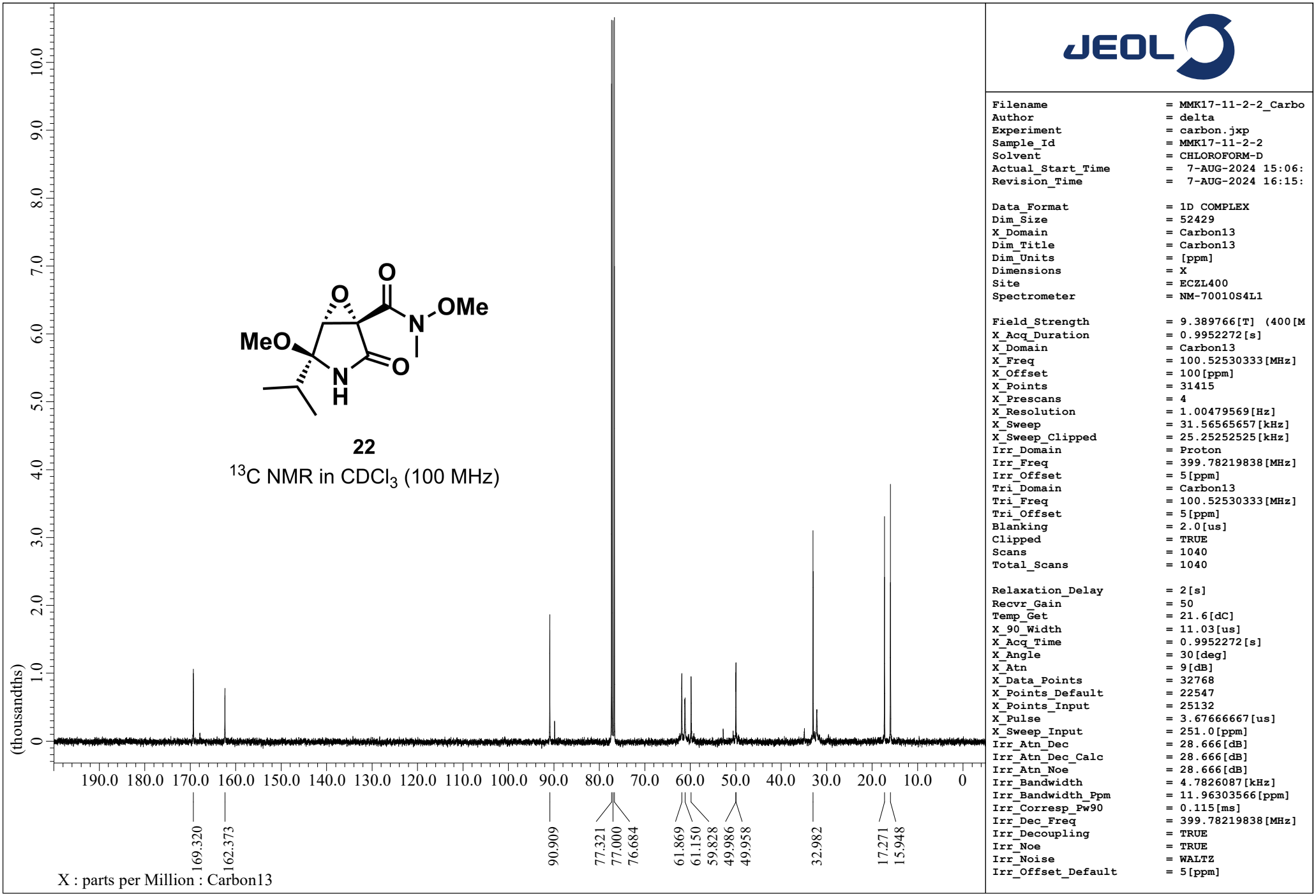
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.30 usec
IRNUC 1H
CTEMP 25.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23



JEOL

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Author	= delta
Experiment	= carbon.jxp
Sample_Id	= MMK17-11-2-2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 7-AUG-2024 15:06:
Revision_Time	= 7-AUG-2024 16:15:
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECZL400
Spectrometer	= NM-70010S4L1
Field_Strength	= 9.389766[T] (400[M
X_Acq_Duration	= 0.9952272[s]
X_Domain	= Carbon13
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 31415
X_Prescans	= 4
X_Resolution	= 1.00479569[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
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Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Carbon13
Tri_Freq	= 100.52530333[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= TRUE
Scans	= 1040
Total_Scans	= 1040
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.6[dC]
X_90_Width	= 11.03[us]
X_Acq_Time	= 0.9952272[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Data_Points	= 32768
X_Points_Default	= 22547
X_Points_Input	= 25132
X_Pulse	= 3.67666667[us]
X_Sweep_Input	= 251.0[ppm]
Irr_Atn_Dec	= 28.666[dB]
Irr_Atn_Dec_Calc	= 28.666[dB]
Irr_Atn_Noe	= 28.666[dB]
Irr_Bandwidth	= 4.7826087[kHz]
Irr_Bandwidth_Ppm	= 11.96303566[ppm]
Irr_Corresp_Pw90	= 0.115[ms]
Irr_Dec_Freq	= 399.78219838[MHz]
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
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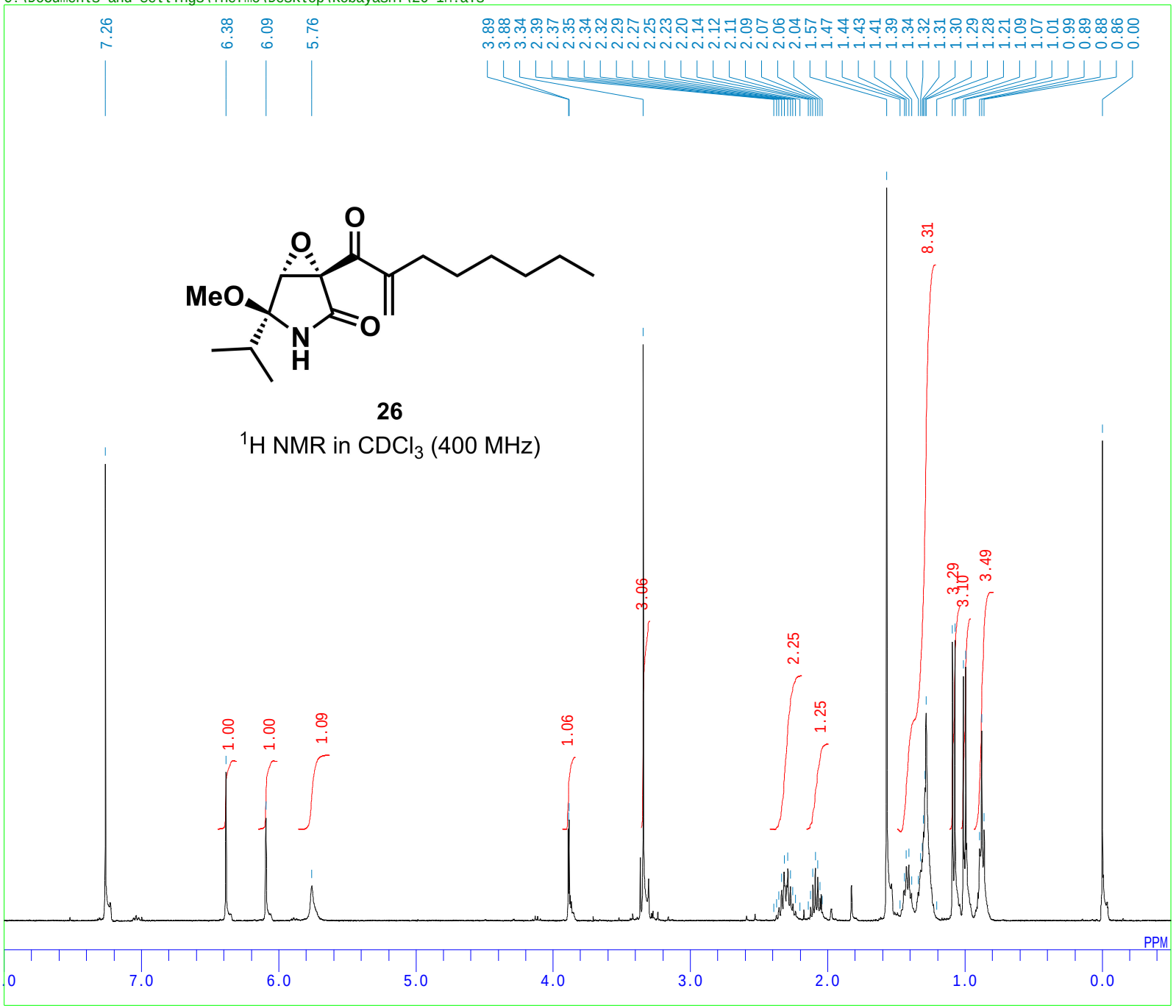
26-1H

C:\Documents and Settings\Thermo\Desktop\kobayashi\26-1H.als



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DATIM Sat Apr 07 10:22:32 2018
OBNUC 1H
EXMOD NON
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OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 26
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

26-13C

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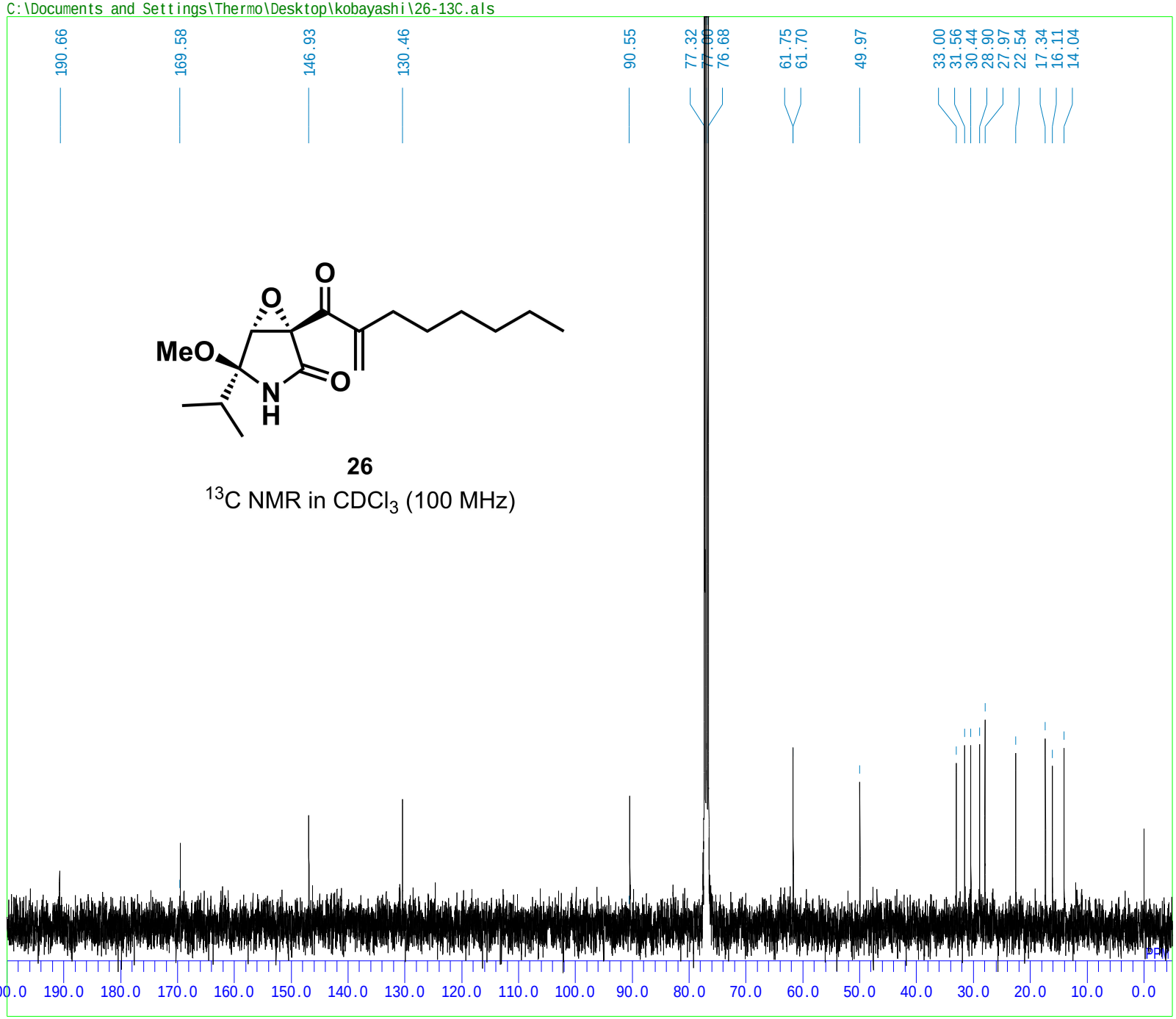


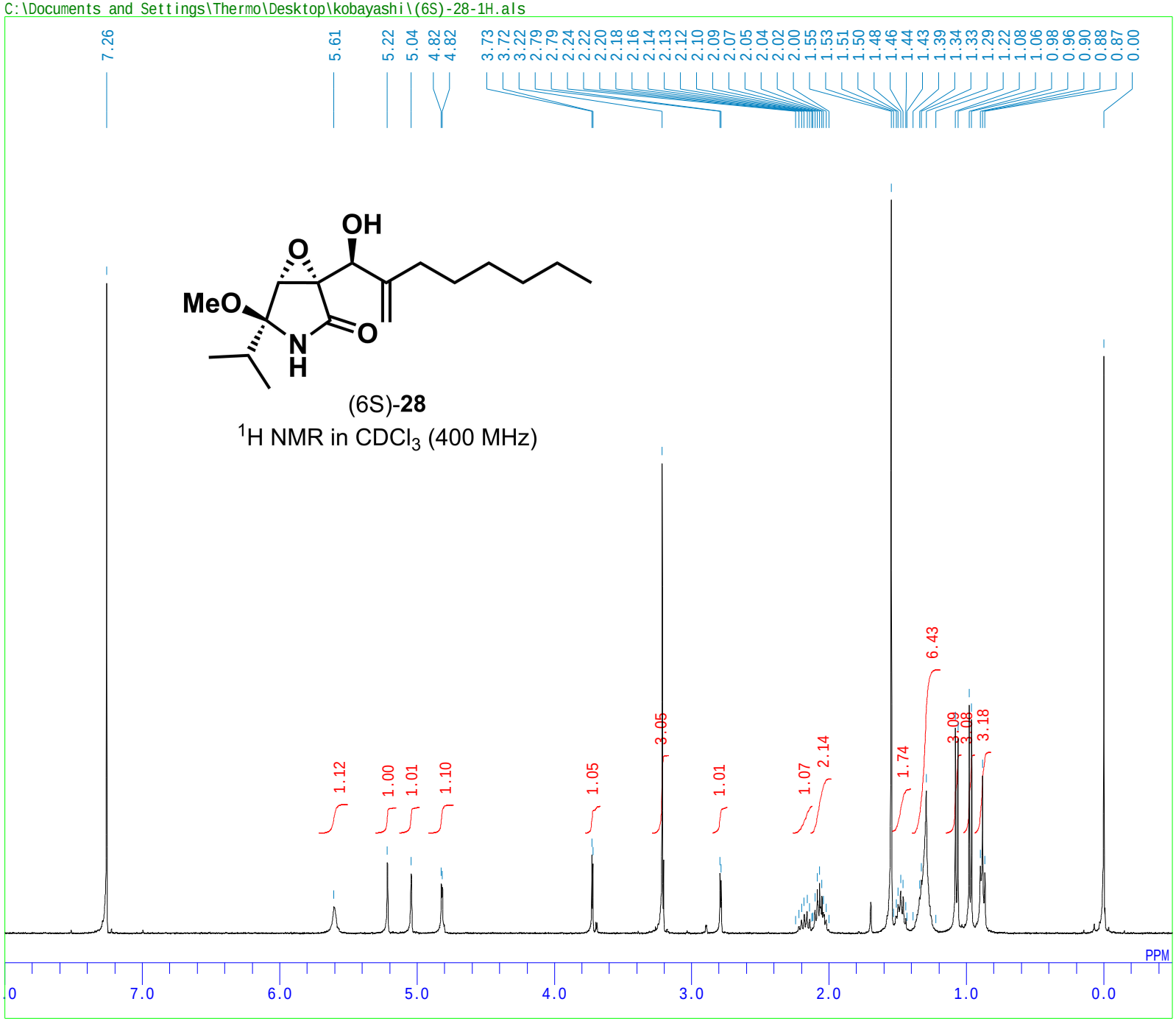
26

^{13}C NMR in $CDCl_3$ (100 MHz)

```
DFILE  26-13C.als
COMNT  26-13C
DATIM  Sat Apr 07 13:29:08 2018
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         3636
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP        23.4 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           25
```

(6S)-28-1H

C:\Documents and Settings\Thermo\Desktop\kobayashi\(6S)-28-1H.als



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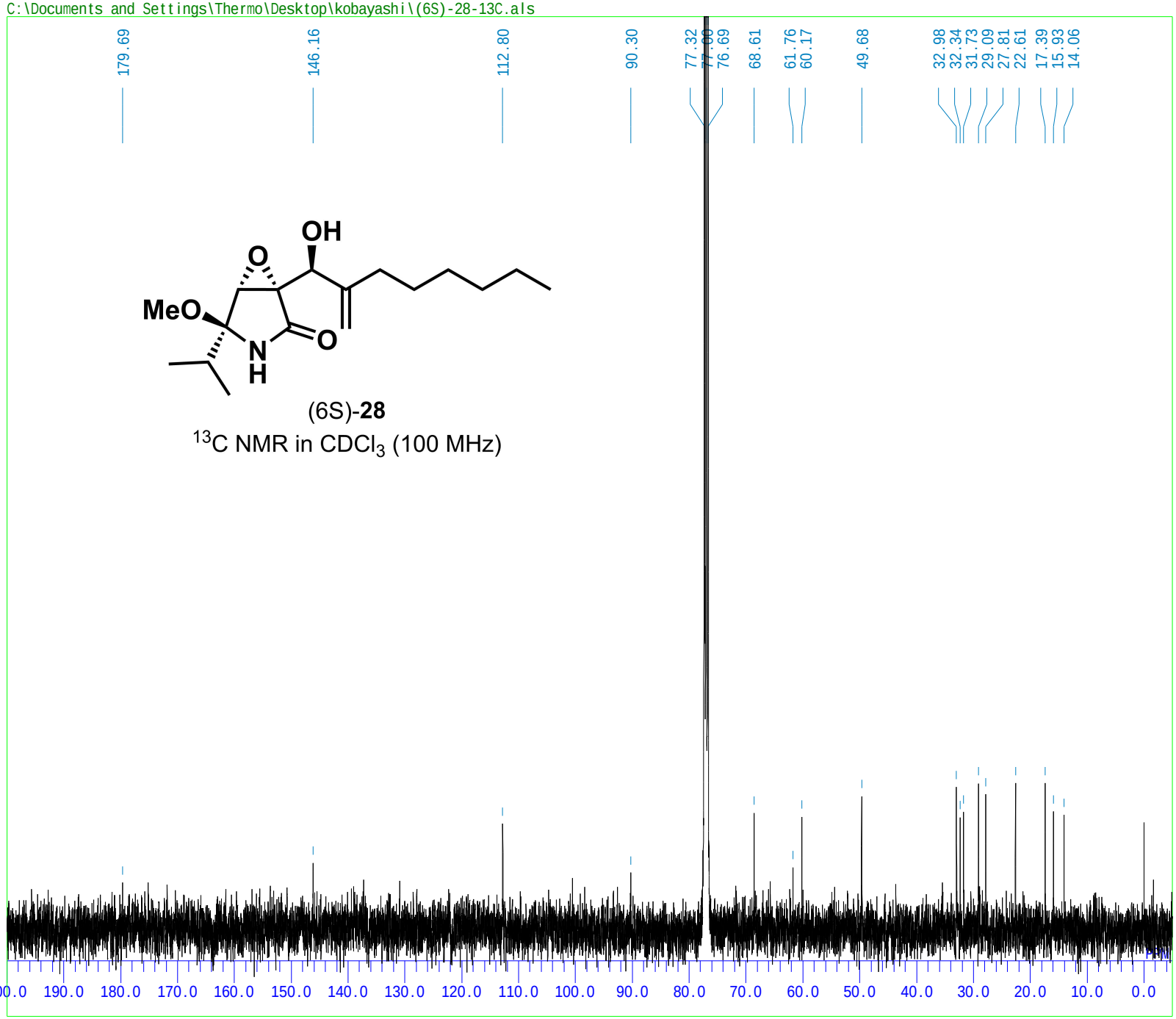
COMNT (6S)-28-1H
DATIM Mon Apr 09 12:49:24 2018
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 27
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 24.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22

(6S)-28-13C

C:\Documents and Settings\Thermo\Desktop\kobayashi\(6S)-28-13C.als



(6S)-**28**

^{13}C NMR in $CDCl_3$ (100 MHz)

```
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COMNT  (6S)-28-13C
DATIM  Mon Apr 09 15:42:32 2018
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         3303
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP         24.5 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           25
```
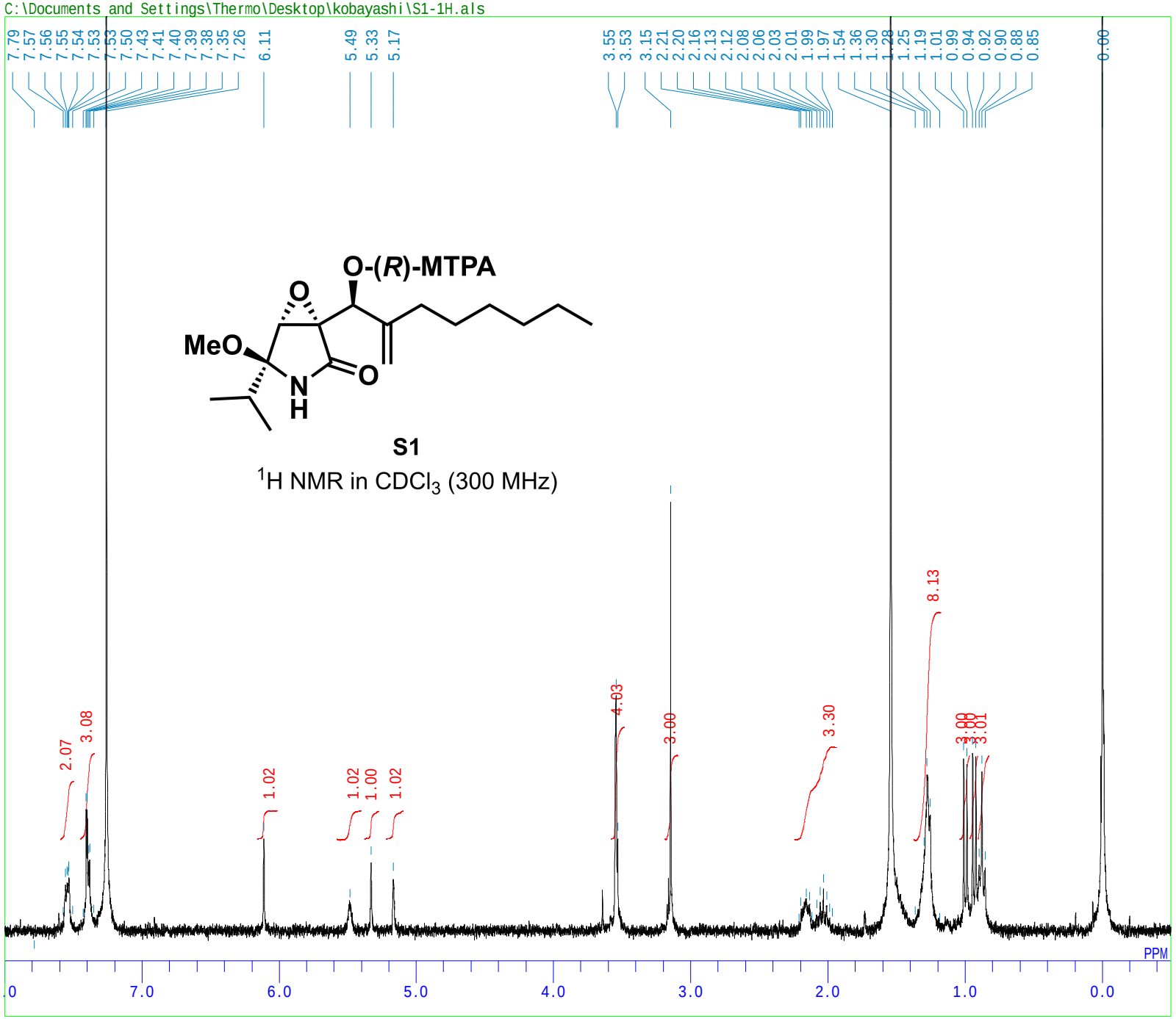

S1-1H

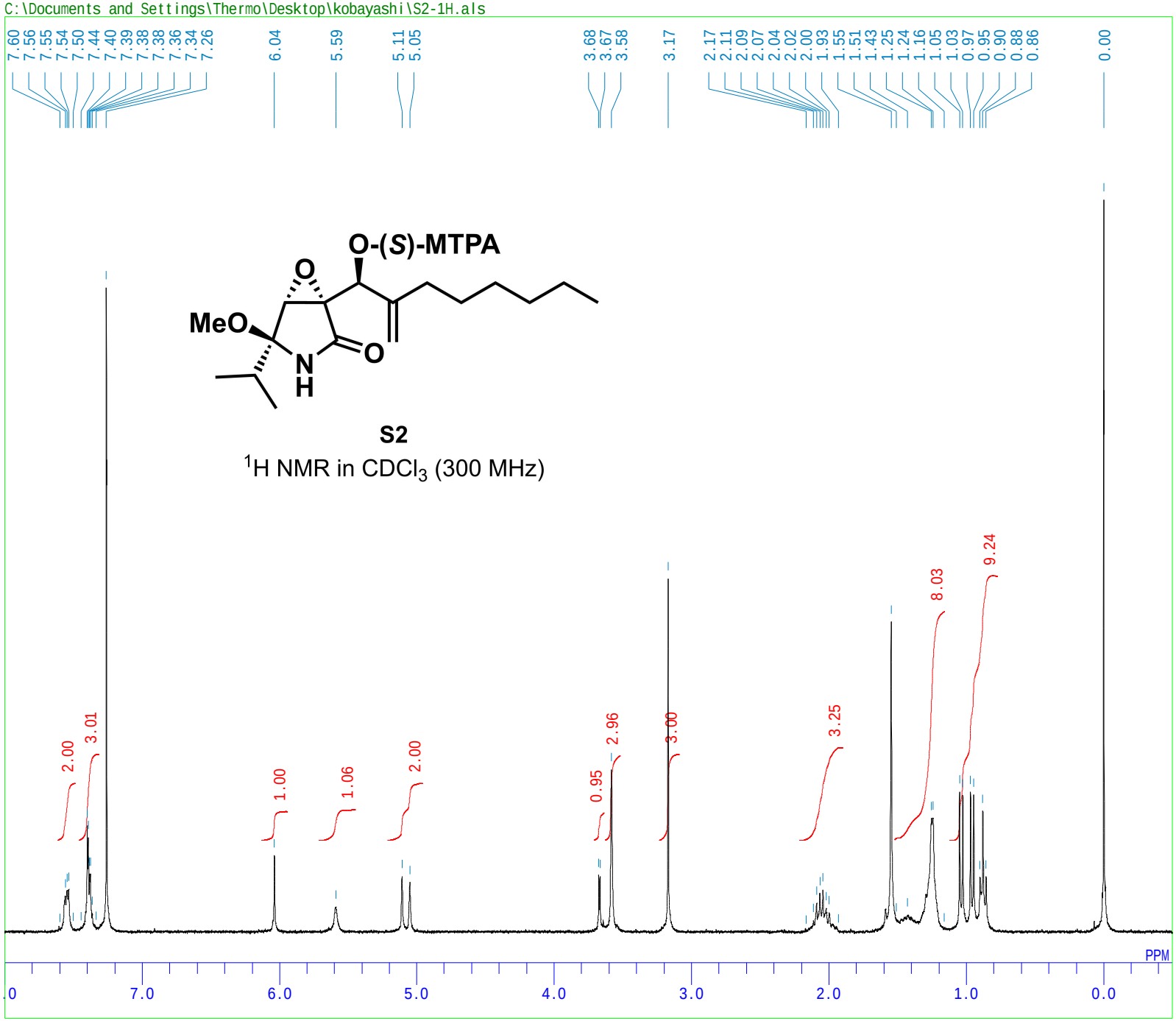
C:\Documents and Settings\Thermo\Desktop\kobayashi\S1-1H.als



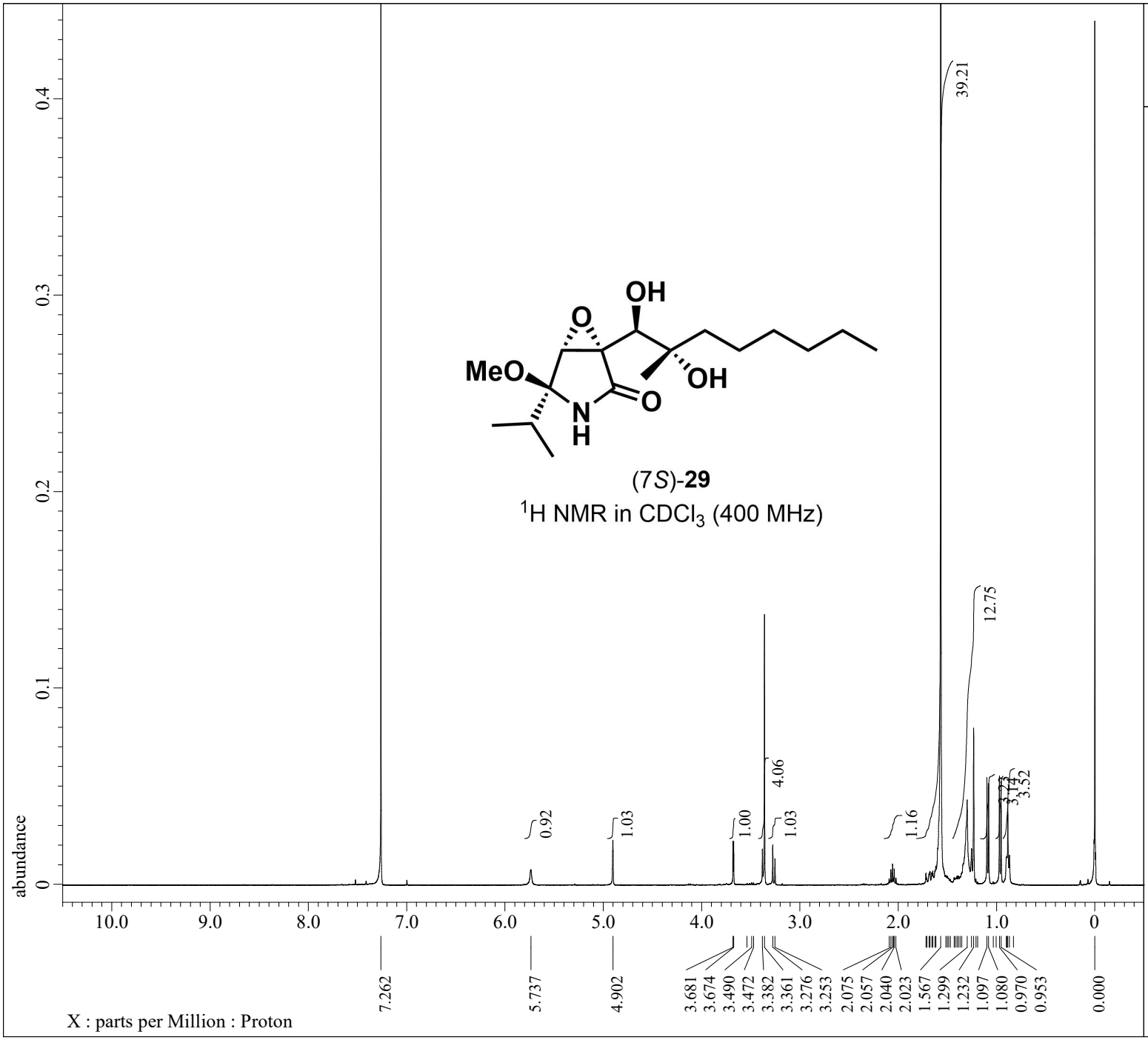
DFILE S1-1H.als
COMNT S1-1H
DATIM Thu Nov 15 20:18:47 2018
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 24

S2-1H

C:\Documents and Settings\Thermo\Desktop\kobayashi\S2-1H.als



DFILE S2-1H.als
COMNT S2-1H
DATIM Thu Nov 15 20:24:53 2018
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22



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Author	= delta
Experiment	= proton.jxp
Sample_Id	= MMK17-15
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-AUG-2024 20:16:
Revision_Time	= 14-AUG-2024 10:32:
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECZL400
Spectrometer	= NM-70010S4L1
Field_Strength	= 9.389766[T] (400[M
X_Acq_Duration	= 2.0470176[s]
X_Domain	= Proton
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 15368
X_Prescans	= 1
X_Resolution	= 0.48851558[Hz]
X_Sweep	= 7.50750751[kHz]
X_Sweep_Clipped	= 6.00600601[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 80
Total_Scans	= 80
Relaxation_Delay	= 5[s]
Recvr_Gain	= 66
Temp_Get	= 21.7[dC]
X_90_Width	= 6.65[us]
X_Acq_Time	= 2.0470176[s]
X_Angle	= 45[deg]
X_Atn	= 4[dB]
X_Data_Points	= 65536
X_Points_Default	= 23933
X_Points_Input	= 12294
X_Pulse	= 3.325[us]
X_Sweep_Input	= 15[ppm]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Default_X_Resolution	= 0.3137[Hz]
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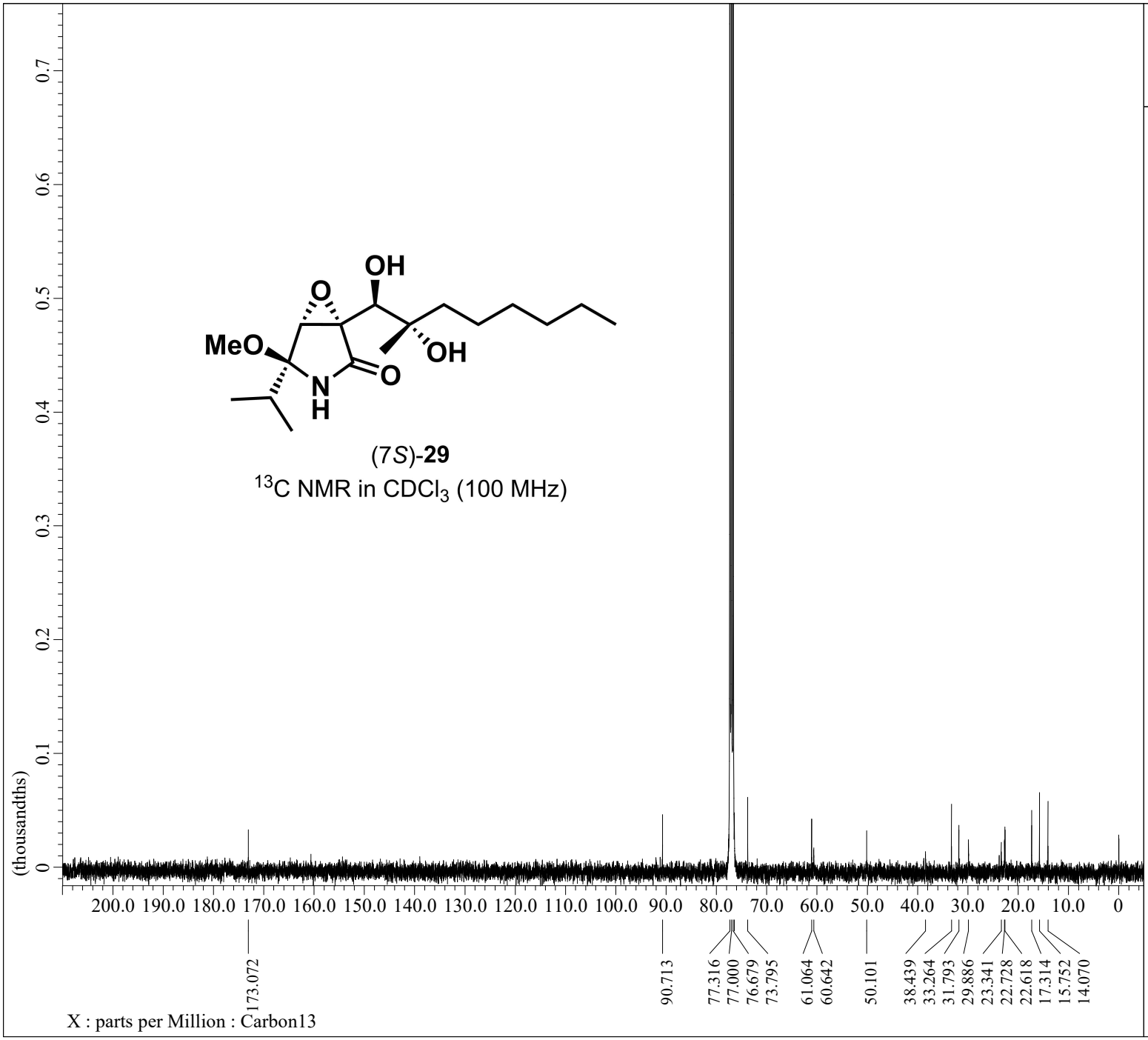
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180,
Presat_Time	= 5[s]
Presat_Time_Flag	= FALSE



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Filename	= MMK17-15-s_Carbon-
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Experiment	= carbon.jxp
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Solvent	= CHLOROFORM-D
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Revision_Time	= 14-AUG-2024 10:46:
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Dim_Size	= 52429
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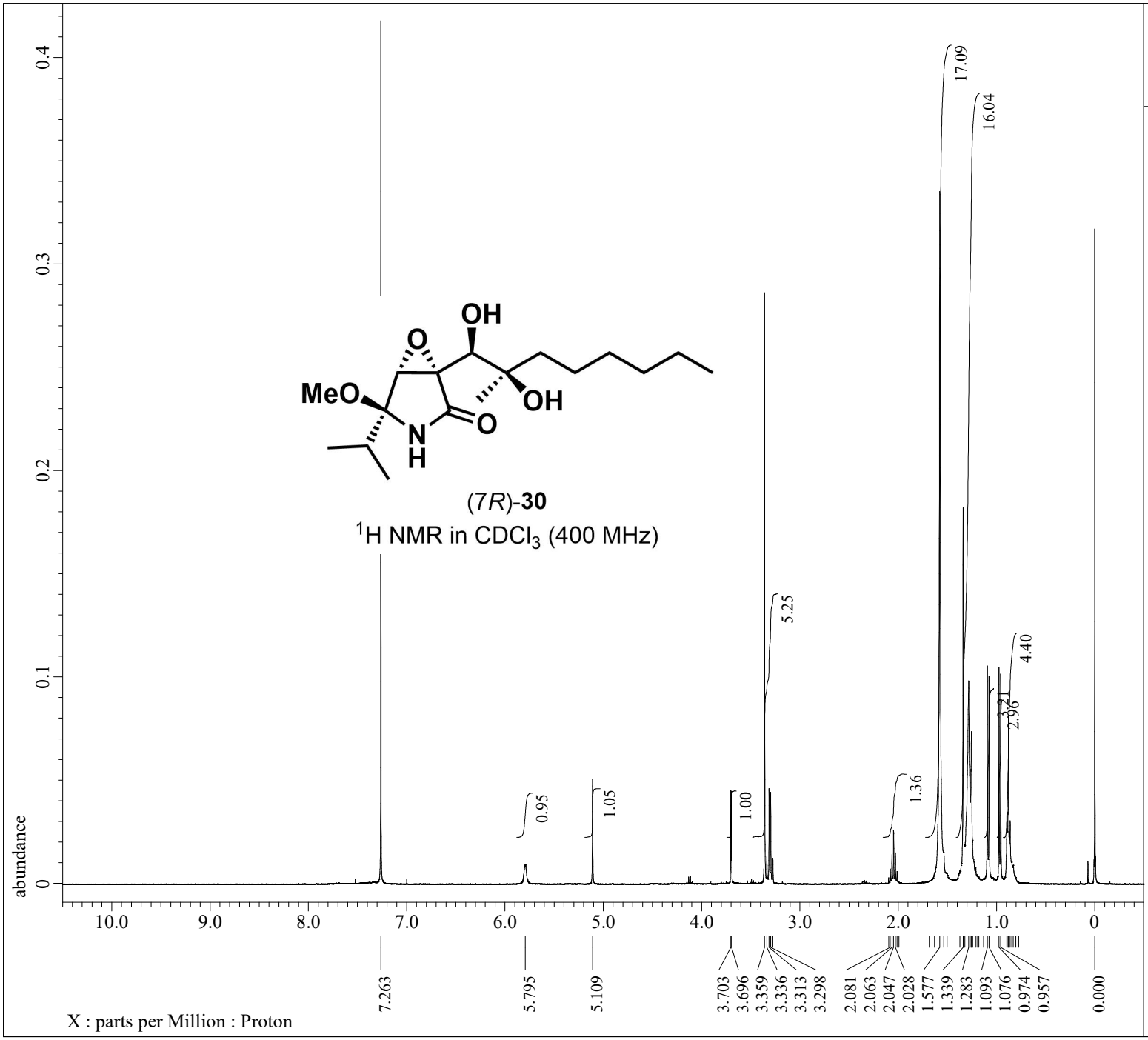
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECZL400
Spectrometer	= NM-70010S4L1
Field_Strength	= 9.389766[T] (400[M
X_Acq_Duration	= 0.9952272[s]
X_Domain	= Carbon13
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 31415
X_Prescans	= 4
X_Resolution	= 1.00479569[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Carbon13
Tri_Freq	= 100.52530333[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= TRUE
Scans	= 29146
Total_Scans	= 29146
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.7[dC]
X_90_Width	= 11.03[us]
X_Acq_Time	= 0.9952272[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Data_Points	= 32768
X_Points_Default	= 22547
X_Points_Input	= 25132
X_Pulse	= 3.67666667[us]
X_Sweep_Input	= 251.0[ppm]
Irr_Atn_Dec	= 28.666[dB]
Irr_Atn_Dec_Calc	= 28.666[dB]
Irr_Atn_Noe	= 28.666[dB]
Irr_Bandwidth	= 4.7826087[kHz]
Irr_Bandwidth_Ppm	= 11.96303566[ppm]
Irr_Corresp_Pw90	= 0.115[ms]
Irr_Dec_Freq	= 399.78219838[MHz]
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]



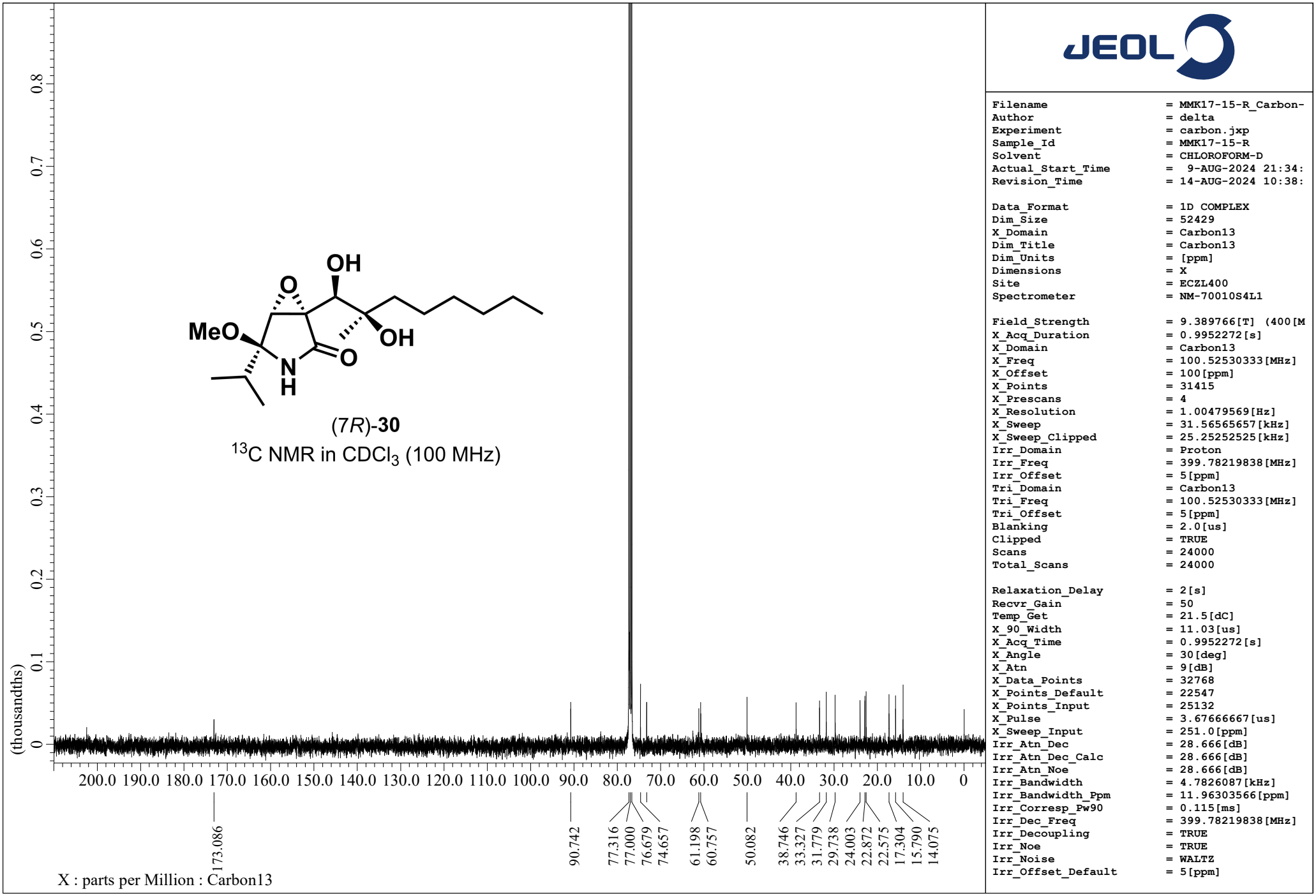
JEOL

```
Filename                = MMK17-15-R_Proton-
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = MMK17-15-R
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  9-AUG-2024 21:21:
Revision_Time           = 14-AUG-2024 10:24:

Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Site                    = ECZL400
Spectrometer            = NM-70010S4L1

Field_Strength          = 9.389766[T] (400[M
X_Acq_Duration          = 2.0470176[s]
X_Domain                = Proton
X_Freq                  = 399.78219838[MHz]
X_Offset                = 5[ppm]
X_Points                = 15368
X_Prescans              = 1
X_Resolution            = 0.48851558[Hz]
X_Sweep                 = 7.50750751[kHz]
X_Sweep_Clipped         = 6.00600601[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.78219838[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 399.78219838[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2.0[us]
Clipped                 = FALSE
Scans                   = 80
Total_Scans             = 80

Relaxation_Delay        = 5[s]
Recvr_Gain              = 66
Temp_Get                = 21.7[dC]
X_90_Width              = 6.65[us]
X_Acq_Time              = 2.0470176[s]
X_Angle                 = 45[deg]
X_Atn                   = 4[dB]
X_Data_Points           = 65536
X_Points_Default        = 23933
X_Points_Input          = 12294
X_Pulse                 = 3.325[us]
X_Sweep_Input           = 15[ppm]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Default_X_Resolution    = 0.3137[Hz]
Experiment_Path         = C:\Program Files\J
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180,
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
```

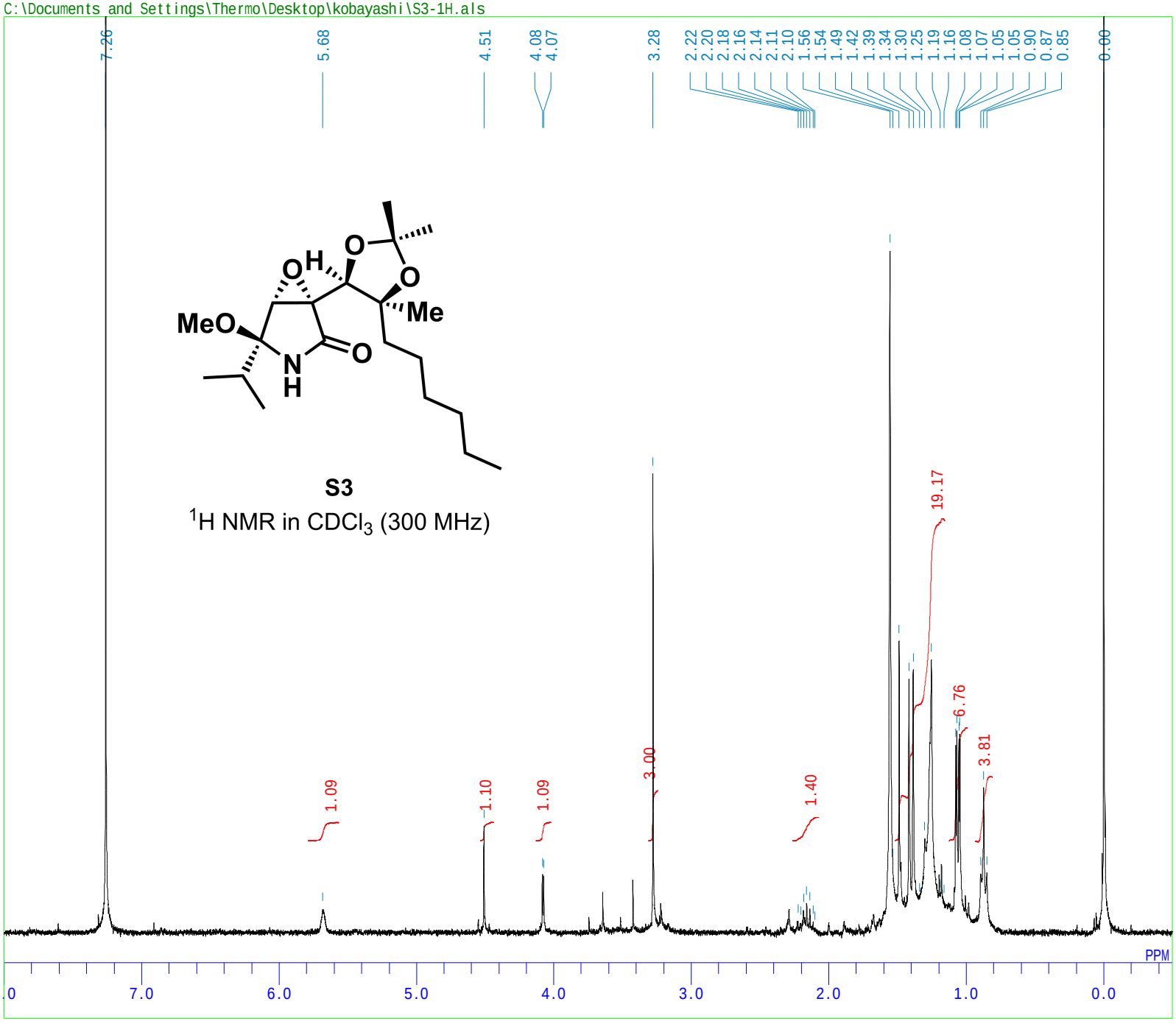


JEOL

Filename	= MMK17-15-R_Carbon-
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= MMK17-15-R
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-AUG-2024 21:34:
Revision_Time	= 14-AUG-2024 10:38:
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECZL400
Spectrometer	= NM-70010S4L1
Field_Strength	= 9.389766[T] (400[M
X_Acq_Duration	= 0.9952272[s]
X_Domain	= Carbon13
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 31415
X_Prescans	= 4
X_Resolution	= 1.00479569[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Carbon13
Tri_Freq	= 100.52530333[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= TRUE
Scans	= 24000
Total_Scans	= 24000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.5[dC]
X_90_Width	= 11.03[us]
X_Acq_Time	= 0.9952272[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Data_Points	= 32768
X_Points_Default	= 22547
X_Points_Input	= 25132
X_Pulse	= 3.67666667[us]
X_Sweep_Input	= 251.0[ppm]
Irr_Atn_Dec	= 28.666[dB]
Irr_Atn_Dec_Calc	= 28.666[dB]
Irr_Atn_Noe	= 28.666[dB]
Irr_Bandwidth	= 4.7826087[kHz]
Irr_Bandwidth_Ppm	= 11.96303566[ppm]
Irr_Corresp_Pw90	= 0.115[ms]
Irr_Dec_Freq	= 399.78219838[MHz]
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]

S3-1H

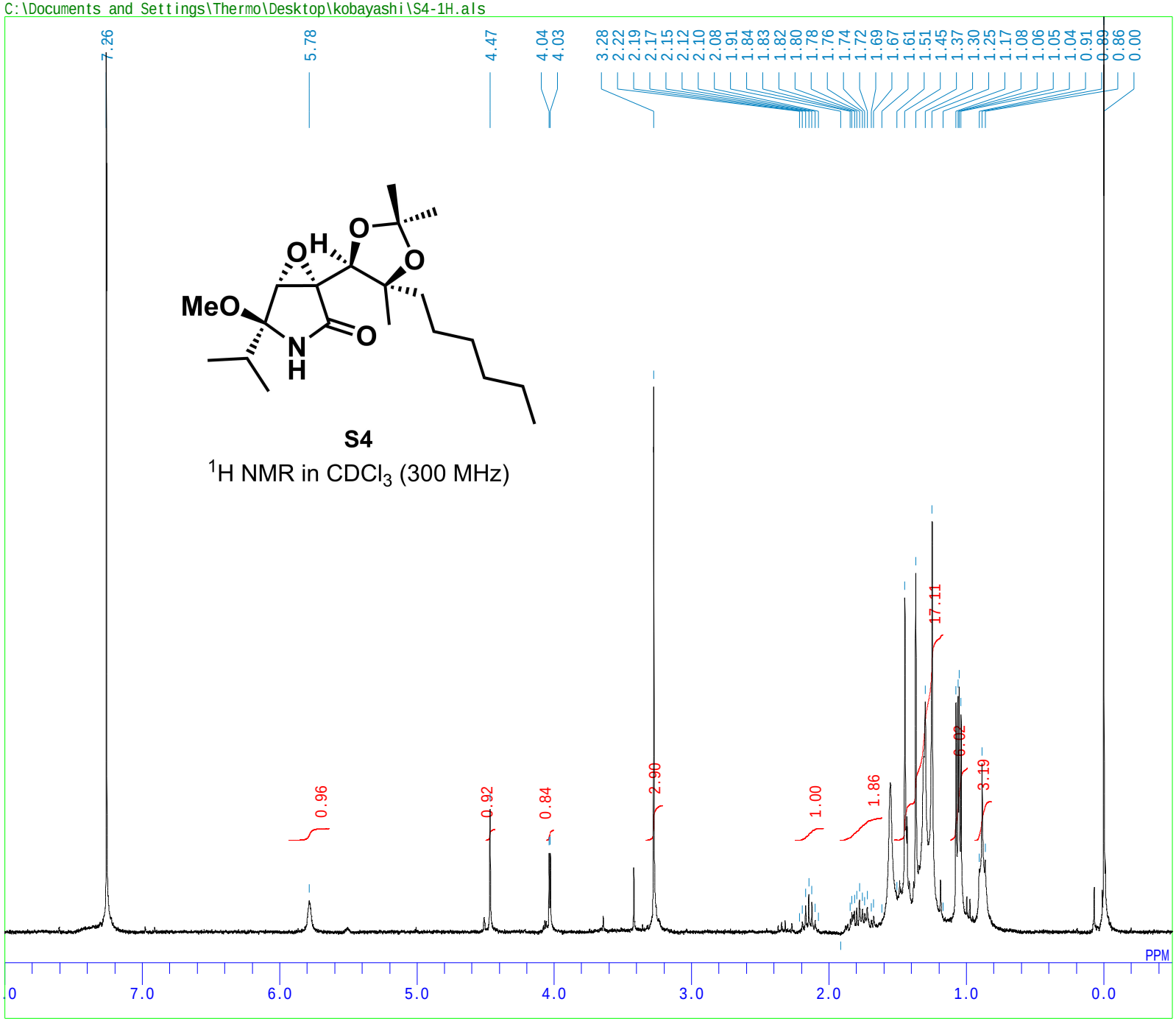
C:\Documents and Settings\Thermo\Desktop\kobayashi\S3-1H.als




```
DFILE  S3-1H.als
COMNT  S3-1H
DATIM  Mon Nov 19 13:15:48 2018
OBNUC  1H
EXMOD  NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN       1150.00 Hz
POINT         32768
FREQU       6006.01 Hz
SCANS           16
ACQTM        5.4559 sec
PD           1.5440 sec
PW1             5.20 usec
IRNUC  1H
CTEMP          23.8 c
SLVNT  CDCL3
EXREF           0.00 ppm
BF              0.12 Hz
RGAIN             22
```

S4-1H

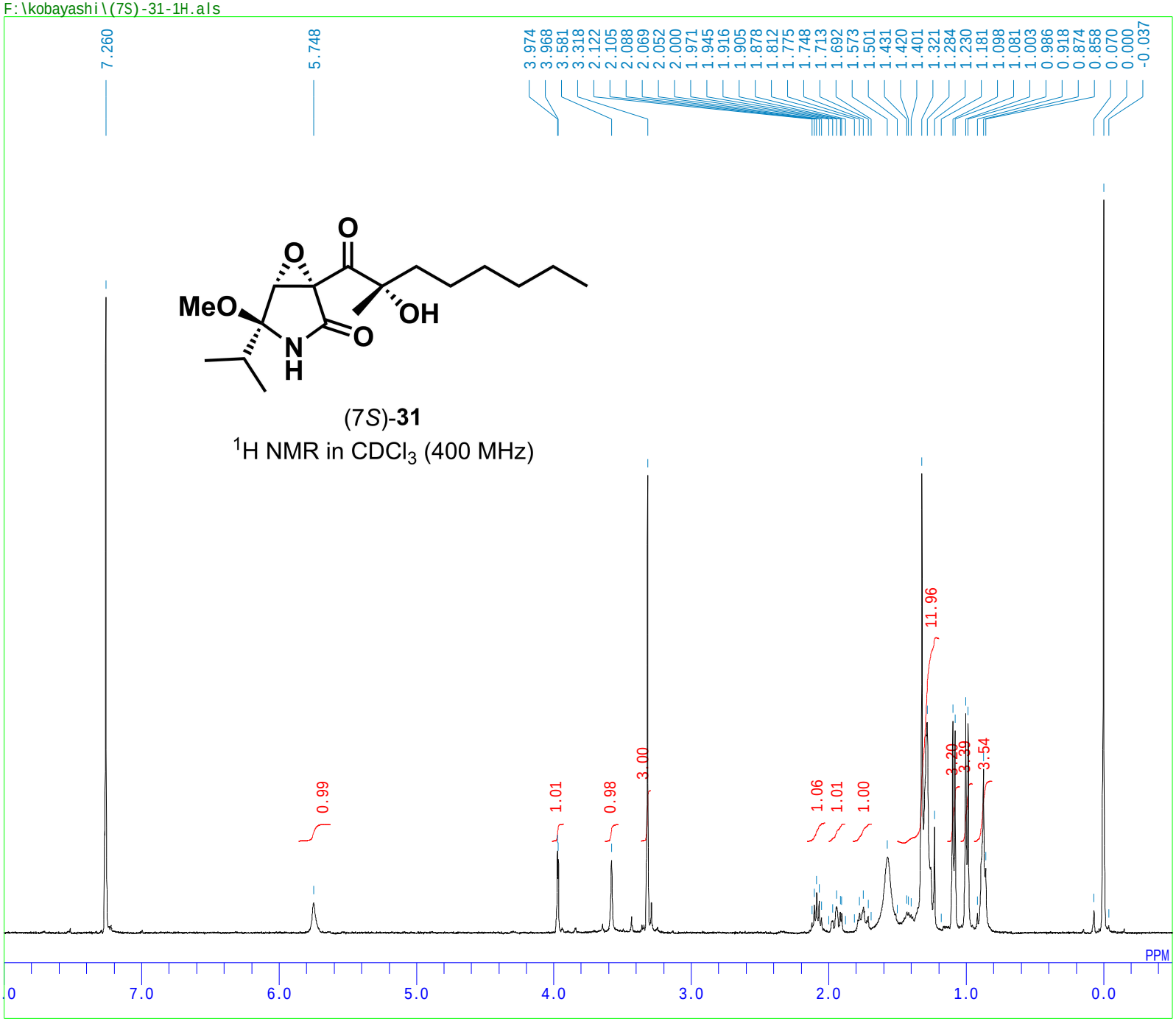
C:\Documents and Settings\Thermo\Desktop\kobayashi\S4-1H.als




```
DFILE  S4-1H.als
COMNT  S4-1H
DATIM  Fri Nov 16 19:28:38 2018
OBNUC  1H
EXMOD  NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN       1150.00 Hz
POINT         32768
FREQU       6006.01 Hz
SCANS           16
ACQTM        5.4559 sec
PD           1.5440 sec
PW1             5.20 usec
IRNUC  1H
CTEMP          25.0 c
SLVNT  CDCL3
EXREF           0.00 ppm
BF              0.12 Hz
RGAIN             21
```

(7S)-31-1H

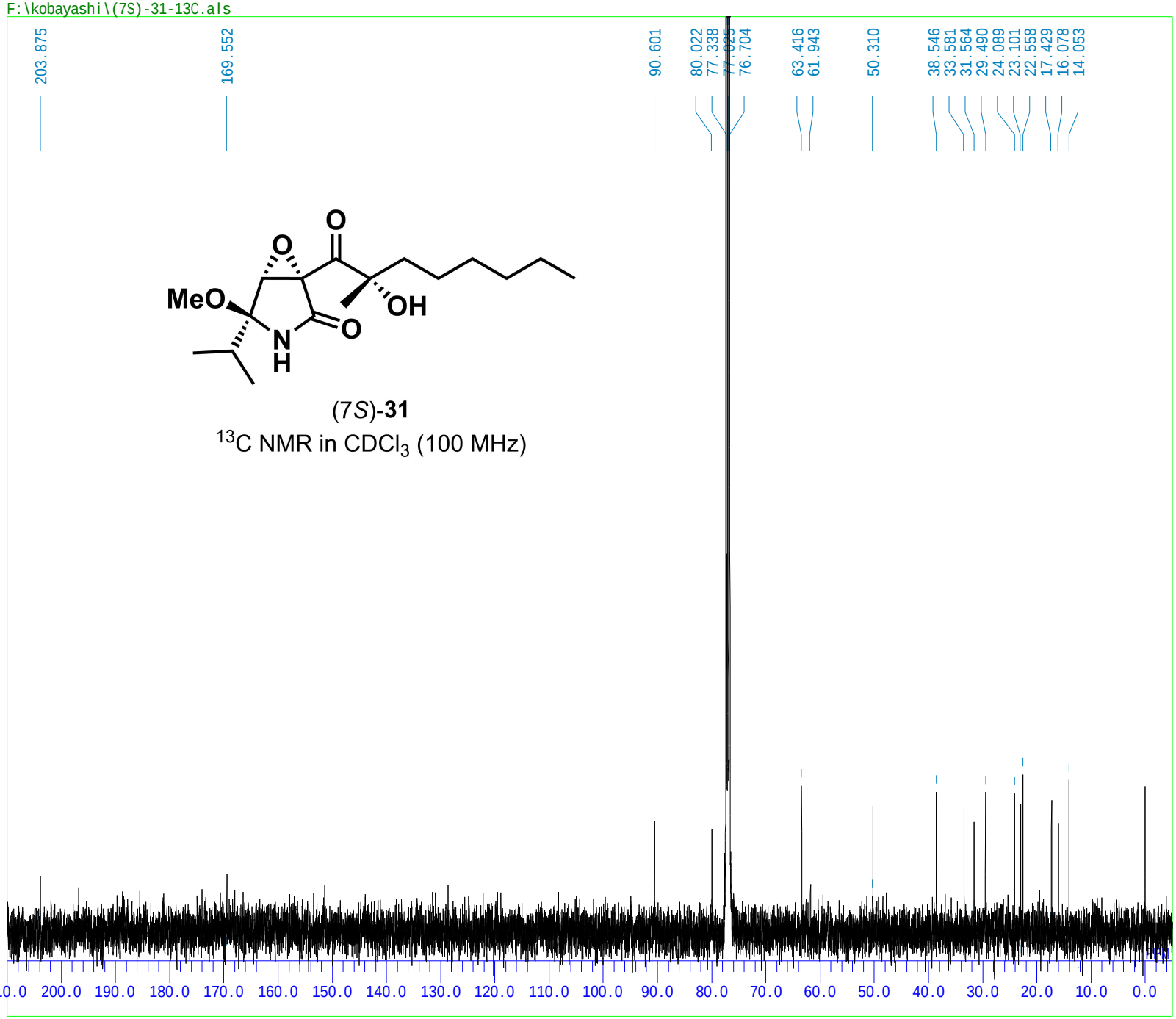
F:\kobayashi\(7S)-31-1H.als



DFILE (7S)-31-1H.als
COMNT (7S)-31-1H
DATIM Mon May 14 11:05:30 2018
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 31
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 24.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

(7S)-31-13C

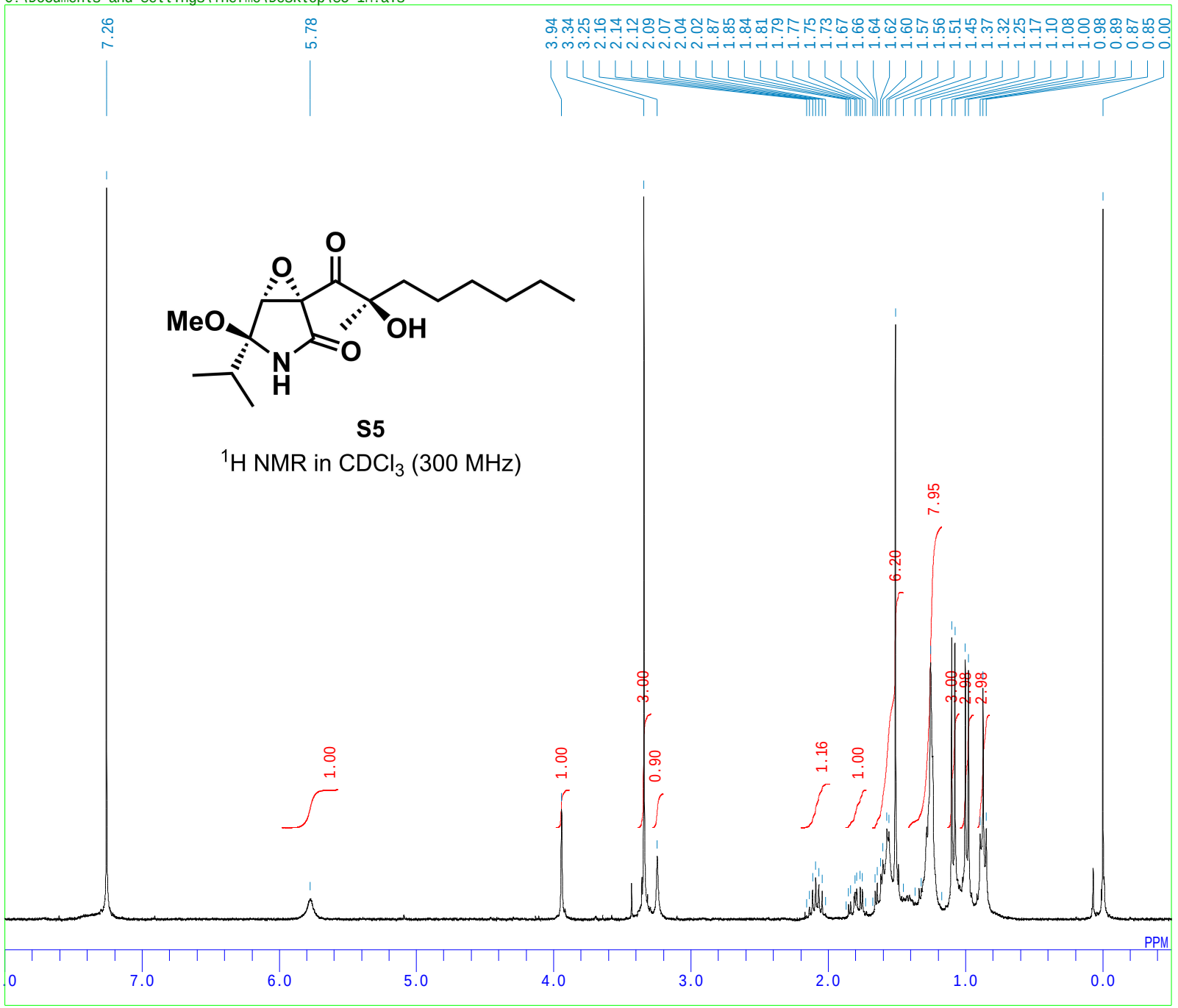
F:\kobayashi\(7S)-31-13C.als




```
DFILE  (7S)-31-13C.als
COMNT  (7S)-31-13C
DATIM  Mon May 14 14:49:26 2018
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         4400
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP         24.4 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            1.20 Hz
RGAIN           25
```

S5-1H

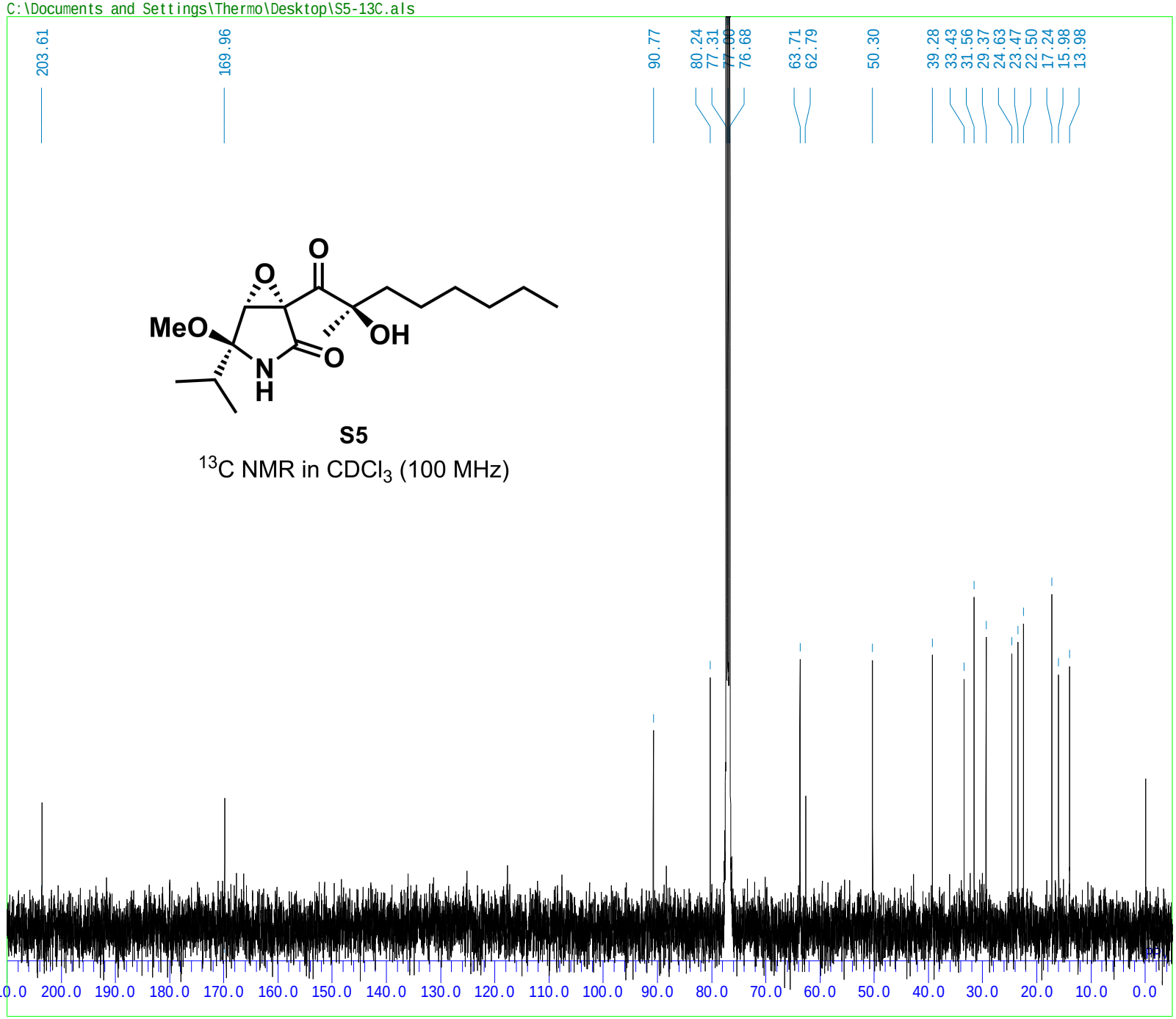
C:\Documents and Settings\Thermo\Desktop\S5-1H.als



DFILE S5-1H.als
COMNT S5-1H
DATIM Tue Apr 10 10:15:30 2018
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 26.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

S5-13C

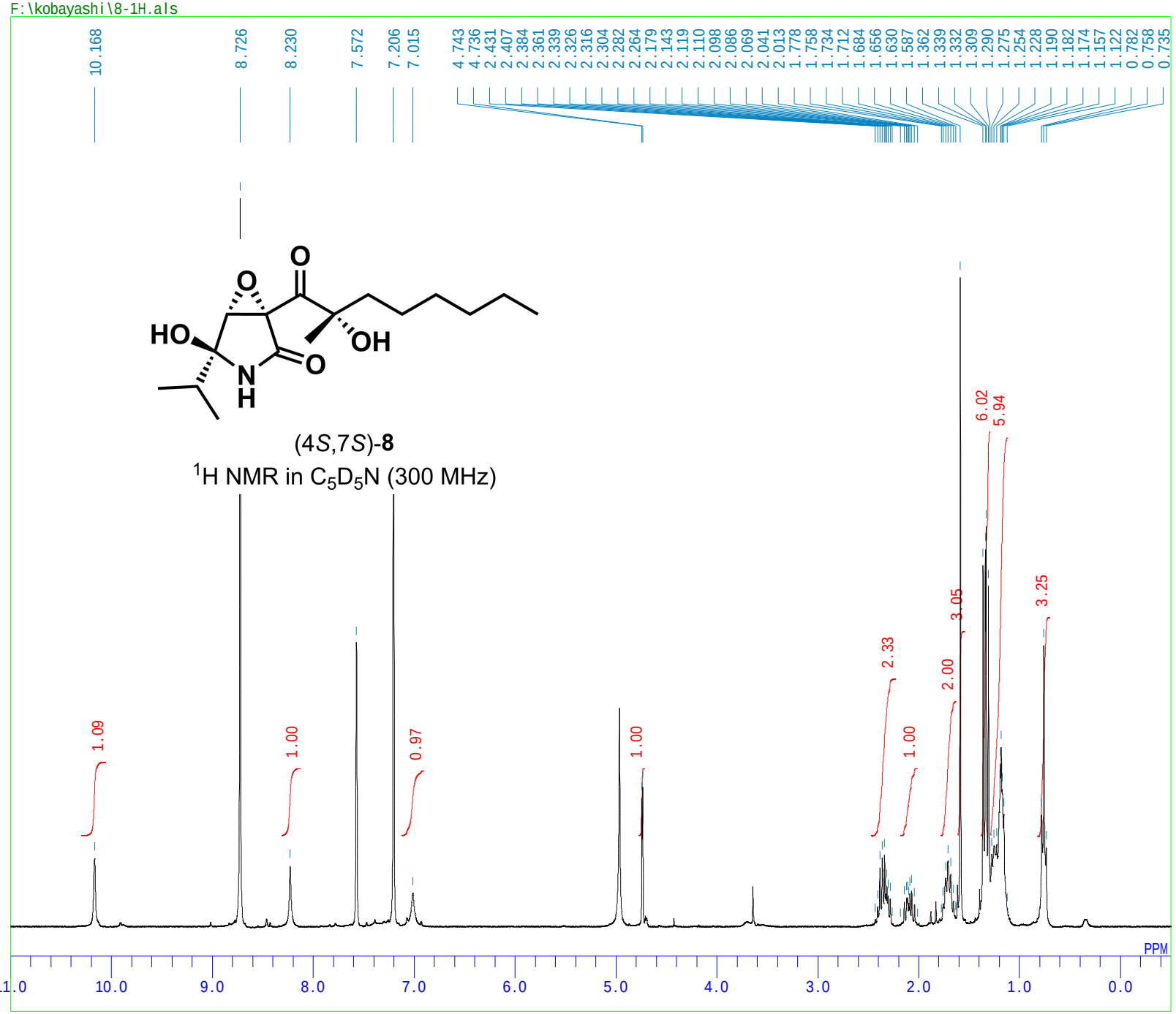
C:\Documents and Settings\Thermo\Desktop\S5-13C.als




```
DFILE  S5-13C.als
COMNT  S5-13C
DATIM  Tue Apr 10 13:15:01 2018
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         3241
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP        24.4 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           25
```

8-1H

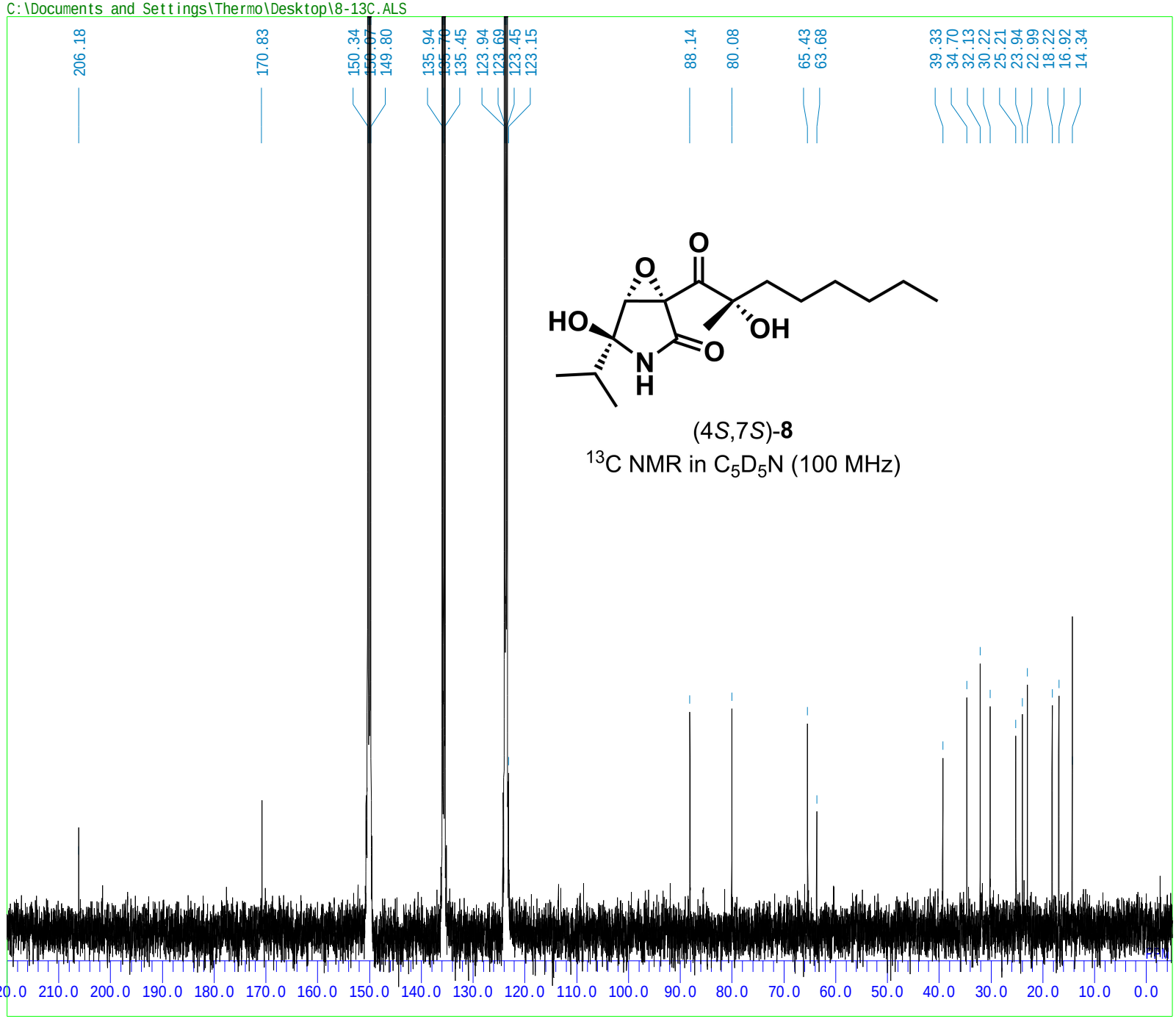
F:\kobayashi\8-1H.als



DFILE 8-1H.als
COMNT 8-1H
DATIM Wed Feb 13 17:55:11 2019
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT C5D5N
EXREF 8.73 ppm
BF 0.12 Hz
RGAIN 17

8-13C

C:\Documents and Settings\Thermo\Desktop\8-13C.ALS



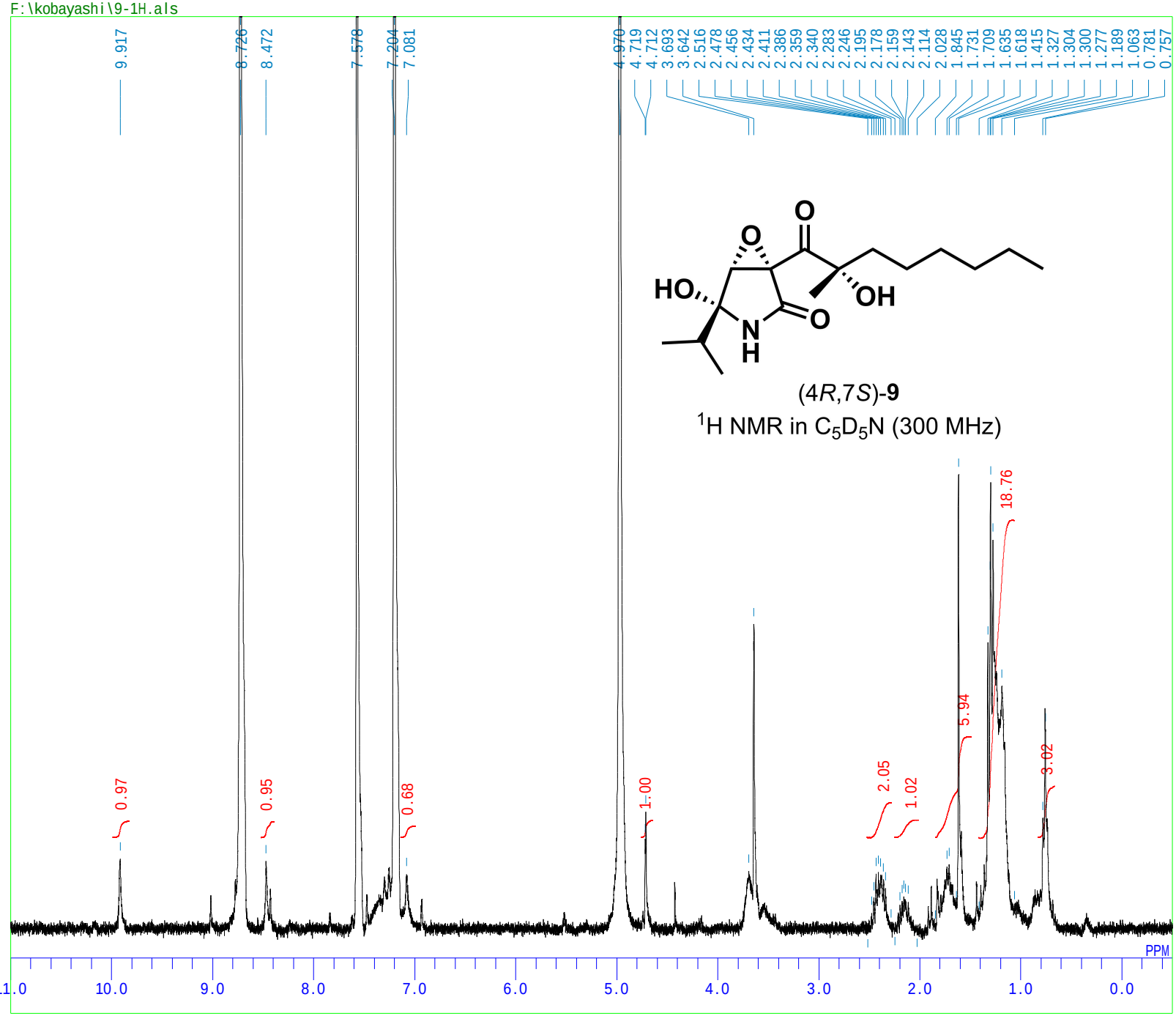
(4*S*,7*S*)-**8**

^{13}C NMR in C_5D_5N (100 MHz)

```
DFILE  8-13C.ALS
COMNT  8-13C
DATIM  Sat Feb 16 11:00:00 2019
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         1949
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP        23.3 c
SLVNT  CDCL3
EXREF       150.07 ppm
BF            1.20 Hz
RGAIN           23
```

9-1H

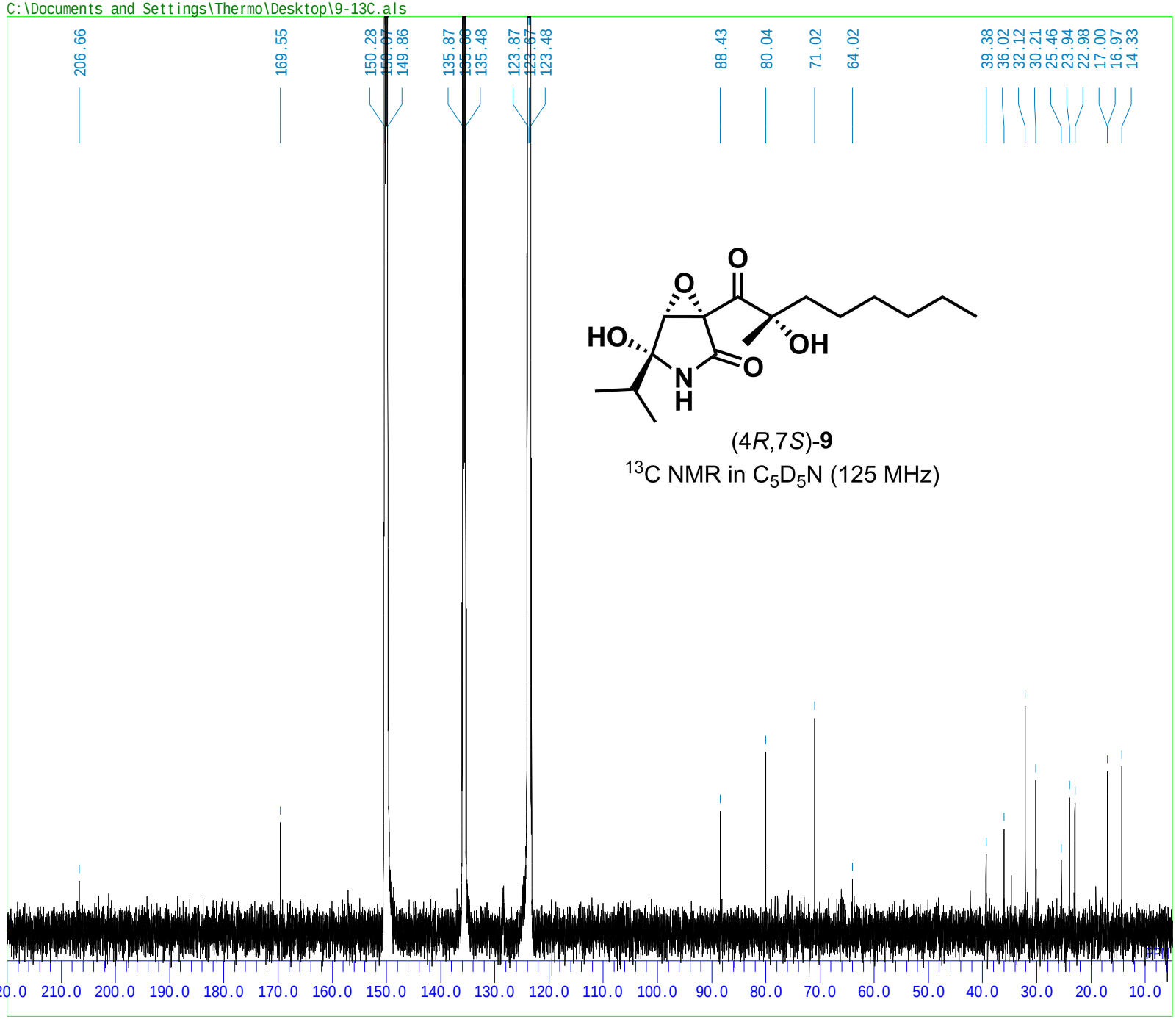
F:\kobayashi\9-1H.als



DFILE 9-1H.als
COMNT 9-1H
DATIM Wed Feb 13 18:01:45 2019
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 23.9 c
SLVNT C5D5N
EXREF 8.73 ppm
BF 0.12 Hz
RGAIN 19

9-13C

C:\Documents and Settings\Thermo\Desktop\9-13C.als



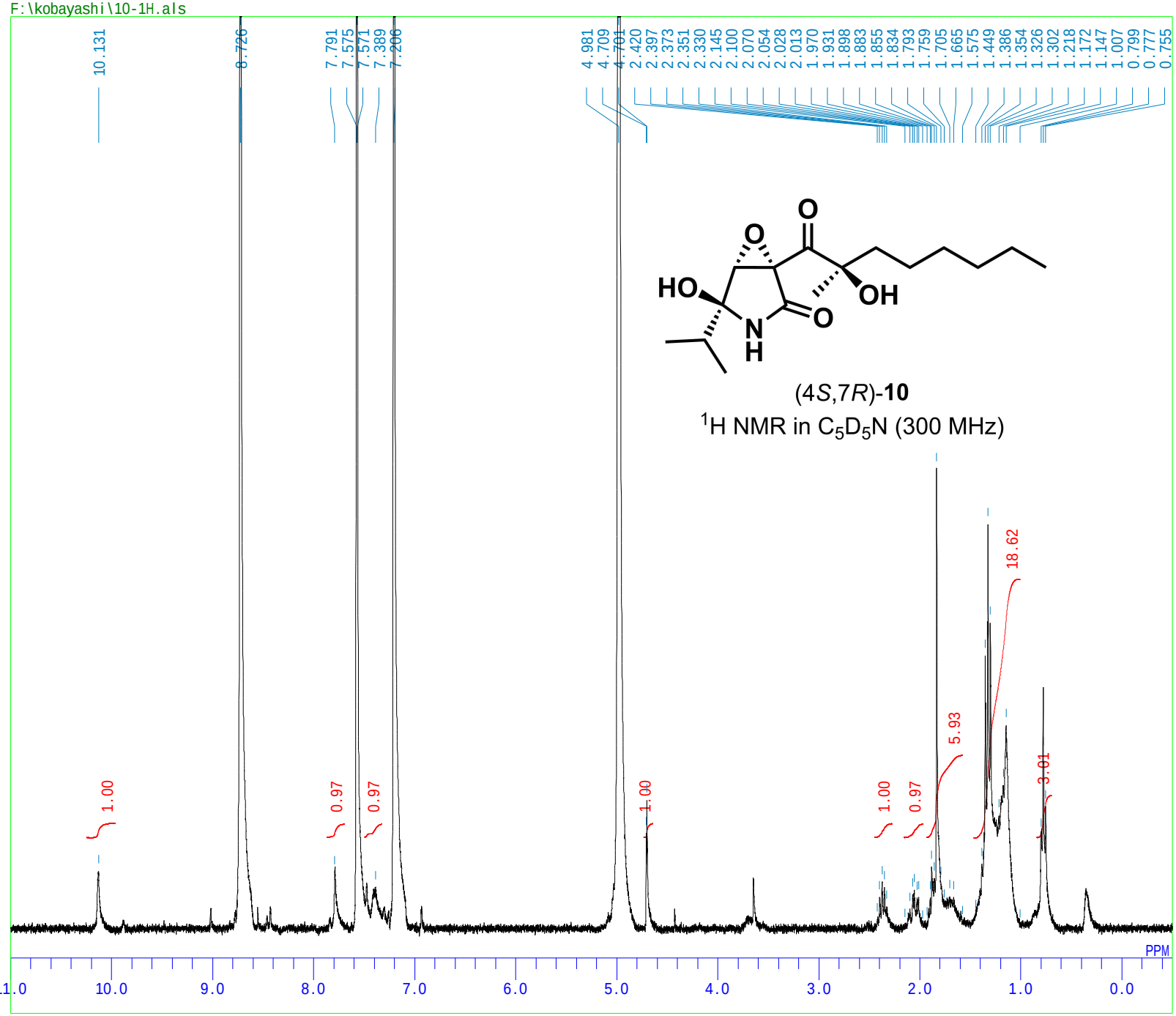
(4*R*,7*S*)-**9**

^{13}C NMR in C_5D_5N (125 MHz)

```
DFILE  9-13C.als
COMNT  9-13C
DATIM  Fri Feb 15 18:53:16 2019
OBNUC  13C
EXMOD  bcm
OBFRQ        125.65 MHz
OBSET        120.00 KHz
OBFIN       7958.00 Hz
POINT         32768
FREQU      33898.30 Hz
SCANS         27000
ACQTM        0.9667 sec
PD           2.0333 sec
PW1             4.90 usec
IRNUC  1H
CTEMP          28.7 c
SLVNT  C5D5N
EXREF        150.07 ppm
BF             0.12 Hz
RGAIN            27
```

10-1H

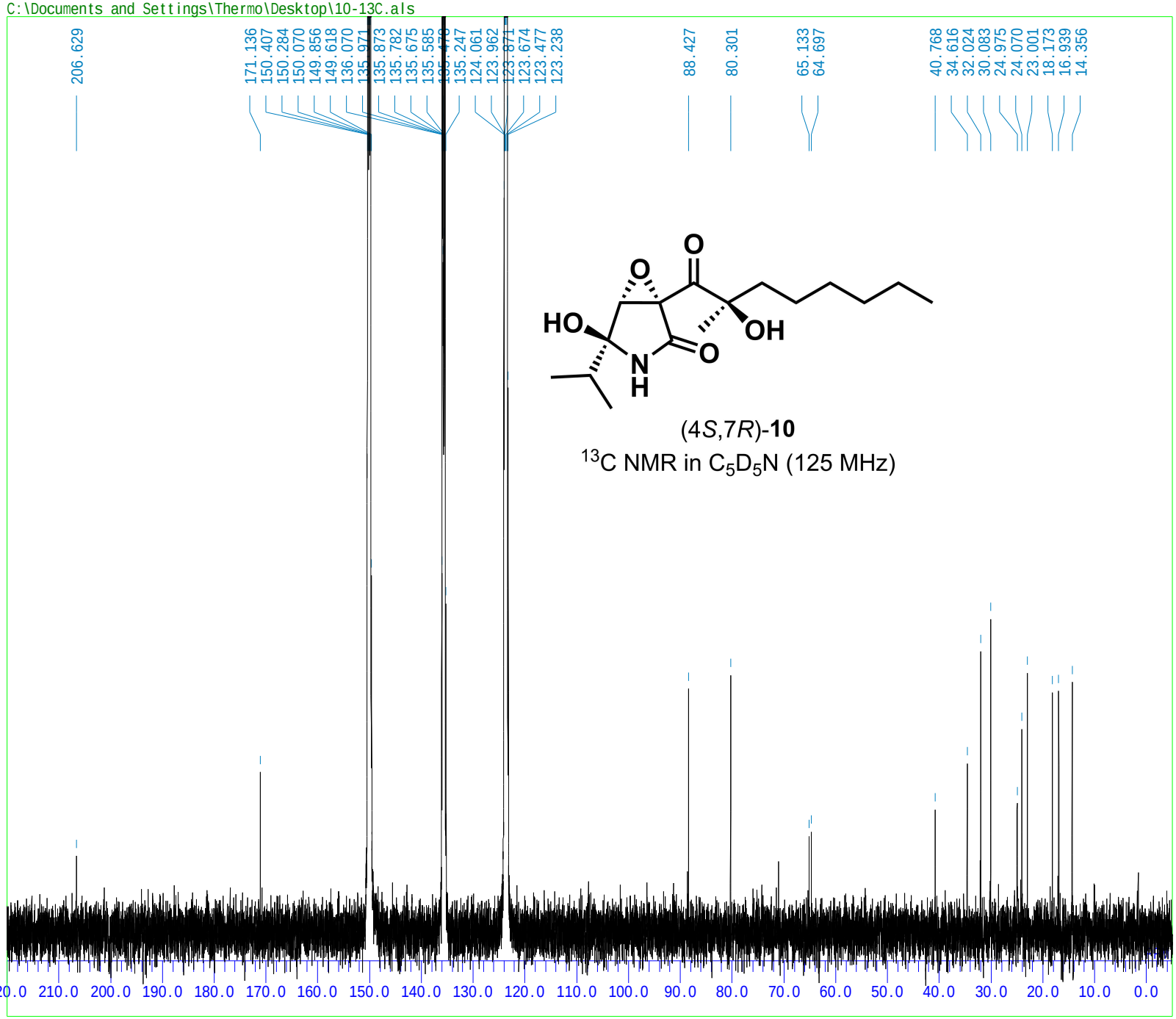
F:\kobayashi\10-1H.als



DFILE 10-1H.als
COMNT 10-1H
DATIM Wed Feb 13 17:47:49 2019
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 25.1 c
SLVNT C5D5N
EXREF 8.73 ppm
BF 0.12 Hz
RGAIN 19

10-13C

C:\Documents and Settings\Thermo\Desktop\10-13C.als



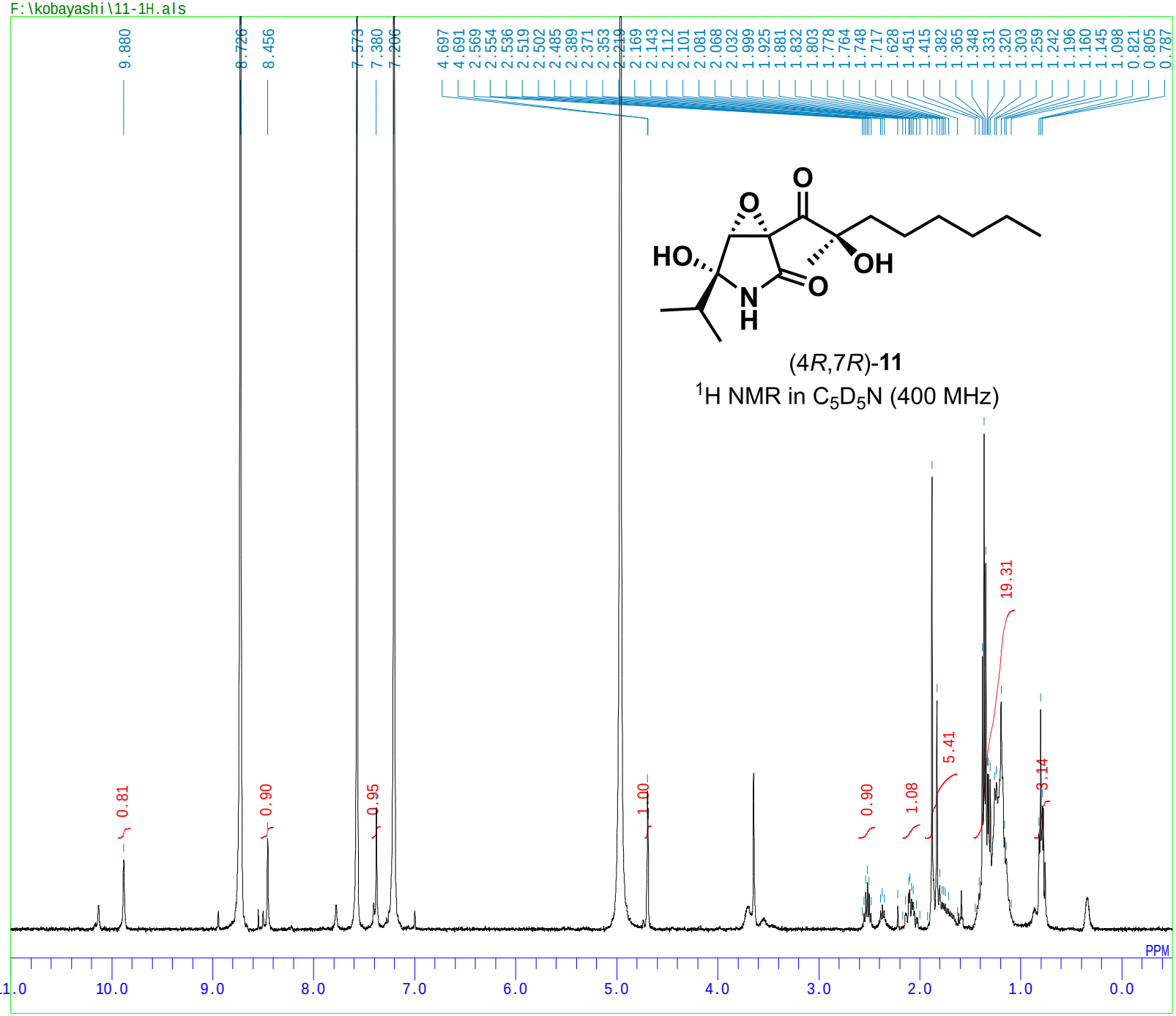
(4*S*,7*R*)-**10**

^{13}C NMR in C_5D_5N (125 MHz)

```
DFILE  10-13C.als
COMNT  10-13C
DATIM  Mon Feb  4 18:23:14 2019
OBNUC  13C
EXMOD  bcm
OBFRQ        125.65 MHz
OBSET        120.00 KHz
OBFIN       7958.00 Hz
POINT         32768
FREQU      33898.30 Hz
SCANS         24000
ACQTM        0.9667 sec
PD           2.0333 sec
PW1            4.90 usec
IRNUC  1H
CTEMP         28.0 c
SLVNT  C5D5N
EXREF       150.07 ppm
BF            0.12 Hz
RGAIN           27
```

11-1H

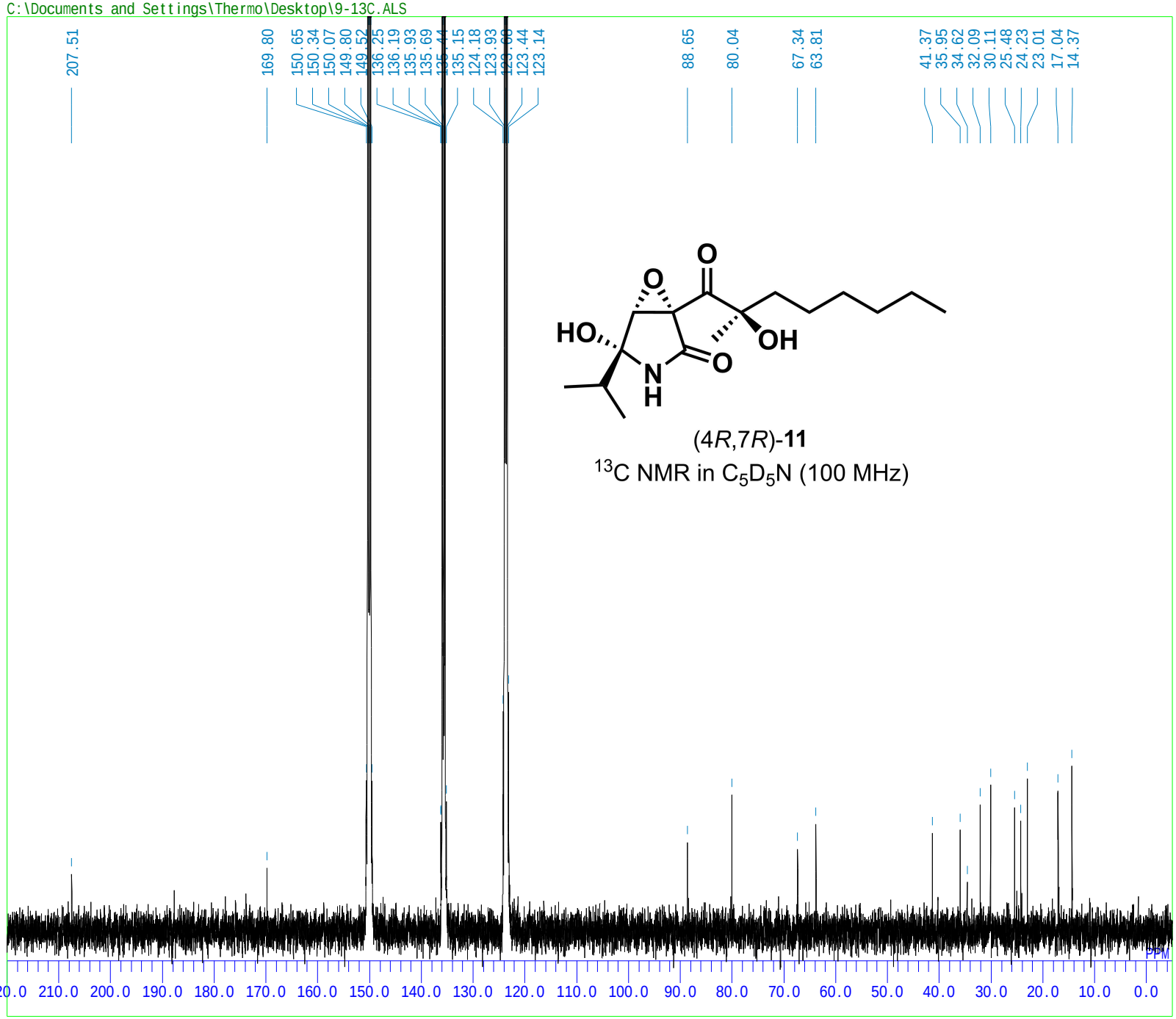
F:\kobayashi\11-1H.als



DFILE 11-1H.als
COMNT 11-1H
DATIM Mon Feb 25 17:23:11 2019
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 23.9 c
SLVNT C5D5N
EXREF 8.73 ppm
BF 0.12 Hz
RGAIN 19

11-13C

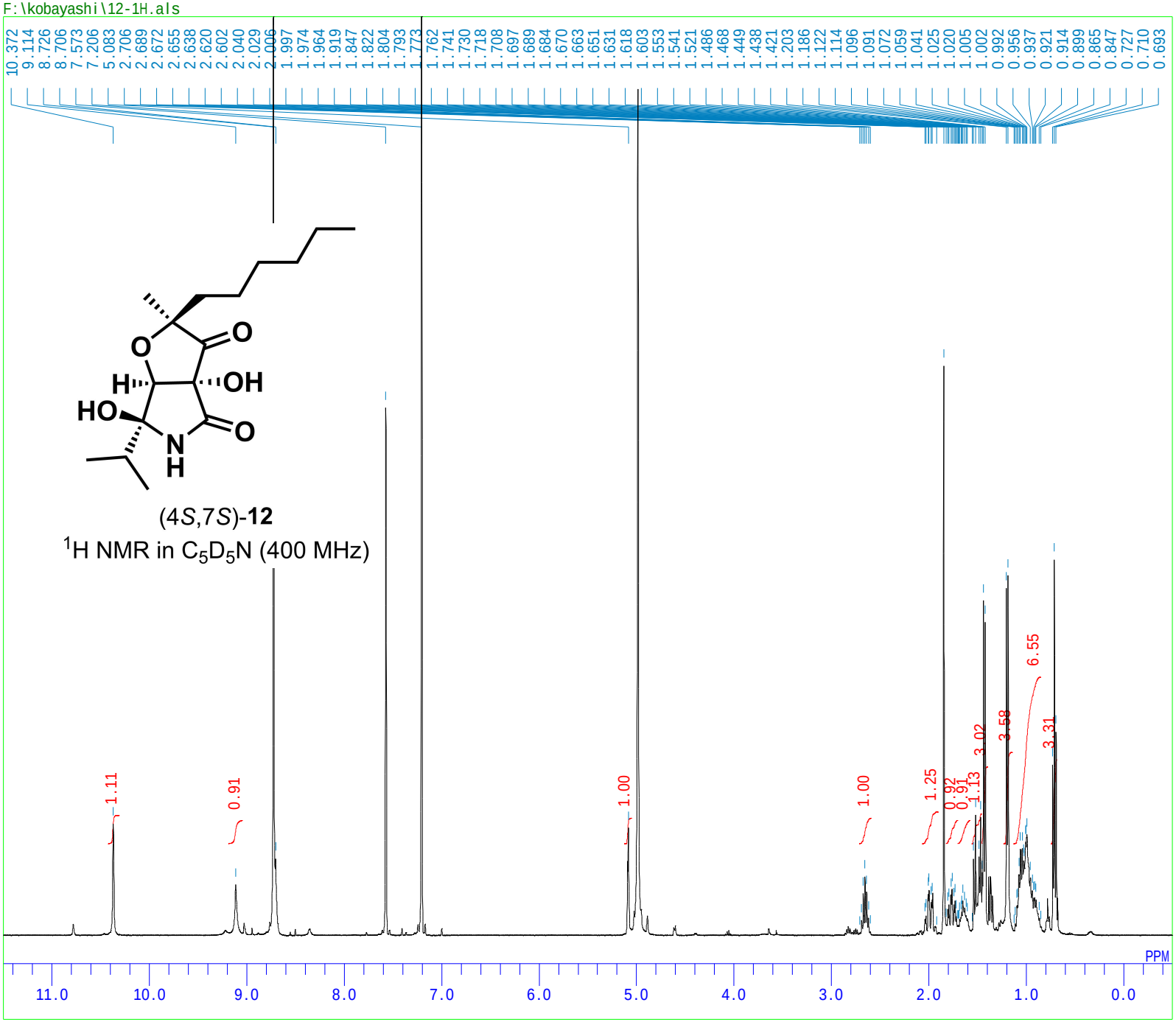
C:\Documents and Settings\Thermo\Desktop\9-13C.ALS



DFILE	9-13C.ALS	
COMNT	11-13C	
DATIM	Sat Feb 16 19:21:32 2019	
OBNUC	13C	
EXMOD	BCM	
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	9889	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	23.3	c
SLVNT	C5D5N	
EXREF	150.07	ppm
BF	1.20	Hz
RGAIN	23	

12-1H

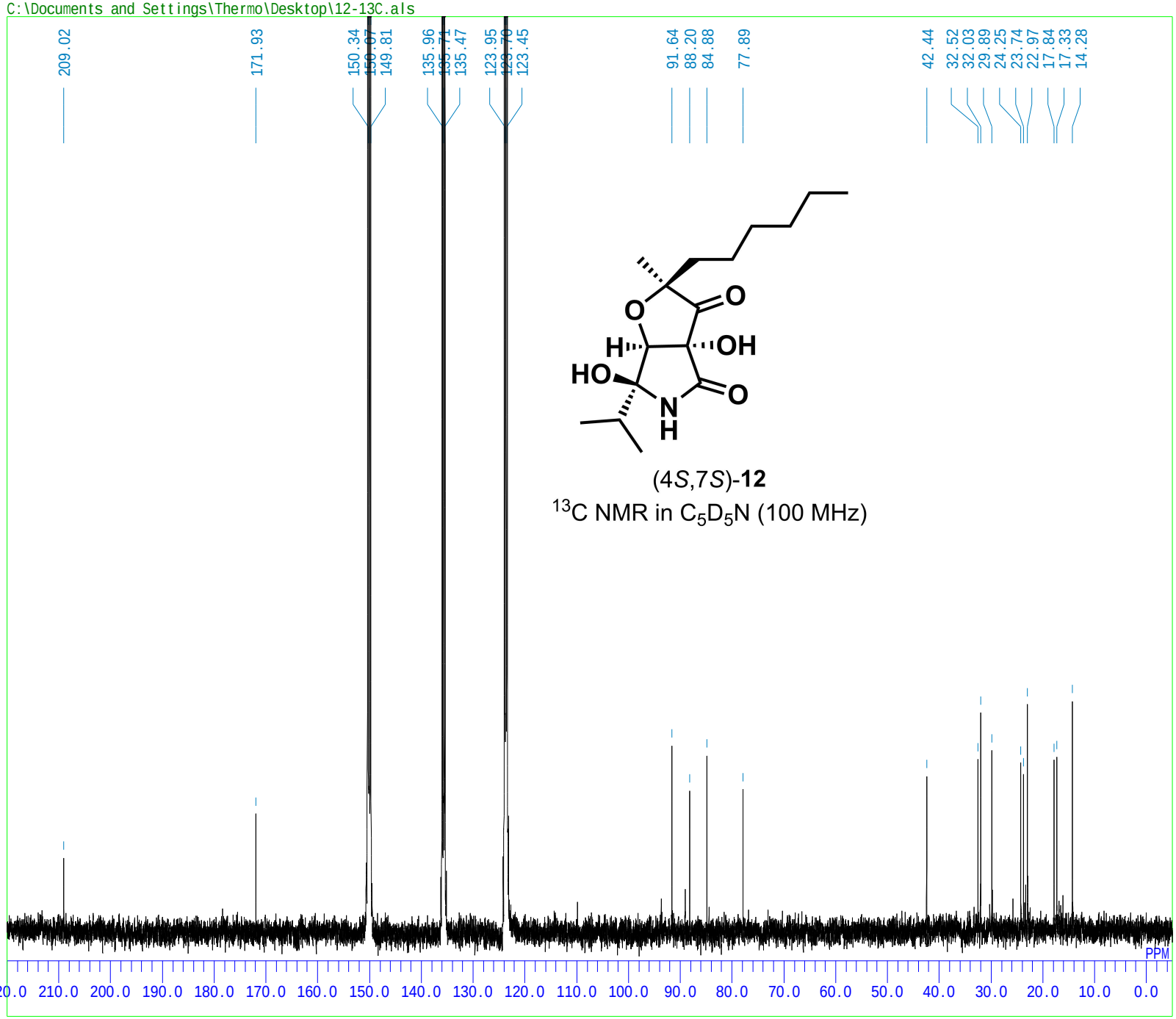
F:\kobayashi\12-1H.als



DFILE 12-1H.als
COMNT 12-1H
DATIM Mon Mar 05 15:05:29 2018
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 64
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT C5D5N
EXREF 0.71 ppm
BF 0.12 Hz
RGAIN 19

12-13C

C:\Documents and Settings\Thermo\Desktop\12-13C.als



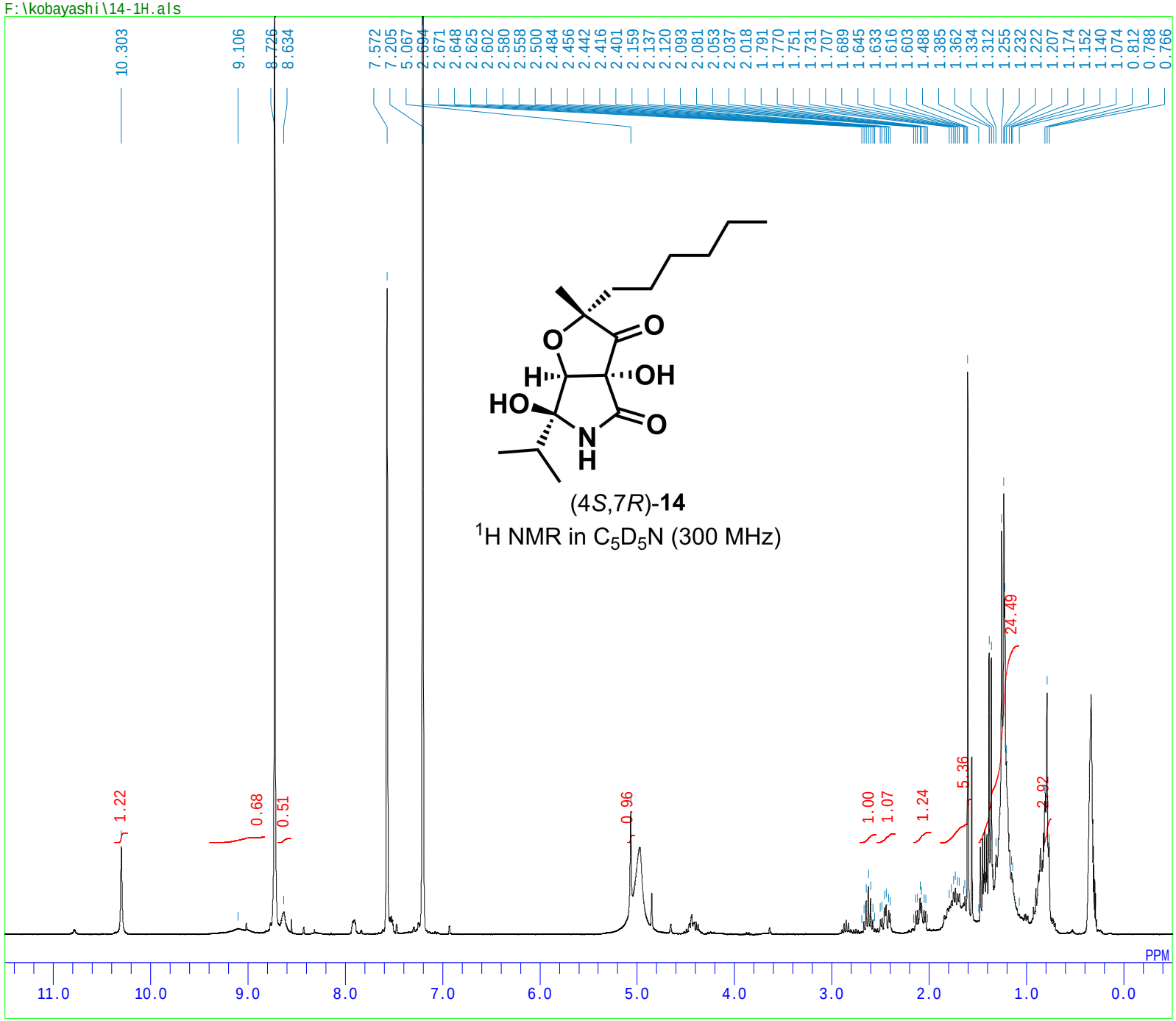
(4*S*,7*S*)-**12**

^{13}C NMR in C_5D_5N (100 MHz)

```
DFILE  12-13C.als
COMNT  12-13C
DATIM  Mon Mar 05 16:50:51 2018
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         2000
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP         22.1 c
SLVNT  C5D5N
EXREF       150.07 ppm
BF            1.20 Hz
RGAIN           22
```

14-1H

F:\kobayashi\14-1H.als



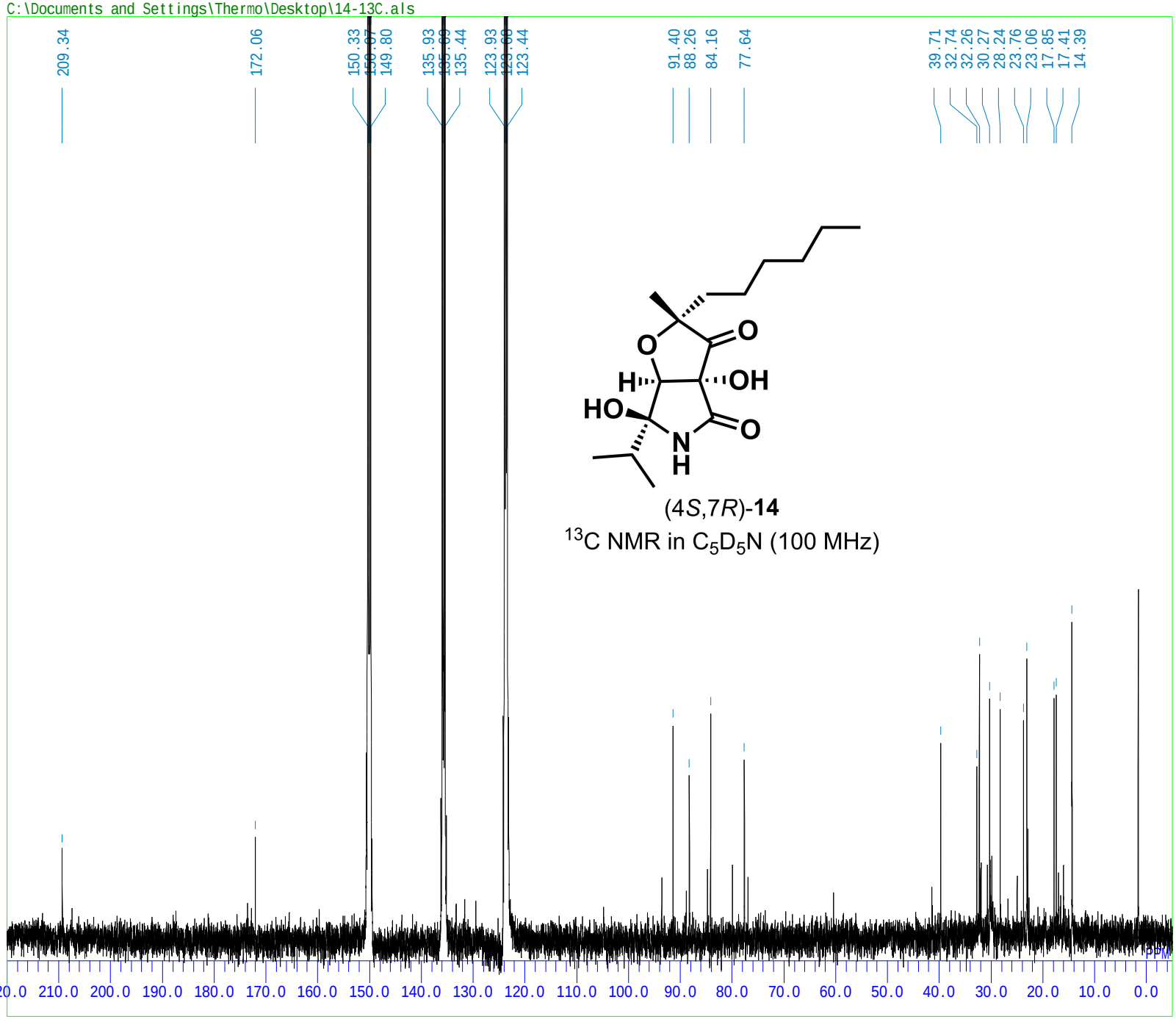
(4*S*,7*R*)-**14**

^{1}H NMR in C_5D_5N (300 MHz)

```
DFILE  14-1H.als
COMNT  14-1H
DATIM  Wed Apr 11 15:47:07 2018
OBNUC  1H
EXMOD  NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN       1150.00 Hz
POINT         32768
FREQU       6006.01 Hz
SCANS           58
ACQTM        5.4559 sec
PD           1.5440 sec
PW1             5.20 usec
IRNUC  1H
CTEMP          23.4 c
SLVNT  C5D5N
EXREF           8.73 ppm
BF              0.12 Hz
RGAIN             17
```

14-13C

C:\Documents and Settings\Thermo\Desktop\14-13C.als




```
DFILE  14-13C.als
COMNT  14-13C
DATIM  Sat Apr 07 20:51:06 2018
OBNUC  13C
EXMOD  BCM
OBFRQ        100.40 MHz
OBSET        125.00 KHz
OBFIN      10500.00 Hz
POINT        32768
FREQU      27118.64 Hz
SCANS         5383
ACQTM         1.2083 sec
PD            1.7920 sec
PW1           5.80 usec
IRNUC  1H
CTEMP         24.0 c
SLVNT  C5D5N
EXREF       150.07 ppm
BF            1.20 Hz
RGAIN           23
```