Data-driven catalyst optimization for stereodivergent asymmetric synthesis of α-allyl carboxylic acids by iridium/boron hybrid catalysis

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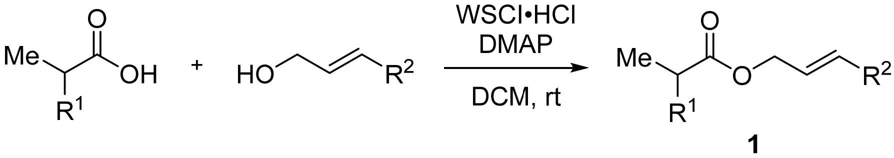
1. General Methods

^{1}H and ^{13}C NMR spectra were recorded on a JEOL ECX500 (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR) or a JEOL ECS400 (400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR) spectrometer. Chemical shifts were reported in the scale relative to the solvent used as an internal reference for ^{1}H (δ = 7.26 ppm for $CDCl_3$) and ^{13}C NMR (δ = 77.00 ppm for $CDCl_3$). Electrospray ionization (ESI)-mass spectra were measured on a JEOL JMS-T100LC AccuTOF spectrometer for HRMS. Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. The enantiomeric excesses (ee's) were determined by HPLC analysis conducted by JASCO HPLC systems (pump: PU-2080; detector: UV-2075, measured at 254 nm or at 210 nm; chiral column OZ-H, IG, IB N-3, IB N-5, IC-3; mobile phase: hexane/2-propanol). Optical rotations were measured on a JASCO P-1010 polarimeter. Column chromatographies were performed with silica gel Merck 60 (230-400 mesh ASTM) or neutral silica gel 60 N (KANTO CHEMICAL, spherical, neutral, 40-100 μm), Biotage Isolera One and Biotage SNAP Ultra, or Yamazen Smart Flash and Universal Column Premium. DBU was purified by distillation from CaH_2 and used under argon atmosphere. LiCl was dried under vacuum at 80 °C for 24 h and used under argon atmosphere. Reagents that were not further described were purchased from Aldrich, Tokyo Chemical Industry Co., Ltd. (TCI), Kanto Chemical Co., Inc., and Wako Pure Chemical Industries, Ltd. and used without further purification.

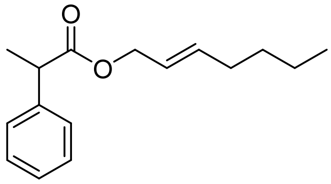
2. Synthesis of Substrates

Procedure for preparation of 1a, 1d, 1e, 1p-w, 1y



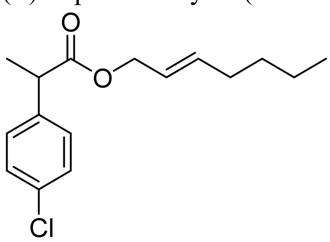
In a flame-dried flask, a solution of carboxylic acid (1.2 equiv) in DCM (ca. 1 M) was cooled to 0 °C. Alcohol (1.0 equiv), WSCI•HCl (1.2 equiv) and DMAP (0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford pure product **1**.

(*E*)-hept-2-en-1-yl 2-phenylpropanoate (**1a**)



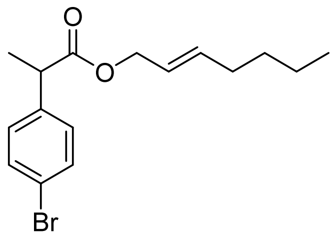
Yield: 80%
^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.2 Hz, 3H), 1.25-1.36 (m, 4H), 1.50 (d, *J* = 7.0 Hz, 3H), 2.02 (dt, *J* = 6.9, 6.9 Hz, 2H), 3.73 (q, *J* = 7.0 Hz, 1H), 4.46-4.55 (m, 2H), 5.47-5.53 (m, 1H), 5.65-5.71 (m, 1H), 7.23-7.28 (m, 1H), 7.30-7.32 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.9, 18.6, 22.1, 31.0, 31.9, 45.5, 65.4, 123.6, 127.0, 127.5, 128.5, 136.4, 140.6, 174.3; IR (neat): 2930, 1736, 1455, 1165, 972, 698 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_2$ $[M+Na]^+$ 269.1512. Found 269.1525.

(*E*)-hept-2-en-1-yl 2-(4-chlorophenyl)propanoate (**1d**)



Yield: 54%
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 7.3 Hz, 3H), 1.19-1.35 (m, 4H), 1.46 (d, *J* = 7.3 Hz, 3H), 2.00 (dt, *J* = 7.3, 7.3 Hz, 2H), 3.68 (q, *J* = 7.3 Hz, 1H), 4.44-4.53 (m, 2H), 5.43-5.50 (m, 1H), 5.63-5.70 (m, 1H), 7.21-7.28 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 18.5, 22.1, 30.9, 31.8, 44.9, 65.6, 123.4, 128.6, 128.9, 132.9, 136.6, 139.0, 173.8; IR (neat): 2930, 1736, 1493, 1164, 972, 834 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115.

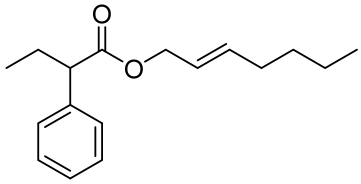
(*E*)-hept-2-en-1-yl 2-(4-bromophenyl)propanoate (**1e**)



Yield: 69%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.1 Hz, 3H), 1.23-1.36 (m, 4H), 1.47 (d, *J* = 7.3 Hz, 3H), 2.01 (dt, *J* = 6.9, 6.9 Hz, 2H), 3.68 (q, *J* = 7.3 Hz, 1H), 4.45-4.54 (m, 2H), 5.45-5.52 (m, 1H), 5.64-5.72 (m, 1H), 7.18 (d, *J* = 8.7 Hz, 2H), 7.44 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 18.4, 22.1, 30.9, 31.8, 45.0, 65.6, 121.0, 123.4, 129.3, 131.6, 136.7, 139.5, 173.8; IR (neat): 2929, 1735, 1489, 1165, 972, 831 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}BrO_2$ $[M+Na]^+$ 347.0617. Found 347.0615.

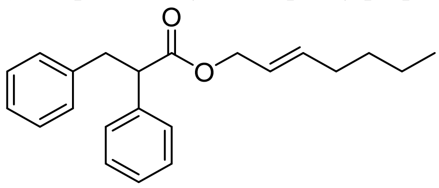
(*E*)-hept-2-en-1-yl 2-phenylbutanoate (**1p**)



Yield: 70%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.82-0.87 (m, 6H), 1.19-1.30 (m, 4H), 1.70-1.81 (m, 1H), 1.94-2.11 (m, 3H), 3.41 (t, *J* = 7.8 Hz, 1H), 4.40-4.52 (m, 2H), 5.42-5.49 (m, 1H), 5.60-5.68 (m, 1H), 7.18-7.27 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 12.1, 13.9, 22.1, 26.8, 30.9, 31.8, 53.5, 65.3, 123.6, 127.1, 128.0, 128.5, 136.4, 139.1, 173.8; IR (neat): 2961, 1734, 1455, 1162, 970, 698 cm^{-1}; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1665.

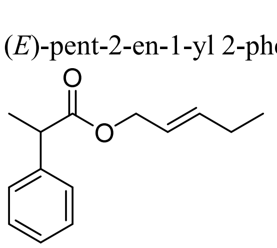
(*E*)-hept-2-en-1-yl 2,3-diphenylpropanoate (**1q**)



Yield: 62%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.84 (t, *J* = 7.3 Hz, 3H), 1.20-1.28 (m, 4H), 1.93 (dt, *J* = 6.7, 6.7 Hz, 2H), 2.98 (dd, *J* = 6.9, 13.7 Hz, 1H), 3.37 (dd, *J* = 9.2, 13.7 Hz, 1H), 3.82 (dd, *J* = 6.9, 9.2 Hz, 1H), 4.38-4.46 (m, 2H), 5.33-5.39 (m, 1H), 5.52-5.58 (m, 1H), 7.07-7.29 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 22.1, 30.9, 31.8, 39.8, 53.7, 65.5, 123.4, 126.3, 127.3, 128.0, 128.3, 128.6, 129.0, 136.4, 138.7, 139.0, 173.1; IR (neat): 3030, 2927, 1734, 1455, 1150, 971, 698 cm^{-1}; HRMS (ESI): m/z calcd for $C_{22}H_{26}O_2$ $[M+Na]^+$ 345.1825. Found 345.1832.

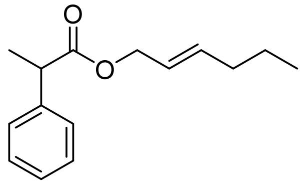
(*E*)-pent-2-en-1-yl 2-phenylpropanoate (**1r** [**1Et**])



Yield: 89%

^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.97 (t, *J* = 7.4 Hz, 3H), 1.50 (d, *J* = 7.1 Hz, 3H), 2.01-2.06 (m, 2H), 3.73 (q, *J* = 7.1 Hz, 1H), 4.46-4.50 (m, 1H), 4.52-4.56 (m, 1H), 5.48-5.51 (m, 1H), 5.70-5.76 (m, 1H), 7.23-7.27(m, 1H), 7.28-7.34 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.1, 18.6, 25.2, 45.5, 65.5, 122.6, 127.0, 127.5, 128.5, 137.8, 140.6, 174.3; IR (neat): 2968, 1734, 1455, 1166, 969, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{14}H_{18}O_2$ [M+Na]$^+$ 241.1199. Found 241.1190.

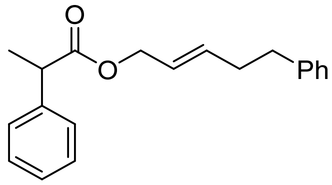
(*E*)-hex-2-en-1-yl 2-phenylpropanoate (**1s** [**1Pr**])



Yield: 85%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.82 (t, *J* = 7.4 Hz, 3H), 1.32 (tq, *J* = 7.4, 7.4 Hz, 2H), 1.45 (d, *J* = 7.3 Hz, 3H), 1.94 (dt, *J* = 7.4, 7.4 Hz, 2H), 3.68 (q, *J* = 7.3 Hz, 1H), 4.41-4.51 (m, 2H), 5.41-5.49 (m, 1H), 5.59-5.67 (m, 1H), 7.18-7.22 (m, 1H), 7.25-7.27 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.6, 18.6, 22.0, 34.2, 45.5, 65.5, 123.8, 127.0, 127.5, 128.5, 136.2, 140.6, 174.3; IR (neat): 2959, 1735, 1455, 1165, 972, 698 cm^{-1}; HRMS (ESI): m/z calcd for $C_{15}H_{20}O_2$ [M+Na]$^+$ 255.1356. Found 255.1365.

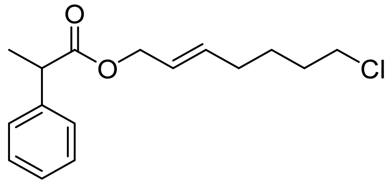
(*E*)-5-phenylpent-2-en-1-yl 2-phenylpropanoate (**1t**)



Yield: 72%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.48 (d, *J* = 7.3 Hz, 3H), 2.32 (dt, *J* = 7.3, 7.3 Hz, 2H), 2.64 (t, *J* = 8.0 Hz, 2H), 3.71 (q, *J* = 7.3 Hz, 1H), 4.43-4.54 (m, 2H), 5.48-5.55 (m, 1H), 5.66-5.74 (m, 1H), 7.13-7.19 (m, 3H), 7.24-7.33 (m, 7H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 18.6, 34.0, 35.3, 45.5, 65.2, 124.3, 125.9, 127.1, 127.5, 128.3, 128.4, 128.6, 135.0, 140.5, 141.5, 174.3; IR (neat): 2931, 1733, 1456, 1163, 1075, 698 cm^{-1}; HRMS (ESI): m/z calcd for $C_{20}H_{22}O_2$ [M+Na]$^+$ 317.1512. Found 317.1497.

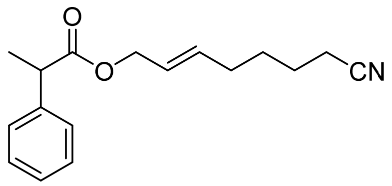
(*E*)-7-chlorohept-2-en-1-yl 2-phenylpropanoate (**1u**)



Yield: 75%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.41-1.49 (m, 5H), 1.65-1.72 (m, 2H), 2.00 (dt, *J* = 6.9, 6.9 Hz, 2H), 3.47 (t, *J* = 6.9 Hz, 2H), 3.68 (q, *J* = 7.3 Hz, 1H), 4.41-4.51 (m, 2H), 5.43-5.51 (m, 1H), 5.56-5.63 (m, 1H), 7.18-7.23 (m, 1H), 7.24-7.30 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.6, 26.0, 31.3, 31.9, 44.8, 45.5, 65.2, 124.4, 127.1, 127.5, 128.6, 135.2, 140.6, 174.2; IR (neat): 2937, 1733, 1454, 1165, 971, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ [M+Na]$^+$ 303.1122. Found 303.1127.

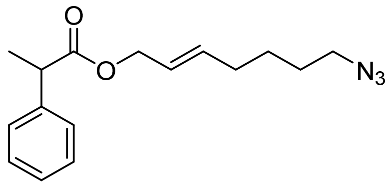
(*E*)-7-cyanohept-2-en-1-yl 2-phenylpropanoate (**1v**)



Yield: 34%

^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.48-1.64 (m, 7H), 2.06 (dt, J = 6.9, 6.9 Hz, 2H), 2.32 (t, J = 7.2 Hz, 2H), 3.73 (q, J = 7.1 Hz, 1H), 4.47-4.54 (m, 2H), 5.49-5.55 (m, 1H), 5.58-5.64 (m, 1H), 7.24-7.34 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 16.9, 18.5, 24.6, 27.6, 31.2, 45.5, 65.0, 119.6, 124.8, 127.1, 127.5, 128.5, 134.4, 140.5, 174.2; IR (neat): 3030, 2936, 2246, 1733, 1454, 1166, 1076, 973, 700 cm^{-1}; HRMS (ESI): m/z calcd for $C_{17}H_{21}NO_2$ $[M+Na]^+$ 294.1465. Found 294.1476.

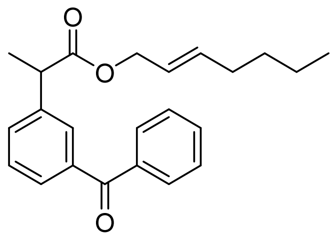
(*E*)-7-azidohept-2-en-1-yl 2-phenylpropanoate (**1w**)



Yield: 62%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34-1.42 (m, 2H), 1.45 (d, J = 7.3 Hz, 3H), 1.48-1.55 (m, 2H), 2.00 (dt, J = 7.2, 7.2 Hz, 2H), 3.20 (t, J = 7.2 Hz, 2H), 3.68 (q, J = 7.3 Hz, 1H), 4.41-4.51 (m, 2H), 5.43-5.50 (m, 1H), 5.56-5.63 (m, 1H), 7.18-7.30 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.5, 25.8, 28.1, 31.6, 45.5, 51.2, 65.1, 124.4, 127.0, 127.5, 128.5, 135.1, 140.6, 174.2; IR (neat): 2934, 2096, 1733, 1453, 1165, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}N_3O_2$ $[M+Na]^+$ 310.1526. Found 310.1524.

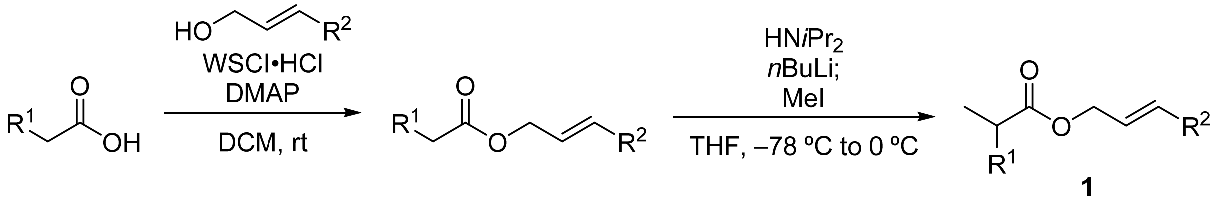
(*E*)-hept-2-en-1-yl 2-(3-benzoylphenyl)propanoate (**1y**)



Yield: 85%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, J = 7.3 Hz, 3H), 1.26-1.34 (m, 4H), 1.53 (d, J = 7.3 Hz, 3H), 2.01 (dt, J = 6.7, 6.7 Hz, 2H), 3.80 (q, J = 7.3 Hz, 1H), 4.48-4.54 (m, 2H), 5.48-5.53 (m, 1H), 5.66-5.72 (m, 1H), 7.42-7.50 (m, 3H), 7.54-7.61 (m, 2H), 7.67-7.69 (m, 1H), 7.75-7.81 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.9, 18.5, 22.1, 30.9, 31.9, 45.4, 65.7, 123.4, 128.3, 128.5, 129.0, 129.3, 130.1, 131.5, 132.5, 136.7, 137.8, 140.9, 173.9, 196.5; IR (neat): 2930, 1733, 1660, 1448, 1282, 1166, 971, 721 cm^{-1}; HRMS (ESI): m/z calcd for $C_{23}H_{26}O_3$ $[M+Na]^+$ 373.1774. Found 373.1780.

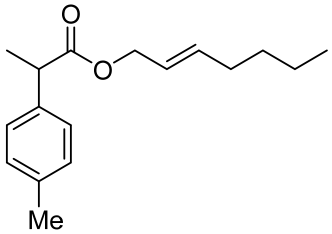
Procedure for preparation of 1b, 1c, 1f-j, 1l, 1m



In a flame-dried flask, a solution of carboxylic acid (1.2 equiv) in DCM (ca. 1 M) was cooled to 0 °C. Alcohol (1.0 equiv), WSCI•HCl (1.2 equiv) and DMAP (0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The combined organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford allyl carboxylate.

In a flame-dried flask, to a solution of HN*i*Pr_2 (1.2 equiv) in THF (0.2 M) was added *n*BuLi (1.2 equiv, 1.6 M in hexane) dropwise at –78 °C. The reaction mixture was warmed to 0 °C and stirred for 15 min at the temperature. The mixture was cooled to –78 °C again and allyl carboxylate (1.0 equiv) was added. After stirring for 1 h at –78 °C, MeI (3.0 equiv) was added at the same temperature. After stirring at room temperature for 4 h, the reaction was quenched with aqueous HCl (1 M), and products were extracted with EtOAc. The organic layer was washed with H_2O and brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford pure product **1**.

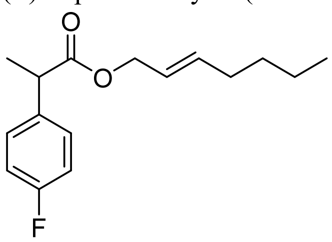
(*E*)-hept-2-en-1-yl 2-(*p*-tolyl)propanoate (**1b**)



Yield: 32% (2 steps)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.3 Hz, 3H), 1.23-1.37 (m, 4H), 1.48 (d, *J* = 7.3 Hz, 3H), 2.02 (dt, *J* = 6.9, 6.9 Hz, 2H), 2.33 (s, 3H), 3.69 (q, *J* = 7.3 Hz, 1H), 4.44-4.55 (m, 2H), 5.46-5.53 (m, 1H), 5.64-5.71 (m, 1H), 7.13 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 18.6, 21.0, 22.1, 31.0, 31.9, 45.1, 65.4, 123.6, 127.3, 129.2, 136.3, 136.6, 137.6, 174.5; IR (neat): 2929, 1735, 1457, 1163, 972, 819 cm^{-1}; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1665.

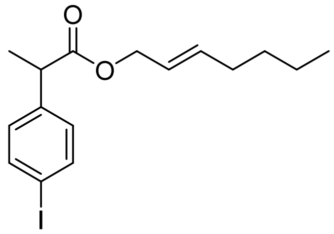
(*E*)-hept-2-en-1-yl 2-(4-fluorophenyl)propanoate (**1c**)



Yield: 44% (2 steps)

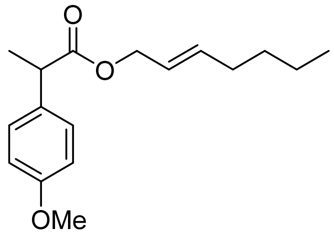
^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.86 (t, *J* = 7.2 Hz, 3H), 1.23-1.34 (m, 4H), 1.46 (d, *J* = 7.1 Hz, 3H), 1.97-2.02 (m, 2H), 3.68 (q, *J* = 7.1 Hz, 1H), 4.45-4.52 (m, 2H), 5.44-5.50 (m, 1H), 5.63-5.69 (m, 1H), 6.96-7.00 (m, 2H), 7.23-7.28 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.8, 18.6, 22.1, 30.9, 31.8, 44.8, 65.5, 115.3 (d, J = 20.3 Hz), 123.5, 129.0 (d, J = 8.3 Hz), 136.2 (d, J = 3.6 Hz), 136.5, 161.9 (d, J = 242.2 Hz), 174.1; ^{19}F NMR (369 MHz, CDCl$_3$): δ = -115.77; IR (neat): 2930, 1735, 1509, 1226, 1156, 972, 839 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}FO_2$ [M+Na]$^+$ 287.1418. Found 287.1434.

(*E*)-hept-2-en-1-yl 2-(4-iodophenyl)propanoate (**1f**)



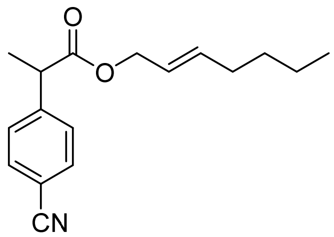
Yield: 59% (2 steps)
^{1}H NMR (500 MHz, CDCl$_3$): δ = 0.89 (t, J = 7.2 Hz, 3H), 1.26-1.34 (m, 4H), 1.47 (d, J = 7.3 Hz, 3H), 2.02 (dt, J = 6.9, 6.9 Hz, 2H), 3.66 (q, J = 7.3 Hz, 1H), 4.46-4.53 (m, 2H), 5.46-5.51 (m, 1H), 5.65-5.71 (m, 1H), 7.05-7.07 (m, 2H), 7.63-7.65 (m, 2H); ^{13}C NMR (125 MHz, CDCl$_3$): δ = 13.9, 18.4, 22.1, 30.9, 31.8, 45.1, 65.6, 92.5, 123.4, 129.5, 136.7, 137.6, 140.2, 173.7; IR (neat): 2928, 1734, 1486, 1164, 1007, 825 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}IO_2$ [M+Na]$^+$ 395.0478. Found 395.0495.

(*E*)-hept-2-en-1-yl 2-(4-methoxyphenyl)propanoate (**1g**)



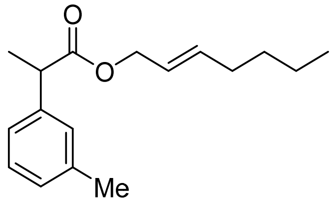
Yield: 39% (2 steps)
^{1}H NMR (500 MHz, CDCl$_3$): δ = 0.90 (t, J = 7.2 Hz, 3H), 1.28-1.37 (m, 4H), 1.49 (d, J = 7.1 Hz, 3H), 2.04 (dt, J = 6.7, 6.7 Hz, 2H), 3.69 (q, J = 7.1 Hz, 1H), 3.81 (s, 3H), 4.48-4.54 (m, 2H), 5.50-5.54 (m, 1H), 5.67-5.71 (m, 1H), 6.86-6.88 (m, 2H), 7.23-7.26 (m, 2H); ^{13}C NMR (125 MHz, CDCl$_3$): δ = 13.9, 18.6, 22.1, 31.0, 31.9, 44.6, 55.2, 65.4, 113.9, 123.6, 128.5, 132.7, 136.3, 158.6, 174.6; IR (neat): 2931, 1734, 1613, 1513, 1248, 1163, 1037, 972, 834 cm^{-1}; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_3$ [M+Na]$^+$ 299.1618. Found 299.1618.

(*E*)-hept-2-en-1-yl 2-(4-cyanophenyl)propanoate (**1h**)



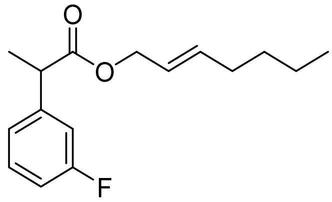
Yield: 23% (2 steps)
^{1}H NMR (400 MHz, CDCl$_3$): δ = 0.88 (t, J = 7.3 Hz, 3H), 1.23-1.37 (m, 4H), 1.51 (d, J = 7.3 Hz, 3H), 2.02 (dt, J = 6.9, 6.9 Hz, 2H), 3.75-3.80 (m, 1H), 4.46-4.55 (m, 2H), 5.44-5.51 (m, 1H), 5.66-5.73 (m, 1H), 7.42 (d, J = 8.5 Hz, 2H), 7.62 (d, J = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 13.9, 18.3, 22.1, 30.9, 31.8, 45.6, 65.9, 111.1, 118.7, 123.2, 128.4, 132.4, 137.1, 145.9, 173.2; IR (neat): 2931, 2229, 1734, 1457, 1167, 1075, 972, 845 cm^{-1}; HRMS (ESI): m/z calcd for $C_{17}H_{21}NO_2$ [M+Na]$^+$ 294.1465. Found 294.1454.

(E)-hept-2-en-1-yl 2-(m-tolyl)propanoate (**1i**)



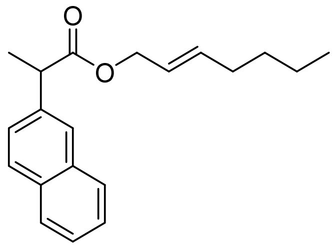
Yield: 32% (2 steps)
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, J = 7.1 Hz, 3H), 1.24-1.36 (m, 4H), 1.48 (d, J = 7.3 Hz, 3H), 2.02 (dt, J = 6.9, 6.9 Hz, 2H), 2.34 (s, 3H), 3.69 (q, J = 7.3 Hz, 1H), 4.45-4.56 (m, 2H), 5.46-5.54 (m, 1H), 5.64-5.72 (m, 1H), 7.06-7.14 (m, 3H), 7.21 (t, J = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 18.6, 21.4, 22.1, 31.0, 31.9, 45.4, 65.4, 123.6, 124.5, 127.8, 128.2, 128.4, 136.3, 138.2, 140.7, 174.4; IR (neat): 2929, 1736, 1457, 1176, 971, 700 cm^{-1}; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ [M+Na]$^+$ 283.1669. Found 283.1665.

(E)-hept-2-en-1-yl 2-(3-fluorophenyl)propanoate (**1j**)



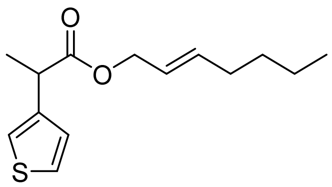
Yield: 46% (2 steps)
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, J = 7.1 Hz, 3H), 1.25-1.35 (m, 4H), 1.49 (d, J = 7.3 Hz, 3H), 2.02 (dt, J = 6.9, 6.9 Hz, 2H), 3.72 (q, J = 7.3 Hz, 1H), 4.50-4.54 (m, 2H), 5.46-5.53 (m, 1H), 5.66-5.73 (m, 1H), 6.92-6.97 (m, 1H), 7.01-7.04 (m, 1H), 7.07 (d, J = 7.8 Hz, 1H), 7.25-7.30 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 18.4, 22.1, 30.9, 31.8, 45.2, 65.6, 114.0 (d, J = 21.0 Hz), 114.5 (d, J = 21.9 Hz), 123.2, 123.3 (d, J = 17.2 Hz), 129.9 (d, J = 8.6 Hz), 136.7, 142.9 (d, J = 7.6 Hz), 162.8 (d, J = 249 Hz), 173.7; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -113.15; IR (neat): 2931, 1736, 1450, 1176, 972, 780 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}FO_2$ [M+Na]$^+$ 287.1418. Found 287.1434.

(E)-hept-2-en-1-yl 2-(naphthalen-2-yl)propanoate (**1l**)



Yield: 57% (2 steps)
^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.86 (t, J = 7.2 Hz, 3H), 1.24-1.33 (m, 4H), 1.60 (d, J = 7.0 Hz, 3H), 2.00 (dt, J = 6.9, 6.9 Hz, 2H), 3.90 (q, J = 7.0 Hz, 1H), 4.49-4.57 (m, 2H), 5.48-5.53 (m, 1H), 5.65-5.70 (m, 1H), 7.44-7.49 (m, 3H), 7.75 (s, 1H), 7.80-7.82 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.8, 18.6, 22.1, 30.9, 31.8, 45.6, 65.5, 123.5, 125.7, 125.8, 126.1, 126.1, 127.6, 127.8, 128.2, 132.5, 133.4, 136.5, 138.0, 174.3; IR (neat): 2929, 2871, 1733, 1456, 1179, 971, 746 cm^{-1}; HRMS (ESI): m/z calcd for $C_{20}H_{24}O_2$ [M+Na]$^+$ 319.1669. Found 319.1676.

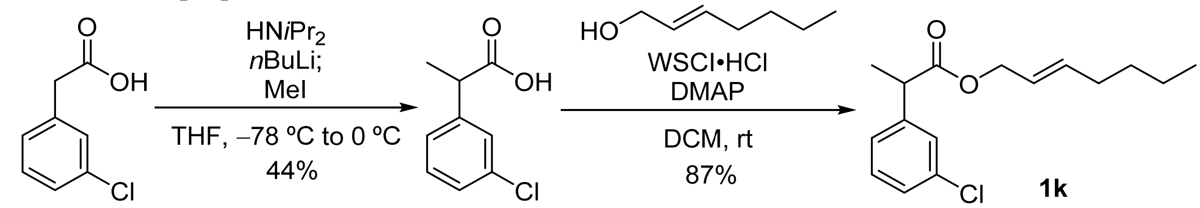
(E)-hept-2-en-1-yl 2-(thiophen-3-yl)propanoate (**1m**)



Yield: 56% (2 steps)

^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.9 Hz, 3H), 1.27-1.37 (m, 4H), 1.51 (d, *J* = 7.0 Hz, 3H), 2.03 (dt, *J* = 6.7, 6.7 Hz, 2H), 3.84 (q, *J* = 7.0 Hz, 1H), 4.50-4.53 (m, 2H), 5.49-5.55 (m, 1H), 5.68-5.74 (m, 1H), 7.07 (dd, *J* = 2.3, 5.2 Hz, 1H), 7.13 (d, *J* = 2.3 Hz, 1H), 7.26-7.27 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 18.2, 22.1, 31.0, 31.9, 41.0, 65.5, 121.1, 123.5, 125.6, 127.1, 136.5, 140.6, 173.9; IR (neat): 2929, 1736, 1457, 1173, 972, 768 cm^{-1}; HRMS (ESI): m/z calcd for $C_{14}H_{20}O_2S$ $[M+Na]^+$ 275.1076. Found 275.1068.

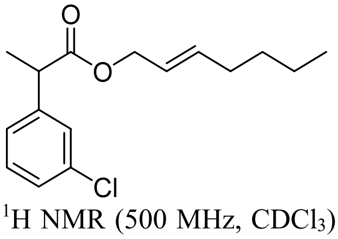
Procedure for preparation of 1k



In a flame-dried flask, to a solution of HN*i*Pr_2 (2.5 mL, 17.6 mmol, 1.2 equiv) in THF (88 mL, 0.2 M) was added *n*BuLi (11 mL, 17.6 mmol, 1.2 equiv, 1.6 M in hexane) dropwise at –78 °C. The reaction mixture was warmed to 0 °C and stirred for 15 min at the temperature. The mixture was cooled to –78 °C again and 2-(3-chlorophenyl)acetic acid (2.5 g, 14.7 mmol, 1.0 equiv) was added. After stirring for 1 h at –78 °C, MeI (2.7 mL, 44.1 mmol, 3.0 equiv) was added at the same temperature. After stirring at room temperature for 4 h, the reaction was quenched with aqueous HCl (1 M) and extracted with EtOAc. The combined organic layer was washed with H_2O and brine, and dried over $MgSO_4$. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford 2-(3-chlorophenyl)propanoic acid (1.2 g, 44%).

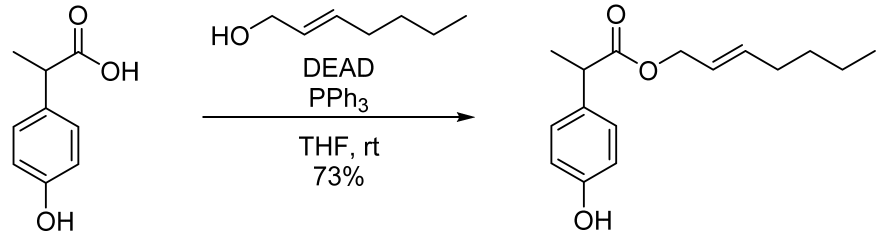
In a flame-dried flask, a solution of 2-(3-chlorophenyl)propanoic acid (1.2 g, 6.4 mmol, 1.2 equiv) in DCM (ca. 1 M) was cooled to 0 °C. (*E*)-Hept-2-en-1-ol (0.71 ml, 5.3 mmol, 1.0 equiv), WSCI•HCl (1.2 g, 6.4 mmol, 1.2 equiv) and DMAP (64.7 mg, 0.53 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1k** (1.30g, 87%).

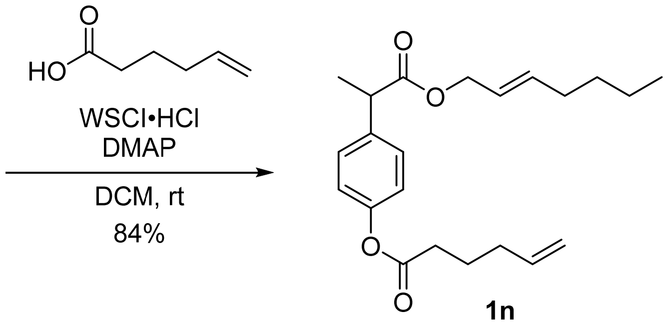
(*E*)-hept-2-en-1-yl 2-(3-chlorophenyl)propanoate (**1k**)



^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.91 (t, *J* = 7.2 Hz, 3H), 1.28-1.39 (m, 4H), 1.51 (d, *J* = 7.3 Hz, 3H), 2.05 (dt, *J* = 6.9, 6.9 Hz, 2H), 3.72 (q, *J* = 7.3 Hz, 1H), 4.50-4.58 (m, 2H), 5.50-5.55 (m, 1H), 5.69-5.75 (m, 1H), 7.20-7.33 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.9, 18.4, 22.1, 30.9, 31.9, 45.2, 65.6, 123.4, 125.7, 127.3, 127.7, 129.8, 134.3, 136.7, 142.4, 173.6; IR (neat): 2930, 1736, 1457, 1165, 972, 694 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115.

Procedure for preparation of 1n



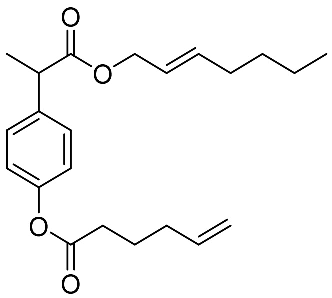


1n

In a flame-dried flask, a solution of 2-(4-hydroxyphenyl)propanoic acid (1.75 g, 10.5 mmol, 1.2 equiv), (*E*)-hept-2-en-1-ol (1.20 mL, 8.76 mmol, 1.0 equiv) and triphenylphosphine (2.76 g, 10.5 mmol, 1.2 equiv) in THF (50 mL) was cooled to 0 °C. Diethyl azodicarboxylate (4.78 mL, 10.5 mmol, 1.2 equiv) was added dropwise at a rate such that the temperature of the reaction mixture was maintained at 0 °C. The solution temperature was allowed to increase at room temperature, and the mixture was stirred for overnight. The reaction mixture was diluted with ether, and washed with saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford (*E*)-hept-2-en-1-yl 2-(4-hydroxyphenyl)propanoate (1.68 g, 73%).

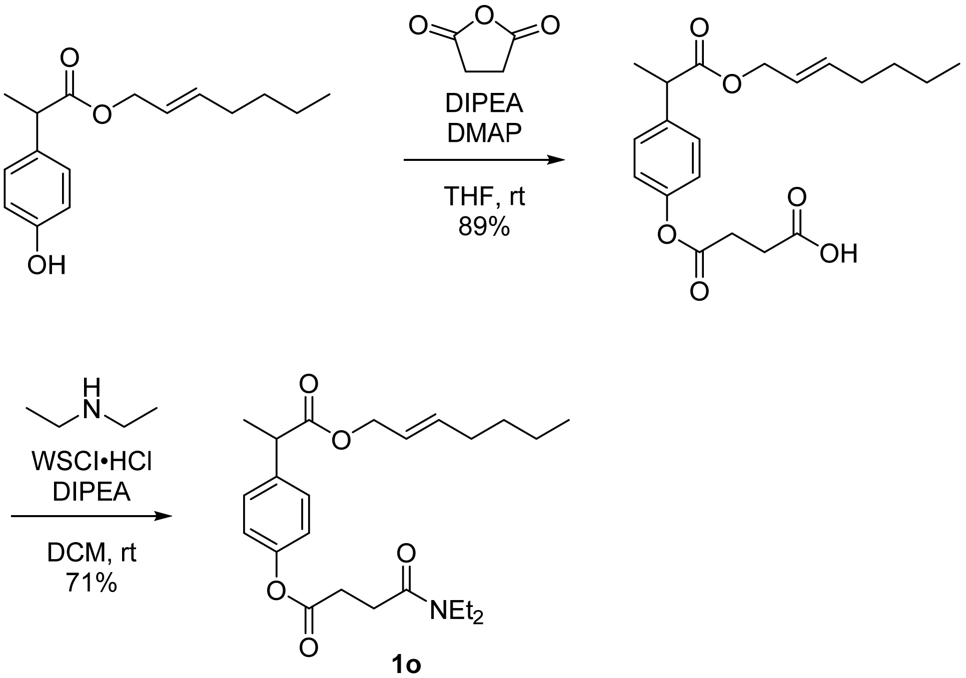
In a flame-dried flask, a solution of hex-5-enoic acid (148 mg, 1.3 mmol, 1.2 equiv) in DCM (ca. 1 M) was cooled to 0 °C. (*E*)-Hept-2-en-1-yl 2-(4-hydroxyphenyl)propanoate (300 mg, 1.1 mmol, 1.0 equiv), WSCI•HCl (244 mg, 1.3 mmol, 1.2 equiv) and DMAP (13.4 mg, 0.11 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1n** (333 mg, 84%).

(*E*)-4-(1-(hept-2-en-1-yloxy)-1-oxopropan-2-yl)phenyl hex-5-enoate (**1n**)



^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.2 Hz, 3H), 1.25-1.36 (m, 4H), 1.49 (d, *J* = 7.2 Hz, 3H), 1.85 (tt, *J* = 7.6, 7.6 Hz, 2H), 2.02 (dt, *J* = 6.9, 6.9 Hz, 2H), 2.18 (dt, *J* = 7.6, 7.6 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 3.72 (q, *J* = 7.2 Hz, 1H), 4.45-4.49 (m, 1H), 4.51-4.55 (m, 1H), 5.02-5.10 (m, 2H), 5.47-5.53 (m, 1H), 5.67-5.72 (m, 1H), 5.78-5.86 (m, 1H), 7.02 (d, *J* = 8.6 Hz, 2H), 7.31 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.8, 18.6, 22.1, 23.9, 30.9, 31.8, 32.9, 33.5, 44.9, 65.5, 115.6, 121.5, 123.5, 128.5, 136.5, 137.4, 137.9, 149.6, 172.0, 174.0; IR (neat): 2931, 1735, 1507, 1204, 914, 847 cm^{-1}; HRMS (ESI): m/z calcd for $C_{22}H_{30}O_4$ $[M+Na]^+$ 381.2036. Found 381.2037.

Procedure for preparation of 1o



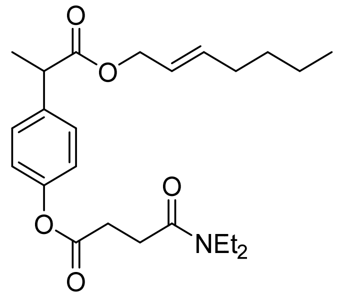
1o

(*E*)-Hept-2-en-1-yl 2-(4-hydroxyphenyl)propanoate (4.0 g, 15.2 mmol, 1 equiv) and succinic anhydride (7.63 mg, 76.2 mmol, 5 equiv) were stirred together in DCM (100 mL) with diisopropylethylamine (5.3 mL, 30.5 mmol, 2.0 equiv) and DMAP (373 mg, 3.05 mmol, 0.2 equiv) at room temperature. After stirring for 2.5 h, the reaction was diluted with H_2O, and products were extracted with EtOAc. The organic layer was washed with H_2O and brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford (*E*)-4-(4-(1-(hept-2-en-1-yloxy)-1-oxopropan-2-yl)phenoxy)-4-oxobutanoic acid (4.92 g, 89%).

In a flame-dried flask, a solution of (*E*)-4-(4-(1-(hept-2-en-1-yloxy)-1-oxopropan-2-yl)phenoxy)-4-oxobutanoic acid (500 mg, 1.4 mmol, 1.2 equiv) in DCM (15 mL) was cooled to 0 °C. Diethylamine (0.17 mL, 1.7 mmol, 1.0 equiv), WSCI•HCl (320mg, 1.7 mmol, 1.2 equiv) and diisopropylethylamine

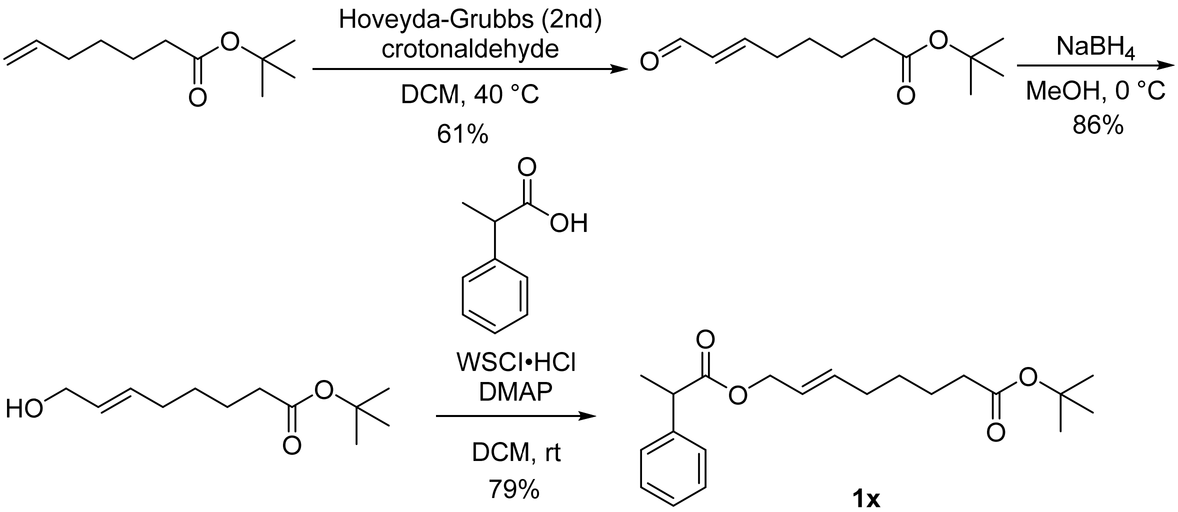
(0.29 mL, 1.7 mmol, 1.2 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1o** (411.5 mg, 71%) as a colorless oil.

(*E*)-4-(1-(hept-2-en-1-yloxy)-1-oxopropan-2-yl)phenyl 4-(diethylamino)-4-oxobutanoate (**1o**)



^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.9 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H), 1.20 (t, *J* = 7.1 Hz, 3H), 1.26-1.35 (m, 4H), 1.48 (d, *J* = 7.1 Hz, 3H), 2.02 (dt, *J* = 6.9, 6.9 Hz, 2H), 2.71 (t, *J* = 6.6 Hz, 2H), 2.92 (t, *J* = 6.6 Hz, 2H), 3.34-3.40 (m, 4H), 3.71 (q, *J* = 7.1 Hz, 1H), 4.44-4.47 (m, 1H), 4.50-4.53 (m, 1H), 5.47-5.51 (m, 1H), 5.67-5.71 (m, 1H), 7.05 (d, *J* = 8.6 Hz, 2H), 7.30 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.0, 13.8, 14.1, 18.6, 22.1, 27.8, 29.6, 30.9, 31.8, 40.3, 41.8, 44.9, 65.5, 121.6, 123.5, 128.4, 136.5, 137.8, 149.7, 169.9, 171.9, 174.1; IR (neat): 2931, 1733, 1645, 1458, 1205, 1132, 973, 791 cm^{-1}; HRMS (ESI): m/z calcd for $C_{24}H_{35}NO_5$ $[M+Na]^+$ 440.2407. Found 440.2426.

Procedure for preparation of 1x



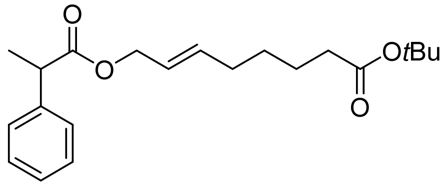
1x

A solution of crotonaldehyde (4.5 mL, 54 mmol, 10 equiv) in DCM (30 mL) was added to a solution of the *tert*-butyl hept-6-enoate (1.0 mg, 5.4 mmol, 1.0 equiv) in DCM (5 mL). Second generation Hoveyda-Grubbs catalyst (100 mg, 0.16 mmol, 0.03 equiv) was added, and the mixture was stirred for 5 h at 40 °C. DMSO (1 mL) was added to the mixture, and the mixture was stirred at room temperature for overnight. Subsequently, the solvent was removed under reduced pressure, and the crude product was purified by silica gel chromatography to yield *tert*-butyl (*E*)-8-oxooct-6-enoate (699 mg, 61% yield).

To a solution of *tert*-butyl (*E*)-8-oxooct-6-enoate (699 mg, 3.29 mmol, 1.0 equiv) in MeOH (20 mL) at 0 °C was added $NaBH_4$ (155 mg, 3.95 mmol, 1.2 equiv). After stirring for 1 h at 0 °C, the reaction was quenched by the addition of saturated NH_4Cl aq. Subsequently the mixture was extracted with DCM. The organic layer was dried over Na_2SO_4. After filtration, the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography to afford *tert*-butyl (*E*)-8-hydroxyoct-6-enoate (604 mg, 86% yield).

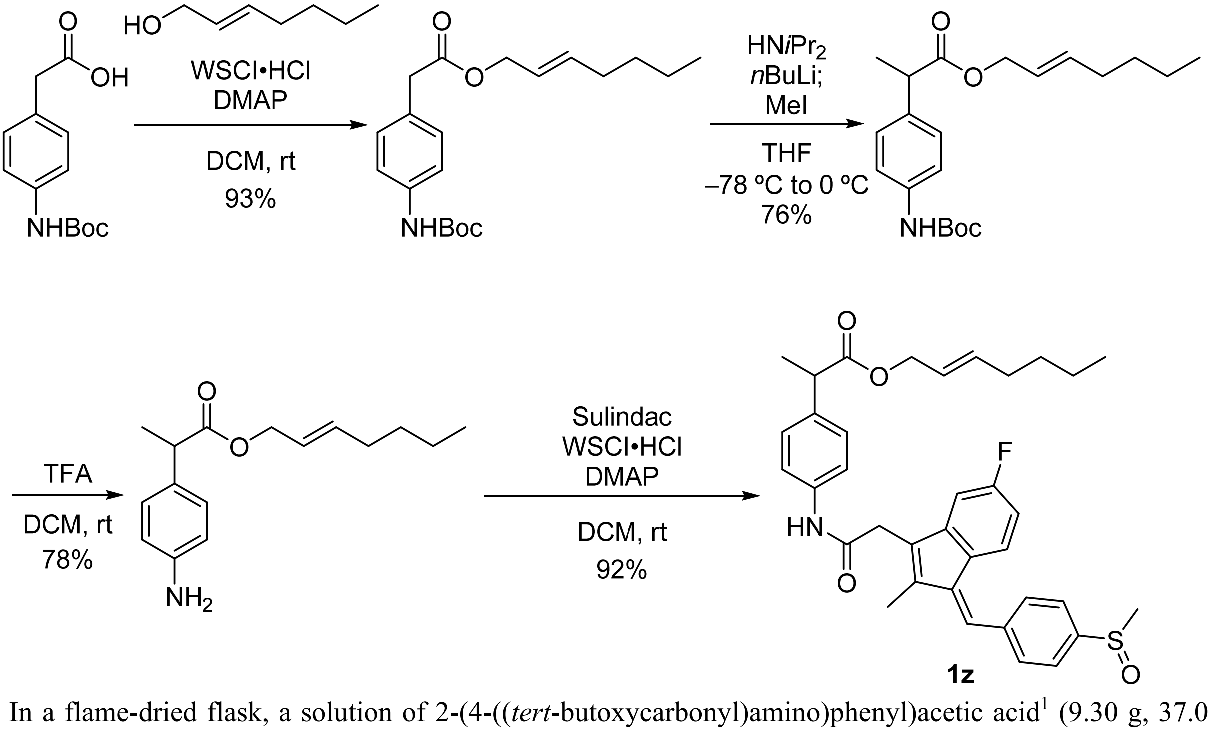
In a flame-dried flask, a solution of 2-phenylpropanoic acid (593 mg, 3.95 mmol, 1.2 equiv) in DCM (15 mL) was cooled to 0 °C. *tert*-Butyl (*E*)-8-hydroxyoct-6-enoate (705.3 mg, 3.29 mmol, 1.0 equiv), WSCI•HCl (757 mg, 3.95 mmol, 1.2 equiv) and DMAP (40.2 mg, 0.33 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by neural silica gel column chromatography to afford **1x** (895 mg, 79%) as a colorless oil.

tert-butyl (*E*)-8-((2-phenylpropanoyl)oxy)oct-6-enoate (**1x**)



^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.33-1.41 (m, 2H), 1.44 (s, 9H), 1.50 (d, *J* = 7.3 Hz, 3H), 1.53-1.59 (m, 2H), 2.03 (dt, *J* = 7.1, 7.1 Hz, 2H), 2.19 (t, *J* = 7.4 Hz, 2H), 3.72 (q, *J* = 7.3 Hz, 1H), 4.45-4.55 (m, 2H), 5.48-5.53 (m, 1H), 5.63-5.68 (m, 1H), 7.23-7.26 (m, 1H), 7.29-7.33 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 18.6, 24.5, 28.1, 28.2, 31.8, 35.3, 45.5, 65.3, 80.0, 124.0, 127.0, 127.5, 128.5, 135.6, 140.6, 173.0, 174.3; IR (neat): 2978, 2934, 1732, 1455, 1153, 972, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{21}H_{30}O_4$ $[M+Na]^+$ 369.2036. Found 369.2024.

Procedure for preparation of 1z



In a flame-dried flask, a solution of 2-(4-((*tert*-butoxycarbonyl)amino)phenyl)acetic acid[1] (9.30 g, 37.0 mmol, 1.05 equiv) in DCM (300 mL) was cooled to 0 °C. (*E*)-hept-2-en-1-ol (4.8 mL, 35.2 mmol, 1.0

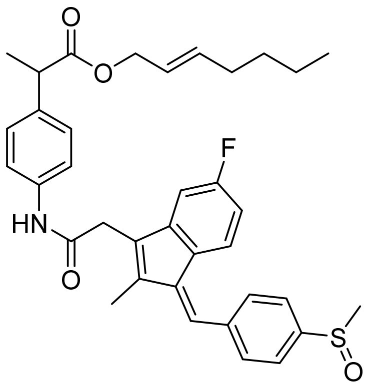
equiv), WSCI•HCl (7.09 g, 37.0 mmol, 1.05 equiv) and DMAP (431 mg, 3.52 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by neural silica gel column chromatography to afford (*E*)-hept-2-en-1-yl 2-(4-((*tert*-butoxycarbonyl)amino)phenyl)acetate (11.3 g, 93%) as a colorless oil.

In a flame-dried flask, to a solution of HN*i*Pr_2 (10.7 mL, 76.0 mmol, 2.2 equiv) in THF (200 mL) was added *n*BuLi (47.8 mL, 76.0 mmol, 2.2 equiv, 1.6 M in hexane) dropwise at –78 °C. The reaction mixture was warmed to 0 °C and stirred for 15 min at the temperature. The mixture was cooled to –78 °C again and (*E*)-hept-2-en-1-yl 2-(4-((tert-butoxycarbonyl)amino)phenyl)acetate (12.0 g, 34.5 mmol, 1.0 equiv) was added. After stirring for 1 h at –78 °C, MeI (2.58 mL, 41.4 mmol, 1.2 equiv) was added at the same temperature. After stirring at room temperature for overnight, the reaction was quenched with aqueous HCl (1 M) and extracted with EtOAc. The combined organic layer was washed with H_2O and brine, and dried over $MgSO_4$. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford (*E*)-hept-2-en-1-yl 2-(4-((*tert*-butoxycarbonyl)amino)phenyl)propanoate (9.53 g, 76%).

(*E*)-hept-2-en-1-yl 2-(4-((*tert*-butoxycarbonyl)amino)phenyl)propanoate (9.53 mL, 26.4 mmol, 1 equiv) was dissolved in DCM (100 mL). TFA (50 mL) was added, and the mixture was stirred for overnight at room temperature. The solvent was removed under reduced pressure, and the crude product was purified by silica gel chromatography to yield (*E*)-hept-2-en-1-yl 2-(4-aminophenyl)propanoate (5.38 g, 78% yield).

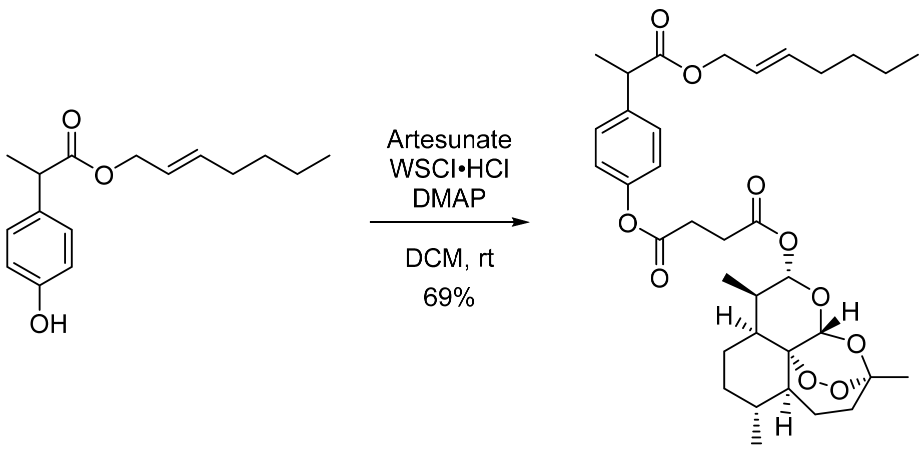
In a flame-dried flask, a solution of Sulindac (409 mg, 1.15 mmol, 1.0 equiv) in DCM (5 mL) was cooled to 0 °C. (*E*)-hept-2-en-1-yl 2-(4-aminophenyl)propanoate (300 mg, 1.15 mmol, 1.0 equiv), WSCI•HCl (231 mg, 1.21 mmol, 1.05 equiv), triethylamine (0.32 mL, 2.30 mmol, 2.0 equiv) and DMAP (14.0 mg, 0.115 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by neural silica gel column chromatography to afford **1z** (630 mg, 92%) as a yellow powder.

(*E*)-hept-2-en-1-yl 2-(4-(2-(5-fluoro-2-methyl-1-((*Z*)-4-(methylsulfinyl)benzylidene)-1H-inden-3-yl)acetamido)phenyl)propanoate (**1z**)



^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.91 (t, *J* = 7.3 Hz, 3H), 1.26-1.40 (m, 4H), 1.49 (d, *J* = 7.3 Hz, 3H), 2.04 (dt, *J* = 6.9, 6.9 Hz, 2H), 2.27 (s, 3H), 2.87 (s, 3H), 3.67-3.74 (m, 3H), 4.46-4.58 (m, 2H), 5.49-5.56 (m, 1H), 5.68-5.76 (m, 1H), 6.60 (ddd, *J* = 2.3, 8.9, 8.9 Hz, 1H), 6.98 (dd, *J* = 2.3, 9.2 Hz, 1H), 7.20-7.26 (m, 4H), 7.49 (d, *J* = 8.7 Hz, 2H), 7.70 (d, *J* = 8.7 Hz, 2H), 7.77 (d, *J* = 8.7 Hz, 2H), 8.25 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 10.5, 13.8, 18.4, 22.0, 30.8, 31.7, 34.3, 43.7, 44.8, 65.4, 106.0 (d, *J* = 23.8 Hz), 110.9 (d, *J* = 22.9 Hz), 120.2, 123.5 (d, *J* = 22.9 Hz), 123.7, 127.8, 128.5, 129.4, 129.4, 130.2, 132.3, 132.4, 136.4, 136.6, 138.7, 139.4, 141.4, 145.1, 146.4 (d, J = 8.6 Hz), 163.2 (d, J = 250.8 Hz), 167.7, 174.2; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -111.94; IR (neat): 3300, 2929, 1731, 1604, 1538, 1324, 1166, 855 cm^{-1}; HRMS (ESI): m/z calcd for $C_{36}H_{38}FNO_4S$ $[M+Na]^+$ 622.2398. Found 622.2377.

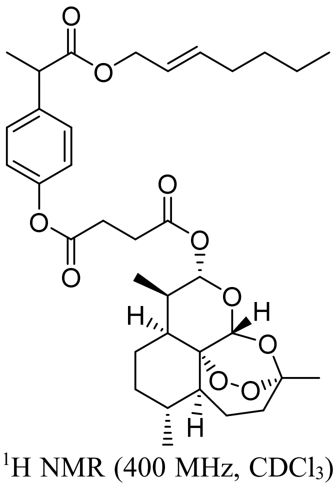
Procedure for preparation of 1aa



1aa

In a flame-dried flask, a solution of Artesunate (586.1 mg, 1.53 mmol, 1.0 equiv) in DCM (10 mL) was cooled to 0 °C. (*E*)-hept-2-en-1-yl 2-(4-hydroxyphenyl)propanoate (400 mg, 1.53 mmol, 1.0 equiv), WSCI•HCl (351 mg, 1.83 mmol, 1.2 equiv) and DMAP (18.6 mg, 0.153 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1aa** (664 mg, 69%) as a colorless liquid.

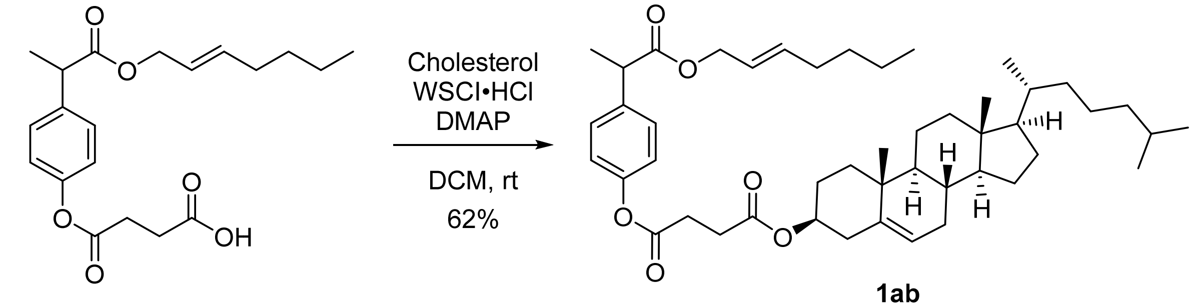
4-(1-(((*E*)-hept-2-en-1-yl)oxy)-1-oxopropan-2-yl)phenyl ((*3R*,*5aS*,*6R*,*8aS*,*9R*,*10S*,*12R*,*12aR*)-3,6,9-trimethyldecahydro-12H-3,12-epoxy[1,2]dioxepino[4,3-i]isochromen-10-yl) succinate (**1aa**)



^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85 (t, J = 8.0 Hz, 3H), 0.96 (d, J = 6.4 Hz, 3H), 1.25-1.48 (m, 13H), 1.59-1.64 (m, 2H), 1.69-1.79 (m, 2H), 1.84-1.86 (m, 3H), 1.99-2.06 (m, 3H), 2.38 (dt, J = 4.1, 14.2 Hz, 1H), 2.53-2.62 (m, 1H), 2.80-2.99 (m, 4H), 3.68-3.76 (m, 3H), 4.43-4.54 (m, 2H), 5.44-5.53 (m, 2H), 5.65-5.73 (m, 1H), 5.81 (d, J = 10.1 Hz, 1H), 7.04 (d, J = 8.7 Hz, 2H), 7.30 (d, J = 8.7 Hz, 2H); ^{13}C NMR

(100 MHz, $CDCl_3$): δ = 11.9, 13.7, 18.5, 20.1, 21.8, 22.0, 24.4, 25.8, 28.9, 29.0, 30.8, 31.6, 31.7, 33.9, 36.0, 37.1, 44.8, 45.0, 51.4, 65.4, 79.9, 91.3, 92.1, 104.3, 121.4, 123.4, 128.4, 136.4, 137.9, 149.4,170.6, 170.8, 173.9; IR (neat): 2928, 1750, 1507, 1376, 1203, 1138, 1017 cm^{-1}; HRMS (ESI): m/z calcd for $C_{35}H_{48}O_{10}$ $[M+Na]^+$ 651.3140. Found 651.3121.

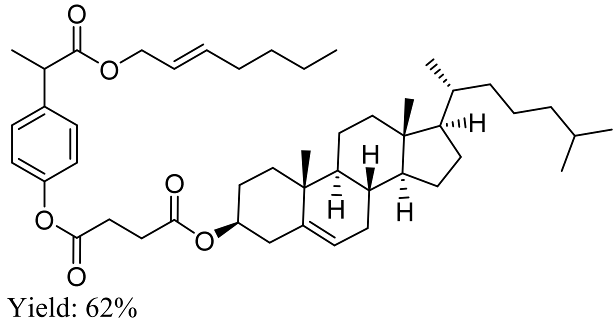
Procedure for preparation of 1ab



1ab

In a flame-dried flask, a solution of (*E*)-4-(4-(1-(hept-2-en-1-yloxy)-1-oxopropan-2-yl)phenoxy)-4-oxobutanoic acid (500 mg, 1.38 mmol, 1.0 equiv) in DCM (10 mL) was cooled to 0 °C. Cholesterol (533 mg, 1.38 mmol, 1.0 equiv), WSCI•HCl (317 mg, 1.66 mmol, 1.2 equiv) and DMAP (16.9 mg, 0.138 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1ab** (621 mg, 62%) as a white solid.

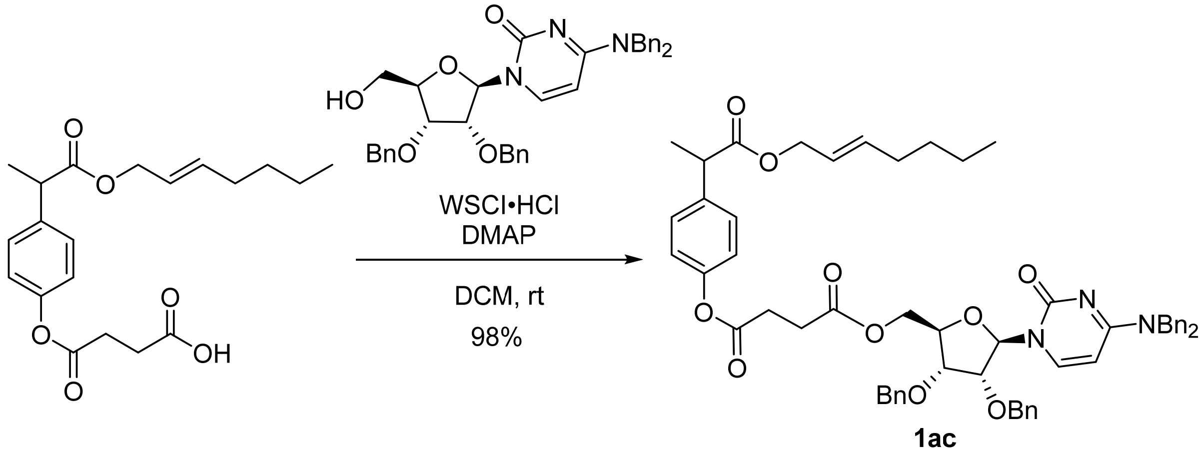
(3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl (4-(1-(((*E*)-hept-2-en-1-yl)oxy)-1-oxopropan-2-yl)phenyl) succinate (**1ab**)



Yield: 62%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.67 (s, 3H), 0.85-1.64 (m, 43H), 1.78-1.87 (m, 3H), 1.94-2.05 (m, 4H), 2.32 (d, J = 7.8 Hz, 2H), 2.71 (t, J = 6.6 Hz, 2H), 2.86 (t, J = 6.6 Hz, 2H), 3.65-3.74 (m, 1H), 4.43-4.55 (m, 2H), 4.61-4.68 (m, 1H), 5.36-5.38 (m, 1H), 5.44-5.53 (m, 1H), 5.64-5.73 (m, 1H), 7.04 (d, J = 8.7 Hz, 2H), 7.30 (d, J = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.8, 13.9, 18.7, 19.3, 21.0, 22.1, 22.5, 22.8, 23.8, 24.2, 26.6, 27.7, 28.0, 28.2, 29.4, 29.4, 30.9, 31.8, 31.9, 35.8, 36.1, 36.5, 36.9, 38.0, 39.5, 39.7, 42.3, 44.9, 50.0, 56.1, 56.6, 65.6, 74.5, 121.5, 122.7, 123.5, 126.8, 128.5, 136.6, 139.5, 149.6, 171.0, 171.4, 174.0; IR (neat): 2935, 2869, 1735, 1467, 1311, 1204, 1137, 1014, 853 cm^{-1}; HRMS (ESI): m/z calcd for $C_{47}H_{70}O_6$ $[M+Na]^+$ 753.5065. Found 753.5075.

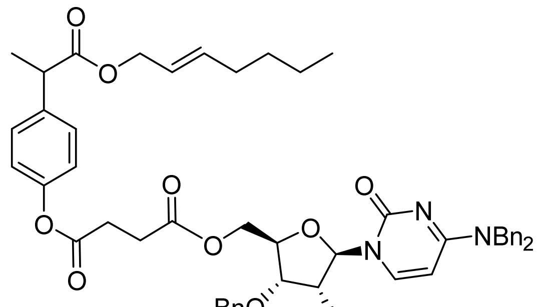
Procedure for preparation of 1ac



1ac

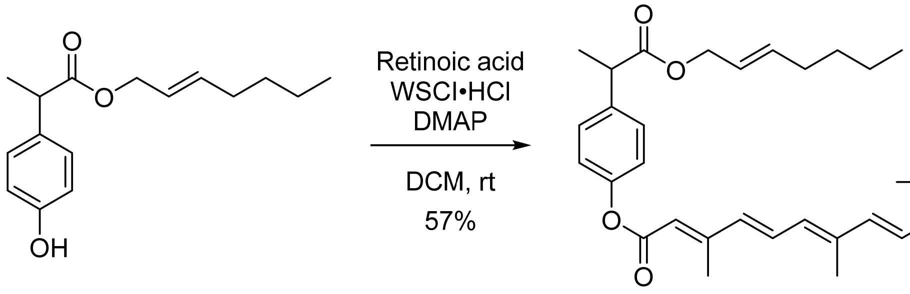
In a flame-dried flask, a solution of (*E*)-4-(4-(1-(hept-2-en-1-yloxy)-1-oxopropan-2-yl)phenoxy)-4-oxobutanoic acid (400 mg, 1.10 mmol, 1.0 equiv) in DCM (8 mL) was cooled to 0 °C. 1-((2*R*,3*R*,4*R*,5*R*)-3,4-Bis(benzyloxy)-5-(hydroxymethyl)tetrahydrofuran-2-yl)-4-(dibenzylamino)pyrimidin-2(1*H*)-one (*31*) (700 mg, 1.16 mmol, 1.05 equiv), WSCI•HCl (254 mg, 1.32 mmol, 1.2 equiv) and DMAP (13.5 mg, 0.11 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1ac** (1.02g, 98%) as a colorless oil.

((2*R*,3*R*,4*R*,5*R*)-3,4-bis(benzyloxy)-5-(4-(dibenzylamino)-2-oxopyrimidin-1(2*H*)-yl)tetrahydrofuran-2-yl)methyl (4-(1-(((*E*)-hept-2-en-1-yl)oxy)-1-oxopropan-2-yl)phenyl) succinate (**1ac**)



Yield: 98%
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 7.3 Hz, 3H), 1.23-1.37 (m, 4H), 1.44-1.46 (m, 3H), 2.01 (dt, *J* = 6.9, 6.9 Hz, 2H), 2.48-2.62 (m, 2H), 2.79 (t, *J* = 6.9 Hz, 2H), 3.66-3.71 (m, 1H), 3.75 (q, *J* = 4.6 Hz, 1H), 4.17 (d, *J* = 11.9 Hz, 1H), 4.27 (d, *J* = 5.0 Hz, 1H), 4.37-4.54 (m, 9H), 4.93-4.99 (m, 3H), 5.09-5.13 (m, 1H), 5.44-5.52 (m, 1H), 5.65-5.73 (m, 1H), 5.77 (d, *J* = 7.8 Hz, 1H), 5.95 (s, 1H), 6.94-6.97 (m, 2H), 7.09-7.12 (m, 2H), 7.19-7.35 (m, 17H), 7.52-7.54 (m, 2H), 7.65-7.68 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.6, 18.4, 21.8, 21.8, 26.3, 28.4, 28.7, 30.6, 31.6, 44.6, 45.8, 49.7, 50.1, 62.4, 65.2, 71.1, 71.6, 74.4, 78.9, 90.5, 91.2, 120.8, 121.1, 123.2, 126.0, 126.6, 127.3, 127.4, 127.5, 127.6, 128.1, 128.2, 128.3, 128.5, 128.6, 135.6, 136.0, 136.3, 136.7, 137.0, 137.6, 137.9, 140.5, 142.0, 149.2, 155.0, 164.0, 170.5, 171.2, 173.7; IR (neat): 2929, 1735, 1654, 1497, 1205, 1139, 736, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{57}H_{61}N_3O_{10}$ $[M+Na]^+$ 970.4249. Found 970.4263.

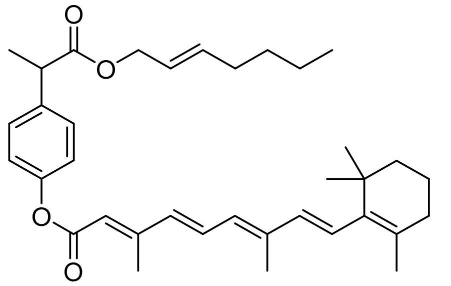
Procedure for preparation of 1ad



1ad

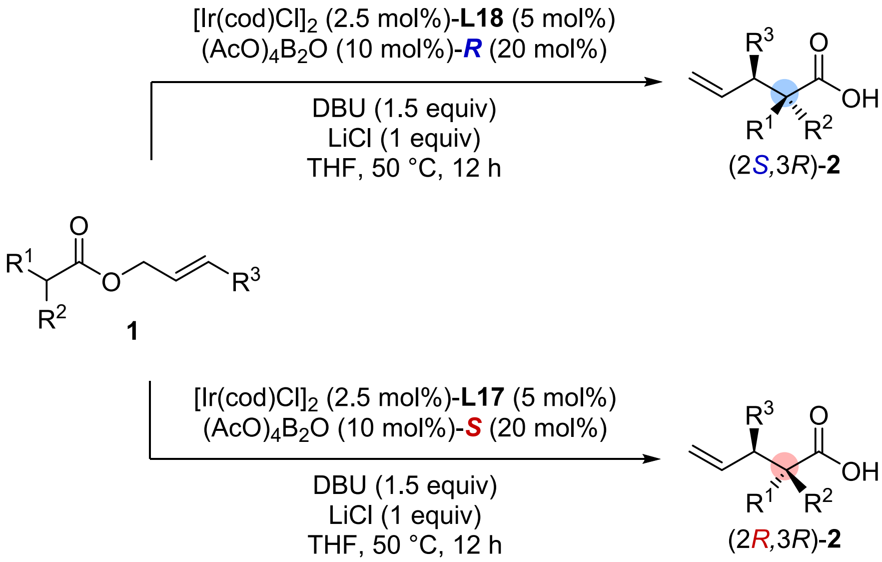
In a flame-dried flask, a solution of Retinoic acid (458.1 mg, 1.53 mmol, 1.0 equiv) in DCM (10 mL) was cooled to 0 °C. (*E*)-hept-2-en-1-yl 2-(4-hydroxyphenyl)propanoate (400 mg, 1.53 mmol, 1.0 equiv), WSCI•HCl (351 mg, 1.83 mmol, 1.2 equiv) and DMAP (18.6 mg, 0.153 mmol, 0.1 equiv) were added sequentially at 0 °C to the solution, and the mixture was stirred at room temperature for overnight. The resulting mixture was diluted with DCM and washed with aqueous HCl (1 M) and saturated $NaHCO_3$ aq. The organic layer was washed with brine, and dried over Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to afford **1ad** (474 mg, 57%) as a yellow liquid.

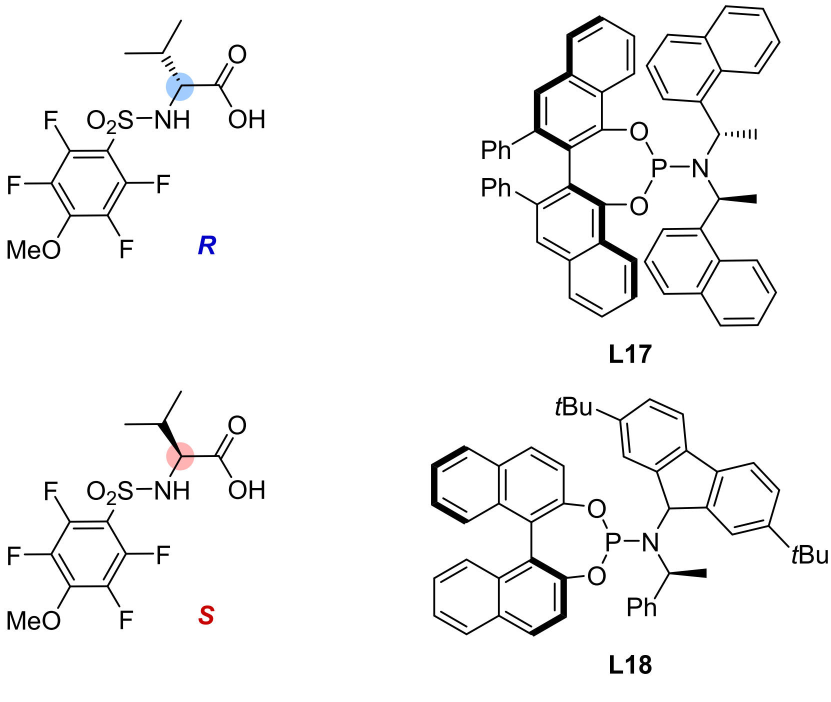
4-(1-(((*E*)-hept-2-en-1-yl)oxy)-1-oxopropan-2-yl)phenyl (2*E*,4*E*,6*E*,8*E*)-3,7-dimethyl-9-(2,6,6-trimethylcyclohex-1-en-1-yl)nona-2,4,6,8-tetraenoate (**1ad**)



^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.3 Hz, 3H), 1.04 (s, 6H), 1.24-1.37 (m, 4H), 1.46-1.50 (m, 5H), 1.58-1.65 (m, 2H), 1.72 (s, 3H), 1.83-1.87 (m, 2H), 1.99-2.04 (m, 5H), 2.40 (s, 3H), 3.73-3.75 (m, 1H), 4.44-4.55 (m, 2H), 5.46-5.54 (m, 1H), 5.66-5.74 (m, 1H), 5.98 (s, 1H), 6.14-6.20 (m, 2H), 6.29-6.39 (m, 2H), 7.05-7.11 (m, 3H), 7.32 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 12.9, 13.8, 14.0, 18.6, 19.1, 21.7, 22.1, 28.9, 30.9, 31.8, 33.1, 34.2, 39.5, 44.9, 65.5, 117.1, 121.8, 123.5, 128.4, 129.0, 129.4, 130.1, 131.9, 134.7, 136.5, 137.1, 137.6, 137.6, 140.3, 149.7, 155.5, 165.4, 174.1; IR (neat): 2931, 1733, 1507, 1457, 1203, 1165 cm^{-1}; HRMS (ESI): m/z calcd for $C_{36}H_{48}O_4$ $[M+Na]^+$ 567.3445. Found 567.3452.

3. General Procedure for the Reaction Conditions





A flame-dried 10 ml test tube A, equipped with a magnetic stirring bar, was charged with $[Ir(cod)Cl]_2$ (5.0 mg, 0.0075 mmol, 0.025 equiv), ligand **L17** or **L18** (0.015 mmol, 0.05 equiv), LiCl (12.8 mg, 0.3 mmol, 1 equiv), DBU (67.2 μL, 0.45 mmol, 1.5 equiv), and anhydrous THF (125 μL). Another flame-dried 10 ml test tube B, equipped with a magnetic stirring bar, was charged with $(AcO)_4B_2O$ (8.2 mg, 0.030 mmol, 0.1 equiv), (*R*)- or (*S*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***R*** or ***S***, 21.6 mg, 0.060 mmol, 0.2 equiv), and anhydrous THF (125 μL). After stirring the solution in test tube A for 1 h at 50 °C and stirring the solution in test tube B for 1 h at room temperature, the solution in test tube B and allyl ester **1** (0.30 mmol, 1 equiv) were added sequentially to the test tube A. The reaction mixture was stirred for 12 h at 50 °C under argon atmosphere. The reaction was quenched with aq. HCl (1.0 M) and products were extracted with EtOAc. The organic layer was washed with aqueous HCl (1.0 M), dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. Diastereomeric ratio (dr) and branch/linear (b/l) selectivity were determined by ^{1}H NMR analysis of crude mixture. The crude product was purified by column chromatography (hexane/EtOAc = 5/1).

(2*S*,3*R*)-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2a**)

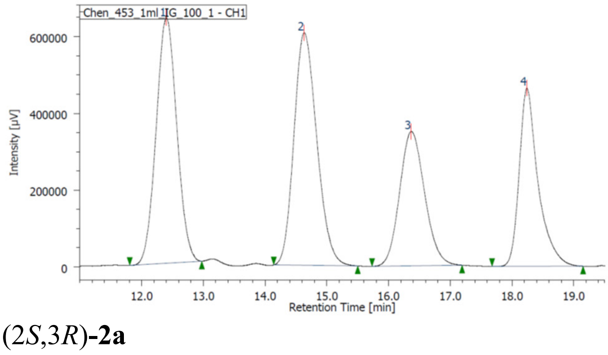
Yield: 82%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.9 Hz, 3H), 1.19-1.45 (m, 6H), 1.54 (s, 3H), 2.86-2.90 (m, 1H), 4.75 (dd, *J* = 18.0, 1.4 Hz, 1H), 4.88 (dd, *J* = 10.3, 1.4 Hz, 1H), 5.32 (ddd, *J* = 18.0, 10.3, 9.2 Hz, 1H), 7.21-7.24 (m, 1H), 7.29-7.32 (m, 2H), 7.40-7.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.1, 17.0, 22.6, 30.3, 30.5, 51.2, 53.7, 117.9, 126.7, 126.9, 128.1, 137.1, 141.6, 181.7; IR (neat): 2930, 1697, 1270, 1103, 917, 698 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 12.3 min; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_2$ $[M+Na]^+$ 269.1512. Found 269.1521; $[\alpha]_D^{20}$ = 59.4 (*c* = 0.47, $CHCl_3$).

(2*R*,3*R*)-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2a**)

Yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.03-1.26 (m, 6H), 1.53 (s, 3H), 2.92-2.96 (m, 1H), 5.11-5.18 (m, 2H), 5.59 (ddd, *J* = 19.2, 10.1, 10.1 Hz, 1H), 7.26 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.34 (dd, *J* = 7.6, 7.6 Hz, 2H), 7.47 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.1, 22.4, 28.2, 30.0, 50.3, 53.9, 118.5, 127.0, 127.2, 128.1, 138.2, 140.9, 181.4; IR (neat): 2933, 1699, 1274, 1134, 921, 697 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.4 min; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_2$ $[M+Na]^+$ 269.1512. Found 269.1506; $[\alpha]_D^{20}$ = 51.4 (*c* = 1.8, $CHCl_3$).

HPLC chart

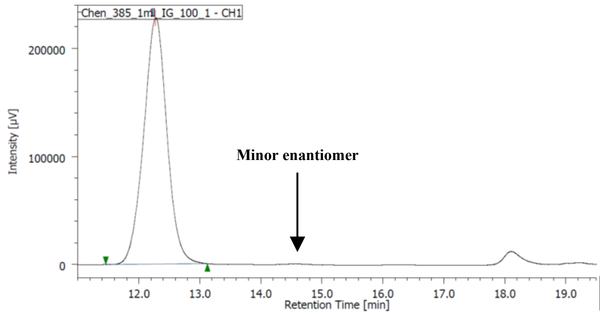
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_453_1ml_IG_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	12.400	14889228	639757	29.694	31.068	N/A	6467	3.457	1.049	
2	Unknown	1	14.633	15590735	605591	31.093	29.409	N/A	7435	2.452	1.194	
3	Unknown	1	16.367	9832434	349908	19.609	16.993	N/A	7850	3.011	1.138	
4	Unknown	1	18.242	9830198	463928	19.604	22.530	N/A	20473	N/A	1.462	

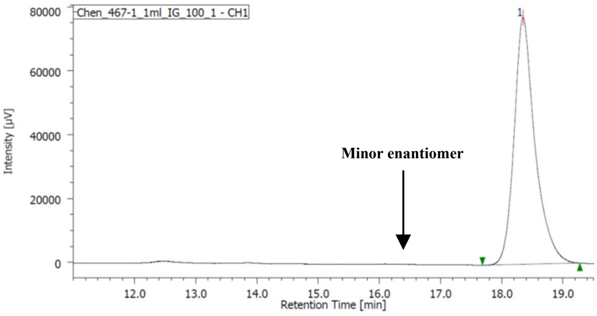
(2*S*,3*R*)-**2a**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_385_1ml_IG_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	12.275	6044866	228207	100.000	100.000	N/A	5171	N/A	1.006	

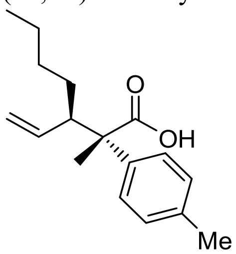
(2*R*,3*R*)-**2a**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_467-1_1ml_IG_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

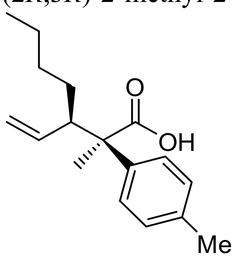
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.350	1834428	77355	100.000	100.000	N/A	16168	N/A	1.311	

(2*S*,3*R*)-2-methyl-2-(*p*-tolyl)-3-vinylheptanoic acid ((2*S*,3*R*)-**2b**)



Yield: 84%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.6 Hz, 3H), 1.20-1.43 (m, 6H), 1.52 (s, 3H), 2.31 (s, 3H), 2.85-2.90 (m, 1H), 4.77 (dd, *J* = 18.5, 2.1 Hz, 1H), 4.89 (dd, *J* = 10.5, 2.1 Hz, 1H), 5.32 (ddd, *J* = 18.5, 10.5, 9.6 Hz, 1H), 7.11 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 17.0, 20.9, 22.6, 30.3, 30.4, 51.0, 53.3, 117.8, 126.6, 128.8, 136.5, 137.1, 138.6, 181.3; IR (neat): 2928, 1698, 1514, 1270, 1099, 730 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 13.5 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1673; $[\alpha]_D^{20}$ = -20.9 (*c* = 0.53, $CHCl_3$).

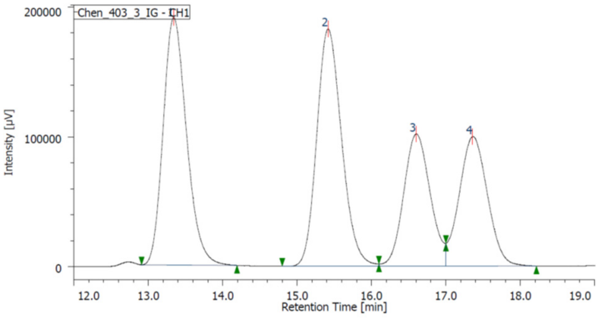
(2*R*,3*R*)-2-methyl-2-(*p*-tolyl)-3-vinylheptanoic acid ((2*R*,3*R*)-**2b**)



Yield: 75%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 7.2 Hz, 3H), 1.00-1.25 (m, 6H), 1.50 (s, 3H), 2.33 (s, 3H), 2.90-2.93 (m, 1H), 5.11-5.16 (m, 2H), 5.58 (ddd, *J* = 18.0, 9.0, 9.0 Hz, 1H), 7.13 (d, *J* = 8.6 Hz, 2H), 7.33 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.2, 20.9, 22.4, 28.2, 30.0, 50.1, 53.6, 118.4, 127.1, 128.9, 136.6, 137.8, 138.2, 181.7; IR (neat): 2931, 1698, 1514, 1273, 919, 730 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 17.2 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1676; $[\alpha]_D^{20}$ = 57.3 (*c* = 2.7, $CHCl_3$).

<u>HPLC chart</u>

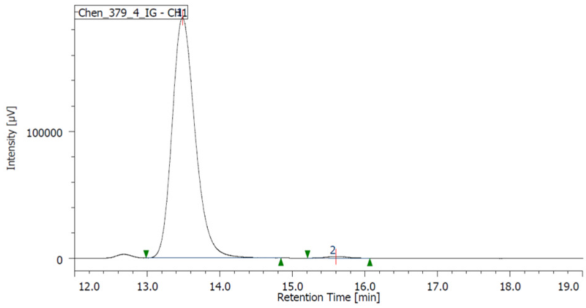
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_403_3_IG-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.342	4161898	190383	30.735	33.153	N/A	8770	3.534	1.216	
2	Unknown	1	15.417	4237341	182528	31.292	31.785	N/A	10299	1.877	1.177	
3	Unknown	1	16.600	2517596	101698	18.592	17.710	N/A	10234	1.121	N/A	
4	Unknown	1	17.358	2624255	99648	19.380	17.352	N/A	9845	N/A	N/A	

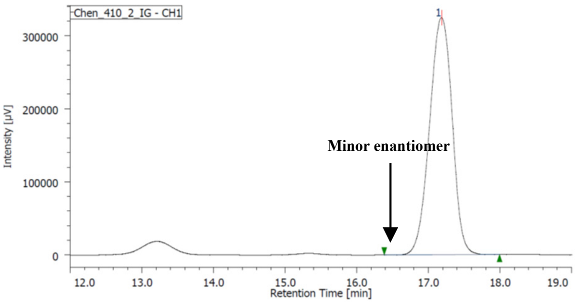
(2*S*,3*R*)-**2b**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_379_4_IG-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.483	4173769	189478	99.286	99.297	N/A	9020	3.635	1.227	
2	Unknown	1	15.600	30012	1341	0.714	0.703	N/A	10826	N/A	1.096	

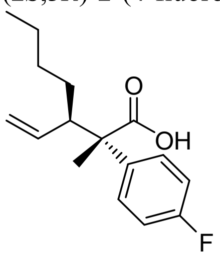
(2*R*,3*R*)-**2b**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_410_2_IG-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

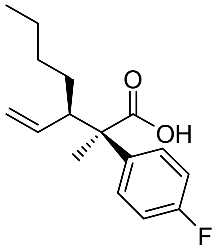
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	17.183	7114671	324556	100.000	100.000	N/A	13673	N/A	0.945	

(2*S*,3*R*)-2-(4-fluorophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2c**)



Yield: 81%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.9 Hz, 3H), 1.19-1.43 (m, 6H), 1.53 (s, 3H), 2.84-2.88 (m, 1H), 4.73 (dd, *J* = 16.9, 1.4 Hz, 1H), 4.89 (dd, *J* = 9.5, 1.4 Hz, 1H), 5.29 (ddd, *J* = 16.9, 9.5, 8.3 Hz, 1H), 6.97-7.00 (m, 2H), 7.37-7.40 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 16.8, 22.5, 30.2, 30.5, 51.3, 53.2, 114.9 (d, *J* = 20.3 Hz), 118.2, 128.5 (d, *J* = 7.2 Hz), 136.8, 137.3 (d, *J* = 2.4 Hz), 161.7 (d, *J* = 246.8 Hz), 181.7; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -115.96; IR (neat): 2932, 1699, 1510, 1237, 919, 830 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 0.30 mL/min; detection: at 254 nm; rt): t_R = 39.5 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}FO_2$ $[M+Na]^+$ 287.1418. Found 287.1428; $[\alpha]_D^{20}$ = 47.1 (*c* = 1.1, $CHCl_3$).

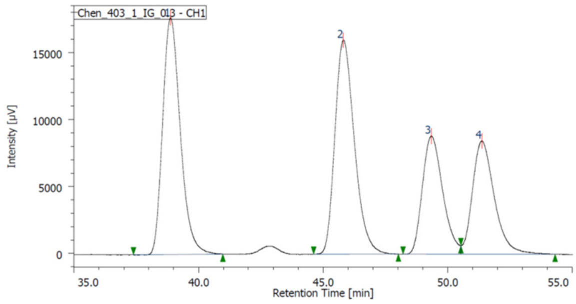
(2*R*,3*R*)-2-(4-fluorophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2c**)



Yield: 76%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.01-1.26 (m, 6H), 1.52 (s, 3H), 2.86-2.90 (m, 1H), 5.13-5.17 (m, 2H), 5.55 (ddd, *J* = 17.8, 8.6, 8.3 Hz, 1H), 7.00-7.03 (m, 2H), 7.41-7.44 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 17.2, 22.4, 28.2, 29.9, 50.6, 53.5, 114.9 (d, *J* = 21.0 Hz), 118.7, 129.0 (d, *J* = 7.6 Hz), 136.5 (d, *J* = 3.8 Hz), 137.9, 161.9 (d, *J* = 236.5 Hz), 181.6; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -115.86; IR (neat): 2932, 1700, 1510, 1237, 920, 830 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 0.30 mL/min; detection: at 254 nm; rt): t_R = 51.8 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}FO_2$ $[M+Na]^+$ 287.1418. Found 287.1410; $[\alpha]_D^{20}$ = 49.4 (*c* = 1.7, $CHCl_3$).

<u>HPLC chart</u>

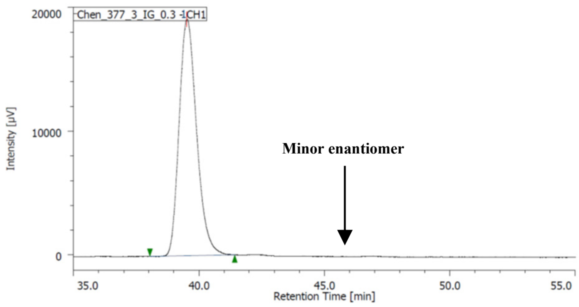
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_403_1_IG_0.3-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	38.875	871027	17697	31.599	34.734	N/A	14765	5.153	1.271	
2	Unknown	1	45.808	869504	16002	31.544	31.406	N/A	16685	2.422	1.300	
3	Unknown	1	49.333	498776	8806	18.095	17.283	N/A	17319	1.332	N/A	
4	Unknown	1	51.367	517178	8446	18.762	16.577	N/A	17300	N/A	N/A	

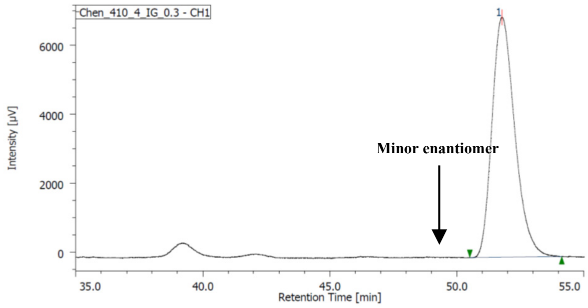
(2*S*,3*R*)-**2c**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_377_3_IG_0.3-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	39.517	925751	19098	100.000	100.000	N/A	15703	N/A	1.261	

(2*R*,3*R*)-**2c**



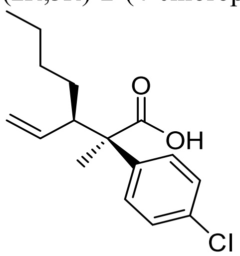
チャンネル情報＋ピーク情報	
クロマトグラム名	Chen_410_4_IG_0.3-CH1
サンプル名	
チャンネル名	CH1
データ取り込み間隔	500 [msec]
波形処理メソッド	（マニュアル）
数値計算式	
判定式	

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	51.783	434050	6943	100.000	100.000	N/A	16210	N/A	1.280	

(2*S*,3*R*)-2-(4-chlorophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2d**)

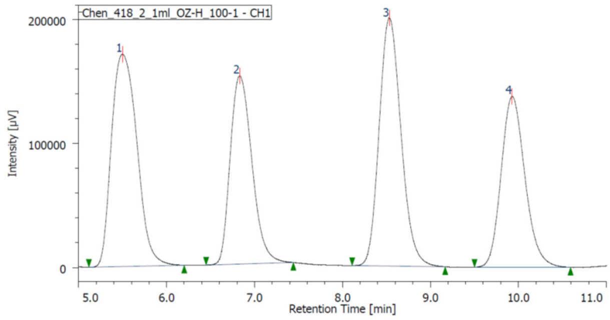
Yield: 87%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.6 Hz, 3H), 1.18-1.45 (m, 6H), 1.52 (s, 3H), 2.82-2.86 (m, 1H), 4.73-4.90 (m, 2H), 5.28 (ddd, *J* = 16.6, 8.6, 8.6 Hz, 1H), 7.27 (d, *J* = 8.6 Hz, 2H), 7.34 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 16.8, 22.5, 30.2, 30.5, 51.2, 53.4, 118.4, 128.2, 128.2, 132.8, 136.6, 140.1, 181.3; IR (neat): 2931, 1699, 1494, 1270, 1098, 919 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 8.5 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1125; $[\alpha]_D^{20}$ = 57.5 (*c* = 1.6, $CHCl_3$).

(2*R*,3*R*)-2-(4-chlorophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2d**)



Yield: 91%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 7.2 Hz, 3H), 1.01-1.28 (m, 6H), 1.51 (s, 3H), 2.85-2.90 (m, 1H), 5.13-5.17 (m, 2H), 5.54 (ddd, *J* = 16.6, 8.6, 8.6 Hz, 1H), 7.30 (d, *J* = 7.7 Hz, 2H), 7.40 (d, *J* = 7.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.1, 22.4, 28.3, 29.9, 50.5, 53.7, 118.8, 128.3, 128.8, 133.0, 137.8, 139.3, 181.4; IR (neat): 2932, 1700, 1495, 1276, 1099, 921 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 7.0 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115; $[\alpha]_D^{20}$ = 56.7 (*c* = 1.6, $CHCl_3$).

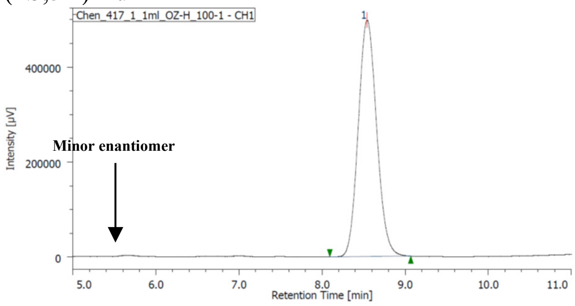
<u>HPLC chart</u>
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_418_2_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.500	3445282	171116	28.497	25.906	N/A	1594	2.655	1.260	
2	Unknown	1	6.833	2603270	151692	21.532	22.965	N/A	3595	3.750	1.275	
3	Unknown	1	8.533	3454461	200064	28.573	30.288	N/A	5673	2.959	1.166	
4	Unknown	1	9.925	2587014	137659	21.398	20.841	N/A	6564	N/A	1.194	

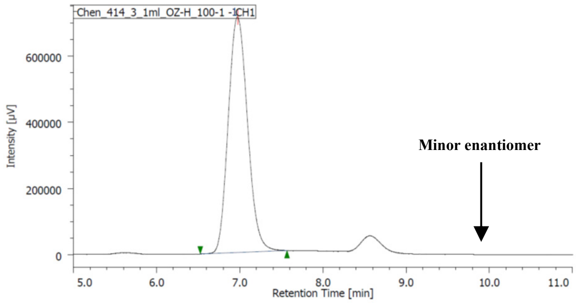
(2*S*,3*R*)-**2d**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_417_1_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	8.542	7655641	498301	100.000	100.000	N/A	7123	N/A	1.104	

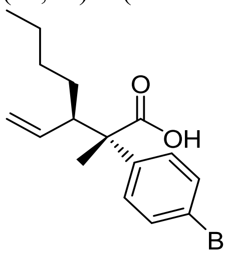
(2*R*,3*R*)-**2d**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_414_3_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

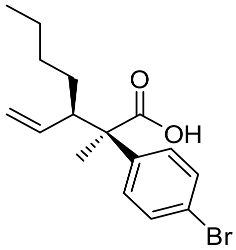
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.975	11453436	709971	100.000	100.000	N/A	4234	N/A	1.125	

(2*S*,3*R*)-2-(4-bromophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2e**)



Yield: 90%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.9 Hz, 3H), 1.18-1.42 (m, 6H), 1.52 (s, 3H), 2.81-2.85 (m, 1H), 4.75 (dd, *J* = 17.2, 1.7 Hz, 1H), 4.90 (dd, *J* = 9.5, 1.7 Hz, 1H), 5.28 (ddd, *J* = 17.2, 9.5, 8.6 Hz, 1H), 7.28 (d, *J* = 8.6 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 16.7, 22.5, 30.2, 30.5, 51.2, 53.5, 118.5, 121.1, 128.6, 131.2, 136.6, 140.7, 181.3; IR (neat): 2931, 1698, 1491, 1270, 919, 747 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 8.0 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}BrO_2$ $[M+Na]^+$ 347.0617. Found 347.0618; $[\alpha]_D^{20}$ = 48.9 (*c* = 1.8, $CHCl_3$).

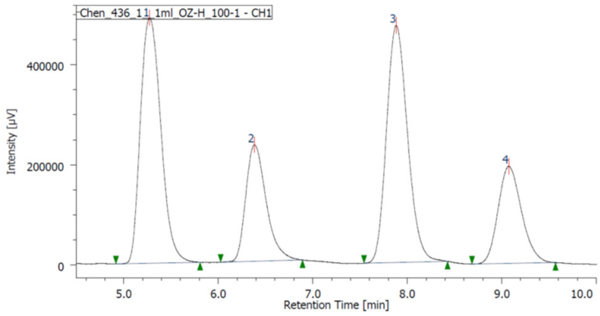
(2*R*,3*R*)-2-(4-bromophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2e**)



Yield: 95%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.78 (t, J = 7.2 Hz, 3H), 1.04-1.25 (m, 6H), 1.50 (s, 3H), 2.85-2.89 (m, 1H), 5.13-5.16 (m, 2H), 5.54 (ddd, J = 17.8, 8.6, 8.6 Hz, 1H), 7.33 (d, J = 8.6 Hz, 2H), 7.45 (d, J = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 17.2, 22.4, 28.3, 29.9, 50.4, 53.7, 118.8, 121.3, 129.1, 131.2, 137.7, 140.0, 180.6; IR (neat): 2931, 1699, 1491, 1276, 921, 747 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 6.3 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}BrO_2$ $[M+Na]^+$ 347.0617. Found 347.0627; $[\alpha]_D^{20}$ = 55.5 (c = 0.91, $CHCl_3$).

<u>HPLC chart</u>

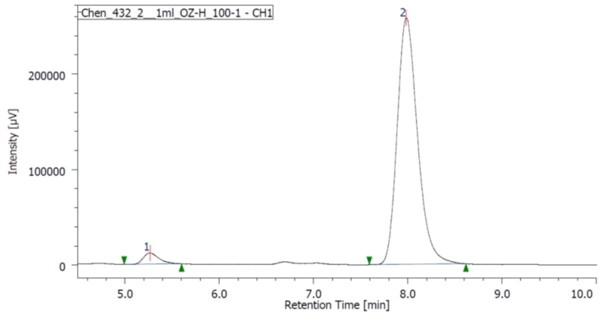
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_436_1_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.275	7348297	489590	34.235	35.266	N/A	2746	2.815	1.250	
2	Unknown	1	6.383	3483232	232579	16.228	16.753	N/A	4356	3.792	1.381	
3	Unknown	1	7.883	7303881	472659	34.028	34.047	N/A	6022	2.784	1.175	
4	Unknown	1	9.075	3328940	193436	15.509	13.934	N/A	6449	N/A	1.180	

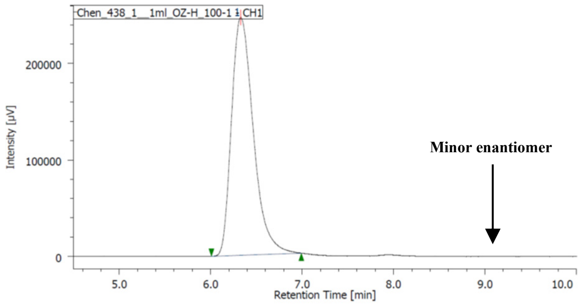
(2*S*,3*R*)-**2e**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_432_2_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.267	132654	11514	3.319	4.272	N/A	5022	7.977	1.408	
2	Unknown	1	7.983	3863802	257996	96.681	95.728	N/A	6855	N/A	1.261	

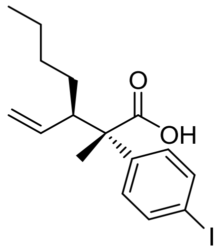
(2*R*,3*R*)-**2e**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_438_1_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

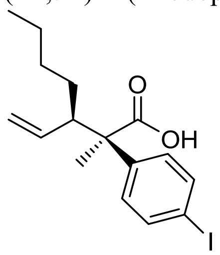
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.325	4095505	246483	100.000	100.000	N/A	3436	N/A	1.431	

(2*S*,3*R*)-2-(4-iodophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2f**)



Yield: 75%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.9 Hz, 3H), 1.17-1.42 (m, 6H), 1.50 (s, 3H), 2.81-2.85 (m, 1H), 4.75 (dd, *J* = 18.6, 1.6 Hz, 1H), 4.90 (dd, *J* = 10.3, 1.6 Hz, 1H), 5.28 (ddd, *J* =18.6, 10.3, 9.2 Hz, 1H), 7.15 (d, *J* = 8.9 Hz, 2H), 7.62 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 16.7, 22.5, 30.2, 30.5, 51.1, 53.6, 92.7, 118.5, 128.9, 136.6, 137.2, 141.4, 181.2; IR (neat): 2928, 1698, 1270, 1100, 919, 742 cm^{-1}; HPLC (chiral column: CHIRALPAK IB N-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 12.6 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}IO_2$ $[M+Na]^+$ 395.0478. Found 395.0464; $[\alpha]_D^{20}$ = 38.6 (*c* = 1.3, $CHCl_3$).

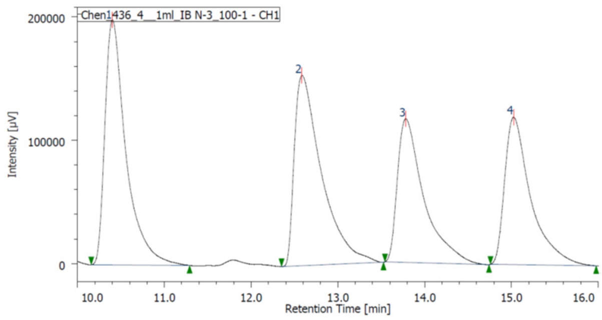
(2*R*,3*R*)-2-(4-iodophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2f**)



Yield: 81%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.00-1.28 (m, 6H), 1.49 (s, 3H), 2.83-2.89 (m, 1H), 5.12-5.16 (m, 2H), 5.48-5.58 (m, 1H), 7.20 (d, *J* = 8.7 Hz, 2H), 7.65 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.1, 22.4, 28.3, 29.9, 50.3, 53.8, 92.9, 118.8, 129.4, 137.2, 137.7, 140.6, 181.0; IR (neat): 2931, 1698, 1275, 1004, 920, 742 cm^{-1}; HPLC (chiral column: CHIRALPAK IB N-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 15.1 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}IO_2$ $[M+Na]^+$ 395.0478. Found 395.0493; $[\alpha]_D^{20}$ = 50.0 (*c* = 1.5, $CHCl_3$).

<u>HPLC chart</u>

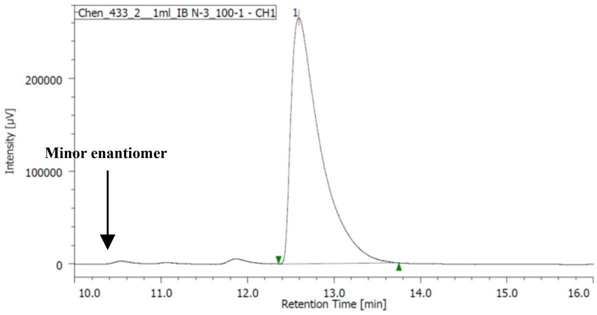
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_436_4__1ml_IB N-3_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.400	3338336	198524	28.632	33.746	N/A	10663	4.656	1.901	
2	Unknown	1	12.583	3359986	154081	28.818	26.191	N/A	8769	2.314	2.528	
3	Unknown	1	13.783	2465735	116270	21.148	19.764	N/A	12052	2.493	2.204	
4	Unknown	1	15.025	2495278	119419	21.402	20.299	N/A	14640	N/A	1.980	

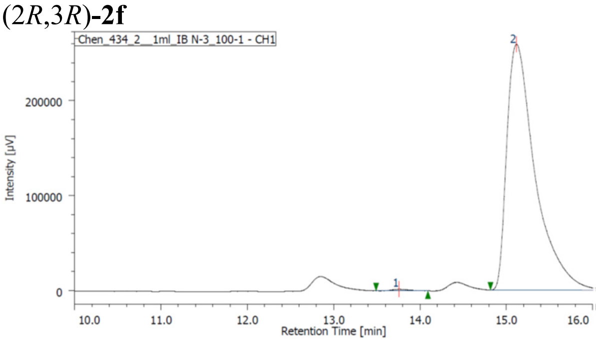
(2*S*,3*R*)-**2f**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_433_2_1ml_IB N-3_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	12.592	6259043	265136	100.000	100.000	N/A	7616	N/A	2.796	

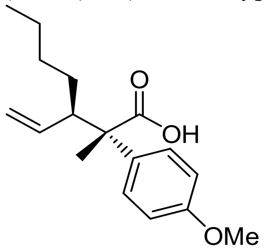
(2*R*,3*R*)-**2f**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_434_2_1ml_IB N-3_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

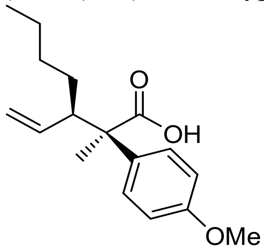
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.758	24711	1692	0.416	0.649	N/A	20390	2.892	1.385	
2	Unknown	1	15.117	5914814	258861	99.584	99.351	N/A	11805	N/A	2.015	

(2*S*,3*R*)-2-(4-methoxyphenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2g**)



Yield: 50%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.9 Hz, 3H), 1.19-1.42 (m, 6H), 1.51 (s, 3H), 2.85-2.90 (m, 1H), 3.79 (s, 3H), 4.76 (dd, *J* = 18.8, 1.8 Hz, 1H), 4.89 (dd, *J* = 10.5, 1.8 Hz, 1H), 5.31 (ddd, *J* = 18.8, 10.5, 9.6 Hz, 1H), 6.84 (d, *J* = 9.2 Hz, 2H), 7.34 (d, *J* = 9.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 16.8, 22.6, 30.3, 30.5, 51.0, 53.0, 55.1, 113.4, 117.9, 127.9, 133.6, 137.1, 158.3, 181.7; IR (neat): 2932, 1697, 1610, 1513, 1253, 1187, 1036, 917, 826 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol/TFA = 100/1/0.05; flow rate: 2.0 mL/min; detection: at 210 nm; rt): t_R = 13.2 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_3$ $[M+Na]^+$ 299.1618. Found 299.1618; $[\alpha]_D^{20}$ = 42.3 (*c* = 1.4, $CHCl_3$).

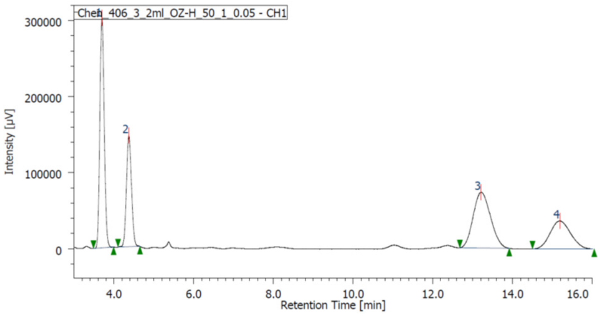
(2*R*,3*R*)-2-(4-methoxyphenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2g**)



Yield: 80%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.00-1.28 (m, 6H), 1.49 (s, 3H), 2.88-2.92 (m, 1H), 3.80 (s, 3H), 5.11-5.16 (m, 2H), 5.57 (ddd, *J* = 17.2, 8.6, 8.6 Hz, 1H), 6.86 (d, *J* = 8.9 Hz, 2H), 7.38 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.1, 22.4, 28.2, 30.0, 50.3, 53.2, 55.2, 113.4, 118.4, 128.4, 138.2, 138.2, 158.4, 181.5; IR (neat): 2933, 1697, 1609, 1513, 1254, 1187, 1035, 919, 827 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol/TFA = 100/1/0.05; flow rate: 2.0 mL/min; detection: at 210 nm; rt): t_R = 4.4 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_3$ $[M+Na]^+$ 299.1618. Found 299.1618; $[\alpha]_D^{20}$ = 63.5 (c = 1.6, $CHCl_3$).

<u>HPLC chart</u>

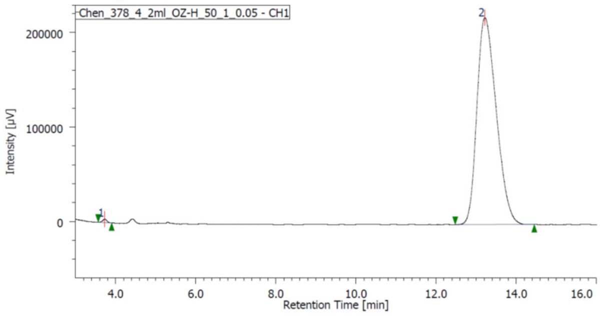
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_406_3_2ml_OZ-H_50_1_0.05-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	3.700	2301623	302380	32.299	54.135	N/A	5420	3.117	1.095	
2	Unknown	1	4.375	1289124	146114	18.091	26.159	N/A	5629	16.868	1.062	
3	Unknown	1	13.208	2219567	73184	31.148	13.102	N/A	4183	2.235	1.148	
4	Unknown	1	15.183	1315645	36890	18.463	6.604	N/A	4042	N/A	1.097	

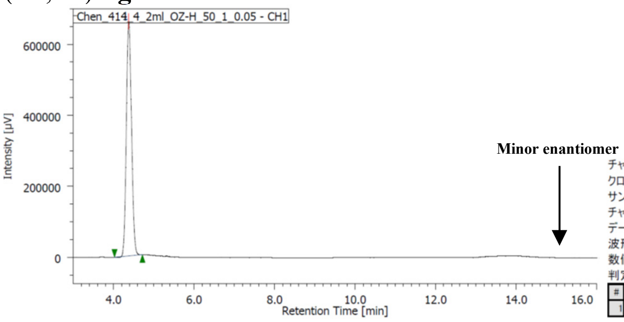
(2*S*,3*R*)-**2g**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_378_4_2ml_OZ-H_50_1_0.05-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	3.725	25938	3553	0.352	1.597	N/A	5745	17.451	1.045	
2	Unknown	1	13.217	7344714	218895	99.648	98.403	N/A	3496	N/A	1.306	

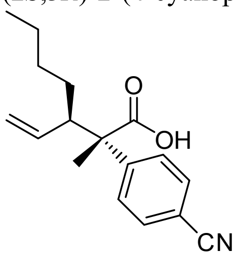
(2*R*,3*R*)-**2g**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_414_4_2ml_OZ-H_50_1_0.05-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	4.375	5915240	660653	100.000	100.000	N/A	5412	N/A	1.134	

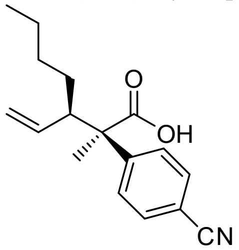
(2*S*,3*R*)-2-(4-cyanophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2h**)



Yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, J = 6.9 Hz, 3H), 1.17-1.49 (m, 6H), 1.56 (s, 3H), 2.82-2.87 (m, 1H), 4.71 (dd, J = 17.9, 1.8 Hz, 1H), 4.88 (dd, J = 9.6, 1.8 Hz, 1H), 5.26 (ddd, J = 17.9, 9.6, 8.7 Hz, 1H), 7.53 (d, J = 8.5 Hz, 2H), 7.60 (d, J = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 16.7, 22.4, 30.1, 30.5, 51.5, 54.1, 110.9, 118.6, 118.9, 127.7, 131.9, 136.1, 147.0, 180.7; IR (neat): 2931, 2229, 1702, 1466, 1270, 1099, 920, 830 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent:

hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 30.2 min; HRMS (ESI): m/z calcd for $C_{17}H_{21}NO_2$ $[2M+Na]^+$ 565.3037. Found 565.3024; $[\alpha]_D^{20}$ = 55.0 (*c* = 2.1, $CHCl_3$).

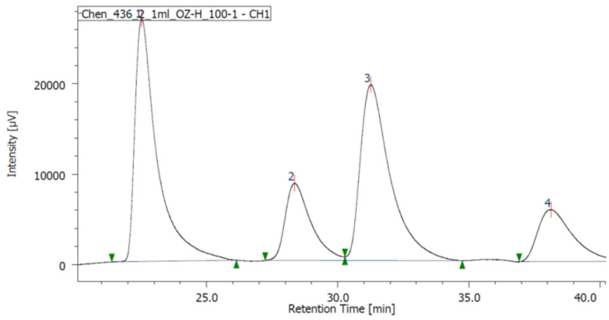
(2*R*,3*R*)-2-(4-cyanophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2h**)



Yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 7.1 Hz, 3H), 1.04-1.39 (m, 6H), 1.55 (s, 3H), 2.86-2.90 (m, 1H), 5.13-5.17 (m, 2H), 5.47-5.56 (m, 1H), 7.58 (d, *J* = 8.7 Hz, 2H), 7.64 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 17.3, 22.3, 28.4, 29.8, 50.7, 54.4, 111.0, 118.6, 119.2, 128.2, 131.9, 137.2, 146.2, 180.4; IR (neat): 2932, 2229, 1702, 1458, 1272, 1091, 921, 830 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 27.5 min; HRMS (ESI): m/z calcd for $C_{17}H_{21}NO_2$ $[M+Na]^+$ 294.1465. Found 294.1476; $[\alpha]_D^{20}$ = 57.9 (*c* = 0.55, $CHCl_3$).

<u>HPLC chart</u>

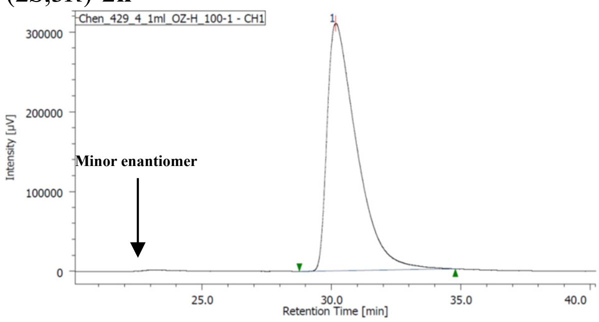
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_436_2_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	22.525	1586575	26849	37.251	44.308	N/A	4610	3.856	2.599	
2	Unknown	1	28.350	581709	8557	13.658	14.122	N/A	4437	1.592	1.702	
3	Unknown	1	31.258	1555231	19443	36.515	32.086	N/A	4067	3.169	1.888	
4	Unknown	1	38.125	535669	5747	12.577	9.484	N/A	4092	N/A	1.697	

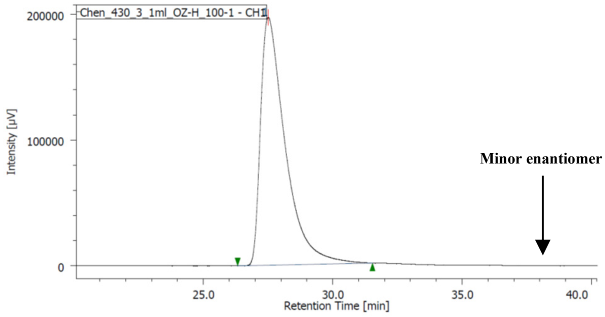
(2*S*,3*R*)-**2h**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_429_4_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	30.158	24665943	310332	100.000	100.000	N/A	3589	N/A	2.201	

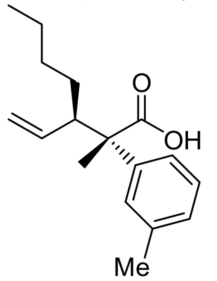
(2*R*,3*R*)-**2h**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_430_3_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

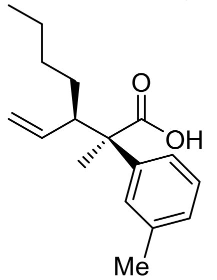
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	27.508	13065100	197100	100.000	100.000	N/A	4534	N/A	2.132	

(2*S*,3*R*)-2-methyl-2-(*m*-tolyl)-3-vinylheptanoic acid ((2*S*,3*R*)-**2i**)



Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, J = 6.9 Hz, 3H), 1.15-1.49 (m, 6H), 1.53 (s, 3H), 2.34 (s, 3H), 2.87-2.91 (m, 1H), 4.77 (dd, J = 18.2, 1.6 Hz, 1H), 4.89 (dd, J = 10.1, 1.6 Hz, 1H), 5.32 (ddd, J = 18.2, 10.1, 8.0 Hz, 1H), 7.04-7.05 (m, 1H), 7.20-7.21 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 17.1, 21.7, 22.6, 30.3, 30.4, 51.0, 53.6, 117.8, 123.8, 127.4, 127.6, 128.0, 137.1, 137.6, 141.5, 181.8; IR (neat): 2930, 1697, 1465, 1270, 916, 723 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 11.0 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1673; $[\alpha]_D^{20}$ = 71.9 (c = 1.8, $CHCl_3$).

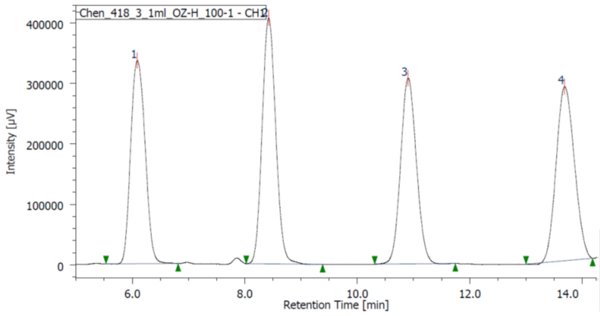
(2*R*,3*R*)-2-methyl-2-(*m*-tolyl)-3-vinylheptanoic acid ((2*R*,3*R*)-**2i**)



Yield: 69%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.79 (t, J = 6.6 Hz, 3H), 1.02-1.26 (m, 6H), 1.52 (s, 3H), 2.36 (s, 3H), 2.93-2.96 (m, 1H), 5.12-5.18 (m, 2H), 5.60 (ddd, J = 17.2, 8.4, 8.4 Hz, 1H), 7.08 (d, J = 6.9 Hz, 1H), 7.20-7.27 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.9, 17.1, 21.7, 22.3, 28.1, 29.9, 50.1, 53.8, 118.4, 124.3, 127.7, 127.9, 128.0, 137.7, 138.3, 140.8, 181.2; IR (neat): 2930, 1698, 1457, 1273, 920, 721 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 8.4 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1673; $[\alpha]_D^{20}$ = 41.3 (c = 1.1, $CHCl_3$).

HPLC chart

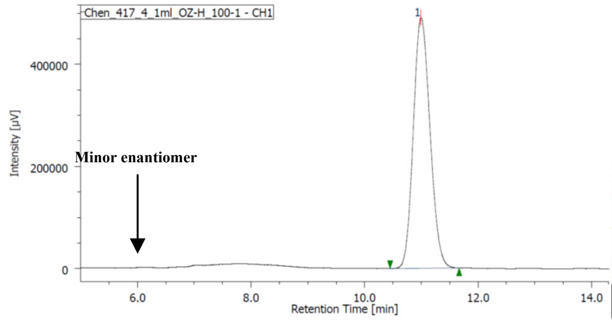
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_418_3_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.092	6000999	336102	23.691	25.104	N/A	2492	5.023	1.121	
2	Unknown	1	8.425	6841283	407124	27.008	30.409	N/A	5773	5.179	1.099	
3	Unknown	1	10.900	5991625	307525	23.654	22.969	N/A	7175	4.969	1.097	
4	Unknown	1	13.683	6496512	288097	25.647	21.518	N/A	8091	N/A	1.106	

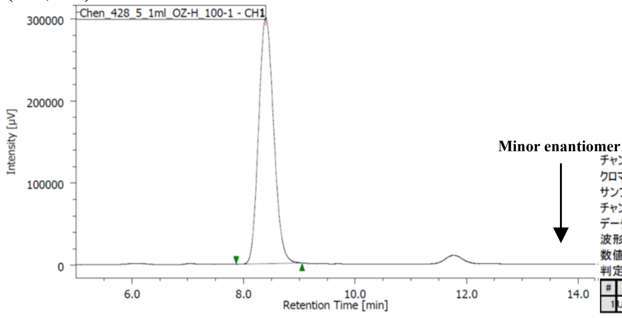
(2*S*,3*R*)-**2i**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_417_4_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.992	10035043	490849	100.000	100.000	N/A	6564	N/A	1.145	

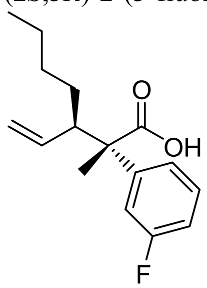
(2*R*,3*R*)-**2i**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_428_5_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

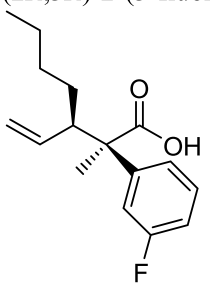
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	8.400	5629295	299217	100.000	100.000	N/A	4518	N/A	1.128	

(2*S*,3*R*)-2-(3-fluorophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2j**)



Yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.9 Hz, 3H), 1.19-1.50 (m, 6H), 1.53 (s, 3H), 2.83-2.88 (m, 1H), 4.75 (dd, *J* = 18.1, 1.6 Hz, 1H), 4.89 (dd, *J* = 10.0, 1.6 Hz, 1H), 5.30 (ddd, *J* = 18.1, 10.0, 8.0 Hz, 1H), 6.90-6.95 (m, 1H), 7.12-7.16 (m, 1H), 7.20 (d, *J* = 8.2 Hz, 1H), 7.24-7.30 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 16.7, 22.5, 30.2, 30.5, 51.3, 53.7, 113.8 (d, *J* = 21.0 Hz), 114.1 (d, *J* = 23.8 Hz), 118.3, 122.4 (d, *J* = 2.9 Hz), 129.4 (d, *J* = 8.6 Hz), 136.6, 144.3 (d, *J* = 7.6 Hz), 162.7 (d, *J* = 248 Hz),181.4; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -41.90; IR (neat): 2932, 1700, 1590, 1269, 920, 726 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 12.6 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}FO_2$ $[M+Na]^+$ 287.1418. Found 287.1434; $[\alpha]_D^{20}$ = 72.9 (*c* = 0.93, $CHCl_3$).

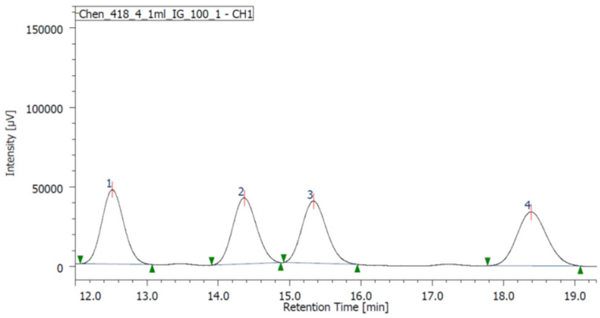
(2*R*,3*R*)-2-(3-fluorophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2j**)



Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.06-1.25 (m, 6H), 1.51 (s, 3H), 2.85-2.91 (m, 1H), 5.13-5.18 (m, 2H), 5.56 (ddd, *J* = 17.4, 8.7, 8.7 Hz, 1H), 6.94-6.98 (m, 1H), 7.18-7.32 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 17.0, 22.4, 28.2, 29.9, 50.6, 53.9, 114.0 (d, *J* = 21.9 Hz), 114.6 (d, *J* = 23.8 Hz), 118.8, 123.0 (d, *J* = 2.9 Hz), 129.5 (d, *J* = 8.6 Hz), 137.8, 143.5, 162.7 (d, *J* = 249 Hz), 180.9; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -41.84; IR (neat): 2933, 1702, 1589, 1269, 921, 724 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.2 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}FO_2$ $[M+Na]^+$ 287.1418. Found 287.1428; $[\alpha]_D^{20}$ = 50.3 (*c* = 1.2, $CHCl_3$).

HPLC chart

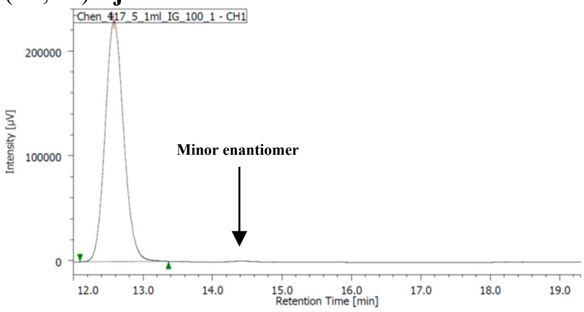
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_418_4_1ml_IG_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	12.517	1005850	46707	25.766	28.961	N/A	7698	3.107	1.086	
2	Unknown	1	14.367	967205	41563	24.776	25.771	N/A	8494	1.533	1.075	
3	Unknown	1	15.333	940296	39106	24.087	24.248	N/A	9166	4.316	1.151	
4	Unknown	1	18.383	990471	33899	25.372	21.019	N/A	8967	N/A	1.099	

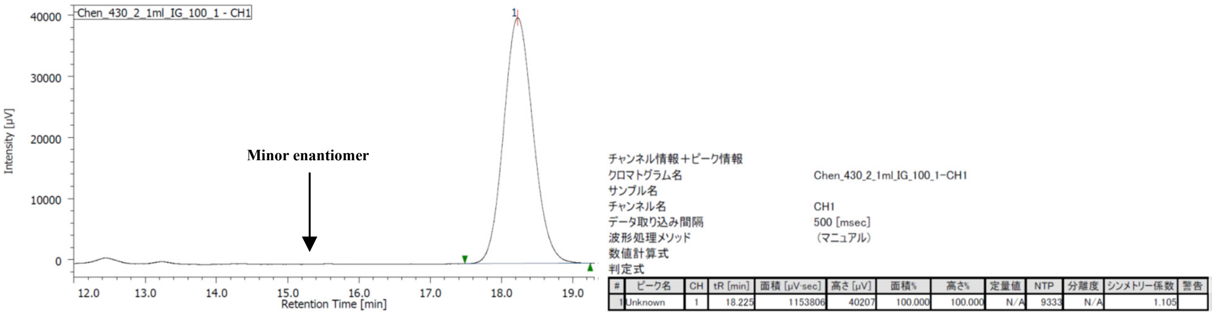
(2*S*,3*R*)-**2j**



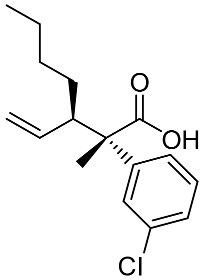
チャンネル情報＋ピーク情報
クロマトグラム名 Chen_417_5_1ml_IG_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	12.583	4259543	229269	100.000	100.000	N/A	10722	N/A	1.104	

(2*R*,3*R*)-**2j**

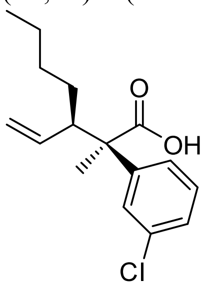


(2*S*,3*R*)-2-(3-chlorophenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2k**)



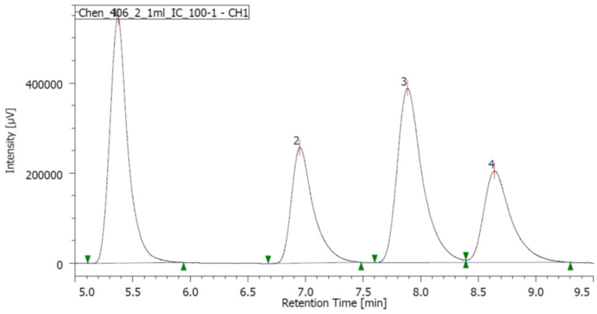
Yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.9 Hz, 3H), 1.20-1.42 (m, 6H), 1.53 (s, 3H), 2.83-2.87 (m, 1H), 4.75 (dd, *J* = 18.5, 1.6 Hz, 1H), 4.90 (dd, *J* = 10.5, 1.6 Hz, 1H), 5.29 (ddd, *J* = 18.5, 10.5, 9.2 Hz, 1H), 7.19-7.24 (m, 2H), 7.28-7.33 (m, 1H), 7.39-7.40 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 16.7, 22.5, 30.2, 30.4, 51.2, 53.7, 118.5, 125.1, 127.1, 127.1, 129.3, 134.1, 136.5, 143.7, 181.4; IR (neat): 2931, 1699, 1467, 1270, 920, 715 cm^{-1}; HPLC (chiral column: CHIRALPAK IC-3-IC-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 17.2 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115; $[\alpha]_D^{20}$ = 76.6 (*c* = 0.74, $CHCl_3$).

(2*R*,3*R*)-2-(3-chlorophenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2k**)



Yield: 91%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.79 (t, *J* = 6.6 Hz, 3H), 1.07-1.26 (m, 6H), 1.52 (s, 3H), 2.87-2.92 (m, 1H), 5.14-5.18 (m, 2H), 5.56 (ddd, *J* = 17.4, 8.4, 8.4 Hz, 1H), 7.24-7.29 (m, 2H), 7.37 (d, *J* = 7.4 Hz, 1H), 7.46 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 17.0, 22.3, 28.2, 29.9, 50.5, 54.0, 118.9, 125.6, 127.2, 127.6, 129.3, 134.2, 137.7, 143.0, 181.1; IR (neat): 2932, 1701, 1468, 1277, 922, 706 cm^{-1}; HPLC (chiral column: CHIRALPAK IC-3-IC-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 15.4 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115; $[\alpha]_D^{20}$ = 49.2 (*c* = 1.4, $CHCl_3$).

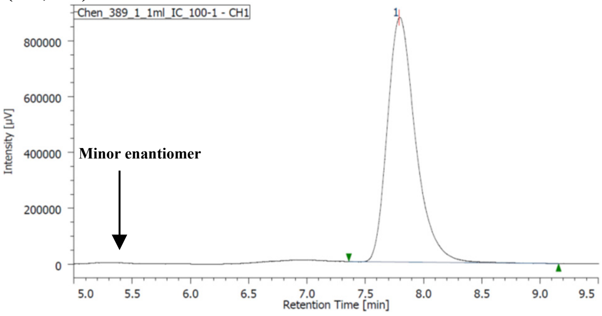
<u>HPLC chart</u>
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_406_2_1ml_IC_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.375	5934067	544867	31.886	39.143	N/A	6465	5.347	1.308	
2	Unknown	1	6.950	3388040	256906	18.205	18.456	N/A	7392	2.662	1.617	
3	Unknown	1	7.883	5908525	387150	31.749	27.813	N/A	6892	1.924	1.506	
4	Unknown	1	8.642	3379657	203052	18.160	14.587	N/A	7096	N/A	1.527	

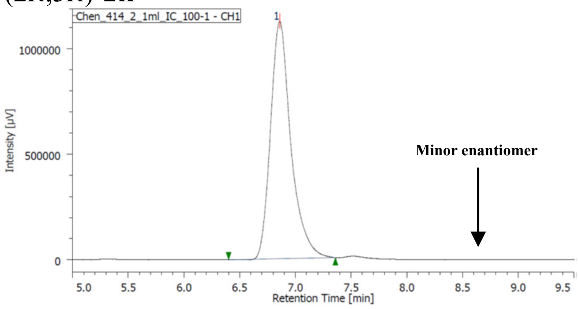
(2*S*,3*R*)-**2k**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_389_1_1ml_IC_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	7.792	14770630	876181	100.000	100.000	N/A	5309	N/A	1.353	

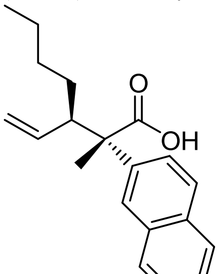
(2*R*,3*R*)-**2k**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_414_2_1ml_IC_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

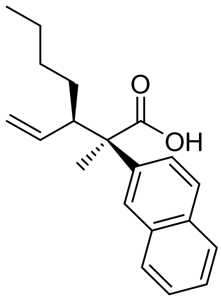
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.858	14301574	1126729	100.000	100.000	N/A	7611	N/A	1.326	

(2*S*,3*R*)-2-methyl-2-(naphthalen-2-yl)-3-vinylheptanoic acid ((2*S*,3*R*)-**2l**)



Yield: 62%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.6 Hz, 3H), 1.21-1.52 (m, 6H), 1.64 (s, 3H), 3.00-3.04 (m, 1H), 4.75 (dd, *J* = 17.4, 1.6 Hz, 1H), 4.81 (dd, *J* = 10.1, 1.6 Hz, 1H), 5.32 (ddd, *J* = 17.4, 10.1, 10.1 Hz, 1H), 7.42-7.44 (m, 2H), 7.60 (dd, *J* = 9.2, 1.8 Hz, 1H), 7.75-7.81 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 17.1, 22.6, 30.4, 30.5, 50.9, 53.8, 118.0, 125.1, 125.5, 125.9, 127.3, 127.7, 128.2, 132.3, 133.1, 136.9, 139.1, 181.1; IR (neat): 2930, 2858, 1697, 1457, 1272, 918, 746 cm^{-1}; HPLC (chiral column: CHIRALPAK IB N-5; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 27.8 min; HRMS (ESI): m/z calcd for $C_{20}H_{24}O_2$ $[M+Na]^+$ 319.1669. Found 319.1664; $[\alpha]_D^{20}$ = 34.1 (*c* = 0.74, $CHCl_3$).

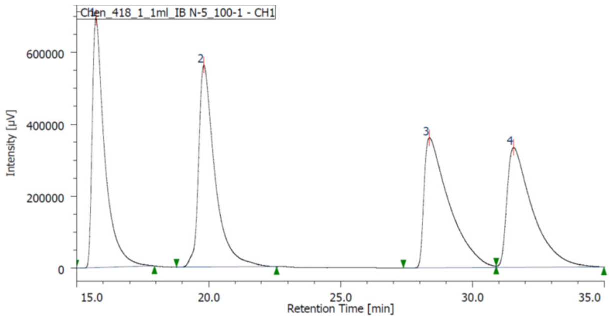
(2*R*,3*R*)-2-methyl-2-(naphthalen-2-yl)-3-vinylheptanoic acid ((2*R*,3*R*)-**2l**)



Yield: 77%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.75 (t, *J* = 6.9 Hz, 3H), 1.07-1.25 (m, 6H), 1.65 (s, 3H), 3.05-3.09 (m, 1H), 5.14-5.21 (m, 2H), 5.63 (ddd, *J* = 17.2, 8.4, 8.4 Hz, 1H), 7.46-7.49 (m, 2H), 7.66 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.79-7.85 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.2, 22.4, 28.3, 30.0, 50.2, 54.1, 118.6, 125.4, 126.0, 126.0, 126.3, 127.3, 127.6, 128.2, 132.3, 133.1, 138.1, 138.4, 181.6; IR (neat): 2931, 2858, 1698, 1465, 1275, 920, 746 cm^{-1}; HPLC (chiral column: CHIRALPAK IB N-5; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 19.9 min; HRMS (ESI): m/z calcd for $C_{20}H_{24}O_2$ $[M+Na]^+$ 319.1669. Found 319.1676; $[\alpha]_D^{20}$ = 33.7 (*c* = 0.82, $CHCl_3$).

HPLC chart

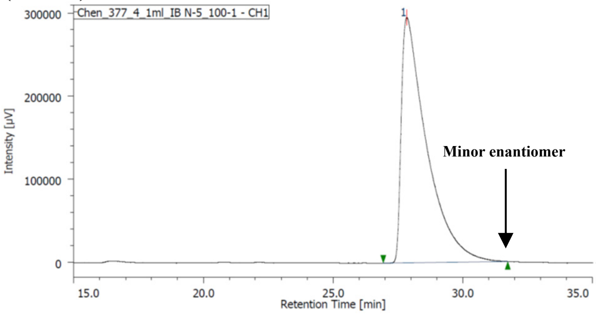
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_418_1_1ml_IB N-5_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.725	22992195	695702	24.580	35.624	N/A	6673	4.621	2.408	
2	Unknown	1	19.817	24191645	561481	25.862	28.751	N/A	6212	6.473	1.772	
3	Unknown	1	28.367	23804147	362091	25.448	18.541	N/A	4769	1.944	3.167	
4	Unknown	1	31.558	22552888	333641	24.110	17.084	N/A	5855	N/A	2.580	

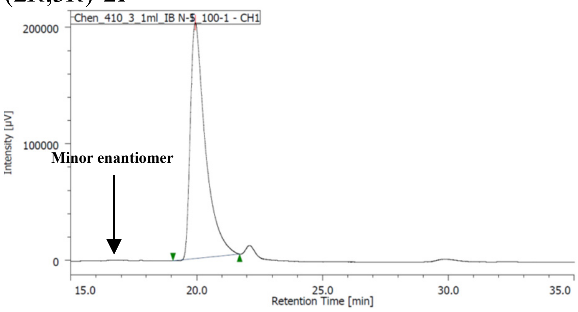
(2*S*,3*R*)-**2l**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_377_4_1ml_IB N-5_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	27.842	20025515	295485	100.000	100.000	N/A	4629	N/A	3.315	

(2*R*,3*R*)-**2l**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_410_3_1ml_IB N-5_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	19.942	8409539	202403	100.000	100.000	N/A	6254	N/A	2.072	

(2*S*,3*R*)-2-methyl-2-(thiophen-3-yl)-3-vinylheptanoic acid ((2*S*,3*R*)-**2m**)

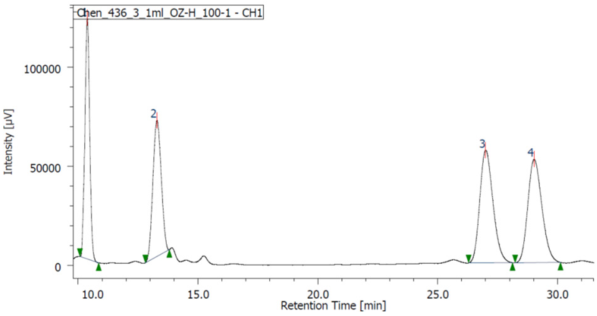
Yield: 97%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.6 Hz, 3H), 1.18-1.38 (m, 6H), 1.53 (s, 3H), 2.84-2.88 (m, 1H), 4.80 (dd, *J* = 17.7, 1.4 Hz, 1H), 4.93 (dd, *J* = 9.7, 1.4 Hz, 1H), 5.36 (ddd, *J* = 17.7, 9.7, 7.9 Hz, 1H), 7.11-7.12 (m, 1H), 7.18-7.19 (m, 1H), 7.23-7.25 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.1, 22.5, 30.1, 30.2, 51.3, 52.1, 118.0, 121.6, 124.9, 127.3, 137.0, 142.9, 181.2; IR (neat): 2930, 1699, 1465, 1273, 917, 714 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 29.1 min; HRMS (ESI): m/z calcd for $C_{14}H_{20}O_2S$ $[M+Na]^+$ 275.1076. Found 275.1082; $[\alpha]_D^{20}$ = 46.4 (*c* = 0.87, $CHCl_3$).

(2*R*,3*R*)-2-methyl-2-(thiophen-3-yl)-3-vinylheptanoic acid ((2*R*,3*R*)-**2m**)

Yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.03-1.25 (m, 6H), 1.51 (s, 3H), 2.81-2.86 (m, 1H), 5.11-5.16 (m, 2H), 5.54 (ddd, *J* = 16.9, 8.7, 8.7 Hz, 1H), 7.15-7.16 (m, 1H), 7.21-7.22 (m, 1H), 7.26-7.28 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.2, 22.4, 28.2, 29.9, 51.2, 52.4, 118.6, 122.1, 125.0, 127.5, 137.8, 142.4, 181.0; IR (neat): 2931, 1701, 1278, 1091, 921, 773 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 10.7 min; HRMS (ESI): m/z calcd for $C_{14}H_{20}O_2S$ $[M+Na]^+$ 275.1076. Found 275.1084; $[\alpha]_D^{20}$ = 51.9 (*c* = 1.2, $CHCl_3$).

<u>HPLC chart</u>

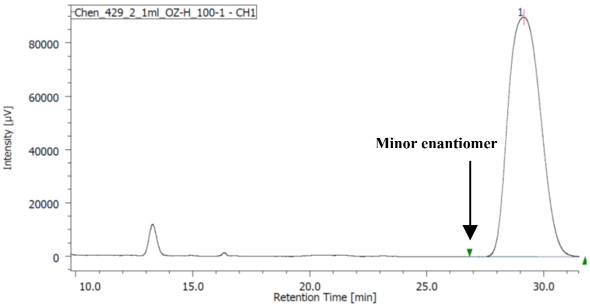
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_436_3_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.383	1692117	121755	22.096	40.668	N/A	13183	5.697	1.020	
2	Unknown	1	13.283	1666705	68635	21.765	22.925	N/A	6499	16.537	1.050	
3	Unknown	1	26.992	2170359	56882	28.342	19.000	N/A	11581	1.949	1.225	
4	Unknown	1	29.017	2128689	52115	27.797	17.407	N/A	11538	N/A	1.201	

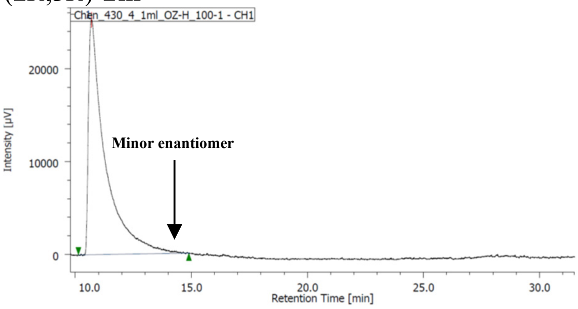
(2*S*,3*R*)-**2m**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_429_2_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	29.142	8664264	89819	100.000	100.000	N/A	1895	N/A	1.145	

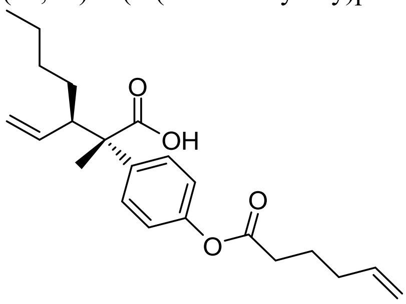
(2*R*,3*R*)-**2m**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_430_4_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

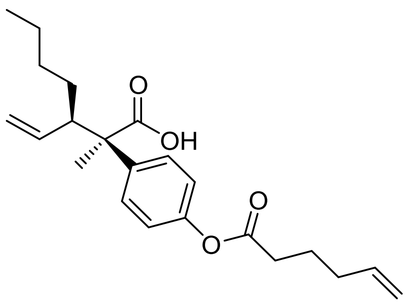
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.700	1262051	25259	100.000	100.000	N/A	1760	N/A	5.293	

(2*S*,3*R*)-2-(4-(hex-5-enoyloxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2n**)



Yield: 84%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.6 Hz, 3H), 1.22-1.43 (m, 6H), 1.53 (s, 3H), 1.85 (tt, *J* = 7.3, 7.3 Hz, 2H), 2.18 (dt, *J* = 7.3, 7.3 Hz, 2H), 2.55 (t, *J* = 7.3 Hz, 2H), 2.85-2.88 (m, 1H), 4.75 (dd, *J* = 17.2, 1.4 Hz, 1H), 4.89 (dd, *J* = 10.3, 1.4 Hz, 1H), 5.03 (dd, *J* = 9.2, 1.4 Hz, 1H), 5.07 (dd, *J* = 17.2, 1.4 Hz, 1H), 5.30 (ddd, *J* = 17.2, 9.2, 6.9 Hz, 1H), 5.78-5.86 (m, 1H), 7.02 (d, *J* = 8.9 Hz, 2H), 7.42 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 16.9, 22.5, 24.0, 30.3, 30.4, 33.0, 33.6, 51.3, 53.4, 115.6, 118.2, 121.0, 127.9, 136.8, 137.5, 139.0, 149.5, 171.9, 181.4; IR (neat): 2932, 1698, 1508, 1173, 1017, 917 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*S*,3*R*)-**2n**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 23.5 min. HRMS (ESI): m/z calcd for $C_{22}H_{30}O_4$ $[M+Na]^+$ 381.2036. Found 381.2038; $[\alpha]_D^{20}$ = 53.7 (*c* = 1.1, $CHCl_3$).

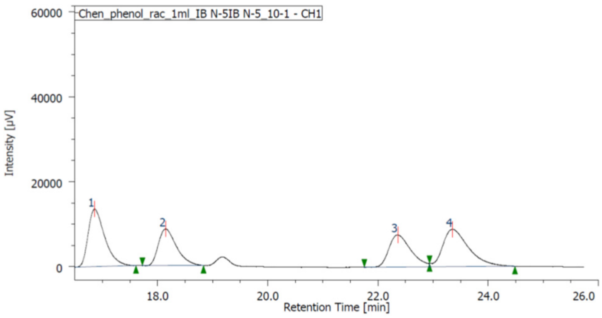
(2*R*,3*R*)-2-(4-(hex-5-enoyloxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2n**)



Yield: 84%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.04-1.23 (m, 6H), 1.52 (s, 3H), 1.86 (tt, *J* = 7.4, 7.4 Hz, 2H), 2.18 (dt, *J* = 7.4, 7.4 Hz, 2H), 2.57 (t, *J* = 7.4 Hz, 2H), 2.88-2.92 (m, 1H), 5.02-5.10 (m, 2H), 5.12-5.17 (m, 2H), 5.52-5.61 (m, 1H), 5.77-5.87 (m, 1H), 7.05 (d, *J* = 8.9 Hz, 2H), 7.47 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.2, 22.4, 24.0, 28.2, 30.0, 33.0, 33.6, 50.5, 53.6, 115.6, 118.7, 121.0, 128.4, 137.5, 137.9, 138.2, 149.6, 172.0, 181.0; IR (neat): 2933, 1699, 1508, 1173, 1017, 918 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*R*,3*R*)-**2n**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.3 min. HRMS (ESI): m/z calcd for $C_{22}H_{30}O_4$ $[M+Na]^+$ 381.2036. Found 381.2039; $[\alpha]_D^{20}$ = 42.6 (*c* = 1.2, $CHCl_3$).

<u>HPLC chart</u>

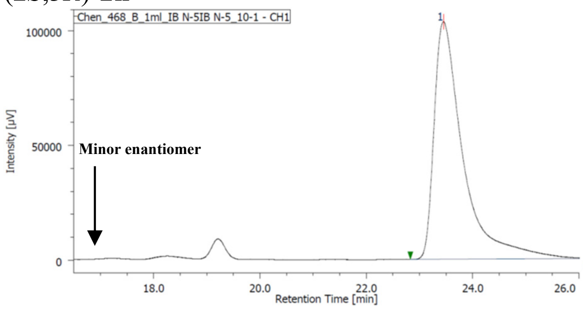
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_phenol_rac_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.858	283172	13532	29.012	35.184	N/A	15669	2.284	1.536	
2	Unknown	1	18.150	196944	8597	20.177	22.354	N/A	14879	6.397	1.459	
3	Unknown	1	22.358	207407	7543	21.249	19.612	N/A	15254	1.281	N/A	
4	Unknown	1	23.350	288545	8788	29.562	22.849	N/A	12709	N/A	N/A	

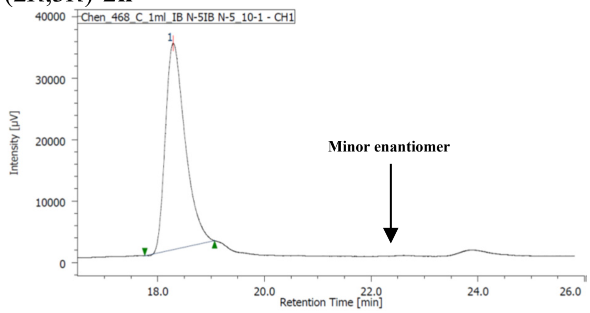
(2*S*,3*R*)-**2n**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_468_B_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	23.450	3912552	103381	100.000	100.000	N/A	11546	N/A	2.404	

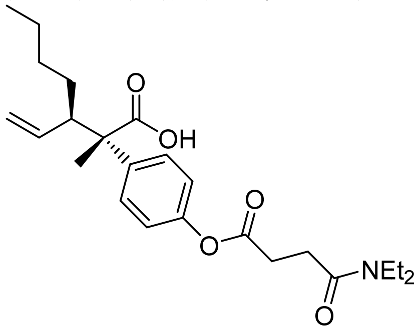
(2*R*,3*R*)-**2n**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_468_C_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

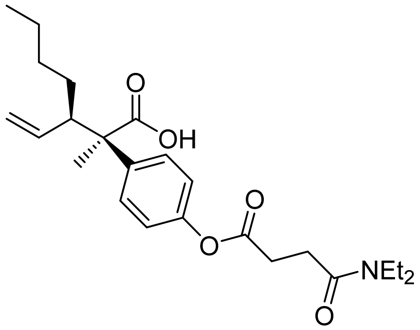
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.292	867781	33610	100.000	100.000	N/A	12010	N/A	1.480	

(2*S*,3*R*)-2-(4-((4-(diethylamino)-4-oxobutanoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2o**)



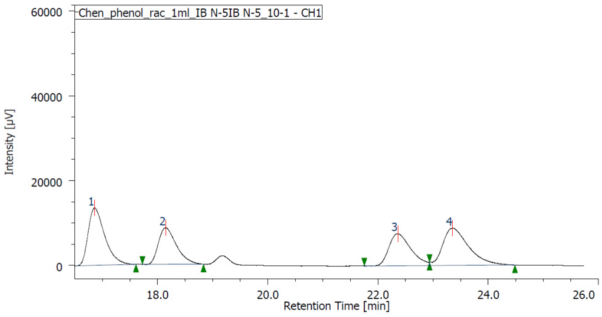
Yield: 89%; [1]H NMR (500 MHz, $CDCl_3$): δ = 0.86 (t, *J* = 6.9 Hz, 3H), 1.10 (t, *J* = 7.0 Hz, 3H), 1.20 (t, *J* = 7.0 Hz, 3H), 1.24-1.44 (m, 6H), 1.51 (s, 3H), 2.70 (t, *J* = 6.7 Hz, 2H), 2.84-2.88 (m, 1H), 2.90 (t, *J* = 6.7 Hz, 2H), 3.32-3.41 (m, 4H), 4.74 (d, *J* = 18.1 Hz, 1H), 4.87 (d, *J* = 10.9 Hz, 1H), 5.30 (ddd, *J* = 18.1, 10.9, 10.9 Hz, 1H), 7.01 (d, *J* = 8.6 Hz, 2H), 7.40 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.0, 14.0, 14.1, 17.1, 22.5, 27.8, 29.6, 30.3, 30.4, 40.5, 41.9, 51.3, 53.3, 118.0, 121.0, 127.8, 137.0, 139.2, 149.5, 170.3, 171.8, 180.2; IR (neat): 2934, 1715, 1615, 1456, 1210, 1138, 917, 737 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*S*,3*R*)-**2o**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 23.8 min.
HRMS (ESI): m/z calcd for $C_{24}H_{35}NO_5$ $[M+Na]^+$ 440.2407. Found 440.2428; $[\alpha]_D^{20}$ = 39.7 (*c* = 1.8, $CHCl_3$).

(2*R*,3*R*)-2-(4-((4-(diethylamino)-4-oxobutanoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2o**)



Yield: 85%; [1]H NMR (400 MHz, $CDCl_3$): δ = 0.77 (t, *J* = 6.9 Hz, 3H), 1.00-1.27 (m, 12H), 1.51 (s, 3H), 2.71 (t, *J* = 6.9 Hz, 2H), 2.88-2.92 (m, 3H), 3.31-3.41 (m, 4H), 5.10-5.16 (m, 2H), 5.56 (ddd, *J* = 19.2, 10.1, 10.1 Hz, 1H), 7.05 (d, *J* = 9.2 Hz, 2H), 7.45 (d, *J* = 9.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.0, 14.0, 14.1, 17.3, 22.4, 27.9, 28.2, 29.6, 30.0, 40.4, 41.9, 50.4, 53.5, 118.5, 121.0, 128.3, 138.1, 138.3, 149.6, 170.2, 171.8, 180.1; IR (neat): 2933, 1731, 1614, 1457, 1211, 1136, 919, 737 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*R*,3*R*)-**2o**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.5 min.
HRMS (ESI): m/z calcd for $C_{24}H_{35}NO_5$ $[M+Na]^+$ 440.2407. Found 440.2426; $[\alpha]_D^{20}$ = 36.1 (*c* = 1.1, $CHCl_3$).

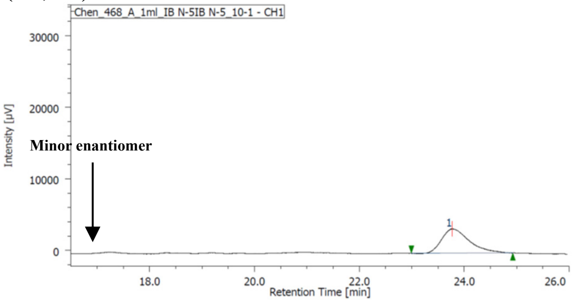
<u>HPLC chart</u>
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_phenol_rac_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.858	283172	13532	29.012	35.184	N/A	15669	2.284	1.536	
2	Unknown	1	18.150	196944	8597	20.177	22.354	N/A	14879	6.397	1.459	
3	Unknown	1	22.358	207407	7543	21.249	19.612	N/A	15254	1.281	N/A	
4	Unknown	1	23.350	288545	8788	29.562	22.849	N/A	12709	N/A	N/A	

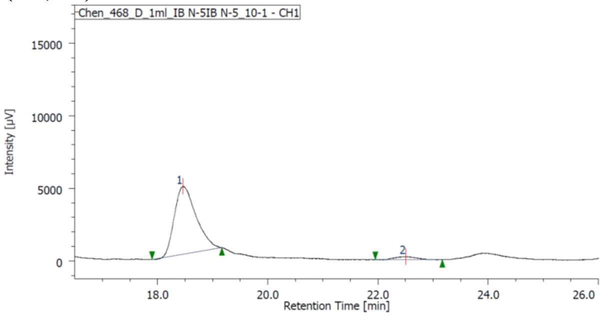
(2*S*,3*R*)-**2o**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_468_A_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	23.767	125190	3391	100.000	100.000	N/A	10183	N/A	1.562	

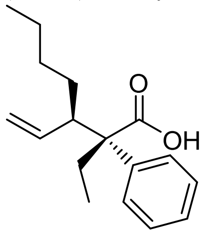
(2*R*,3*R*)-**2o**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_468_D_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

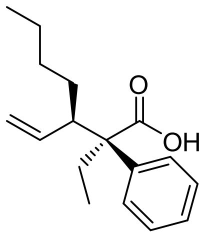
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.458	128231	4669	95.729	95.709	N/A	10380	5.447	1.408	
2	Unknown	1	22.508	5721	209	4.271	4.291	N/A	13805	N/A	1.000	

(2*S*,3*R*)-2-ethyl-2-phenyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2p**)



Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.70 (t, *J* = 7.6 Hz, 3H), 0.80 (t, *J* = 7.1 Hz, 3H), 1.01-1.30 (m, 5H), 1.46-1.52 (m, 1H), 1.93-2.12 (m, 2H), 2.62-2.68 (m, 1H), 5.07-5.16 (m, 2H), 5.55 (ddd, *J* =17.4, 10.1, 10.1 Hz, 1H), 7.24-7.26 (m, 3H), 7.30-7.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 9.1, 14.1, 22.5, 29.2, 30.2, 30.4, 50.5, 59.2, 117.8, 126.5, 127.7, 128.4, 138.5, 139.2, 180.8; IR (neat): 2934, 1698, 1461, 1255, 918, 699 cm^{-1}; HPLC (chiral column: CHIRALPAK IB N-5; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 8.9 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1673; $[\alpha]_D^{20}$ = -1.1 (*c* = 1.5, $CHCl_3$).

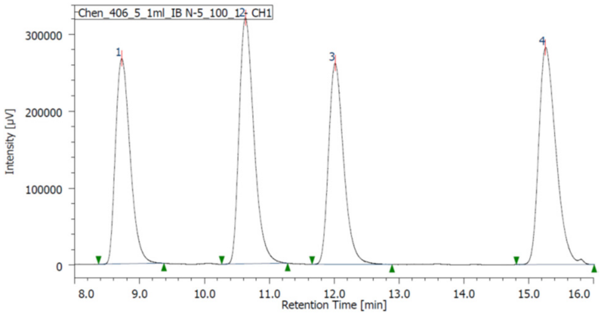
(2*R*,3*R*)-2-ethyl-2-phenyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2p**)



Yield: 77%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.83 (t, J = 7.2 Hz, 3H), 0.89 (t, J = 7.4 Hz, 3H), 1.13-1.34 (m, 5H), 1.59-1.65 (m, 1H), 2.12-2.16 (m, 2H), 2.65-2.69 (m, 1H), 5.01-5.13 (m, 3H), 7.23-7.26 (m, 3H), 7.30-7.33 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 9.0, 14.1, 22.5, 29.6, 30.0, 30.9, 49.6, 58.8, 102.9, 117.7, 126.7, 127.3, 129.1, 138.0, 181.4; IR (neat): 2932, 1699, 1459, 1251, 917, 702 cm^{-1}; HPLC (chiral column: CHIRALPAK IB N-5; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 15.1 min; HRMS (ESI): m/z calcd for $C_{17}H_{24}O_2$ $[M+Na]^+$ 283.1669. Found 283.1681; $[\alpha]_D^{20}$ = 1.5 (c = 2.0, $CHCl_3$).

<u>HPLC chart</u>

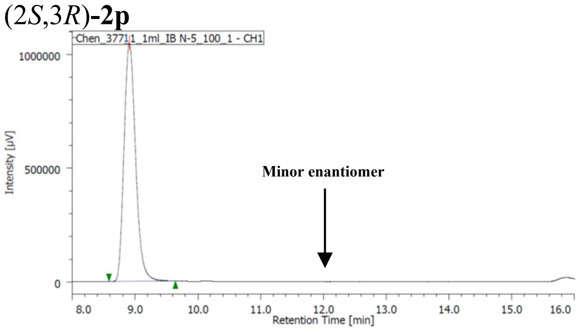
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_406_5_1ml_IB N-5_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	8.725	4169076	266663	22.361	23.613	N/A	7128	4.636	1.358	
2	Unknown	1	10.633	5072495	319640	27.207	28.304	N/A	10648	3.345	1.366	
3	Unknown	1	12.017	4216320	261136	22.615	23.123	N/A	13276	7.252	1.300	
4	Unknown	1	15.258	5186299	281872	27.817	24.960	N/A	16221	N/A	1.388	

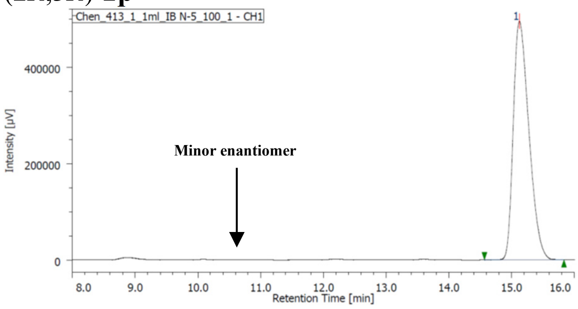
(2S,3R)-**2p**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_377_1_1ml_IB N-5_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	8.908	13459753	1048480	100.000	100.000	N/A	11362	N/A	1.219	

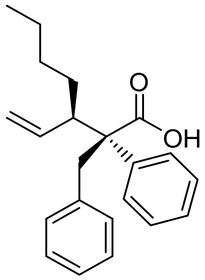
(2R,3R)-**2p**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_413_1_1ml_IB N-5_100_1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

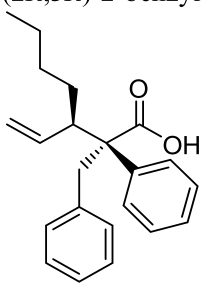
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.125	8576268	495778	100.000	100.000	N/A	17375	N/A	1.406	

(2S,3R)-2-benzyl-2-phenyl-3-vinylheptanoic acid ((2S,3R)-**2q**)



Yield: 72%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85 (t, *J* = 7.1 Hz, 3H), 1.10-1.41 (m, 5H), 1.66-1.71 (m, 1H), 2.77-2.81 (m, 1H), 3.08 (d, *J* = 14.2 Hz, 1H), 3.45 (d, *J* = 14.2 Hz, 1H), 5.25 (dd, *J* = 17.2, 1.8 Hz, 1H), 5.34 (dd, *J* =10.3, 1.8 Hz, 1H), 5.72 (ddd, *J* = 17.2, 10.3, 10.3 Hz, 1H), 6.71 (d, *J* = 7.3 Hz, 2H), 7.03-7.11 (m, 5H), 7.25-7.27 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.1, 22.6, 29.6, 30.3, 44.1, 49.3, 60.4, 118.7, 126.1, 126.7, 127.4, 127.6, 128.7, 130.8, 137.2, 138.2, 139.1, 180.1; IR (neat): 3031, 2930, 1698, 1496, 1234, 918, 700 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 14.9 min; HRMS (ESI): m/z calcd for $C_{22}H_{26}O_2$ $[M+Na]^+$ 345.1825. Found 345.1813; $[\alpha]_D^{20}$ = 5.8 (*c* = 2.0, $CHCl_3$).

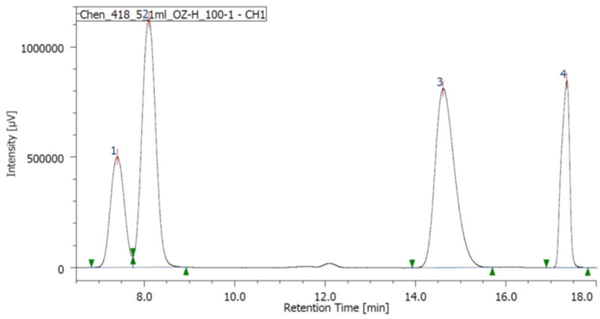
(2*R*,3*R*)-2-benzyl-2-phenyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2q**)



Yield: 69%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.90 (t, *J* = 6.9 Hz, 3H), 1.20-1.36 (m, 5H), 1.77-1.80 (m, 1H), 2.76-2.80 (m, 1H), 3.40 (d, *J* = 13.7 Hz, 1H), 3.47 (d, *J* = 13.7 Hz, 1H), 5.14 (dd, *J* = 17.1, 1.9 Hz, 1H), 5.21 (dd, *J* = 9.3, 1.9 Hz, 1H), 5.43 (ddd, *J* = 17.1, 9.3, 8.4 Hz, 1H), 7.04 (d, *J* = 6.9 Hz, 2H), 7.15-7.20 (m, 3H), 7.25 (d, *J* = 7.4 Hz, 2H), 7.29-7.35 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 22.6, 29.9, 30.5, 42.8, 48.8, 60.0, 118.5, 126.5, 126.8, 127.2, 127.8, 129.4, 130.4, 137.0, 138.1, 138.6, 180.6; IR (neat): 3031, 2929, 1700, 1497, 1265, 917, 701 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 7.6 min; HRMS (ESI): m/z calcd for $C_{22}H_{26}O_2$ $[M+Na]^+$ 345.1825. Found 345.1813; $[\alpha]_D^{20}$ = 10.6 (*c* = 3.8, $CHCl_3$).

<u>HPLC chart</u>

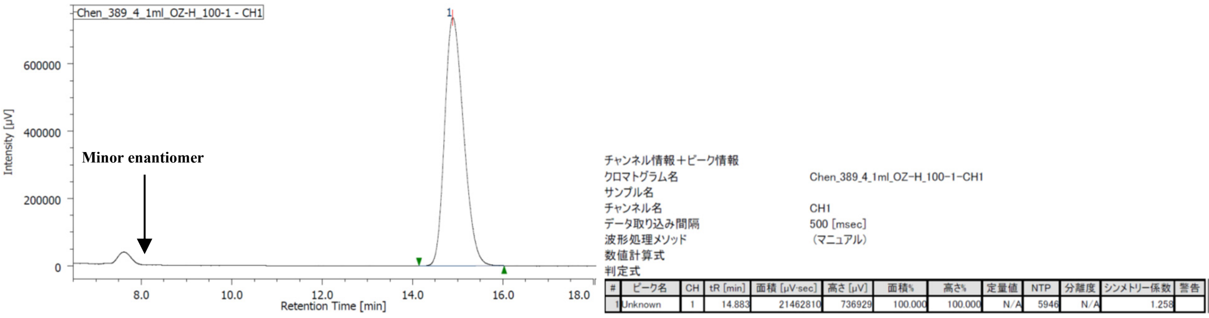
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_418_5_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

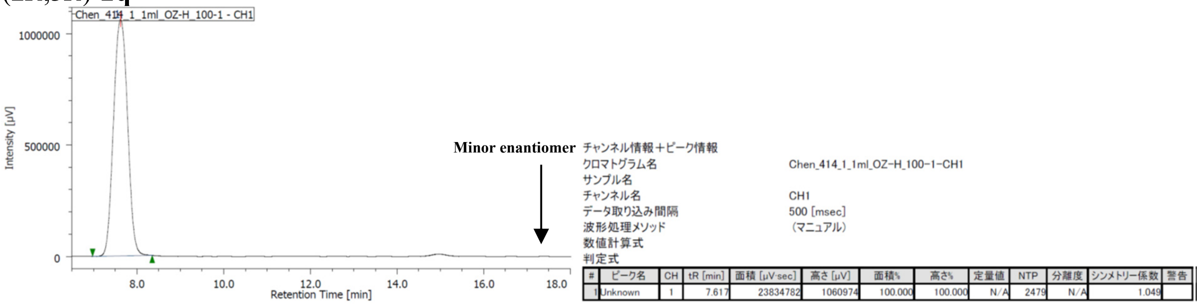
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	7.400	10694772	500871	15.348	15.262	N/A	2584	1.219	N/A	
2	Unknown	1	8.100	23818664	1123126	34.182	34.224	N/A	3245	9.552	1.034	
3	Unknown	1	14.608	24520154	810281	35.189	24.691	N/A	5368	4.730	1.327	
4	Unknown	1	17.342	10647597	847437	15.280	25.823	N/A	36885	N/A	0.838	

(2*S*,3*R*)-**2q**



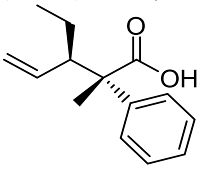
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.883	21462810	736929	100.000	100.000	N/A	5946	N/A	1.258	

(2*R*,3*R*)-**2q**



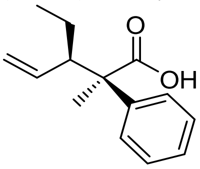
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	7.617	23834782	1060974	100.000	100.000	N/A	2479	N/A	1.049	

(2*S*,3*R*)-3-ethyl-2-methyl-2-phenylpent-4-enoic acid ((2*S*,3*R*)-**2r** [(2*S*,3*R*)-**2Et**])



Yield: 89%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.90 (t, *J* = 7.3 Hz, 3H), 1.26-1.39 (m, 2H), 1.54 (s, 3H), 2.77-2.82 (m, 1H), 4.77 (dd, *J* = 18.5, 1.8 Hz, 1H), 4.90 (dd, *J* = 10.3, 1.8 Hz, 1H), 5.30 (ddd, *J* = 18.5, 10.3, 9.2 Hz, 1H), 7.21-7.25 (m, 1H), 7.29-7.33 (m, 2H), 7.41-7.43 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 12.8, 17.0, 23.7, 53.3, 53.7, 118.2, 126.7, 126.9, 128.1, 136.6, 141.6, 181.8; IR (neat): 2964, 1696, 1458, 1272, 918, 699 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 15.1 min; HRMS (ESI): m/z calcd for $C_{14}H_{18}O_2$ $[M+Na]^+$ 241.1199. Found 241.1189; $[\alpha]_D^{20}$ = 98.9 (*c* = 0.69, $CHCl_3$).

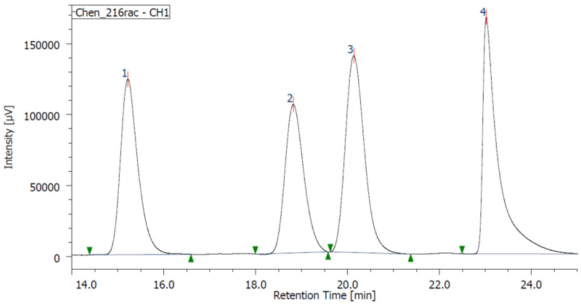
(2*R*,3*R*)-3-ethyl-2-methyl-2-phenylpent-4-enoic acid ((2*R*,3*R*)-**2r** [(2*R*,3*R*)-**2Et**])



Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.76 (t, *J* = 7.6 Hz, 3H), 0.96-1.07 (m, 1H), 1.13-1.24 (m, 1H), 1.52 (s, 3H), 2.79-2.84 (m, 1H), 5.12-5.17 (m, 2H), 5.55 (ddd, *J* = 19.7, 10.5, 9.2 Hz, 1H), 7.22-7.26 (m, 1H), 7.30-7.34 (m, 2H), 7.44 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 12.5, 17.2, 21.6, 52.5, 53.9, 118.8, 127.0, 127.2, 128.2, 137.7, 140.9, 181.2; IR (neat): 2963, 1698, 1457, 1273, 921, 697 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 21.3 min; HRMS (ESI): m/z calcd for $C_{14}H_{18}O_2$ $[M+Na]^+$ 241.1199. Found 241.1198; $[\alpha]_D^{20}$ = 79.2 (*c* = 0.85, $CHCl_3$).

HPLC chart

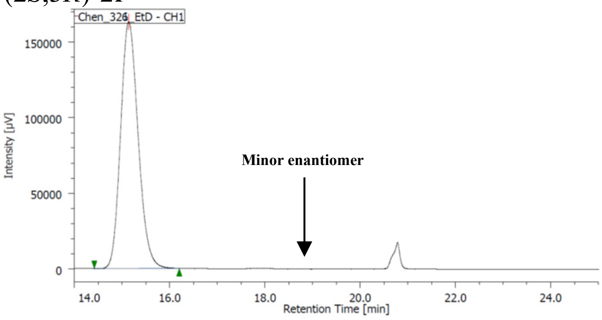
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_216rac−CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.217	3274956	123667	23.232	23.179	N/A	8165	5.078	1.358	
2	Unknown	1	18.817	2964459	104582	21.030	19.602	N/A	10126	1.758	1.245	
3	Unknown	1	20.133	3991837	138650	28.318	25.987	N/A	11415	4.695	1.271	
4	Unknown	1	23.017	3865227	166641	27.420	31.233	N/A	37135	N/A	3.840	

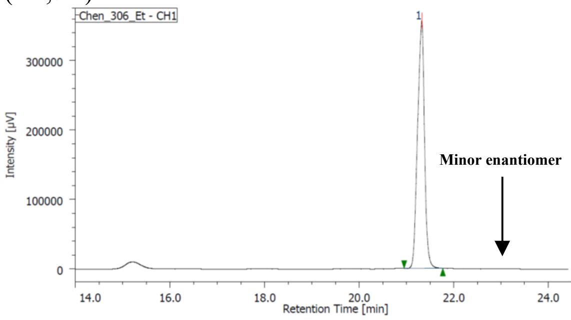
(2*S*,3*R*)-**2r**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_326_EtD−CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.142	4174378	163099	100.000	100.000	N/A	8176	N/A	1.163	

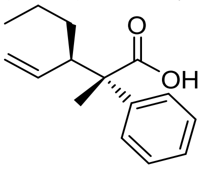
(2*R*,3*R*)-**2r**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_306_Et−CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

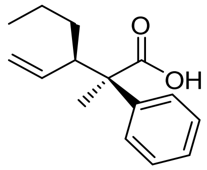
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	21.325	3633479	356696	100.000	100.000	N/A	98794	N/A	0.907	

(2*S*,3*R*)-2-methyl-2-phenyl-3-vinylhexanoic acid ((2*S*,3*R*)-**2s** [(2*S*,3*R*)-**2Pr**])



Yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.90 (t, *J* = 7.1 Hz, 3H), 1.19-1.43 (m, 4H), 1.55 (s, 3H), 2.88-2.93 (m, 1H), 4.75 (dd, *J* = 17.4, 1.8 Hz, 1H), 4.88 (dd, *J* = 10.1, 1.8 Hz, 1H), 5.32 (ddd, *J* = 17.4, 10.1, 10.1 Hz, 1H), 7.20-7.25 (m, 1H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.42 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.1, 21.1, 33.0, 51.0, 53.6, 117.9, 126.7, 126.9, 128.1, 137.0, 141.7, 181.0; IR (neat): 2961, 1697, 1457, 1267, 925, 697 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 12.9 min; HRMS (ESI): m/z calcd for $C_{15}H_{20}O_2$ $[M+Na]^+$ 255.1356. Found 255.1344; $[\alpha]_D^{20}$ = 97.4 (*c* = 0.39, $CHCl_3$).

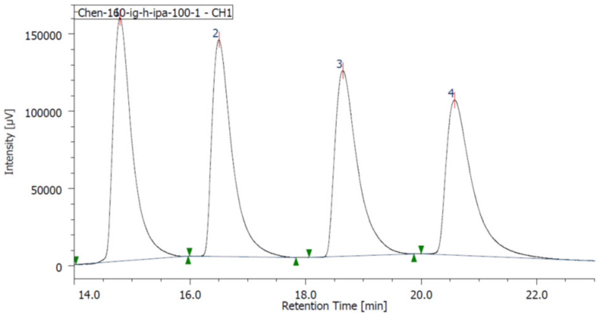
(2*R*,3*R*)-2-methyl-2-phenyl-3-vinylhexanoic acid ((2*R*,3*R*)-**2s** [(2*R*,3*R*)-**2Pr**])



Yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.77 (t, *J* = 7.1 Hz, 3H), 1.02-1.36 (m, 4H), 1.53 (s, 3H), 2.93-2.99 (m, 1H), 5.11-5.18 (m, 2H), 5.59 (ddd, *J* = 19.7, 10.5, 9.2 Hz, 1H), 7.24-7.28 (m, 1H), 7.32-7.36 (m, 2H), 7.45-7.48 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 17.2, 20.8, 30.7, 50.1, 53.9, 118.5, 127.0, 127.2, 128.1, 138.1, 140.8, 181.2; IR (neat): 2957, 1698, 1458, 1277, 921, 697 cm^{-1}; HPLC (chiral column: CHIRALPAK IG; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 16.4 min; HRMS (ESI): m/z calcd for $C_{15}H_{20}O_2$ $[M+Na]^+$ 255.1356. Found 255.1363; $[\alpha]_D^{20}$ = 52.1 (*c* = 1.1, $CHCl_3$).

HPLC chart

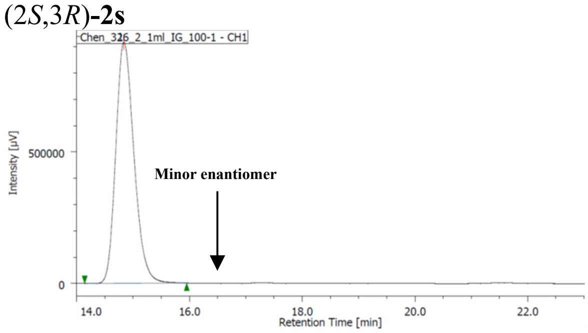
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen-160-ig-h-ipa-100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.792	3607596	157496	26.785	30.408	N/A	10992	2.933	1.908	
2	Unknown	1	16.500	3416800	140133	25.369	27.056	N/A	11961	3.331	2.055	
3	Unknown	1	18.642	3278221	120007	24.340	23.170	N/A	11819	2.668	1.963	
4	Unknown	1	20.575	3166012	100305	23.507	19.366	N/A	11512	N/A	2.236	

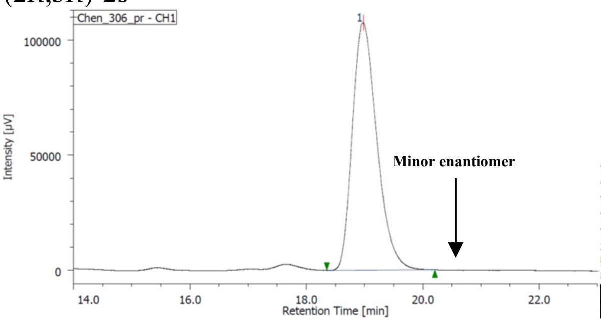
(2*S*,3*R*)-**2s**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_326_2_1ml_IG_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.833	20614938	917107	100.000	100.000	N/A	10281	N/A	1.243	

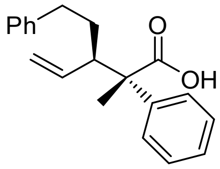
(2*R*,3*R*)-**2s**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_306_pr-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.975	3155925	107599	100.000	100.000	N/A	9784	N/A	1.300	

(2*S*,3*R*)-2-methyl-3-phenethyl-2-phenylpent-4-enoic acid ((2*S*,3*R*)-**2t**)



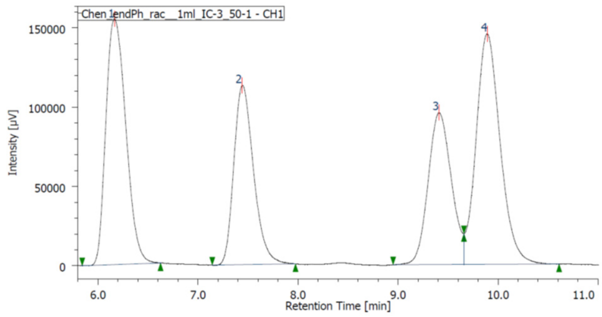
Yield: 63%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.53 (s, 3H), 1.61-1.71 (m, 1H), 1.76-1.84 (m, 1H), 2.46-2.54 (m, 1H), 2.66-2.74 (m, 1H), 2.89-2.93 (m, 1H), 4.85 (dd, J = 18.8, 1.6 Hz, 1H), 4.99 (dd, J = 10.5, 1.6 Hz, 1H), 5.41 (ddd, J = 18.8, 10.5, 9.6 Hz, 1H), 7.13-7.36 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.7, 32.7, 34.3, 51.1, 53.7, 118.6, 125.7, 126.6, 126.9, 128.2, 128.3, 128.5, 136.7, 141.5, 142.2, 181.3; IR (neat): 2930, 1697, 1496, 1268, 920, 699 cm^{-1}; HPLC (chiral column: CHIRALPAK IC-3; solvent: hexane/2-propanol = 50/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 9.5 min; HRMS (ESI): m/z calcd for $C_{20}H_{22}O_2$ $[M+Na]^+$ 317.1512. Found 317.1523; $[\alpha]_D^{20}$ = 35.0 (c = 1.8, $CHCl_3$).

(2*R*,3*R*)-2-methyl-3-phenethyl-2-phenylpent-4-enoic acid ((2*R*,3*R*)-**2t**)

Yield: 89%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.33-1.48 (m, 2H), 1.53 (s, 3H), 2.32-2.40 (m, 1H), 2.61-2.68 (m, 1H), 2.96-3.01 (m, 1H), 5.20-5.25 (m, 2H), 5.63-5.72 (m, 1H), 6.99-7.01 (m, 2H), 7.11-7.33 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 16.9, 30.3, 33.7, 49.4, 53.7, 119.3, 125.7, 127.0, 127.2, 128.2, 128.2, 128.5, 137.7, 140.5, 141.9, 181.1; IR (neat): 2945, 1698, 1496, 1274, 924, 698 cm^{-1}; HPLC (chiral column: CHIRALPAK IC-3; solvent: hexane/2-propanol = 50/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 6.5 min; HRMS (ESI): m/z calcd for $C_{20}H_{22}O_2$ $[M+Na]^+$ 317.1512. Found 317.1523; $[\alpha]_D^{20}$ = 30.5 (c = 1.4, $CHCl_3$).

<u>HPLC chart</u>

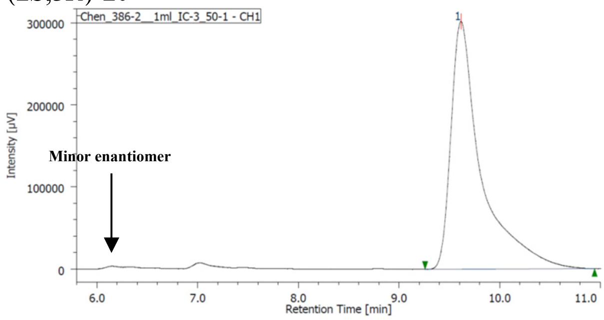
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_endPh_rac__1ml_IC-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.167	2149261	154992	27.985	30.445	N/A	4339	3.502	1.268	
2	Unknown	1	7.442	1549030	113030	20.170	22.202	N/A	7005	5.015	1.311	
3	Unknown	1	9.408	1546753	95738	20.140	18.806	N/A	7635	1.123	N/A	
4	Unknown	1	9.892	2434941	145336	31.705	28.548	N/A	8374	N/A	N/A	

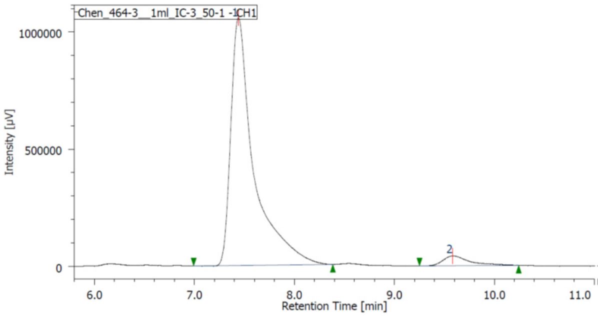
(2*S*,3*R*)-**2t**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_386-2__1ml_IC-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	9.617	6328751	301912	100.000	100.000	N/A	7769	N/A	2.332	

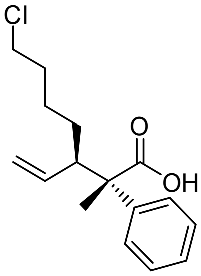
(2*R*,3*R*)-**2t**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_464-3_1ml_IC-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

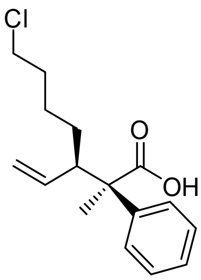
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	7.442	16857034	1057841	95.528	96.158	N/A	8022	5.632	2.364	
2	Unknown	1	9.583	789120	42271	4.472	3.842	N/A	7938	N/A	1.782	

(2*S*,3*R*)-7-chloro-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2u**)



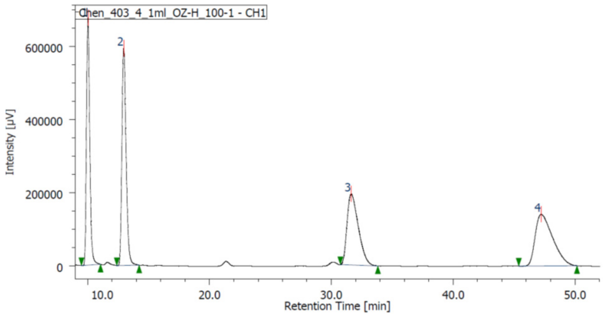
Yield: 84%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.32-1.53 (m, 4H), 1.55 (s, 3H), 1.68-1.86 (m, 2H), 2.85-2.90 (m, 1H), 3.52 (t, *J* = 6.9 Hz, 2H), 4.77 (dd, *J* = 18.5, 1.6 Hz, 1H), 4.91 (dd, *J* = 10.3, 1.6 Hz, 1H), 5.32 (ddd, *J* = 18.5, 10.3, 9.2 Hz, 1H), 7.22-7.26 (m, 1H), 7.32 (t, *J* = 7.3 Hz, 2H), 7.40 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.1, 25.3, 29.9, 32.3, 45.0, 51.1, 53.7, 118.4, 126.6, 127.0, 128.2, 136.6, 141.4, 181.9; IR (neat): 2940, 1697, 1445, 1273, 920, 700 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 31.8 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115; $[\alpha]_D^{20}$ = 54.4 (*c* = 0.90, $CHCl_3$).

(2*R*,3*R*)-7-chloro-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2u**)



Yield: 81%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.07-1.13 (m, 2H), 1.17-1.26 (m, 1H), 1.39-1.46 (m, 1H), 1.56 (s, 3H), 1.56-1.59 (m, 1H), 1.64-1.72 (m, 1H), 2.92-2.95 (m, 1H), 3.40 (td, *J* = 6.7, 1.9 Hz, 2H), 5.15-5.19 (m, 2H), 5.57 (ddd, *J* = 17.2, 8.3, 8.3 Hz, 1H), 7.25-7.28 (m, 1H), 7.34 (t, *J* = 7.7 Hz, 2H), 7.44-7.46 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.2, 25.0, 27.8, 32.2, 44.8, 50.2, 53.8, 119.0, 127.1, 127.2, 128.2, 137.6, 140.5, 181.4; IR (neat): 2940, 1698, 1445, 1273, 923, 699 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 13.0 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}ClO_2$ $[M+Na]^+$ 303.1122. Found 303.1115; $[\alpha]_D^{20}$ = 40.2 (*c* = 1.8, $CHCl_3$).

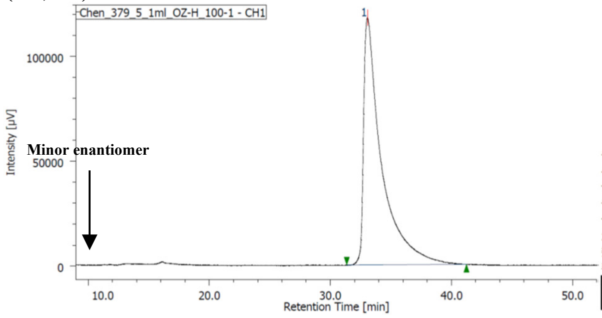
<u>HPLC chart</u>
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_403_4_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.033	12783168	677013	23.870	42.140	N/A	6813	5.339	1.246	
2	Unknown	1	12.967	14124874	593835	26.375	36.963	N/A	7098	16.076	1.293	
3	Unknown	1	31.625	12529722	194359	23.397	12.098	N/A	5461	7.141	1.541	
4	Unknown	1	47.217	14115628	141373	26.358	8.800	N/A	5016	N/A	1.647	

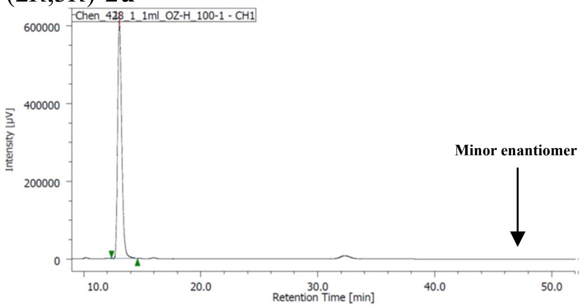
(2*S*,3*R*)-**2u**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_379_5_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	33.058	12106289	117756	100.000	100.000	N/A	3794	N/A	3.858	

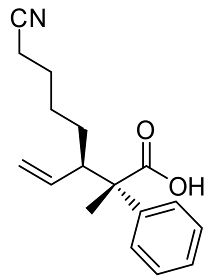
(2*R*,3*R*)-**2u**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_428_1_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

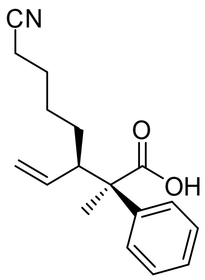
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.042	14819429	612925	100.000	100.000	N/A	6994	N/A	1.314	

(2*S*,3*R*)-7-cyano-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2v**)



Yield: 54%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.36-1.55 (m, 9H), 2.31 (t, *J* = 6.9 Hz, 2H), 2.82-2.86 (m, 1H), 4.77 (dd, *J* = 17.2, 1.1 Hz, 1H), 4.91 (dd, *J* = 9.3, 1.7 Hz, 1H), 5.32 (ddd, *J* = 17.2, 9.3, 8.3 Hz, 1H), 7.23-7.25 (m, 1H), 7.30-7.33 (m, 2H), 7.34-7.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.0, 17.3, 25.0, 27.0, 29.7, 51.1, 53.5, 118.6, 119.7, 126.5, 127.1, 128.3, 136.4, 141.3, 180.8; IR (neat): 3074, 2935, 2247, 1698, 1446, 1270, 1095, 921, 701 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 10.0 min; HRMS (ESI): m/z calcd for $C_{17}H_{21}NO_2$ $[M+Na]^+$ 294.1465. Found 294.1473; $[\alpha]_D^{20}$ = 55.6 (*c* = 0.77, $CHCl_3$).

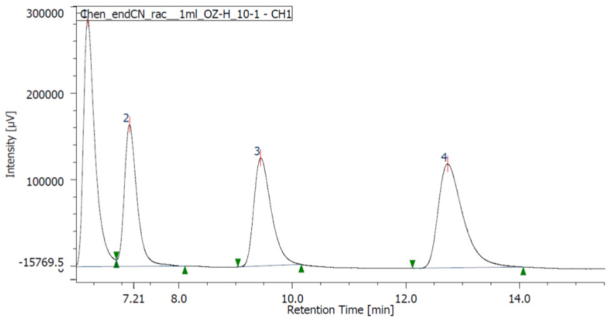
(2*R*,3*R*)-7-cyano-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2v**)



Yield: 82%; [1]H NMR (400 MHz, $CDCl_3$): δ = 1.01-1.15 (m, 2H), 1.18-1.26 (m, 1H), 1.37-1.57 (m, 6H), 2.18 (t, J = 7.1 Hz, 2H), 2.89-2.94 (m, 1H), 5.15-5.19 (m, 2H), 5.56 (ddd, J = 16.9, 9.0, 9.0 Hz, 1H), 7.26-7.29 (m, 1H), 7.33-7.36 (m, 2H), 7.43-7.45 (m, 2H); [13]C NMR (100 MHz, $CDCl_3$): δ = 16.9, 17.2, 24.9, 26.7, 27.6, 50.1, 53.7, 119.2, 119.6, 127.1, 127.2, 128.3, 137.4, 140.4, 181.2; IR (neat): 3062, 2942, 2247, 1697, 1445, 1271, 1121, 925, 701 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 7.1 min; HRMS (ESI): m/z calcd for $C_{17}H_{21}NO_2$ $[M+Na]^+$ 294.1465. Found 294.1492; $[\alpha]_D^{20}$ = 40.0 (c = 1.9, $CHCl_3$).

<u>HPLC chart</u>

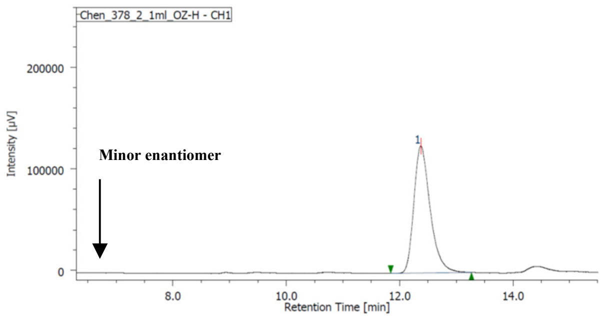
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_endCN_rac__1ml_OZ-H_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.400	4082512	286579	31.532	41.176	N/A	5348	2.002	1.710	
2	Unknown	1	7.133	2544690	164069	19.655	23.573	N/A	5510	4.963	1.299	
3	Unknown	1	9.442	2663016	125081	20.568	17.972	N/A	4744	5.007	1.488	
4	Unknown	1	12.733	3656889	120264	28.245	17.279	N/A	4374	N/A	1.624	

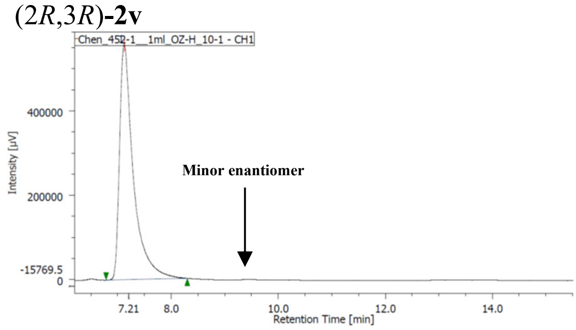
(2*S*,3*R*)**-2v**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_378_2_1ml_OZ-H-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	12.375	2498674	124835	100.000	100.000	N/A	9457	N/A	1.346	

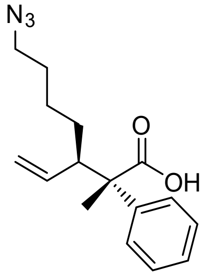
(2*R*,3*R*)**-2v**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_452-1__1ml_OZ-H_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

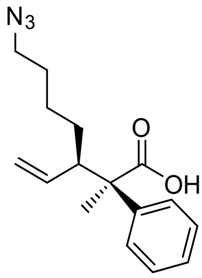
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	7.125	10019854	558439	100.000	100.000	N/A	4741	N/A	2.080	

(2*S*,3*R*)-7-azido-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2w**)



Yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.26-1.71 (m, 9H), 2.84-2.89 (m, 1H), 3.24 (t, *J* = 7.1 Hz, 2H), 4.76 (dd, *J* = 18.8, 1.8 Hz, 1H), 4.91 (dd, *J* = 10.5, 1.8 Hz, 1H), 5.32 (ddd, *J* = 18.8, 10.5, 10.5 Hz, 1H), 7.22-7.26 (m, 1H), 7.32 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.39-7.41 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.2, 25.1, 28.6, 30.2, 51.2, 51.3, 53.6, 118.4, 126.6, 127.0, 128.2, 136.6, 141.4, 181.5; IR (neat): 2939, 2095, 1697, 1269, 920, 700 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 34.0 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}N_3O_2$ $[M+Na]^+$ 310.1526. Found 310.1541; $[\alpha]_D^{20}$ = 67.5 (*c* = 1.1, $CHCl_3$).

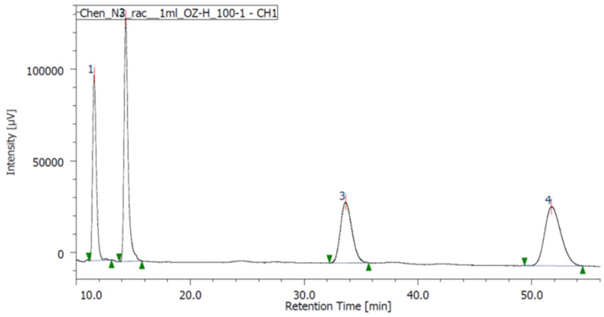
(2*R*,3*R*)-7-azido-2-methyl-2-phenyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2w**)



Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.01-1.19 (m, 3H), 1.32-1.54 (m, 6H), 2.90-2.96 (m, 1H), 3.13 (t, *J* = 7.1 Hz, 2H), 5.14-5.19 (m, 2H), 5.57 (ddd, *J* = 19.7, 10.5, 9.2 Hz, 1H), 7.25-7.29 (m, 1H), 7.32-7.36 (m, 2H), 7.44-7.47 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.2, 24.8, 28.0, 28.4, 50.3, 51.2, 53.8, 119.0, 127.1, 127.2, 128.2, 137.6, 140.5, 181.3; IR (neat): 2941, 2095, 1698, 1272, 924, 699 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 14.4 min; HRMS (ESI): m/z calcd for $C_{16}H_{21}N_3O_2$ $[M+Na]^+$ 310.1526. Found 310.1534; $[\alpha]_D^{20}$ = 43.1 (*c* = 1.3, $CHCl_3$).

<u>HPLC chart</u>

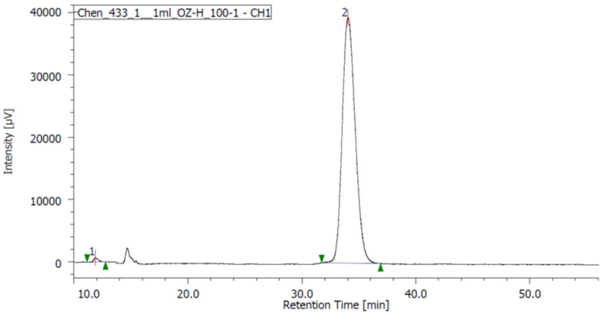
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_N3_rac__1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	11.567	2273641	101081	20.561	33.775	N/A	6521	4.766	1.345	
2	Unknown	1	14.333	3250967	132800	29.399	44.374	N/A	9408	15.833	1.534	
3	Unknown	1	33.633	2327054	33178	21.044	11.086	N/A	5269	8.083	1.193	
4	Unknown	1	51.725	3206391	32218	28.996	10.765	N/A	6165	N/A	1.235	

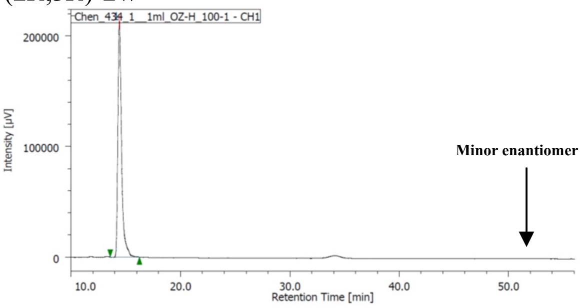
(2*S*,3*R*)-**2w**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_433_1_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	11.892	19050	673	0.607	1.679	N/A	3985	15.580	1.174	
2	Unknown	1	34.042	3121035	39383	99.393	98.321	N/A	4215	N/A	1.192	

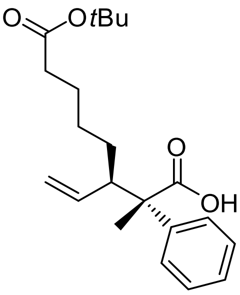
(2*R*,3*R*)-**2w**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_434_1_1ml_OZ-H_100-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.417	5024657	213109	100.000	100.000	N/A	10252	N/A	1.567	

(2*S*,3*R*)-8-(*tert*-butoxy)-2-methyl-8-oxo-2-phenyl-3-vinyloctanoic acid ((2*S*,3*R*)-**2x**)



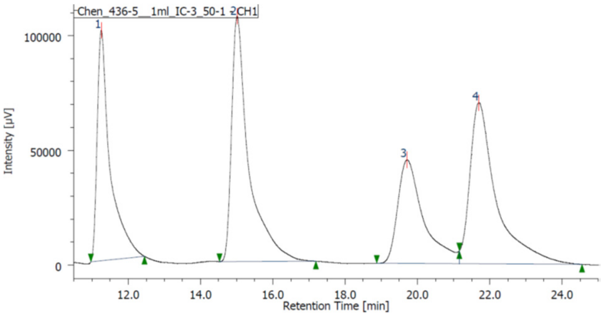
Yield: 97%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.24-1.41 (m, 5H), 1.44 (s, 9H), 1.54 (s, 3H), 1.59-1.66 (m, 1H), 2.17-2.21(m, 2H), 2.84-2.88 (m, 1H), 4.75 (dd, *J* = 17.6, 1.6 Hz, 1H), 4.88 (dd, *J* = 9.8, 1.6 Hz, 1H), 5.31 (ddd, *J* = 17.6, 9.8, 8.0 Hz, 1H), 7.21-7.24 (m, 1H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.39-7.41 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 17.2, 25.0, 27.5, 28.1, 30.4, 35.5, 51.2, 53.7, 79.9, 118.1, 126.6, 126.9, 128.2, 136.8, 141.6, 173.3, 181.3; IR (neat): 2979, 2935, 1731, 1257, 1154, 918, 700 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 72.9 min; HRMS (ESI): m/z calcd for $C_{21}H_{30}O_4$ $[M+Na]^+$ 369.2036. Found 369.2024; $[\alpha]_D^{20}$ = 51.6 (*c* = 1.4, $CHCl_3$).

(2*R*,3*R*)-8-(*tert*-butoxy)-2-methyl-8-oxo-2-phenyl-3-vinyloctanoic acid ((2*R*,3*R*)-**2x**)

Yield: 88%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.06-1.13 (m, 3H), 1.25-1.38 (m, 3H), 1.40 (s, 9H), 1.53 (s, 3H), 2.06-2.09 (m, 2H), 2.91-2.95 (m, 1H), 5.12-5.17 (m, 2H), 5.57 (ddd, J = 16.0, 8.6, 8.6 Hz, 1H), 7.24-7.27 (m, 1H), 7.33 (t, J = 7.7 Hz, 2H), 7.45 (d, J = 7.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 17.1, 24.8, 27.2, 28.0, 28.2, 35.4, 50.2, 53.8, 79.9, 118.7, 127.0, 127.2, 128.2, 137.9, 140.7, 173.1, 181.1; IR (neat): 2979, 2936, 1731, 1257, 1154, 921, 699 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 23.5 min; HRMS (ESI): m/z calcd for $C_{21}H_{30}O_4$ $[M+Na]^+$ 369.2036. Found 369.2024; $[\alpha]_D^{20}$ = 37.0 (c = 1.5, $CHCl_3$).

HPLC chart

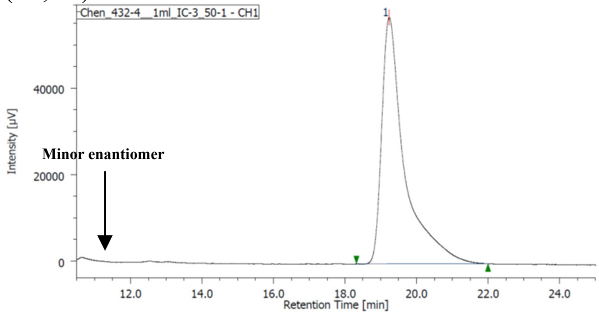
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_436-5_1ml_IC-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	11.258	2493042	100441	21.049	31.111	N/A	7517	6.208	2.395	
2	Unknown	1	15.008	3535501	106986	29.850	33.139	N/A	7528	5.155	2.421	
3	Unknown	1	19.708	2242475	45178	18.933	13.994	N/A	4811	1.782	N/A	
4	Unknown	1	21.700	3573031	70240	30.167	21.757	N/A	6176	N/A	N/A	

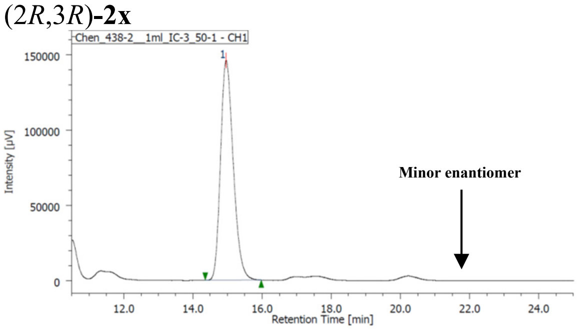
(2*S*,3*R*)-**2x**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_432-4_1ml_IC-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	19.233	2520190	56983	100.000	100.000	N/A	6745	N/A	2.403	

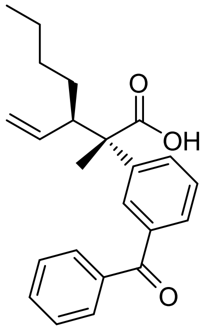
(2*R*,3*R*)-**2x**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_438-2_1ml_IC-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

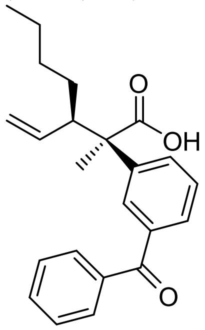
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.958	3728213	145816	100.000	100.000	N/A	8030	N/A	1.260	

(2*S*,3*R*)-2-(3-benzoylphenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2y**)



Yield: 81%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.86 (t, *J* = 6.6 Hz, 3H), 1.20-1.45 (m, 6H), 1.58 (s, 3H), 2.87-2.92 (m, 1H), 4.75 (dd, *J* = 17.4, 1.8 Hz, 1H), 4.91 (dd, *J* = 10.3, 1.8 Hz, 1H), 5.33 (ddd, *J* = 17.4, 10.3, 10.3 Hz, 1H), 7.41-7.46 (m, 3H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.67 (t, *J* = 8.2 Hz, 2H), 7.77 (d, *J* = 8.2 Hz, 2H), 7.85 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 17.0, 22.6, 30.2, 30.5, 51.3, 53.8, 118.5, 128.1, 128.2, 128.5, 128.8, 130.1, 131.0, 132.5, 136.7, 137.4, 137.4, 142.0, 181.1, 196.7; IR (neat): 2930, 1698, 1661, 1448, 1279, 1106, 920, 714 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 37.2 min; HRMS (ESI): m/z calcd for $C_{23}H_{26}O_3$ $[M+Na]^+$ 373.1774. Found 373.1788; $[\alpha]_D^{20}$ = 65.1 (*c* = 1.6, $CHCl_3$).

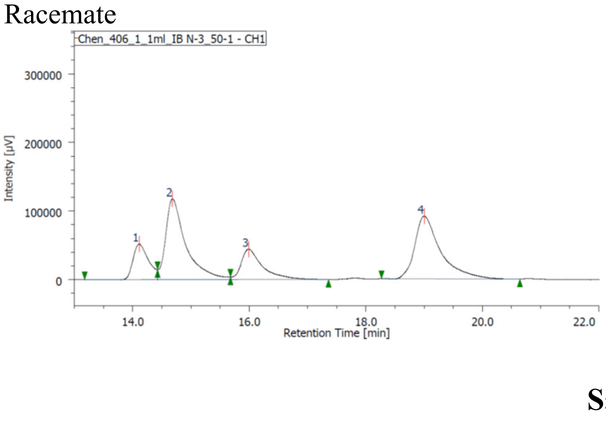
(2*R*,3*R*)-2-(3-benzoylphenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2y**)



Yield: 75%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.9 Hz, 3H), 1.05-1.28 (m, 6H), 1.57 (s, 3H), 2.90-2.94 (m, 1H), 5.13-5.16 (m, 2H), 5.53-5.60 (m, 1H), 7.42-7.48 (m, 3H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.69-7.72 (m, 2H), 7.78-7.80 (m, 2H), 7.91 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 17.4, 22.4, 28.4, 29.9, 50.5, 54.1, 118.9, 128.1, 128.2, 128.9, 129.1, 130.1, 131.5, 132.5, 137.4, 137.4, 137.7, 141.2, 181.1, 196.6; IR (neat): 2931, 1700, 1661, 1448, 1278, 1133, 922, 710 cm^{-1}; HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 55.9 min; HRMS (ESI): m/z calcd for $C_{23}H_{26}O_3$ $[M+Na]^+$ 373.1774. Found 373.1788; $[\alpha]_D^{20}$ = 47.7 (*c* = 1.7, $CHCl_3$).

HPLC chart

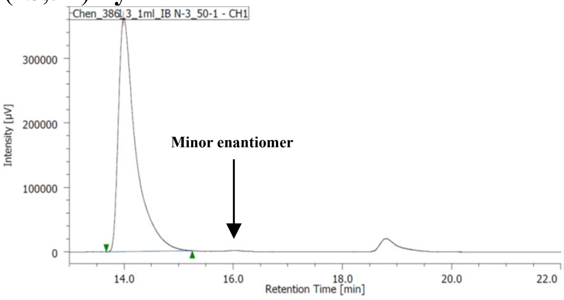
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_406_1_1ml_IB N-3_50-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.108	958222	51910	12.377	16.999	N/A	12971	1.112	N/A	
2	Unknown	1	14.683	2835251	117807	36.621	38.577	N/A	11778	2.342	N/A	
3	Unknown	1	15.992	1136386	44323	14.678	14.514	N/A	12199	4.694	N/A	
4	Unknown	1	19.000	2812240	91339	36.324	29.910	N/A	11587	N/A	1.693	

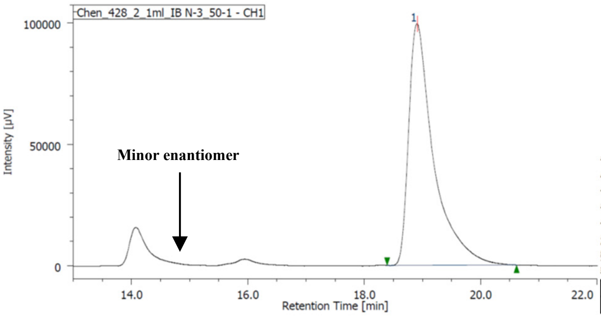
(2*S*,3*R*)-**2y**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_386_3_1ml_IB N-3_50-1-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　(マニュアル)
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.992	7986513	361646	100.000	100.000	N/A	12157	N/A	2.345	

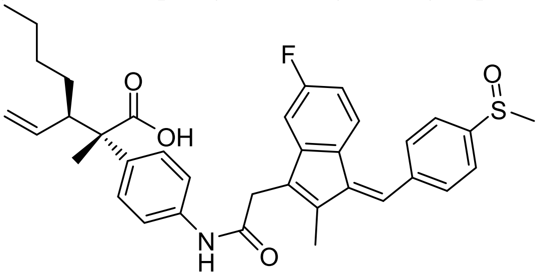
(2*R*,3*R*)-**2y**



チャンネル情報＋ピーク情報
クロマトグラム名　Chen_428_2_1ml_IB N-3_50-1-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　(マニュアル)
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.908	2969663	99508	100.000	100.000	N/A	12167	N/A	2.118	

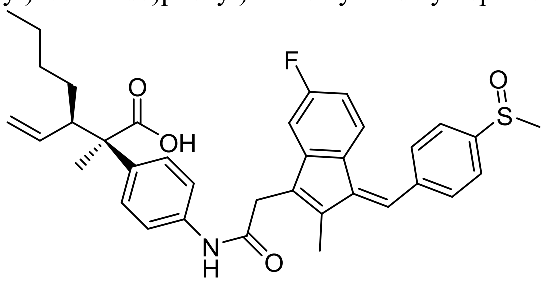
(2*S*,3*R*)-2-(4-(2-(5-fluoro-2-methyl-1-((*Z*)-4-(methylsulfinyl)benzylidene)-1*H*-inden-3-yl)acetamido)phenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2z**)



$[Ir(cod)Cl]_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L18** (0.030 mmol, 0.10 equiv), $(AcO)_4B_2O$ (16.4 mg, 0.060 mmol, 0.2 equiv) and (*R*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***R***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.

Yield: 74%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85 (t, *J* = 6.6 Hz, 3H), 1.18-1.45 (m, 6H), 1.49 (s, 3H), 2.23 (s, 3H), 2.79-2.82 (m, 4H), 3.65 (s, 2H), 4.71 (dd, *J* = 18.5, 1.8 Hz, 1H), 4.84 (dd, *J* = 9.6, 1.8 Hz, 1H), 5.27 (ddd, *J* = 18.5, 9.6, 9.6 Hz, 1H), 6.55-6.60 (m, 1H), 6.90 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.16-7.19 (m, 2H), 7.29 (d, *J* = 9.2 Hz, 2H), 7.38 (d, *J* = 8.7 Hz, 2H), 7.59 (s, 1H), 7.66 (d, *J* = 8.2 Hz, 2H), 7.73 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 10.6, 14.0, 16.8, 22.6, 30.3, 30.5, 34.5, 43.6, 51.1, 53.3, 106.0 (d, *J* = 23.9 Hz), 111.3 (d, *J* = 22.0 Hz), 118.0, 119.8, 123.9 (d, *J* = 11.5 Hz), 127.3, 128.9, 129.5, 129.5, 130.3, 132.2, 136.1, 137.0, 138.3, 139.0, 139.5, 141.4, 145.1, 146.3 (d, *J* = 7.7 Hz), 163.4 (d, *J* = 246.1 Hz), 167.7, 180.0; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -112.01; IR (neat): 2930, 2360, 1699, 1603, 1540, 1088, 756 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl amide (2*S*,3*R*)-**2z**. HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 4/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 22.5 min. HRMS (ESI): m/z calcd for $C_{36}H_{38}FNO_4S$ $[M+Na]^+$ 622.2398. Found 622.2375; $[\alpha]_D^{20}$ = 20.8 (*c* = 2.05, $CHCl_3$).

(2*R*,3*R*)-2-(4-(2-(5-fluoro-2-methyl-1-((*Z*)-4-(methylsulfinyl)benzylidene)-1*H*-inden-3-yl)acetamido)phenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2z**)

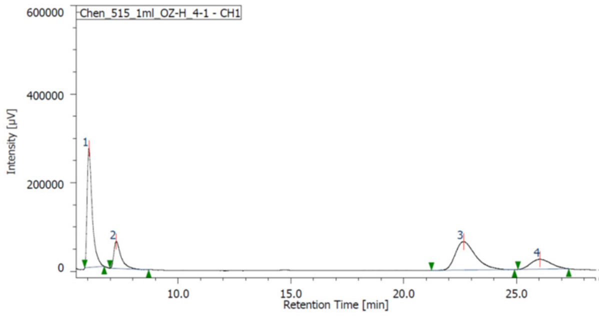


$[Ir(cod)Cl]_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L17** (0.030 mmol, 0.10 equiv), $(AcO)_4B_2O$ (16.4 mg, 0.060 mmol, 0.2 equiv) and (*S*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***S***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.

Yield: 45%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.76 (t, *J* = 6.9 Hz, 3H), 1.01-1.21 (m, 6H), 1.49 (s, 3H), 2.26 (s, 3H), 2.78-2.83 (m, 4H), 3.69 (s, 2H), 5.11-5.15 (m, 2H), 5.55 (ddd, *J* = 16.0, 6.9, 6.9 Hz, 1H), 6.57-6.62 (m, 1H), 6.92 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.18-7.22 (m, 2H), 7.35-7.41 (m, 4H), 7.50 (s, 1H), 7.68 (d, *J* = 8.6 Hz, 2H), 7.74 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 10.6, 14.2, 17.0, 22.4, 28.2, 30.0, 34.5, 43.5, 50.2, 53.5, 106.0 (d, *J* = 23.9 Hz), 111.3 (d, *J* = 22.0 Hz), 118.5, 120.0, 124.0, 127.8, 128.9, 129.5, 129.5, 130.3, 132.1, 136.1, 137.5, 138.1, 139.1, 139.5, 141.4, 144.9, 146.2 (d, *J* = 7.7 Hz), 163.4 (d, *J* = 246.1 Hz), 167.9, 179.9; ^{19}F NMR (369 MHz, $CDCl_3$): δ = -111.88; IR (neat): 2930, 2359, 1716, 1652, 1541, 1087, 757 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl amide (2*R*,3*R*)-**2z**. HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 4/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 7.2 min. HRMS (ESI): m/z calcd for $C_{36}H_{38}FNO_4S$ $[M+Na]^+$ 622.2398. Found 622.2375; $[\alpha]_D^{20}$ = 25.7 (*c* = 0.46, $CHCl_3$).

<u>HPLC chart</u>

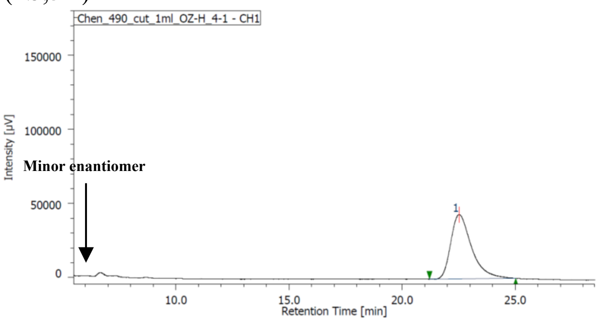
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_515_1ml_OZ-H_4-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	6.058	4248567	268802	38.544	64.550	N/A	3977	2.673	1.913	
2	Unknown	1	7.267	1345523	61215	12.207	14.700	N/A	3098	14.557	2.061	
3	Unknown	1	22.650	4030033	64299	36.561	15.441	N/A	3219	2.050	1.461	
4	Unknown	1	26.042	1398559	22109	12.688	5.309	N/A	3663	N/A	1.158	

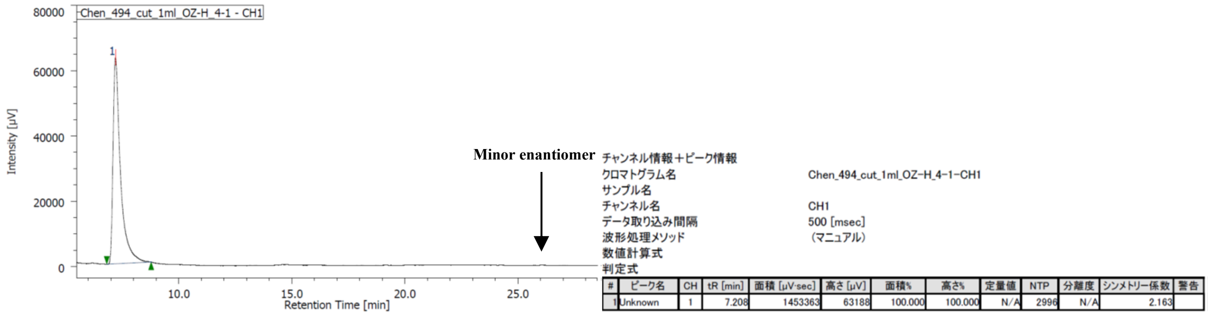
(2*S*,3*R*)-**2z**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_490_cut_1ml_OZ-H_4-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

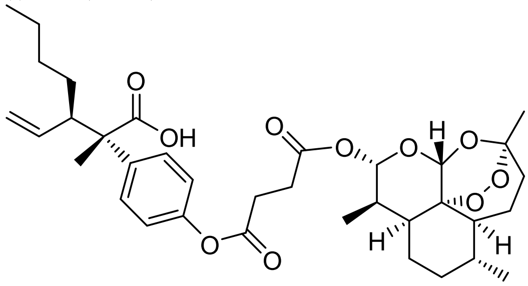
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	22.517	2777933	43420	100.000	100.000	N/A	3157	N/A	1.523	

(2*R*,3*R*)-**2z**



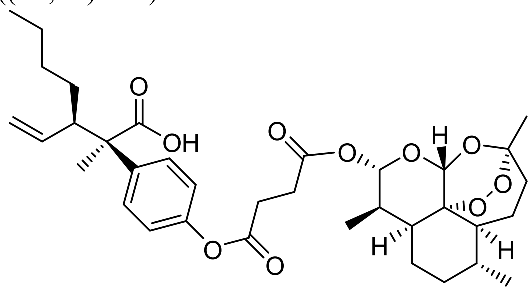
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	7.208	1453363	63188	100.000	100.000	N/A	2996	N/A	2.163	

(2*S*,3*R*)-2-methyl-2-(4-((4-oxo-4-(((3*R*,5a*S*,6*R*,8a*S*,9*R*,10*S*,12*R*,12a*R*)-3,6,9-trimethyldecahydro-12*H*-3,12-epoxy[1,2]dioxepino[4,3-*i*]isochromen-10-yl)oxy)butanoyl)oxy)phenyl)-3-vinylheptanoic acid ((2*S*,3*R*)-**2aa**)



[Ir(cod)Cl]$_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L18** (0.030 mmol, 0.10 equiv), (AcO)$_4$B$_2$O (16.4 mg, 0.060 mmol, 0.2 equiv) and (*R*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***R***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.
Yield: 77%; ^{1}H NMR (400 MHz, CDCl$_3$): δ = 0.83-0.88 (m, 6H), 0.95-1.05 (m, 4H), 1.15-1.52 (m, 16H), 1.59-1.65 (m, 1H), 1.69-1.80 (m, 2H), 1.87-1.92 (m, 1H), 2.00-2.06 (m, 1H), 2.37 (dt, *J* = 4.1, 14.2 Hz, 1H), 2.53-2.62 (m, 1H), 2.77-2.98 (m, 5H), 4.74 (dd, *J* = 18.3, 1.8 Hz, 1H), 4.88 (dd, *J* = 9.6, 1.8 Hz, 1H), 5.29 (ddd, *J* = 18.3, 9.6, 9.6 Hz, 1H), 5.45 (s, 1H), 5.81 (d, *J* = 10.1 Hz, 1H), 7.04 (d, *J* = 9.2 Hz, 2H), 7.40 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 12.1, 14.0, 17.0, 20.2, 22.0, 22.5, 24.6, 25.9, 29.1, 29.2, 30.3, 30.4, 31.8, 34.0, 36.2, 37.2, 45.2, 51.3, 51.5, 53.3, 80.1, 91.5, 92.3, 104.5, 118.2, 121.0, 127.9, 136.8, 139.1, 149.4, 170.6, 170.9, 180.6; IR (neat): 2926, 2357, 1748, 1507, 1095, 1013, 757 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*S*,3*R*)-**2aa**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 23.9 min. HRMS (ESI): m/z calcd for $C_{35}H_{48}O_{10}$ [M+Na]$^+$ 651.3140. Found 651.3131; $[\alpha]_D^{20}$ = 29.2 (*c* = 0.76, CHCl$_3$).

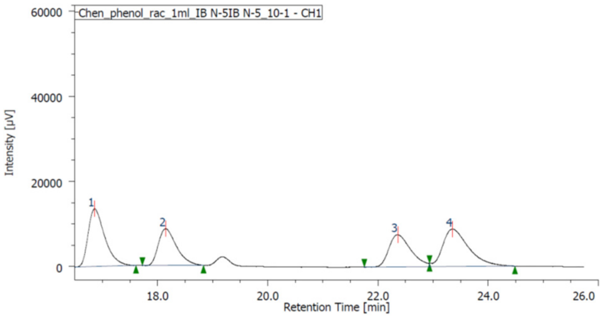
(2*R*,3*R*)-2-methyl-2-(4-((4-oxo-4-(((3*R*,5a*S*,6*R*,8a*S*,9*R*,10*S*,12*R*,12a*R*)-3,6,9-trimethyldecahydro-12*H*-3,12-epoxy[1,2]dioxepino[4,3-*i*]isochromen-10-yl)oxy)butanoyl)oxy)phenyl)-3-vinylheptanoic acid ((2*R*,3*R*)-**2aa**)



[Ir(cod)Cl]$_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L17** (0.030 mmol, 0.10 equiv), $(AcO)_4B_2O$ (16.4 mg, 0.060 mmol, 0.2 equiv) and (*S*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***S***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.
Yield: 73%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.77 (t, *J* = 7.2 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H), 0.92-1.11 (m, 8H), 1.18-1.51 (m, 12H), 1.62 (dt, *J* = 13.7, 4.6 Hz, 1H), 1.69-1.80 (m, 2H), 1.86-1.92 (m, 1H), 2.01-2.05 (m, 1H), 2.38 (dt, *J* = 3.4, 13.7 Hz, 1H), 2.54-2.61 (m, 1H), 2.77-2.98 (m, 5H), 5.12-5.16 (m, 2H), 5.45 (s, 1H), 5.55 (ddd, *J* = 16.6, 8.3, 8.3 Hz, 1H), 5.82 (d, *J* = 9.7 Hz, 1H), 7.07 (d, *J* = 9.2 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 12.1, 14.0, 17.3, 20.2, 22.0, 22.4, 24.6, 25.9, 28.2, 29.1, 29.2, 30.0, 31.8, 34.1, 36.2, 37.2, 45.2, 50.5, 51.5, 53.6, 80.1, 91.5, 92.3, 104.5, 118.6, 121.0, 128.4, 137.9, 138.4, 149.5, 170.6, 171.0, 180.4; IR (neat): 2928, 2359, 1749, 1507, 1135, 1016, 757 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*R*,3*R*)-**2aa**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.3 min. HRMS (ESI): m/z calcd for $C_{35}H_{48}O_{10}$ $[M+Na]^+$ 651.3140. Found 651.3165; $[\alpha]_D^{20}$ = 21.1 (*c* = 0.97, $CHCl_3$).

HPLC chart

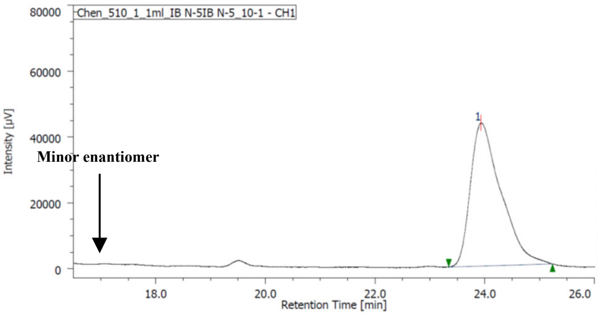
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_phenol_rac_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.858	283172	13532	29.012	35.184	N/A	15669	2.284	1.536	
2	Unknown	1	18.150	196944	8597	20.177	22.354	N/A	14879	6.397	1.459	
3	Unknown	1	22.358	207407	7543	21.249	19.612	N/A	15254	1.281	N/A	
4	Unknown	1	23.350	288545	8788	29.562	22.849	N/A	12709	N/A	N/A	

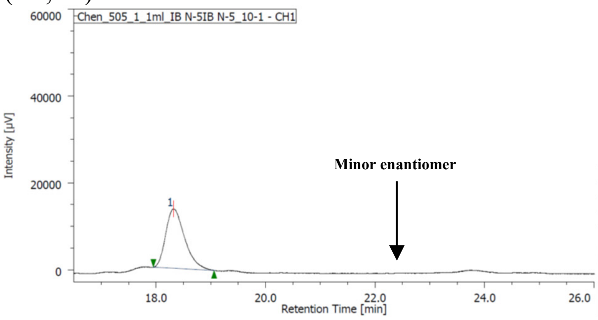
(2*S*,3*R*)-**2aa**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_510_1_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	23.933	1683306	43443	100.000	100.000	N/A	9137	N/A	1.663	

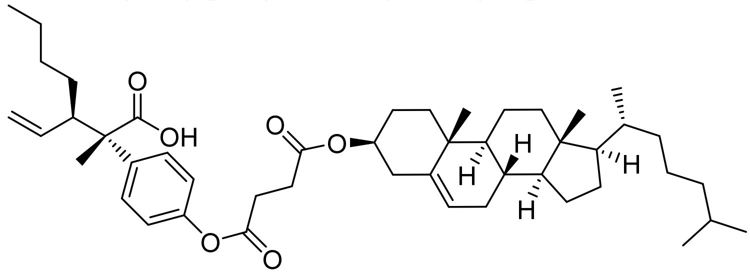
(2*R*,3*R*)-**2aa**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_505_1_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

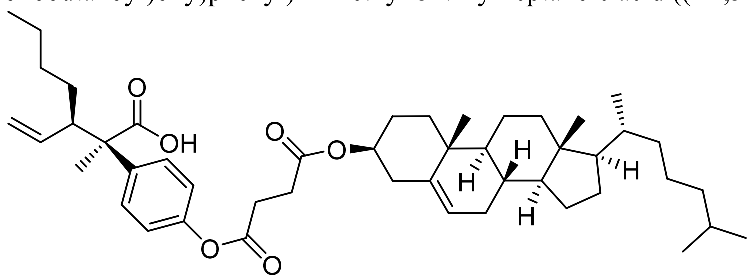
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.317	328161	13659	100.000	100.000	N/A	13754	N/A	1.400	

(2*S*,3*R*)-2-(4-((4-(((3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)oxy)-4-oxobutanoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2ab**)



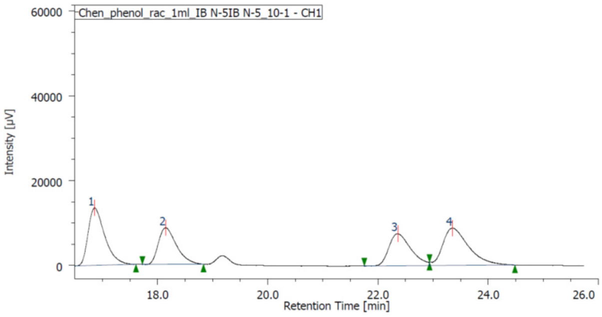
Yield: 67%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.68 (s, 3H), 0.86-1.60 (m, 45H), 1.80-1.87 (m, 3H), 1.95-2.02 (m, 2H), 2.33 (d, *J* = 6.9 Hz, 2H), 2.70 (t, *J* = 6.6 Hz, 2H), 2.84-2.87 (m, 3H), 4.62-4.68 (m, 1H), 4.75 (m, 1H), 4.89 (dd, *J* = 8.6, 1.7 Hz, 1H), 5.29 (ddd, *J* = 16.6, 8.6, 8.6 Hz, 1H), 5.37 (d, *J* = 4.6 Hz, 1H), 7.04 (d, *J* = 8.9 Hz, 2H), 7.41 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 11.8, 14.0, 17.0, 18.7, 19.3, 21.0, 22.5, 22.5, 22.8, 23.8, 24.3, 27.7, 28.0, 28.2, 29.4, 30.3, 30.4, 31.8, 31.9, 35.8, 36.2, 36.5, 36.9, 38.0, 39.5, 39.7, 42.3, 50.0, 51.3, 53.4, 56.1, 56.7, 74.5, 118.2, 120.9, 122.7, 127.9, 136.8, 139.1, 139.5, 149.5, 170.9, 171.5, 181.0; IR (neat): 2935, 2868, 1734, 1507, 1209, 1173, 1138, 1017, 738 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*S*,3*R*)-**2z**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 23.4 min. HRMS (ESI): m/z calcd for $C_{47}H_{70}O_6$ $[M+Na]^+$ 753.5065. Found 753.5053; $[\alpha]_D^{20}$ = 2.2 (*c* = 0.95, $CHCl_3$).

(2*R*,3*R*)-2-(4-((4-(((3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)oxy)-4-oxobutanoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2ab**)



Yield: 76%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.68 (s, 3H), 0.77 (t, *J* = 6.9 Hz, 3H), 0.86 (d, *J* = 1.8 Hz, 3H), 0.87 (d, *J* = 1.4 Hz, 3H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.94-1.68 (m, 33H), 1.79-1.87 (m, 3H), 1.93-2.04 (m, 2H), 2.33 (d, *J* = 7.3 Hz, 2H), 2.71 (t, *J* = 6.6 Hz, 2H), 2.85-2.92 (m, 3H), 4.62-4.69 (m, 1H), 5.12-5.16 (m, 2H), 5.37 (d, *J* = 4.6 Hz, 1H), 5.55 (ddd, *J* = 19.7, 9.2, 9.2 Hz, 1H), 7.06 (d, *J* = 8.9 Hz, 2H), 7.46 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.8, 14.0, 17.2, 18.7, 19.3, 21.0, 22.4, 22.5, 22.8, 23.8, 24.3, 27.7, 28.0, 28.2, 29.4, 29.4, 30.0, 31.8, 31.9, 35.8, 36.1, 36.5, 36.9, 38.0, 39.5, 39.7, 42.3, 50.0, 50.5, 53.6, 56.1, 56.6, 74.5, 118.7, 121.0, 122.7, 128.4, 137.9, 138.3, 139.5, 149.5, 170.9, 171.5, 181.0; IR (neat): 2951, 2868, 1732, 1508, 1267, 1210, 1138, 1017, 739 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*R*,3*R*)-**2ab**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.3 min. HRMS (ESI): m/z calcd for $C_{47}H_{70}O_6$ $[M+Na]^+$ 753.5065. Found 753.5070; $[\alpha]_D^{20}$ = 0.71 (*c* = 1.8, $CHCl_3$).

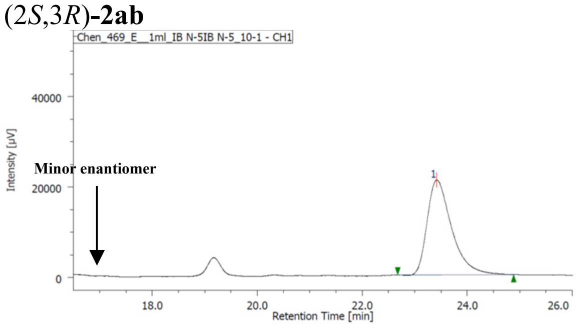
<u>HPLC chart</u>
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_phenol_rac_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.858	283172	13532	29.012	35.184	N/A	15669	2.284	1.536	
2	Unknown	1	18.150	196944	8597	20.177	22.354	N/A	14879	6.397	1.459	
3	Unknown	1	22.358	207407	7543	21.249	19.612	N/A	15254	1.281	N/A	
4	Unknown	1	23.350	288545	8788	29.562	22.849	N/A	12709	N/A	N/A	

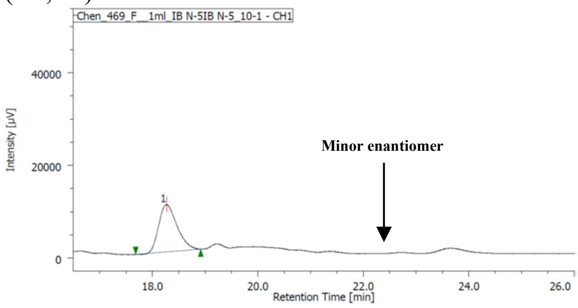
(2*S*,3*R*)-**2ab**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_469_E__1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	23.417	679218	20970	100.000	100.000	N/A	13148	N/A	1.541	

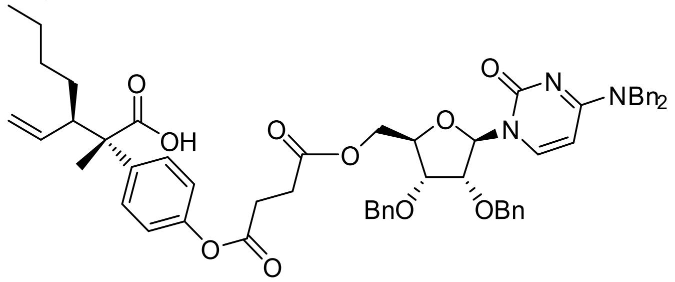
(2*R*,3*R*)-**2ab**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_469_F__1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.267	247754	10226	100.000	100.000	N/A	13111	N/A	1.323	

(2*S*,3*R*)-2-(4-((4-(((2*R*,3*R*,4*R*,5*R*)-3,4-bis(benzyloxy)-5-(4-(dibenzylamino)-2-oxopyrimidin-1(2*H*)-yl)tetrahydrofuran-2-yl)methoxy)-4-oxobutanoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2ac**)

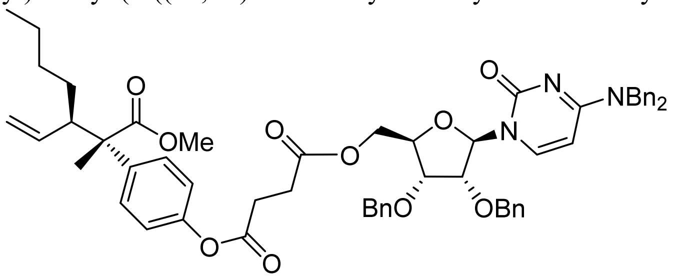


$[Ir(cod)Cl]_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L18** (0.030 mmol, 0.10 equiv), $(AcO)_4B_2O$ (16.4 mg, 0.060 mmol, 0.2 equiv) and (*R*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***R***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.
NMR yield: 53%; Product was isolated after methyl esterification.

Procedure of methylation

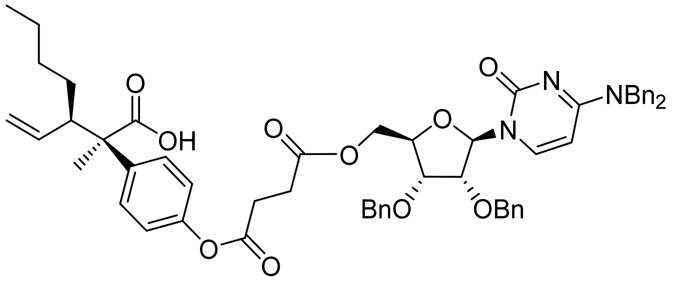
A solution of crude (2*S*,3*R*)-**2ac** in MeOH (1.0 mL) and toluene (1.0 mL) was treated with $TMSCHN_2$ (2.0 M in Et_2O, 0.45 mL, 0.9 mmol), which resulted in a solution color change to yellow. After stirring for 30 min at room temperature, a few drops of acetic acid were added until the color of the solution disappeared. The reaction mixture was concentrated under reduced pressure and the remaining residue was purified by column chromatography on silica gel (EtOAc/hexane = 1/1), affording the pure (2*S*,3*R*)-**2ae**.

((2*R*,3*R*,4*R*,5*R*)-3,4-bis(benzyloxy)-5-(4-(dibenzylamino)-2-oxopyrimidin-1(2*H*)-yl)tetrahydrofuran-2-yl)methyl (4-((2*S*,3*R*)-1-methoxy-2-methyl-1-oxo-3-vinylheptan-2-yl)phenyl) succinate ((2*S*,3*R*)-**2ae**)



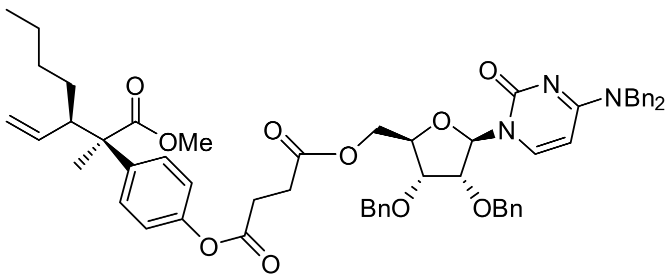
Yield: 49%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.83 (t, *J* = 6.6 Hz, 3H), 1.14-1.29 (m, 6H), 1.44 (s, 3H), 2.42-2.56 (m, 2H), 2.72-2.81 (m, 3H), 3.60 (s, 3H), 3.70-3.74 (m, 1H), 4.13 (d, *J* = 11.4 Hz, 1H), 4.25 (d, *J* = 4.6 Hz, 1H), 4.33-4.46 (m, 5H), 4.65 (dd, *J* = 18.8, 1.8 Hz, 1H), 4.80 (dd, *J* = 10.5, 1.8 Hz, 1H), 4.90-4.95 (m, 3H), 5.08-5.12 (m, 1H), 5.22 (ddd, *J* = 18.8, 10.5, 9.2 Hz, 1H), 5.74 (d, *J* = 8.2 Hz, 1H), 5.91 (s, 1H), 6.89-6.91 (m, 2H), 7.06-7.08 (m, 2H), 7.16-7.18 (m, 2H), 7.22-7.31 (m, 17H), 7.49 (d, *J* = 6.9 Hz, 2H), 7.65 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.0, 17.2, 22.5, 28.7, 29.0, 30.3, 30.5, 50.5, 50.7, 51.5, 51.6, 52.1, 53.5, 62.7, 71.5, 72.0, 73.9, 74.6, 79.2, 90.9, 91.5, 118.0, 120.7, 126.3, 127.5, 127.7, 127.8, 127.8, 127.9, 128.4, 128.5, 128.6, 128.8, 129.0, 135.7, 136.6, 137.0, 137.2, 137.8, 140.0, 140.9, 149.0, 154.8, 163.9, 170.7, 171.5, 175.6; IR (neat): 2929, 1746, 1652, 1520, 1362, 1139, 981, 699 cm^{-1}; Enantiomeric excess was determined after hydrolysis of methyl ester and phenyl ester of (2*S*,3*R*)-**2ae**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 23.5 min. HRMS (ESI): m/z calcd for $C_{58}H_{63}N_3O_{10}$ $[M+Na]^+$ 984.4406. Found 984.4391; $[\alpha]_D^{20}$ = 120.5 (*c* = 0.70, $CHCl_3$).

(2*R*,3*R*)-2-(4-((4-(((2*R*,3*R*,4*R*,5*R*)-3,4-bis(benzyloxy)-5-(4-(dibenzylamino)-2-oxopyrimidin-1(2*H*)-yl)tetrahydrofuran-2-yl)methoxy)-4-oxobutanoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2ac**)



$[Ir(cod)Cl]_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L17** (0.030 mmol, 0.10 equiv), $(AcO)_4B_2O$ (16.4 mg, 0.060 mmol, 0.2 equiv) and (*S*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***S***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.
NMR yield: 40%; Product was isolated after methyl esterification.

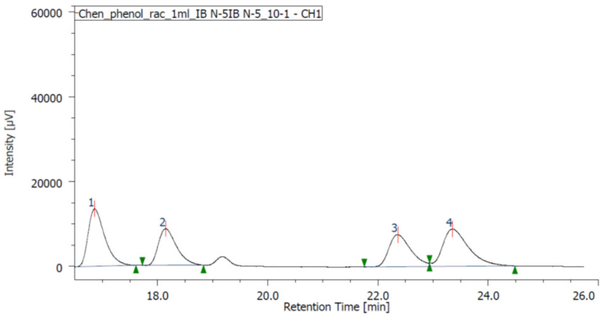
((2*R*,3*R*,4*R*,5*R*)-3,4-bis(benzyloxy)-5-(4-(dibenzylamino)-2-oxopyrimidin-1(2*H*)-yl)tetrahydrofuran-2-yl)methyl (4-((2*R*,3*R*)-1-methoxy-2-methyl-1-oxo-3-vinylheptan-2-yl)phenyl) succinate ((2*R*,3*R*)-**2ae**)



Yield: 34%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.76 (t, *J* = 6.6 Hz, 3H), 1.02-1.35 (m, 6H), 1.52 (s, 3H), 2.50-2.61 (m, 2H), 2.79 (t, *J* = 6.6 Hz, 2H), 2.88-2.91 (m, 1H), 3.56 (s, 3H), 3.75-3.78 (m, 1H), 4.19 (d, *J* = 12.0 Hz, 1H), 4.28 (d, *J* = 4.6 Hz, 1H), 4.39-4.49 (m, 5H), 4.94-5.02 (m, 3H), 5.08-5.12 (m, 3H), 5.55 (ddd, *J* = 16.6, 9.2, 9.2 Hz, 1H), 5.77 (d, *J* = 8.0 Hz, 1H), 5.95 (s, 1H), 6.98 (d, *J* = 8.6 Hz, 2H), 7.11 (br, 1H), 7.20-7.38 (m, 19H), 7.52 (d, *J* = 7.4 Hz, 3H), 7.66 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.9, 14.0, 17.5, 22.4, 25.3, 28.2, 28.7, 29.0, 29.9, 50.7, 51.9, 53.8, 62.9, 64.4, 65.6, 71.5, 72.0, 74.8, 79.1, 91.0, 91.4, 118.2, 120.8, 121.4, 126.3, 127.6, 127.8, 127.8, 127.9, 128.2, 128.3, 128.4, 128.5, 128.6, 128.8, 129.0, 137.2, 137.9, 138.4, 139.3, 140.7, 149.2, 155.3, 164.3, 170.7, 171.5, 175.4; IR (neat): 2924, 1735, 1653, 1496, 1453, 1094, 839, 698 cm^{-1}; Enantiomeric excess was determined after hydrolysis of methyl ester and phenyl ester of (2*R*,3*R*)-**2ae**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.4 min. HRMS (ESI): m/z calcd for $C_{58}H_{63}N_3O_{10}$ $[M+Na]^+$ 984.4406. Found 984.4391;

HPLC chart

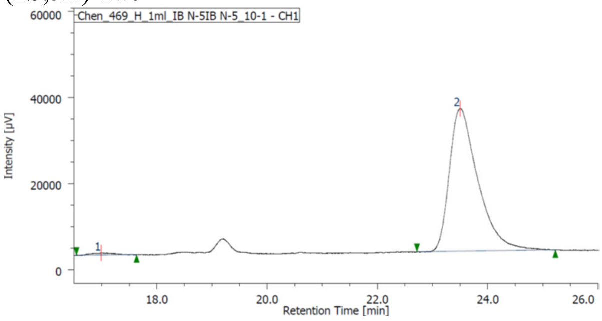
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_phenol_rac_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.858	283172	13532	29.012	35.184	N/A	15669	2.284	1.536	
2	Unknown	1	18.150	196944	8597	20.177	22.354	N/A	14879	6.397	1.459	
3	Unknown	1	22.358	207407	7543	21.249	19.612	N/A	15254	1.281	N/A	
4	Unknown	1	23.350	288545	8788	29.562	22.849	N/A	12709	N/A	N/A	

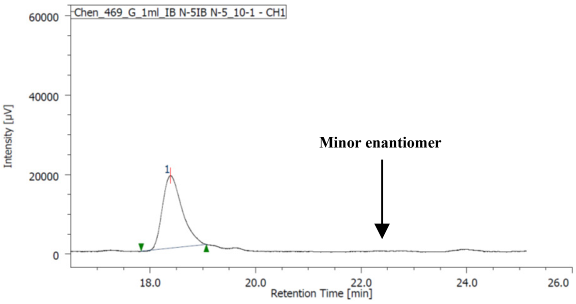
(2*S*,3*R*)-**2ac**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_469_H_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.992	13251	499	1.102	1.482	N/A	6877	7.568	1.340	
2	Unknown	1	23.500	1189231	33140	98.898	98.518	N/A	10788	N/A	1.569	

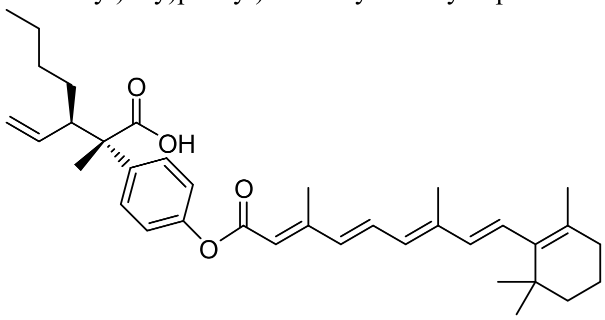
(2*R*,3*R*)-**2ac**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_469_G_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.383	471949	18264	100.000	100.000	N/A	11946	N/A	1.422	

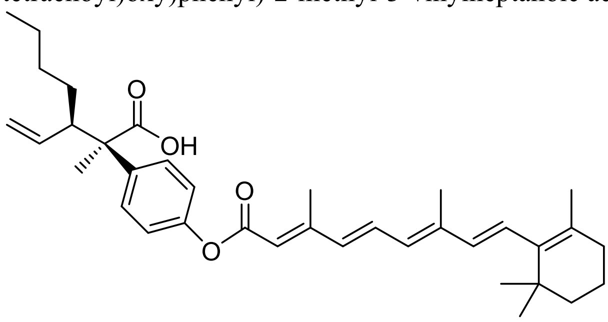
(2*S*,3*R*)-2-(4-(((2*E*,4*E*,6*E*,8*E*)-3,7-dimethyl-9-(2,6,6-trimethylcyclohex-1-en-1-yl)nona-2,4,6,8-tetraenoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*S*,3*R*)-**2ad**)



[Ir(cod)Cl]$_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L18** (0.030 mmol, 0.10 equiv), (AcO)$_4$B$_2$O (16.4 mg, 0.060 mmol, 0.2 equiv) and (*R*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***R***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.

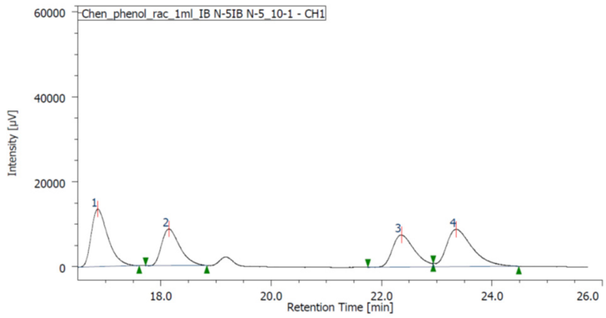
Yield: 72%; ^{1}H NMR (400 MHz, CDCl$_3$): δ = 0.88 (t, *J* = 6.9 Hz, 3H), 1.02-1.04 (m, 6H), 1.22-1.49 (m, 8H), 1.54 (s, 3H), 1.59-1.64 (m, 2H), 1.70-1.73 (m, 3H), 2.01-2.05 (m, 5H), 2.40 (s, 3H), 2.86-2.90 (m, 1H), 4.77 (dd, *J* = 18.8, 1.6 Hz, 1H), 4.90 (dd, *J* = 10.7, 1.6 Hz, 1H), 5.32 (ddd, *J* = 18.8, 10.7, 9.6 Hz, 1H), 5.97 (s, 1H), 6.11-6.23 (m, 2H), 6.26-6.39 (m, 2H), 7.05-7.11 (m, 3H), 7.43 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 12.9, 14.0, 14.1, 17.0, 19.2, 21.8, 22.6, 29.0, 30.3, 30.4, 33.1, 34.2, 39.6, 51.2, 53.4, 117.2, 118.1, 121.3, 127.8, 129.1, 129.4, 130.2, 131.9, 134.8, 136.9, 137.2, 137.6, 138.7, 140.3, 149.6, 155.5, 165.3, 181.0; IR (neat): 2930, 2359, 1717, 1507, 1213, 1124, 752 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*S*,3*R*)-**2ad**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 24.4 min. HRMS (ESI): m/z calcd for $C_{36}H_{48}O_4$ [M+Na]$^+$ 567.3445. Found 567.3452; $[\alpha]_D^{20}$ = 26.0 (*c* = 1.02, CHCl$_3$).

(2*R*,3*R*)-2-(4-(((2*E*,4*E*,6*E*,8*E*)-3,7-dimethyl-9-(2,6,6-trimethylcyclohex-1-en-1-yl)nona-2,4,6,8-tetraenoyl)oxy)phenyl)-2-methyl-3-vinylheptanoic acid ((2*R*,3*R*)-**2ad**)



[Ir(cod)Cl]$_2$ (10.0 mg, 0.015 mmol, 0.05 equiv), ligand **L17** (0.030 mmol, 0.10 equiv), $(AcO)_4B_2O$ (16.4 mg, 0.060 mmol, 0.2 equiv) and (*S*)-((2,3,5,6-tetrafluoro-4-methoxyphenyl)sulfonyl)-valine (***S***, 43.2 mg, 0.120 mmol, 0.4 equiv) were used.
Yield: 67%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.79 (t, *J* = 6.9 Hz, 3H), 1.03-1.27 (m, 12H), 1.47-1.49 (m, 2H), 1.53 (s, 3H), 1.60-1.65 (m, 2H), 1.73 (s, 3H), 2.01-2.05 (m, 5H), 2.41 (s, 3H), 2.90-2.94 (m, 1H), 5.13-5.17 (m, 2H), 5.57 (ddd, *J* = 19.5, 8.9, 8.9 Hz, 1H), 5.99 (s, 1H), 6.15-6.20 (m, 2H), 6.30-6.39 (m, 2H), 7.06-7.11 (m, 3H), 7.48 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 12.9, 14.0, 14.1, 17.3, 19.2, 21.7, 22.4, 28.2, 28.9, 30.0, 33.1, 34.2, 39.6, 50.4, 53.6, 117.2, 118.6, 121.3, 128.3, 129.1, 129.4, 130.2, 132.0, 134.7, 137.2, 137.6, 137.9, 138.0, 140.4, 149.7, 155.6, 165.4, 181.1; IR (neat): 2930, 2359, 1717, 1507, 1218, 1122, 757 cm^{-1}; Enantiomeric excess was determined after hydrolysis of phenyl ester (2*R*,3*R*)-**2ad**. HPLC (chiral column: CHIRALPAK IB N-5-IB N-5; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): t_R = 18.6 min. HRMS (ESI): m/z calcd for $C_{36}H_{48}O_4$ $[M+Na]^+$ 567.3445. Found 567.3454 $[\alpha]_D^{20}$ = 24.6 (*c* = 1.56, $CHCl_3$).

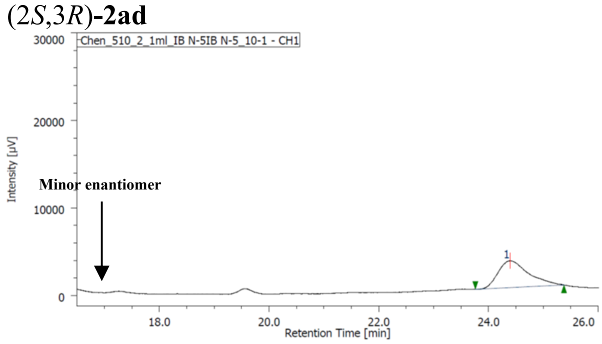
<u>HPLC chart</u>
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_phenol_rac_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	16.858	283172	13532	29.012	35.184	N/A	15669	2.284	1.536	
2	Unknown	1	18.150	196944	8597	20.177	22.354	N/A	14879	6.397	1.459	
3	Unknown	1	22.358	207407	7543	21.249	19.612	N/A	15254	1.281	N/A	
4	Unknown	1	23.350	288545	8788	29.562	22.849	N/A	12709	N/A	N/A	

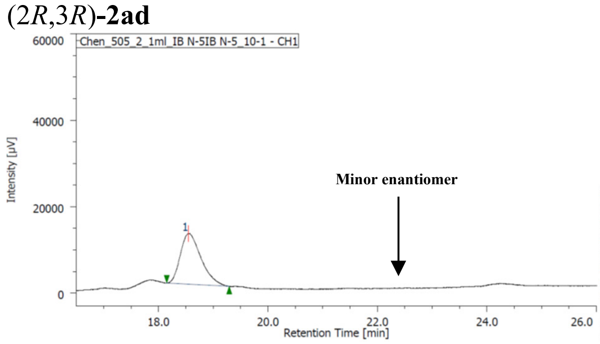
(2*S*,3*R*)-**2ad**



チャンネル情報＋ピーク情報
クロマトグラム名 Chen_510_2_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	24.392	119073	3067	100.000	100.000	N/A	9772	N/A	1.489	

(2*R*,3*R*)-**2ad**



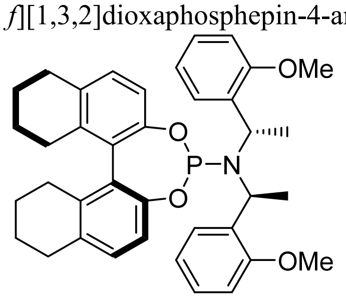
チャンネル情報＋ピーク情報
クロマトグラム名 Chen_505_2_1ml_IB N-5IB N-5_10-1-CH1
サンプル名
チャンネル名 CH1
データ取り込み間隔 500 [msec]
波形処理メソッド （マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	18.550	307635	11726	100.000	100.000	N/A	11659	N/A	1.394	

4. Procedure for Phosphoramidite Ligand Synthesis

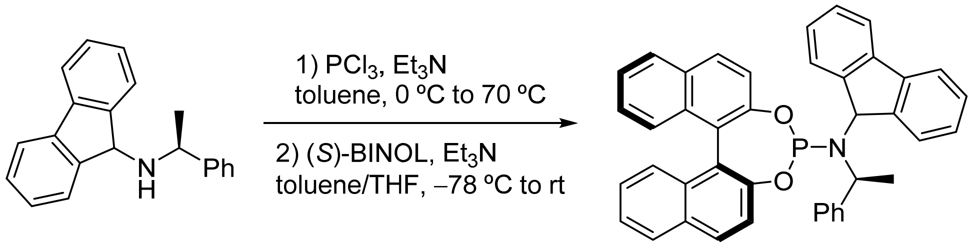
L1 and **L3** were purchased from Aldrich. **L2**, **L4**, **L13** and **L15** were prepared according to the reported methods.[2-5]

(11b*S*)-*N*,*N*-bis((*S*)-1-(2-methoxyphenyl)ethyl)-8,9,10,11,12,13,14,15-octahydrodinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L4**)



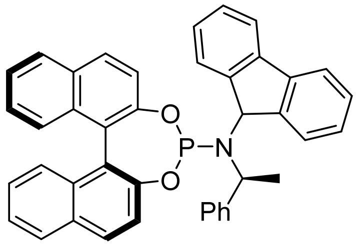
Yield: 30%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.40 (d, *J* = 7.3 Hz, 6H), 1.72-1.79 (m, 8H), 2.21-2.27 (m, 2H), 2.59-2.82 (m, 6H), 3.62 (s, 6H), 4.78-4.86 (m, 2H), 6.48 (d, *J* = 7.8 Hz, 2H), 6.71-6.79 (m, 4H), 6.99-7.10 (m, 4H), 7.50-7.52 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.9, 22.1, 22.6, 22.7, 22.8, 27.7, 27.9, 29.0, 29.2, 50.7 (d, *J* = 13.4 Hz), 54.7, 109.1, 118.8, 119.1, 119.5, 127.2, 127.6, 127.7, 128.6, 129.3, 129.6, 132.0, 132.8, 133.8, 137.2, 138.0, 149.1, 149.6 (d, *J* = 8.6 Hz), 156.1; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 152.0; IR (neat): 2933, 1600, 1466, 1239, 1096, 938, 833, 751 cm^{-1}; HRMS (ESI): m/z calcd for $C_{38}H_{42}NO_4P$ $[M+Na]^+$ 630.2749. Found 630.2750; $[\alpha]_D^{20}$ = 78.7 (*c* = 0.86, $CHCl_3$).

Preparation of L5-12, 14, 16



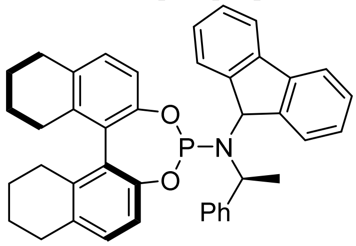
A flame-dried 250 mL flask, equipped with a magnetic stirring bar, was charged with toluene (50 mL) and PCl_3 (0.67 mL, 7.7 mmol, 1.1 equiv), and then the solution was cooled to 0 °C. Another flame-dried, 25 mL flask was charged with secondary amine (7.7 mmol, 1.1 equiv), toluene (8.0 mL), and Et_3N (1.8 mL, 12.9 mmol, 1.84 equiv). This mixture was added dropwise to the above mentioned PCl_3 solution at 0 °C. After the addition was complete, the reaction mixture was heated at 70 °C for 6 h, and then was cooled to –78 °C. To this flask at –78 °C, a solution of (*S*)-BINOL (2.0 g, 7.0 mmol, 1 equiv) and Et_3N (3.5 mL, 25.2 mmol, 3.6 equiv) in toluene (30 mL) and THF (6.0 mL) was added slowly. The resulting mixture was stirred at room temperature for overnight, then filtered through celite, and the residue was washed with Et_2O. The filtrate was concentrated in vacuo. The product was purified by column chromatography (hexane/EtOAc = 15/1). The solvent was evaporated in vacuo to afford the pure ligand.

(11b*S*)-*N*-(9*H*-fluoren-9-yl)-*N*-((*S*)-1-phenylethyl)dinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L5**)



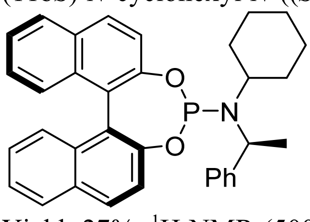
Yield: 32%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.54 (d, *J* = 13.7 Hz, 3H), 3.37-3.45 (m, 1H), 5.30 (s, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 7.12-7.35 (m, 14H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.57-7.62 (m, 3H), 7.72-7.76 (m, 3H), 7.91-7.96 (m, 2H), 8.06 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 26.1 (d, *J* = 25.0 Hz), 56.5 (d, *J* = 28.6 Hz), 62.1, 119.4, 119.5, 121.5 (d, *J* = 2.4 Hz), 122.3, 124.1 (d, *J* = 4.8 Hz), 124.4, 124.8, 125.9, 126.1, 126.3, 126.5, 126.9, 127.1, 127.1, 127.4, 127.9 (d, *J* = 4.8 Hz), 128.1, 128.2, 128.3, 129.7, 130.4 (d, *J* = 2.4 Hz), 131.4, 132.6, 132.8 (d, *J* = 2.4 Hz), 140.8, 140.9, 142.7, 144.5, 145.5, 149.5; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 144.7; IR (neat): 2919, 1590, 1449, 1385, 1092, 947, 746, 695 cm^{-1}; HRMS (ESI): m/z calcd for $C_{41}H_{30}NO_2P$ $[M+Na]^+$ 622.1906. Found 622.1914; $[\alpha]_D^{20}$ = -24.9 (*c* = 0.73, $CHCl_3$).

(11b*S*)-*N*-(9*H*-fluoren-9-yl)-*N*-((*S*)-1-phenylethyl)-8,9,10,11,12,13,14,15-octahydrodinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L6**)



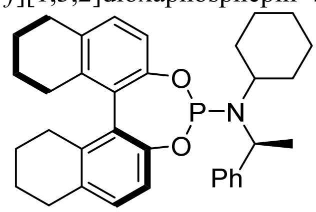
Yield: 71%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.44-1.55 (m, 5H), 1.64-1.70 (m, 3H), 1.75-1.82 (m, 3H), 2.18-2.28 (m, 2H), 2.54-2.72 (m, 4H), 2.79-2.89 (m, 2H), 3.27-3.34 (m, 1H), 5.27 (s, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.98 (t, *J* = 7.4 Hz, 1H), 7.05-7.28 (m, 10H), 7.31-7.39 (m, 2H), 7.64 (dd, *J* = 16.0, 7.4 Hz, 2H), 7.94 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 22.5, 22.5, 22.6, 22.7, 26.0 (d, *J* = 28.6 Hz), 27.7, 27.8, 28.9, 29.2, 56.3 (d, *J* = 25.0 Hz), 62.0, 118.8, 119.1 (d, *J* = 2.4 Hz), 119.2, 119.4, 126.3 (d, *J* = 6.0 Hz), 126.8, 126.9 (d, *J* = 2.4 Hz), 127.0, 127.1 (d, *J* = 2.4 Hz), 127.3, 127.8 (d, *J* = 6.0 Hz), 128.0, 128.9, 129.3 (d, *J* = 4.8 Hz), 129.4, 132.8, 134.2, 137.3, 138.1 (d, *J* = 2.4 Hz), 140.8, 140.9, 143.1, 145.2, 145.7, 147.8 (d, *J* = 7.2 Hz), 149.0; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 138.5; IR (neat): 2928, 1580, 1469, 1218, 1092, 937, 759, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{41}H_{38}NO_2P$ $[M+Na]^+$ 630.2532. Found 630.2554; $[\alpha]_D^{20}$ = -122.4 (*c* = 1.2, $CHCl_3$).

(11b*S*)-*N*-cyclohexyl-*N*-((*S*)-1-phenylethyl)dinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L7**)



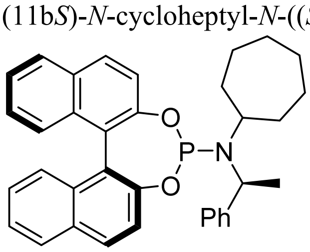
Yield: 27%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.48-0.56 (m, 1H), 0.73-0.81 (m, 1H), 0.86-0.94 (m, 1H), 1.31-1.34 (m, 1H), 1.37-1.43 (m, 1H), 1.49-1.57 (m, 4H), 1.69 (d, *J* = 6.9 Hz, 3H), 1.82-1.84 (m, 1H), 2.71-2.77 (m, 1H), 4.49-4.56 (m, 1H), 7.21-7.34 (m, 5H), 7.37-7.42 (m, 5H), 7.53 (d, *J* = 8.6 Hz, 1H), 7.58 (d, *J* = 7.4 Hz, 2H), 7.76 (d, *J* = 8.6 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.97 (d, *J* = 9.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 24.4 (d, *J* = 17.9 Hz), 25.3, 26.1, 33.9 (d, *J* = 6.0 Hz), 34.6, 53.7 (d, *J* = 16.7 Hz), 55.4 (d, *J* = 3.6 Hz), 121.6 (d, *J* = 2.4 Hz), 122.0, 122.4 (d, *J* = 2.4 Hz), 124.0, 124.1, 124.3, 124.6, 125.8, 125.9, 126.5, 127.0 (t, J = 2.4 Hz), 128.0, 128.2, 128.3, 129.4, 130.2, 130.4, 131.3, 132.6, 132.8, 145.5, 150.0, 150.4, 150.4; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 150.7; IR (neat): 2927, 1590, 1463, 1231, 1071, 944, 822, 750 cm^{-1}; HRMS (ESI): m/z calcd for $C_{34}H_{32}NO_2P$ $[M+Na]^+$ 540.2063. Found 540.2054; $[\alpha]_D^{20}$ = 177.8 (c = 0.82, $CHCl_3$).

(11b*S*)-*N*-cyclohexyl-*N*-((*S*)-1-phenylethyl)-8,9,10,11,12,13,14,15-octahydrodinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L8**)



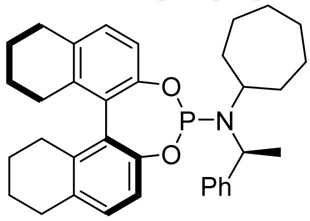
Yield: 60%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.50-0.58 (m, 1H), 0.79-0.93 (m, 2H), 1.16-1.24 (m, 1H), 1.30-1.33 (m, 1H), 1.37-1.43 (m, 2H), 1.46-1.58 (m, 4H), 1.67-1.84 (m, 10H), 2.18-2.24 (m, 1H), 2.32-2.37 (m, 1H), 2.49-2.56 (m, 1H), 2.57-2.67 (m, 2H), 2.69-2.86 (m, 4H), 4.39-4.47 (m, 1H), 6.78 (d, J = 8.0 Hz, 1H), 6.88 (d, J = 8.0 Hz, 1H), 7.01 (d, J = 8.0 Hz, 1H), 7.06 (d, J = 8.0 Hz, 1H), 7.24 (t, J = 7.2 Hz, 1H), 7.36 (t, J = 7.7 Hz, 2H), 7.54 (d, J = 7.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 22.6, 22.7, 25.4 (d, J = 22.7 Hz), 25.6, 26.2, 26.4, 27.5, 27.8, 29.0, 29.2, 33.4 (d, J = 2.4 Hz), 33.8, 53.8 (d, J = 22.7 Hz), 55.4, 118.3, 118.8 (d, J = 2.4 Hz), 126.3, 126.9 (d, J = 3.6 Hz), 127.6, 128.1, 128.7, 129.3 (d, J = 4.8 Hz), 132.6, 133.7, 137.5, 137.9, 146.6, 148.6, 148.6, 149.4; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 146.1; IR (neat): 2923, 1581, 1469, 1221, 1094, 937, 757 cm^{-1}; HRMS (ESI): m/z calcd for $C_{34}H_{40}NO_2P$ $[M+Na]^+$ 548.2689. Found 548.2692; $[\alpha]_D^{20}$ = 62.2 (c = 0.88, $CHCl_3$).

(11b*S*)-*N*-cycloheptyl-*N*-((*S*)-1-phenylethyl)dinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L9**)



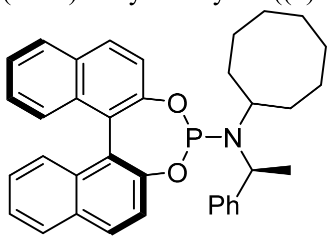
Yield: 37%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.62-0.71 (m, 1H), 1.03-1.56 (m, 9H), 1.67-1.76 (m, 4H), 1.91-1.96 (m, 1H), 2.90-2.97 (m, 1H), 4.47-4.54 (m, 1H), 7.21-7.31 (m, 5H), 7.36-7.42 (m, 5H), 7.53-7.58 (m, 3H), 7.75 (d, J = 8.6 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.98 (d, J = 8.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 24.9, 25.1, 26.8, 27.2, 31.6, 35.3, 37.1, 54.4 (d, J = 16.7 Hz), 57.3 (d, J = 3.6 Hz), 121.6 (d, J = 2.4 Hz), 122.0, 122.4 (d, J = 2.4 Hz), 124.0, 124.0, 124.3, 124.6, 125.8, 125.9, 126.5, 127.0 (t, J = 3.6 Hz), 128.0, 128.2, 128.3, 129.5, 130.2, 130.4, 131.3, 132.6, 132.8, 145.7, 150.0, 150.3, 150.3; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 149.7; IR (neat): 2925, 1462, 1231, 1071, 946, 822, 750, 626 cm^{-1}; HRMS (ESI): m/z calcd for $C_{35}H_{34}NO_2P$ $[M+Na]^+$ 554.2219. Found 554.2197; $[\alpha]_D^{20}$ = 163.5 (c = 1.2, $CHCl_3$).

(11b*S*)-*N*-cycloheptyl-*N*-((*S*)-1-phenylethyl)-8,9,10,11,12,13,14,15-octahydrodinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L10**)



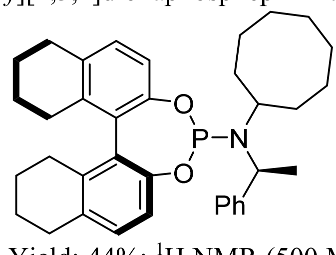
Yield: 29%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.70-0.78 (m, 1H), 1.05-1.37 (m, 8H), 1.43-1.53 (m, 3H), 1.58-1.84 (m, 11H), 2.17-2.23 (m, 1H), 2.32-2.37 (m, 1H), 2.57-2.72 (m, 3H), 2.74-2.86 (m, 4H), 4.37-4.44 (m, 1H), 6.74 (d, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 7.02 (d, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.54 (d, *J* = 7.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 22.6, 22.7 (d, *J* = 2.4 Hz), 25.2, 25.4, 26.6, 27.4, 27.6, 27.9, 29.0, 29.2, 34.5, 36.4, 54.5 (d, *J* = 21.5 Hz), 57.0, 118.3, 118.8 (d, *J* = 2.4 Hz), 126.3, 126.8 (d, *J* = 3.6 Hz), 127.7, 128.2, 128.9, 129.2, 132.5, 133.7, 137.5, 137.9, 146.8, 148.5, 148.5, 149.5; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 144.5; IR (neat): 2924, 1716, 1541, 1221, 1094, 938, 758 cm^{-1}; HRMS (ESI): m/z calcd for $C_{35}H_{42}NO_2P$ [M+Na]$^+$ 562.2845. Found 562.2844; $[\alpha]_D^{20}$ = 67.0 (*c* = 0.99, $CHCl_3$).

(11b*S*)-*N*-cyclooctyl-*N*-((*S*)-1-phenylethyl)dinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L11**)



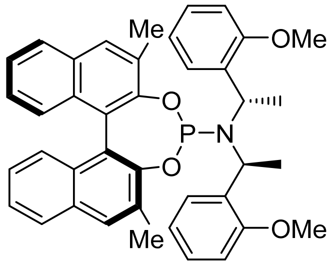
Yield: 16%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.47-0.61 (br, 1H), 0.85-1.12 (m, 6H), 1.21-1.58 (m, 5H), 1.69 (d, *J* = 6.9 Hz, 3H), 1.77-1.87 (m, 2H), 3.11-3.18 (m, 1H), 4.44-4.53 (m, 1H), 7.21-7.27 (m, 2H), 7.29-7.32 (m, 2H), 7.34-7.43 (m, 6H), 7.54-7.59 (m, 3H), 7.78 (d, *J* = 9.2 Hz, 1H), 7.84 (d, *J* = 8.2 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.98 (d, *J* = 9.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 23.7, 25.0, 25.7, 25.7, 26.0, 28.9, 35.0, 36.0, 54.7 (d, *J* = 17.2 Hz), 55.3 (d, *J* = 2.9 Hz), 121.7 (d, *J* = 1.9 Hz), 122.1, 122.4 (d, *J* = 1.9 Hz), 123.9, 124.0, 124.3, 124.6, 125.8, 125.9, 126.5, 126.9 (t, *J* = 2.9 Hz), 127.0, 128.0, 128.2 (d, *J* = 1.9 Hz), 129.7, 130.2, 130.5, 131.2, 132.8, 132.8, 132.8, 150.0, 150.2, 150.3; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 149.8; IR (neat): 2920, 1716, 1507, 1231, 1070, 947, 822, 749 cm^{-1}; HRMS (ESI): m/z calcd for $C_{36}H_{36}NO_2P$ [M+Na]$^+$ 568.2376. Found 568.2366; $[\alpha]_D^{20}$ = 146.5 (*c* = 0.97, $CHCl_3$).

(11b*S*)-*N*-cyclooctyl-*N*-((*S*)-1-phenylethyl)-8,9,10,11,12,13,14,15-octahydrodinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L12**)



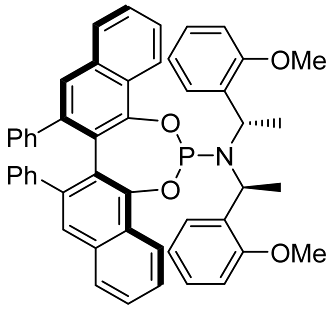
Yield: 44%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.86-0.96 (m, 2H), 1.07-1.44 (m, 10H), 1.49-1.56 (m, 3H), 1.69-1.78 (m, 10H), 2.20-2.24 (m, 1H), 2.30-2.35 (m, 1H), 2.59-2.71 (m, 3H), 2.75-2.86 (m, 3H), 3.07-3.12 (m, 1H), 4.34-4.41 (m, 1H), 6.80 (d, *J* = 8.3 Hz, 1H), 6.91 (d, *J* = 8.3 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 1H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.36 (t, *J* = 7.7 Hz, 2H), 7.55 (d, *J* = 7.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 22.6, 22.7, 22.7, 22.8, 23.5, 24.7, 25.9, 26.0, 26.6, 27.6, 28.0, 29.1, 29.2, 34.7, 35.4, 54.8 (d, *J* = 20.3 Hz), 55.1, 118.4, 118.9 (d, *J* = 2.4 Hz), 126.3, 126.7 (d, *J* = 3.6 Hz), 127.7, 128.2, 129.2, 129.2, 132.5, 133.8, 137.3, 137.9, 147.3, 148.5, 148.5, 149.5; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 143.1; IR (neat): 2924, 1653, 1470, 1221, 1101, 939, 758, 699 cm^{-1}; HRMS (ESI): m/z calcd for $C_{36}H_{44}NO_2P$ [M+Na]$^+$ 576.3002. Found 576.2986; $[\alpha]_D^{20}$ = 73.6 (*c* = 1.0, $CHCl_3$).

(11b*S*)-*N*,*N*-bis((*S*)-1-(2-methoxyphenyl)ethyl)-2,6-dimethyldinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L14**)



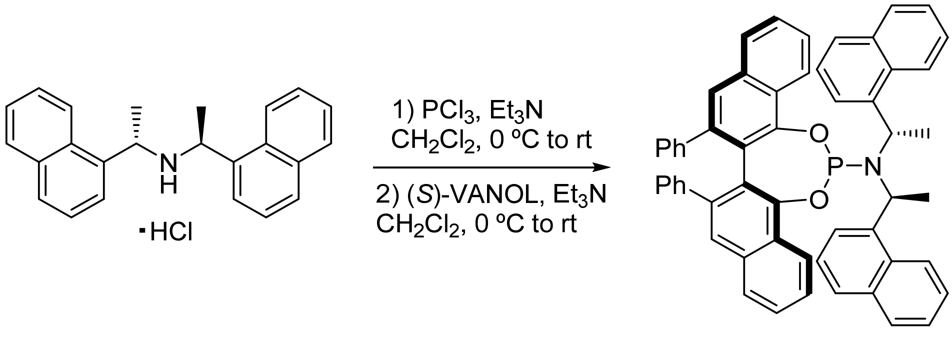
Yield: 64%; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.56 (d, *J* = 7.8 Hz, 6H), 2.14 (s, 3H), 2.76 (s, 3H), 3.55 (s, 6H), 4.99-5.06 (m, 2H), 6.60 (d, *J* = 8.2 Hz, 2H), 6.77 (t, *J* = 7.8 Hz, 2H), 7.05-7.12 (m, 5H), 7.21-7.34 (m, 3H), 7.46 (s, 1H), 7.60 (d, *J* = 7.8 Hz, 2H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.81-7.83 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 17.8 (d, *J* = 14.3 Hz), 22.3, 51.5 (d, *J* = 15.5 Hz), 54.7, 109.4, 119.7, 121.3 (d, *J* = 3.6 Hz), 123.9, 124.1, 124.4, 124.6 (d, *J* = 2.4 Hz), 124.9, 127.1 (d, *J* = 3.6 Hz), 127.4, 127.6 (d, *J* = 4.8 Hz), 129.0, 129.2, 129.6, 129.7, 130.1, 130.3, 130.8, 131.2, 131.8, 131.8, 132.8, 149.3, 150.4, 150.4, 156.0; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 150.7; IR (neat): 2930, 1601, 1461, 1240, 1094, 1034, 903, 750 cm^{-1}; HRMS (ESI): m/z calcd for $C_{40}H_{38}NO_4P$ $[M+Na]^+$ 650.2431. Found 650.2431; $[\alpha]_D^{20}$ = -242.0 (*c* = 1.14, $CHCl_3$).

(9a*S*)-*N*,*N*-bis((*S*)-1-(2-methoxyphenyl)ethyl)-9,10-diphenyldinaphtho[1,2-*d*:2',1'-*f*][1,3,2]dioxaphosphepin-2-amine (**L16**)



Yield: 91%; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.49 (d, *J* = 5.2 Hz, 6H), 3.61 (s, 6H), 5.06-5.12 (m, 2H), 6.48 (d, *J* = 6.9 Hz, 2H), 6.56 (t, *J* = 7.2 Hz, 4H), 6.84-6.92 (m, 6H), 7.03-7.09 (m, 4H), 7.16-7.19 (m, 1H), 7.26 (s, 1H), 7.34-7.37 (m, 1H), 7.42 (s, 1H), 7.55-7.58 (m, 1H), 7.65-7.70 (m, 2H), 7.72-7.74 (m, 2H), 7.83 (d, *J* = 8.6 Hz, 1H), 7.93 (d, *J* = 8.6 Hz, 1H), 8.58 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 22.3 (d, *J* = 10.7 Hz), 52.1 (d, *J* = 15.5 Hz), 54.7, 109.5, 119.8, 122.3, 122.3 (d, *J* = 2.4 Hz), 123.2, 123.8, 125.1, 125.5, 125.5, 125.8, 126.0, 126.1, 126.3, 126.6, 126.7, 126.8, 127.0, 127.4, 127.4, 127.6 (d, *J* = 4.8 Hz), 128.0, 129.1, 129.2, 132.9 (d, *J* = 2.4 Hz), 133.7, 134.2, 140.5, 140.6, 140.8, 140.8, 147.9, 149.1, 149.2, 156.0; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 157.2; IR (neat): 2925, 2378, 1716, 1489, 1362, 1240, 1087, 750 cm^{-1}; HRMS (ESI): m/z calcd for $C_{50}H_{42}NO_4P$ $[M+Na]^+$ 774.2744. Found 774.2720; $[\alpha]_D^{20}$ = 109.1 (*c* = 1.4, $CHCl_3$).

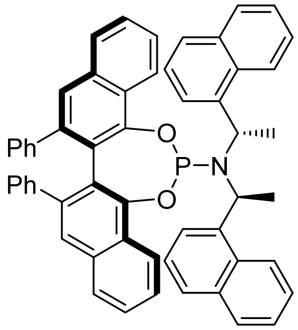
Preparation of L17



L17

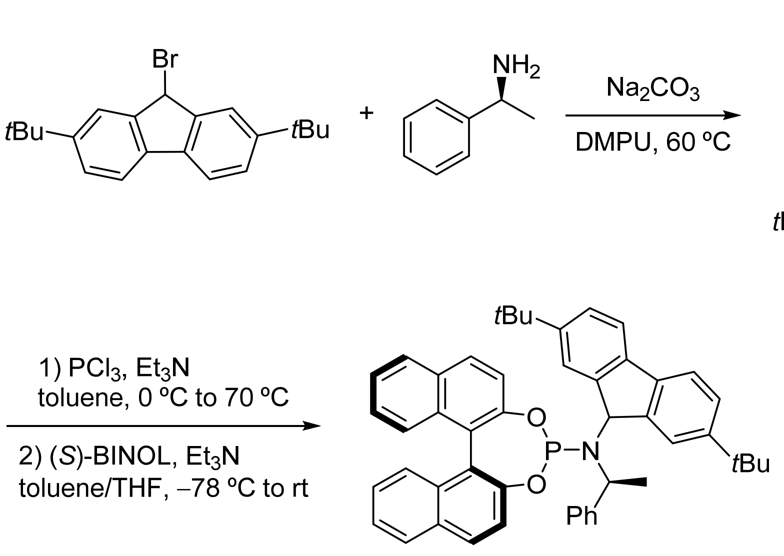
A solution of bis[(*S*)-(−)-(1-naphthyl)ethyl]amine hydrochloride (74 mg, 0.20 mmol, 1.1 equiv) and Et_3N (120 µL, 0.84 mmol, 6 equiv) in dry CH_2Cl_2 (2.0 mL) was added to a dry Schlenck tube containing 3A molecular sieves (600 mg) under argon atmosphere. The mixture was cooled down to 0 °C and PCl_3 (162 µL, 1.86 mmol, 10.3 equiv) was added dropwise. The mixture was stirred at room temperature for 2 h and the volatiles were carefully removed connecting the Schlenck tube to high vacuum. The resulting yellowish solid was dissolved in dry CH_2Cl_2 (2.0 mL) under argon atmosphere and then volatiles where removed again under vacuum. This procedure was repeated twice in order to remove excess of PCl_3. Then, the resulting residue was dissolved in dry CH_2Cl_2 (2.0 mL), Et_3N (120 µL, 0.84 mmol, 6 equiv) was added and the mixture was cooled down to 0 °C. A solution of (*S*)-VANOL (79 mg, 0.18 mmol, 1 equiv) and Et_3N (120 µL, 0.84 mmol, 6 equiv) in dry CH_2Cl_2 (2.0 mL) was then added dropwise at 0 °C. The resulting mixture was allowed to warm to room temperature and stirred for 16 h. The solvent was evaporated and the crude residue was purified by column chromatography (hexane/EtOAc = 15/1) to yield the pure ligand **L17** (69 mg, 48%).

(9a*S*)-*N*,*N*-bis((*S*)-1-(naphthalen-1-yl)ethyl)-9,10-diphenyldinaphtho[1,2-*d*:2',1'-*f*][1,3,2]dioxaphosphepin-2-amine (**L17**)



^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.60-1.70 (m, 6H), 5.49-5.58 (m, 2H), 6.48-6.51 (m, 4H), 6.87-6.92 (m, 4H), 6.99 (t, *J* = 7.7 Hz, 2H), 7.03-7.09 (m, 2H), 7.15-7.19 (m, 2H), 7.27-7.33 (m, 3H), 7.38-7.41 (m, 4H), 7.46 (s, 1H), 7.53 (d, *J* = 8.6 Hz, 1H), 7.63-7.75 (m, 5H), 7.78-7.84 (m, 3H), 7.89 (d, *J* = 8.6 Hz, 1H), 8.05 (d, *J* = 8.6 Hz, 1H), 8.66 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 22.9 (d, *J* = 8.3 Hz), 54.1 (d, *J* = 19.2 Hz), 122.3, 122.4 (d, *J* = 2.4 Hz), 122.7, 122.9 (d, *J* = 3.6 Hz), 124.2, 124.4 (d, *J* = 2.4 Hz), 124.8, 125.0, 125.3, 125.5, 125.6, 125.6, 126.1, 126.2, 126.5, 126.6, 126.6, 126.9, 127.0, 127.0, 127.3, 127.4 (d, *J* = 3.6 Hz), 128.2, 128.7, 129.2, 129.2, 130.5 (d, *J* = 2.4 Hz), 133.3, 133.8, 134.4, 139.3 (d, *J* = 2.4 Hz), 140.4, 140.5, 140.7, 140.8, 147.7, 149.1, 149.2; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 154.2; IR (neat): 2926, 2374, 1716, 1508, 1362, 1086, 761, 698 cm^{-1}; HRMS (ESI): m/z calcd for $C_{56}H_{42}NO_2P$ $[M+Na]^+$ 814.2845. Found 814.2863; $[\alpha]_D^{20}$ = 169.6 (*c* = 1.3, $CHCl_3$).

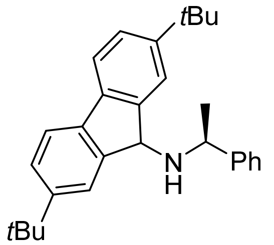
Preparation of L18



L18

Under argon atmosphere, 9-bromo-2,7-di-*tert*-butyl-9*H*-fluorene (2.0 g, 5.6 mmol, 1.19 equiv) and Na_2CO_3 (1.0 g, 9.3 mmol, 1.98 equiv) were mixed with 19 mL of dry DMPU. The resulting white suspension was then treated with (*S*)-1-phenylethan-1-amine (0.57 g, 4.7 mmol, 1 equiv), and the reaction mixture was stirred in an oil bath at 60 °C for 12 hours. At the end of the reaction, the suspension was cooled down to 0 °C, and diluted with 15 mL of H_2O. Products were extracted with Et_2O (15 mL x 3). The combined organic extracts were washed with H_2O (15 mL x 3), dried over Na_2SO_4, filtered, and evaporated. The resulting residue was directly subjected to silica gel flash chromatography (hexane/EtOAc = 10/1) to give (*S*)-2,7-di-*tert*-butyl-*N*-(1-phenylethyl)-9*H*-fluoren-9-amine (1.8 g, 97%).

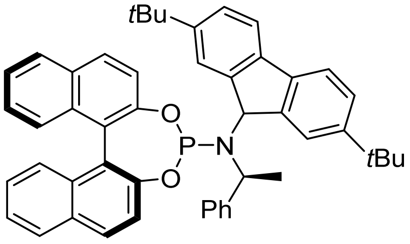
(*S*)-2,7-di-*tert*-butyl-*N*-(1-phenylethyl)-9*H*-fluoren-9-amine



^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.32 (s, 12H), 1.39 (s, 9H), 4.08 (q, *J* = 6.5 Hz, 1H), 4.73 (s, 1H), 7.21-7.24 (m, 1H), 7.31-7.41 (m, 6H), 7.47 (s, 1H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.60 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 25.5, 31.5, 31.6, 34.7, 34.9, 55.6, 61.8, 118.8, 119.0, 122.0, 122.1, 124.8, 124.9, 126.9, 126.9, 128.4, 137.8, 137.9, 146.2, 146.4, 146.5, 149.6, 150.0; IR (neat): 3335, 2961, 1477, 1362, 1257, 820, 700 cm^{-1}; HRMS (ESI): m/z calcd for $C_{29}H_{35}N$ $[M+H]^+$ 398.2842. Found 398.2827;

A flame-dried 200 mL flask, equipped with a magnetic stirring bar, was charged with toluene (30 mL) and PCl_3 (0.39 mL, 4.5 mmol, 1.1 equiv), and then the solution was cooled to 0 °C. Another flame-dried, 20 mL flask was charged with secondary amine (1.8 g, 4.5 mmol, 1.1 equiv), toluene (4.8 mL), and Et_3N (1.1 mL, 7.4 mmol, 1.8 equiv). This mixture was added dropwise to the above mentioned PCl_3 solution at 0 °C. After the addition was complete, the reaction mixture was heated at 70 °C for 6 h, and then was cooled to –78 °C. To this flask at –78 °C, a solution of (*S*)-BINOL (1.2 g, 4.1 mmol, 1 equiv) and Et_3N (2.2 mL, 14.8 mmol, 3.6 equiv) in toluene (18 mL) and THF (3.5 mL) was added slowly. The resulting mixture was stirred at room temperature overnight, then filtered through celite, and washed with Et_2O. The organic phase was concentrated in vacuo. The product was purified by column chromatography (hexane/EtOAc = 15/1). The solvent was evaporated in vacuo to afford the pure ligand **L18** (2.4 g, 80%).

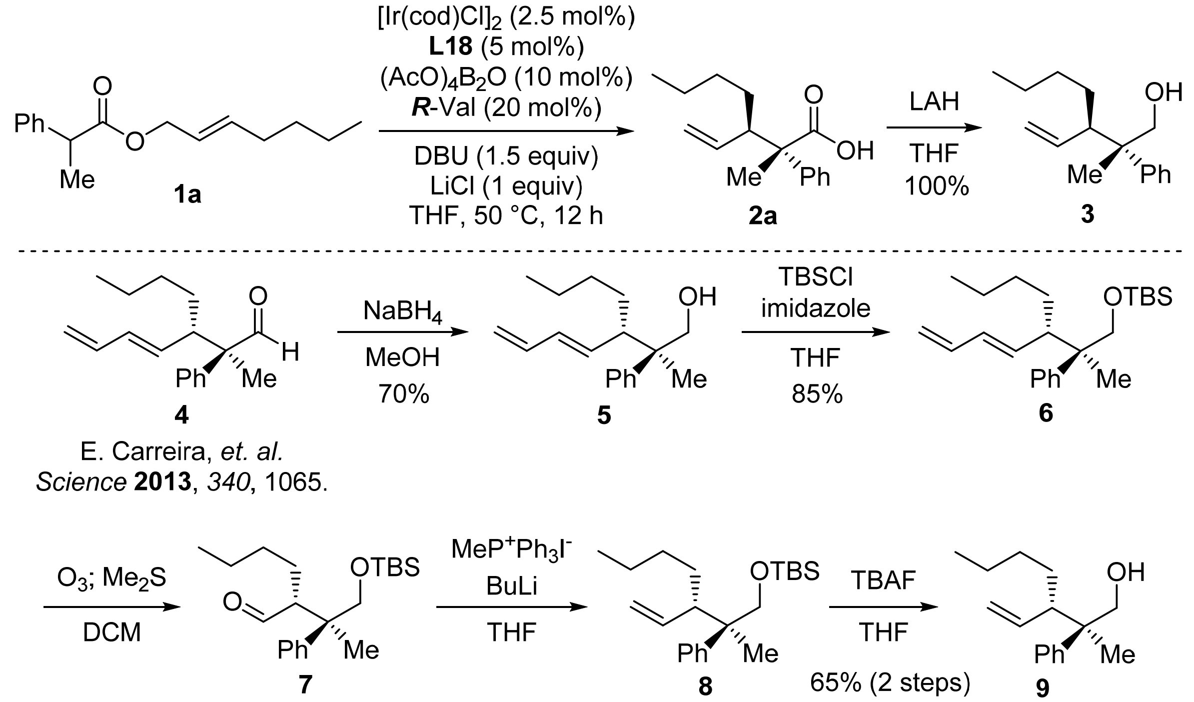
(11b*S*)-*N*-(2,7-di-*tert*-butyl-9*H*-fluoren-9-yl)-*N*-((*S*)-1-phenylethyl)dinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-amine (**L18**)



^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.31 (s, 9H), 1.45 (s, 9H), 1.57 (d, *J* = 6.9 Hz, 3H), 3.39-3.47 (m, 1H), 5.32 (s, 1H), 7.20-7.41 (m, 13H), 7.48 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 8.6 Hz, 2H), 7.67 (d, *J* = 9.2 Hz, 1H), 7.77-7.82 (m, 3H), 8.01 (d, *J* = 8.6 Hz, 1H), 8.11-8.13 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 14.2, 22.7, 26.5 (d, *J* = 25.0 Hz), 31.5 (d, *J* = 9.5 Hz), 34.8 (d, *J* = 20.3 Hz), 56.6 (d, *J* = 25.0 Hz), 62.4 (d, *J* = 4.8 Hz), 118.7, 118.8, 121.9 (d, *J* = 2.4 Hz), 122.0, 122.5, 123.4, 123.8, 124.0, 124.1, 124.5, 124.8, 125.2 (d, *J* = 3.6 Hz), 125.9, 126.1, 126.5, 126.7 (d, *J* = 4.8 Hz), 127.1 (d, *J* = 6.0 Hz), 128.1, 128.1, 128.3, 129.5, 130.5, 130.6, 131.4, 132.7, 132.9, 138.2, 138.5, 142.7, 144.6, 145.9, 148.9, 149.3, 149.3, 149.7, 149.9; ^{31}P NMR (159 MHz, $CDCl_3$): δ = 146.4; IR (neat): 2961, 1458, 1230, 1093, 948, 819, 771, 695 cm^{-1}; HRMS (ESI): m/z calcd for $C_{49}H_{46}NO_2P$ $[M+Na]^+$ 734.3158. Found 734.3152; $[\alpha]_D^{20}$ = -41.0 (*c* = 1.3, $CHCl_3$).

5. Determination of Relative and Absolute Configuration of the Products

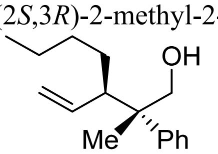
The relative and absolute configuration of **2a** was determined by comparing with the spectra data of **4** (Ref.14 in main text) after conversion into **9** as shown in the following scheme. NMR spectra of **3** matched with that of **9**, suggesting **3** has the same relative configuration with **9**. HPLC analysis revealed that absolute configuration of **3** and **9** were opposite. From these results, we confirmed product **2a** in the reaction with Ir/**L18** and B/***R*** to be (2*S*,3*R*)-**2a**. Those of other products **2** were assigned by analogy.



Procedure for preparation of 3 from 2a

To a solution of $LiAlH_4$ (77.0 mg, 2.0 mmol, 10 equiv) in THF (2 ml), **2a** (50.0 mg, 0.20 mmol, 1 equiv) was added at –78 °C and the reaction mixture was stirred at reflux temperature for overnight. The reaction was quenched with H_2O (0.05 mL). 15% NaOH solution in H_2O (0.05 mL) and H_2O (0.15 mL) were added successively. The mixture was stirred for 1 h and the solid was filtered through pad of celite. The crude solution was evaporated to give **3** (47 mg, 10/1 dr, 100%) as a colorless oil.

(2*S*,3*R*)-2-methyl-2-phenyl-3-vinylheptan-1-ol (**3**)



^{1}H NMR (500 MHz, $CDCl_3$): major diastereomer δ = 0.78-1.50 (m, 9H), 1.55 (s, 3H), 2.32-2.42 (m, 1H), 3.71 (d, *J* = 9.2 Hz, 1H), 3.78 (d, *J* = 9.2 Hz, 1H), 4.87 (dd, *J* = 17.2, 2.3 Hz, 1H), 4.96 (dd, *J* = 10.0, 2.3 Hz, 1H), 5.42 (ddd, *J* = 17.2, 10.0, 9.7 Hz, 1H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.18-7.27 (m, 2H), 7.30-7.36 (m, 2H); HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): 99% ee, t_R = 11.2 min (major), t_R = 11.6 min (minor)

Procedure for preparation of 9 from 4

To a solution of **4** (217.1 mg, 0.85 mmol, 1equiv) in MeOH (4 ml), $NaBH_4$ (64.1 mg, 1.69 mmol, 2 equiv) was added at 0 °C and the reaction mixture was stirred at room temperature for 2 h. The reaction was quenched with NH_4Cl aq and products were extracted with EtOAc. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. The crude product was purified by column chromatography (hexane/EtOAc). **5** was obtained as a colorless oil (153.8 mg, 70%).

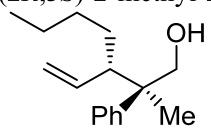
To a solution of **5** (153.8 mg, 0.60 mmol, 1 equiv) and imidazole (101.3 mg, 1.49 mmol, 2.47 equiv) in DCM (3 ml), TBSCl (134.6 mg, 0.89 mmol, 1.48 equiv) was added at room temperature and the reaction mixture was stirred for 2 h. The reaction was quenched with H_2O and products were extracted with EtOAc. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. The crude product was purified by column chromatography (hexane/EtOAc). **6** was obtained as a colorless oil (187.5 mg, 85%).

Ozone was bubbled through a solution of **6** (187.5 mg, 0.50 mmol) in DCM (4 ml) at −78 °C until the solution took on a characteristic blue color. Two drops of Me_2S was added to the reaction mixture. The mixture was stirred at room temperature for 1 h. After evaporation, the crude product was purified by column chromatography (hexane/EtOAc). **7** was obtained as a colorless oil (82.3 mg, 50%).

A flame-dried round-bottom flask was charged with methyltriphenylphosphonium iodide (487 mg, 1.20 mmol, 5 equiv) and THF (8 mL), and the solution was cooled to −78 °C. *n*BuLi (0.74 ml, 1.59 M in hexane, 1.18 mmol, 4.9 equiv) was added dropwise over 3 min, and the reaction was stirred for 30 min at 0 °C, then 1 h at room temperature. The solution was then cooled to −78 °C, and **7** (82.3 mg, 0.24 mmol, 1 equiv) in THF (1 mL) was added via syringe with rinsing with THF (2 × 0.5 mL). The reaction mixture was allowed to warm to room temperature, and the mixture was stirred for overnight. H_2O was added, and the solution was extracted with EtOAc. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. The crude product was purified by column chromatography (hexane/EtOAc). **8** was obtained as a colorless oil.

To a solution of **8** in THF (2 mL), TBAF (0.38ml, 1.0 M in THF, 0.38 mmol, 1.6 equiv) was added at room temperature and the reaction mixture was stirred for 5 h. The reaction was quenched with H_2O and products were extracted with EtOAc. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. The crude product was purified by column chromatography (hexane/EtOAc). **9** was obtained as a colorless oil (22.9 mg, 4/1 dr, 65% in 2 steps).

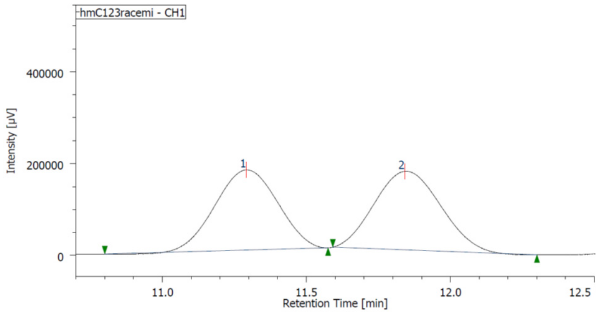
(2*R*,3*S*)-2-methyl-2-phenyl-3-vinylheptan-1-ol (**9**)



^{1}H NMR (500 MHz, $CDCl_3$): major diastereomer δ = 0.78-1.50 (m, 9H), 1.55 (s, 3H), 2.32-2.42 (m, 1H), 3.71 (d, *J* = 9.2 Hz, 1H), 3.78 (d, *J* = 9.2 Hz, 1H), 4.87 (dd, *J* = 17.2, 2.3 Hz, 1H), 4.96 (dd, *J* = 10.0, 2.3 Hz, 1H), 5.42 (ddd, *J* = 17.2, 10.0, 9.7 Hz, 1H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.18-7.27 (m, 2H), 7.30-7.36 (m, 2H); HPLC (chiral column: CHIRALCEL OZ-H; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm; rt): 99% ee, t_R = 10.8 min (minor), t_R = 11.6 min (major)

HPLC chart

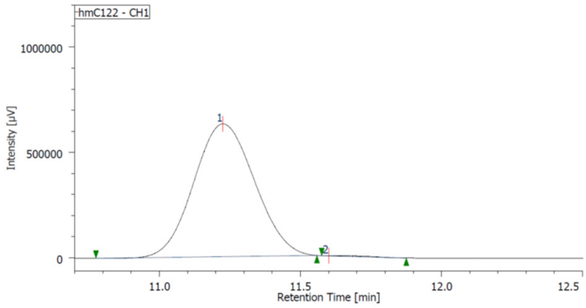
Racemate



チャンネル情報＋ピーク情報
クロマトグラム名　hmC123racemi-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	11.292	2642422	174955	49.082	50.511	N/A	11964	1.296	0.985	
2	Unknown	1	11.842	2741273	171413	50.918	49.489	N/A	11686	N/A	1.119	

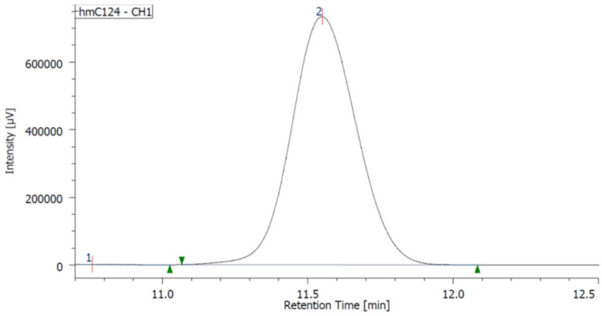
3



チャンネル情報＋ピーク情報
クロマトグラム名　hmC122-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	11.225	9459911	630757	99.724	99.930	N/A	12394	0.964	1.064	
2	Unknown	1	11.600	26229	439	0.276	0.070	N/A	15154	N/A	6.211	

9

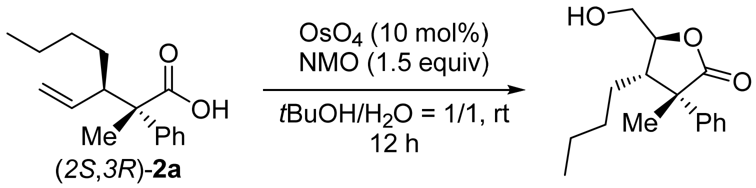


チャンネル情報＋ピーク情報
クロマトグラム名　hmC124-CH1
サンプル名
チャンネル名　CH1
データ取り込み間隔　500 [msec]
波形処理メソッド　（マニュアル）
数値計算式
判定式

#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.758	14648	1484	0.128	0.202	N/A	22877	2.281	1.068	
2	Unknown	1	11.550	11419560	733304	99.872	99.798	N/A	12611	N/A	1.070	

6. Stereodivergent Transformation of 2a

Procedure for preparation of (*3S*,*4S*,*5S*)-3a

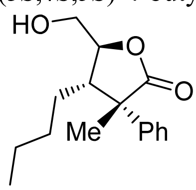


(*2S*,*3R*)-**2a**

(*3S*,*4S*,*5S*)-**3a**

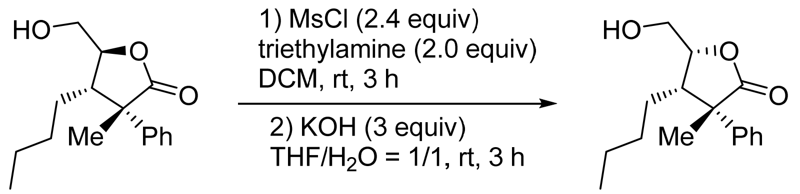
To a solution of (*2S*,*3R*)-**2a** (20.0 mg, 0.0812 mmol, 1 equiv) and *N*-methylmorphiline (14.3 mg, 0.122 mmol, 1.5 equiv) in *t*BuOH (0.4 mL) and H_2O (0.5 mL), OsO_4 (105 μL, 8.12 μmol, 10 mol%, 2.5 w% in *t*BuOH) was added at rt and the reaction mixture was stirred for 12 h. The reaction was diluted with H_2O and EtOAc and products were extracted with EtOAc three times. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. Diastereomeric ratio was determined by ^{1}H NMR analysis (>50/1 dr). The crude product was purified by column chromatography (hexane/EtOAc). (*3S*,*4S*,*5S*)-**3a** was obtained as a colorless oil (22.8 mg, 100%).

(*3S*,*4S*,*5S*)-4-butyl-5-(hydroxymethyl)-3-methyl-3-phenyldihydrofuran-2(3H)-one ((*3S*,*4S*,*5S*)-**3a**)



^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.84 (t, *J* = 7.1 Hz, 3H), 1.21-1.38 (m, 6H), 1.74 (s, 3H), 2.39-2.42 (m, 1H), 3.73 (dd, *J* = 12.8, 4.1 Hz, 1H), 4.08-4.11 (m, 1H), 4.19-4.22 (m, 1H), 7.25 (d, *J* = 8.2 Hz, 2H), 7.32-7.43 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.7, 22.7, 23.2, 27.8, 29.8, 47.3, 52.6, 62.6, 83.1, 126.5, 127.5, 128.6, 137.6, 180.6; IR (neat): 3405, 2932, 1771, 1230, 1033, 700 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_3$ $[M+Na]^+$ 285.1461. Found 285.1468; $[\alpha]_D^{20}$ = 45.4 (*c* = 1.59, $CHCl_3$).

Procedure for preparation of (*3S*,*4S*,*5R*)-3a



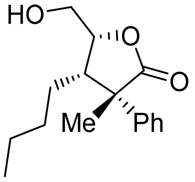
(*3S*,*4S*,*5S*)-**3a** (*3S*,*4S*,*5R*)-**3a**

To a solution of (*3S*,*4S*,*5S*)-**3a** (20.2 mg, 0.077 mmol, 1 equiv) and methanesulfonyl chloride (14.4 μL, 0.185 mmol, 2.4 equiv) in DCM (0.4 mL), triethylamine (21.5 μL, 0.154 mmol, 2.0 equiv) was added at 0 °C and the reaction mixture was stirred at room temperature for 3 h. The reaction was quench with saturated $NaHCO_3$ aq. and products were extracted with EtOAc three times. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. The crude product was purified by column chromatography (hexane/EtOAc) to afford mesylated product.

To a solution of mesylated product (0.077 mmol, 1 equiv) in THF (0.25 mL) and H_2O (0.25 mL), KOH (13.0 mg, 0.231 mmol, 3.0 equiv) was added at 0 °C and the reaction mixture was stirred at room temperature for 3 h. The reaction was quench with aqueous HCl (1M) and products were extracted with EtOAc three times. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. Diastereomeric ratio was determined by ^{1}H NMR analysis (10/1 dr).

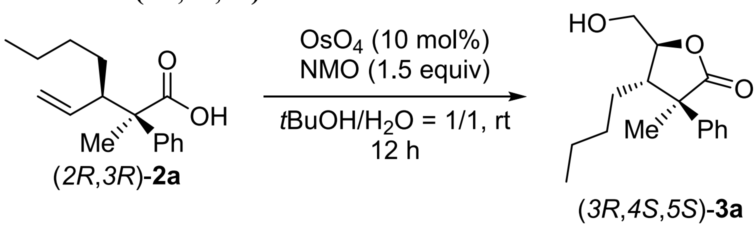
The crude product was purified by column chromatography (DCM/EtOAc). (*3S*,*4S*,*5R*)-**3a** was obtained as a colorless oil (18.6 mg, 92%).

(*3S*,*4S*,*5R*)-4-butyl-5-(hydroxymethyl)-3-methyl-3-phenyldihydrofuran-2(3H)-one ((*3S*,*4S*,*5R*)-**3a**)



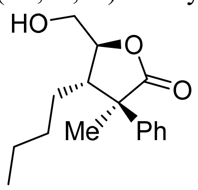
^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.85 (t, *J* = 7.2 Hz, 3H), 1.22-1.30 (m, 5H), 1.44-1.50 (m, 1H), 1.65 (s, 3H), 2.60-2.64 (m, 1H), 3.44-3.46 (m, 1H), 3.60 (dd, *J* = 12.6, 9.7 Hz, 1H), 4.68-4.73 (m, 1H), 7.22-7.24 (m, 2H), 7.27-7.35 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.8, 22.8, 26.0, 26.1, 30.0, 49.2, 51.0, 61.5, 81.7, 127.4, 127.4, 128.4, 139.1, 180.3; IR (neat): 3409, 2932, 1769, 1383, 1069, 704 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_3$ $[M+Na]^+$ 285.1461. Found 285.1468; $[\alpha]_D^{20}$ = -74.9 (*c* = 1.50, $CHCl_3$).

Procedure for preparation of (*3R*,*4S*,*5S*)-3a



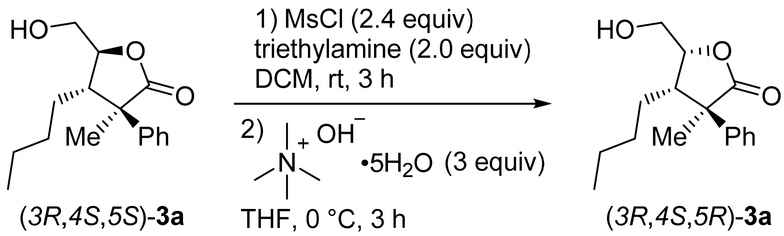
To a solution of (*2R*,*3R*)-**2a** (26.8 mg, 0.109 mmol, 1 equiv) and *N*-methylmorphiline (19.1 mg, 0.163 mmol, 1.5 equiv) in *t*BuOH (0.55 mL) and H_2O (0.7 mL), OsO_4 (142 μL, 10.9 μmol, 10 mol%, 2.5 w% in *t*BuOH) was added at rt and the reaction mixture was stirred for 12 h. The reaction was diluted with H_2O and EtOAc and products were extracted with EtOAc three times. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. Diastereomeric ratio was determined by ^{1}H NMR analysis (>50/1 dr). The crude product was purified by column chromatography (hexane/EtOAc). (*3R*,*4S*,*5S*)-**3a** was obtained as a colorless oil (28.1 mg, 92%).

(*3R*,*4S*,*5S*)-4-butyl-5-(hydroxymethyl)-3-methyl-3-phenyldihydrofuran-2(3H)-one ((*3R*,*4S*,*5S*)-**3a**)



^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.77 (t, *J* = 7.3 Hz, 3H), 1.09-1.18 (m, 4H), 1.40-1.52 (m, 2H), 1.58 (s, 3H), 2.68-2.74 (m, 1H), 3.69-3.75 (m, 1H), 4.04-4.07 (m, 1H), 4.26-4.29 (m, 1H), 7.28-7.38 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.6, 16.4, 22.6, 26.8, 29.4, 47.5, 51.6, 62.5, 83.4, 126.7, 127.2, 128.5, 142.0, 180.7; IR (neat): 3396, 2926, 1761, 1383, 1093, 700 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_3$ $[M+Na]^+$ 285.1461. Found 285.1460; $[\alpha]_D^{20}$ = 51.1 (*c* = 0.69, $CHCl_3$).

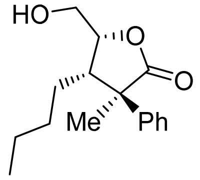
Procedure for preparation of (*3R*,*4S*,*5R*)-3a



To a solution of (*3R*,*4S*,*5S*)-**3a** (31.0 mg, 0.118 mmol, 1 equiv) and methanesulfonyl chloride (22.1 μL, 0.284 mmol, 2.4 equiv) in DCM (1.0 mL), triethylamine (32.9 μL, 0.236 mmol, 2.0 equiv) was added at 0 °C and the reaction mixture was stirred at room temperature for 3 h. The reaction was quench with saturated $NaHCO_3$ aq. and products were extracted with EtOAc three times. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. The crude product was purified by column chromatography (hexane/EtOAc) to afford mesylated product as a colorless oil (40.2 mg, 100%).

To a solution of mesylated product (8.1 mg, 0.024 mmol, 1 equiv) in THF (0.25 mL), tetramethylammonium hydroxide pentahydrate (12.9 mg, 0.714 mmol, 3.0 equiv) was added at 0 °C and the reaction mixture was stirred at 0 °C for 3 h. The reaction was quench with aqueous HCl (1M) and products were extracted with EtOAc three times. The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. Diastereomeric ratio was determined by ^{1}H NMR analysis (6.4/1 dr). The crude product was purified by column chromatography (DCM/EtOAc). (*3R*,*4S*,*5R*)-**3a** was obtained as a colorless oil (4.35 mg, 70%).

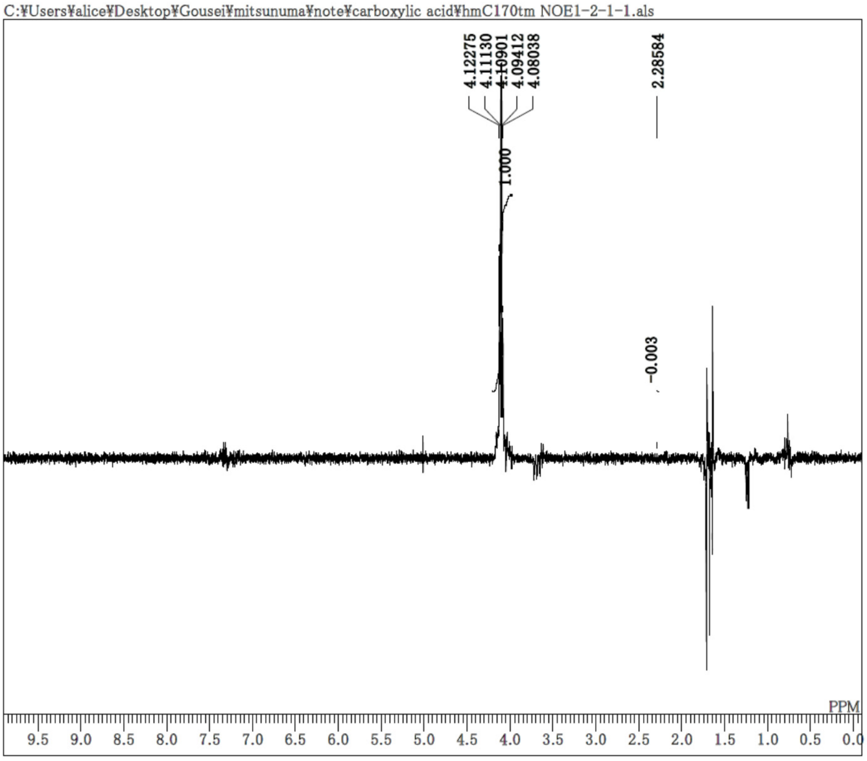
(*3R*,*4S*,*5R*)-4-butyl-5-(hydroxymethyl)-3-methyl-3-phenyldihydrofuran-2(3H)-one ((*3R*,*4S*,*5R*)-**3a**)

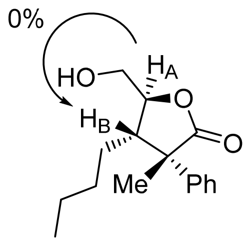


^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 7.2 Hz, 3H), 1.25-1.29 (m, 6H), 1.57 (s, 3H), 2.78-2.82 (m, 1H), 3.83-3.85 (m, 1H), 3.89-3.93 (m, 1H), 4.58-4.62 (m, 1H), 7.27-7.38 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 13.8, 19.8, 22.9, 24.7, 30.2, 49.2, 51.7, 62.3, 81.1, 126.1, 127.3, 128.8, 143.2, 180.4; IR (neat): 3374, 2926, 1769, 1652, 1092, 700 cm^{-1}; HRMS (ESI): m/z calcd for $C_{16}H_{22}O_3$ $[M+Na]^+$ 285.1461. Found 285.1460; $[\alpha]_D^{20}$ = -3.91 (*c* = 0.44, $CHCl_3$).

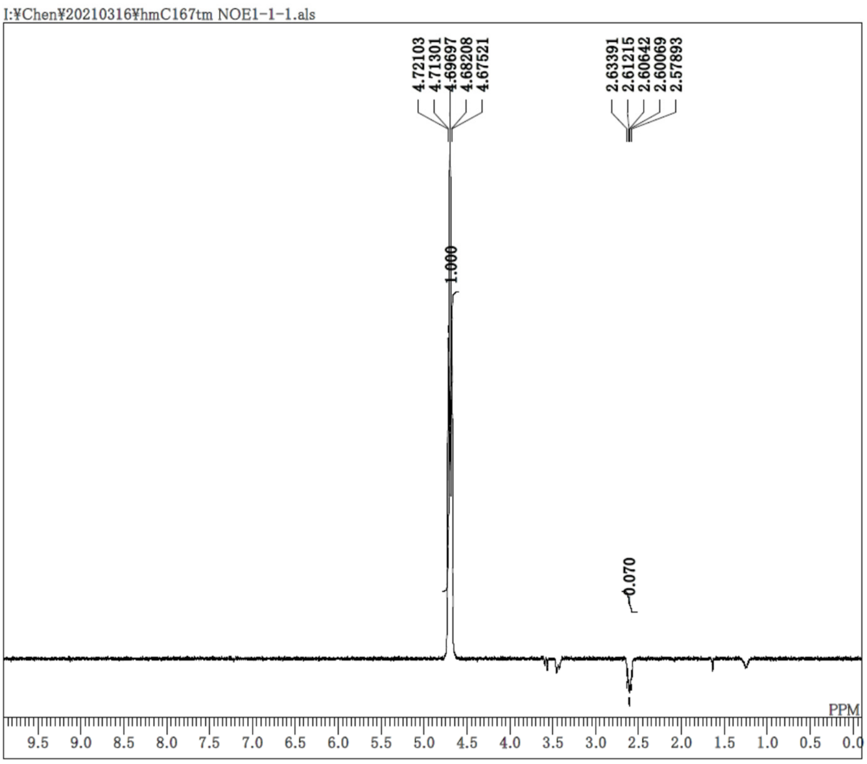
Determination of relative configuration of 3a

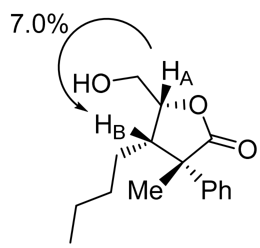
The relative configurations of **3a** were determined by ^{1}H NOE analysis. The results indicated that the proton H_A was located close to the proton H_B (*syn*-configuration) in the case of (*3S*,*4S*,*5R*)-**3a** and (*3R*,*4S*,*5R*)-**3a**.





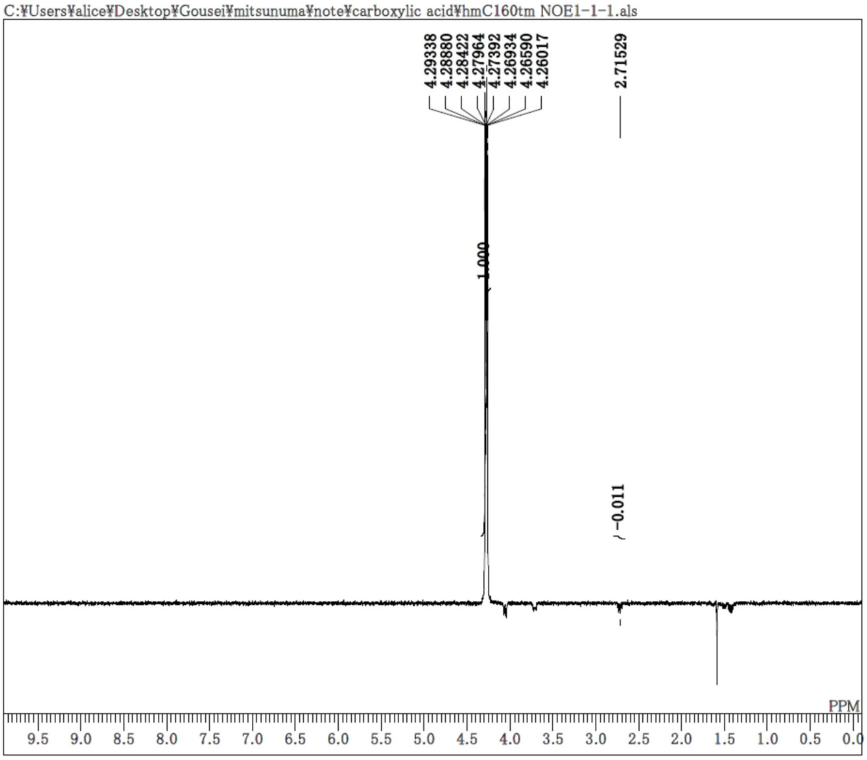
(3*S*,4*S*,5*S*)-**3a**





(3*S*,4*S*,5*R*)-**3a**

C:¥Users¥alice¥Desktop¥Gousei¥mitsunuma¥note¥carboxylic acid¥hmC160tm NOE1-1-1.als



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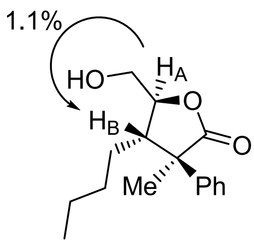
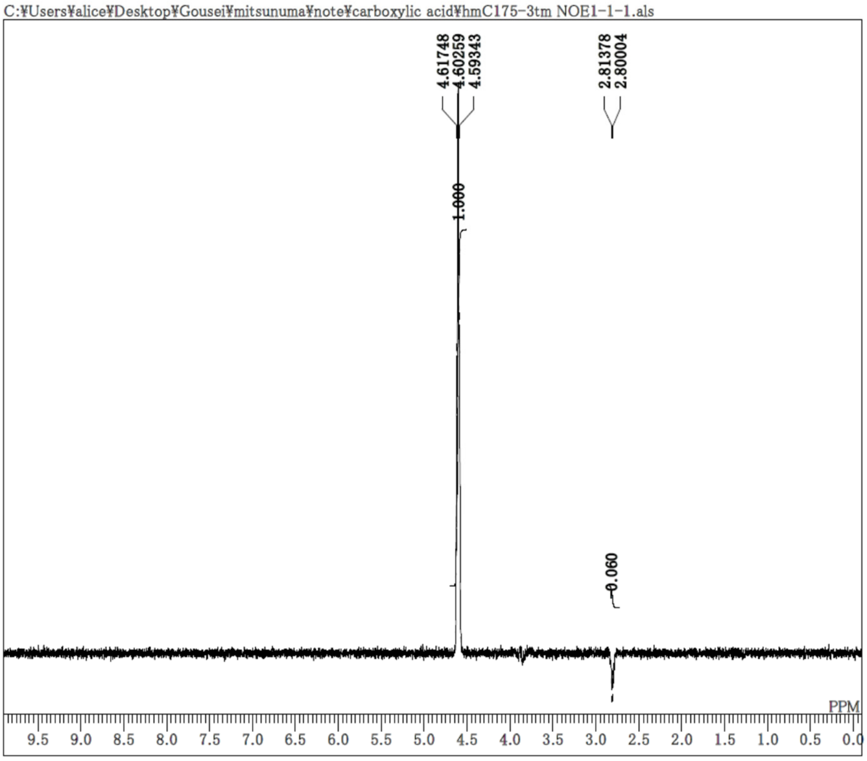
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 100
ACQTM 1.7459 sec
PD 7.0000 sec
PW1 11.10 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 38



(*3R*,*4S*,*5S*)-**3a**

C:¥Users¥alice¥Desktop¥Gousei¥mitsunuma¥note¥carboxylic acid¥hmC175-3tm NOE1-1-1.als



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OBFRQ 500.16 MHz
OBSET 2.41 KHz
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POINT 13107
FREQU 7507.51 Hz
SCANS 100
ACQTM 1.7459 sec
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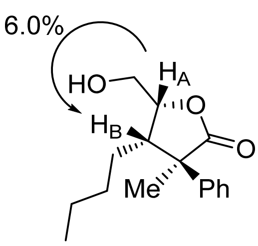
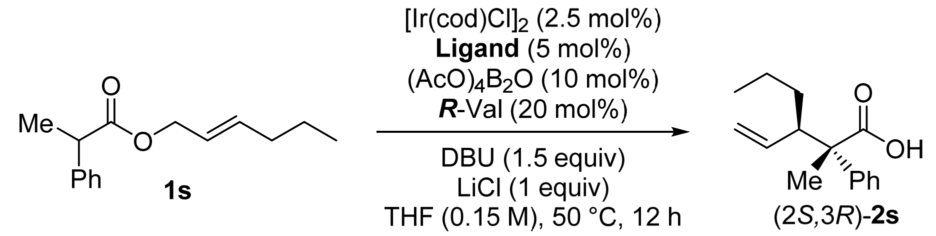
RGAIN 38

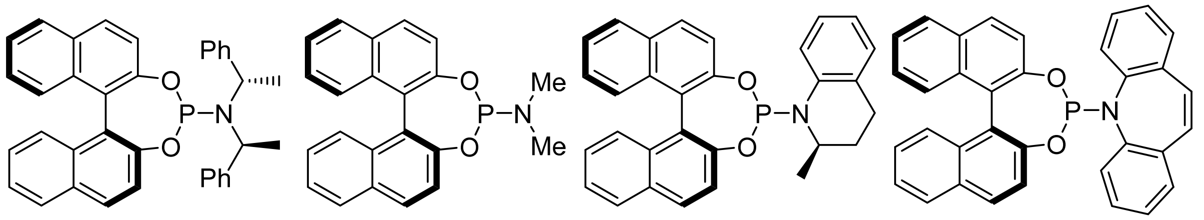


(*3R*,*4S*,*5R*)-**3a**

7. Initial Screening Results

Screening of phosphoramidite ligand





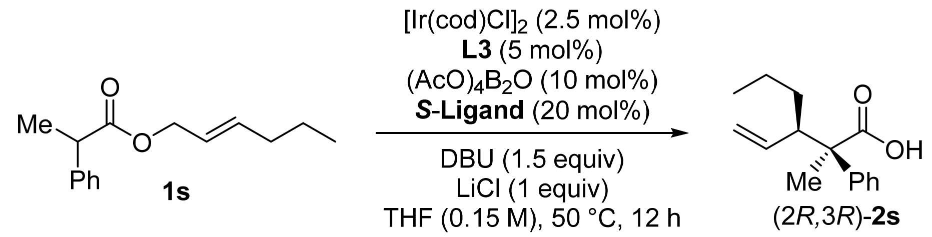
L1
54%, 22/1 dr, 2.2/1 b/l

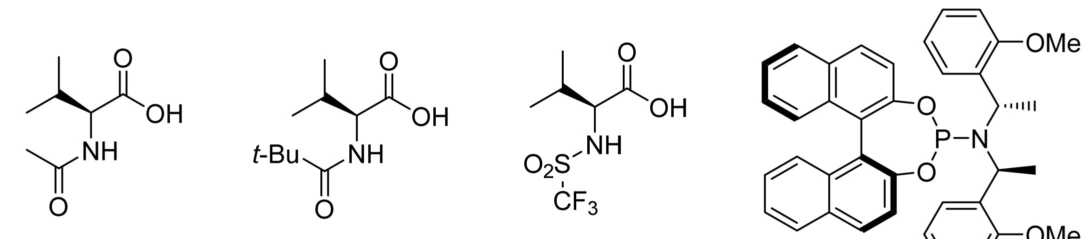
No reaction

No reaction

No reaction

Screening of boron ligand



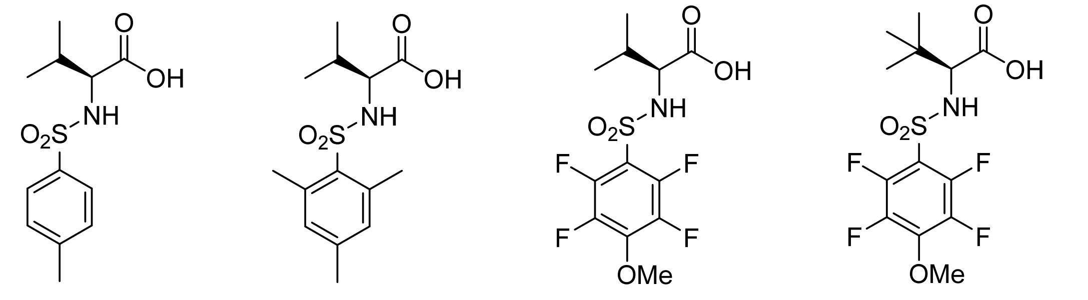


11%
5.5/1 dr, 3.1/1 b/l

14%
3.5/1 dr, 1.4/1 b/l

59%
2.3/1 dr, 1/2.3 b/l

L3



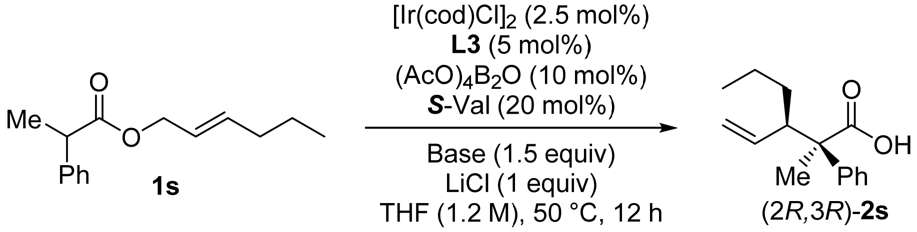
53%
2.3/1 dr, 1/3.5 b/l

71%
1/1.7 dr, 1/5.6 b/l

43%
11/1 dr, 2.6/1 b/l

24%
9.4/1 dr, 2.8/1 b/l

Screening of base

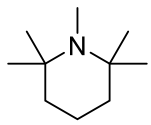


Et_3N

NR

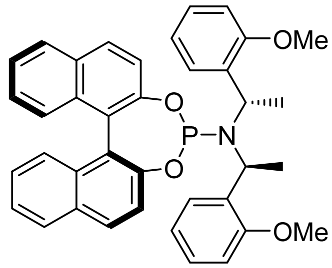
iPr_2EtN

NR

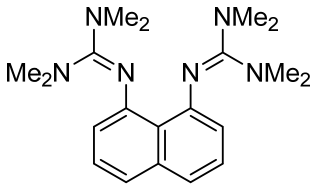


PMP

NR

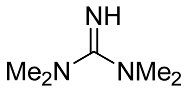


L3



TMGN

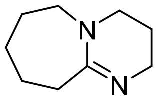
NR



TMG

22%

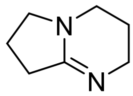
6.0/1 dr, 1.5/1 b/l



DBU

54%

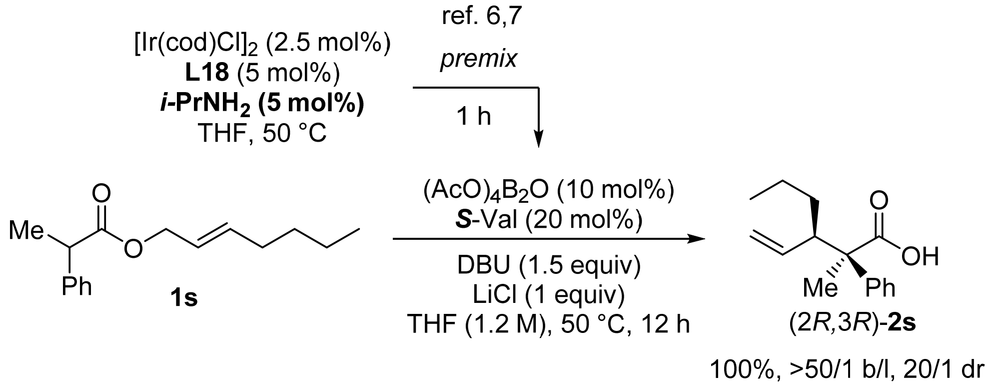
7.3/1 dr, 3.8/1 b/l



DBN

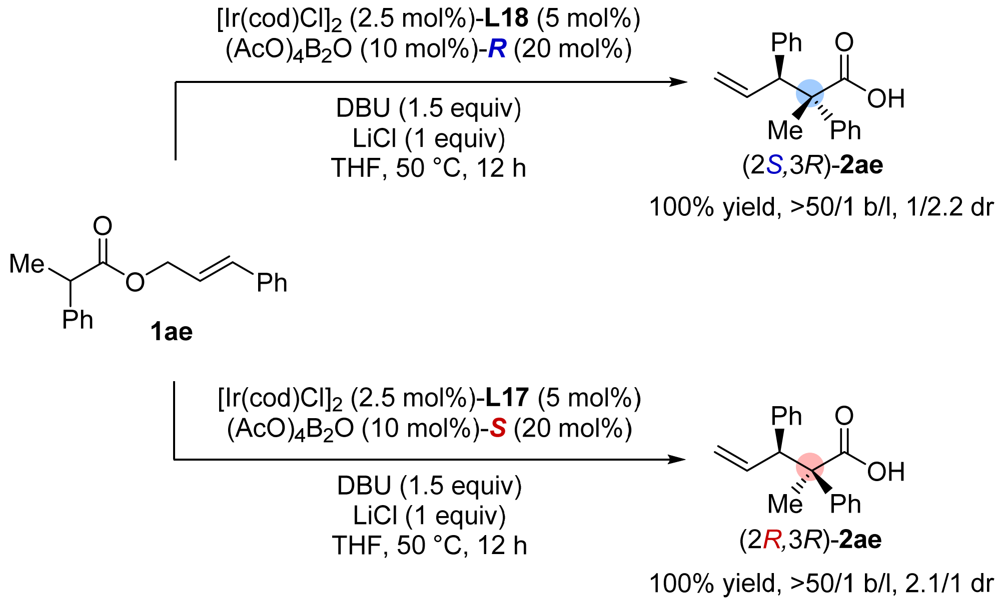
NR

Screening of activation method



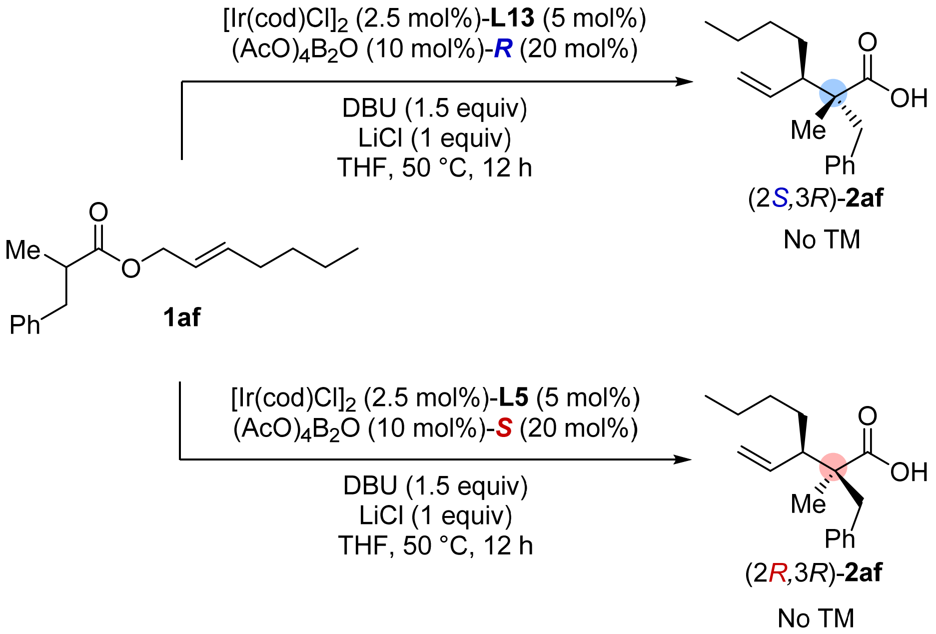
8. Limitations

<u>Application to cinnamyl 2-phenylpropanoate (**1ae**)</u>

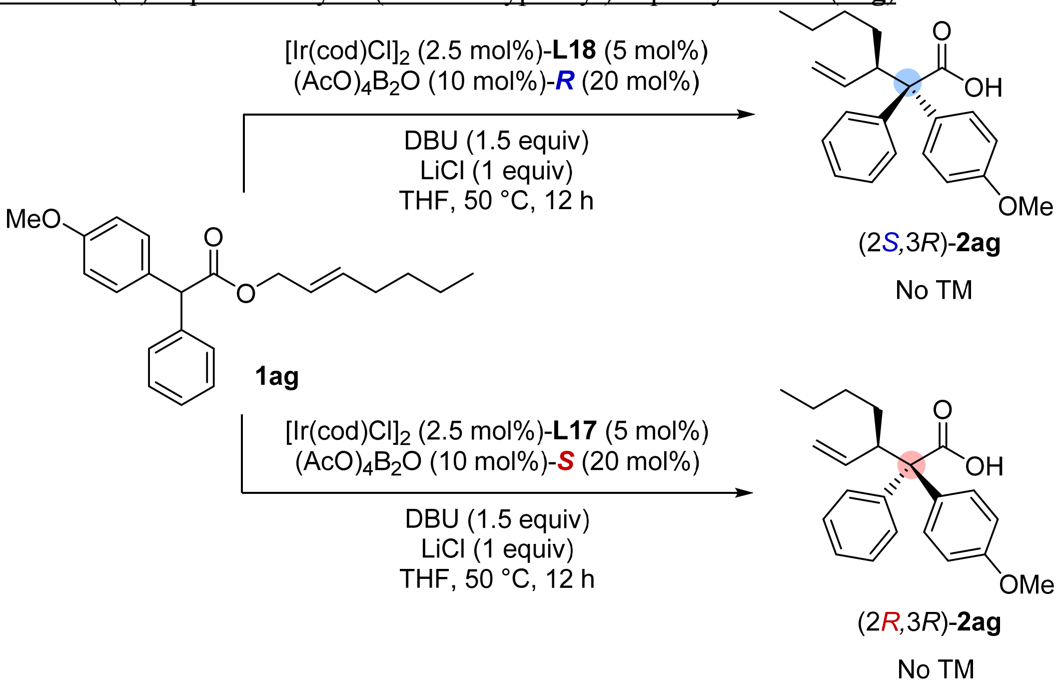


The current optimized condition could not be applied to the stereodivergent synthesis of β-aryl substituted carboxylic acid.

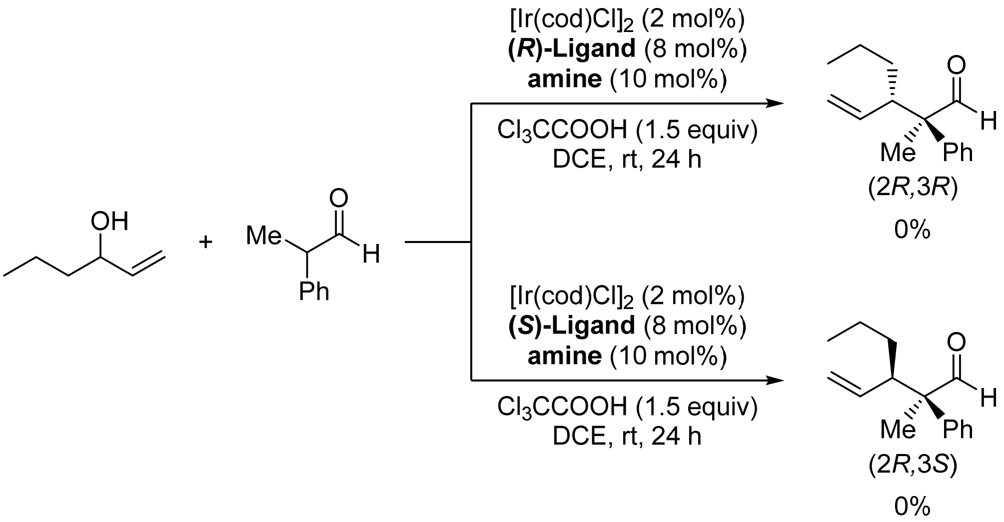
<u>Application to (*E*)-hept-2-en-1-yl 2-methyl-3-phenylpropanoate (**1af**)</u>

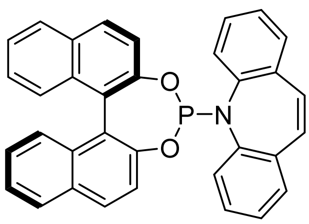


Application to (*E*)-hept-2-en-1-yl 2-(4-methoxyphenyl)-2-phenylacetate (**1ag**)

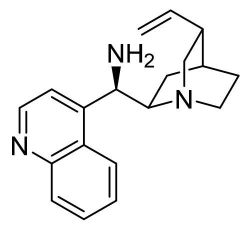


9. Control Experiment using Carreira's Reaction Conditions





(*R*)-Ligand

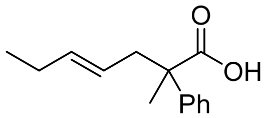


amine

10. Dataset

All the experiment was conducted twice under the same condition in S3. Yield, diastereomeric ratio (dr) and branch/linear (b/l) selectivity were determined by ^{1}H NMR analysis of crude mixture using 1,1,2,2-tetrachloroethane (10.0 μL, 0.0953 mmol) as an internal standard. Enantiomeric excess (ee) was determined by HPLC analysis. The ^{1}H NMR peaks of linear-**2Pr** were determined by reported spectra (Ref. 34 in main text). Linear-**2Et** was synthesized according to a related procedure (Ref. 34 in main text). All the crude NMR charts are listed in Chapter **11. NMR Charts**.
For Tables of the dataset, please see from the next page.

(*E*)-2-methyl-2-phenylhept-4-enoic acid



^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.91 (t, *J* = 7.4 Hz, 3H), 1.54 (s, 3H), 1.96 (dq, *J* = 7.0, 7.1 Hz, 2H), 2.59 (dd, *J* = 13.7, 7.4 Hz, 1H), 2.77 (dd, *J* = 13.7, 7.4 Hz, 1H), 5.24 (dt, *J* = 15.2, 7.1 Hz, 1H), 5.52 (dt, *J* = 15.2, 7.4 Hz, 1H), 7.25-7.39 (m, 5H).; IR (neat): 2962, 1700, 1277, 971, 697 cm^{-1}; HRMS (ESI): m/z calcd for $C_{14}H_{18}O_2$ $[M-H+2Na]^+$ 263.1018. Found 263.1012.

Table S1. Phosphoramidite ligands used in this study and experimental results. Ar = 2-MeOC$_6$H$_4$. Reaction conditions: [Ir(cod)Cl]$_2$ (2.5 mol% Ir), phosphoramidite ligand (5 mol%), (AcO)$_4$B$_2$O (10 mol%), ***S*** (20 mol%), DBU (1.5 equiv), LiCl (1.0 equiv), THF, 50 °C, 12 h. Each reaction was performed twice. Average values of selectivity data in the 1st and 2nd reactions were used for the MFA.

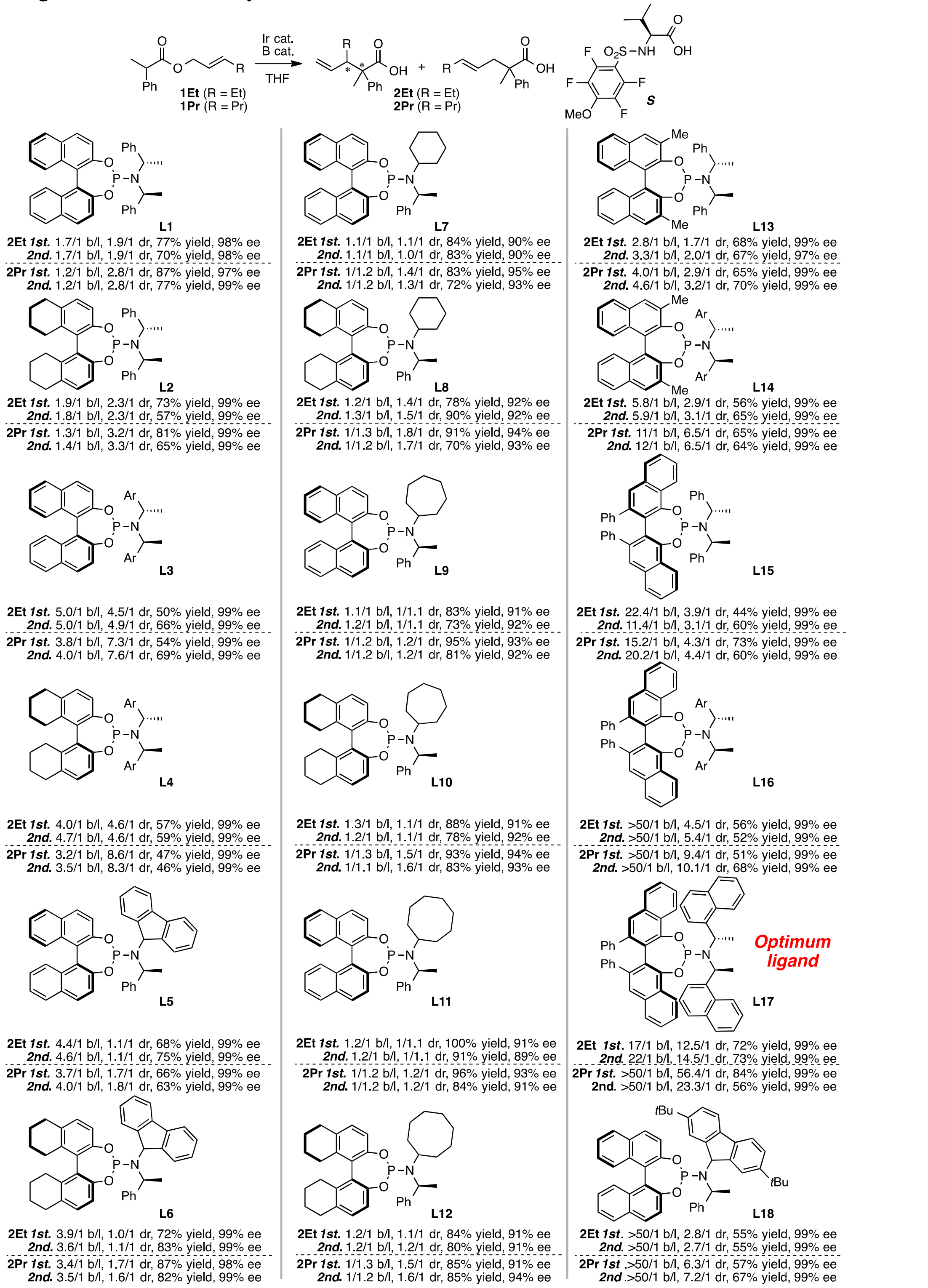
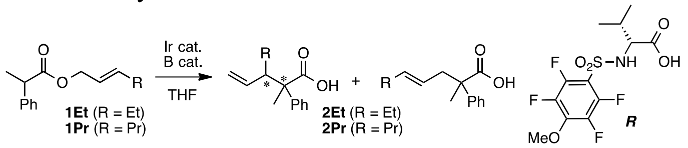
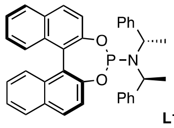


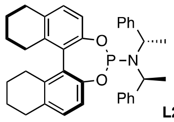
Table S2. Phosphoramidite ligands used in this study and experimental results. Ar = 2-$MeOC_6H_4$. Reaction conditions: $[Ir(cod)Cl]_2$ (2.5 mol% Ir), phosphoramidite ligand (5 mol%), $(AcO)_4B_2O$ (10 mol%), ***R*** (20 mol%), DBU (1.5 equiv), LiCl (1.0 equiv), THF, 50 °C, 12 h. Each reaction was performed twice. Average values of selectivity data in the 1st and 2nd reactions were used for the MFA.





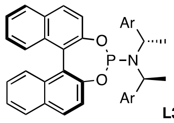
L1

2Et ***1st.*** 2.0/1 b/l, 5.2/1 dr, 94% yield, 97% ee
2nd. 2.1/1 b/l, 6.5/1 dr, 85% yield, 97% ee
2Pr ***1st.*** 2.4/1 b/l, 9.6/1 dr, 100% yield, 99% ee
2nd. 2.7/1 b/l, 10.0/1 dr, 77% yield, 97% ee



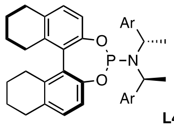
L2

2Et ***1st.*** 2.4/1 b/l, 6.1/1 dr, 97% yield, 98% ee
2nd. 2.5/1 b/l, 7.4/1 dr, 76% yield, 97% ee
2Pr ***1st.*** 2.6/1 b/l, 11.3/1 dr, 100% yield, 98% ee
2nd. 2.7/1 b/l, 14.1/1 dr, 94% yield, 99% ee



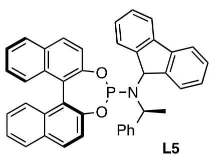
L3

2Et ***1st.*** 4.6/1 b/l, 6.7/1 dr, 70% yield, 99% ee
2nd. 5.3/1 b/l, 6.0/1 dr, 74% yield, 98% ee
2Pr ***1st.*** 4.5/1 b/l, 10.4/1 dr, 65% yield, 99% ee
2nd. 4.8/1 b/l, 9.8/1 dr, 85% yield, 98% ee



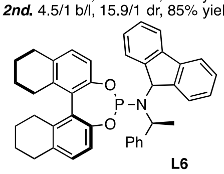
L4

2Et ***1st.*** 4.8/1 b/l, 6.5/1 dr, 81% yield, 98% ee
2nd. 5.4/1 b/l, 6.8/1 dr, 72% yield, 99% ee
2Pr ***1st.*** 4.0/1 b/l, 10.3/1 dr, 56% yield, 99% ee
2nd. 5.3/1 b/l, 10.5/1 dr, 84% yield, 99% ee



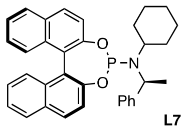
L5

2Et ***1st.*** 4.0/1 b/l, 11.0/1 dr, 83% yield, 98% ee
2nd. 4.2/1 b/l, 10.8/1 dr, 83% yield, 98% ee
2Pr ***1st.*** 4.2/1 b/l, 16.7/1 dr, 95% yield, 98% ee
2nd. 4.5/1 b/l, 15.9/1 dr, 85% yield, 97% ee



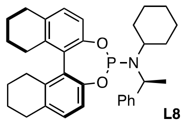
L6

2Et ***1st.*** 3.9/1 b/l, 10.6/1 dr, 100% yield, 98% ee
2nd. 3.4/1 b/l, 9.9/1 dr, 96% yield, 98% ee
2Pr ***1st.*** 3.8/1 b/l, 16.4/1 dr, 96% yield, 98% ee
2nd. 4.1/1 b/l, 17.7/1 dr, 98% yield, 97% ee



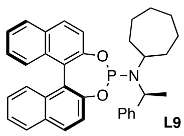
L7

2Et ***1st.*** 1/1.5 b/l, 3.2/1 dr, 87% yield, 92% ee
2nd. 1/1.6 b/l, 3.5/1 dr, 68% yield, 92% ee
2Pr ***1st.*** 1/1.5 b/l, 4.6/1 dr, 82% yield, 93% ee
2nd. 1/1.4 b/l, 5.1/1 dr, 66% yield, 93% ee



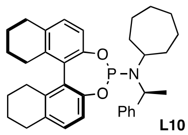
L8

2Et ***1st.*** 1.0/1 b/l, 3.6/1 dr, 87% yield, 95% ee
2nd. 1.1/1 b/l, 2.5/1 dr, 98% yield, 94% ee
2Pr ***1st.*** 1/1.1 b/l, 6.2/1 dr, 98% yield, 95% ee
2nd. 1/1.0 b/l, 7.5/1 dr, 86% yield, 95% ee



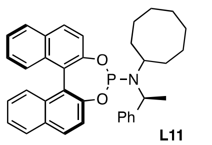
L9

2Et ***1st.*** 1/1.5 b/l, 2.7/1 dr, 83% yield, 93% ee
2nd. 1/1.6 b/l, 4.0/1 dr, 84% yield, 93% ee
2Pr ***1st.*** 1/1.5 b/l, 4.6/1 dr, 95% yield, 94% ee
2nd. 1/1.5 b/l, 5.9/1 dr, 85% yield, 93% ee



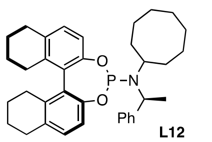
L10

2Et ***1st.*** 1/1.1 b/l, 3.8/1 dr, 88% yield, 95% ee
2nd. 1/1.1 b/l, 5.2/1 dr, 80% yield, 95% ee
2Pr ***1st.*** 1/1.2 b/l, 6.7/1 dr, 100% yield, 95% ee
2nd. 1/1.0 b/l, 6.4/1 dr, 86% yield, 95% ee



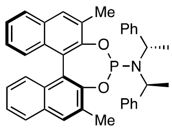
L11

2Et ***1st.*** 1/1.4 b/l, 2.7/1 dr, 86% yield, 93% ee
2nd. 1/1.6 b/l, 4.9/1 dr, 87% yield, 94% ee
2Pr ***1st.*** 1/1.6 b/l, 5.4/1 dr, 85% yield, 95% ee
2nd. 1/1.5 b/l, 6.2/1 dr, 84% yield, 94% ee



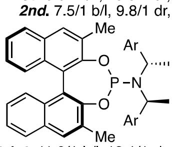
L12

2Et ***1st.*** 1/1.1 b/l, 3.8/1 dr, 88% yield, 95% ee
2nd. 1.0/1 b/l, 2.5/1 dr, 100% yield, 94% ee
2Pr ***1st.*** 1/1.2 b/l, 6.7/1 dr, 100% yield, 95% ee
2nd. 1/1.2 b/l, 9.1/1 dr, 95% yield, 95% ee



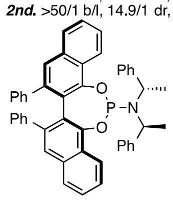
L13

2Et ***1st.*** 4.1/1 b/l, 9.9/1 dr, 89% yield, 98% ee
2nd. 4.7/1 b/l, 8.5/1 dr, 80% yield, 98% ee
2Pr ***1st.*** 9.9/1 b/l, 10.0/1 dr, 81% yield, 99% ee
2nd. 7.5/1 b/l, 9.8/1 dr, 64% yield, 99% ee



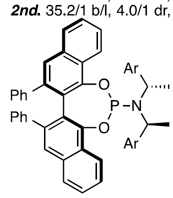
L14

2Et ***1st.*** 11.0/1 b/l, 13,1/1 dr, 77% yield, 99% ee
2nd.10.7/1 b/l, 10.6/1 dr, 93% yield, 99% ee
2Pr ***1st.*** >50/1 b/l, 15.2/1 dr, 80% yield, 99% ee
2nd. >50/1 b/l, 14.9/1 dr, 88% yield, 99% ee



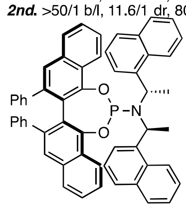
L15

2Et ***1st.*** 19.8/1 b/l, 3.0/1 dr, 85% yield, 99% ee
2nd. 22.8/1 b/l, 3.1/1 dr, 73% yield, 99% ee
2Pr ***1st.*** 38.5/1 b/l, 3.9/1 dr, 87% yield, 99% ee
2nd. 35.2/1 b/l, 4.0/1 dr, 84% yield, 99% ee



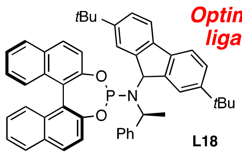
L16

2Et ***1st.*** >50/1 b/l, 8.2/1 dr, 69% yield, 99% ee
2nd. >50/1 b/l, 8.8/1 dr, 76% yield, 99% ee
2Pr ***1st.*** >50/1 b/l, 10.9/1 dr, 71% yield, 99% ee
2nd. >50/1 b/l, 11.6/1 dr, 80% yield, 99% ee



L17

2Et ***1st.*** >50/1 b/l, 1.5/1 dr, 77% yield, 99% ee
2nd. >50/1 b/l, 1.5/1 dr, 85% yield, 99% ee
2Pr ***1st.*** >50/1 b/l, 1.9/1 dr, 85% yield, 99% ee
2nd. >50/1 b/l, 1.9/1 dr, 98% yield, 99% ee



Optimum ligand

L18

2Et ***1st.*** >50/1 b/l, 19.8/1 dr, 91% yield, 99% ee
2nd. >50/1 b/l, 23.2/1 dr, 89% yield, 99% ee
2Pr ***1st.*** >50/1 b/l, 19.9/1 dr, 97% yield, 99% ee
2nd. >50/1 b/l, 23.1/1 dr, 100% yield, 99% ee

11. Details of the Molecular Field Analysis.

Scripts for the MFA are available at https://github.com/sh-yamaguchi/MFA. Brief summary about how to run the scripts are shown on page S95. Important structural information with all the 36 Ir-π-allyl intermediate structures (xyz files) obtained by running the scripts is found in "output" folder of SI. Under the output folder, three major folders "text", "test", "another" can be found. The "text" folder includes xyz files described in the text (Figure 2, figs S4-S6). The "test" folder includes xyz files described in the external validation section (fig S12). The "another" folder includes xyz files described in another design pathway (figs S14-S17). Two types of descriptors were employed for the MFA described in the text (For the details, see: Calculations of the molecular fields section below). Descriptor 1 was used for the MFA in the text and MFA using descriptor 2 and the same training samples with the MFA shown in the text was described in the SI (figs S23-S25). Thus, two folders "descriptor 1" and descriptor 2" can be found under the "text" folder. For example, xyz files generated by the MFA of the 24 constitutional selectivity data in the reaction using the ligand ***S*** shown in the text are found in output/text/descriptor1/S/24/bl. A few lines from the bottom of the xyz files correspond to the important structural information visualized. N/C atoms are the structural information corresponding to the positive coefficients that overlap(N)/do not overlap(C) with the intermediate structure. O/F atoms are the structural information corresponding to the negative coefficients that overlap(O)/do not overlap(F) with the intermediate structure.

DFT Calculations. All calculations were performed using Gaussian 16, Revision C.01[8]. We performed optimization of ground state geometries using the B3LYP[9–13] functional and the LANL2DZ[14] effective core potential for Ir, and 6-31G(d) for all other atoms. The MFA using the intermediate structures optimized using the B3LYP[9-13] functional augmented with Grimme's D3 empirical dispersion term[15] could not lead to design of the ligands **L14**, **L16** (fig. S40) and **L18** (fig. S42). One of the possible explanations is that some of weak non-covalent interactions in the Ir-π-allyl complexes do not exist in the corresponding transition state structures consisting of the Ir-π-allyl complexes and the boron enolate species or overestimation of the dispersion effects. Frequency calculations were performed to characterize the structures as minima (no imaginary frequency) on the potential surface, and to obtain thermal corrections to the Gibbs free energies. We employed the most stable conformers for the MFA unless otherwise noted (discussion about conformers, see fig. S13). To find the most stable conformers, conformational analysis was performed with the Merck molecular force field implemented in Spartan'18. Conformers within 4 kcal/mol were considered for further computations.

Calculations of the molecular fields. In the typical MFA, probe atoms are employed to calculate molecular (interaction) fields[16]. In this study, however, indicator fields were employed, which are one of the molecular fields for MFA[17] and often used for the MFA in asymmetric catalysis[18–21]. (Further discussion for the reason that we employed the indicator fields, see the text.) The protocol for calculations of indicator fields is as follows (fig. S1a). (I) A set of the Ir-π-allyl intermediates was optimized using the DFT method at the B3LYP/LANL2DZ (Ir) and 6-31G(d) level. As a reference structure, we used an X-ray crystal structure of well-established intermediates in the related allylic substitution reactions[22,23]. Superimposed structures of the reference X-ray structure and **L1/1Et** aligned based on the allyl group are shown in fig. S1b. (II) The coordinates of the set of the molecules obtained in step I were aligned. For alignment, first, we defined an xy plane based on the mean plane of the allyl group of **L1/1Et** as shown in fig. S1c. The central carbon atom of the allyl group was set as the origin. Then, alignment was performed through the least squares method by minimizing the distances between the allyl groups (the 7 atoms highlighted by red shown in fig. S1b) of **1L/1Et** and other intermediates. (III) The structures were placed in a grid space. The unit cell size is 1 Å per side. We used the molecular structures around the reaction center for calculations of the molecular fields instead of the use of the whole molecular structures to reduce dimensions of descriptors and avoid overfitting. The size of the grid space, which is centered at the origin, is $10 \times 12 \times 6$ Å^3. Each unit cell is regarded as an element of the descriptor vectors. The unit cells that included Bondi van der Waals radii of C (1.70 Å), H (1.20 Å), O (1.52 Å) atoms were counted as 1, or were otherwise counted as 0. Columns of all 0 and all 1 were removed to give the descriptor matrix (**descriptor 1**). We also employed the descriptor obtained by removal of the columns showing $|r| \leqq 0.3$

(**descriptor 2**: $|r|$ is an absolute value of a correlation coefficient with the target variables.). In the text, we show the results obtained by using descriptor 1. For the MFA using descriptor 2, the important structural information used for the design was also observed (figs S23-S25). In the models built by using descriptor 1, some regression coefficients for the descriptors showing $|r| \leqq 0.3$ took non-zero values. In some cases, however, the important structural information with $|r| \leqq 0.3$ was useful for the molecular design. For example, the important structural information visualized by the MFA from 20 b/l ratio and dr values in the reactions using ligand ***R*** (figs S14D and E) showed $r = 0.29$ for b/l ratios and $r = 0.23$ for dr. Thus, we recommend to perform MFA using both the descriptors and to compare both the results. All the regression coefficient values including standardized regression coefficients in the regression models used for the molecular design along with correlation coefficient r and coordinates of unit cell centers are summarized in parameters folder. In the folder, two excel files, text_test.xlsx and another.xlsx can be found. In the "text_test" folder, parameters such as regression coefficients of the MFA using descriptor1, descriptor2, and test samples for the molecular design are summarized. In the "another" folder, parameters such as regression coefficients of the MFA described in another design pathway (figs S14-S17) are summarized. Correlation coefficients between the descriptors are also summarized in the excel files.

LASSO and Elastic Net regression. Logarithms of constitutional selectivity (b/l) and diastereoselectivity (dr) were employed as target variables (Tables S3 and S4), which correspond to energy differences between the transition states that lead to each isomer ($\Delta\Delta G^{\ddagger} = -RT\log(\text{b/l or dr})$. R is the gas constant and T is the temperature of the reactions, 323.15 K. The indicator fields and $\Delta\Delta G^{\ddagger}$ values (kcal/mol) were correlated using LASSO (Least Absolute Shrinkage and Selection Operator)[24] or Elastic Net[25] to generate the regression models. The LASSO and Elastic Net regressions were performed according to the reported procedure[19] using the R package, glmnet[24]. By minimizing the

$$E(\boldsymbol{\beta}) = \sum_{i=1}^{n}(y_i - \mathbf{x}_i^T\boldsymbol{\beta})^2 + \lambda\sum_{j=1}^{p}\left\{(1-\alpha)\beta_j^2 + \alpha|\beta_j|\right\}$$

above loss function, we can estimate coefficients $\boldsymbol{\beta} = (\beta_1, \beta_2, \ldots, \beta_j, \ldots, \beta_p)^T$ while simultaneously assigning unimportant coefficients for reaction outcomes to be 0 (In this study, y_i is $\Delta\Delta G^{\ddagger}$. $\mathbf{x}_i = (x_{i1}, x_{i2}, \ldots, x_{ij}, \ldots, x_{ip})^T$ is the indicator field. n and p denote the number of samples and descriptors, respectively.). If α is 1, this method corresponds to LASSO regression[24]. If α is $0 < \alpha < 1$, this method corresponds to Elastic Net regression[25]. In all cases in this study, we selected values of the hyper parameter λ that minimized the mean squared error calculated from predicted values obtained from leave-one-out cross-validation by using glmnet[19,26]. Among descriptors that are correlated, LASSO will select one. In contrast, Elastic Net can extract multiple correlated descriptors[25]. We performed Elastic Net regression if structural information that led to molecular design could not be found through LASSO regression. For Elastic Net, we varied the parameter α from 0.1 to 0.9 in steps of 0.1, and for each choice of α we selected the parameter λ according to the procedure described above using glmnet. Among the models in which the important structural information that led to molecular design was included, we employed the model showing the highest q^2 (coefficient of determination calculated from predicted values of leave-one-out cross-validation [LOOCV]). Coefficients of determination calculated from the resulting regression models (R^2) and q^2 are shown in fig. S2 (24 reactions) and fig. S3 (32 reactions) along with plots of the measured and predicted values. The numbers of all descriptors and extracted descriptors are also shown in figs S2 and S3. The measured and predicted values are summarized in Table S3 and S4. We also performed 4-fold CV and y-randomization. The analysis was repeated 100 times for 4-fold CV and 50 times for y-randomization. The average values of the coefficients of determination are shown in fig. S2 and S3 (Q^2 for 4-fold CV and R^2_{yradom} for y-randomization). In all cases, Q^2 showed good values over 0.6, indicating the models are robust. Low values of R^2_{yrandom} close to 0 indicate the probability of chance correlation is low.

Molecular design. We describe the workflow for the molecular design in this study.
(I) MFA using the sets of selectivity data and the intermediate structures is performed and the quality of the resulting regression models is checked. In this study, we have successfully designed the molecules based on structural information visualized by the MFA. All the regression models showed R^2, q^2, $Q^2 > 0.6$ and $R^2_{\text{y-random}} < 0.2$. Thus, we tentatively employ these metrics (R^2, q^2, $Q^2 > 0.6$ and $R^2_{\text{y-random}} < 0.2$) to determine whether the models are used for the design. (*In the QSAR [Quantitative Structure-Activity Relationships]/QSPR [Quantitative Structure-Property Relationships] fields, various discussions about metrics to evaluate regression models have been reported[27,28]. However, data-driven design of molecules showing improved selectivity in asymmetric catalysis through the visualized information by MFA is still rare[17]. Thus, further discussion about metrics to evaluate the regression models in this method should be needed after sufficient accumulation of related examples.)
(II) Extracted structural information is visualized on the intermediate structures and all the intermediate structures in the training samples and visualized structural information are thoroughly compared. By considering synthetic accessibility, substituents are introduced to the intermediate structures to overlap light blue points as shown in the text. For the design, we basically employ the structural information that fulfills $|r| > 0.3$ and shows the same sign with correlation coefficient r (i.e. the structural information corresponding to positive regression coefficients should show a positive value of correlation coefficient r with the target variables). If predicted $\Delta\Delta G^{\ddagger}$ values of the designed molecules are higher than those of the template molecules, the designed ligands are synthesized and the reactions using the ligands are examined.
(III) If the selectivity values are not satisfactory, MFA using all the sets of target variables including those in the reactions with the designed ligands is again performed. This workflow is repeated until designed molecules show high selectivity.
The important structural information with intermediate structures used for the design is shown in Fig. 2, and figs. S4–S6. Structural information from coefficients with absolute values of <0.01 was not visualized (We discuss threshold values in figs. S7, S27c, and S28c.). In figs S4–S6, we also show the origins of the structural information. As shown in fig. S12, we confirmed that the regression model validated by a test set included the structural information corresponding to the information used for the molecular design.

Interpretation of the important structural information. We can obtain insights into the diastereoinduction mechanisms from the visualized structural information as shown in figs. S19 and S20.

Python and R scripts for MFA.

Scripts for the molecular field analysis are available at https://github.com/sh-yamaguchi/MFA. Here, we describe a brief summary about how to run the scripts. To run the scripts, Mac OS (the environment in which we checked the scripts: Mac OS 10.14), python 3.7, numpy, R, glmnet are required.

<u>Calculations of the descriptors</u>

The descriptors (molecular fields) are calculated as follows; Download the folder “data” from SI to a directory. To the same directory, put the lib folder downloaded from the above github site. Then, run the python script shown below.

```
#!/usr/bin/env python
# -*- coding: utf-8 -*-

import numpy as np
import lib.pymfa as pymfa

setting = np.loadtxt("data/setting.txt",str)
mean_plane = int(setting[0,1]);  origin = int(setting[1,1]); x_axis = int(setting[2,1]); n = list(setting[3,1]); n = [int(i)
for i in n]
grid_x = int(setting[4,1]); grid_y = int(setting[5,1]); grid_z = int(setting[6,1])
threshold = float(setting[7,1])

ex = pymfa.MolecularFieldAnalysis(mean_plane, origin, x_axis, n, grid_x, grid_y, grid_z, threshold)
mol_name = np.loadtxt("data/mol_name_36.txt",str)

#text/descriptor1
descriptor24 = ex.indicator_field(mol_name[range(24)])
descriptor32 = ex.indicator_field(mol_name[range(32)])
np.savetxt("output/text/descriptor1/descriptor24.txt",descriptor24[1])
np.savetxt("output/text/descriptor1/descriptor32.txt",descriptor32[1])

#text/descriptor2
R36bl = np.loadtxt("data/target_variables/R36bl.txt",usecols = [1,])
R24bl = R36bl[range(24)]
R32bl = R36bl[range(32)]
R36dr = np.loadtxt("data/target_variables/R36dr.txt",usecols = [1,])
R24dr = R36dr[range(24)]
R32dr = R36dr[range(32)]
S36bl = np.loadtxt("data/target_variables/S36bl.txt",usecols = [1,])
S24bl = S36bl[range(24)]
S32bl = S36bl[range(32)]
S36dr = np.loadtxt("data/target_variables/S36dr.txt",usecols = [1,])
S24dr = S36dr[range(24)]
S32dr = S36dr[range(32)]
S36ee = np.loadtxt("data/target_variables/S36ee.txt",usecols = [1,])
S24ee = S36ee[range(24)]
S32ee = S36ee[range(32)]
R36ee = np.loadtxt("data/target_variables/R36ee.txt",usecols = [1,])
```

```
R24ee = R36ee[range(24)]
R32ee = R36ee[range(32)]

descriptorS24bl = ex.indicator_field_corr(mol_name[range(24)],S24bl)
descriptorS32bl = ex.indicator_field_corr(mol_name[range(32)],S32bl)
descriptorS24dr = ex.indicator_field_corr(mol_name[range(24)],S24dr)
descriptorS32dr = ex.indicator_field_corr(mol_name[range(32)],S32dr)
descriptorR24bl = ex.indicator_field_corr(mol_name[range(24)],R24bl)
descriptorR32bl = ex.indicator_field_corr(mol_name[range(32)],R32bl)
descriptorR24dr = ex.indicator_field_corr(mol_name[range(24)],R24dr)
descriptorR32dr = ex.indicator_field_corr(mol_name[range(32)],R32dr)
descriptorR24ee = ex.indicator_field_corr(mol_name[range(24)],R24ee)
descriptorR32ee = ex.indicator_field_corr(mol_name[range(32)],R32ee)
descriptorS24ee = ex.indicator_field_corr(mol_name[range(24)],S24ee)
descriptorS32ee = ex.indicator_field_corr(mol_name[range(32)],S32ee)

np.savetxt("output/text/descriptor2/descriptorS24bl.txt",descriptorS24bl[1])
np.savetxt("output/text/descriptor2/descriptorS32bl.txt",descriptorS32bl[1])
np.savetxt("output/text/descriptor2/descriptorS24dr.txt",descriptorS24dr[1])
np.savetxt("output/text/descriptor2/descriptorS32dr.txt",descriptorS32dr[1])
np.savetxt("output/text/descriptor2/descriptorR24bl.txt",descriptorR24bl[1])
np.savetxt("output/text/descriptor2/descriptorR32bl.txt",descriptorR32bl[1])
np.savetxt("output/text/descriptor2/descriptorR24dr.txt",descriptorR24dr[1])
np.savetxt("output/text/descriptor2/descriptorR32dr.txt",descriptorR32dr[1])
np.savetxt("output/text/descriptor2/descriptorS24ee.txt",descriptorS24ee[1])
np.savetxt("output/text/descriptor2/descriptorS32ee.txt",descriptorS32ee[1])
np.savetxt("output/text/descriptor2/descriptorR24ee.txt",descriptorR24ee[1])
np.savetxt("output/text/descriptor2/descriptorR32ee.txt",descriptorR32ee[1])

#test
mol_name_training24 = np.loadtxt("data/mol_name_24_training.txt",str)
mol_name_training32 = np.loadtxt("data/mol_name_32_training.txt",str)

descriptor_training24 = ex.indicator_field(mol_name_training24)
descriptor_training32 = ex.indicator_field(mol_name_training32)
np.savetxt("output/test/descriptor_training24.txt",descriptor_training24[1])
np.savetxt("output/test/descriptor_training32.txt",descriptor_training32[1])

#another design path
mol_name = np.loadtxt("data/mol_name_28.txt",str)

descriptor20 = ex.indicator_field(mol_name[range(20)])
descriptor22 = ex.indicator_field(mol_name[range(22)])
descriptor26 = ex.indicator_field(mol_name[range(26)])
np.savetxt("output/another/descriptor20.txt",descriptor20[1])
np.savetxt("output/another/descriptor22.txt",descriptor22[1])
np.savetxt("output/another/descriptor26.txt",descriptor26[1])
```

Regression analysis

The R script for LASSO and Elastic Net regression using the descriptors and the calculated $\Delta\Delta G^{\ddagger}$ values is shown below. The text files of the regression coefficients can be obtained by running the R script. All the coefficient values along with 3-dimensional coordinates of unit cell centers etc. are summarized as excel files which can be found in "parameters" folder (SI)

```
#text/descriptor1
S24dr <- read.table("data/target_variables/S36dr.txt",row.names=1,nrows=24)
S24bl <- read.table("data/target_variables/S36bl.txt",row.names=1,nrows=24)
S24ee <- read.table("data/target_variables/S36ee.txt",row.names=1,nrows=24)
R24dr <- read.table("data/target_variables/R36dr.txt",row.names=1,nrows=24)
R24bl <- read.table("data/target_variables/R36bl.txt",row.names=1,nrows=24)
R24ee <- read.table("data/target_variables/R36ee.txt",row.names=1,nrows=24)

S32dr <- read.table("data/target_variables/S36dr.txt",row.names=1,nrows=32)
S32bl <- read.table("data/target_variables/S36bl.txt",row.names=1,nrows=32)
S32ee <- read.table("data/target_variables/S36ee.txt",row.names=1,nrows=32)
R32dr <- read.table("data/target_variables/R36dr.txt",row.names=1,nrows=32)
R32bl <- read.table("data/target_variables/R36bl.txt",row.names=1,nrows=32)
R32ee <- read.table("data/target_variables/R36ee.txt",row.names=1,nrows=32)

S24dr <- as.matrix(S24dr$V2)
S24bl <- as.matrix(S24bl$V2)
S24ee <- as.matrix(S24ee$V2)
R24dr <- as.matrix(R24dr$V2)
R24bl <- as.matrix(R24bl$V2)
R24ee <- as.matrix(R24ee$V2)

S32dr <- as.matrix(S32dr$V2)
S32bl <- as.matrix(S32bl$V2)
S32ee <- as.matrix(S32ee$V2)
R32dr <- as.matrix(R32dr$V2)
R32bl <- as.matrix(R32bl$V2)
R32ee <- as.matrix(R32ee$V2)

descriptor24 <- read.table("output/text/descriptor1/descriptor24.txt", header=TRUE)
descriptor32 <- read.table("output/text/descriptor1/descriptor32.txt", header=TRUE)
library(glmnet)
lasso_S24dr <- cv.glmnet(as.matrix(descriptor24), as.matrix(S24dr), nfold=24, alpha=1.0, grouped=FALSE)
lasso_S24bl <- cv.glmnet(as.matrix(descriptor24), as.matrix(S24bl), nfold=24, alpha=1.0, grouped=FALSE)
lasso_S24ee <- cv.glmnet(as.matrix(descriptor24), as.matrix(S24ee), nfold=24, alpha=1.0, grouped=FALSE)
lasso_R24dr <- cv.glmnet(as.matrix(descriptor24), as.matrix(R24dr), nfold=24, alpha=1.0, grouped=FALSE)
lasso_R24bl <- cv.glmnet(as.matrix(descriptor24), as.matrix(R24bl), nfold=24, alpha=1.0, grouped=FALSE)
lasso_R24ee <- cv.glmnet(as.matrix(descriptor24), as.matrix(R24ee), nfold=24, alpha=1.0, grouped=FALSE)

lasso_S32dr <- cv.glmnet(as.matrix(descriptor32), as.matrix(S32dr), nfold=32, alpha=1.0, grouped=FALSE)
lasso_S32bl <- cv.glmnet(as.matrix(descriptor32), as.matrix(S32bl), nfold=32, alpha=1.0, grouped=FALSE)
lasso_S32ee <- cv.glmnet(as.matrix(descriptor32), as.matrix(S32ee), nfold=32, alpha=1.0, grouped=FALSE)
```

```
lasso_R32dr <- cv.glmnet(as.matrix(descriptor32), as.matrix(R32dr), nfold=32, alpha=1.0, grouped=FALSE)
lasso_R32bl <- cv.glmnet(as.matrix(descriptor32), as.matrix(R32bl), nfold=32, alpha=1.0, grouped=FALSE)
lasso_R32ee <- cv.glmnet(as.matrix(descriptor32), as.matrix(R32ee), nfold=32, alpha=1.0, grouped=FALSE)

s_S24dr = lasso_S24dr$lambda.min
s_S24bl = lasso_S24bl$lambda.min
s_S24ee = lasso_S24ee$lambda.min
s_R24dr = lasso_R24dr$lambda.min
s_R24bl = lasso_R24bl$lambda.min
s_R24ee = lasso_R24ee$lambda.min

s_S32dr = lasso_S32dr$lambda.min
s_S32bl = lasso_S32bl$lambda.min
s_S32ee = lasso_S32ee$lambda.min
s_R32dr = lasso_R32dr$lambda.min
s_R32bl = lasso_R32bl$lambda.min
s_R32ee = lasso_R32ee$lambda.min

coefficient_S24dr = coef(lasso_S24dr, s_S24dr)
coefficient_S24bl = coef(lasso_S24bl, s_S24bl)
coefficient_S24ee = coef(lasso_S24ee, s_S24ee)
coefficient_R24dr = coef(lasso_R24dr, s_R24dr)
coefficient_R24bl = coef(lasso_R24bl, s_R24bl)
coefficient_R24ee = coef(lasso_R24ee, s_R24ee)

coefficient_S32dr = coef(lasso_S32dr, s_S32dr)
coefficient_S32bl = coef(lasso_S32bl, s_S32bl)
coefficient_S32ee = coef(lasso_S32ee, s_S32ee)
coefficient_R32dr = coef(lasso_R32dr, s_R32dr)
coefficient_R32bl = coef(lasso_R32bl, s_R32bl)
coefficient_R32ee = coef(lasso_R32ee, s_R32ee)

write.table(as.matrix(coefficient_S24dr), file="output/text/descriptor1/coefficient_S24dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S24bl), file="output/text/descriptor1/coefficient_S24bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S24ee), file="output/text/descriptor1/coefficient_S24ee.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R24dr), file="output/text/descriptor1/coefficient_R24dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R24bl), file="output/text/descriptor1/coefficient_R24bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R24ee), file="output/text/descriptor1/coefficient_R24ee.txt",
col.name=FALSE,quote=FALSE)

write.table(as.matrix(coefficient_S32dr), file="output/text/descriptor1/coefficient_S32dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S32bl), file="output/text/descriptor1/coefficient_S32bl.txt", col.name=FALSE,
quote=FALSE)
```

```
write.table(as.matrix(coefficient_S32ee), file="output/text/descriptor1/coefficient_S32ee.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32dr), file="output/text/descriptor1/coefficient_R32dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32bl), file="output/text/descriptor1/coefficient_R32bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32ee), file="output/text/descriptor1/coefficient_R32ee.txt",
col.name=FALSE,quote=FALSE)


#text/descriptor2
descriptorS24bl <- read.table("output/text/descriptor2/descriptorS24bl.txt", header=TRUE)
descriptorS32bl <- read.table("output/text/descriptor2/descriptorS32bl.txt", header=TRUE)
descriptorS24dr <- read.table("output/text/descriptor2/descriptorS24dr.txt", header=TRUE)
descriptorS32dr <- read.table("output/text/descriptor2/descriptorS32dr.txt", header=TRUE)
descriptorR24bl <- read.table("output/text/descriptor2/descriptorR24bl.txt", header=TRUE)
descriptorR32bl <- read.table("output/text/descriptor2/descriptorR32bl.txt", header=TRUE)
descriptorR24dr <- read.table("output/text/descriptor2/descriptorR24dr.txt", header=TRUE)
descriptorR32dr <- read.table("output/text/descriptor2/descriptorR32dr.txt", header=TRUE)
descriptorS24ee <- read.table("output/text/descriptor2/descriptorS24ee.txt", header=TRUE)
descriptorS32ee <- read.table("output/text/descriptor2/descriptorS32ee.txt", header=TRUE)
descriptorR24ee <- read.table("output/text/descriptor2/descriptorR24ee.txt", header=TRUE)
descriptorR32ee <- read.table("output/text/descriptor2/descriptorR32ee.txt", header=TRUE)

lasso_S24dr <- cv.glmnet(as.matrix(descriptorS24dr), as.matrix(S24dr), nfold=24, alpha=1.0, grouped=FALSE)
lasso_S24bl <- cv.glmnet(as.matrix(descriptorS24bl), as.matrix(S24bl), nfold=24, alpha=0.5, grouped=FALSE)
lasso_S24ee <- cv.glmnet(as.matrix(descriptorS24ee), as.matrix(S24ee), nfold=24, alpha=1.0, grouped=FALSE)
lasso_R24dr <- cv.glmnet(as.matrix(descriptorR24dr), as.matrix(R24dr), nfold=24, alpha=1.0, grouped=FALSE)
lasso_R24bl <- cv.glmnet(as.matrix(descriptorR24bl), as.matrix(R24bl), nfold=24, alpha=1.0, grouped=FALSE)
lasso_R24ee <- cv.glmnet(as.matrix(descriptorR24ee), as.matrix(R24ee), nfold=24, alpha=1.0, grouped=FALSE)

lasso_S32dr <- cv.glmnet(as.matrix(descriptorS32dr), as.matrix(S32dr), nfold=32, alpha=1.0, grouped=FALSE)
lasso_S32bl <- cv.glmnet(as.matrix(descriptorS32bl), as.matrix(S32bl), nfold=32, alpha=1.0, grouped=FALSE)
lasso_S32ee <- cv.glmnet(as.matrix(descriptorS32ee), as.matrix(S32ee), nfold=32, alpha=1.0, grouped=FALSE)
lasso_R32dr <- cv.glmnet(as.matrix(descriptorR32dr), as.matrix(R32dr), nfold=32, alpha=1.0, grouped=FALSE)
lasso_R32bl <- cv.glmnet(as.matrix(descriptorR32bl), as.matrix(R32bl), nfold=32, alpha=1.0, grouped=FALSE)
lasso_R32ee <- cv.glmnet(as.matrix(descriptorR32ee), as.matrix(R32ee), nfold=32, alpha=1.0, grouped=FALSE)

s_S24dr = lasso_S24dr$lambda.min
s_S24bl = lasso_S24bl$lambda.min
s_S24ee = lasso_S24ee$lambda.min
s_R24dr = lasso_R24dr$lambda.min
s_R24bl = lasso_R24bl$lambda.min
s_R24ee = lasso_R24ee$lambda.min

s_S32dr = lasso_S32dr$lambda.min
s_S32bl = lasso_S32bl$lambda.min
s_S32ee = lasso_S32ee$lambda.min
s_R32dr = lasso_R32dr$lambda.min
```

```
s_R32bl = lasso_R32bl$lambda.min
s_R32ee = lasso_R32ee$lambda.min

coefficient_S24dr = coef(lasso_S24dr, s_S24dr)
coefficient_S24bl = coef(lasso_S24bl, s_S24bl)
coefficient_S24ee = coef(lasso_S24ee, s_S24ee)
coefficient_R24dr = coef(lasso_R24dr, s_R24dr)
coefficient_R24bl = coef(lasso_R24bl, s_R24bl)
coefficient_R24ee = coef(lasso_R24ee, s_R24ee)

coefficient_S32dr = coef(lasso_S32dr, s_S32dr)
coefficient_S32bl = coef(lasso_S32bl, s_S32bl)
coefficient_S32ee = coef(lasso_S32ee, s_S32ee)
coefficient_R32dr = coef(lasso_R32dr, s_R32dr)
coefficient_R32bl = coef(lasso_R32bl, s_R32bl)
coefficient_R32ee = coef(lasso_R32ee, s_R32ee)

write.table(as.matrix(coefficient_S24dr), file="output/text/descriptor2/coefficient_S24dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S24bl), file="output/text/descriptor2/coefficient_S24bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S24ee), file="output/text/descriptor2/coefficient_S24ee.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R24dr), file="output/text/descriptor2/coefficient_R24dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R24bl), file="output/text/descriptor2/coefficient_R24bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R24ee), file="output/text/descriptor2/coefficient_R24ee.txt", col.name=FALSE,
quote=FALSE)

write.table(as.matrix(coefficient_S32dr), file="output/text/descriptor2/coefficient_S32dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S32bl), file="output/text/descriptor2/coefficient_S32bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S32ee), file="output/text/descriptor2/coefficient_S32ee.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32dr), file="output/text/descriptor2/coefficient_R32dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32bl), file="output/text/descriptor2/coefficient_R32bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32ee), file="output/text/descriptor2/coefficient_R32ee.txt", col.name=FALSE,
quote=FALSE)

#another design path
R20dr <- read.table("data/target_variables/R28dr.txt",row.names=1,nrows=20)
R20bl <- read.table("data/target_variables/R28bl.txt",row.names=1,nrows=20)

S22bl <- read.table("data/target_variables/S28bl.txt",row.names=1,nrows=22)
```

```
S26dr <- read.table("data/target_variables/S28dr.txt",row.names=1,nrows=26)
S26bl <- read.table("data/target_variables/S28bl.txt",row.names=1,nrows=26)

R20dr <- as.matrix(R20dr$V2)
R20bl <- as.matrix(R20bl$V2)
S22bl <- as.matrix(S22bl$V2)
S26dr <- as.matrix(S26dr$V2)
S26bl <- as.matrix(S26bl$V2)

descriptor20 <- read.table("output/another/descriptor20.txt", header=TRUE)
descriptor22 <- read.table("output/another/descriptor22.txt", header=TRUE)
descriptor26 <- read.table("output/another/descriptor26.txt", header=TRUE)

lasso_R20dr <- cv.glmnet(as.matrix(descriptor20), as.matrix(R20dr), nfold=20, alpha=1.0, grouped=FALSE)
lasso_R20bl <- cv.glmnet(as.matrix(descriptor20), as.matrix(R20bl), nfold=20, alpha=1.0, grouped=FALSE)
lasso_S22bl <- cv.glmnet(as.matrix(descriptor22), as.matrix(S22bl), nfold=22, alpha=1.0, grouped=FALSE)
lasso_S26dr <- cv.glmnet(as.matrix(descriptor26), as.matrix(S26dr), nfold=26, alpha=0.4, grouped=FALSE)
lasso_S26bl <- cv.glmnet(as.matrix(descriptor26), as.matrix(S26bl), nfold=26, alpha=1.0, grouped=FALSE)

s_R20dr = lasso_R20dr$lambda.min
s_R20bl = lasso_R20bl$lambda.min
s_S22bl = lasso_S22bl$lambda.min
s_S26dr = lasso_S26dr$lambda.min
s_S26bl = lasso_S26bl$lambda.min

coefficient_R20dr = coef(lasso_R20dr, s_R20dr)
coefficient_R20bl = coef(lasso_R20bl, s_R20bl)
coefficient_S22bl = coef(lasso_S22bl, s_S22bl)
coefficient_S26dr = coef(lasso_S26dr, s_S26dr)
coefficient_S26bl = coef(lasso_S26bl, s_S26bl)

write.table(as.matrix(coefficient_R20dr), file="output/another/coefficient_R20dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R20bl), file="output/another/coefficient_R20bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S22bl), file="output/another/coefficient_S22bl.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S26dr), file="output/another/coefficient_S26dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_S26bl), file="output/another/coefficient_S26bl.txt", col.name=FALSE,
quote=FALSE)


#test
S24bl_training <- read.table("data/target_variables/S24bl_training.txt",row.names=1)

S32dr_training <- read.table("data/target_variables/S32dr_training.txt",row.names=1)
S32bl_training <- read.table("data/target_variables/S32bl_training.txt",row.names=1)
R32dr_training <- read.table("data/target_variables/R32dr_training.txt",row.names=1)
```

```
R32bl_training <- read.table("data/target_variables/R32bl_training.txt",row.names=1)

S24bl_training <- as.matrix(S24bl_training$V2)
S32dr_training <- as.matrix(S32dr_training$V2)
S32bl_training <- as.matrix(S32bl_training$V2)
R32dr_training <- as.matrix(R32dr_training$V2)
R32bl_training <- as.matrix(R32bl_training$V2)

descriptor24_training <- read.table("output/test/descriptor_training24.txt", header=TRUE)
descriptor32_training <- read.table("output/test/descriptor_training32.txt", header=TRUE)

lasso_S24bl <- cv.glmnet(as.matrix(descriptor24_training), as.matrix(S24bl_training), nfold=24, alpha=0.5,
grouped=FALSE)
lasso_S32dr <- cv.glmnet(as.matrix(descriptor32_training), as.matrix(S32dr_training), nfold=32, alpha=1.0,
grouped=FALSE)
lasso_S32bl <- cv.glmnet(as.matrix(descriptor32_training), as.matrix(S32bl_training), nfold=32, alpha=1.0,
grouped=FALSE)
lasso_R32dr <- cv.glmnet(as.matrix(descriptor32_training), as.matrix(R32dr_training), nfold=32, alpha=1.0,
grouped=FALSE)
lasso_R32bl <- cv.glmnet(as.matrix(descriptor32_training), as.matrix(R32bl_training), nfold=32, alpha=1.0,
grouped=FALSE)

s_S24bl = lasso_S24bl$lambda.min
s_S32dr = lasso_S32dr$lambda.min
s_S32bl = lasso_S32bl$lambda.min
s_R32dr = lasso_R32dr$lambda.min
s_R32bl = lasso_R32bl$lambda.min

coefficient_S24bl = coef(lasso_S24bl, s_S24bl)
coefficient_S32dr = coef(lasso_S32dr, s_S32dr)
coefficient_S32bl = coef(lasso_S32bl, s_S32bl)
coefficient_R32dr = coef(lasso_R32dr, s_R32dr)
coefficient_R32bl = coef(lasso_R32bl, s_R32bl)

write.table(as.matrix(coefficient_S24bl), file="output/test/coefficient_S24bl.txt", col.name=FALSE, quote=FALSE)
write.table(as.matrix(coefficient_S32dr), file="output/test/coefficient_S32dr.txt", col.name=FALSE, quote=FALSE)
write.table(as.matrix(coefficient_S32bl), file="output/test/coefficient_S32bl.txt", col.name=FALSE, quote=FALSE)
write.table(as.matrix(coefficient_R32dr), file="output/test/coefficient_R32dr.txt", col.name=FALSE,
quote=FALSE)
write.table(as.matrix(coefficient_R32bl), file="output/test/coefficient_R32bl.txt", col.name=FALSE,
quote=FALSE)
```

Visualization

The python script for visualization of the important structural information is shown below. The xyz files that include the extracted structural information can be obtained by running the script. The resulting xyz files can be found in the "output" folder (SI). A few lines from the bottom of the xyz files correspond to the important structural information visualized. N/C atoms are the structural information corresponding to the positive coefficients that overlap(N)/do not overlap(C) with the intermediate structure. O/F atoms are the structural information corresponding to the negative coefficients that overlap(O)/do not overlap(F) with the intermediate structure. Before running the script, download the “output” folder from SI to the directory where the “data” folder was downloaded and remove all the xyz files in the “output” folder.

```
#!/usr/bin/env python
# -*- coding: utf-8 -*-

import numpy as np
import lib.pymfa as pymfa

setting = np.loadtxt("data/setting.txt",str)
mean_plane = int(setting[0,1]);  origin = int(setting[1,1]); x_axis = int(setting[2,1]); n = list(setting[3,1]); n = [int(i)
for i in n]
grid_x = int(setting[4,1]); grid_y = int(setting[5,1]); grid_z = int(setting[6,1])
threshold = float(setting[7,1])

ex = pymfa.MolecularFieldAnalysis(mean_plane, origin, x_axis, n, grid_x, grid_y, grid_z, threshold)
mol_name = np.loadtxt("data/mol_name_36.txt",str)

#descriptor1
descriptor24 = ex.indicator_field(mol_name[range(24)])
descriptor32 = ex.indicator_field(mol_name[range(32)])

directory24_1 = "output/text/descriptor1/S/24/dr/"; directory24_2 = "output/text/descriptor1/S/24/bl/";
directory24_3 = "output/text/descriptor1/R/24/dr/"; directory24_4 = "output/text/descriptor1/R/24/bl/";directory24_5
= "output/text/descriptor1/S/24/ee/";directory24_6 = "output/text/descriptor1/R/24/ee/"
directory32_1 = "output/text/descriptor1/S/32/dr/"; directory32_2 = "output/text/descriptor1/S/32/bl/";
directory32_3 = "output/text/descriptor1/R/32/dr/"; directory32_4 = "output/text/descriptor1/R/32/bl/";directory32_5
= "output/text/descriptor1/S/32/ee/";directory32_6 = "output/text/descriptor1/R/32/ee/"

for i in range(len(mol_name)):
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_S24dr.txt",mol
_name,i,descriptor24[0]),directory24_1)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_S24bl.txt",mol
_name,i,descriptor24[0]),directory24_2)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_R24dr.txt",mol
_name,i,descriptor24[0]),directory24_3)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_R24bl.txt",mol
_name,i,descriptor24[0]),directory24_4)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_S24ee.txt",mol
_name,i,descriptor24[0]),directory24_5)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_R24ee.txt",mol
_name,i,descriptor24[0]),directory24_6)

        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_S32dr.txt",mol
_name,i,descriptor32[0]),directory32_1)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_S32bl.txt",mol
_name,i,descriptor32[0]),directory32_2)
```

```
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_R32dr.txt",mol
_name,i,descriptor32[0]),directory32_3)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_R32bl.txt",mol
_name,i,descriptor32[0]),directory32_4)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_S32ee.txt",mol
_name,i,descriptor32[0]),directory32_5)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/text/descriptor1/coefficient_R32ee.txt",mol
_name,i,descriptor32[0]),directory32_6)

#descriptor2

R36bl = np.loadtxt("data/target_variables/R36bl.txt",usecols = [1,])
R24bl = R36bl[range(24)]
R32bl = R36bl[range(32)]
R36dr = np.loadtxt("data/target_variables/R36dr.txt",usecols = [1,])
R24dr = R36dr[range(24)]
R32dr = R36dr[range(32)]
S36bl = np.loadtxt("data/target_variables/S36bl.txt",usecols = [1,])
S24bl = S36bl[range(24)]
S32bl = S36bl[range(32)]
S36dr = np.loadtxt("data/target_variables/S36dr.txt",usecols = [1,])
S24dr = S36dr[range(24)]
S32dr = S36dr[range(32)]
S36ee = np.loadtxt("data/target_variables/S36ee.txt",usecols = [1,])
S24ee = S36ee[range(24)]
S32ee = S36ee[range(32)]
R36ee = np.loadtxt("data/target_variables/R36ee.txt",usecols = [1,])
R24ee = R36ee[range(24)]
R32ee = R36ee[range(32)]

directory24_1 = "output/text/descriptor2/S/24/dr/"; directory24_2 = "output/text/descriptor2/S/24/bl/";
directory24_3 = "output/text/descriptor2/R/24/dr/"; directory24_4 = "output/text/descriptor2/R/24/bl/";directory24_5
= "output/text/descriptor2/S/24/ee/";directory24_6 = "output/text/descriptor2/R/24/ee/"
directory32_1 = "output/text/descriptor2/S/32/dr/"; directory32_2 = "output/text/descriptor2/S/32/bl/";
directory32_3 = "output/text/descriptor2/R/32/dr/"; directory32_4 = "output/text/descriptor2/R/32/bl/";directory32_5
= "output/text/descriptor2/S/32/ee/";directory32_6 = "output/text/descriptor2/R/32/ee/"

for i in range(len(mol_name)):
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_S24dr.txt
",mol_name,i,descriptor24[0],S24dr),directory24_1)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_S24bl.txt
",mol_name,i,descriptor24[0],S24bl),directory24_2)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_R24dr.txt
",mol_name,i,descriptor24[0],R24dr),directory24_3)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_R24bl.txt
",mol_name,i,descriptor24[0],R24bl),directory24_4)
        ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_S24ee.txt
",mol_name,i,descriptor24[0],S24ee),directory24_5)
```

```
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_R24ee.txt
",mol_name,i,descriptor24[0],R24ee),directory24_6)

	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_S32dr.txt
",mol_name,i,descriptor32[0],S32dr),directory32_1)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_S32bl.txt
",mol_name,i,descriptor32[0],S32bl),directory32_2)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_R32dr.txt
",mol_name,i,descriptor32[0],R32dr),directory32_3)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_R32bl.txt
",mol_name,i,descriptor32[0],R32bl),directory32_4)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_S32ee.txt
",mol_name,i,descriptor32[0],S32ee),directory32_5)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info_corr("output/text/descriptor2/coefficient_R32ee.txt
",mol_name,i,descriptor32[0],R32ee),directory32_6)

#another
mol_name = np.loadtxt("data/mol_name_28.txt",str)

descriptor20 = ex.indicator_field(mol_name[range(20)])
descriptor22 = ex.indicator_field(mol_name[range(22)])
descriptor26 = ex.indicator_field(mol_name[range(26)])

directory26_1 = "output/another/S/26/dr/"; directory26_2 = "output/another/S/26/bl/";directory22_1 =
"output/another/S/22/bl/"; directory20_1 = "output/another/R/20/dr/"; directory20_2 = "output/another/R/20/bl/"

for i in range(len(mol_name)):
	ex.xyz_file("data/mol_name_28.txt",i,ex.structure_info("output/another/coefficient_S26dr.txt",mol_name,i,
descriptor26[0]),directory26_1)
	ex.xyz_file("data/mol_name_28.txt",i,ex.structure_info("output/another/coefficient_S26bl.txt",mol_name,i,
descriptor26[0]),directory26_2)
	ex.xyz_file("data/mol_name_28.txt",i,ex.structure_info("output/another/coefficient_S22bl.txt",mol_name,i,
descriptor22[0]),directory22_1)
	ex.xyz_file("data/mol_name_28.txt",i,ex.structure_info("output/another/coefficient_R20dr.txt",mol_name,i
,descriptor20[0]),directory20_1)
	ex.xyz_file("data/mol_name_28.txt",i,ex.structure_info("output/another/coefficient_R20bl.txt",mol_name,i
,descriptor20[0]),directory20_2)

#test
mol_name = np.loadtxt("data/mol_name_36.txt",str)
mol_name_training24 = np.loadtxt("data/mol_name_24_training.txt",str)
mol_name_training32 = np.loadtxt("data/mol_name_32_training.txt",str)

descriptor_training24 = ex.indicator_field(mol_name_training24)
descriptor_training32 = ex.indicator_field(mol_name_training32)

directory24_2 = "output/test/S/24/bl/"
directory32_1 = "output/test/S/32/dr/"; directory32_2 = "output/test/S/32/bl/"; directory32_3 =
"output/test/R/32/dr/"; directory32_4 = "output/test/R/32/bl/"
```

```
for i in range(len(mol_name)):
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/test/coefficient_S24bl.txt",mol_name,i,descriptor_training24[0]),directory24_2)

	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/test/coefficient_S32dr.txt",mol_name,i,descriptor_training32[0]),directory32_1)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/test/coefficient_S32bl.txt",mol_name,i,descriptor_training32[0]),directory32_2)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/test/coefficient_R32dr.txt",mol_name,i,descriptor_training32[0]),directory32_3)
	ex.xyz_file("data/mol_name_36.txt",i,ex.structure_info("output/test/coefficient_R32bl.txt",mol_name,i,descriptor_training32[0]),directory32_4)
```

Calculations of the molecular fields.

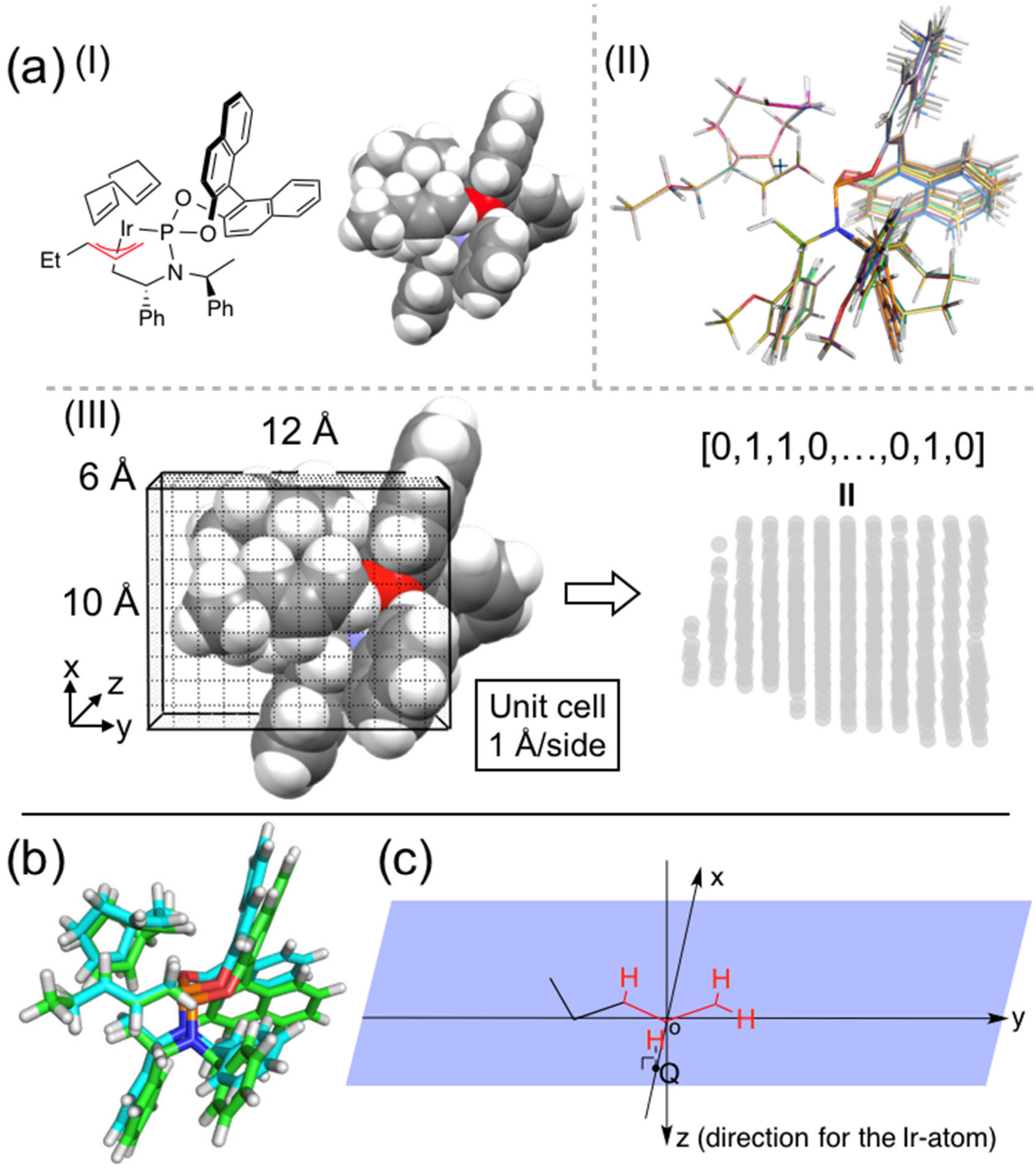


Fig. S1. (a) Protocol for molecular field calculations. (I) Optimization of the intermediate structures. (II) Alignment of the intermediate structures (Superposed intermediate structures are shown.). (III) Conversion of 3D-molecular structures to 0,1 vectors. More details are described on page S92 (Calculations of the molecular fields), (b) Superimposed structures of **L1/1Et** (color of carbon atoms: green) and the X-ray crystal structure from Ref 21 (**L1/1Me** : the substituents of allyl group is Me group instead of Et group in **L1/1Et**. color of carbon atoms: light blue). The structures were aligned based on the allyl group highlighted by red in fig. S1a. (c) Definition of the xy plane.

For the alignment of the molecular structures, the xy plane was defined as follows. A mean plane of 7 atoms that compose the π-allyl moiety of **1L/1Et** was defined as the xy plane (fig. S1c). The central π-allyl carbon atom was set as an origin. A hydrogen atom on the central π-allyl carbon atom was used to define an x-axis. A point Q shown in fig. S1b is a foot of a perpendicular drawn from the hydrogen atom to the xy plane. A straight line passing through the origin and point Q was defined as the x axis.

Plots of the measured vs predicted values

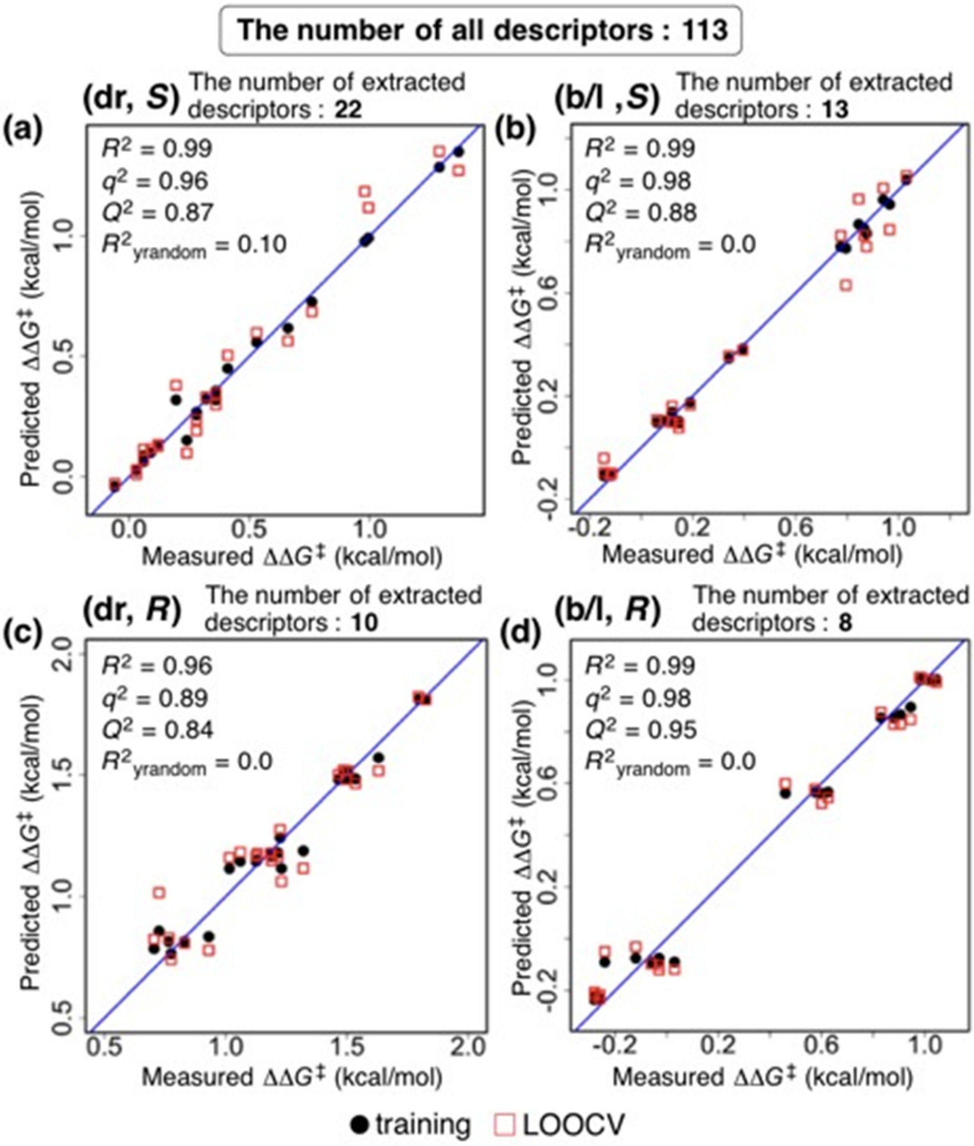


Fig. S2. Results of the MFA using 24 training samples. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

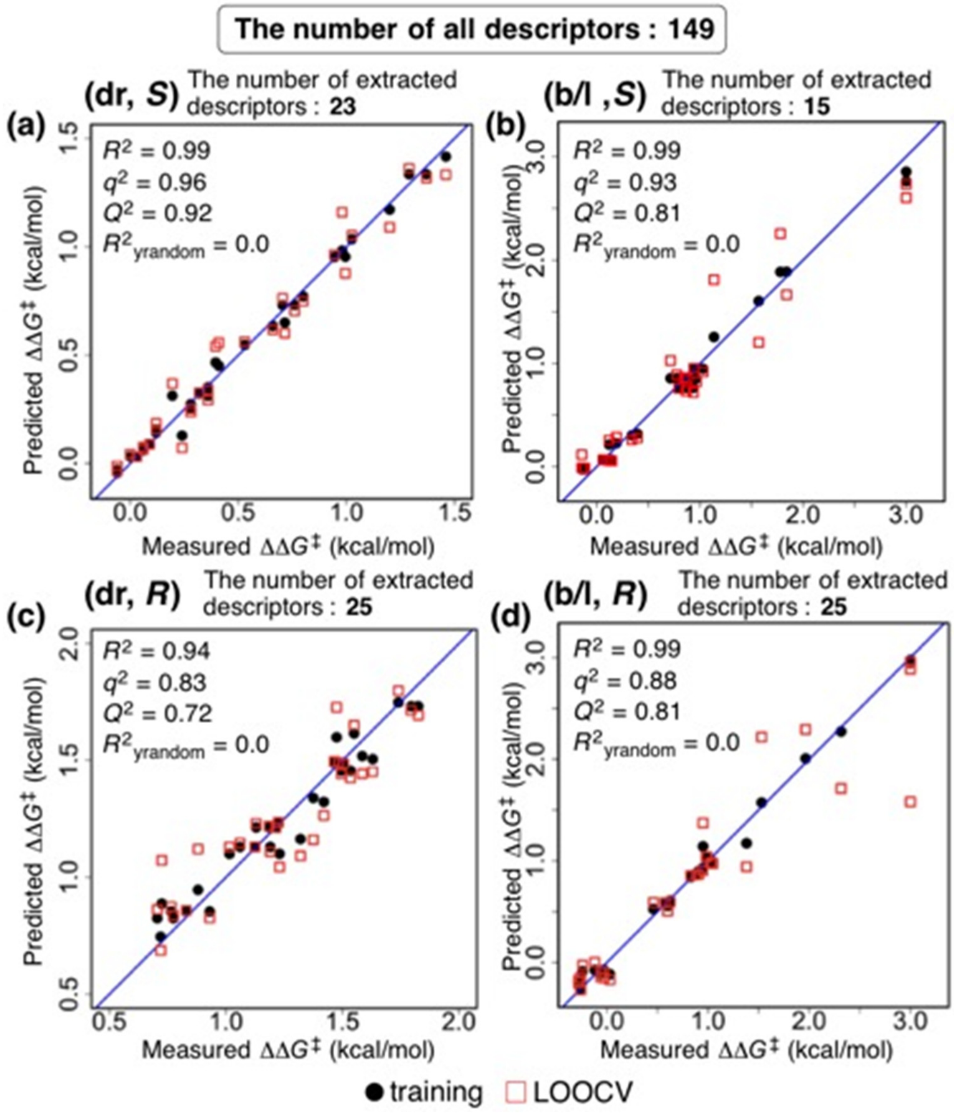


Fig. S3. Results of the MFA using 32 training samples. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

Table S3. Data of the reactions using valine derived ligand *S* and predicted values.
Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** is the optimum ligand for the reactions using valine derived ligand ***S***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (b/l)			24 reactions (dr)			32 reactions (b/l)			32 reactions (dr)		
	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)
L1/1Et	0.34	0.346	0.355	0.41	0.449	0.504	0.34	0.304	0.260	0.41	0.451	0.557
L1/1Pr	0.12	0.138	0.160	0.66	0.617	0.564	0.12	0.212	0.257	0.66	0.634	0.615
L2/1Et	0.395	0.384	0.378	0.53	0.558	0.598	0.395	0.317	0.277	0.53	0.547	0.561
L2/1Pr	0.19	0.175	0.164	0.76	0.726	0.685	0.19	0.226	0.287	0.76	0.731	0.701
L3/1Et	1.03	1.039	1.056	0.995	0.991	1.118	1.03	0.948	0.917	0.995	0.956	0.878
L3/1Pr	0.875	0.831	0.779	1.29	1.286	1.352	0.875	0.857	0.849	1.29	1.335	1.362
L4/1Et	0.94	0.962	1.007	0.98	0.977	1.185	0.94	0.948	0.953	0.98	0.984	1.161
L4/1Pr	0.775	0.779	0.822	1.37	1.350	1.272	0.775	0.857	0.890	1.37	1.335	1.318
L5/1Et	0.965	0.944	0.845	0.06	0.062	0.070	0.965	0.855	0.821	0.06	0.065	0.077
L5/1Pr	0.865	0.851	0.824	0.36	0.356	0.348	0.865	0.764	0.733	0.36	0.351	0.338
L6/1Et	0.845	0.867	0.965	0.03	0.029	0.028	0.845	0.855	0.863	0.03	0.040	0.043
L6/1Pr	0.795	0.774	0.631	0.32	0.323	0.332	0.795	0.764	0.758	0.32	0.326	0.339
L7/1Et	0.06	0.098	0.108	0.03	0.023	0.010	0.06	0.067	0.067	0.03	0.040	0.043
L7/1Pr	-0.12	-0.111	-0.108	0.195	0.319	0.379	-0.12	-0.025	-0.014	0.195	0.315	0.370
L8/1Et	0.145	0.098	0.076	0.24	0.151	0.098	0.145	0.067	0.055	0.24	0.131	0.073
L8/1Pr	-0.145	-0.111	-0.097	0.36	0.319	0.298	-0.145	-0.025	-0.011	0.36	0.315	0.295
L9/1Et	0.09	0.102	0.105	-0.06	-0.040	-0.029	0.09	0.067	0.063	-0.06	-0.024	-0.006
L9/1Pr	-0.12	-0.106	-0.103	0.12	0.127	0.134	-0.12	-0.025	-0.014	0.12	0.160	0.190
L10/1Et	0.145	0.103	0.093	0.06	0.088	0.114	0.145	0.067	0.055	0.06	0.067	0.071
L10/1Pr	-0.115	-0.106	-0.099	0.28	0.256	0.238	-0.115	-0.025	-0.015	0.28	0.251	0.232
L11/1Et	0.12	0.102	0.098	-0.06	-0.043	-0.028	0.12	0.067	0.059	-0.06	-0.042	-0.034
L11/1Pr	-0.12	-0.106	-0.103	0.12	0.125	0.126	-0.12	-0.025	-0.014	0.12	0.142	0.156
L12/1Et	0.12	0.102	0.098	0.09	0.100	0.115	0.12	0.067	0.059	0.09	0.091	0.092
L12/1Pr	-0.145	-0.106	-0.041	0.28	0.268	0.191	-0.145	-0.025	0.115	0.28	0.275	0.251
L13/1Et	0.715	1.060		0.395	0.277		0.715	0.855	1.029	0.395	0.466	0.541
L13/1Pr	0.935	0.851		0.715	0.444		0.935	0.764	0.718	0.715	0.649	0.599
L14/1Et	1.135	1.123		0.705	0.818		1.135	1.256	1.811	0.705	0.729	0.764
L14/1Pr	1.57	0.914		1.2	1.112		1.57	1.603	1.203	1.2	1.172	1.087
L15/1Et	1.78	1.060		0.8	0.320		1.78	1.886	2.256	0.8	0.773	0.750
L15/1Pr	1.84	0.851		0.945	0.488		1.84	1.887	1.665	0.945	0.950	0.964
L16/1Et	3	1.123		1.025	0.862		3	2.854	2.734	1.025	1.037	1.054
L16/1Pr	3	0.914		1.46	1.156		3	2.763	2.603	1.46	1.416	1.338
L17/1Et	1.82			1.62			1.82	2.014		1.62	0.773	
L17/1Pr	3			2.59			3	1.923		2.59	0.957	
L18/1Et	3			0.66			3	1.695		0.66	0.366	
L18/1Pr	3			1.18			3	1.603		1.18	0.549	

Table S4. Data of the reactions using valine derived ligand *R* and predicted values.

Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol) used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L18** is the optimum ligand for the reactions using valine derived ligand ***R***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (b/l)			**24 reactions (dr)**			**32 reactions (b/l)**			**32 reactions (dr)**		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Et	0.46	0.561	0.599	1.13	1.151	1.176	0.46	0.526	0.589	1.13	1.213	1.230
L1/1Pr	0.6	0.561	0.522	1.465	1.481	1.501	0.6	0.557	0.505	1.465	1.489	1.495
L2/1Et	0.575	0.566	0.577	1.225	1.242	1.275	0.575	0.576	0.587	1.225	1.229	1.236
L2/1Pr	0.625	0.566	0.544	1.63	1.572	1.517	0.625	0.607	0.594	1.63	1.506	1.452
L3/1Et	1.025	1.003	0.999	1.185	1.179	1.177	1.025	0.993	0.981	1.185	1.213	1.218
L3/1Pr	0.99	1.003	1.009	1.485	1.509	1.520	0.99	1.024	1.038	1.485	1.489	1.490
L4/1Et	1.045	1.003	0.992	1.215	1.179	1.164	1.045	0.993	0.973	1.215	1.213	1.212
L4/1Pr	0.98	1.003	1.011	1.505	1.509	1.518	0.98	1.024	1.042	1.505	1.489	1.486
L5/1Et	0.905	0.867	0.830	1.535	1.484	1.465	0.905	0.895	0.866	1.535	1.456	1.425
L5/1Pr	0.945	0.896	0.848	1.795	1.814	1.824	**0.945**	**0.926**	0.905	**1.795**	**1.732**	1.715
L6/1Et	0.83	0.855	0.877	1.495	1.484	1.482	0.83	0.842	0.854	1.495	1.456	1.442
L6/1Pr	0.88	0.855	0.830	1.825	1.814	1.812	0.88	0.873	0.862	1.825	1.732	1.694
L7/1Et	-0.28	-0.226	-0.207	0.775	0.765	0.741	-0.28	-0.223	-0.178	0.775	0.825	0.838
L7/1Pr	-0.24	-0.090	-0.049	1.015	1.115	1.160	-0.24	-0.085	-0.026	1.015	1.101	1.130
L8/1Et	0.03	-0.090	-0.118	0.705	0.785	0.824	0.03	-0.116	-0.170	0.705	0.825	0.862
L8/1Pr	-0.03	-0.090	-0.103	1.23	1.115	1.061	-0.03	-0.085	-0.100	1.23	1.101	1.044
L9/1Et	-0.28	-0.226	-0.207	0.765	0.814	0.829	-0.28	-0.223	-0.195	0.765	0.854	0.874
L9/1Pr	-0.26	-0.226	-0.216	1.06	1.145	1.182	-0.26	-0.192	-0.149	1.06	1.130	1.147
L10/1Et	-0.06	-0.090	-0.096	0.93	0.835	0.779	-0.06	-0.116	-0.144	0.93	0.854	0.826
L10/1Pr	-0.06	-0.090	-0.096	1.19	1.165	1.148	-0.06	-0.085	-0.092	1.19	1.130	1.109
L11/1Et	-0.26	-0.236	-0.232	0.83	0.814	0.808	-0.26	-0.255	-0.270	0.83	0.854	0.858
L11/1Pr	-0.28	-0.236	-0.220	1.125	1.145	1.165	-0.28	-0.224	-0.182	1.125	1.130	1.130
L12/1Et	-0.03	-0.075	-0.120	0.725	0.858	1.015	-0.03	-0.073	-0.151	0.725	0.888	1.072
L12/1Pr	-0.12	-0.075	-0.031	1.32	1.188	1.116	-0.12	-0.073	0.003	1.32	1.164	1.091
L13/1Et	0.95	0.867		1.42	1.484		0.95	1.142	1.372	1.42	1.323	1.264
L13/1Pr	1.38	0.867		1.475	1.814		1.38	1.173	0.942	1.475	1.599	1.727
L14/1Et	1.53	0.872		1.585	1.575		1.53	1.574	2.216	1.585	1.519	1.443
L14/1Pr	3	0.872		1.74	1.905		3	2.957	1.581	1.74	1.748	1.798
L15/1Et	1.965	0.867		0.72	1.484		1.965	2.007	2.292	0.72	0.746	0.686
L15/1Pr	2.315	0.867		0.88	1.814		2.315	2.273	1.711	0.88	0.946	1.121
L16/1Et	3	0.872		1.375	1.575		3	2.944	2.887	1.375	1.339	1.161
L16/1Pr	3	0.872		1.55	1.905		3	2.975	2.956	1.55	1.615	1.650
L17/1Et	3			0.26			3	2.123		0.26	0.712	
L17/1Pr	3			0.41			3	2.154		0.41	0.988	
L18/1Et	3			1.92			3	2.926		1.92	1.472	
L18/1Pr	3			1.92			**3**	**2.957**		**1.92**	**1.748**	

Details of the molecular design

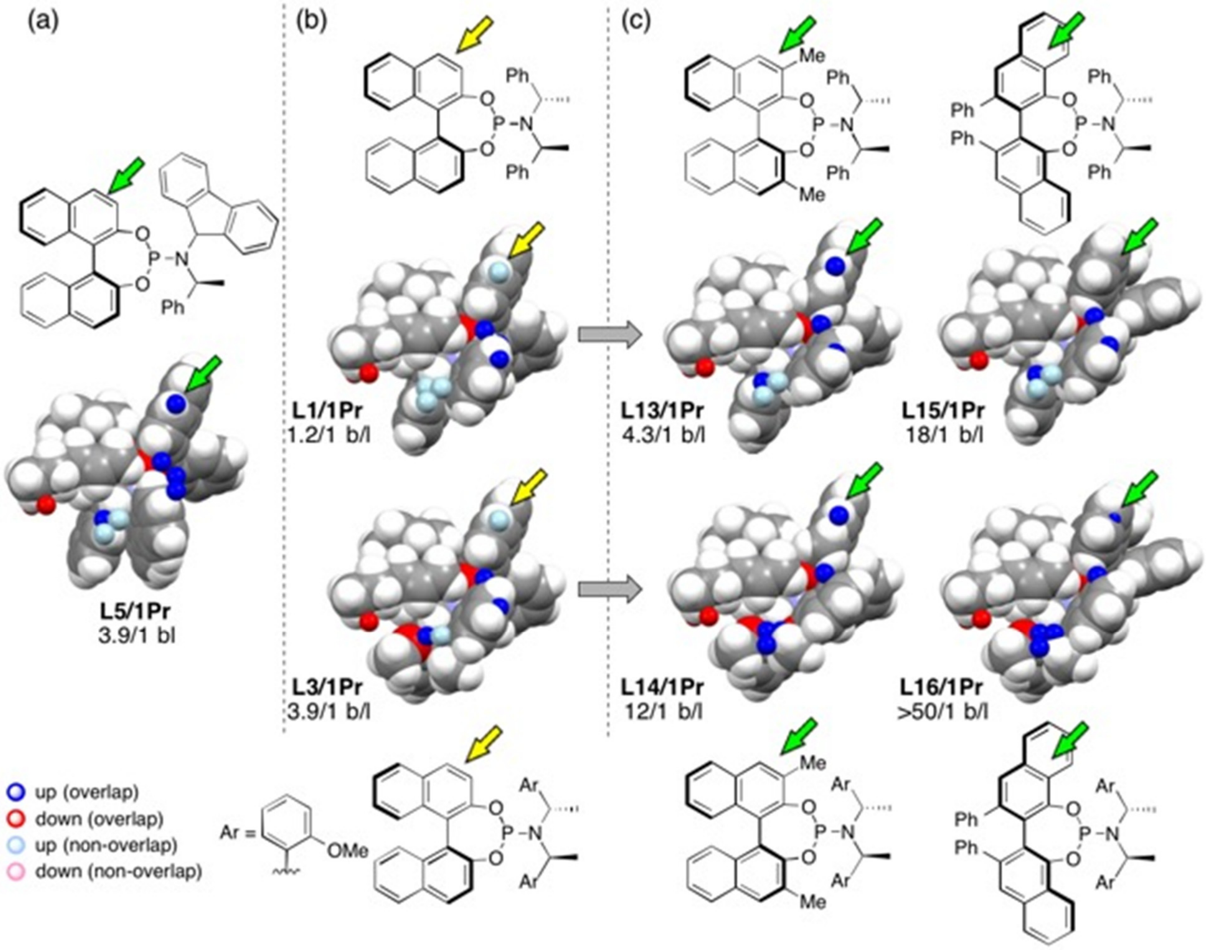


Fig. S4. Results of the MFA using b/l data obtained from the 24 reactions with valine ligand ***S***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules.

The blue point used for the design is derived from, for example, **L5/1Pr**. In comparison to the binaphthyl skeletons of **L1/1Pr** and **L3/1Pr**, that of **L5/1Pr** is closer to the reactive site that affords the linear product, due to the steric repulsion with the fluorene group. On the basis of the light blue point on **L1/1Pr** and **L3/1Pr** indicated by yellow arrow, we designed **L13/1Pr-L16/1Pr** bearing the 3,3'-dimethyl BINOL or VANOL skeleton.

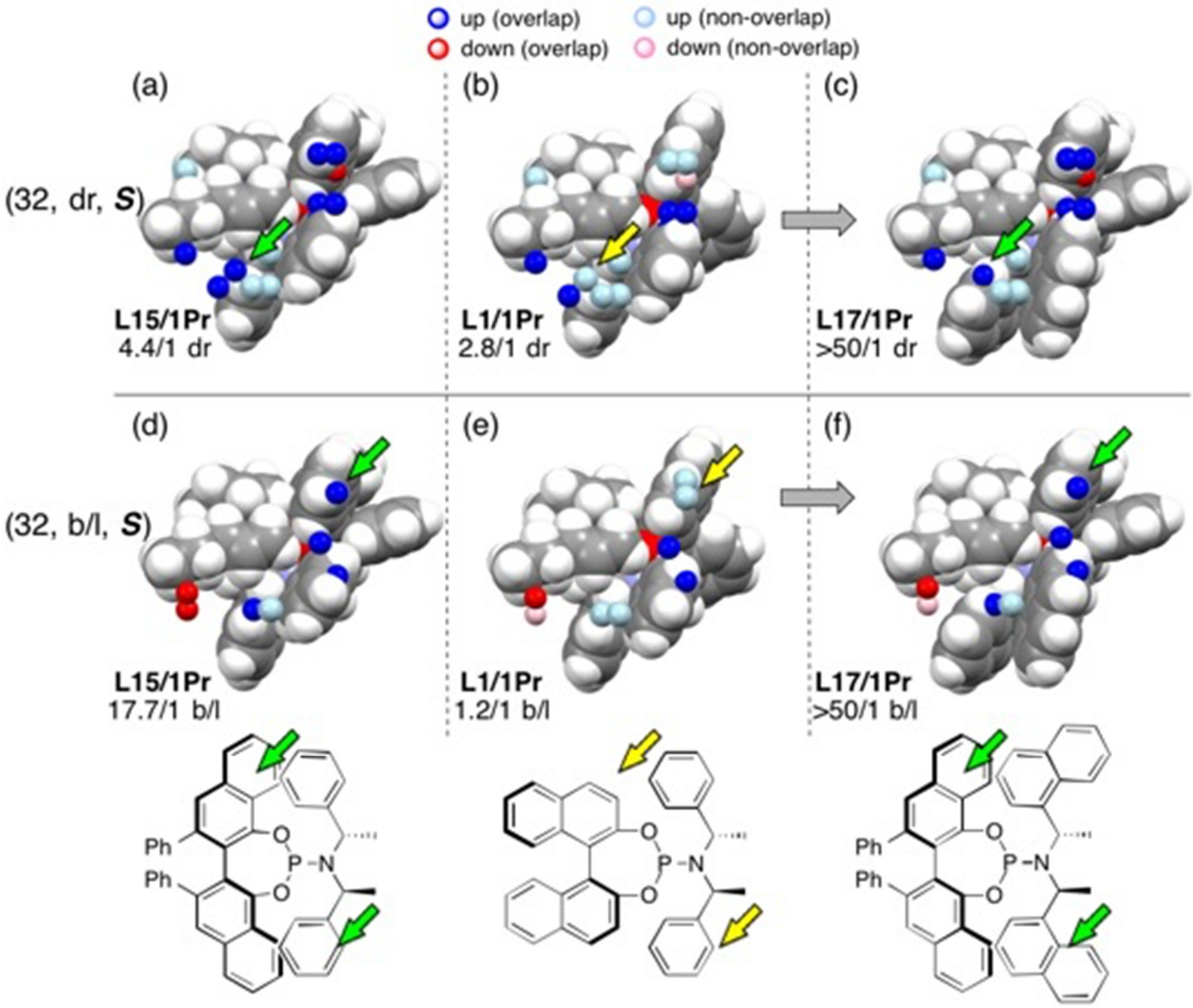


Fig. S5. Results of the MFA using b/l and dr data obtained from the 32 reactions with valine ligand ***S***. (a), (d) The origin of the blue point used for the molecular design. (b), (e) Design templates. (c), (f) Designed molecules.

As to the structural information for dr, the blue point used for the design is derived from, for example, **L15/1Pr**. The phenyl group of **L15/1Pr** is closer to the allyl group in comparison to that of **L1/1Pr** due to steric repulsion with the VANOL skeleton, indicating increase of the steric demand under the allyl group improves dr. Thus, we designed **L17/1Pr** by introducing the naphthyl group in place of the Ph group of **L1/1Pr** along with employment of the VANOL skeleton.

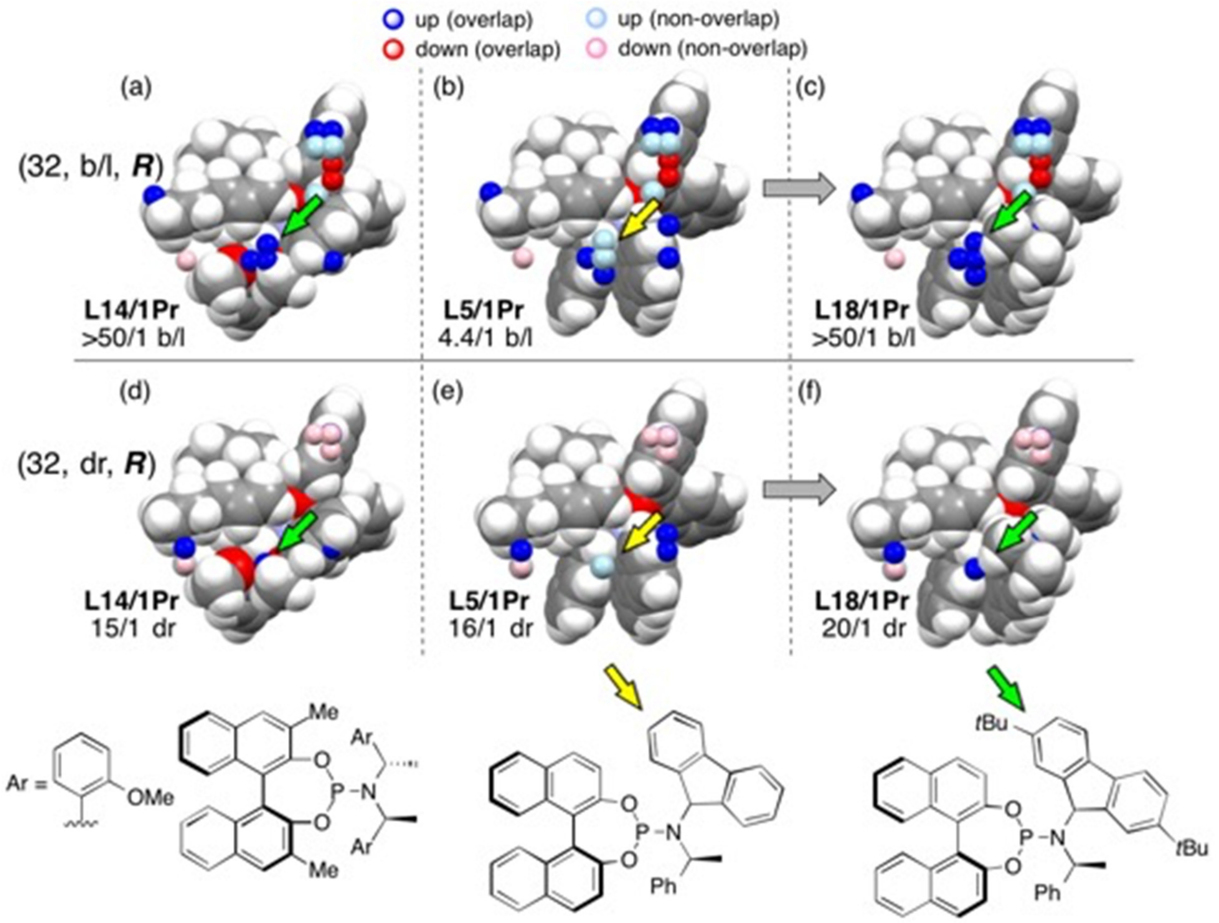


Fig. S6. Results of the MFA using b/l and dr data obtained from the 32 reactions with valine ligand ***R***. (a), (d) The origin of the blue point used for the molecular design. (b), (e) Design templates. (c), (f) Designed molecules.

The blue points used for the design are derived from, for example, the methoxy group of **L14/1Pr** as shown in figs. **S6a** and **S6d**. On the basis of the light blue point around the 2-position of the fluorene group of **L5/1Pr**, we designed **L18/1Pr** by introducing the *t*Bu group.

Determination of the threshold value

In the MFA, we did not visualize structural information from coefficients with absolute values of <0.01. As described in the molecular design section on page S94, we carefully evaluated the regression models and selected the structural information for the molecular design. After the evaluation, practitioners can determine threshold values based on one's intuition to make it easy to obtain insights into reactions (see also figs. S24, S27 and S28). All the results of the MFA used for the design were shown in figs. S8–S11 with threshold values of 0.01 and 0.1. Note that our molecular design is not based on predicted values obtained from the regression models. From the visualized structural information, we obtained the insights into a direction in chemical space where molecules showing improved selectivity would exist, and designed molecules by considering synthetic accessibility. In all cases, however, we confirmed predicted $\Delta\Delta G^{\ddagger}$ values of the designed molecules were at least higher than those of the template molecules used for the design (Tables S3, S4 and figs. S7–S11). In medicinal chemistry, it is known that prediction of the activities of molecules that have largely different structures from those included in a training set is difficult by MFA in comparison to conventional QSAR/free energy relationships. MFA is, however, useful to obtain insights into biological activities of molecules from visualized structural information[29]. Although some groups tried to design molecules through visualized structural information by MFA in asymmetric catalysis, enantioselectivity of the designed molecules lay within the range of those in the training samples[19, 30–32]. Most MFA has employed molecular structures without complexation to substrates (e.g. catalyst alone) in calculations of molecular fields. In our study, however, we employed the structures of catalyst-substrate complexes (i.e., Ir-π-allyl intermediates) in the selectivity determining step [16]. Formation of the Ir-π-allyl intermediates markedly reduced conformational flexibilities and induced structural changes compared to the catalysts alone and substrates alone (as shown in figs S4–S6). These features led to extraction of highly interpretable structural information that resulted in designing the molecules showing improved selectivity.

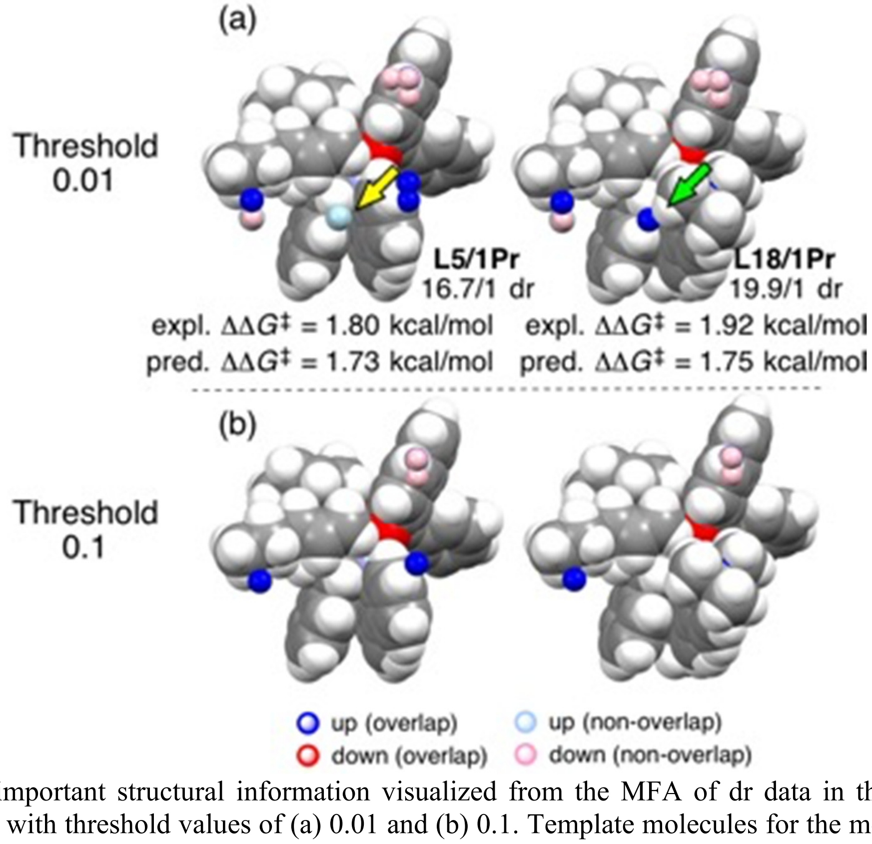


Fig. S7. The important structural information visualized from the MFA of dr data in the 32 reactions using ligand ***R*** with threshold values of (a) 0.01 and (b) 0.1. Template molecules for the molecular design and the designed molecules are shown on the left and right sides, respectively.

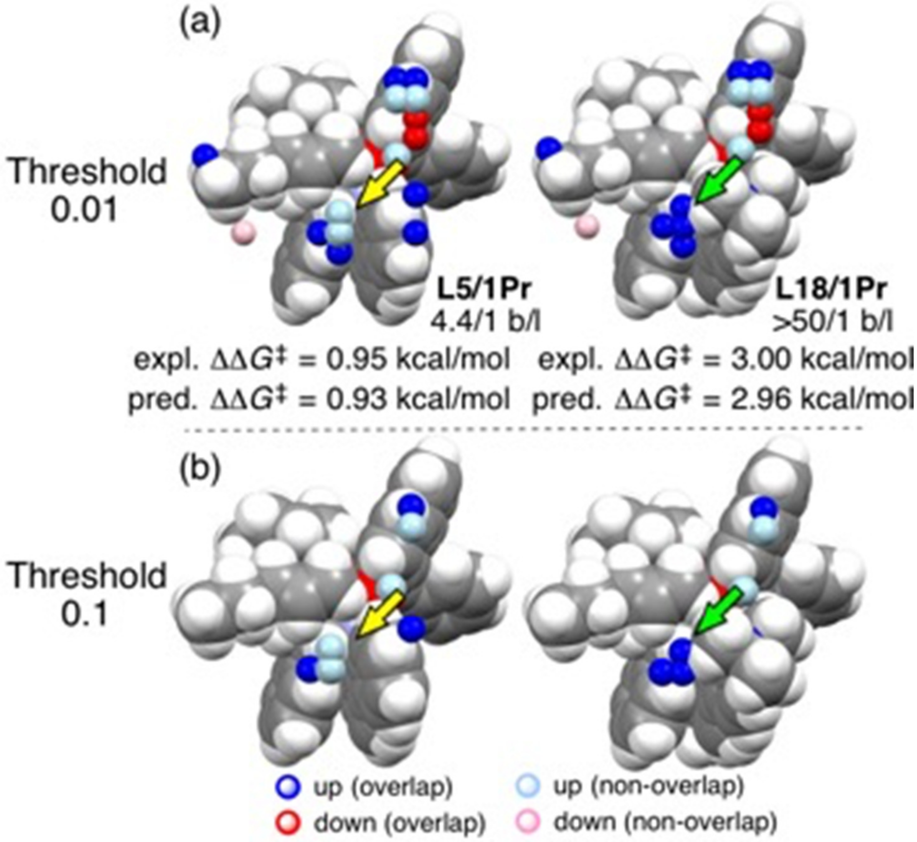


Fig. S8. The important structural information visualized from the MFA of b/l data in the 32 reactions using ligand ***R*** with threshold values of (a) 0.01 and (b) 0.1. Template molecules for the molecular design and the designed molecules are shown on the left and right sides, respectively.

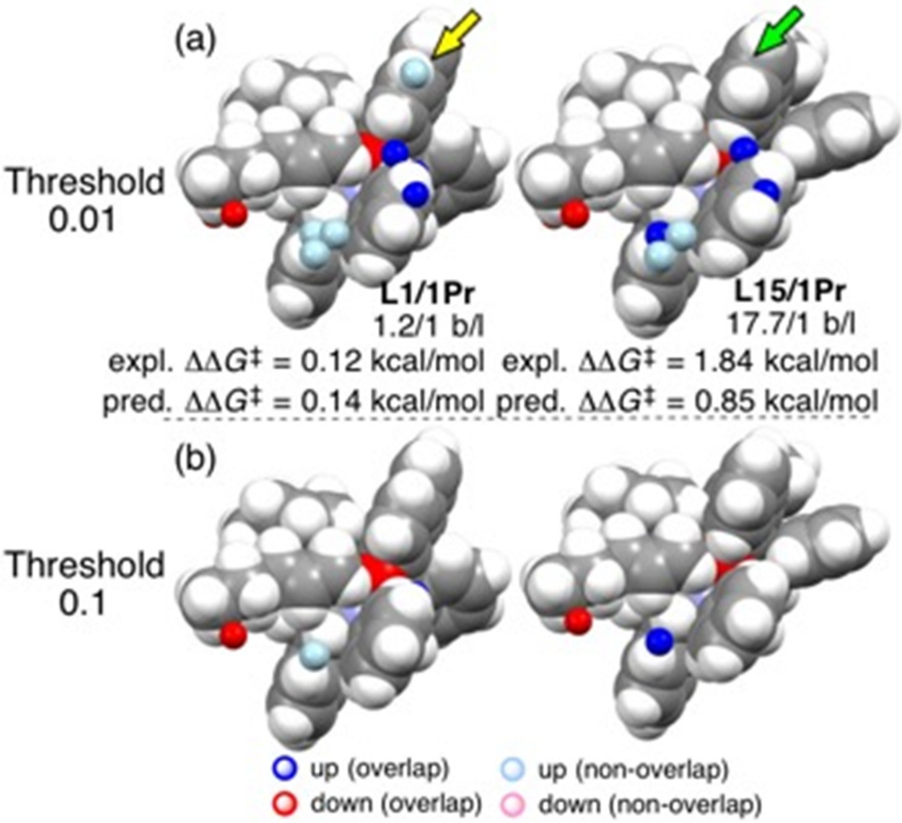


Fig. S9. The important structural information visualized from the MFA of b/l data in the 24 reactions using ligand ***S*** with threshold values of (a) 0.01 and (b) 0.1. Template molecules for the molecular design and the designed molecules are shown on the left and right sides, respectively.

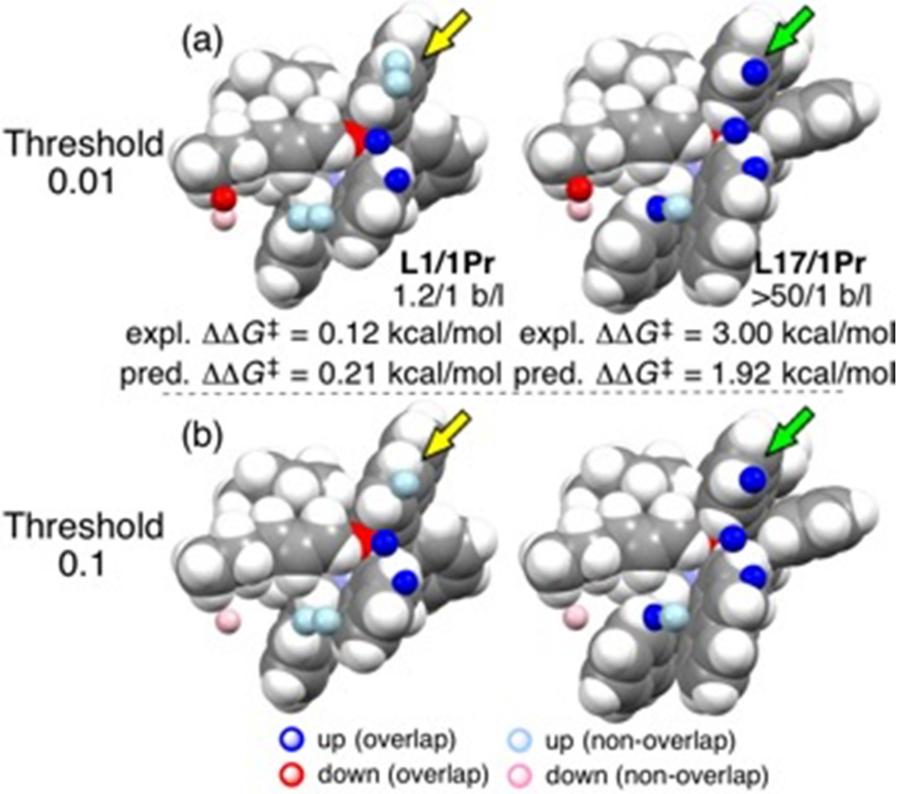


Fig. S10. The important structural information visualized from the MFA of b/l data in the 32 reactions using ligand ***S*** with threshold values of (a) 0.01 and (b) 0.1. Template molecules for the molecular design and the designed molecules are shown on the left and right sides, respectively.

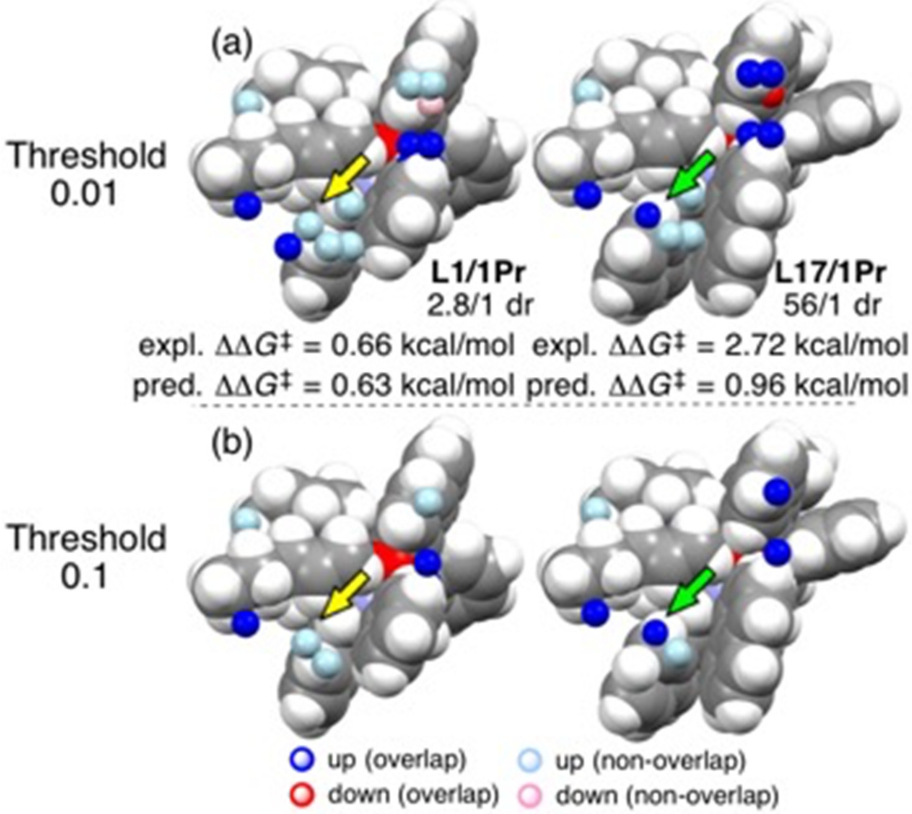


Fig. S11. The important structural information visualized from the MFA of dr data in the 32 reactions using ligand ***S*** with threshold values of (a) 0.01 and (b) 0.1. Template molecules for the molecular design and the designed molecules are shown on the left and right sides, respectively.

Validation of the regression models using test samples

We evaluate the regression models using leave-one-out cross validation, 4-fold cross-validation and y-randomization (figs S2 and S3). Here, we also evaluate the regression models using test samples. We divided the samples into 19 training and 5 test samples (24 samples), and into 25 training and 7 test samples (32 samples). Test samples were selected to cover the entire region of the sample space visualized on the descriptor space reduced from 113 (24 samples) and 149 (32 samples) to 2 dimensions by PCA (principle component analysis) (fig. S12a and S12b). PCA was performed using scikit-learn[33]. All the 5 regression models used for the molecular design fulfilled the Golbraikh-Trophsa criteria[27] (Table S4) that are frequently used to evaluate QSAR/QSPR models. As shown in figs. S12c–S12g, we can find important structural information corresponding to the information used for the molecular design as indicated by yellow and green arrows.

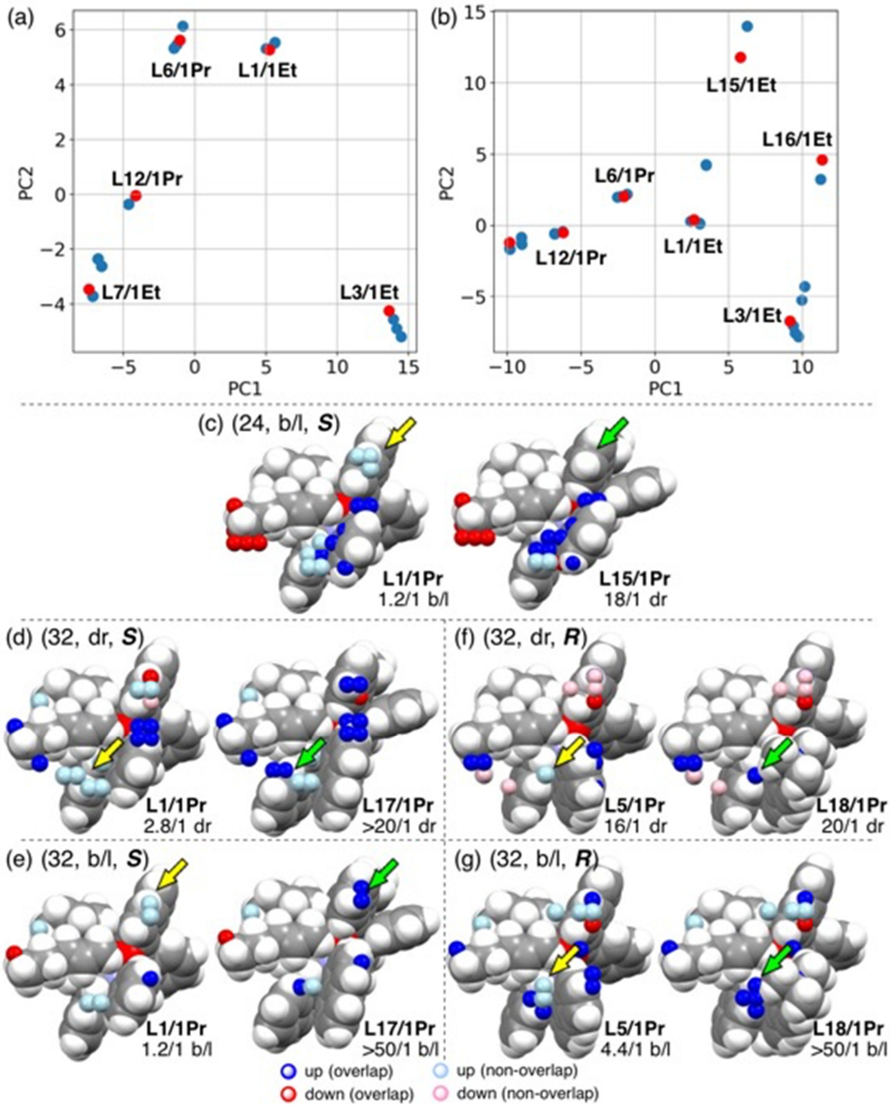


Fig. S12. The samples visualized on a descriptor space compressed to 2-dimesion by PCA for (a) 24 samples and (b) 32 samples. Blue and red dots indicate training and test samples, respectively. (c)–(g) Visualized important information from the regression models using 19 and 25 training samples. In parenthesis, the numbers of all samples including test samples are shown. The structural information from coefficients with absolute values of <0.01 was not visualized.

Table S5. Summary of the MFA using test samples. N_{sel} means the number of extracted descriptors. α is the hyperparamenter of Elastic Net shown in LASSO and Elastic Net regression section on page S93. The numbers of all the descriptors are 113 (24 samples) and 149 (36 samples). We evaluated the model based on Golbraikh-Trophsa criteria shown below[27]. Although some regression models show low q^2 values in comparison to R^2 due to the small sample size and the sample selection method, all models fulfil Golbraikh-Tropsha criteria.

1. Coefficient of determination for a test set $R^2_{pred} > 0.6$;
2. Leave-one-out cross-validated coefficient of determination $q^2 > 0.5$;
3. One of the coefficients of determination for regressions of a test set through the origin (either predicted vs observed values $R_o{}^2{}_{pred}$ or observed vs predicted values $R'_o{}^2{}_{pred}$) should have value close to R^2_{pred};
   $(R^2_{pred} - R_o{}^2{}_{pred})/R^2_{pred}$ or $(R^2_{pred} - R'_o{}^2{}_{pred})/R^2_{pred} < 0.1$ and $0.85 < k$ or $k' < 1.15$, where k and k' are slopes of the regression line through the origin.

	Valine derived ligand ***S*** (24 samples)	Valine derived ligand ***S*** (32 samples)		Valine derived ligand ***R*** (32 samples)	
	b/l	dr	b/l	dr	b/l
α	0.5	1	1	1	1
R^2	0.99	0.99	0.98	0.97	0.99
q^2	0.97	0.93	0.64	0.67	0.73
R^2_{pred}	0.96	0.95	0.90	0.87	0.96
N_{sel}	65	32	12	43	24
$R_o{}^2{}_{pred}$	0.96	0.94	0.90	0.87	0.96
$R'_o{}^2{}_{pred}$	0.96	0.95	0.90	0.84	0.96
k	1.04	1.06	1.14	1.07	1.02
k’	0.94	0.93	0.83	0.92	0.96
$(R^2_{pred} - R_o{}^2{}_{pred})/R^2_{pred}$	<0.01	<0.01	<0.01	<0.01	<0.01
$(R^2_{pred} - R_0{}'^2{}_{pred})/R^2_{pred}$	<0.01	<0.01	<0.01	0.03	<0.01

Table S6. Data of the reactions using valine derived ligand *S* and predicted values (external validation).

Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Training and test samples are highlighted by yellow and purple respectively. Ligands designed are highlighted by green. **L17** is the optimum ligand for the reactions using valine derived ligand ***S***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (b/l)				32 reactions (b/l)			32 reactions (dr)		
	Exp.	Pred.	Pred. (CV)		Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)
L1/1Pr	0.12	0.144	0.189	L1/1Pr	0.12	0.187	0.271	0.66	0.659	0.697
L2/1Et	0.395	0.403	0.417	L2/1Et	0.395	0.289	0.189	0.53	0.554	0.587
L2/1Pr	0.19	0.176	0.159	L2/1Pr	0.19	0.237	0.327	0.76	0.733	0.697
L3/1Pr	0.875	0.826	0.757	L3/1Pr	0.875	0.842	0.822	1.29	1.306	1.355
L4/1Et	0.94	0.937	1.002	L4/1Et	0.94	0.893	0.870	0.98	0.977	1.165
L4/1Pr	0.775	0.801	0.858	L4/1Pr	0.775	0.842	0.878	1.37	1.337	1.278
L5/1Et	0.965	0.951	0.841	L5/1Et	0.965	0.844	0.815	0.06	0.060	0.036
L5/1Pr	0.865	0.847	0.706	L5/1Pr	0.865	0.792	0.769	0.36	0.360	0.238
L6/1Et	0.845	0.849	0.946	L6/1Et	0.845	0.844	0.850	0.03	0.037	0.059
L7/1Pr	-0.12	-0.110	-0.109	L7/1Pr	-0.12	0.005	0.023	0.195	0.316	0.382
L8/1Et	0.145	0.117	0.094	L8/1Et	0.145	0.056	0.007	0.24	0.137	0.051
L8/1Pr	-0.145	-0.110	-0.103	L8/1Pr	-0.145	0.005	0.027	0.36	0.316	0.292
L9/1Et	0.09	0.117	0.125	L9/1Et	0.09	0.056	0.049	-0.06	-0.027	-0.006
L9/1Pr	-0.12	-0.110	-0.109	L9/1Pr	-0.12	0.005	0.023	0.12	0.152	0.182
L10/1Et	0.145	0.117	0.107	L10/1Et	0.145	0.056	0.037	0.06	0.073	0.084
L10/1Pr	-0.115	-0.110	-0.110	L10/1Pr	-0.115	0.005	0.022	0.28	0.252	0.228
L11/1Et	0.12	0.117	0.117	L11/1Et	0.12	0.056	0.043	-0.06	-0.041	-0.029
L11/1Pr	-0.12	-0.110	-0.103	L11/1Pr	-0.12	0.005	0.103	0.12	0.138	0.150
L12/1Et	0.12	0.119	0.104	L12/1Et	0.12	0.126	0.056	0.09	0.090	0.041
L1/1Et	0.34	0.384		L13/1Et	0.715	0.844	0.981	0.395	0.474	0.548
L3/1Et	1.03	1.052		L13/1Pr	0.935	0.792	0.745	0.715	0.653	0.579
L6/1Pr	0.795	0.622		L14/1Et	1.135	1.181	1.697	0.705	0.702	0.999
L7/1Et	0.06	0.117		L14/1Pr	1.57	1.571	1.640	1.2	1.180	1.138
L12/1Pr	-0.145	-0.089		L15/1Pr	1.84	1.927	2.237	0.945	0.921	0.834
L13/1Et	0.715	0.964		L16/1Pr	3	2.706	1.013	1.46	1.447	1.201
L13/1Pr	0.935	0.737		L1/1Et	0.34	0.238		0.41	0.554	
L14/1Et	1.135	1.100		L3/1Et	1.03	0.451		0.995	0.828	
L14/1Pr	1.57	0.873		L6/1Pr	0.795	0.792		0.32	0.338	
L15/1Et	1.78	0.964		L7/1Et	0.06	0.056		0.03	0.037	
L15/1Pr	1.84	0.737		L12/1Pr	-0.145	0.126		0.28	0.218	
L16/1Et	3	1.100		L15/1Et	1.78	1.978		0.8	0.742	
L16/1Pr	3	0.873		L16/1Et	3	2.315		1.025	0.969	
L17/1Et	1.82			L17/1Et	1.82	2.014		1.62	0.684	
L17/1Pr	3			L17/1Pr	3	1.923		2.59	0.863	
L18/1Et	3			L18/1Et	3	1.695		0.66	0.374	
L18/1Pr	3			L18/1Pr	3	1.603		1.18	0.553	

Table S7. Data of the reactions using valine derived ligand *R* and predicted values (external validation).

Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol) used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Training and test samples are highlighted by yellow and purple respectively. Ligands designed are highlighted by green. **L18** is the optimum ligand for the reactions using valine derived ligand ***R***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	32 reactions (b/l)			**32 reactions (dr)**		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Pr	0.6	0.607	0.609	1.465	1.489	1.544
L2/1Et	0.575	0.590	0.604	1.225	1.279	1.346
L2/1Pr	0.625	0.607	0.596	1.63	1.553	1.475
L3/1Pr	0.99	1.011	1.022	1.485	1.493	1.497
L4/1Et	1.045	0.994	0.955	1.215	1.218	1.221
L4/1Pr	0.98	1.011	1.029	1.505	1.493	1.487
L5/1Et	0.905	0.907	0.832	1.535	1.500	1.465
L5/1Pr	**0.945**	**0.927**	0.904	**1.795**	**1.783**	1.767
L6/1Et	0.83	0.833	0.910	1.495	1.497	1.502
L7/1Pr	-0.24	-0.084	0.003	1.015	1.082	1.117
L8/1Et	0.03	-0.100	-0.152	0.705	0.807	0.885
L8/1Pr	-0.03	-0.084	-0.100	1.23	1.082	0.997
L9/1Et	-0.28	-0.212	-0.133	0.765	0.805	0.841
L9/1Pr	-0.26	-0.196	-0.121	1.06	1.079	1.102
L10/1Et	-0.06	-0.101	-0.156	0.93	0.897	0.857
L10/1Pr	-0.06	-0.084	-0.118	1.19	1.172	1.148
L11/1Et	-0.26	-0.253	-0.262	0.83	0.833	0.840
L11/1Pr	-0.28	-0.237	-0.199	1.125	1.108	1.087
L12/1Et	-0.03	-0.032	-0.216	0.725	0.740	0.842
L13/1Et	0.95	1.153	1.369	1.42	1.313	1.179
L13/1Pr	1.38	1.170	0.951	1.475	1.588	1.719
L14/1Et	1.53	1.544	2.438	1.585	1.573	1.456
L14/1Pr	3	2.964	1.547	1.74	1.746	1.851
L15/1Pr	2.315	2.303	2.168	0.88	0.892	1.607
L16/1Pr	3	2.962	1.430	1.55	1.555	1.634
L1/1Et	0.46	0.590		1.13	1.215	
L3/1Et	1.025	0.726		1.185	1.218	
L6/1Pr	0.88	0.853		1.825	1.771	
L7/1Et	-0.28	-0.212		0.775	0.715	
L12/1Pr	-0.12	-0.032		1.32	1.015	
L15/1Et	1.965	2.286		0.72	0.629	
L16/1Et	3	2.677		1.375	1.126	
L17/1Et	3	2.122		0.26	0.629	
L17/1Pr	3	2.139		0.41	0.904	
L18/1Et	3	2.444		1.92	1.569	
L18/1Pr	**3**	**2.461**		**1.92**	**1.844**	

Discussion about conformers

Here, we discuss conformers. Our analysis employed intermediate structures in the selectivity-determining step. Complexation with the Ir salt largely reduces conformational flexibility of the phosphoramidite ligands and the substrates. In some cases, however, conformers with similar energies were found through conformational analysis and the result of the MFA will change depending on conformers. In such a case, we selected conformers according to our intuition as described below. **L18/1Et** and **L18/1Pr** are included in the MFA shown on the next page (fig. S14). Directions of *t*Bu moiety in the most stable conformers **L18/1Et_1** and **L18/1Pr_1** were different. Thus, we employed **L18/1Et_4** for the analysis that had the same conformation with the most stable conformer of **L18/1Pr**.

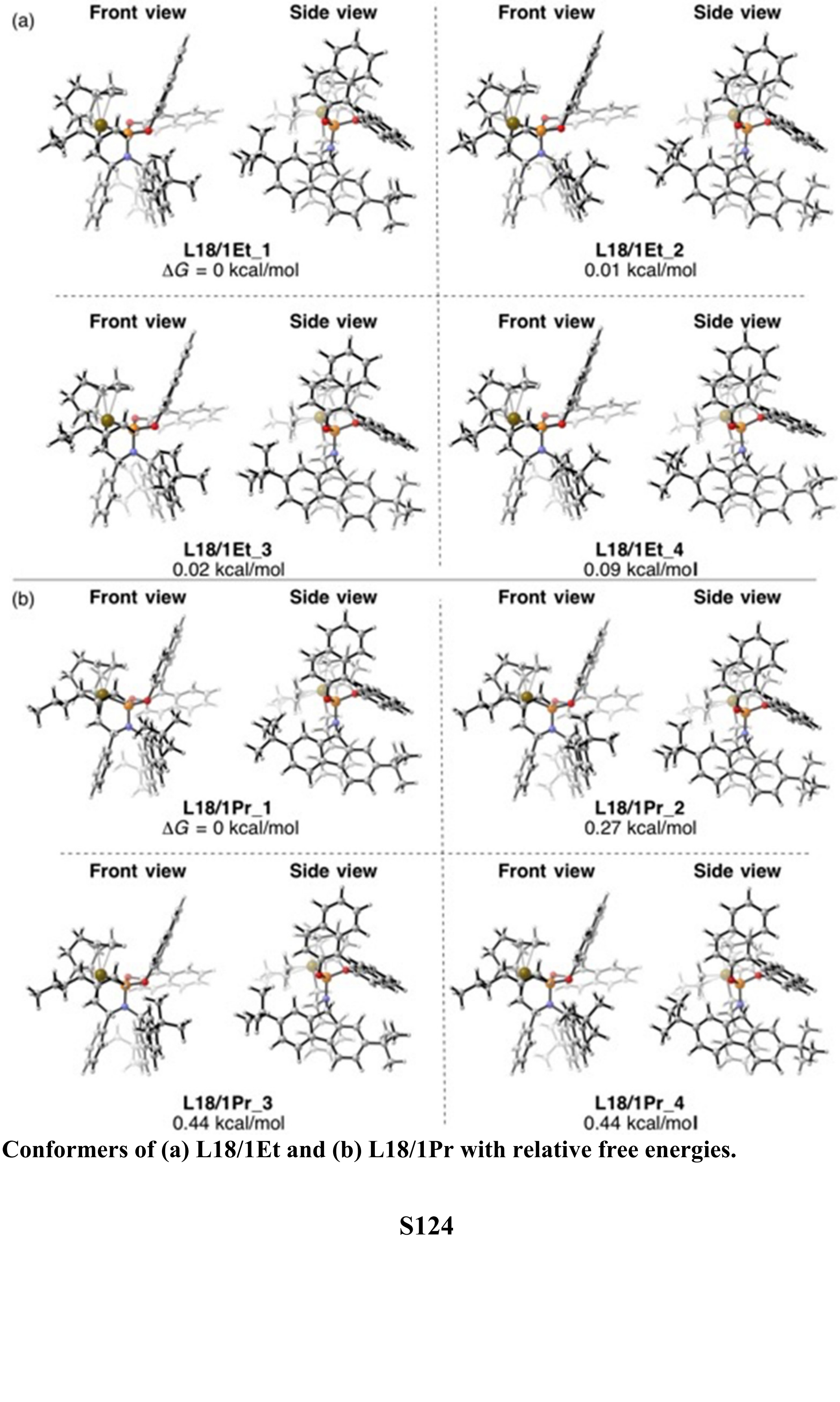


Fig. S13. Conformers of (a) L18/1Et and (b) L18/1Pr with relative free energies.

Another design pathway

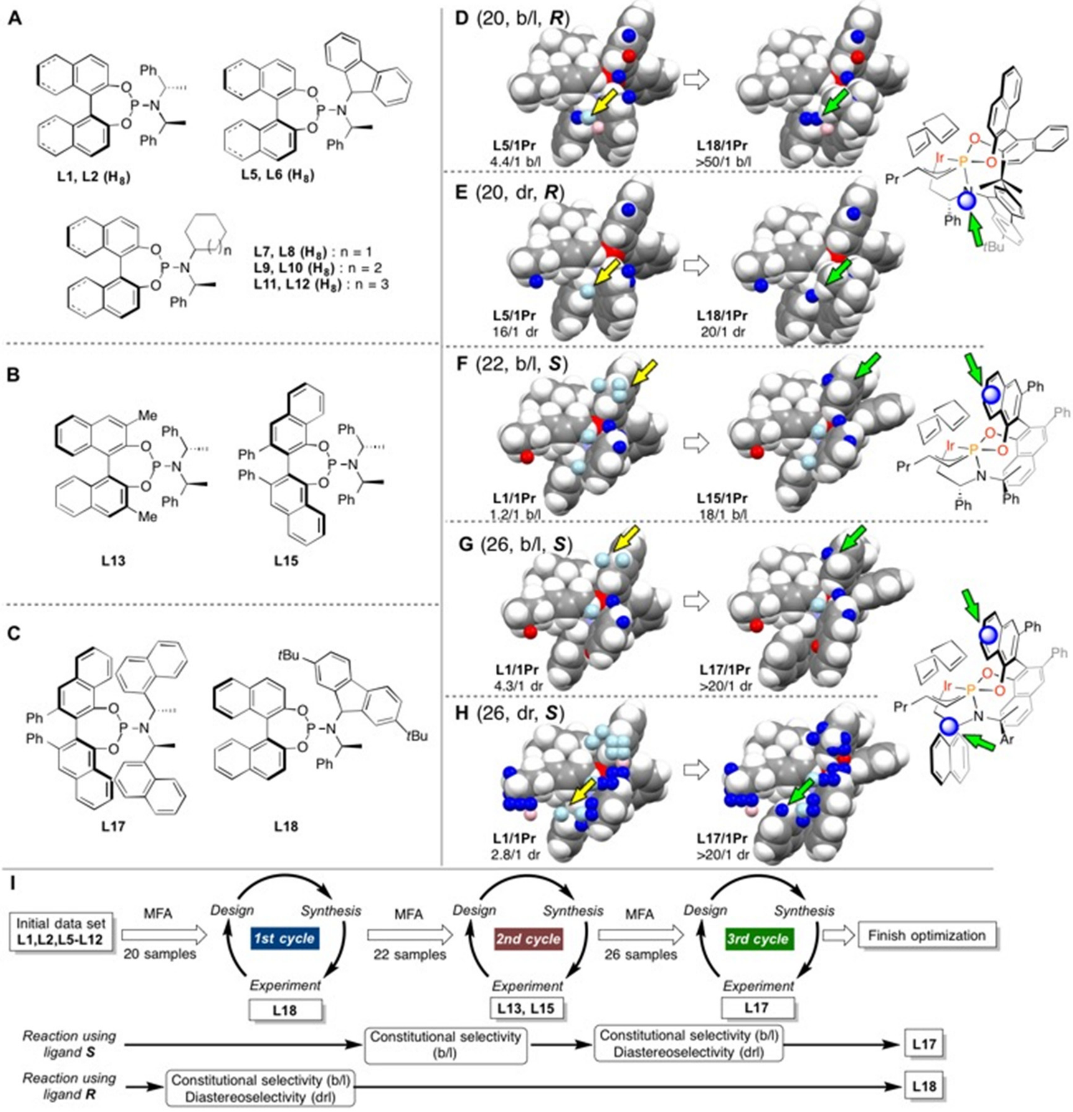


Fig. S14. Another design pathway. **(A)** An initial training set. **(B)**, **(C)** Designed ligands. **(D)–(H)** Important structural information (threshold = 0.01) shown on the template molecules (left sides) and the designed molecules (right sides). Structural information shown in fig S14H was extracted using Elastic Net ($\alpha = 0.4$). All the other information was extracted by LASSO. **(I)** Overall design flow.

In this design pathway, first, we performed the MFA using the 20 reactions with the 10 phosphoramidite ligands shown in fig. S14A. For b/l and dr in the reactions using ligand ***R***, a light blue point existed around the 2-position of the fluorene moiety of **L5/1Pr** as shown in figs. S14D, S14E and S15. Thus, we designed **L18/1Pr** by introducing a *t*Bu group at the 2-postion of the fluorene moiety of **L5/1Pr**. We again performed MFA using the 22 reactions including those with ligand **L18**. As shown in figs. S14F and S16, we could design **L13** and **L15** from **L1/1Pr** based on the information extracted from b/l data in the reactions using the ligand ***S***. We then performed the MFA using the 26 reactions. As shown in figs. S14G, S14H and S17, we could design **L17** from **L1/1Pr** based on the information extracted from b/l and dr data in the reactions using ligand ***S***. Coefficients of determination calculated from the resulting regression models used for the design are shown in fig. S18 along with plots of the measured and predicted values.

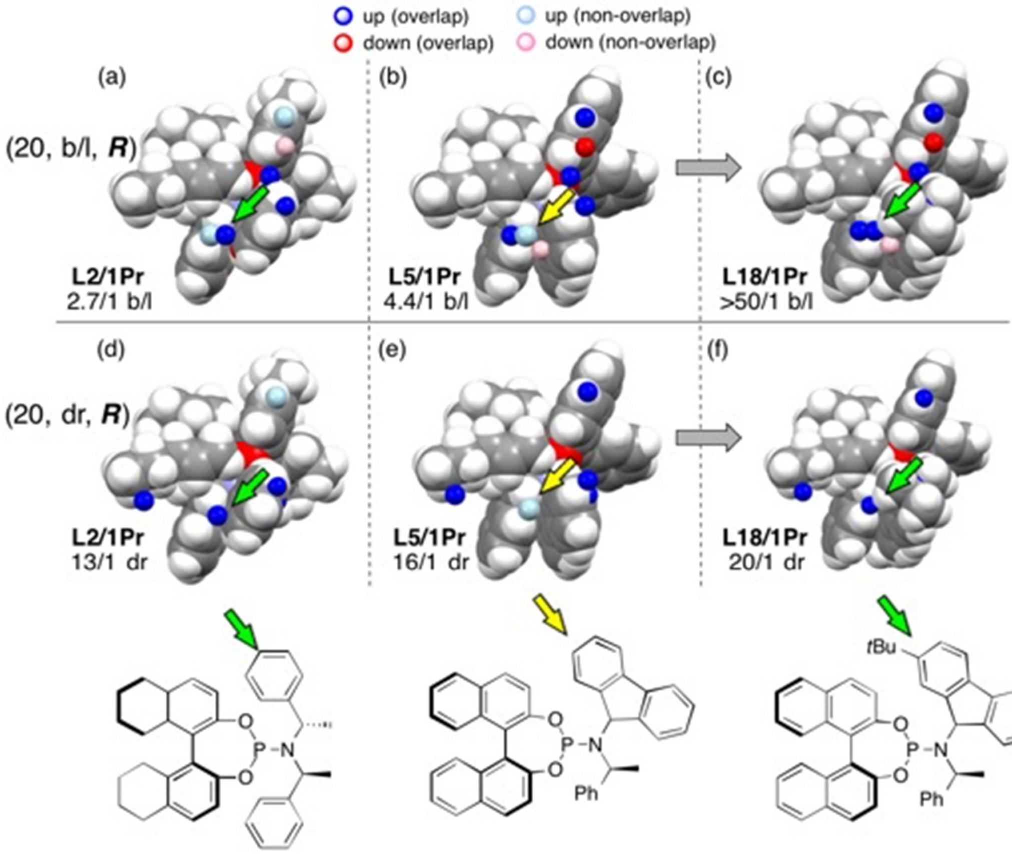


Fig. S15. Results of the MFA using b/l and dr data obtained from the 20 reactions with valine ligand ***R***. (a), (d) The origin of the blue point used for the molecular design. (b), (e) Design templates. (c), (f) Designed molecules.

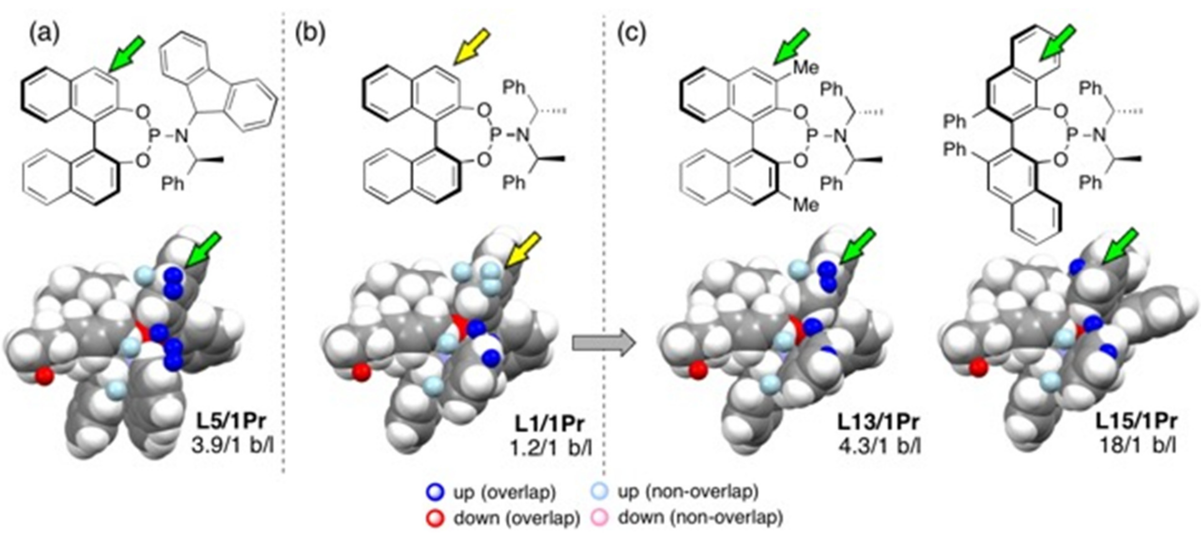


Fig. S16. Results of the MFA using b/l data obtained form the 22 reactions with valine ligand ***S***. (a), The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules.

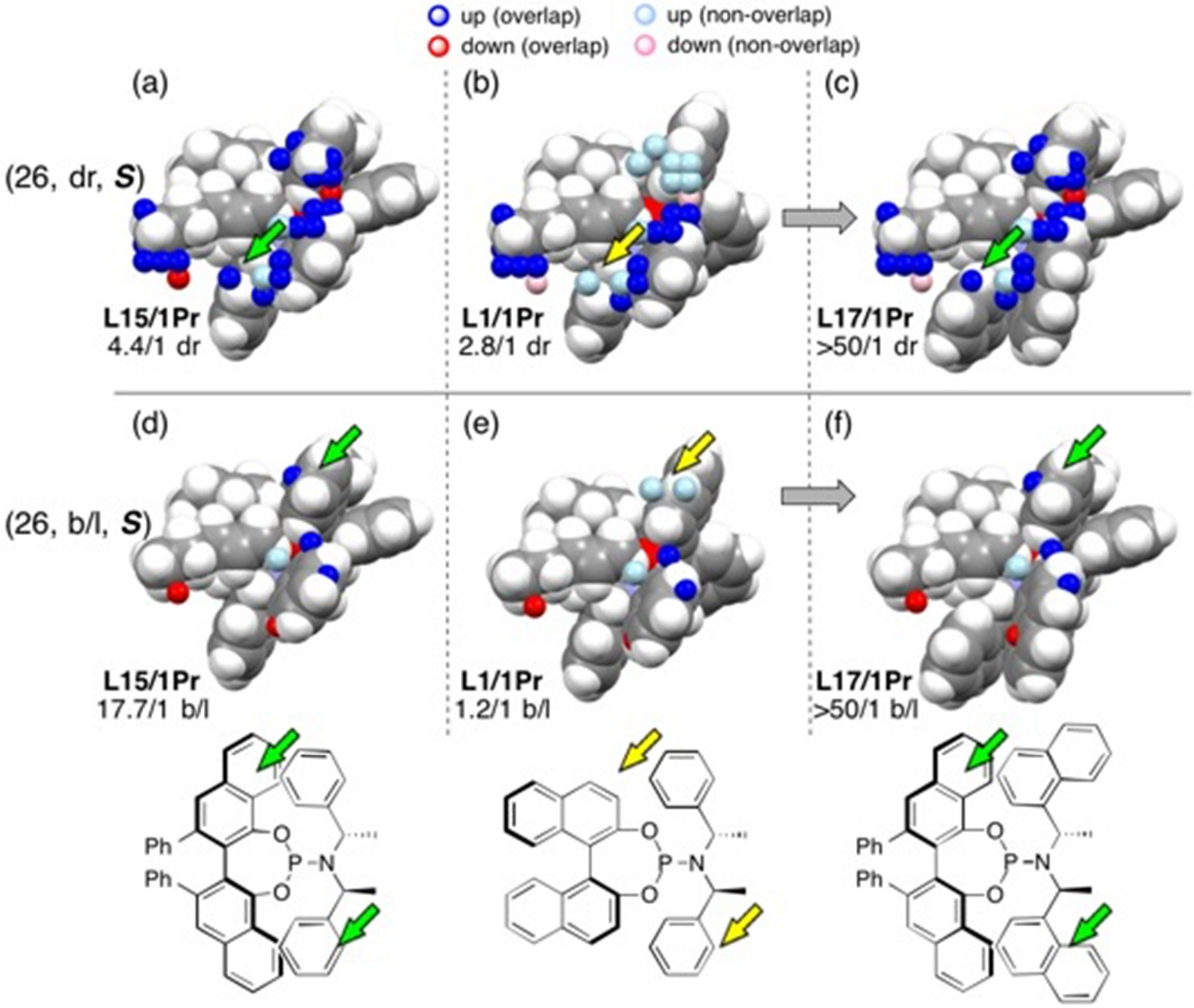


Fig. S17. Results of the MFA using b/l and dr data obtained from the 26 reactions with valine ligand ***S***. (a), (d) The origin of the blue point used for the molecular design. (b), (e) Design templates. (c), (f) Designed molecules.

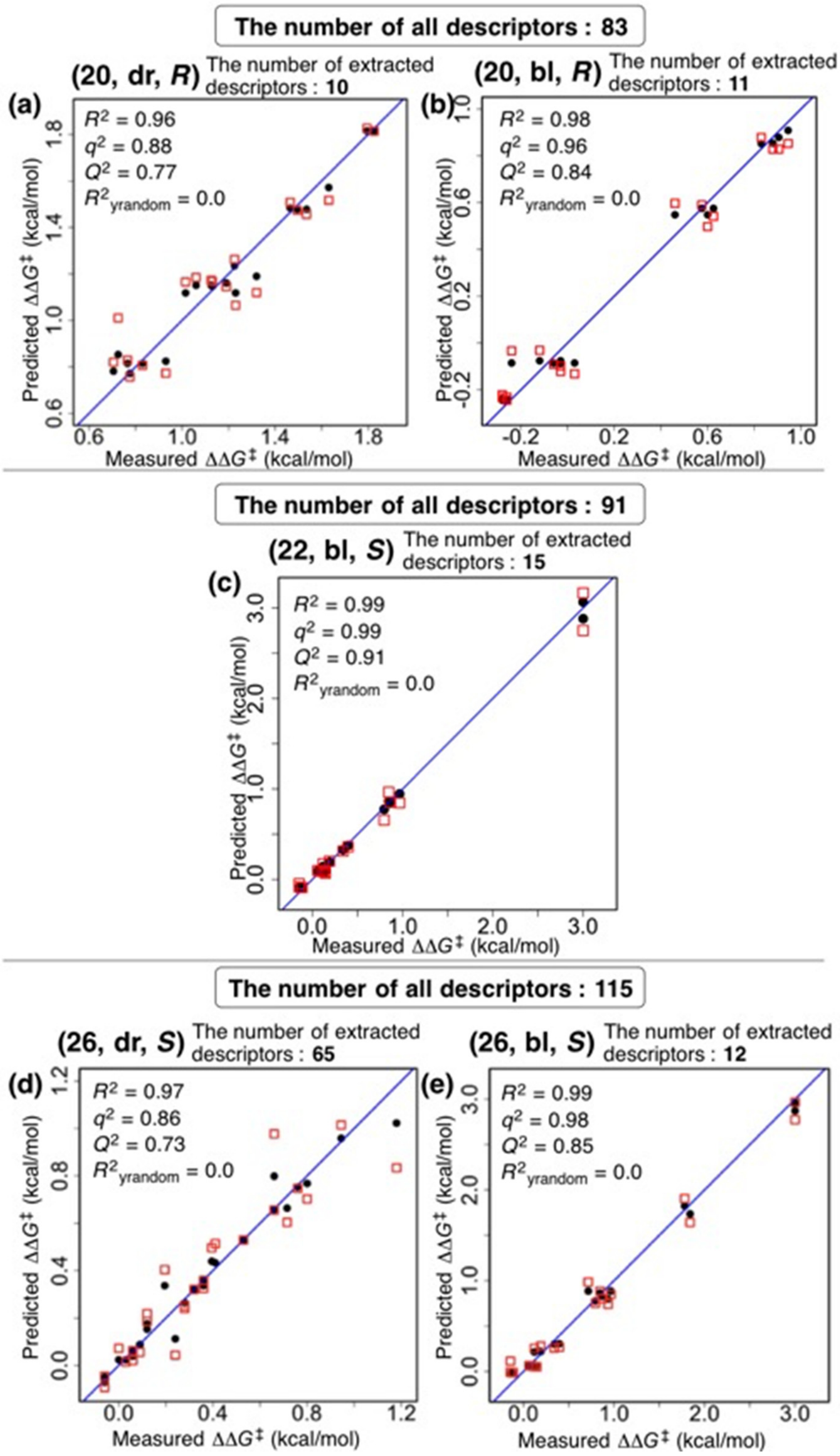


Fig. S18. Results of the MFA in another design pathway. (a)–(e) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. $R^2_{yrandom}$: Coefficient of determination calculated by y-randomization. The blue line is the x = y line

Table S8. Data of the reactions using valine derived ligand *S* and predicted values (another design pathway).

Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** is the optimum ligand for the reactions using valine derived ligand ***S***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values of the reactions using the template and designed molecules are highlighted by red.

	22 reactions (b/l)			26 reactions (b/l)			26 reactions (dr)		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Et	0.34	0.327	0.314	0.34	0.301	0.257	0.41	0.433	0.514
L1/1Pr	**0.12**	**0.143**	0.173	**0.12**	**0.214**	0.254	**0.66**	**0.658**	0.655
L2/1Et	0.395	0.375	0.361	0.395	0.305	0.267	0.53	0.528	0.529
L2/1Pr	0.19	0.191	0.201	0.19	0.218	0.280	0.76	0.752	0.746
L5/1Et	0.965	0.946	0.846	0.965	0.886	0.848	0.06	0.060	0.062
L5/1Pr	0.865	0.856	0.850	0.865	0.830	0.801	0.36	0.357	0.359
L6/1Et	0.845	0.864	0.965	0.845	0.864	0.885	0.03	0.023	0.072
L6/1Pr	0.795	0.775	0.656	0.795	0.776	0.754	0.32	0.320	0.321
L7/1Et	0.06	0.092	0.098	0.06	0.063	0.063	0.03	0.023	0.015
L7/1Pr	-0.12	-0.092	-0.088	-0.12	-0.024	-0.013	0.195	0.336	0.404
L8/1Et	0.145	0.092	0.070	0.145	0.063	0.051	0.24	0.112	0.043
L8/1Pr	-0.145	-0.092	-0.084	-0.145	-0.024	-0.009	0.36	0.336	0.325
L9/1Et	0.09	0.092	0.093	0.09	0.063	0.059	-0.06	-0.050	-0.046
L9/1Pr	-0.12	-0.092	-0.088	-0.12	-0.024	-0.013	0.12	0.175	0.219
L10/1Et	0.145	0.092	0.084	0.145	0.063	0.051	0.06	0.039	0.021
L10/1Pr	-0.115	-0.092	-0.089	-0.115	-0.024	-0.014	0.28	0.263	0.253
L11/1Et	0.12	0.092	0.088	0.12	0.063	0.055	-0.06	-0.071	-0.093
L11/1Pr	-0.12	-0.092	-0.088	-0.12	-0.024	-0.013	0.12	0.153	0.186
L12/1Et	0.12	0.092	0.088	0.12	0.063	0.055	0.09	0.088	0.055
L12/1Pr	-0.145	-0.092	-0.047	-0.145	-0.024	0.113	0.28	0.263	0.241
L18/1Et	3	3.066	3.166	3	2.959	2.965	0.66	0.798	0.977
L18/1Pr	3	2.882	2.751	3	2.872	2.773	1.18	1.023	0.834
L13/1Et	0.715	1.040		0.715	0.886	0.986	0.395	0.439	0.496
L13/1Pr	**0.935**	**0.856**		0.935	0.799	0.740	0.715	0.664	0.603
L15/1Et	1.78	1.064		1.78	1.823	1.905	0.8	0.768	0.702
L15/1Pr	**1.84**	**0.880**		1.84	1.735	1.641	0.945	0.959	1.015
L17/1Et	1.82			1.82	1.823		1.62	0.768	
L17/1Pr	3			**3**	**1.735**		**2.59**	**0.992**	

Table S9. Data of the reactions using valine derived ligand *R* and predicted values (another design pathway).

Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol) used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L18** is the optimum ligand for the reactions using valine derived ligand ***R***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values of the reactions using the template and designed molecules are highlighted by red.

	20 reactions (b/l)			20 reactions (dr)		
	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)
L1/1Et	0.46	0.548	0.597	1.13	1.146	1.168
L1/1Pr	0.6	0.548	0.497	1.465	1.483	1.508
L2/1Et	0.575	0.575	0.590	1.225	1.235	1.262
L2/1Pr	0.625	0.575	0.541	1.63	1.572	1.517
L5/1Et	0.905	0.879	0.828	1.535	1.478	1.456
L5/1Pr	**0.945**	**0.909**	0.853	**1.795**	**1.815**	1.827
L6/1Et	0.83	0.852	0.878	1.495	1.478	1.473
L6/1Pr	0.88	0.854	0.828	1.825	1.815	1.814
L7/1Et	-0.28	-0.240	-0.222	0.775	0.771	0.756
L7/1Pr	-0.24	-0.086	-0.034	1.015	1.118	1.166
L8/1Et	0.03	-0.086	-0.132	0.705	0.781	0.820
L8/1Pr	-0.03	-0.086	-0.099	1.23	1.118	1.065
L9/1Et	-0.28	-0.240	-0.222	0.765	0.814	0.829
L9/1Pr	-0.26	-0.240	-0.232	1.06	1.151	1.185
L10/1Et	-0.06	-0.086	-0.091	0.93	0.824	0.773
L10/1Pr	-0.06	-0.086	-0.091	1.19	1.161	1.145
L11/1Et	-0.26	-0.245	-0.245	0.83	0.814	0.807
L11/1Pr	-0.28	-0.245	-0.234	1.125	1.151	1.173
L12/1Et	-0.03	-0.076	-0.122	0.725	0.853	1.011
L12/1Pr	-0.12	-0.076	-0.032	1.32	1.190	1.120
L18/1Et	3	0.936		1.92	1.566	
L18/1Pr	**3**	**0.936**		**1.92**	**1.903**	

Possible diastereoinduction mechanism

The blue points for b/l were observed near the reactive site that afforded the linear product. Here we explain interpretation of the important structural information for dr. This reaction proceeded via nucleophilic attack of the boron enolate species to the Ir-π-allyl complex. For sake of simplicity, here we employ a mononuclear boron enolate structure for the explanation while the structure of the boron enolate species is under discussion. In this section, we performed DFT calculations of the boron enolate structures using the B3LYP functional augmented with Grimme's D3 empirical dispersion term[15], and 6-31G(d) basis set. Frequency calculations were performed at the same level of theory described above to obtain thermal corrections to the Gibbs free energies. Single point energy calculations were performed with the M06 functional[34] using 6-311++G(d,p) basis set. Solvation effects were included using the SMD[35] solvation model with tetrahydrofuran as the solvent. Although we have not considered the boron enolate structures explicitly, the information about the boron enolates is included in the experimental selectivity data. Therefore, on the basis of the visualized information, we can discuss diastereoinduction mechanisms including the facial selectivity of the boron enolates as shown in figs. S19 and S20.

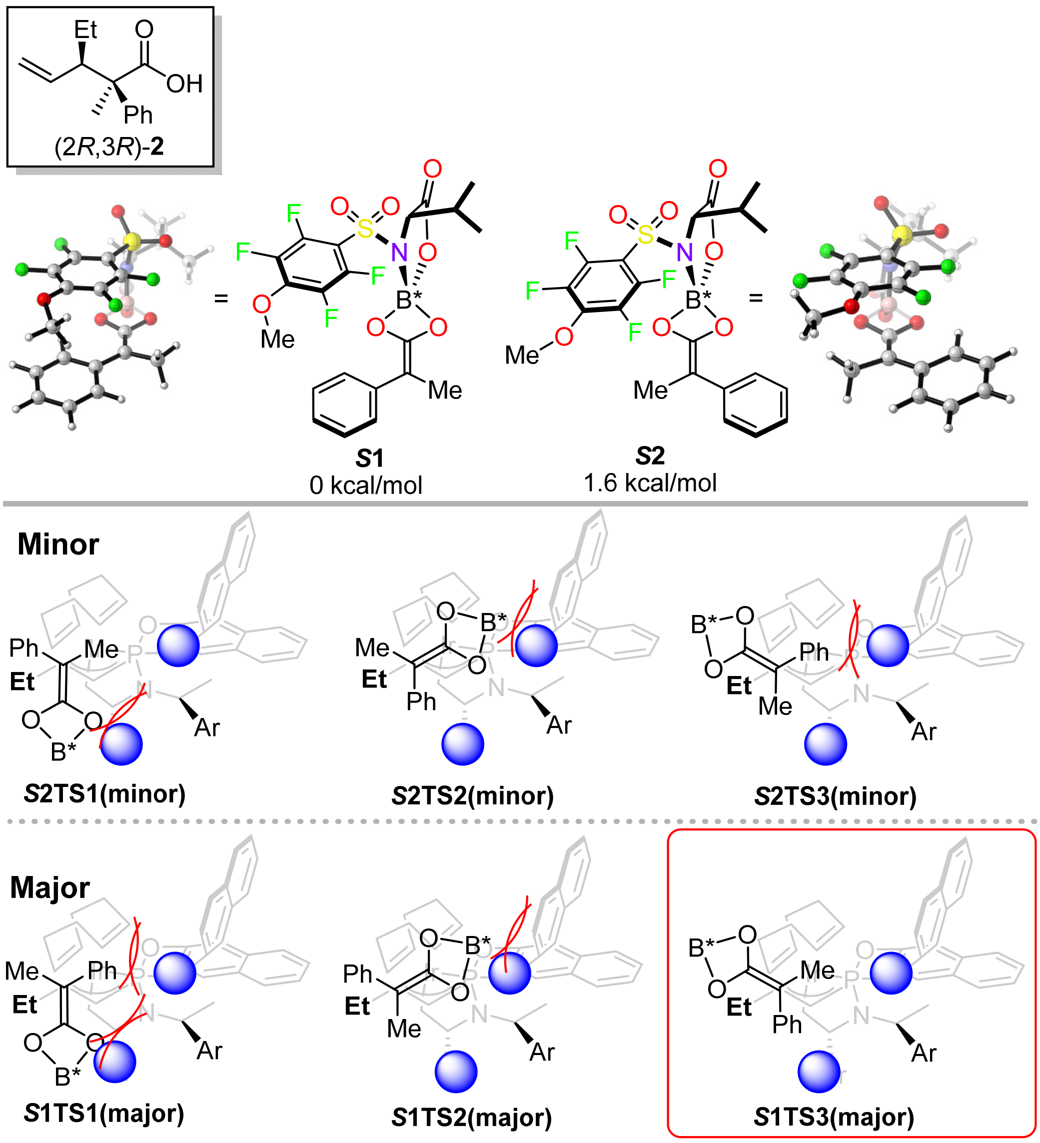


Fig. S19. The possible diastereoinduction mechanism for the reactions using ligand ***S***.

S*1** and ***S*2** are the conformers of the boron enolate having valine-derived ligand ***S. According to the DFT calculations, the conformer ***S*1**, in which *Si*-face is covered by the substituent on the boron catalyst, is more stable than the conformer ***S*2**. We showed possible 6 transition state geometries having the staggered conformation along with the important structural information extracted by the MFA of the dr data obtained from the 32 reactions. When substituents are introduced to the blue points, only ***S*1TS3** is not destabilized by the substituents.

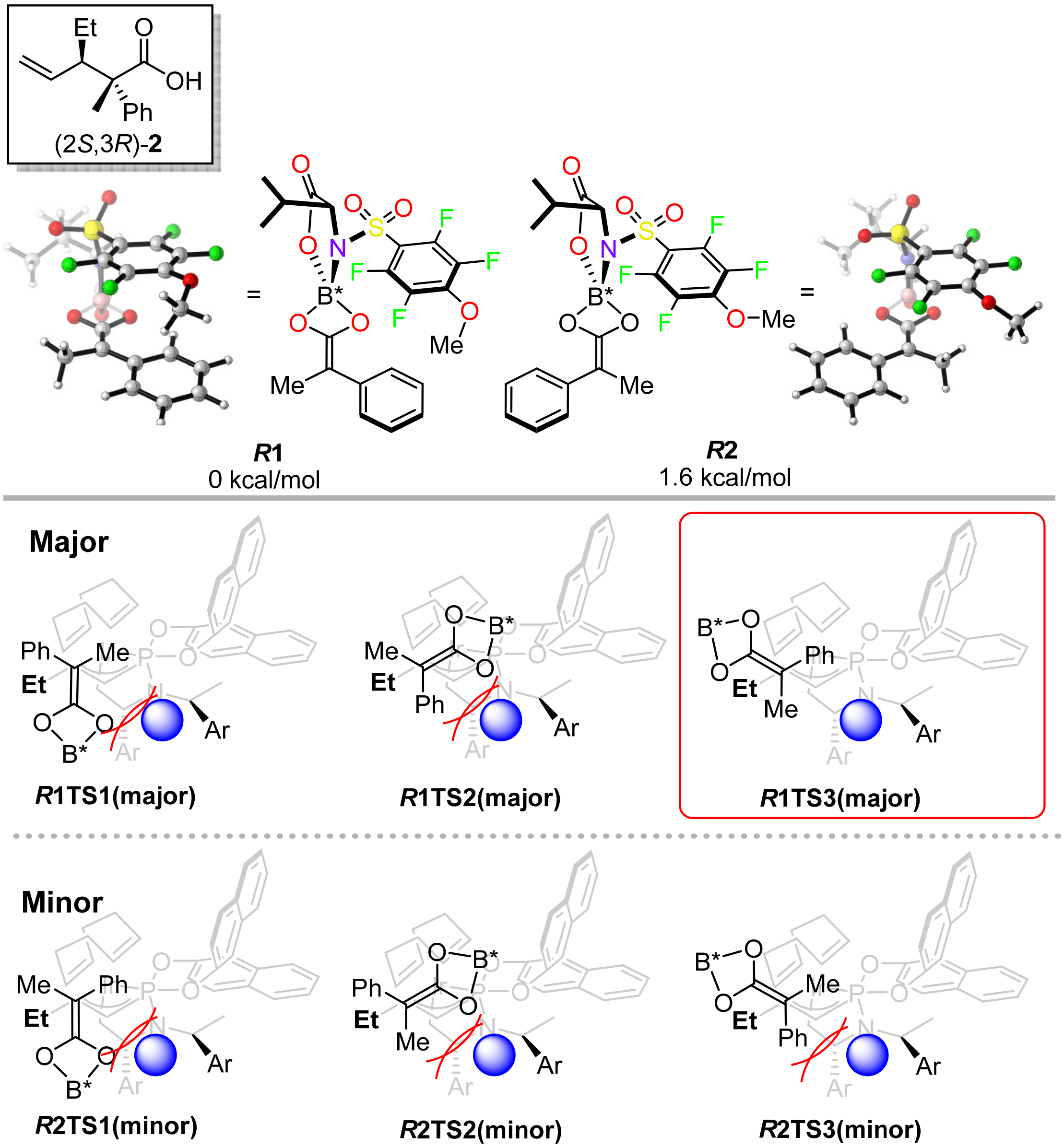


Fig. S20. The possible diastereoinduction mechanism for the reactions using ligand ***R***.

R*1** and ***R*2** are the conformers of the boron enolate having valine-derived ligand ***R. According to the DFT calculations, the conformer ***R*1**, in which *Re*-face is covered by the substituent on the boron catalyst, is more stable than the conformer ***R*2**. We showed possible 6 transition state geometries having the staggered conformation along with the important structural information extracted by the MFA of the dr data obtained from the 32 reactions. When substituents are introduced to the blue points, only ***R*1TS3** is not destabilized by the substituents.

MFA using descriptor2 (descriptors calculated by removing the columns showing |*r*| ≦ 0.3 (|*r*| is an absolute value of a correlation coefficient with the target variables.)

Plots of the measured vs predicted values

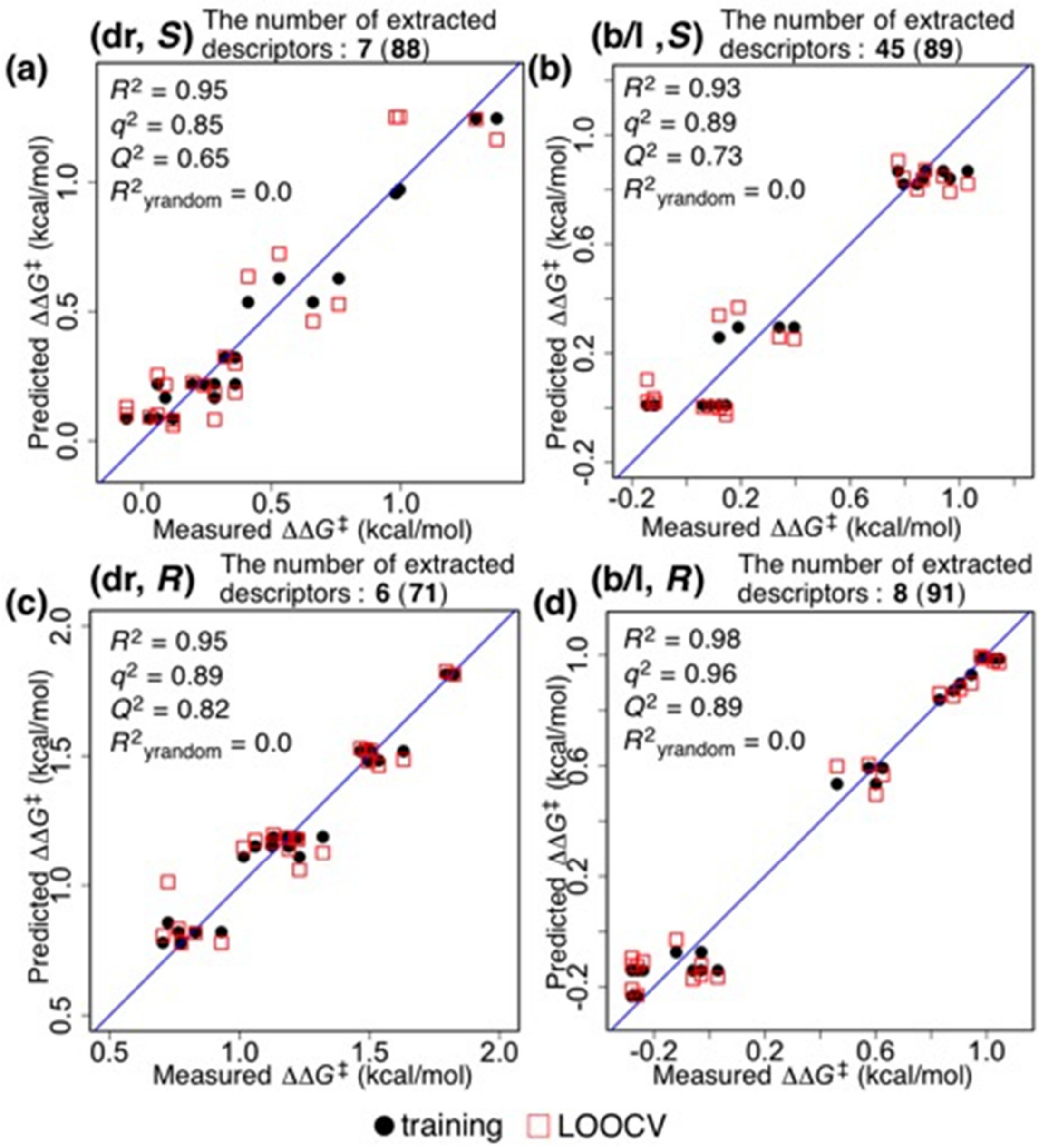


Fig. S21. Results of the MFA using 24 training samples and descriptor 2. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. The number in parenthesis is the number of all descriptors. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

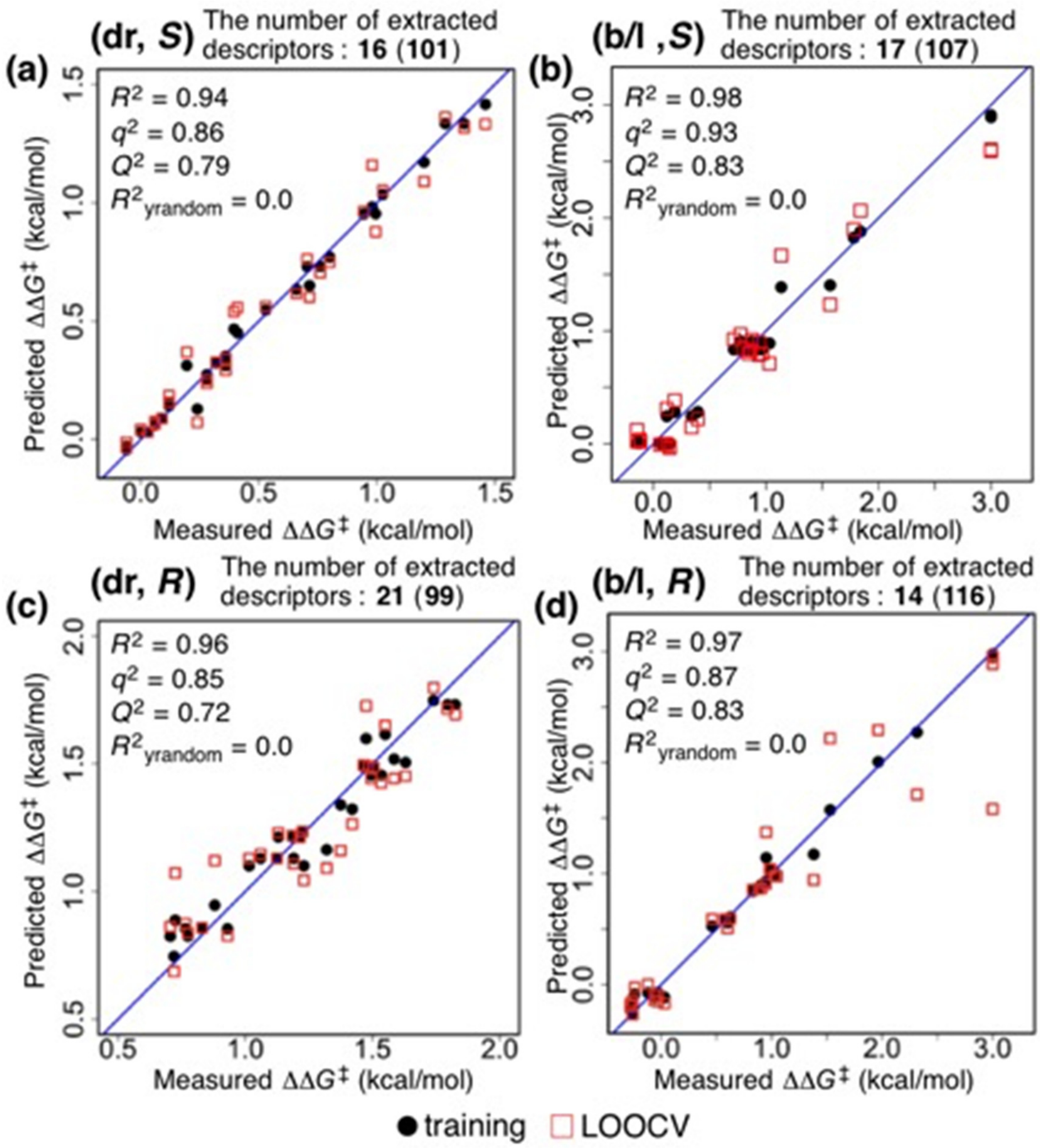


Fig. S22. Results of the MFA using 32 training samples and descriptor 2. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. The number in parenthesis is the number of all descriptors. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

Table S10. Data of the reactions using valine derived ligand *S* and predicted values. Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** is the optimum ligand for the reactions using valine derived ligand ***S***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (b/l)			24 reactions (dr)			32 reactions (b/l)			32 reactions (dr)		
	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)
L1/1Et	0.34	0.295	0.259	0.41	0.536	0.635	0.34	0.244	0.147	0.41	0.584	0.648
L1/1Pr	**0.12**	**0.257**	0.339	0.66	0.536	0.462	**0.12**	**0.244**	0.310	**0.66**	**0.584**	0.535
L2/1Et	0.395	0.295	0.252	0.53	0.628	0.724	0.395	0.282	0.218	0.53	0.592	0.641
L2/1Pr	0.19	0.295	0.368	0.76	0.628	0.527	0.19	0.282	0.383	0.76	0.592	0.536
L3/1Et	1.03	0.868	0.821	0.995	0.971	1.252	1.03	0.890	0.710	0.995	0.947	0.892
L3/1Pr	**0.875**	**0.868**	0.872	1.29	1.246	1.243	0.875	0.907	0.923	1.29	1.278	1.279
L4/1Et	0.94	0.868	0.848	0.98	0.956	1.251	0.94	0.907	0.897	0.98	1.061	1.316
L4/1Pr	0.775	0.868	0.906	1.37	1.246	1.163	0.775	0.907	0.969	1.37	1.278	1.241
L5/1Et	0.965	0.840	0.792	0.06	0.086	0.102	0.965	0.835	0.799	0.06	0.136	0.181
L5/1Pr	0.865	0.840	0.837	0.36	0.322	0.299	0.865	0.835	0.827	0.36	0.341	0.320
L6/1Et	0.845	0.820	0.801	0.03	0.086	0.092	0.845	0.822	0.799	0.03	0.136	0.146
L6/1Pr	0.795	0.820	0.843	0.32	0.322	0.325	0.795	0.822	0.839	0.32	0.341	0.361
L7/1Et	0.06	0.010	0.002	0.03	0.086	0.092	0.06	0.007	-0.007	0.03	0.136	0.146
L7/1Pr	-0.12	0.010	0.021	0.195	0.219	0.226	-0.12	0.007	0.018	0.195	0.136	0.130
L8/1Et	0.145	0.010	-0.026	0.24	0.219	0.214	0.145	0.007	-0.037	0.24	0.136	0.126
L8/1Pr	-0.145	0.010	0.023	0.36	0.219	0.188	-0.145	0.007	0.020	0.36	0.136	0.115
L9/1Et	0.09	0.010	0.002	-0.06	0.086	0.102	0.09	0.007	-0.002	-0.06	0.136	0.154
L9/1Pr	-0.12	0.010	0.021	0.12	0.086	0.060	-0.12	0.007	0.020	0.12	0.136	0.137
L10/1Et	0.145	0.010	-0.006	0.06	0.219	0.255	0.145	0.007	-0.026	0.06	0.136	0.142
L10/1Pr	-0.115	0.010	0.020	0.28	0.219	0.185	-0.115	0.007	0.019	0.28	0.136	0.090
L11/1Et	0.12	0.010	-0.001	-0.06	0.086	0.131	0.12	0.007	-0.004	-0.06	0.136	0.159
L11/1Pr	-0.12	0.010	0.036	0.12	0.086	0.080	-0.12	0.007	0.036	0.12	0.136	0.137
L12/1Et	0.12	0.010	-0.001	0.09	0.167	0.217	0.12	0.007	-0.004	0.09	0.186	0.254
L12/1Pr	-0.145	0.010	0.103	0.28	0.167	0.082	-0.145	0.007	0.123	0.28	0.186	0.132
L13/1Et	0.715	0.880		0.395	0.403		0.715	0.835	0.925	0.395	0.557	0.696
L13/1Pr	**0.935**	**0.880**		0.715	0.403		0.935	0.835	0.787	0.715	0.557	0.470
L14/1Et	1.135	0.920		0.705	0.838		1.135	1.387	1.668	0.705	0.781	0.852
L14/1Pr	**1.57**	**0.920**		1.2	1.113		1.57	1.404	1.233	1.2	1.111	1.037
L15/1Et	1.78	0.880		0.8	0.403		1.78	1.825	1.900	0.8	0.797	0.828
L15/1Pr	**1.84**	**0.880**		0.945	0.403		1.84	1.880	2.065	0.945	0.827	0.748
L16/1Et	3	0.920		1.025	0.838		3	2.892	2.607	1.025	1.051	1.094
L16/1Pr	**3**	**0.920**		1.46	1.113		3	2.909	2.591	1.46	1.382	1.318
L17/1Et	1.82			1.62			1.82	1.880		1.62	0.827	
L17/1Pr	3			2.59			**3**	**1.880**		**2.59**	**0.827**	
L18/1Et	3			0.66			3	1.387		0.66	0.505	
L18/1Pr	3			1.18			3	1.387		1.18	0.505	

Table S11. Data of the reactions using valine derived ligand *R* and predicted values.
Constitutional selectivity (b/l) and diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol) used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L18** is the optimum ligand for the reactions using valine derived ligand ***R***. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (b/l)			24 reactions (dr)			32 reactions (b/l)			32 reactions (dr)		
	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)
L1/1Et	0.46	0.535	0.598	1.13	1.187	1.198	0.46	0.574	0.610	1.13	1.188	1.232
L1/1Pr	0.6	0.535	0.497	1.465	1.519	1.530	0.6	0.574	0.563	1.465	1.476	1.484
L2/1Et	0.575	0.592	0.605	1.225	1.187	1.179	0.575	0.574	0.572	1.225	1.250	1.273
L2/1Pr	0.625	0.592	0.566	1.63	1.519	1.486	0.625	0.574	0.555	1.63	1.538	1.467
L3/1Et	1.025	0.986	0.978	1.185	1.187	1.187	1.025	0.983	0.977	1.185	1.235	1.299
L3/1Pr	0.99	0.986	0.989	1.485	1.519	1.526	0.99	0.983	0.989	1.485	1.480	1.475
L4/1Et	1.045	0.986	0.972	1.215	1.187	1.181	1.045	0.983	0.970	1.215	1.192	1.178
L4/1Pr	0.98	0.986	0.994	1.505	1.519	1.522	0.98	0.983	0.993	1.505	1.480	1.465
L5/1Et	0.905	0.896	0.876	1.535	1.483	1.464	0.905	0.894	0.846	1.535	1.492	1.475
L5/1Pr	0.945	0.929	0.898	1.795	1.815	1.826	0.945	0.894	0.863	1.795	1.780	1.775
L6/1Et	0.83	0.838	0.861	1.495	1.483	1.480	0.83	0.869	0.896	1.495	1.492	1.492
L6/1Pr	0.88	0.871	0.852	1.825	1.815	1.813	0.88	0.869	0.846	1.825	1.780	1.762
L7/1Et	-0.28	-0.140	-0.095	0.775	0.779	0.778	-0.28	-0.073	-0.056	0.775	0.796	0.803
L7/1Pr	-0.24	-0.140	-0.108	1.015	1.111	1.148	-0.24	-0.073	-0.060	1.015	1.084	1.111
L8/1Et	0.03	-0.140	-0.166	0.705	0.779	0.807	0.03	-0.073	-0.090	0.705	0.796	0.832
L8/1Pr	-0.03	-0.140	-0.157	1.23	1.111	1.060	-0.03	-0.073	-0.084	1.23	1.084	1.023
L9/1Et	-0.28	-0.141	-0.121	0.765	0.820	0.834	-0.28	-0.073	-0.056	0.765	0.842	0.862
L9/1Pr	-0.26	-0.141	-0.124	1.06	1.152	1.176	-0.26	-0.073	-0.058	1.06	1.130	1.148
L10/1Et	-0.06	-0.141	-0.172	0.93	0.820	0.779	-0.06	-0.073	-0.080	0.93	0.842	0.810
L10/1Pr	-0.06	-0.141	-0.172	1.19	1.152	1.141	-0.06	-0.073	-0.080	1.19	1.130	1.109
L11/1Et	-0.26	-0.234	-0.229	0.83	0.820	0.817	-0.26	-0.073	-0.058	0.83	0.842	0.845
L11/1Pr	-0.28	-0.234	-0.209	1.125	1.152	1.167	-0.28	-0.073	-0.056	1.125	1.130	1.131
L12/1Et	-0.03	-0.074	-0.120	0.725	0.857	1.013	-0.03	-0.055	-0.077	0.725	0.834	1.050
L12/1Pr	-0.12	-0.074	-0.030	1.32	1.189	1.128	-0.12	-0.055	-0.011	1.32	1.212	1.103
L13/1Et	0.95	0.929		1.42	1.483		0.95	1.123	1.311	1.42	1.305	1.226
L13/1Pr	1.38	0.929		1.475	1.815		1.38	1.123	0.981	1.475	1.593	1.725
L14/1Et	1.53	0.986		1.585	1.483		1.53	1.766	2.340	1.585	1.527	1.436
L14/1Pr	3	0.986		1.74	1.815		3	2.629	1.544	1.74	1.772	1.833
L15/1Et	1.965	0.929		0.72	1.483		1.965	2.073	2.197	0.72	0.669	0.601
L15/1Pr	2.315	0.929		0.88	1.815		2.315	2.073	1.947	0.88	0.957	1.052
L16/1Et	3	0.986		1.375	1.483		3	2.715	2.535	1.375	1.366	1.356
L16/1Pr	3	0.986		1.55	1.815		3	2.715	2.592	1.55	1.610	1.692
L17/1Et	3			0.26			3	2.054		0.26	0.632	
L17/1Pr	3			0.41			3	2.054		0.41	0.920	
L18/1Et	3			1.92			3	2.400		1.92	1.411	
L18/1Pr	3			1.92			3	2.400		1.92	1.699	

Details of the molecular design

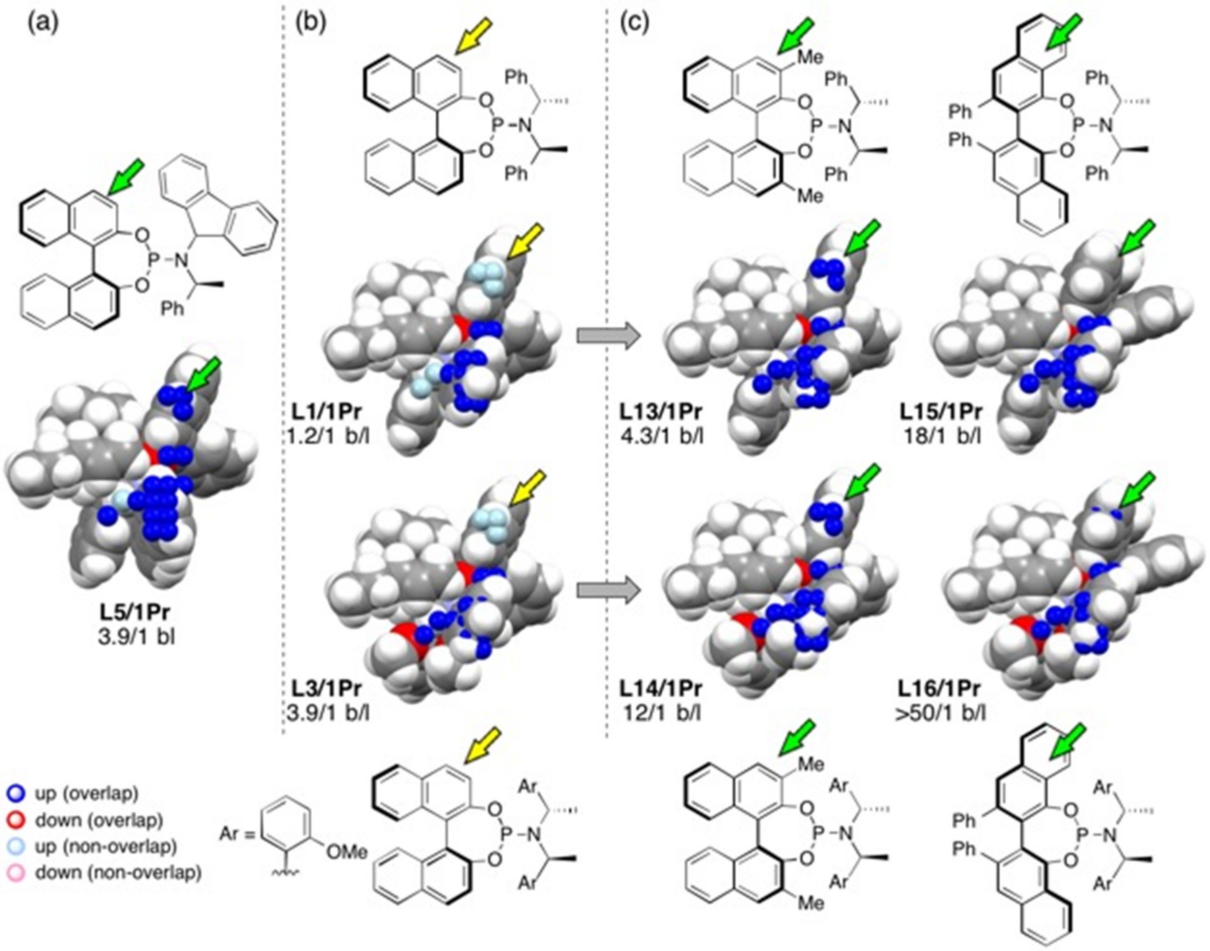


Fig. S23. Results of the MFA using b/l data obtained from the 24 reactions with valine ligand ***S***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. The structural information shown in this figure was extracted using Elastic Net (α = 0.5). The structural information shown in figs. S24 and S25 was extracted using LASSO.

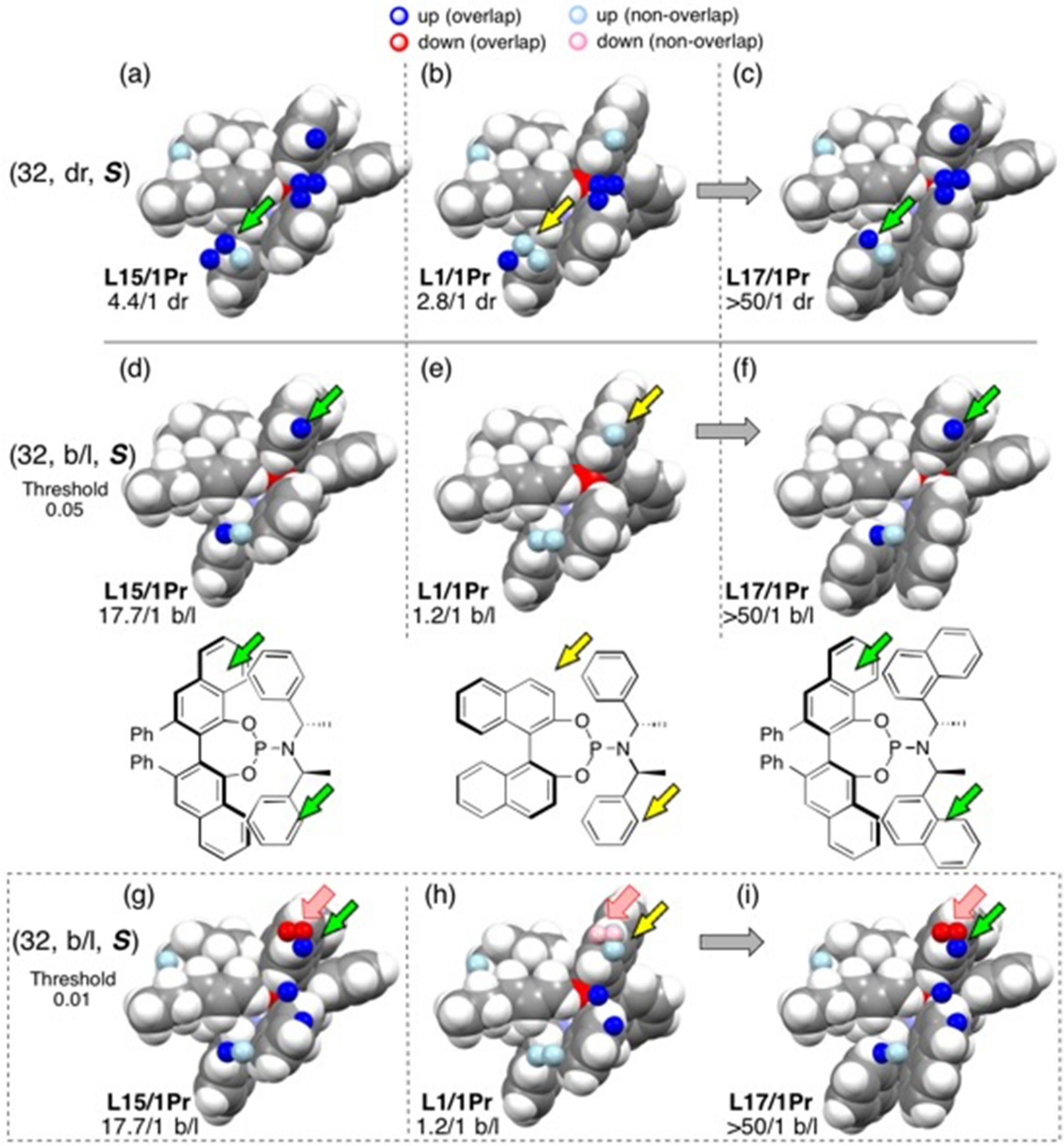


Fig. S24. Results of the MFA using b/l and dr data obtained from the 32 reactions with valine ligand ***S***. (a), (d), (g) The origin of the blue point used for the molecular design. (b), (e), (h) Design templates. (c), (f), (i) Designed molecules. Although the predicted b/l ratio of **L17/1Pr** ($\Delta\Delta G^{\ddagger}$ = 1.88 kcal/mol) is higher than that of the template molecule **L1/1Pr** ($\Delta\Delta G^{\ddagger}$ = 0.24 kcal/mol), **L17/1Pr** overlapped with red points as indicated by red arrow. Thus, for clarity, we set threshold value to 0.05 as shown in Fig. S24d, S24e, and S24f.

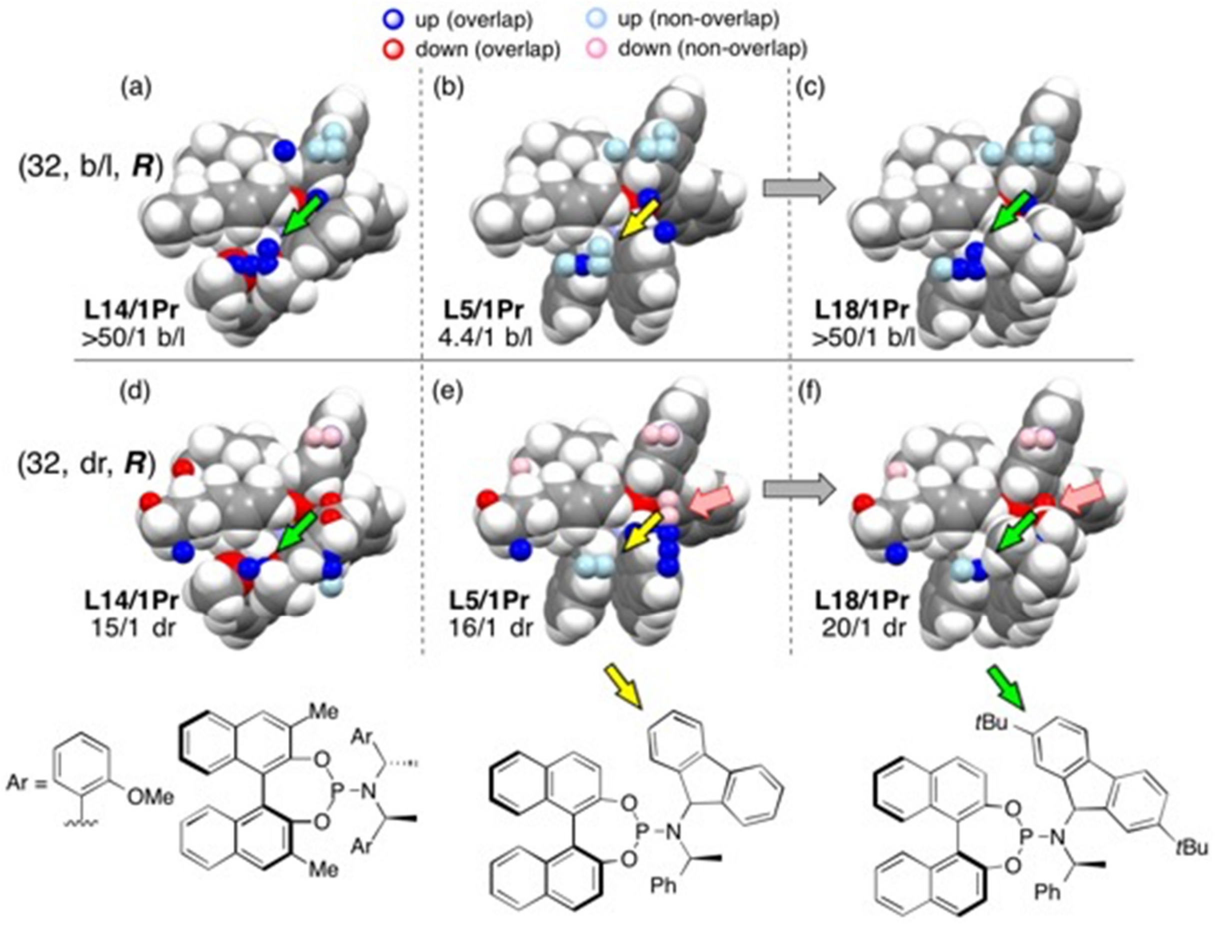


Fig. S25. Results of the MFA using b/l and dr data obtained from the 32 reactions with valine ligand ***R***. (a), (d) The origin of the blue point used for the molecular design. (b), (e) Design templates. (c), (f) Designed molecules. In the case of the MFA using dr data, **L18/1Pr** overlapped with red points as indicated by a red arrow (Fig. S25f) and the predicted value of **L18/1Pr** ($\Delta\Delta G^{\ddagger}$ = 1.7 kcal/mol) is lower than that of the design template **L5/1Pr** ($\Delta\Delta G^{\ddagger}$ = 1.78 kcal/mol) (Table S11). However, the signs of the correlation coefficients of the structural information with target variables (0.309, 0.309) are not consistent with those of the regression coefficients (-0.108, -0.03), meaning low reliability of the structural information (the red points) for the design (see, descriptor 2 of R32dr in text_test.xlsx. The parameters are highlighted by yellow). In the MFA using descriptor 1, all the signs of the regression coefficients are consistent with those of correlation coefficients. Thus, in this case, we employed the information extracted by the MFA using descriptor 1 for the molecular design.

MFA using enantioselectivity data

In this study, we mainly focused on b/l ratio and dr data since enantiomeric excess values in the initial training datasets are high (90% ee–99% ee in the 24 reactions using ligand ***S*** and 92% ee–99% ee in the 24 reactions using ligand ***R***). However, we also performed MFA using the enantioselectivity data to keep enantioselectivity excellent. We checked that, compared to the template molecules, the intermediate structures from the designed ligands did not overlap with the further structural information that lowered the enantioselectivity or if the structure overlapped with the structural information corresponding to negative regression coefficients, the decrease of the enantioselectivity was canceled by overlapping with the important structural information corresponding to positive regression coefficients. (figs. S27-S29). We also checked predicted $\Delta\Delta G^{\ddagger}$ values of all the designed molecules are over 2.9 kcal/mol [>97.8% ee] (Table S12). The information described above also strongly prompted us to synthesize the designed ligands, meaning that the MFA using enantioselectivity data has been also the powerful support to achieve the data-driven optimization of the catalyst structures for stereodivergent asymmetric synthesis.

Plots of the measured vs predicted values

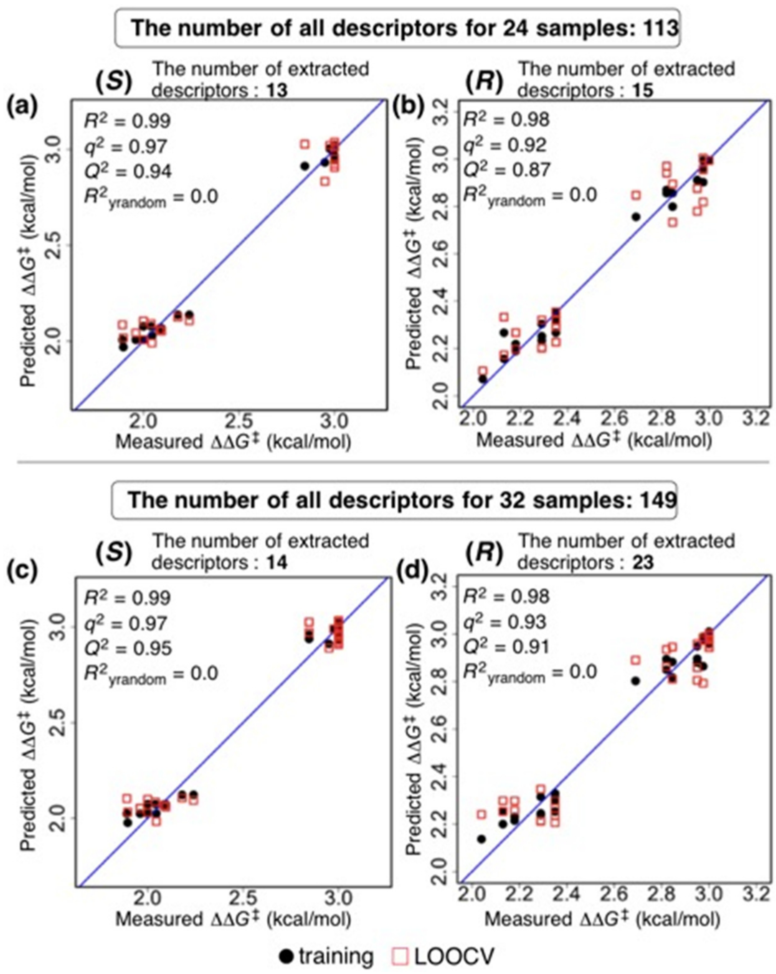


Fig. S26. Results of the MFA using enantioselectivity data. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

Table S12. Enantioselectivity data and predicted values.

Enantioselectivity of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA were highlighted by green. The 2nd MFA was performed including the designed ligands. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (*S*)			**24 reactions (*R*)**			**32 reactions (*S*)**			**32 reactions (*R*)**		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Et	2.95	2.932	2.834	2.69	2.756	2.848	2.95	2.911	2.890	2.69	2.791	2.880
L1/1Pr	**2.845**	**2.913**	3.028	2.845	2.799	2.733	**2.845**	**2.960**	3.025	2.845	2.813	2.787
L2/1Et	3	2.942	2.907	2.82	2.870	2.941	3	2.940	2.908	2.82	2.853	2.883
L2/1Pr	3	3.002	3.022	2.975	2.903	2.819	3	2.988	2.983	2.975	2.875	2.807
L3/1Et	3	2.965	2.952	2.975	2.963	2.954	3	2.975	2.965	2.975	2.970	2.968
L3/1Pr	**3**	**3.025**	3.038	2.975	2.996	3.005	3	3.023	3.033	2.975	2.992	3.000
L4/1Et	3	2.965	2.952	2.975	2.963	2.958	3	2.975	2.965	2.975	2.970	2.965
L4/1Pr	3	3.025	3.038	3	2.996	2.994	3	3.023	3.033	3	2.992	2.988
L5/1Et	3	2.945	2.924	2.95	2.912	2.877	3	2.938	2.920	2.95	2.924	2.861
L5/1Pr	3	3.004	3.011	2.845	2.856	2.895	3	2.987	2.985	**2.845**	**2.864**	2.958
L6/1Et	3	2.945	2.906	2.95	2.912	2.780	3	2.938	2.920	2.95	2.879	2.787
L6/1Pr	2.975	3.004	3.021	2.82	2.856	2.970	2.975	2.987	2.990	2.82	2.892	2.967
L7/1Et	1.89	2.007	2.085	2.04	2.071	2.105	1.89	2.024	2.103	2.04	2.089	2.151
L7/1Pr	2.24	2.136	2.106	2.13	2.267	2.333	2.24	2.122	2.093	2.13	2.260	2.320
L8/1Et	2.04	2.077	2.090	2.29	2.234	2.201	2.04	2.074	2.085	2.29	2.237	2.208
L8/1Pr	2.18	2.136	2.124	2.35	2.267	2.227	2.18	2.122	2.107	2.35	2.260	2.218
L9/1Et	2	2.007	2.007	2.13	2.157	2.173	2	2.024	2.030	2.13	2.170	2.195
L9/1Pr	2.085	2.066	2.058	2.18	2.189	2.197	2.085	2.072	2.067	2.18	2.192	2.202
L10/1Et	2	2.077	2.105	2.35	2.320	2.290	2	2.074	2.098	2.35	2.318	2.291
L10/1Pr	2.18	2.136	2.124	2.35	2.353	2.355	2.18	2.122	2.107	2.35	2.341	2.335
L11/1Et	1.895	1.969	2.016	2.18	2.219	2.267	1.895	1.976	2.032	2.18	2.225	2.275
L11/1Pr	2.045	2.028	1.990	2.29	2.252	2.204	2.045	2.025	1.983	2.29	2.247	2.196
L12/1Et	1.96	2.007	2.043	2.29	2.304	2.321	1.96	2.024	2.053	2.29	2.310	2.332
L12/1Pr	2.095	2.066	2.052	2.35	2.337	2.317	2.095	2.072	2.059	2.35	2.332	2.311
L13/1Et	2.845	3.023		2.95	2.813		2.845	2.938	2.971	2.95	2.941	2.936
L13/1Pr	**3**	**3.082**		3	2.846		3	2.987	2.985	3	2.964	2.940
L14/1Et	3	3.033		3	2.927		3	2.966	2.956	3	2.987	2.973
L14/1Pr	**3**	**3.093**		3	2.960		3	3.015	3.023	3	3.009	3.022
L15/1Et	3	3.023		3	2.813		3	2.950	2.931	3	2.976	2.937
L15/1Pr	**3**	**3.082**		3	2.846		3	2.998	3.006	3	2.998	3.005
L16/1Et	3	3.033		3	2.927		3	2.978	2.962	3	3.004	3.009
L16/1Pr	**3**	**3.093**		3	2.960		3	3.015	3.032	3	3.026	3.047
L17/1Et	3			3			3	2.950		3	2.891	
L17/1Pr	2.5			3			**3**	**2.998**		3	2.913	
L18/1Et	3			3			3	2.966		3	2.905	
L18/1Pr	3			3			3	3.015		**3**	**2.927**	

Details of the support of the molecular design through the MFA using enantioselectivity data

We designed the molecules based on the MFA of b/l ratio and dr data as shown in Fig. 2 and figs S4-fig S6. In figs S27-S29, we show the structural information visualized by the MFA of the enantioselectivity data along with the template and designed molecules shown in figs S4-S6. Compared to the template molecules, the intermediate structures from the designed ligands did not overlap with the further structural information that lowered the enantioselectivity or if the structure overlapped with the structural information corresponding to negative regression coefficients, the decrease of the enantioselectivity was canceled by overlapping with the important structural information corresponding to positive regression coefficients. As shown in Table S12, predicted $\Delta\Delta G^{\ddagger}$ values of the designed molecules were higher than those of the template molecules. All the MFAs in this section were performed using LASSO.

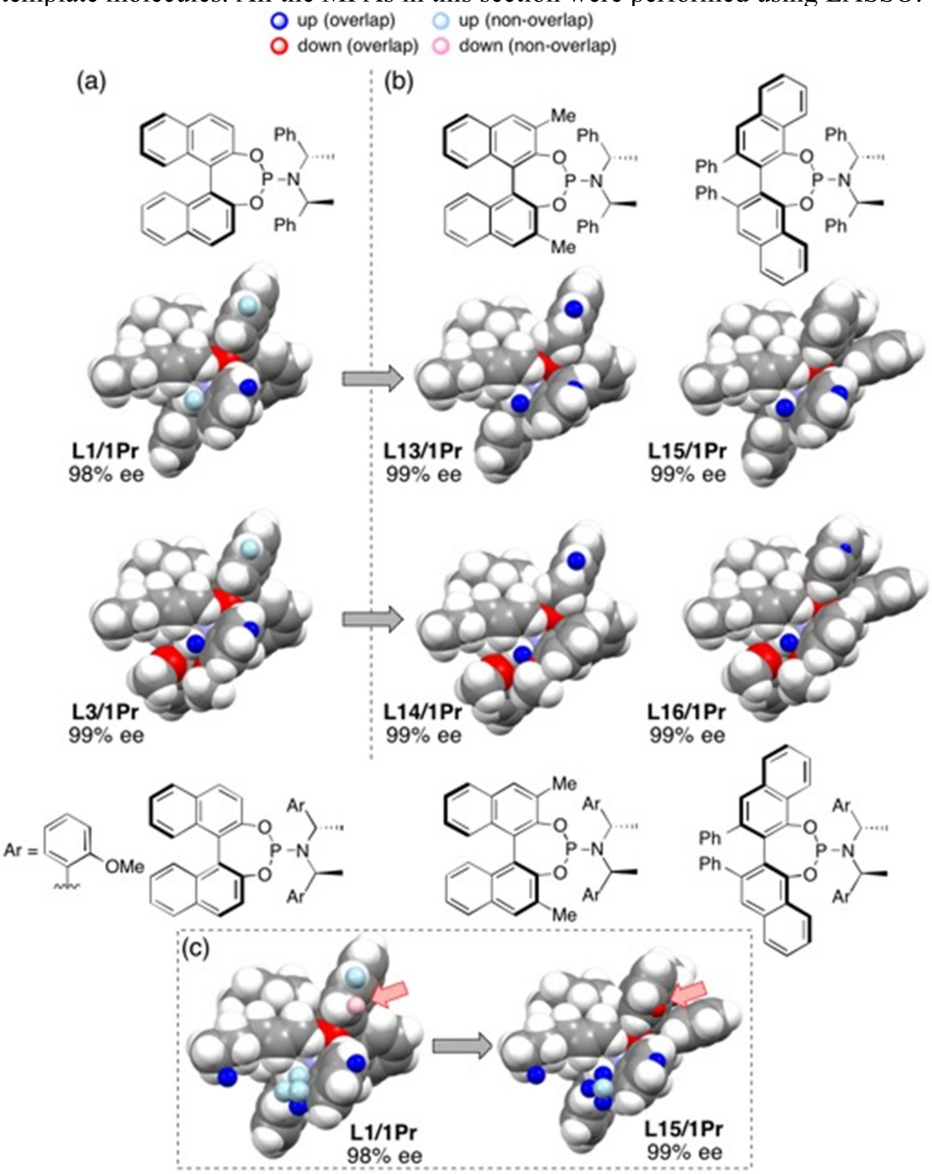


Fig. S27. Results of the MFA using enantioselectivity data obtained from the 24 reactions with valine ligand ***S***. (a) Design templates. (b) Designed molecules. The structural information from coefficients with absolute values of <0.075 was not visualized. In fig. S27c, we also show the structural information from coefficient with absolute values of < 0.01. While a red point was identified as indicated by red arrows, the decrease of the enantioselectivity was canceled by overlapping with the important structural information corresponding to positive regression coefficients as shown in figs. S27a and S27b. Thus, we employed the threshold value of 0.075 for the sake of clarity.

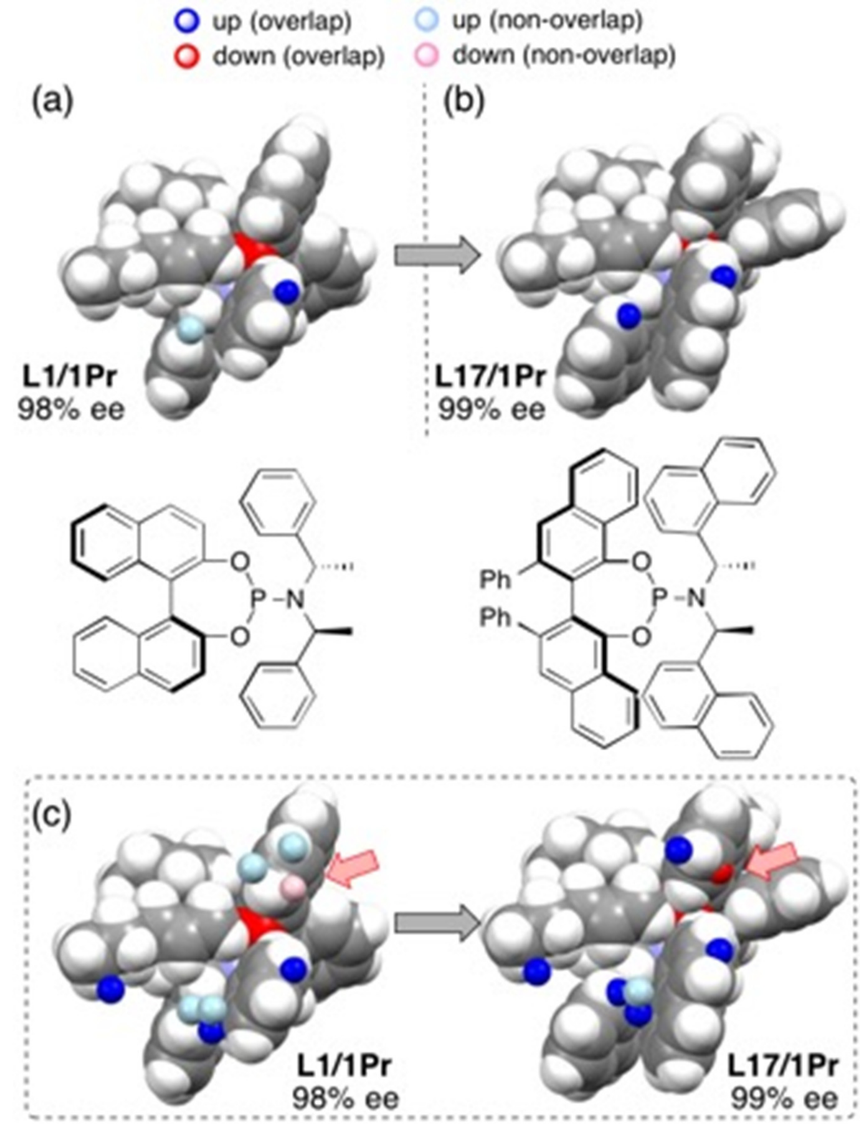


Fig. S28. Results of the MFA using enantioselecitivity data obtained from the 32 reactions with valine ligand ***S***. (a) Design templates. (b) Designed molecules. The structural information from coefficients with absolute values of <0.055 was not visualized. In fig. S28c, we also show the structural information from coefficient with absolute values of < 0.01. While a red point was identified as indicated by red arrows, the decrease of the enantioselectivity was canceled by overlapping with the important structural information corresponding to positive regression coefficients as shown in figs. S28a and S28b. Thus, we employed the threshold value of 0.055 for the sake of clarity.

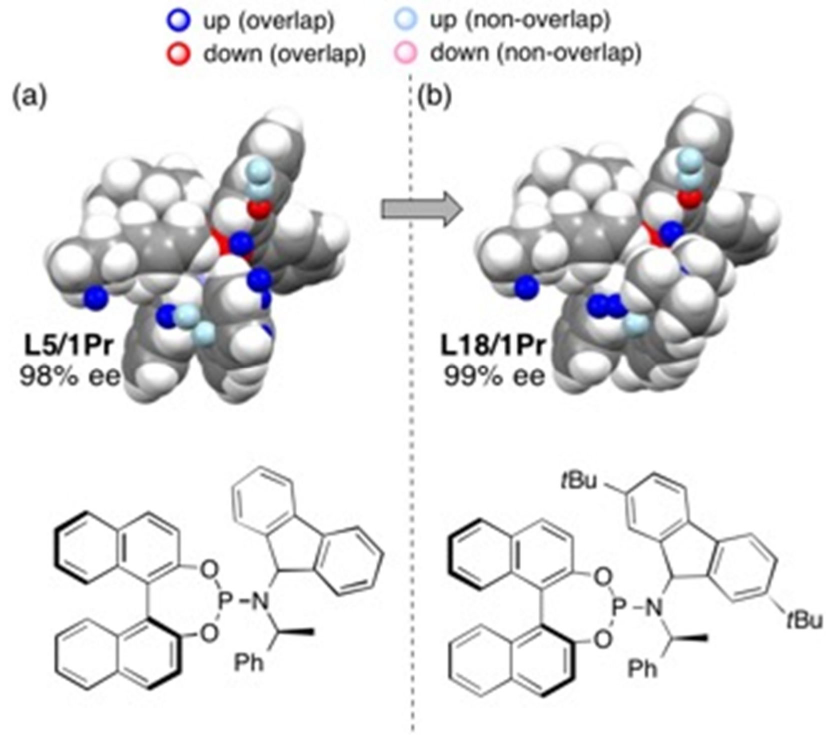
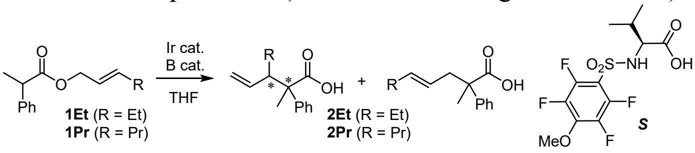


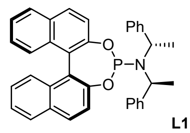
Fig. S29. Results of the MFA using b/l and dr data obtained from the 32 reactions with valine ligand ***R***. (a) Design templates. (b) Designed molecules. The structural information from coefficients with absolute values of <0.01 was not visualized.

MFA using dr data of (2*R*, 3*R*)-2/(2*S*, 3*R*)-2

Since the enantioselectivity of this reaction system is excellent, we employed dr data measured by NMR. We also checked the quality of the regression models generated through analysis of dr obtained from NMR and enantiomeric ratios (more accurate dr of (2*R*, 3*R*)-**2**/(2*S*, 3*R*)-**2**) as shown on page S145-S159. (S148~ MFA using descriptor 1, S151~ MFA using descriptor 2, S154~ MFA using test set, and S157~ MFA in another design pathway.). The results are almost identical with those obtained through analysis of dr data measured by NMR.

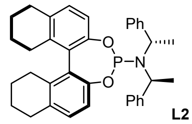
Table S13. Enantioselectivity data of the minor diastereomers in the reactions using valine derived ligand ***S*** (Reaction conditions and enantioselectivity data of the major diastereomers are shown in Table S1.). N.D. = not determined due to small peak size (calculated as a single enantiomer).





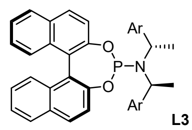
L1

2Et ***1st.*** 1.7/1 b/l, 1.9/1 dr, 77% yield, 80% ee
2nd. 1.7/1 b/l, 1.9/1 dr, 70% yield, 82% ee
2Pr ***1st.*** 1.2/1 b/l, 2.8/1 dr, 87% yield, 80% ee
2nd. 1.2/1 b/l, 2.8/1 dr, 77% yield, 81% ee



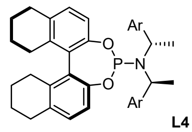
L2

2Et ***1st.*** 1.9/1 b/l, 2.3/1 dr, 73% yield, 80% ee
2nd. 1.8/1 b/l, 2.3/1 dr, 57% yield, 83% ee
2Pr ***1st.*** 1.3/1 b/l, 3.2/1 dr, 81% yield, 76% ee
2nd. 1.4/1 b/l, 3.3/1 dr, 65% yield, 76% ee



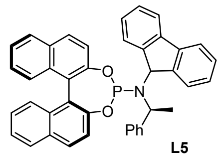
L3

2Et ***1st.*** 5.0/1 b/l, 4.5/1 dr, 50% yield, 80% ee
2nd. 5.0/1 b/l, 4.9/1 dr, 66% yield, 81% ee
2Pr ***1st.*** 3.8/1 b/l, 7.3/1 dr, 54% yield, 65% ee
2nd. 4.0/1 b/l, 7.6/1 dr, 69% yield, 64% ee



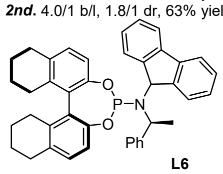
L4

2Et ***1st.*** 4.0/1 b/l, 4.6/1 dr, 57% yield, 75% ee
2nd. 4.7/1 b/l, 4.6/1 dr, 59% yield, 78% ee
2Pr ***1st.*** 3.2/1 b/l, 8.6/1 dr, 47% yield, 61% ee
2nd. 3.5/1 b/l, 8.3/1 dr, 46% yield, 59% ee



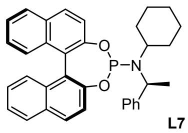
L5

2Et ***1st.*** 4.4/1 b/l, 1.1/1 dr, 68% yield, 87% ee
2nd. 4.6/1 b/l, 1.1/1 dr, 75% yield, 88% ee
2Pr ***1st.*** 3.7/1 b/l, 1.7/1 dr, 66% yield, 84% ee
2nd. 4.0/1 b/l, 1.8/1 dr, 63% yield, 86% ee



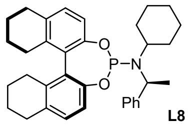
L6

2Et ***1st.*** 3.9/1 b/l, 1.0/1 dr, 72% yield, 88% ee
2nd. 3.6/1 b/l, 1.1/1 dr, 83% yield, 90% ee
2Pr ***1st.*** 3.4/1 b/l, 1.7/1 dr, 87% yield, 86% ee
2nd. 3.5/1 b/l, 1.6/1 dr, 82% yield, 81% ee



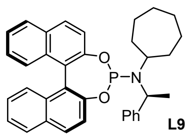
L7

2Et ***1st.*** 1.1/1 b/l, 1.1/1 dr, 84% yield, 77% ee
2nd. 1.1/1 b/l, 1.0/1 dr, 83% yield, 75% ee
2Pr ***1st.*** 1/1.2 b/l, 1.4/1 dr, 83% yield, 67% ee
2nd. 1/1.2 b/l, 1.3/1 dr, 72% yield, 64% ee



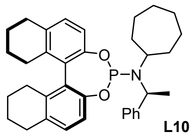
L8

2Et ***1st.*** 1.2/1 b/l, 1.4/1 dr, 78% yield, 77% ee
2nd. 1.3/1 b/l, 1.5/1 dr, 90% yield, 75% ee
2Pr ***1st.*** 1/1.3 b/l, 1.8/1 dr, 91% yield, 67% ee
2nd. 1/1.2 b/l, 1.7/1 dr, 70% yield, 62% ee



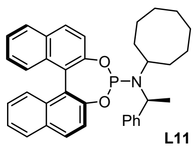
L9

2Et ***1st.*** 1.1/1 b/l, 1/1.1 dr, 83% yield, 82% ee
2nd. 1.2/1 b/l, 1/1.1 dr, 73% yield, 81% ee
2Pr ***1st.*** 1/1.2 b/l, 1.2/1 dr, 95% yield, 71% ee
2nd. 1/1.2 b/l, 1.2/1 dr, 81% yield, 70% ee



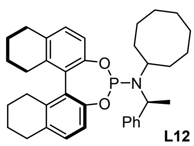
L10

2Et ***1st.*** 1.3/1 b/l, 1.1/1 dr, 88% yield, 80% ee
2nd. 1.2/1 b/l, 1.1/1 dr, 78% yield, 81% ee
2Pr ***1st.*** 1/1.3 b/l, 1.5/1 dr, 93% yield, 71% ee
2nd. 1/1.1 b/l, 1.6/1 dr, 83% yield, 70% ee



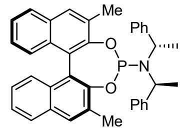
L11

2Et ***1st.*** 1.2/1 b/l, 1/1.1 dr, 100% yield, 91% ee
2nd. 1.2/1 b/l, 1/1.1 dr, 91% yield, 89% ee
2Pr ***1st.*** 1/1.2 b/l, 1.2/1 dr, 96% yield, 93% ee
2nd. 1/1.2 b/l, 1.2/1 dr, 84% yield, 91% ee



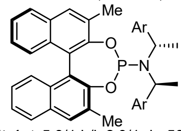
L12

2Et ***1st.*** 1.2/1 b/l, 1.1/1 dr, 84% yield, 82% ee
2nd. 1.2/1 b/l, 1.2/1 dr, 80% yield, 82% ee
2Pr ***1st.*** 1/1.3 b/l, 1.5/1 dr, 85% yield, 74% ee
2nd. 1/1.2 b/l, 1.6/1 dr, 85% yield, 75% ee



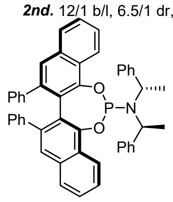
L13

2Et ***1st.*** 2.8/1 b/l, 1.7/1 dr, 68% yield, 81% ee
2nd. 3.3/1 b/l, 2.0/1 dr, 67% yield, 87% ee
2Pr ***1st.*** 4.0/1 b/l, 2.9/1 dr, 65% yield, 62% ee
2nd. 4.6/1 b/l, 3.2/1 dr, 70% yield, 85% ee



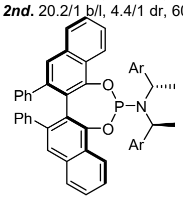
L14

2Et ***1st.*** 5.8/1 b/l, 2.9/1 dr, 56% yield, 91% ee
2nd. 5.9/1 b/l, 3.1/1 dr, 65% yield, 99% ee
2Pr ***1st.*** 11/1 b/l, 6.5/1 dr, 65% yield, 85% ee
2nd. 12/1 b/l, 6.5/1 dr, 64% yield, N.D.



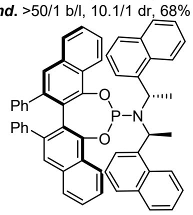
L15

2Et ***1st.*** 22.4/1 b/l, 3.9/1 dr, 44% yield, 93% ee
2nd. 11.4/1 b/l, 3.1/1 dr, 60% yield, 95% ee
2Pr ***1st.*** 15.2/1 b/l, 4.3/1 dr, 73% yield, 94% ee
2nd. 20.2/1 b/l, 4.4/1 dr, 60% yield, 95% ee



L16

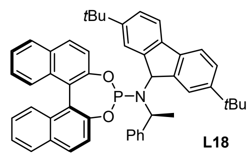
2Et ***1st.*** >50/1 b/l, 4.5/1 dr, 56% yield, 92% ee
2nd. >50/1 b/l, 5.4/1 dr, 52% yield, 99% ee
2Pr ***1st.*** >50/1 b/l, 9.4/1 dr, 51% yield, 92% ee
2nd. >50/1 b/l, 10.1/1 dr, 68% yield, 93% ee



Optimum ligand

L17

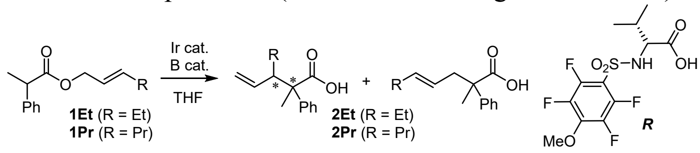
2Et 17/1 b/l, 12.5/1 dr, 72% yield, N.D.
2Pr >50/1 b/l, 56.4/1 dr, 84% yield, N.D.

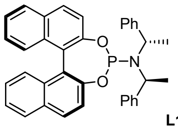


L18

2Et >50/1 b/l, 2.8/1 dr, 55% yield, N.D.
2Pr >50/1 b/l, 6.3/1 dr, 57% yield, N.D.

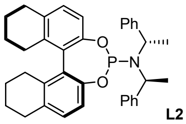
Table S14. Enantioselectivity data of the minor diastereomers in the reactions using valine derived ligand ***R*** (Reaction conditions and enantioselectivity data of the major diastereomers are shown in Table S2.). N.D. = not determined due to small peak size (calculated as a single enantiomer).





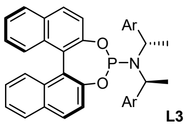
L1

2Et ***1st.*** 2.0/1 b/l, 5.2/1 dr, 94% yield, 88% ee
2nd. 2.1/1 b/l, 6.5/1 dr, 85% yield, 94% ee
2Pr ***1st.*** 2.4/1 b/l, 9.6/1 dr, 100% yield, 99% ee
2nd. 2.7/1 b/l, 10.0/1 dr, 77% yield, N.D.



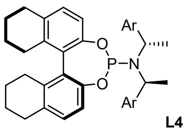
L2

2Et ***1st.*** 2.4/1 b/l, 6.1/1 dr, 97% yield, 99% ee
2nd. 2.5/1 b/l, 7.4/1 dr, 76% yield, 98% ee
2Pr ***1st.*** 2.6/1 b/l, 11.3/1 dr, 100% yield, N.D.
2nd. 2.7/1 b/l, 14.1/1 dr, 94% yield, N.D.



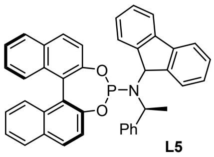
L3

2Et ***1st.*** 4.6/1 b/l, 6.7/1 dr, 70% yield, 99% ee
2nd. 5.3/1 b/l, 6.0/1 dr, 74% yield, 98% ee
2Pr ***1st.*** 4.5/1 b/l, 10.4/1 dr, 65% yield, N.D.
2nd. 4.8/1 b/l, 9.8/1 dr, 85% yield, N.D.



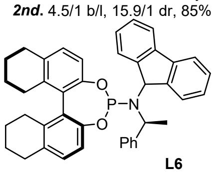
L4

2Et ***1st.*** 4.8/1 b/l, 6.5/1 dr, 81% yield, 99% ee
2nd. 5.4/1 b/l, 6.8/1 dr, 72% yield, 99% ee
2Pr ***1st.*** 4.0/1 b/l, 10.3/1 dr, 56% yield, N.D.
2nd. 5.3/1 b/l, 10.5/1 dr, 84% yield, N.D.



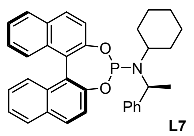
L5

2Et ***1st.*** 4.0/1 b/l, 11.0/1 dr, 83% yield, 99% ee
2nd. 4.2/1 b/l, 10.8/1 dr, 83% yield, N.D.
2Pr ***1st.*** 4.2/1 b/l, 16.7/1 dr, 95% yield, 99% ee
2nd. 4.5/1 b/l, 15.9/1 dr, 85% yield, N.D.



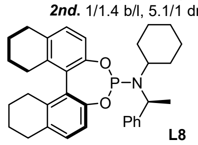
L6

2Et ***1st.*** 3.9/1 b/l, 10.6/1 dr, 100% yield, 91% ee
2nd. 3.4/1 b/l, 9.9/1 dr, 96% yield, 99% ee
2Pr ***1st.*** 3.8/1 b/l, 16.4/1 dr, 96% yield, N.D.
2nd. 4.1/1 b/l, 17.7/1 dr, 98% yield, N.D.



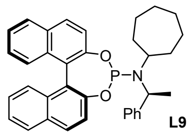
L7

2Et ***1st.*** 1/1.5 b/l, 3.2/1 dr, 87% yield, 41% ee
2nd. 1/1.6 b/l, 3.5/1 dr, 68% yield, 70% ee
2Pr ***1st.*** 1/1.5 b/l, 4.6/1 dr, 82% yield, 46% ee
2nd. 1/1.4 b/l, 5.1/1 dr, 66% yield, N.D.



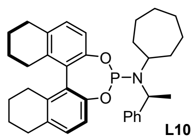
L8

2Et ***1st.*** 1.0/1 b/l, 3.6/1 dr, 87% yield, 49% ee
2nd. 1.1/1 b/l, 2.5/1 dr, 98% yield, 49% ee
2Pr ***1st.*** 1/1.1 b/l, 6.2/1 dr, 98% yield, 45% ee
2nd. 1/1.0 b/l, 7.5/1 dr, 86% yield, N.D.



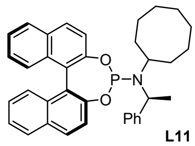
L9

2Et ***1st.*** 1/1.5 b/l, 2.7/1 dr, 83% yield, 48% ee
2nd. 1/1.6 b/l, 4.0/1 dr, 84% yield, 45% ee
2Pr ***1st.*** 1/1.5 b/l, 4.6/1 dr, 95% yield, 54% ee
2nd. 1/1.5 b/l, 5.9/1 dr, 85% yield, 40% ee



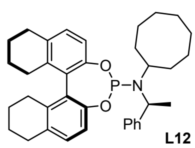
L10

2Et ***1st.*** 1/1.1 b/l, 3.8/1 dr, 88% yield, 43% ee
2nd. 1/1.1 b/l, 5.2/1 dr, 80% yield, 46% ee
2Pr ***1st.*** 1/1.2 b/l, 6.7/1 dr, 100% yield, 35% ee
2nd. 1/1.0 b/l, 6.4/1 dr, 86% yield, N.D.



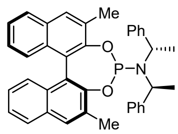
L11

2Et ***1st.*** 1/1.4 b/l, 2.7/1 dr, 86% yield, 52% ee
2nd. 1/1.6 b/l, 4.9/1 dr, 87% yield, 50% ee
2Pr ***1st.*** 1/1.6 b/l, 5.4/1 dr, 85% yield, 72% ee
2nd. 1/1.5 b/l, 6.2/1 dr, 84% yield, N.D.



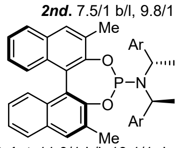
L12

2Et ***1st.*** 1/1.1 b/l, 3.8/1 dr, 88% yield, 49% ee
2nd. 1.0/1 b/l, 2.5/1 dr, 100% yield, 52% ee
2Pr ***1st.*** 1/1.2 b/l, 6.7/1 dr, 100% yield, 35% ee
2nd. 1/1.2 b/l, 9.1/1 dr, 95% yield, N.D.



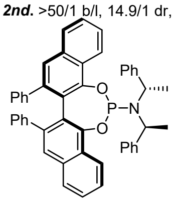
L13

2Et ***1st.*** 4.1/1 b/l, 9.9/1 dr, 89% yield, 99% ee
2nd. 4.7/1 b/l, 8.5/1 dr, 80% yield, 99% ee
2Pr ***1st.*** 9.9/1 b/l, 10.0/1 dr, 81% yield, N.D.
2nd. 7.5/1 b/l, 9.8/1 dr, 64% yield, N.D.



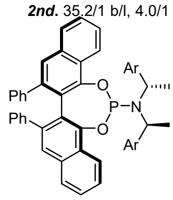
L14

2Et ***1st.*** 11.0/1 b/l, 13,1/1 dr, 77% yield, 99% ee
2nd.10.7/1 b/l, 10.6/1 dr, 93% yield, 94% ee
2Pr ***1st.*** >50/1 b/l, 15.2/1 dr, 80% yield, N.D.
2nd. >50/1 b/l, 14.9/1 dr, 88% yield, 93% ee



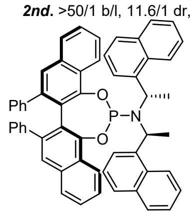
L15

2Et ***1st.*** 19.8/1 b/l, 3.0/1 dr, 85% yield, 99% ee
2nd. 22.8/1 b/l, 3.1/1 dr, 73% yield, 99% ee
2Pr ***1st.*** 38.5/1 b/l, 3.9/1 dr, 87% yield, N.D.
2nd. 35.2/1 b/l, 4.0/1 dr, 84% yield, N.D.



L16

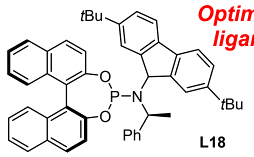
2Et ***1st.*** >50/1 b/l, 8.2/1 dr, 69% yield, 99% ee
2nd. >50/1 b/l, 8.8/1 dr, 76% yield, 99% ee
2Pr ***1st.*** >50/1 b/l, 10.9/1 dr, 71% yield, N.D.
2nd. >50/1 b/l, 11.6/1 dr, 80% yield, N.D.



L17

2Et >50/1 b/l, 1.5/1 dr, 77% yield, N.D.
2Pr >50/1 b/l, 1.9/1 dr, 85% yield, N.D.

Optimum ligand



L18

2Et >50/1 b/l, 19.8/1 dr, 91% yield, N.D.
2Pr >50/1 b/l, 19.9/1 dr, 97% yield, N.D.

Table S15. Experimental and predicted values of dr data determined by NMR and enantioselectivity data. (MFA using descriptor1)

Diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** and **L18** are the optimum ligand for the reactions using valine derived ligands ***S*** and ***R***, respectively. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (*S*, dr)			**24 reactions (*R*, dr)**			**32 reactions (*S*, dr)**			**32 reactions (*R*, dr)**		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Et	0.470	0.492	0.528	1.154	1.204	1.217	0.470	0.497	0.607	1.154	1.222	1.256
L1/1Pr	0.721	0.695	0.662	1.461	1.476	1.481	**0.721**	**0.720**	0.721	1.461	1.469	1.476
L2/1Et	0.594	0.611	0.636	1.221	1.248	1.304	0.594	0.592	0.591	1.221	1.262	1.306
L2/1Pr	0.836	0.813	0.787	1.627	1.52	1.448	0.836	0.816	0.795	1.627	1.510	1.437
L3/1Et	1.056	1.053	1.205	1.185	1.204	1.211	1.056	1.032	0.986	1.185	1.222	1.235
L3/1Pr	1.412	1.408	1.487	1.480	1.476	1.476	1.412	1.445	1.476	1.480	1.469	1.466
L4/1Et	1.057	1.054	1.291	1.215	1.204	1.201	1.057	1.072	1.257	1.215	1.222	1.223
L4/1Pr	1.510	1.490	1.393	1.502	1.476	1.470	1.510	1.461	1.423	1.502	1.469	1.457
L5/1Et	0.099	0.102	0.108	1.531	1.495	1.485	0.099	0.104	0.115	1.531	1.491	1.472
L5/1Pr	0.406	0.402	0.395	1.786	1.767	1.765	0.406	0.394	0.375	**1.786**	**1.745**	1.725
L6/1Et	0.064	0.074	0.078	1.504	1.495	1.496	0.064	0.087	0.096	1.504	1.491	1.486
L6/1Pr	0.372	0.375	0.382	1.813	1.767	1.754	0.372	0.377	0.389	1.813	1.738	1.697
L7/1Et	0.080	0.074	0.070	0.912	0.941	0.947	0.080	0.087	0.089	0.912	0.945	0.954
L7/1Pr	0.295	0.412	0.471	1.084	1.213	1.261	0.295	0.413	0.47	1.084	1.193	1.233
L8/1Et	0.294	0.210	0.164	0.887	0.941	0.957	0.294	0.189	0.134	0.887	0.945	0.964
L8/1Pr	0.464	0.412	0.388	1.314	1.213	1.166	0.464	0.413	0.389	1.314	1.193	1.141
L9/1Et	-0.018	0.000	0.008	0.960	1.01	1.030	-0.018	0.010	0.023	0.960	1.028	1.053
L9/1Pr	0.203	0.202	0.199	1.246	1.282	1.297	0.203	0.233	0.258	1.246	1.275	1.287
L10/1Et	0.096	0.136	0.171	1.136	1.01	0.968	0.096	0.112	0.124	1.136	1.028	0.989
L10/1Pr	0.363	0.338	0.321	1.290	1.282	1.279	0.363	0.335	0.317	1.290	1.275	1.270
L11/1Et	-0.039	-0.018	0.001	1.016	0.991	0.935	-0.039	-0.023	-0.015	1.016	0.992	0.946
L11/1Pr	0.182	0.185	0.181	1.157	1.263	1.307	0.182	0.201	0.214	1.157	1.239	1.289
L12/1Et	0.121	0.131	0.145	0.908	1.032	1.176	0.121	0.120	0.114	0.908	1.045	1.200
L12/1Pr	0.344	0.333	0.272	1.425	1.303	1.246	0.344	0.338	0.308	1.425	1.292	1.208
L13/1Et	0.442	0.314		1.422	1.495		0.442	0.513	0.591	1.422	1.328	1.257
L13/1Pr	0.804	0.516		1.469	1.767		0.804	0.737	0.661	1.469	1.576	1.693
L14/1Et	0.718	0.874		1.596	1.539		0.718	0.743	0.808	1.596	1.554	1.474
L14/1Pr	1.223	1.229		1.749	1.811		1.223	1.198	1.149	1.749	1.753	1.804
L15/1Et	0.821	0.356		0.716	1.495		0.821	0.784	0.728	0.716	0.732	0.692
L15/1Pr	0.959	0.558		0.879	1.767		0.959	0.967	1.003	0.879	0.920	1.057
L16/1Et	1.038	0.916		1.374	1.539		1.038	1.045	1.066	1.374	1.349	1.205
L16/1Pr	1.484	1.271		1.551	1.811		1.484	1.458	1.408	1.551	1.597	1.635
L17/1Et	1.619			0.257			1.619	0.784		0.257	0.689	
L17/1Pr	2.586			0.409			**2.586**	**1.008**		0.409	0.937	
L18/1Et	0.658			1.914			0.658	0.338		1.914	1.538	
L18/1Pr	1.179			1.917			1.179	0.561		**1.917**	**1.785**	

Plots of the measured vs predicted values

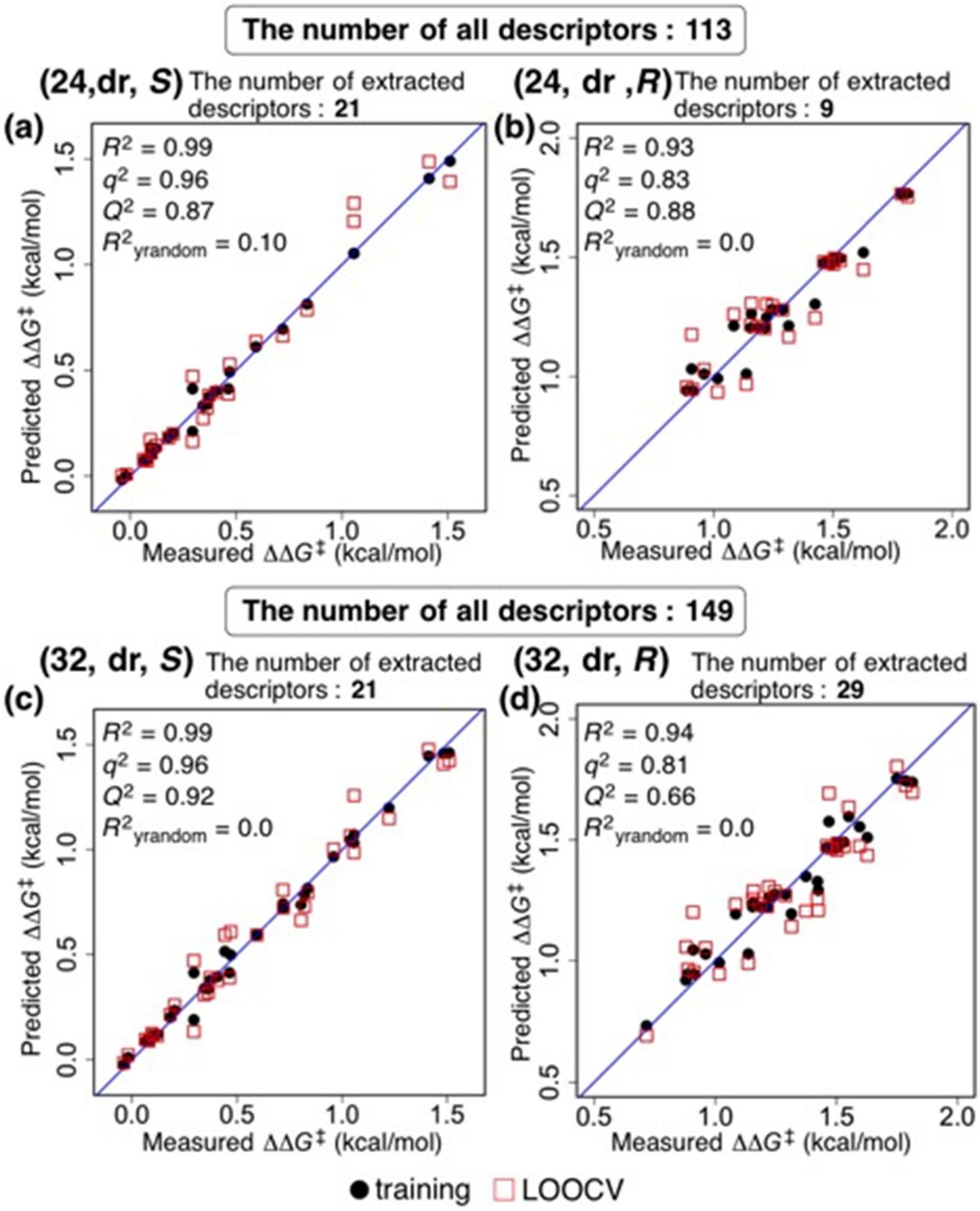


Fig. S30. Results of the MFA using descriptor1 and dr data determined by NMR and enantioselectivity data. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

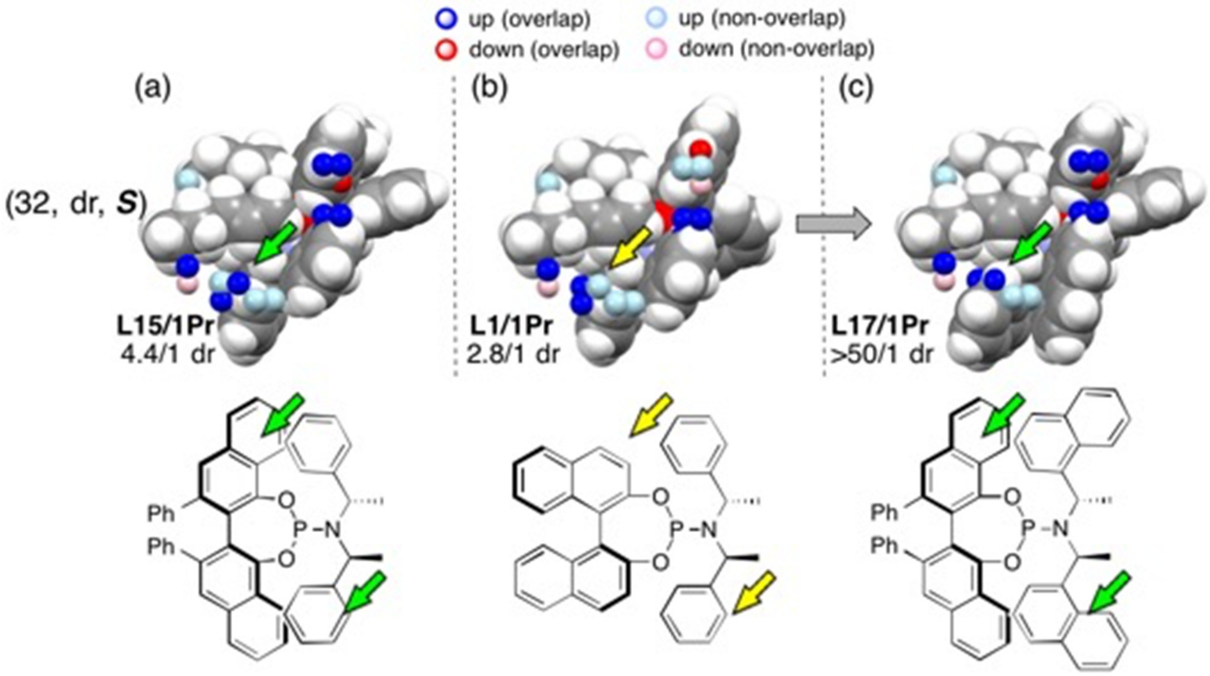


Fig. S31. Results of the MFA using dr data obtained from the 32 reactions with valine ligand ***S***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. Diastereomeric ratios determined by NMR are shown.

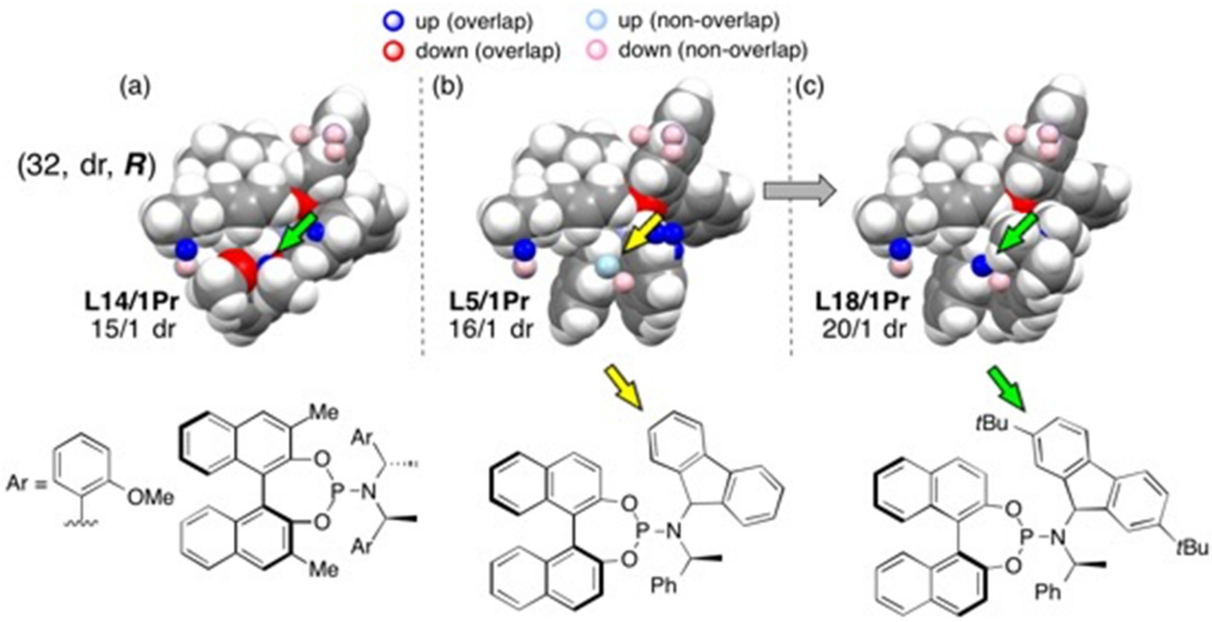


Fig. S32. Results of the MFA using dr data obtained from the 32 reactions with valine ligand ***R***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. Diastereomeric ratios determined by NMR are shown.

Table S16. Experimental and predicted values of dr data determined by NMR and enantioselectivity data. (MFA using descriptor2)

Diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** and **L18** are the optimum ligand for the reactions using valine derived ligands ***S*** and ***R***, respectively. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	24 reactions (*S*, dr)			24 reactions (*R*, dr)			32 reactions (*S*, dr)			32 reactions (*R*, dr)		
	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)	Exp.	Pred.	Pred. (CV)
L1/1Et	0.470	0.494	0.530	1.154	1.212	1.223	0.470	0.494	0.601	1.154	1.235	1.253
L1/1Pr	0.721	0.693	0.661	1.461	1.495	1.502	**0.721**	**0.728**	0.734	1.461	1.472	1.474
L2/1Et	0.594	0.613	0.641	1.221	1.212	1.210	0.594	0.588	0.583	1.221	1.245	1.268
L2/1Pr	0.836	0.812	0.783	1.627	1.495	1.457	0.836	0.822	0.808	1.627	1.482	1.429
L3/1Et	1.056	1.053	1.208	1.185	1.212	1.217	1.056	1.020	0.968	1.185	1.235	1.245
L3/1Pr	1.412	1.407	1.492	1.480	1.495	1.498	1.412	1.448	1.479	1.480	1.472	1.469
L4/1Et	1.057	1.053	1.294	1.215	1.212	1.211	1.057	1.072	1.254	1.215	1.235	1.238
L4/1Pr	1.510	1.491	1.393	1.502	1.495	1.494	1.510	1.467	1.432	1.502	1.472	1.463
L5/1Et	0.099	0.099	0.100	1.531	1.502	1.493	0.099	0.099	0.099	1.531	1.450	1.367
L5/1Pr	0.406	0.404	0.400	1.786	1.785	1.788	0.406	0.394	0.377	**1.786**	**1.743**	1.673
L6/1Et	0.064	0.067	0.076	1.504	1.502	1.504	0.064	0.078	0.097	1.504	1.450	1.424
L6/1Pr	0.372	0.372	0.376	1.813	1.785	1.777	0.372	0.373	0.372	1.813	1.687	1.600
L7/1Et	0.080	0.051	0.009	0.912	0.923	0.924	0.080	0.144	0.172	0.912	0.951	0.963
L7/1Pr	0.295	0.405	0.460	1.084	1.206	1.253	0.295	0.378	0.415	1.084	1.188	1.226
L8/1Et	0.294	0.206	0.154	0.887	0.923	0.934	0.294	0.144	0.080	0.887	0.951	0.973
L8/1Pr	0.464	0.405	0.375	1.314	1.206	1.158	0.464	0.378	0.342	1.314	1.188	1.135
L9/1Et	-0.018	-0.008	-0.003	0.960	0.996	1.005	-0.018	0.054	0.082	0.960	1.021	1.035
L9/1Pr	0.203	0.190	0.177	1.246	1.279	1.288	0.203	0.288	0.322	1.246	1.258	1.261
L10/1Et	0.096	0.147	0.195	1.136	0.996	0.958	0.096	0.054	0.033	1.136	1.021	0.991
L10/1Pr	0.363	0.346	0.331	1.290	1.279	1.276	0.363	0.288	0.254	1.290	1.258	1.250
L11/1Et	-0.039	0.010	0.060	1.016	0.996	0.989	-0.039	0.008	0.061	1.016	1.021	1.021
L11/1Pr	0.182	0.209	0.241	1.157	1.279	1.315	0.182	0.242	0.312	1.157	1.258	1.283
L12/1Et	0.121	0.132	0.146	0.908	1.025	1.164	0.121	0.109	0.097	0.908	1.051	1.211
L12/1Pr	0.344	0.330	0.313	1.425	1.308	1.255	0.344	0.343	0.342	1.425	1.287	1.235
L13/1Et	0.442	0.294		1.422	1.502		0.442	0.506	0.560	1.422	1.377	1.346
L13/1Pr	0.804	0.493		1.469	1.785		0.804	0.740	0.684	1.469	1.614	1.704
L14/1Et	0.718	0.890		1.596	1.502		0.718	0.762	0.804	1.596	1.524	1.460
L14/1Pr	1.223	1.245		1.749	1.785		1.223	1.190	1.151	1.749	1.761	1.794
L15/1Et	0.821	0.294		0.716	1.502		0.821	0.783	0.721	0.716	0.709	0.681
L15/1Pr	0.959	0.493		0.879	1.785		0.959	0.973	1.031	0.879	0.946	1.005
L16/1Et	1.038	0.890		1.374	1.502		1.038	1.039	1.046	1.374	1.386	1.363
L16/1Pr	1.484	1.245		1.551	1.785		1.484	1.467	1.452	1.551	1.624	1.725
L17/1Et	1.619			0.257			1.619	0.713		0.257	0.680	
L17/1Pr	2.586			0.409			**2.586**	**0.947**		0.409	0.917	
L18/1Et	0.658			1.914			0.658	0.418		1.914	1.443	
L18/1Pr	1.179			1.917			1.179	0.652		**1.917**	**1.680**	

Plots of the measured vs predicted values

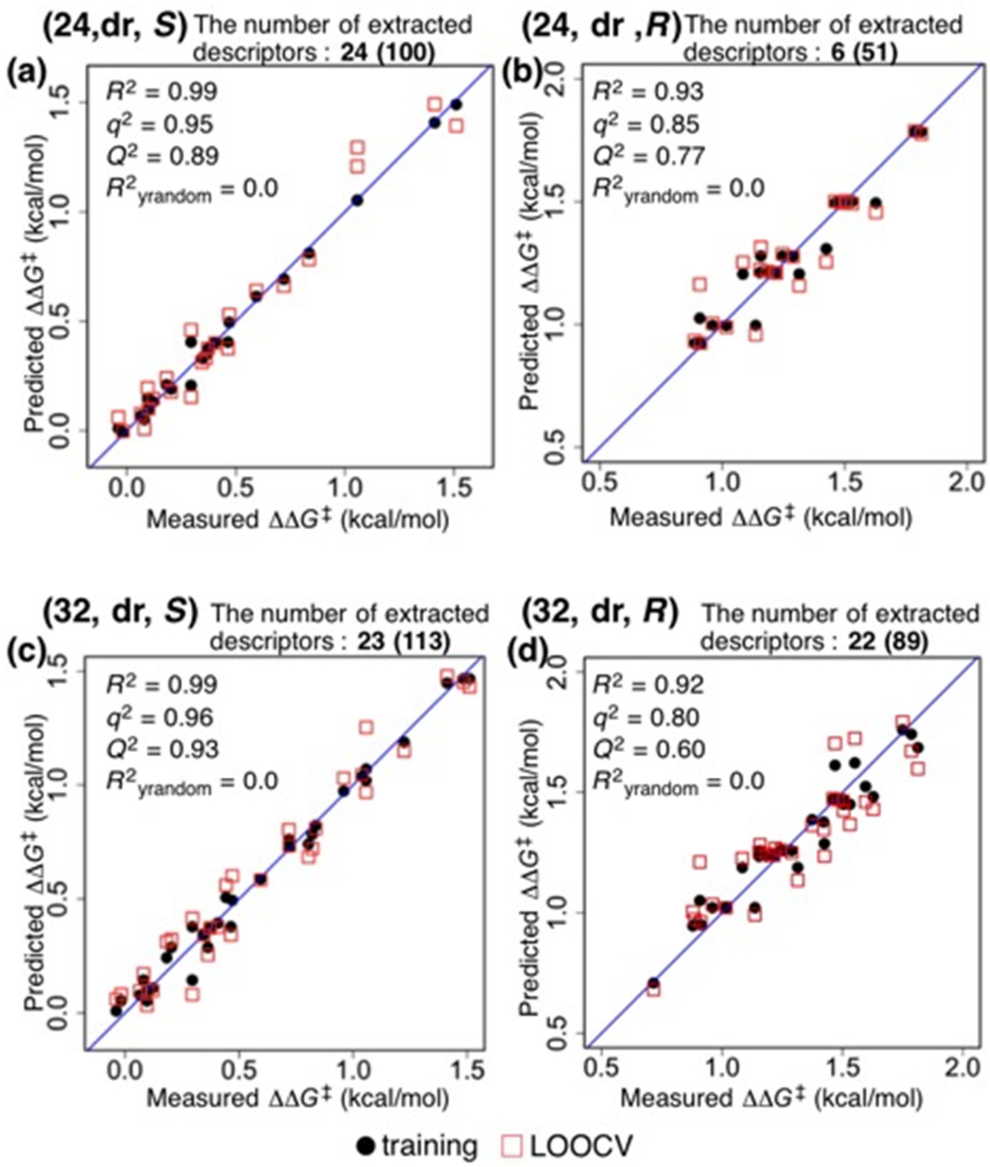


Fig. S33. Results of the MFA using descriptor 2 and dr data detemined by NMR and enantioselectivity data. (a)–(d) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. The number in parenthesis is the number of all descriptors. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x=y line.

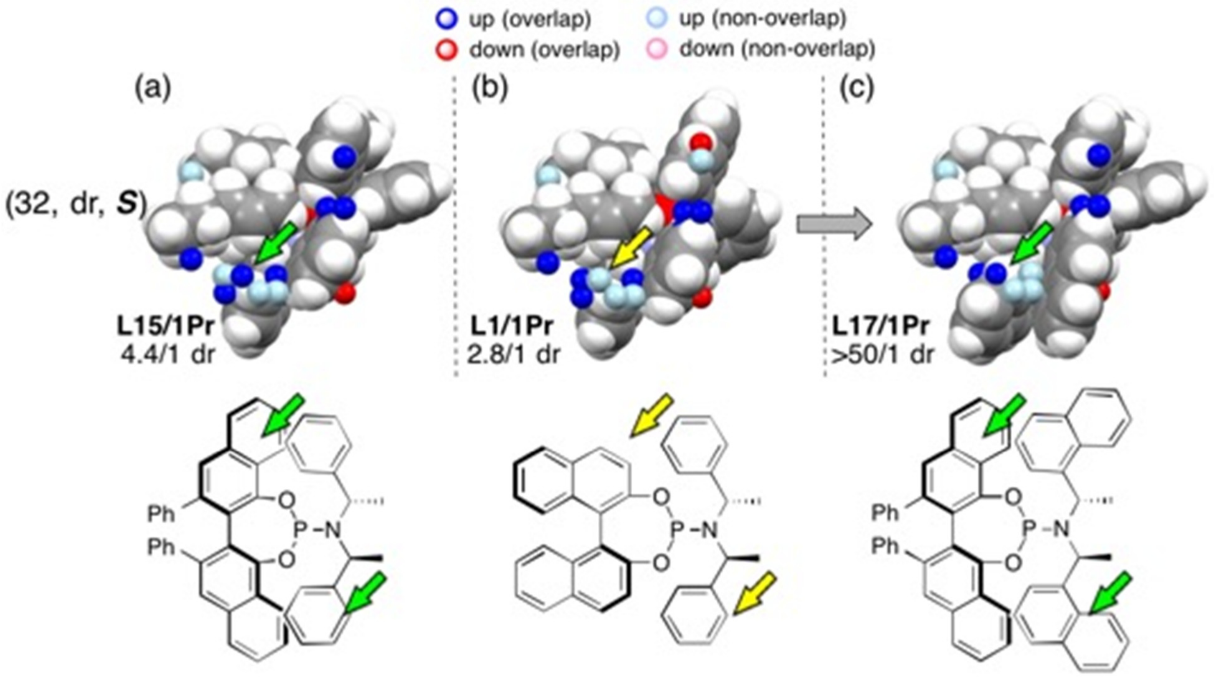


Fig. S34. Results of the MFA using dr data obtained from the 32 reactions with valine ligand ***S***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. Diastereomeric ratios determined by NMR are shown.

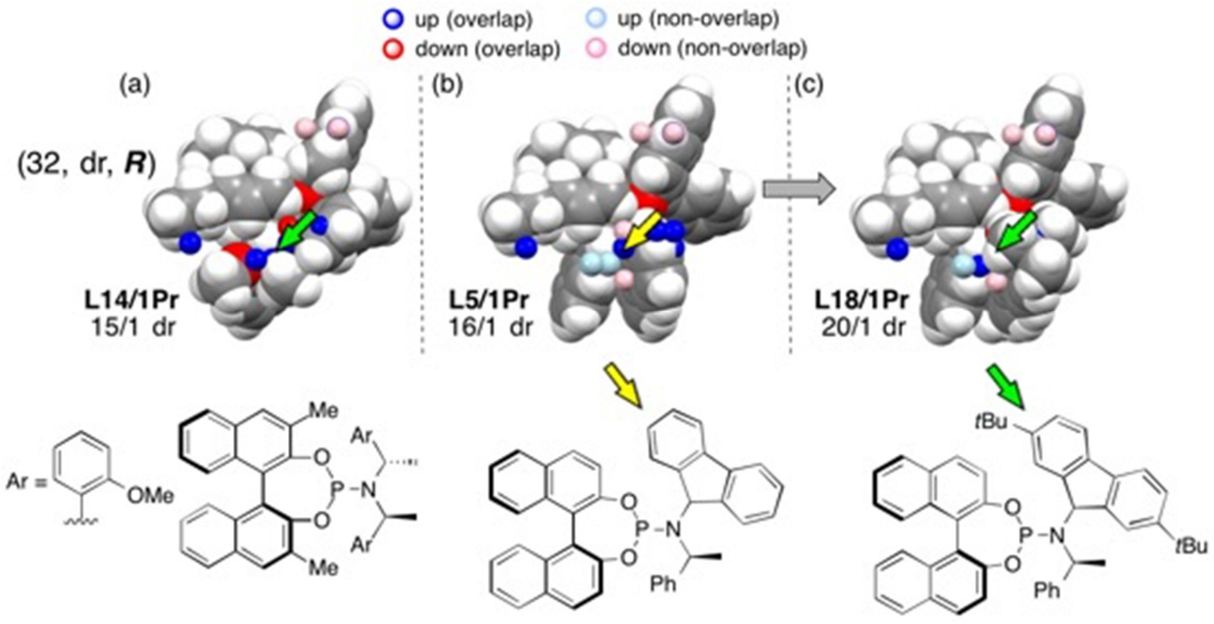


Fig. S35. Results of the MFA using dr data obtained from the 32 reactions with valine ligand ***R***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. Diastereomeric ratios determined by NMR are shown.

Table S17. Experimental and predicted values of dr data determined by NMR and enantioselectivity data. (test set)

Diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** and **L18** are the optimum ligand for the reactions using valine derived ligands ***S*** and ***R***, respectively. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	32 reactions (*S*, dr)			**32 reactions (*R*, dr)**		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Pr	**0.721**	0.72	0.795	1.461	1.471	1.497
L2/1Et	0.594	0.605	0.619	1.221	1.308	1.420
L2/1Pr	0.836	0.822	0.805	1.627	1.529	1.433
L3/1Pr	1.412	1.427	1.489	1.480	1.486	1.492
L4/1Et	1.057	1.055	1.267	1.215	1.226	1.275
L4/1Pr	1.510	1.476	1.398	1.502	1.486	1.471
L5/1Et	0.099	0.099	0.083	1.531	1.511	1.488
L5/1Pr	0.406	0.406	0.316	1.786	**1.777**	1.723
L6/1Et	0.064	0.085	0.104	1.504	1.505	1.513
L7/1Pr	0.295	0.41	0.479	1.084	1.174	1.224
L8/1Et	0.294	0.193	0.117	0.887	0.953	1.007
L8/1Pr	0.464	0.41	0.385	1.314	1.174	1.092
L9/1Et	-0.018	0.01	0.031	0.960	1.007	1.068
L9/1Pr	0.203	0.228	0.250	1.246	1.228	1.211
L10/1Et	0.096	0.119	0.139	1.136	1.084	1.013
L10/1Pr	0.363	0.336	0.311	1.290	1.305	1.324
L11/1Et	-0.039	-0.019	-0.006	1.016	0.976	0.922
L11/1Pr	0.182	0.199	0.209	1.157	1.197	1.243
L12/1Et	0.121	0.121	0.083	0.908	0.924	0.981
L13/1Et	0.442	0.513	0.598	1.422	1.337	1.226
L13/1Pr	0.804	0.73	0.662	1.469	1.558	1.676
L14/1Et	0.718	0.716	1.007	1.596	1.587	1.515
L14/1Pr	1.223	1.224	1.236	1.749	1.747	1.808
L15/1Pr	0.959	0.954	0.852	0.879	0.888	1.544
L16/1Pr	1.484	1.451	1.308	1.551	1.554	1.647
L1/1Et	0.470	0.605		1.154	1.226	
L3/1Et	1.056	0.916		1.185	1.265	
L6/1Pr	0.372	0.392		1.813	1.726	
L7/1Et	0.080	0.085		0.912	0.876	
L12/1Pr	0.344	0.302		1.425	1.145	
L15/1Et	0.821	0.737		0.716	0.669	
L16/1Et	1.038	0.940		1.374	1.333	
L17/1Et	1.619	0.636		0.257	0.669	
L17/1Pr	**2.586**	**0.853**		0.409	0.890	
L18/1Et	0.658	0.398		1.914	1.611	
L18/1Pr	1.179	0.615		1.917	**1.832**	

Table S18. Summary of the MFA using test samples. N_{sel} means the number of extracted descriptors. α is the hyperparamenter of Elastic Net shown in LASSO and Elastic Net regression section on page S93. The numbers of all the descriptors are 149 (25 samples). We evaluated the model based on Golbraikh-Trophsa criteria shown below[26]. Although the regression model generated through analysis of 25 dr data points in the reactions using ligand ***R*** shows low q^2 values in comparison to R^2 due to the small sample size and the sample selection method, all models fulfil Golbraikh-Tropsha criteria.

4. Coefficient of determination for a test set $R^2_{pred} > 0.6$;
5. Leave-one-out cross-validated coefficient of determination $q^2 > 0.5$;
6. One of the coefficients of determination for regressions of a test set through the origin (either predicted vs observed values $R_o{}^2{}_{pred}$ or observed vs predicted values $R'_o{}^2{}_{pred}$) should have value close to R^2_{pred};
   $(R^2_{pred} - R_o{}^2{}_{pred})/R^2_{pred}$ or $(R^2_{pred} - R'_o{}^2{}_{pred})/R^2_{pred} < 0.1$ and $0.85 < k$ or $k' < 1.15$, where k and k' are slopes of the regression line through the origin.

	***S*32dr**	***R*32dr**
α	1	1
R^2	0.99	0.96
q^2	0.94	0.56
R^2_{pred}	0.96	0.89
N_{sel}	28	42
$R_o{}^2{}_{pred}$	0.94	0.88
$R'_o{}^2{}_{pred}$	0.95	0.89
k	1.07	1.03
k'	0.92	0.96
$(R^2_{pred} - R_o{}^2{}_{pred})/R^2_{pred}$	0.01	<0.01
$(R^2_{pred} - R_0'{}^2{}_{pred})/R^2_{pred}$	<0.01	<0.01

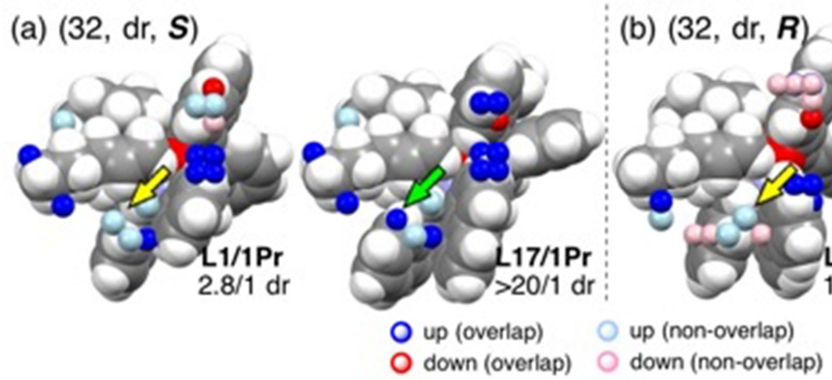


Fig. S36. (a), (b) Visualized important information from the regression models using 25 training samples. In parenthesis, the numbers of all samples including test samples are shown. Diastereomeric ratios determined by NMR are shown.

Table S19. Experimental and predicted values of dr data determined by NMR and enantioselectivity data. (MFA in another design pathway)

Diastereoselectivity (dr) of products **2Et** and **2Pr** are shown as $\Delta\Delta G^{\ddagger}$ values (kcal/mol), which were used as the target variables for the regression analysis (**Exp.**). Predicted values (**Pred.**) and predicted values from leave-one-out cross-validation [**Pred. (CV)**] are also shown. Ligands for the 1st MFA are highlighted by yellow. Ligands designed at the 1st MFA are highlighted by green. The 2nd MFA was performed including the designed ligands. **L17** and **L18** are the optimum ligand for the reactions using valine derived ligands ***S*** and ***R***, respectively. Predicted and measured $\Delta\Delta G^{\ddagger}$ values in the reactions using the template and designed molecules are highlighted by red.

	20 reactions (*R*, dr)			**26 reactions (*S*, dr)**		
	Exp.	**Pred.**	**Pred. (CV)**	**Exp.**	**Pred.**	**Pred. (CV)**
L1/1Et	1.154	1.193	1.242	0.470	0.478	0.562
L1/1Pr	**1.461**	**1.468**	1.482	**0.721**	**0.732**	0.746
L2/1Et	1.221	1.254	1.313	0.594	0.582	0.572
L2/1Pr	1.627	1.529	1.446	0.836	0.836	0.841
L5/1Et	1.531	1.499	1.489	0.099	0.104	0.113
L5/1Pr	1.786	1.774	1.774	0.406	0.400	0.389
L6/1Et	1.504	1.499	1.500	0.064	0.079	0.107
L6/1Pr	1.813	1.774	1.762	0.372	0.376	0.385
L7/1Et	0.912	0.934	0.938	0.080	0.079	0.078
L7/1Pr	1.084	1.209	1.262	0.295	0.430	0.494
L8/1Et	0.887	0.934	0.949	0.294	0.176	0.111
L8/1Pr	1.314	1.209	1.160	0.464	0.430	0.415
L9/1Et	0.960	1.009	1.029	-0.018	-0.008	-0.004
L9/1Pr	1.246	1.285	1.302	0.203	0.246	0.280
L10/1Et	1.136	1.009	0.961	0.096	0.089	0.083
L10/1Pr	1.290	1.285	1.283	0.363	0.343	0.330
L11/1Et	1.016	0.982	0.918	-0.039	-0.045	-0.060
L11/1Pr	1.157	1.258	1.309	0.182	0.209	0.235
L12/1Et	0.908	1.027	1.174	0.121	0.119	0.099
L12/1Pr	1.425	1.303	1.223	0.344	0.333	0.328
L18/1Et	1.914	1.559		0.658	0.783	0.942
L18/1Pr	**1.917**	**1.835**		1.179	1.037	0.868
L13/1Et				0.442	0.492	0.551
L13/1Pr				0.804	0.746	0.679
L15/1Et				0.821	0.791	0.692
L15/1Pr				0.959	0.972	1.065
L17/1Et				1.619	0.791	
L17/1Pr				**2.586**	**1.045**	

Plots of the measured vs predicted values

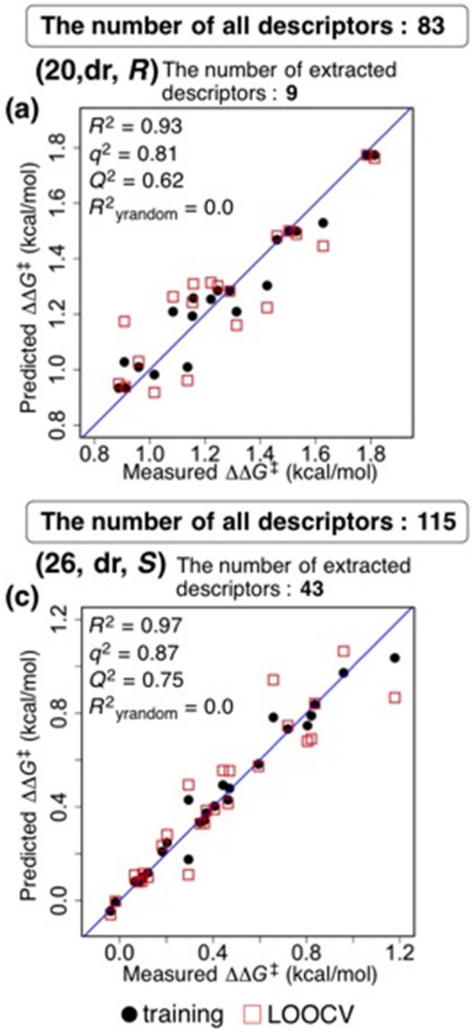


Fig. S37. Results of the MFA in another design pathway. (a), (b) Plots of the predicted (black dots) and the leave-one-out predicted (red squares) vs. measured $\Delta\Delta G^{\ddagger}$ values. R^2 : Coefficient of determination q^2 : Leave-one-out cross-validated coefficient of determination. Q^2 : 4-fold cross-validated coefficient of determination. R^2_{yrandom}: Coefficient of determination calculated by y-randomization. The blue line is the x = y line

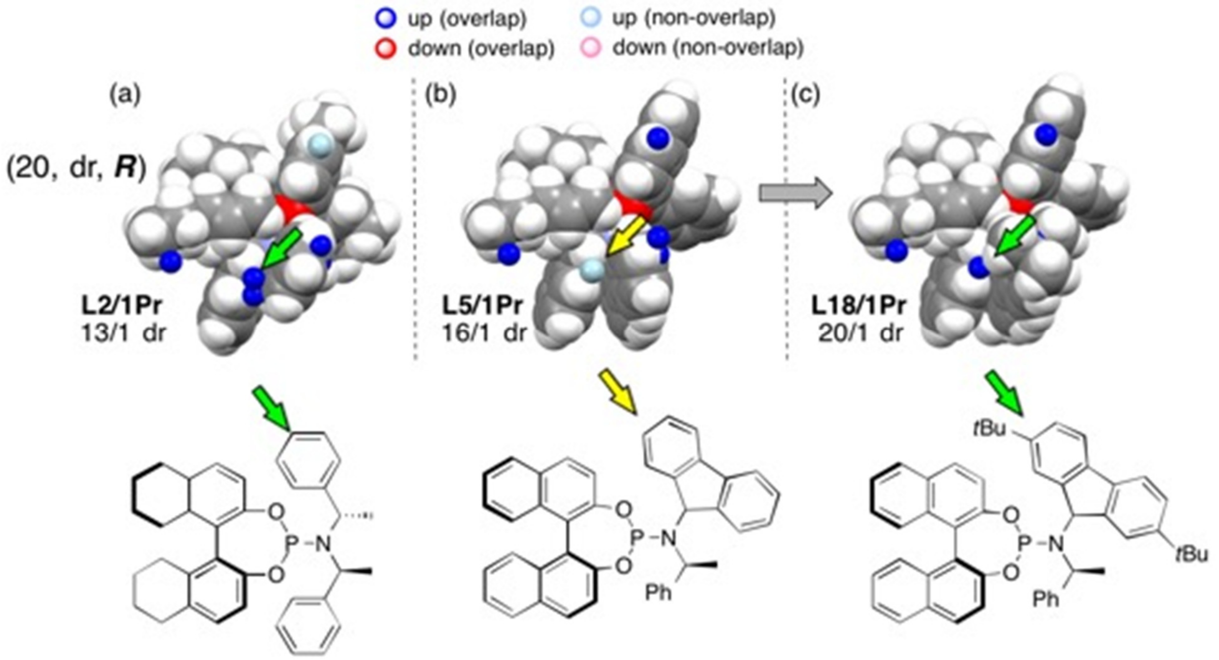


Fig. S38. Results of the MFA using dr data obtained from the 20 reactions with valine ligand ***R***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. Diastereomeric ratios determined by NMR are shown.

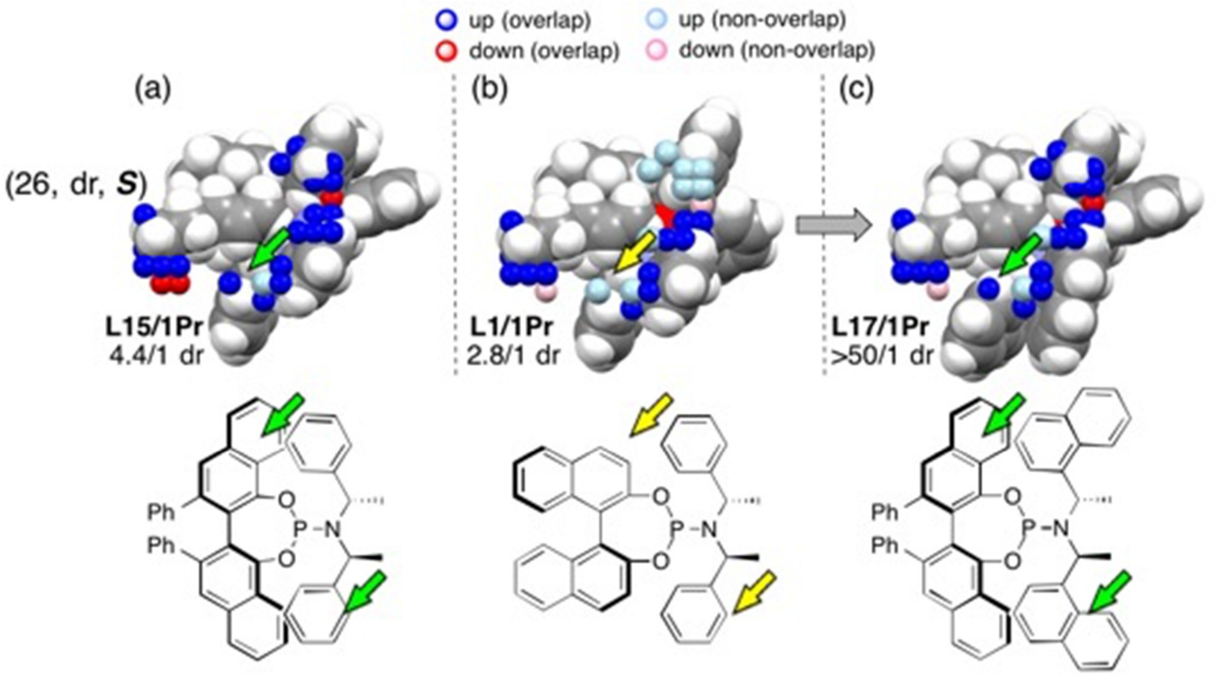


Fig. S39. Results of the MFA obtained from the 26 reactions with valine ligand ***S***. (a) The origin of the blue point used for the molecular design. (b) Design templates. (c) Designed molecules. The MFA was performed using Elastic Net ($\alpha = 0.5$). Diastereomeric ratios determined by NMR are shown.

MFA using structures optimized at the B3LYP-D3/LANL2DZ and 6-31G* level

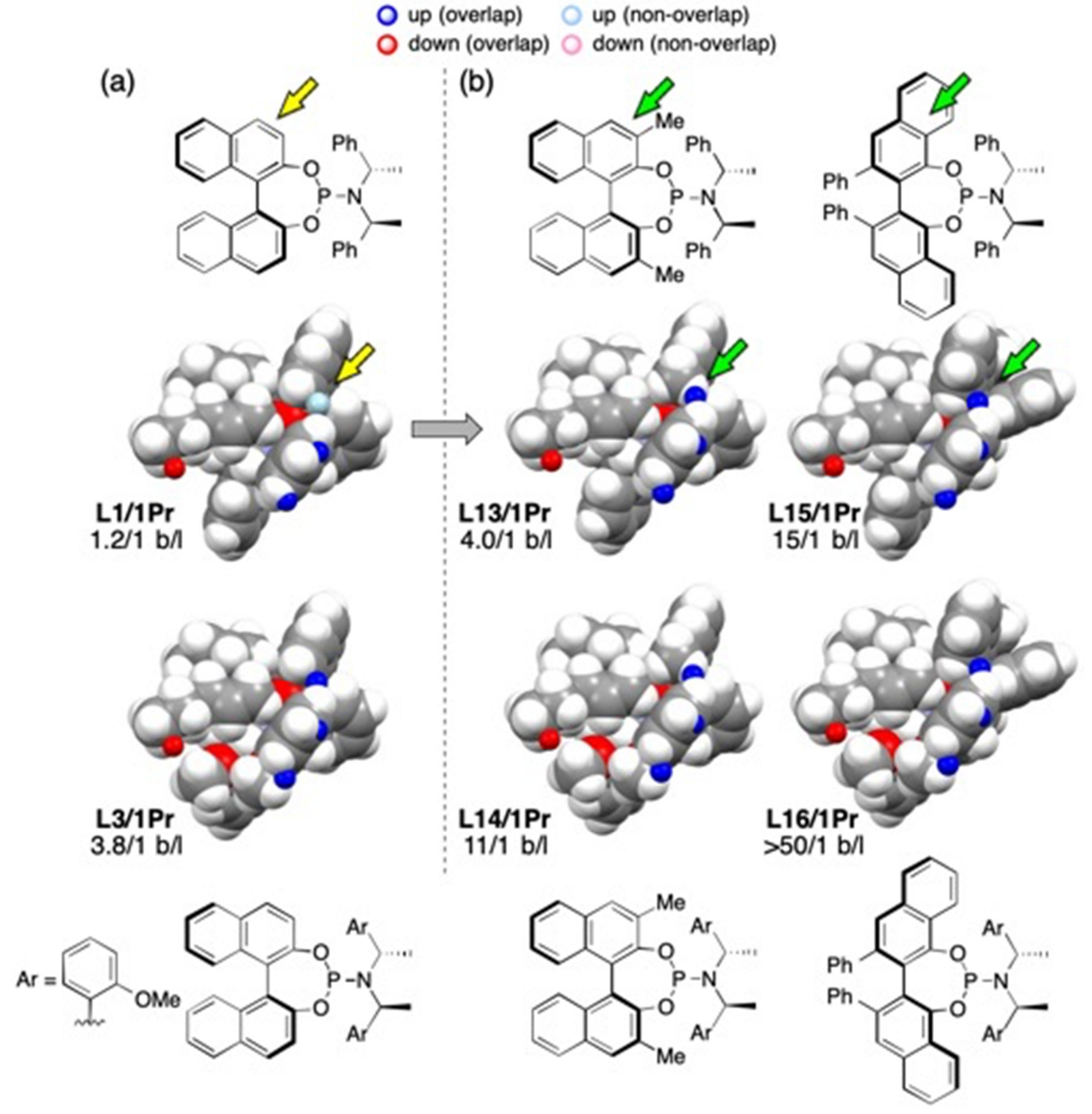


Fig. S40. Results of the MFA using b/l data obtained from the 24 reactions with valine ligand ***S***. (a) Design templates. (b) Designed molecules.

In comparison to the MFA using structures optimized at B3LYP/LANL2DZ and 6-31G* level (Fig. S4), we could not find the structural information that leads to the design of **L14** and **L16**.

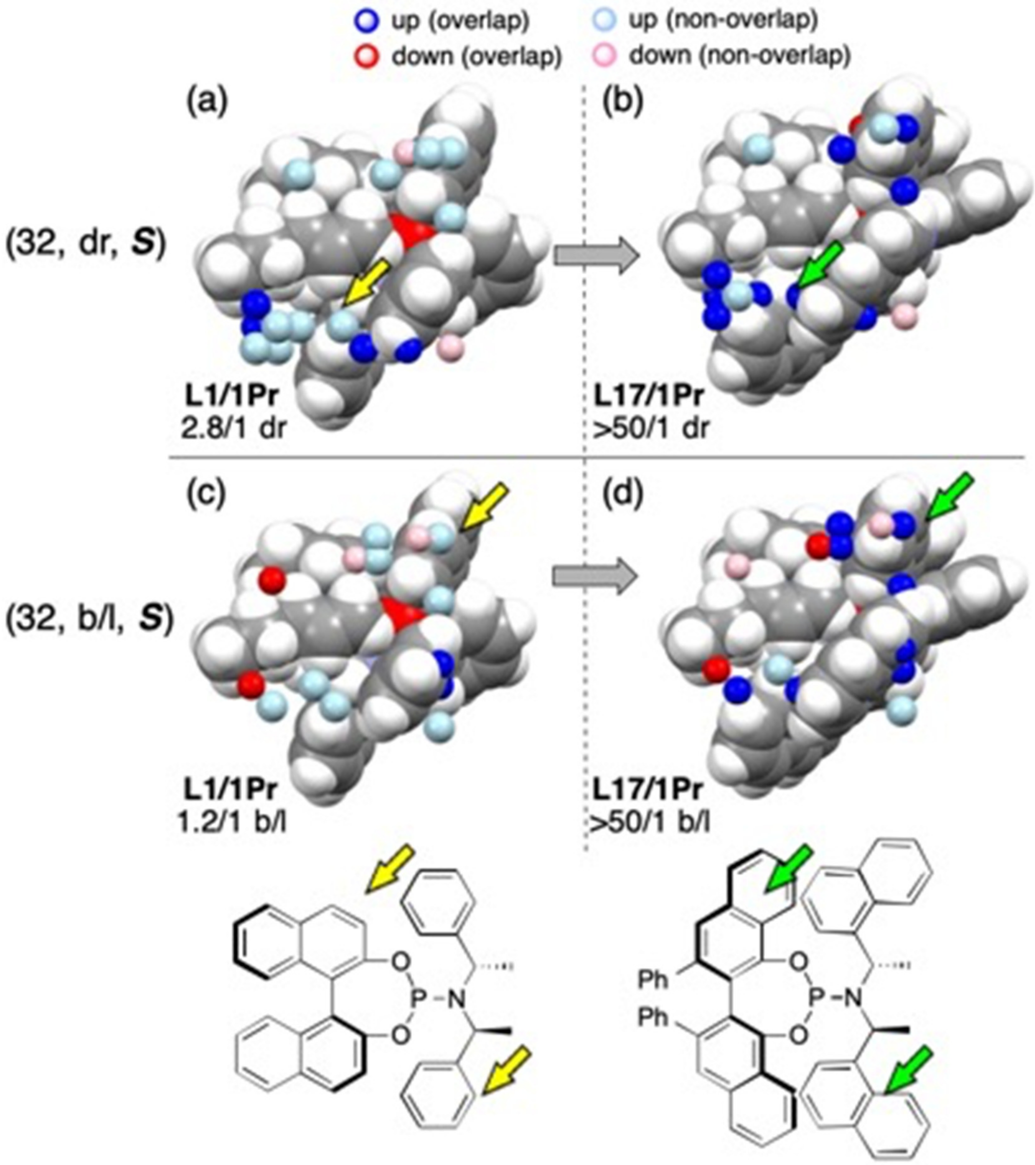


Fig. S41. Results of the MFA using b/l and dr data obtained from the 32 reactions with valine ligand ***S***. (a), (c) Design templates. (b), (e) Designed molecules.

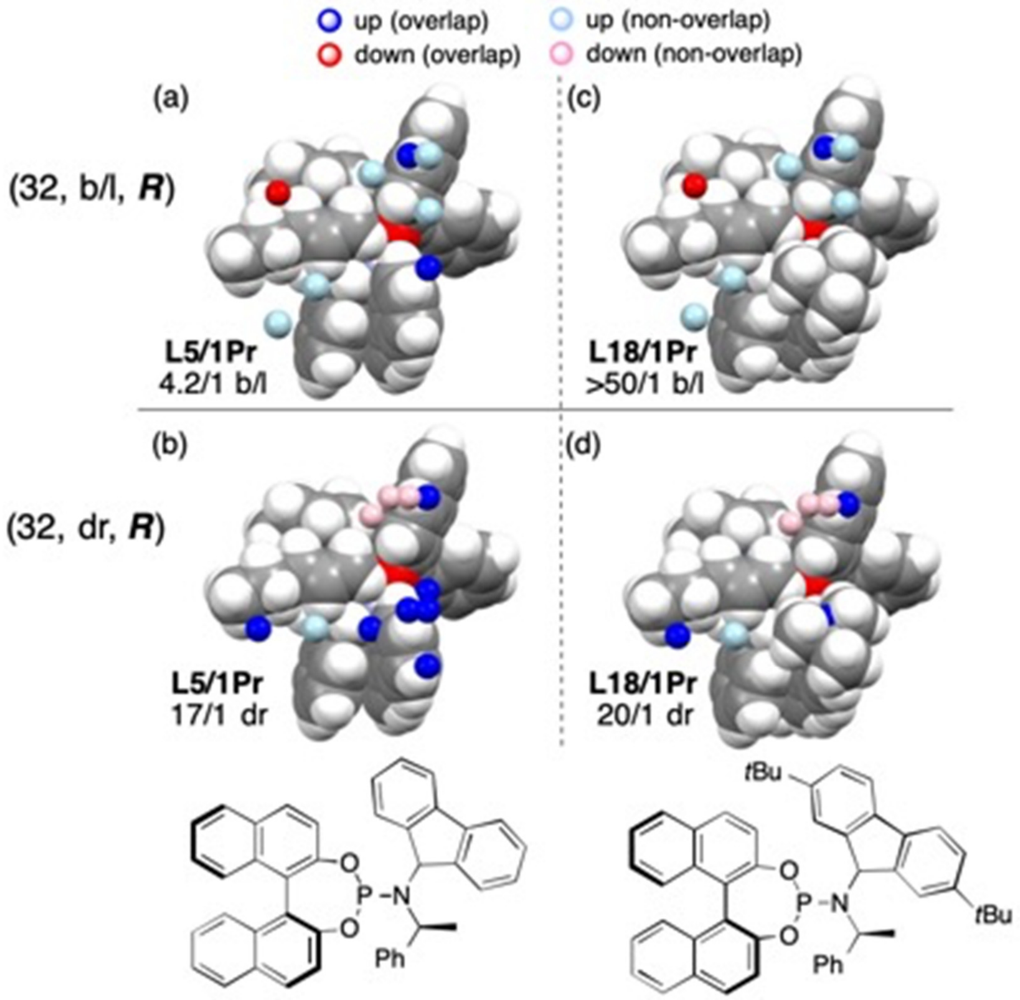


Fig. S42. Results of the MFA using b/l [(a) with **L5/1Pr** and (c) with **L18/1Pr**] and dr data [(b) with **L5/1Pr** and (d) with **L18/1Pr**] obtained from the 32 reactions with valine ligand ***R***.

In comparison to the MFA using structures optimized at B3LYP/LANL2DZ and 6-31G* level (Fig. S6), we could not find the structural information that leads to the design of **L18**.

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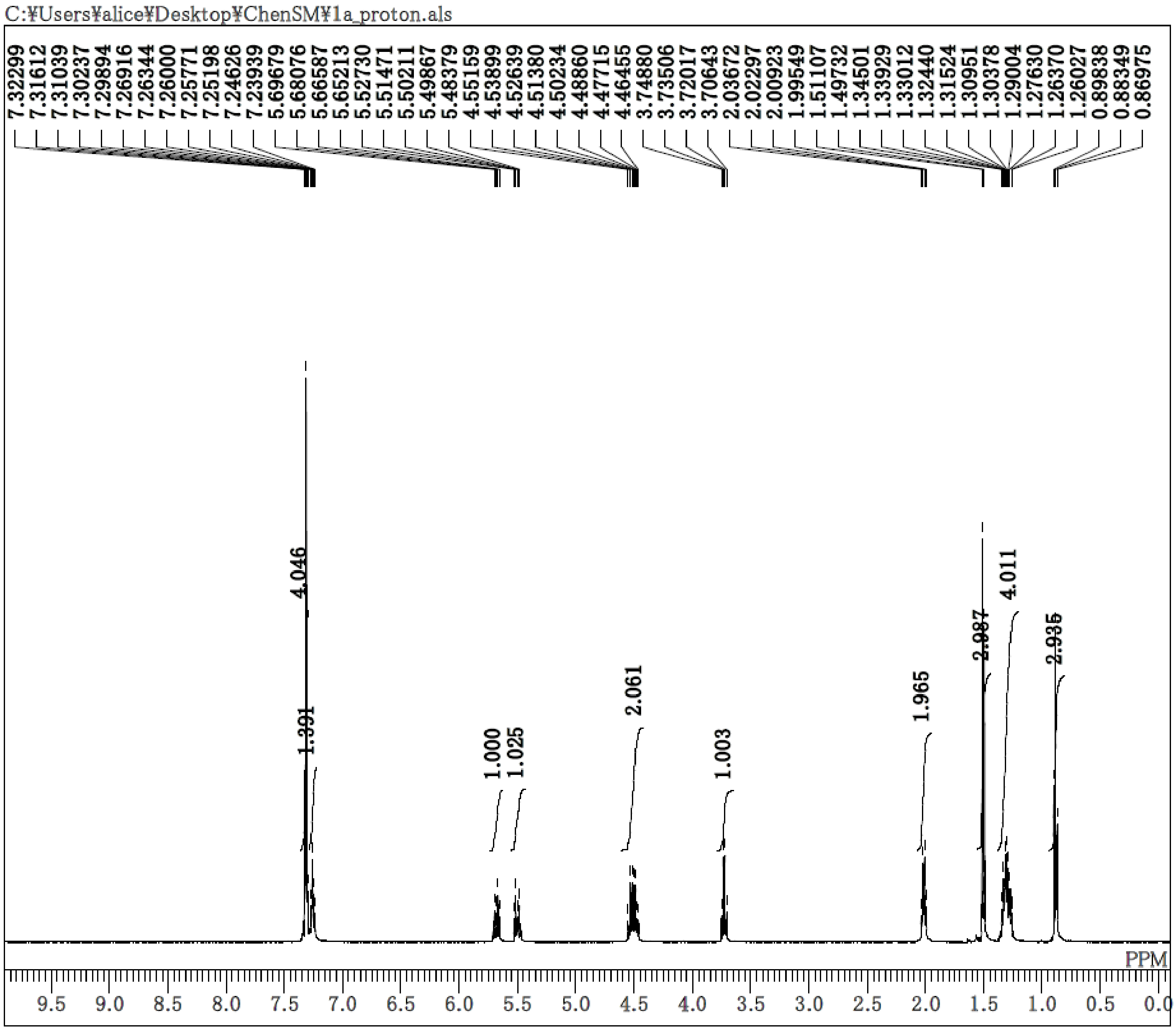
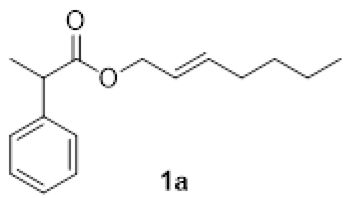
13. NMR Charts





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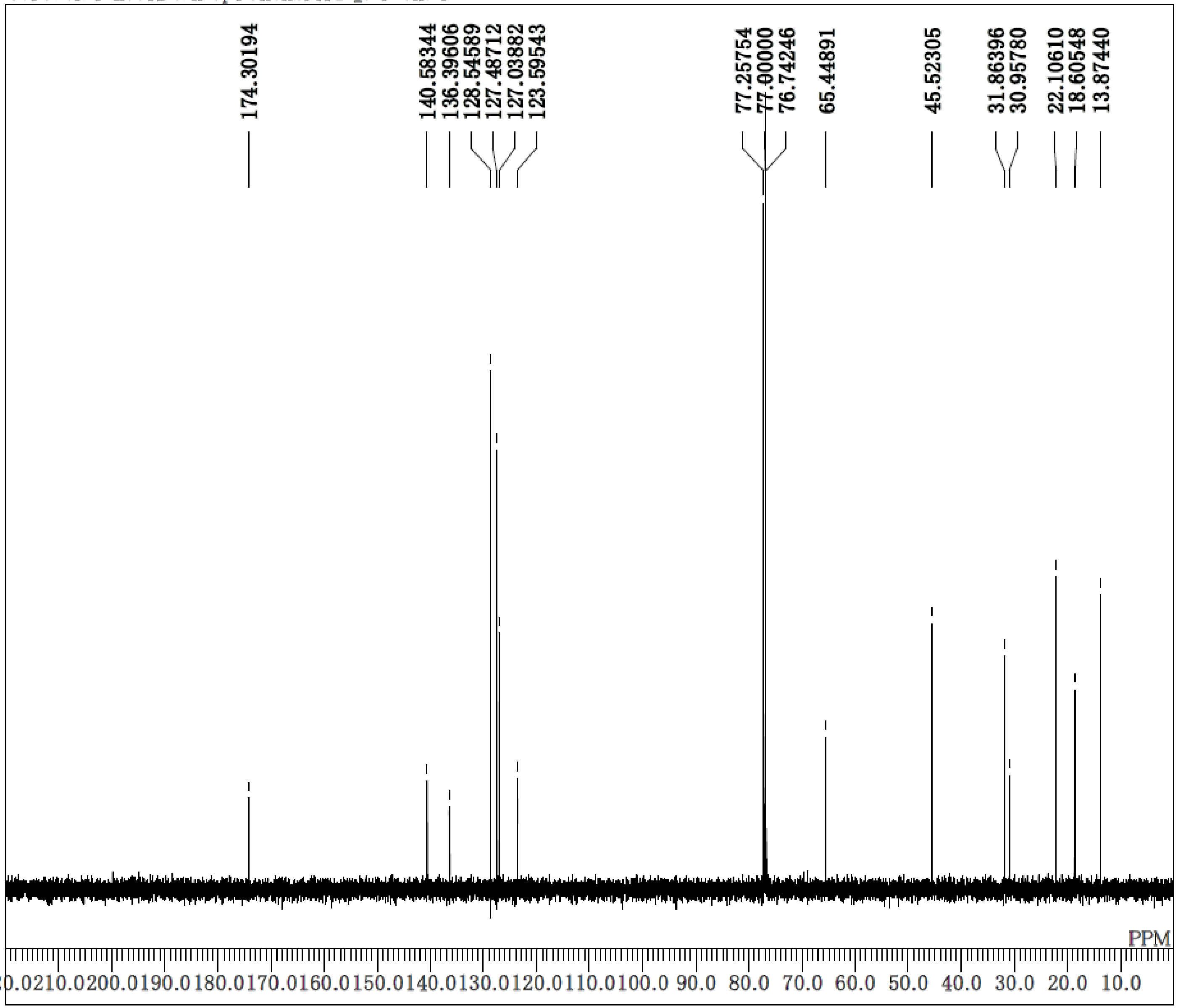

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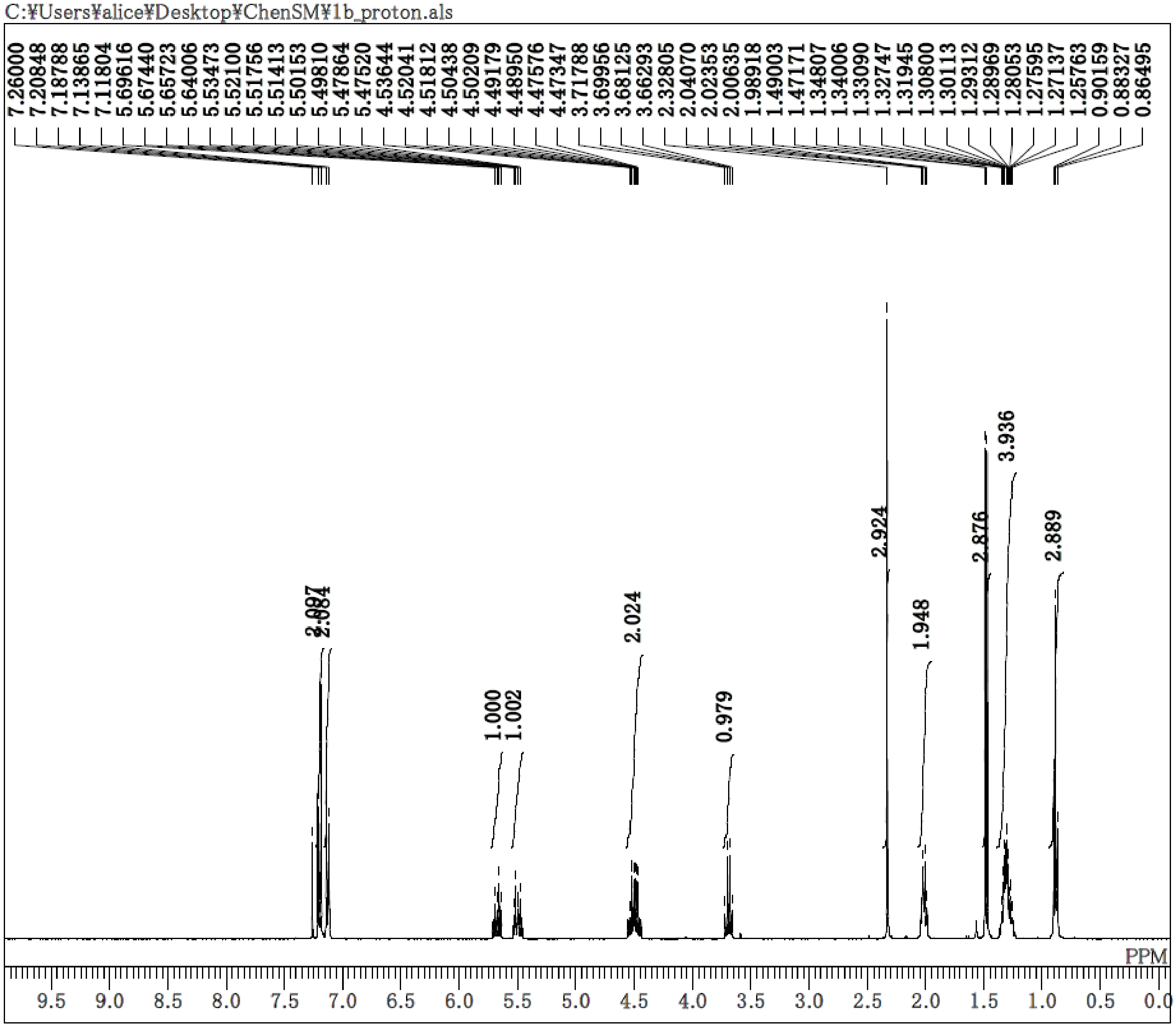

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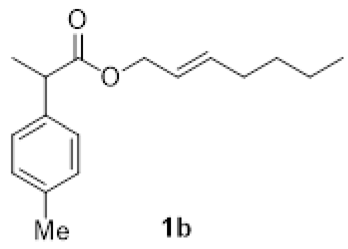

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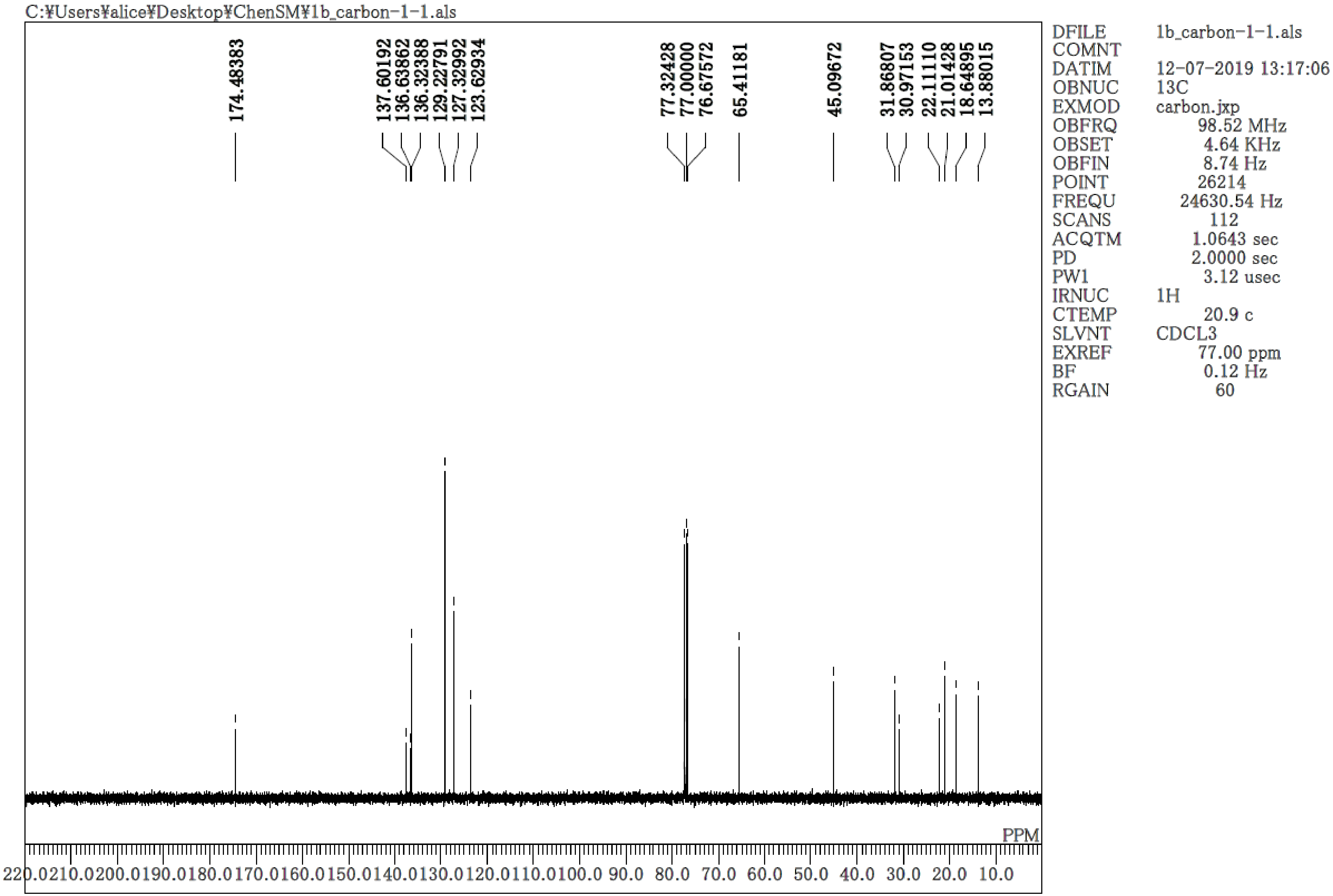
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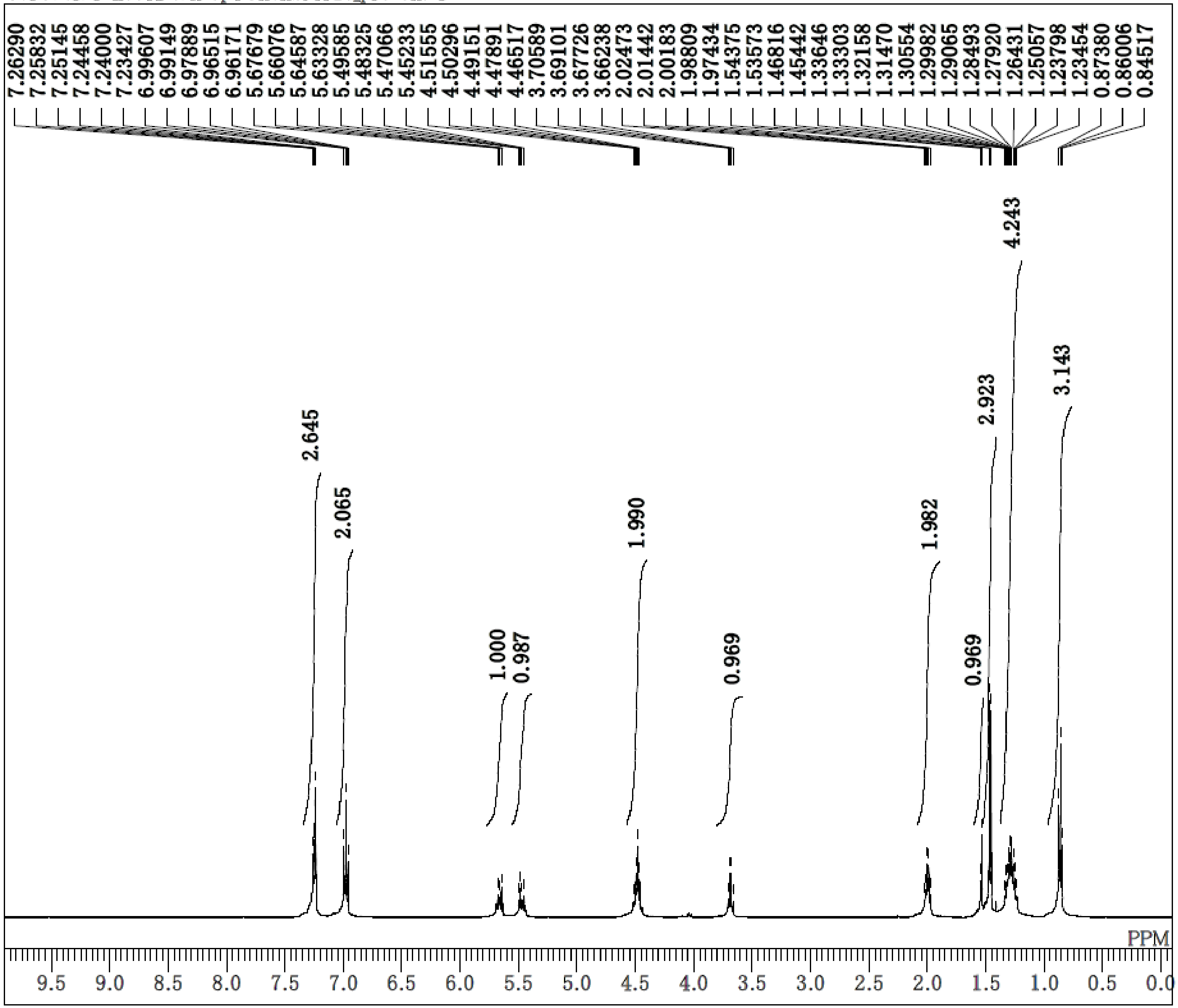

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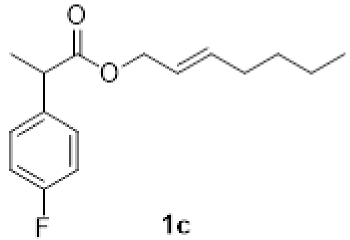

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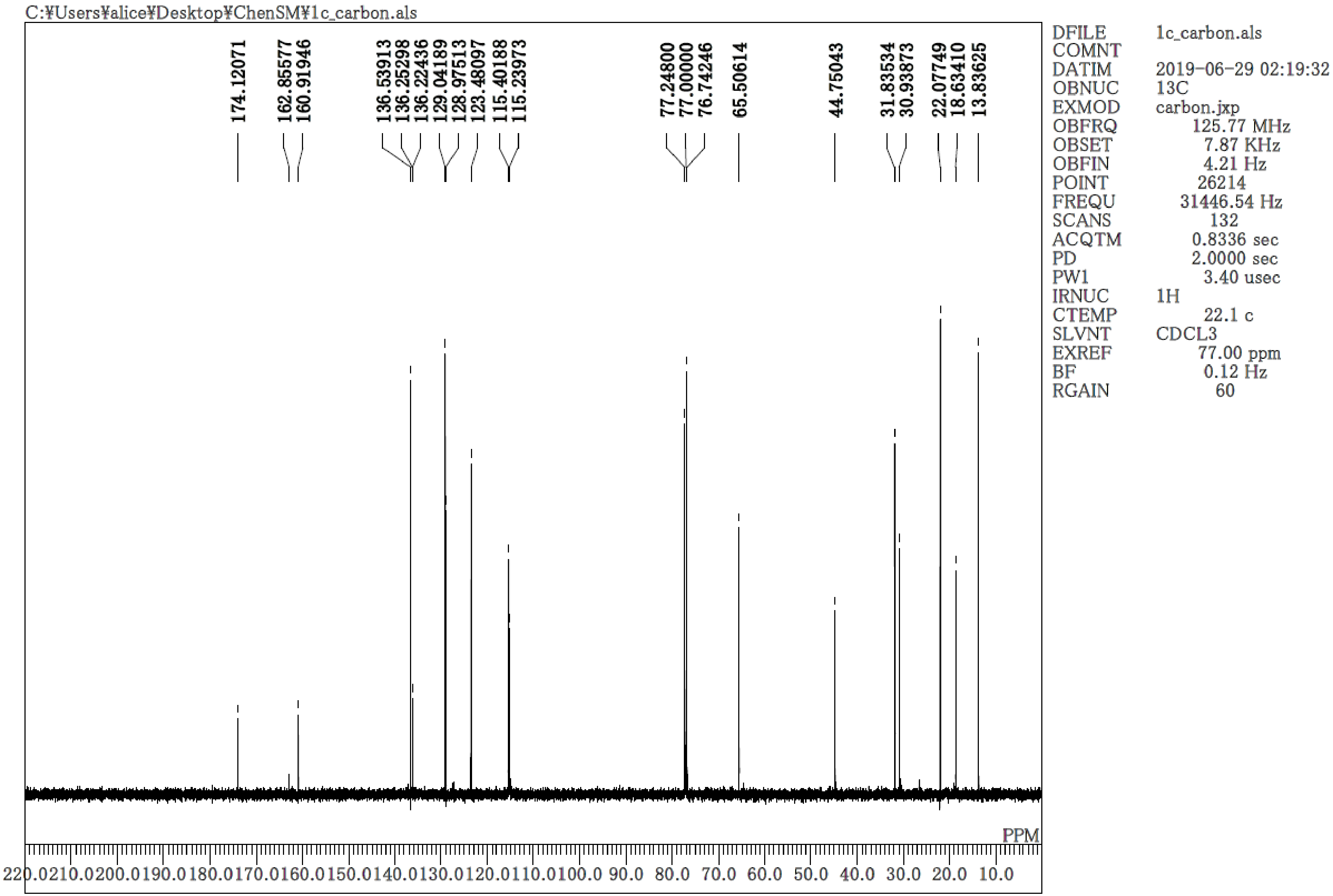
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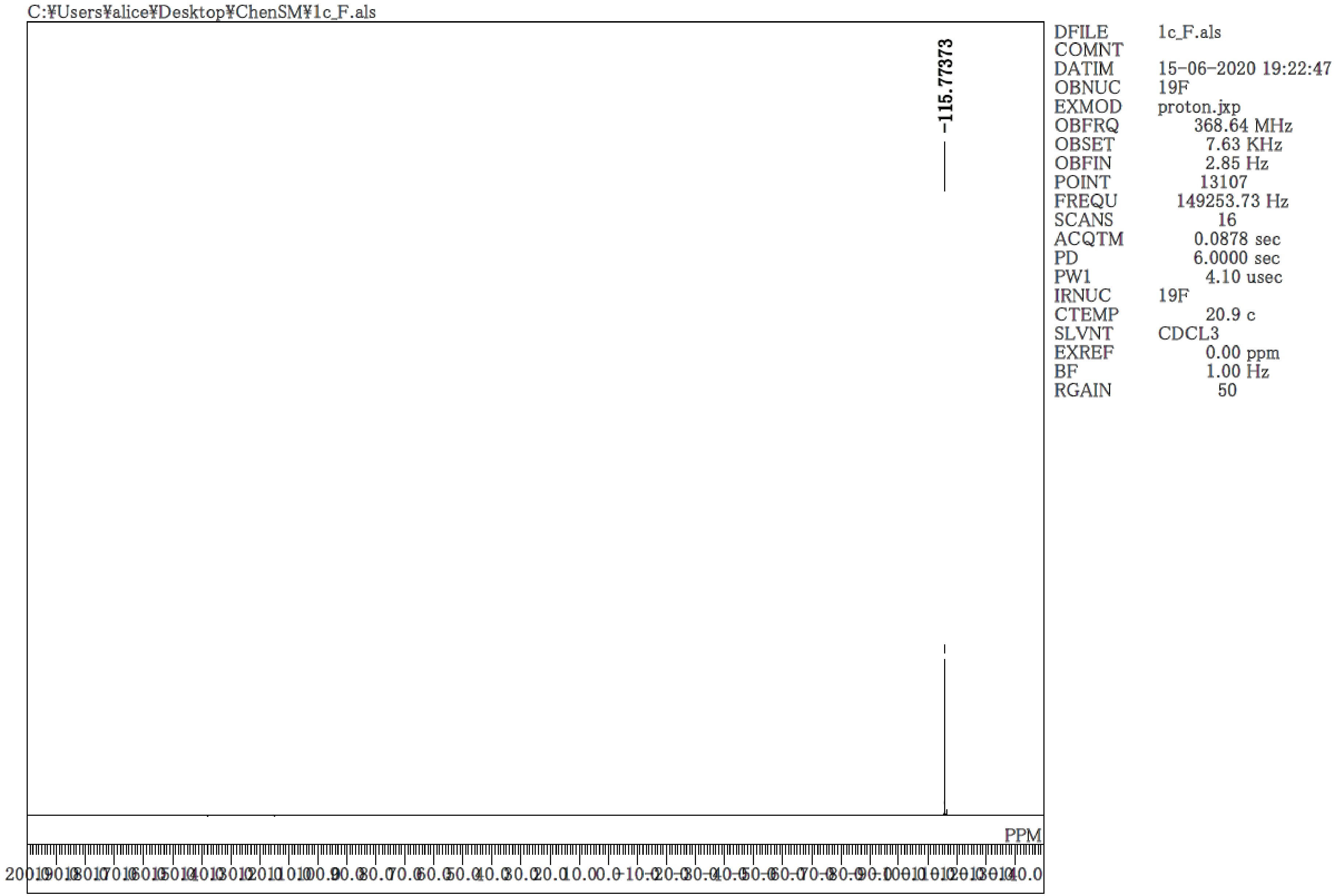
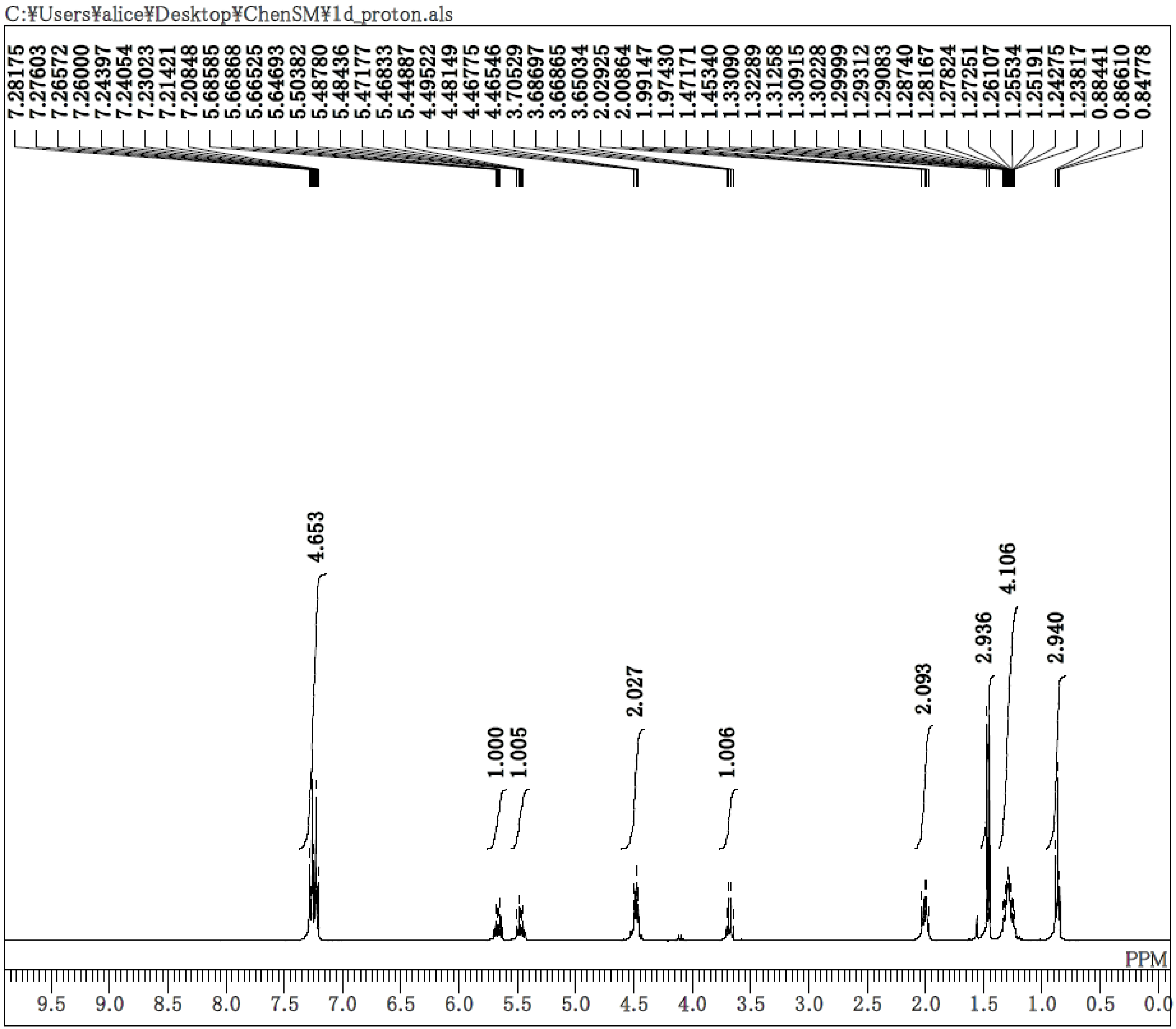
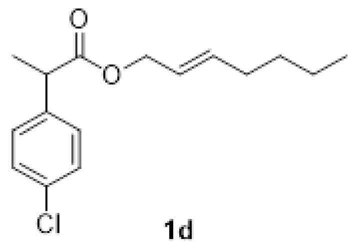
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1c_carbon.als
COMNT
DATIM 2019-06-29 02:19:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 132
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60


C:¥Users¥alice¥Desktop¥ChenSM¥1c_F.als
-115.77373
PPM
DFILE 1c_F.als
COMNT
DATIM 15-06-2020 19:22:47
OBNUC 19F
EXMOD proton.jxp
OBFRQ 368.64 MHz
OBSET 7.63 KHz
OBFIN 2.85 Hz
POINT 13107
FREQU 149253.73 Hz
SCANS 16
ACQTM 0.0878 sec
PD 6.0000 sec
PW1 4.10 usec
IRNUC 19F
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 50

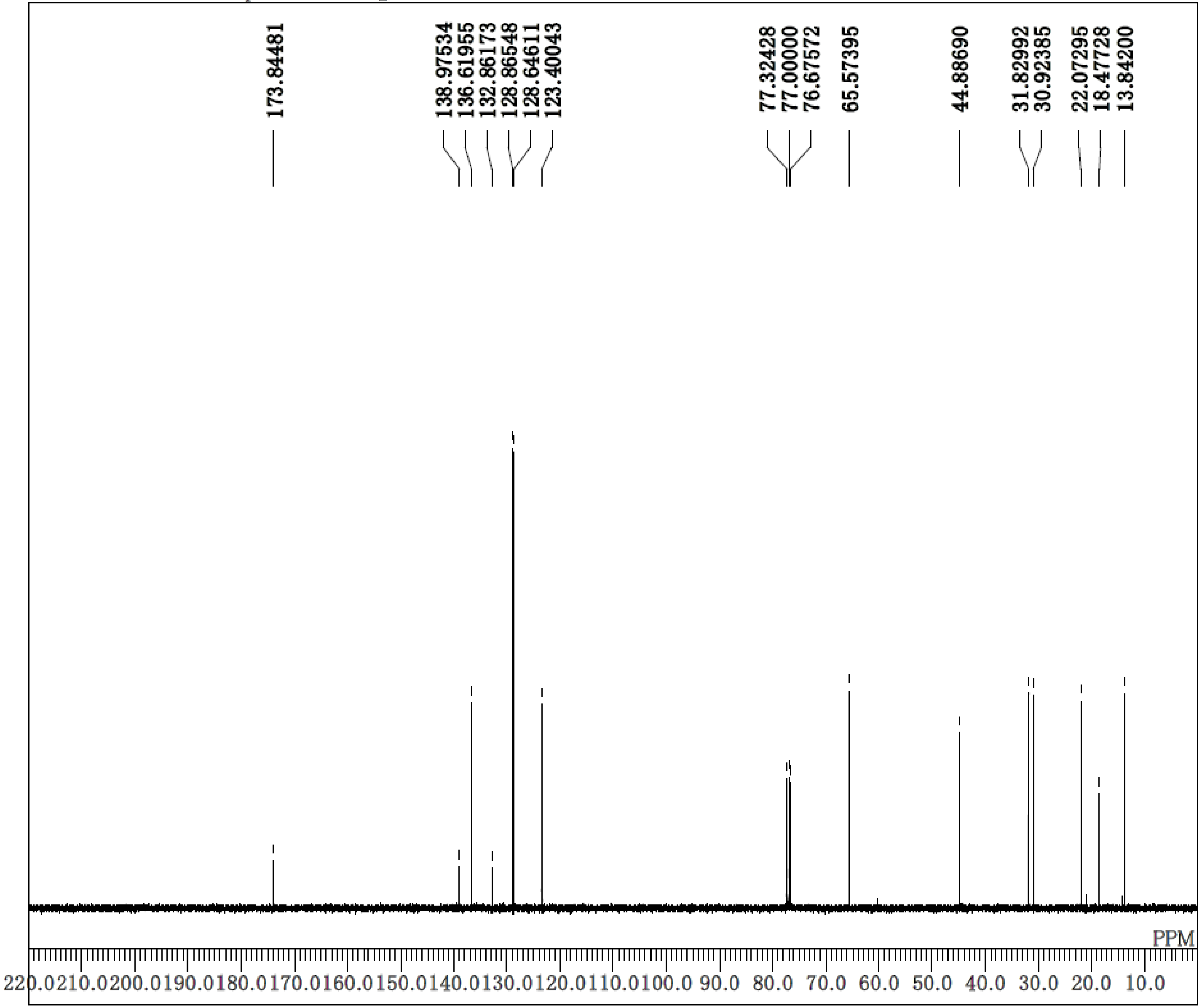


DFILE	1d_proton.als
COMNT	
DATIM	14-08-2019 01:50:31
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38

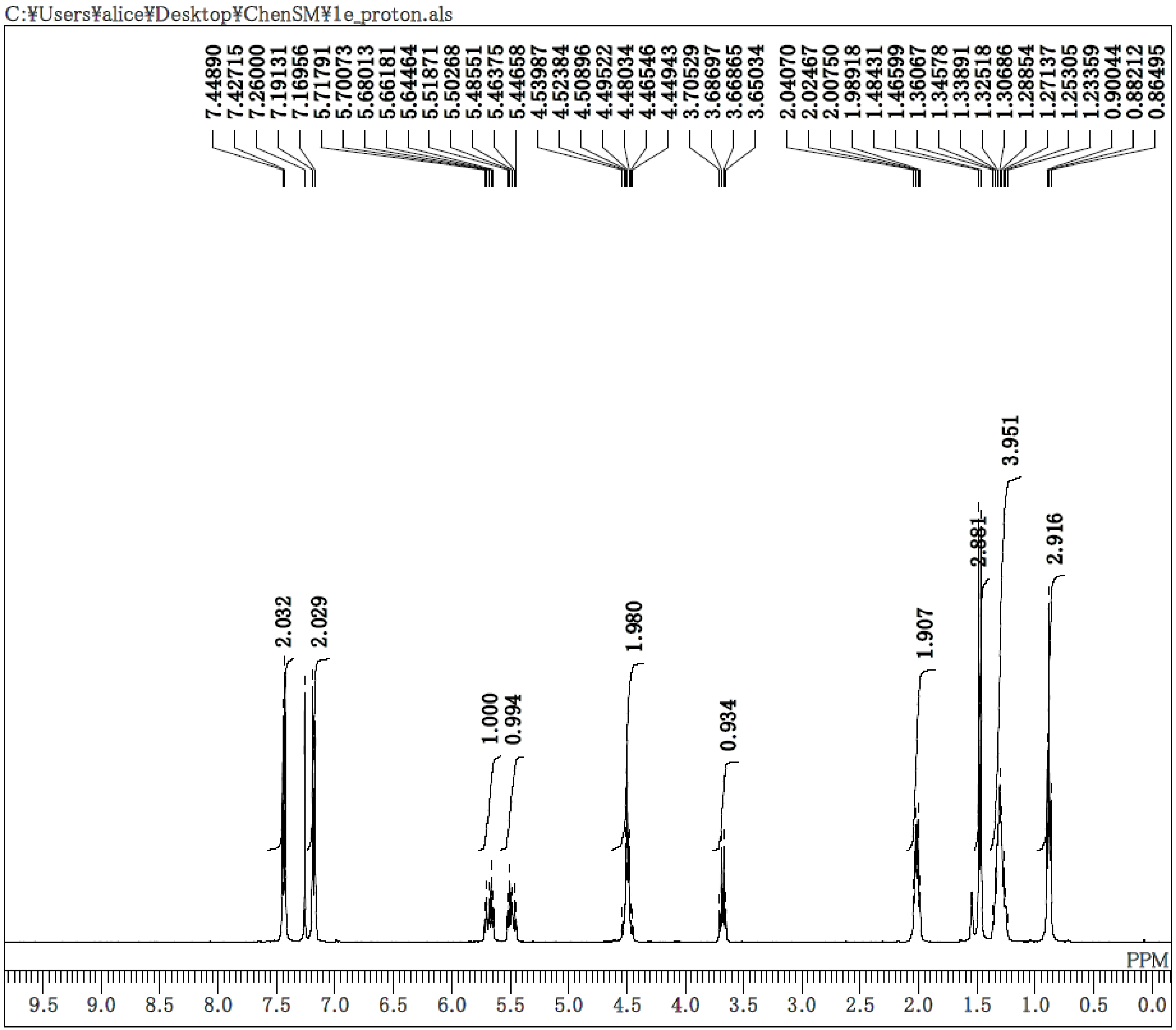


1d

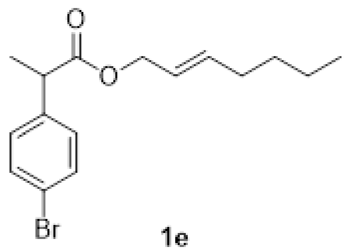
C:¥Users¥alice¥Desktop¥ChenSM¥1d_carbon.als



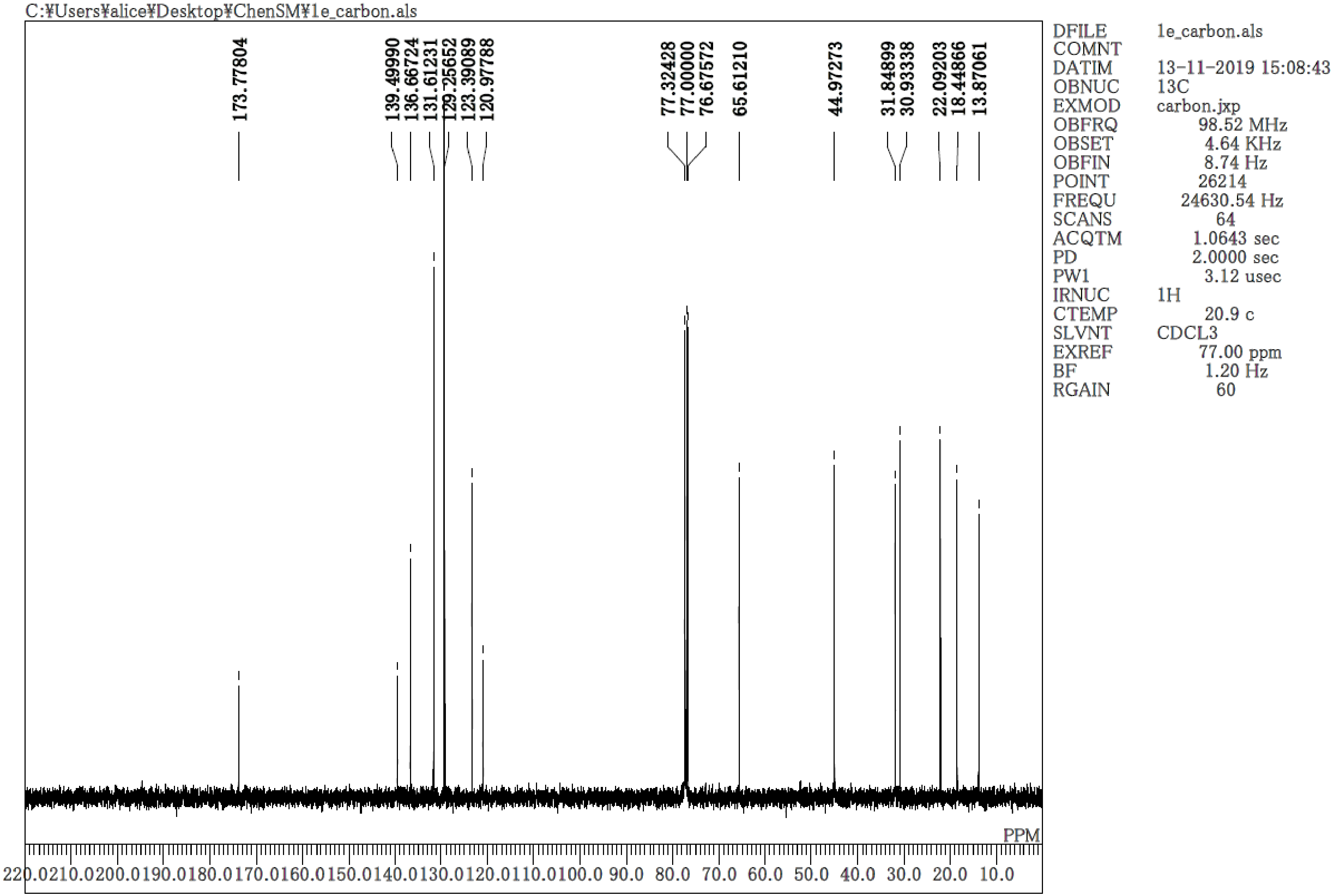
DFILE	1d_carbon.als
COMNT	
DATIM	14-08-2019 02:20:11
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	109
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE	1e_proton.als
COMNT	
DATIM	13-11-2019 14:55:15
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	46



1e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129.25652
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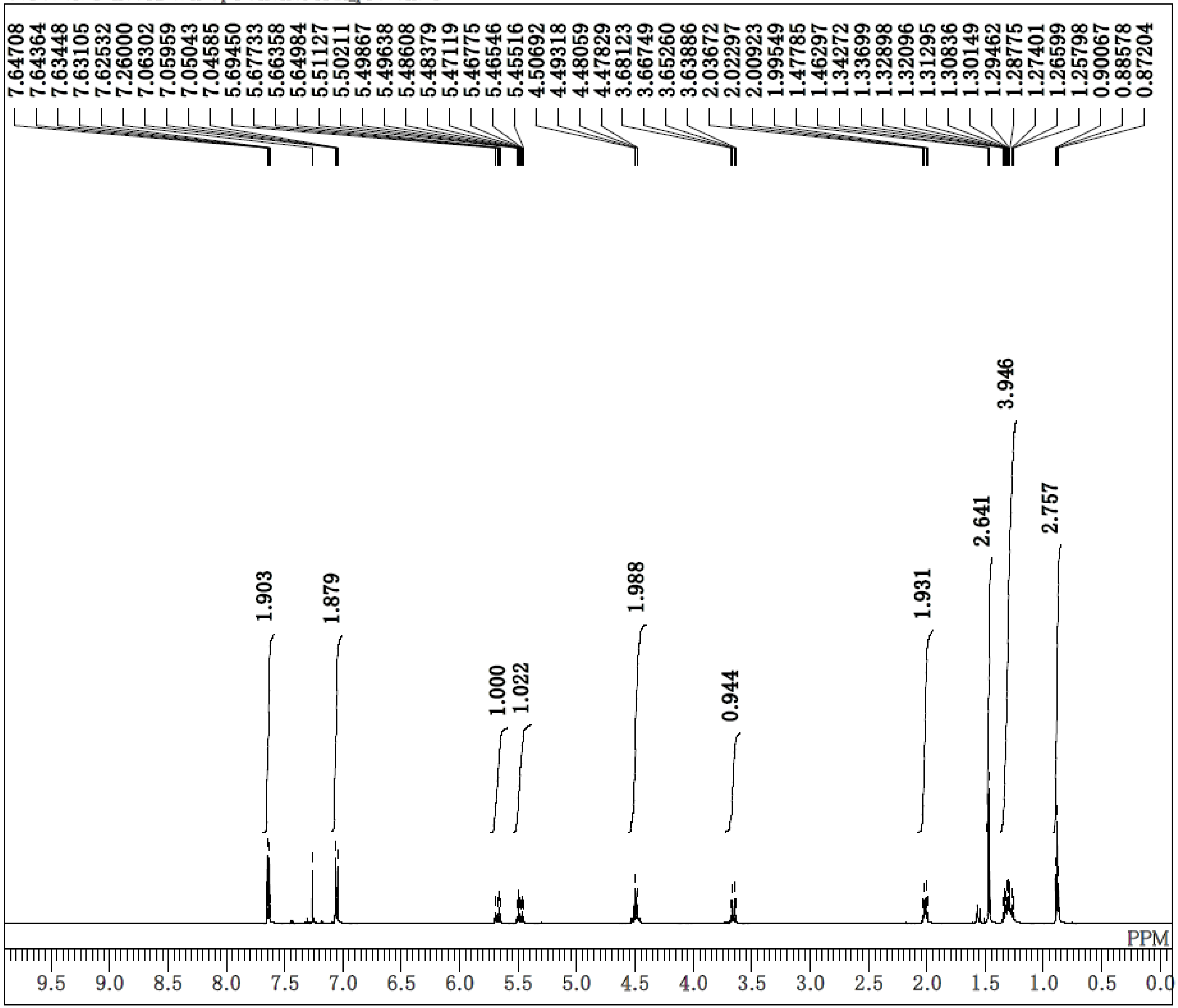
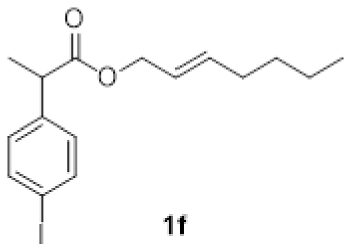
120.97788
77.32428
77.00000
76.67572
65.61210
44.97273
31.84899
30.93338
22.09203
18.44866
13.87061
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1e_carbon.als
COMNT
DATIM 13-11-2019 15:08:43
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 64
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenSM¥1f_proton.als

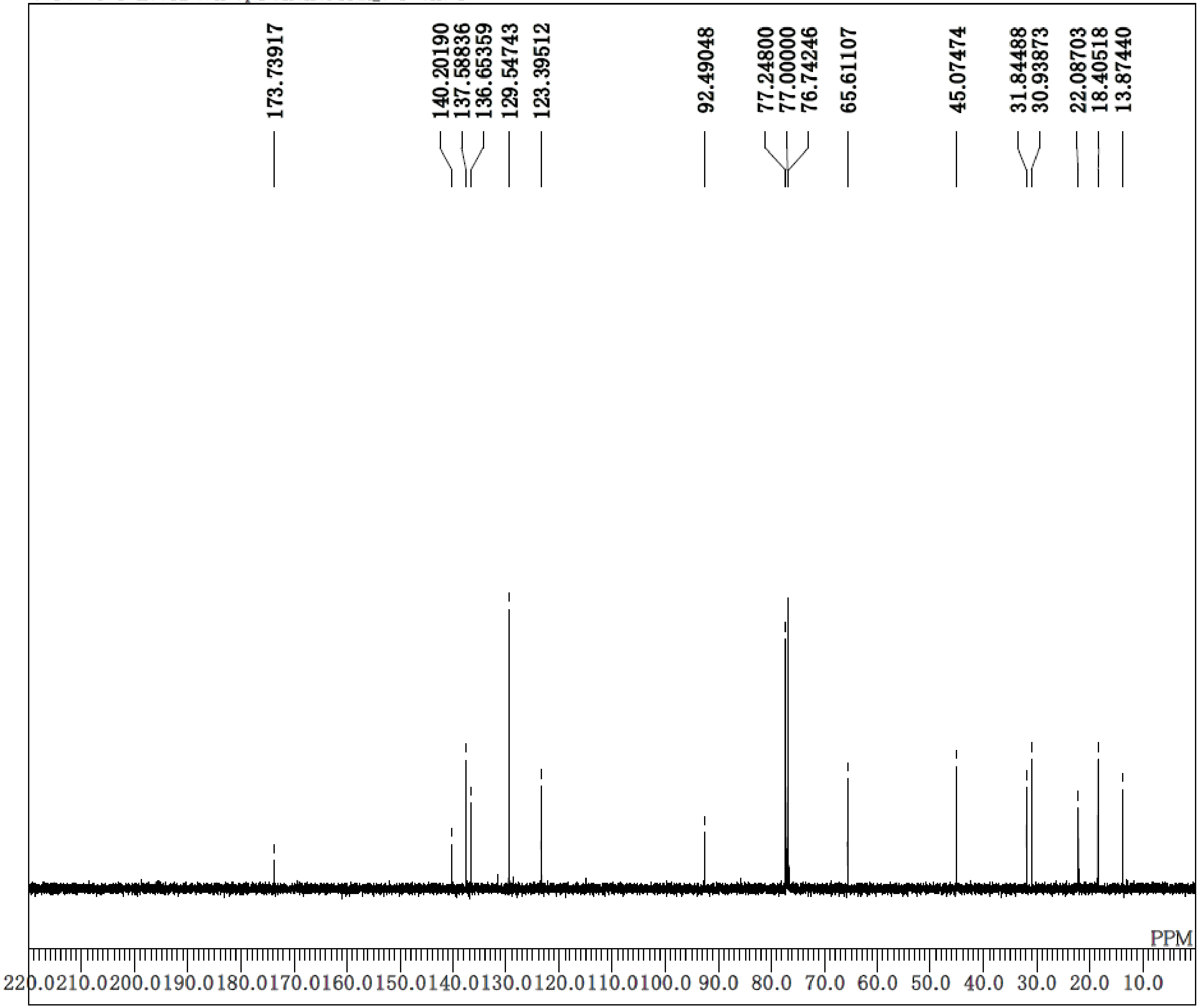


DFILE 1f_proton.als
COMNT
DATIM 2019-11-19 23:34:03
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

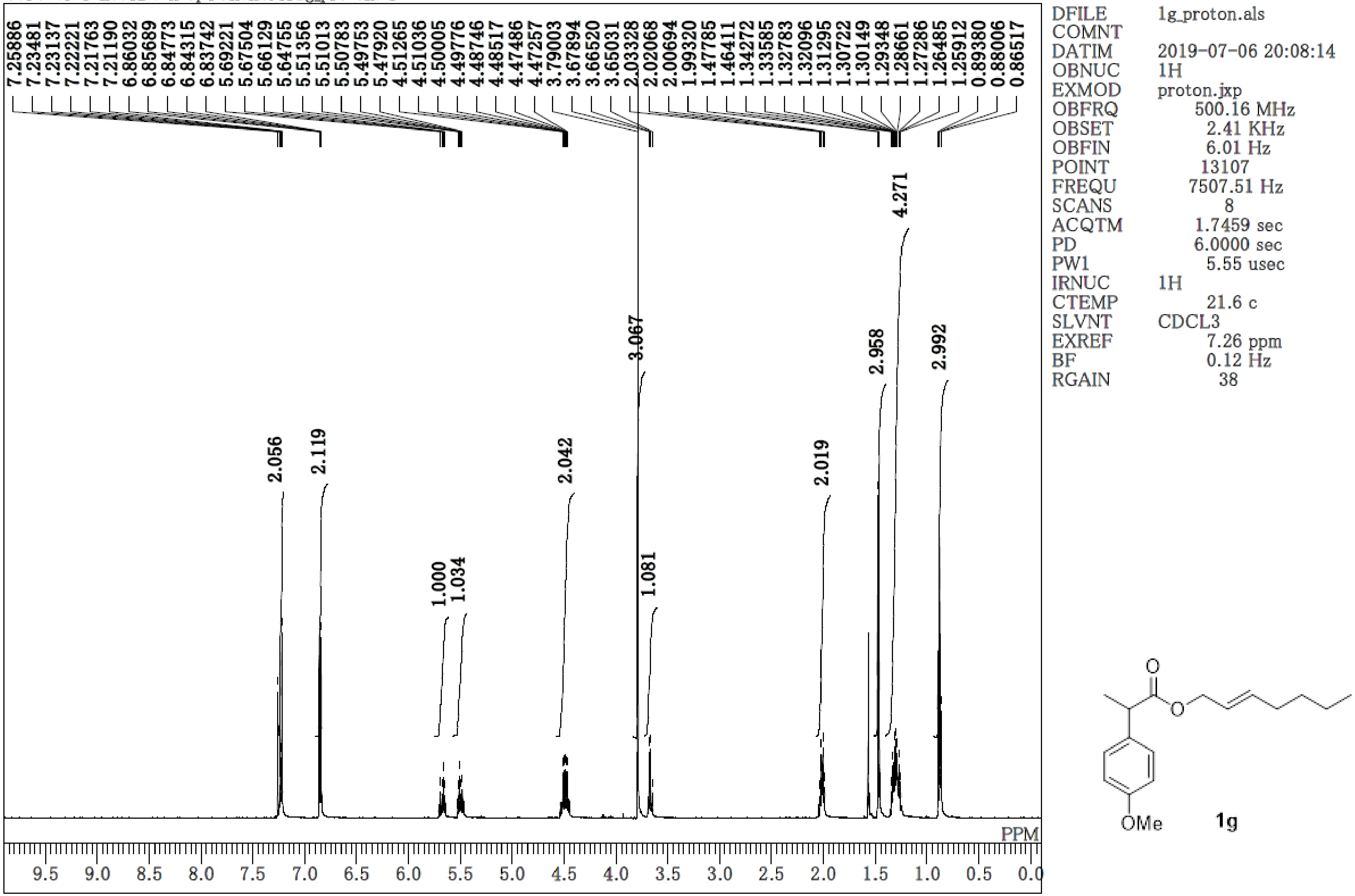


1f

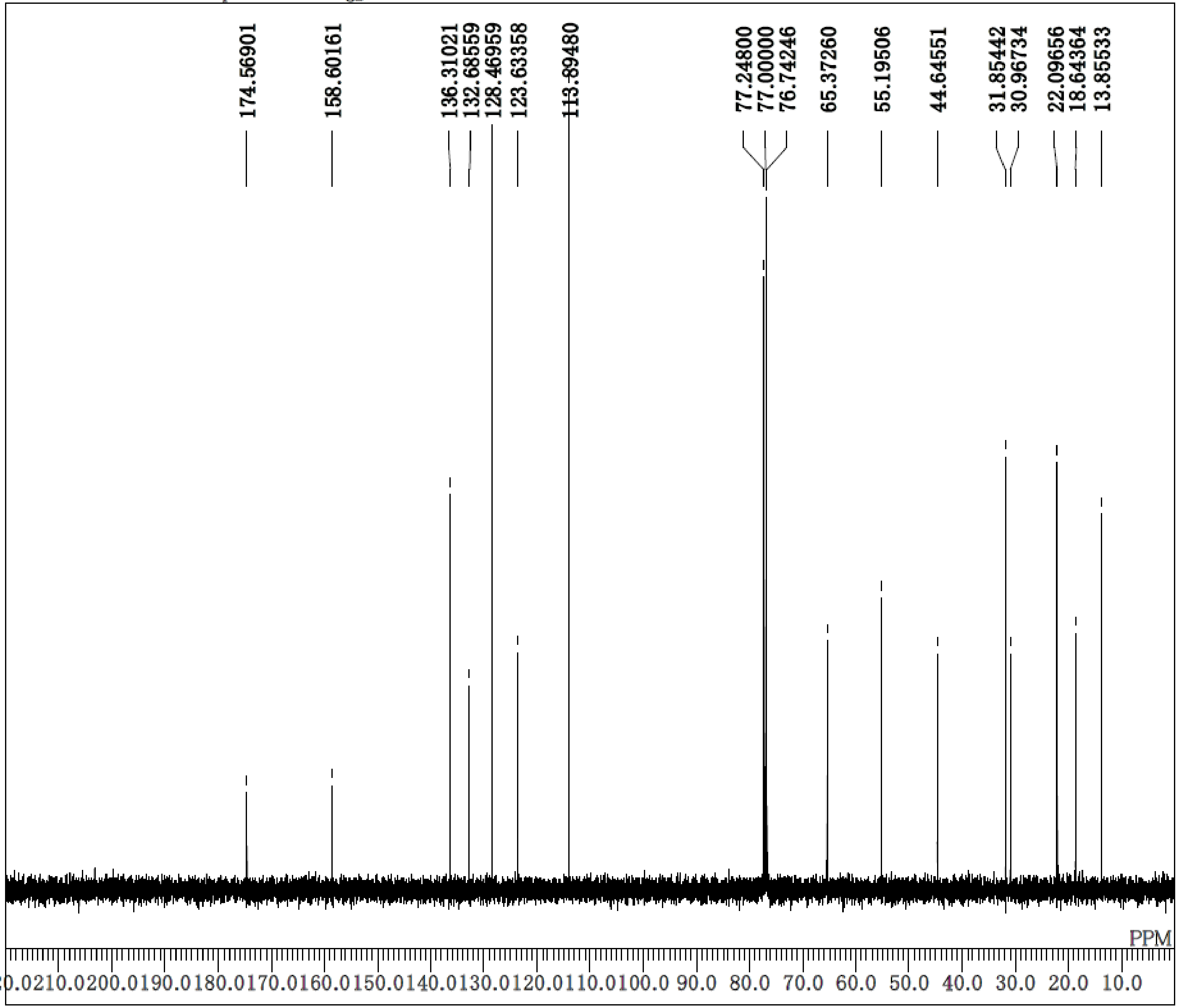
C:¥Users¥alice¥Desktop¥ChenSM¥1f_carbon.als



DFILE	1f_carbon.als
COMNT	
DATIM	2019-11-19 23:47:31
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	66
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

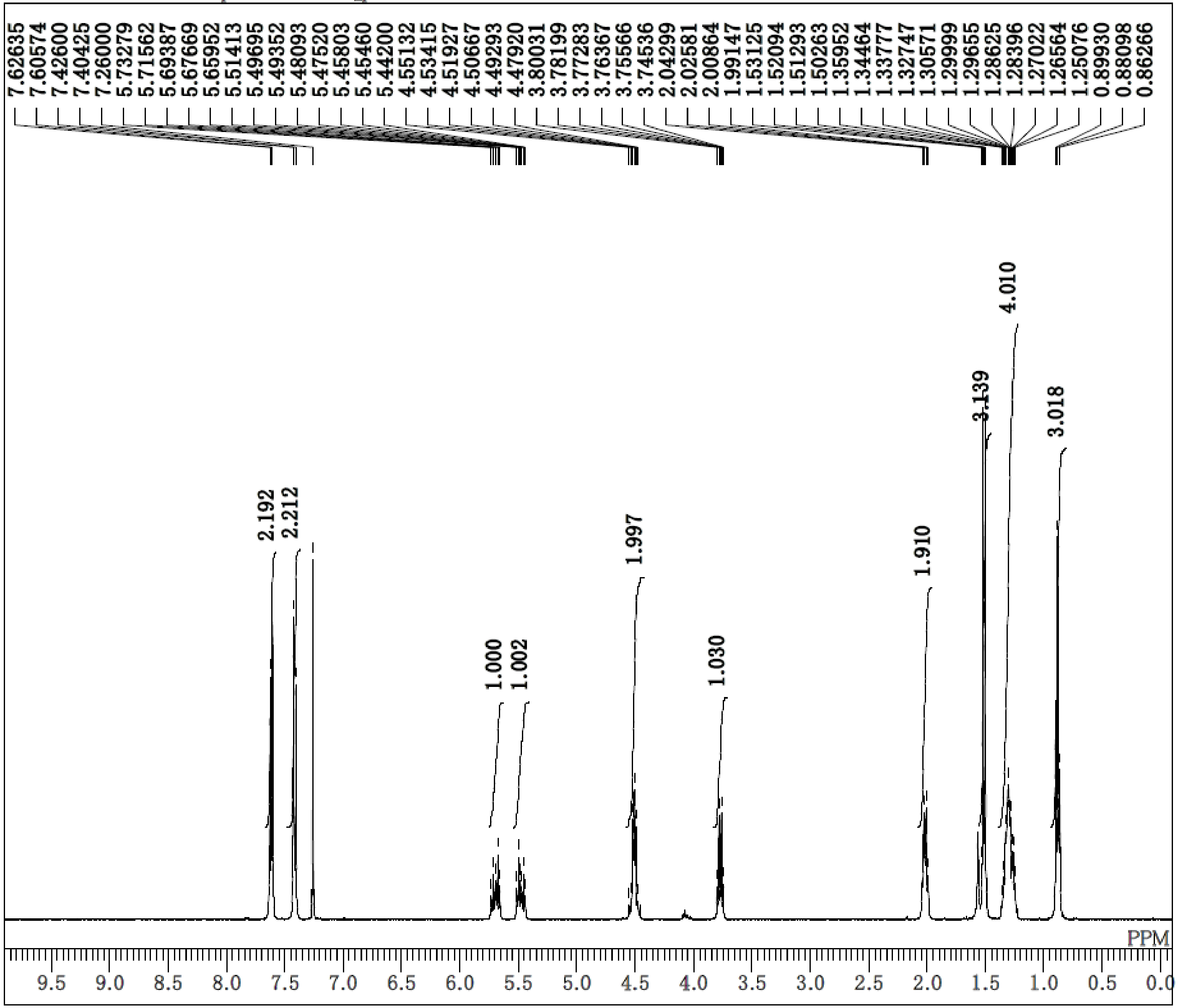

C:¥Users¥alice¥Desktop¥ChenSM¥1g_proton.als
DFILE 1g_proton.als
COMNT
DATIM 2019-07-06 20:08:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38
PPM
OMe
1g

C:¥Users¥alice¥Desktop¥ChenSM¥1g_carbon.als

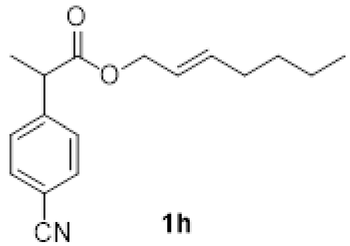


DFILE	1g_carbon.als
COMNT	
DATIM	2019-07-06 20:33:27
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	101
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenSM¥1h_proton.als

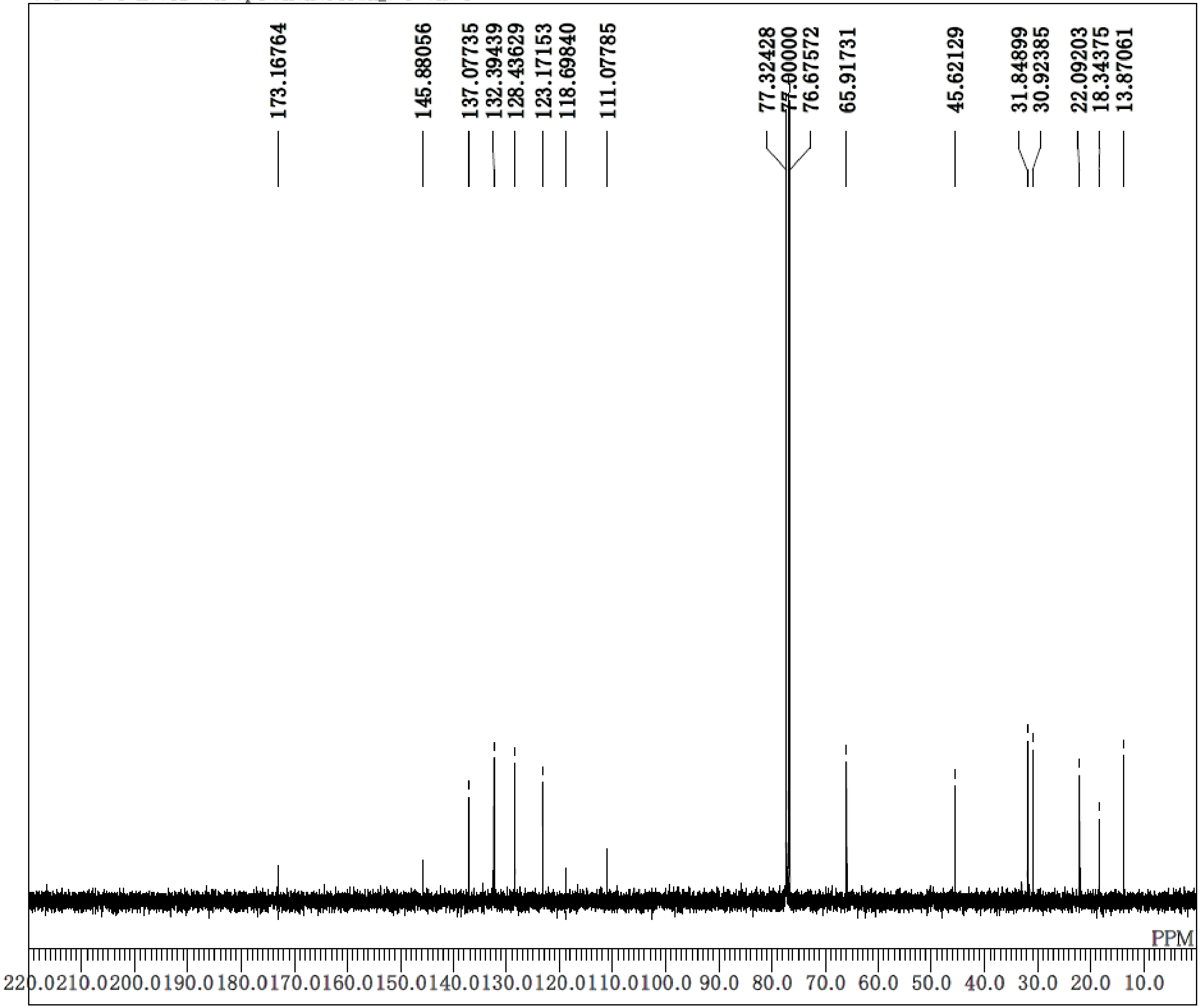


DFILE	1h_proton.als
COMNT	
DATIM	09-11-2019 21:12:19
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	44

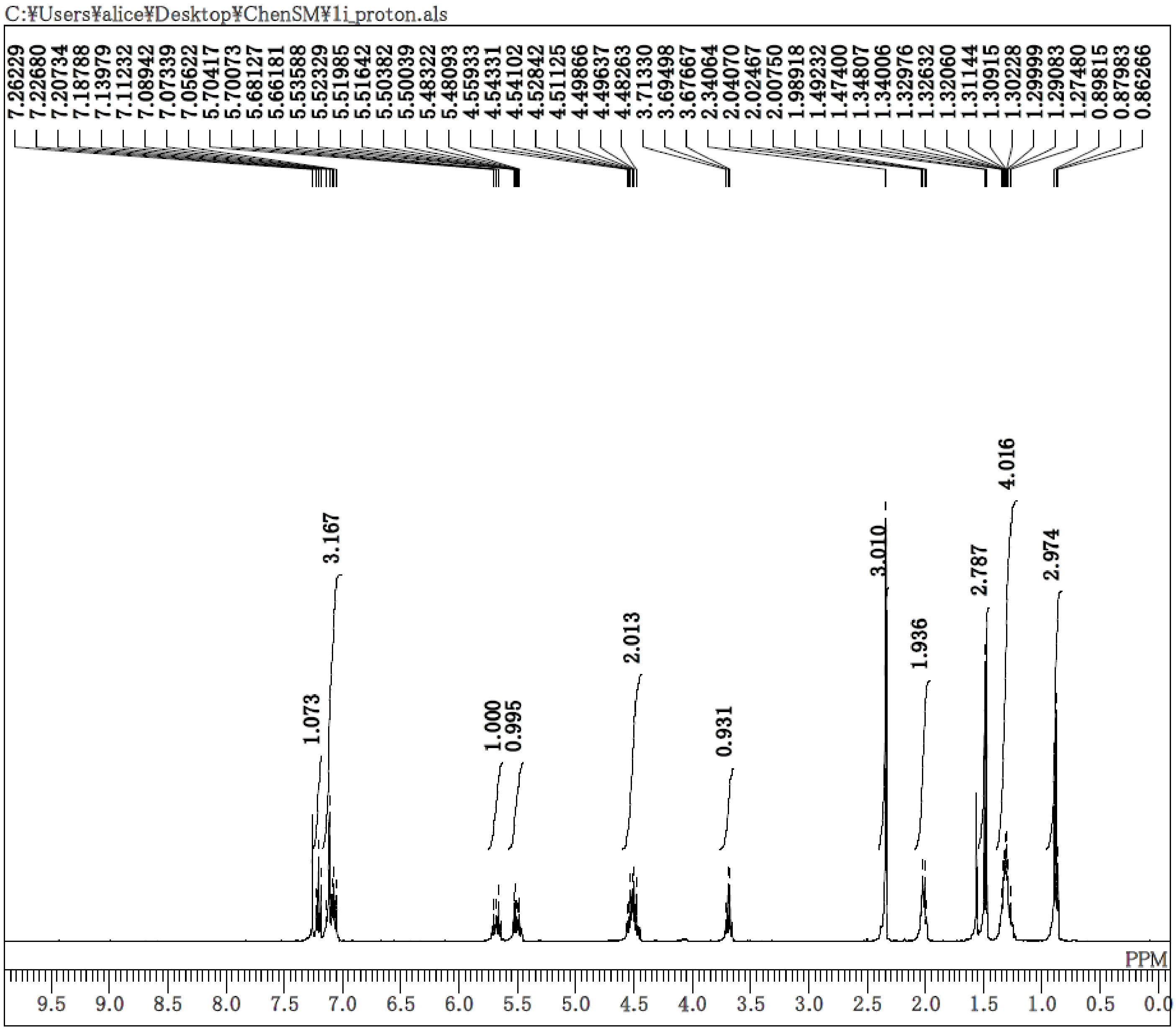


1h

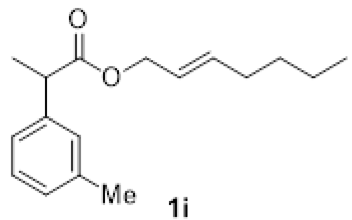
C:¥Users¥alice¥Desktop¥ChenSM¥1h_carbon.als



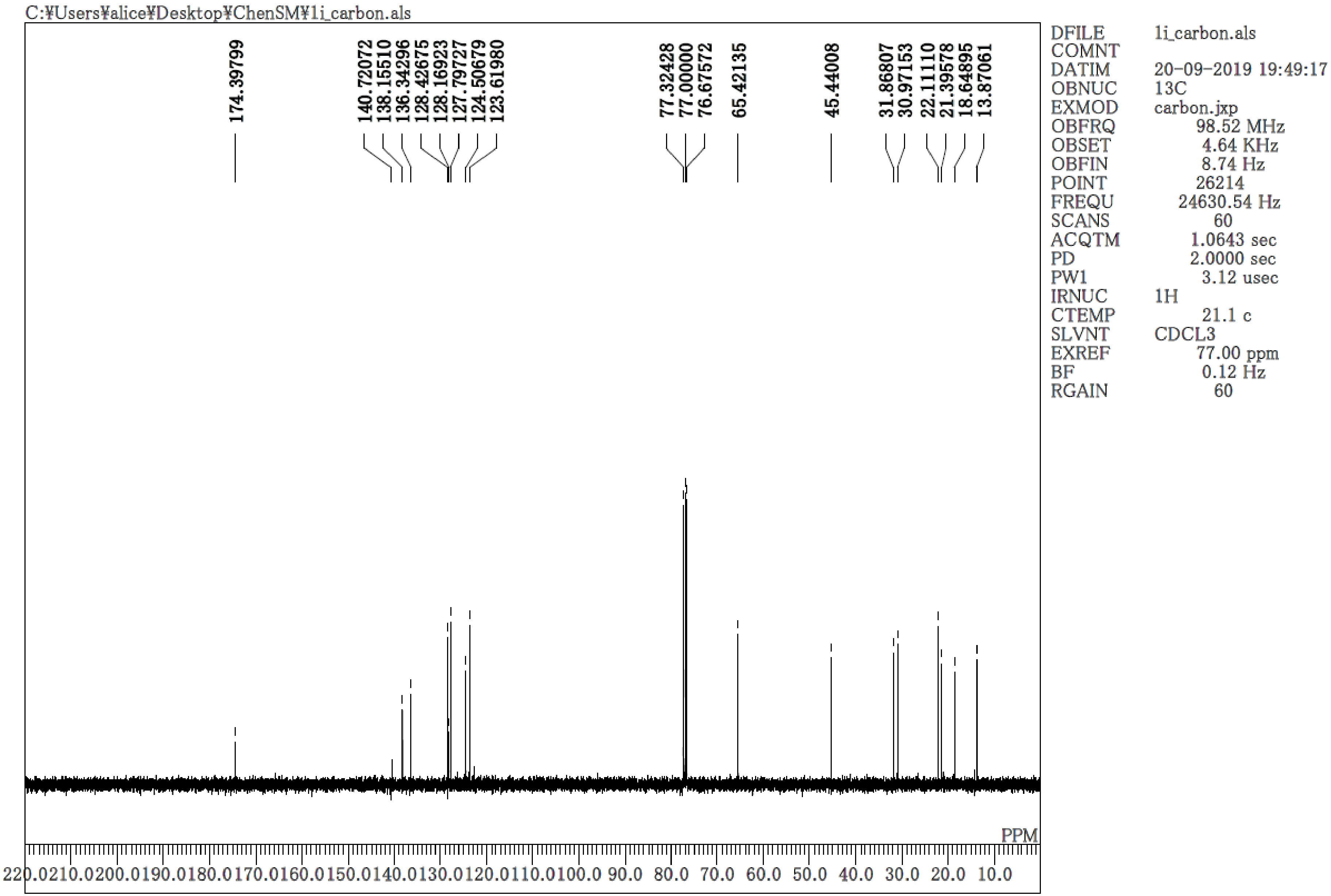
DFILE	1h_carbon.als
COMNT	
DATIM	09-11-2019 21:25:32
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	350
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE 1i_proton.als
COMNT
DATIM 20-09-2019 14:52:13
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



1i



C:¥Users¥alice¥Desktop¥ChenSM¥1i_carbon.als
174.39799
140.72072
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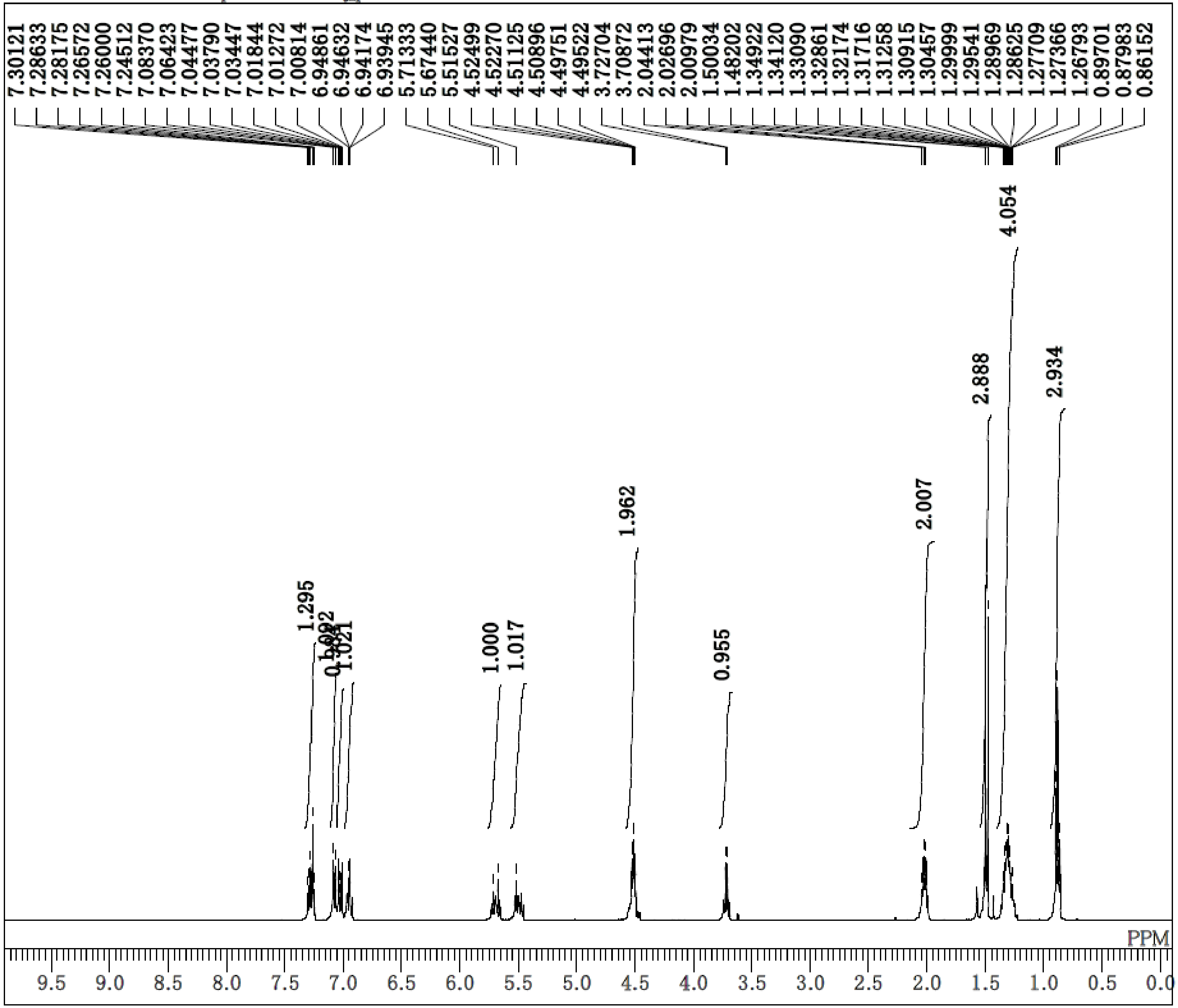
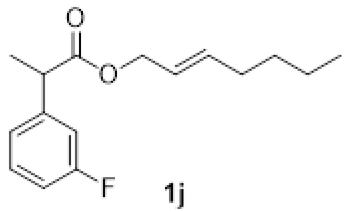
136.34296
128.42675
128.16923
127.79727
124.50679
123.61980
77.32428
77.00000
76.67572
65.42135
45.44008
31.86807
30.97153
22.11110
21.39578
18.64895
13.87061
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1i_carbon.als
COMNT
DATIM 20-09-2019 19:49:17
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 60
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

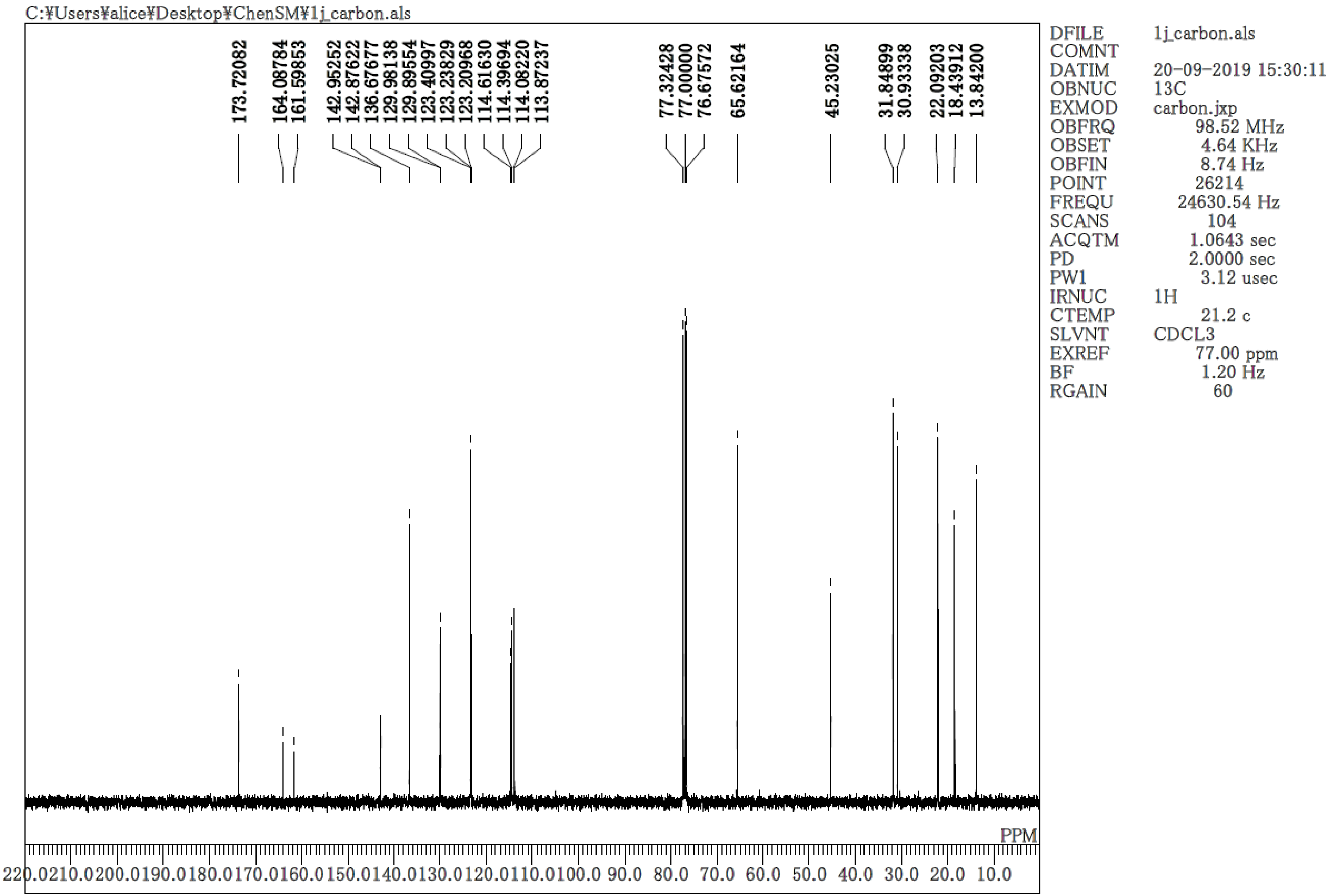
C:¥Users¥alice¥Desktop¥ChenSM¥1j_proton.als



DFILE	1j_proton.als
COMNT	
DATIM	20-09-2019 14:47:23
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



1j


C:¥Users¥alice¥Desktop¥ChenSM¥1j_carbon.als
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129.98138
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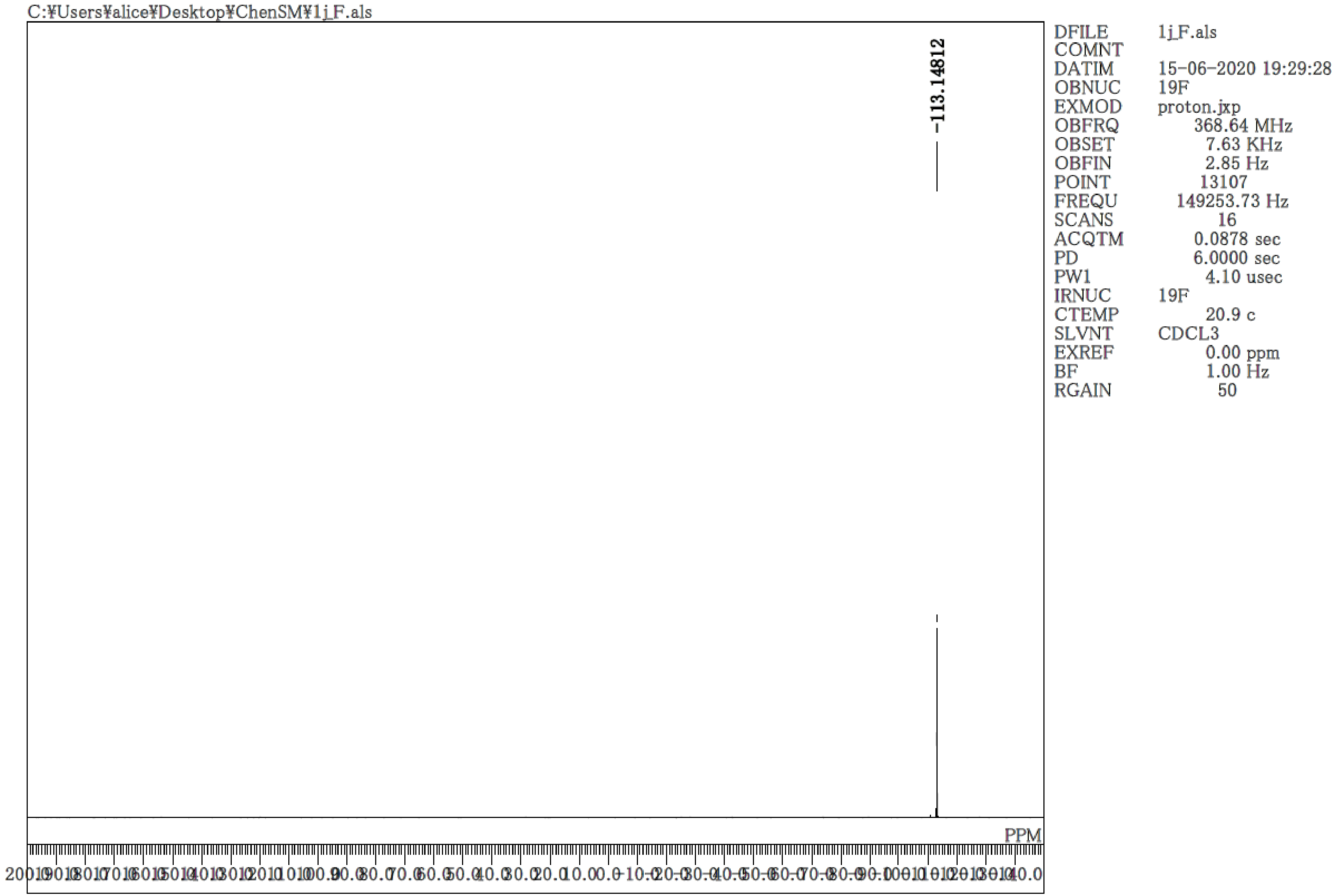
123.23829
123.20968
114.61630
114.39694
114.08220
113.87237
77.32428
77.00000
76.67572
65.62164
45.23025
31.84899
30.93338
22.09203
18.43912
13.84200
DFILE 1j_carbon.als
COMNT
DATIM 20-09-2019 15:30:11
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 104
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0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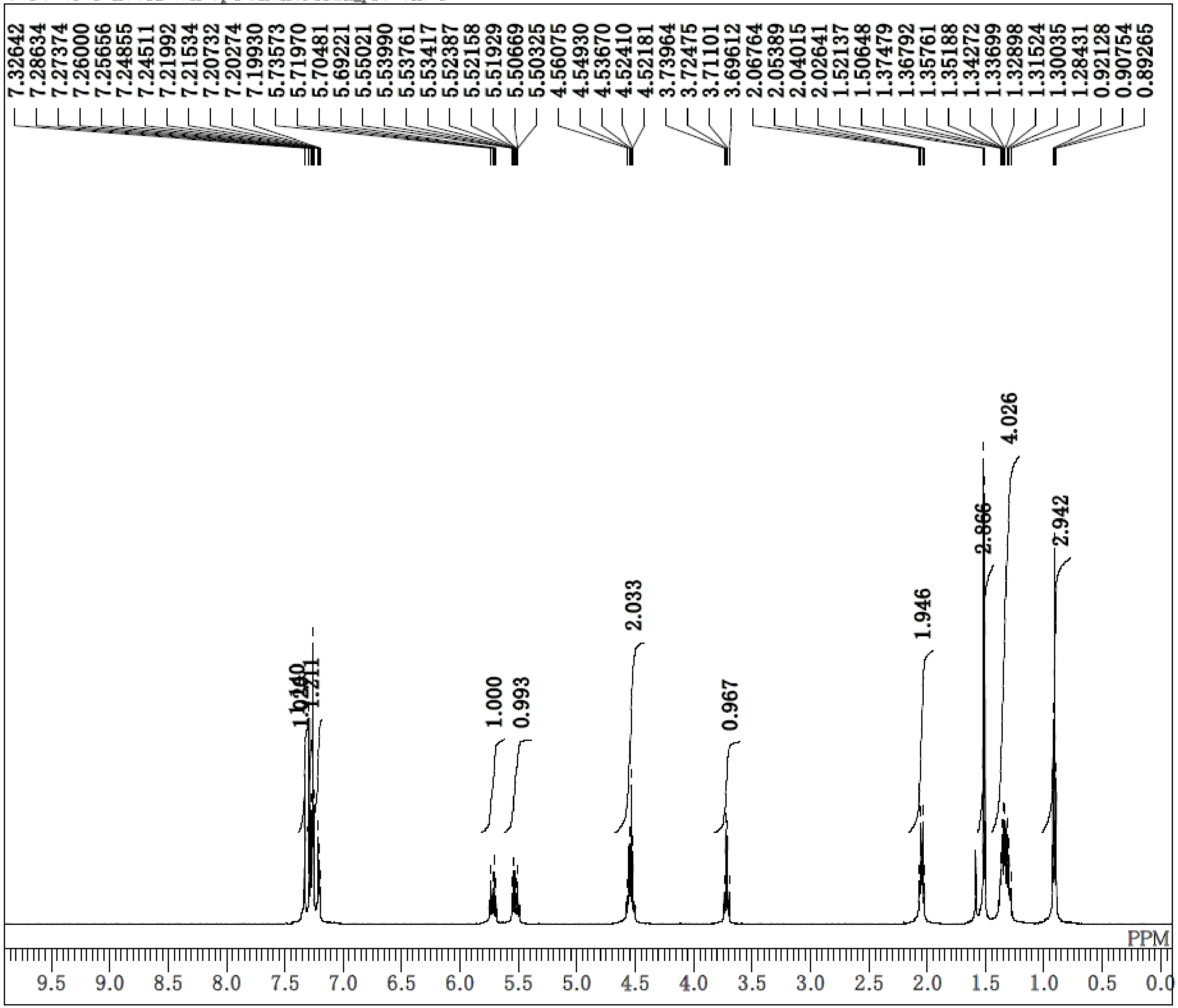
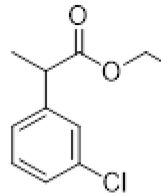
PPM
DFILE 1j_F.als
COMNT
DATIM 15-06-2020 19:29:28
OBNUC 19F
EXMOD proton.jxp
OBFRQ 368.64 MHz
OBSET 7.63 KHz
OBFIN 2.85 Hz
POINT 13107
FREQU 149253.73 Hz
SCANS 16
ACQTM 0.0878 sec
PD 6.0000 sec
PW1 4.10 usec
IRNUC 19F
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 50

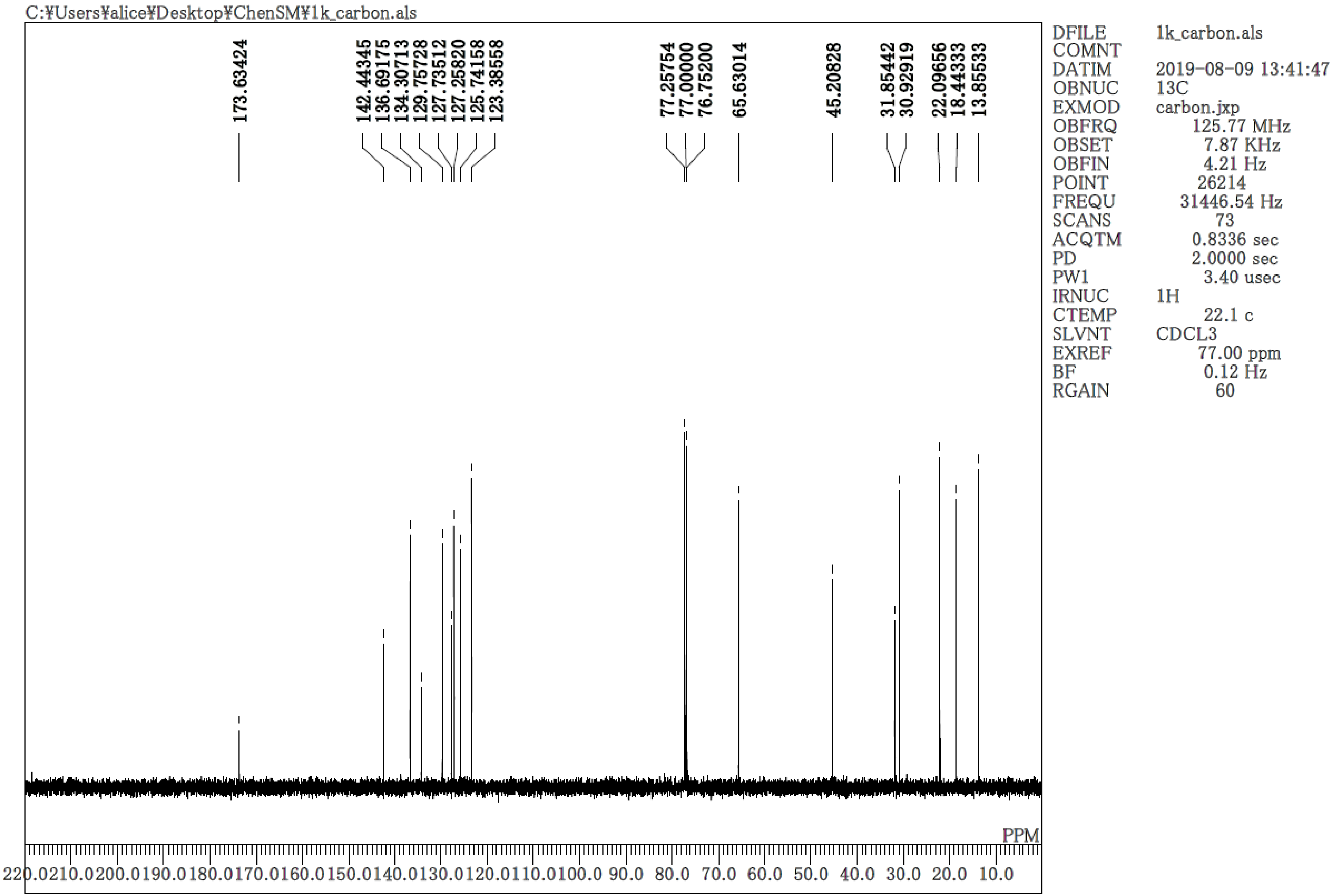
C:¥Users¥alice¥Desktop¥ChenSM¥1k_proton.als



DFILE	1k_proton.als
COMNT	
DATIM	2019-08-09 13:37:28
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



1k


C:¥Users¥alice¥Desktop¥ChenSM¥1k_carbon.als
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129.75728
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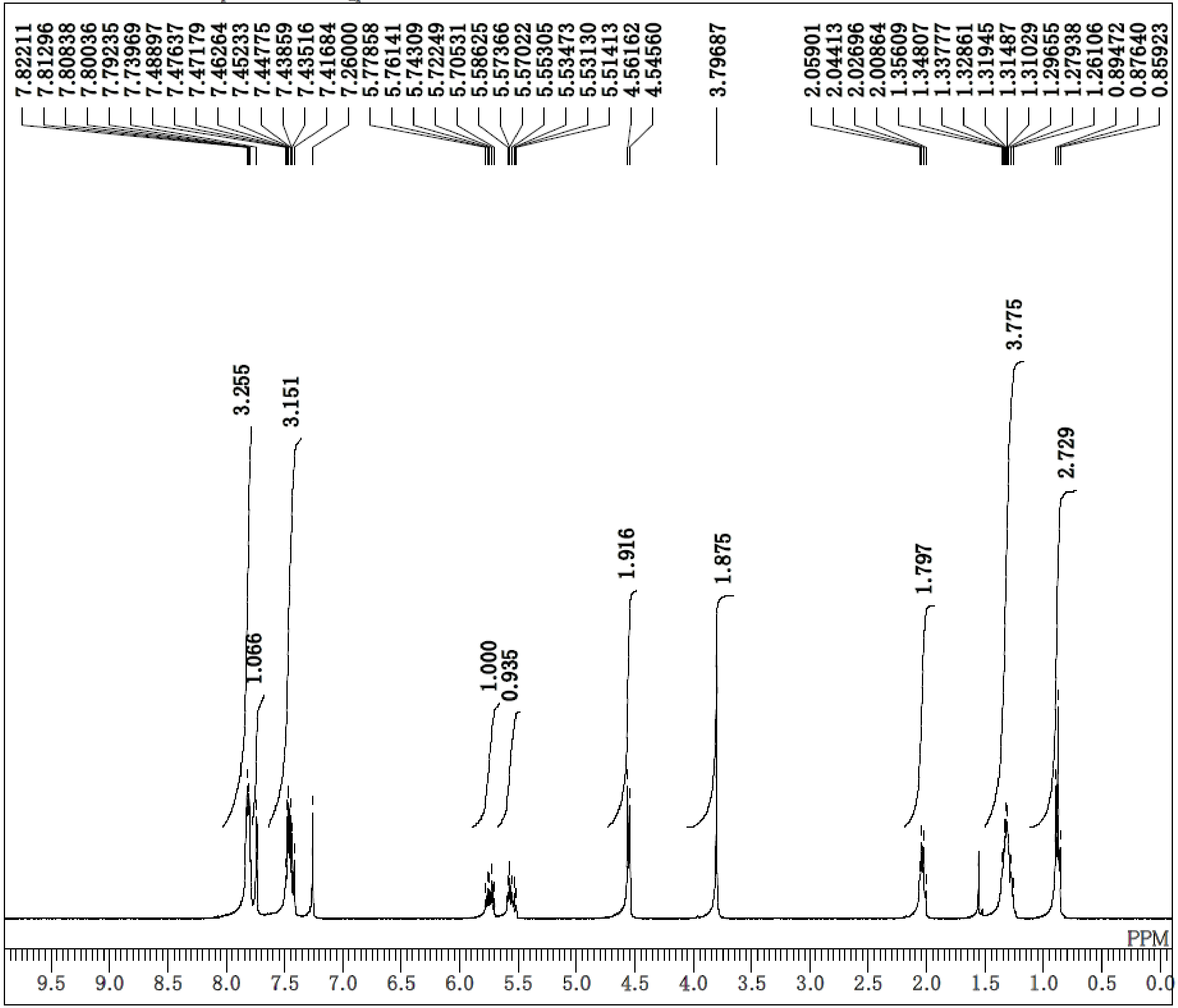
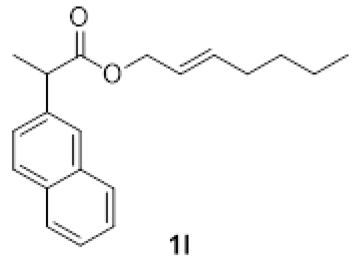
127.25820
125.74158
123.38558
77.25754
77.00000
76.75200
65.63014
45.20828
31.85442
30.92919
22.09656
18.44333
13.85533
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1k_carbon.als
COMNT
DATIM 2019-08-09 13:41:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 73
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

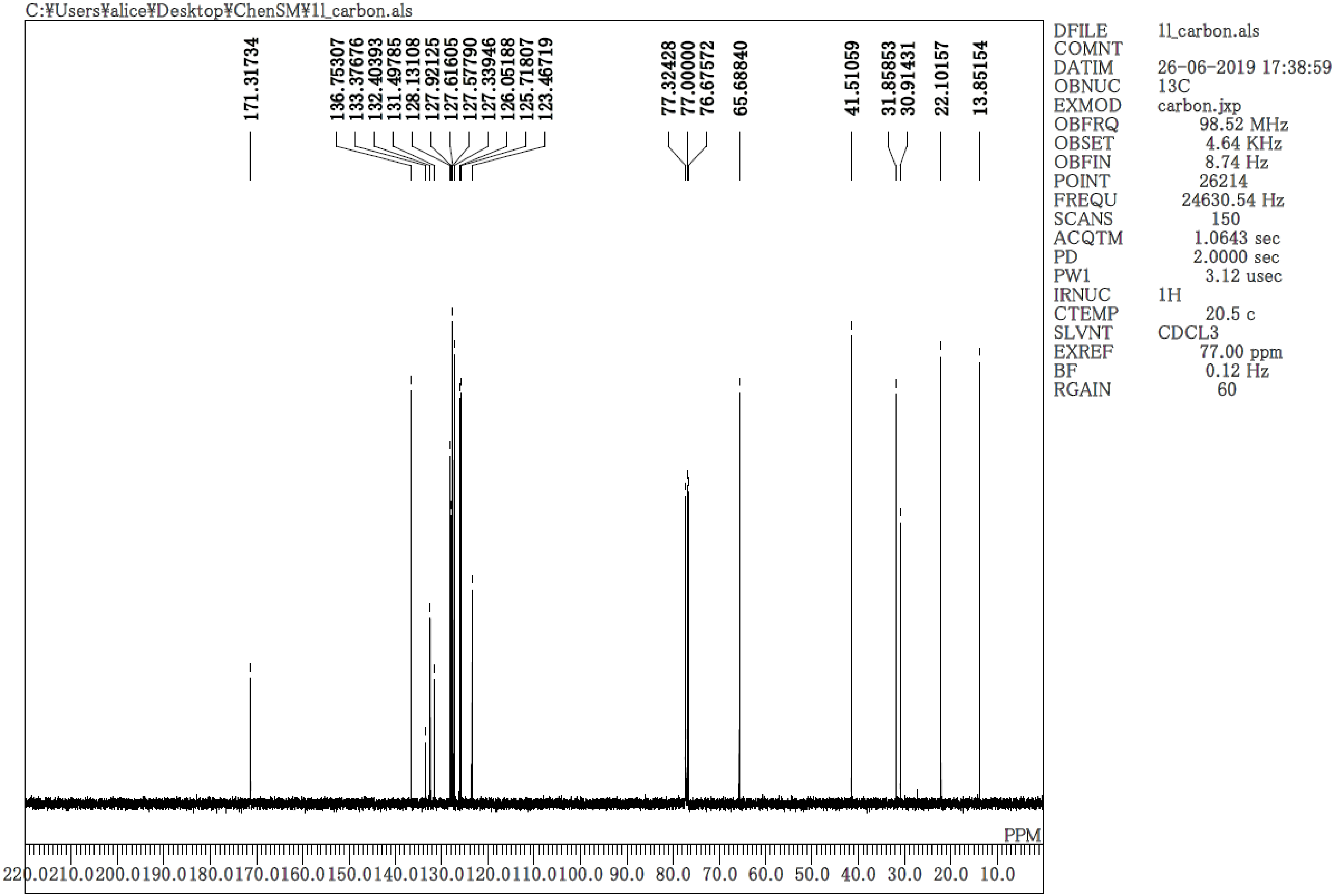
C:¥Users¥alice¥Desktop¥ChenSM¥1l_proton.als



DFILE 1l_proton.als
COMNT
DATIM 26-06-2019 17:18:19
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42



1l


C:¥Users¥alice¥Desktop¥ChenSM¥1l_carbon.als
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127.33946
126.05188
125.71807
123.46719
77.32428
77.00000
76.67572
65.68840
41.51059
31.85853
30.91431
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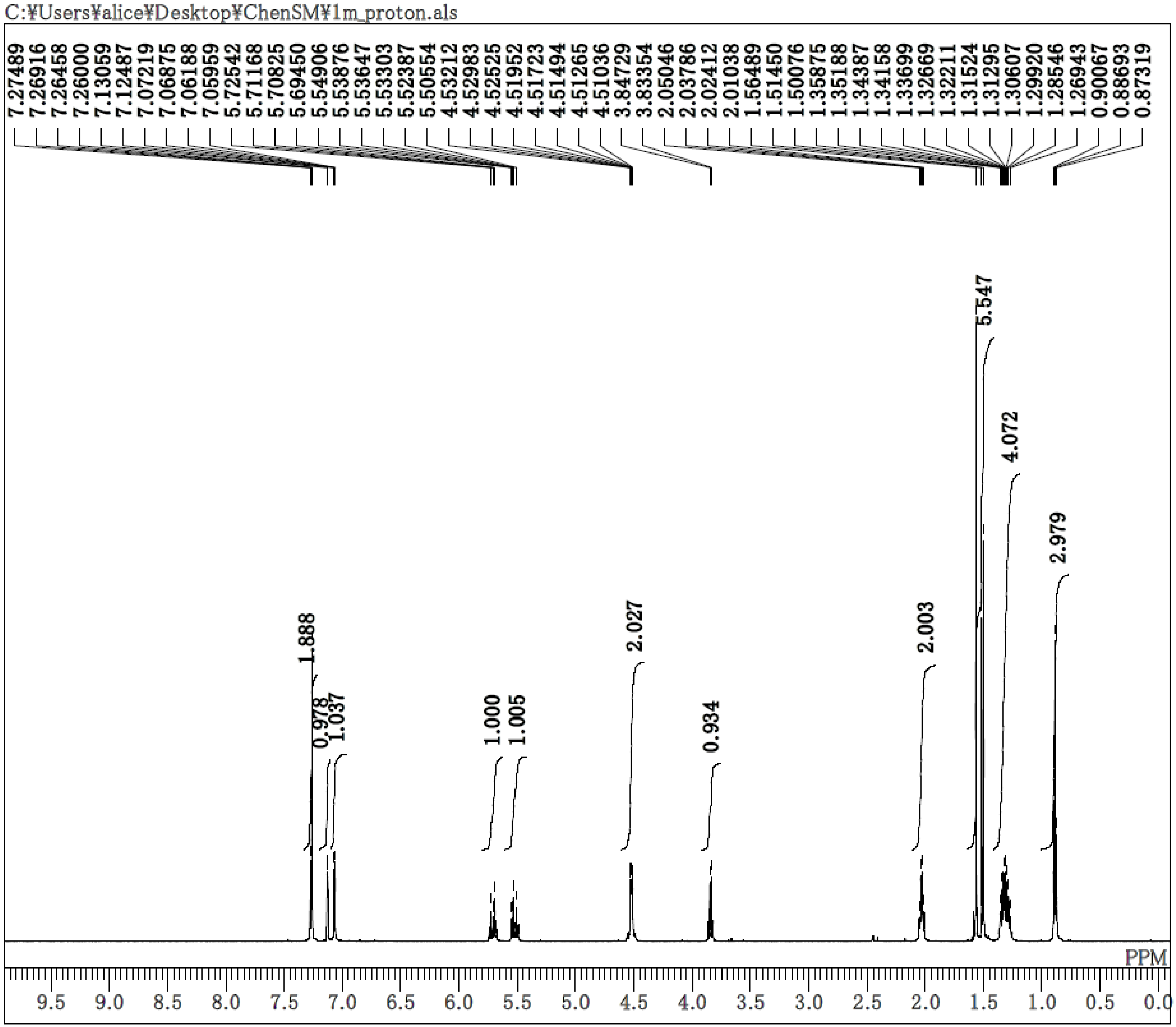
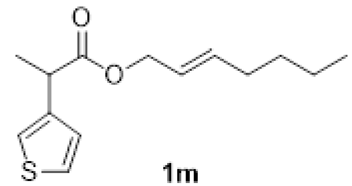
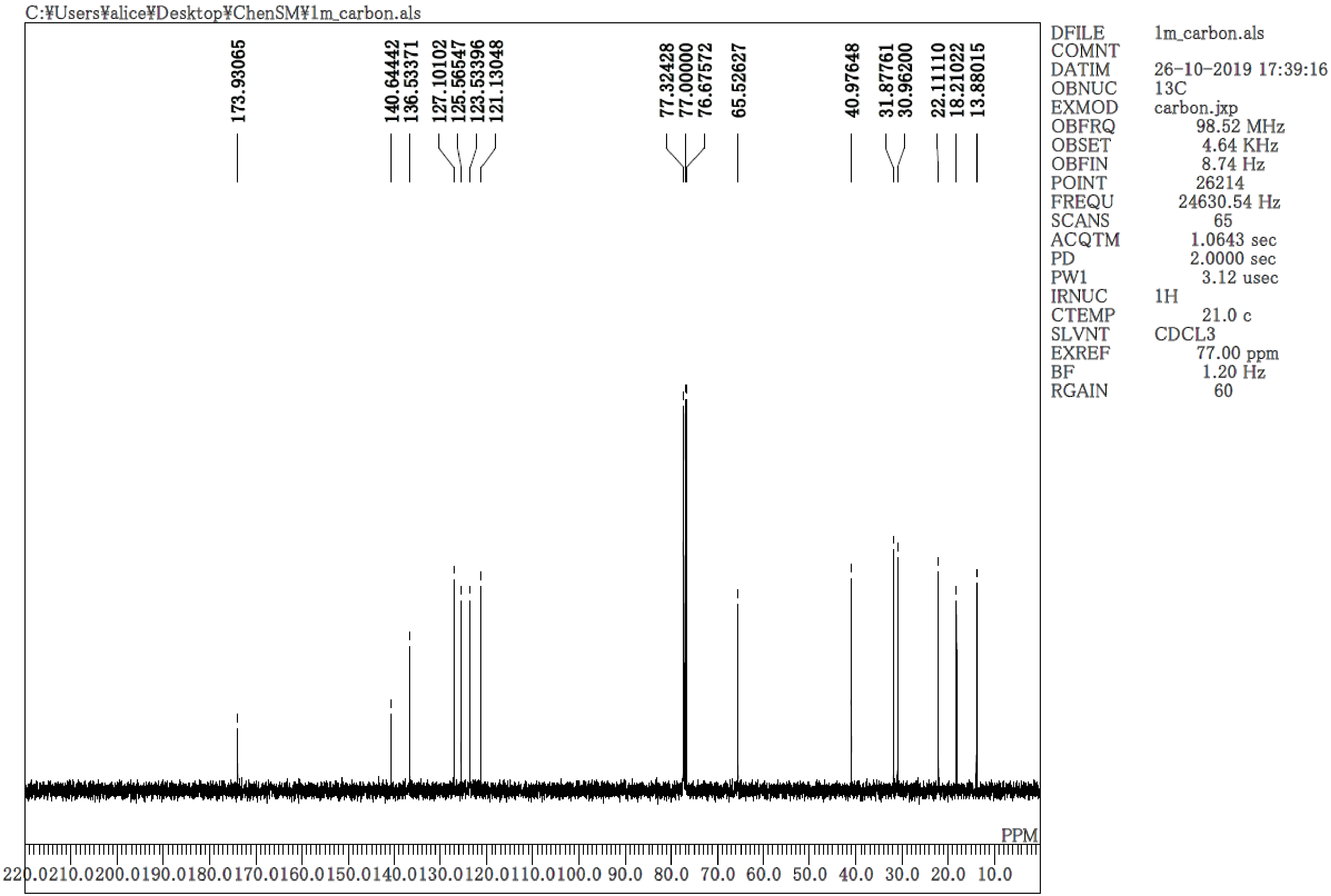
13.85154
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1l_carbon.als
COMNT
DATIM 26-06-2019 17:38:59
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 150
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



DFILE	1m_proton.als
COMNT	
DATIM	2019-10-26 16:41:17
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50



1m


C:¥Users¥alice¥Desktop¥ChenSM¥1m_carbon.als
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121.13048
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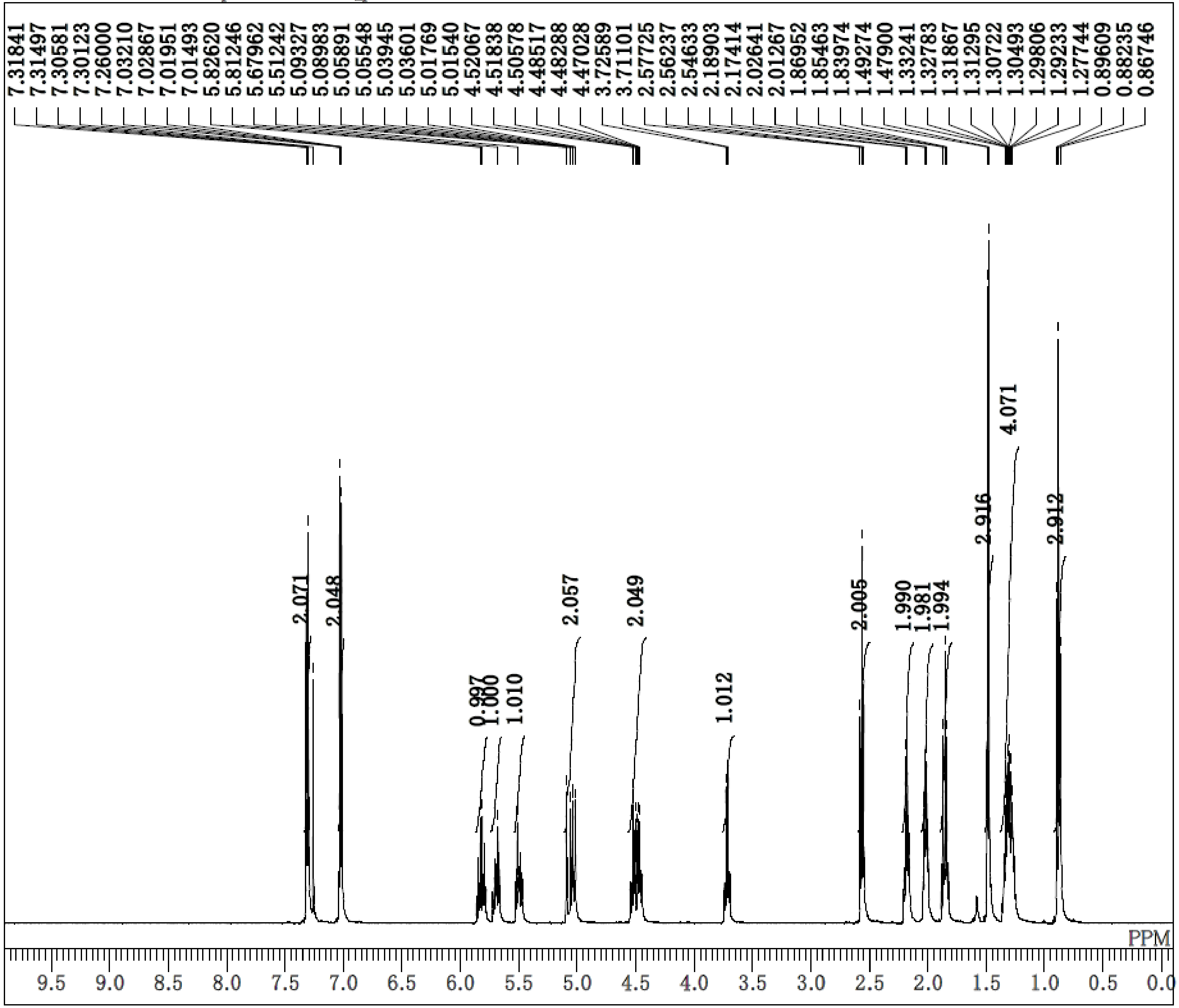
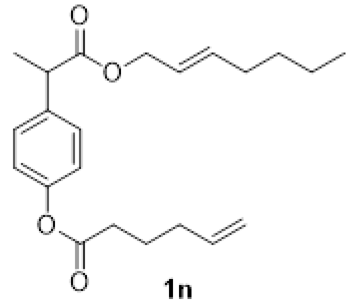
77.00000
76.67572
65.52627
40.97648
31.87761
30.96200
22.11110
18.21022
13.88015
PPM
DFILE 1m_carbon.als
COMNT
DATIM 26-10-2019 17:39:16
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 65
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenSM¥1n_proton.als



DFILE 1n_proton.als
COMNT
DATIM 2019-12-13 23:14:22
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



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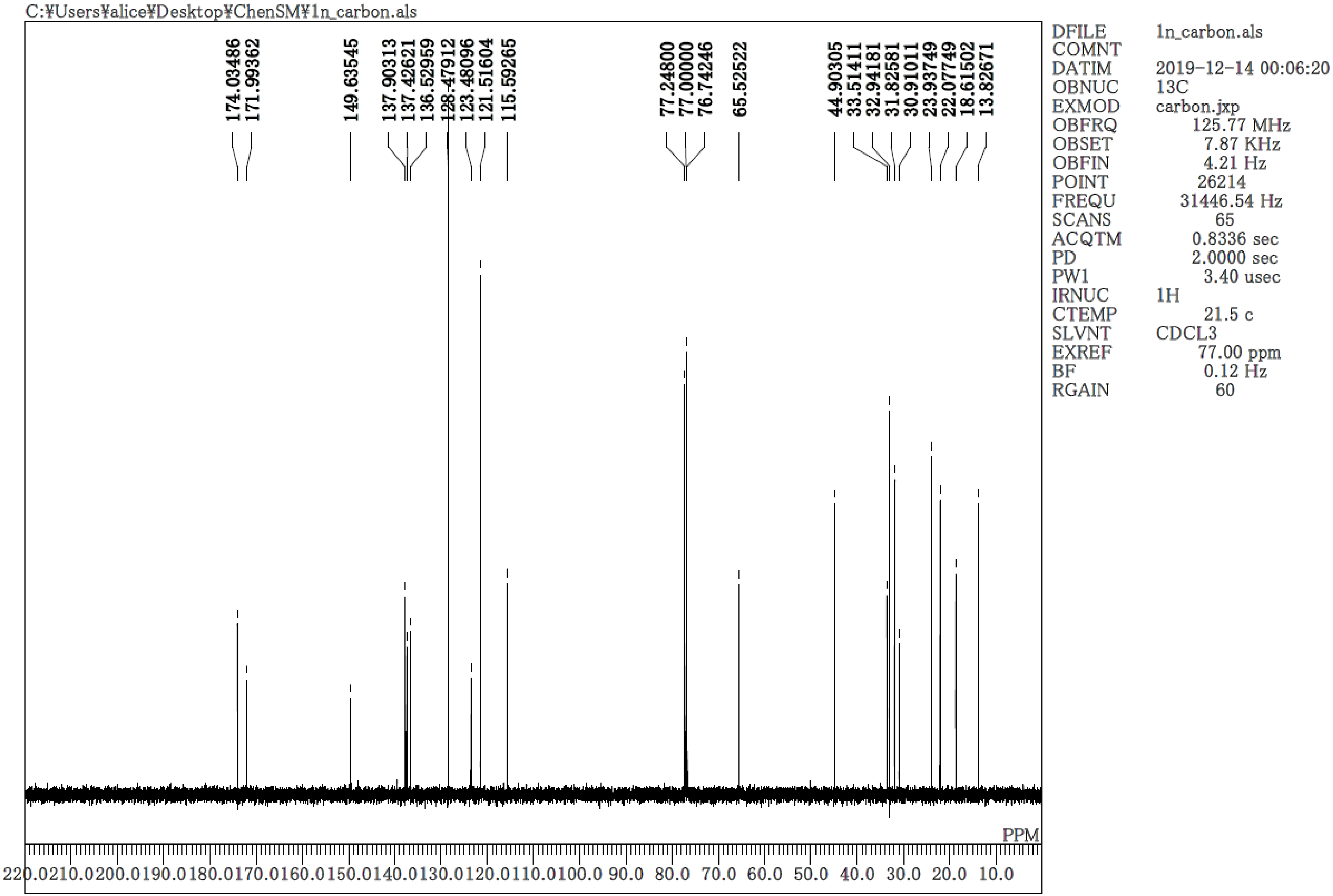

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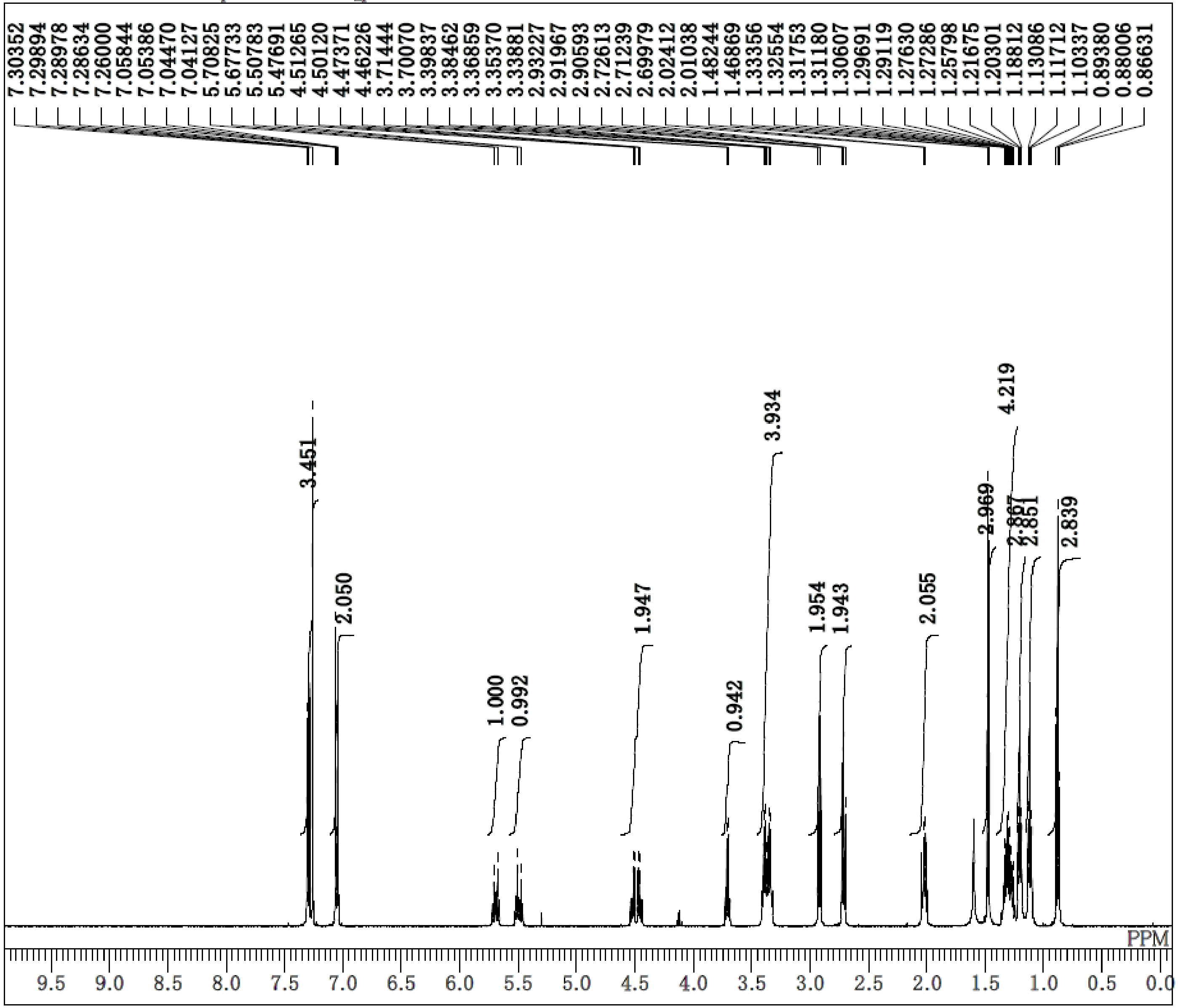
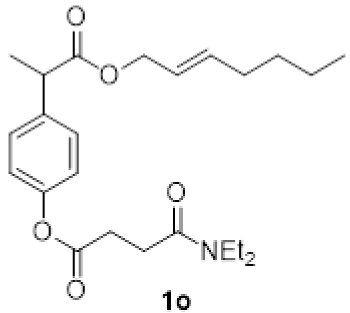
44.90305
33.51411
32.94181
31.82581
30.91011
23.93749
22.07749
18.61502
13.82671
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1n_carbon.als
COMNT
DATIM 2019-12-14 00:06:20
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 65
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenSM¥1o_proton.als



DFILE	1o_proton.als
COMNT	
DATIM	2019-12-05 14:09:11
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



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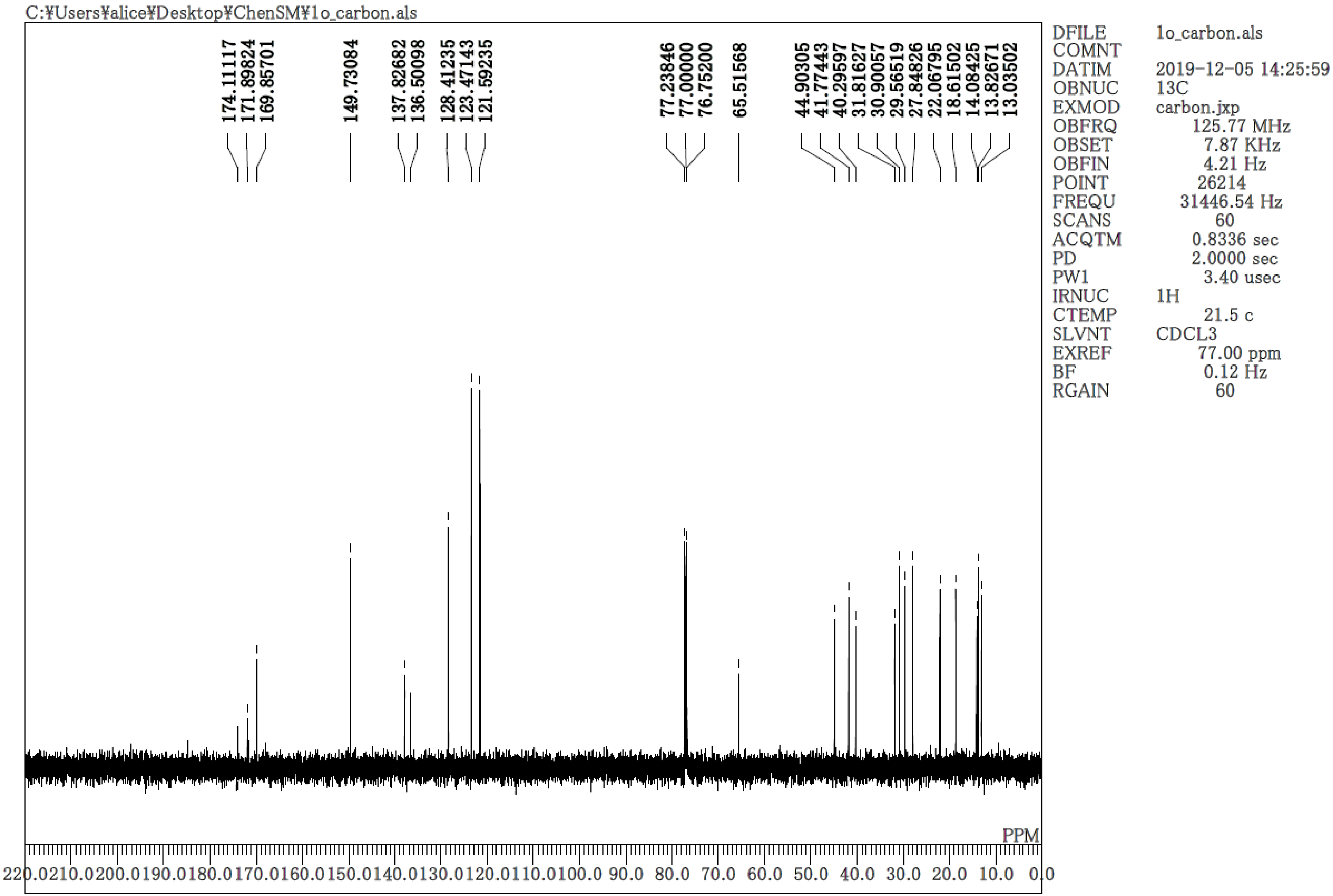
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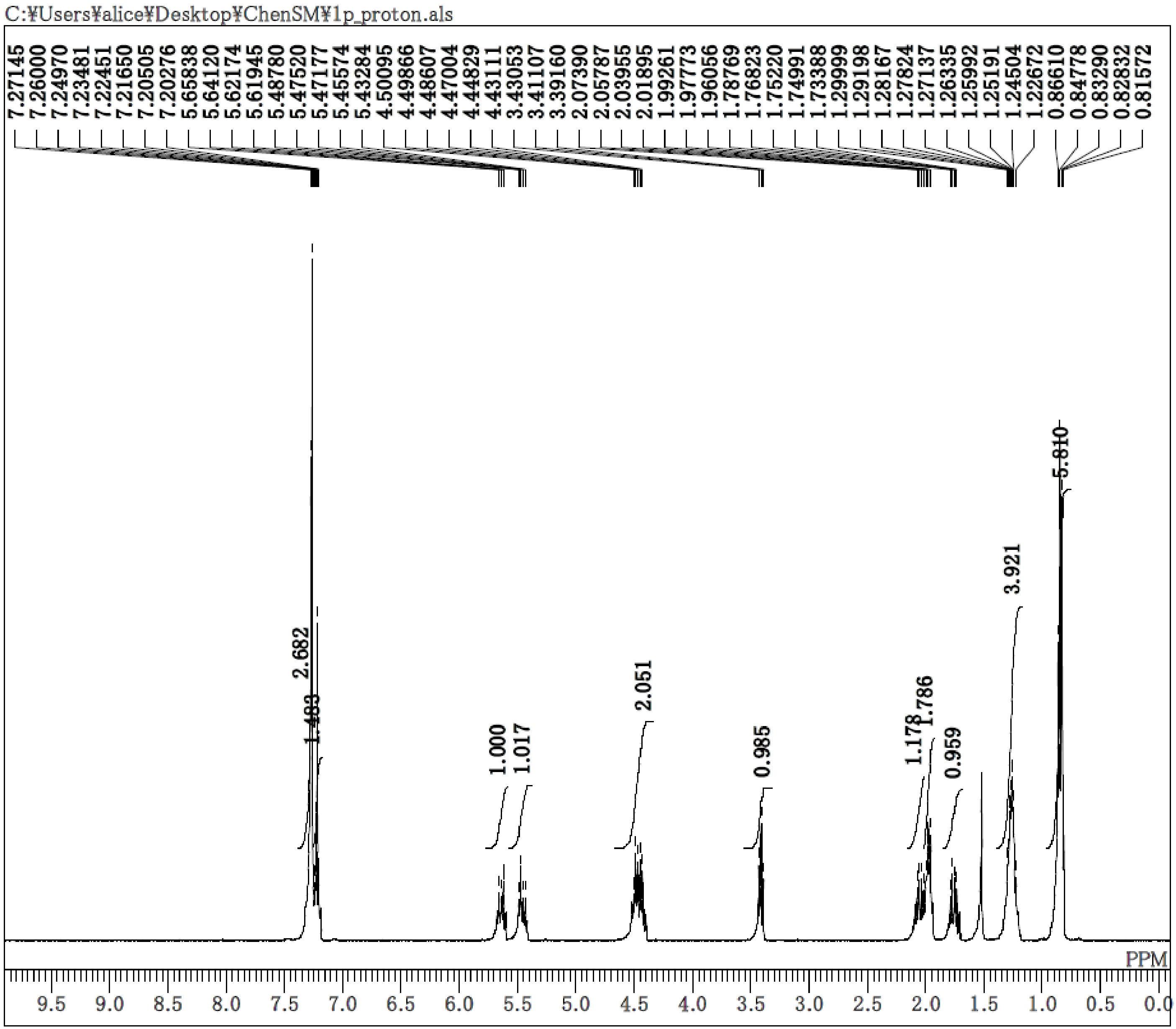
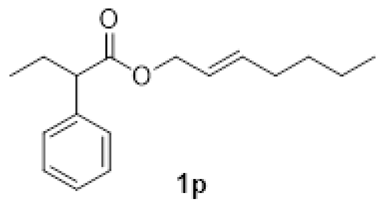
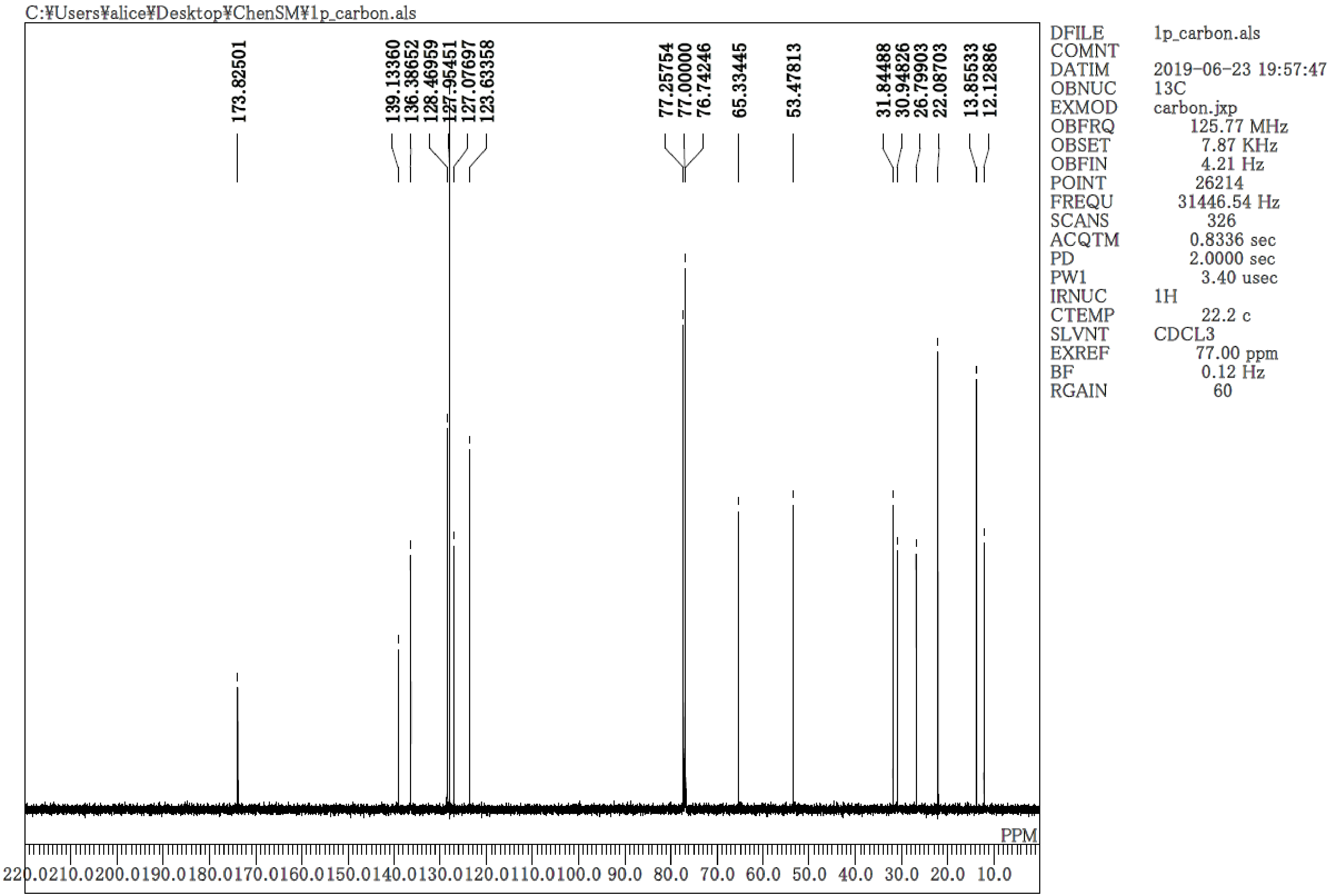
77.23846
77.00000
76.75200
65.51568
44.90305
41.77443
40.29597
31.81627
30.90057
29.56519
27.84826
22.06795
18.61502
14.08425
13.82671
13.03502
DFILE 1o_carbon.als
COMNT
DATIM 2019-12-05 14:25:59
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 60
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0



DFILE	1p_proton.als
COMNT	
DATIM	23-06-2019 19:55:29
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



1p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128.46959
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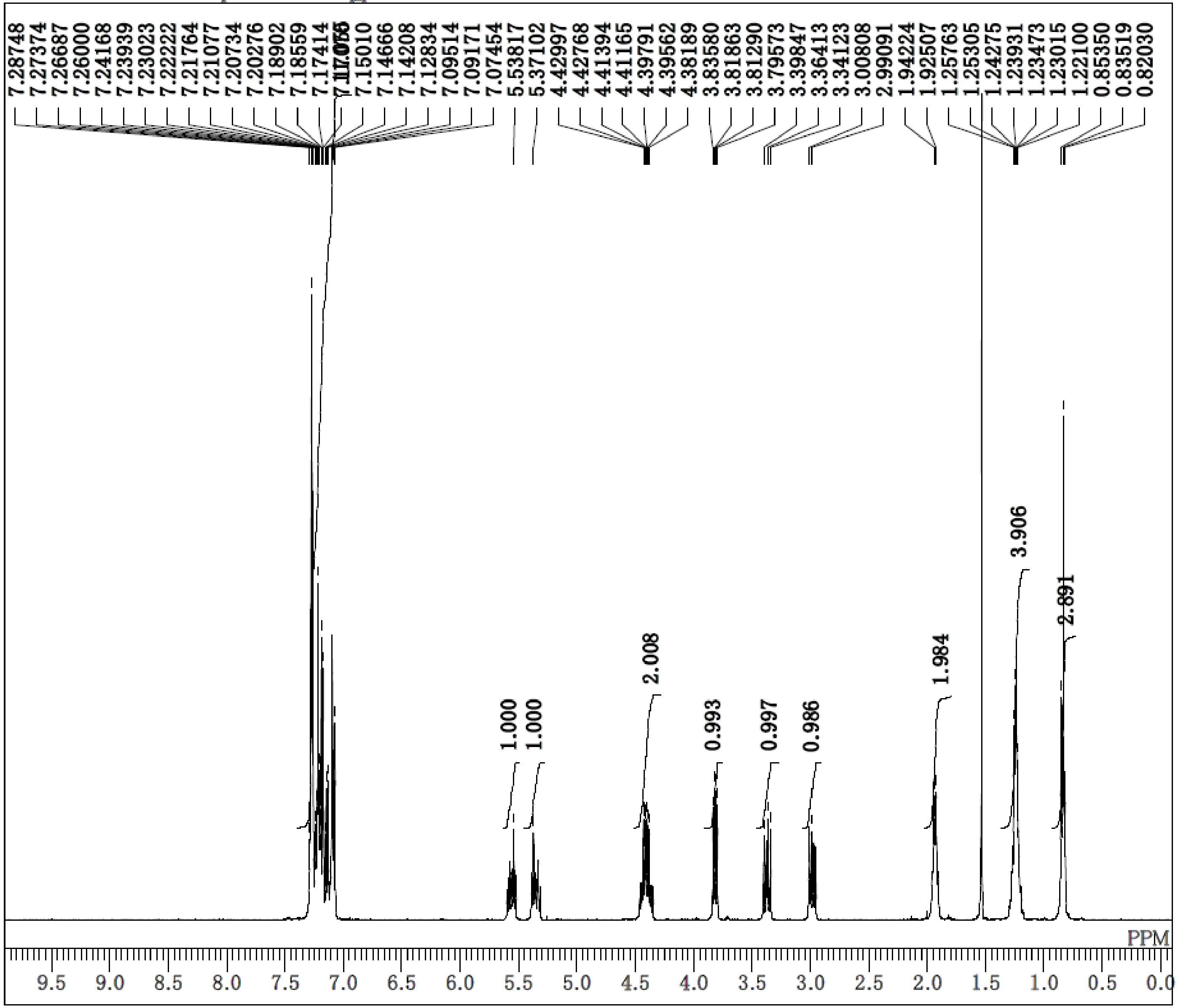
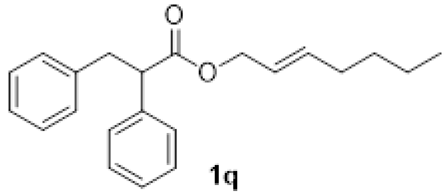
53.47813
31.84488
30.94826
26.79903
22.08703
13.85533
12.12886
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1p_carbon.als
COMNT
DATIM 2019-06-23 19:57:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 326
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenSM¥1q_proton.als

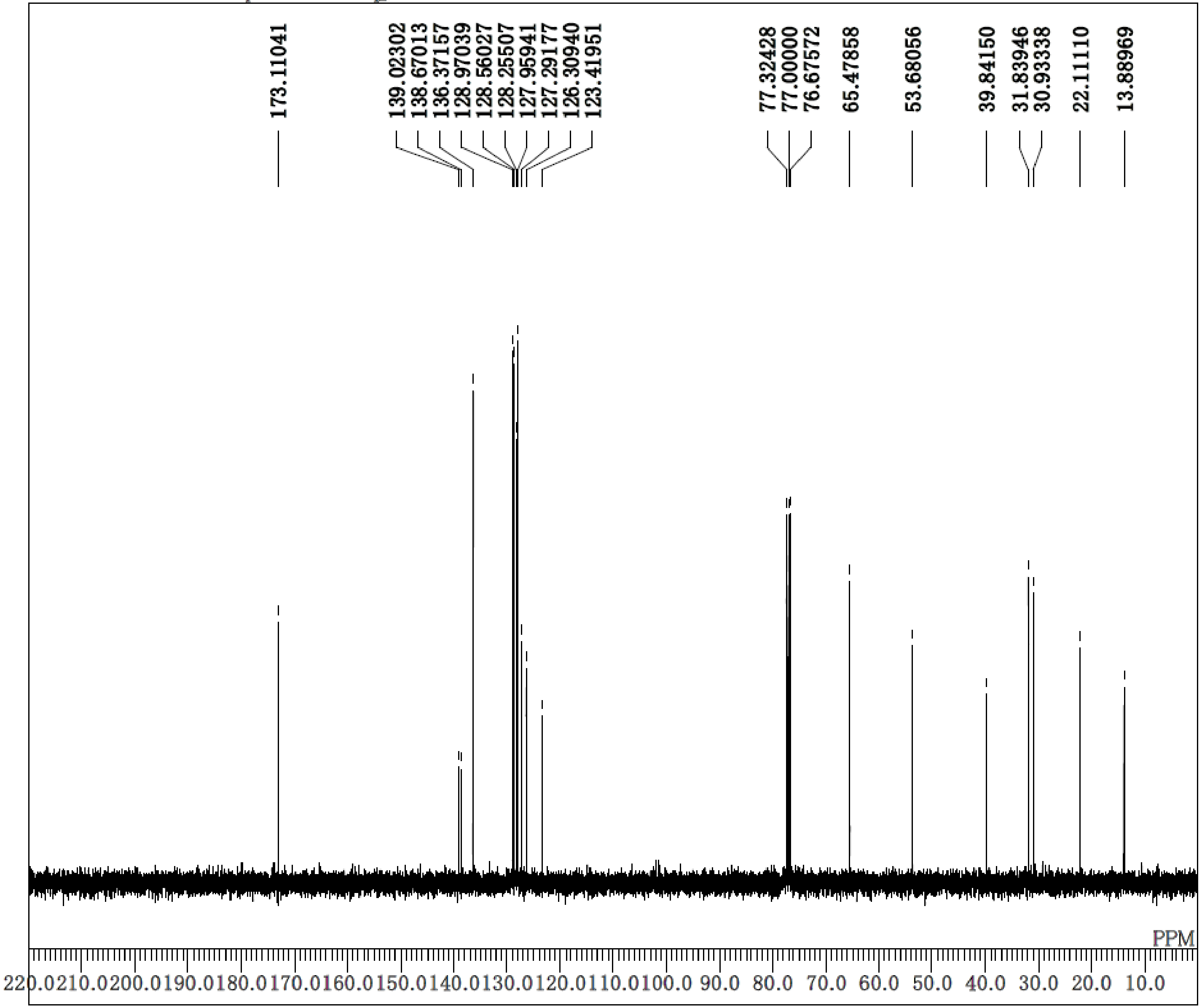


DFILE	1q_proton.als
COMNT	
DATIM	10-08-2019 15:42:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



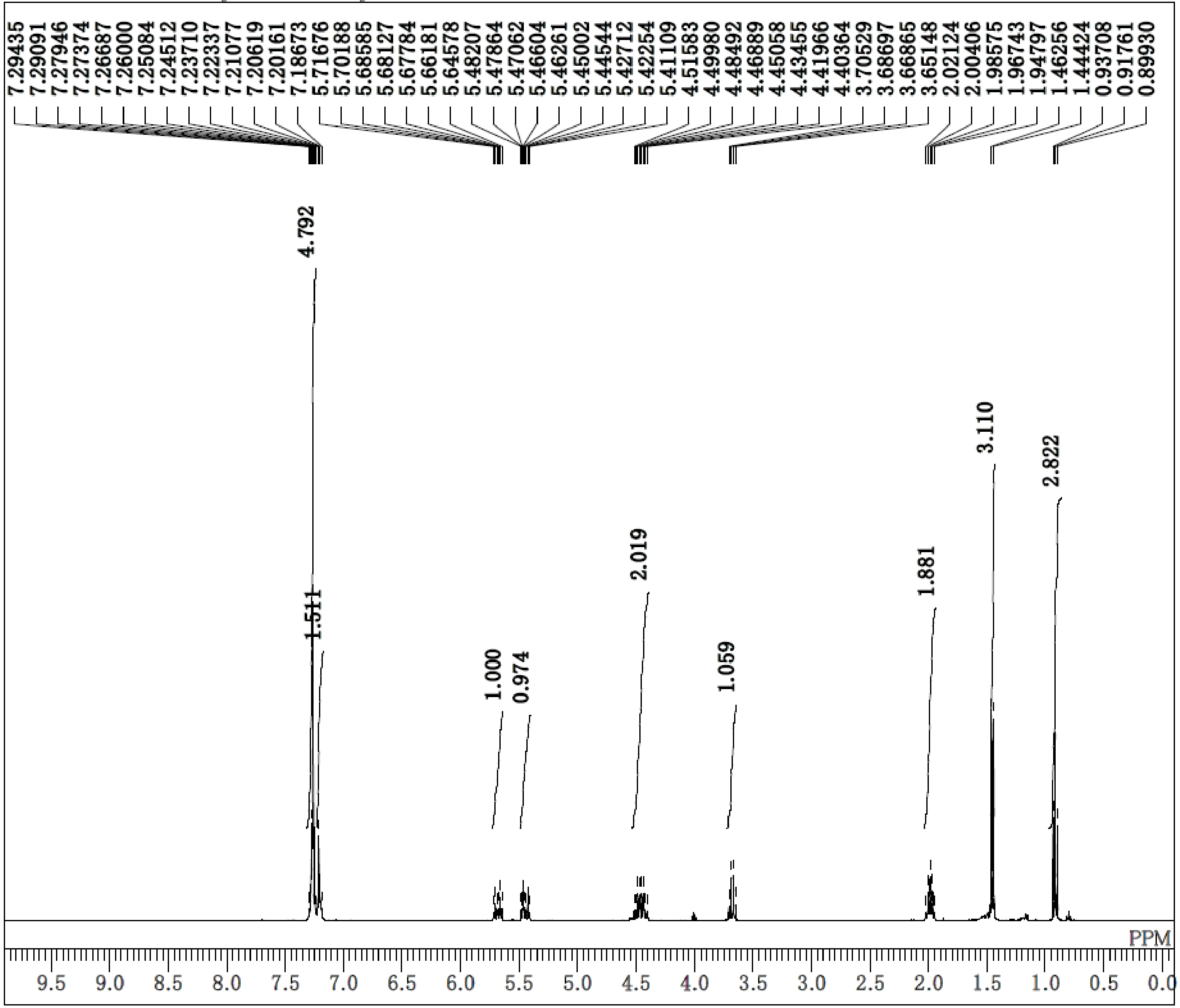
1q

C:¥Users¥alice¥Desktop¥ChenSM¥1q_carbon.als

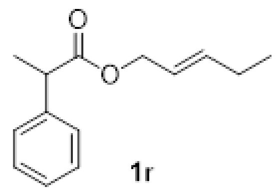


DFILE	1q_carbon.als
COMNT	
DATIM	10-08-2019 15:53:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	120
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenSM¥1r_protoin.als

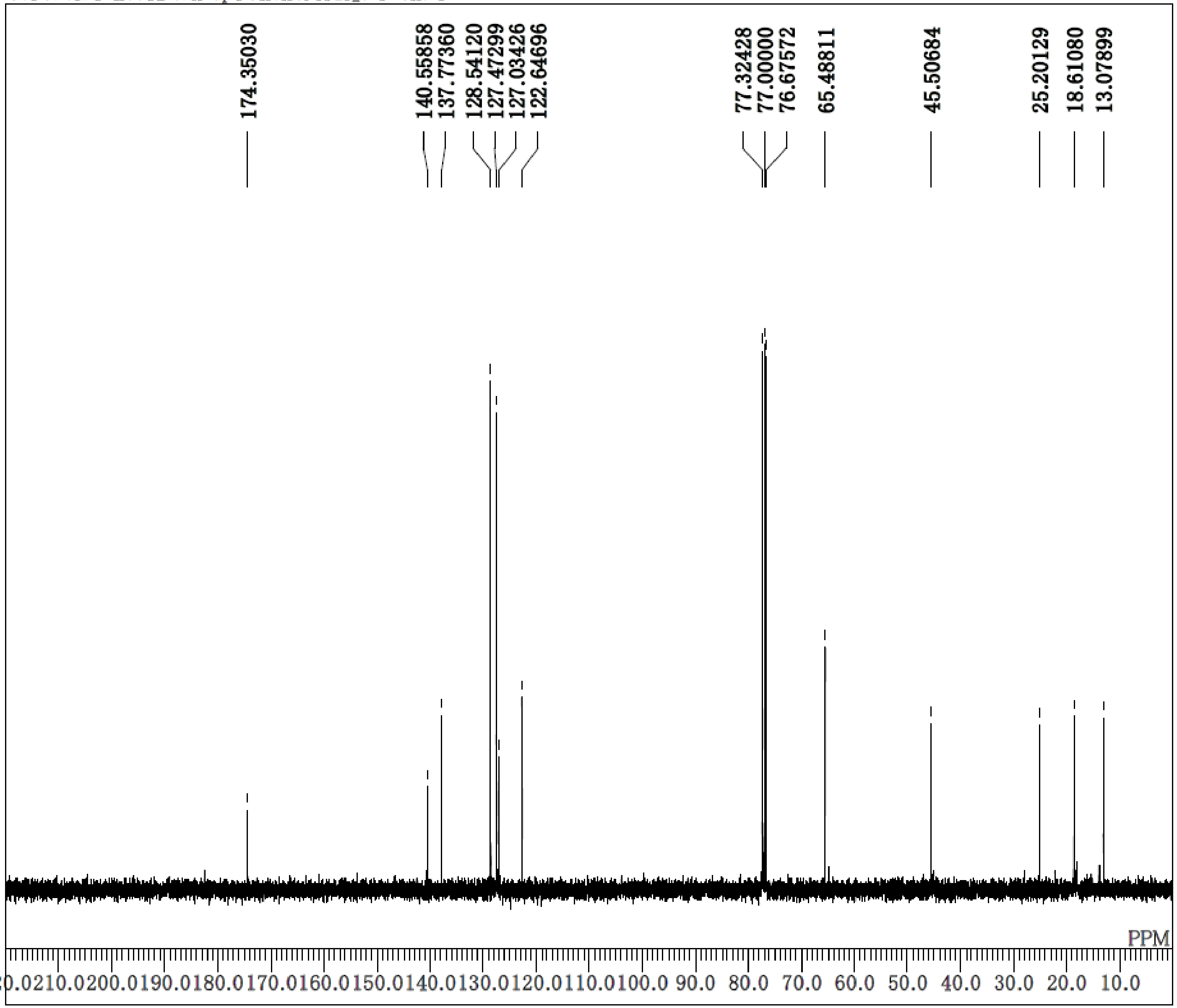


DFILE 1r_protoin.als
COMNT
DATIM 25-03-2020 20:58:12
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



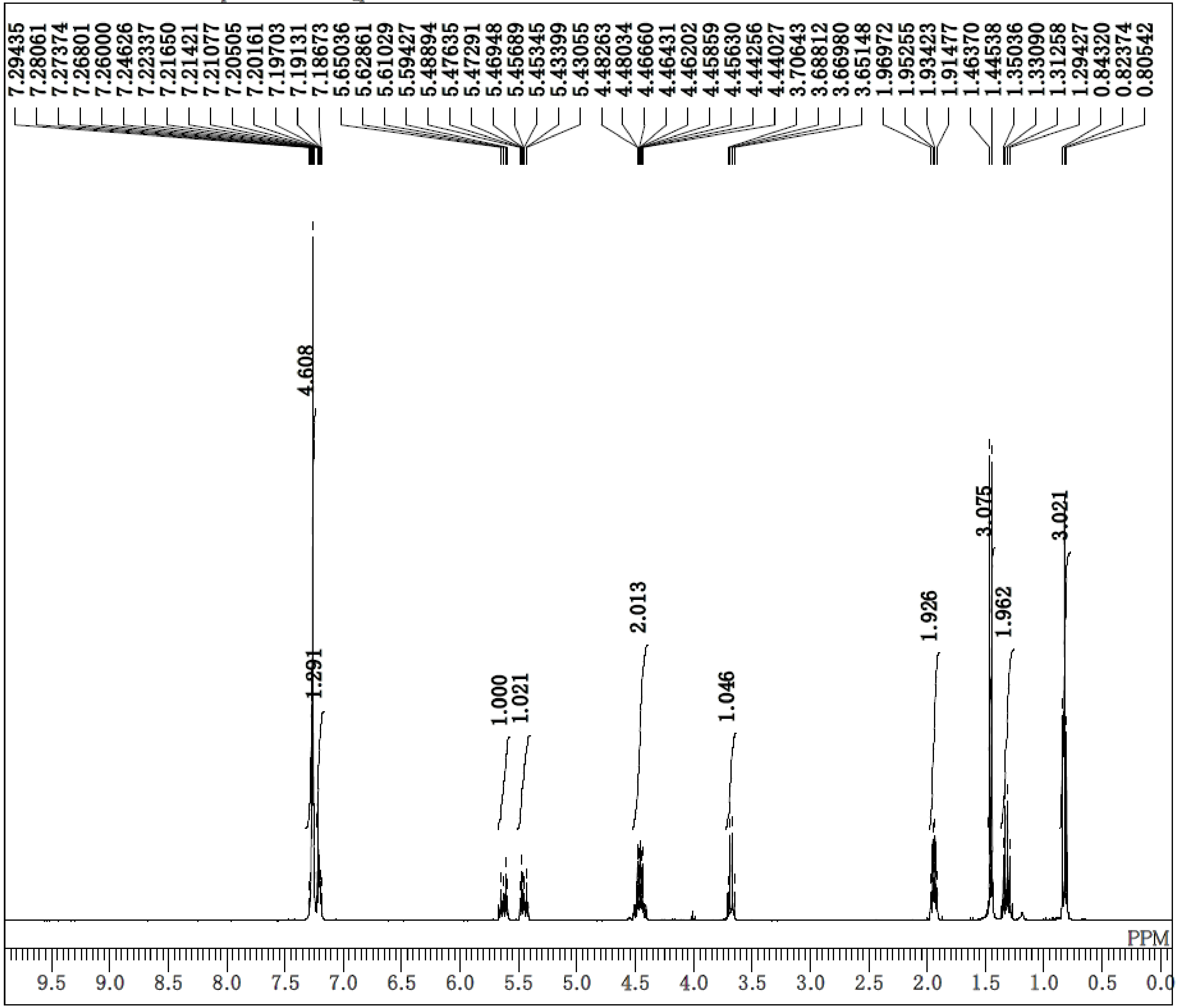
1r

C:¥Users¥alice¥Desktop¥ChenSM¥1r_carbon.als

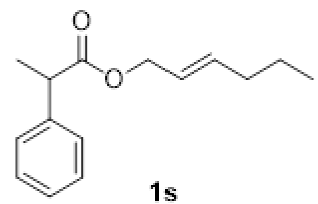


DFILE	1r_carbon.als
COMNT	
DATIM	25-03-2020 13:05:47
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	189
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

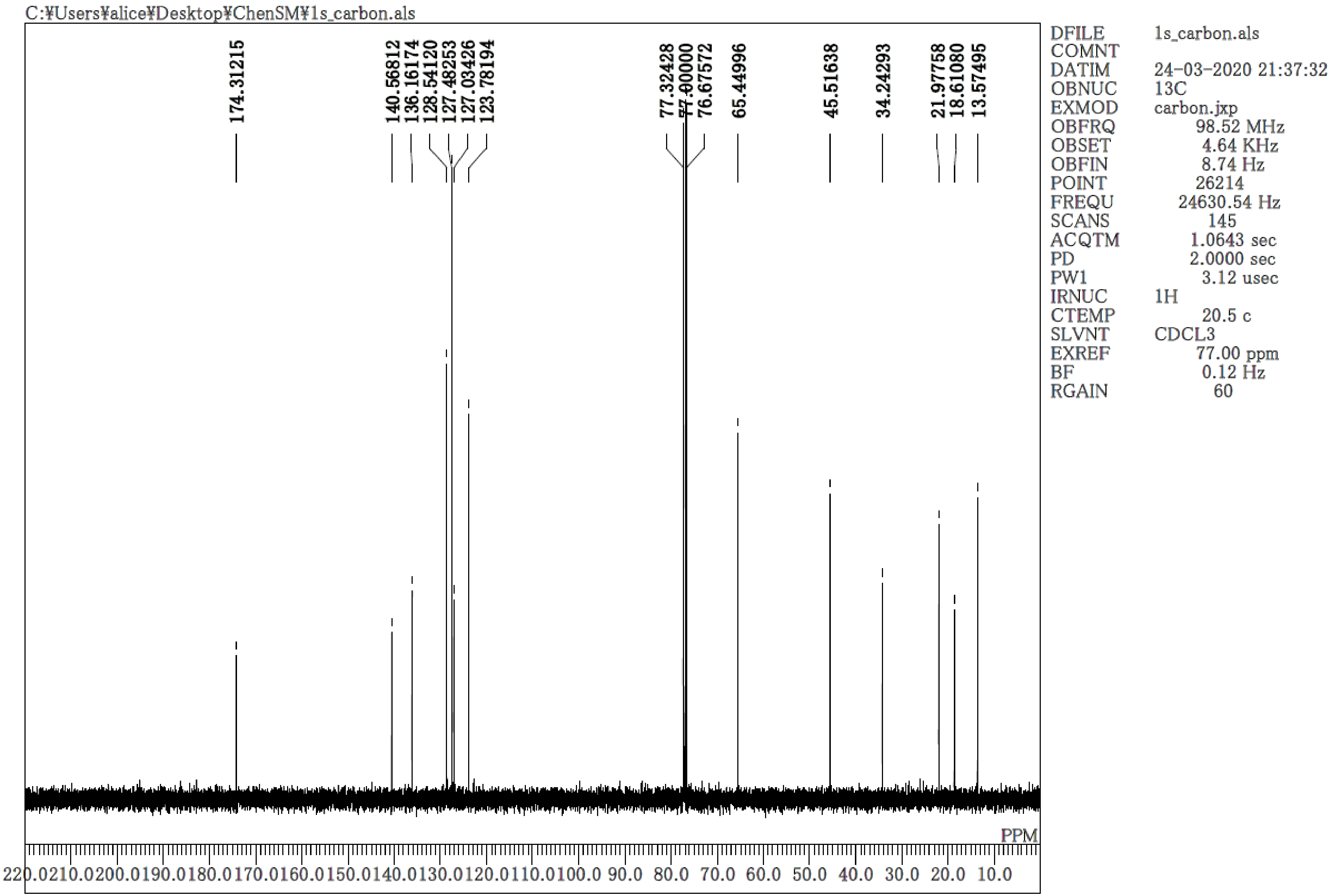
C:¥Users¥alice¥Desktop¥ChenSM¥1s_proton.als



DFILE	1s_proton.als
COMNT	
DATIM	24-03-2020 21:32:43
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34



1s


C:¥Users¥alice¥Desktop¥ChenSM¥1s_carbon.als
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136.16174
128.54120
127.48253
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123.78194
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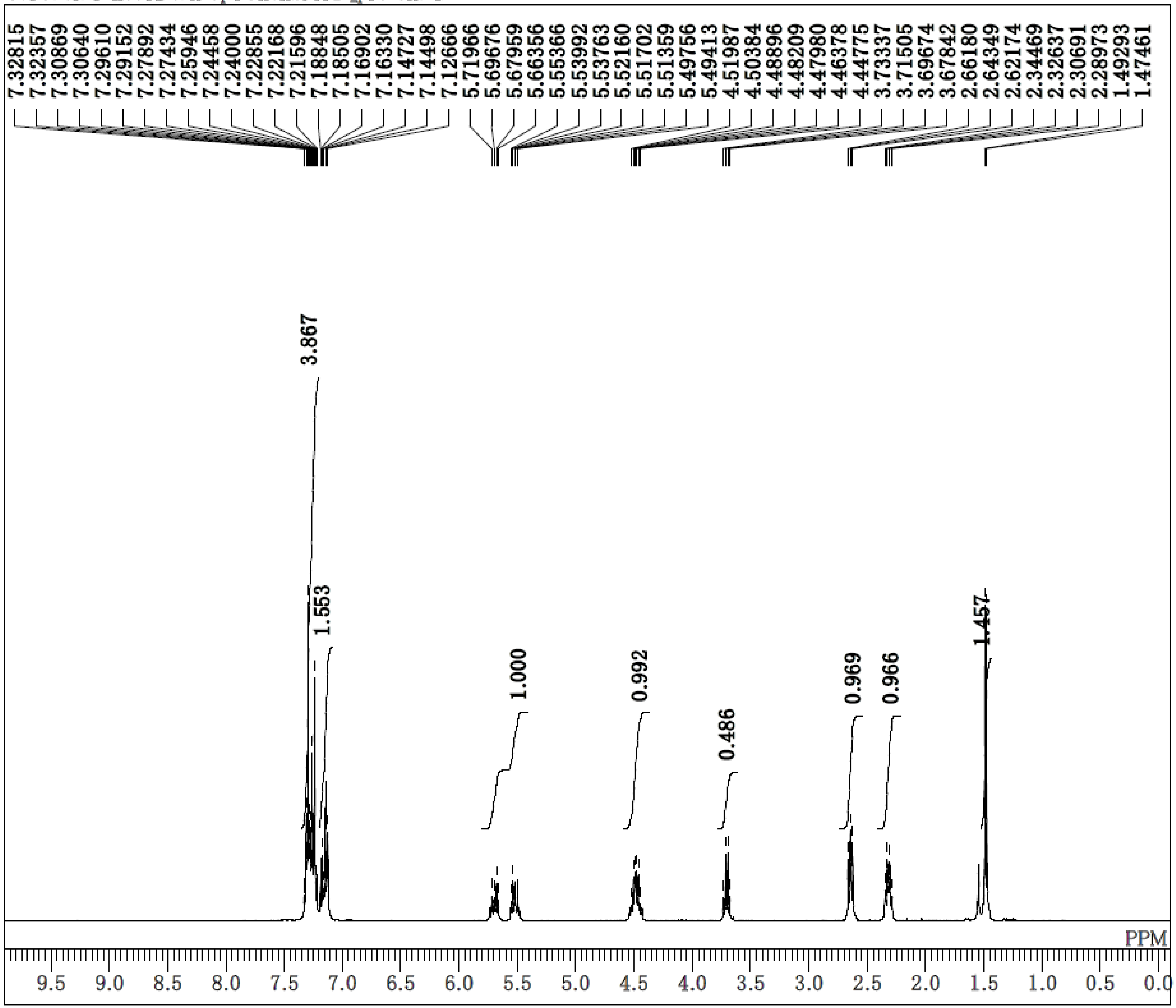
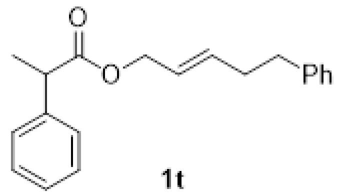
65.44996
45.51638
34.24293
21.97758
18.61080
13.57495
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1s_carbon.als
COMNT
DATIM 24-03-2020 21:37:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 145
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenSM¥1t_proton.als



DFILE	1t_proton.als
COMNT	
DATIM	28-03-2020 17:55:37
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	40



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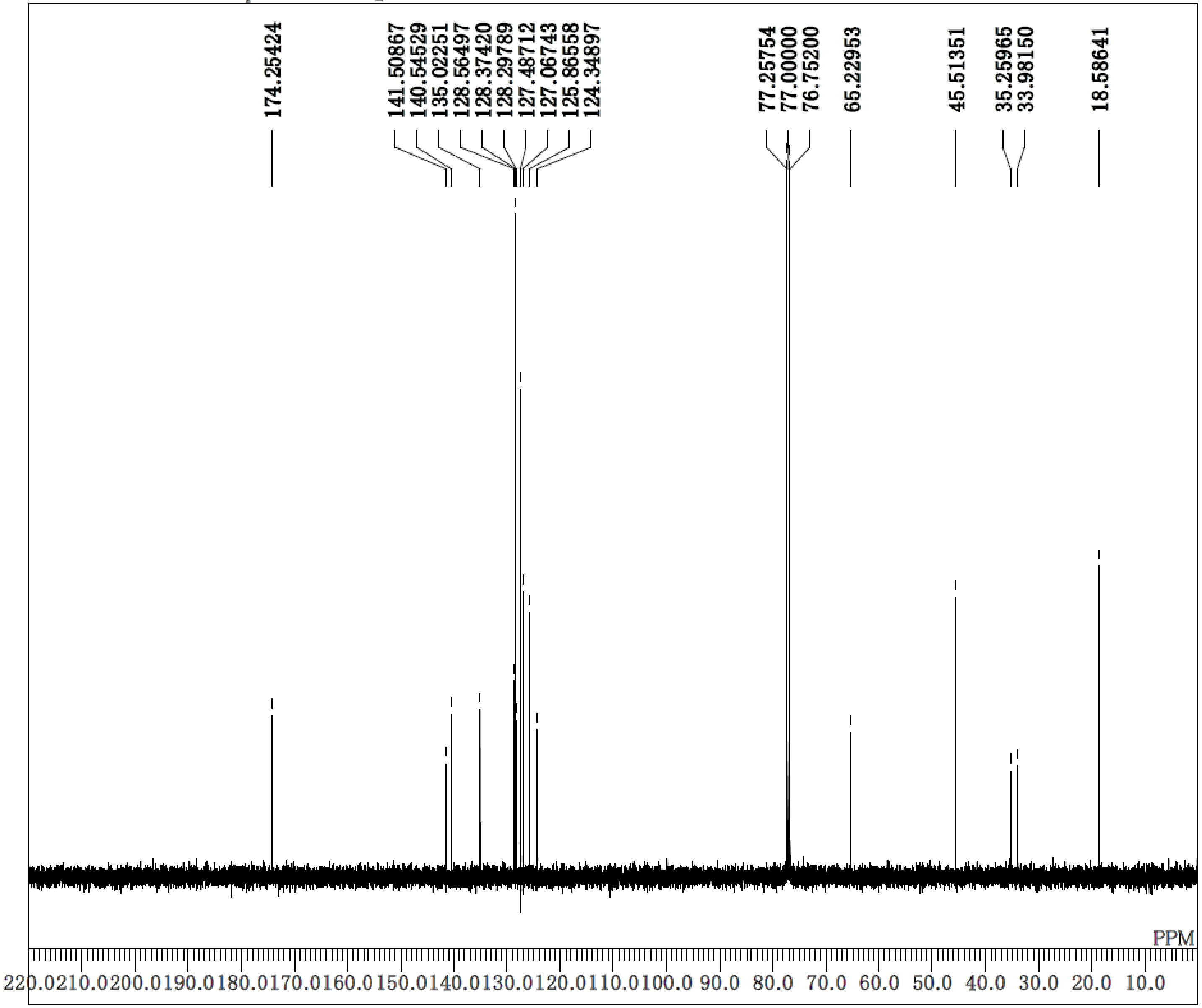
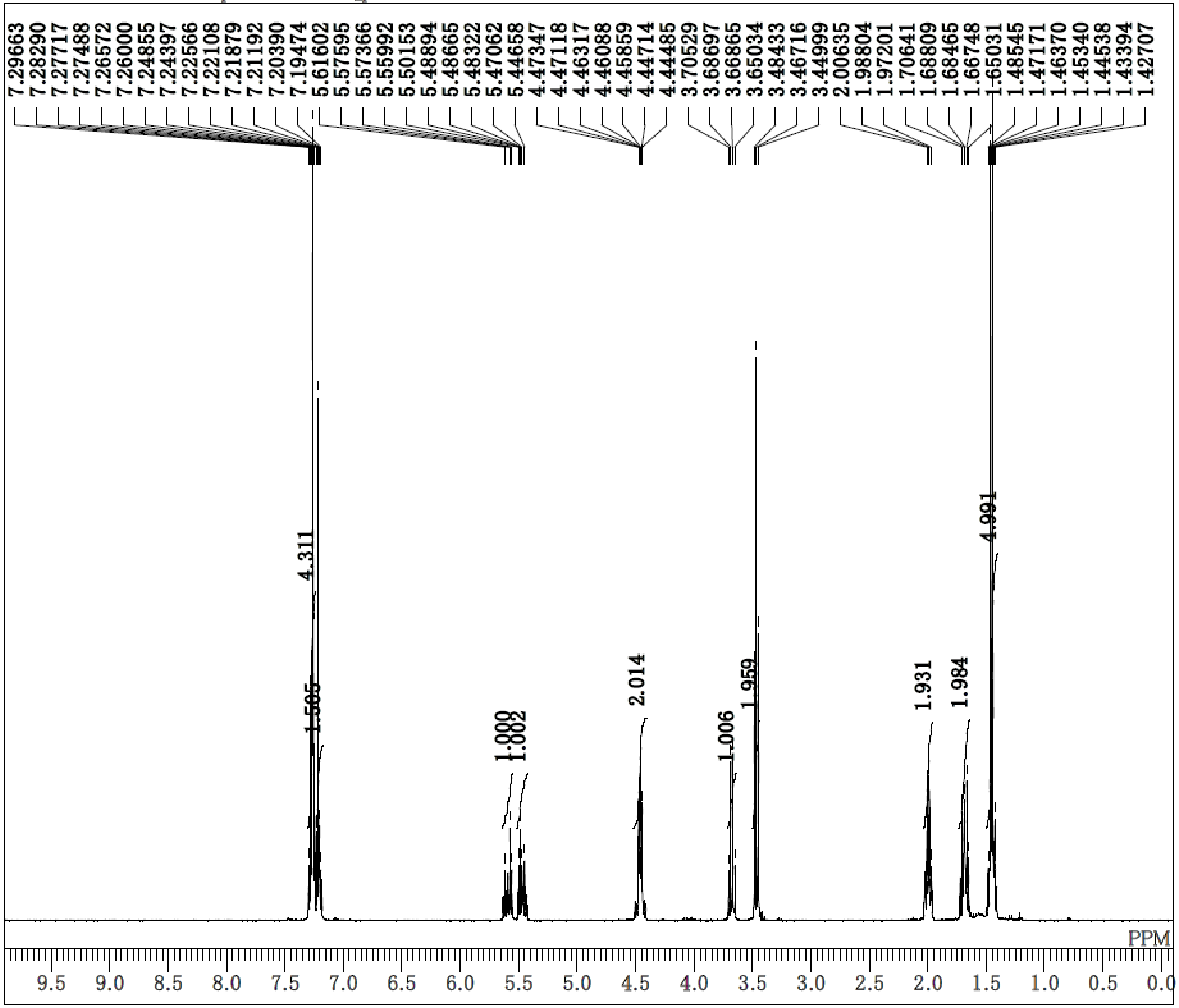
C:¥Users¥alice¥Desktop¥ChenSM¥1t_carbon.als

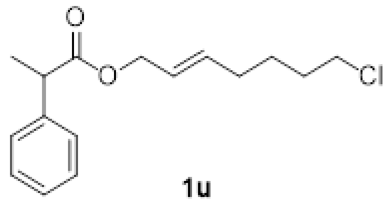


DFILE 1t_carbon.als
COMNT
DATIM 2020-04-09 20:49:16
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 101
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

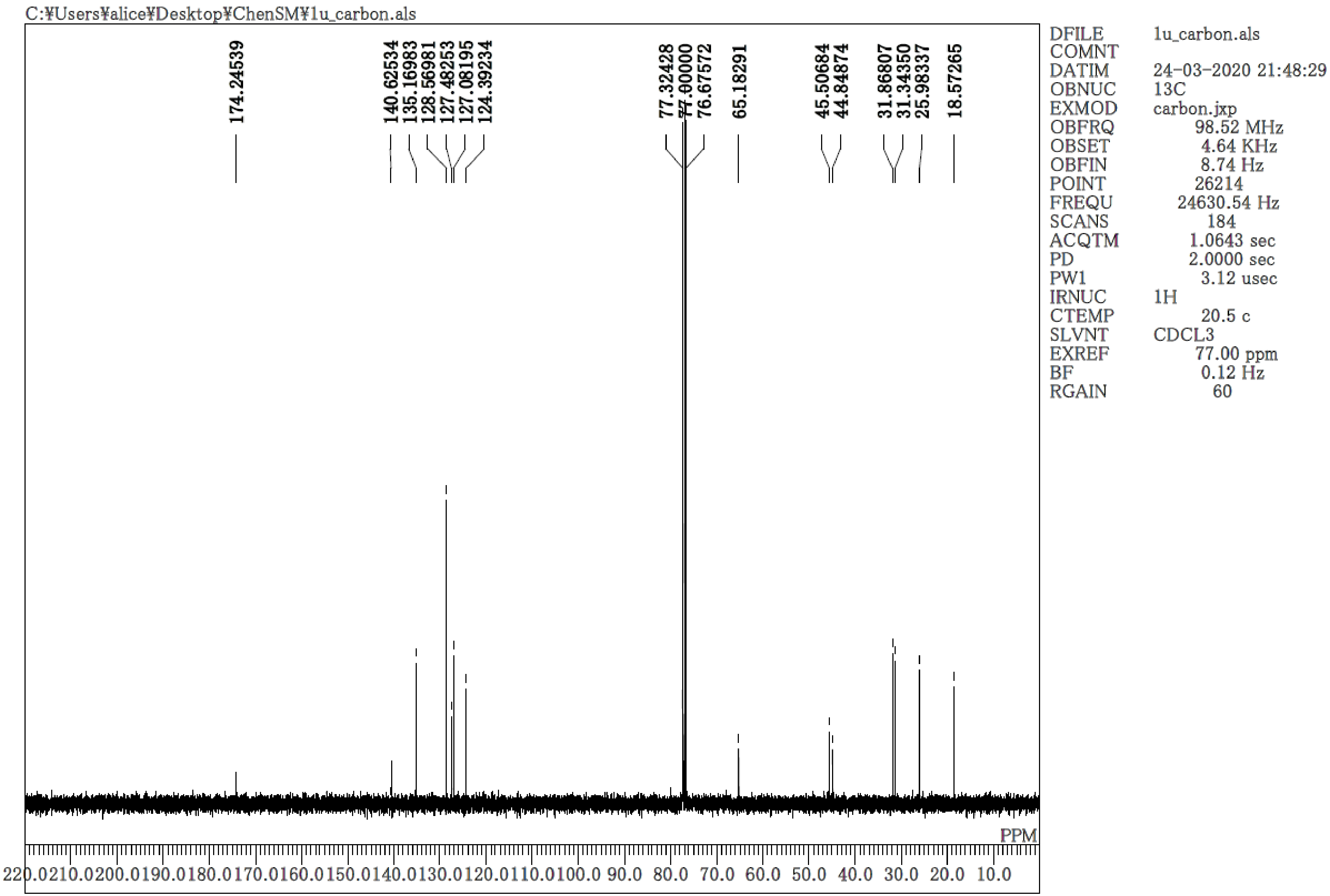
C:¥Users¥alice¥Desktop¥ChenSM¥1u_proton.als



DFILE	1u_proton.als
COMNT	
DATIM	24-03-2020 21:59:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



1u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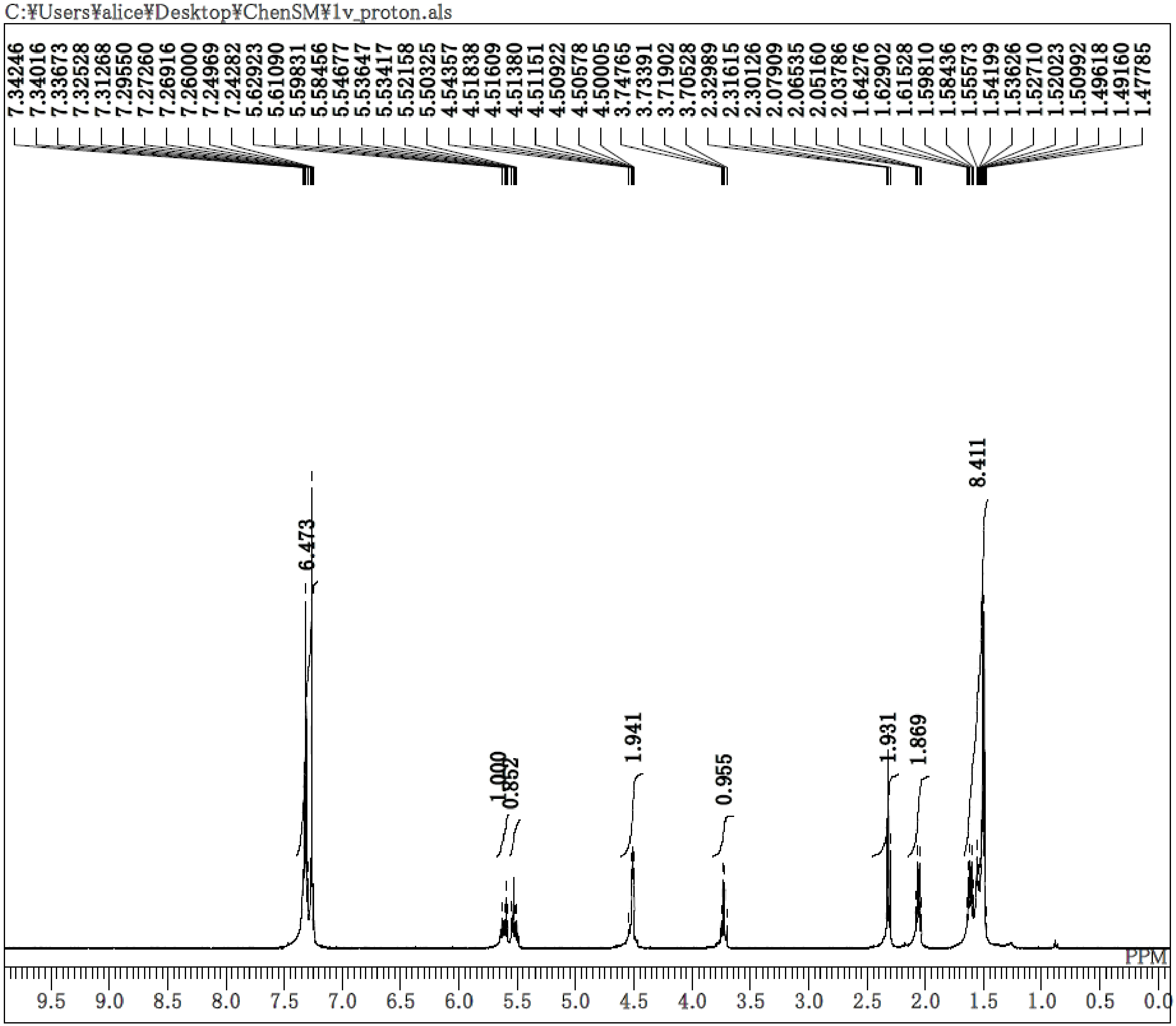
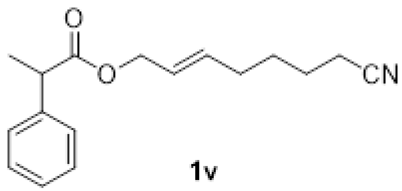
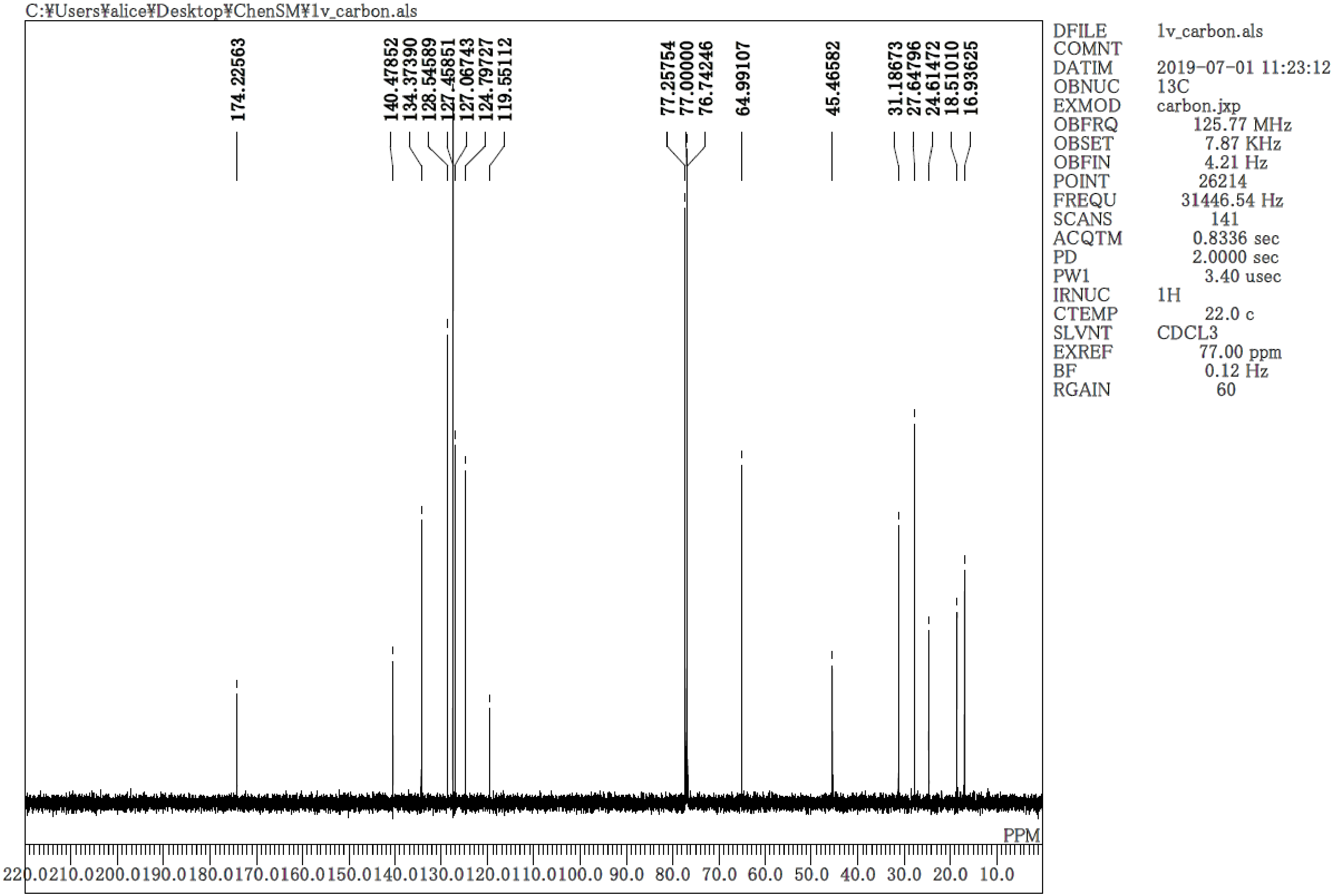
76.67572
65.18291
45.50684
44.84874
31.86807
31.34350
25.98337
18.57265
PPM
DFILE 1u_carbon.als
COMNT
DATIM 24-03-2020 21:48:29
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 184
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



DFILE	1v_proton.als
COMNT	
DATIM	2019-07-01 11:17:52
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



1v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127.45851
127.06743
124.79727
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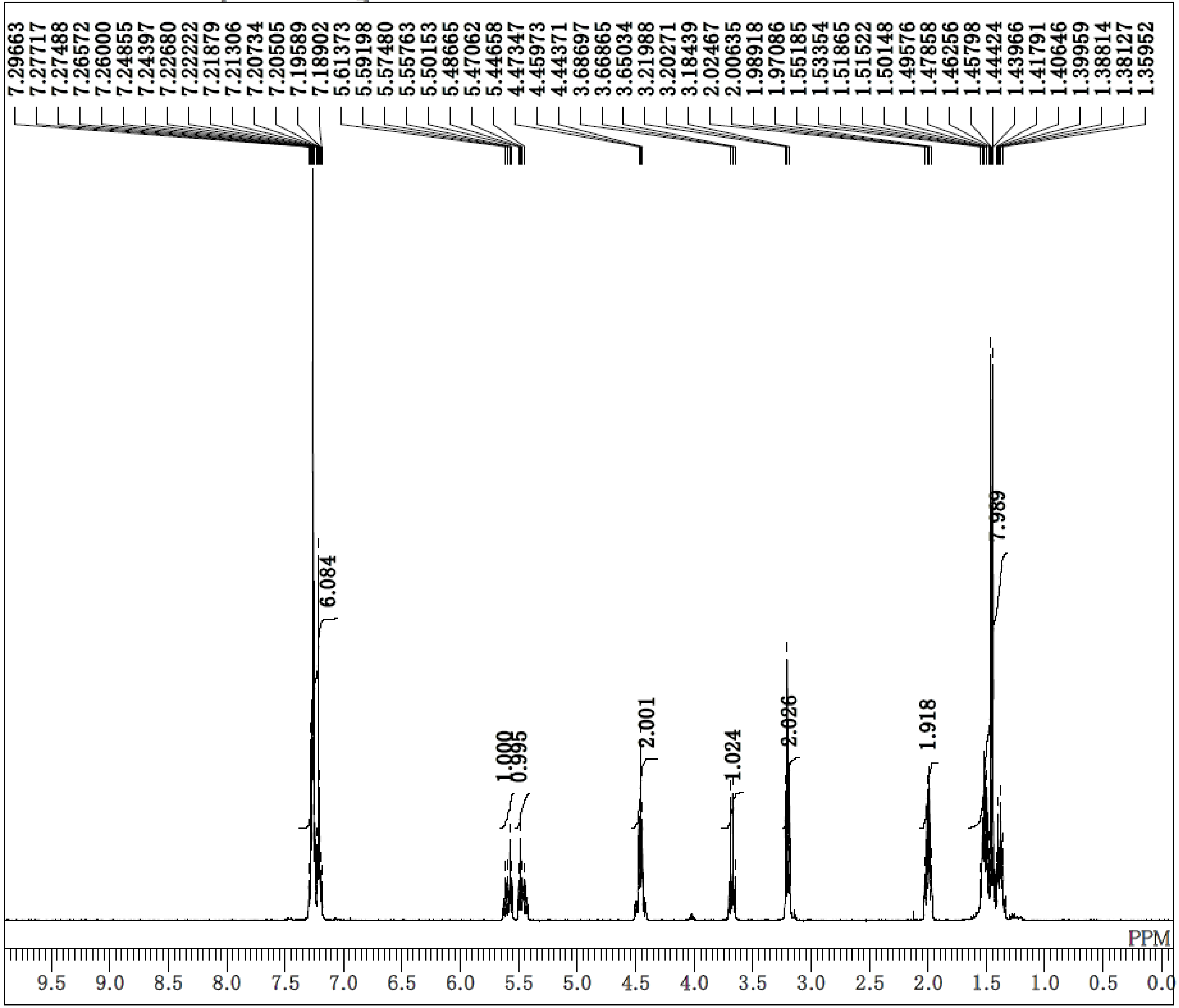
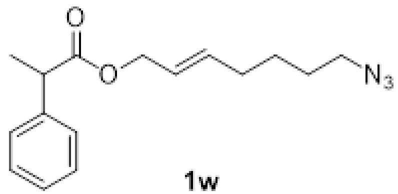
77.25754
77.00000
76.74246
64.99107
45.46582
31.18673
27.64796
24.61472
18.51010
16.93625
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1v_carbon.als
COMNT
DATIM 2019-07-01 11:23:12
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 141
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

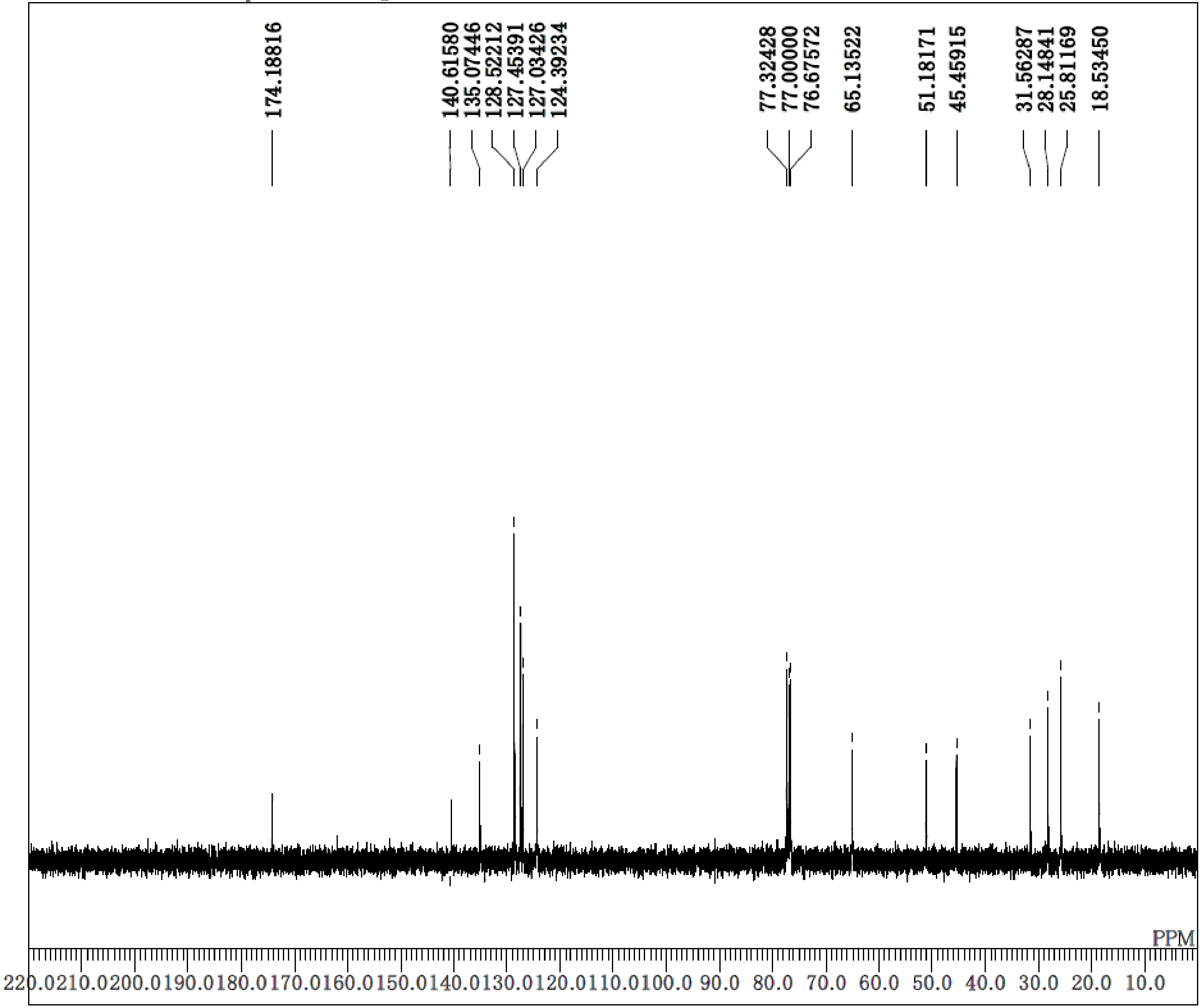
C:¥Users¥alice¥Desktop¥ChenSM¥1w_proton.als



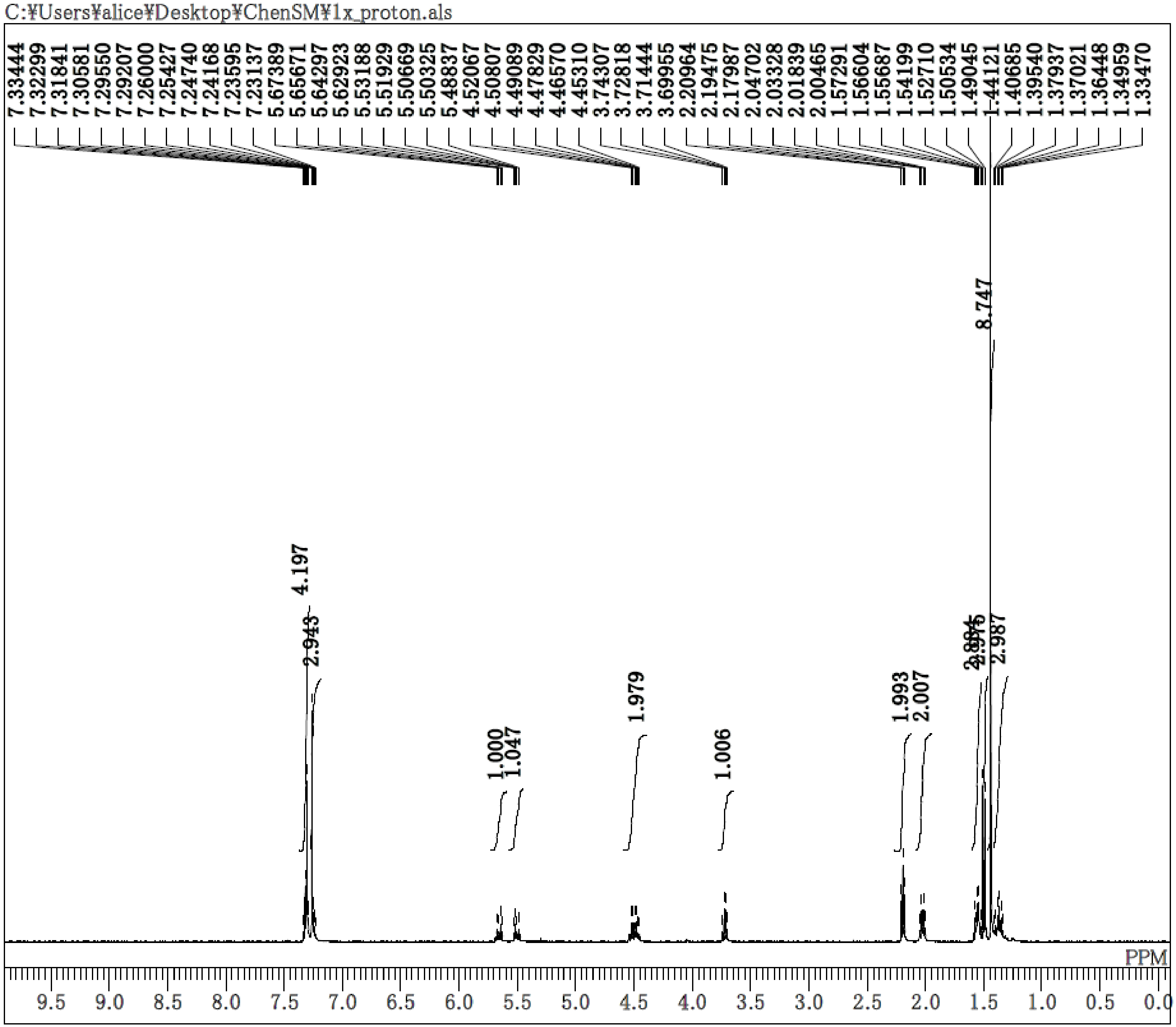


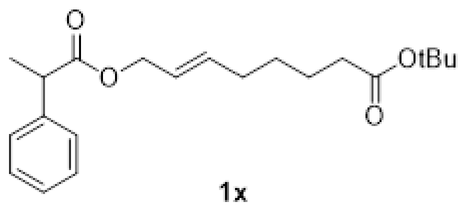
1w

C:¥Users¥alice¥Desktop¥ChenSM¥1w_carbon.als

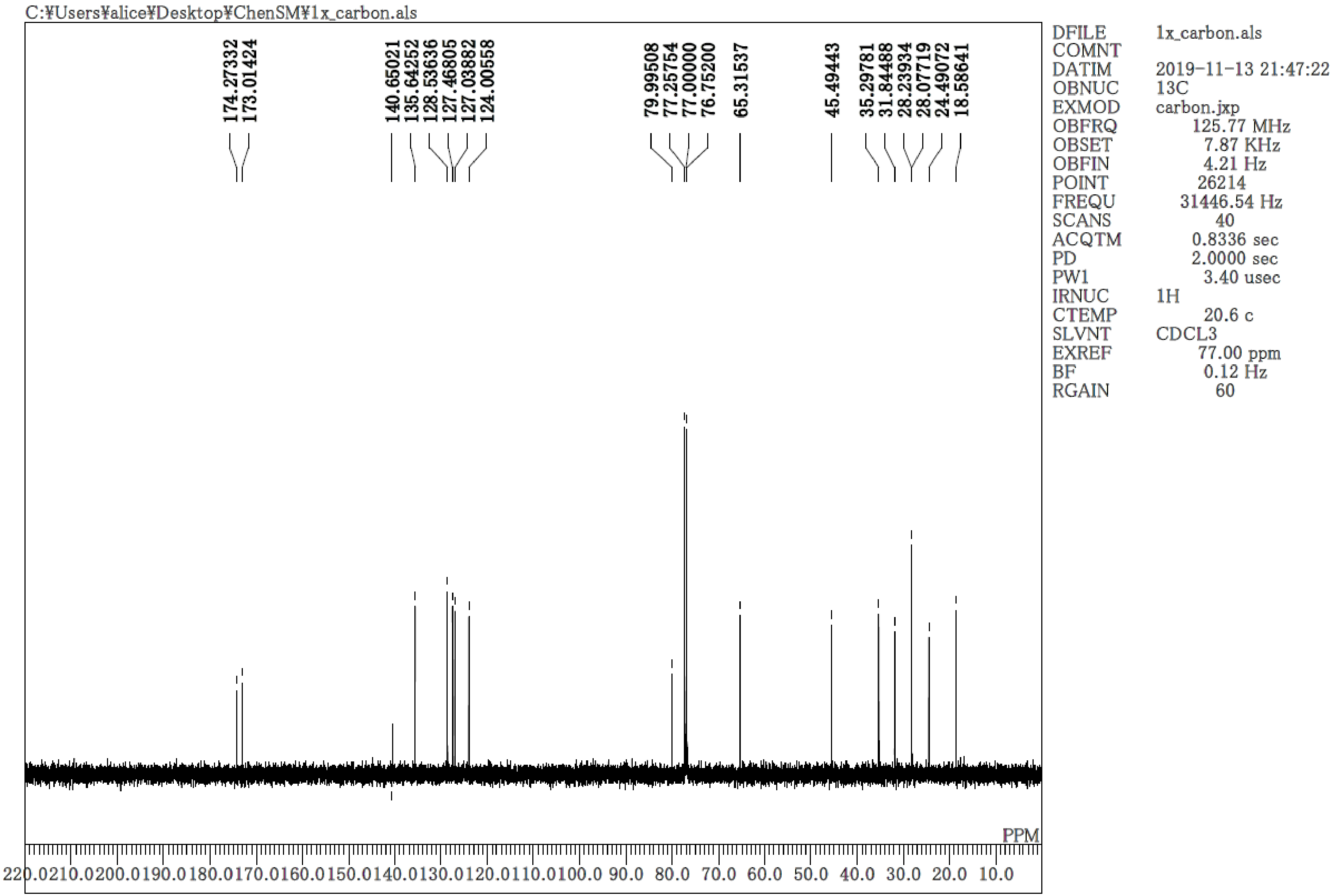


DFILE	1w_carbon.als
COMNT	
DATIM	09-11-2019 13:34:11
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	61
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60


C:¥Users¥alice¥Desktop¥ChenSM¥1x_proton.als
DFILE 1x_proton.als
COMNT
DATIM 2019-11-13 21:36:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42
PPM


OtBu

1x


C:¥Users¥alice¥Desktop¥ChenSM¥1x_carbon.als
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124.00558
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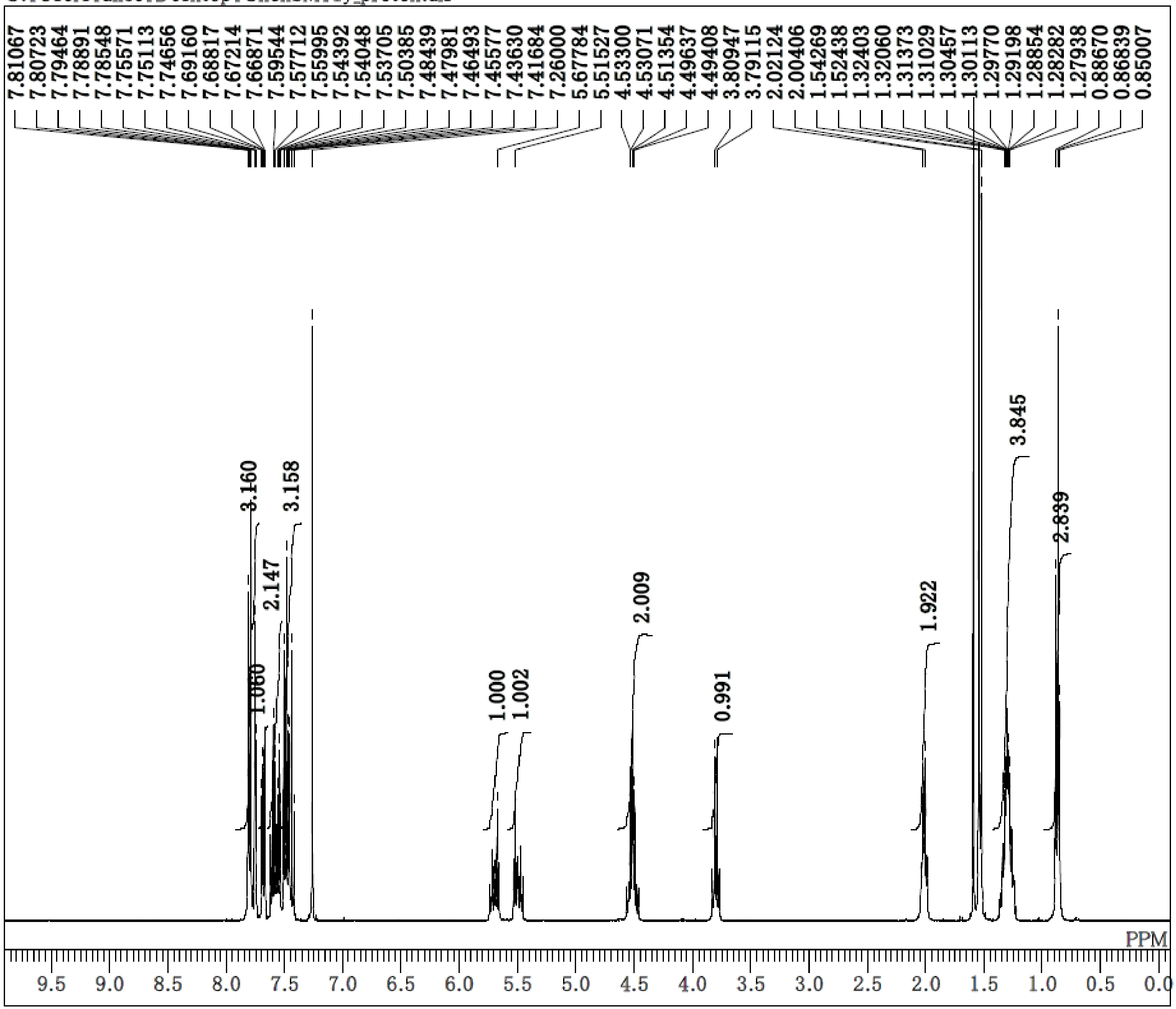
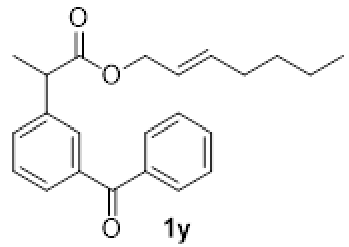
76.75200
65.31537
45.49443
35.29781
31.84488
28.23934
28.07719
24.49072
18.58641
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1x_carbon.als
COMNT
DATIM 2019-11-13 21:47:22
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 40
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

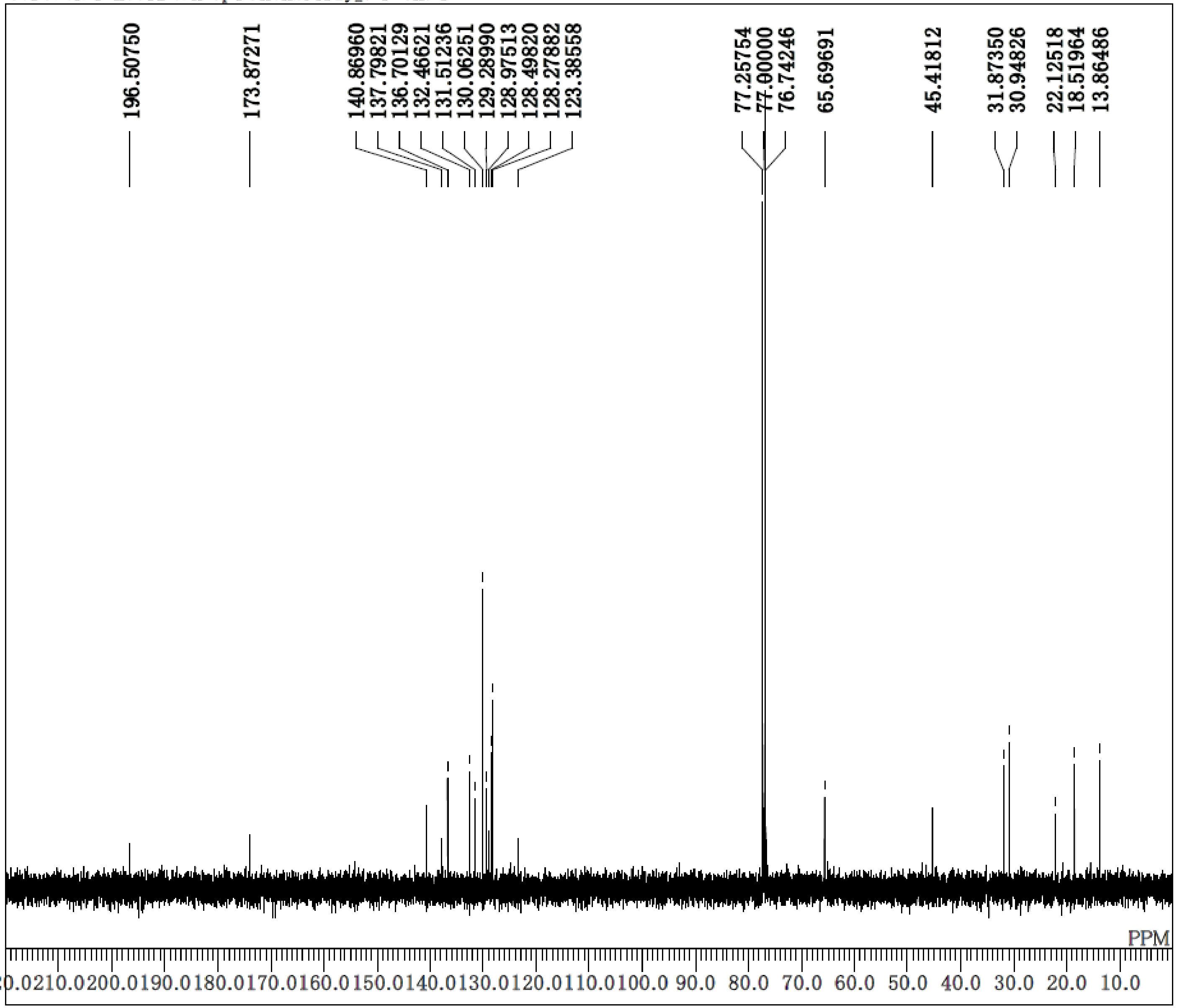


DFILE 1y_proton.als
COMNT
DATIM 27-07-2019 16:04:49
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



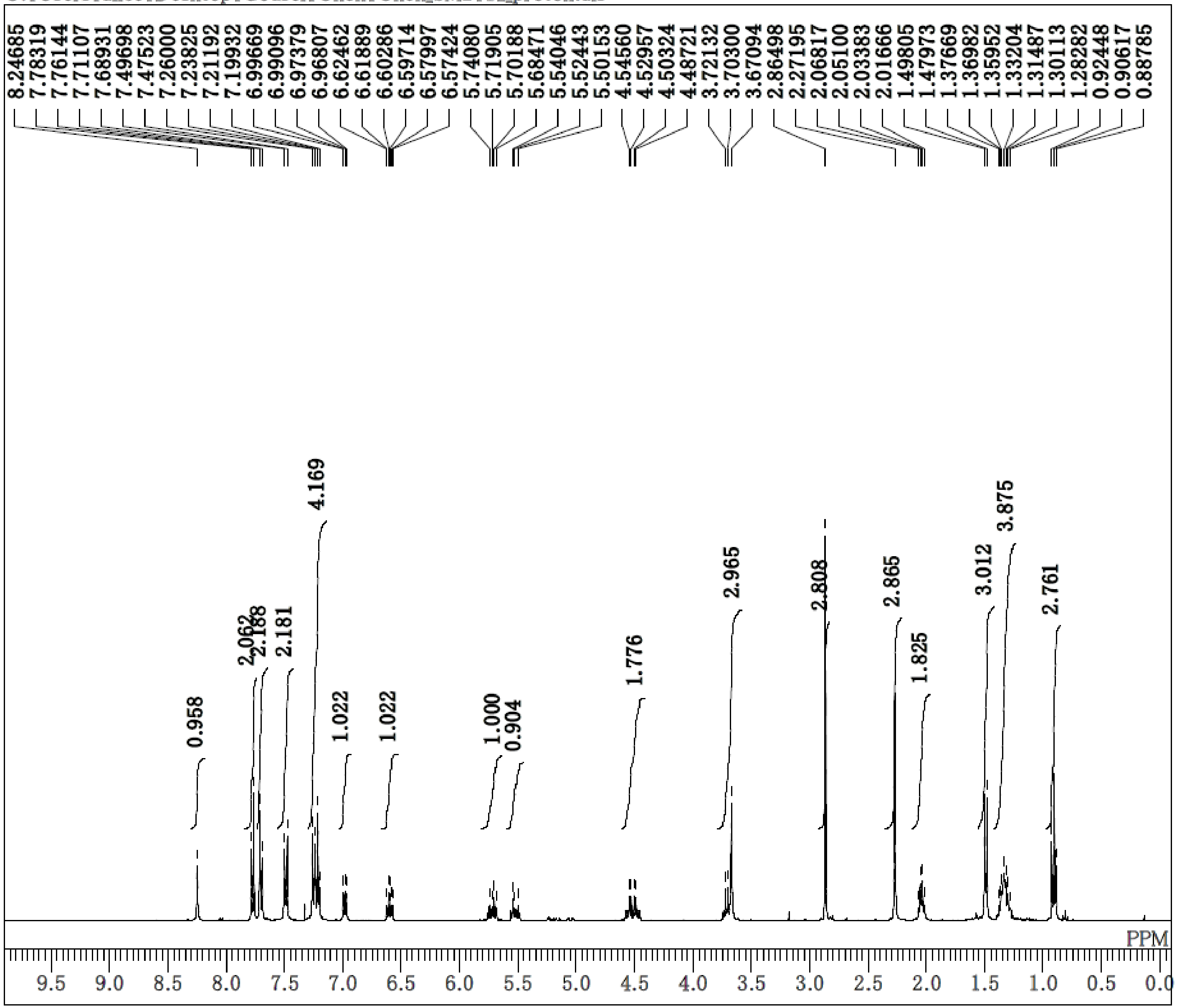
1y

C:¥Users¥alice¥Desktop¥ChenSM¥1y_carbon.als

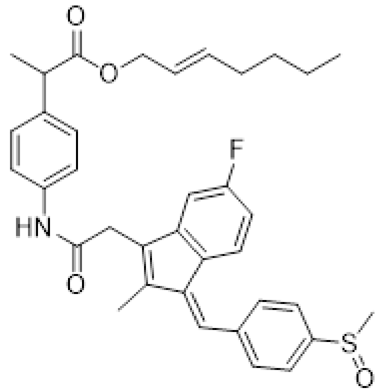


DFILE	1y_carbon.als
COMNT	
DATIM	2019-07-27 16:32:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	60
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥1z_proton.als

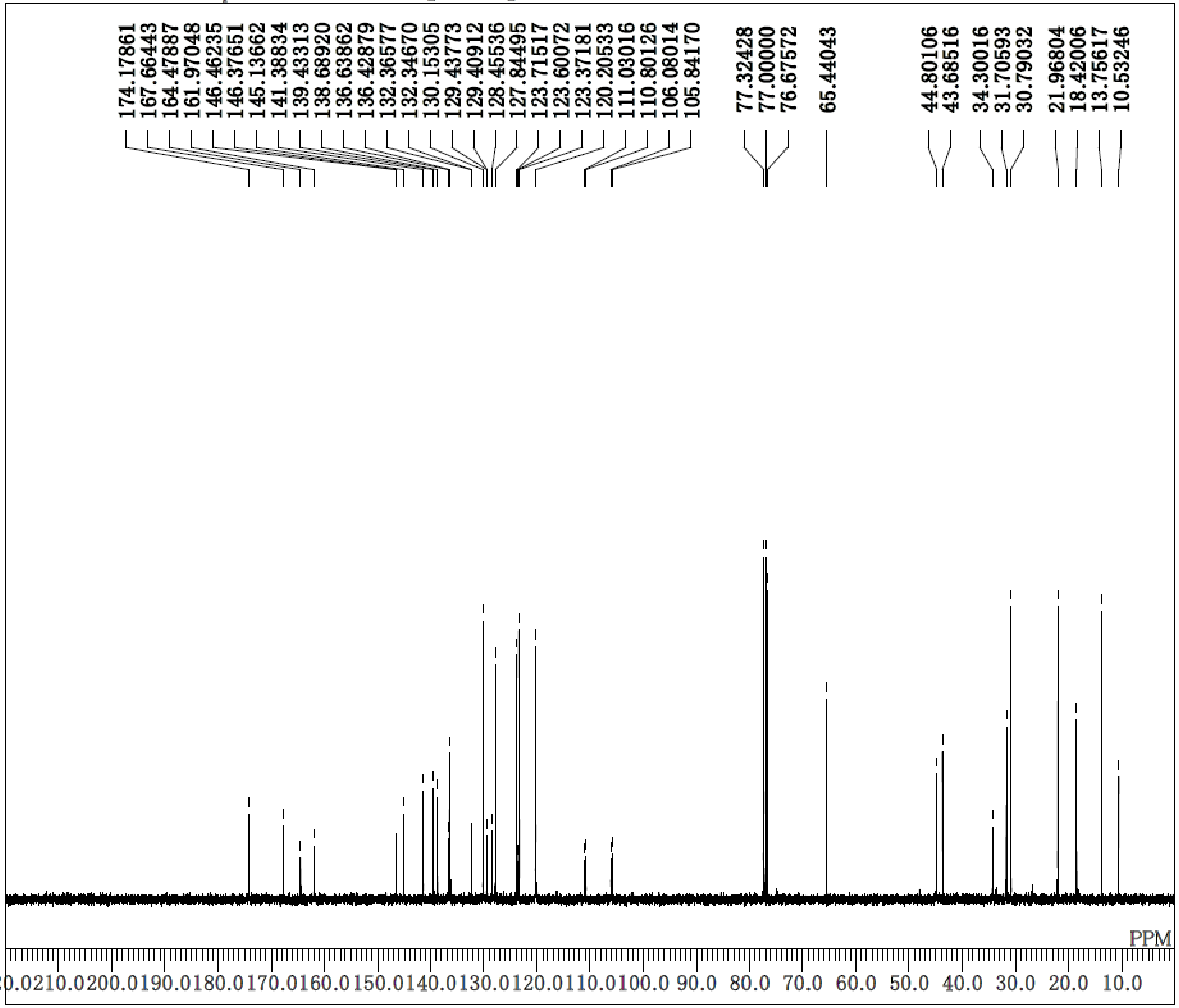


DFILE	1z_proton.als
COMNT	
DATIM	30-10-2020 20:30:50
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	22



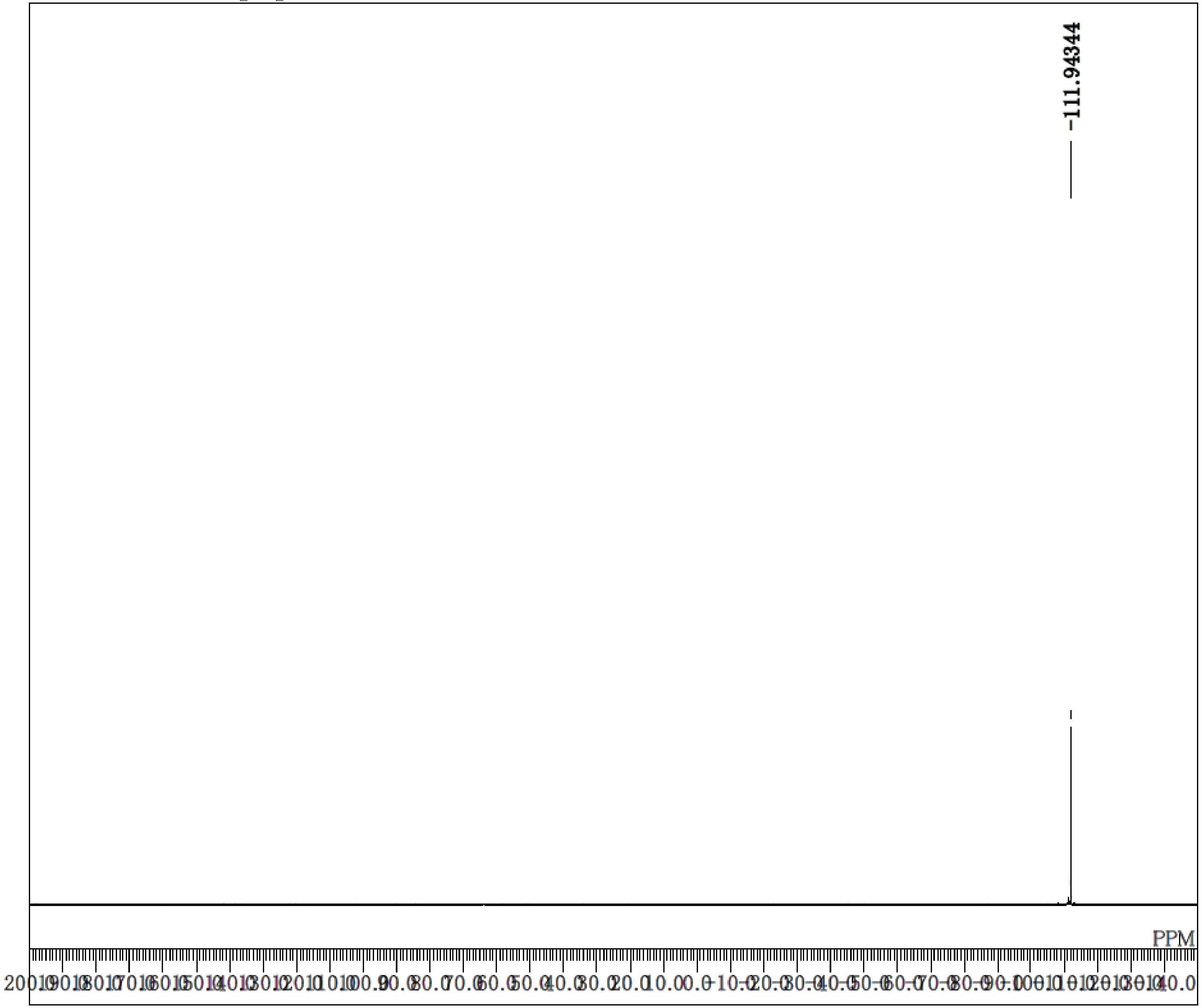
1Z

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥1z_carbon.als



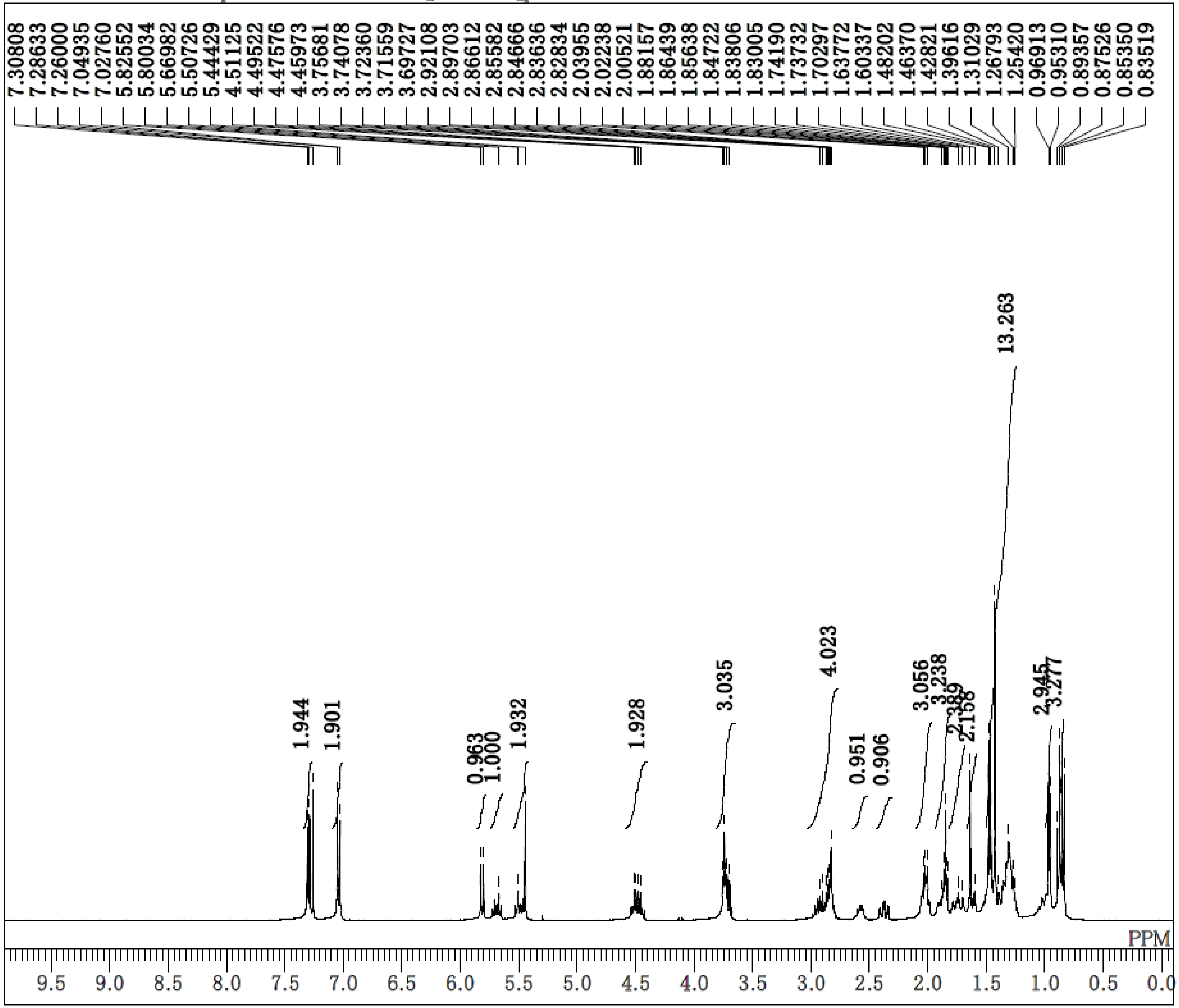
DFILE	1z_carbon.als
COMNT	
DATIM	30-10-2020 20:32:54
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	213
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

I:¥sulundac F¥sulindac_SM_F-1-1.als

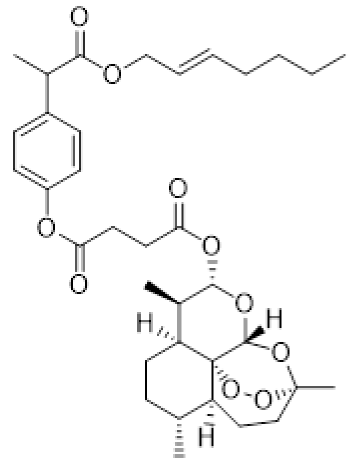


DFILE	sulindac_SM_F-1-1.als
COMNT	
DATIM	14-11-2020 09:10:58
OBNUC	19F
EXMOD	proton.jxp
OBFRQ	368.64 MHz
OBSET	7.63 KHz
OBFIN	2.85 Hz
POINT	13107
FREQU	149253.73 Hz
SCANS	100
ACQTM	0.0878 sec
PD	6.0000 sec
PW1	4.10 usec
IRNUC	19F
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	50

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥1aa_proton.als

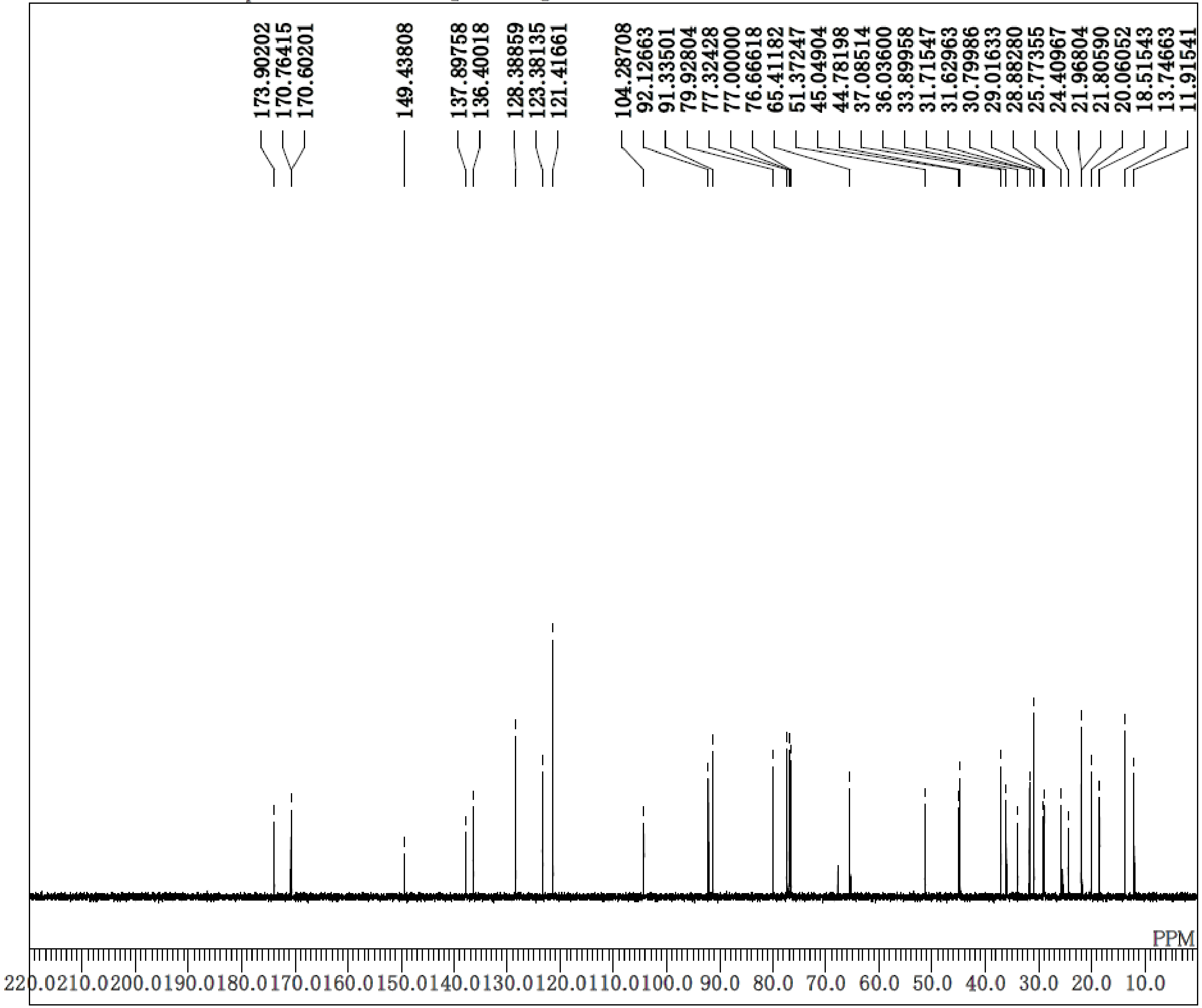


DFILE	1aa_proton.als
COMNT	
DATIM	10-10-2020 15:15:57
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34

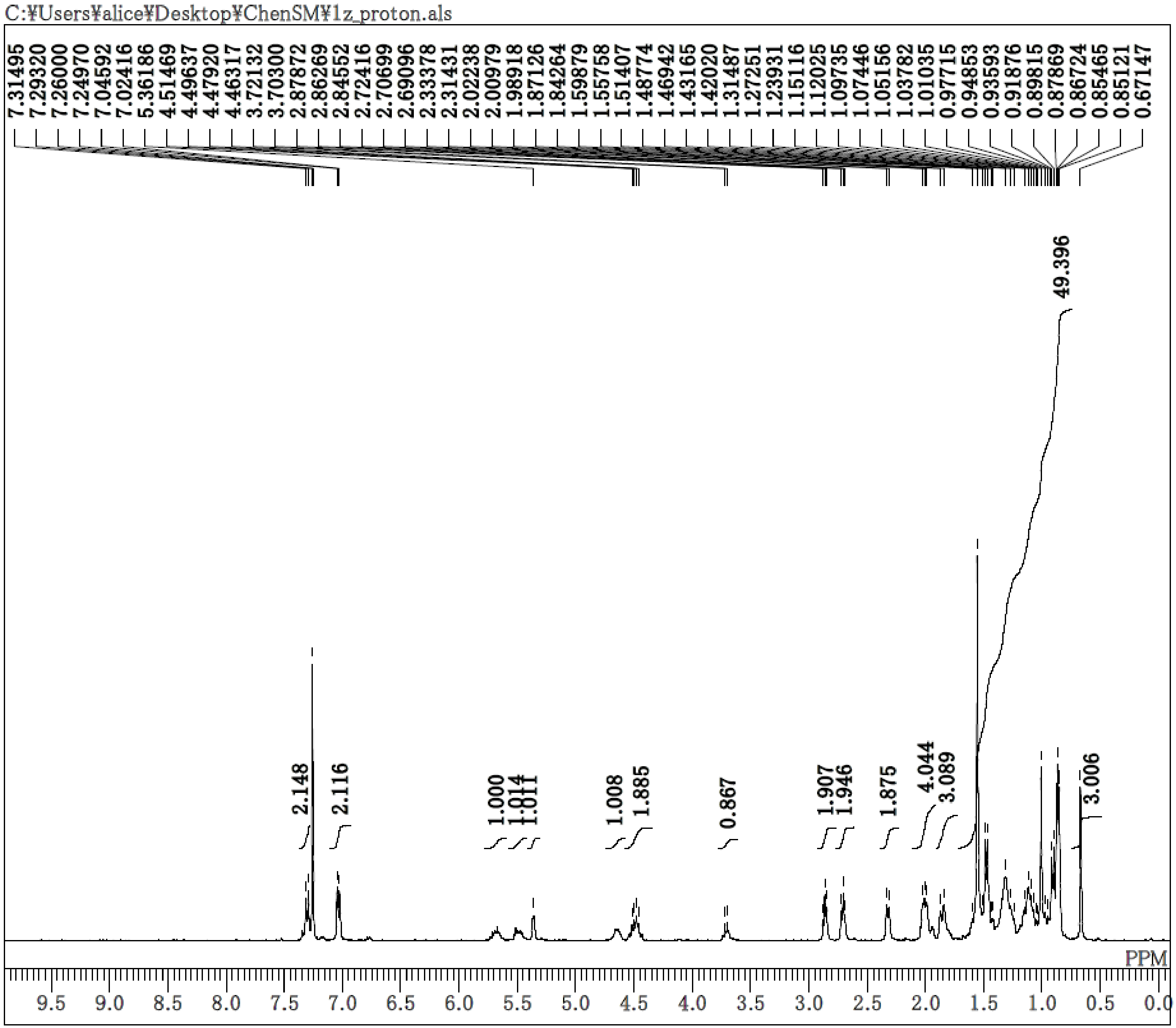


1aa

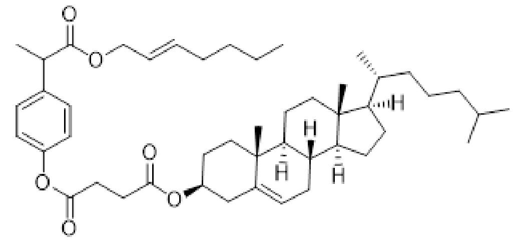
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥1aa_carbon.als



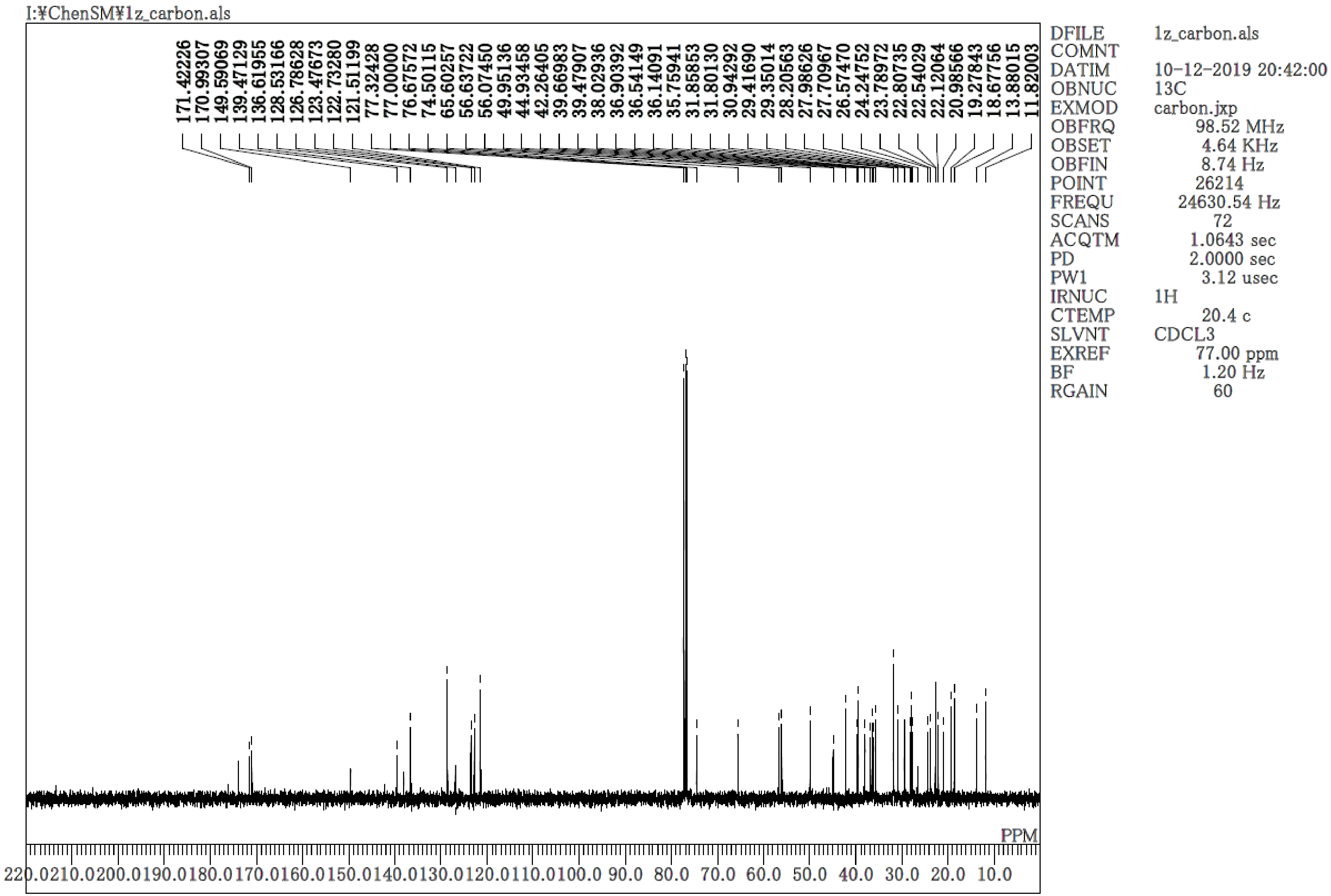
DFILE	1aa_carbon.als
COMNT	
DATIM	12-10-2020 17:35:14
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	134
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE	1z_proton.als
COMNT	
DATIM	10-12-2019 19:19:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	48



1ab


I:¥ChenSM¥1z_carbon.als
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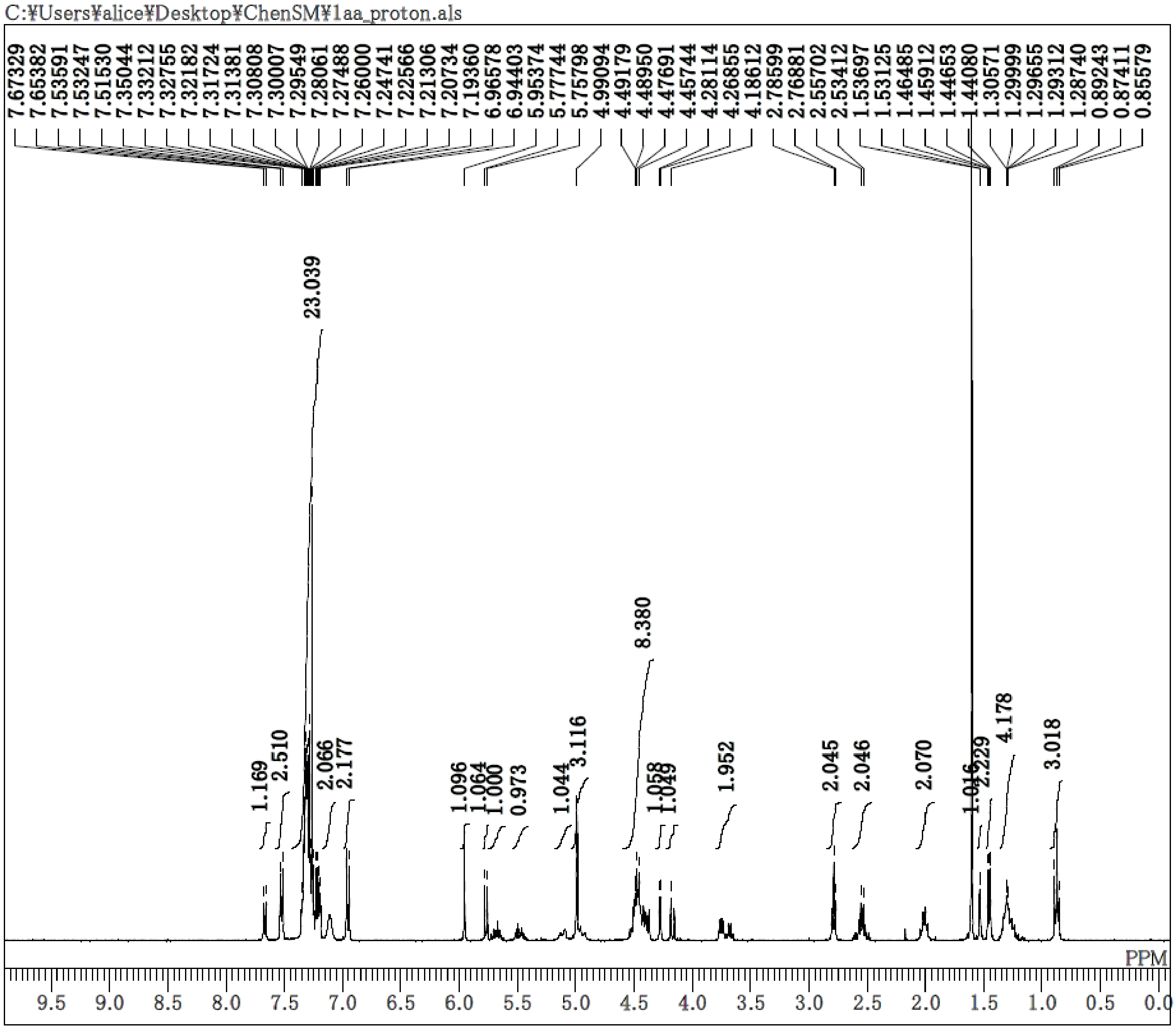
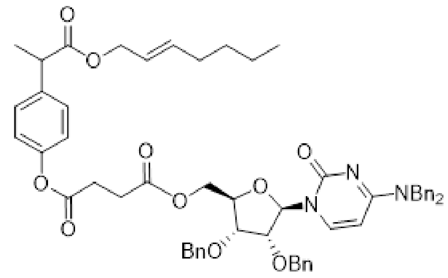
122.73280
121.51199
77.32428
77.00000
76.67572
74.50115
65.60257
56.63722
56.07450
49.95136
44.93458
42.26405
39.66983
39.47907
38.02936
36.90392
36.54149
36.14091
35.75941
31.85853
31.80130
30.94292
29.41690
29.35014
28.20563
27.98626
27.70967
26.57470
24.24752
23.78972
22.80735
22.54029
22.12064
20.98566
19.27843
18.67756
13.88015
11.82003
DFILE 1z_carbon.als
COMNT
DATIM 10-12-2019 20:42:00
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 72
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0

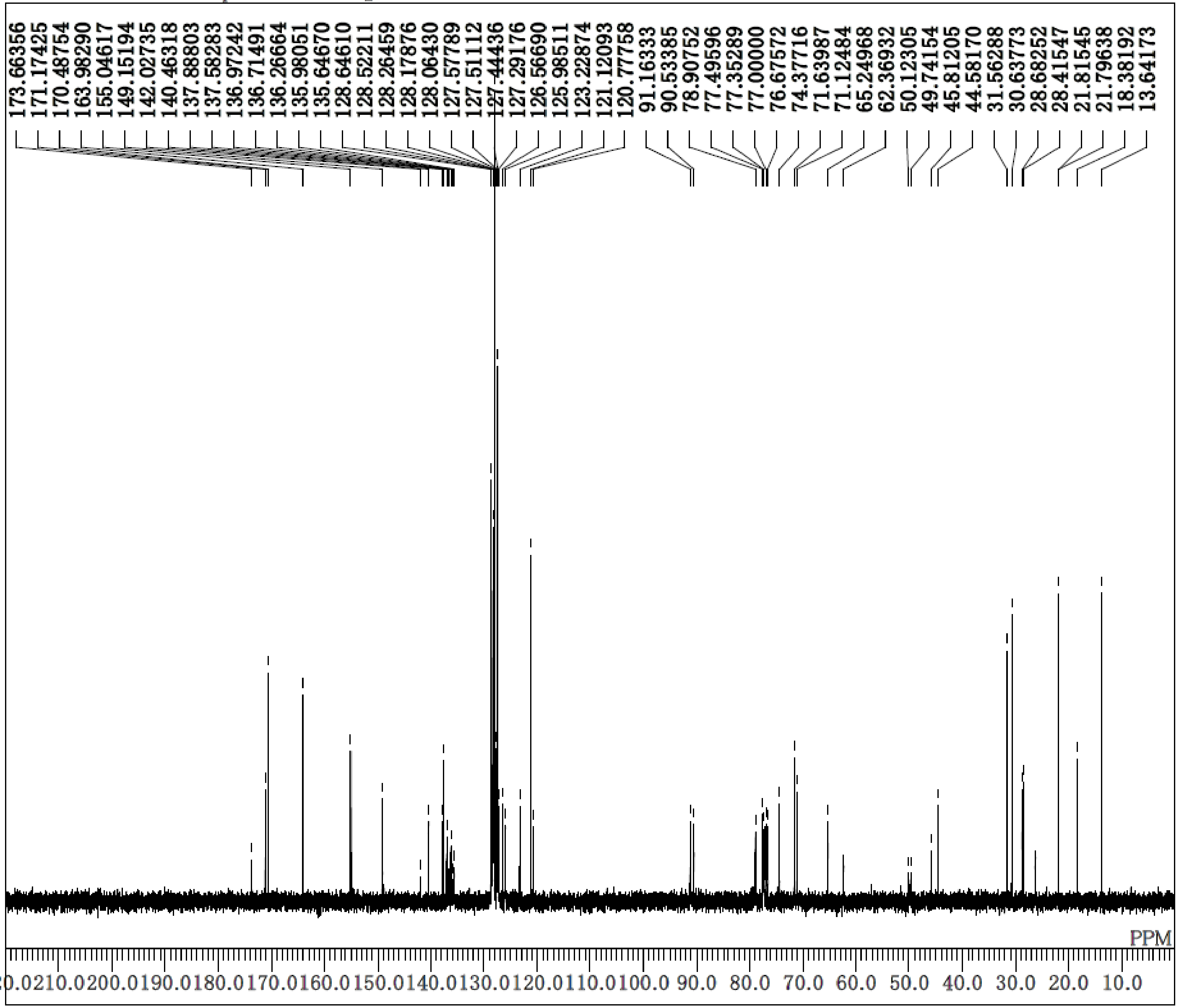


DFILE	1aa_proton.als
COMNT	
DATIM	04-01-2020 17:22:16
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	44



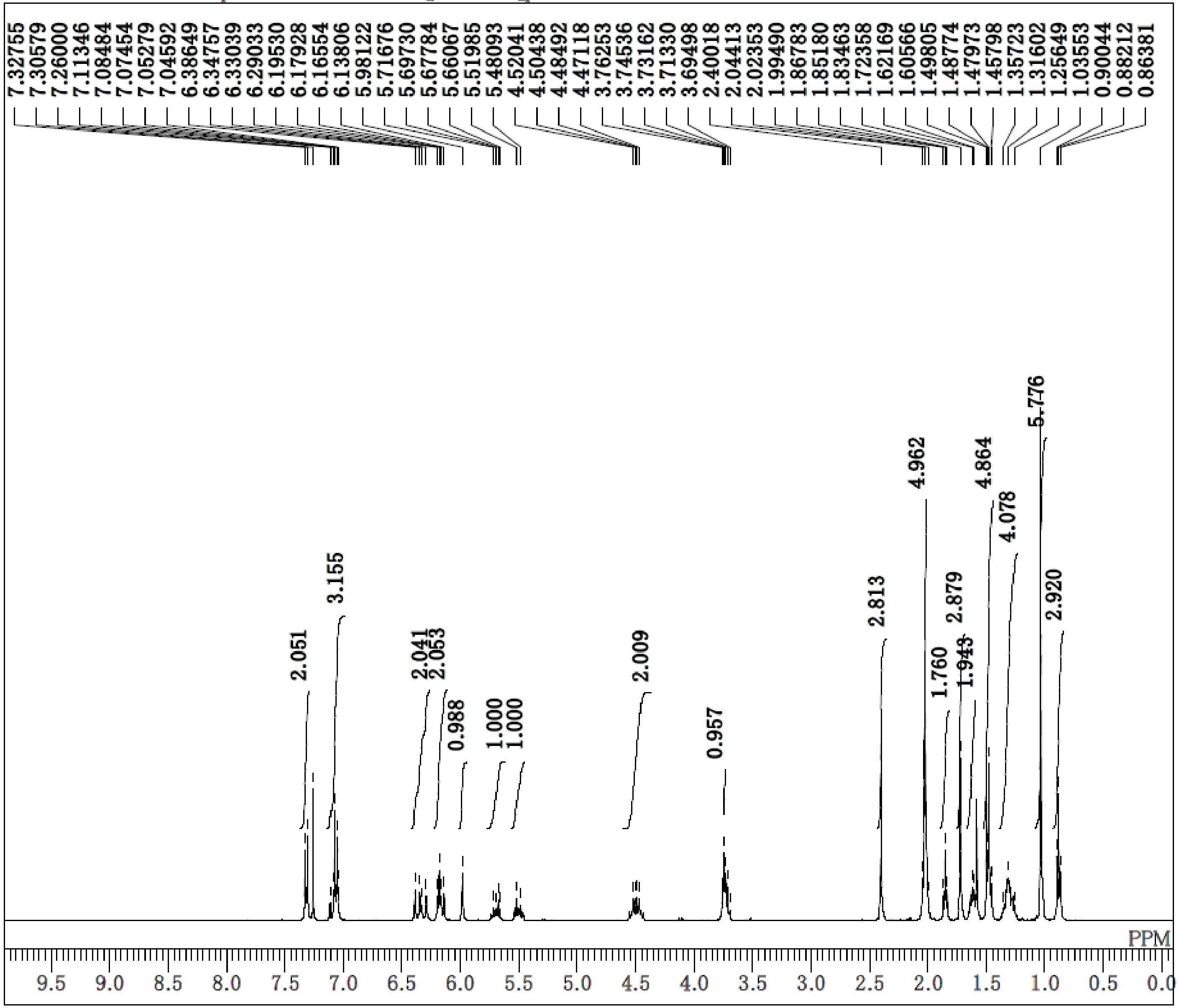
1ac

C:¥Users¥alice¥Desktop¥ChenSM¥1aa_carbon.als

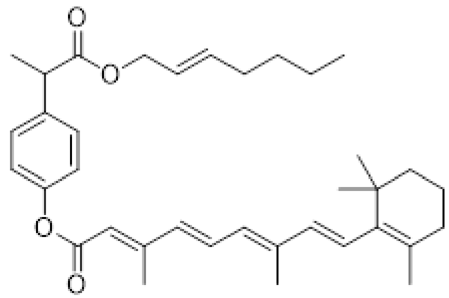


DFILE	1aa_carbon.als
COMNT	
DATIM	04-01-2020 17:42:34
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	60
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

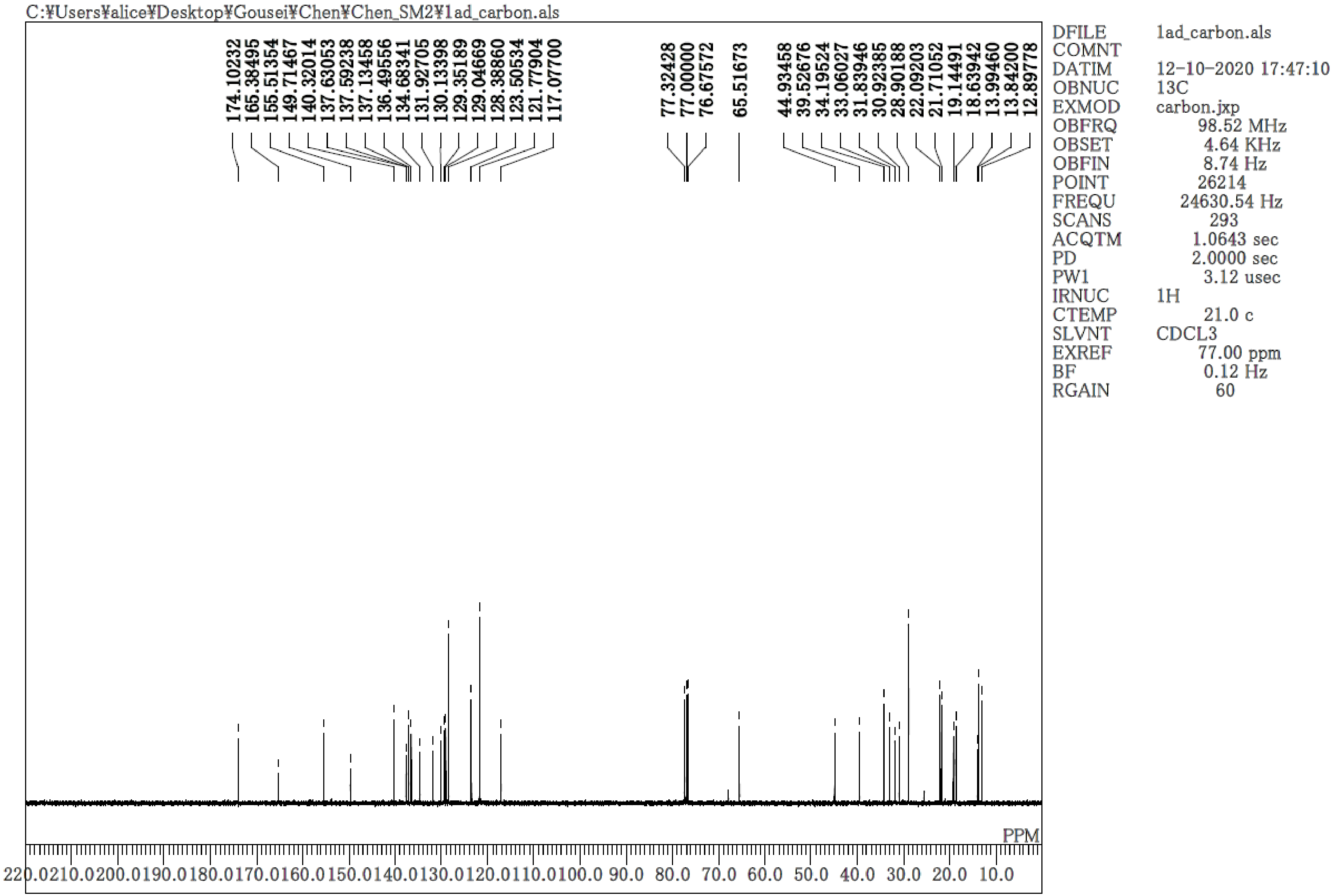
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥1ad_proton.als



DFILE	1ad_proton.als
COMNT	
DATIM	10-10-2020 15:21:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



1ad


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥1ad_carbon.als
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136.49556
134.68341
131.92705
130.13398
129.35189
129.04669
128.38860
123.50534
121.77904
117.07700
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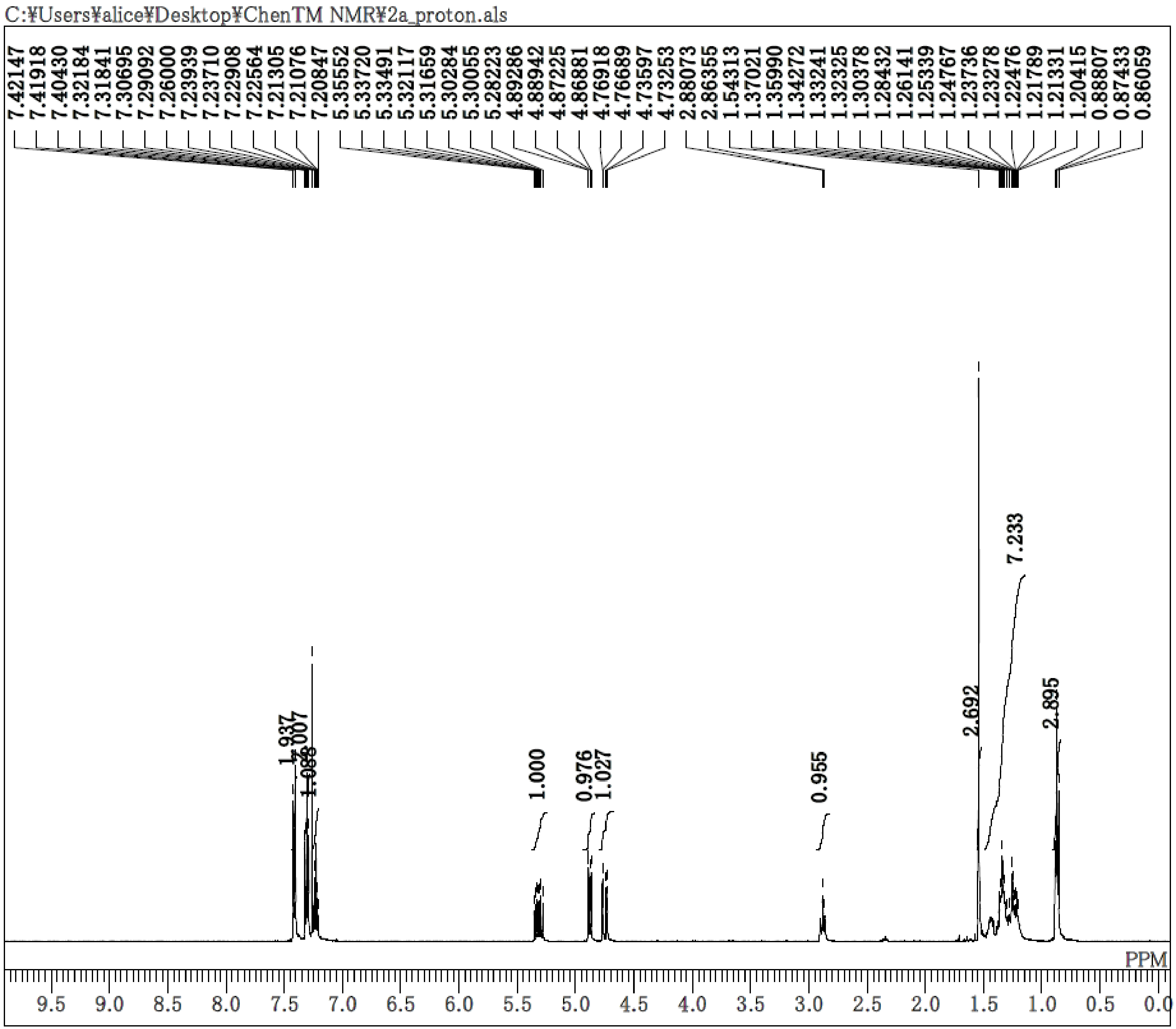
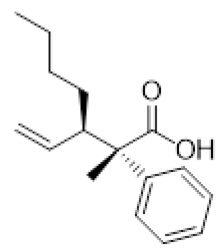
77.00000
76.67572
65.51673
44.93458
39.52676
34.19524
33.06027
31.83946
30.92385
28.90188
22.09203
21.71052
19.14491
18.63942
13.99460
13.84200
12.89778
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 1ad_carbon.als
COMNT
DATIM 12-10-2020 17:47:10
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 293
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

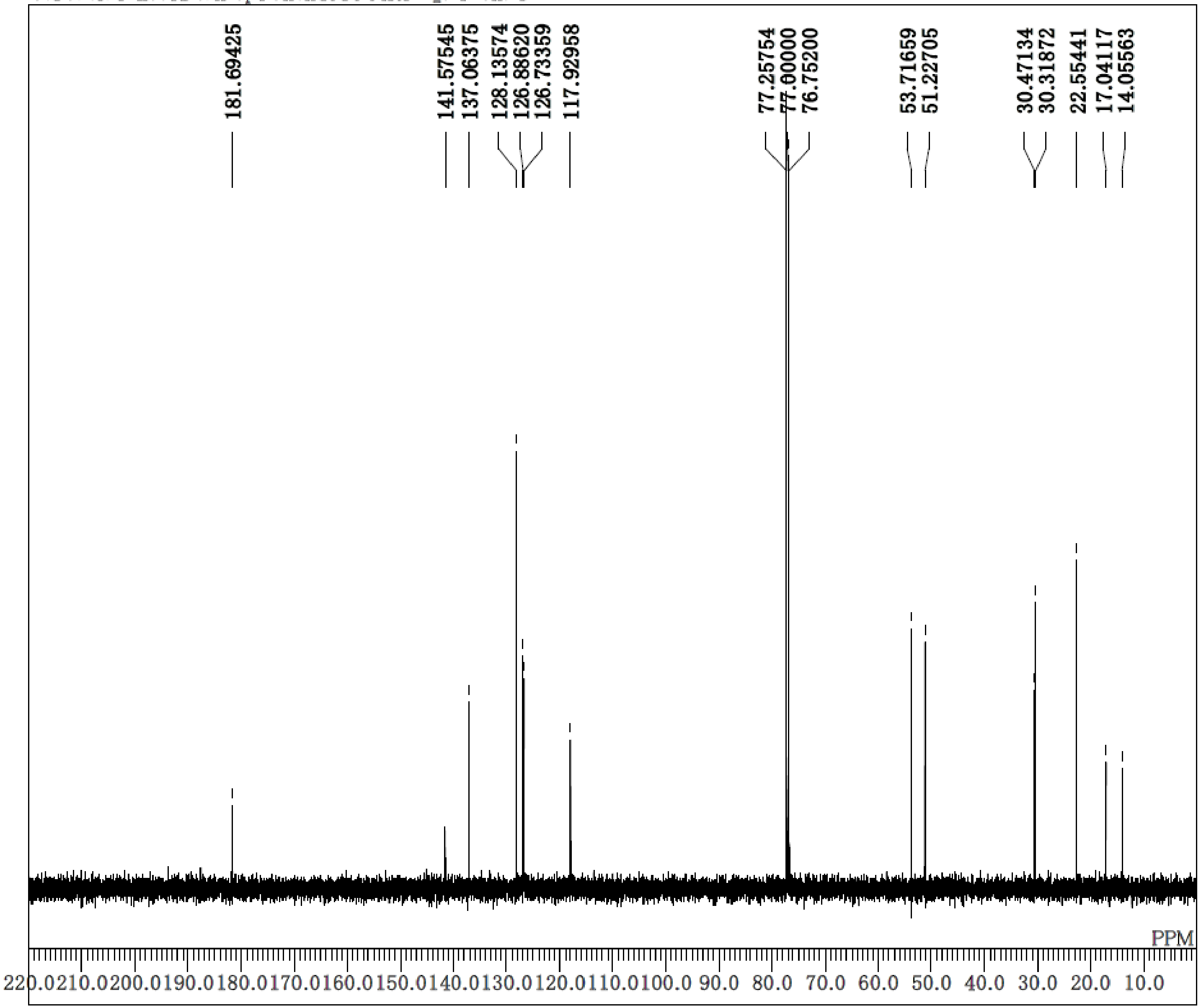


DFILE	2a_proton.als
COMNT	
DATIM	2020-04-15 15:04:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



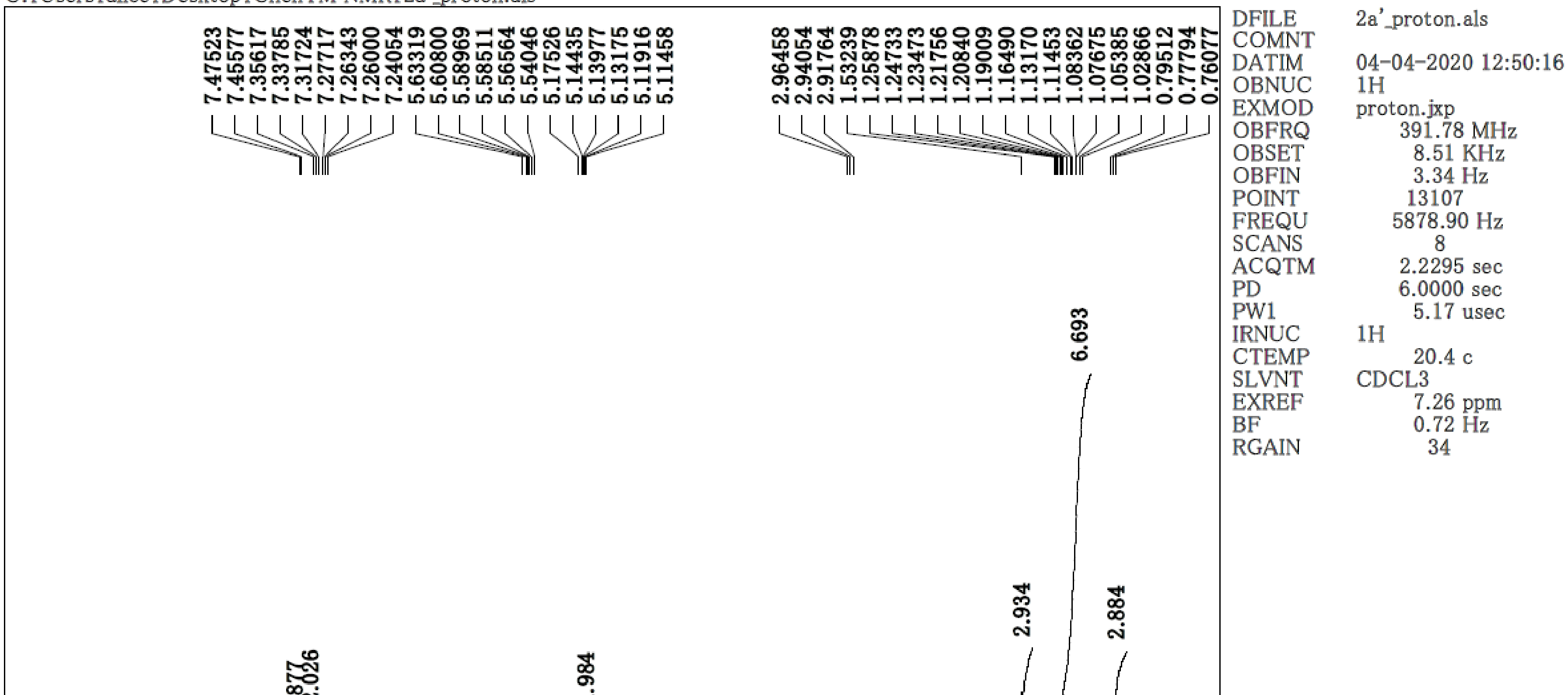
(2*S*,3*R*)-**2a**

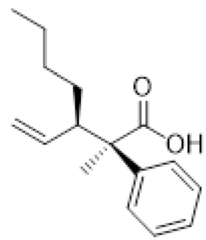
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2a_carbon.als



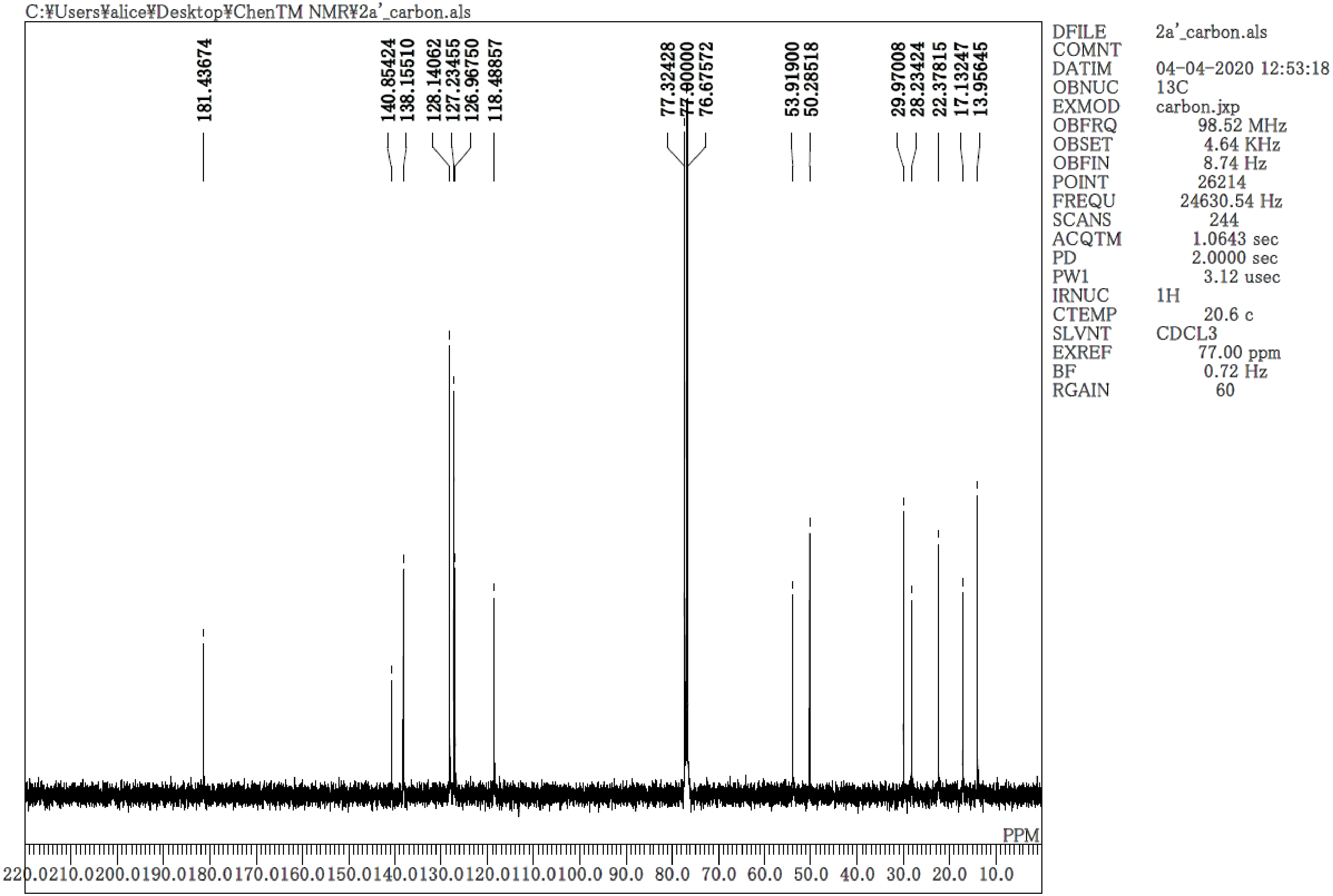
DFILE	2a_carbon.als
COMNT	
DATIM	2020-04-15 16:54:06
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	100
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2a'_proton.als





(2*R*,3*R*)-**2a**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2a'_carbon.als
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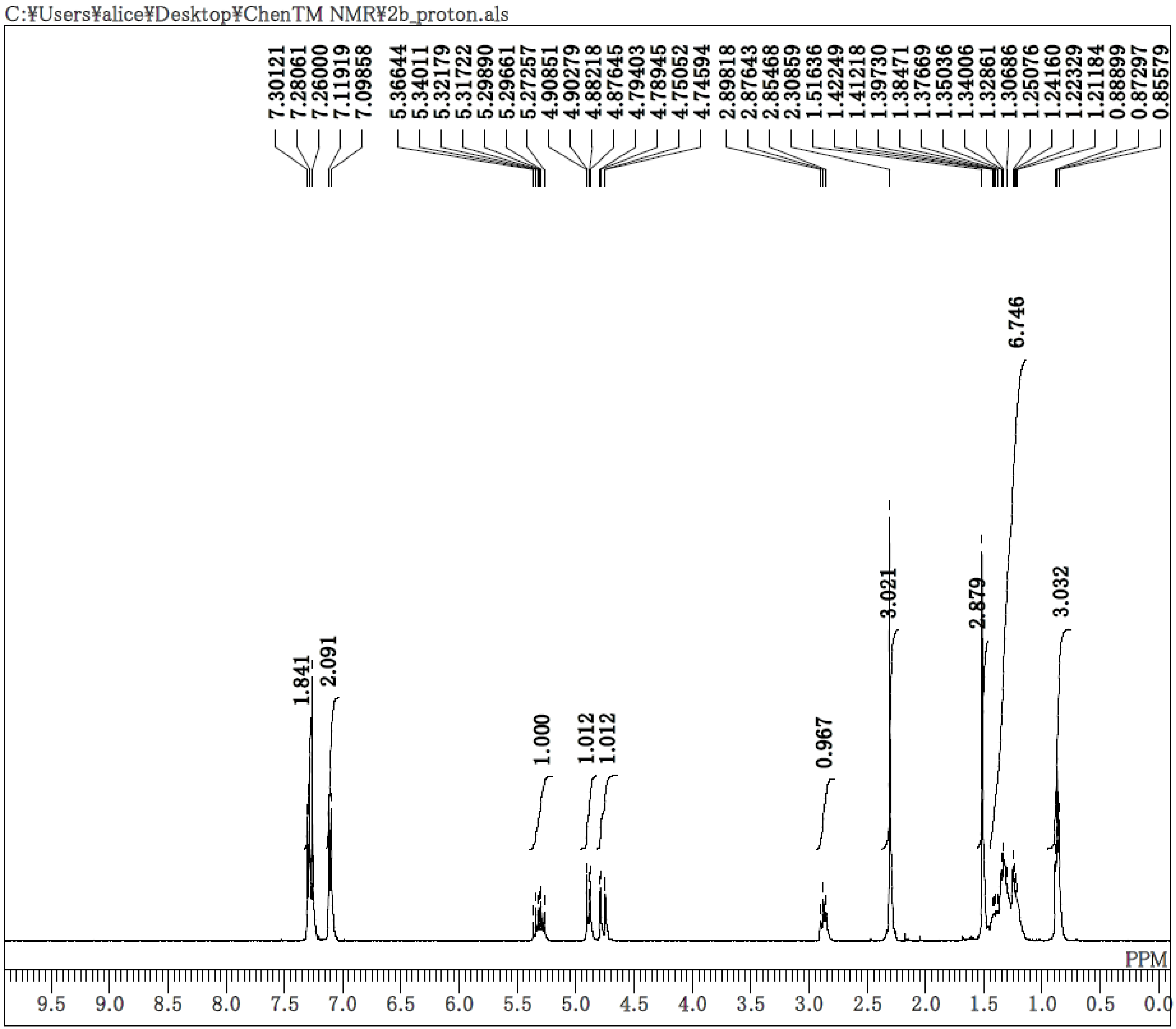
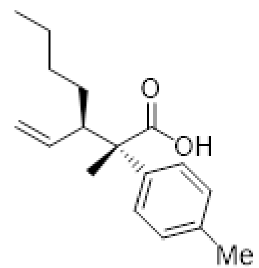
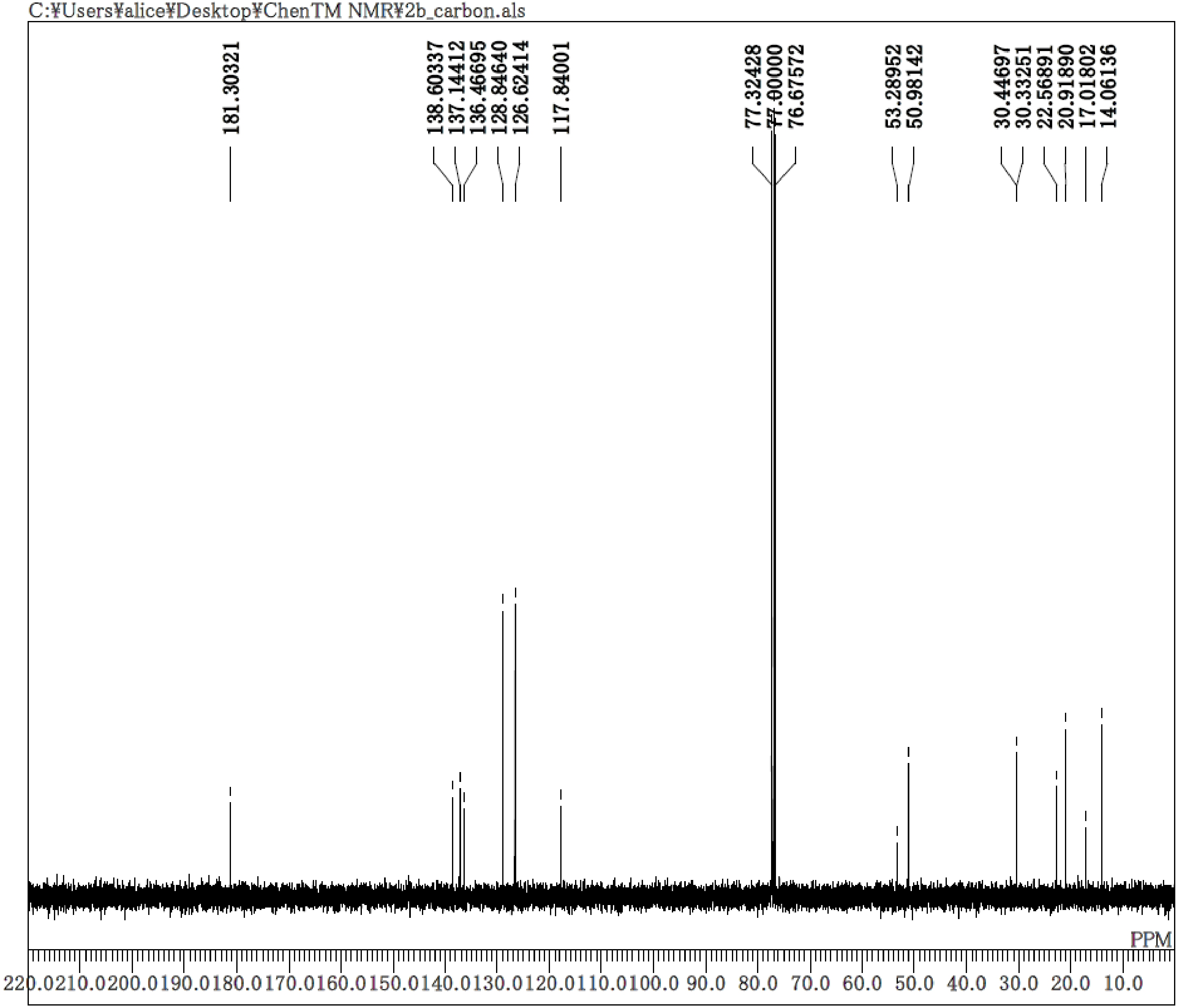
127.23455
126.96750
118.48857
77.32428
77.00000
76.67572
53.91900
50.28518
29.97008
28.23424
22.37815
17.13247
13.95645
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2a'_carbon.als
COMNT
DATIM 04-04-2020 12:53:18
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 244
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.72 Hz
RGAIN 60



DFILE	2b_proton.als
COMNT	
DATIM	16-03-2020 22:12:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	16
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	44

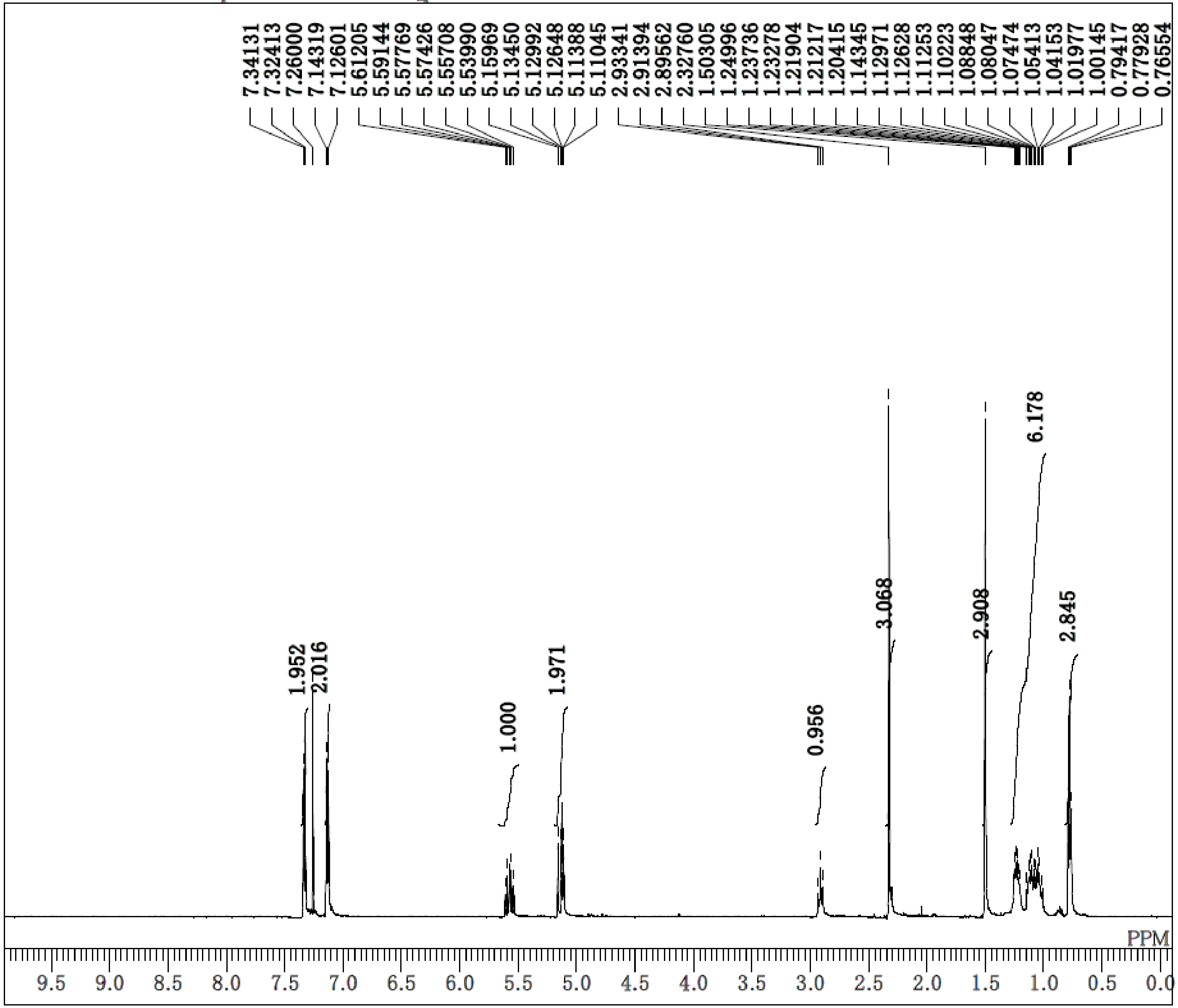


(2*S*,3*R*)-**2b**

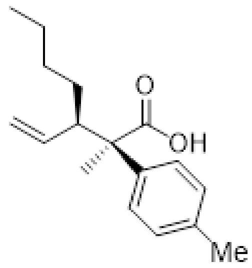


DFILE	2b_carbon.als
COMNT	
DATIM	16-03-2020 22:35:28
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	176
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

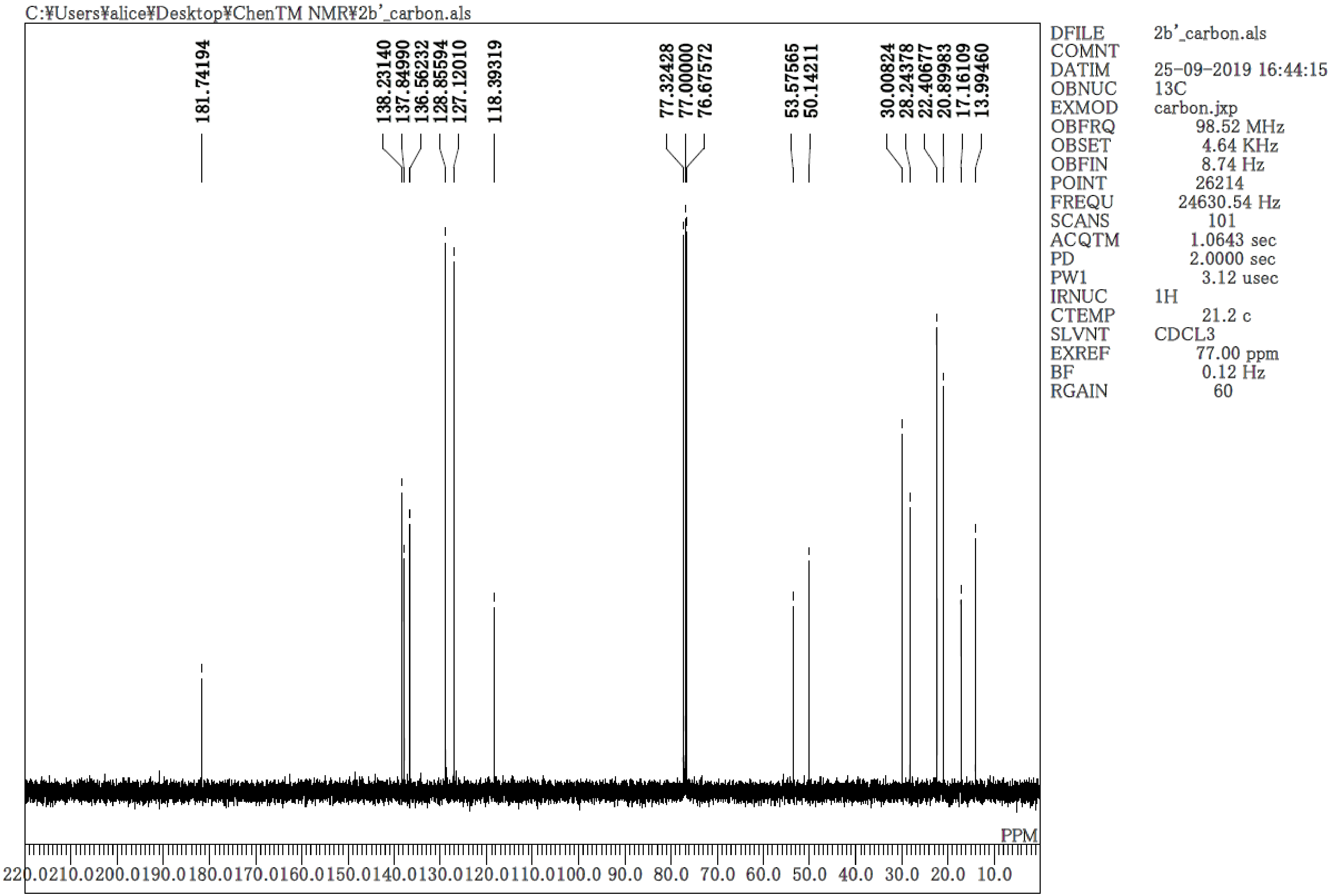
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2b'_proton.als



DFILE	2b'_proton.als
COMNT	
DATIM	2019-09-25 14:42:26
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

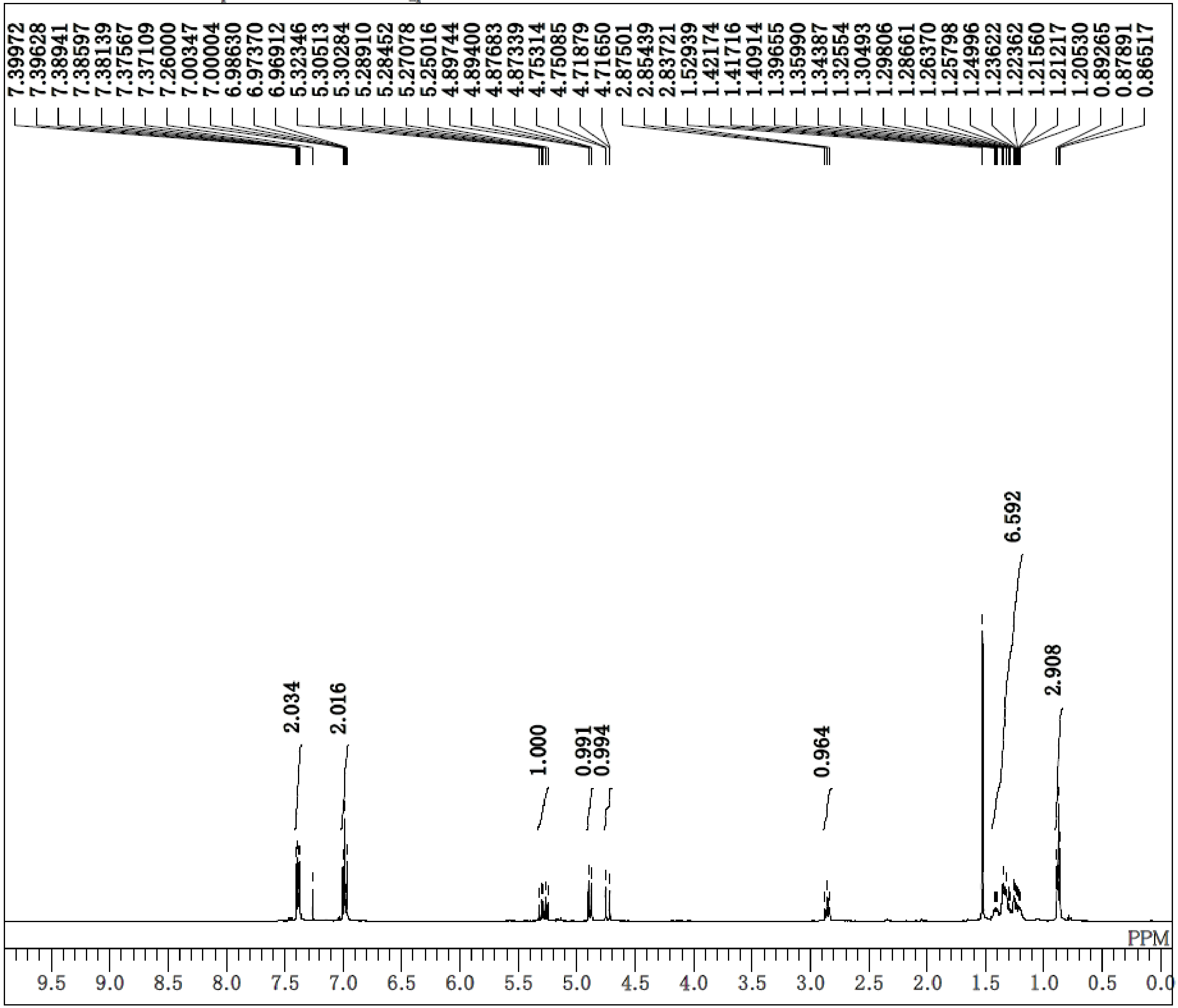


(2*R*,3*R*)-**2b**

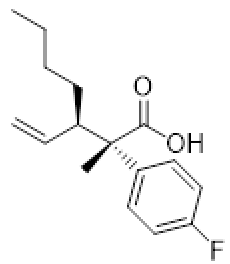


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2b'_carbon.als
181.74194
138.23140
137.84990
136.56232
128.85594
127.12010
118.39319
77.32428
77.00000
76.67572
53.57565
50.14211
30.00824
28.24378
22.40677
20.89983
17.16109
13.99460
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2b'_carbon.als
COMNT
DATIM 25-09-2019 16:44:15
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 101
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2c_proton.als

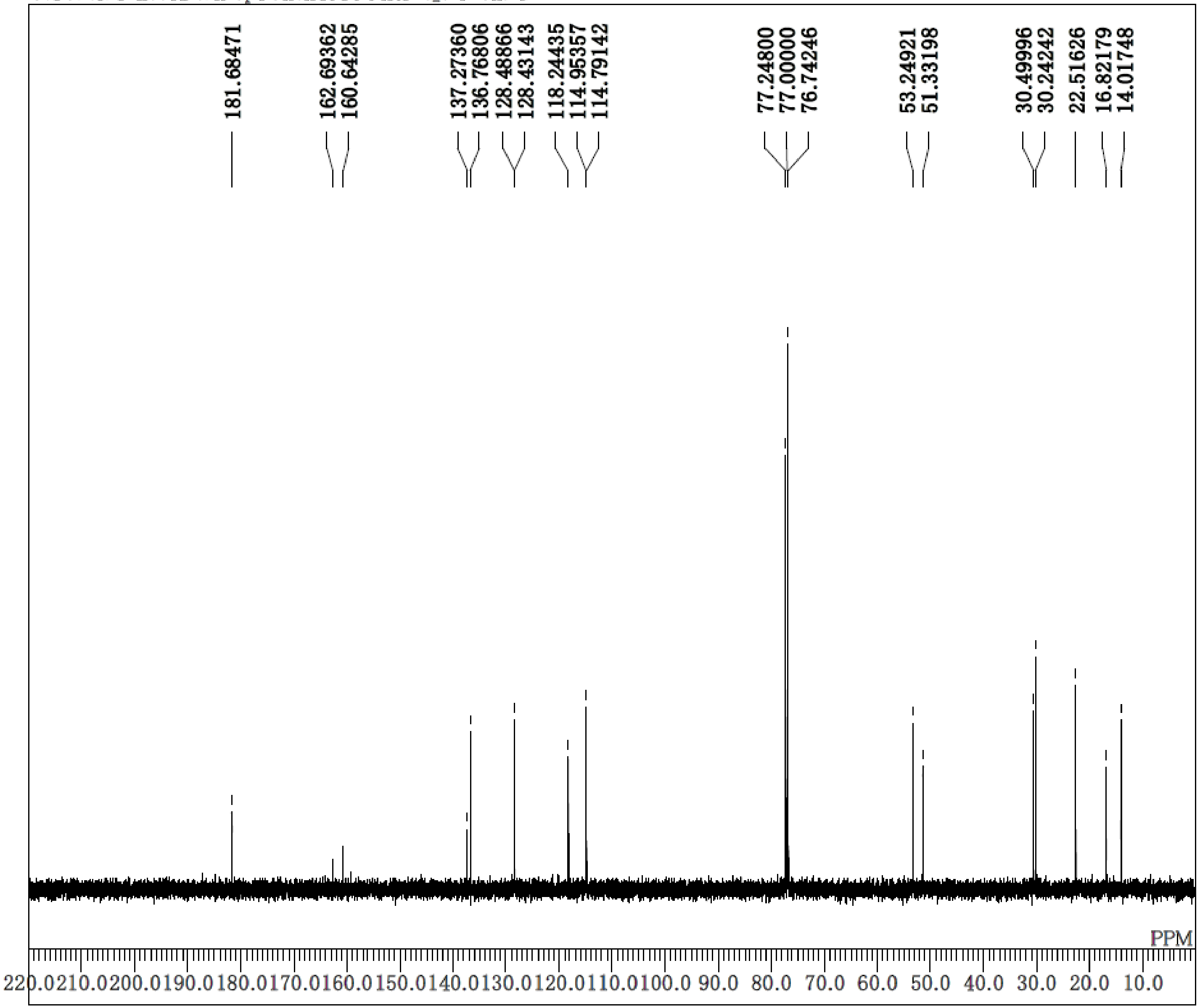


DFILE	2c_proton.als
COMNT	
DATIM	2019-09-26 13:36:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



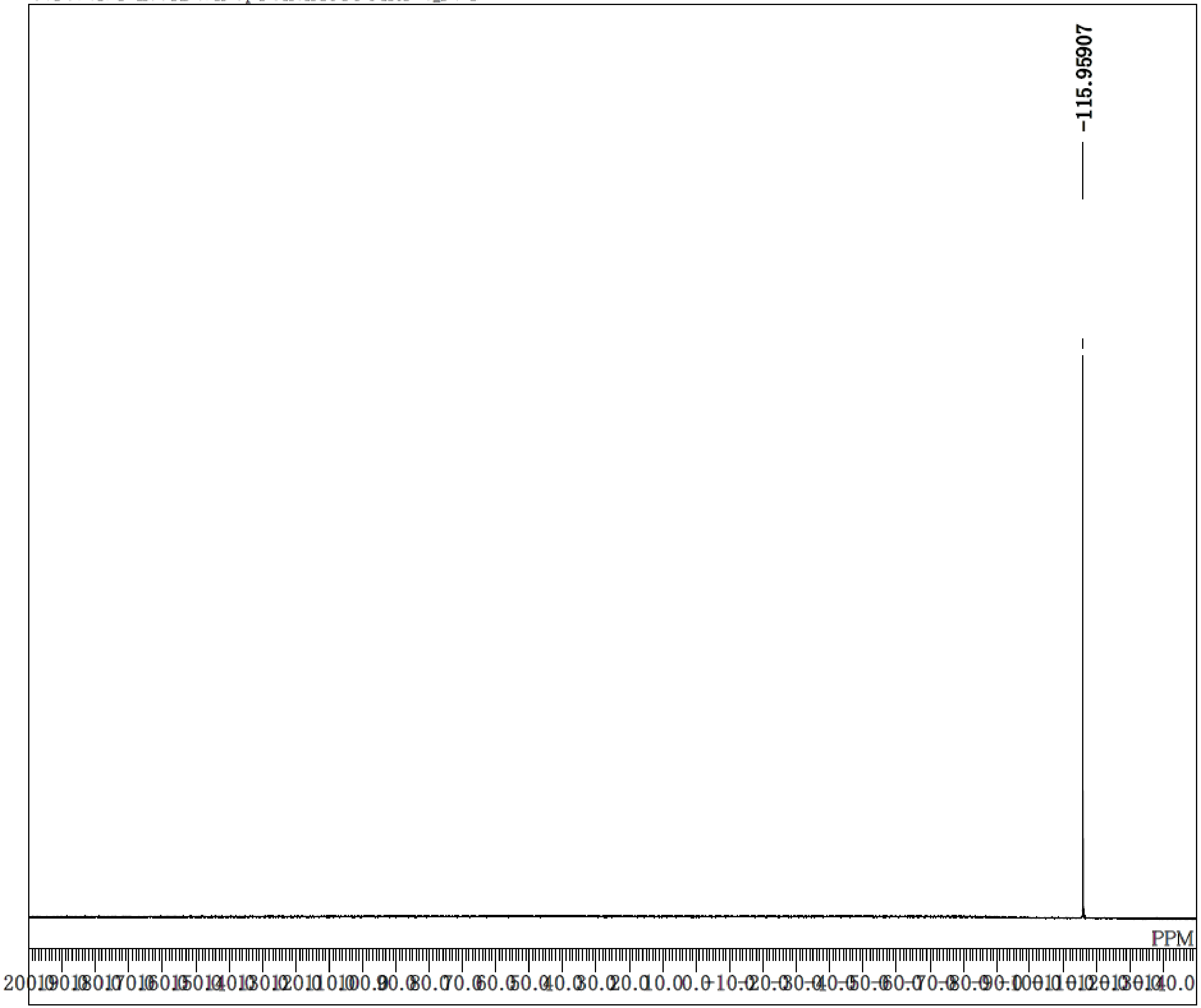
(2*S*,3*R*)-**2c**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2c_carbon.als



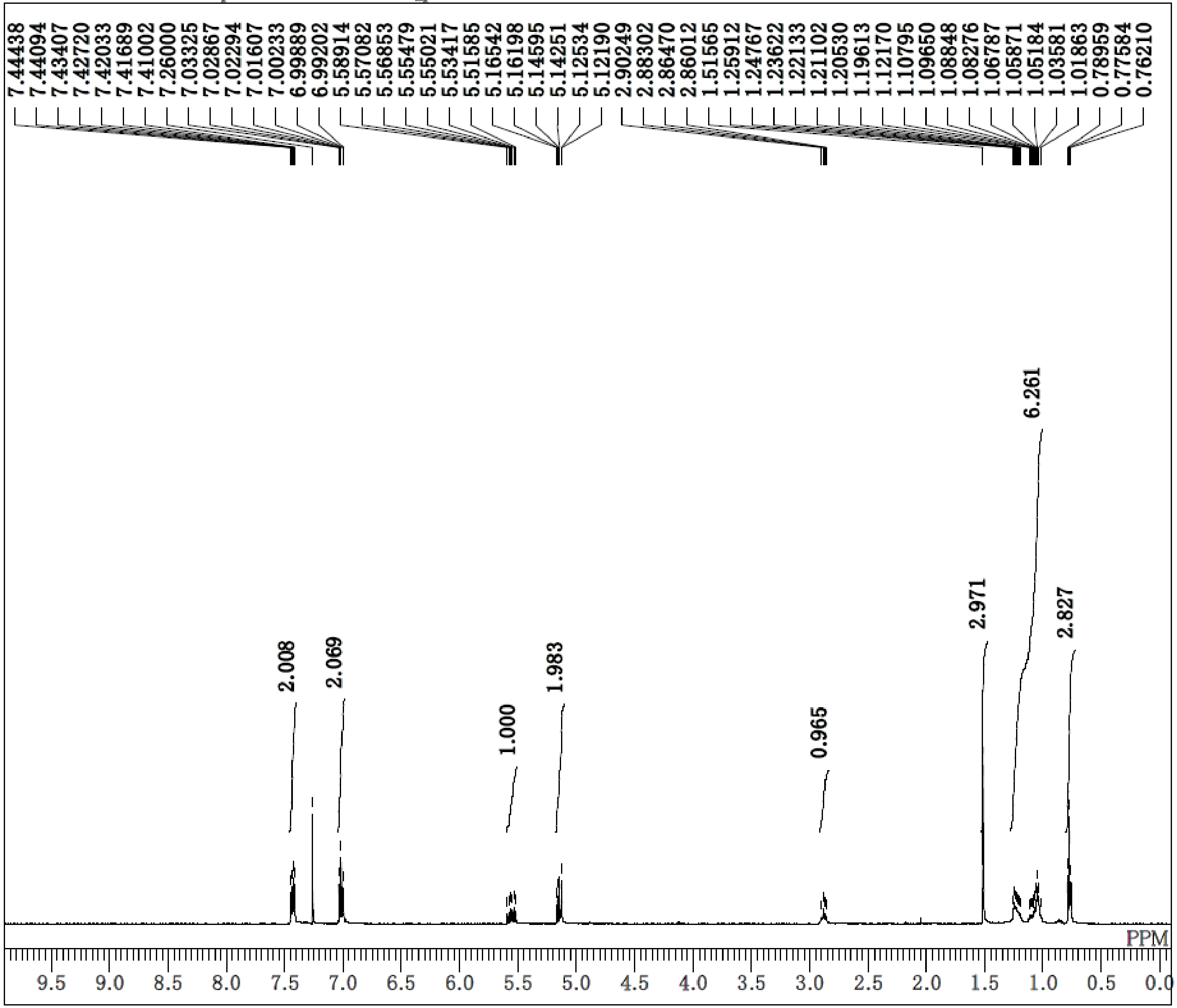
DFILE	2c_carbon.als
COMNT	
DATIM	2019-09-10 21:24:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	99
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2c_F.als

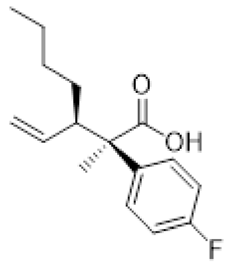


DFILE	2c_F.als
COMNT	
DATIM	2020-06-16 01:37:42
OBNUC	19F
EXMOD	proton.jxp
OBFRQ	368.64 MHz
OBSET	7.63 KHz
OBFIN	2.85 Hz
POINT	13107
FREQU	149253.73 Hz
SCANS	16
ACQTM	0.0878 sec
PD	6.0000 sec
PW1	4.10 usec
IRNUC	19F
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50

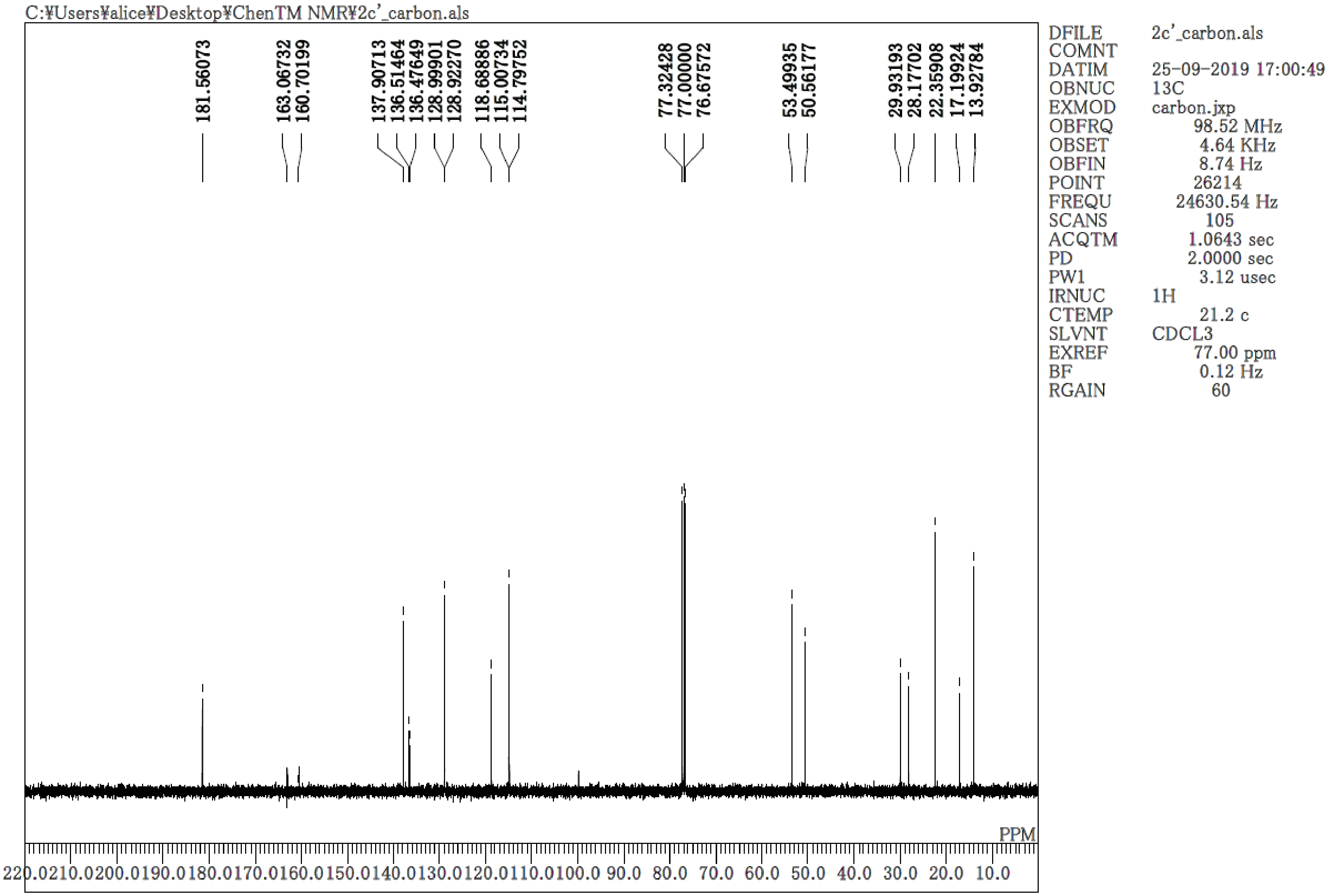
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2c'_proton.als



DFILE	2c'_proton.als
COMNT	
DATIM	2019-09-25 14:56:59
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

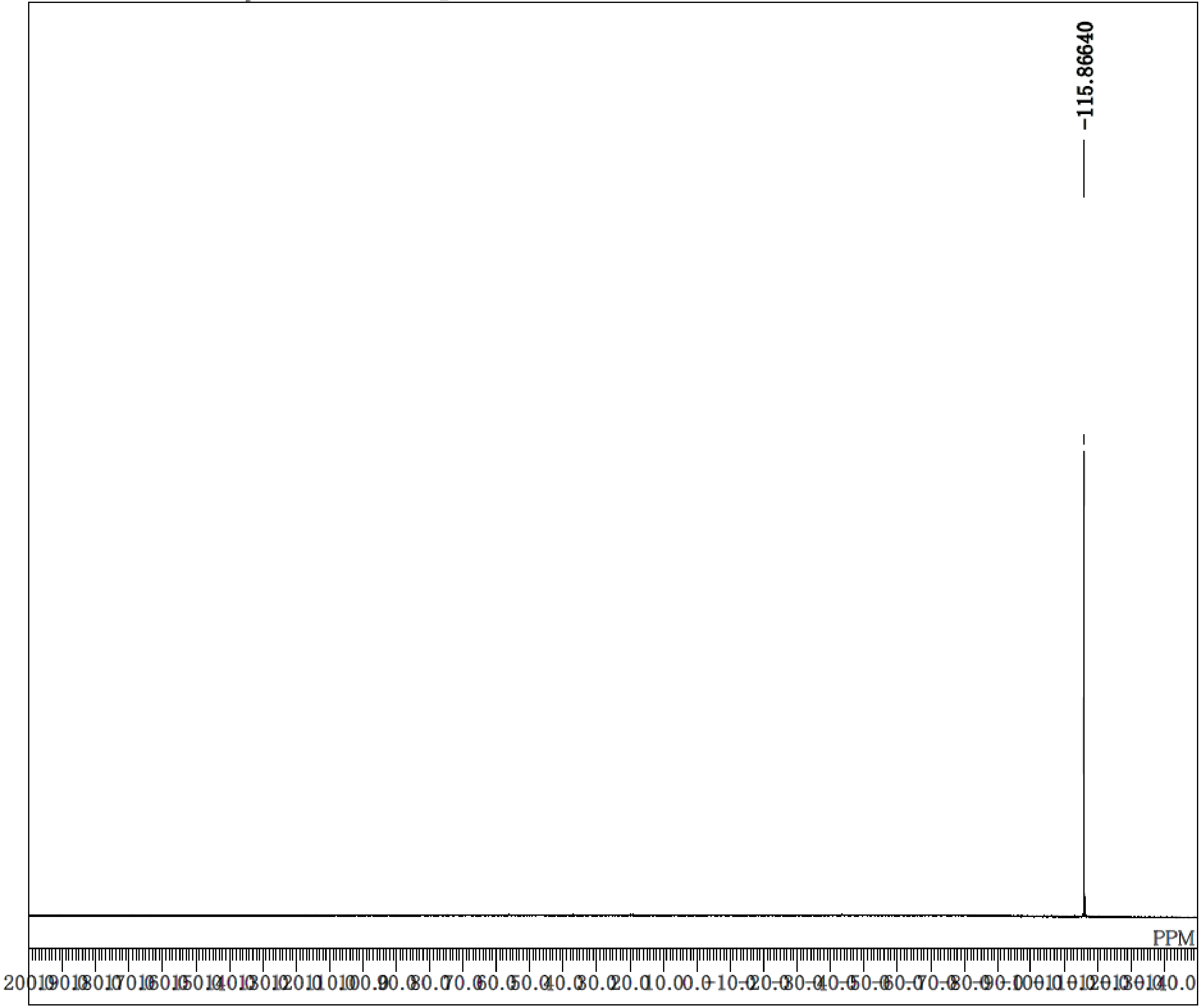


(2*R*,3*R*)-**2c**



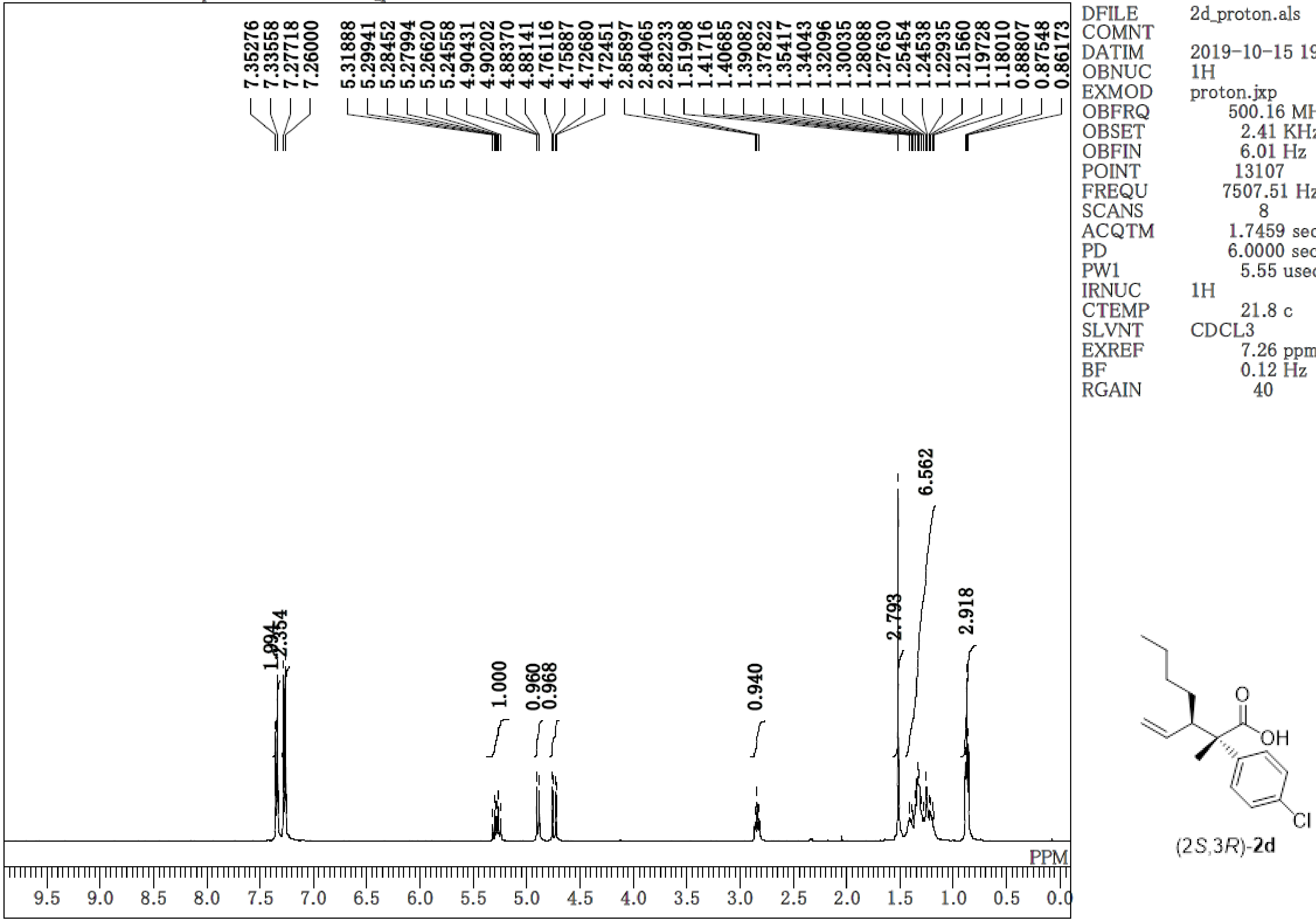
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2c'_carbon.als
181.56073
163.06732
160.70199
137.90713
136.51464
136.47649
128.99901
128.92270
118.68886
115.00734
114.79752
77.32428
77.00000
76.67572
53.49935
50.56177
29.93193
28.17702
22.35908
17.19924
13.92784
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2c'_carbon.als
COMNT
DATIM 25-09-2019 17:00:49
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 105
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2c'_F.als



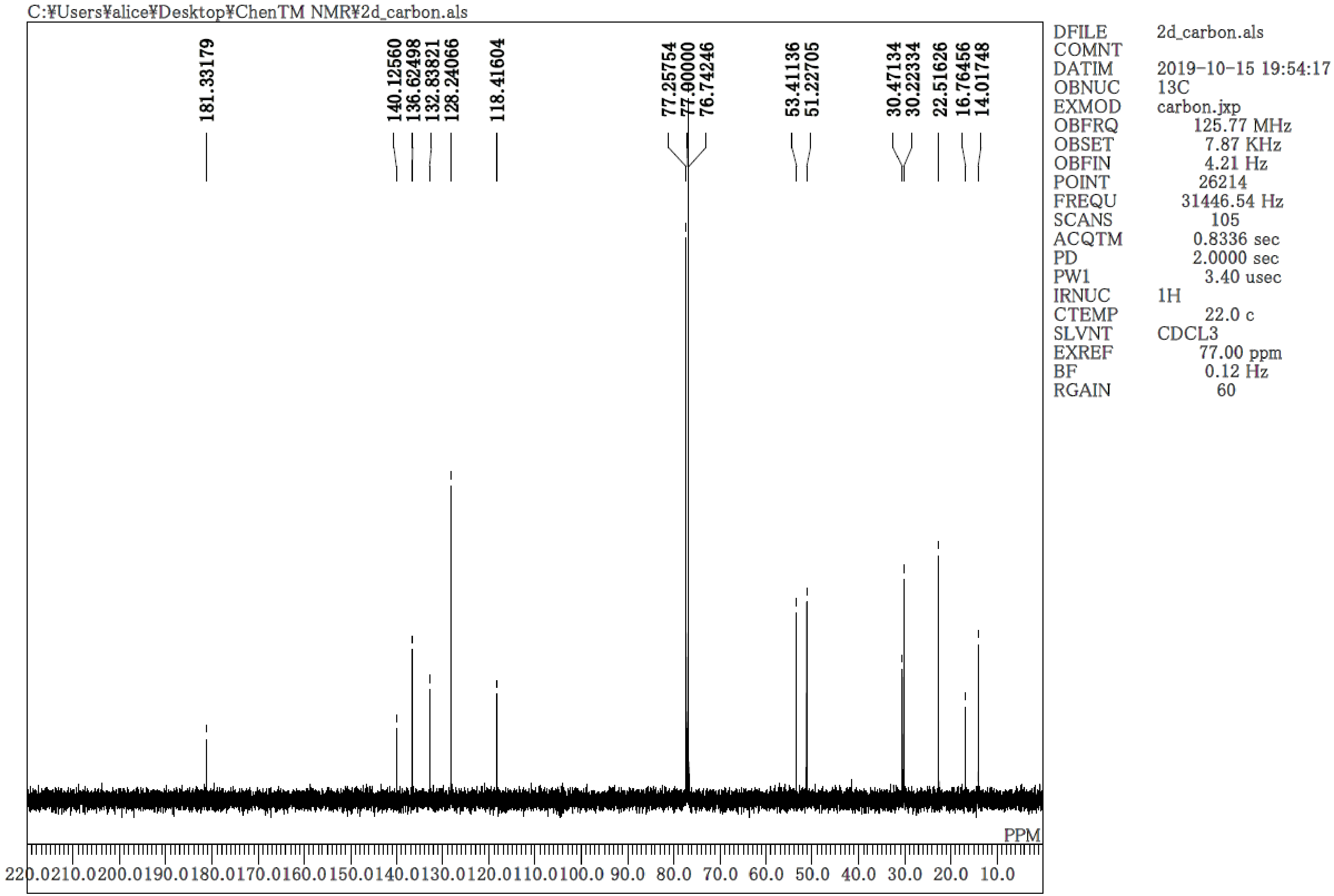
DFILE	2c'_F.als
COMNT	
DATIM	16-06-2020 02:03:46
OBNUC	19F
EXMOD	proton.jxp
OBFRQ	368.64 MHz
OBSET	7.63 KHz
OBFIN	2.85 Hz
POINT	13107
FREQU	149253.73 Hz
SCANS	16
ACQTM	0.0878 sec
PD	6.0000 sec
PW1	4.10 usec
IRNUC	19F
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	50

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2d_proton.als



DFILE	2d_proton.als
COMNT	
DATIM	2019-10-15 19:38:17
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

(2*S*,3*R*)-**2d**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2d_carbon.als
181.33179
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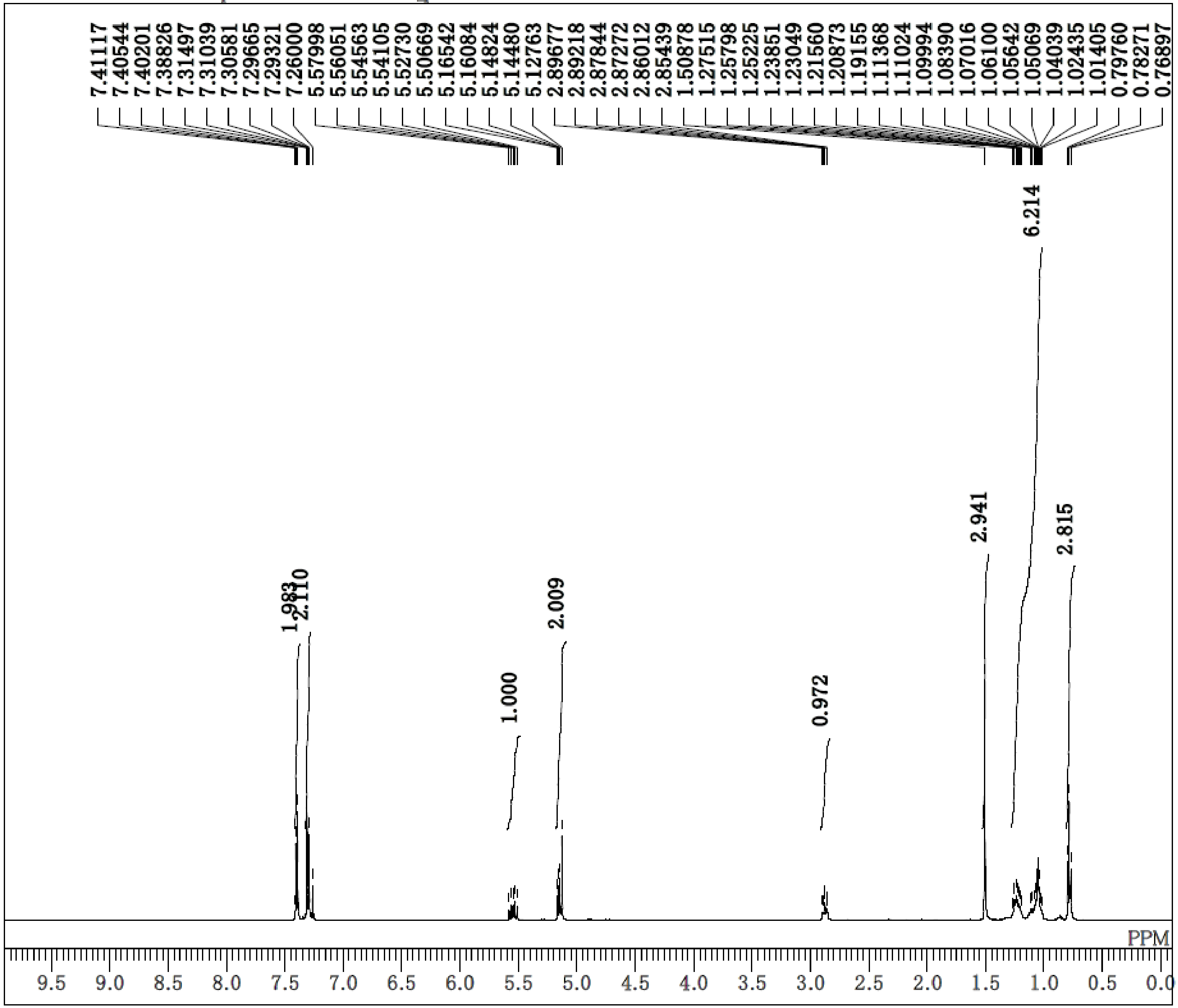
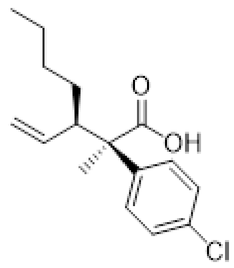
128.24066
118.41604
77.25754
77.00000
76.74246
53.41136
51.22705
30.47134
30.22334
22.51626
16.76456
14.01748
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2d_carbon.als
COMNT
DATIM 2019-10-15 19:54:17
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 105
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

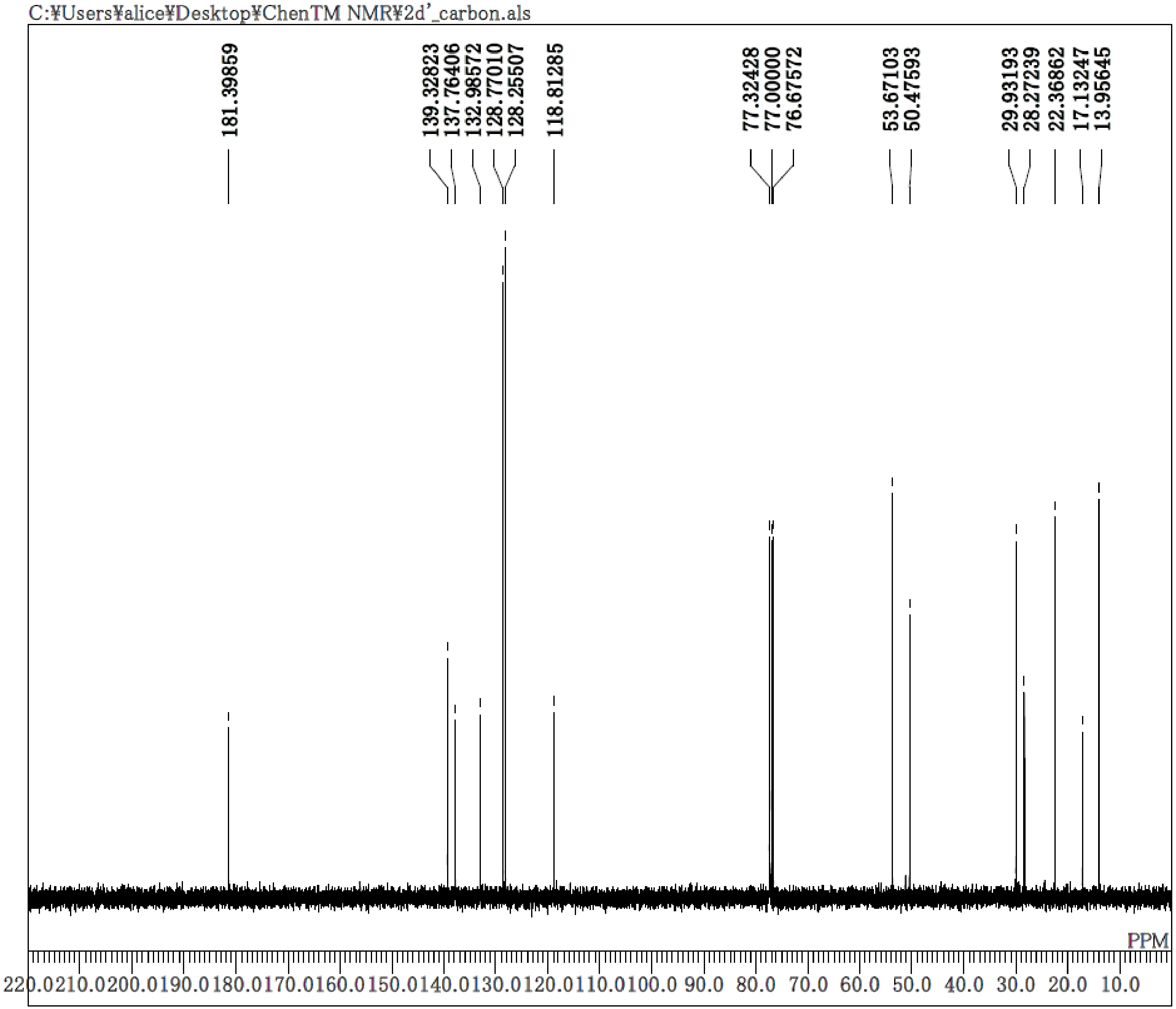
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2d'_proton.als



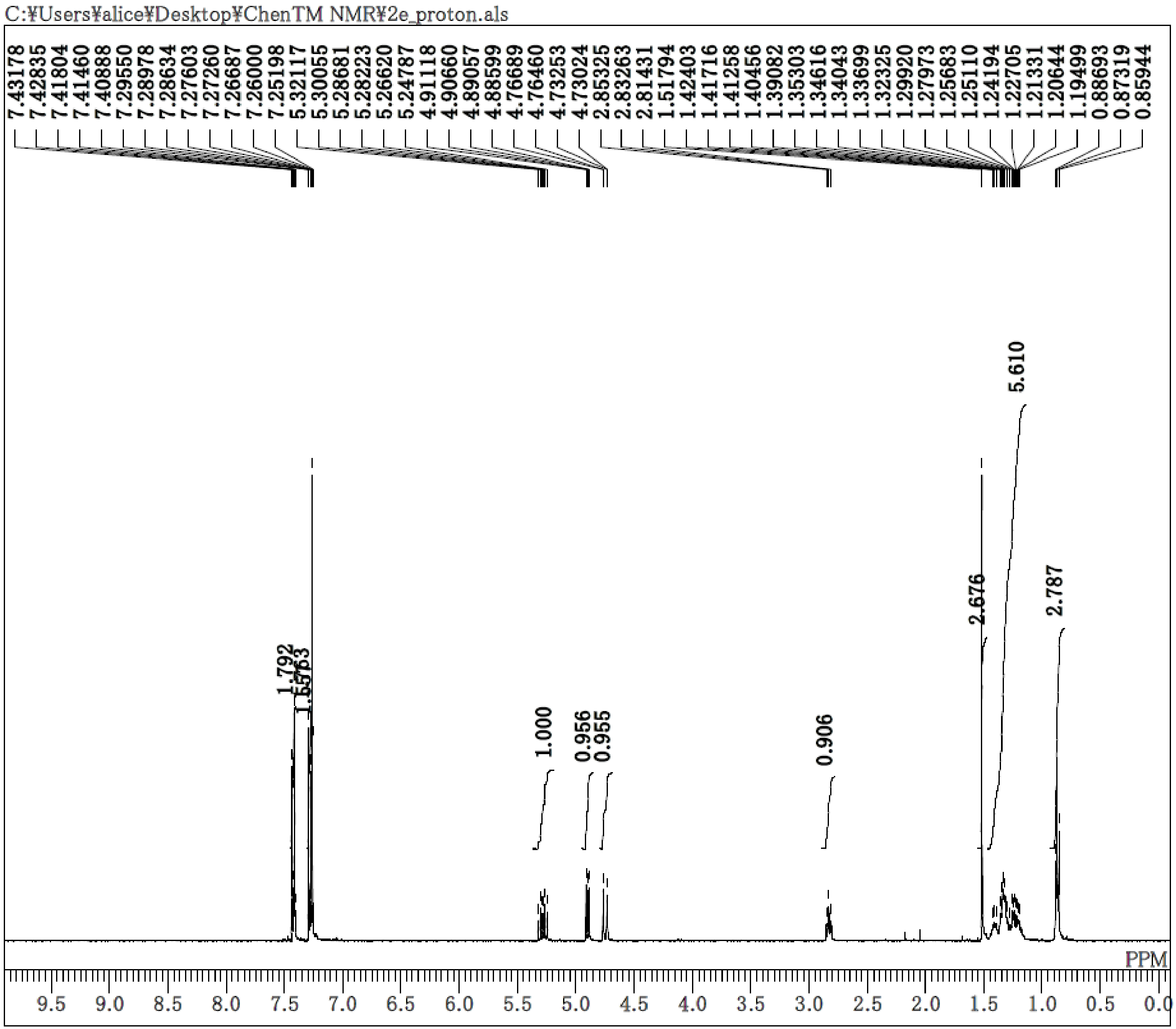
DFILE	2d'_proton.als
COMNT	
DATIM	2019-10-04 15:51:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



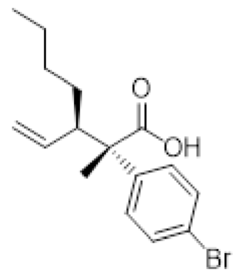
(2*R*,3*R*)-**2d**



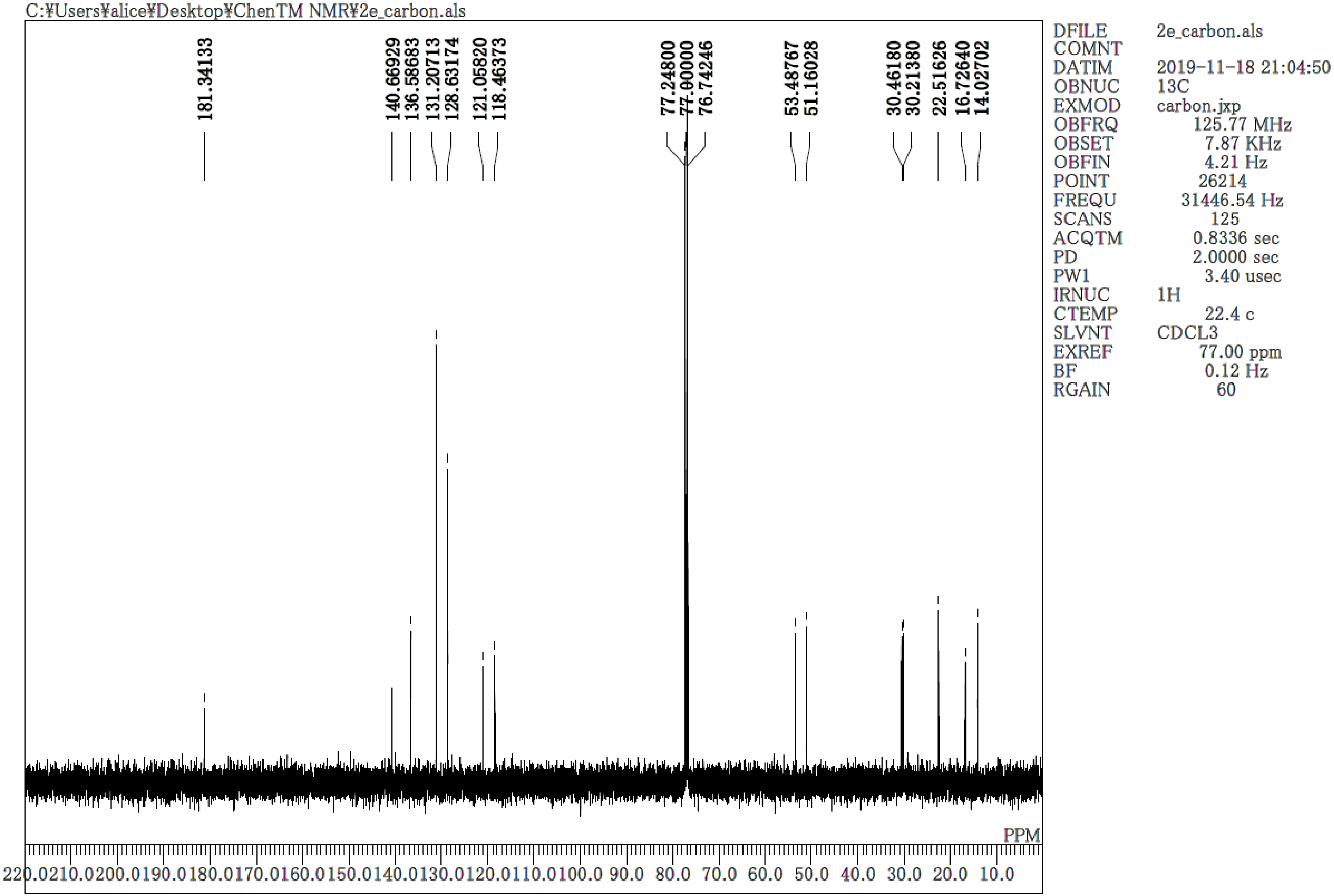
DFILE	2d'_carbon.als
COMNT	
DATIM	04-10-2019 19:12:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	90
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



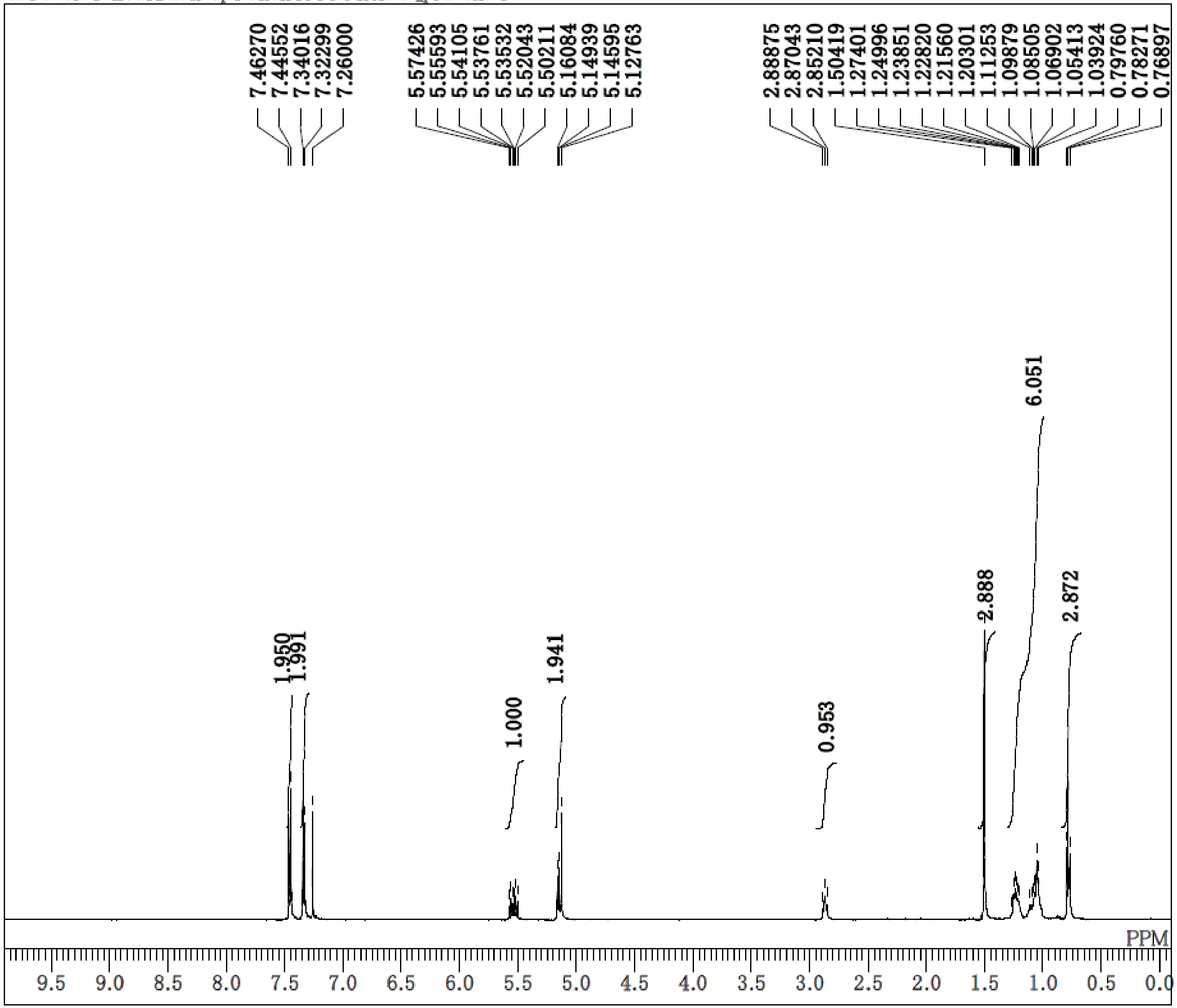
DFILE	2e_proton.als
COMNT	
DATIM	2019-11-18 21:00:17
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



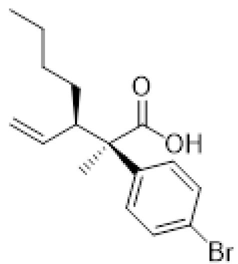
(2*S*,3*R*)-**2e**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2e_carbon.als
181.34133
140.66929
136.58683
131.20713
128.63174
121.05820
118.46373
77.24800
77.00000
76.74246
53.48767
51.16028
30.46180
30.21380
22.51626
16.72640
14.02702
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2e_carbon.als
COMNT
DATIM 2019-11-18 21:04:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 125
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2e'_proton.als

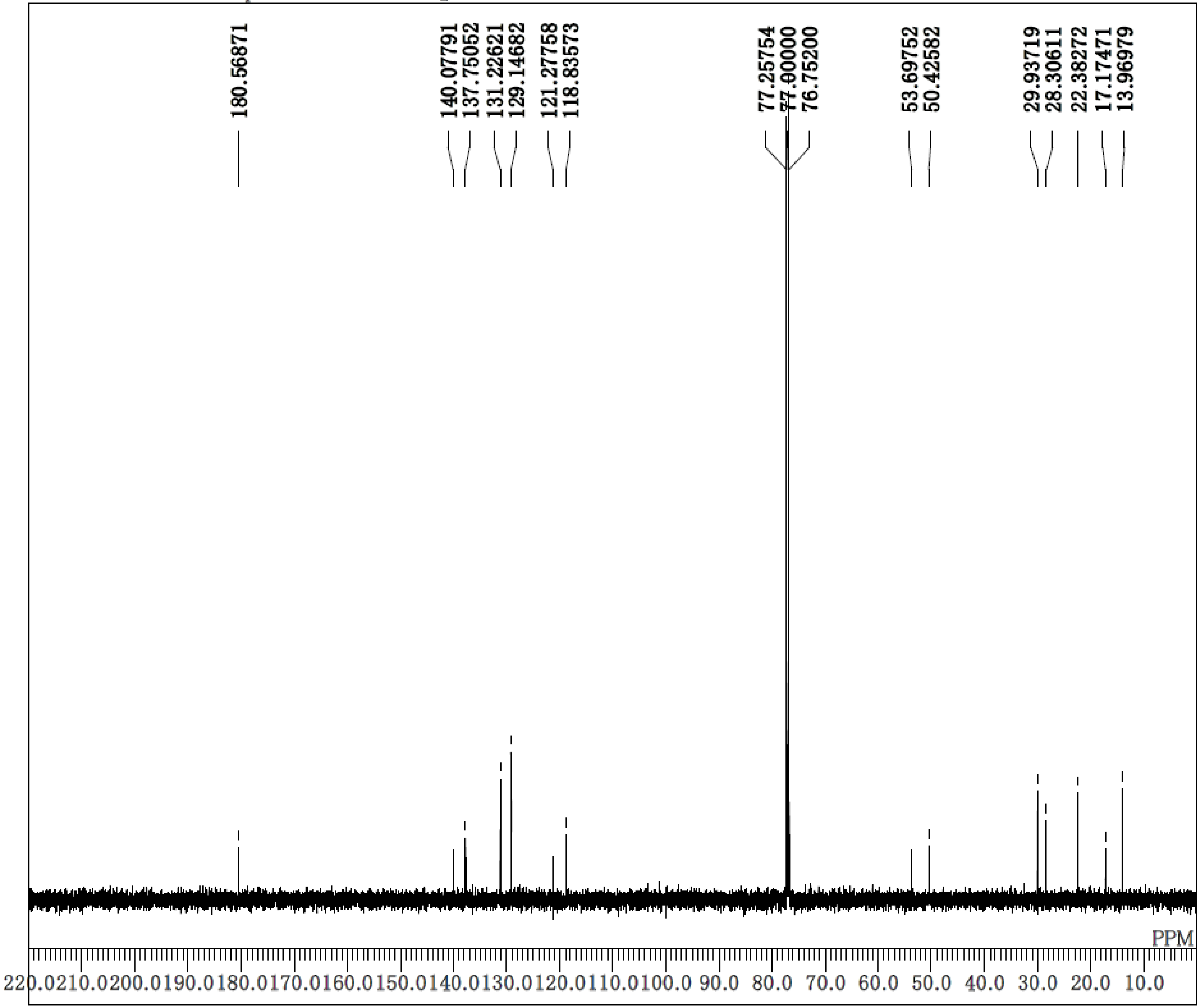


DFILE	2e'_proton.als
COMNT	
DATIM	2019-12-06 22:29:55
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



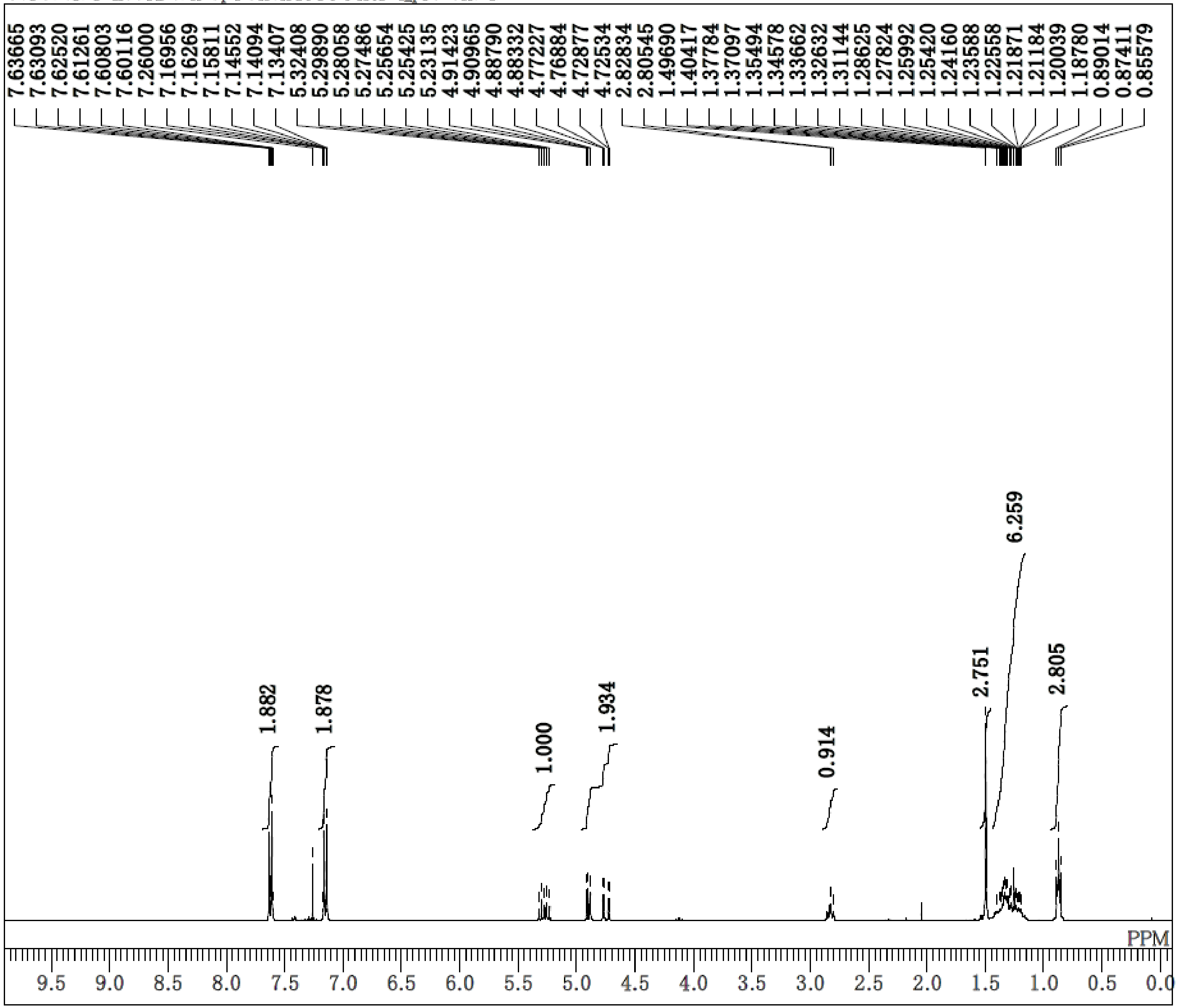
(2*R*,3*R*)-**2e**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2e'_carbon.als

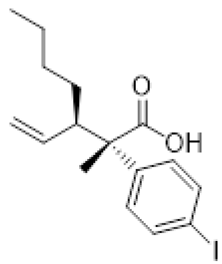


DFILE	2e'_carbon.als
COMNT	
DATIM	2019-12-06 22:36:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	298
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

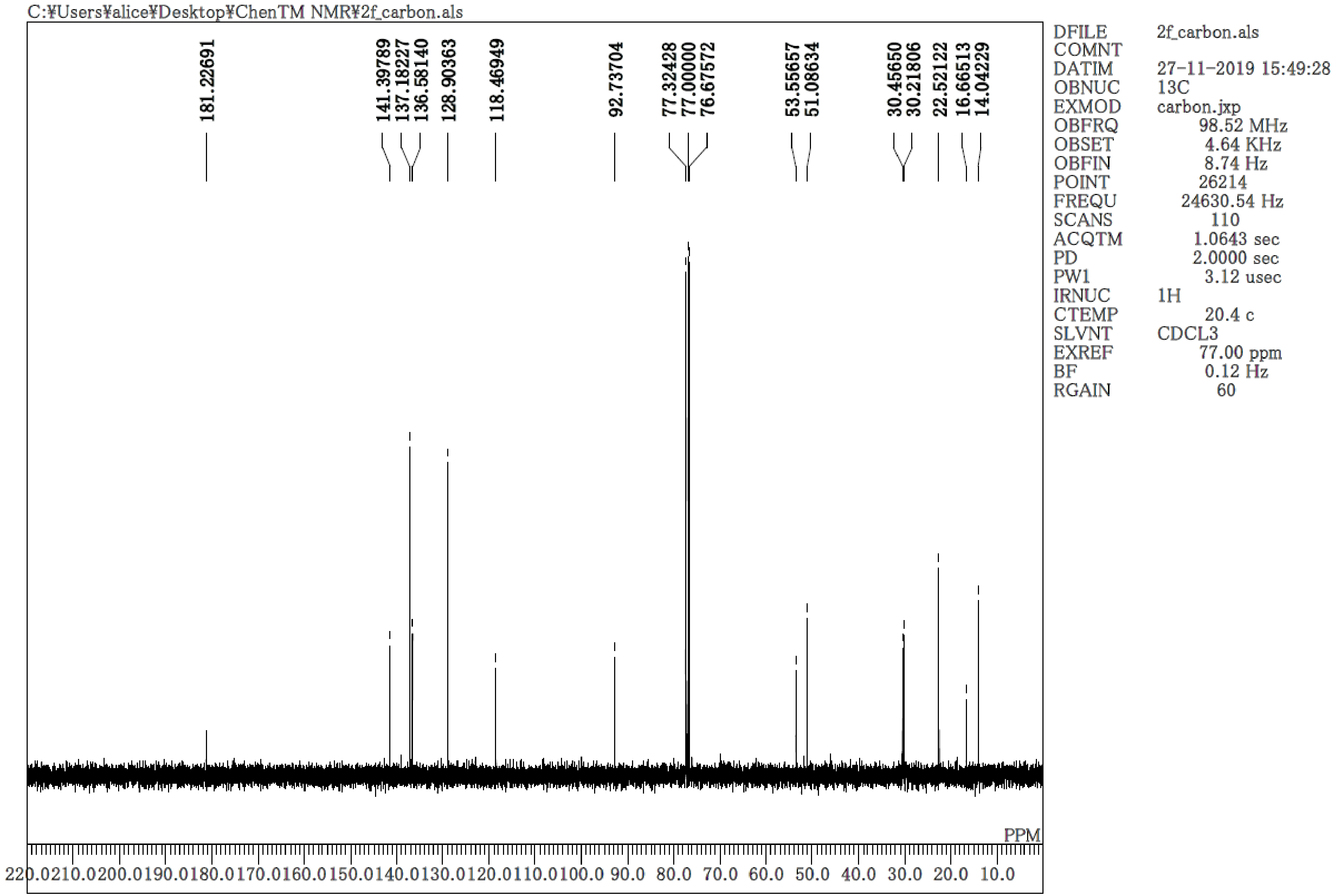
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2f_proton.als

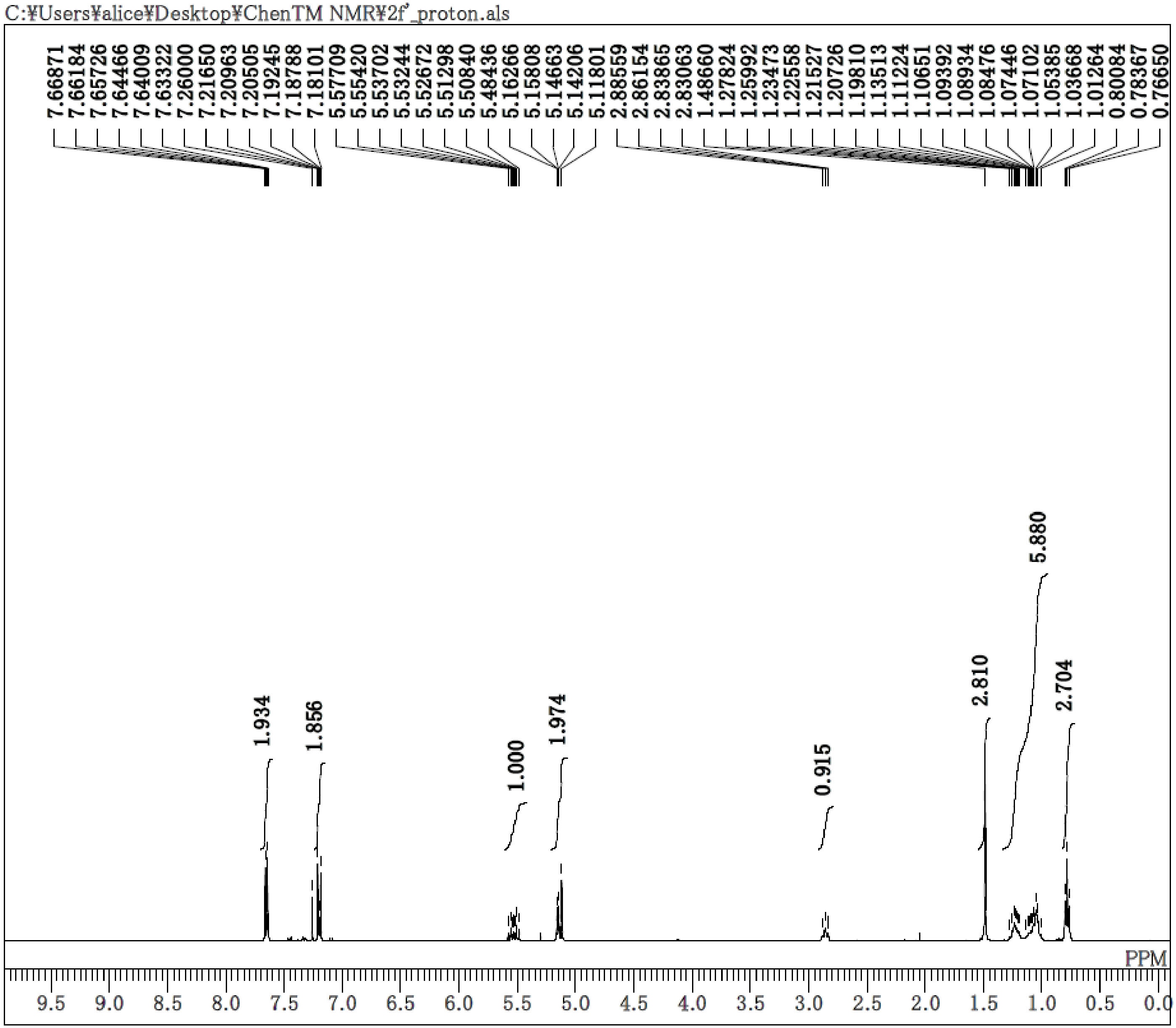


DFILE	2f_proton.als
COMNT	
DATIM	27-11-2019 15:12:13
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36

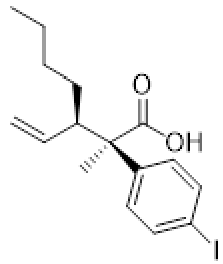


(2*S*,3*R*)-**2f**

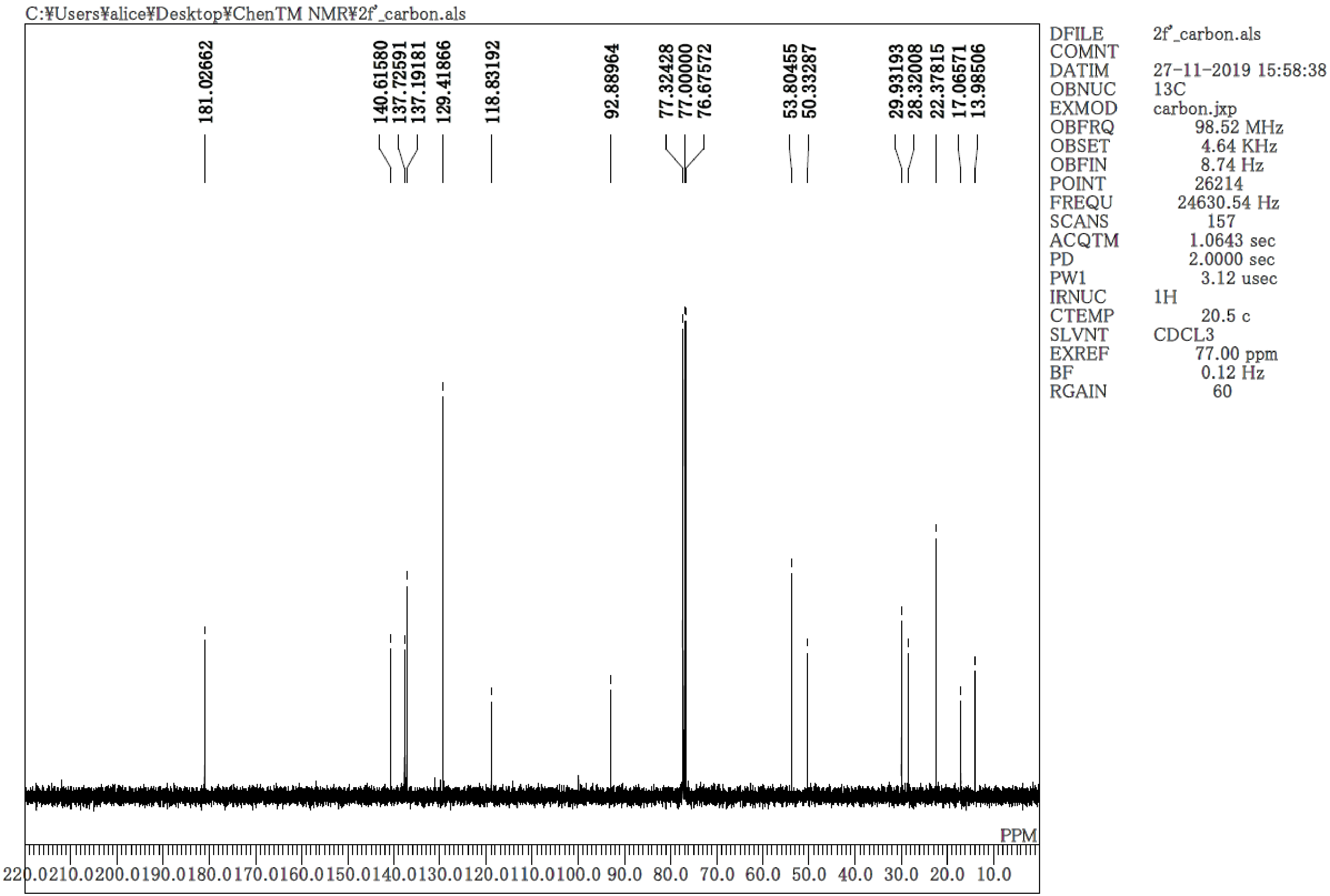

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2f_carbon.als
181.22691
141.39789
137.18227
136.58140
128.90363
118.46949
92.73704
77.32428
77.00000
76.67572
53.55657
51.08634
30.45650
30.21806
22.52122
16.66513
14.04229
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2f_carbon.als
COMNT
DATIM 27-11-2019 15:49:28
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 110
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



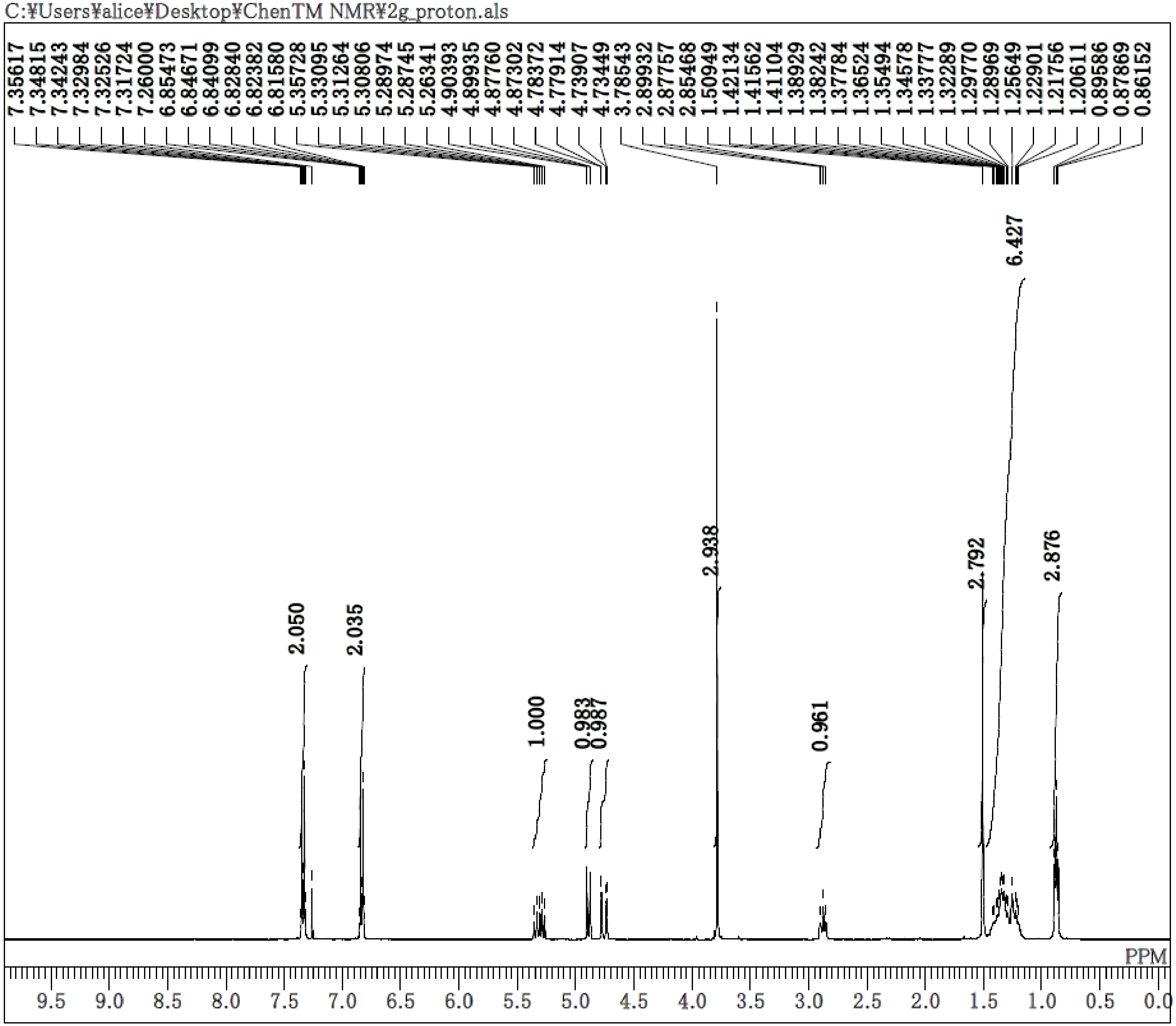
DFILE	2f'_proton.als
COMNT	
DATIM	27-11-2019 15:19:32
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36

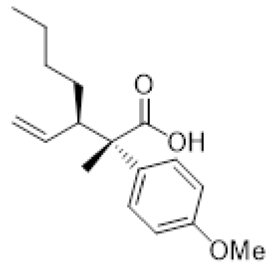


(2*R*,3*R*)-**2f**

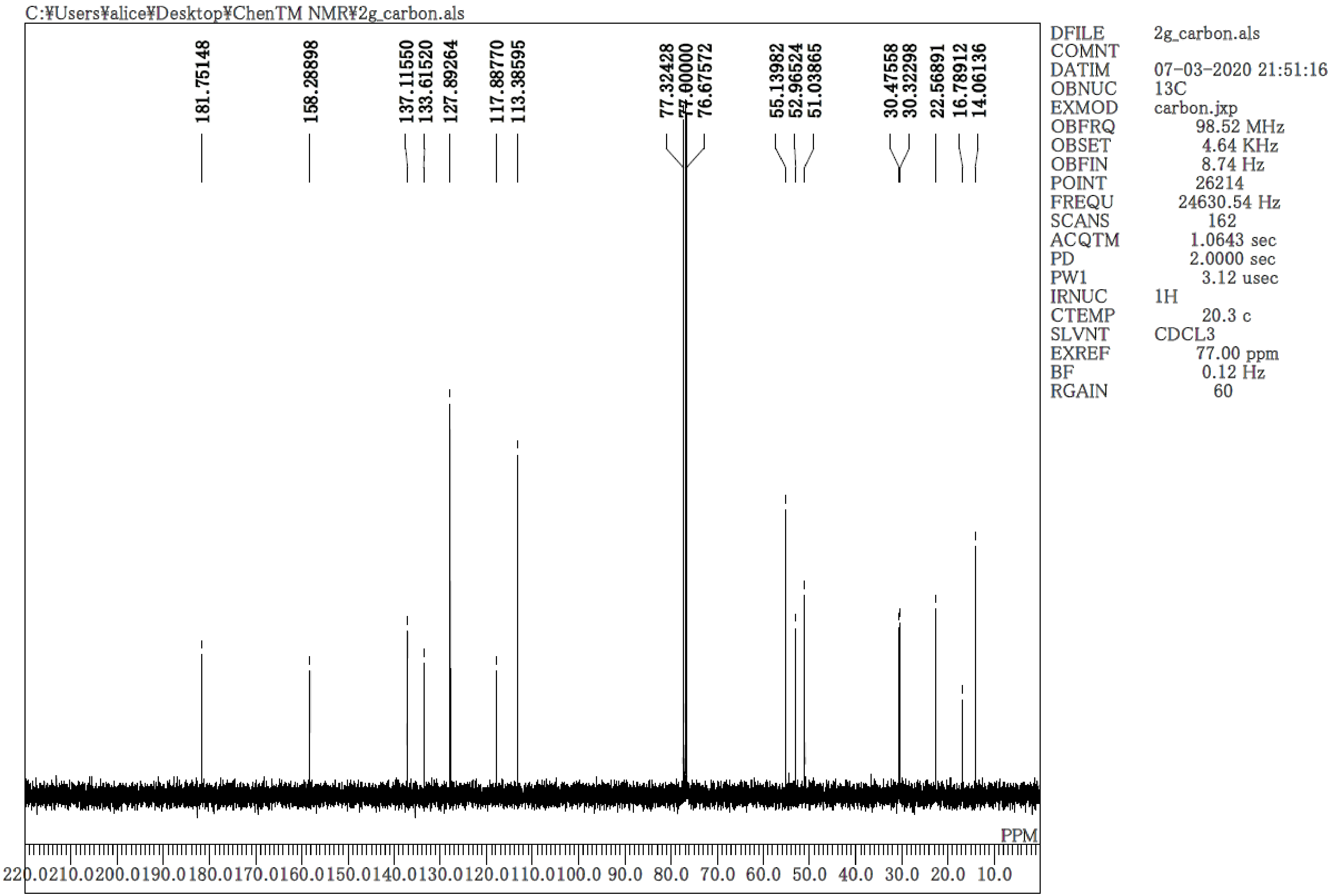


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2f'_carbon.als
181.02662
140.61580
137.72591
137.19181
129.41866
118.83192
92.88964
77.32428
77.00000
76.67572
53.80455
50.33287
29.93193
28.32008
22.37815
17.06571
13.98506
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2f'_carbon.als
COMNT
DATIM 27-11-2019 15:58:38
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 157
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2g_proton.als
DFILE 2g_proton.als
COMNT
DATIM 07-03-2020 21:36:40
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 34
PPM


O
OH
OMe

(2*S*,3*R*)-**2g**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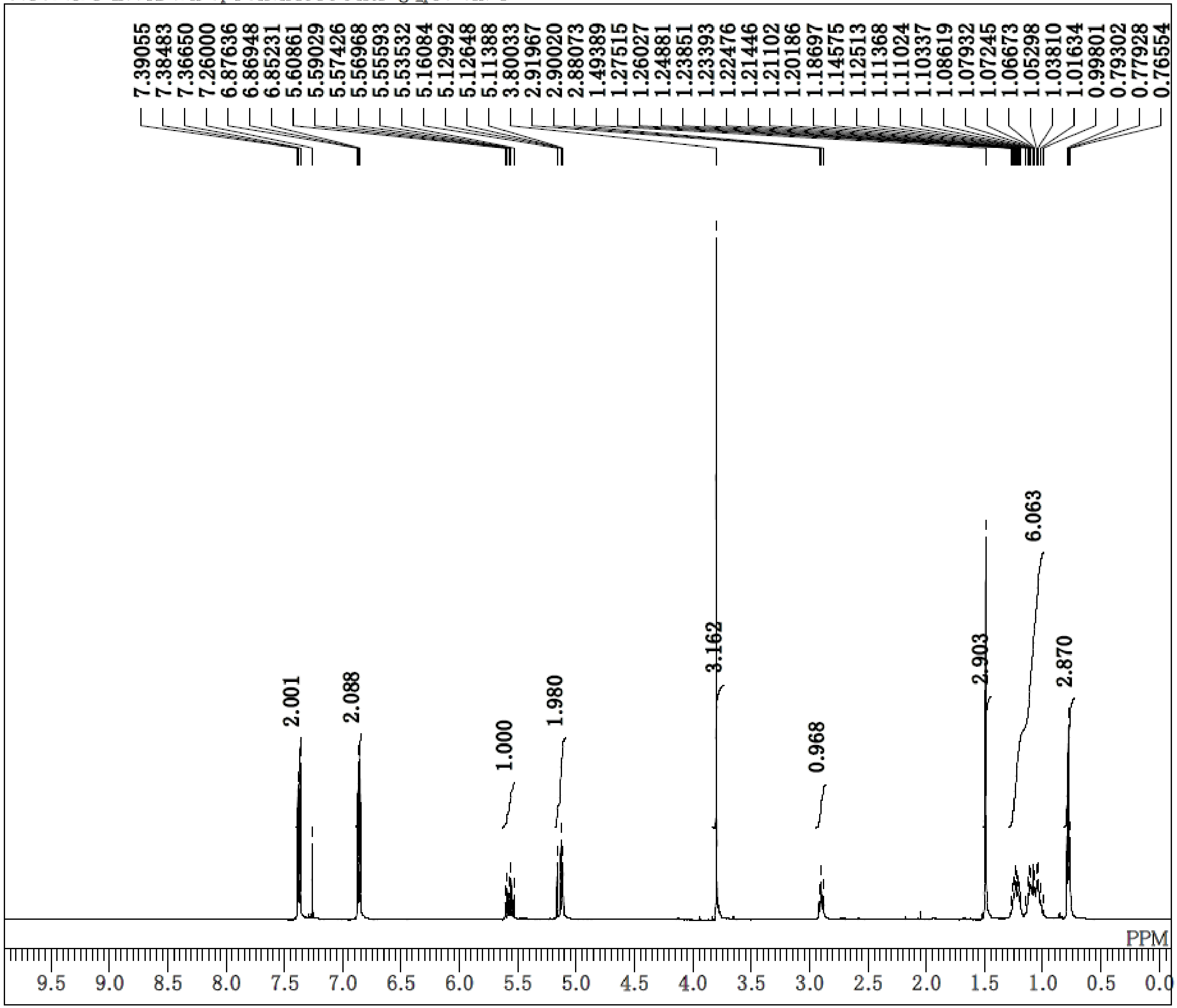
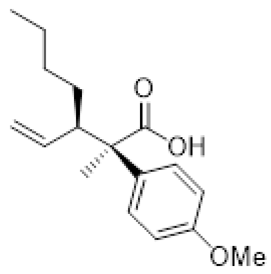
181.75148
158.28898
137.11550
133.61520
127.89264
117.88770
113.38595
77.32428
77.00000
76.67572
55.13982
52.96524
51.03865
30.47558
30.32298
22.56891
16.78912
14.06136
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2g_carbon.als
COMNT
DATIM 07-03-2020 21:51:16
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 162
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

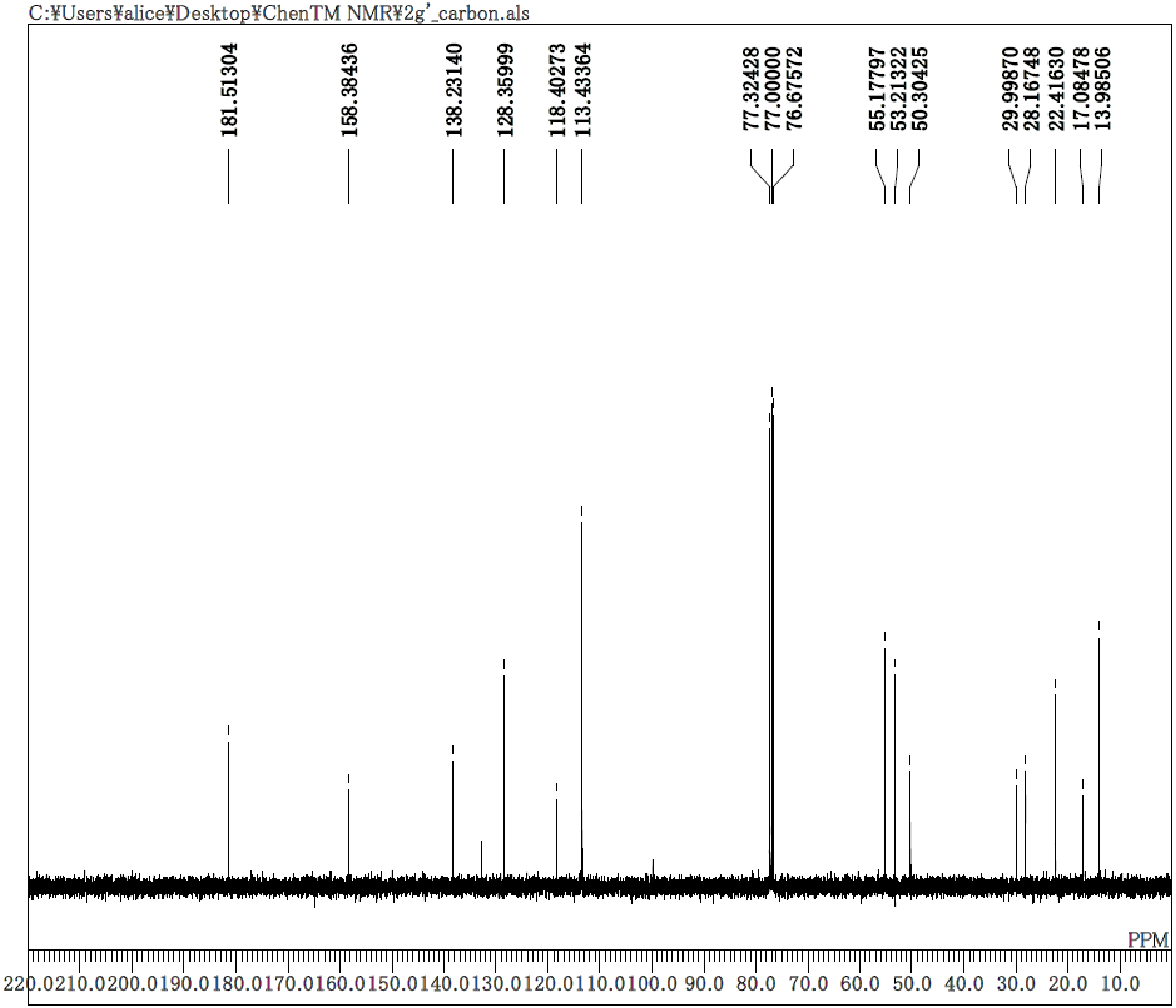
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2g'_proton.als



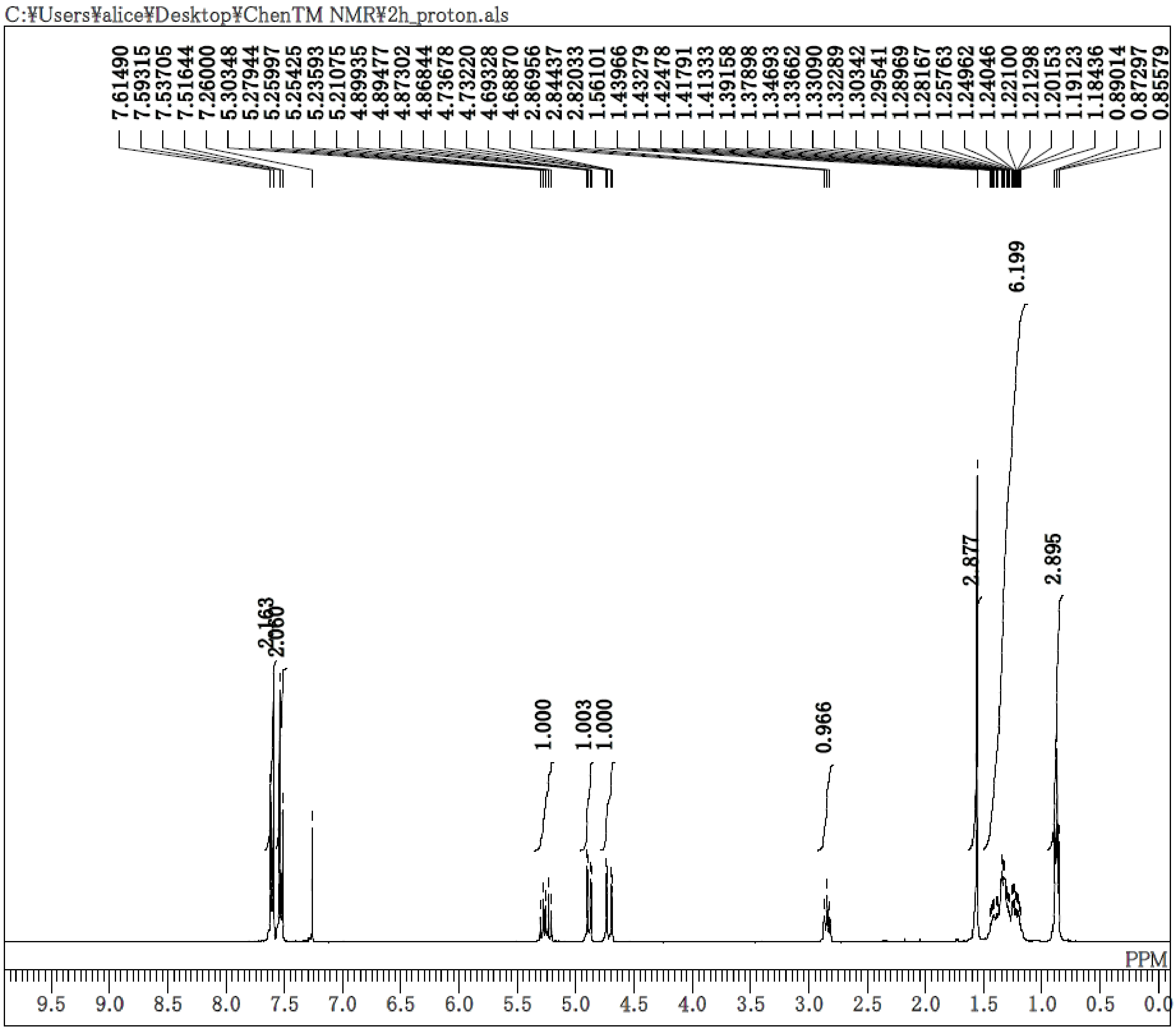
DFILE 2g'_proton.als
COMNT
DATIM 2019-10-04 15:57:51
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



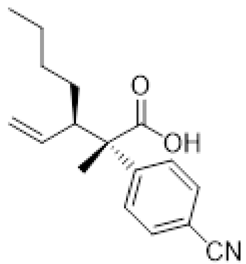
(2*R*,3*R*)-**2g**



DFILE	2g'_carbon.als
COMNT	
DATIM	04-10-2019 19:20:04
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	109
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

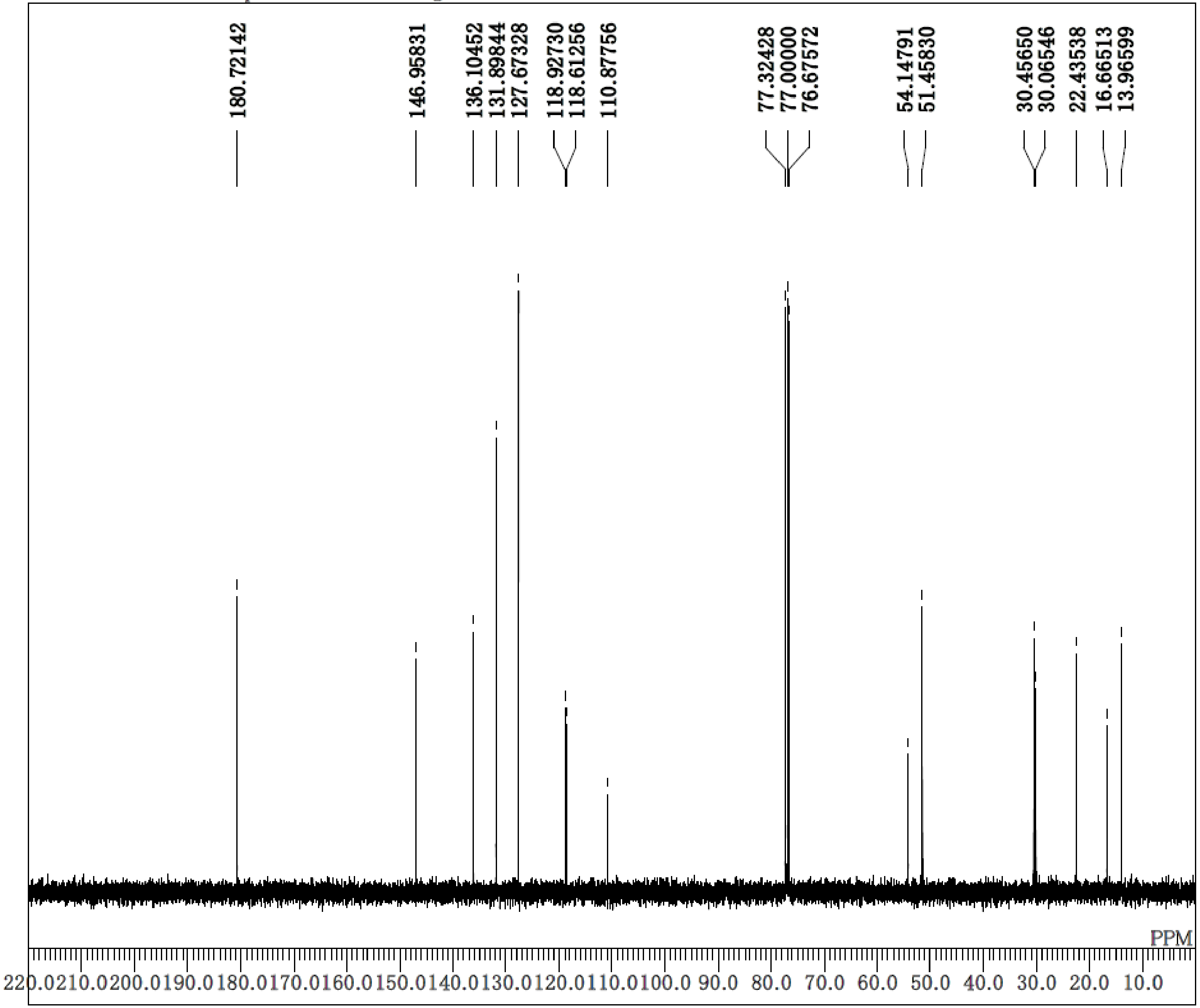


DFILE	2h_proton.als
COMNT	
DATIM	09-11-2019 11:41:53
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38

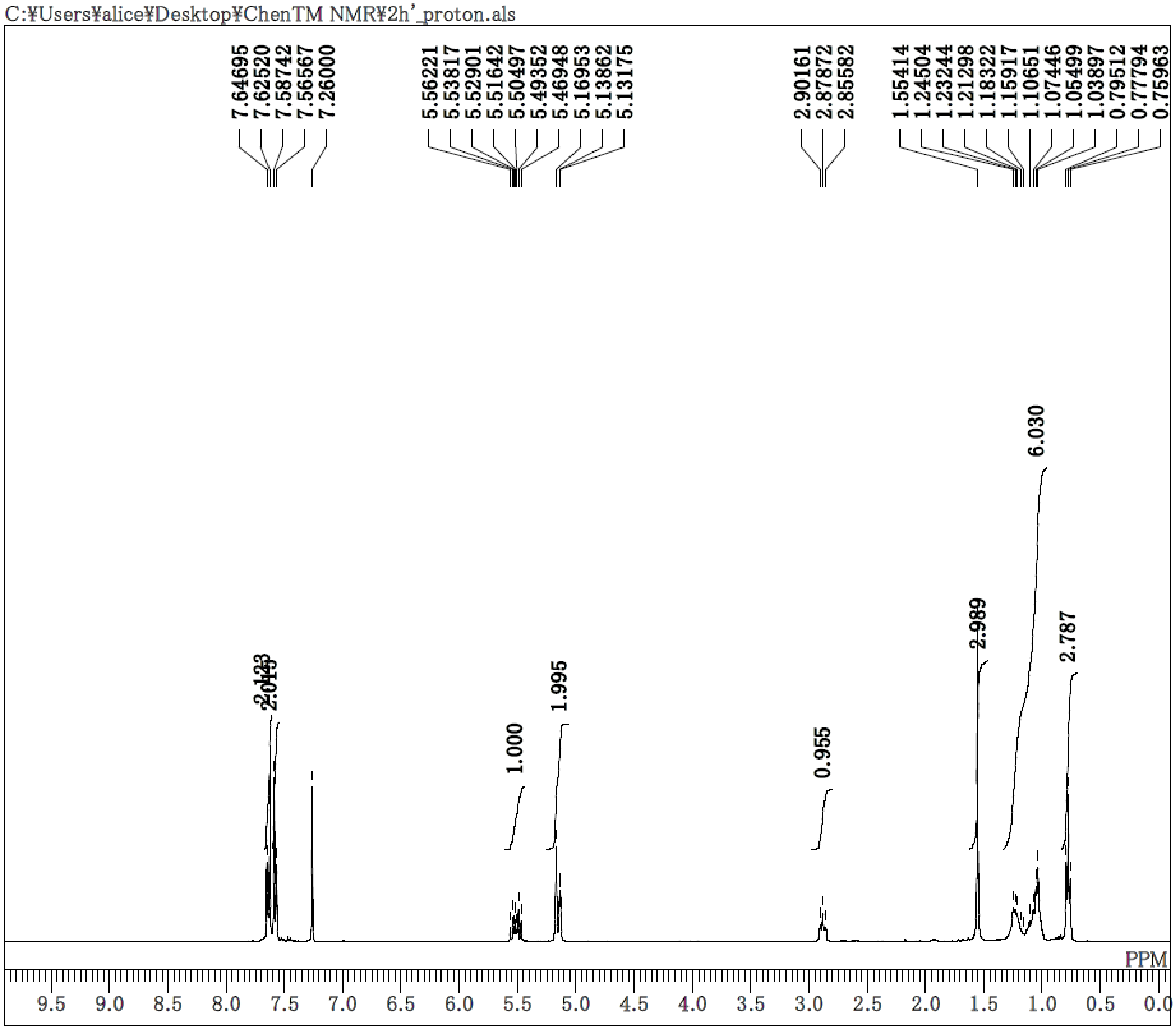


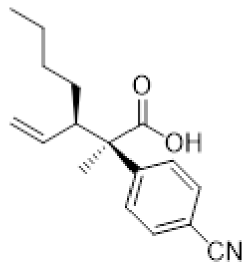
(2*S*,3*R*)-**2h**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2h_carbon.als

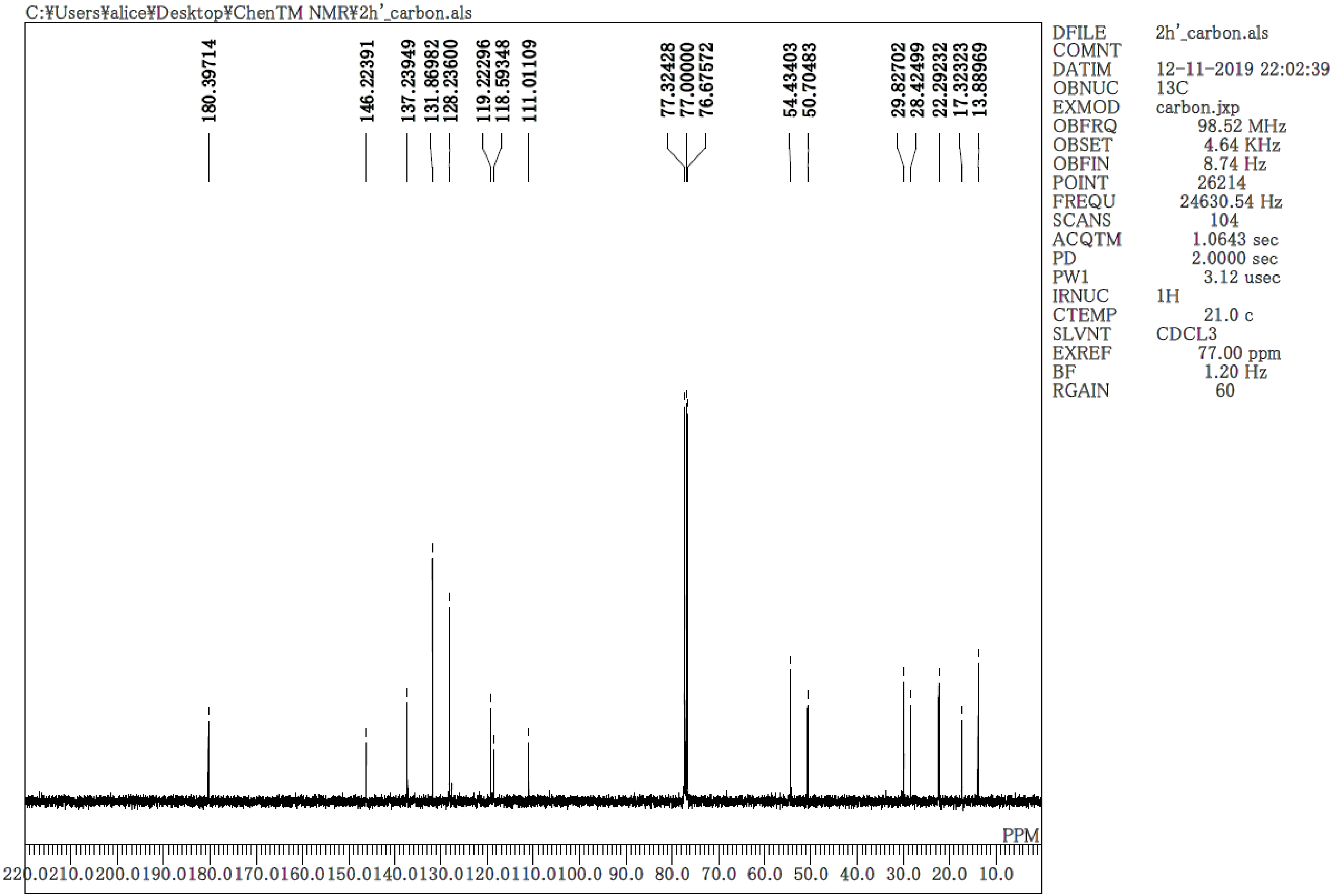


DFILE	2h_carbon.als
COMNT	
DATIM	09-11-2019 11:55:20
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	137
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

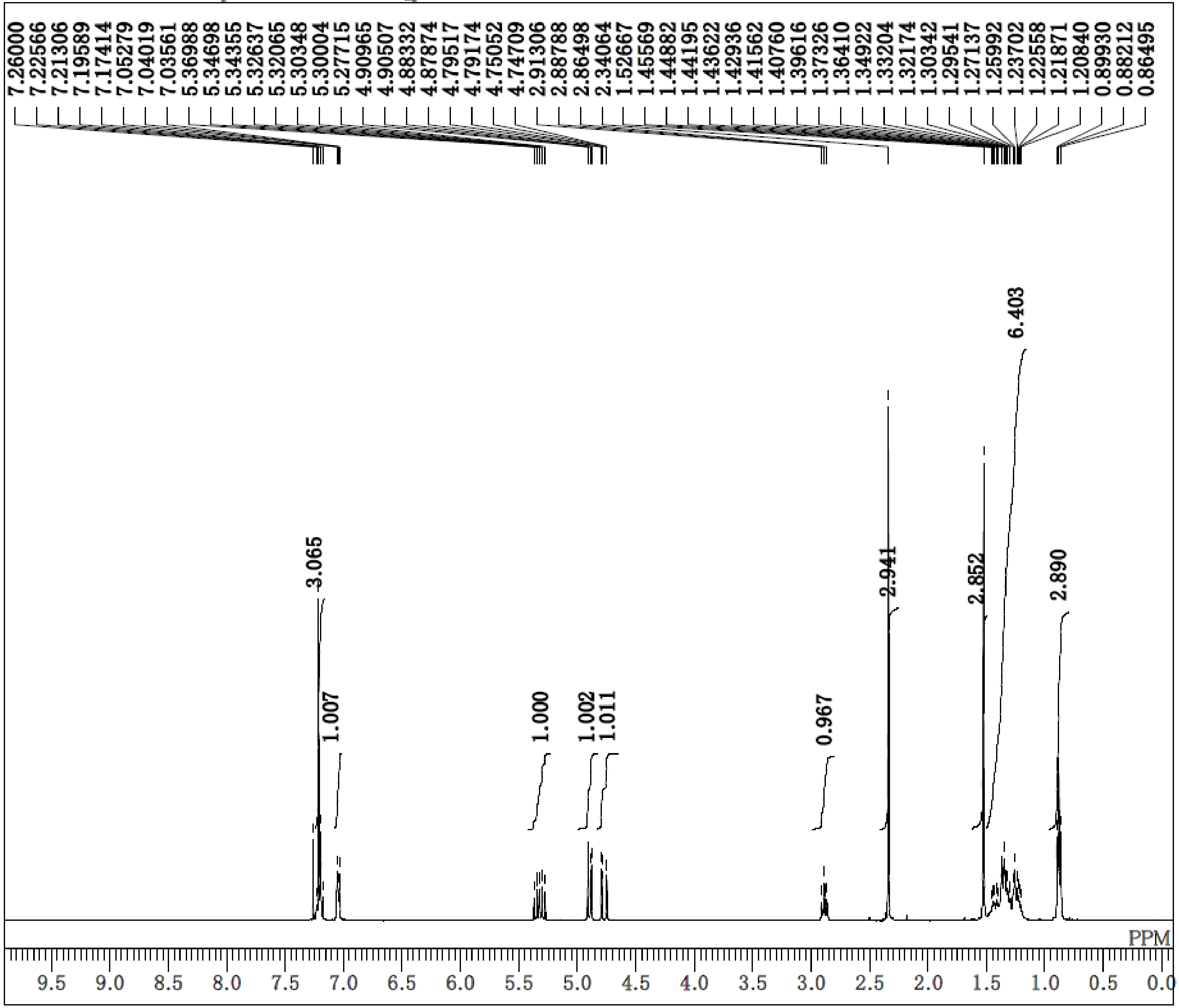

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2h'_proton.als
DFILE 2h'_proton.als
COMNT
DATIM 12-11-2019 20:39:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 44
PPM


O
OH
CN

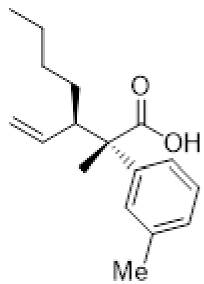
(2*R*,3*R*)-**2h**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2h'_carbon.als
180.39714
146.22391
137.23949
131.86982
128.23600
119.22296
118.59348
111.01109
77.32428
77.00000
76.67572
54.43403
50.70483
29.82702
28.42499
22.29232
17.32323
13.88969
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2h'_carbon.als
COMNT
DATIM 12-11-2019 22:02:39
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 104
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2i_proton.als

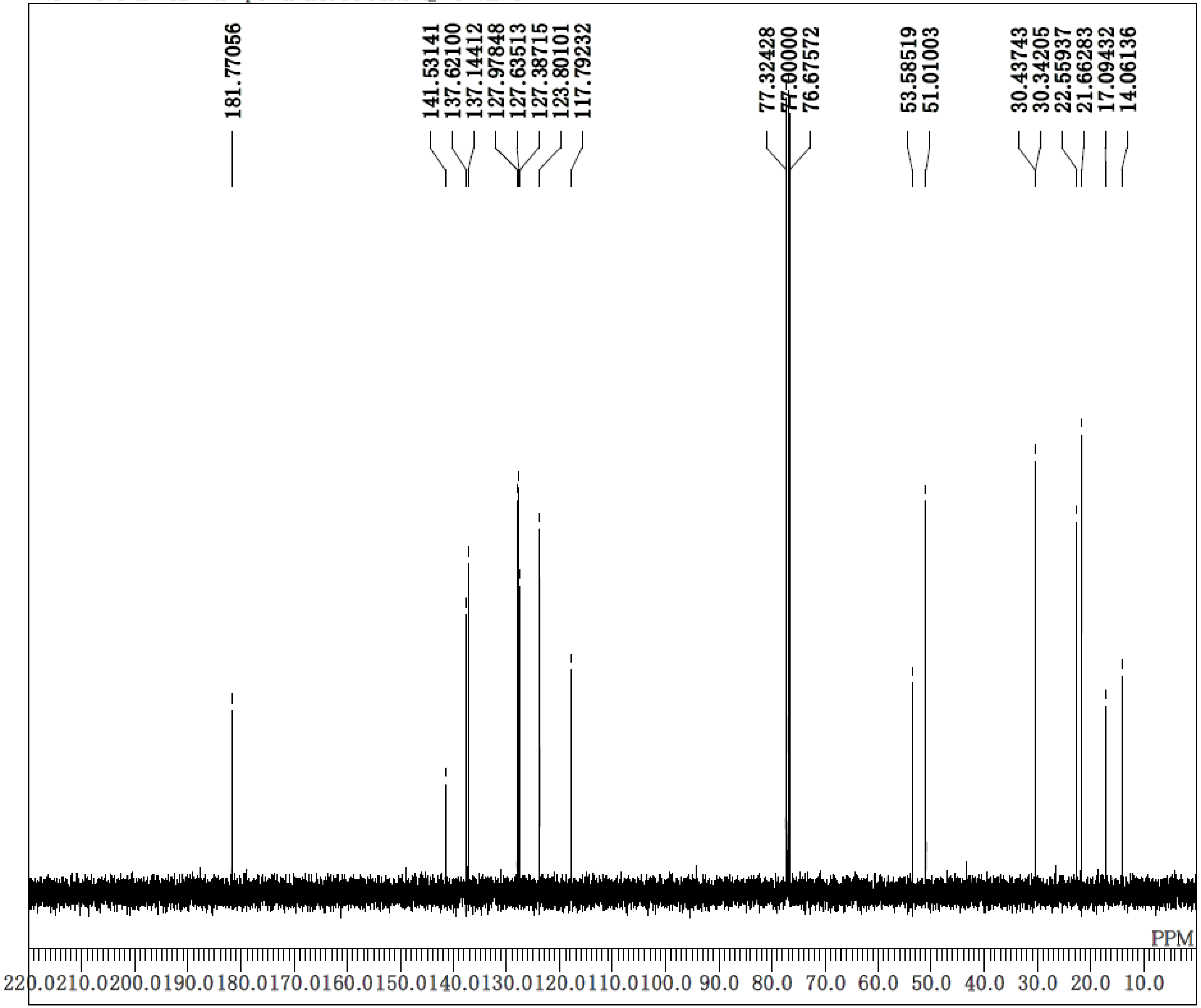


DFILE	2i_proton.als
COMNT	
DATIM	16-10-2019 14:19:30
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32



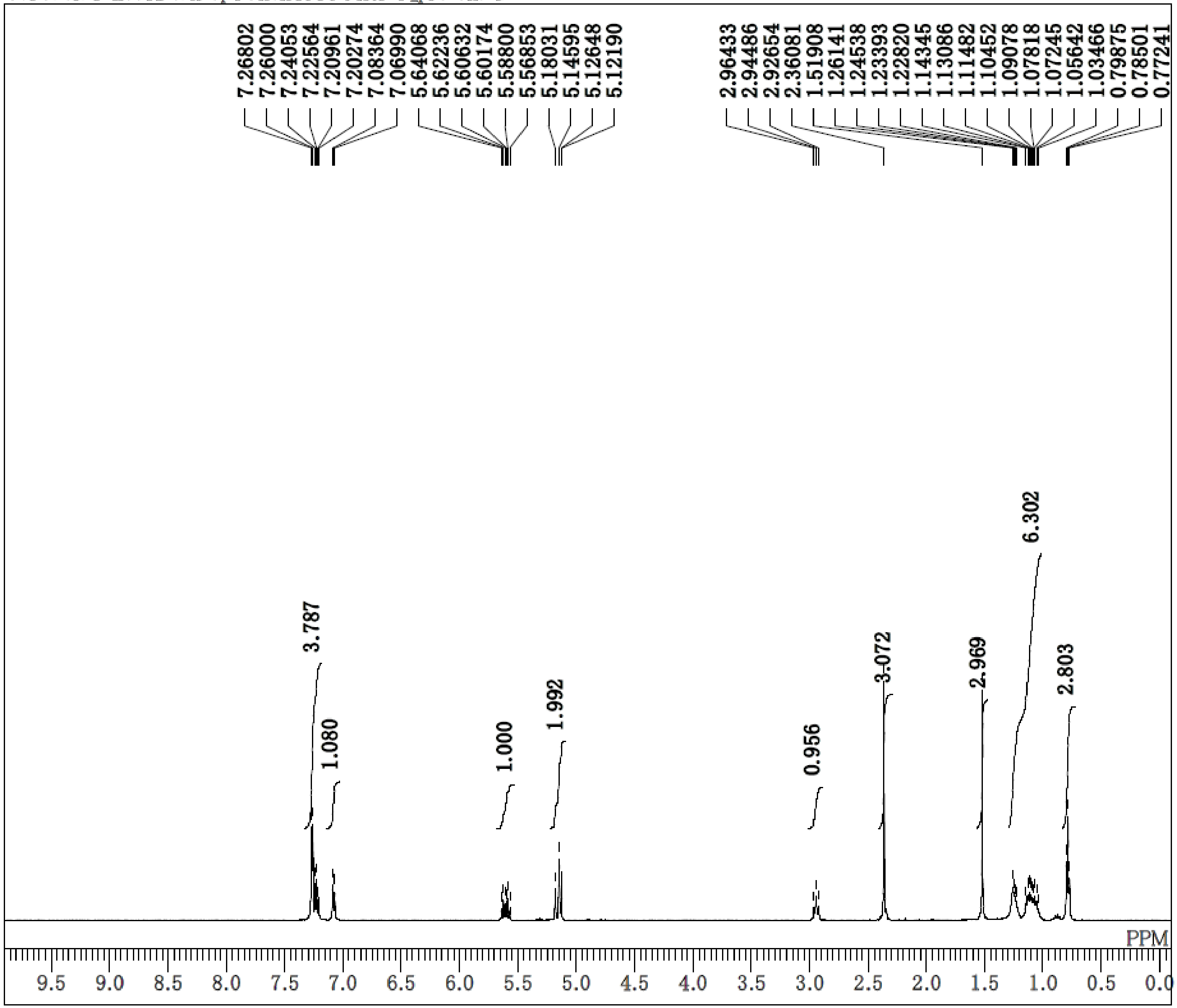
(2*S*,3*R*)-**2i**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2i_carbon.als

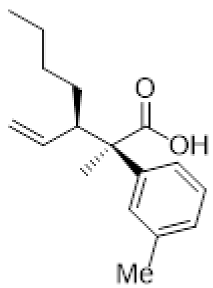


DFILE	2i_carbon.als
COMNT	
DATIM	16-10-2019 14:43:37
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	106
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2i'_proton.als

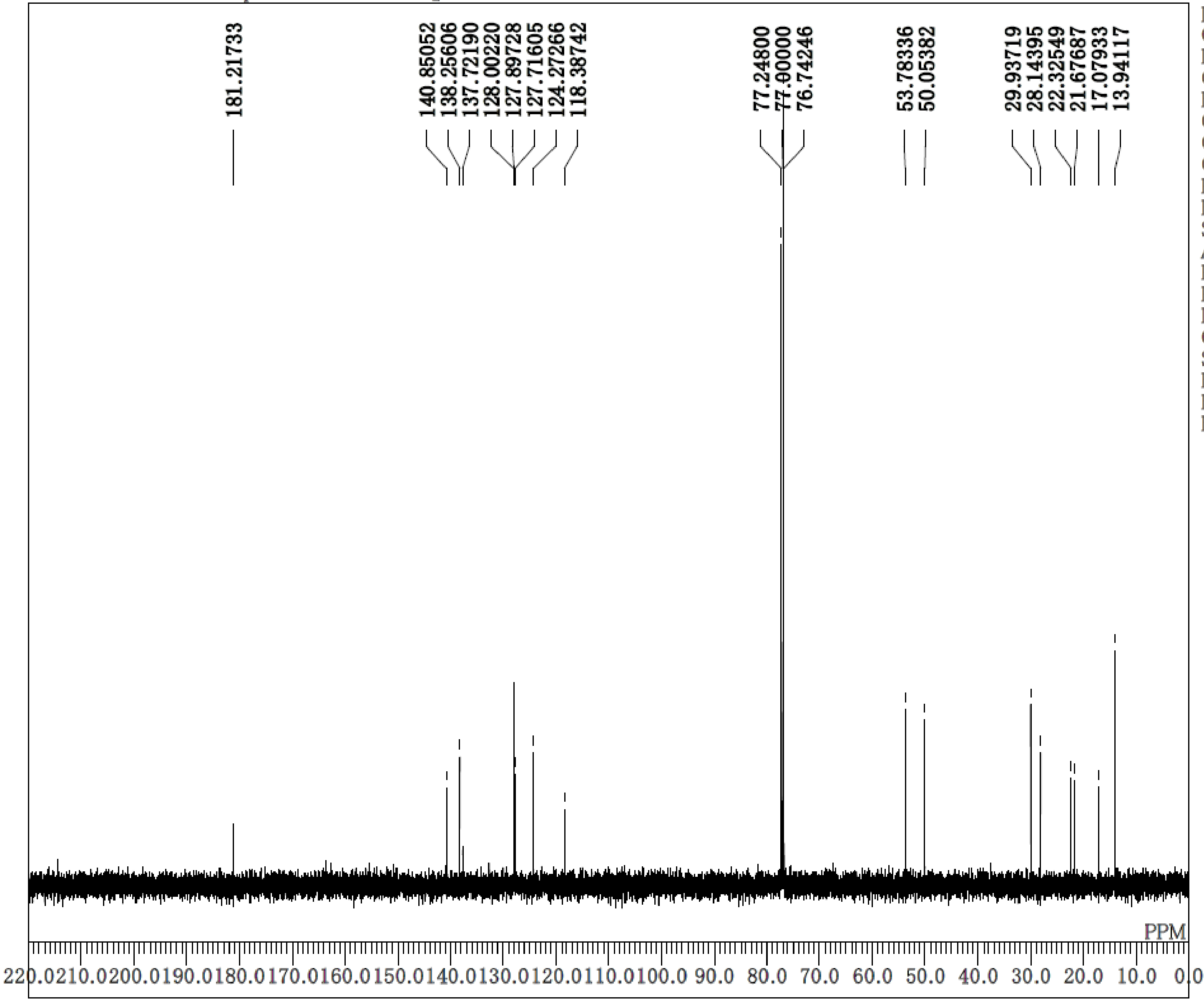


DFILE	2i'_proton.als
COMNT	
DATIM	2019-11-06 12:16:28
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.10 Hz
RGAIN	42



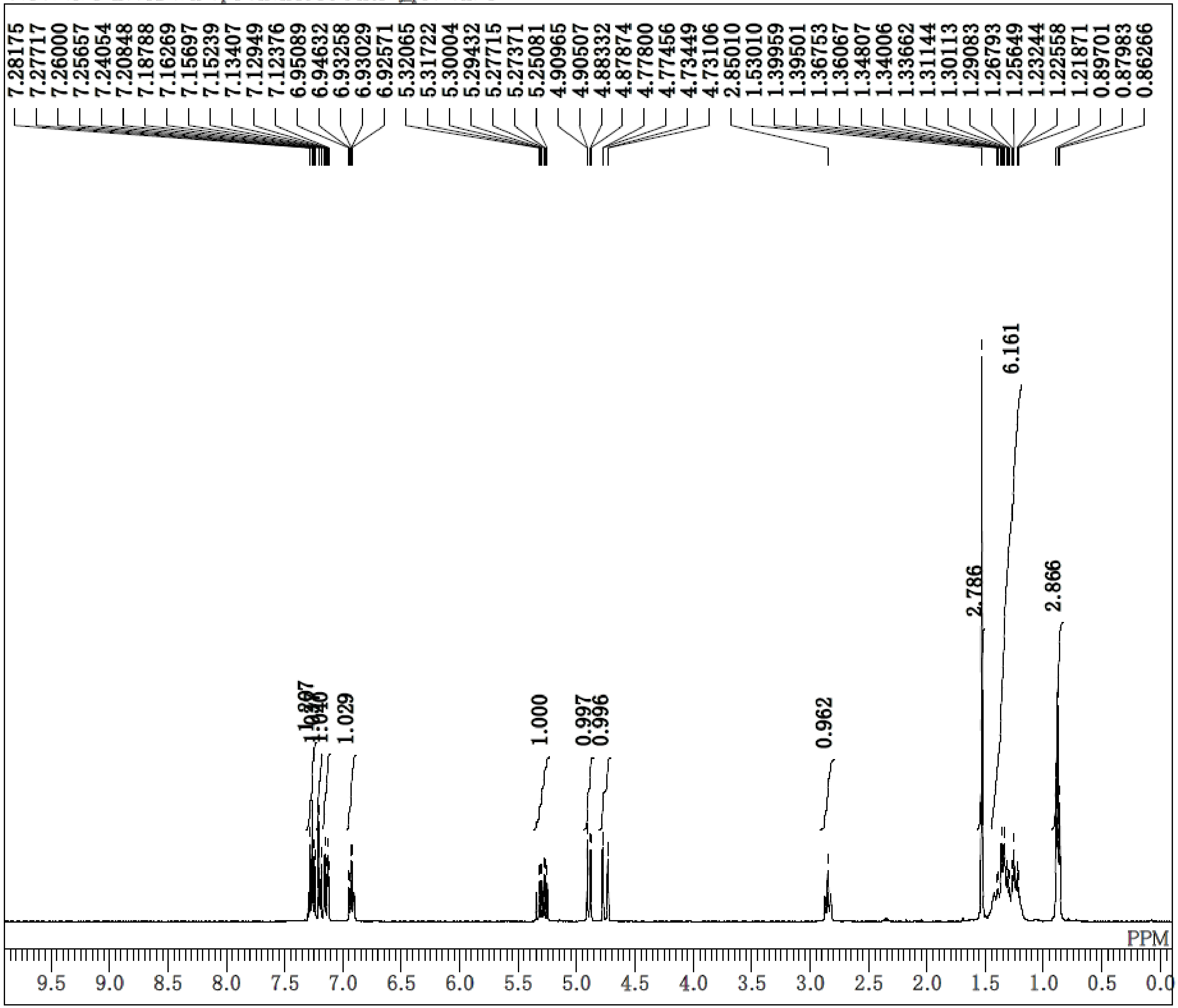
(2*R*,3*R*)-**2l**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2i'_carbon.als

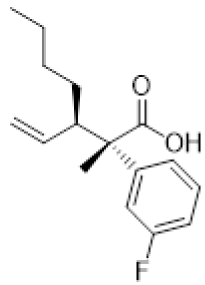


DFILE	2i'_carbon.als
COMNT	
DATIM	2019-11-06 12:37:28
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	102
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.10 Hz
RGAIN	60

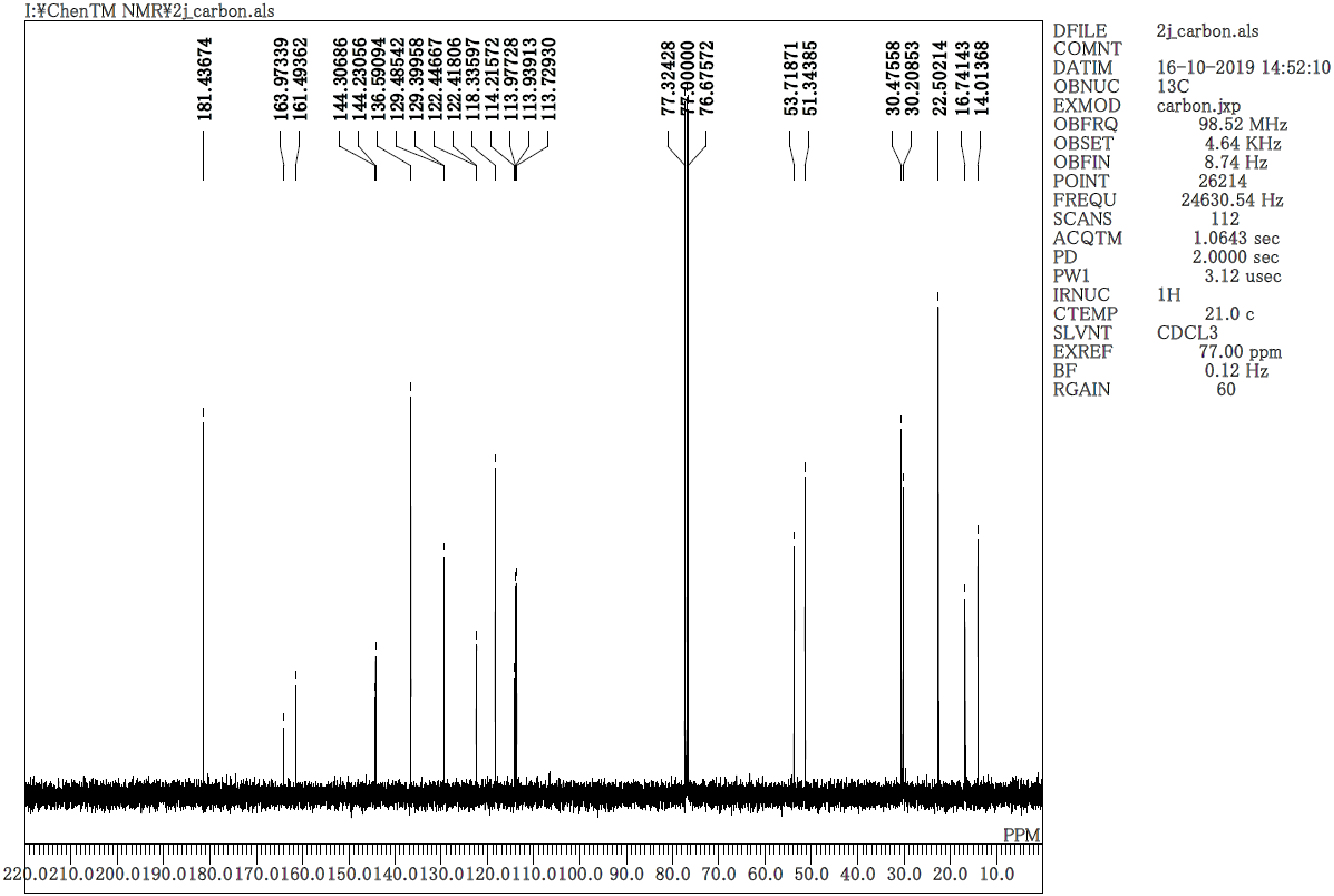
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2j_proton.als



DFILE	2j_proton.als
COMNT	
DATIM	16-10-2019 14:24:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30



(2*S*,3*R*)-**2j**


I:¥ChenTM NMR¥2j_carbon.als
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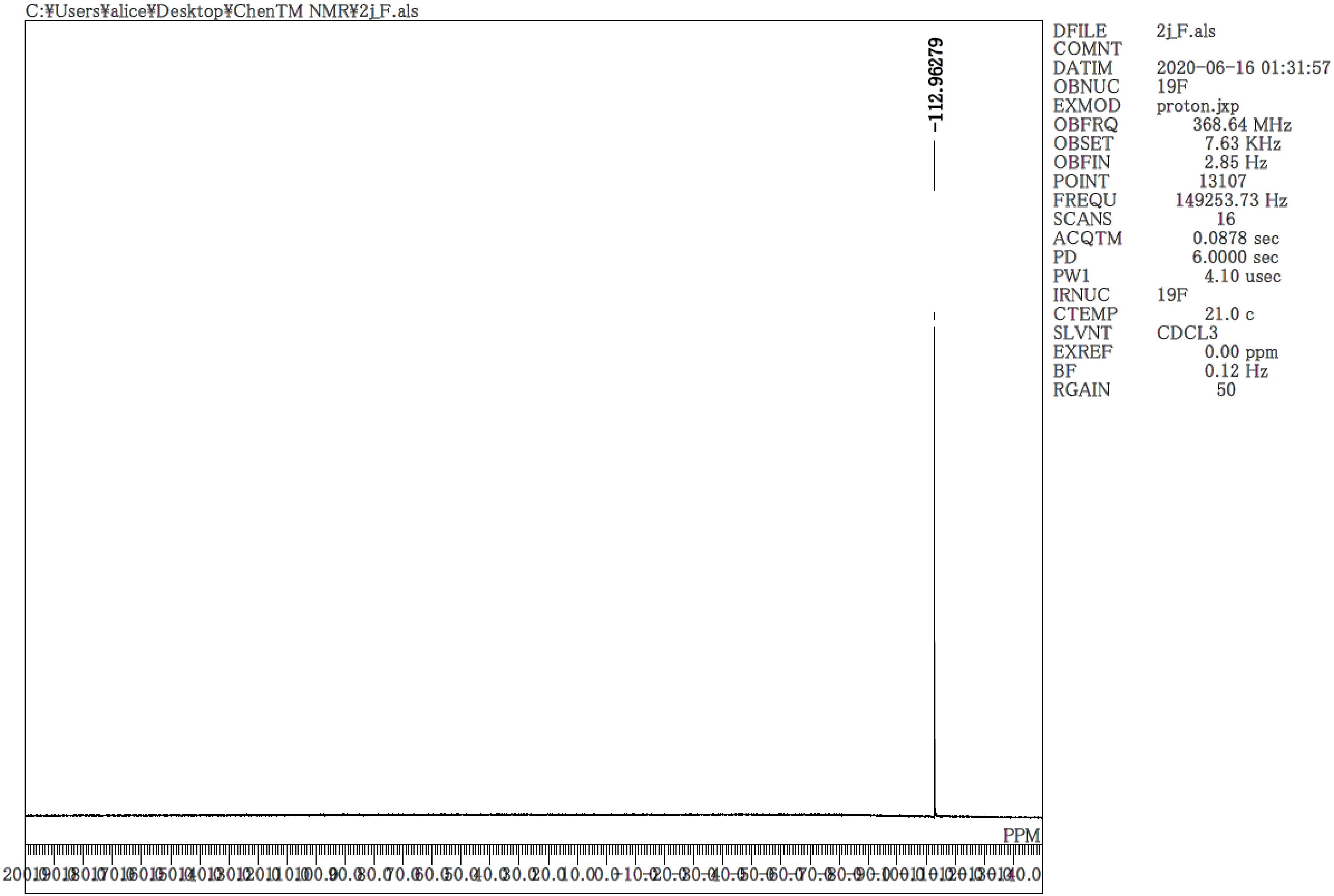
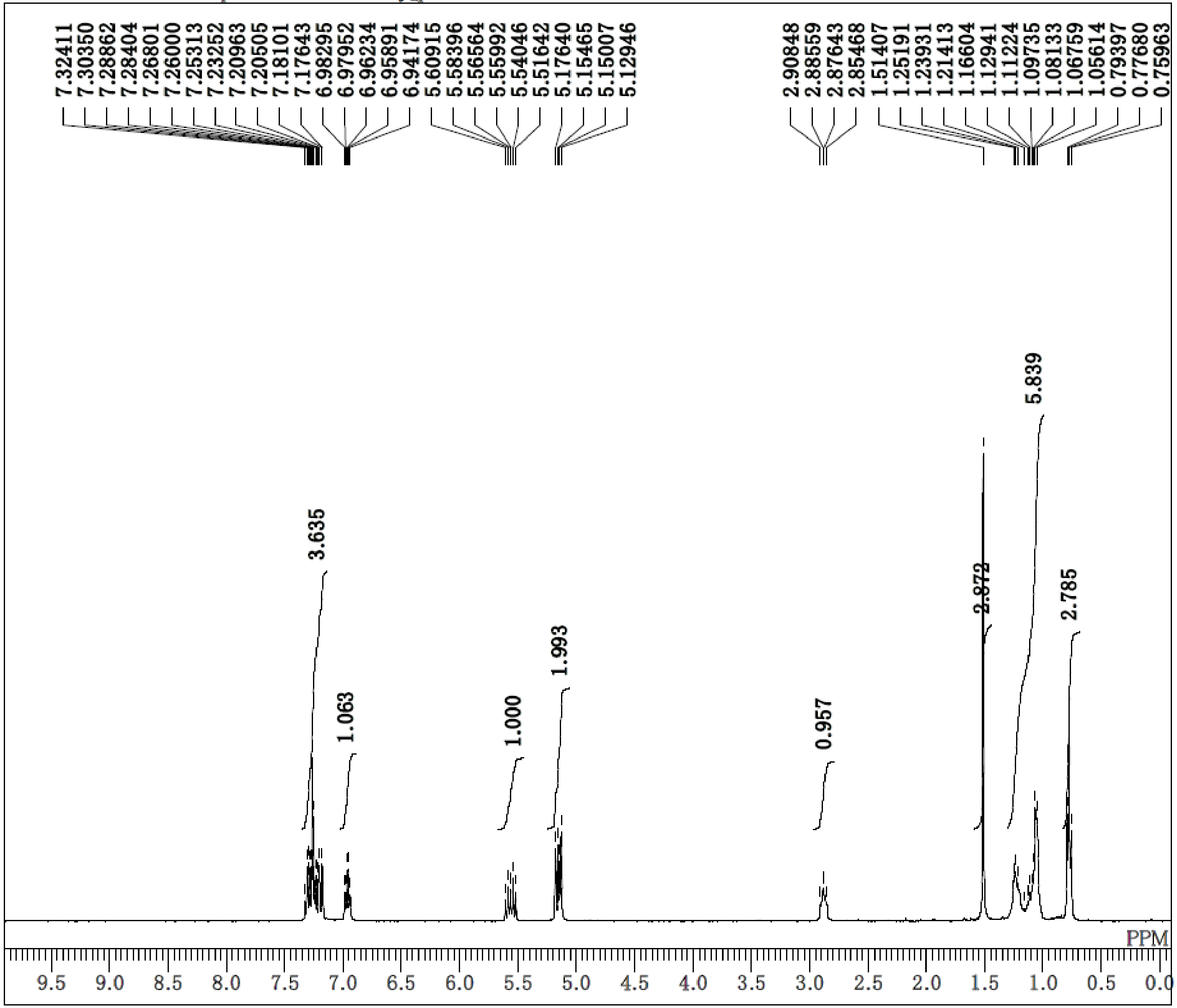
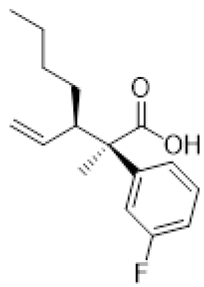
144.30686
144.23056
136.59094
129.48542
129.39958
122.44667
122.41806
118.33597
114.21572
113.97728
113.93913
113.72930
77.32428
77.00000
76.67572
53.71871
51.34385
30.47558
30.20853
22.50214
16.74143
14.01368
DFILE 2j_carbon.als
COMNT
DATIM 16-10-2019 14:52:10
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 112
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2j_F.als
-112.96279
PPM
DFILE 2j_F.als
COMNT
DATIM 2020-06-16 01:31:57
OBNUC 19F
EXMOD proton.jxp
OBFRQ 368.64 MHz
OBSET 7.63 KHz
OBFIN 2.85 Hz
POINT 13107
FREQU 149253.73 Hz
SCANS 16
ACQTM 0.0878 sec
PD 6.0000 sec
PW1 4.10 usec
IRNUC 19F
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2j'_proton.als

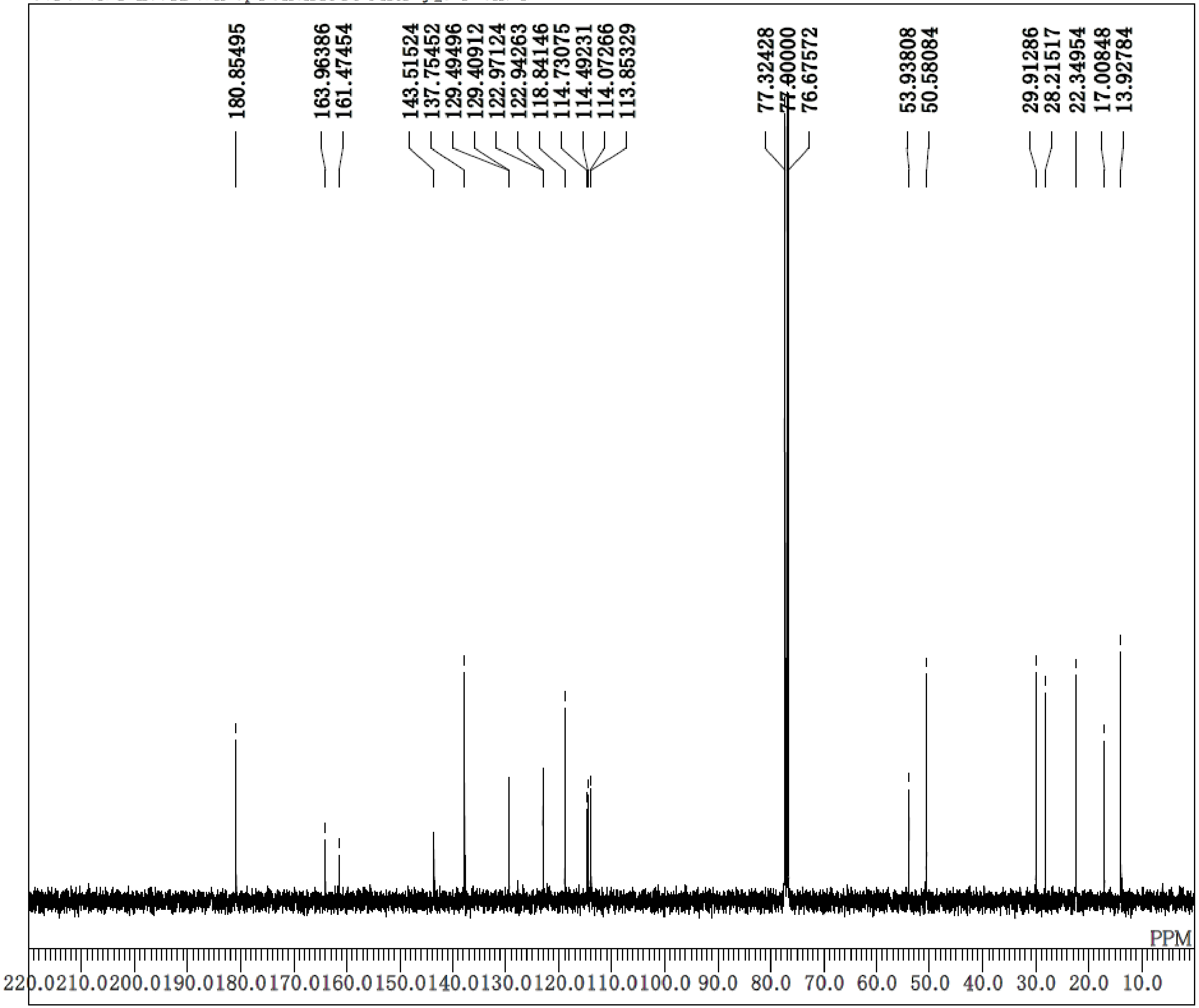


DFILE	2j'_proton.als
COMNT	
DATIM	12-11-2019 20:32:28
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	42



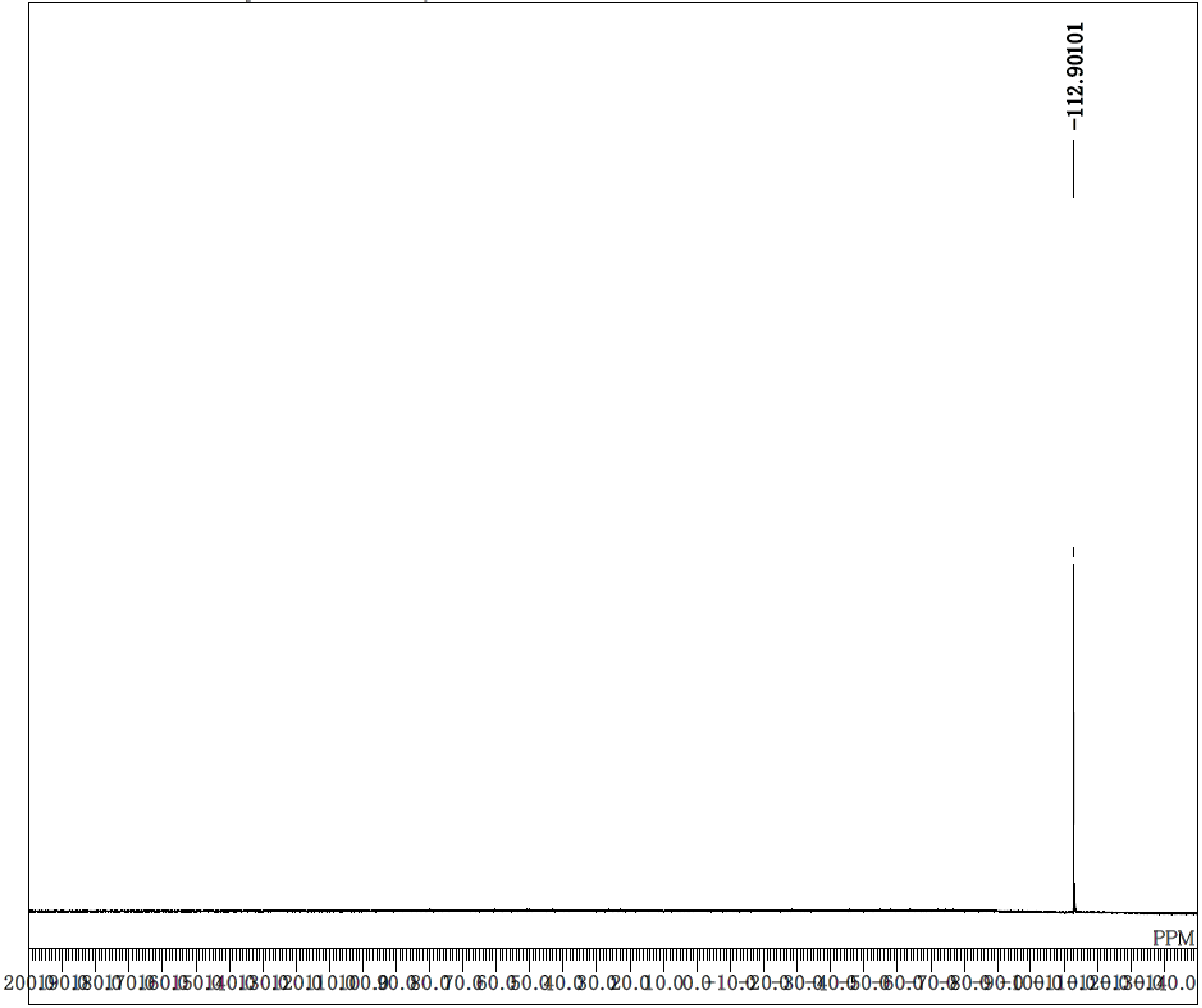
(2*R*,3*R*)-**2j**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2j'_carbon.als



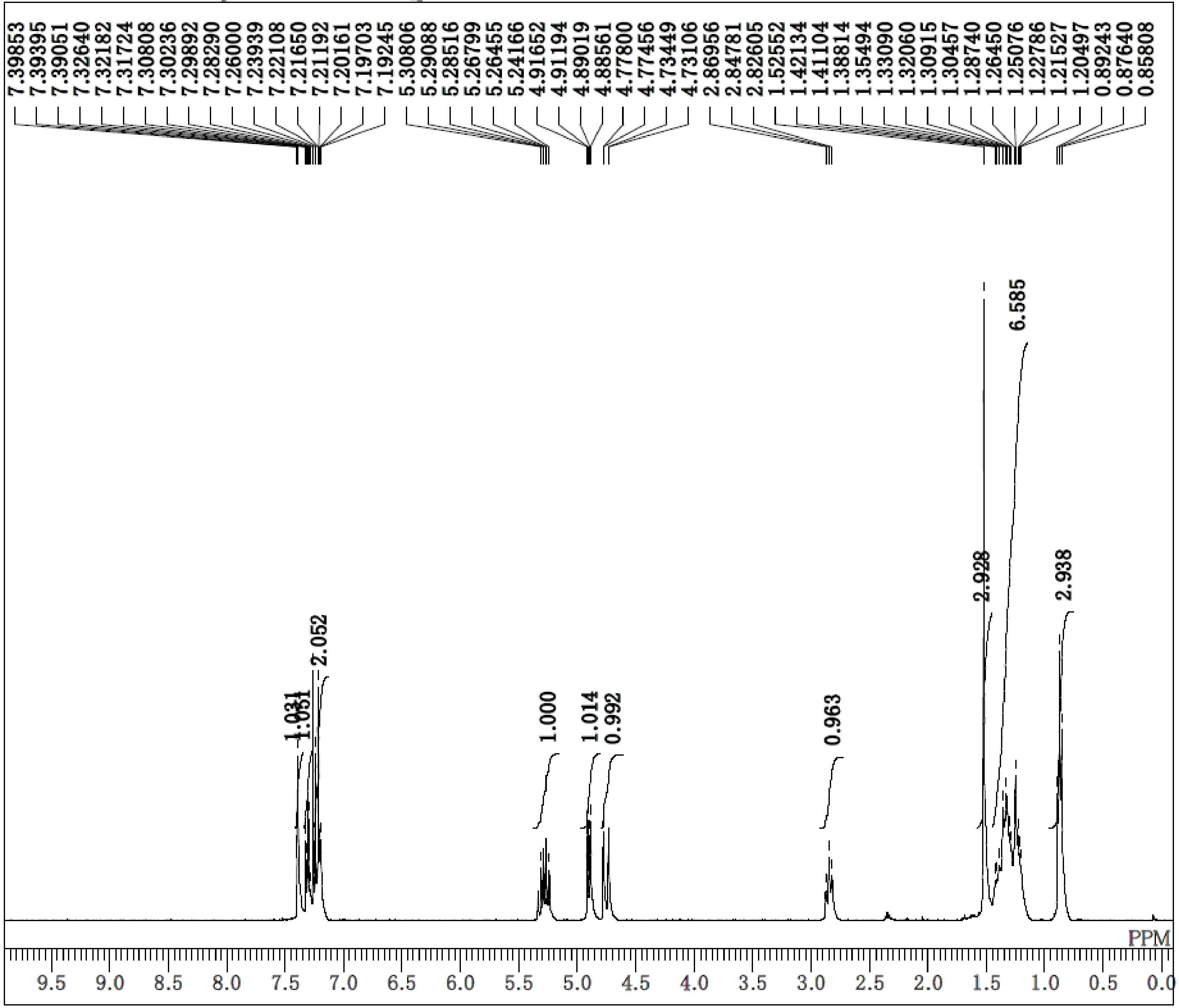
DFILE	2j'_carbon.als
COMNT	
DATIM	12-11-2019 21:04:26
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	121
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2j'_F.als

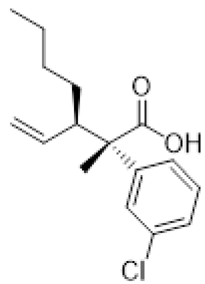


DFILE	2j'_F.als
COMNT	
DATIM	16-06-2020 01:53:47
OBNUC	19F
EXMOD	proton.jxp
OBFRQ	368.64 MHz
OBSET	7.63 KHz
OBFIN	2.85 Hz
POINT	13107
FREQU	149253.73 Hz
SCANS	16
ACQTM	0.0878 sec
PD	6.0000 sec
PW1	4.10 usec
IRNUC	19F
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50

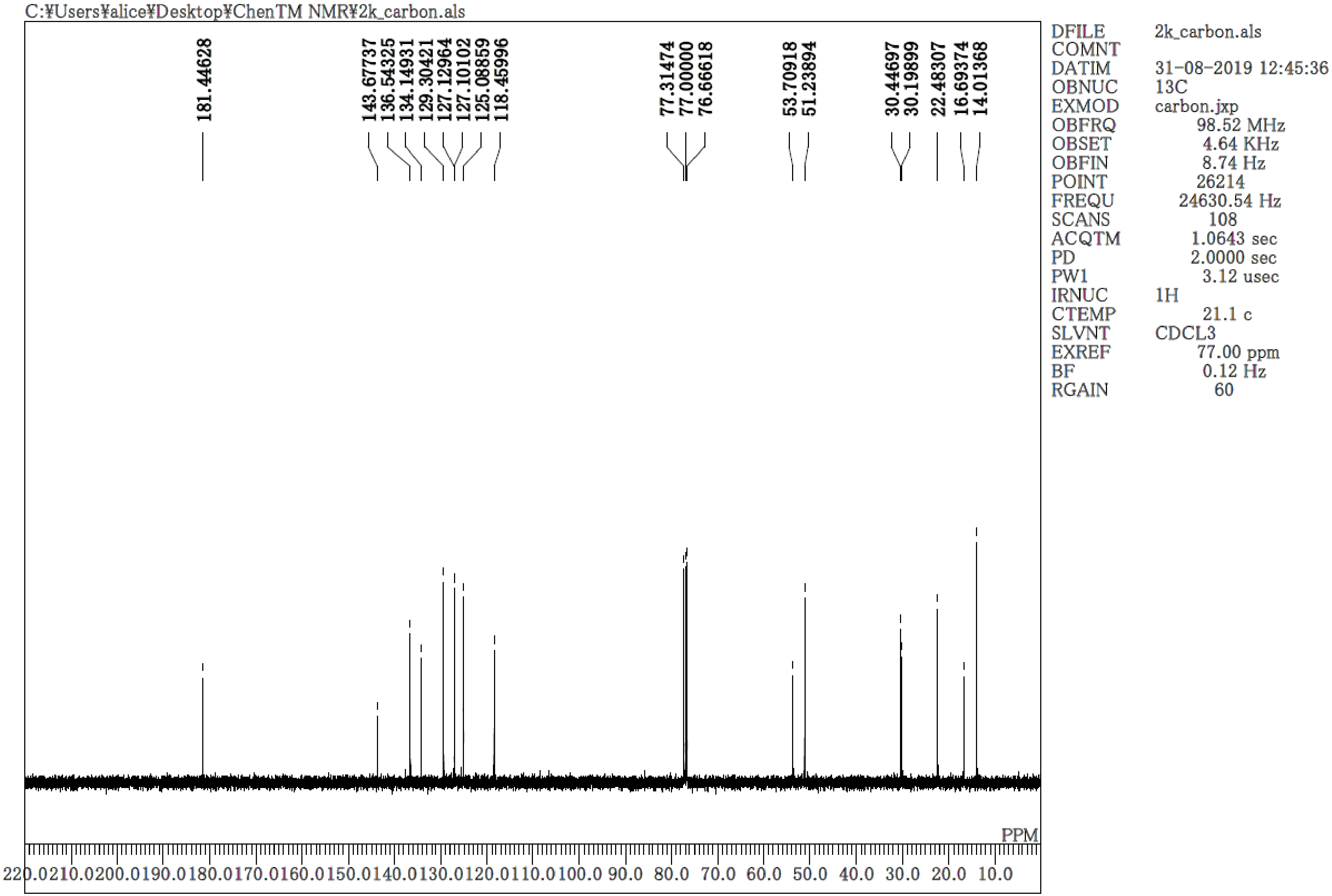
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2k_proton.als



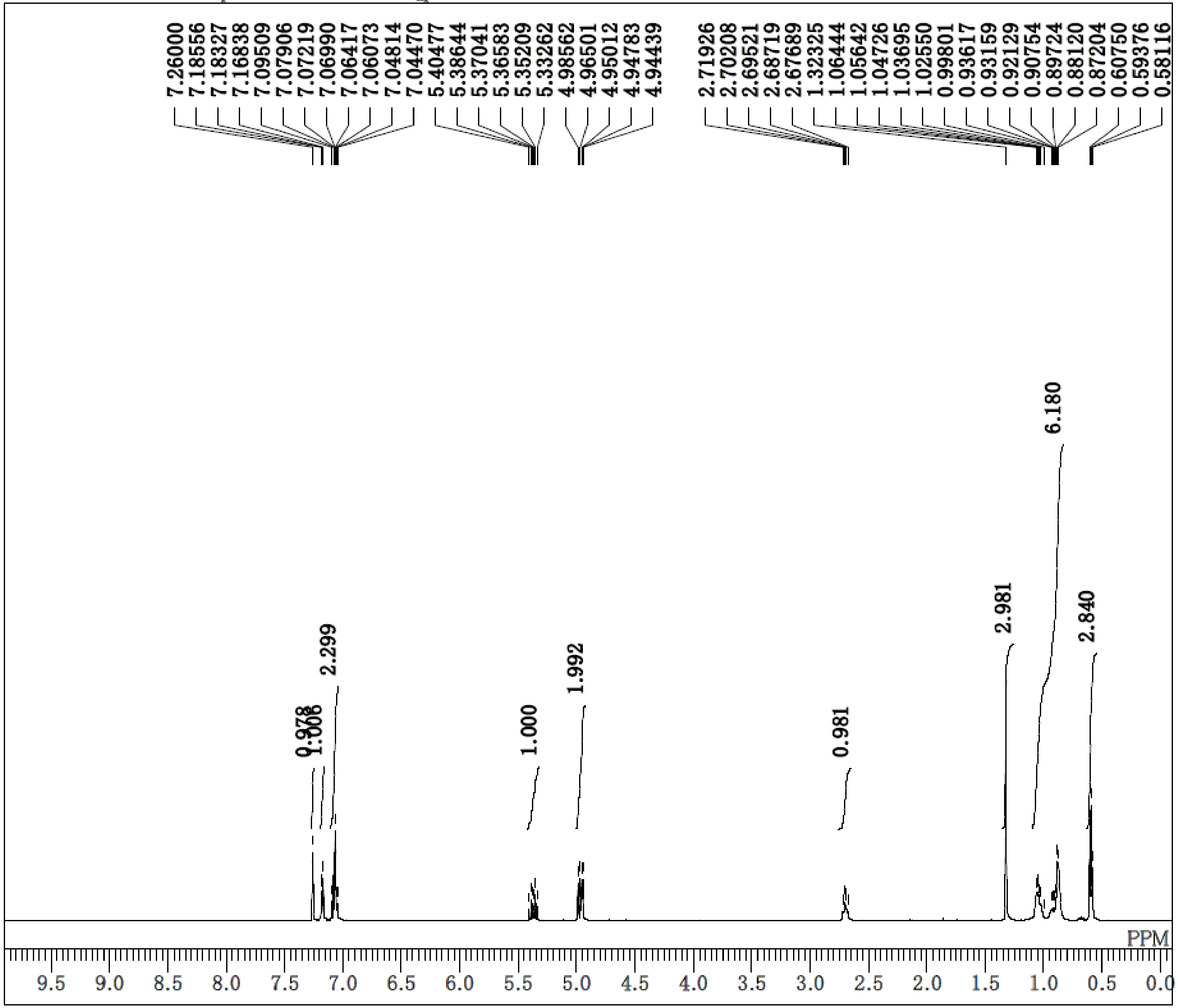
DFILE	2k_proton.als
COMNT	
DATIM	17-03-2020 02:15:58
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



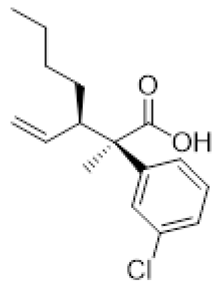
(2*S*,3*R*)-**2k**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2k_carbon.als
181.44628
143.67737
136.54325
134.14931
129.30421
127.12964
127.10102
125.08859
118.45996
77.31474
77.00000
76.66618
53.70918
51.23894
30.44697
30.19899
22.48307
16.69374
14.01368
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2k_carbon.als
COMNT
DATIM 31-08-2019 12:45:36
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 108
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

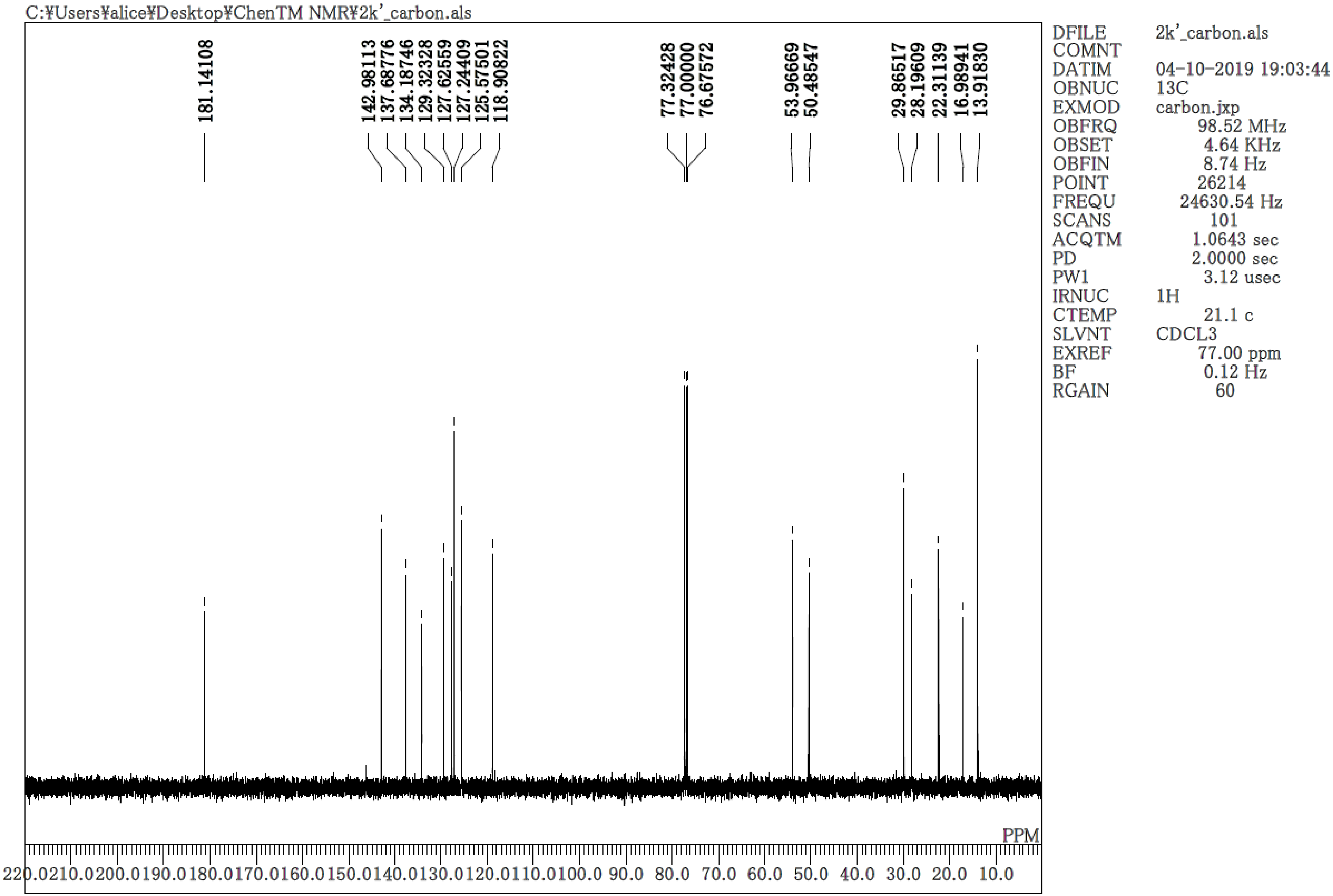
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2k'_proton.als



DFILE	2k'_proton.als
COMNT	
DATIM	2019-10-04 15:44:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



(2*R*,3*R*)-**2k**



C:¥Users¥alice¥Desktop¥ChenTM NMR¥2k'_carbon.als
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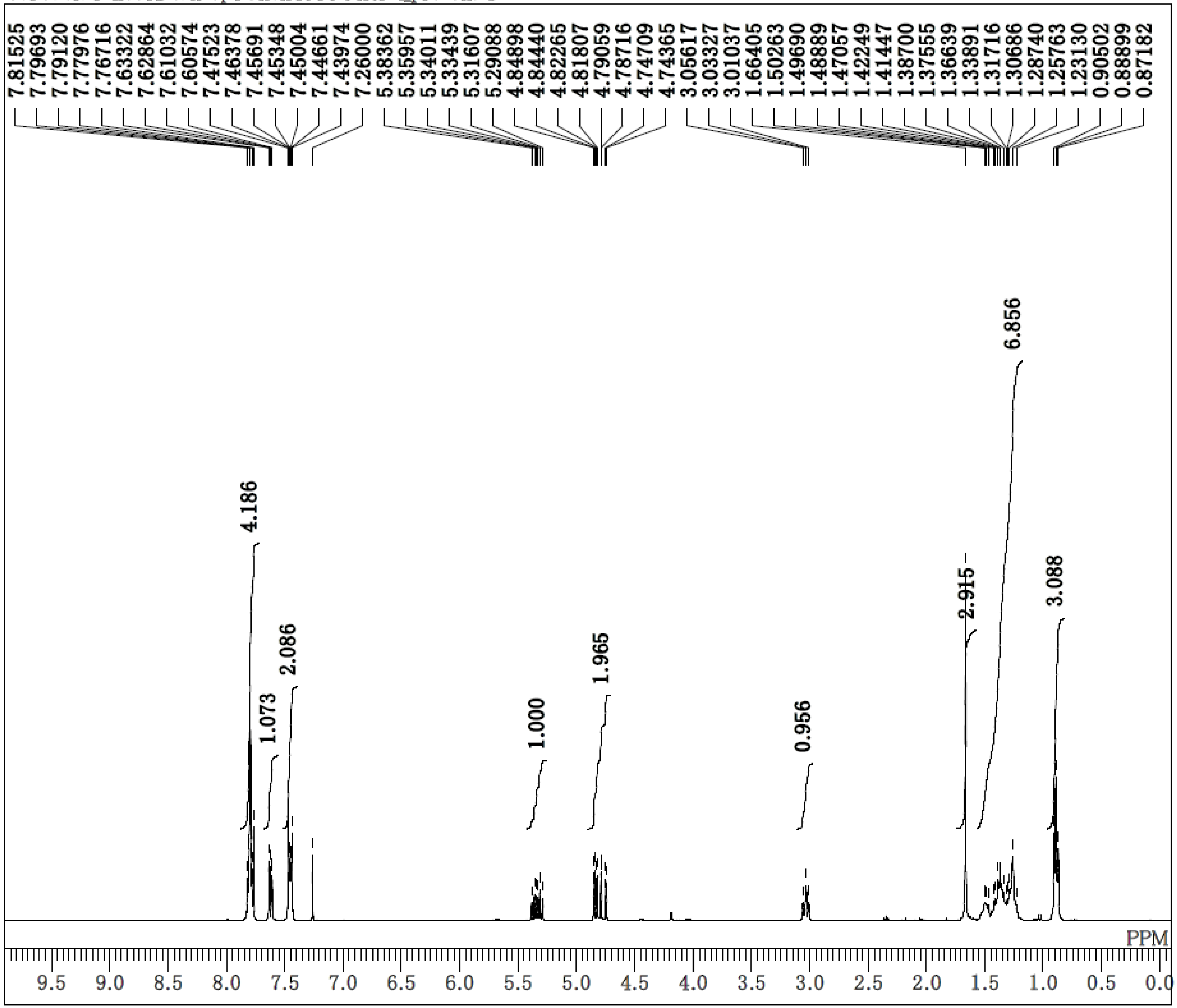
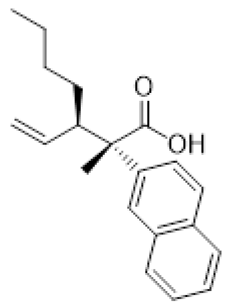
134.18746
129.32328
127.62559
127.24409
125.57501
118.90822
77.32428
77.00000
76.67572
53.96669
50.48547
29.86517
28.19609
22.31139
16.98941
13.91830
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2k'_carbon.als
COMNT
DATIM 04-10-2019 19:03:44
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 101
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

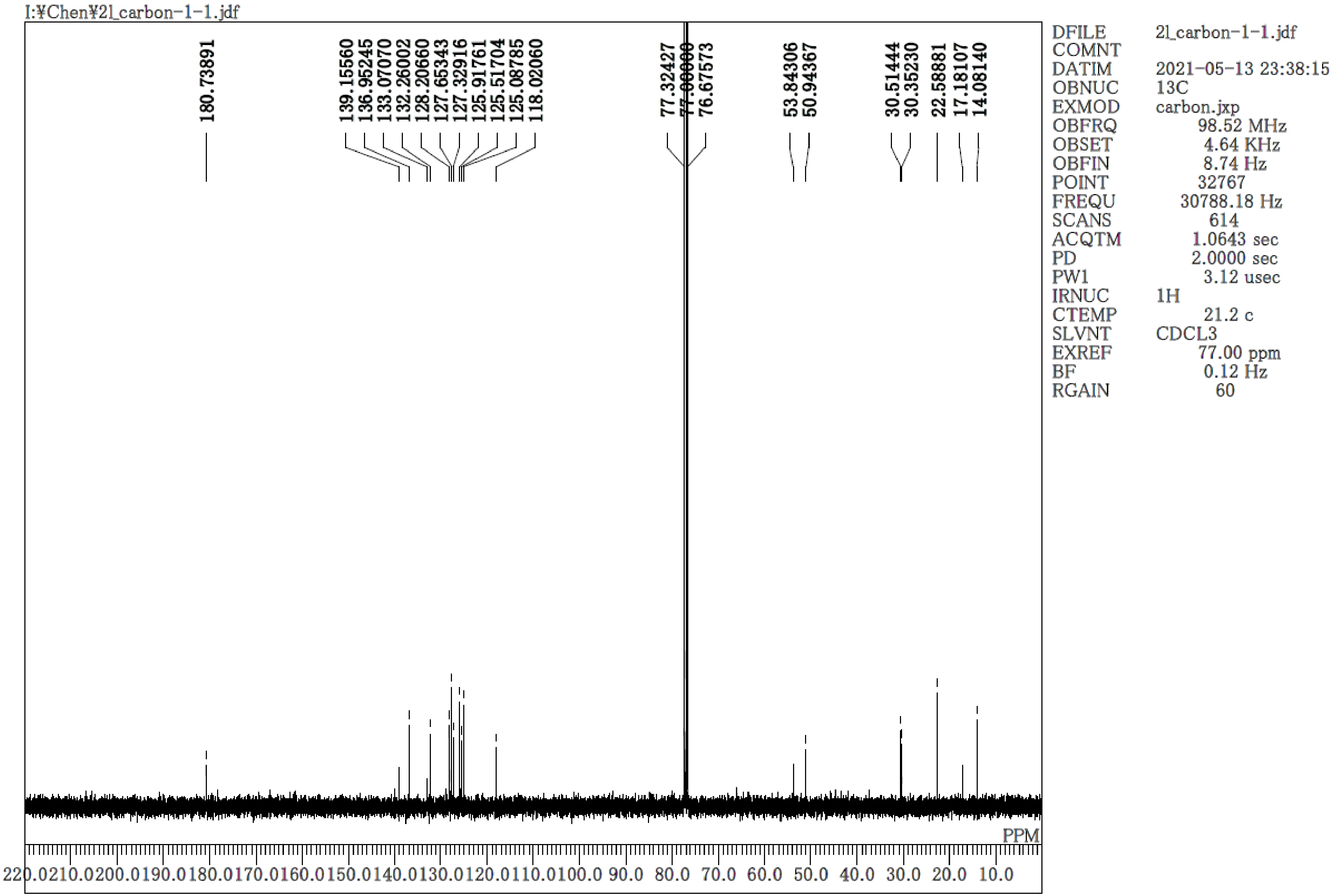
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2l_proton.als



DFILE	2l_proton.als
COMNT	
DATIM	06-03-2020 21:20:56
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



(2*S*,3*R*)-**2l**


I:¥Chen¥2l_carbon-1-1.jdf
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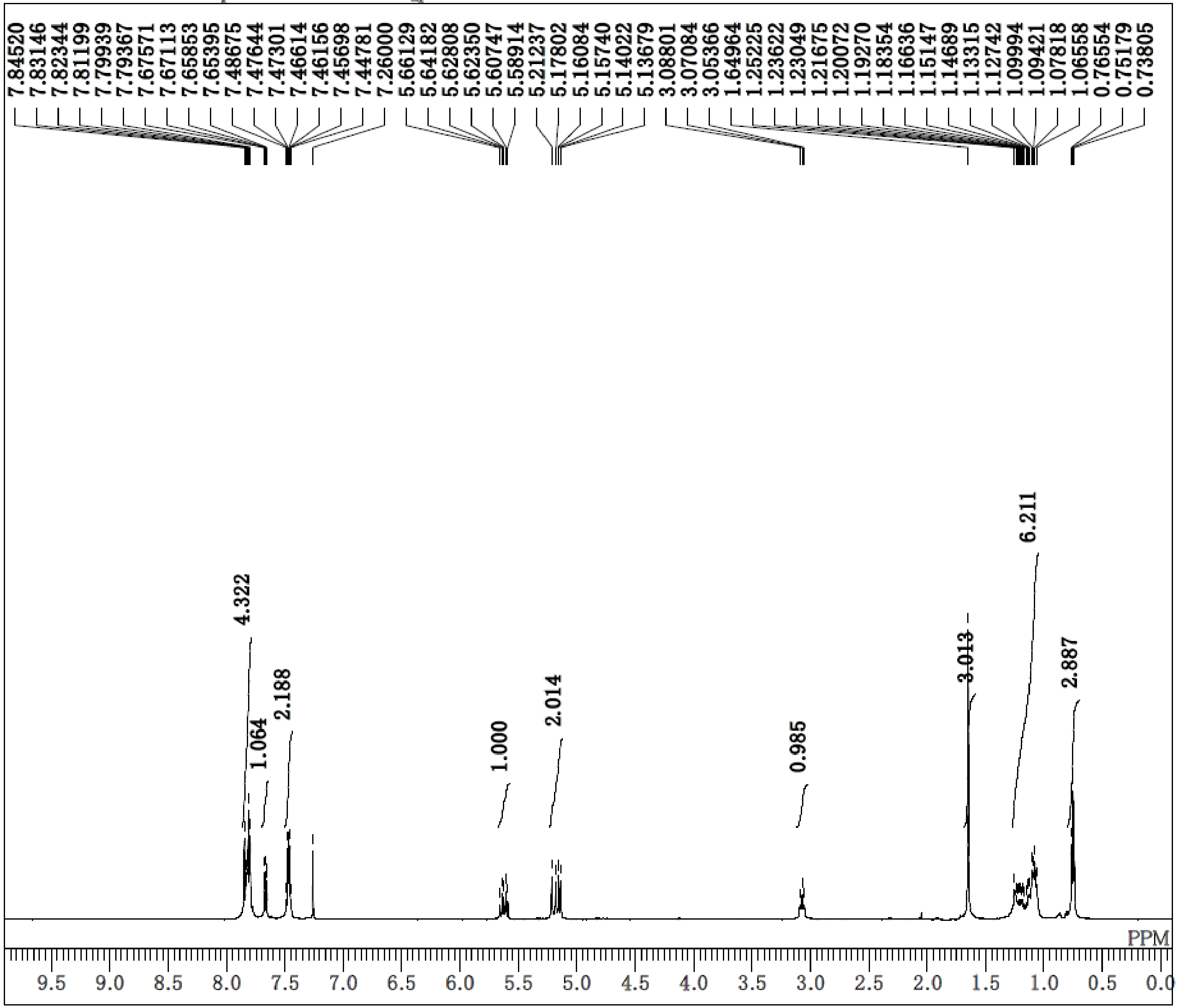
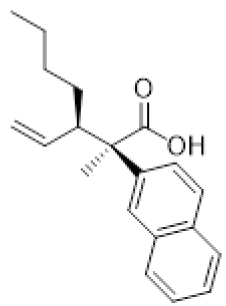
136.95245
133.07070
132.26002
128.20660
127.65343
127.32916
125.91761
125.51704
125.08785
118.02060
77.32427
77.00000
76.67573
53.84306
50.94367
30.51444
30.35230
22.58881
17.18107
14.08140
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2l_carbon-1-1.jdf
COMNT
DATIM 2021-05-13 23:38:15
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 32767
FREQU 30788.18 Hz
SCANS 614
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

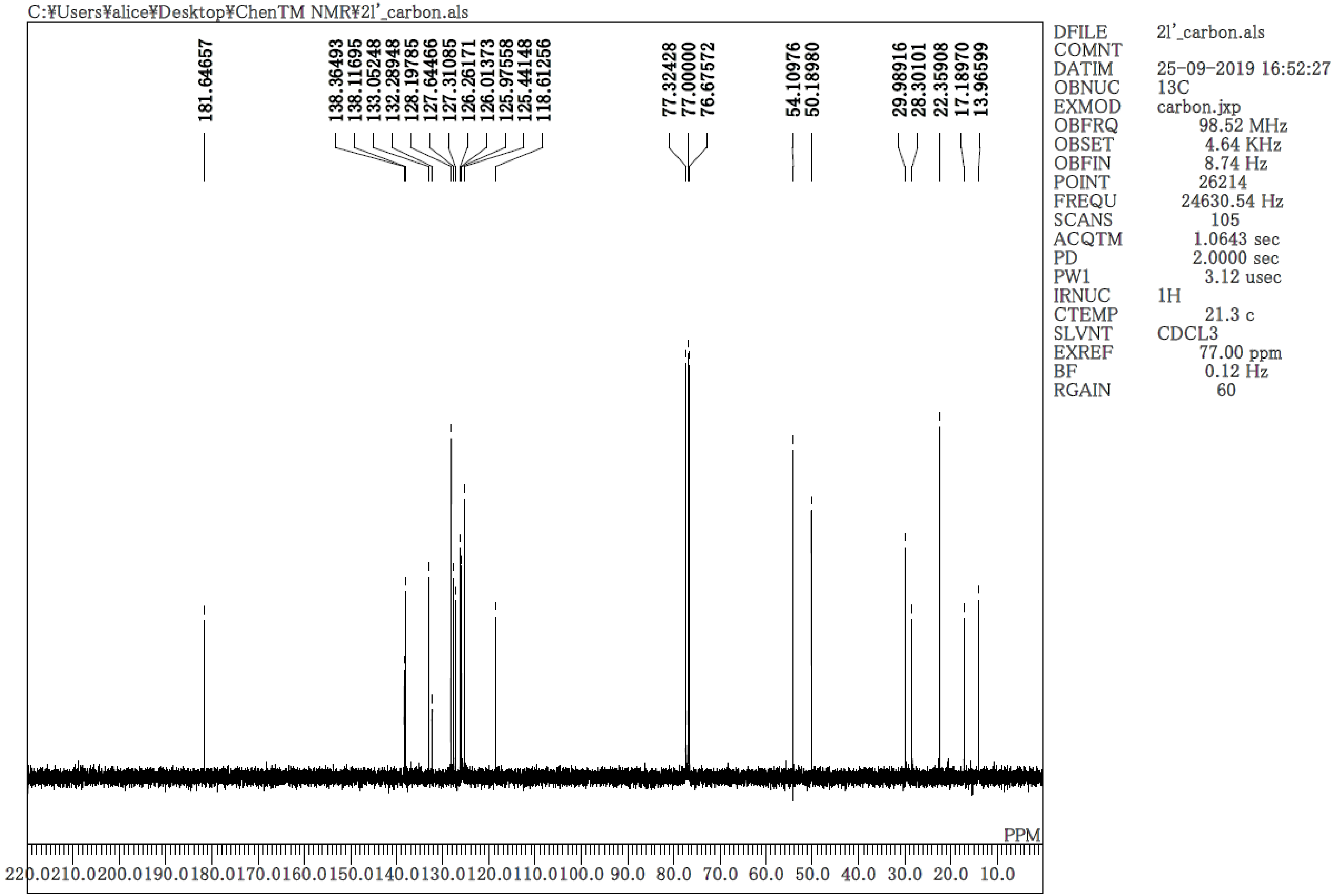
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2l'_proton.als



DFILE	2l'_proton.als
COMNT	
DATIM	2019-09-25 14:47:45
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



(2*R*,3*R*)-**2l**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2l'_carbon.als
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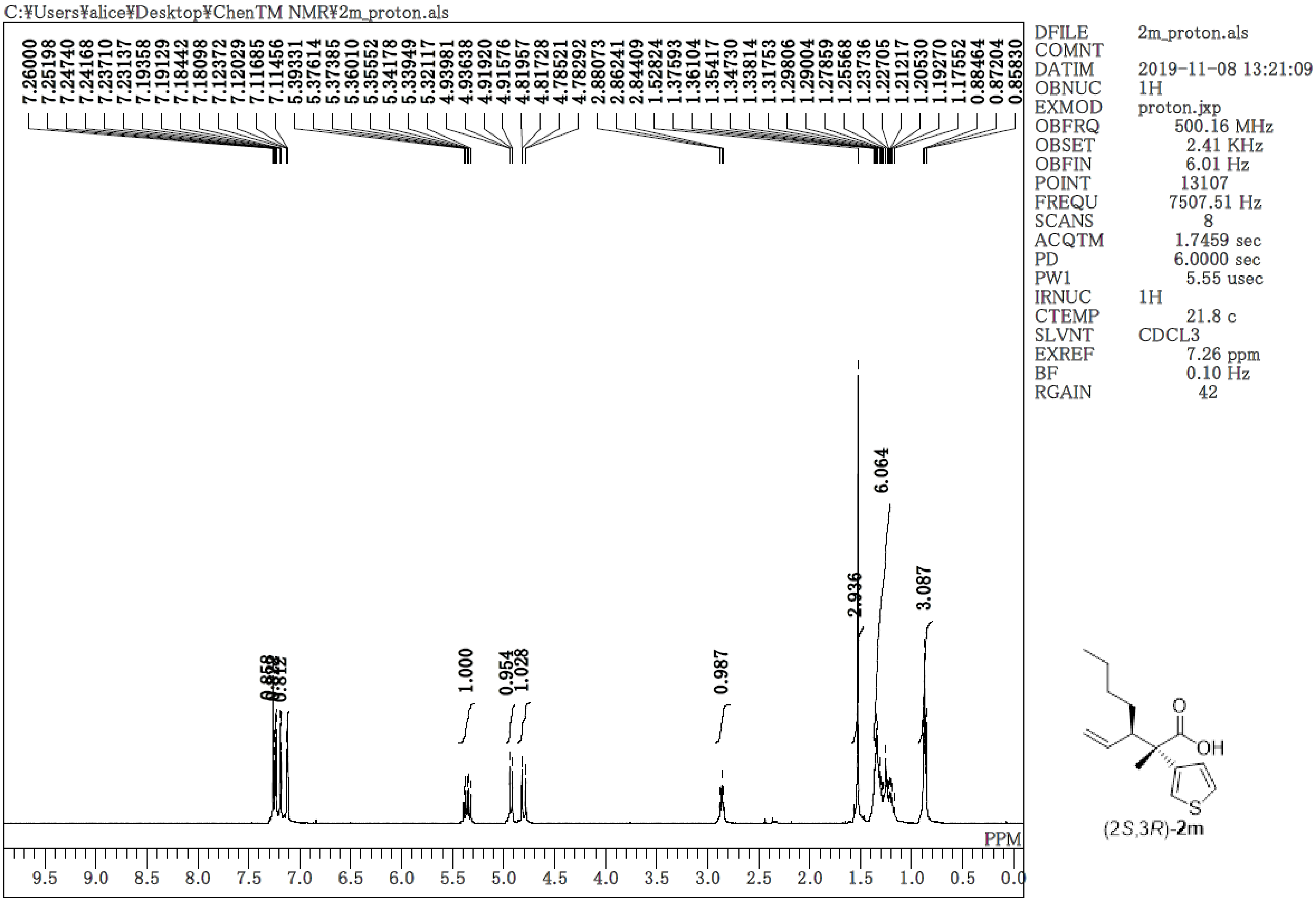
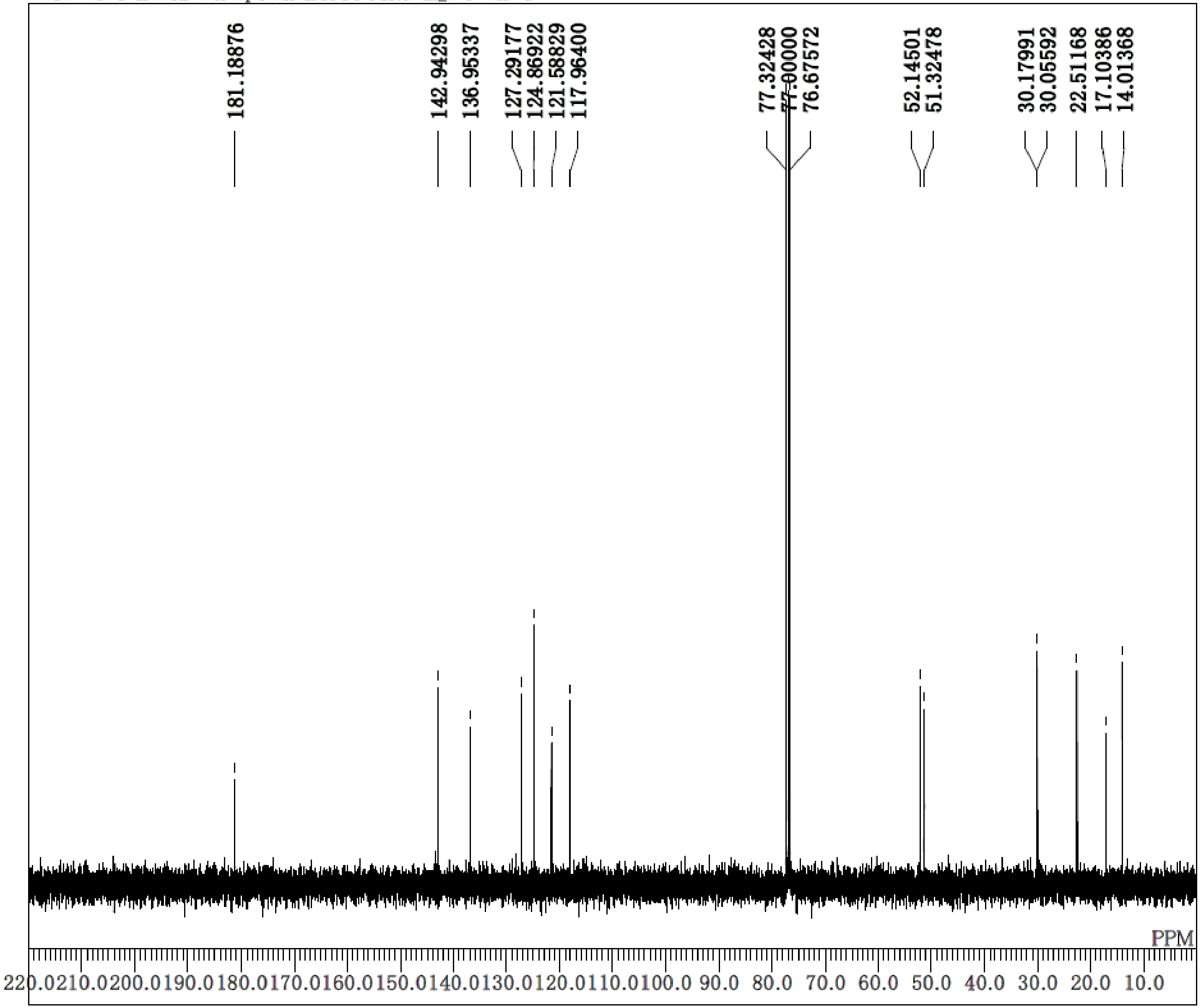
138.11695
133.05248
132.28948
128.19785
127.64466
127.31085
126.26171
126.01373
125.97558
125.44148
118.61256
77.32428
77.00000
76.67572
54.10976
50.18980
29.98916
28.30101
22.35908
17.18970
13.96599
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2l'_carbon.als
COMNT
DATIM 25-09-2019 16:52:27
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 105
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2m_proton.als
DFILE 2m_proton.als
COMNT
DATIM 2019-11-08 13:21:09
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 42
PPM
1.000
0.954
1.028
0.987
2.936
6.064
3.087
OH
O
S

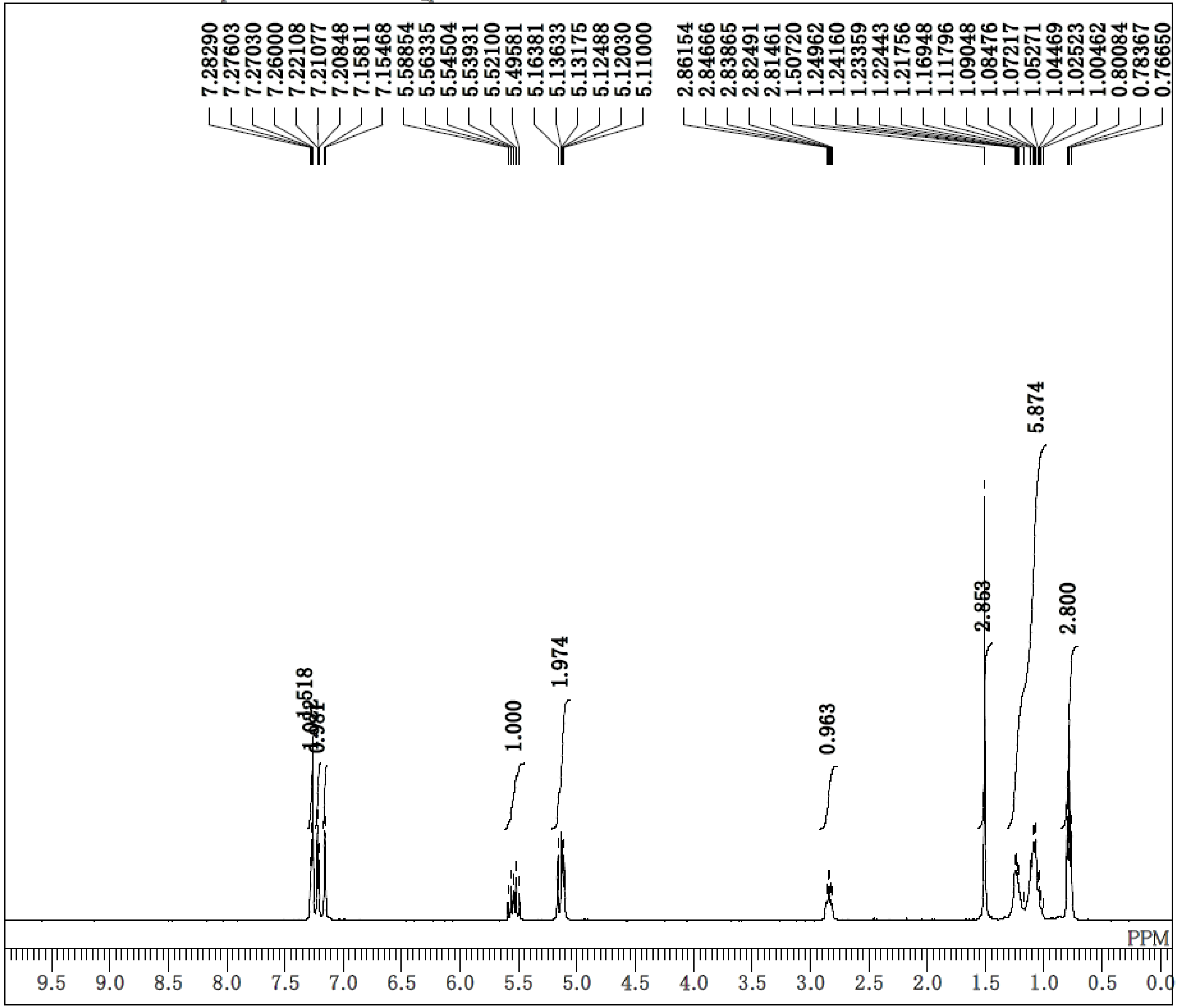
(2*S*,3*R*)-**2m**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2m_carbon.als

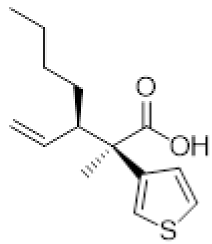


DFILE	2m_carbon.als
COMNT	
DATIM	08-11-2019 14:15:13
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	101
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

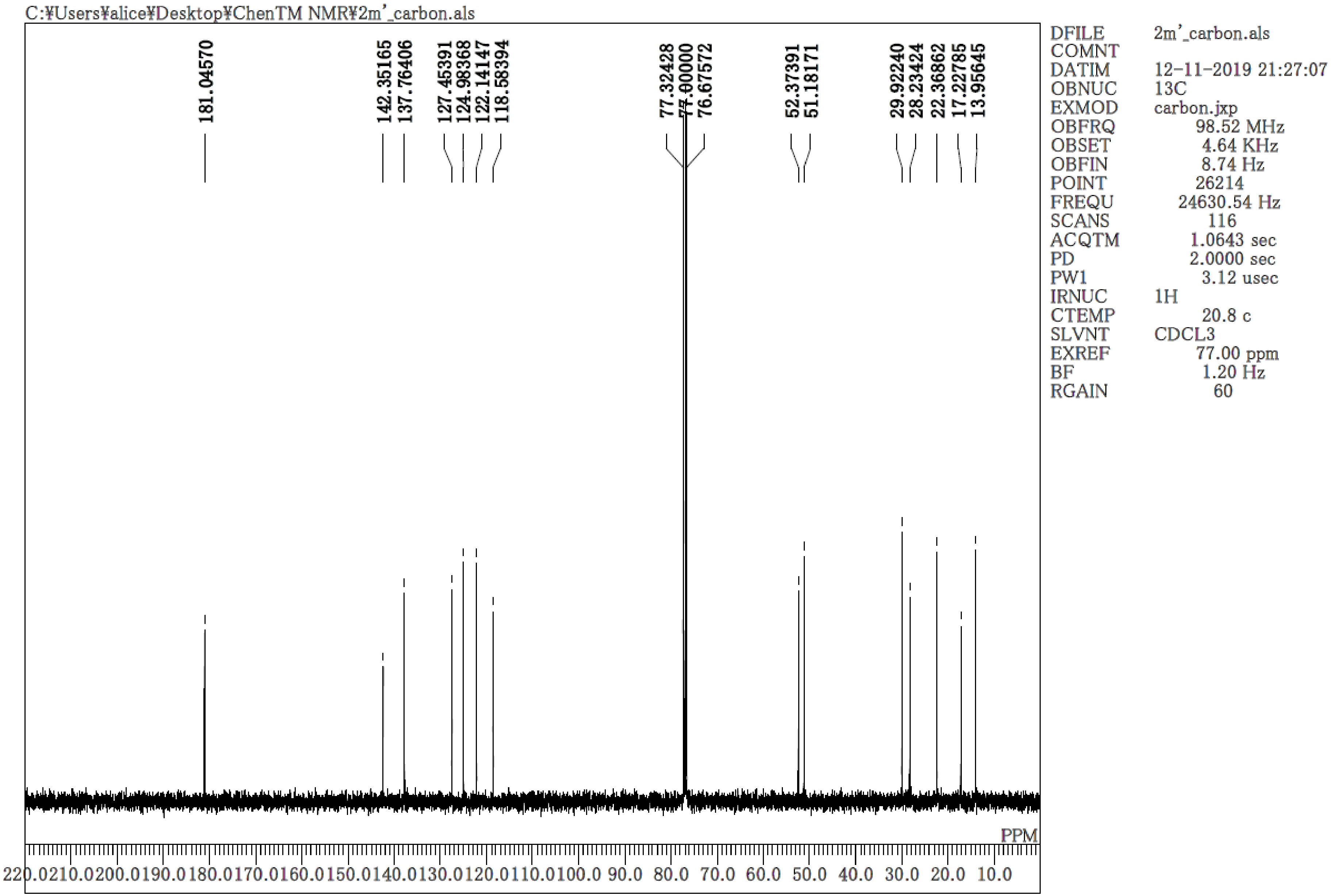
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2m'_proton.als

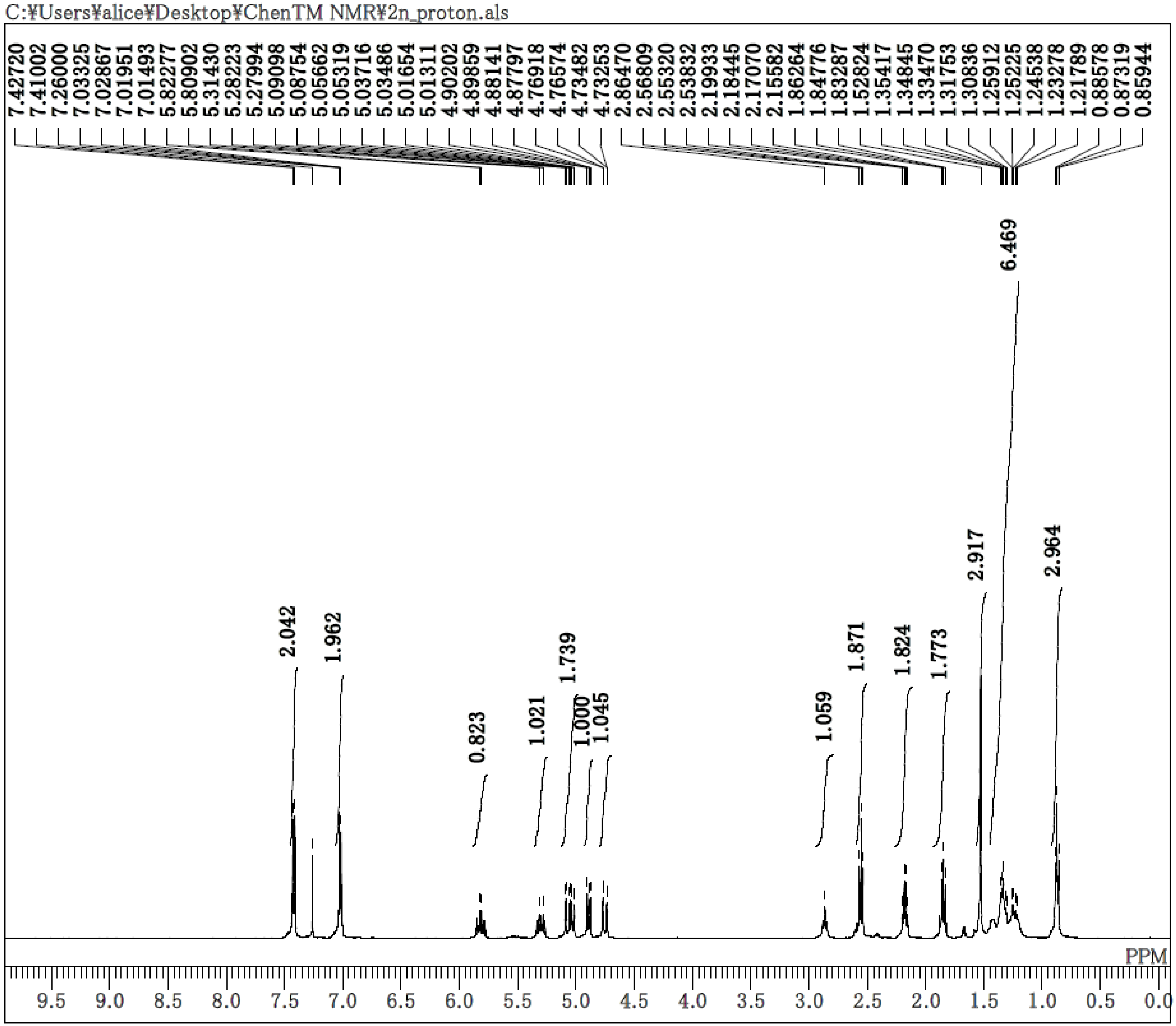


DFILE	2m'_proton.als
COMNT	
DATIM	12-11-2019 20:47:55
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	42

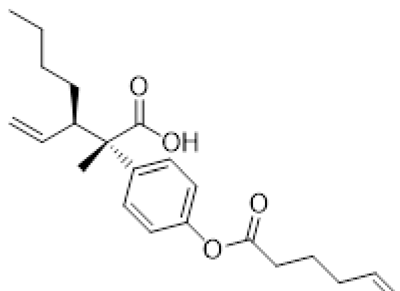


(2*R*,3*R*)-**2m**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2m'_carbon.als
181.04570
142.35165
137.76406
127.45391
124.98368
122.14147
118.58394
77.32428
77.00000
76.67572
52.37391
51.18171
29.92240
28.23424
22.36862
17.22785
13.95645
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2m'_carbon.als
COMNT
DATIM 12-11-2019 21:27:07
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 116
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

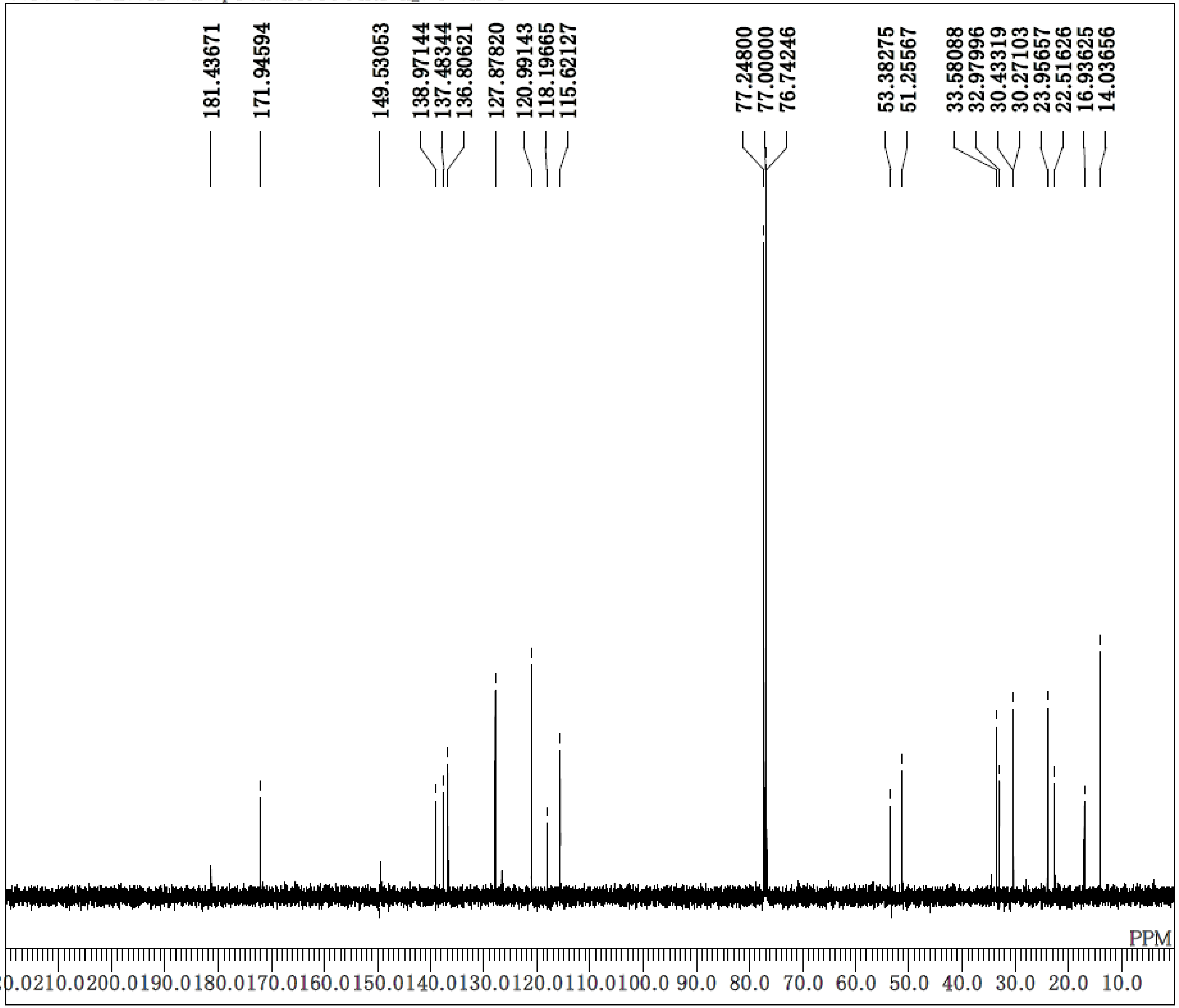


DFILE	2n_proton.als
COMNT	
DATIM	2019-12-24 20:56:28
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	2.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



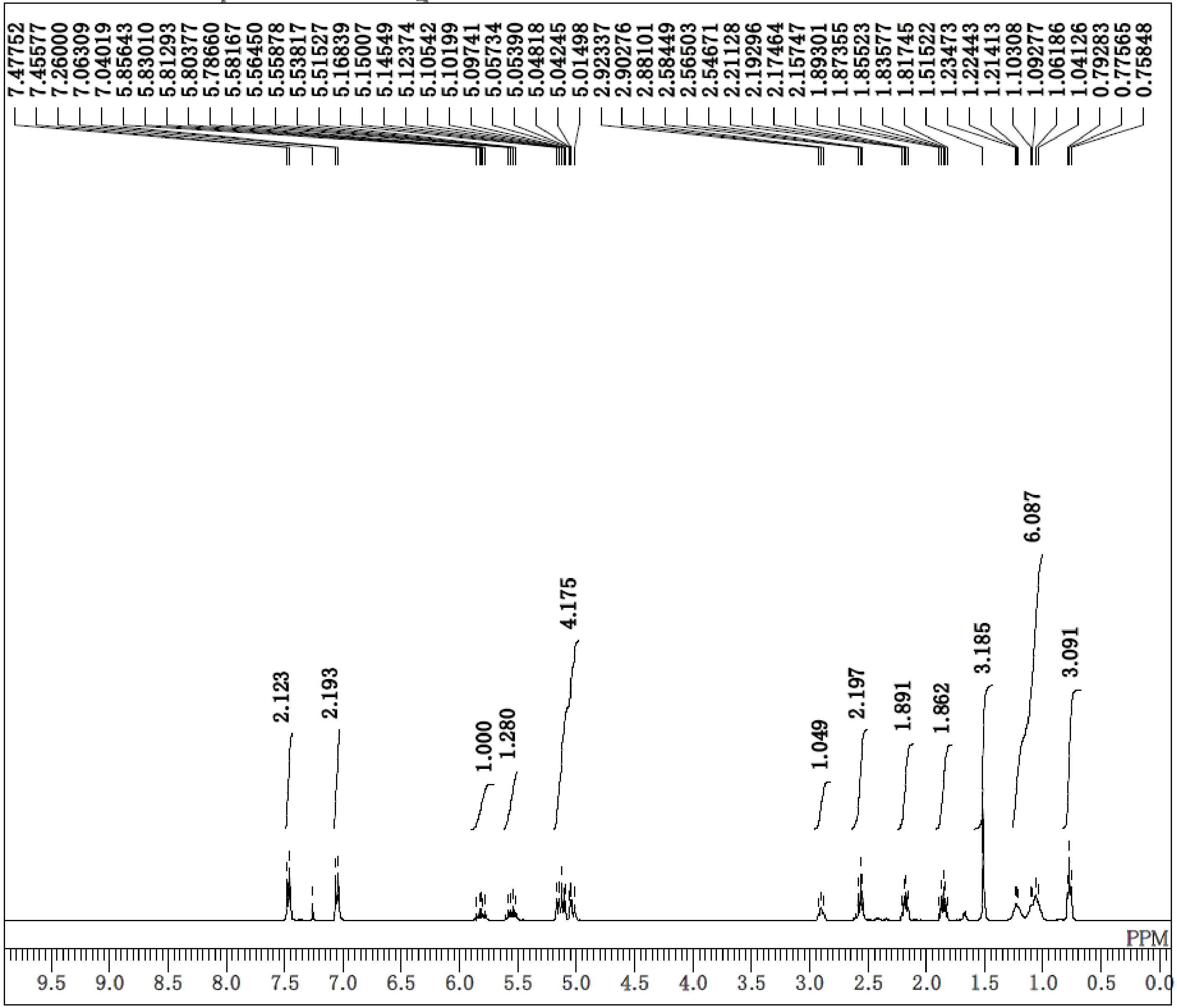
(2*S*,3*R*)-**2n**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2n_carbon.als

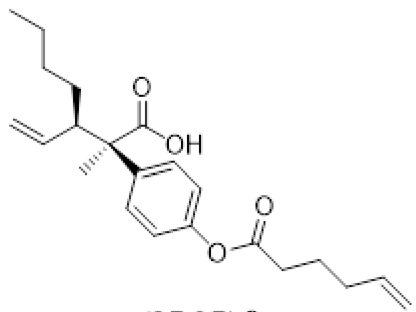


DFILE	2n_carbon.als
COMNT	
DATIM	2019-12-24 21:07:45
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	165
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

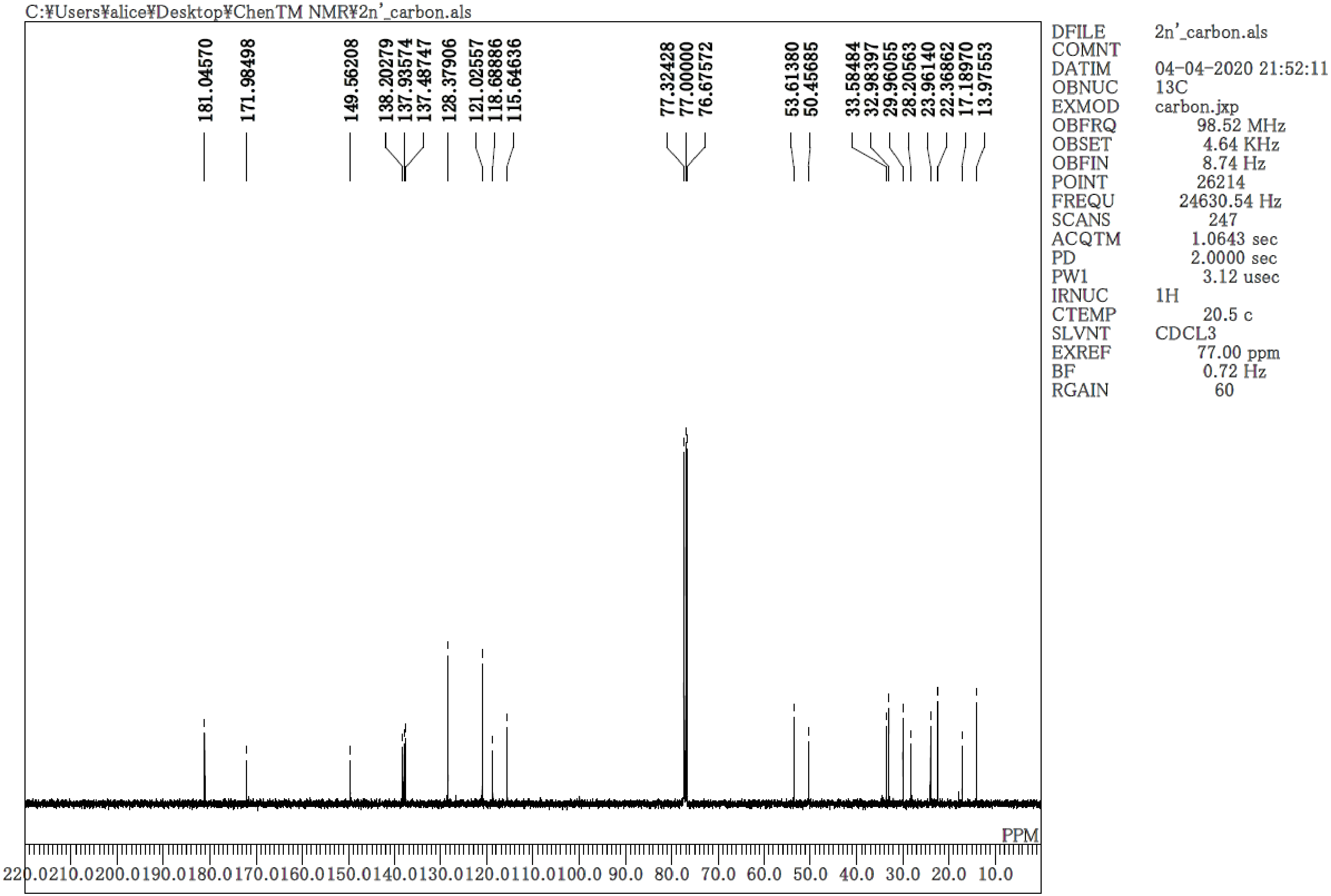
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2n'_proton.als



DFILE	2n'_proton.als
COMNT	
DATIM	04-04-2020 21:50:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.72 Hz
RGAIN	34



(2*R*,3*R*)-**2n**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2n'_carbon.als
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171.98498
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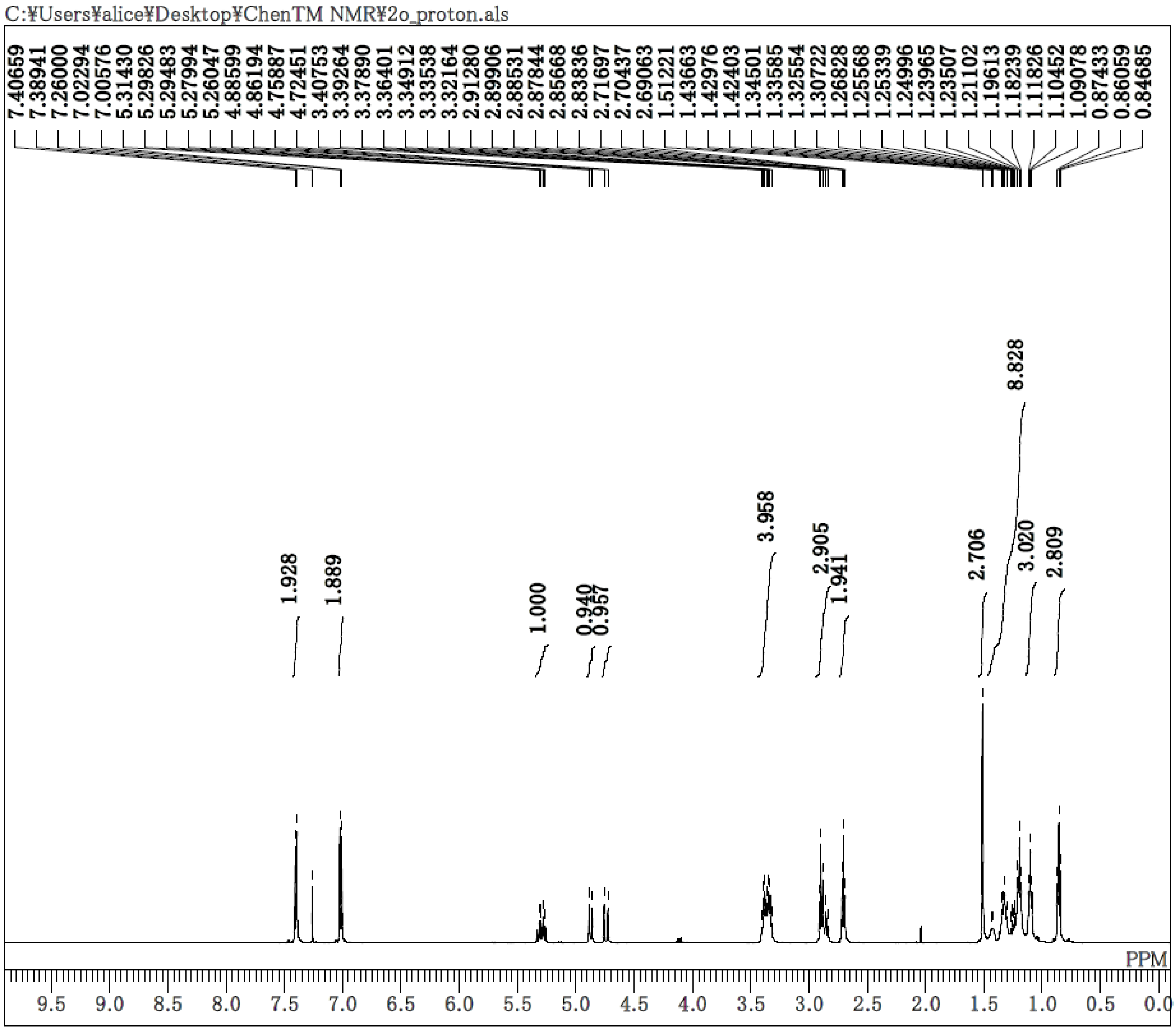
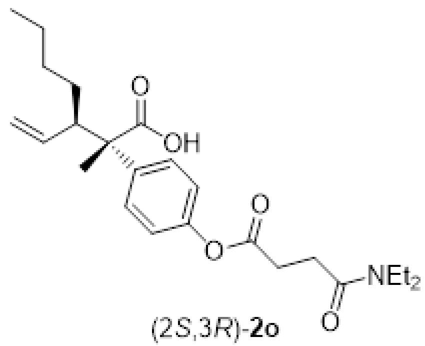
138.20279
137.93574
137.48747
128.37906
121.02557
118.68886
115.64636
77.32428
77.00000
76.67572
53.61380
50.45685
33.58484
32.98397
29.96055
28.20563
23.96140
22.36862
17.18970
13.97553
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2n'_carbon.als
COMNT
DATIM 04-04-2020 21:52:11
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 247
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.72 Hz
RGAIN 60

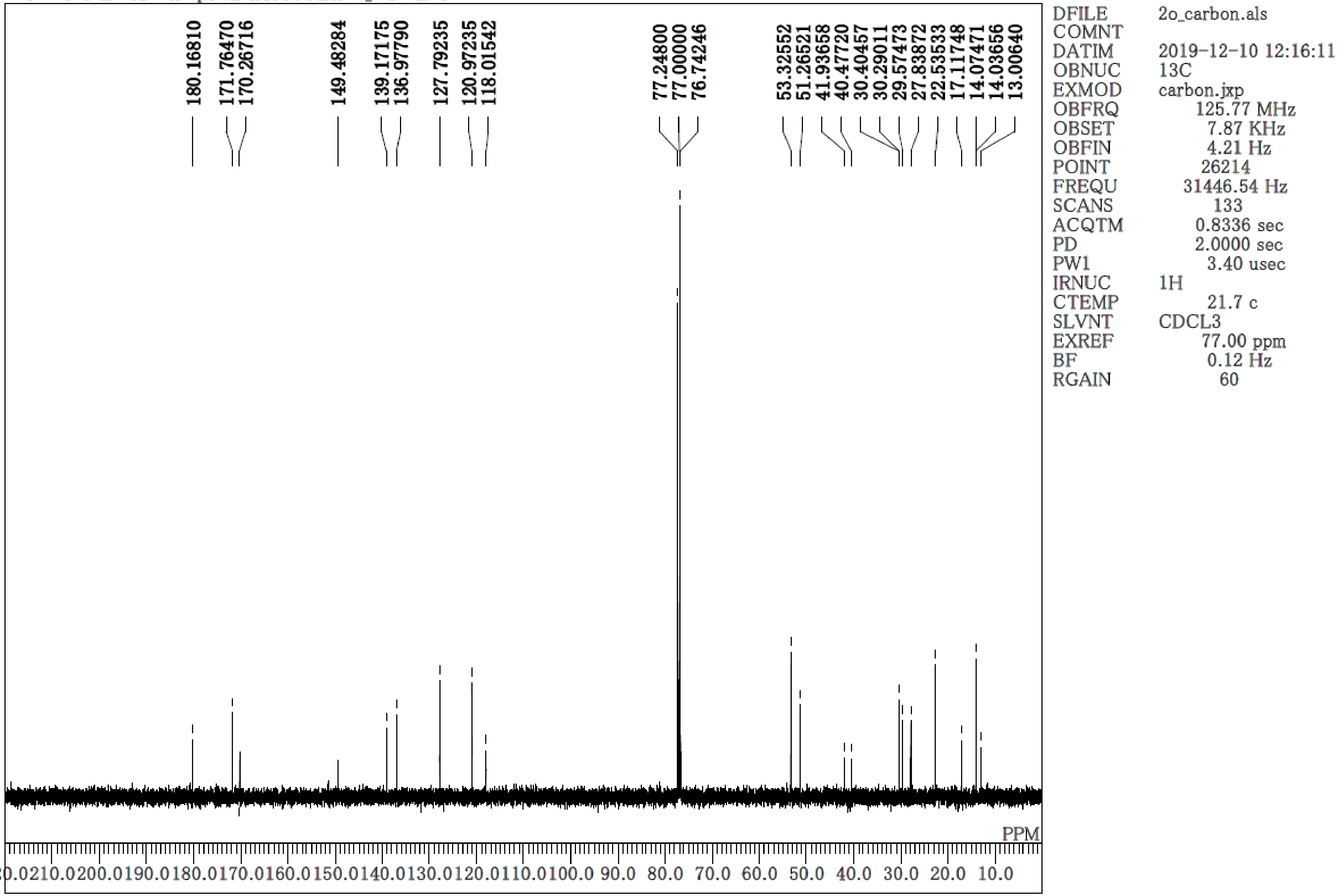


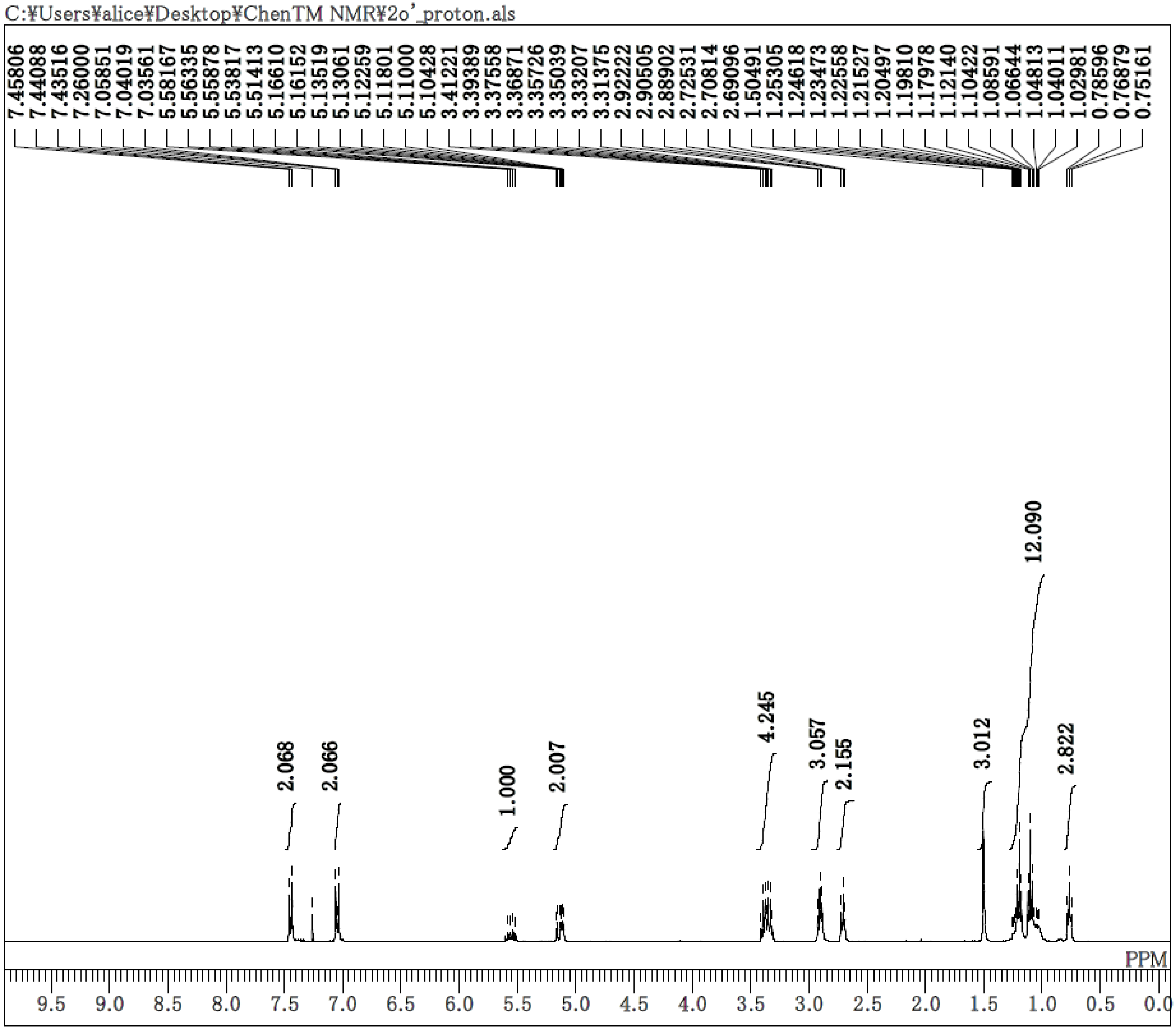
DFILE	2o_proton.als
COMNT	
DATIM	2019-12-10 12:10:39
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



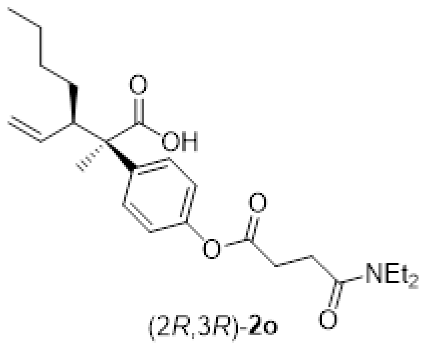
(2*S*,3*R*)-**2o**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2o_carbon.als

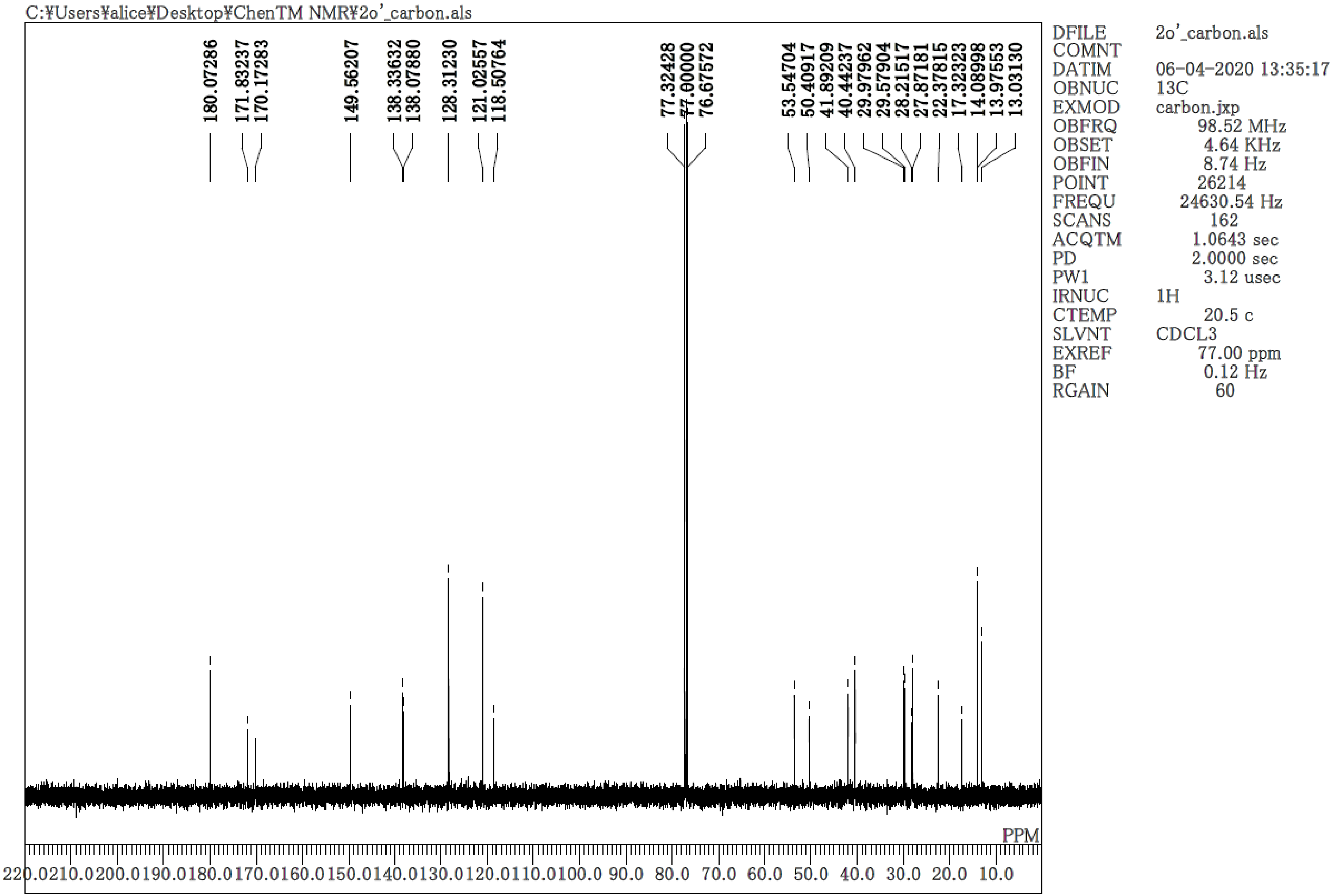




DFILE	2o'_proton.als
COMNT	
DATIM	06-04-2020 13:32:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	13
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32



(2*R*,3*R*)-**2o**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2o'_carbon.als
180.07286
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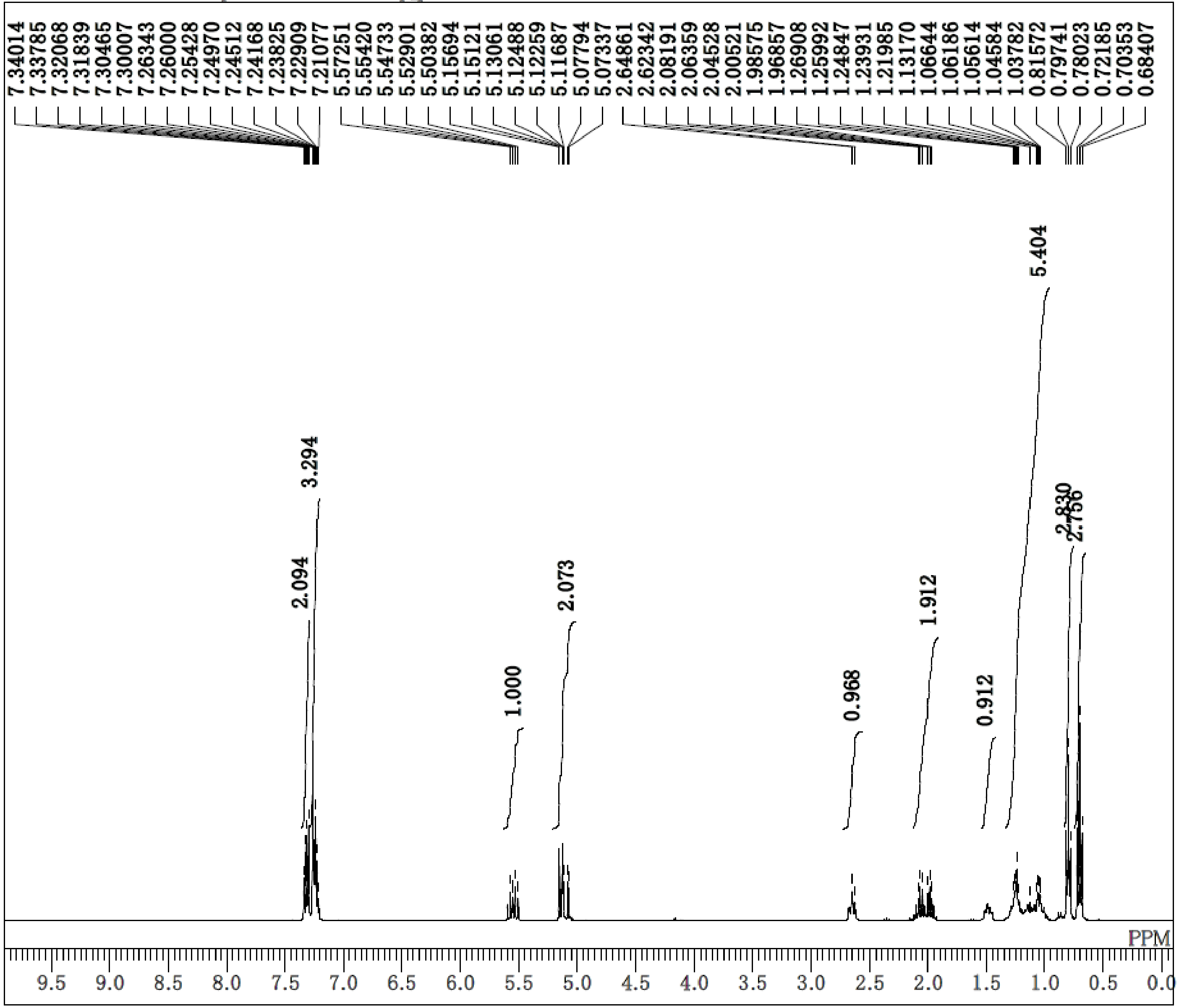
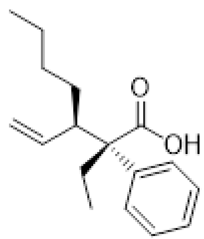
149.56207
138.33632
138.07880
128.31230
121.02557
118.50764
77.32428
77.00000
76.67572
53.54704
50.40917
41.89209
40.44237
29.97962
29.57904
28.21517
27.87181
22.37815
17.32323
14.08998
13.97553
13.03130
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2o'_carbon.als
COMNT
DATIM 06-04-2020 13:35:17
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 162
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2p_proton.als

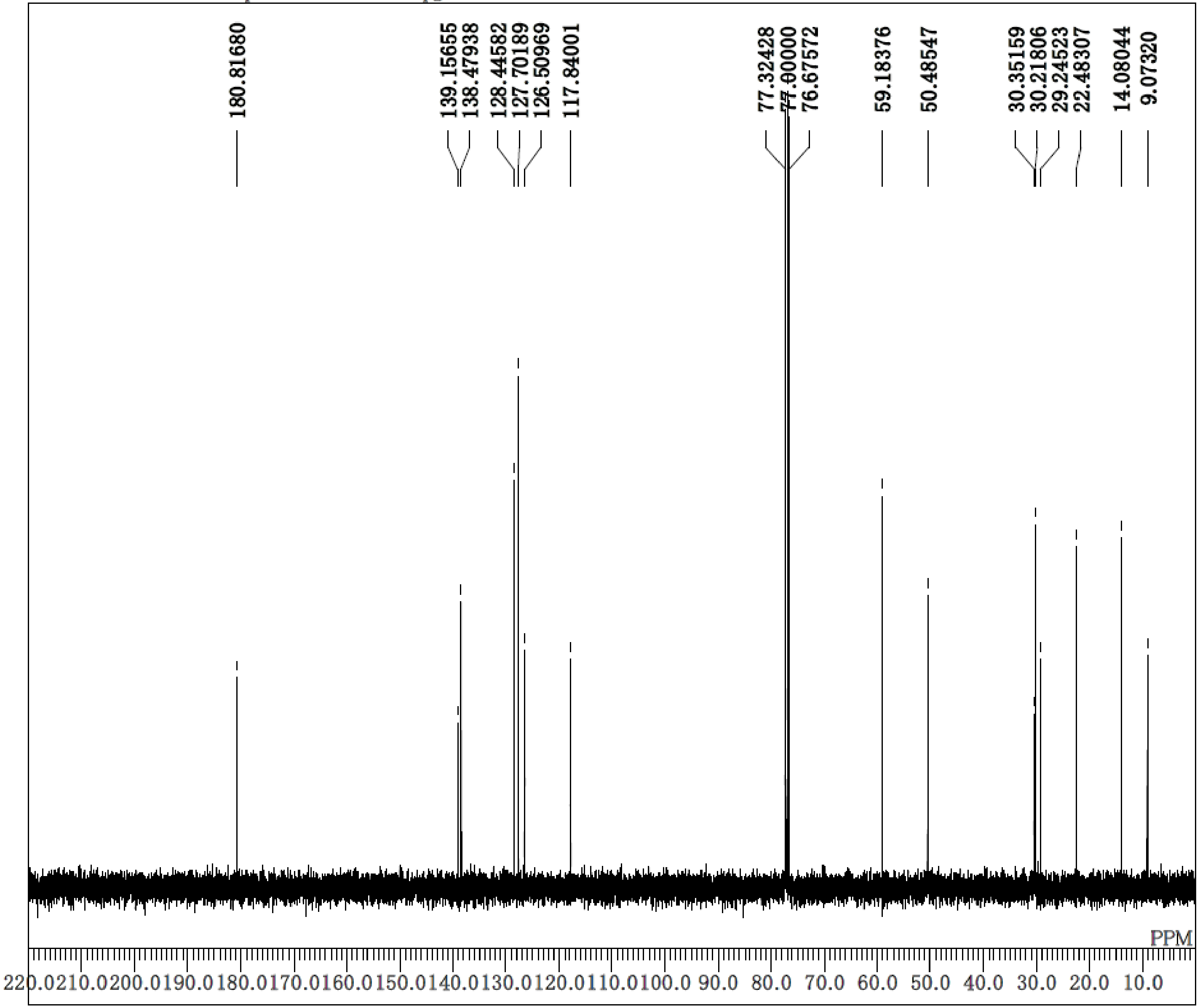


DFILE	2p_proton.als
COMNT	
DATIM	14-03-2020 17:31:34
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32



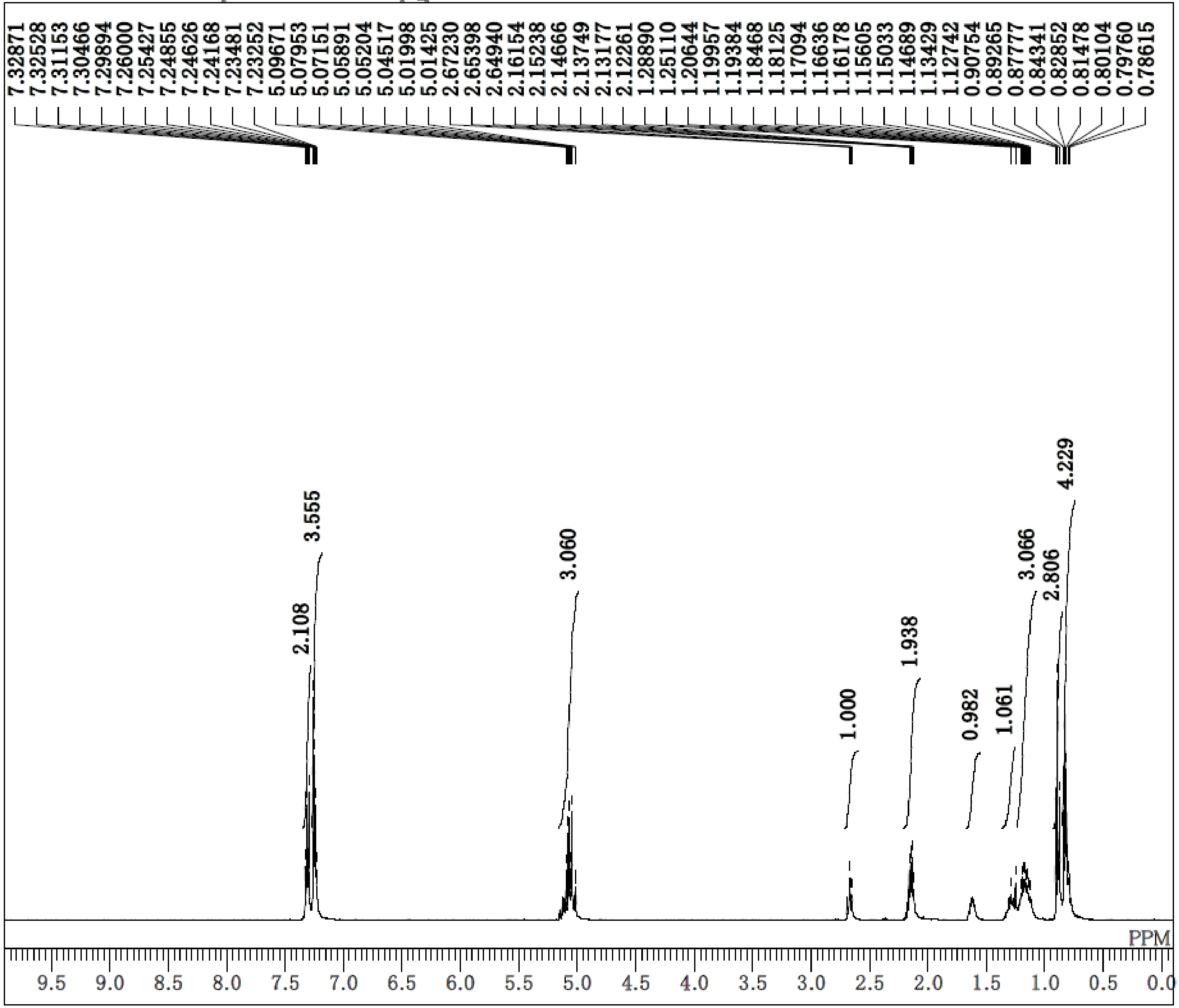
(2*S*,3*R*)-**2p**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2p_carbon.als

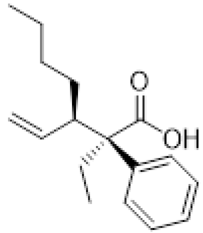


DFILE	2p_carbon.als
COMNT	
DATIM	14-03-2020 17:33:56
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	108
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2p'_proton.als

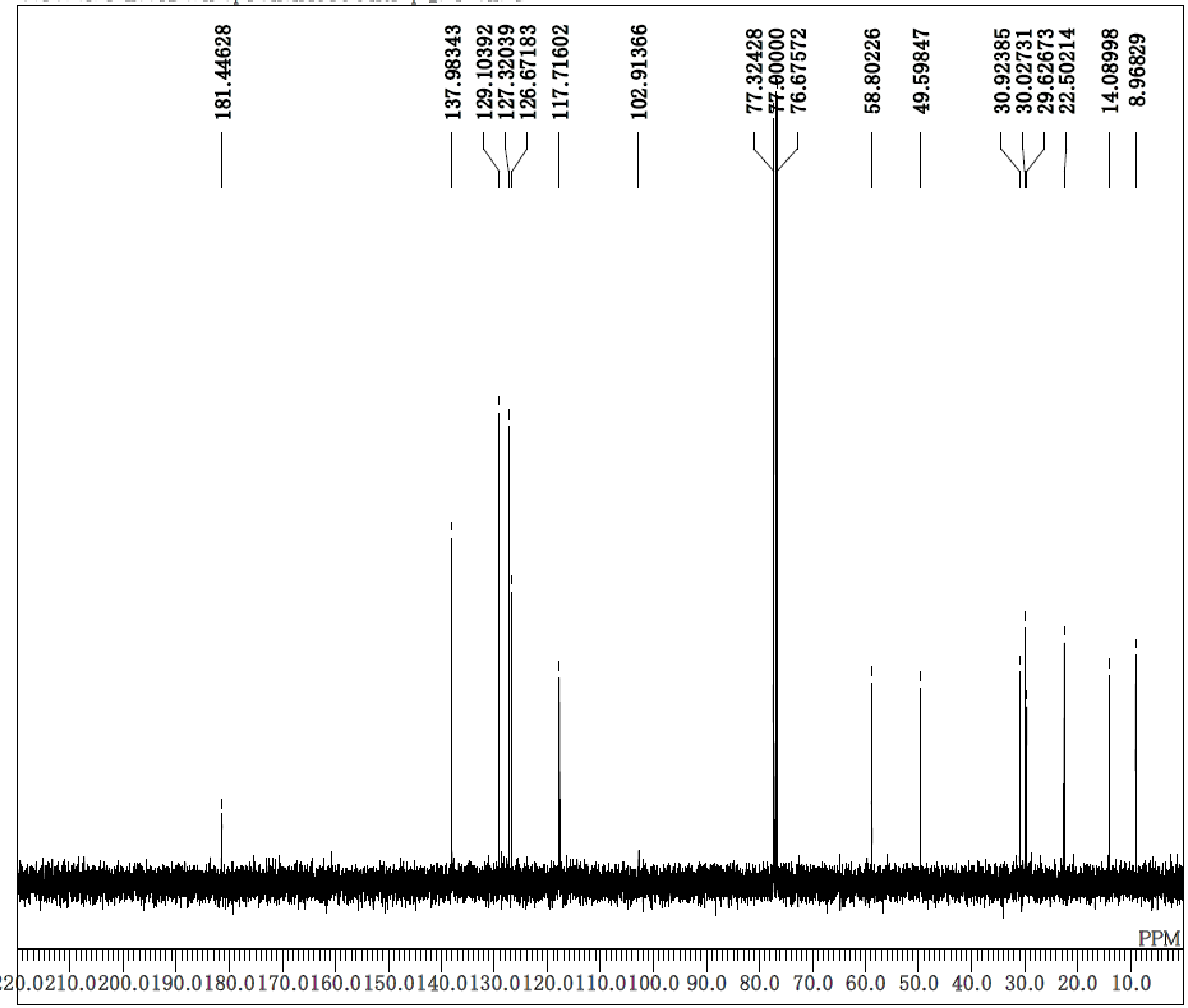


DFILE	2p'_proton.als
COMNT	
DATIM	2019-10-02 13:01:37
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



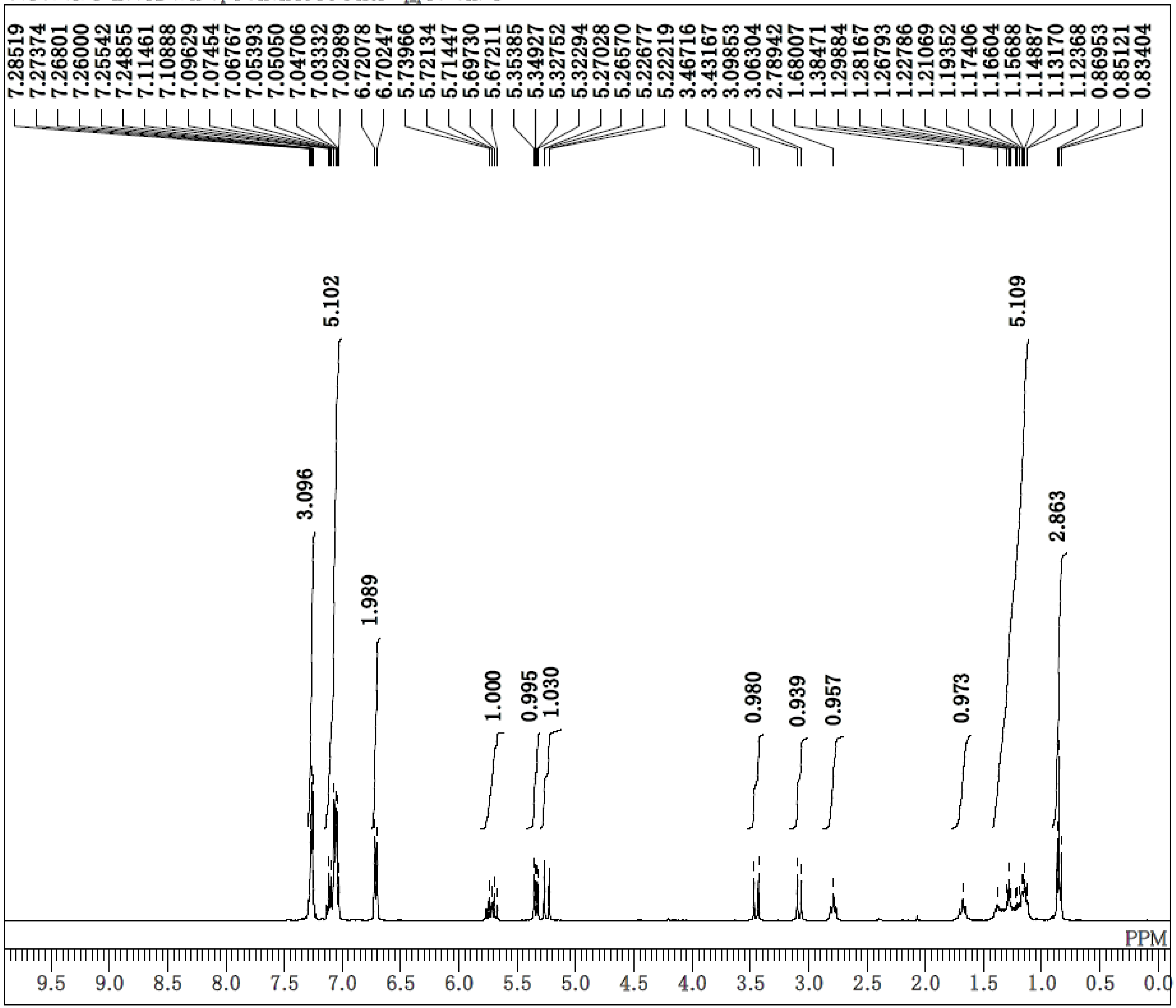
(2*R*,3*R*)-**2p**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2p'_carbon.als

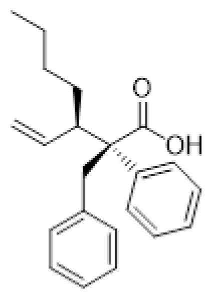


DFILE	2p'_carbon.als
COMNT	
DATIM	02-10-2019 12:05:17
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	120
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

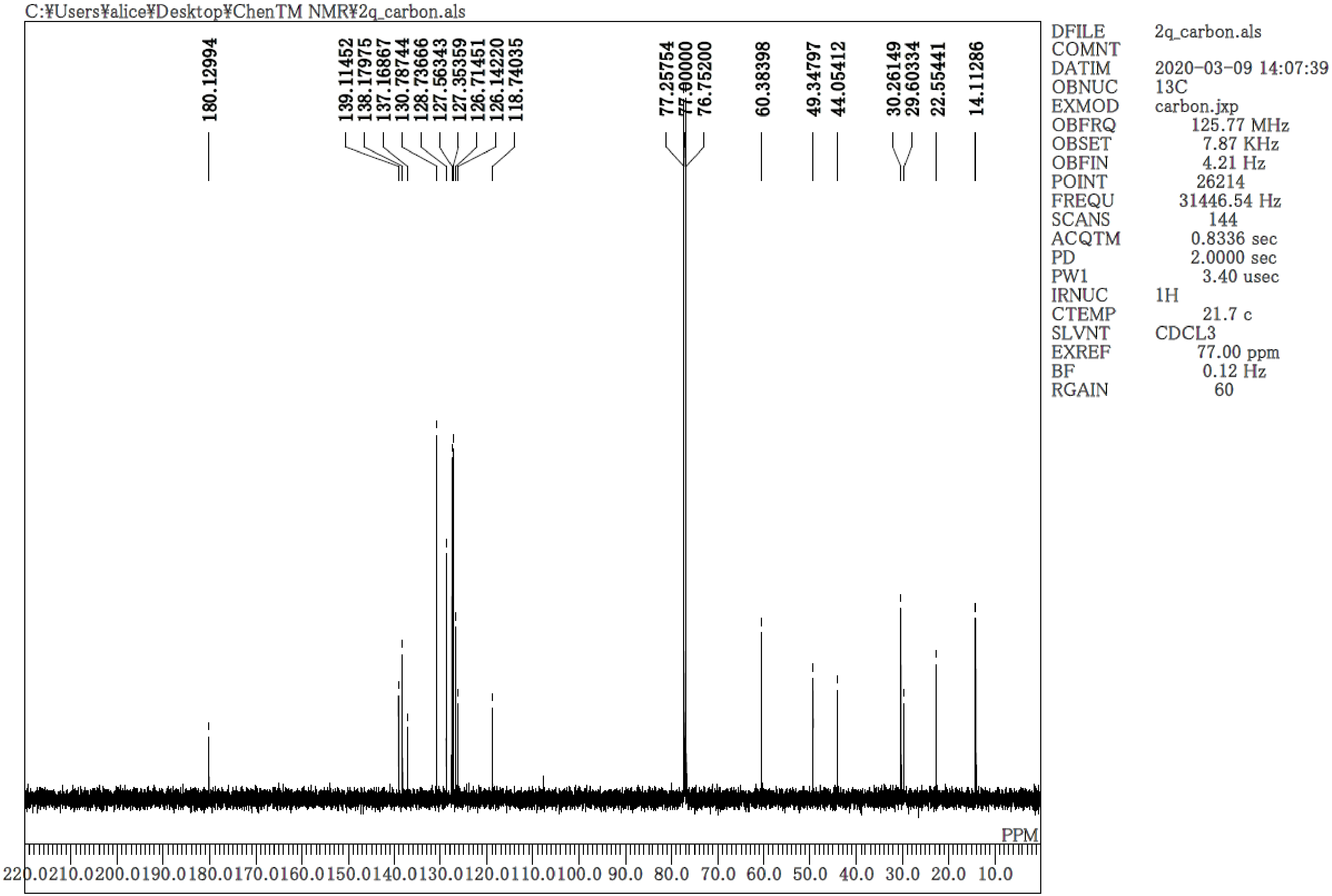
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2q_proton.als



DFILE	2q_proton.als
COMNT	
DATIM	09-03-2020 14:16:54
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30



(2*S*,3*R*)-**2q**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2q_carbon.als
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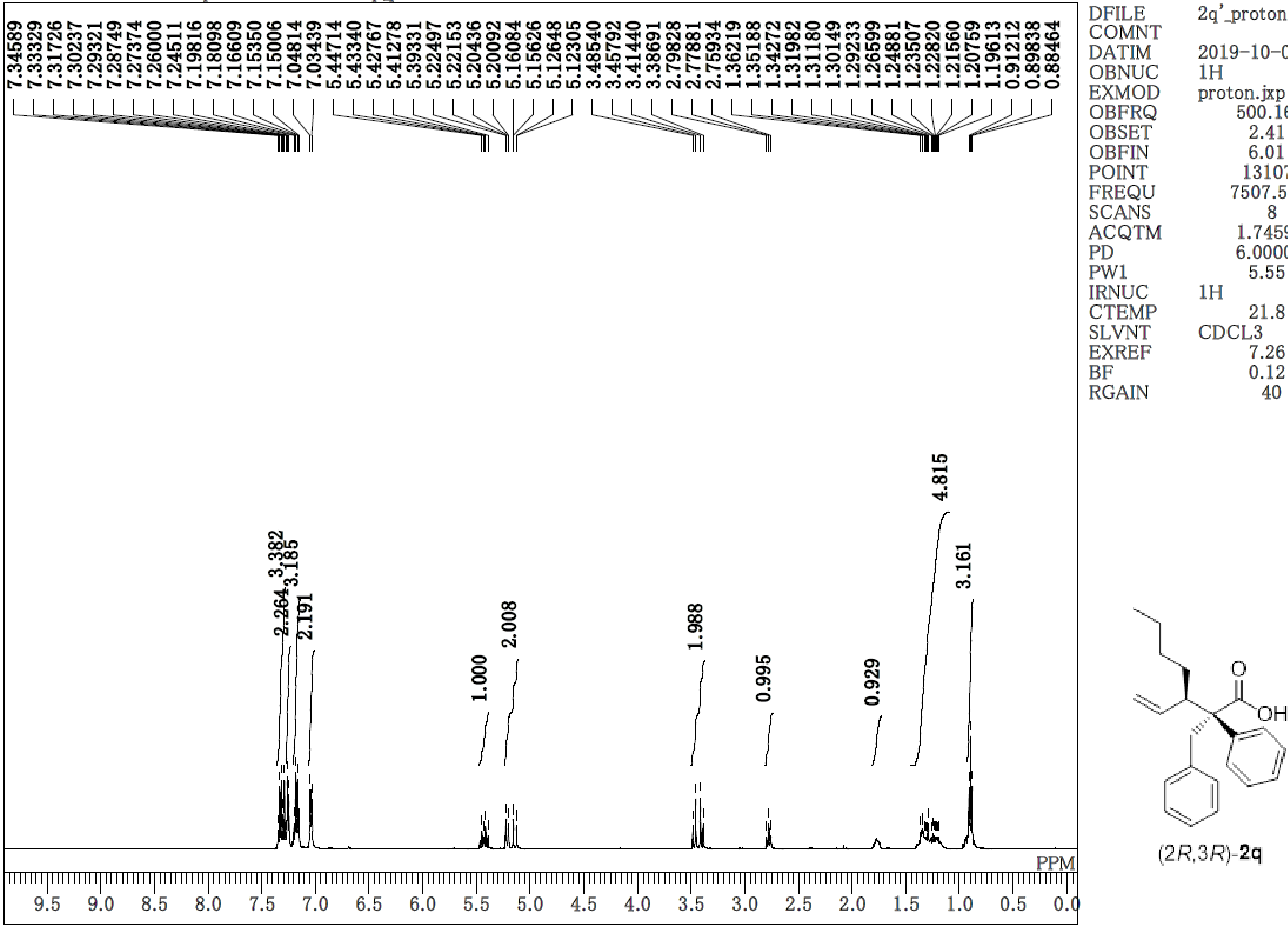
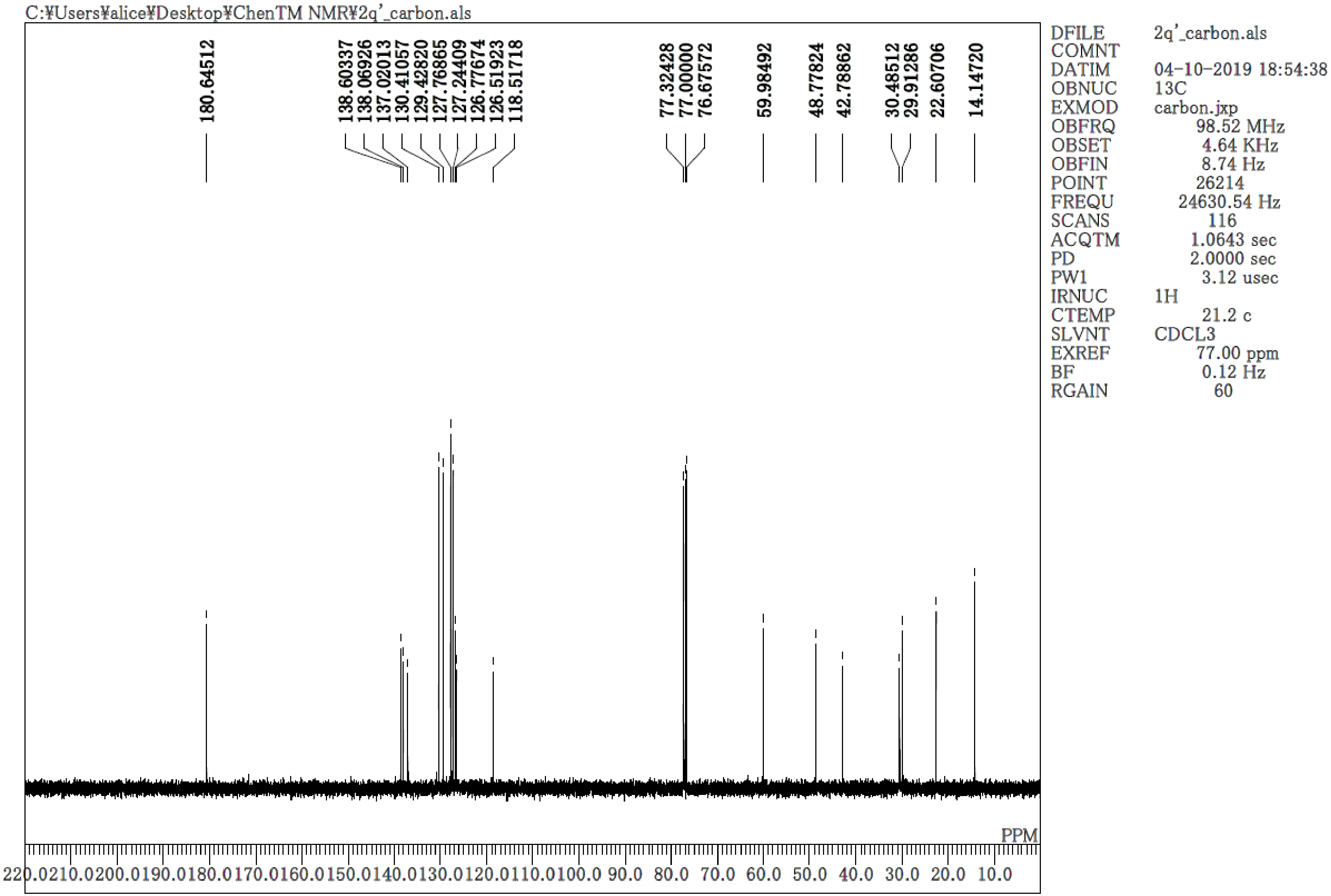
139.11452
138.17975
137.16867
130.78744
128.73666
127.56343
127.35359
126.71451
126.14220
118.74035
77.25754
77.00000
76.75200
60.38398
49.34797
44.05412
30.26149
29.60334
22.55441
14.11286
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2q_carbon.als
COMNT
DATIM 2020-03-09 14:07:39
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 144
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

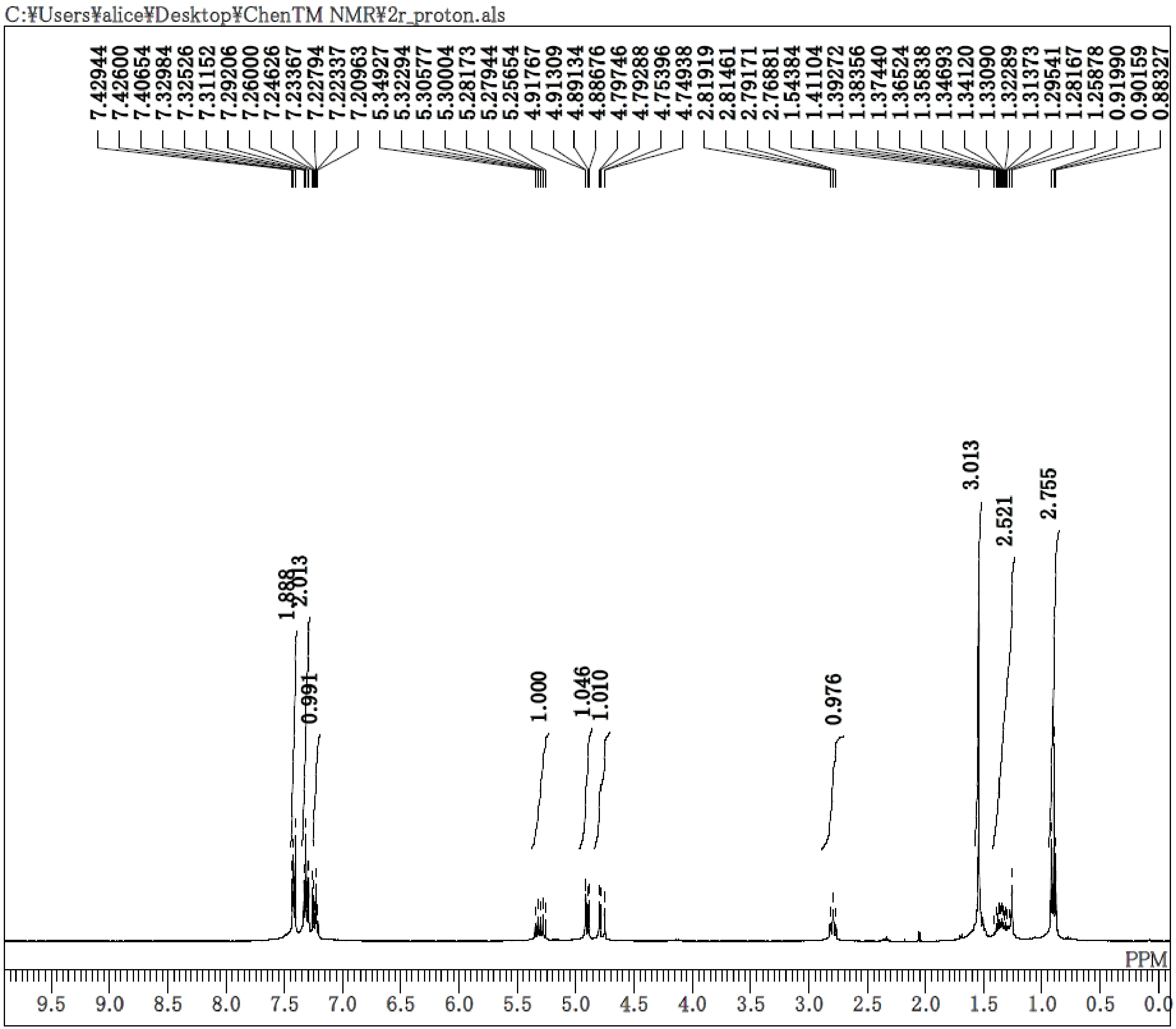
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2q'_proton1.als

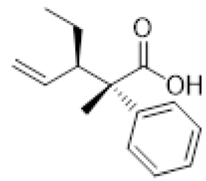


DFILE	2q'_proton1.als
COMNT	
DATIM	2019-10-04 15:39:14
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

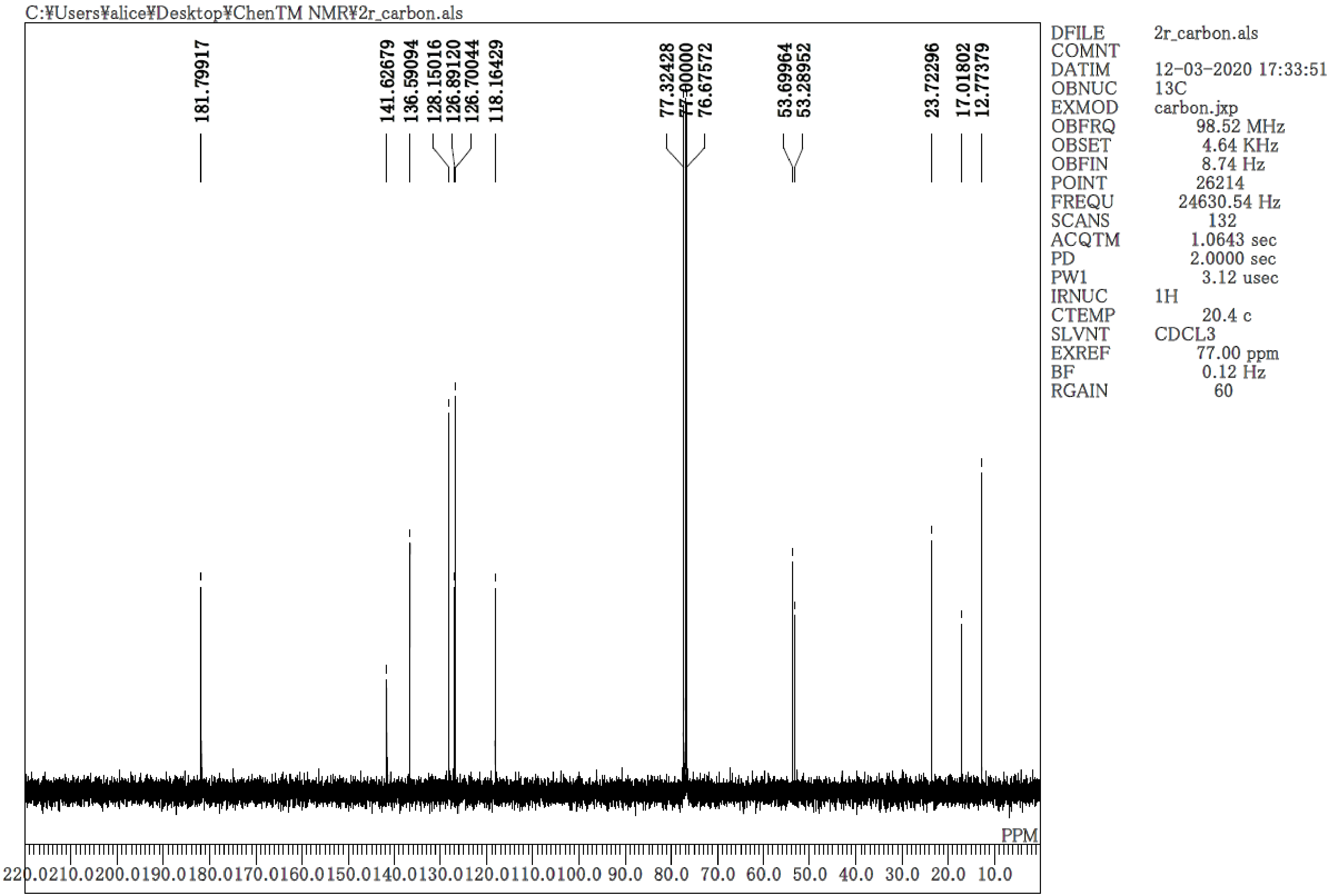
(2*R*,3*R*)-**2q**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2q'_carbon.als
180.64512
138.60337
138.06926
137.02013
130.41057
129.42820
127.76865
127.24409
126.77674
126.51923
118.51718
77.32428
77.00000
76.67572
59.98492
48.77824
42.78862
30.48512
29.91286
22.60706
14.14720
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2q'_carbon.als
COMNT
DATIM 04-10-2019 18:54:38
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 116
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

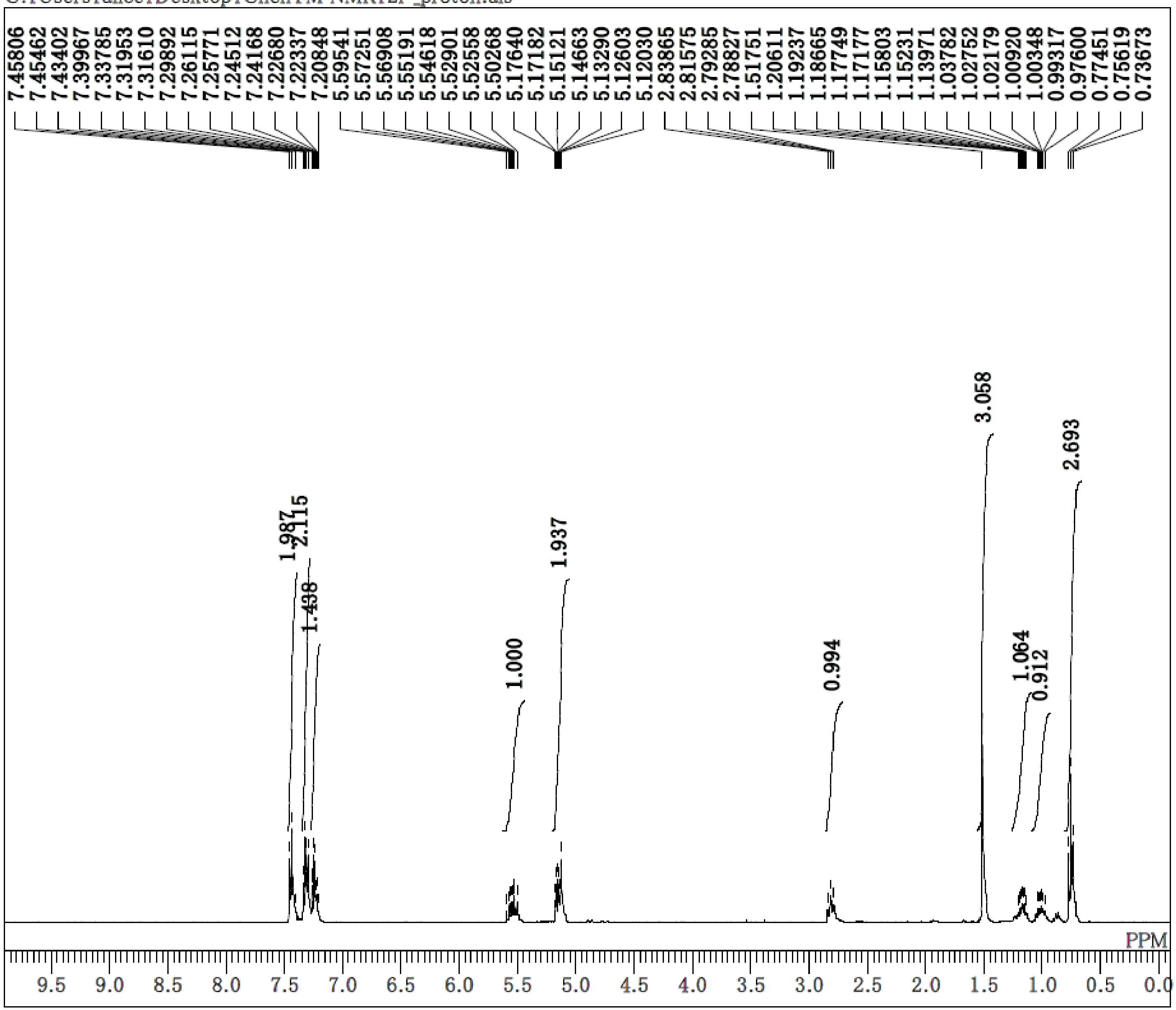

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2r_proton.als
7.42944
7.42600
7.40654
7.32984
7.32526
7.31152
7.29206
7.26000
7.24626
7.23367
7.22794
7.22337
7.20963
5.34927
5.32294
5.30577
5.30004
5.28173
5.27944
5.25654
4.91767
4.91309
4.89134
4.88676
4.79746
4.79288
4.75396
4.74938
2.81919
2.81461
2.79171
2.76881
1.54384
1.41104
1.39272
1.38356
1.37440
1.36524
1.35838
1.34693
1.34120
1.33090
1.32289
1.31373
1.29541
1.28167
1.25878
0.91990
0.90159
0.88327
1.888
2.013
0.991
1.000
1.046
1.010
0.976
3.013
2.521
2.755
PPM
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
DFILE 2r_proton.als
COMNT
DATIM 12-03-2020 17:47:31
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36


O
OH

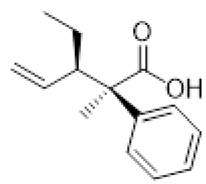
(2*S*,3*R*)-**2r**



C:¥Users¥alice¥Desktop¥ChenTM NMR¥2r_carbon.als
181.79917
141.62679
136.59094
128.15016
126.89120
126.70044
118.16429
77.32428
77.00000
76.67572
53.69964
53.28952
23.72296
17.01802
12.77379
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2r_carbon.als
COMNT
DATIM 12-03-2020 17:33:51
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 132
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

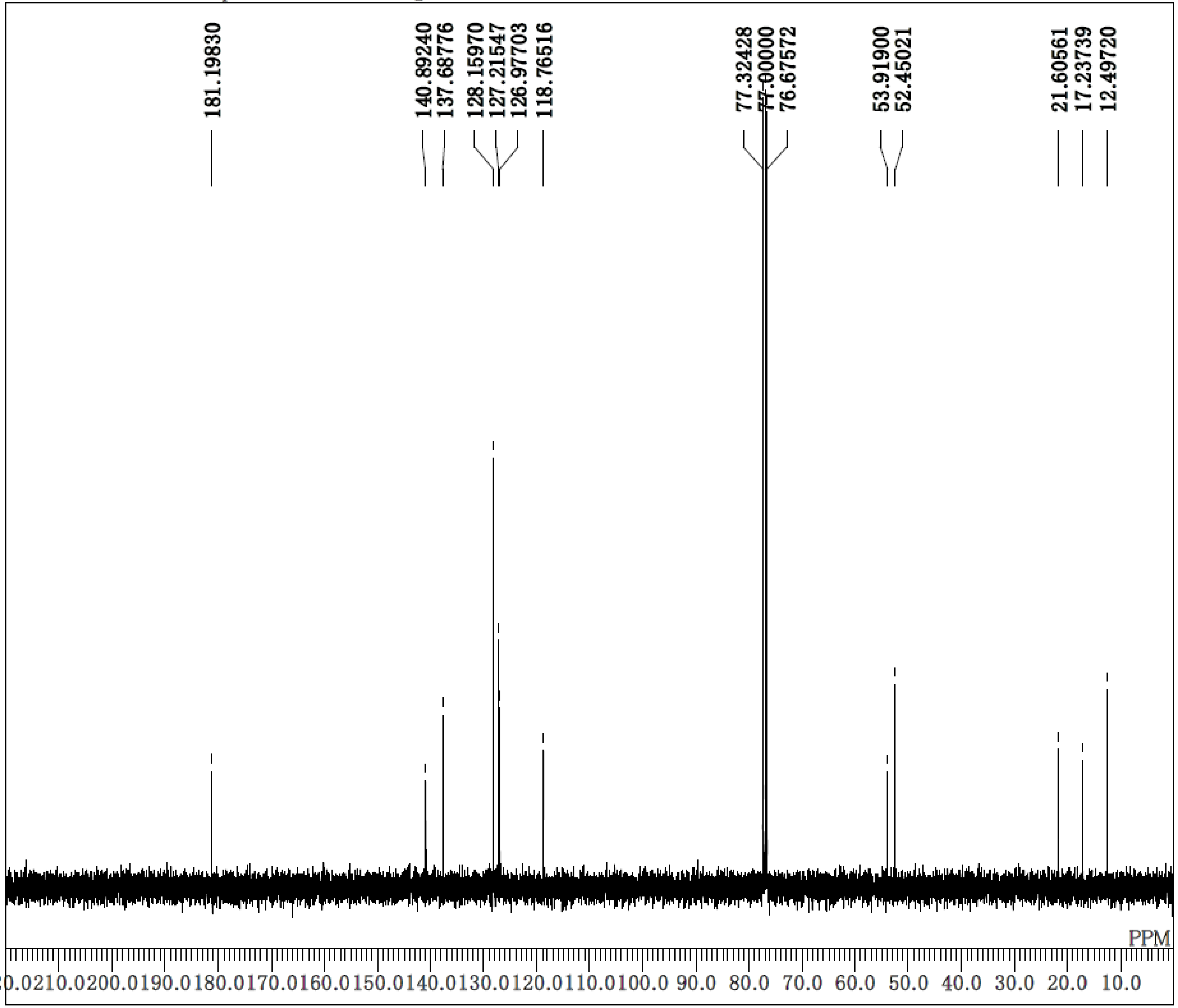


DFILE	2r'_proton.als
COMNT	
DATIM	12-03-2020 22:36:53
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38

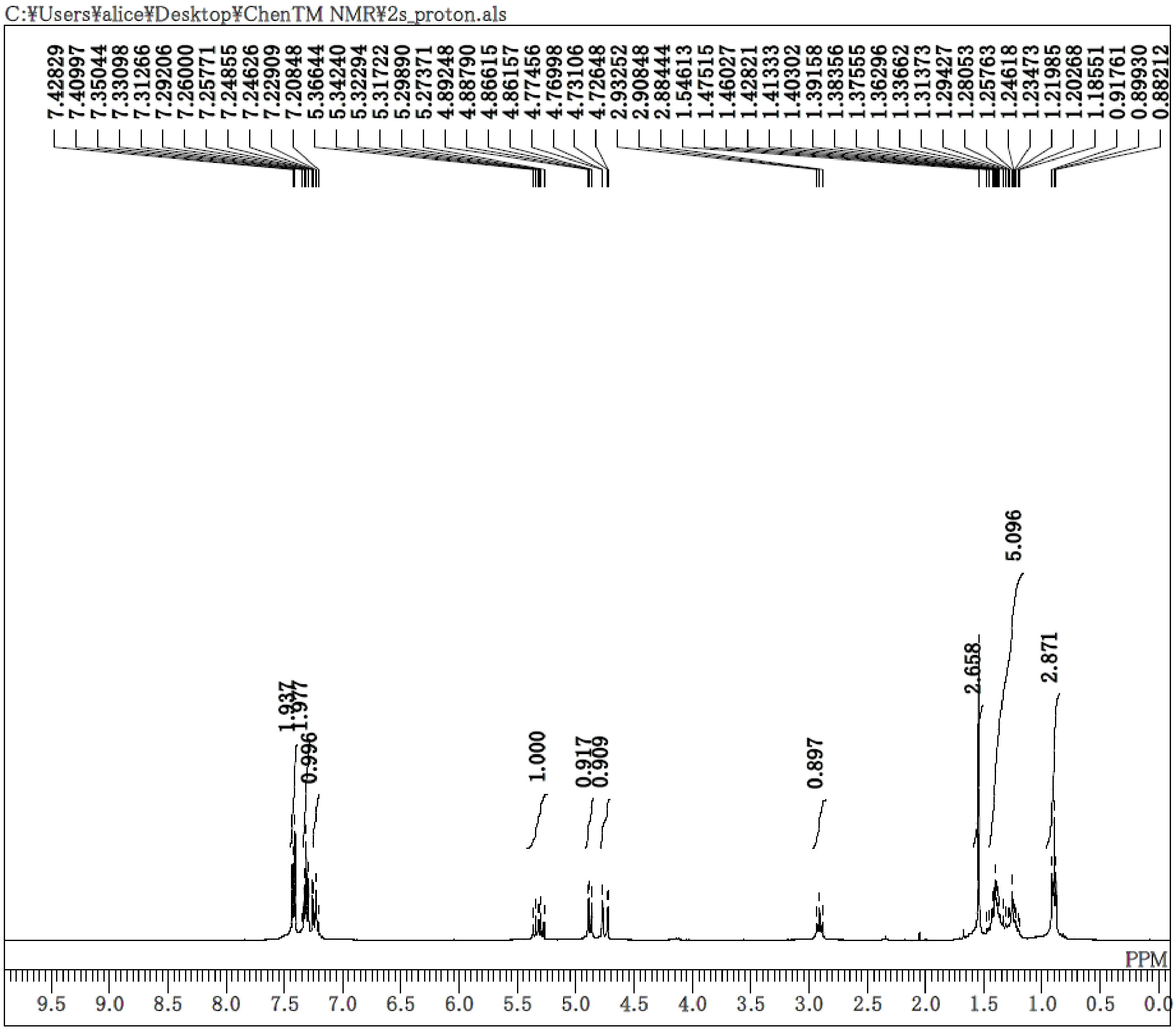


(2*R*,3*R*)-**2r**

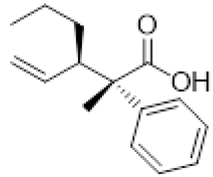
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2r'_carbon.als



DFILE	2r'_carbon.als
COMNT	
DATIM	12-03-2020 22:39:27
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	208
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

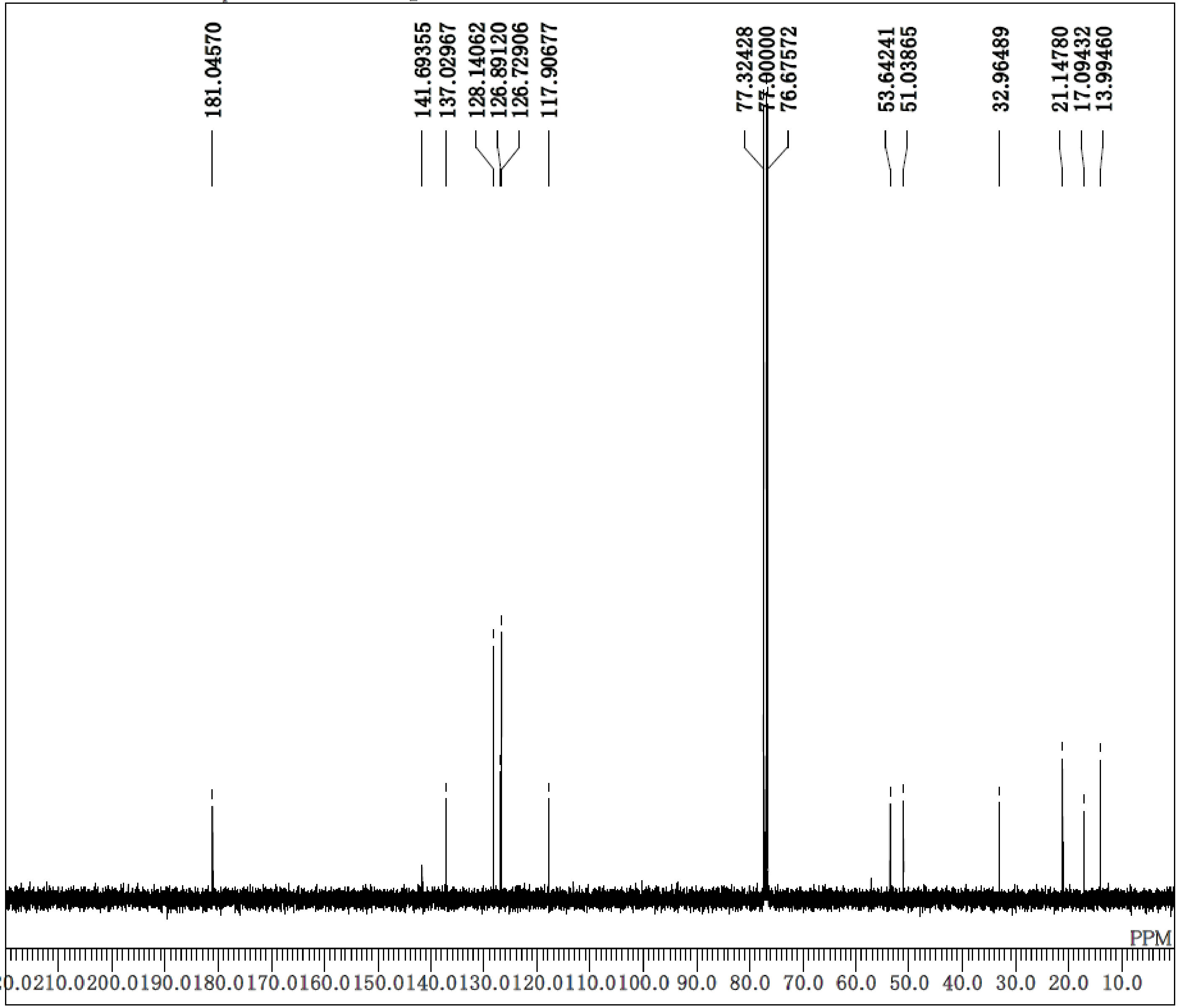


DFILE	2s_proton.als
COMNT	
DATIM	12-03-2020 12:15:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34

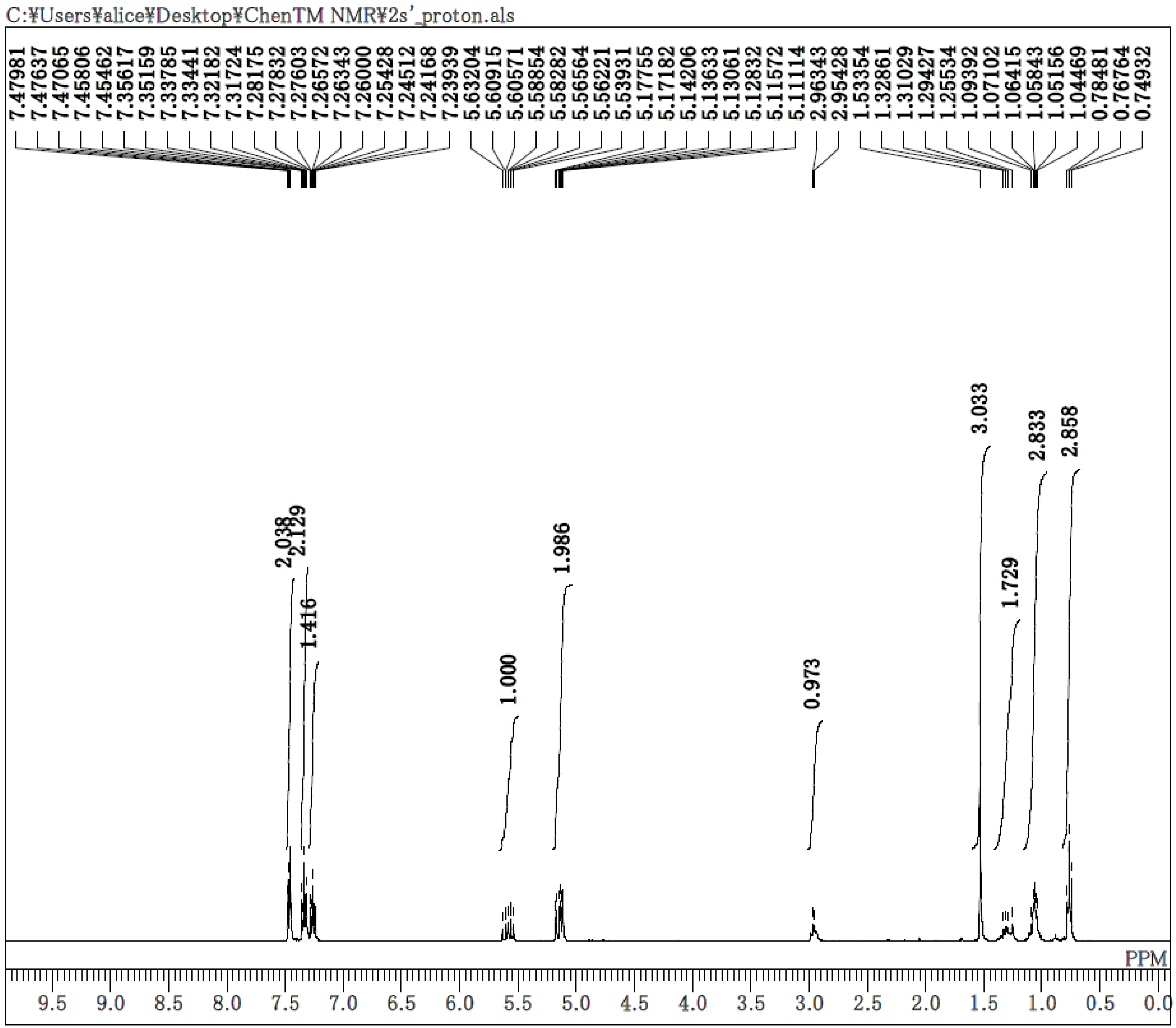


(2*S*,3*R*)-**2s**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2s_carbon.als



DFILE	2s_carbon.als
COMNT	
DATIM	11-03-2020 21:51:47
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	436
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2s'_proton.als
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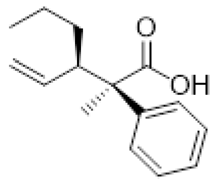
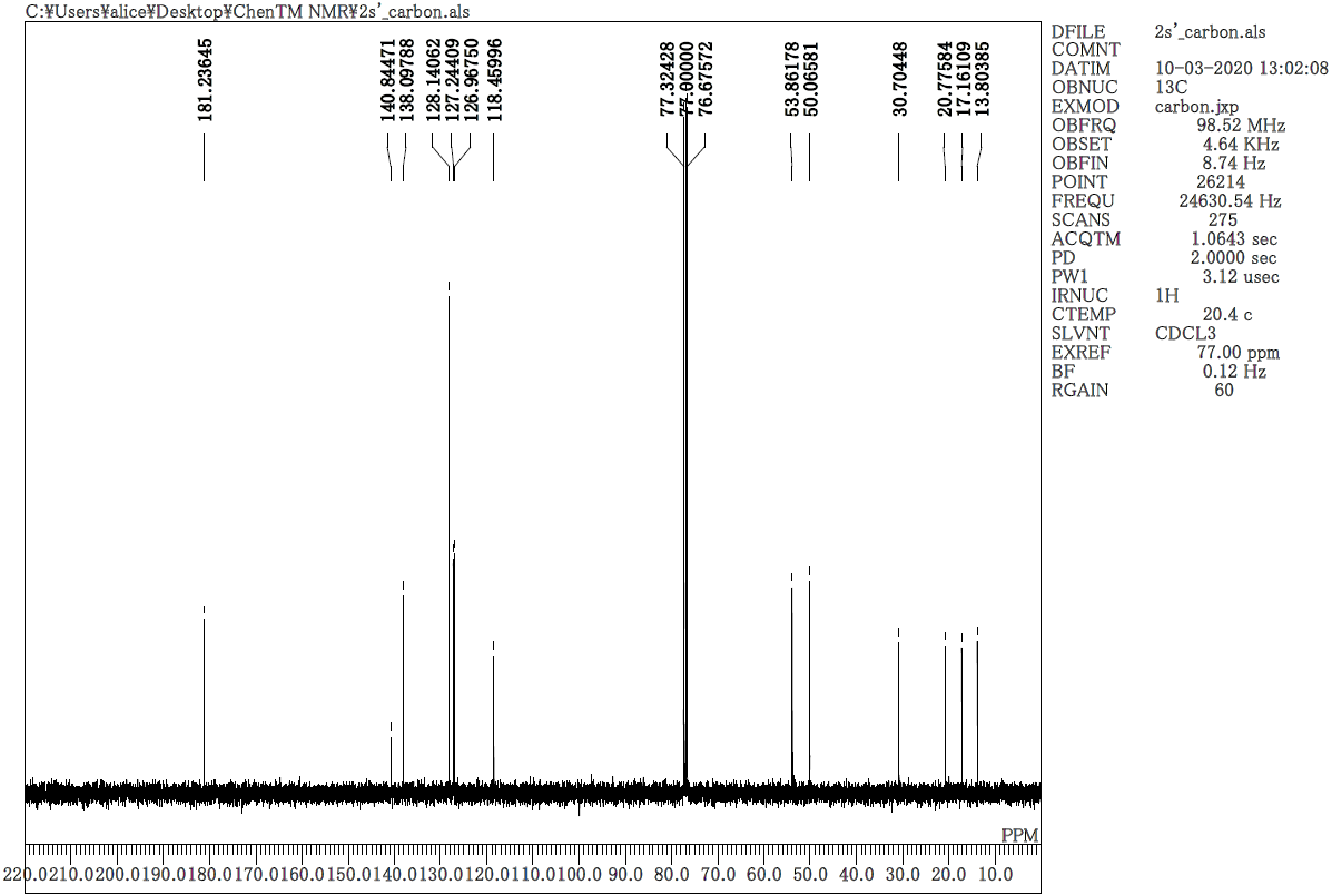
5.63204 5.60915 5.60571 5.58854 5.58282 5.56564 5.56221 5.53931 5.17755 5.17182 5.14206 5.13633 5.13061 5.12832 5.11572 5.11114
2.96343 2.95428
1.53354 1.32861 1.31029 1.29427 1.25534 1.09392 1.07102 1.06415 1.05843 1.05156 1.04469 0.78481 0.76764 0.74932
2.038
2.129
1.416
1.000
1.986
0.973
3.033
1.729
2.833
2.858
PPM
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
DFILE 2s'_proton.als
COMNT
DATIM 10-03-2020 13:19:28
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36


O
OH

(2*R*,3*R*)-**2s**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2s'_carbon.als
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140.84471
138.09788
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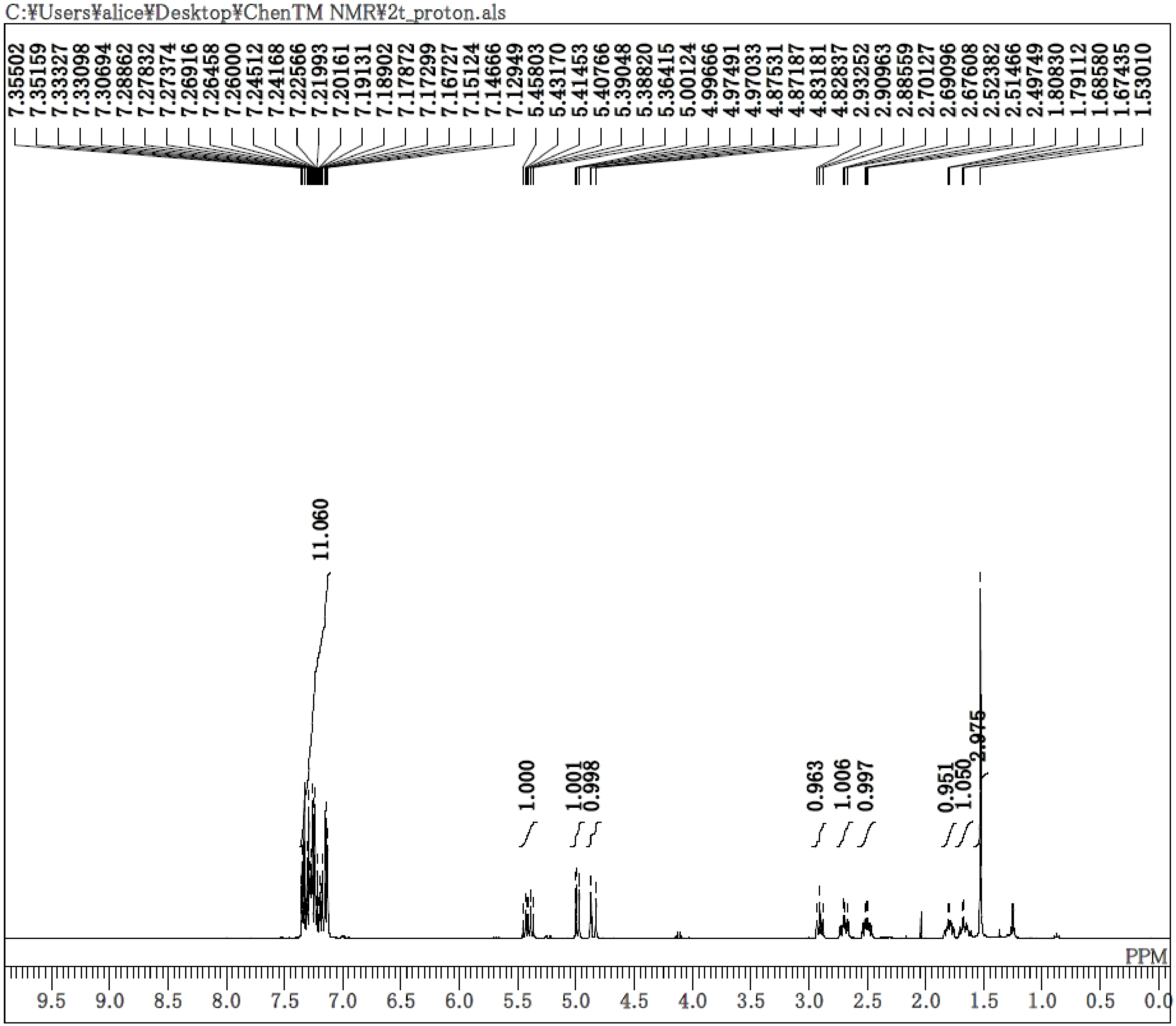
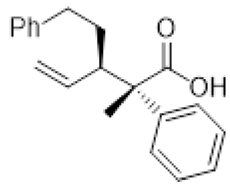
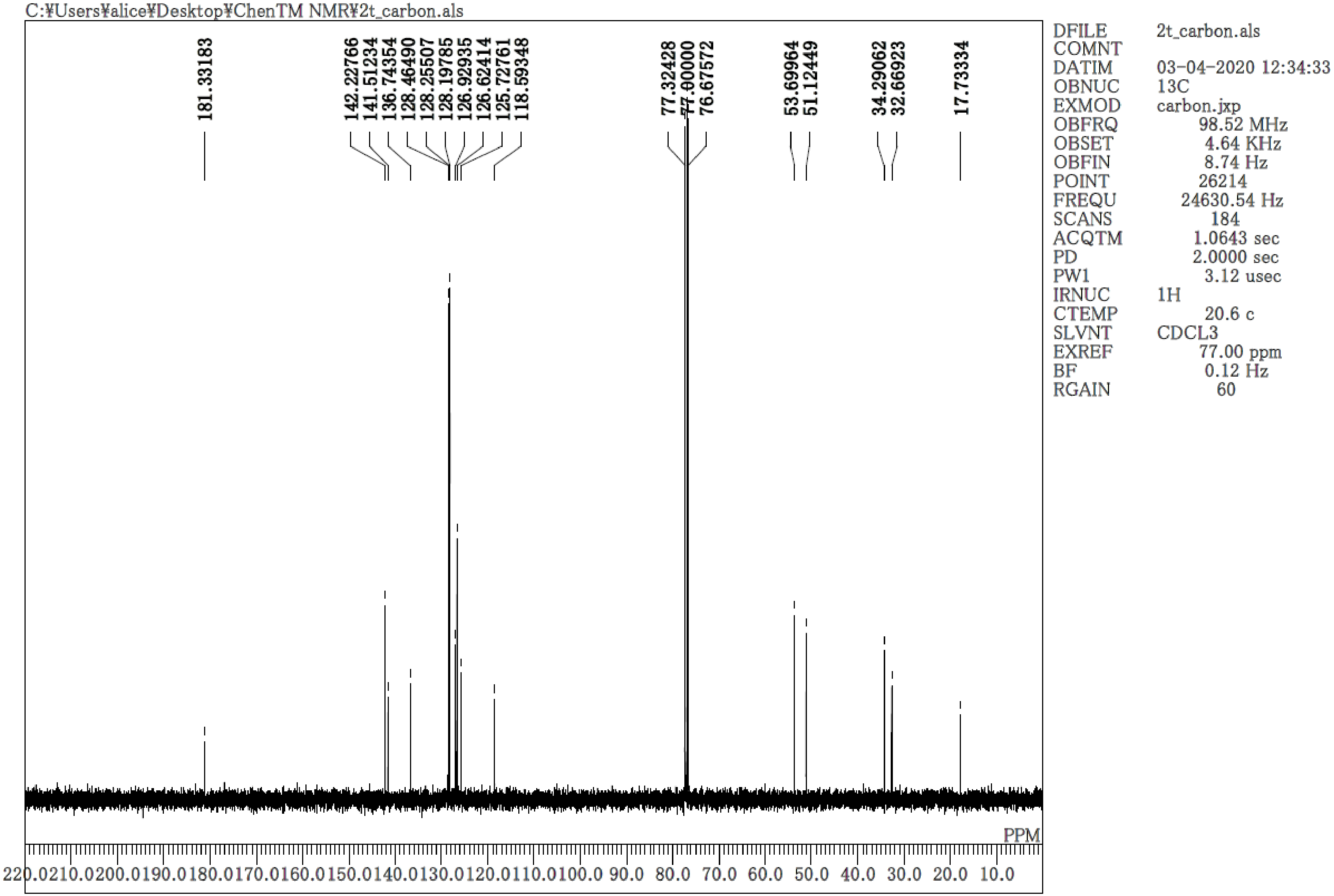
127.24409
126.96750
118.45996
77.32428
77.00000
76.67572
53.86178
50.06581
30.70448
20.77584
17.16109
13.80385
DFILE 2s'_carbon.als
COMNT
DATIM 10-03-2020 13:02:08
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 275
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM



DFILE 2t_proton.als
COMNT
DATIM 03-04-2020 12:45:38
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36



(2*S*,3*R*)-**2t**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2t_carbon.als
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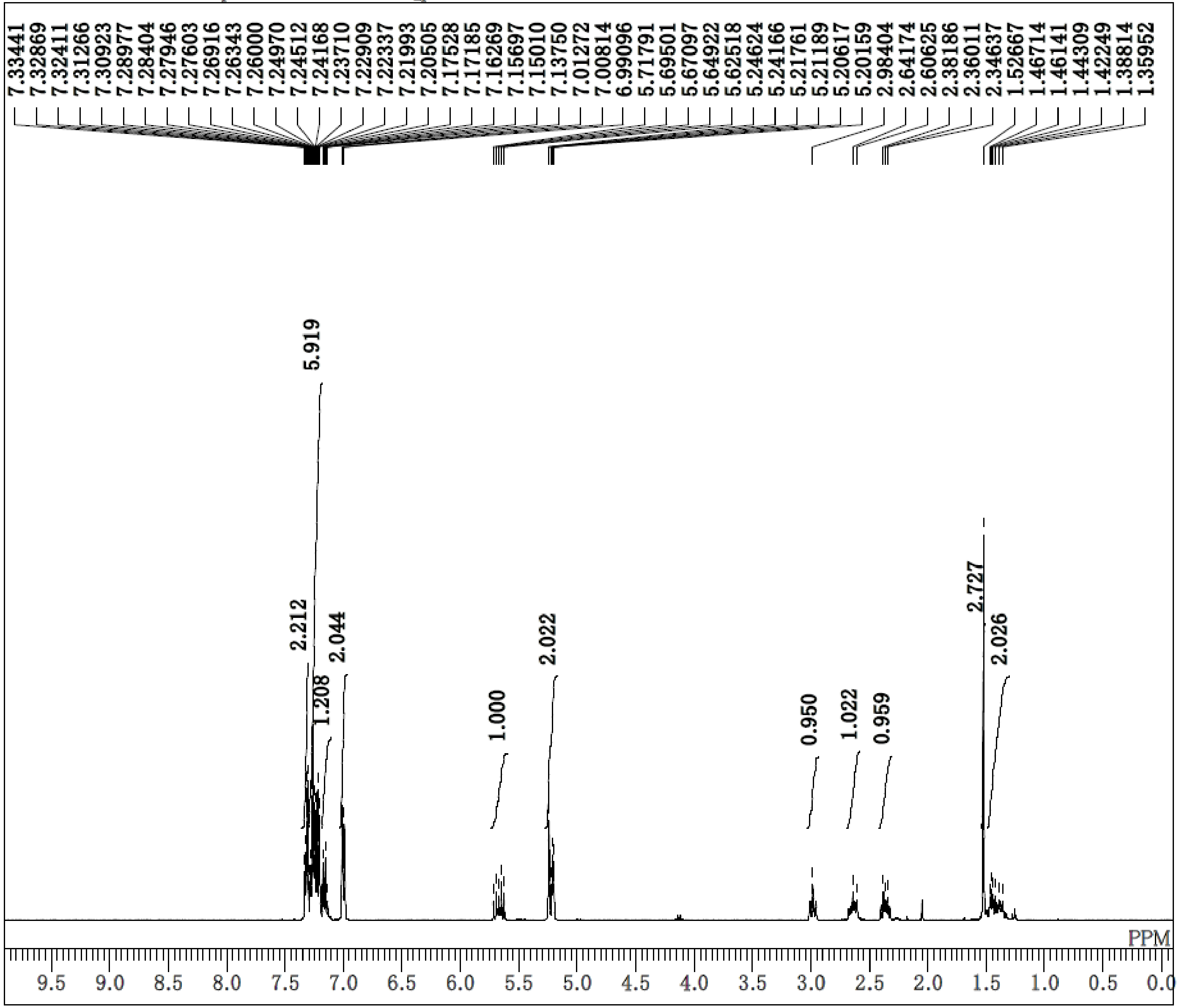
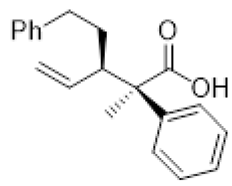
142.22766
141.51234
136.74354
128.46490
128.25507
128.19785
126.92935
126.62414
125.72761
118.59348
77.32428
77.00000
76.67572
53.69964
51.12449
34.29062
32.66923
17.73334
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2t_carbon.als
COMNT
DATIM 03-04-2020 12:34:33
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 184
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

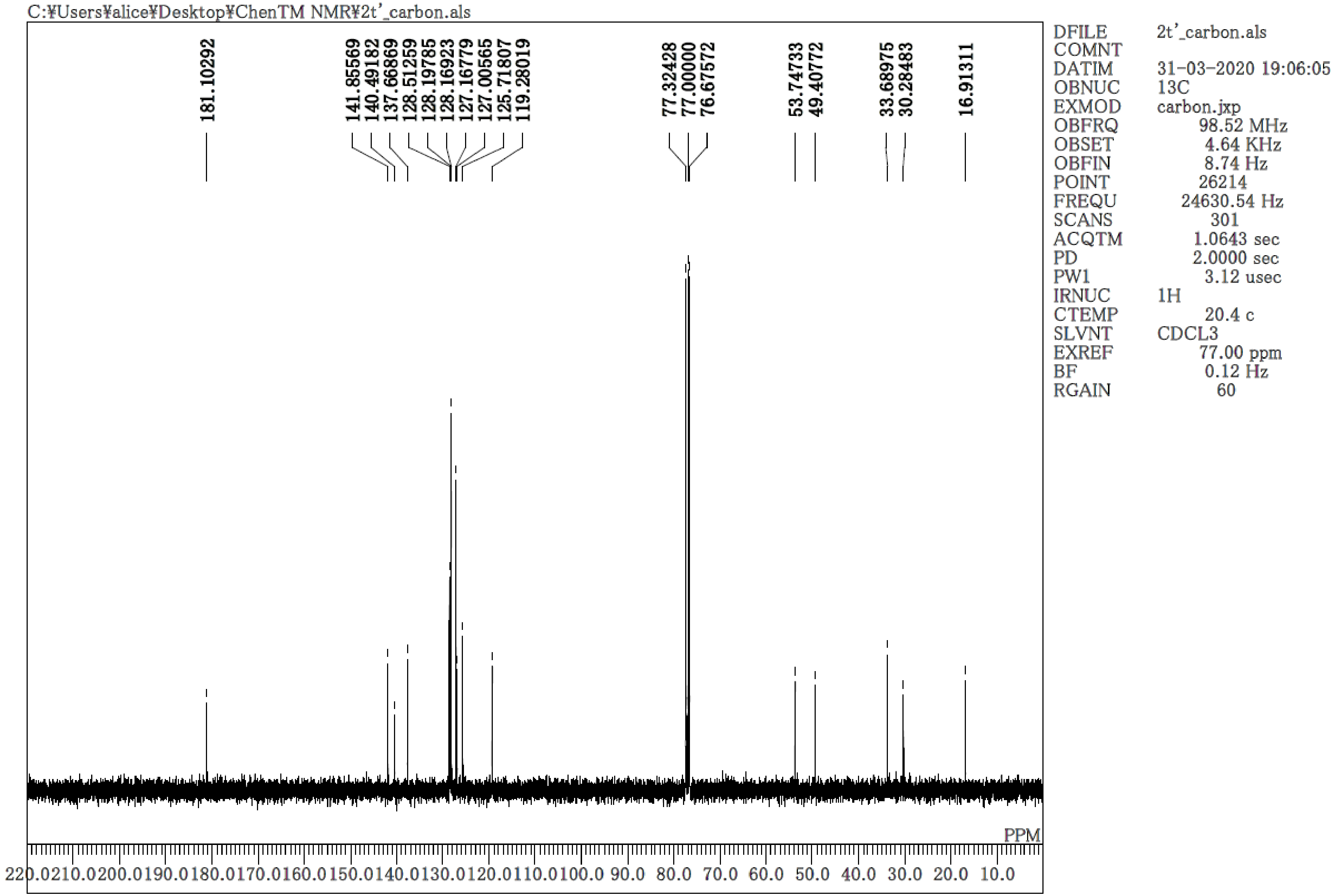
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2t'_proton.als



DFILE	2t'_proton.als
COMNT	
DATIM	31-03-2020 20:09:41
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



(2*R*,3*R*)-**2t**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2t'_carbon.als
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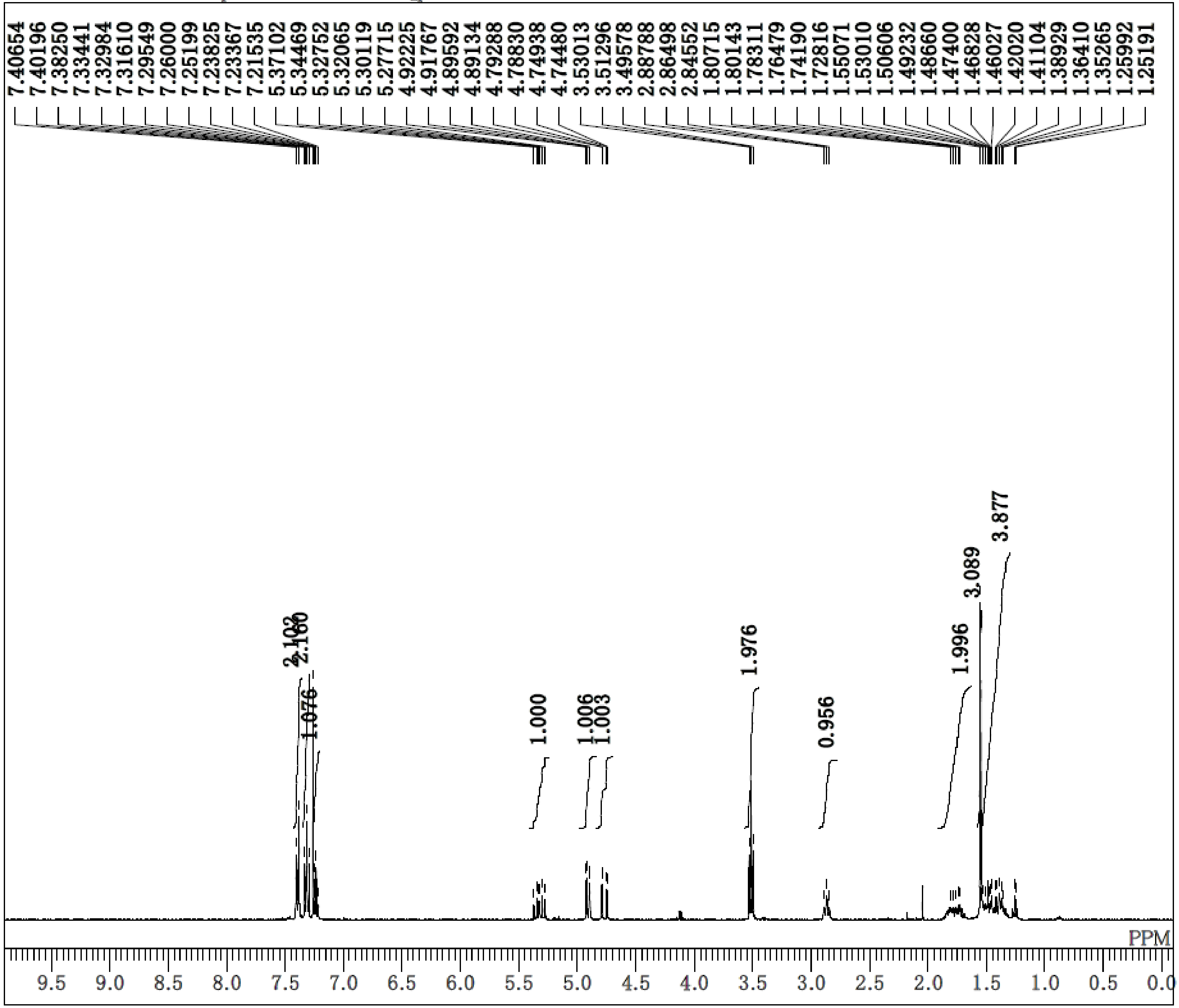
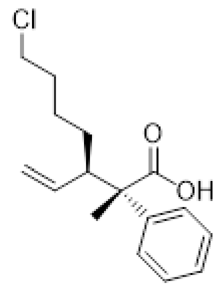
141.85569
140.49182
137.66869
128.51259
128.19785
128.16923
127.16779
127.00565
125.71807
119.28019
77.32428
77.00000
76.67572
53.74733
49.40772
33.68975
30.28483
16.91311
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2t'_carbon.als
COMNT
DATIM 31-03-2020 19:06:05
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 301
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

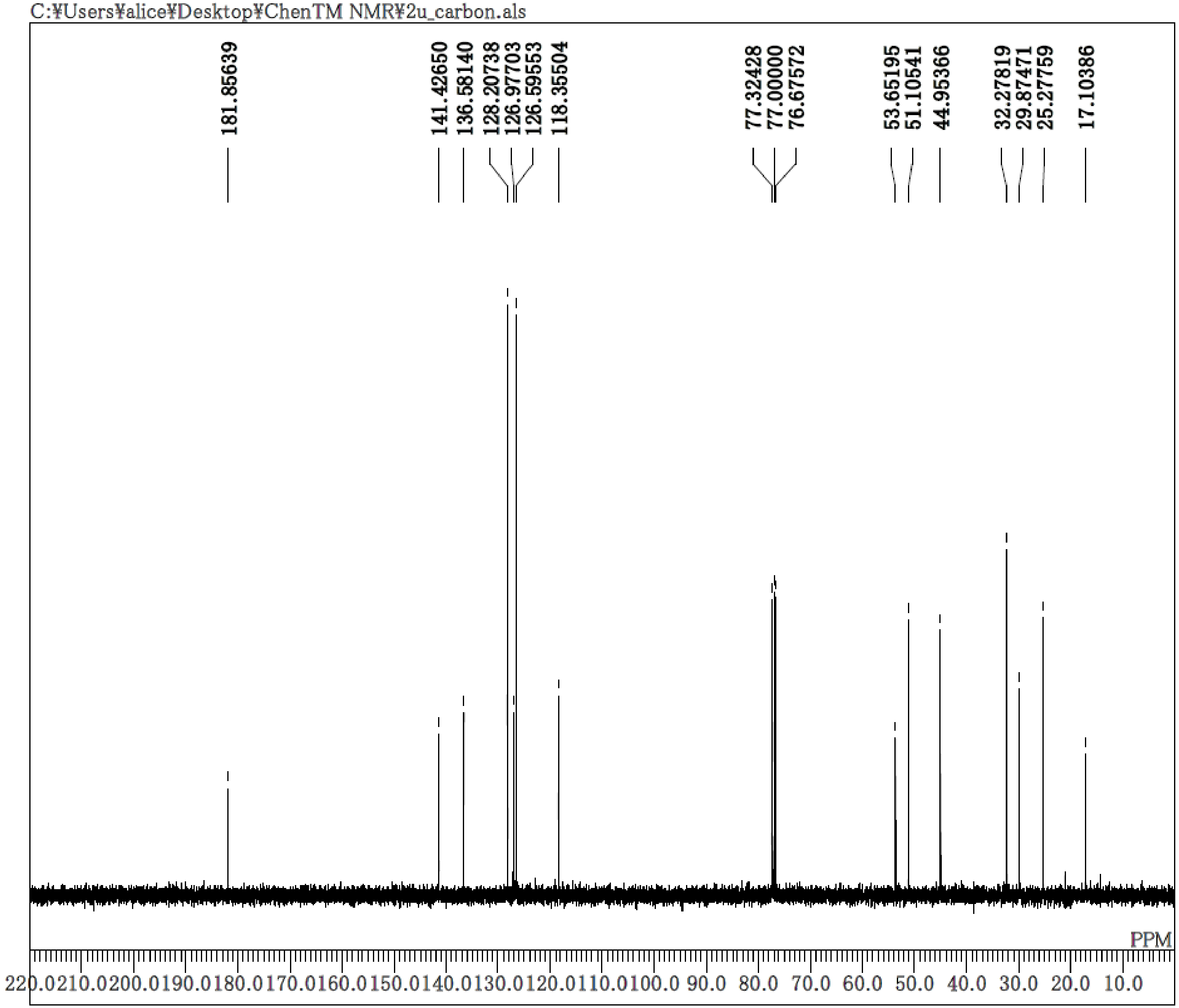
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2u_proton.als



DFILE 2u_proton.als
COMNT
DATIM 09-09-2019 18:24:23
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 46

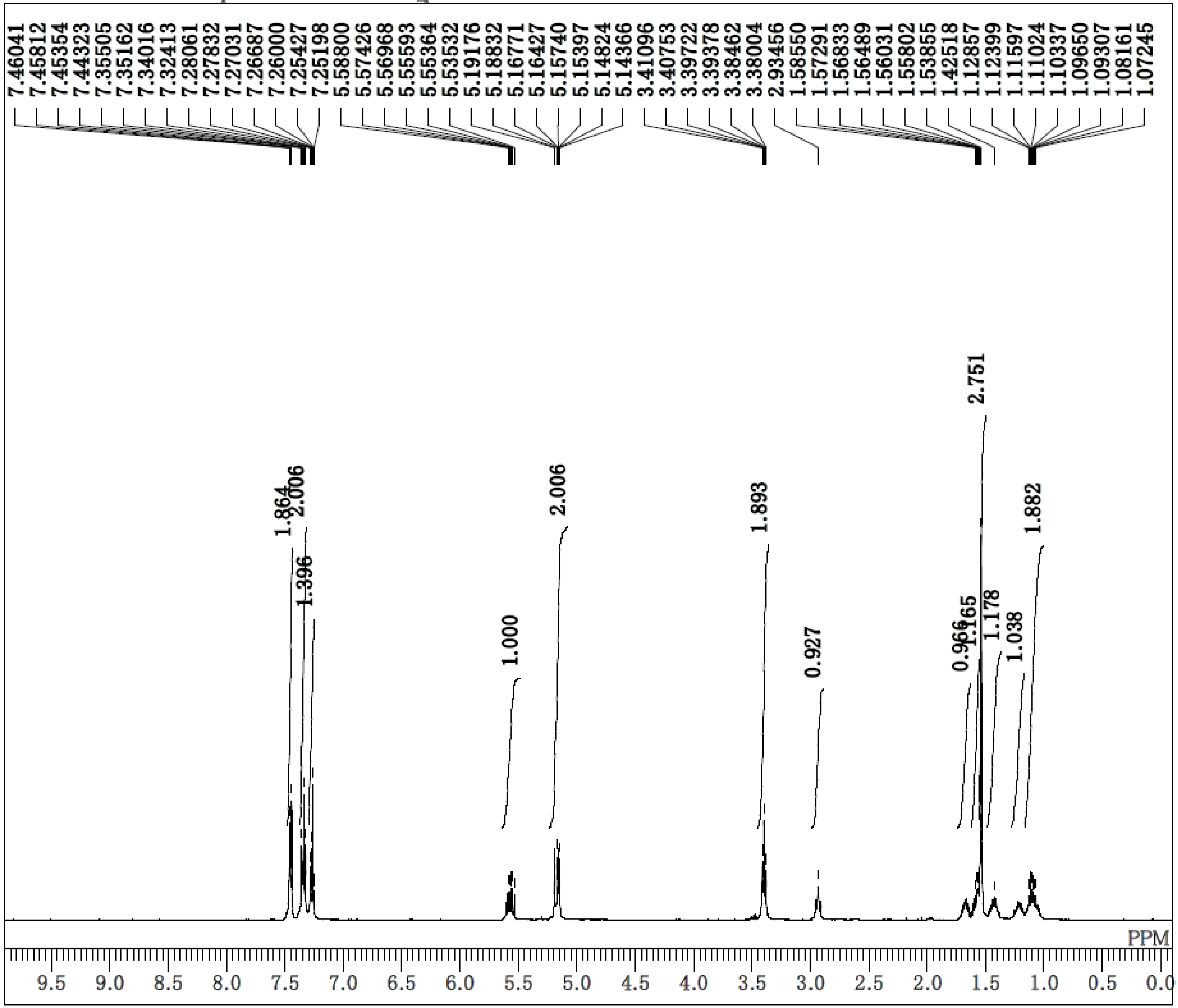


(2*S*,3*R*)-**2u**

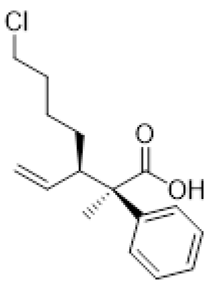


DFILE	2u_carbon.als
COMNT	
DATIM	09-09-2019 16:15:37
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	69
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

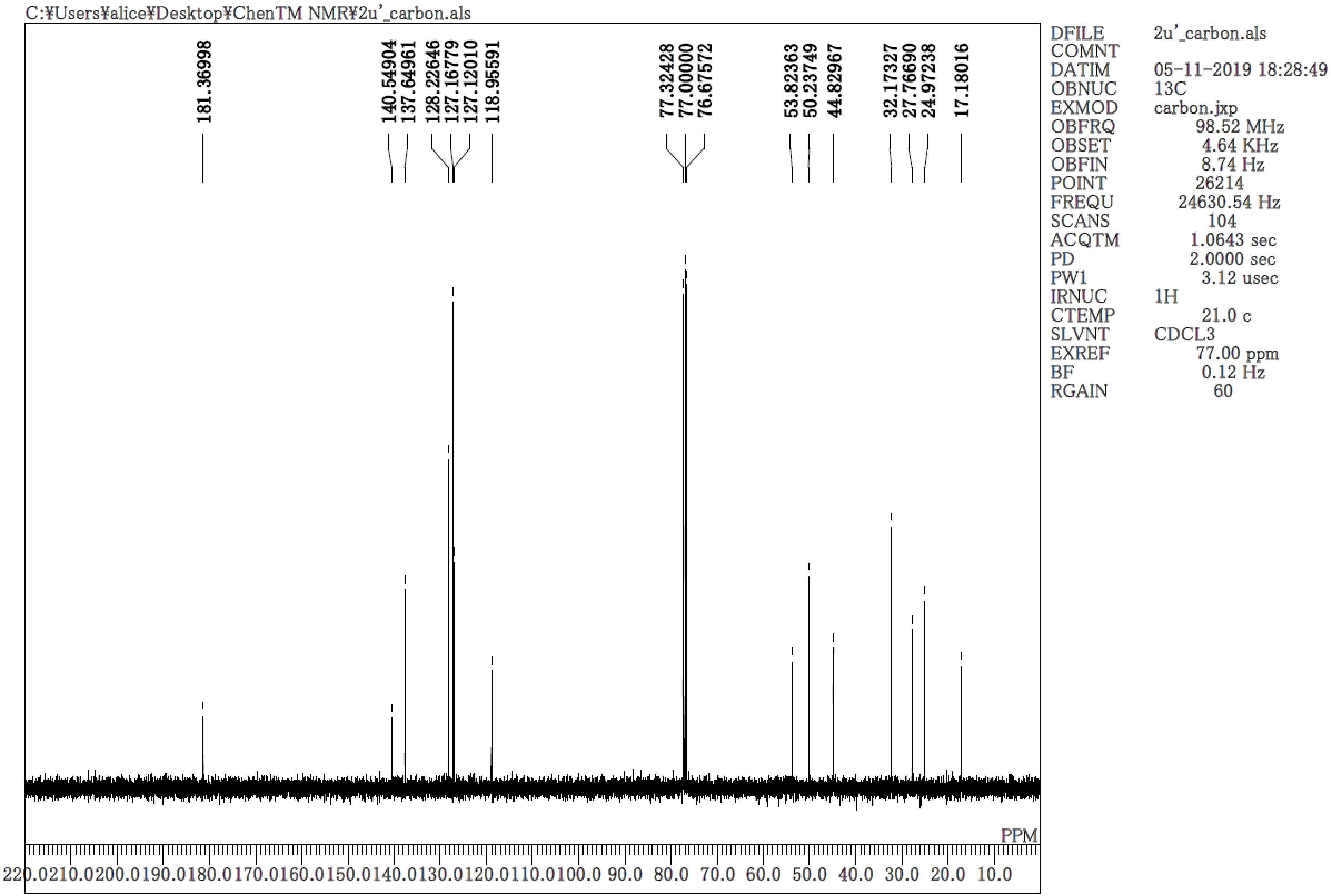
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2u'_proton.als

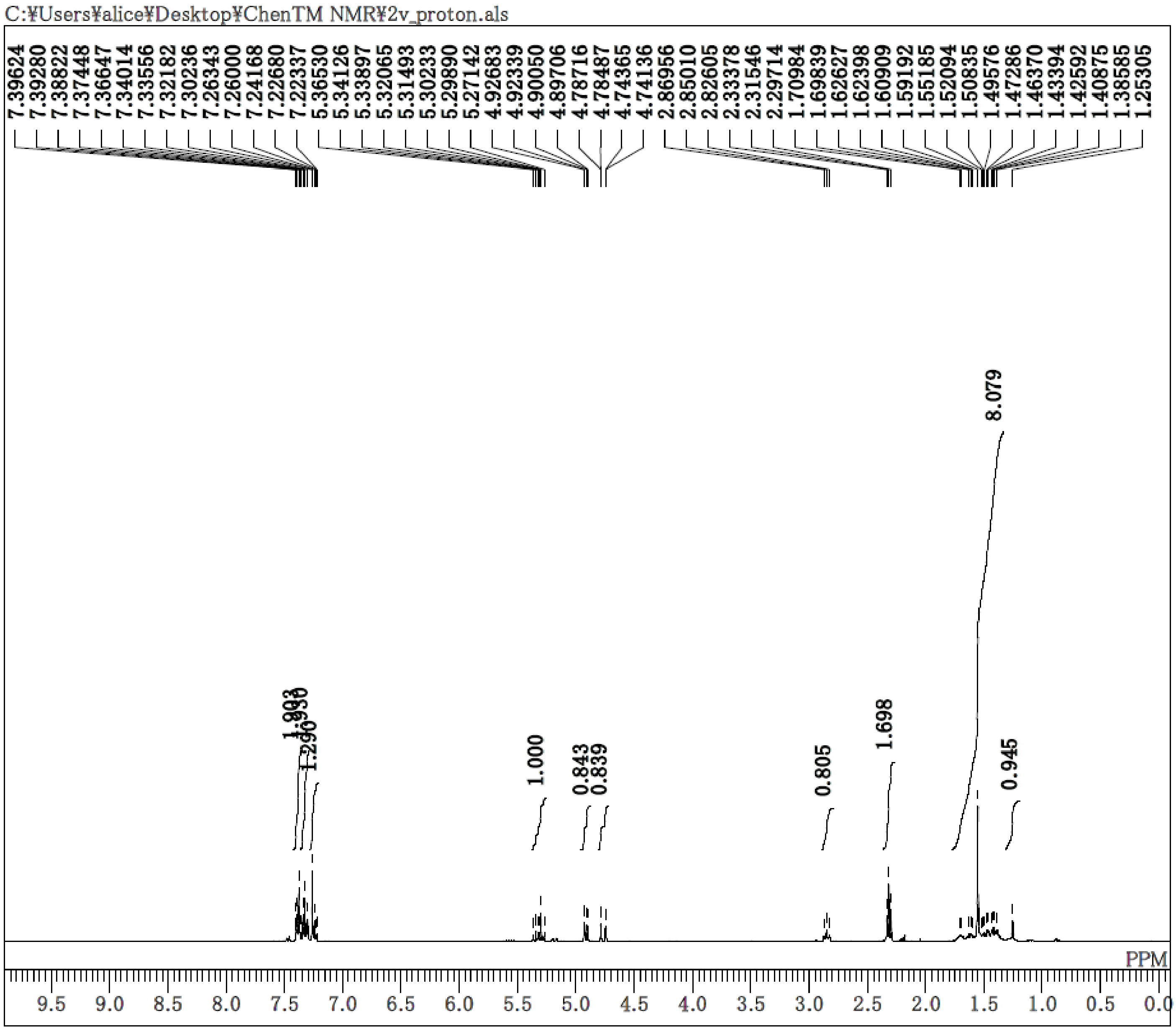


DFILE	2u'_proton.als
COMNT	
DATIM	2019-11-05 16:05:23
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

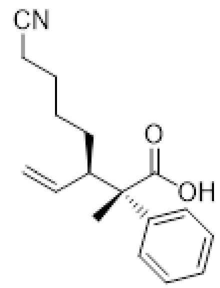


(2*R*,3*R*)-**2u**

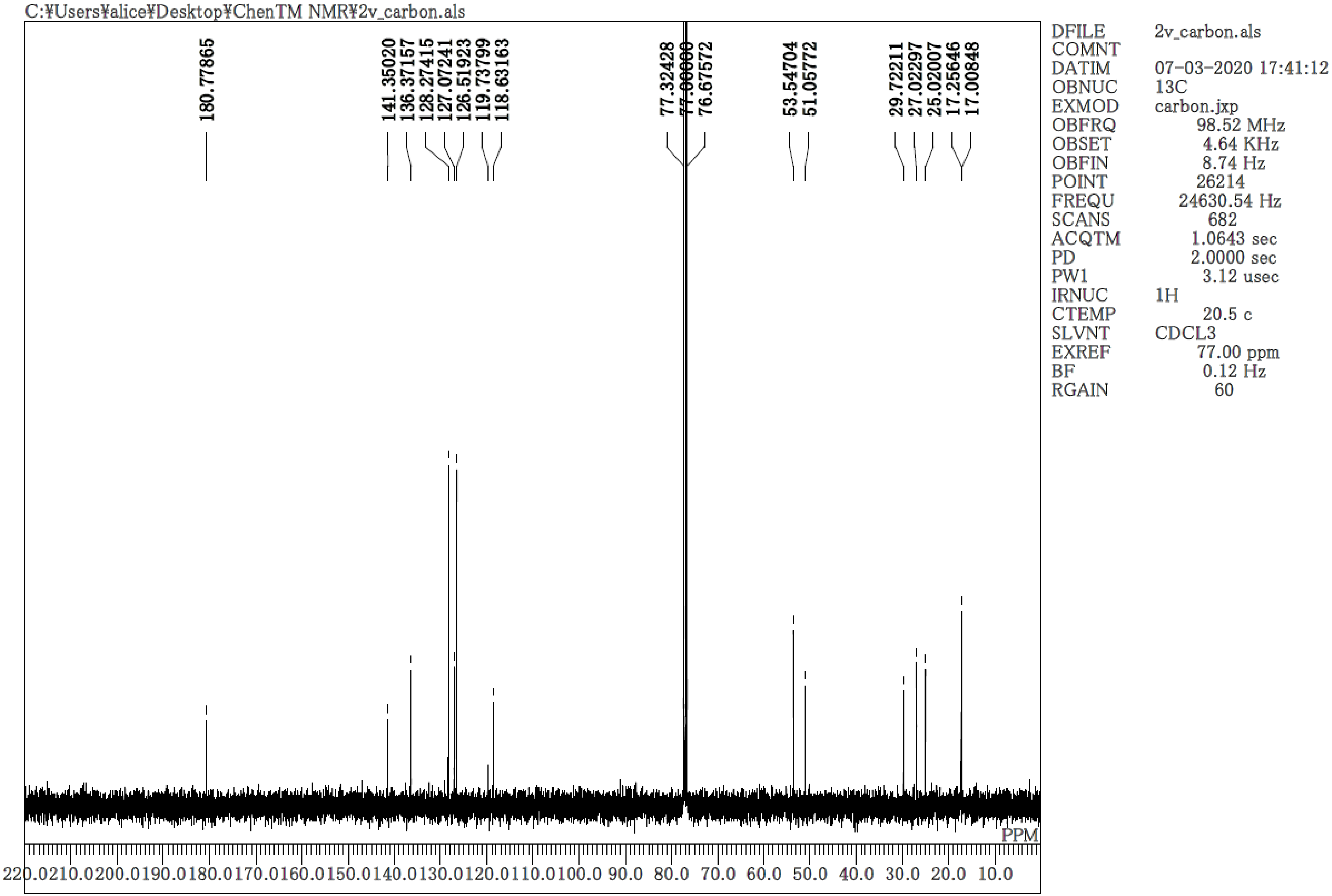

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2u'_carbon.als
181.36998
140.54904
137.64961
128.22646
127.16779
127.12010
118.95591
77.32428
77.00000
76.67572
53.82363
50.23749
44.82967
32.17327
27.76690
24.97238
17.18016
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2u'_carbon.als
COMNT
DATIM 05-11-2019 18:28:49
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 104
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



DFILE	2v_proton.als
COMNT	
DATIM	07-03-2020 16:22:55
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



(2*S*,3*R*)-**2v**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2v_carbon.als
180.77865
141.35020
136.37157
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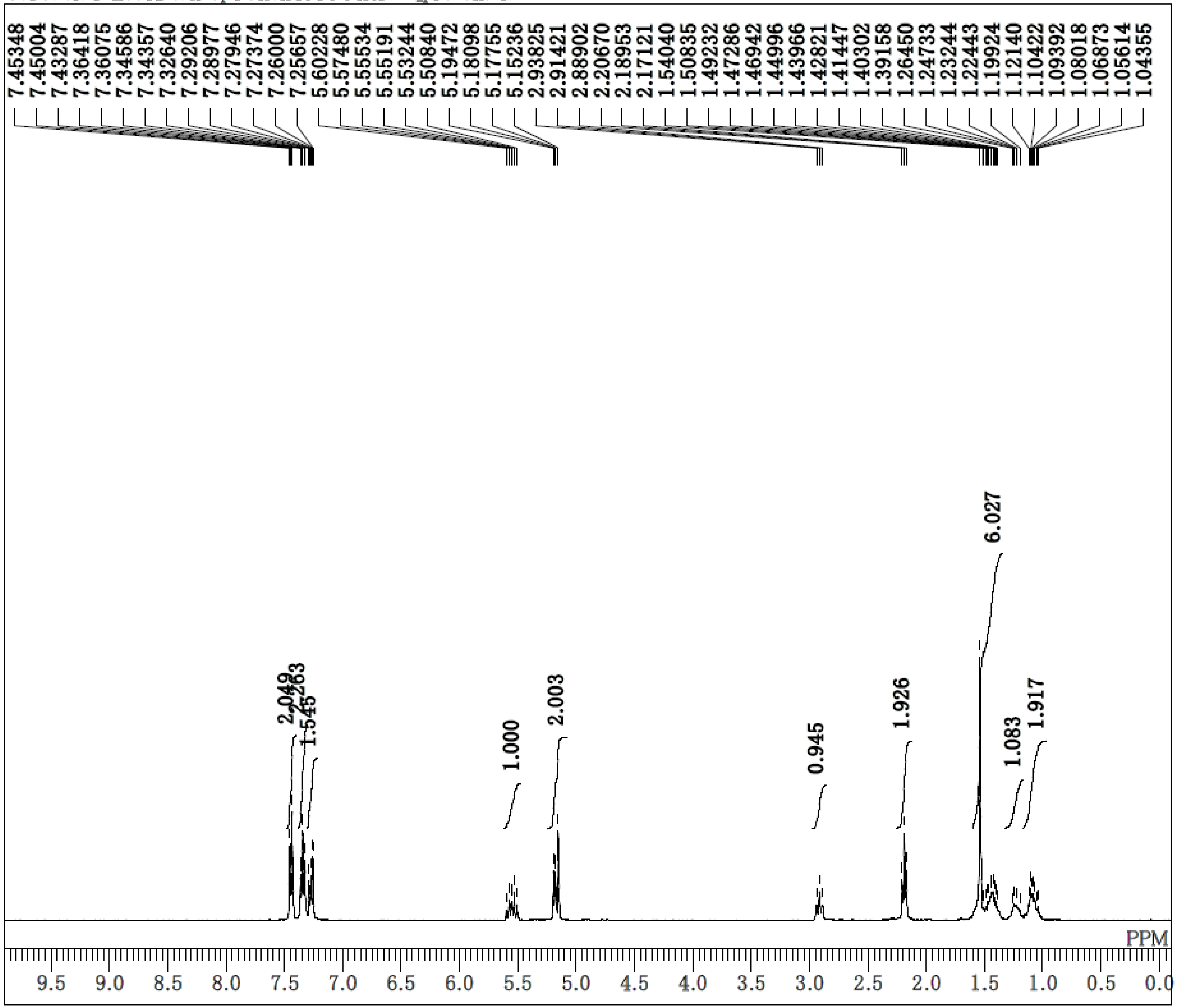
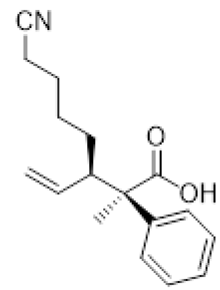
127.07241
126.51923
119.73799
118.63163
77.32428
77.00000
76.67572
53.54704
51.05772
29.72211
27.02297
25.02007
17.25646
17.00848
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2v_carbon.als
COMNT
DATIM 07-03-2020 17:41:12
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 682
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

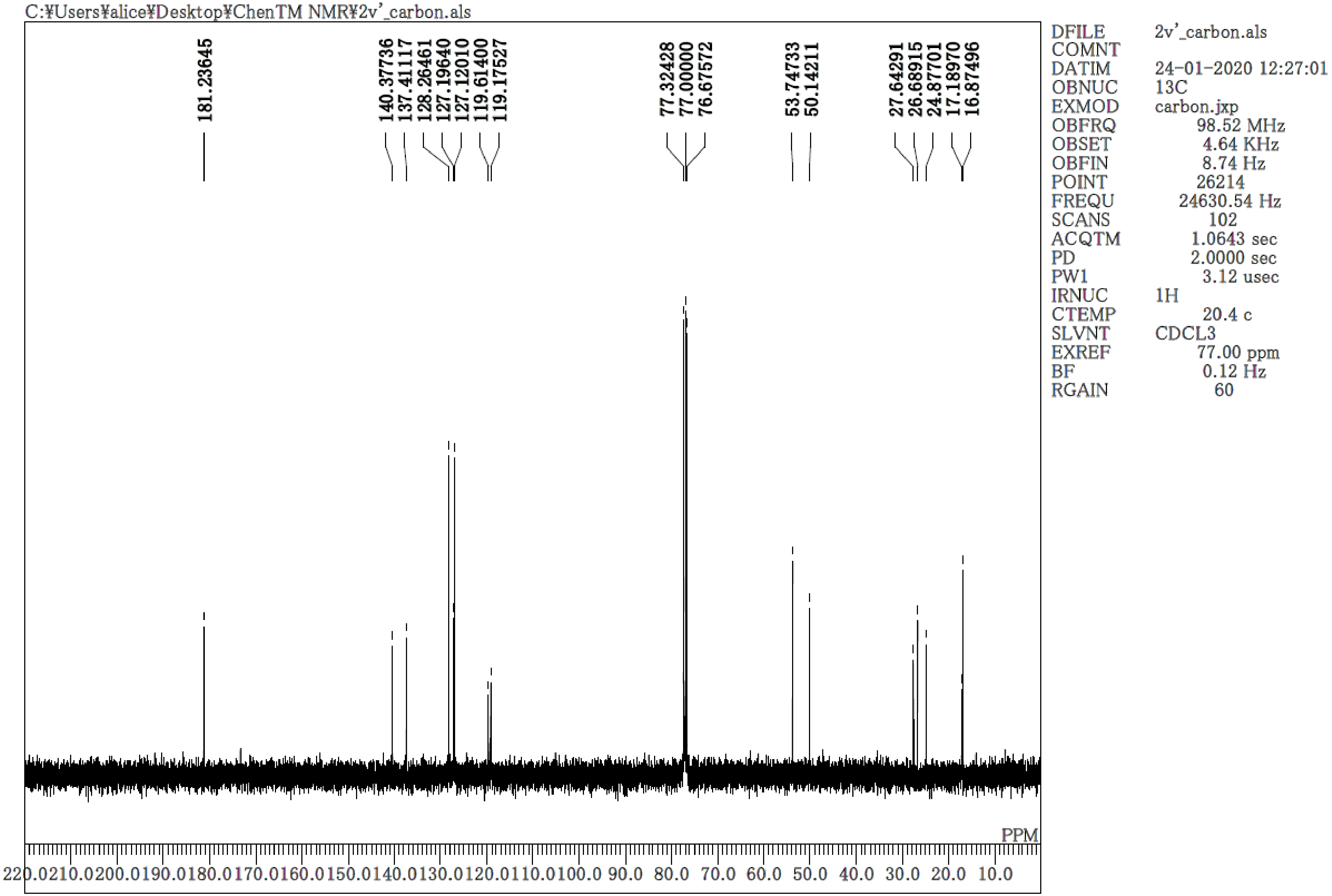
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2v'_proton.als



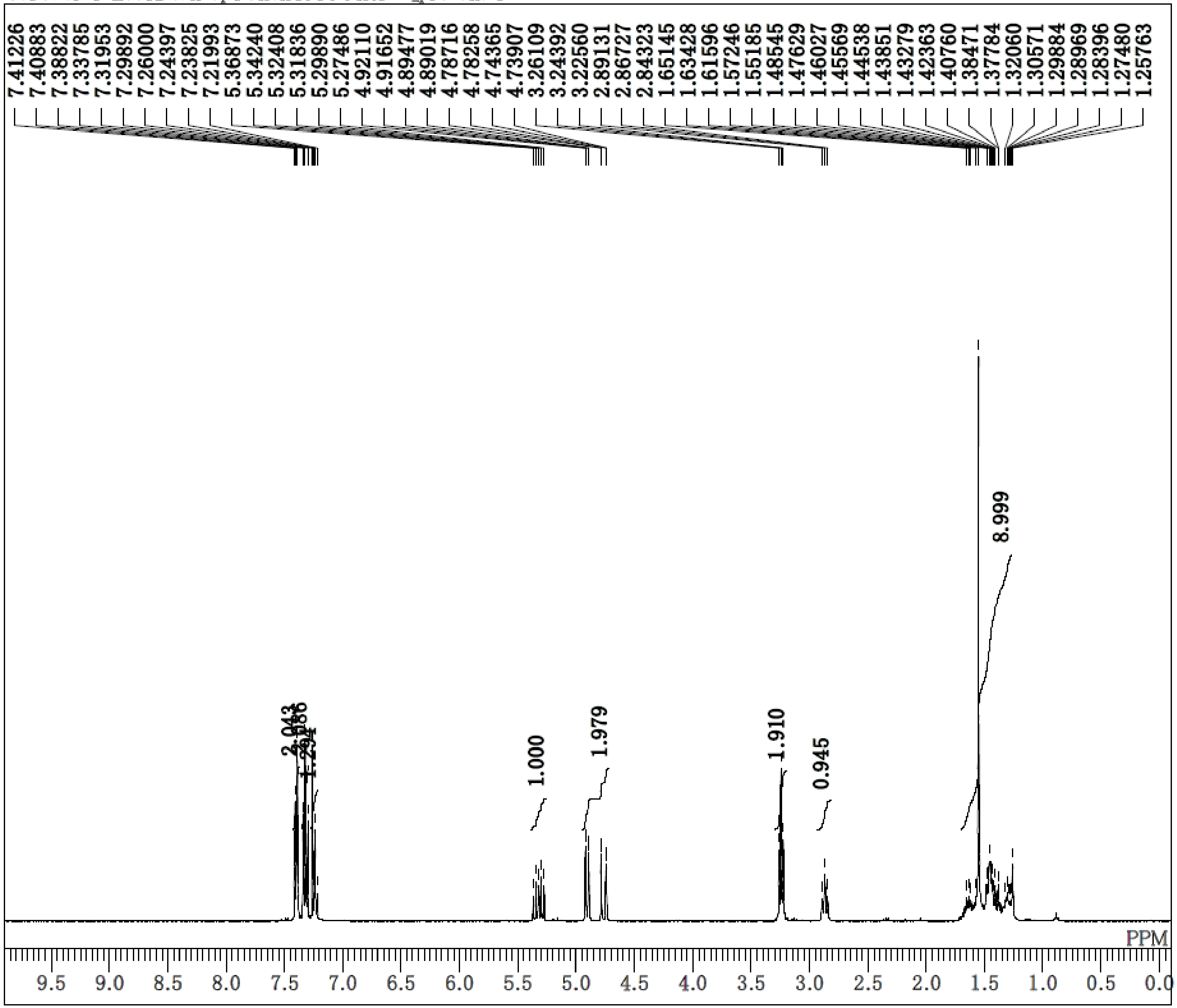
DFILE	2v'_proton.als
COMNT	
DATIM	24-01-2020 12:21:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



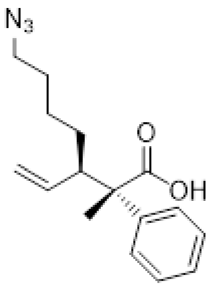
(2*R*,3*R*)-**2v**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2v'_carbon.als
181.23645
140.37736
137.41117
128.26461
127.19640
127.12010
119.61400
119.17527
77.32428
77.00000
76.67572
53.74733
50.14211
27.64291
26.68915
24.87701
17.18970
16.87496
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2v'_carbon.als
COMNT
DATIM 24-01-2020 12:27:01
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 102
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2w_proton.als

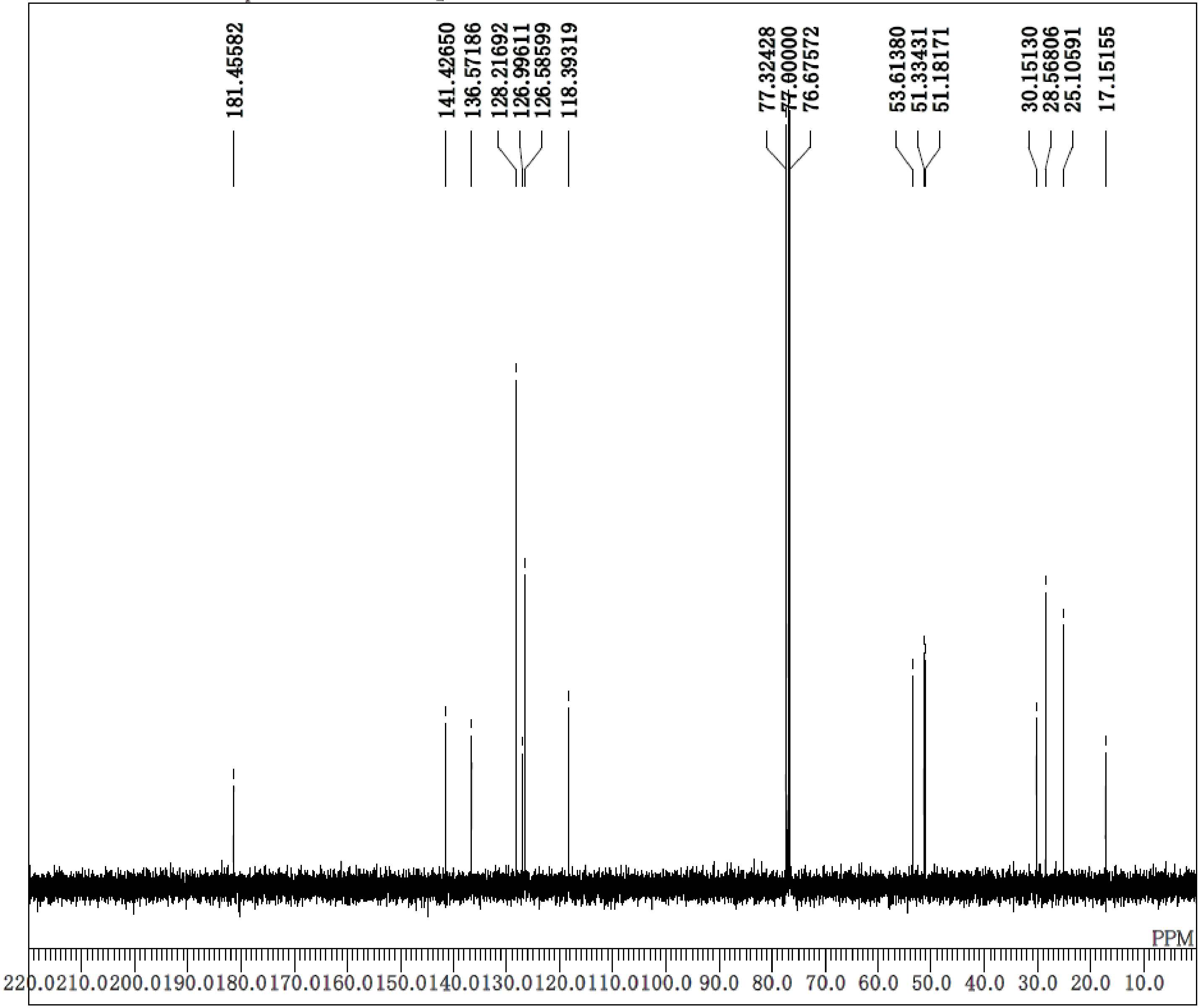


DFILE	2w_proton.als
COMNT	
DATIM	26-11-2019 19:40:58
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



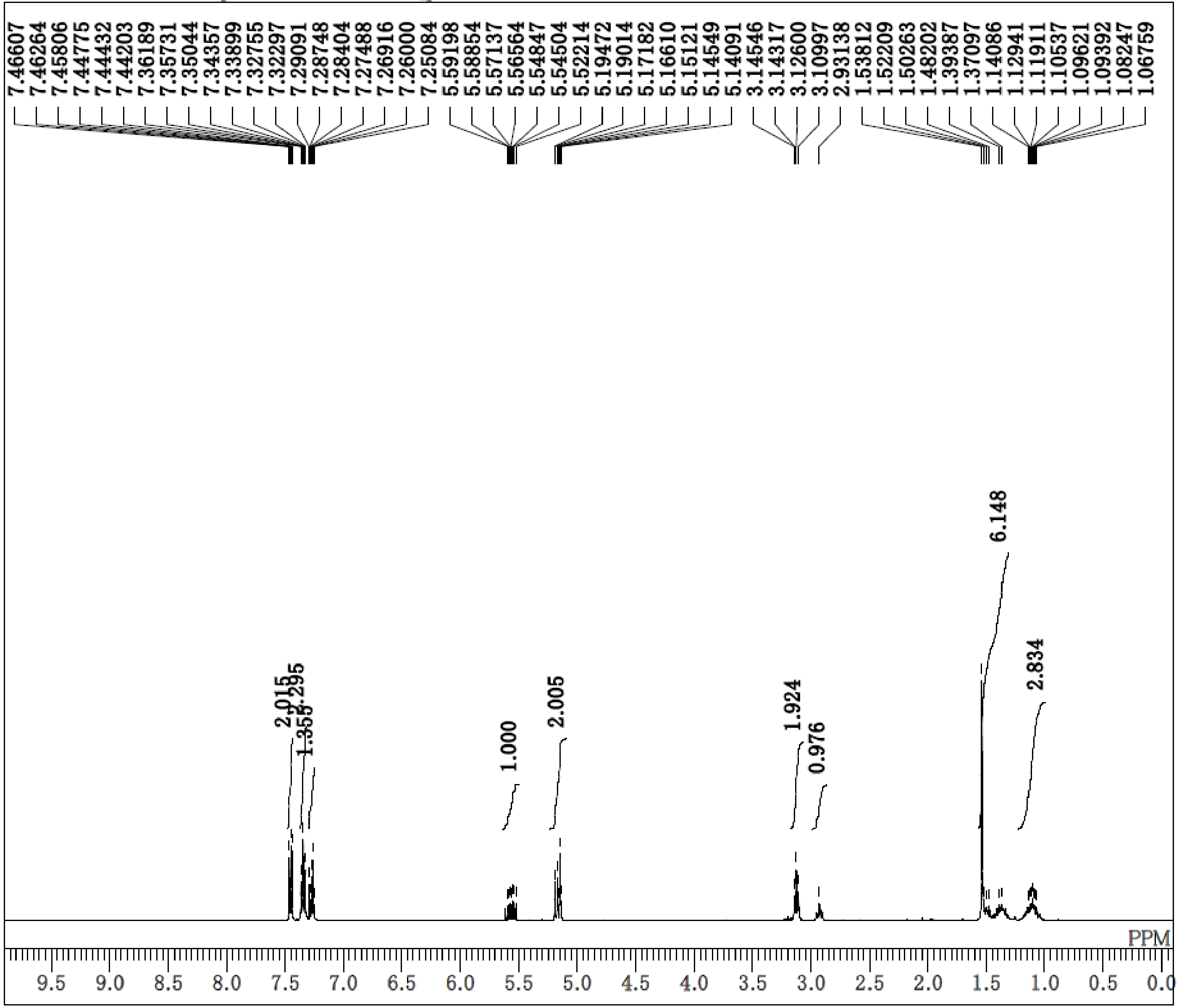
(2*S*,3*R*)-**2w**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2w_carbon.als

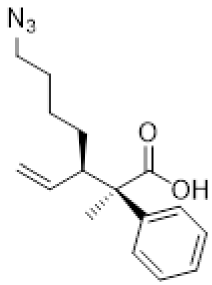


DFILE	2w_carbon.als
COMNT	
DATIM	26-11-2019 20:06:03
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	108
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2w'_proton.als

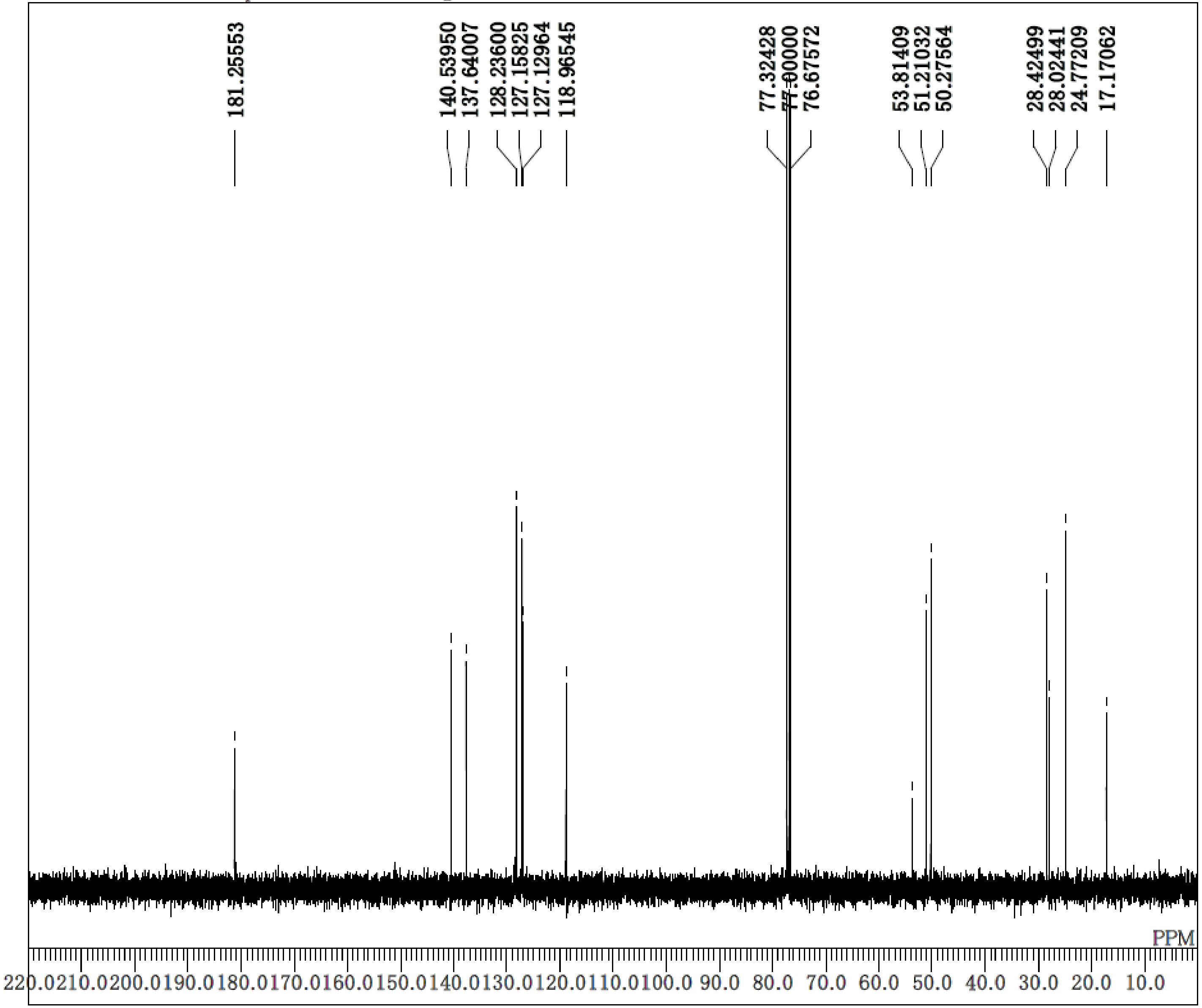


DFILE 2w'_proton.als
COMNT
DATIM 26-11-2019 19:12:37
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36

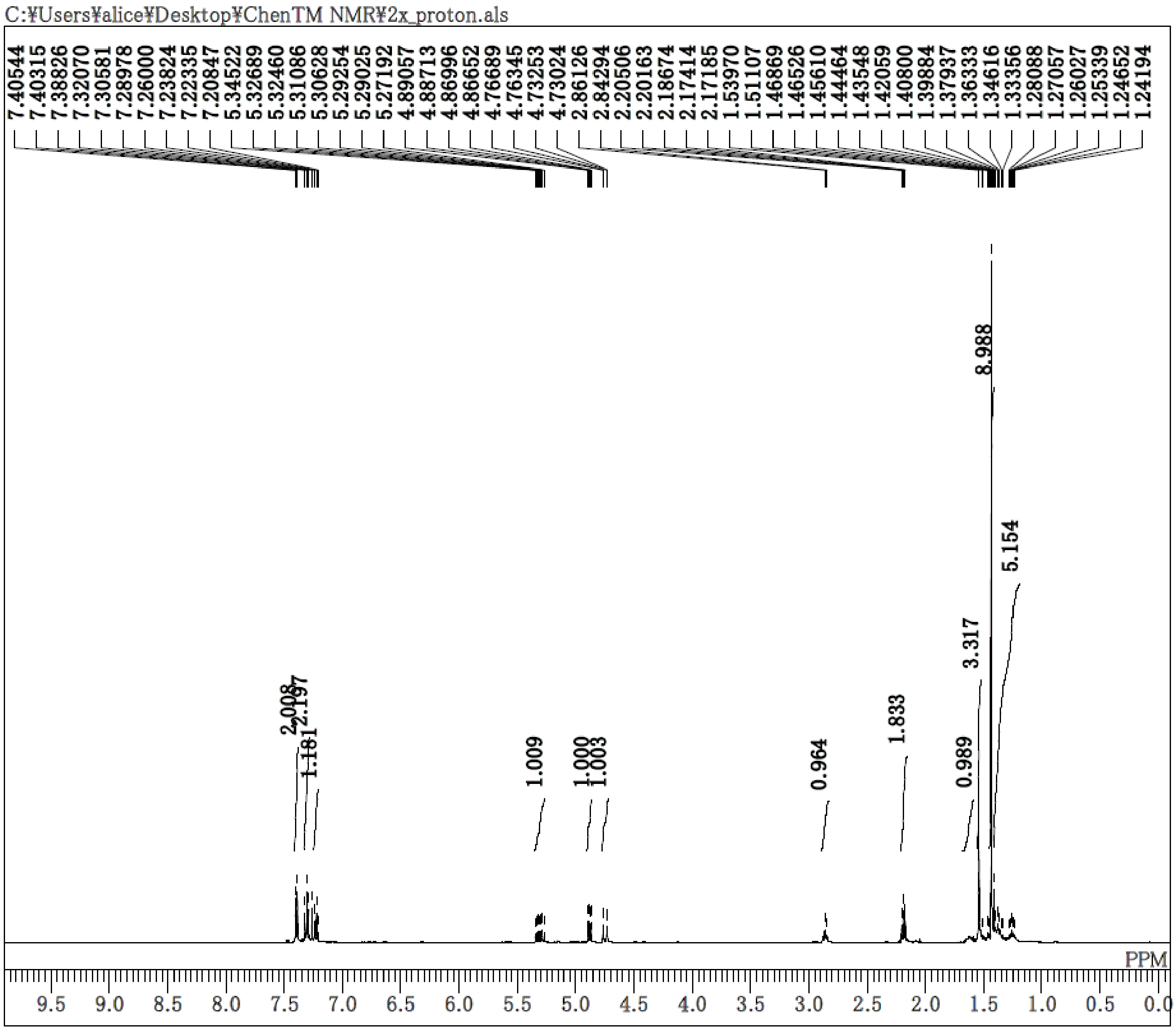


(2*R*,3*R*)-**2w**

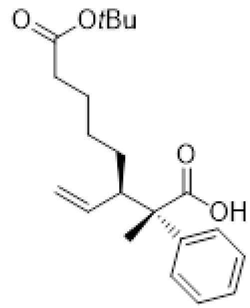
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2w'_carbon.als



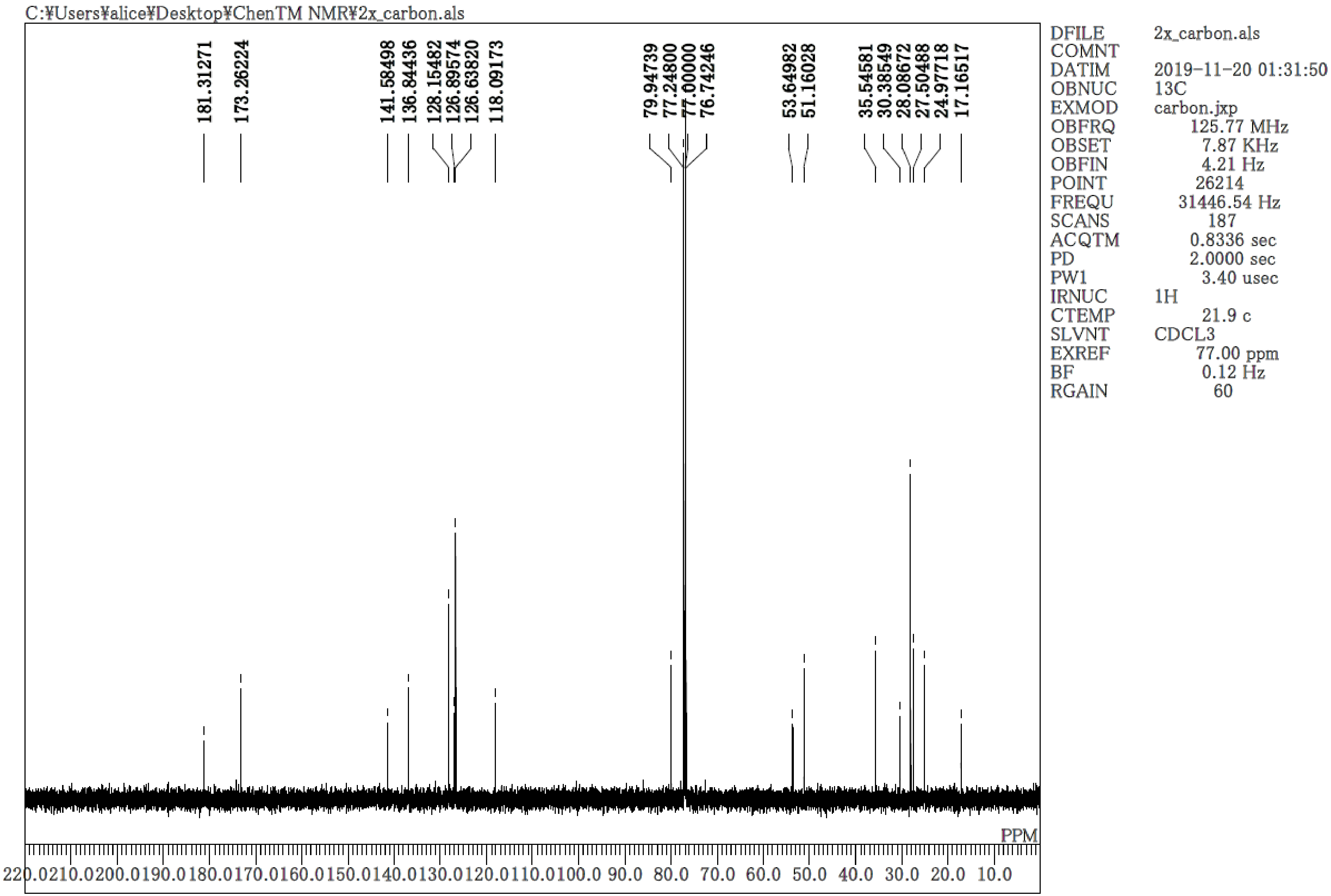
DFILE	2w'_carbon.als
COMNT	
DATIM	26-11-2019 19:46:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	108
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



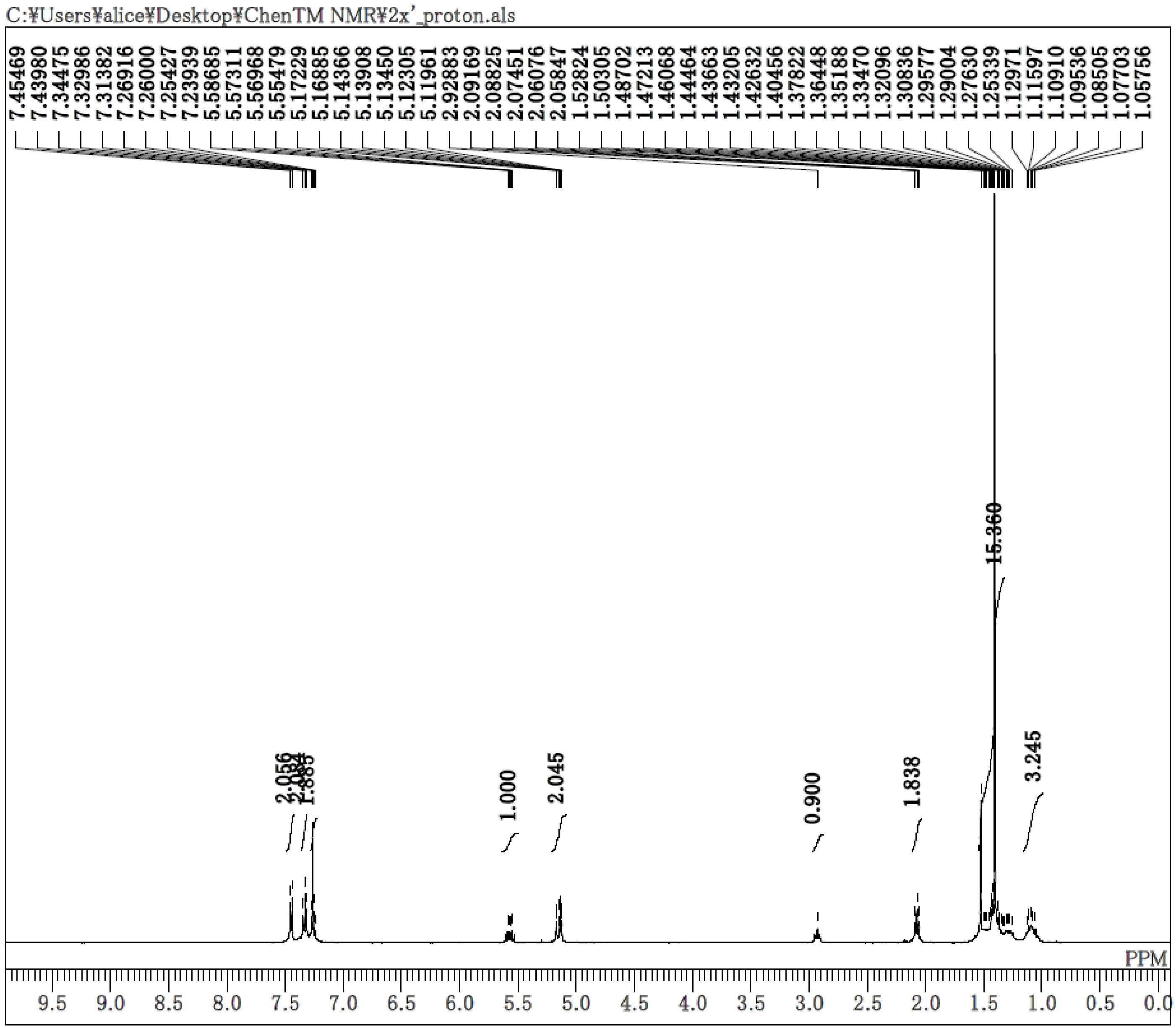
DFILE 2x_proton.als
COMNT
DATIM 2019-11-20 01:26:58
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



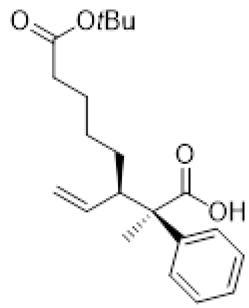
(2*S*,3*R*)-**2x**



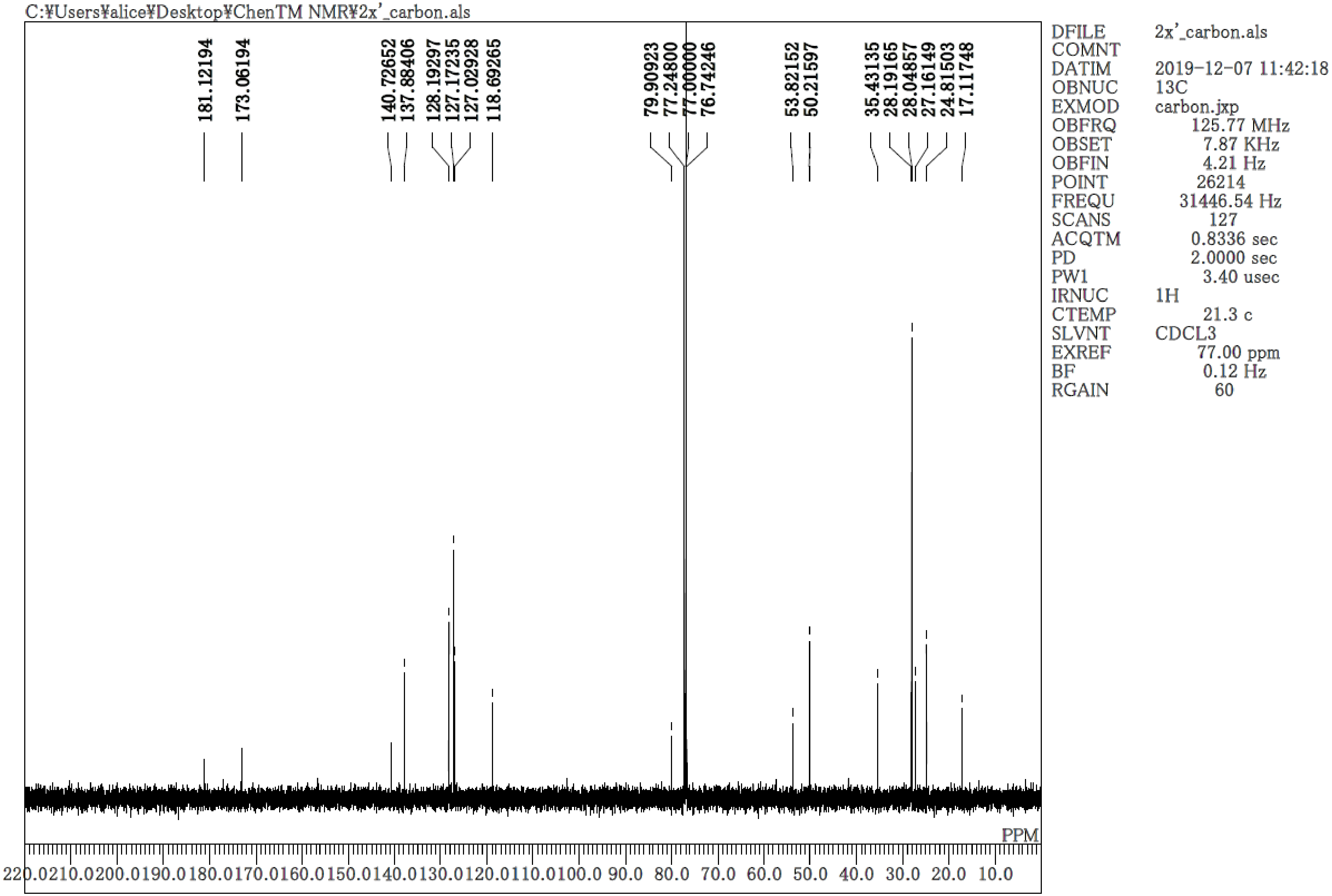
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2x_carbon.als
181.31271
173.26224
141.58498
136.84436
128.15482
126.89574
126.63820
118.09173
79.94739
77.24800
77.00000
76.74246
53.64982
51.16028
35.54581
30.38549
28.08672
27.50488
24.97718
17.16517
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2x_carbon.als
COMNT
DATIM 2019-11-20 01:31:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 187
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



DFILE	2x'_proton.als
COMNT	
DATIM	2019-12-07 11:19:16
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



(2*R*,3*R*)-**2x**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2x'_carbon.als
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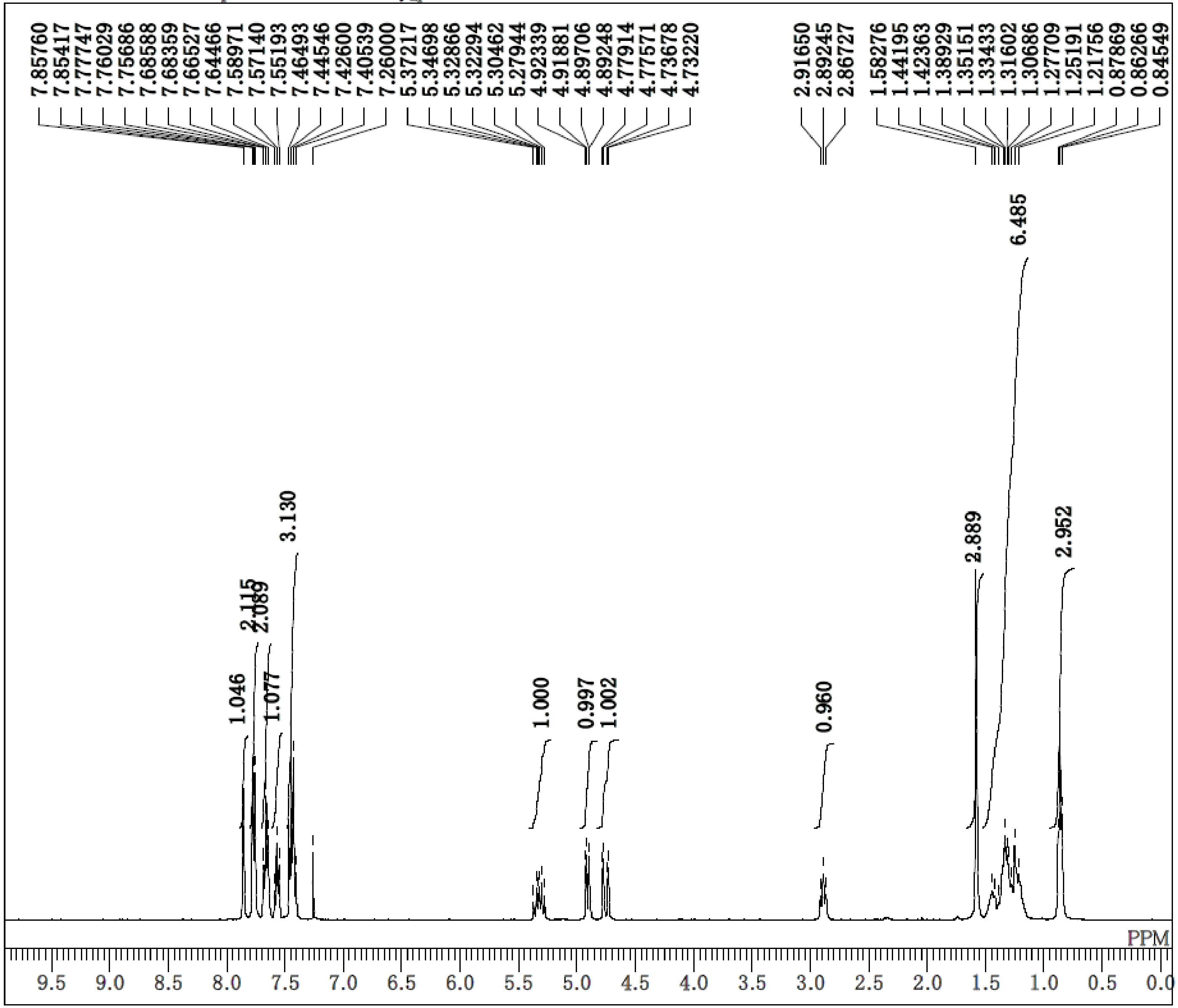
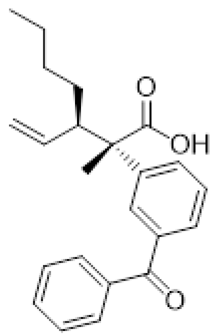
137.88406
128.19297
127.17235
127.02928
118.69265
79.90923
77.24800
77.00000
76.74246
53.82152
50.21597
35.43135
28.19165
28.04857
27.16149
24.81503
17.11748
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2x'_carbon.als
COMNT
DATIM 2019-12-07 11:42:18
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 127
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

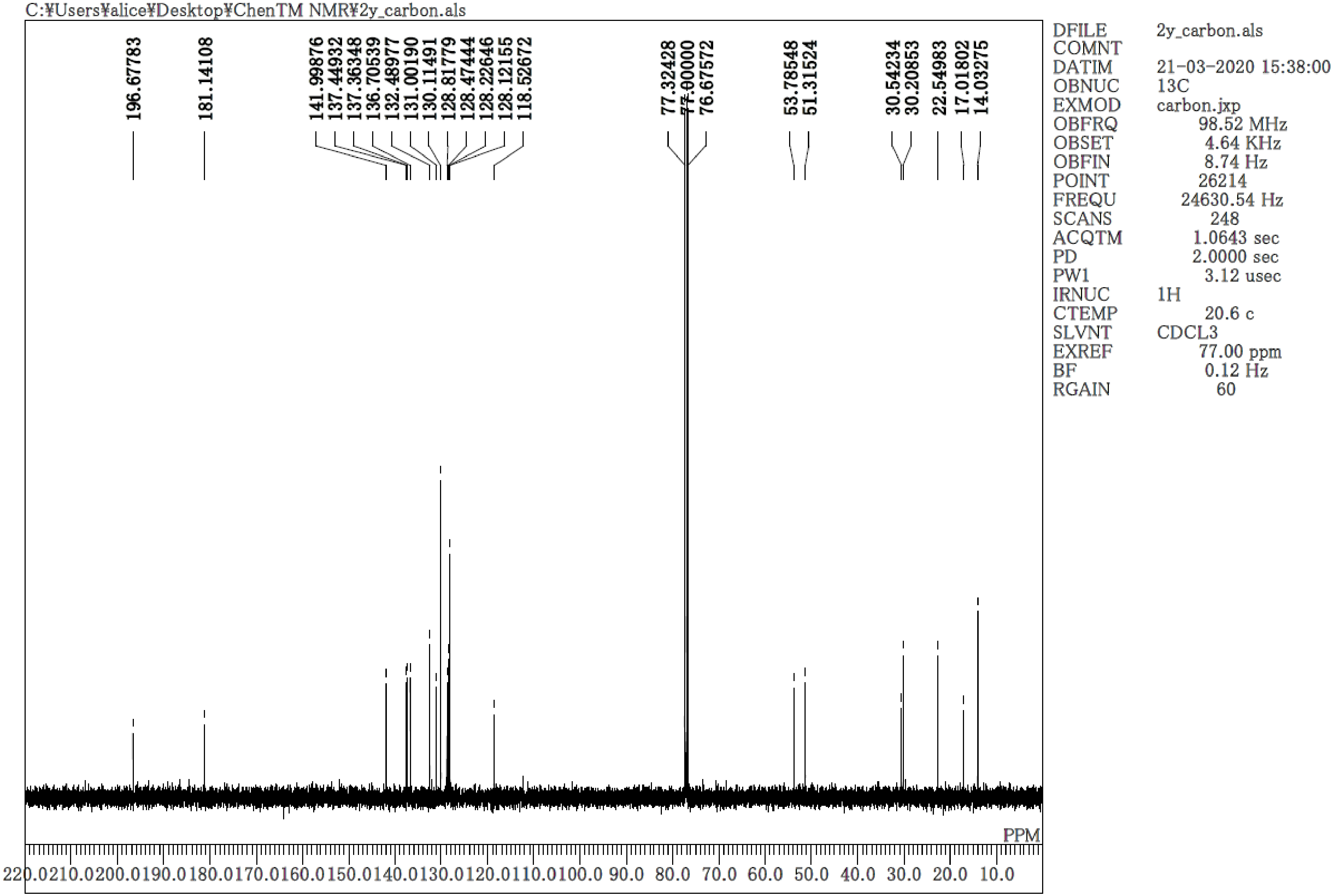
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2y_proton.als



DFILE	2y_proton.als
COMNT	
DATIM	21-03-2020 15:35:59
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34



(2*S*,3*R*)-**2y**



C:¥Users¥alice¥Desktop¥ChenTM NMR¥2y_carbon.als
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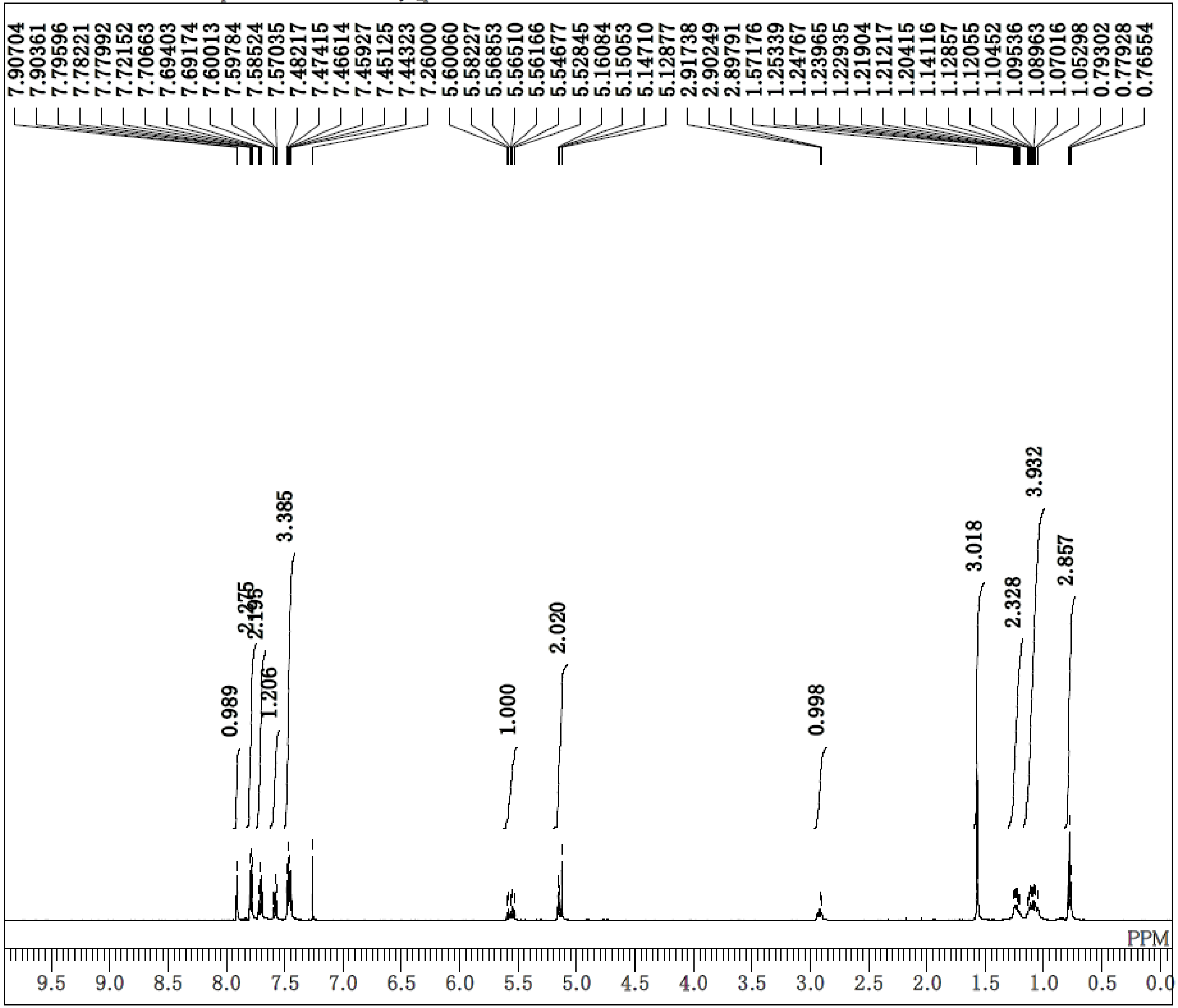
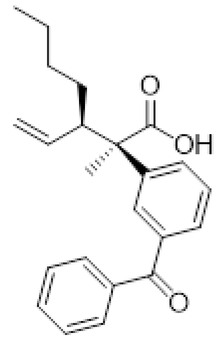
137.44932
137.36348
136.70539
132.48977
131.00190
130.11491
128.81779
128.47444
128.22646
128.12155
118.52672
77.32428
77.00000
76.67572
53.78548
51.31524
30.54234
30.20853
22.54983
17.01802
14.03275
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2y_carbon.als
COMNT
DATIM 21-03-2020 15:38:00
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 248
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

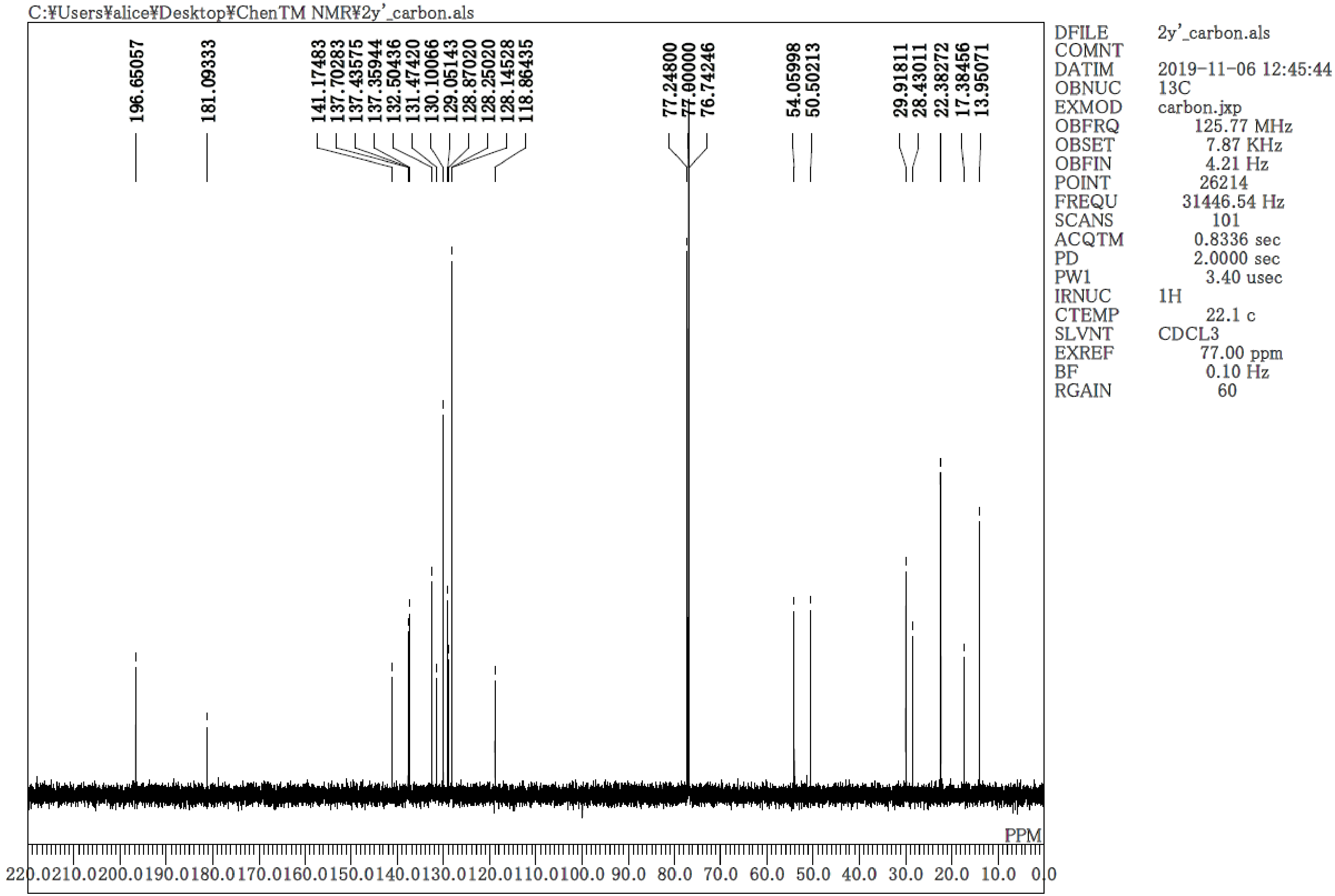
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2y'_proton.als



DFILE	2y'_proton.als
COMNT	
DATIM	2019-11-06 12:10:51
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.10 Hz
RGAIN	42

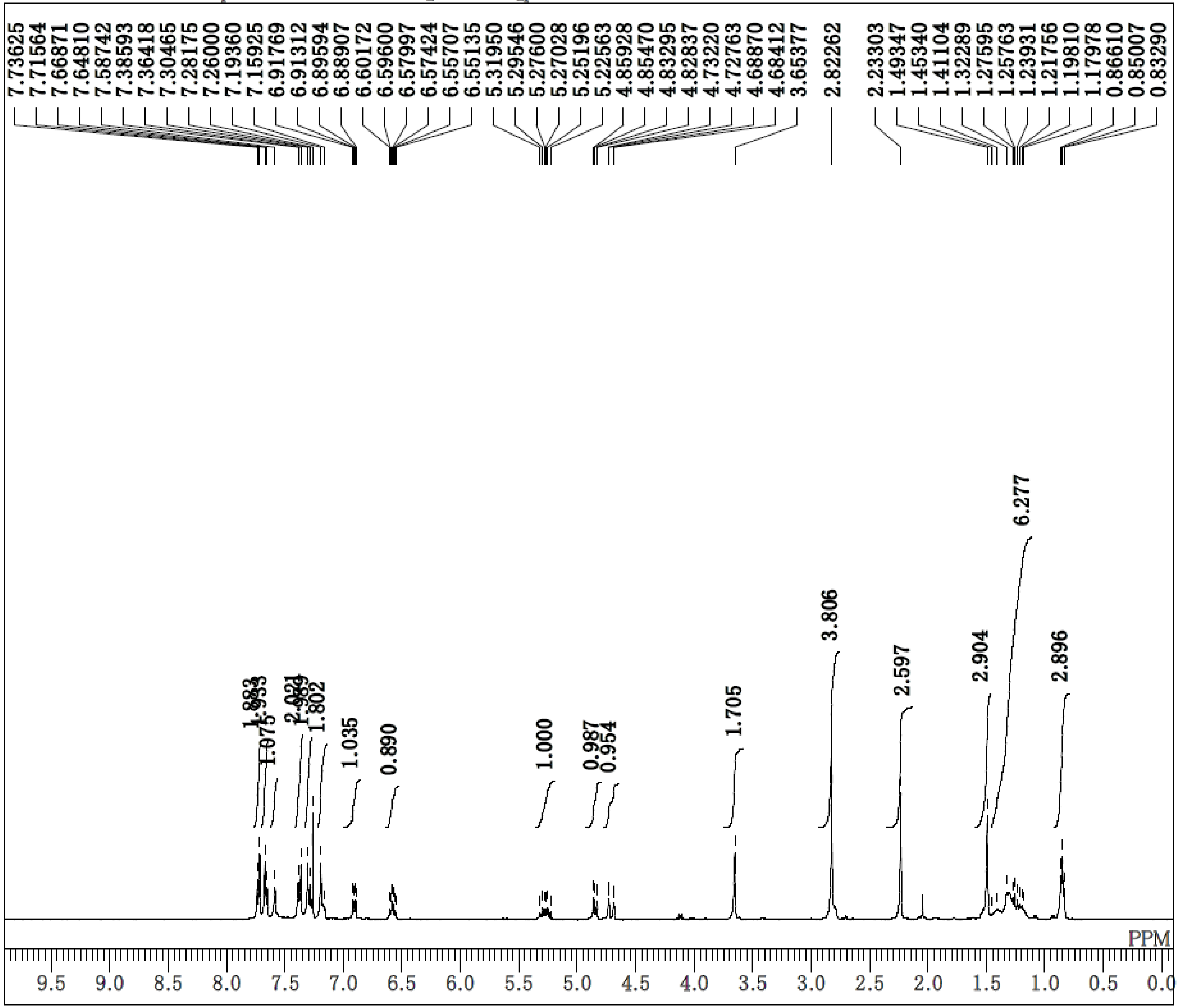


(2*R*,3*R*)-**2y**

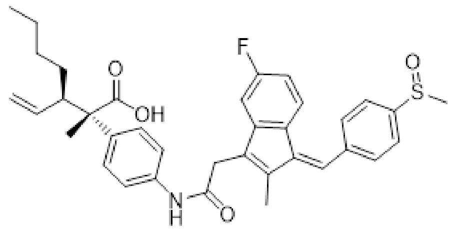


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2y'_carbon.als
196.65057
181.09333
141.17483
137.70283
137.43575
137.35944
132.50436
131.47420
130.10066
129.05143
128.87020
128.25020
128.14528
118.86435
77.24800
77.00000
76.74246
54.05998
50.50213
29.91811
28.43011
22.38272
17.38456
13.95071
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
DFILE 2y'_carbon.als
COMNT
DATIM 2019-11-06 12:45:44
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 101
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 60

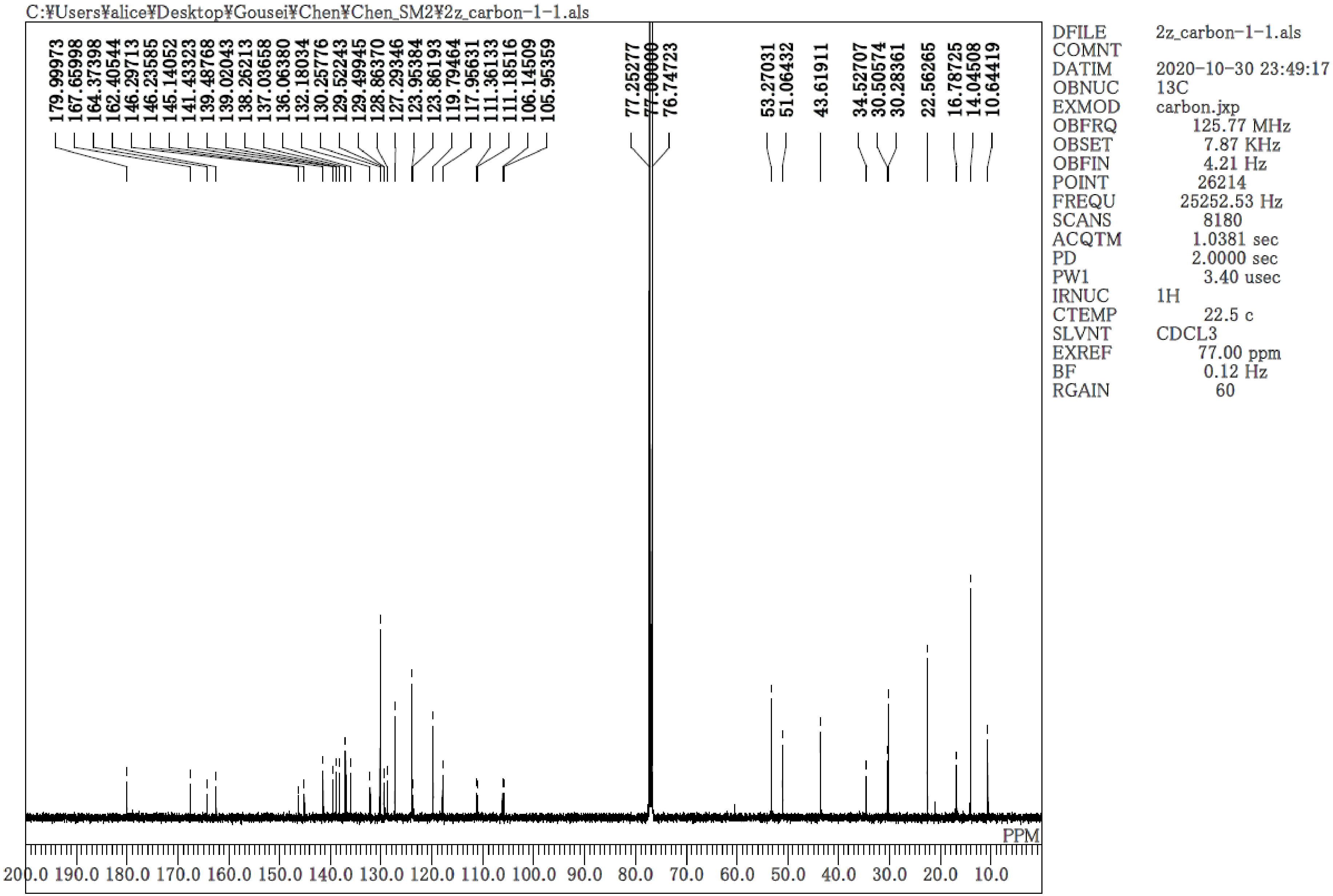
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2aa_proton.als



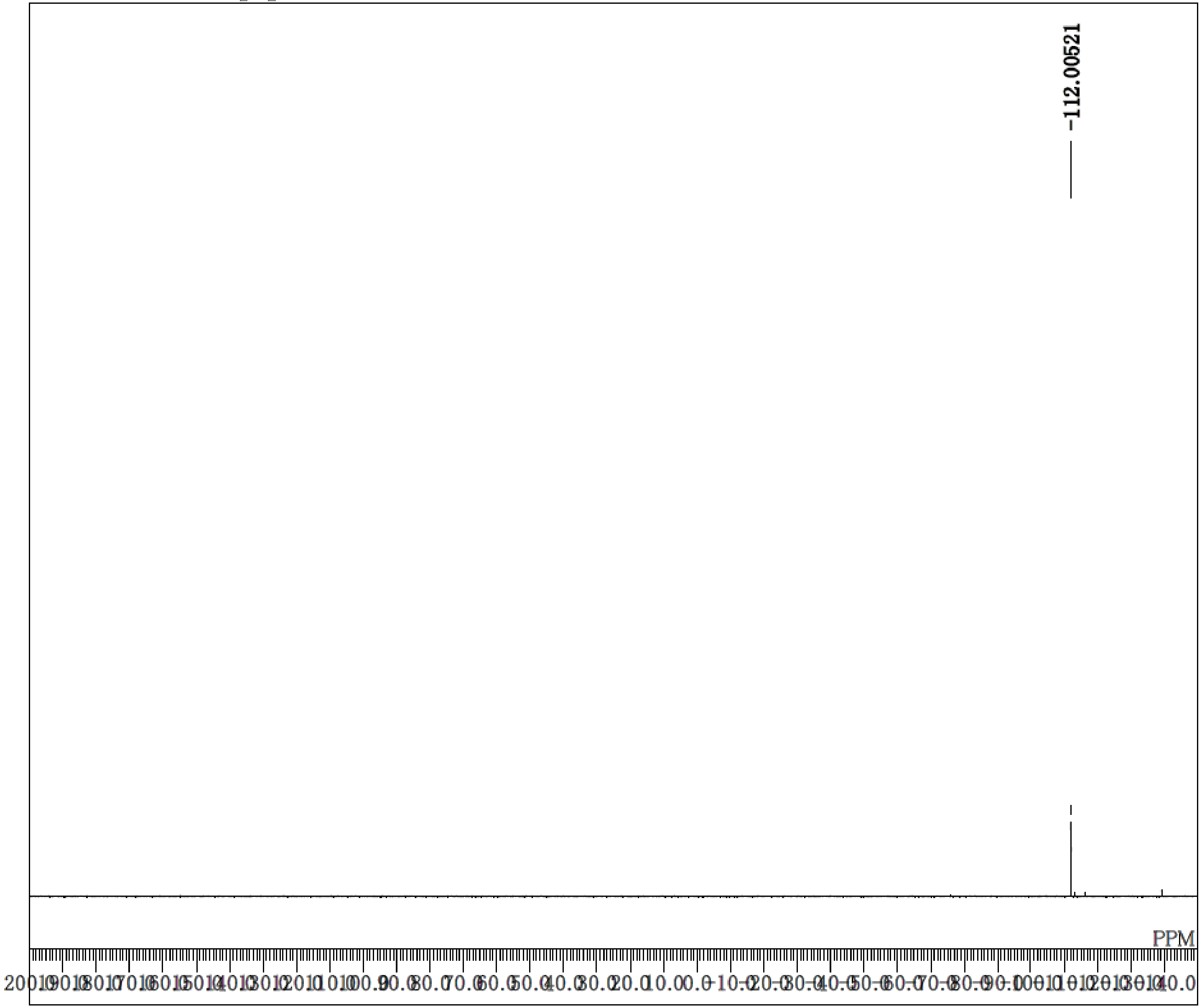
DFILE	2aa_proton.als
COMNT	
DATIM	30-10-2020 20:49:15
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



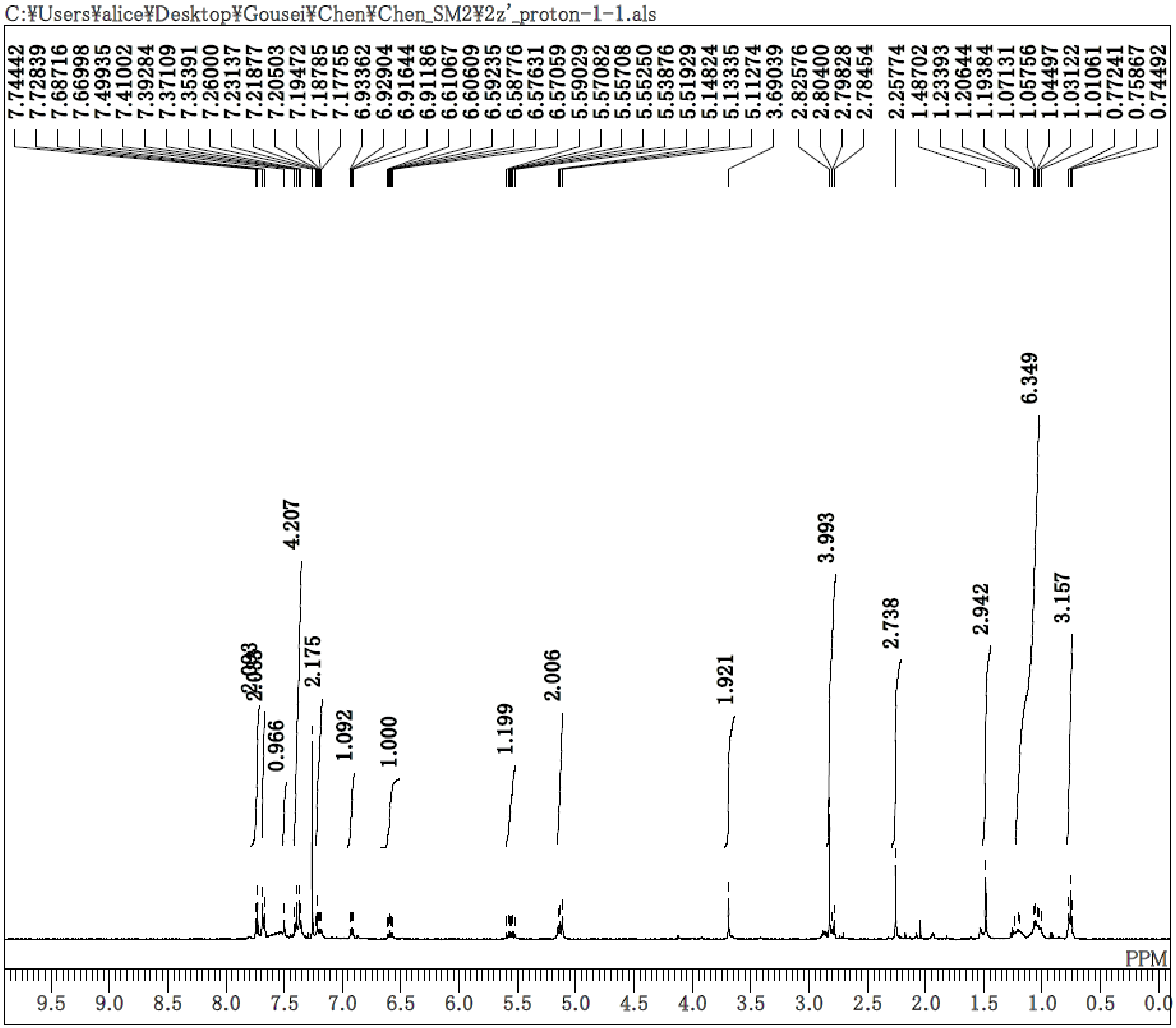
(2*S*,3*R*)-**2z**


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2z_carbon-1-1.als
179.99973
167.65998
164.37398
162.40544
146.29713
146.23585
145.14052
141.43323
139.48768
139.02043
138.26213
137.03658
136.06380
132.18034
130.25776
129.52243
129.49945
128.86370
127.29346
123.95384
123.86193
119.79464
117.95631
111.36133
111.18516
106.14509
105.95359
77.25277
77.00000
76.74723
53.27031
51.06432
43.61911
34.52707
30.50574
30.28361
22.56265
16.78725
14.04508
10.64419
PPM
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2z_carbon-1-1.als
COMNT
DATIM 2020-10-30 23:49:17
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 8180
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

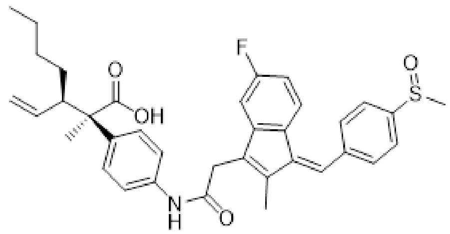
I:¥sulundac F¥sulindac_2z_F-1-1.als



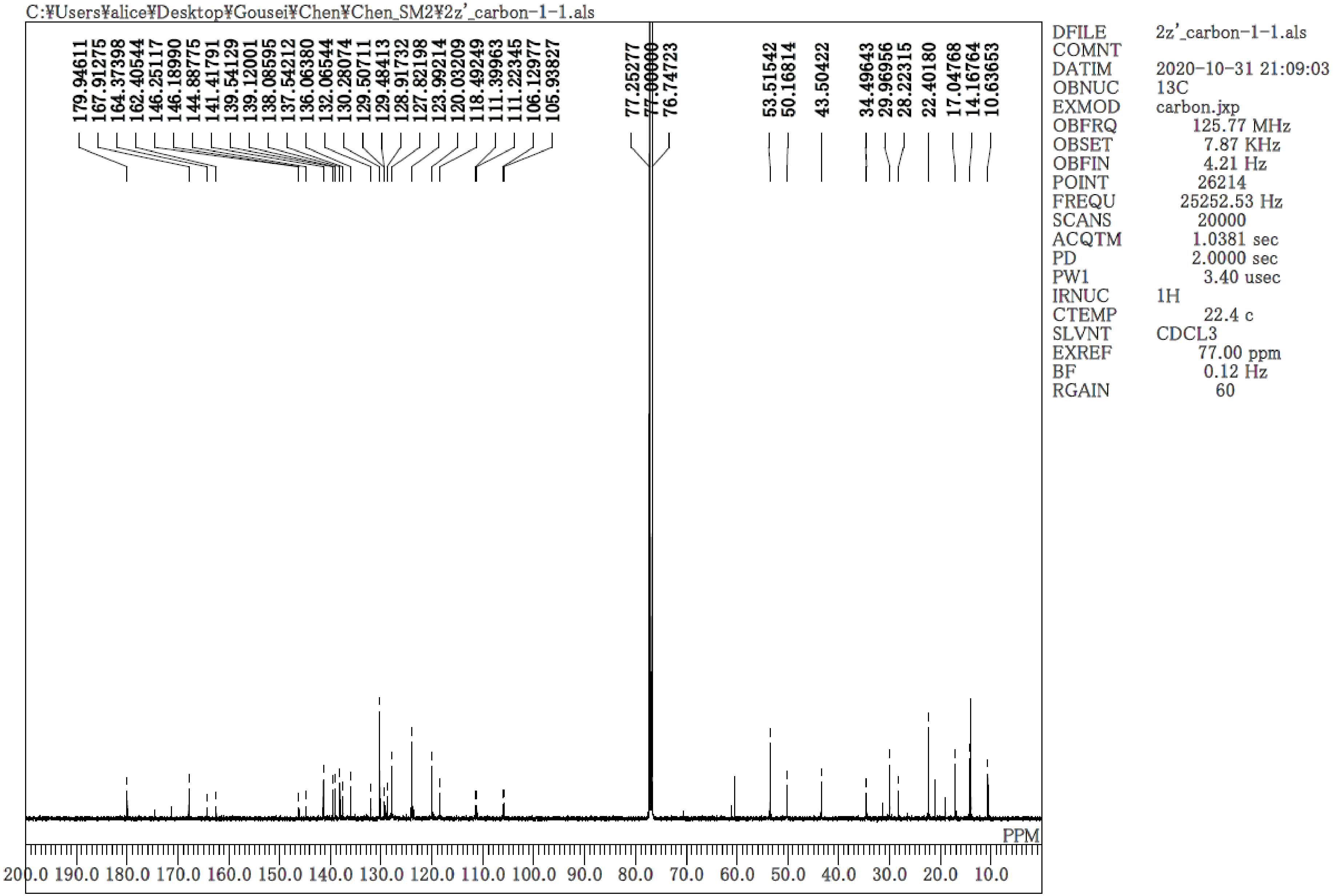
DFILE	sulindac_2z_F-1-1.als
COMNT	
DATIM	14-11-2020 09:26:41
OBNUC	19F
EXMOD	proton.jxp
OBFRQ	368.64 MHz
OBSET	7.63 KHz
OBFIN	2.85 Hz
POINT	13107
FREQU	149253.73 Hz
SCANS	8
ACQTM	0.0000 sec
PD	6.0000 sec
PW1	4.10 usec
IRNUC	19F
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	50



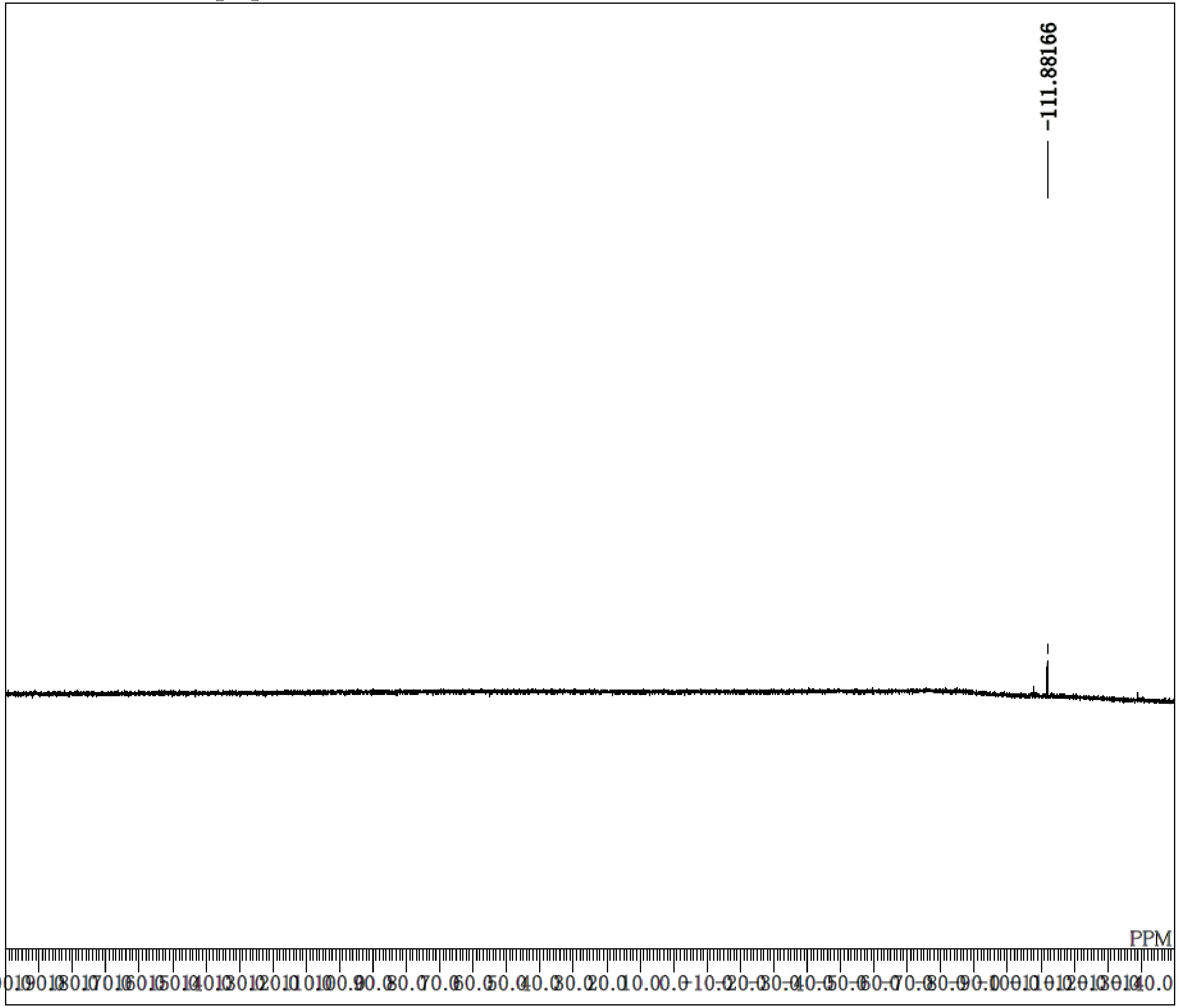
DFILE	2z'_proton-1-1.als
COMNT	
DATIM	2020-10-31 14:51:44
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



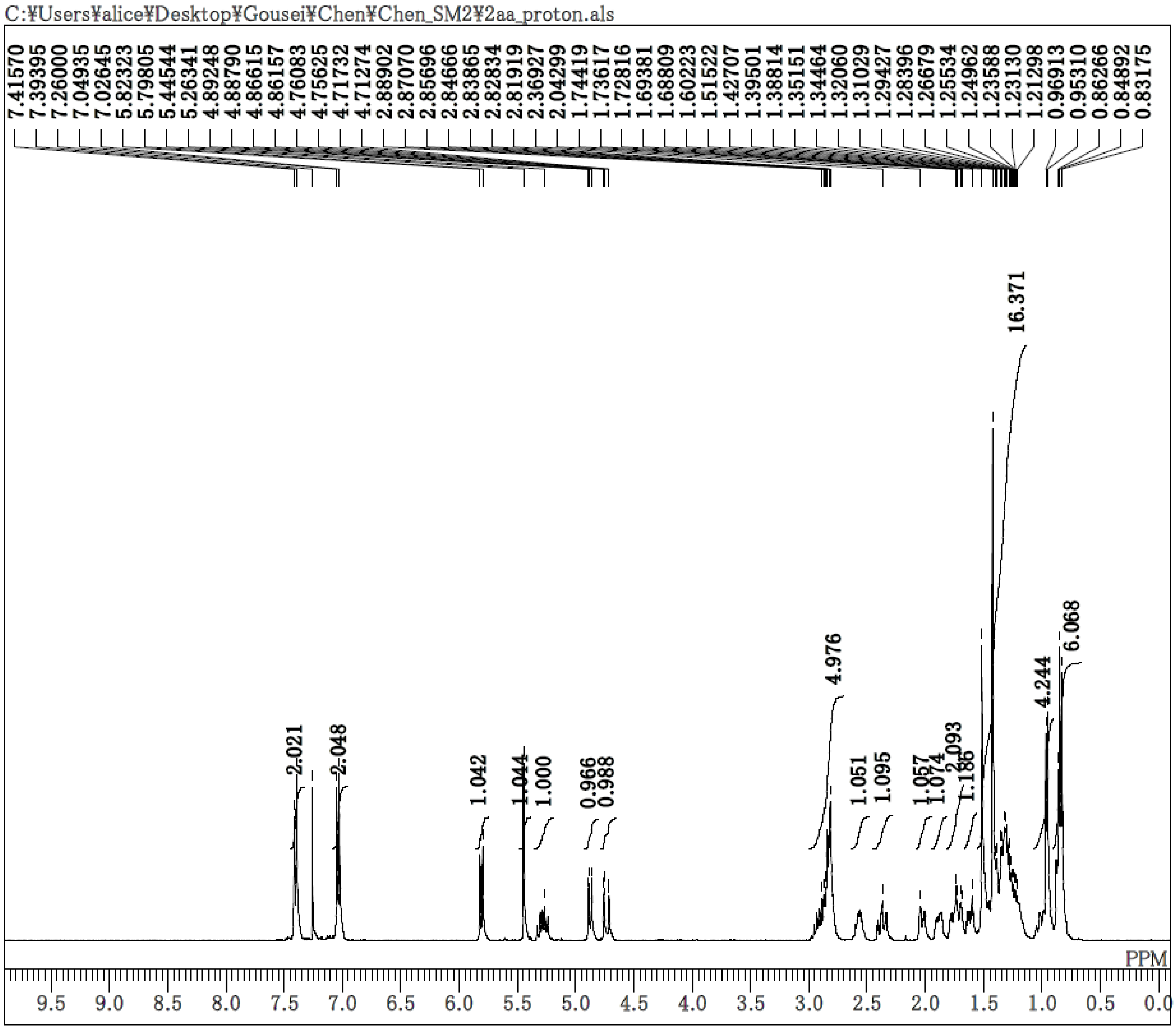
(2*R*,3*R*)-**2z**


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2z'_carbon-1-1.als
179.94611
167.91275
164.37398
162.40544
146.25117
146.18990
144.88775
141.41791
139.54129
139.12001
138.08595
137.54212
136.06380
132.06544
130.28074
129.50711
129.48413
128.91732
127.82198
123.99214
120.03209
118.49249
111.39963
111.22345
106.12977
105.93827
77.25277
77.00000
76.74723
53.51542
50.16814
43.50422
34.49643
29.96956
28.22315
22.40180
17.04768
14.16764
10.63653
PPM
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2z'_carbon-1-1.als
COMNT
DATIM 2020-10-31 21:09:03
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 20000
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

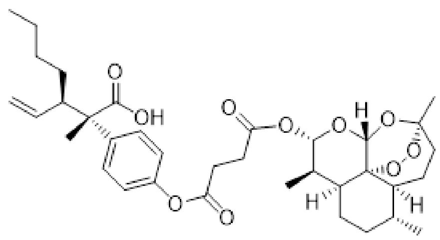
I:¥sulundac F¥sulindac_2z'_F-1-1.als



DFILE	sulindac_2z'_F-1-1.als
COMNT	
DATIM	14-11-2020 09:37:40
OBNUC	19F
EXMOD	proton.jxp
OBFRQ	368.64 MHz
OBSET	7.63 KHz
OBFIN	2.85 Hz
POINT	13107
FREQU	149253.73 Hz
SCANS	20
ACQTM	0.0000 sec
PD	6.0000 sec
PW1	4.10 usec
IRNUC	19F
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	50

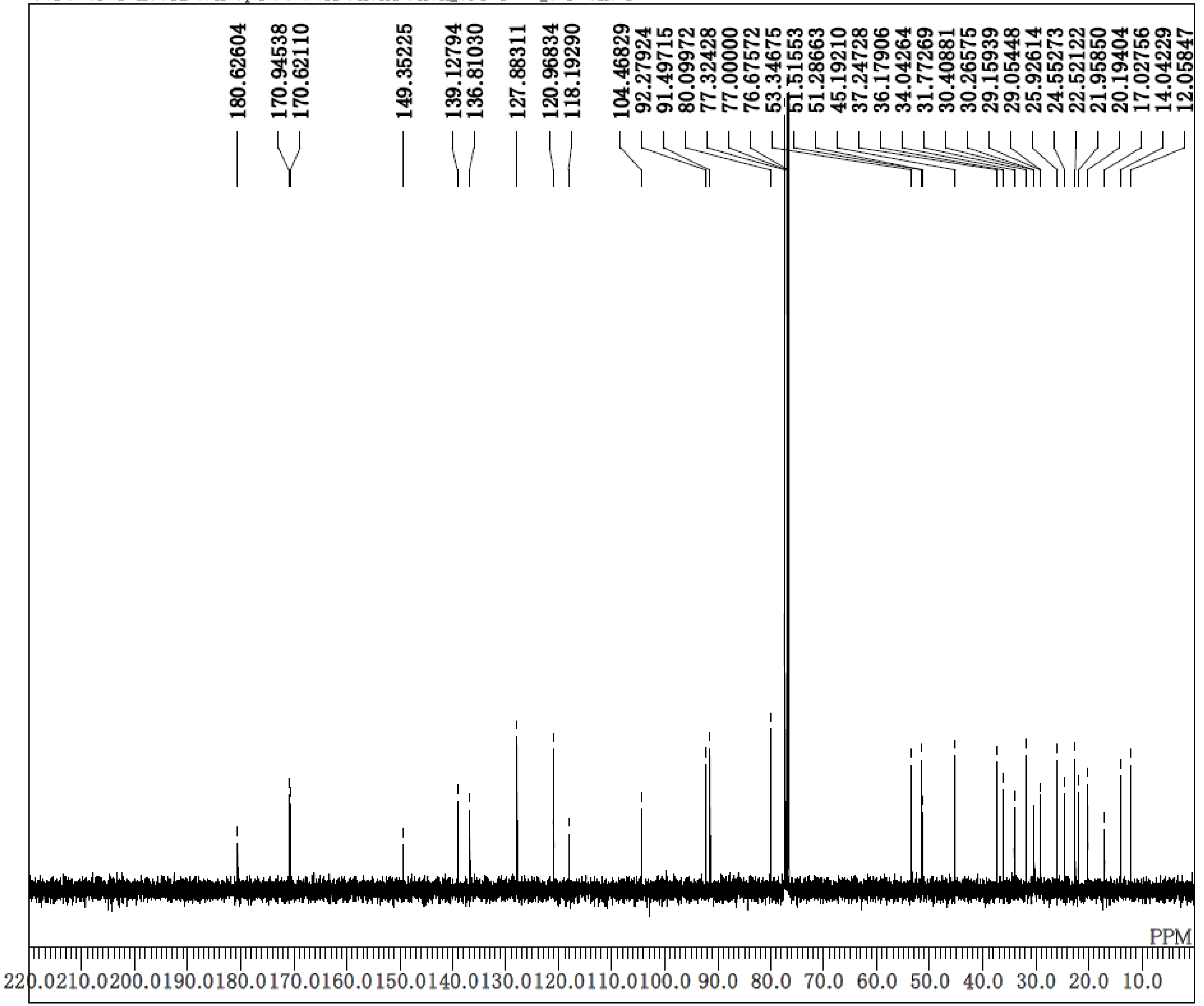


DFILE	2aa_proton.als
COMNT	
DATIM	22-10-2020 19:52:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34



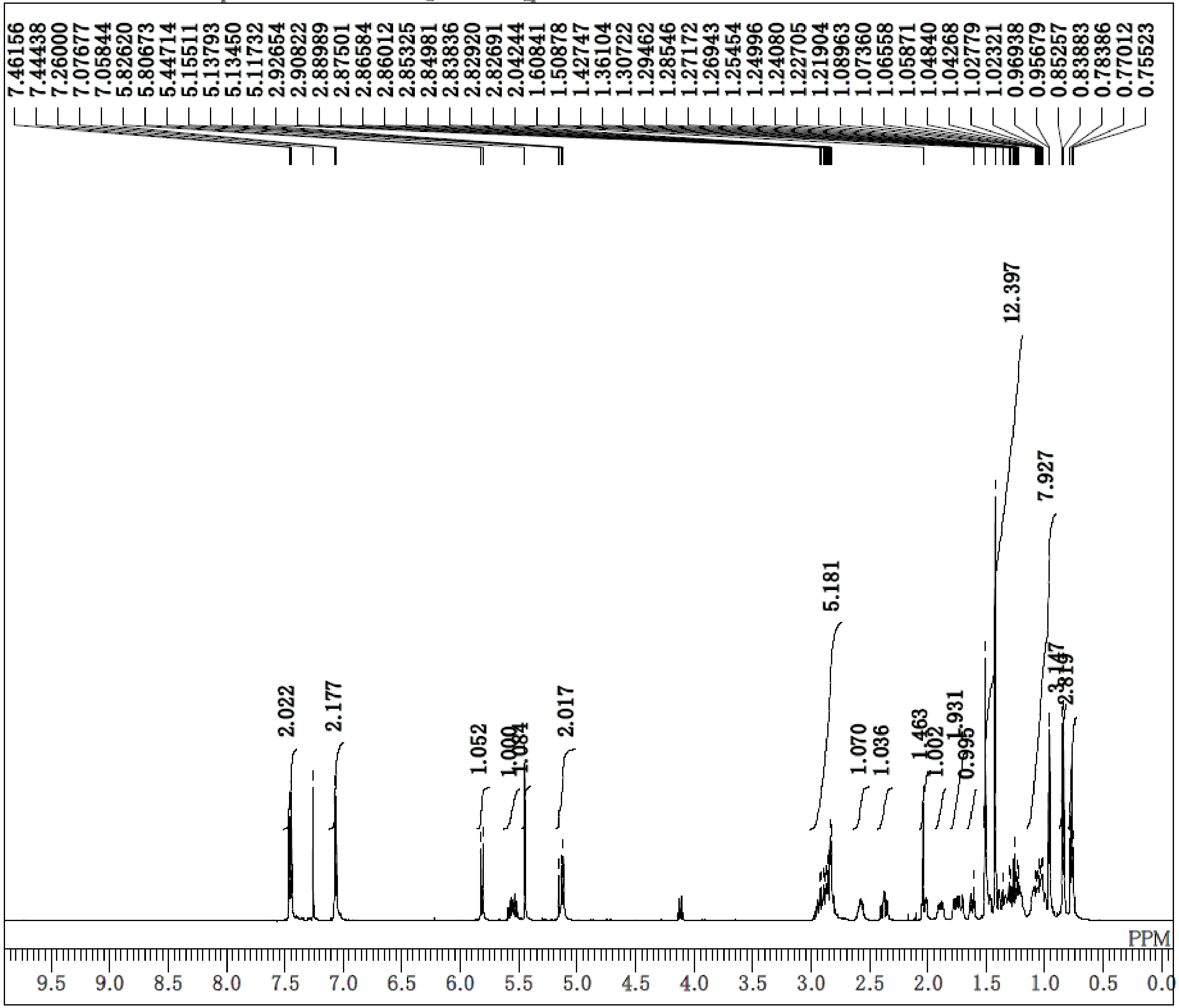
(2*S*,3*R*)-**2aa**

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2aa_carbon.als

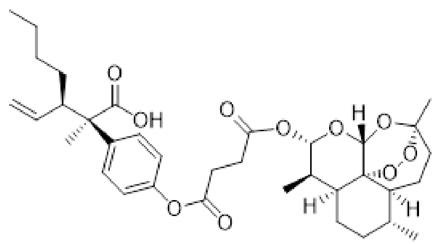


DFILE	2aa_carbon.als
COMNT	
DATIM	22-10-2020 19:54:15
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	464
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2aa'_proton-1-1.als

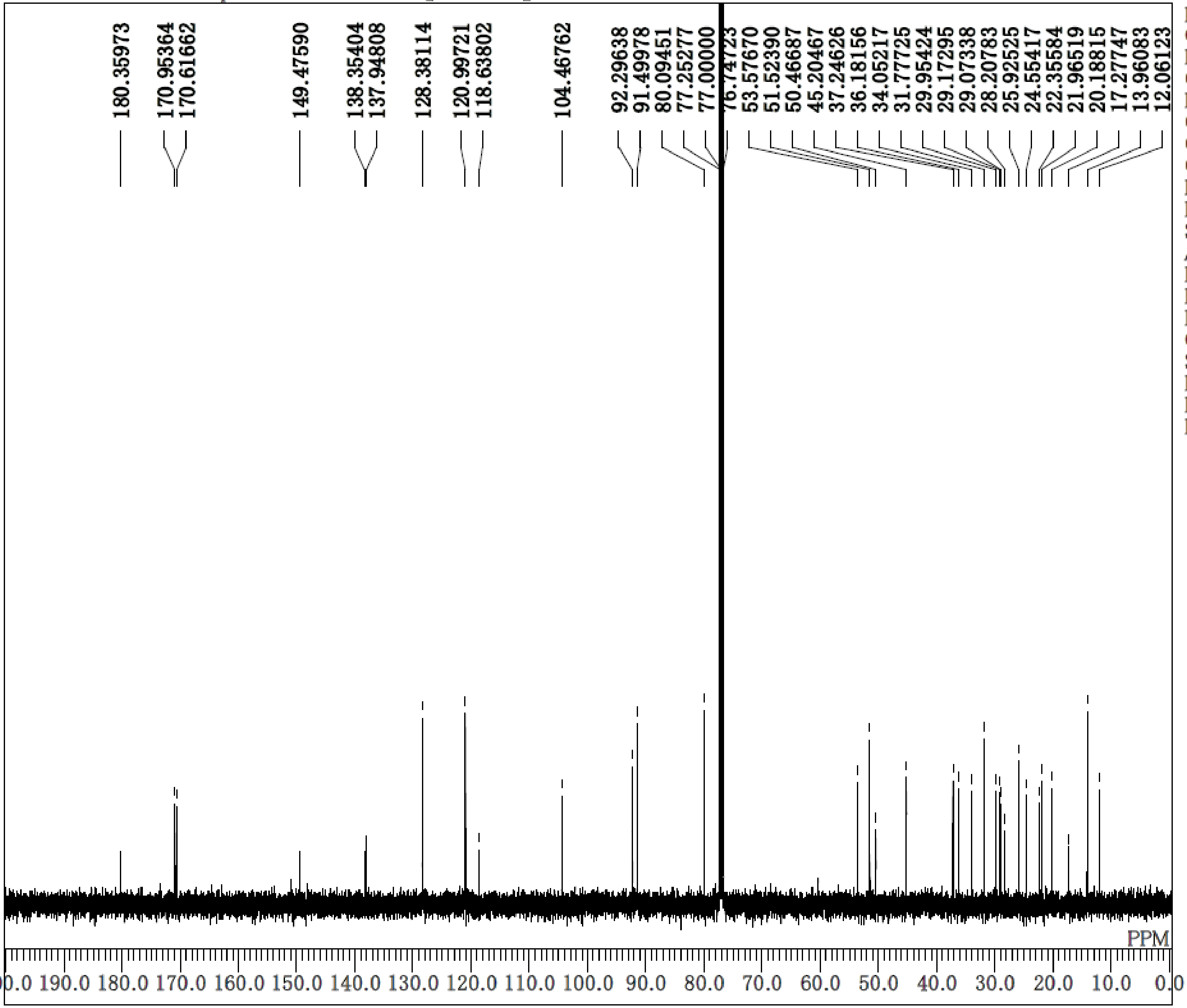


DFILE	2aa'_proton-1-1.als
COMNT	
DATIM	2020-10-20 20:59:03
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.32 Hz
RGAIN	38

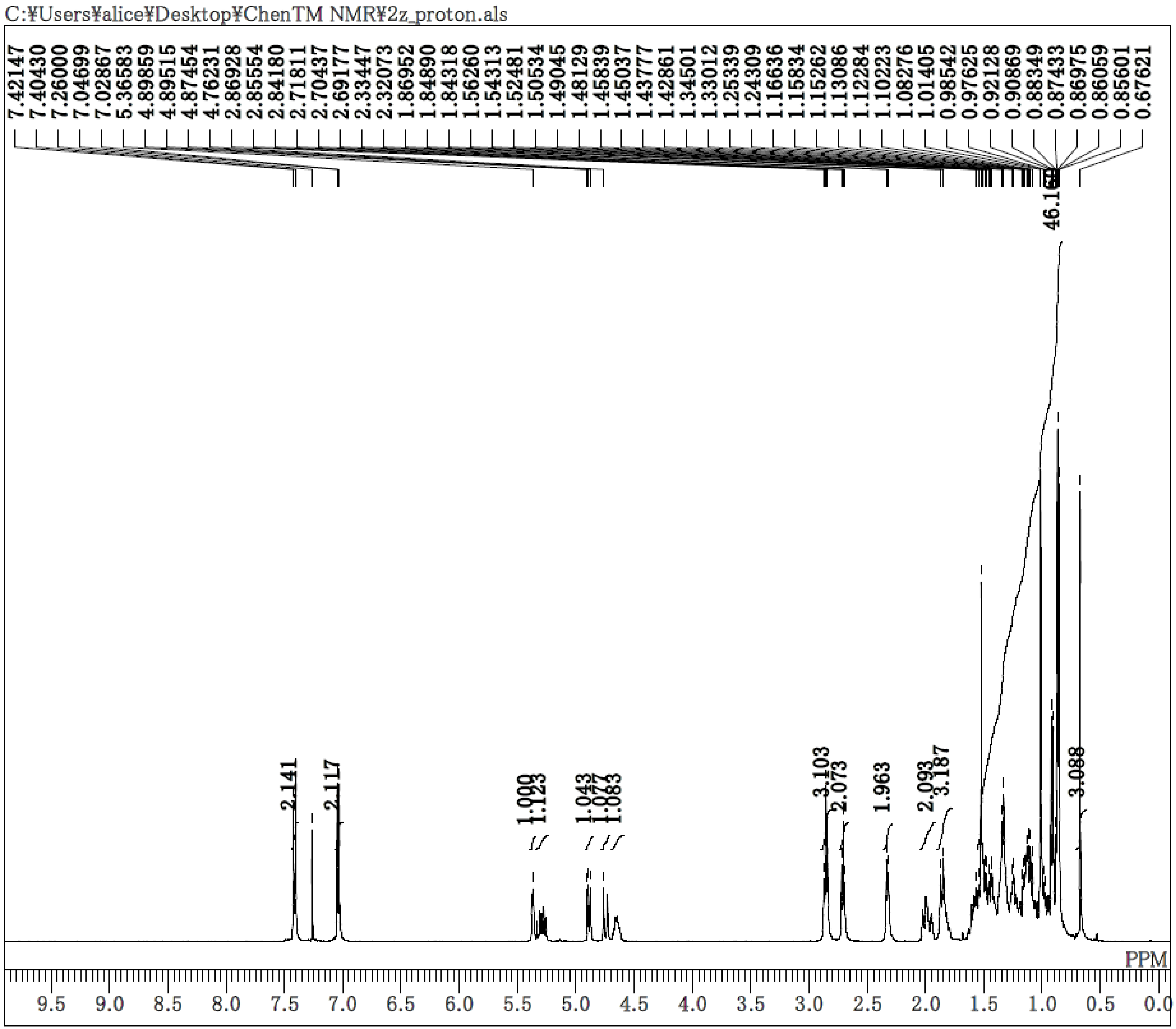


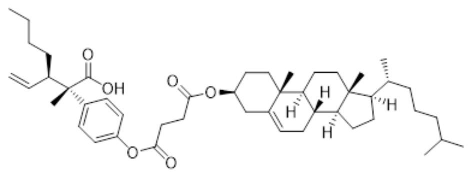
(2*R*,3*R*)-**2aa**

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2aa'_carbon-1-1.als

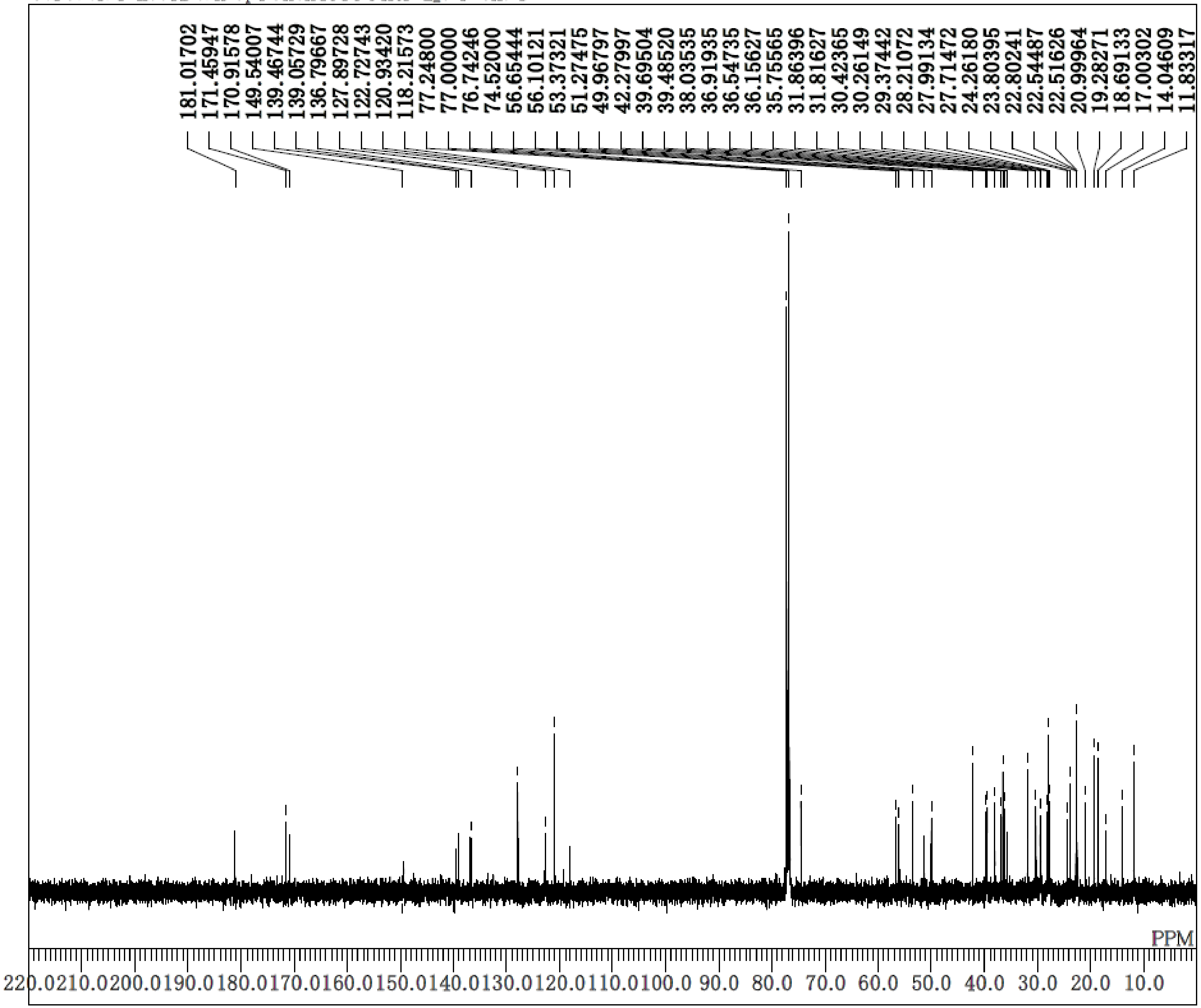


DFILE	2aa'_carbon-1-1.als
COMNT	
DATIM	2020-10-20 21:01:27
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	25252.53 Hz
SCANS	300
ACQTM	1.0381 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.32 Hz
RGAIN	60


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2z_proton.als
DFILE 2z_proton.als
COMNT
DATIM 2019-12-13 12:36:16
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38
7.42147
7.40430
7.26000
7.04699
7.02867
5.36583
4.89859
4.89515
4.87454
4.76231
2.86928
2.85554
2.84180
2.71811
2.70437
2.69177
2.33447
2.32073
1.86952
1.84890
1.84318
1.56260
1.54313
1.52481
1.50534
1.49045
1.48129
1.45839
1.45037
1.43777
1.42861
1.34501
1.33012
1.25339
1.24309
1.16636
1.15834
1.15262
1.13086
1.12284
1.10223
1.08276
1.01405
0.98542
0.97625
0.92128
0.90869
0.88349
0.87433
0.86975
0.86059
0.85601
0.67621
46.16
2.141
2.117
1.000
1.123
1.043
1.077
1.083
3.103
2.073
1.963
2.093
3.187
3.088
PPM
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

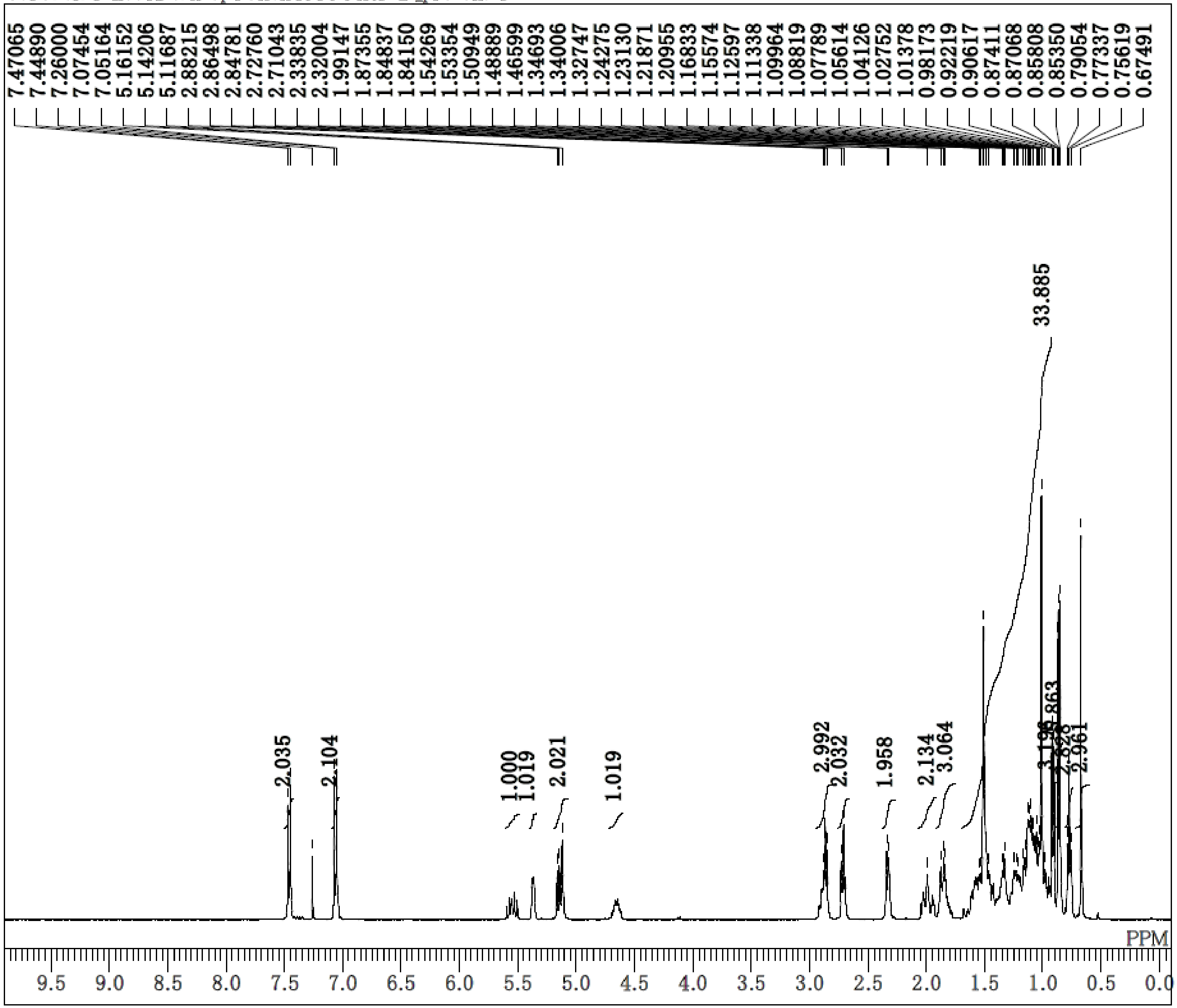

O
OH
O
O
O
O
H
H
H
H
(2S,3R)-**2ab**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2z_carbon.als

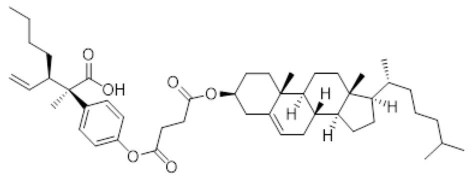


DFILE	2z_carbon.als
COMNT	
DATIM	2019-12-13 12:41:56
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	291
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2z'_proton.als

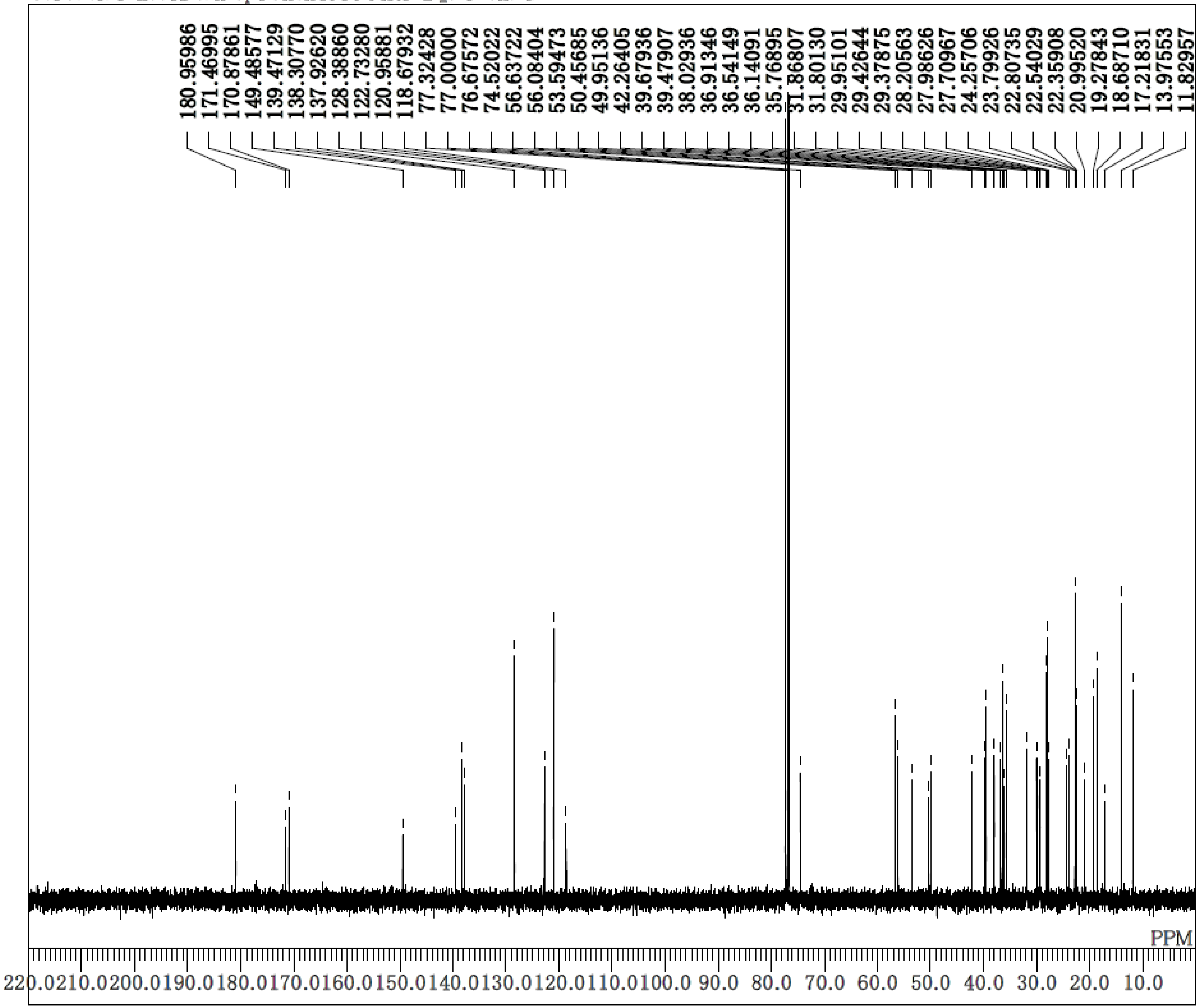


DFILE	2z'_proton.als
COMNT	
DATIM	31-03-2020 02:28:36
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	26



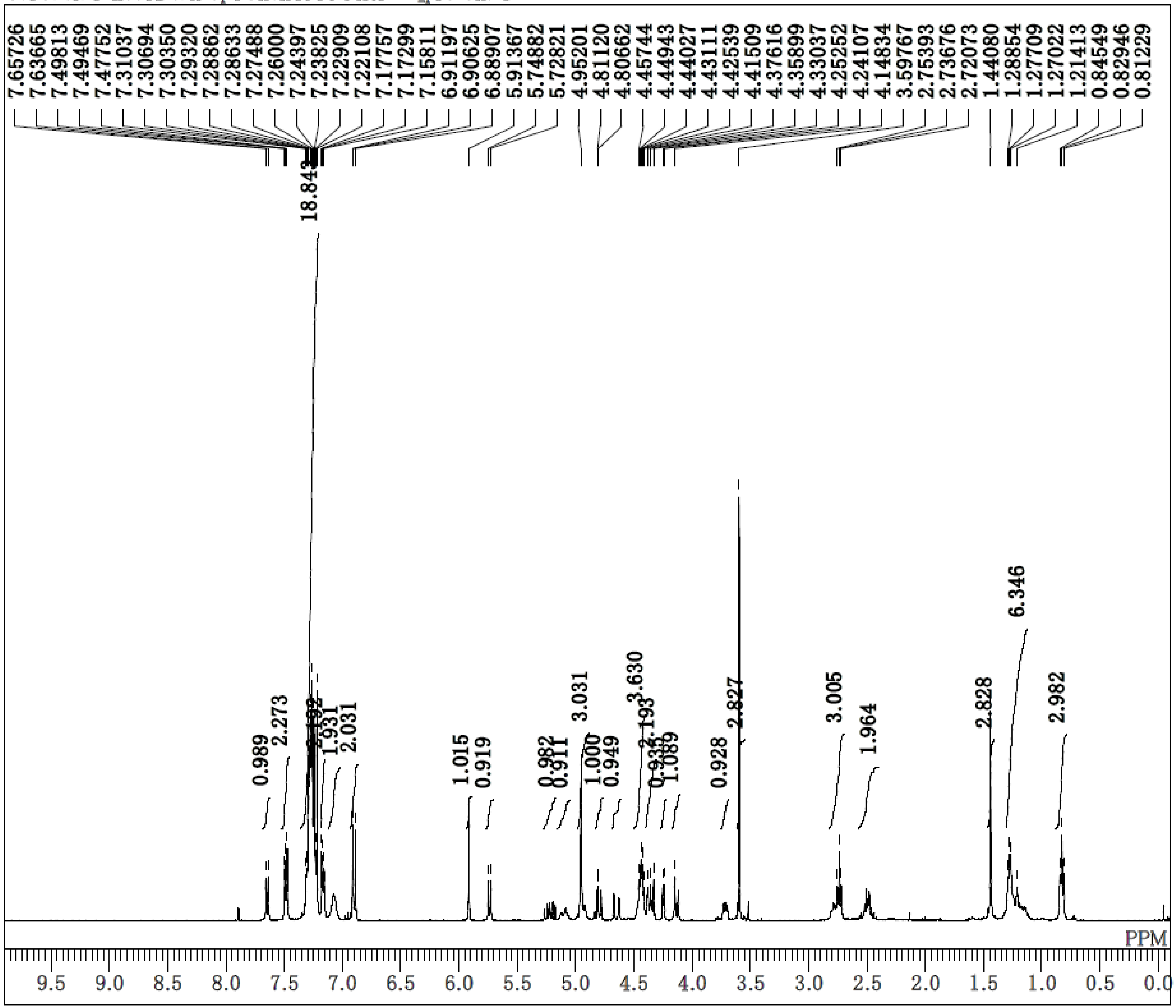
(2*R*,3*R*)-**2ab**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2z'_carbon.als

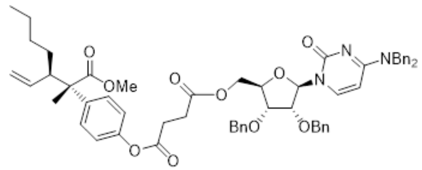


DFILE	2z'_carbon.als
COMNT	
DATIM	31-03-2020 02:37:05
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	241
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

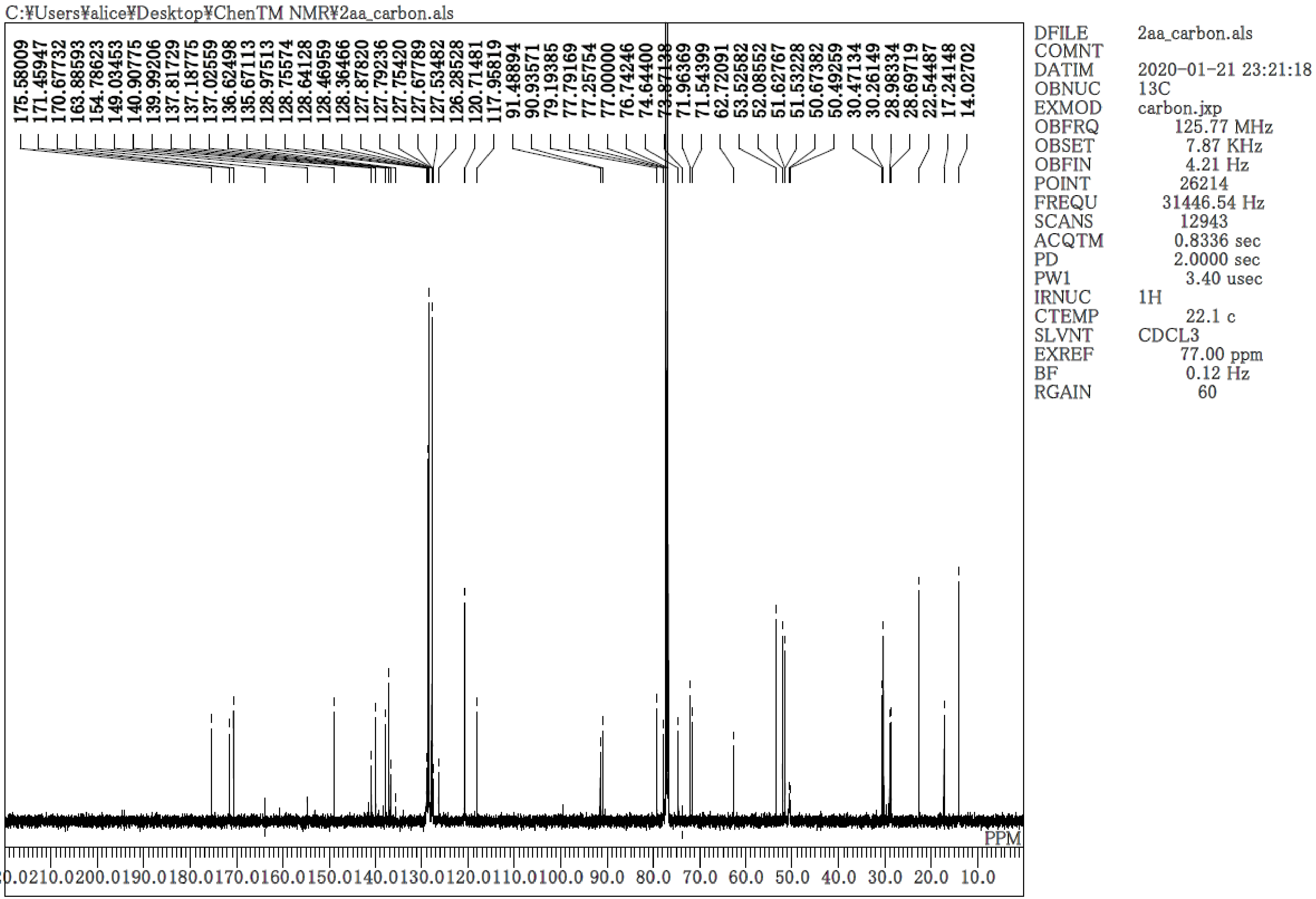
C:¥Users¥alice¥Desktop¥ChenTM NMR¥2aa_proton.als



DFILE	2aa_proton.als
COMNT	
DATIM	20-01-2020 18:53:52
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



(2S,3R)-**2ae**


C:¥Users¥alice¥Desktop¥ChenTM NMR¥2aa_carbon.als
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171.45947
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154.78623
149.03453
140.90775
139.99206
137.81729
137.18775
137.02559
136.62498
135.67113
128.97513
128.75574
128.64128
128.46959
128.36466
127.87820
127.79236
127.75420
127.67789
127.53482
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120.71481
117.95819
91.48894
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79.19385
77.79169
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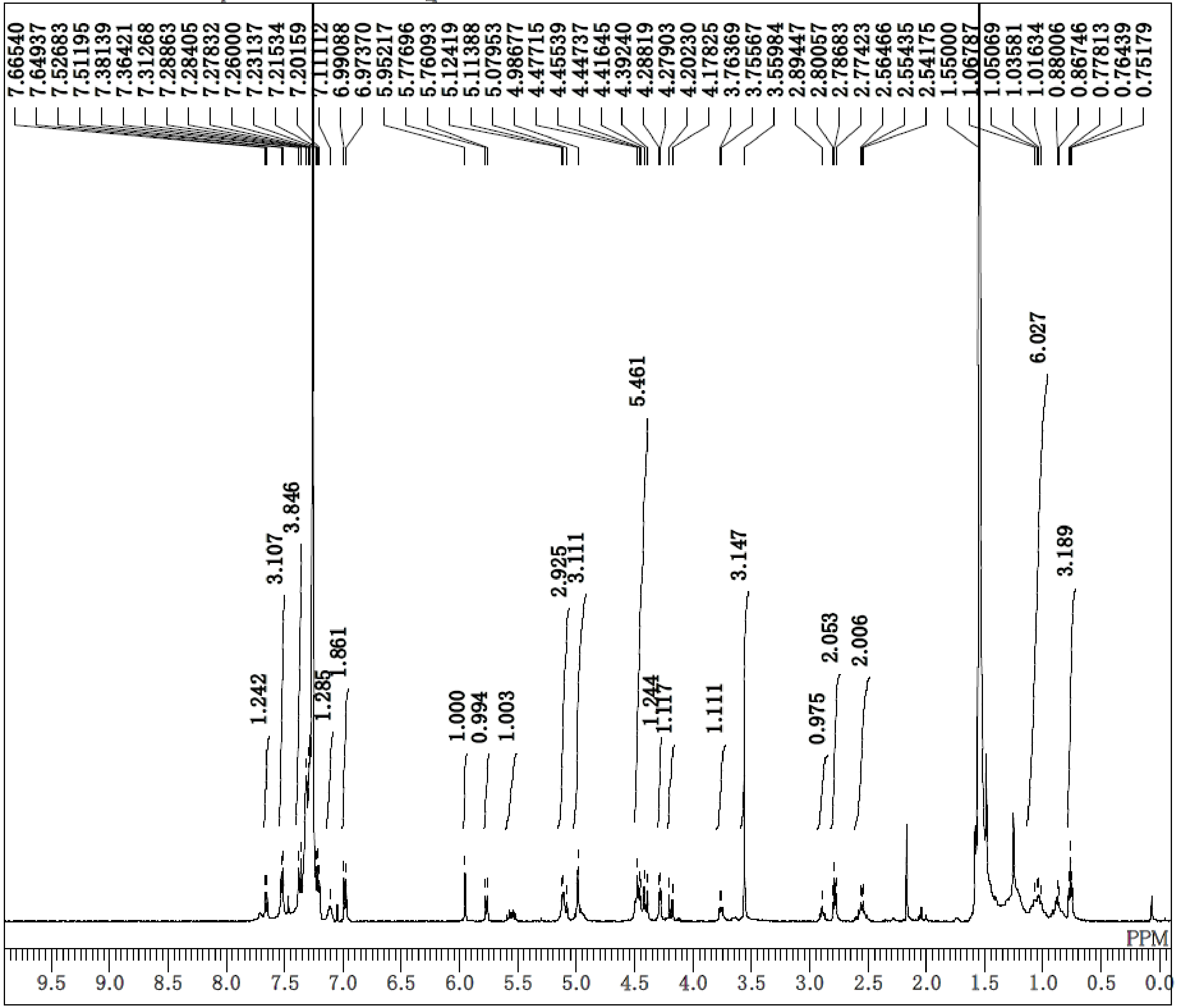
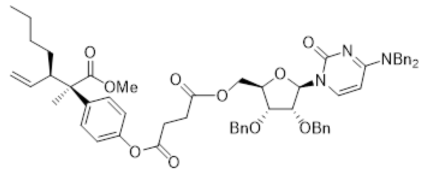
77.00000
76.74246
74.64400
73.87138
71.96369
71.54399
62.72091
53.52582
52.08552
51.62767
51.53228
50.67382
50.49259
30.47134
30.26149
28.98334
28.69719
22.54487
17.24148
14.02702
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE 2aa_carbon.als
COMNT
DATIM 2020-01-21 23:21:18
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 12943
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2aa'_proton.als

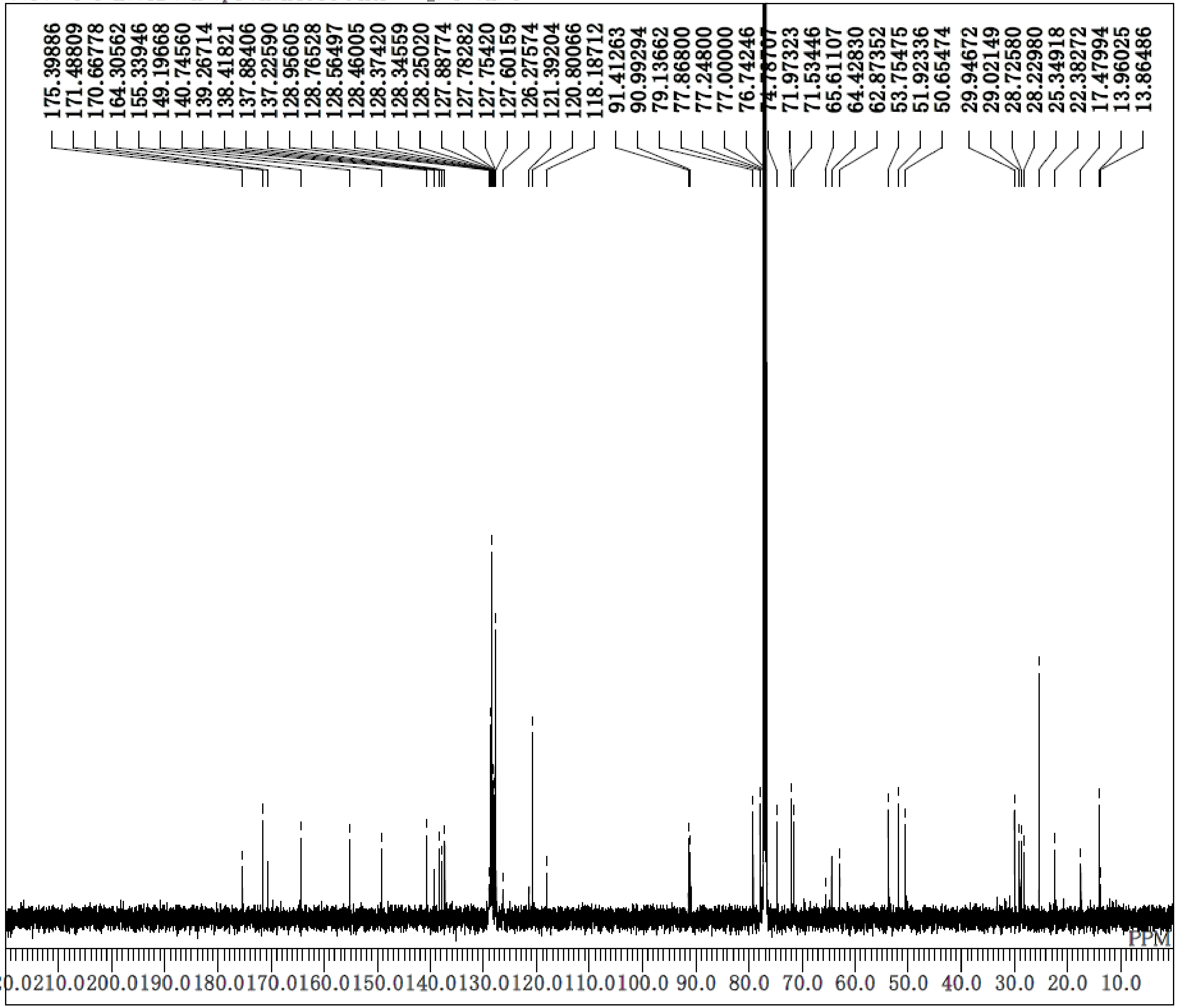


DFILE	2aa'_proton.als
COMNT	
DATIM	2020-06-16 19:58:32
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	256
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	25.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	44



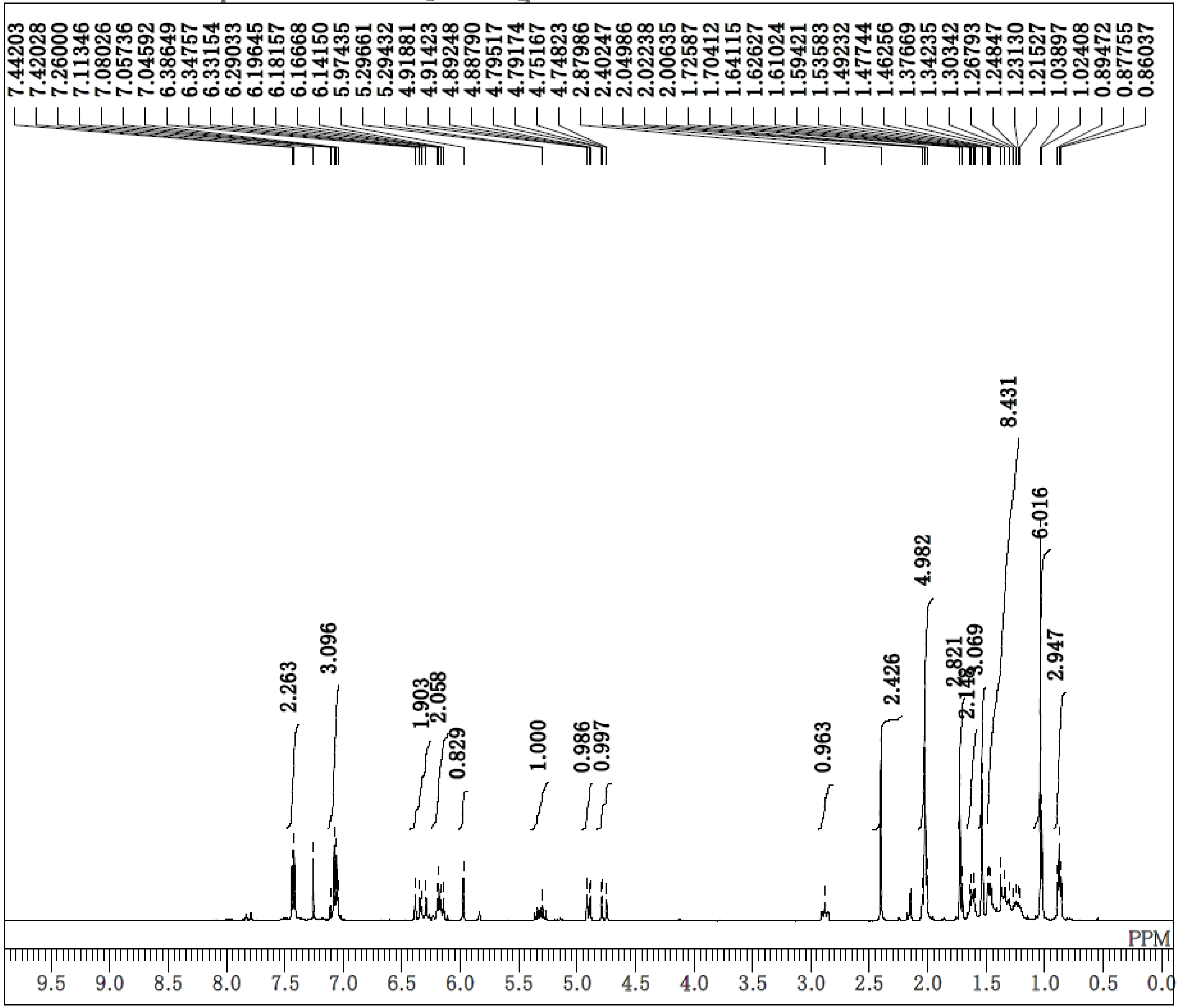
(2*R*,3*R*)-**2ae**

C:¥Users¥alice¥Desktop¥ChenTM NMR¥2aa'_carbon.als

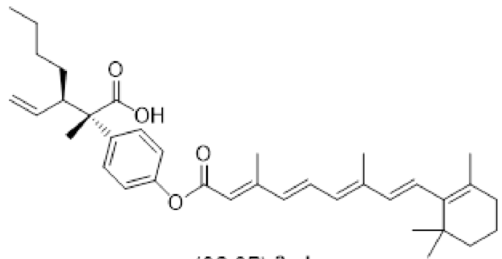


DFILE	2aa'_carbon.als
COMNT	
DATIM	2020-06-16 21:00:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	18211
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	25.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2ad_proton.als

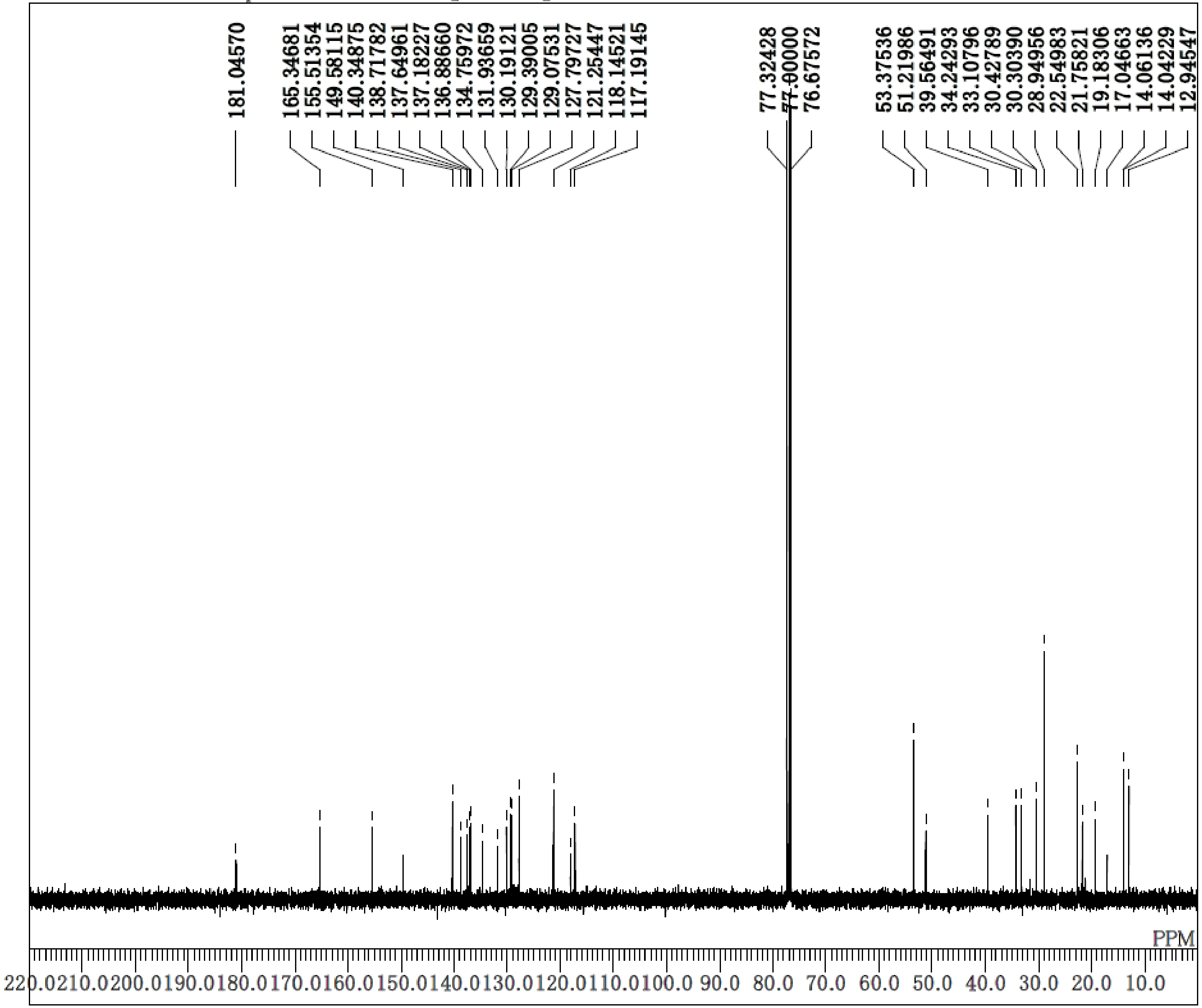


DFILE	2ad_proton.als
COMNT	
DATIM	21-10-2020 21:51:41
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32



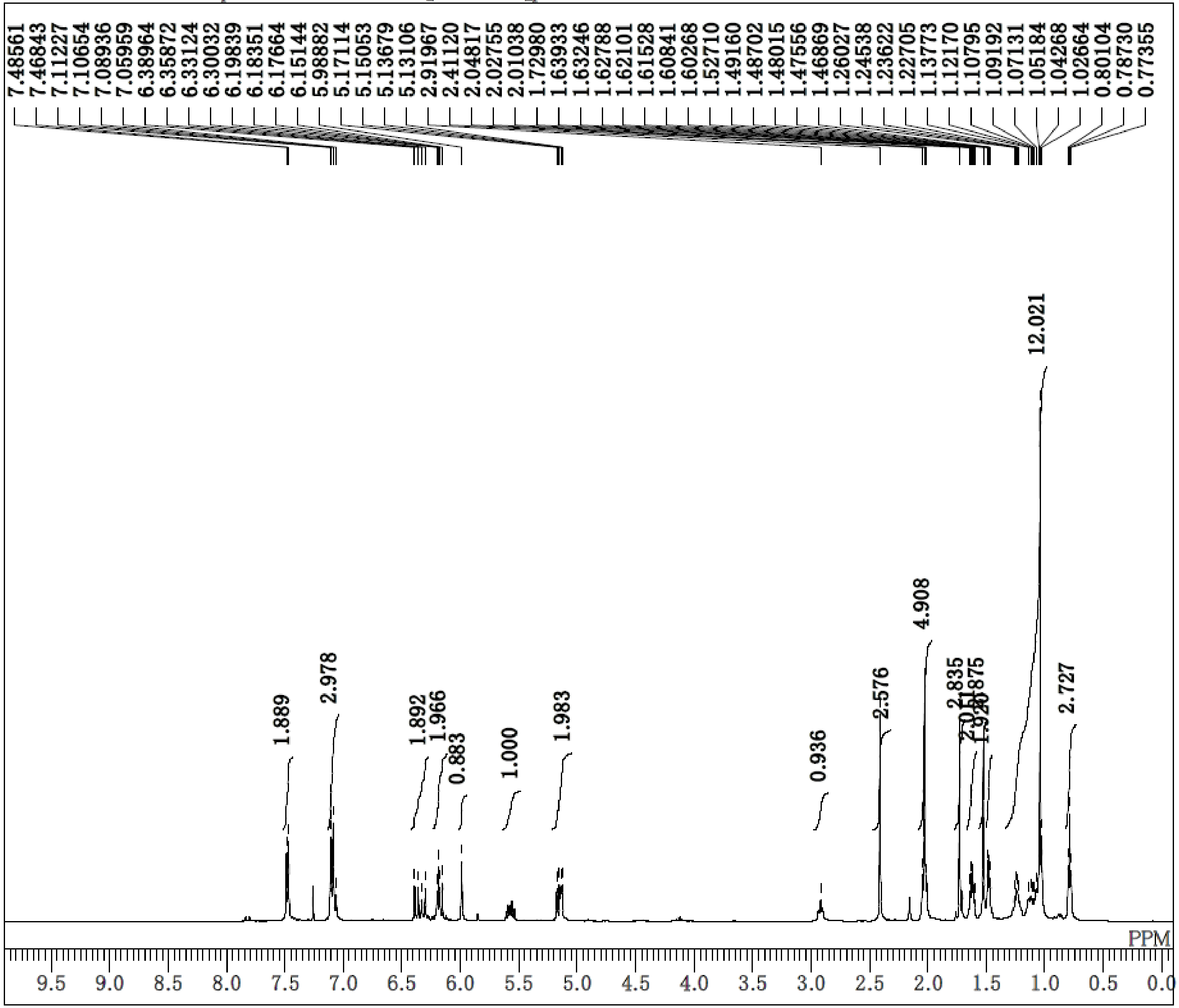
(2*S*,3*R*)-**2ad**

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2ad_carbon.als

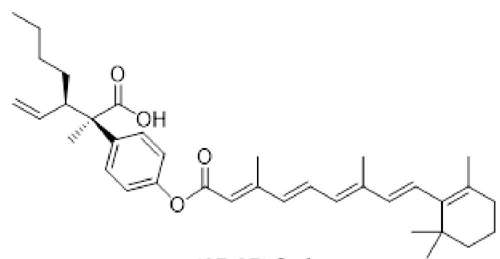


DFILE	2ad_carbon.als
COMNT	
DATIM	21-10-2020 21:55:02
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	353
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2ad'_proton-1-1.als

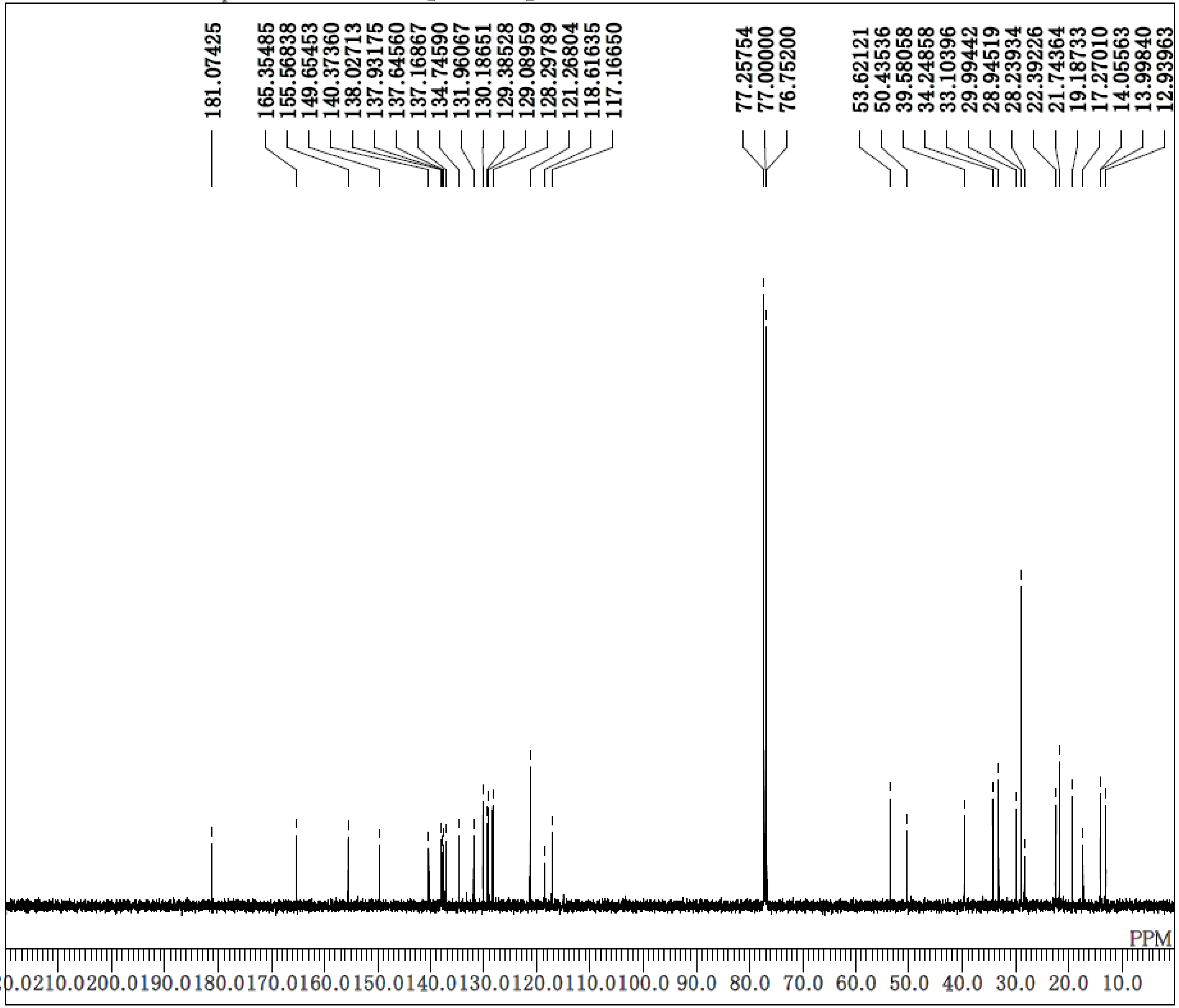


DFILE	2ad'_proton-1-1.als
COMNT	
DATIM	2020-10-15 13:15:60
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.62 Hz
RGAIN	36

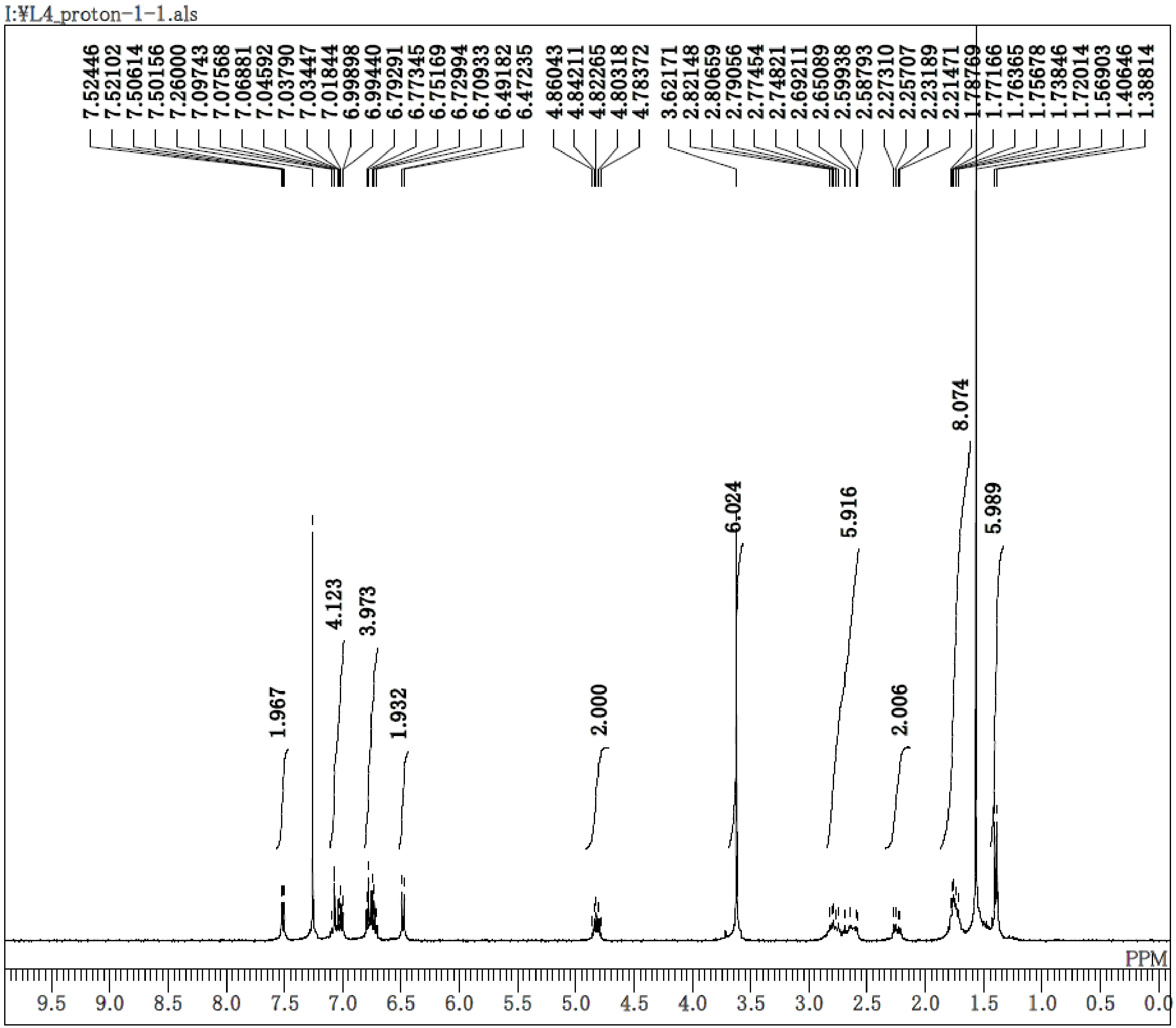


(2*R*,3*R*)-**2ad**

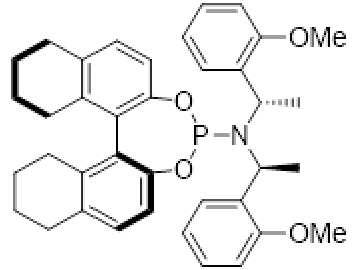
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥2ad'_carbon-1-1.als



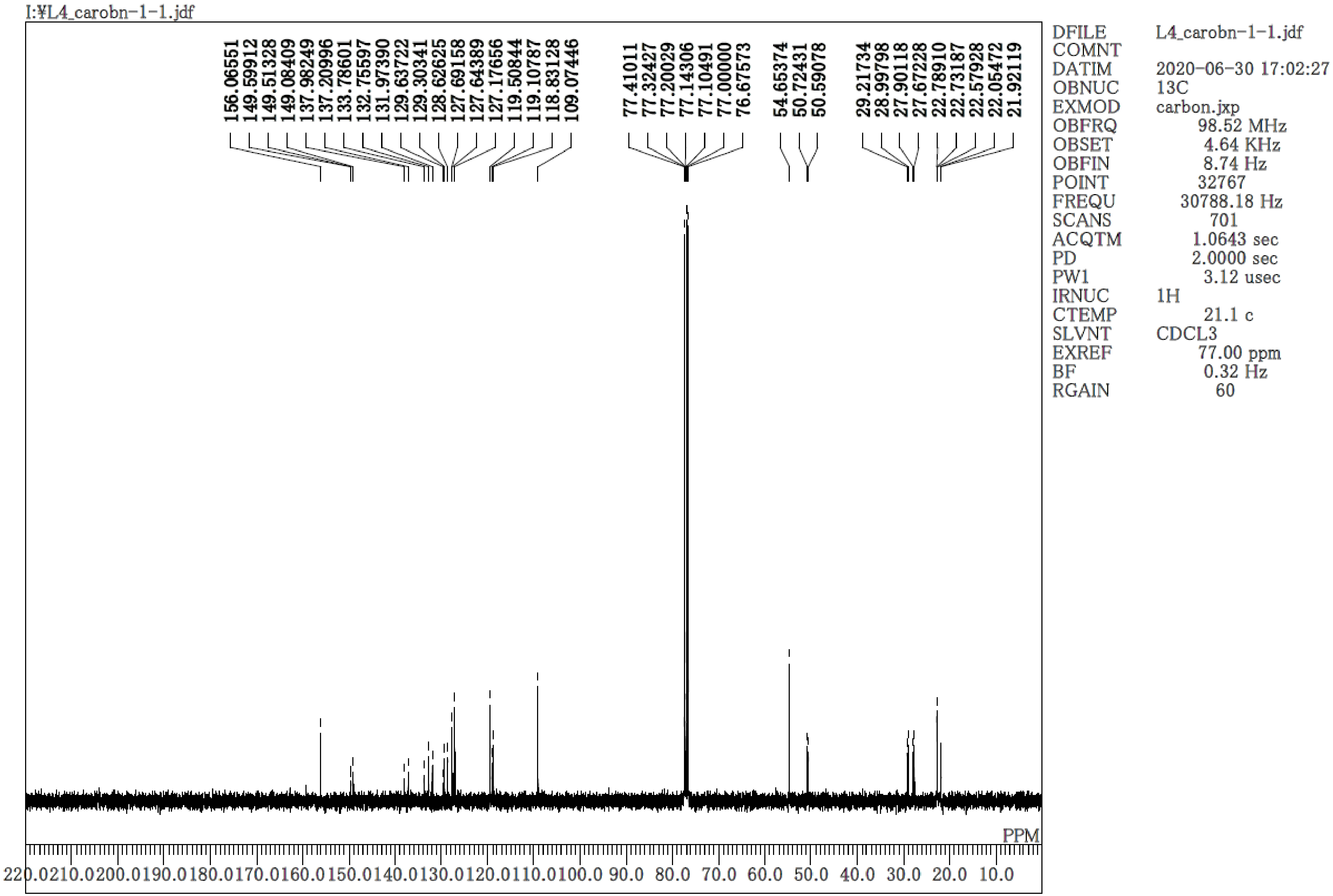
DFILE	2ad'_carbon-1-1.als
COMNT	
DATIM	2020-10-15 13:02:24
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	256
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	60

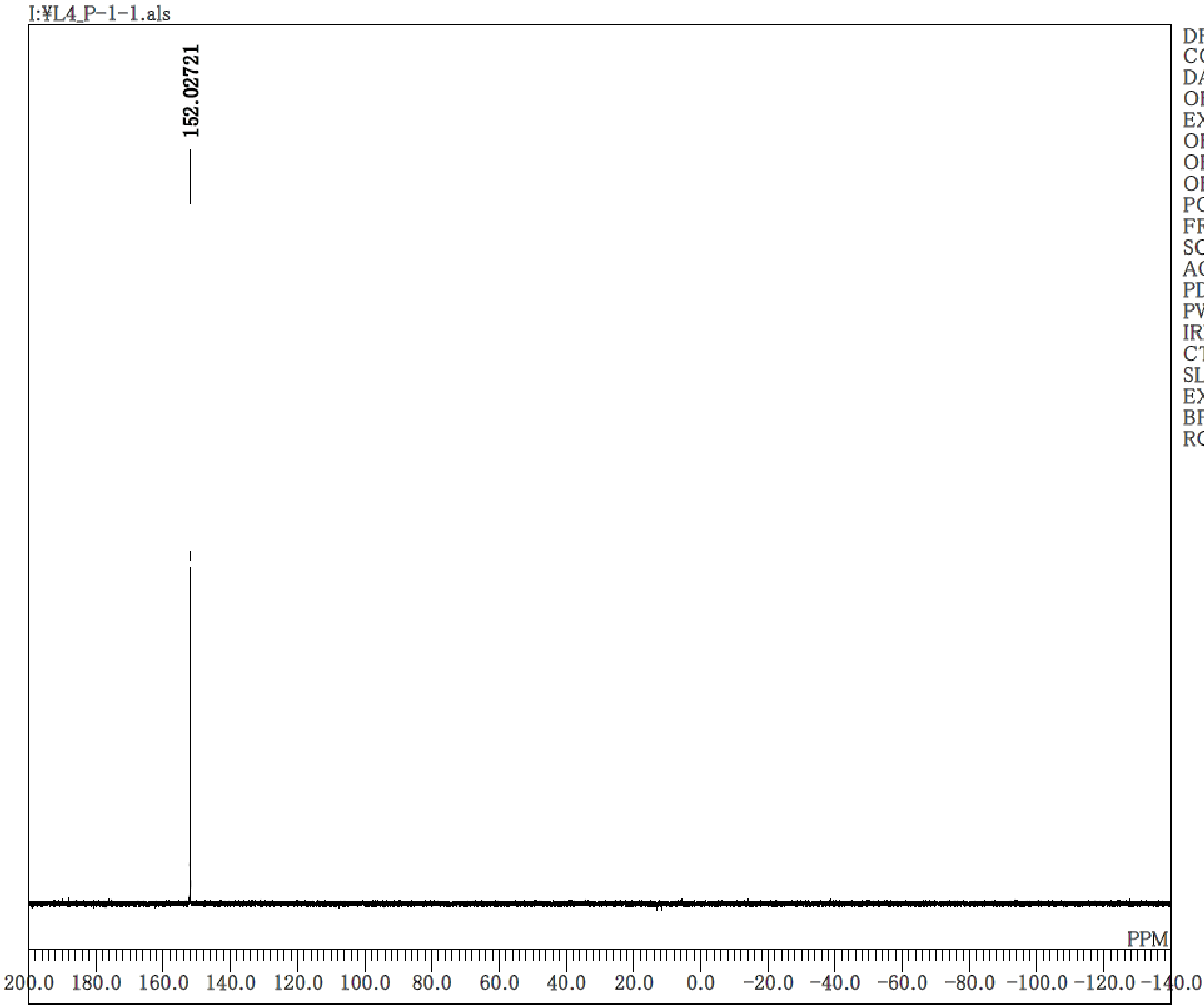


DFILE	L4_proton-1-1.als
COMNT	
DATIM	30-06-2020 16:56:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	46



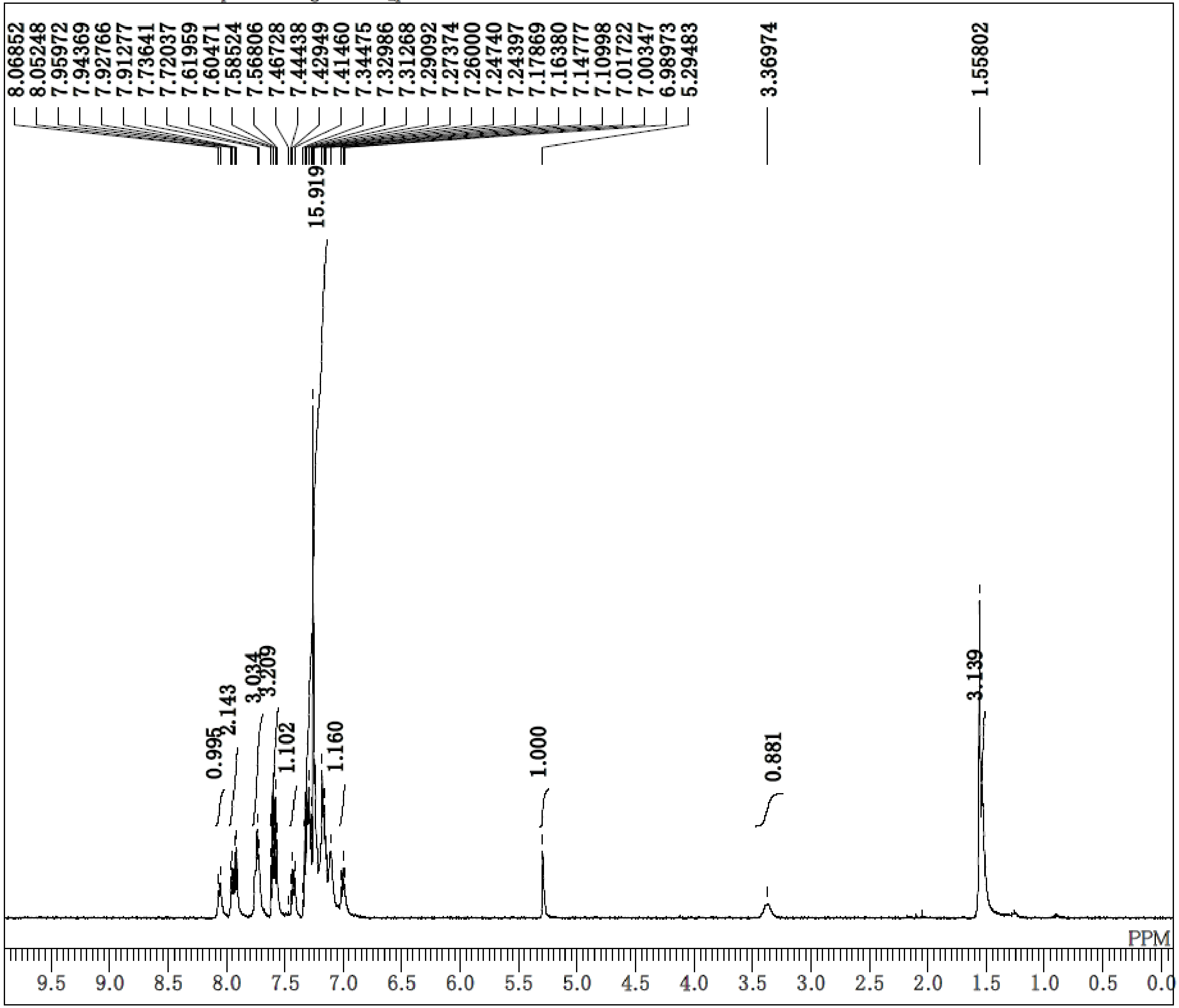
L4


I:¥L4_carobn-1-1.jdf
156.06551
149.59912
149.51328
149.08409
137.98249
137.20996
133.78601
132.75597
131.97390
129.63722
129.30341
128.62625
127.69158
127.64389
127.17656
119.50844
119.10787
118.83128
109.07446
77.41011
77.32427
77.20029
77.14306
77.10491
77.00000
76.67573
54.65374
50.72431
50.59078
29.21734
28.99798
27.90118
27.67228
22.78910
22.73187
22.57928
22.05472
21.92119
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE L4_carobn-1-1.jdf
COMNT
DATIM 2020-06-30 17:02:27
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 32767
FREQU 30788.18 Hz
SCANS 701
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.32 Hz
RGAIN 60

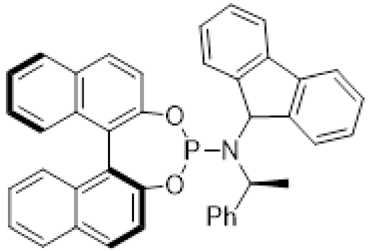


DFILE	L4_P-1-1.als
COMNT	
DATIM	30-06-2020 17:40:53
OBNUC	31P
EXMOD	carbon.jxp
OBFRQ	158.59 MHz
OBSET	7.99 KHz
OBFIN	9.23 Hz
POINT	26214
FREQU	64102.56 Hz
SCANS	101
ACQTM	0.4089 sec
PD	2.0000 sec
PW1	4.80 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.32 Hz
RGAIN	56

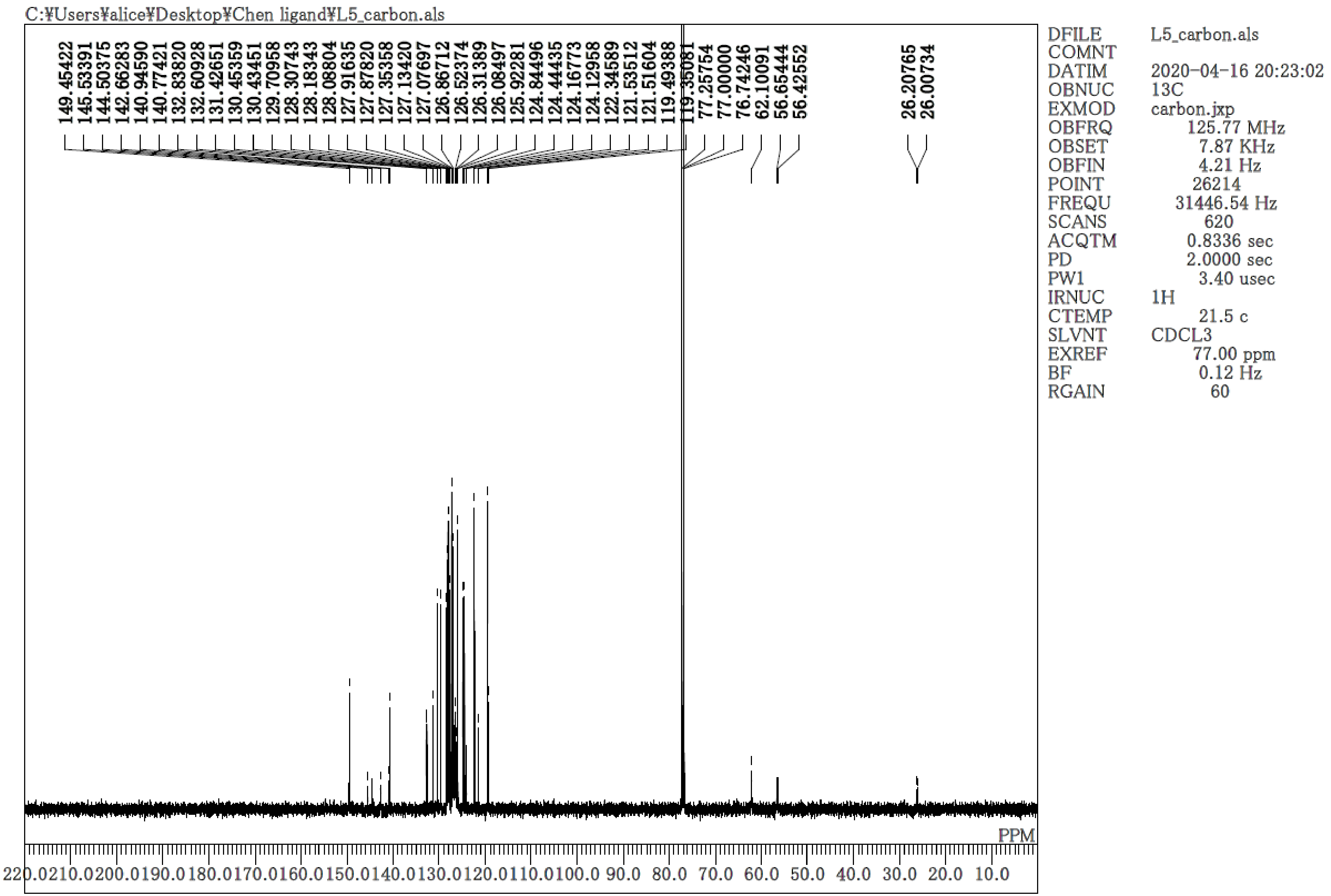
C:¥Users¥alice¥Desktop¥Chen ligand¥L5_proton.als

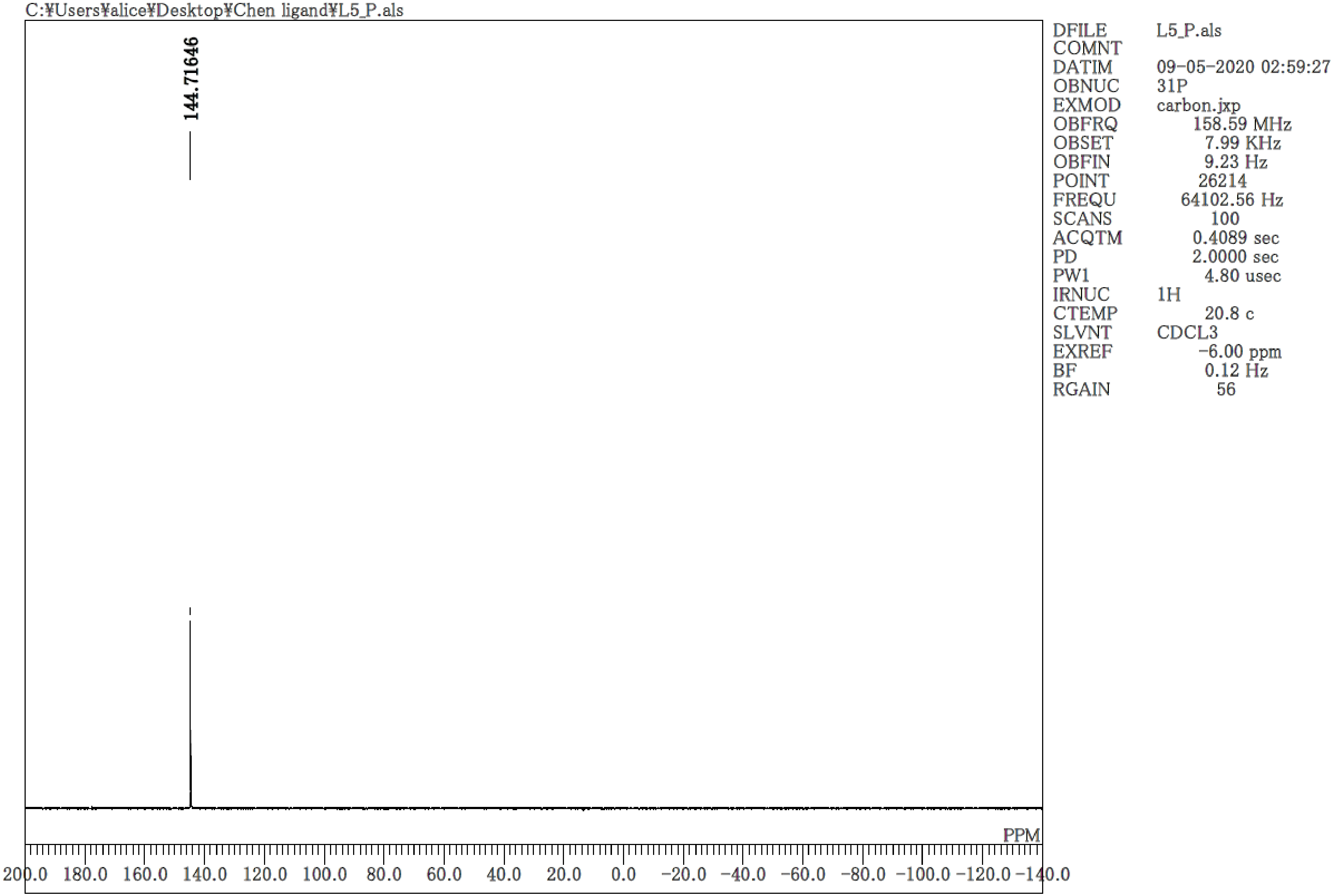


DFILE	L5_proton.als
COMNT	
DATIM	2019-08-19 14:28:30
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

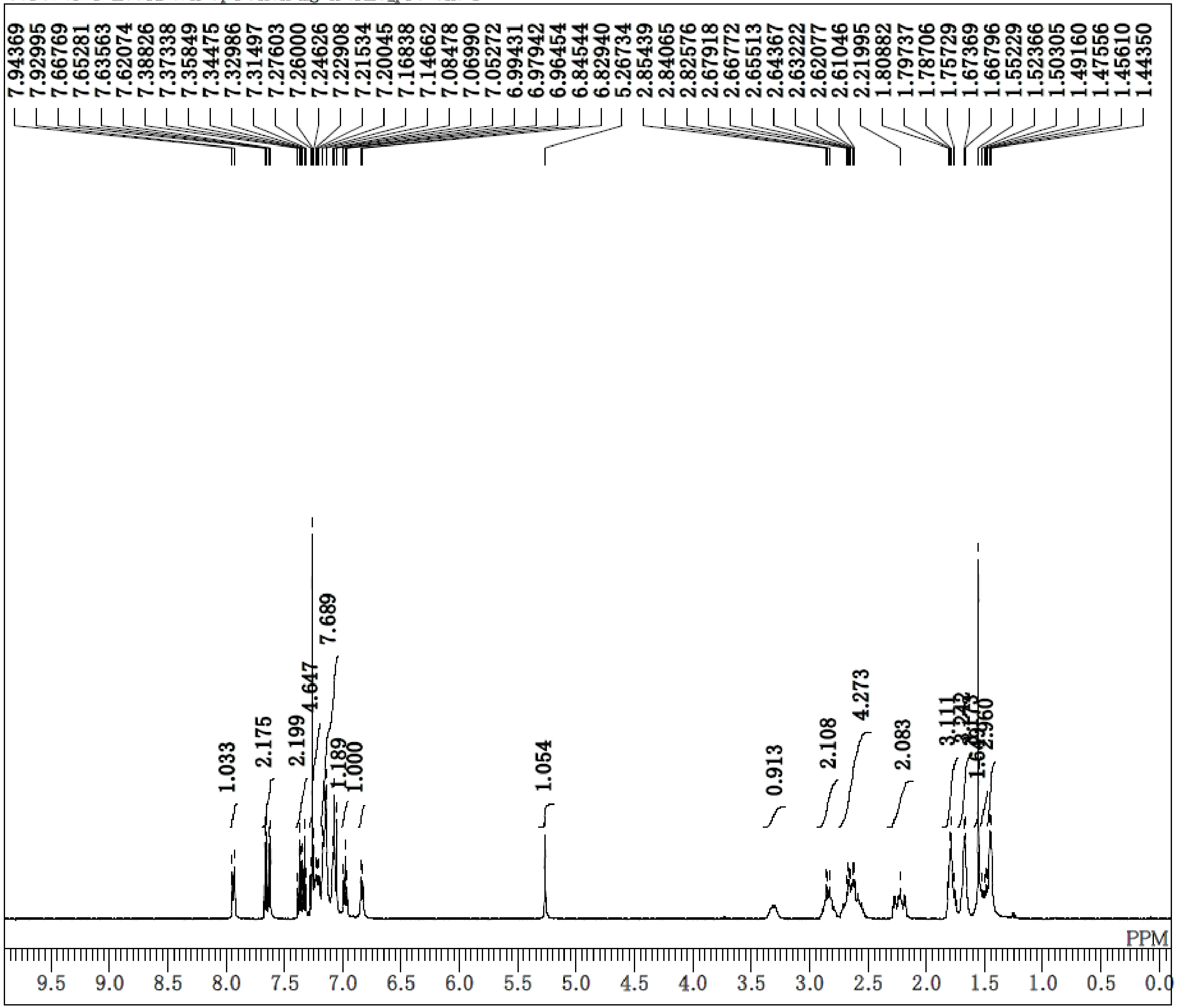


L5

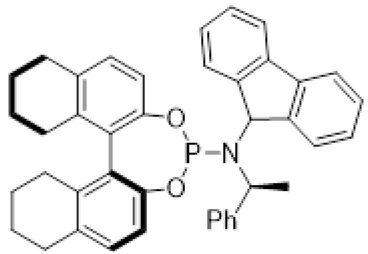

C:¥Users¥alice¥Desktop¥Chen ligand¥L5_carbon.als
149.45422
145.53391
144.50375
142.66283
140.94590
140.77421
132.83820
132.60928
131.42651
130.45359
130.43451
129.70958
128.30743
128.18343
128.08804
127.91635
127.87820
127.35358
127.13420
127.07697
126.86712
126.52374
126.31389
126.08497
125.92281
124.84496
124.44435
124.16773
124.12958
122.34589
121.53512
121.51604
119.49388
119.35081
77.25754
77.00000
76.74246
62.10091
56.65444
56.42552
26.20765
26.00734
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE L5_carbon.als
COMNT
DATIM 2020-04-16 20:23:02
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 620
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60


C:¥Users¥alice¥Desktop¥Chen ligand¥L5_P.als
144.71646
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L5_P.als
COMNT
DATIM 09-05-2020 02:59:27
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 100
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 56

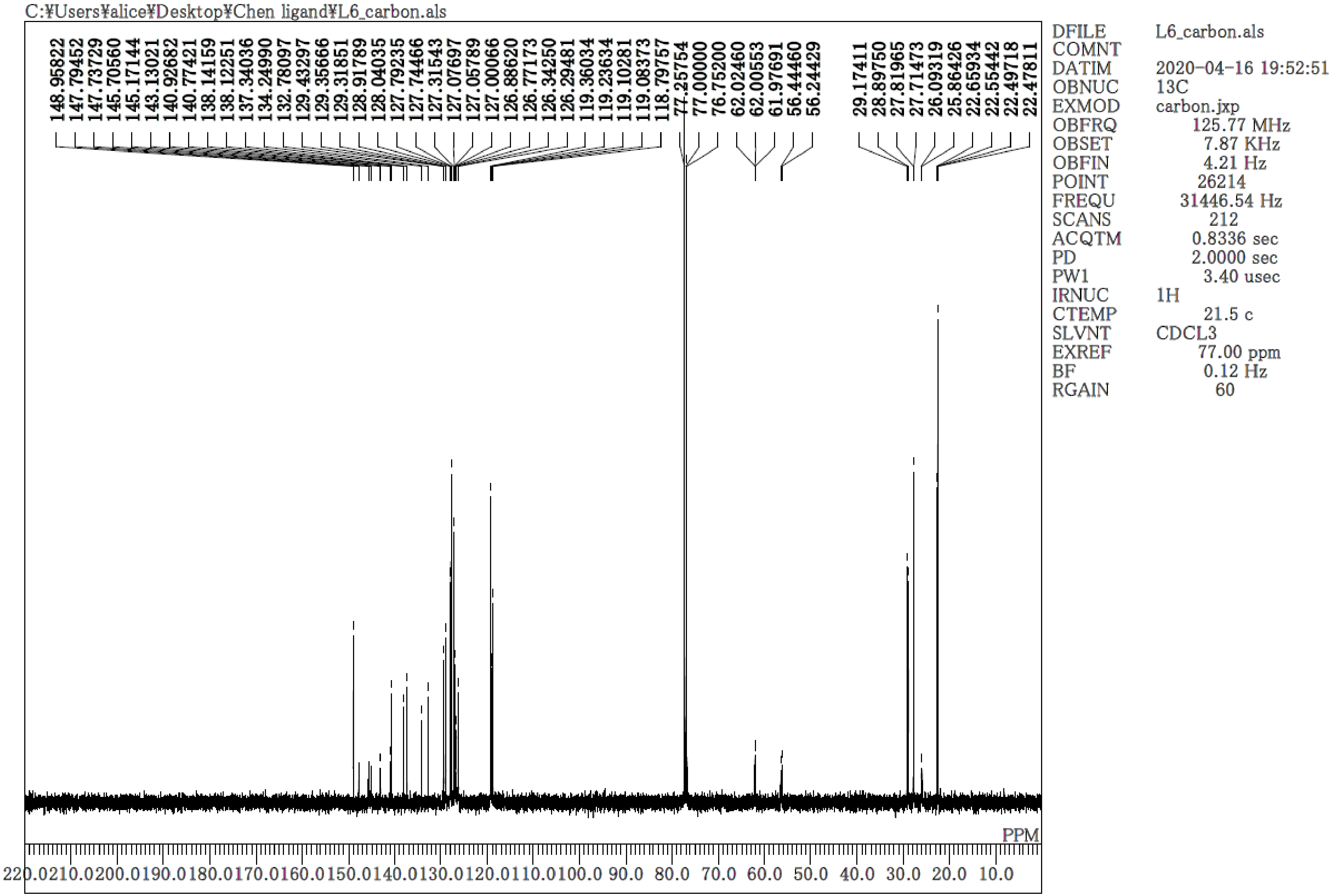
C:¥Users¥alice¥Desktop¥Chen ligand¥L6_proton.als

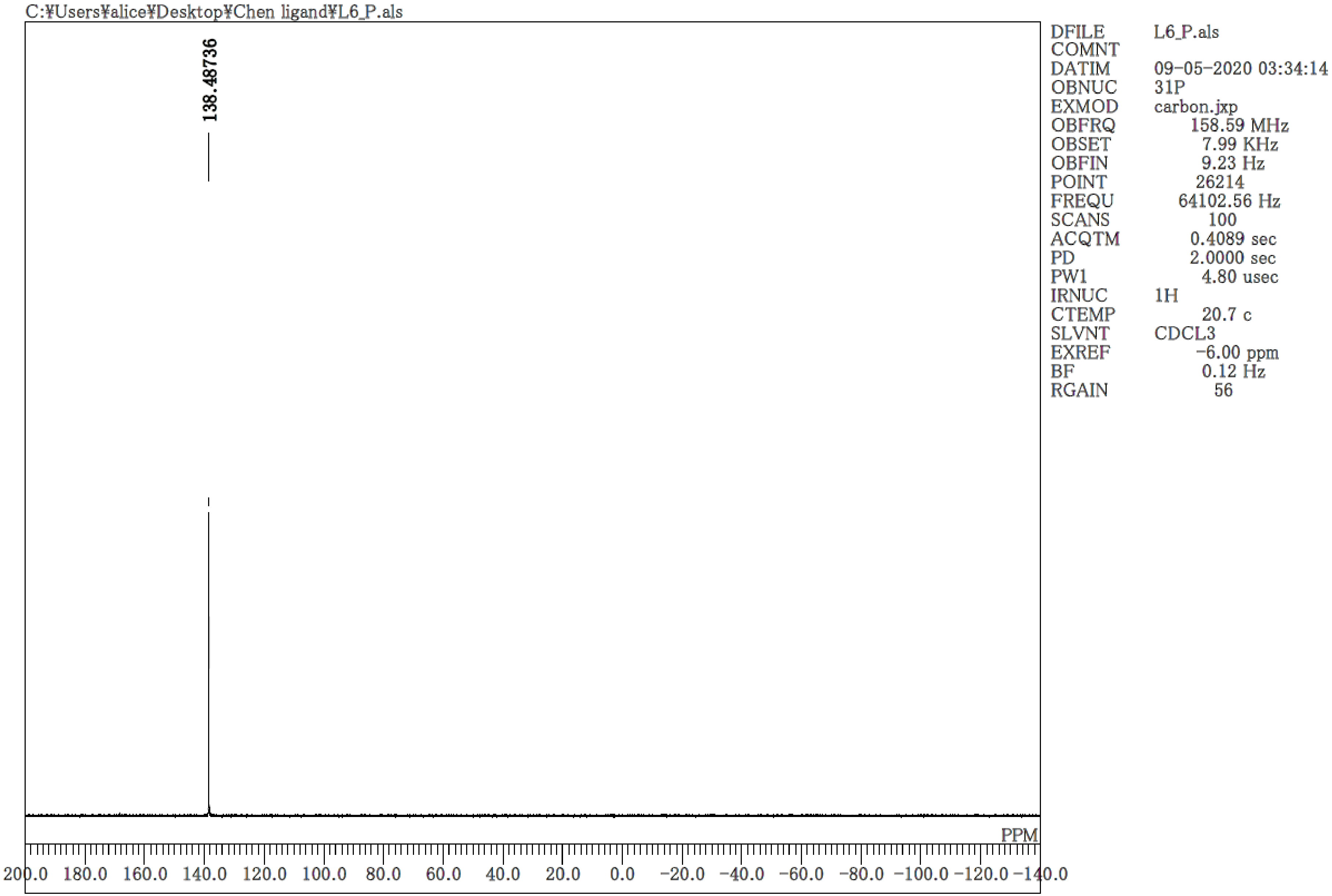


DFILE L6_proton.als
COMNT
DATIM 2020-04-16 15:46:41
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

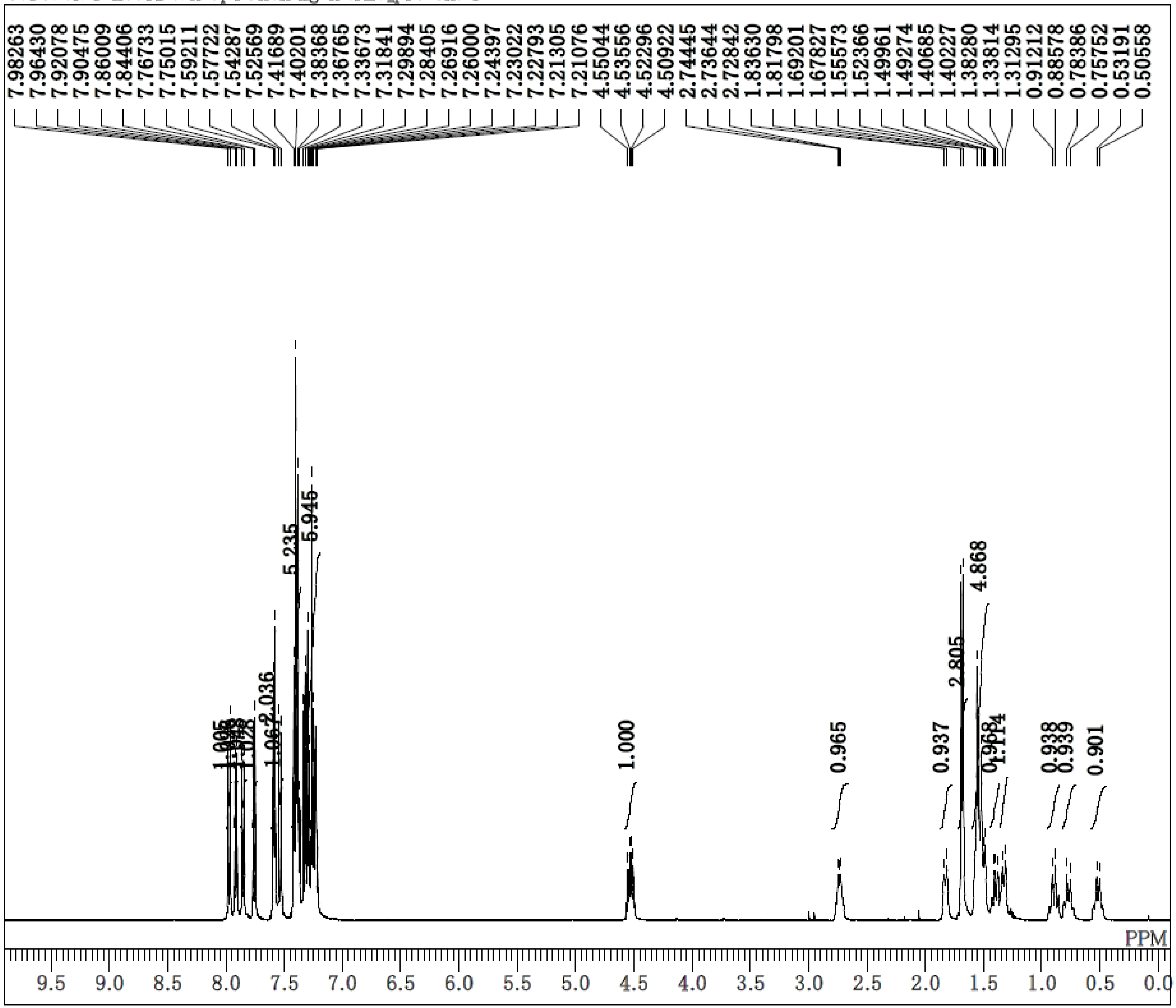


L6

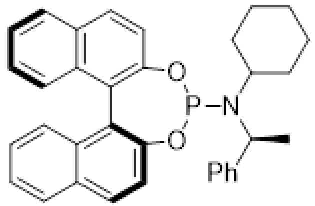

C:¥Users¥alice¥Desktop¥Chen ligand¥L6_carbon.als
148.95822
147.79452
147.73729
145.70560
145.17144
143.13021
140.92682
140.77421
138.14159
138.12251
137.34036
134.24990
132.78097
129.43297
129.35666
129.31851
128.91789
128.04035
127.79235
127.74466
127.31543
127.07697
127.05789
127.00066
126.88620
126.77173
126.34250
126.29481
119.36034
119.23634
119.10281
119.08373
118.79757
77.25754
77.00000
76.75200
62.02460
62.00553
61.97691
56.44460
56.24429
29.17411
28.89750
27.81965
27.71473
26.09319
25.86426
22.65934
22.55442
22.49718
22.47811
DFILE L6_carbon.als
COMNT
DATIM 2020-04-16 19:52:51
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 212
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0


C:¥Users¥alice¥Desktop¥Chen ligand¥L6_P.als
138.48736
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L6_P.als
COMNT
DATIM 09-05-2020 03:34:14
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 100
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 56

C:¥Users¥alice¥Desktop¥Chen ligand¥L7_proton.als

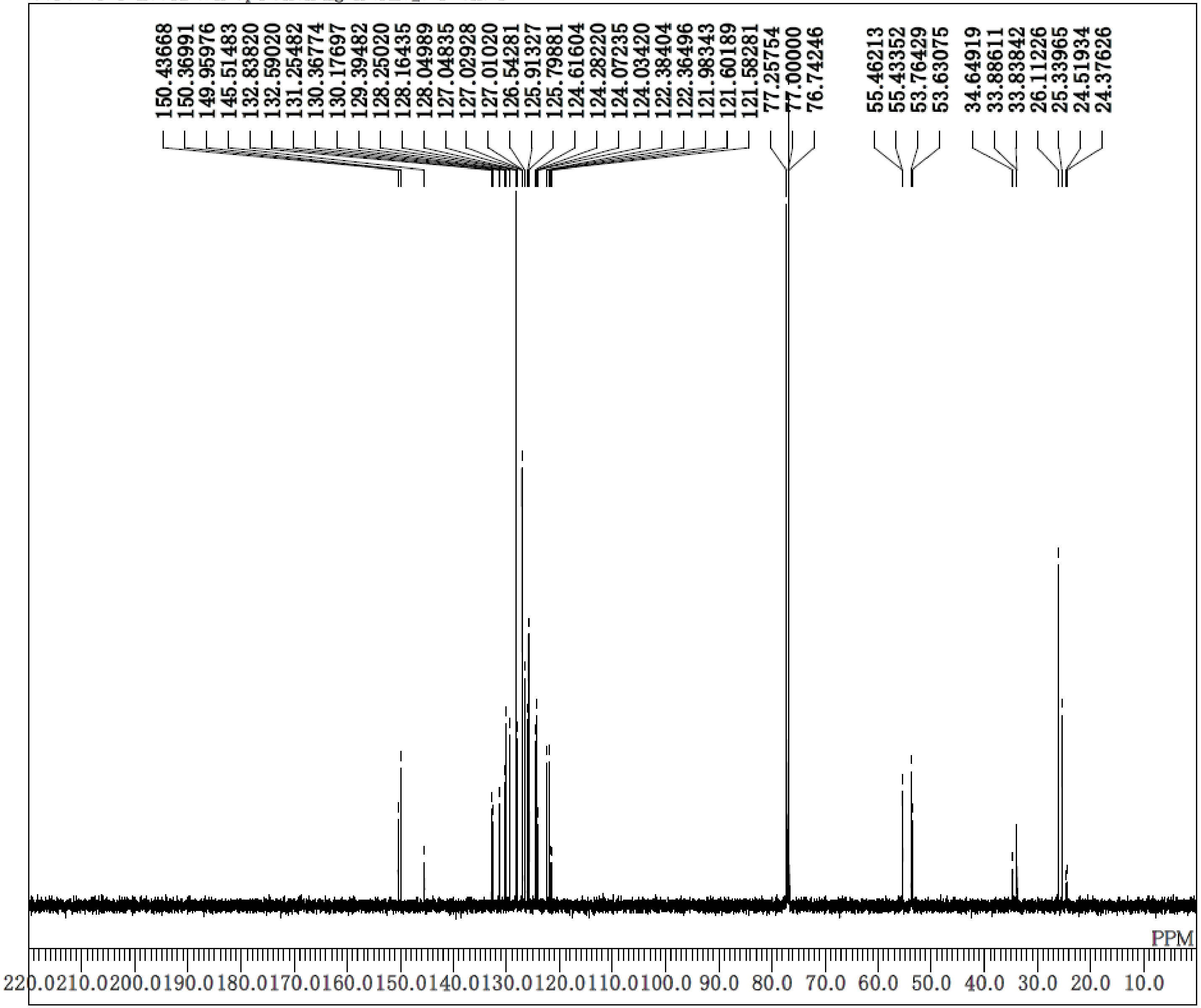


DFILE	L7_proton.als
COMNT	
DATIM	2020-04-17 14:34:21
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

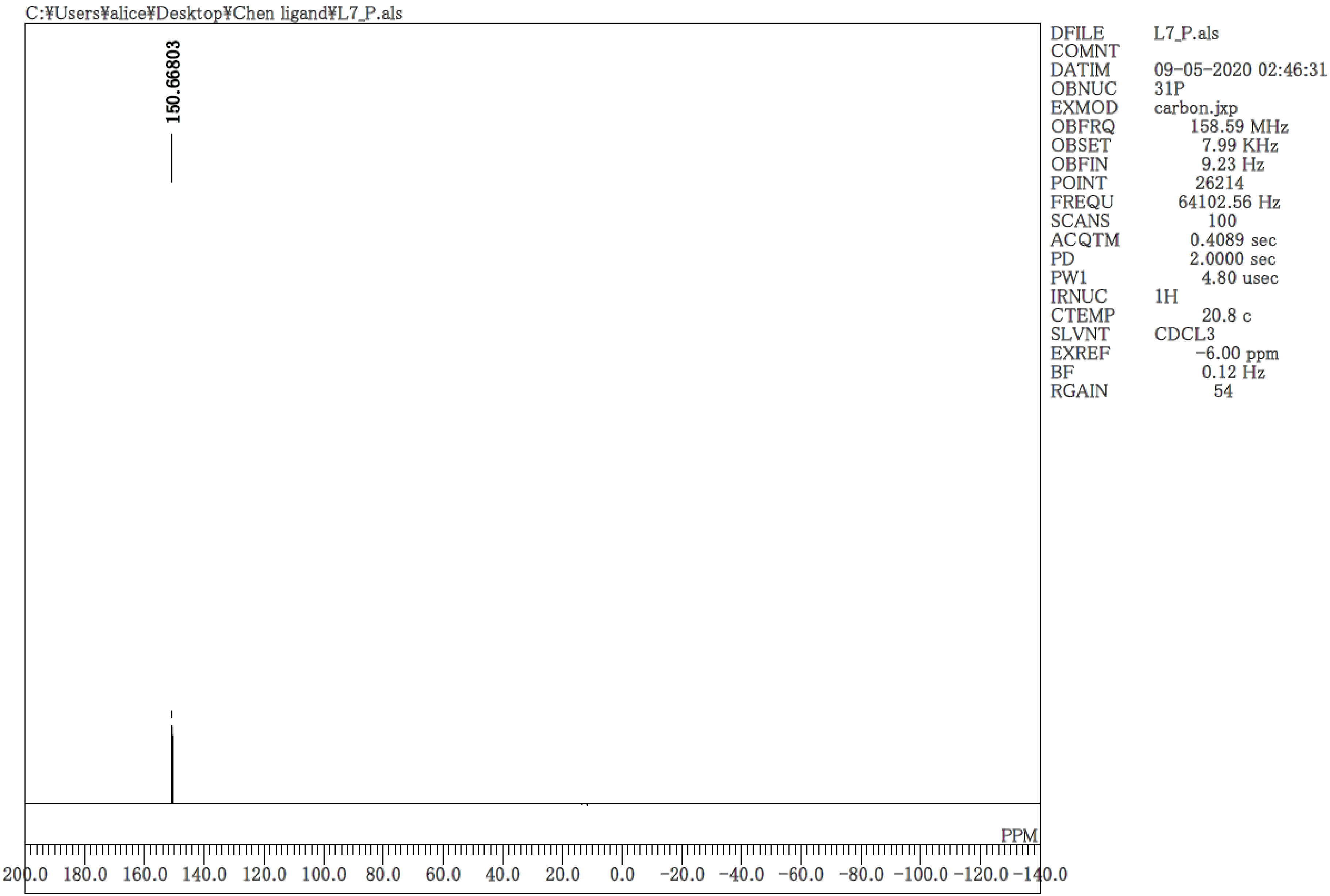


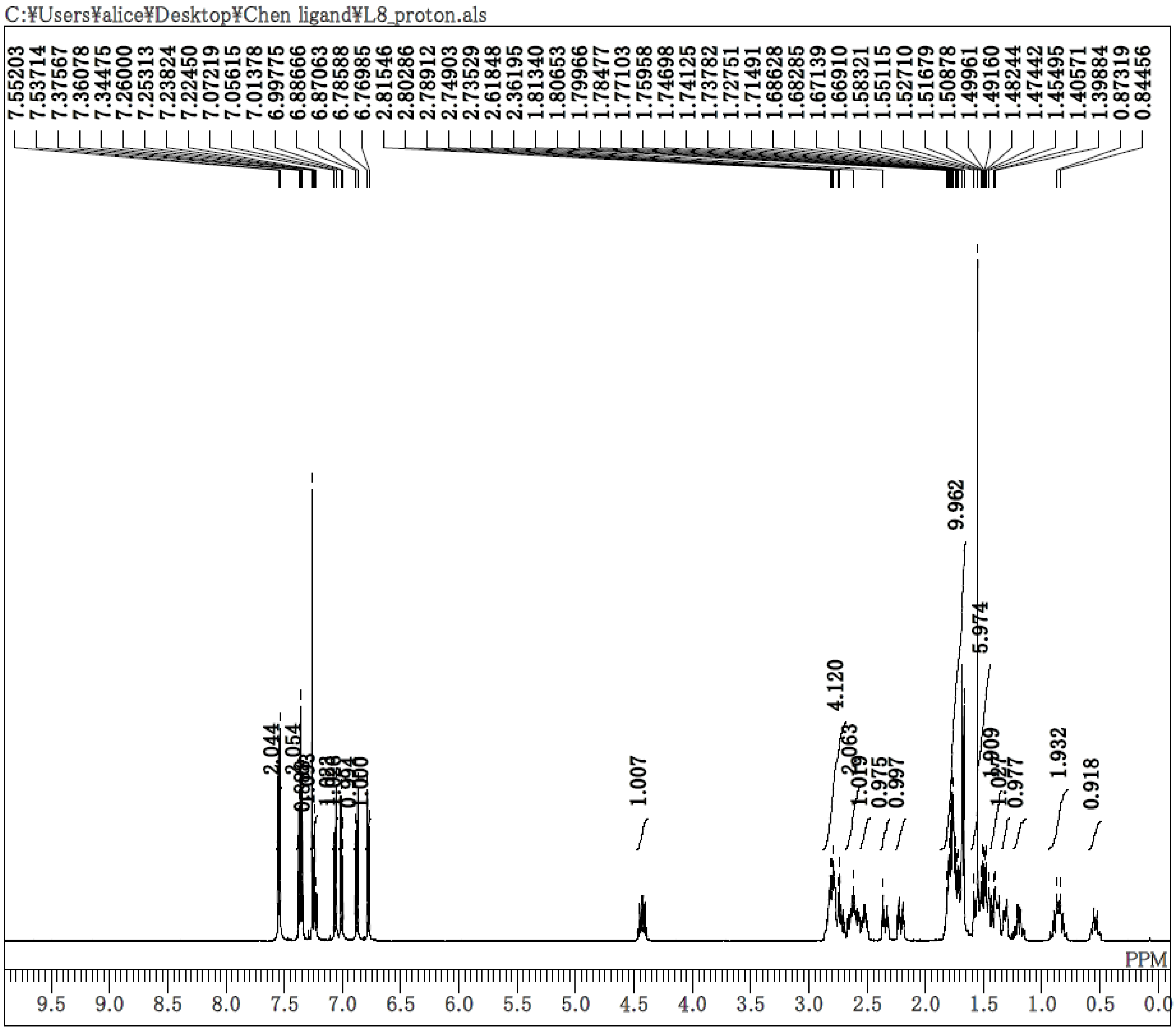
L7

C:¥Users¥alice¥Desktop¥Chen ligand¥L7_carbon.als

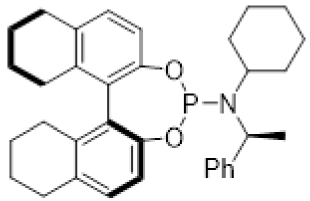


DFILE L7_carbon.als
COMNT
DATIM 2020-04-17 14:47:41
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 361
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

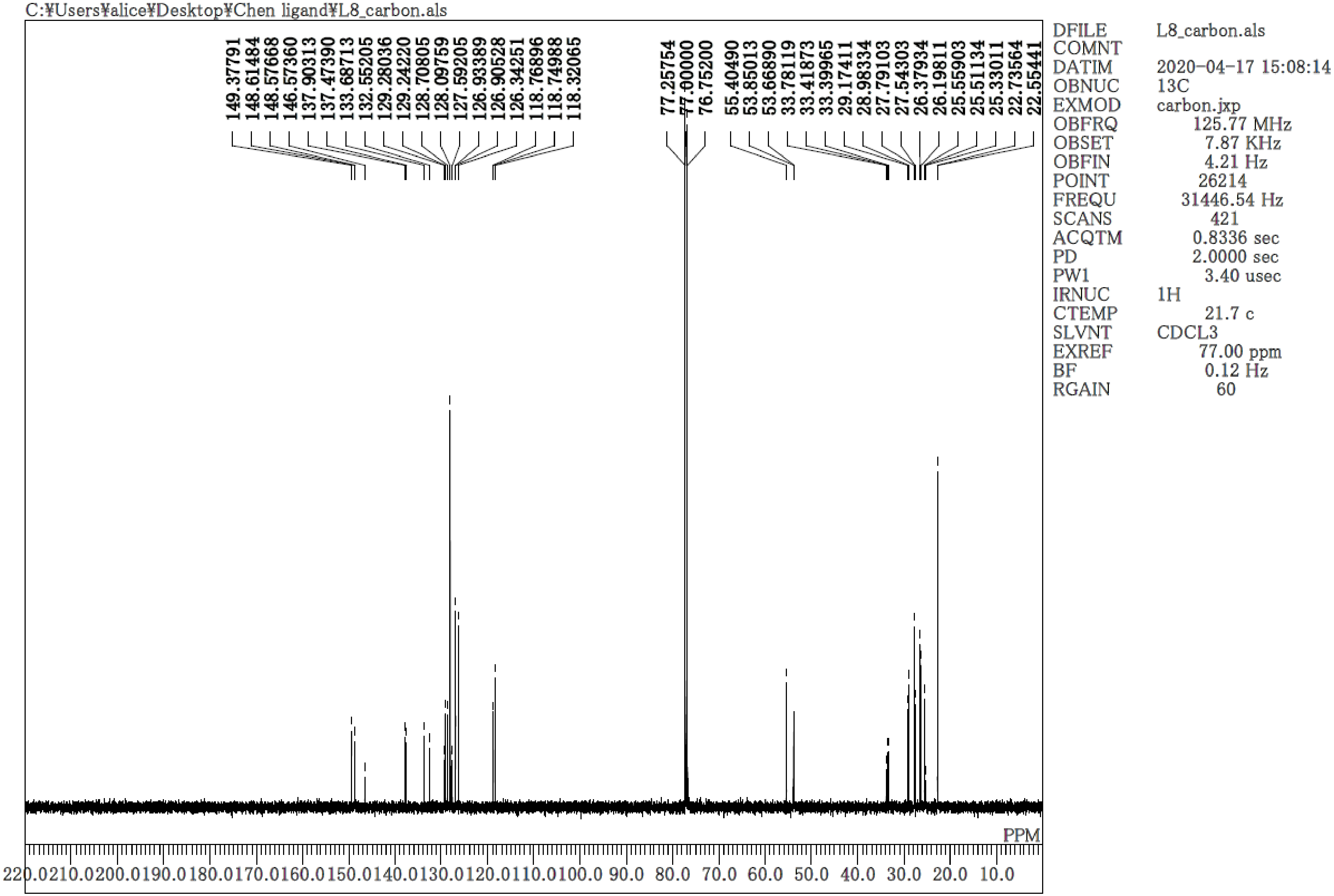

C:¥Users¥alice¥Desktop¥Chen ligand¥L7_P.als
150.66803
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L7_P.als
COMNT
DATIM 09−05−2020 02:46:31
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 100
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 54

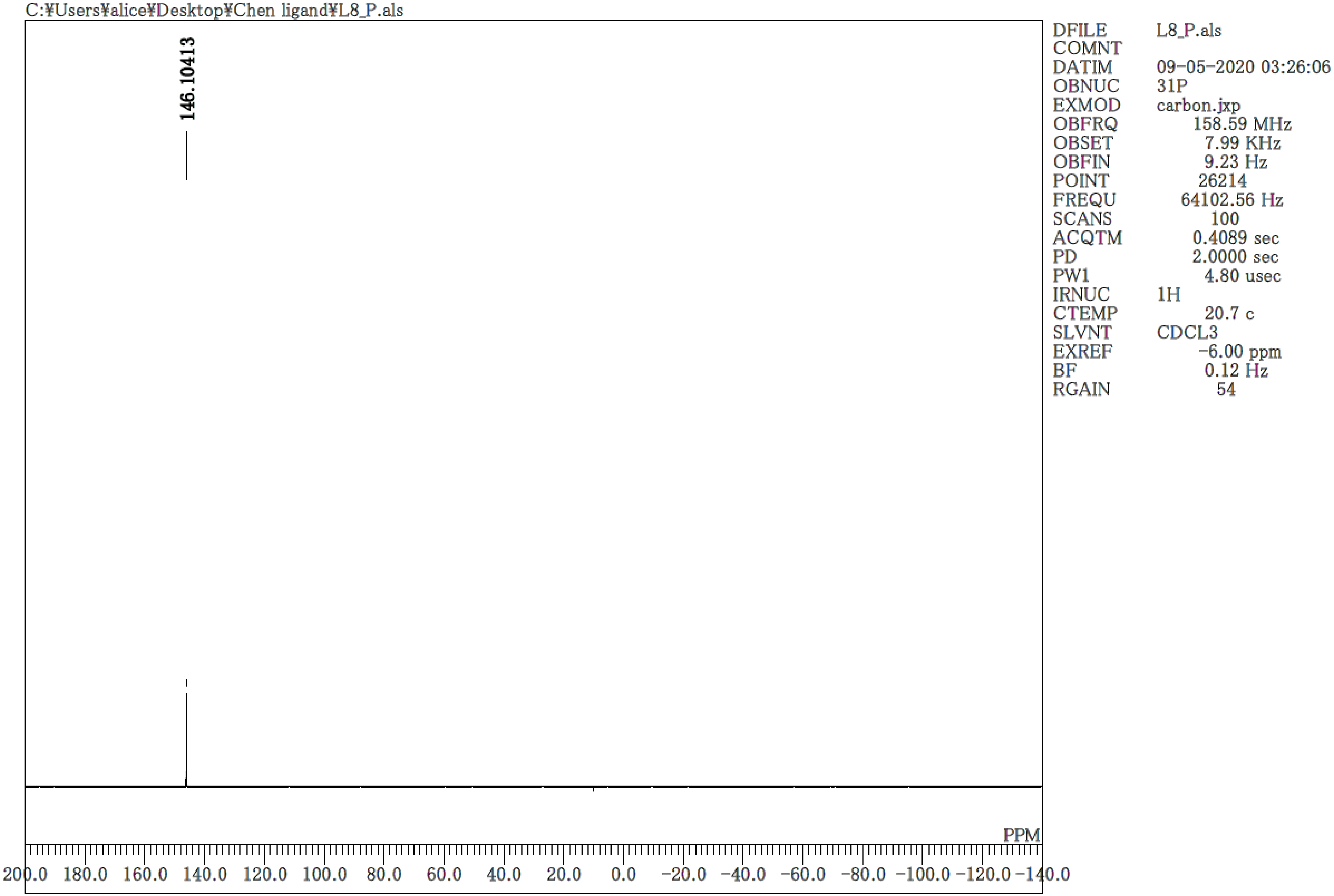


DFILE	L8_proton.als
COMNT	
DATIM	2020-04-17 14:39:33
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

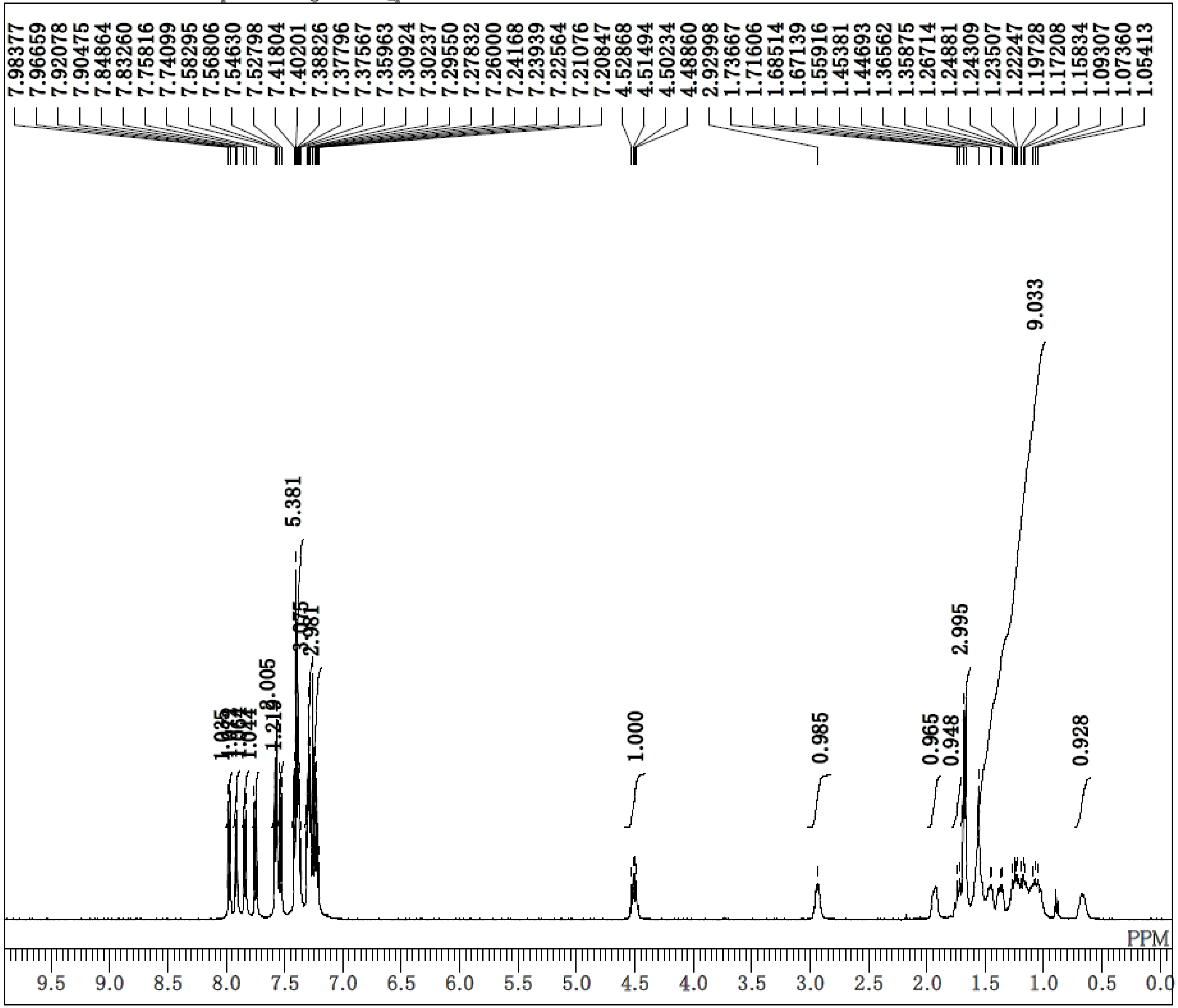


L8

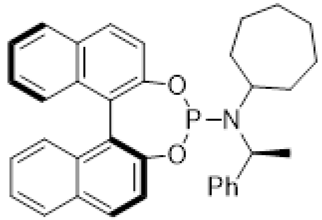

C:¥Users¥alice¥Desktop¥Chen ligand¥L8_carbon.als
149.37791
148.61484
148.57668
146.57360
137.90313
137.47390
133.68713
132.55205
129.28036
129.24220
128.70805
128.09759
127.59205
126.93389
126.90528
126.34251
118.76896
118.74988
118.32065
77.25754
77.00000
76.75200
55.40490
53.85013
53.66890
33.78119
33.41873
33.39965
29.17411
28.98334
27.79103
27.54303
26.37934
26.19811
25.55903
25.51134
25.33011
22.73564
22.55441
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE L8_carbon.als
COMNT
DATIM 2020-04-17 15:08:14
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 421
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60


C:¥Users¥alice¥Desktop¥Chen ligand¥L8_P.als
146.10413
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L8_P.als
COMNT
DATIM 09-05-2020 03:26:06
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 100
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 54

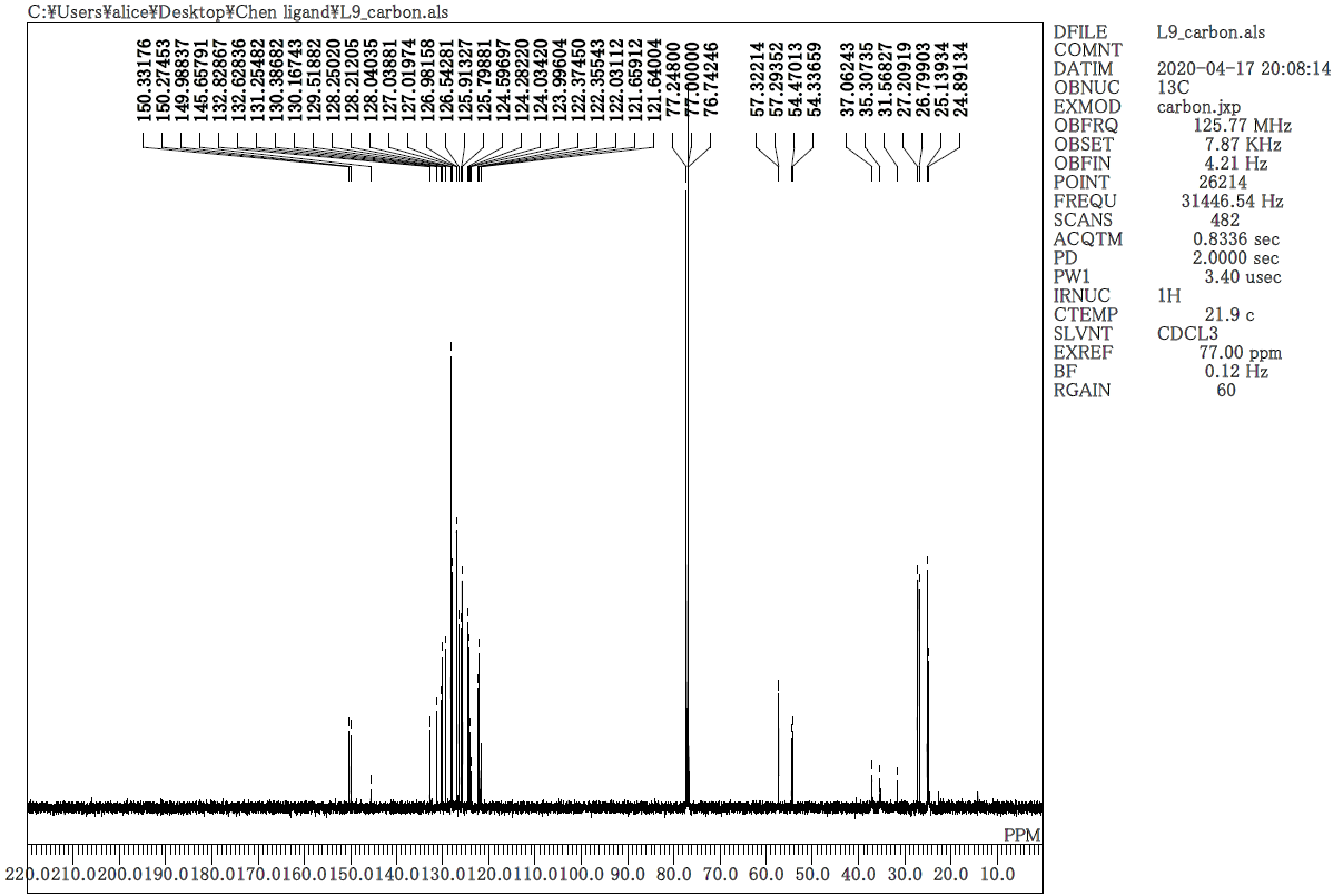
C:¥Users¥alice¥Desktop¥Chen ligand¥L9_proton.als



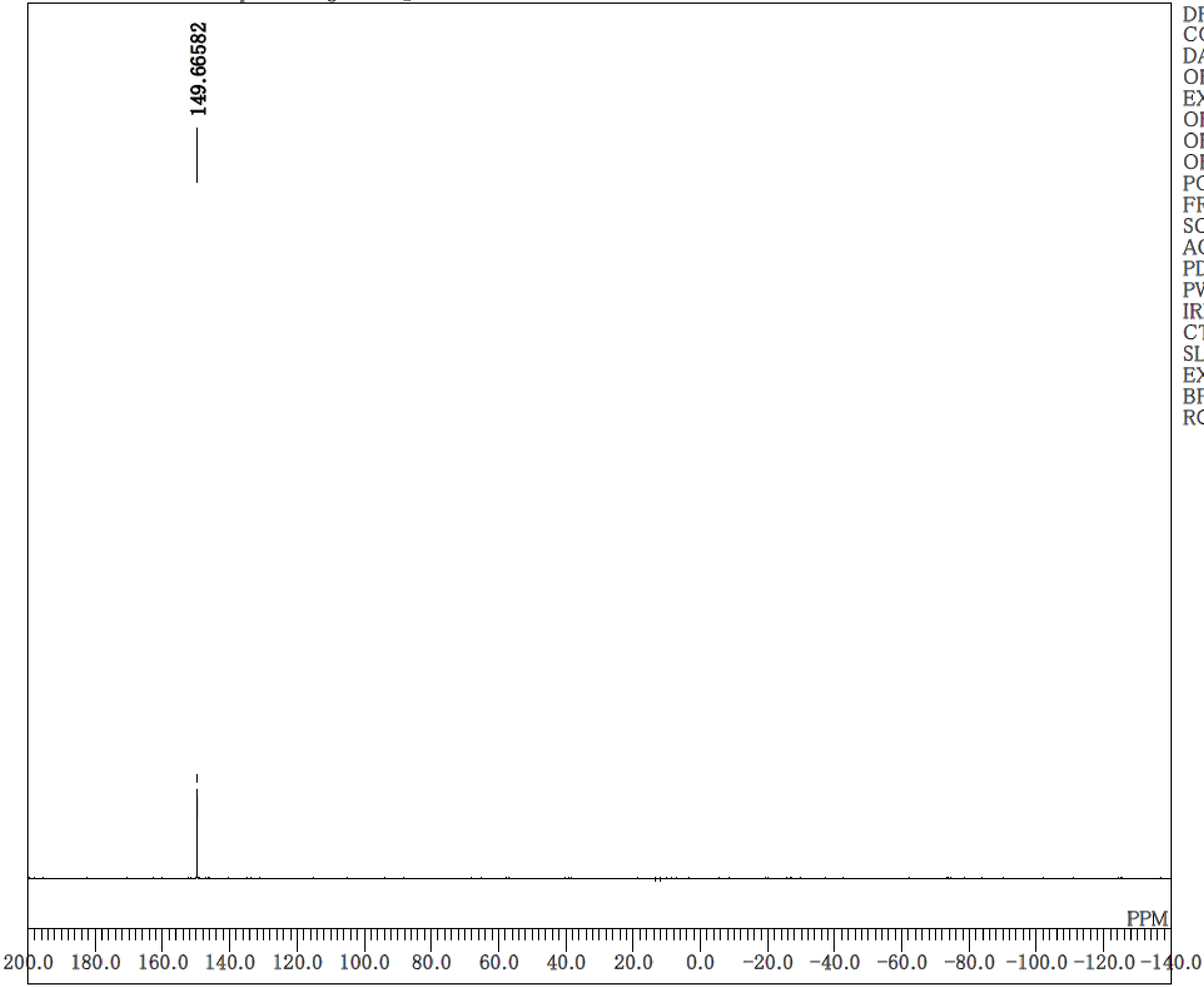
DFILE	L9_proton.als
COMNT	
DATIM	2020-04-17 19:57:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



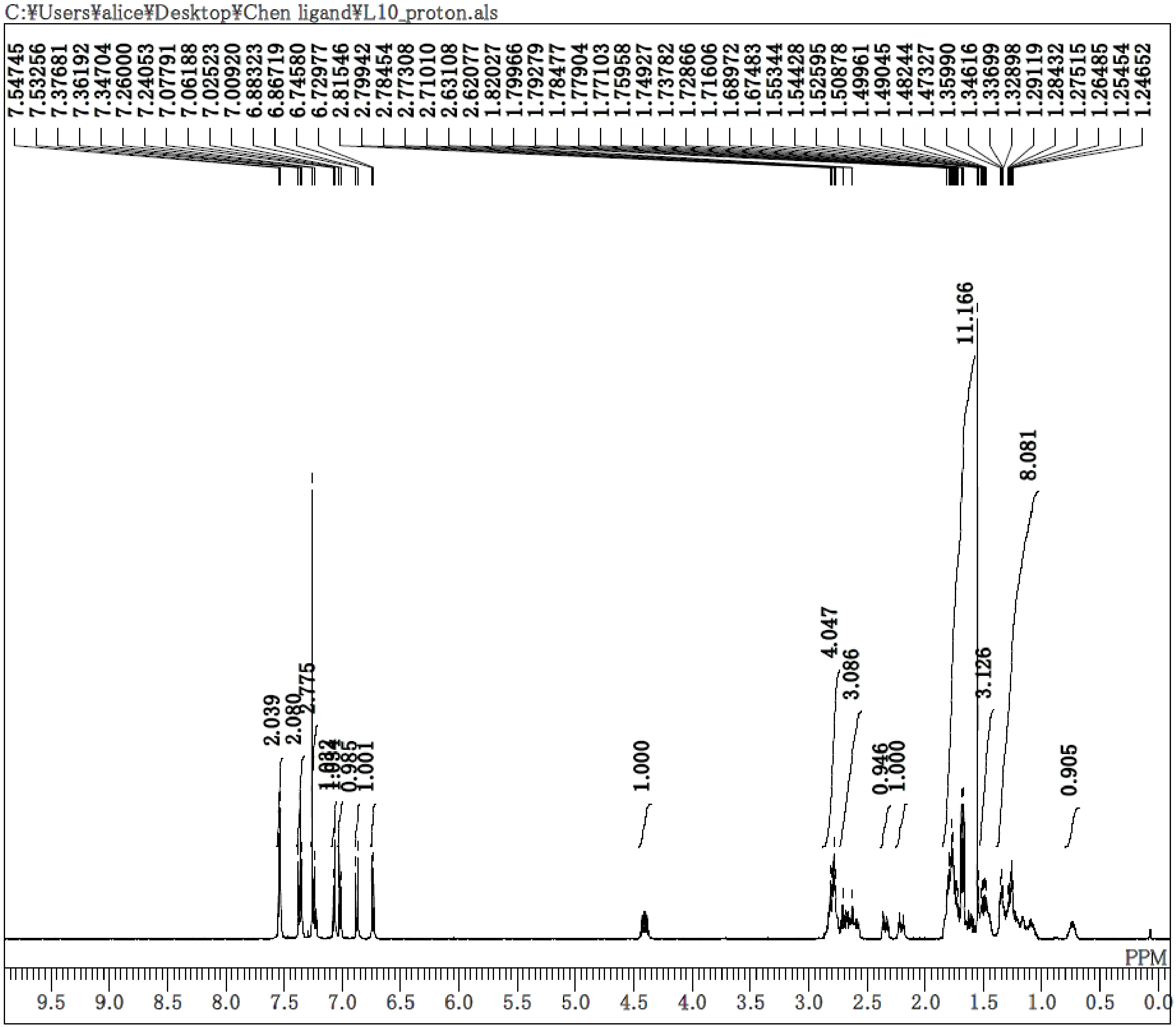
L9


C:¥Users¥alice¥Desktop¥Chen ligand¥L9_carbon.als
150.33176
150.27453
149.98837
145.65791
132.82867
132.62836
131.25482
130.38682
130.16743
129.51882
128.25020
128.21205
128.04035
127.03881
127.01974
126.98158
126.54281
125.91327
125.79881
124.59697
124.28220
124.03420
123.99604
122.37450
122.35543
122.03112
121.65912
121.64004
77.24800
77.00000
76.74246
57.32214
57.29352
54.47013
54.33659
37.06243
35.30735
31.56827
27.20919
26.79903
25.13934
24.89134
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE L9_carbon.als
COMNT
DATIM 2020-04-17 20:08:14
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 482
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

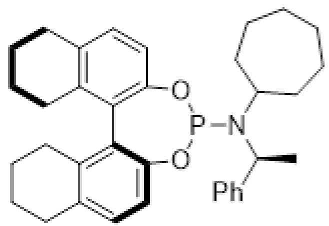
C:¥Users¥alice¥Desktop¥Chen ligand¥L9_P.als



DFILE	L9_P.als
COMNT	
DATIM	09-05-2020 02:24:20
OBNUC	31P
EXMOD	carbon.jxp
OBFRQ	158.59 MHz
OBSET	7.99 KHz
OBFIN	9.23 Hz
POINT	26214
FREQU	64102.56 Hz
SCANS	100
ACQTM	0.4089 sec
PD	2.0000 sec
PW1	4.80 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	−6.00 ppm
BF	0.12 Hz
RGAIN	54

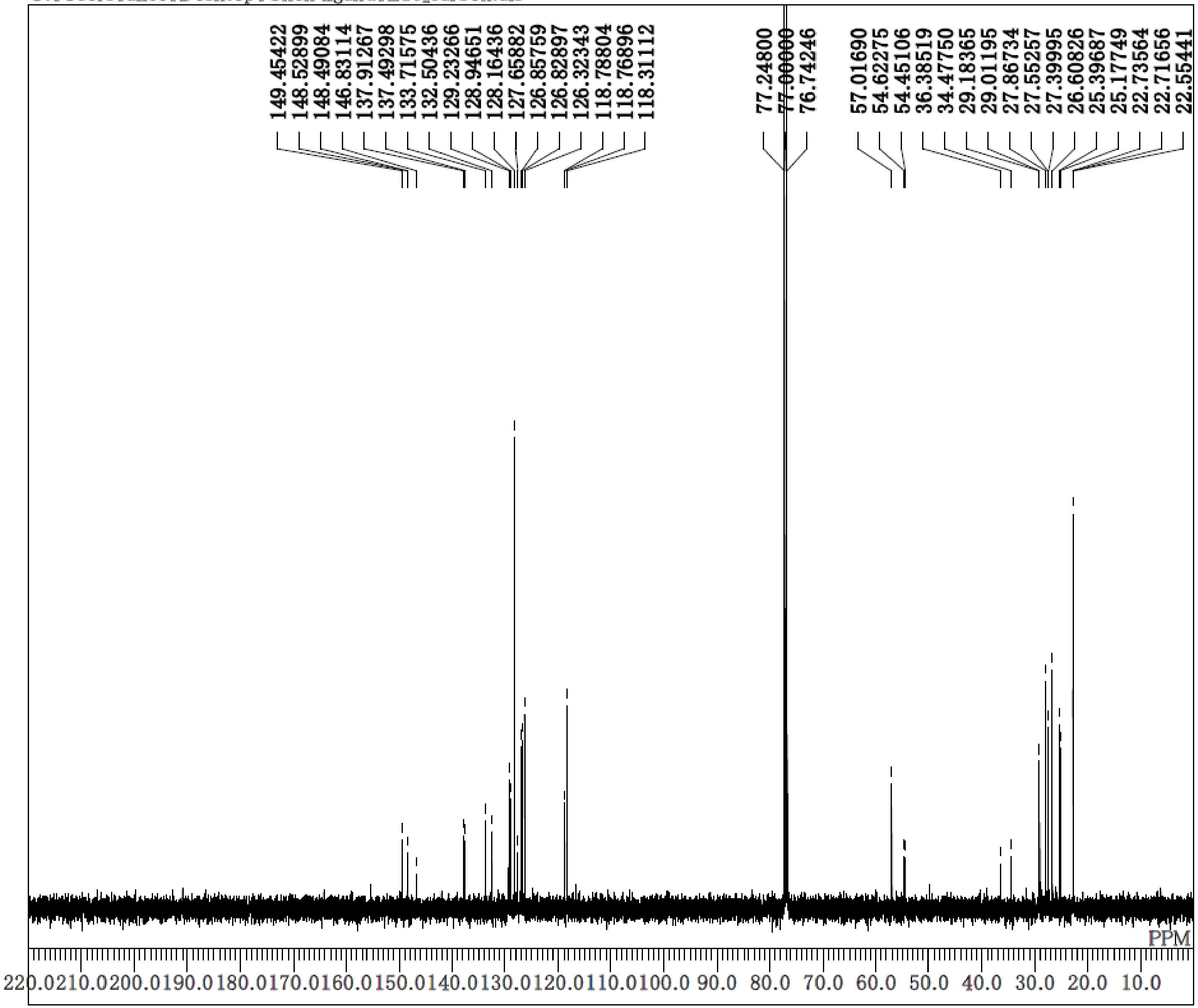


DFILE	L10_proton.als
COMNT	
DATIM	2020-04-17 20:02:60
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



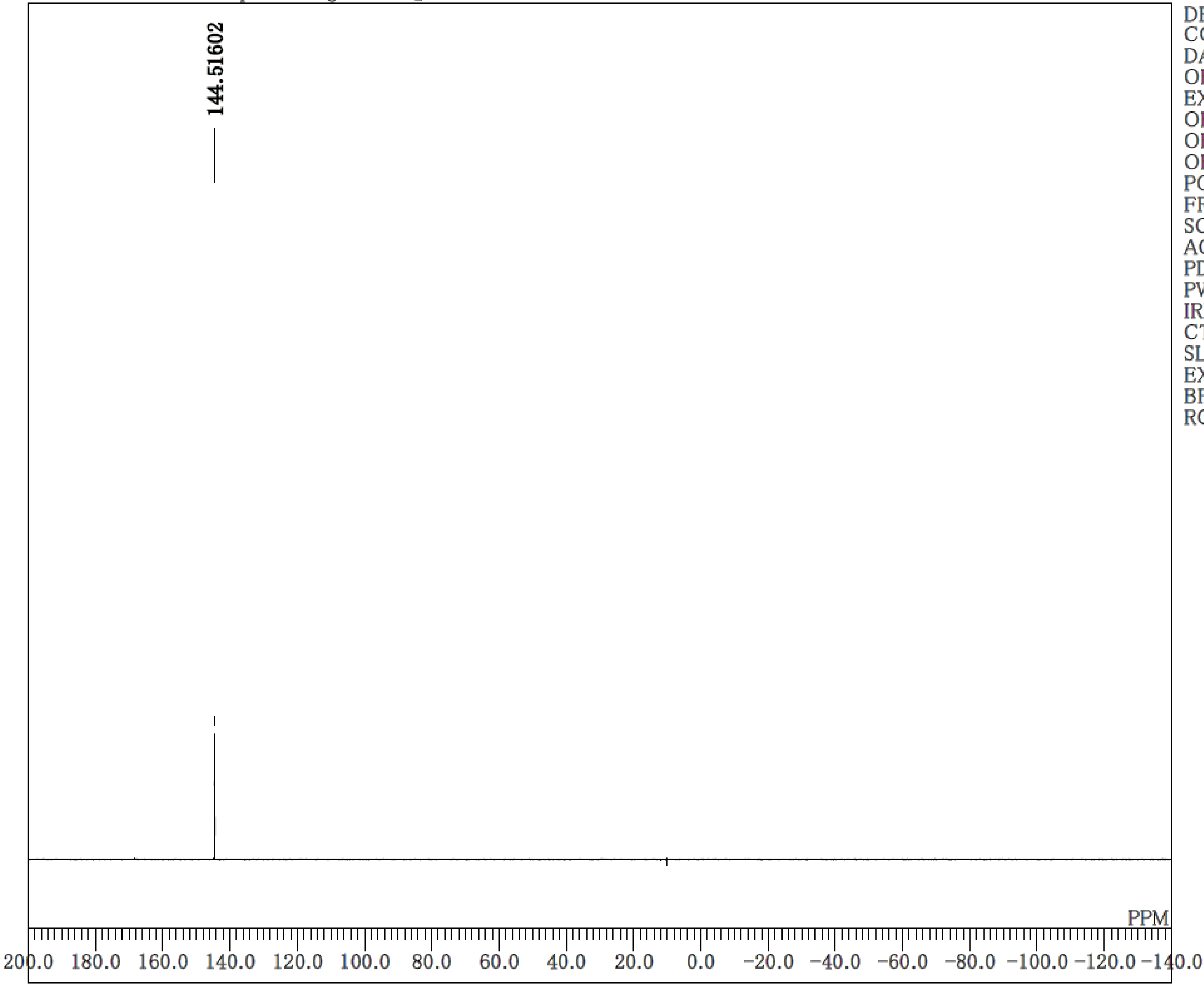
L10

C:¥Users¥alice¥Desktop¥Chen ligand¥L10_carbon.als



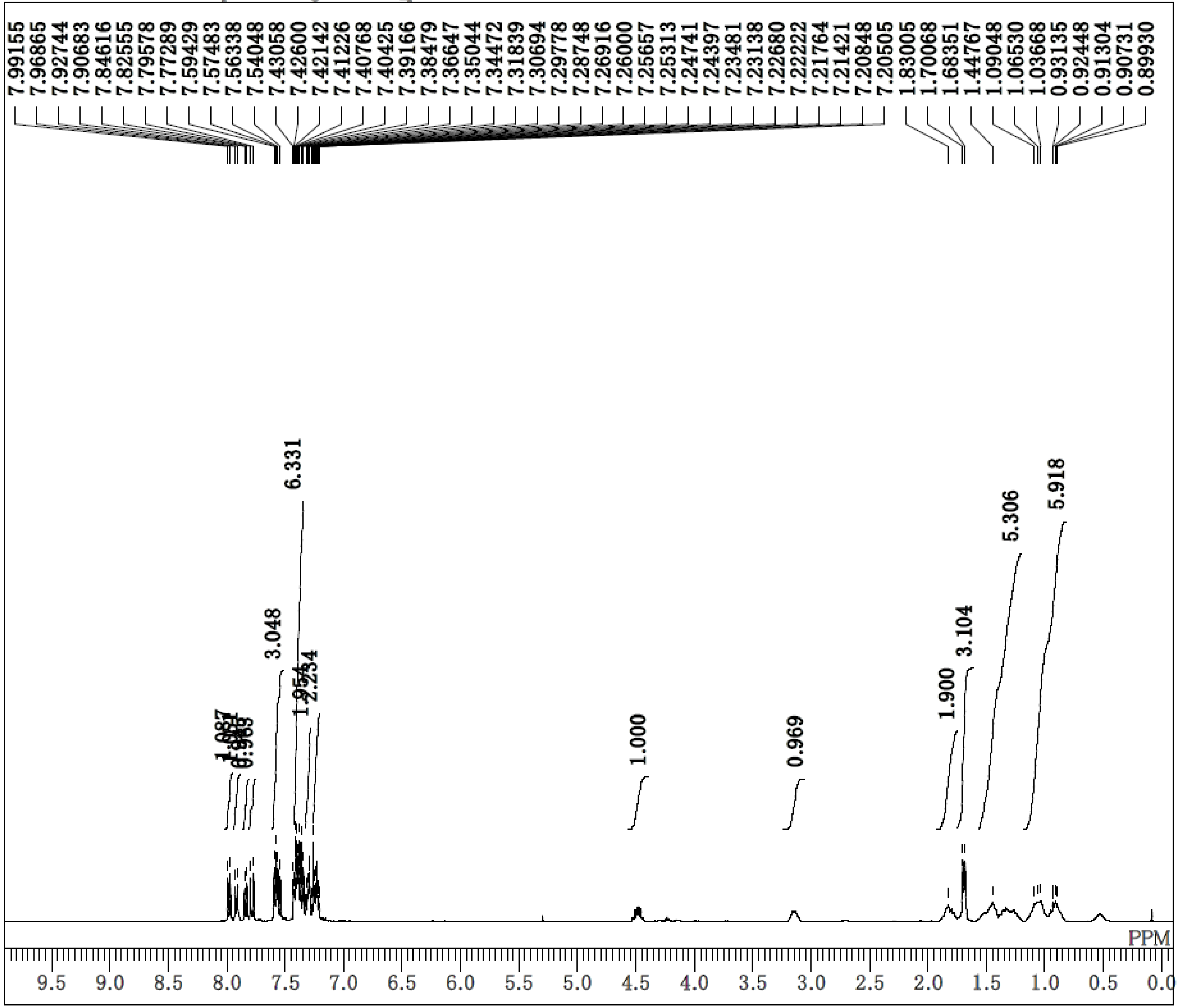
DFILE	L10_carbon.als
COMNT	
DATIM	2020-04-17 20:35:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	584
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥Chen ligand¥L10_P.als

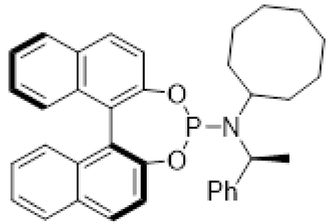


DFILE	L10_P.als
COMNT	
DATIM	09-05-2020 03:11:38
OBNUC	31P
EXMOD	carbon.jxp
OBFRQ	158.59 MHz
OBSET	7.99 KHz
OBFIN	9.23 Hz
POINT	26214
FREQU	64102.56 Hz
SCANS	100
ACQTM	0.4089 sec
PD	2.0000 sec
PW1	4.80 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	−6.00 ppm
BF	0.12 Hz
RGAIN	56

C:¥Users¥alice¥Desktop¥Chen ligand¥L11_proton.als

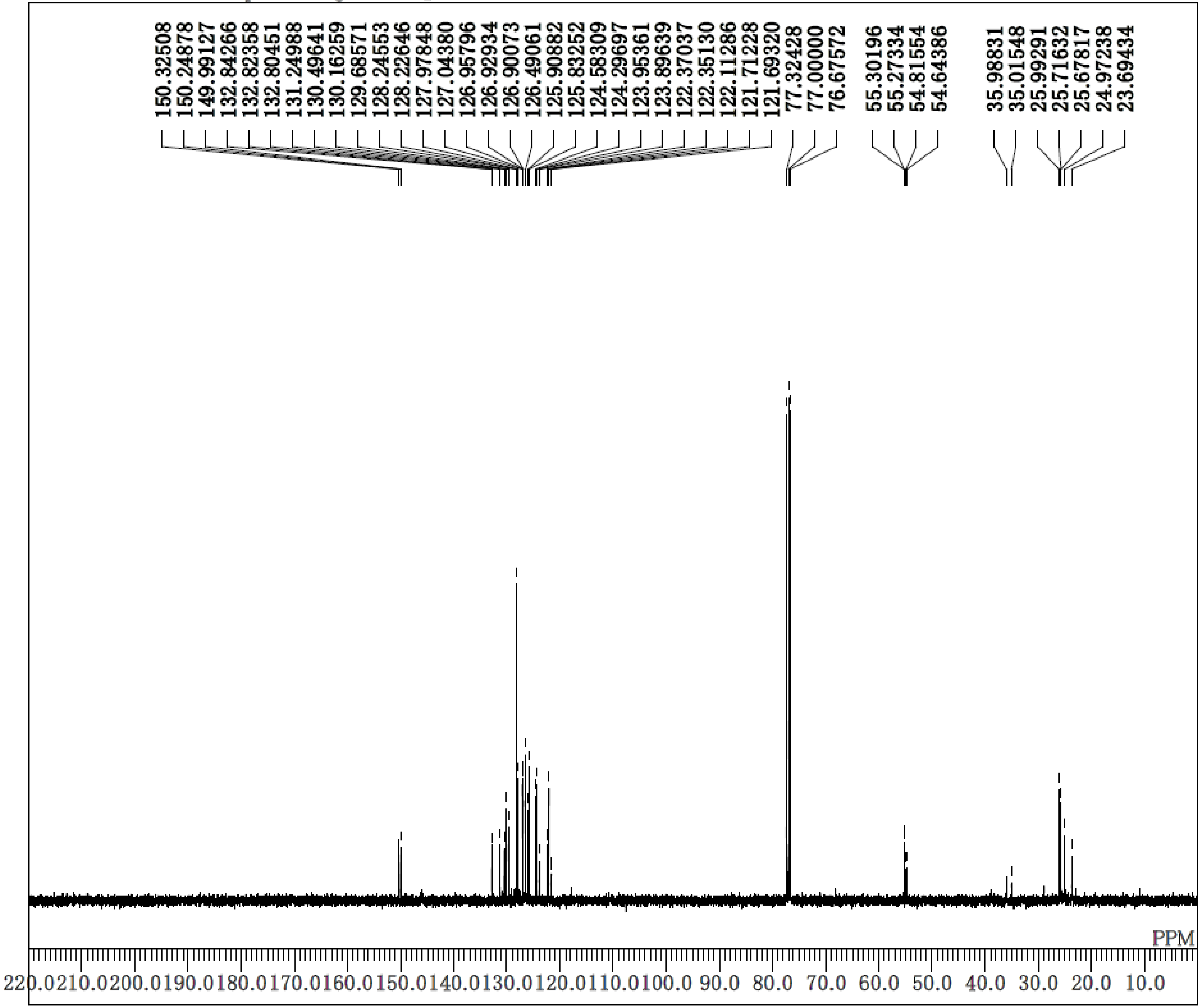


DFILE	L11_proton.als
COMNT	
DATIM	18-04-2020 16:16:58
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34

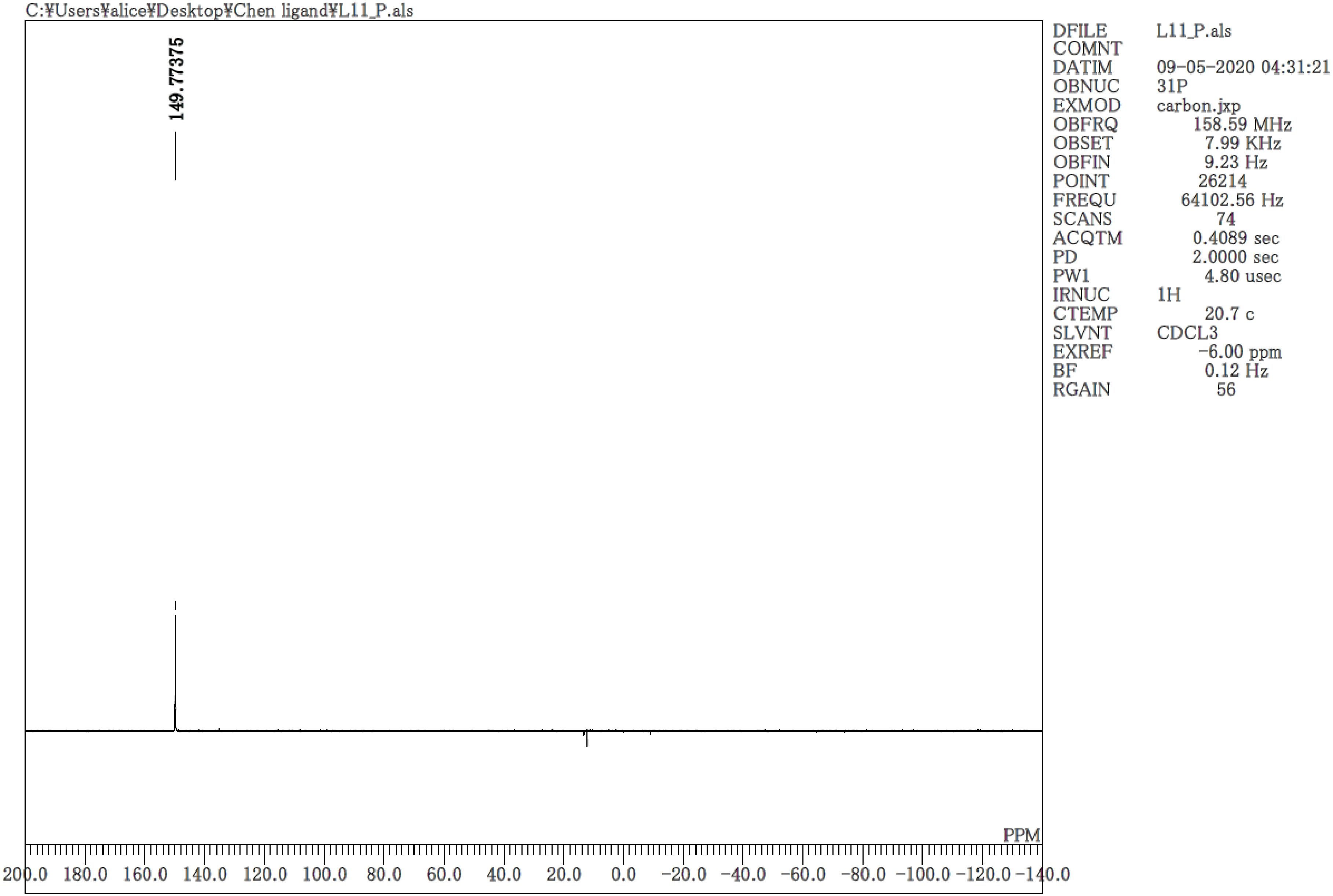


L11

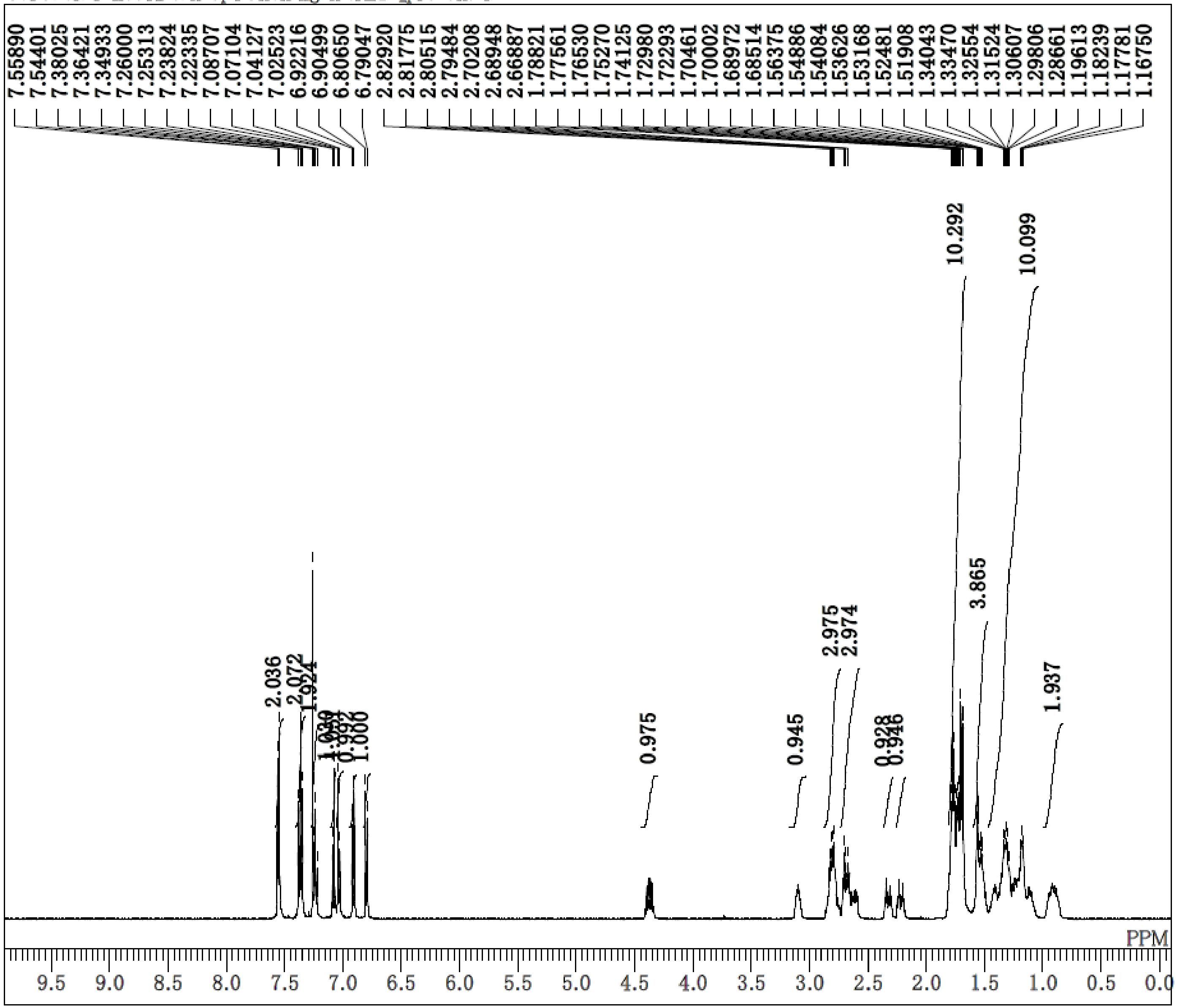
C:¥Users¥alice¥Desktop¥Chen ligand¥L11_carbon.als



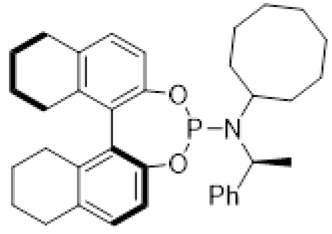
DFILE	L11_carbon.als
COMNT	
DATIM	18-04-2020 16:42:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	434
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60


C:¥Users¥alice¥Desktop¥Chen ligand¥L11_P.als
149.77375
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L11_P.als
COMNT
DATIM 09-05-2020 04:31:21
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 74
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 56

C:¥Users¥alice¥Desktop¥Chen ligand¥L12_proton.als

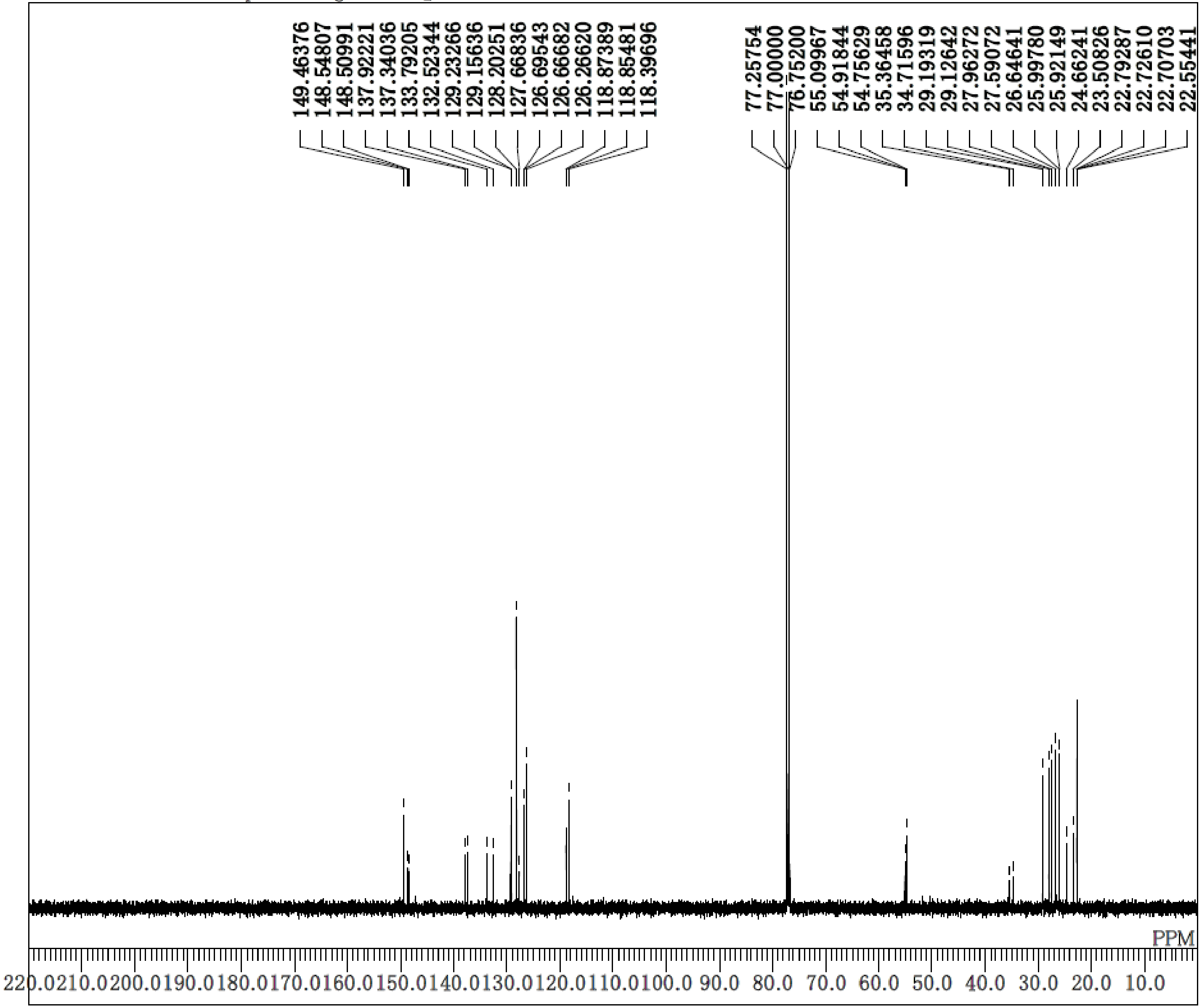


DFILE L12_proton.als
COMNT
DATIM 2020-04-17 22:47:54
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40

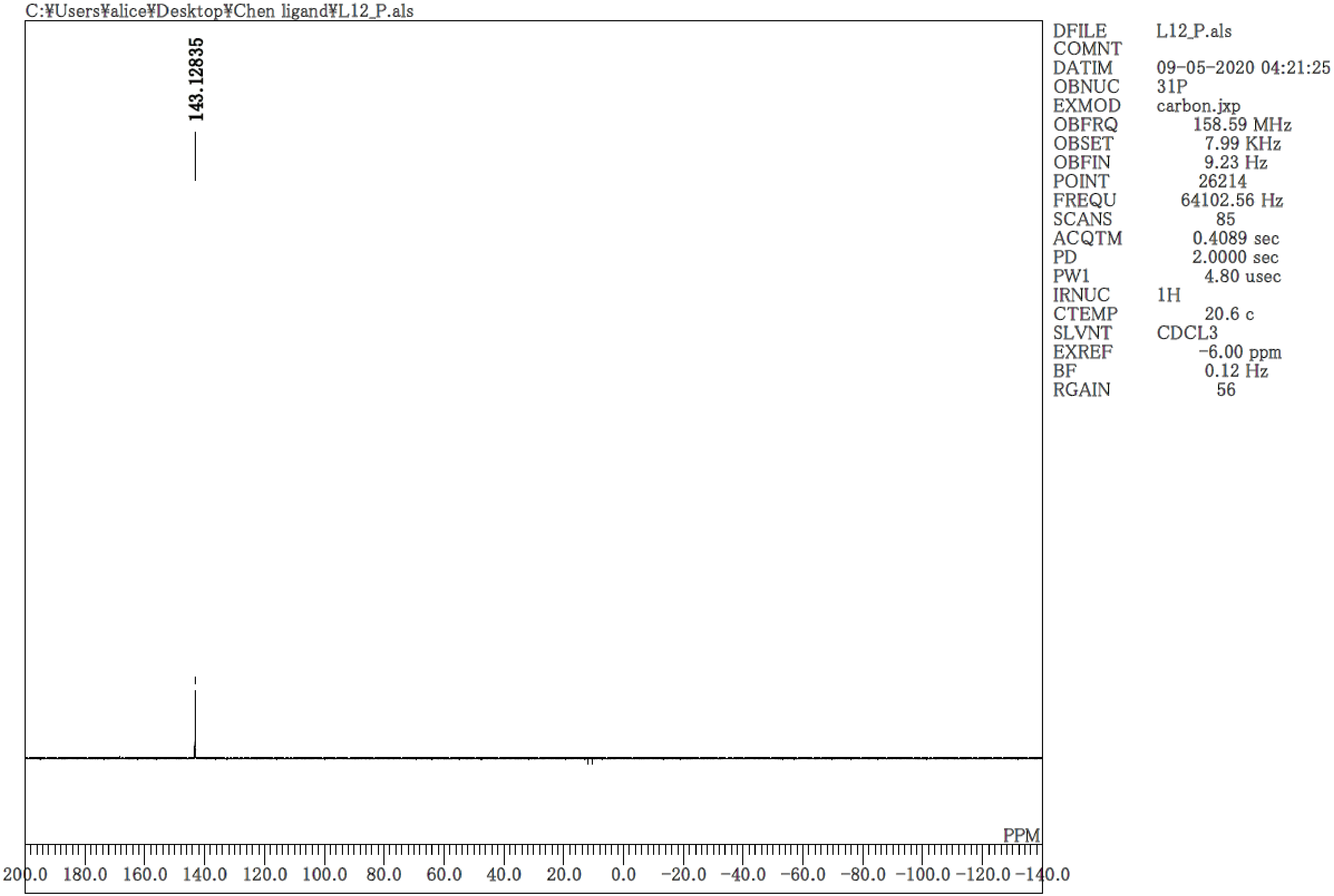


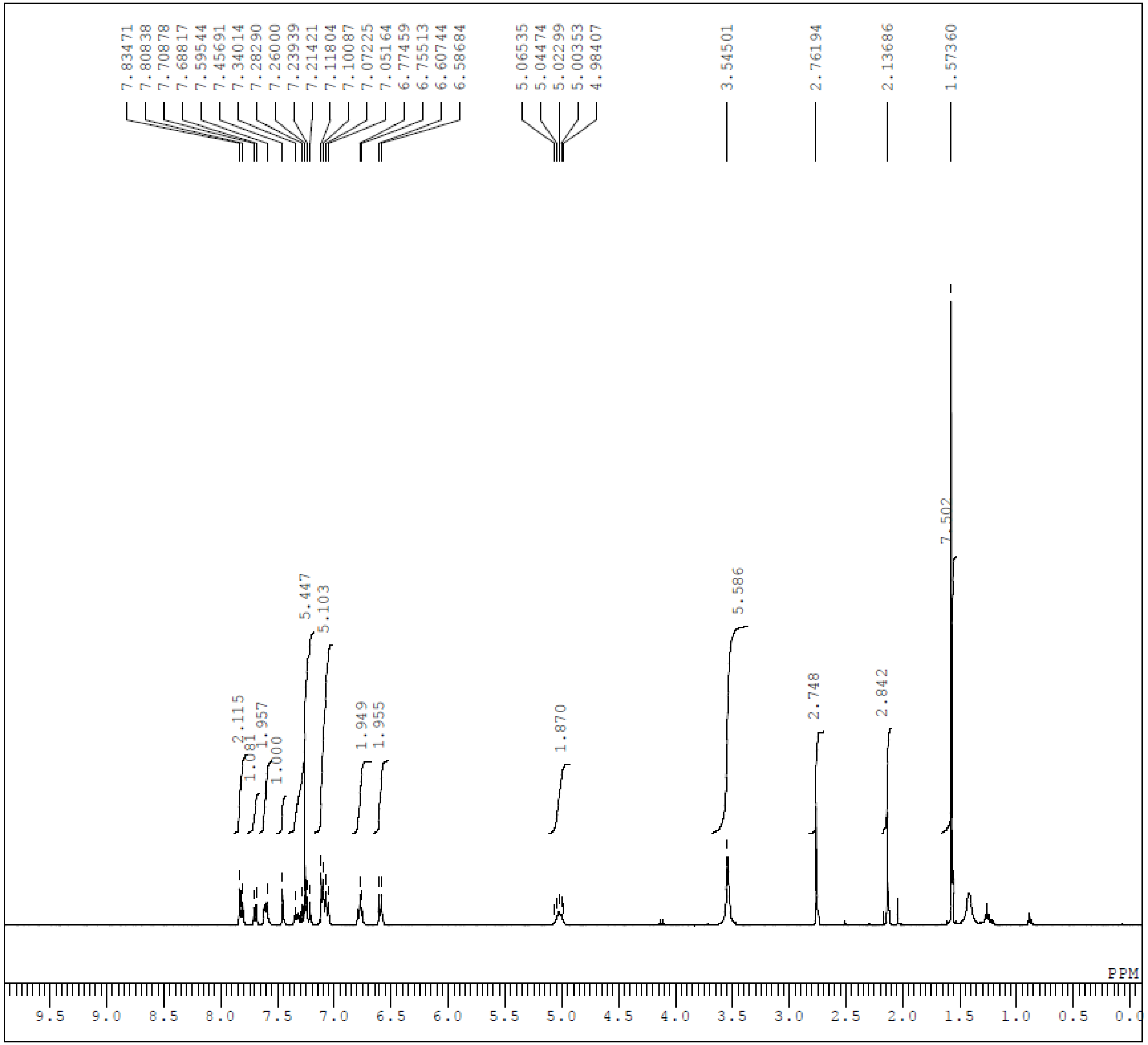
L12

C:¥Users¥alice¥Desktop¥Chen ligand¥L12_carbon.als

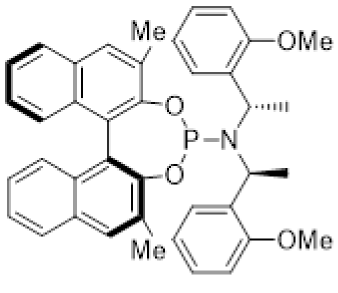


DFILE	L12_carbon.als
COMNT	
DATIM	2020-04-17 22:52:23
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	421
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

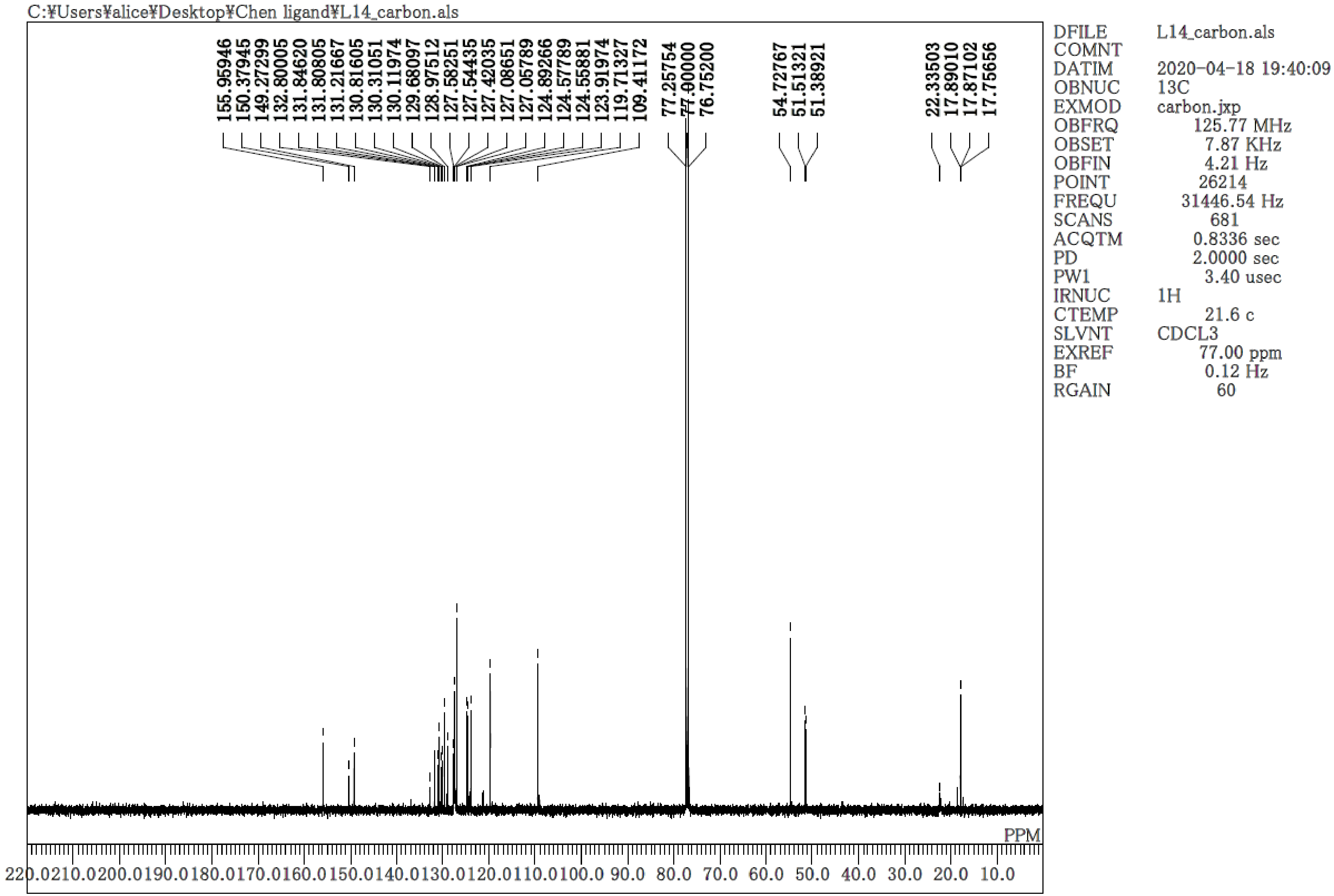

C:¥Users¥alice¥Desktop¥Chen ligand¥L12_P.als
143.12835
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L12_P.als
COMNT
DATIM 09-05-2020 04:21:25
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 85
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 56



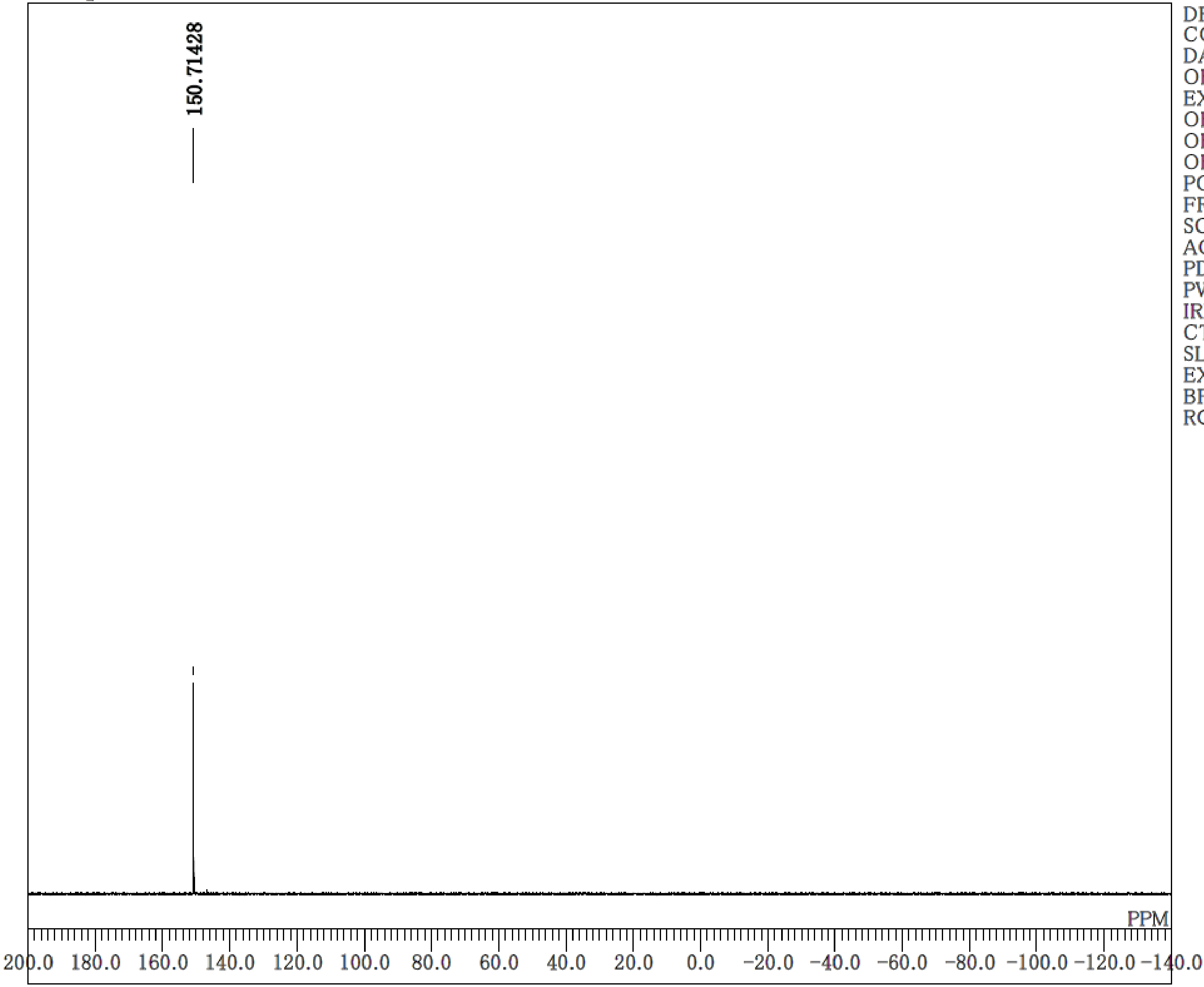
DFILE L14_proton.als
COMNT
DATIM 21-06-2020 10:41:49
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 46



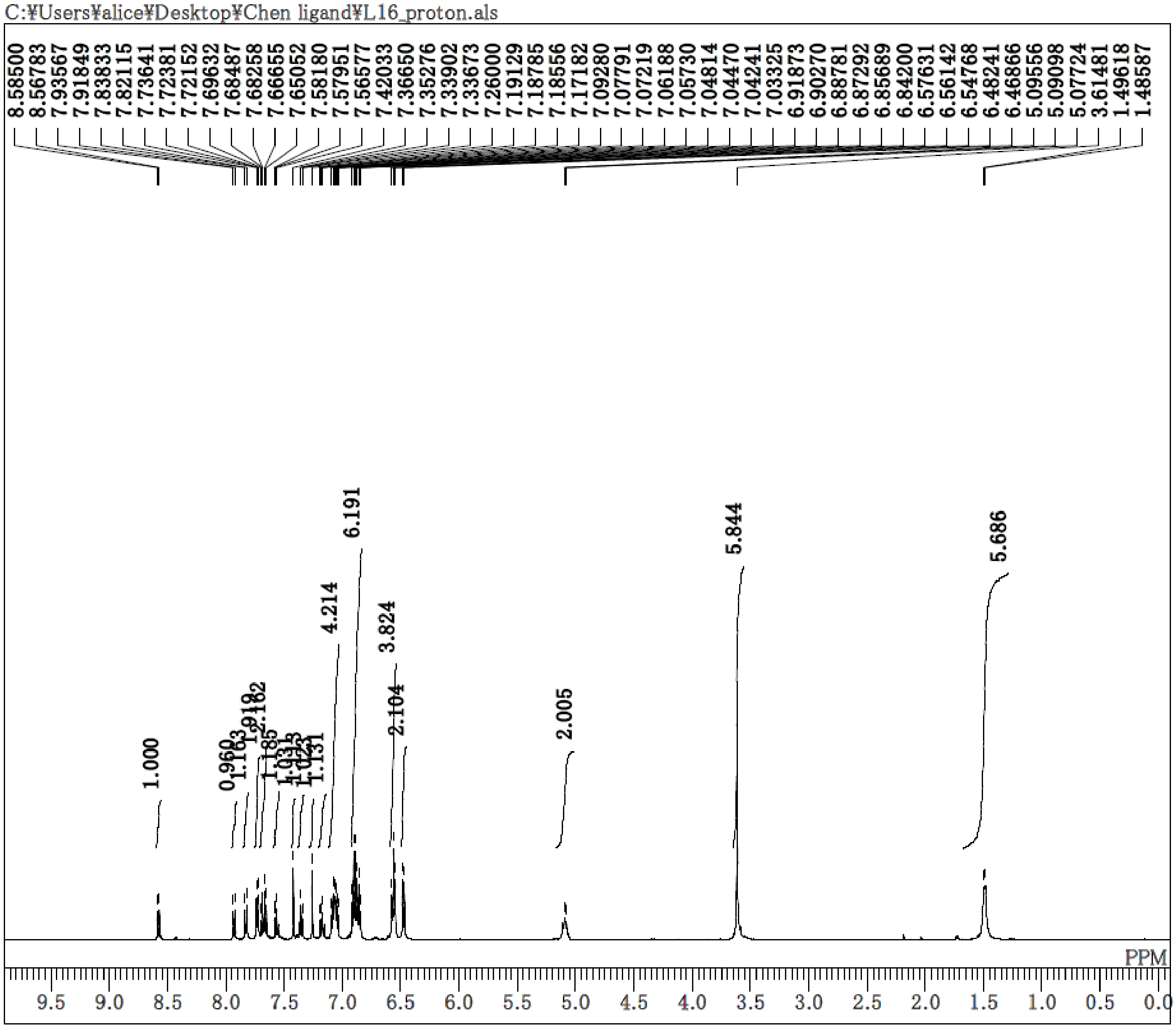
L14


C:¥Users¥alice¥Desktop¥Chen ligand¥L14_carbon.als
155.95946
150.37945
149.27299
132.80005
131.84620
131.80805
131.21667
130.81605
130.31051
130.11974
129.68097
128.97512
127.58251
127.54435
127.42035
127.08651
127.05789
124.89266
124.57789
124.55881
123.91974
119.71327
109.41172
77.25754
77.00000
76.75200
54.72767
51.51321
51.38921
22.33503
17.89010
17.87102
17.75656
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE L14_carbon.als
COMNT
DATIM 2020-04-18 19:40:09
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 681
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

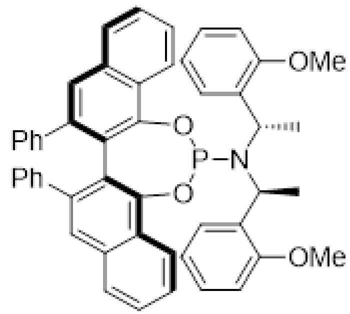
I:¥L14_P-1-1.als



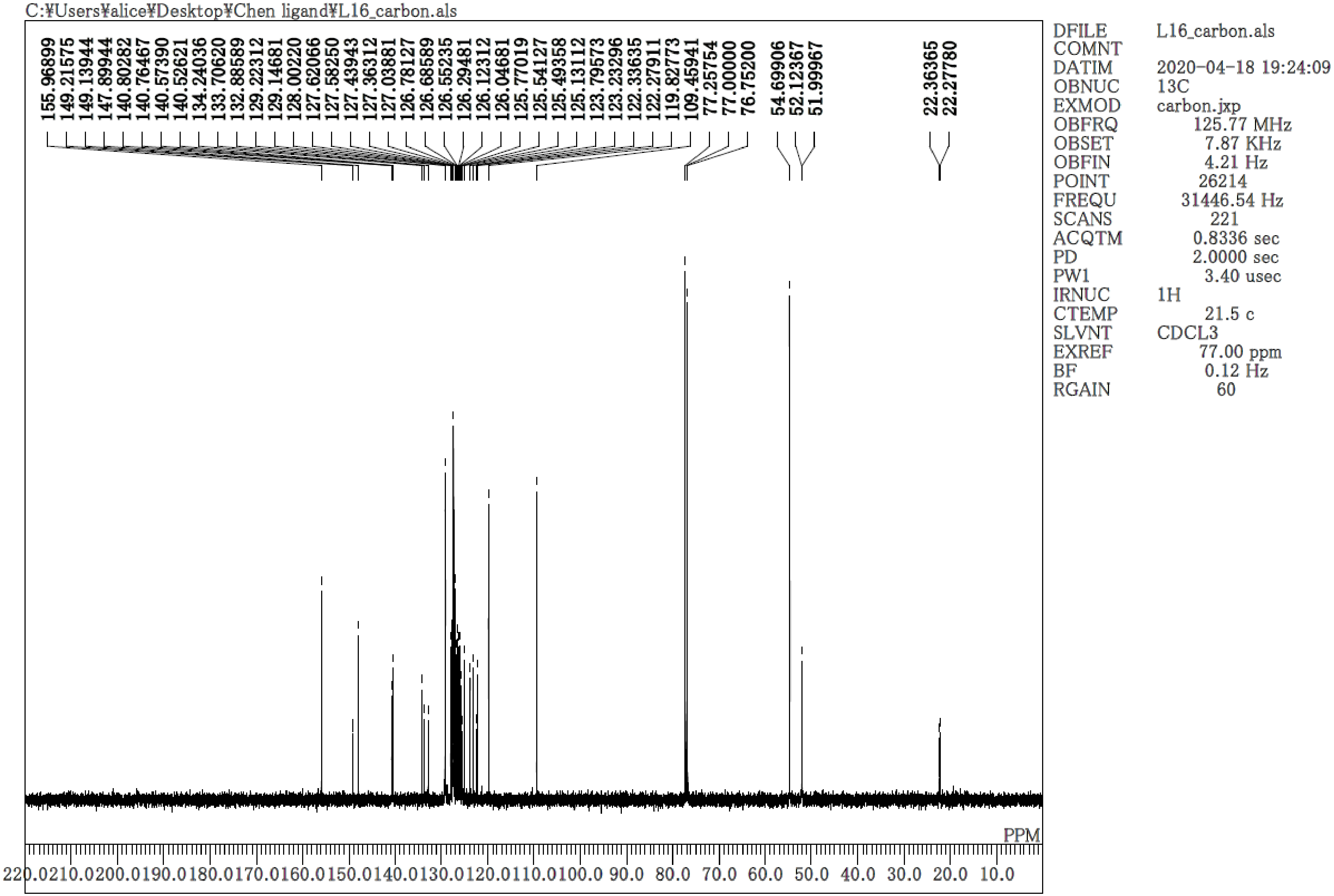
DFILE	L14_P-1-1.als
COMNT	
DATIM	22-06-2020 20:07:04
OBNUC	31P
EXMOD	carbon.jxp
OBFRQ	158.59 MHz
OBSET	7.99 KHz
OBFIN	9.23 Hz
POINT	26214
FREQU	64102.56 Hz
SCANS	102
ACQTM	0.4089 sec
PD	2.0000 sec
PW1	4.80 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	−6.00 ppm
BF	0.02 Hz
RGAIN	56



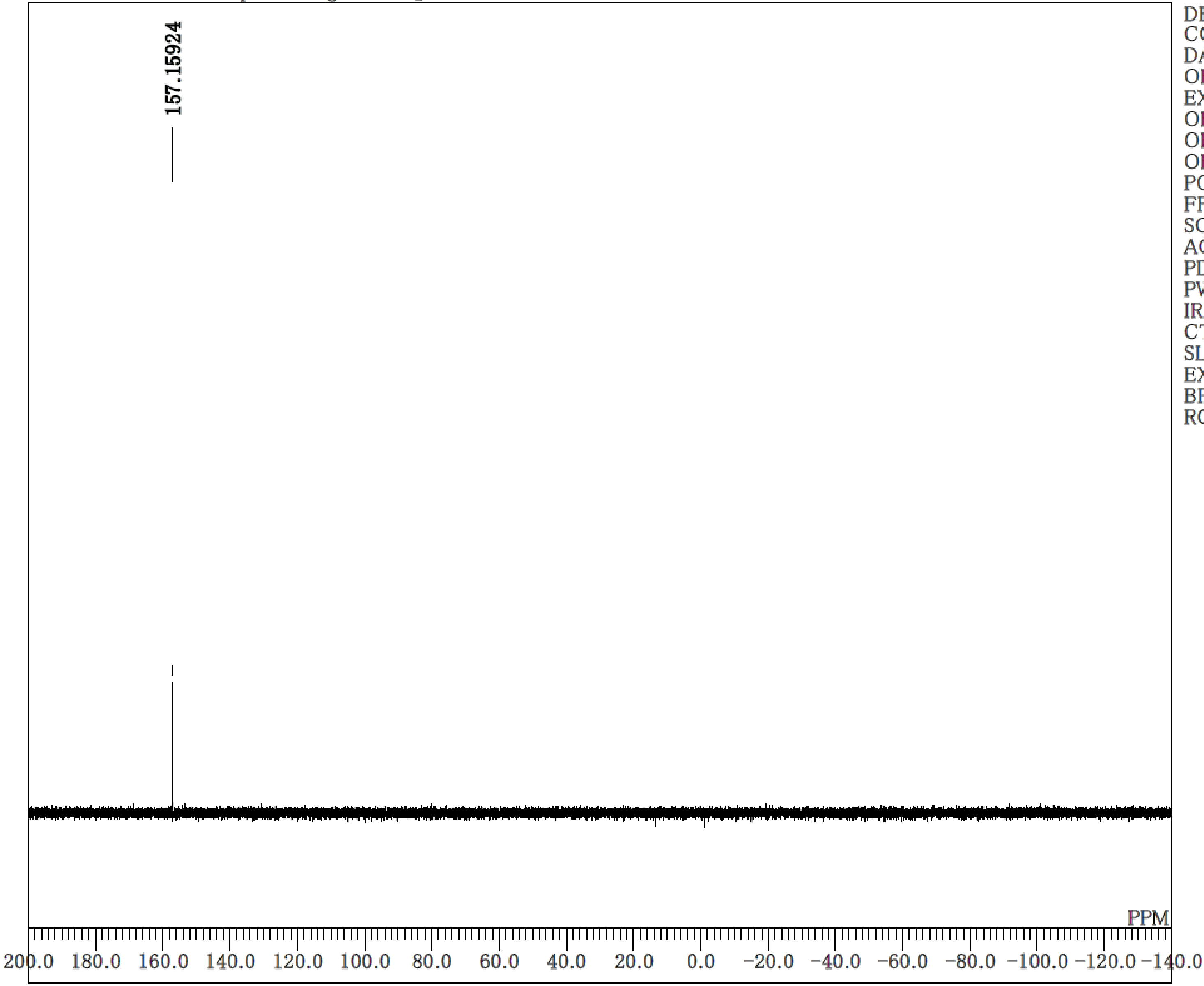
DFILE	L16_proton.als
COMNT	
DATIM	2020-04-18 19:35:43
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30



L16

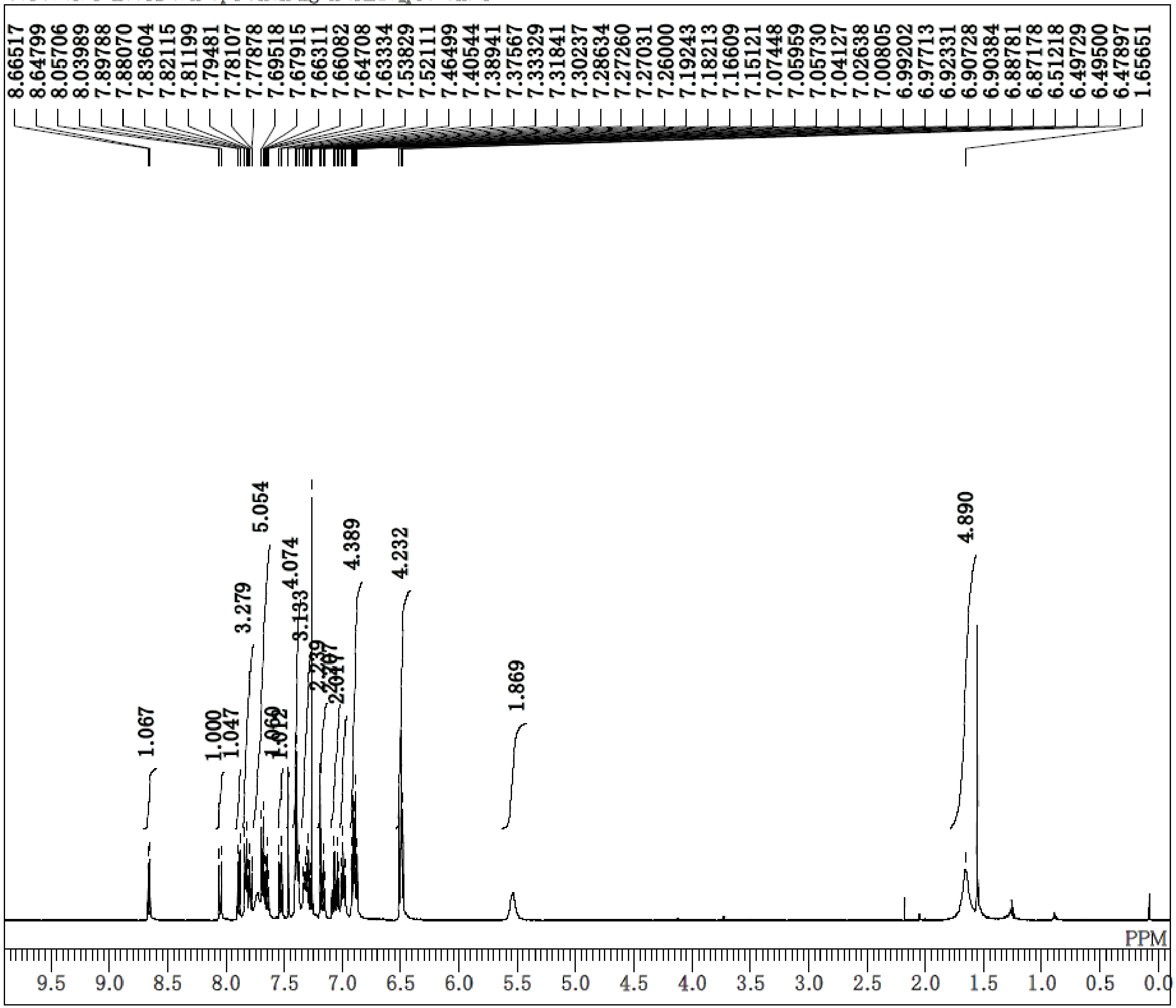

C:¥Users¥alice¥Desktop¥Chen ligand¥L16_carbon.als
155.96899
149.21575
149.13944
147.89944
140.80282
140.76467
140.57390
140.52621
134.24036
133.70620
132.88589
129.22312
129.14681
128.00220
127.62066
127.58250
127.43943
127.36312
127.03881
126.78127
126.68589
126.55235
126.29481
126.12312
126.04681
125.77019
125.54127
125.49358
125.13112
123.79573
123.23296
122.33635
122.27911
119.82773
109.45941
77.25754
77.00000
76.75200
54.69906
52.12367
51.99967
22.36365
22.27780
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE L16_carbon.als
COMNT
DATIM 2020-04-18 19:24:09
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 221
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:¥Users¥alice¥Desktop¥Chen ligand¥L16_P.als

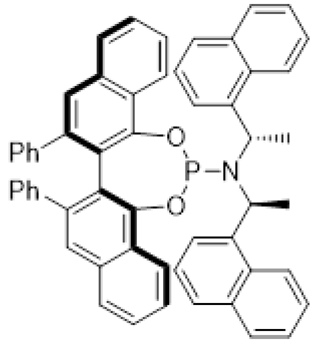


DFILE	L16_P.als
COMNT	
DATIM	09-05-2020 17:53:20
OBNUC	31P
EXMOD	carbon.jxp
OBFRQ	158.59 MHz
OBSET	7.99 KHz
OBFIN	9.23 Hz
POINT	26214
FREQU	64102.56 Hz
SCANS	6
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	4.80 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	−6.00 ppm
BF	0.12 Hz
RGAIN	56

C:¥Users¥alice¥Desktop¥Chen ligand¥L17_proton.als

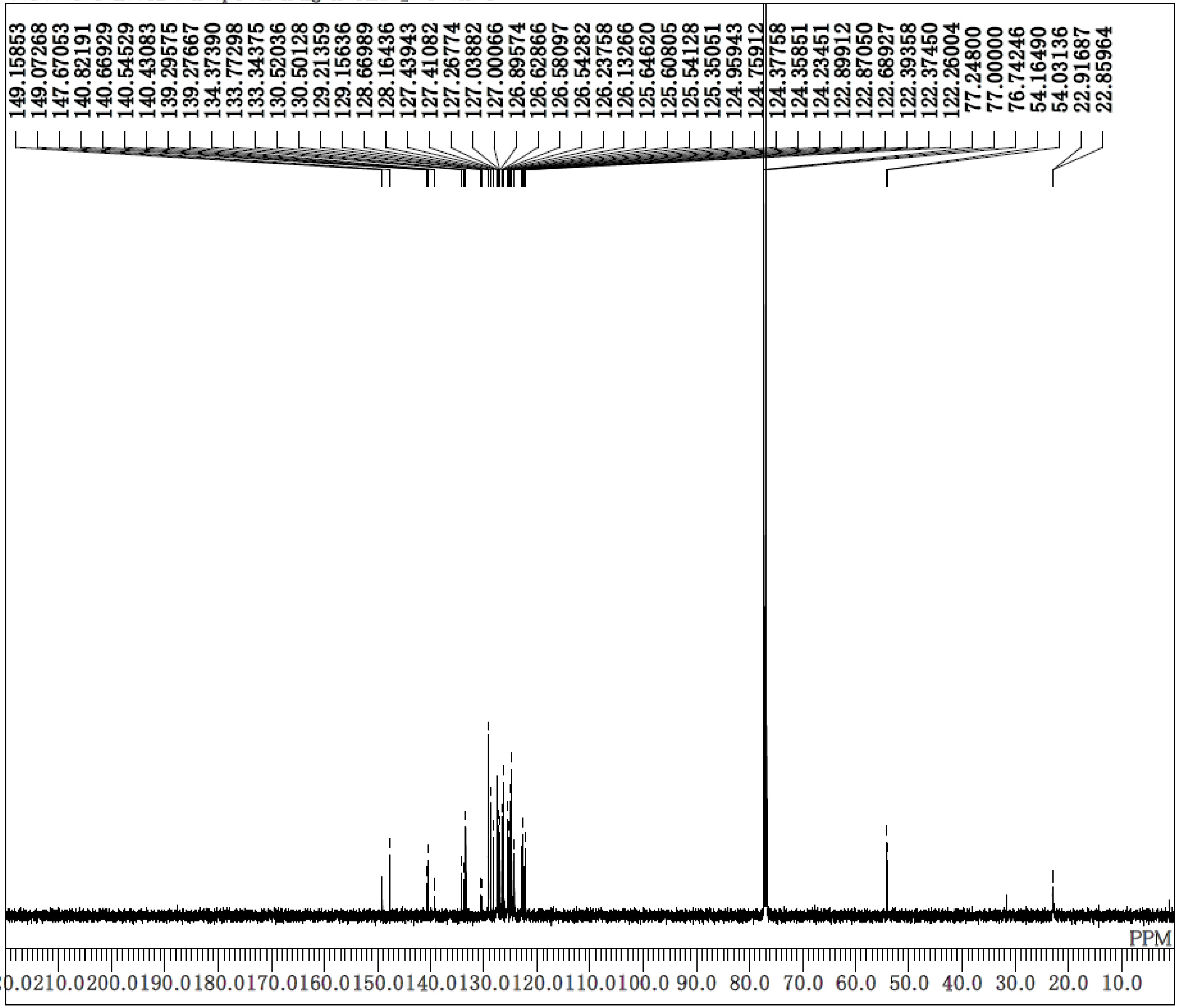


DFILE	L17_proton.als
COMNT	
DATIM	2020-04-24 17:15:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

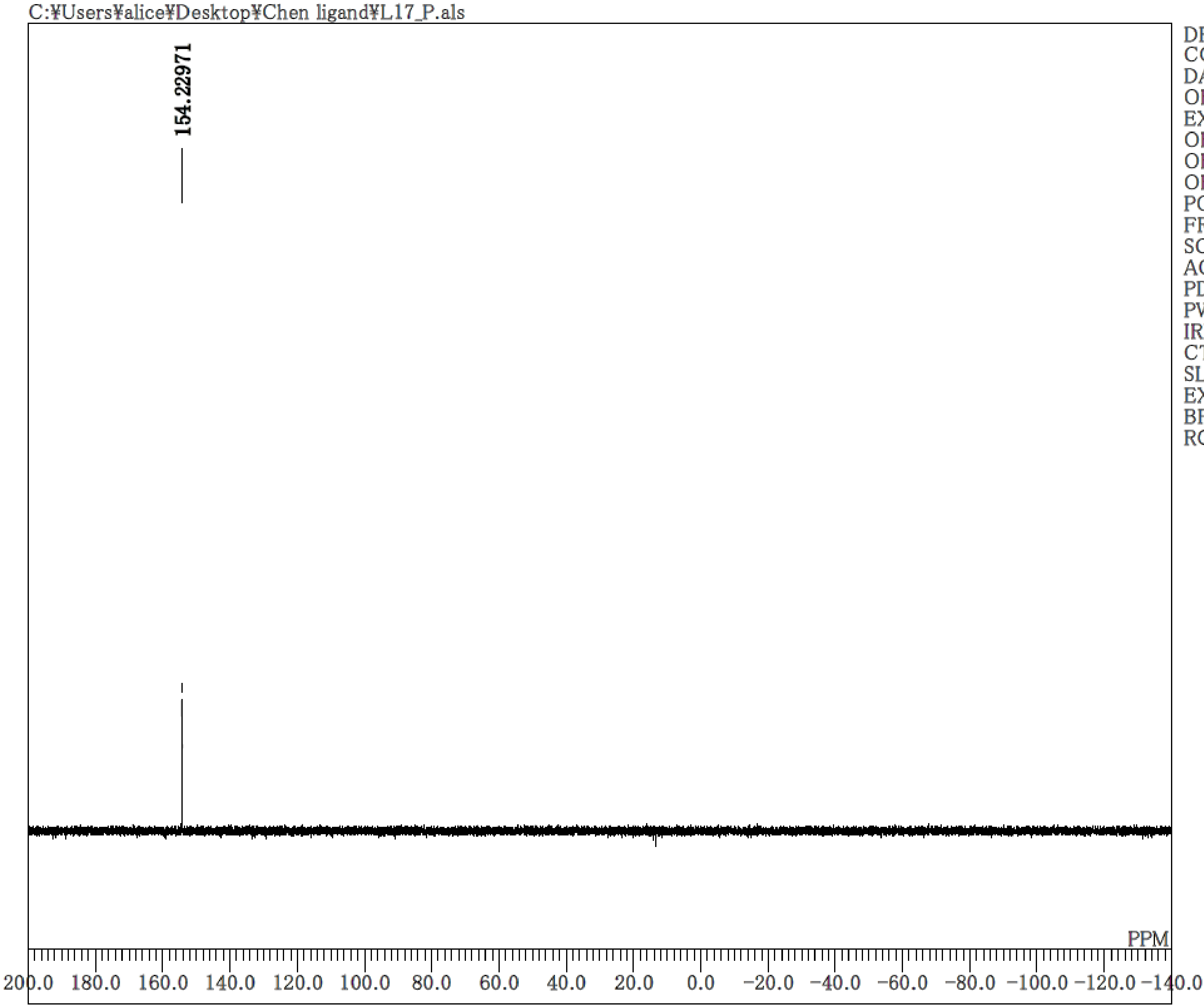


L17

C:¥Users¥alice¥Desktop¥Chen ligand¥L17_carbon.als

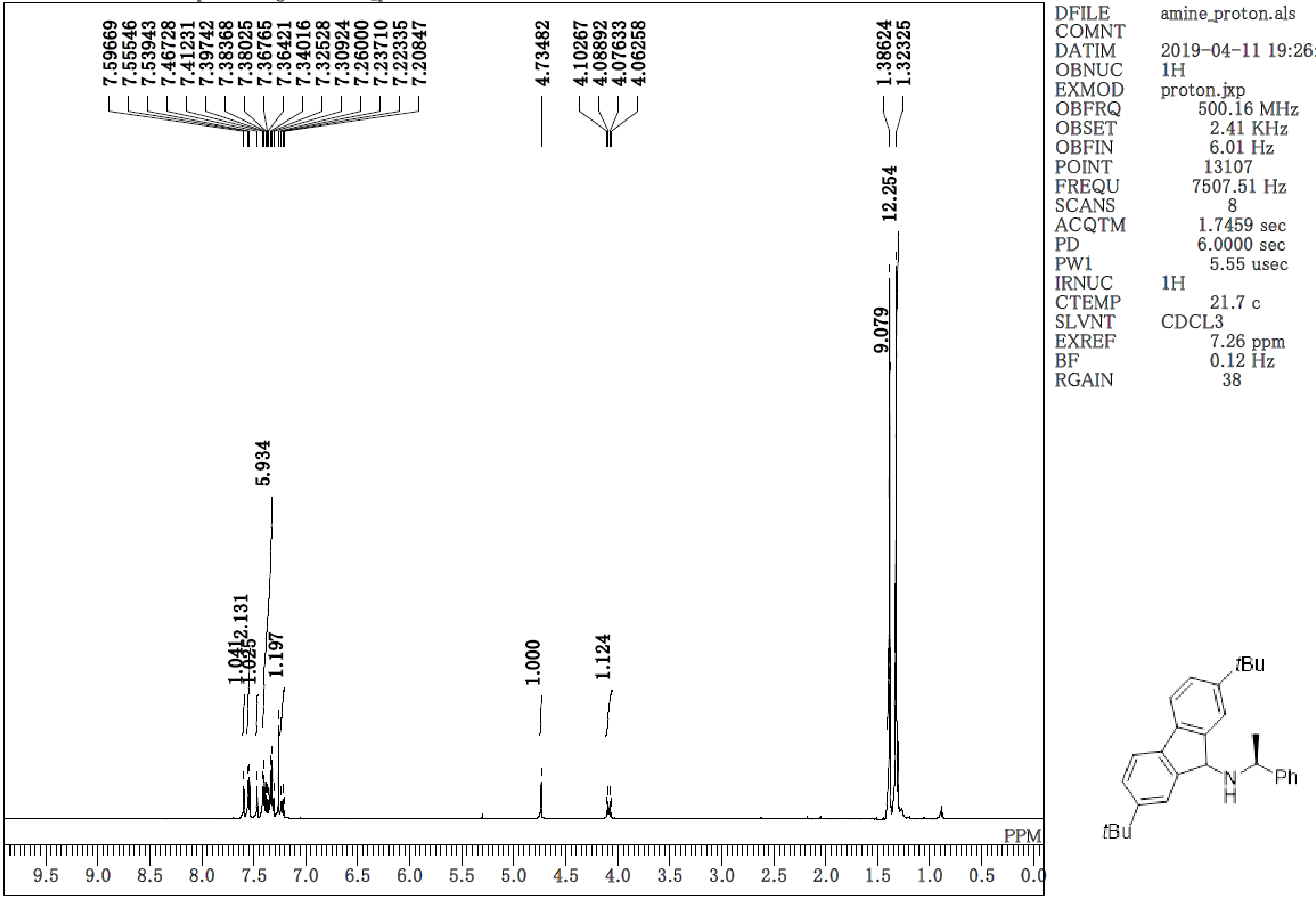


DFILE	L17_carbon.als
COMNT	
DATIM	2020-04-24 20:53:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	4000
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



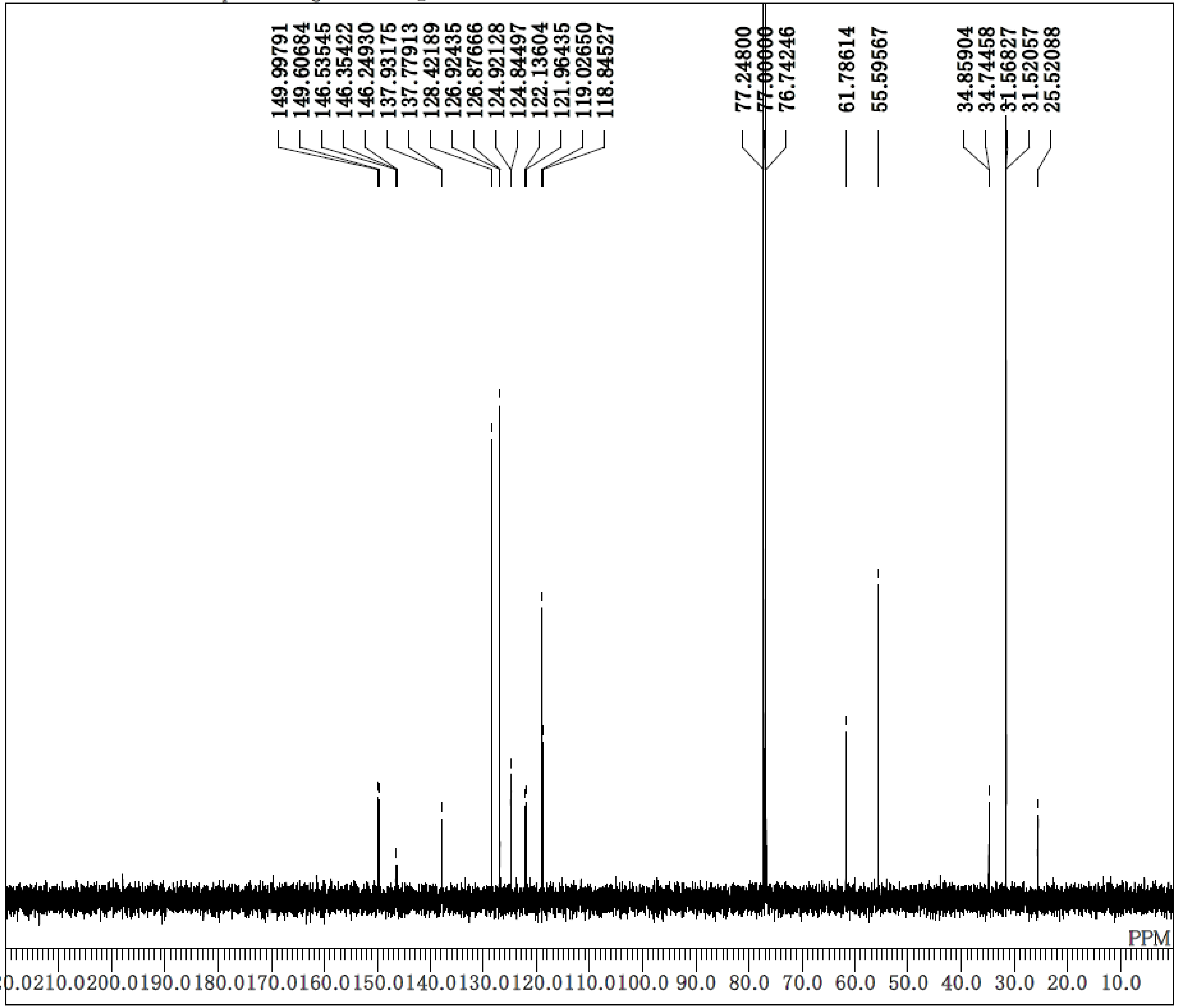
DFILE	L17_P.als
COMNT	
DATIM	09-05-2020 17:12:29
OBNUC	31P
EXMOD	carbon.jxp
OBFRQ	158.59 MHz
OBSET	7.99 KHz
OBFIN	9.23 Hz
POINT	26214
FREQU	64102.56 Hz
SCANS	60
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	4.80 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	−6.00 ppm
BF	0.12 Hz
RGAIN	56

C:¥Users¥alice¥Desktop¥Chen ligand¥amine_proton.als



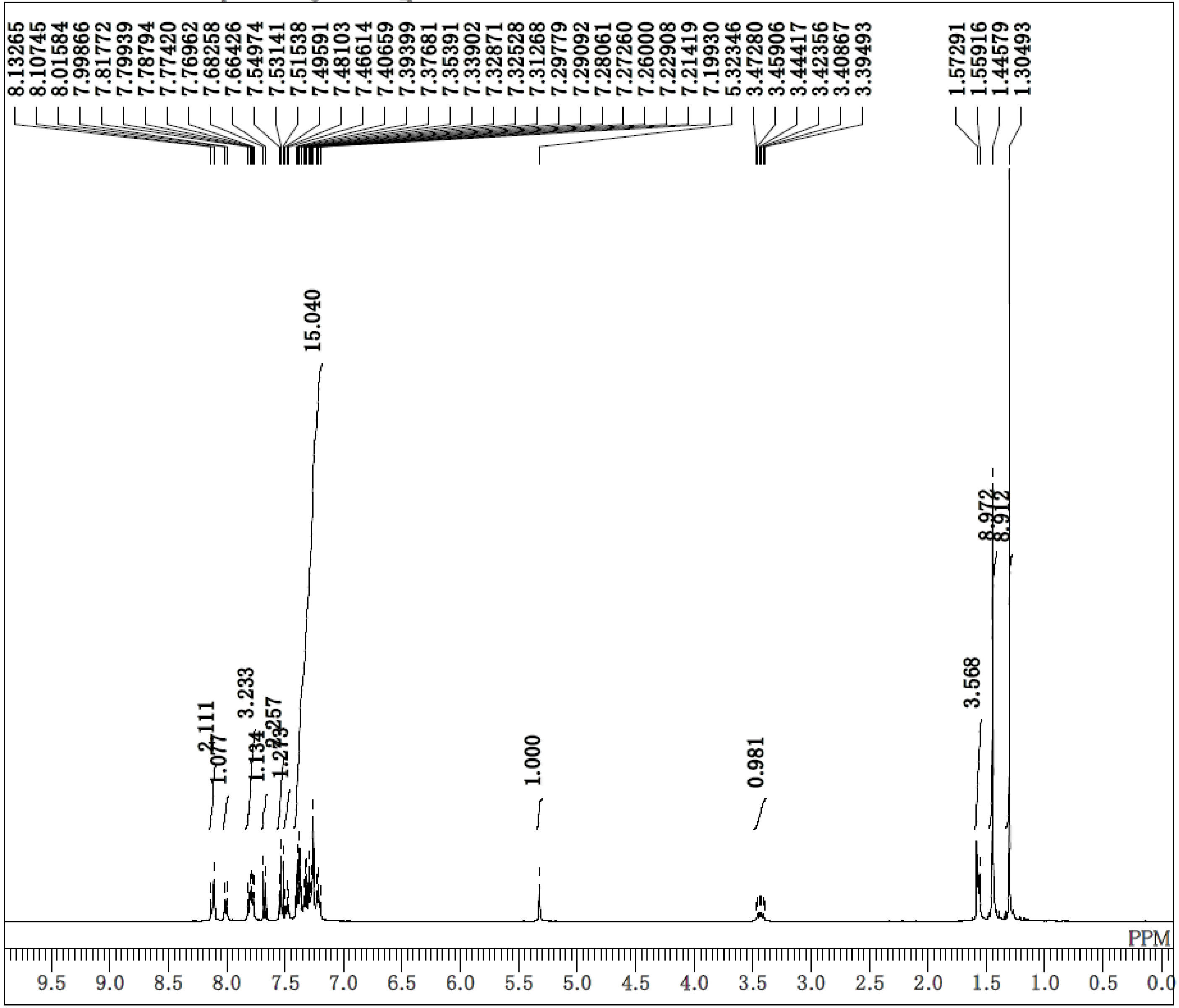
DFILE	amine_proton.als
COMNT	
DATIM	2019-04-11 19:26:22
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38

C:¥Users¥alice¥Desktop¥Chen ligand¥amine_carbon.als

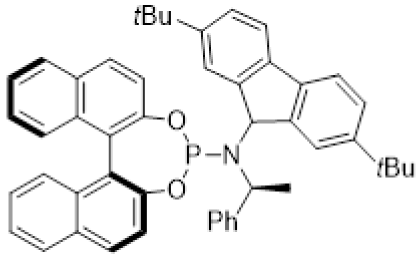


DFILE	amine_carbon.als
COMNT	
DATIM	2019-07-18 10:41:38
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	121
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

C:¥Users¥alice¥Desktop¥Chen ligand¥L18_proton.als

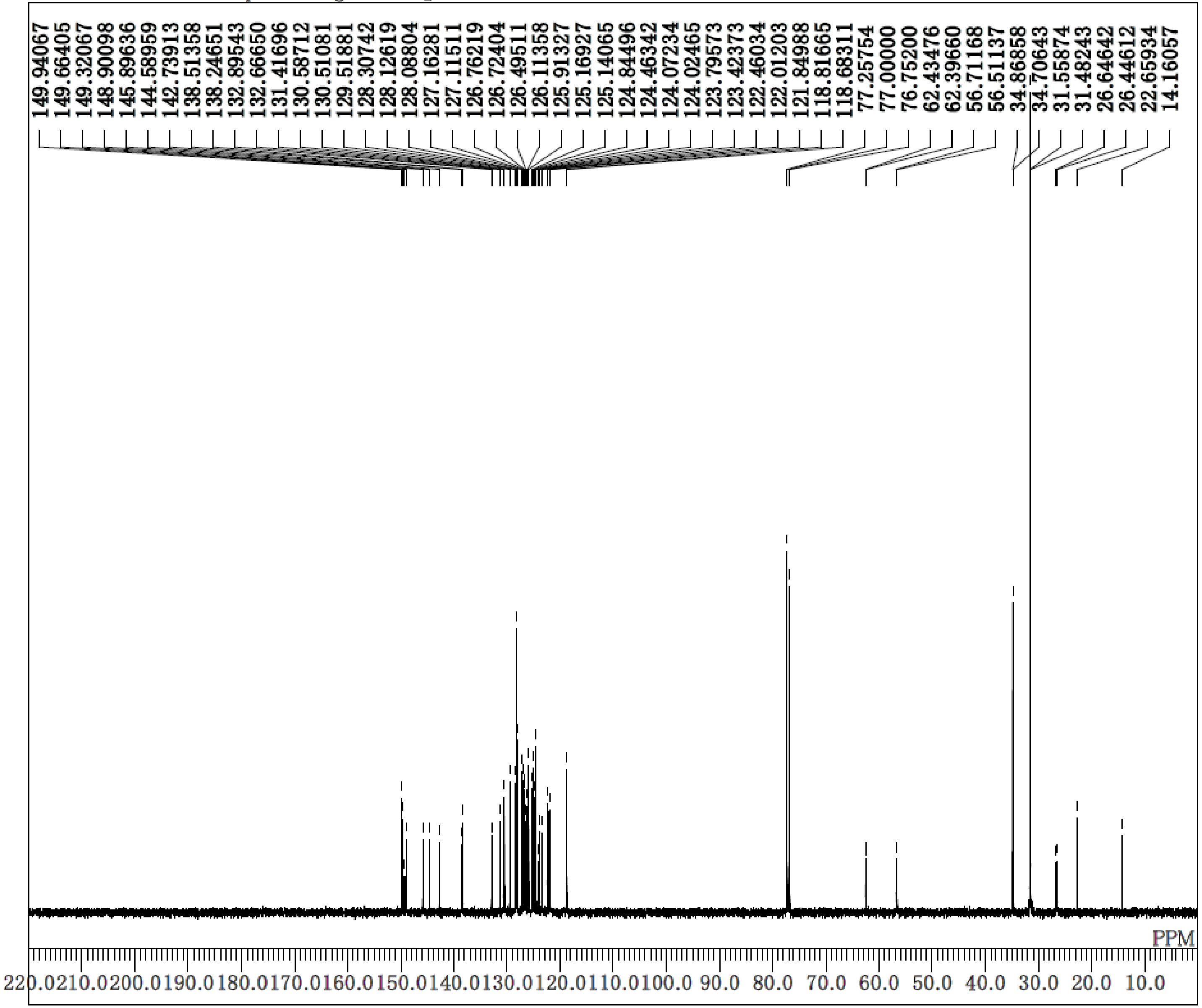


DFILE	L18_proton.als
COMNT	
DATIM	2020-04-19 20:52:29
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36

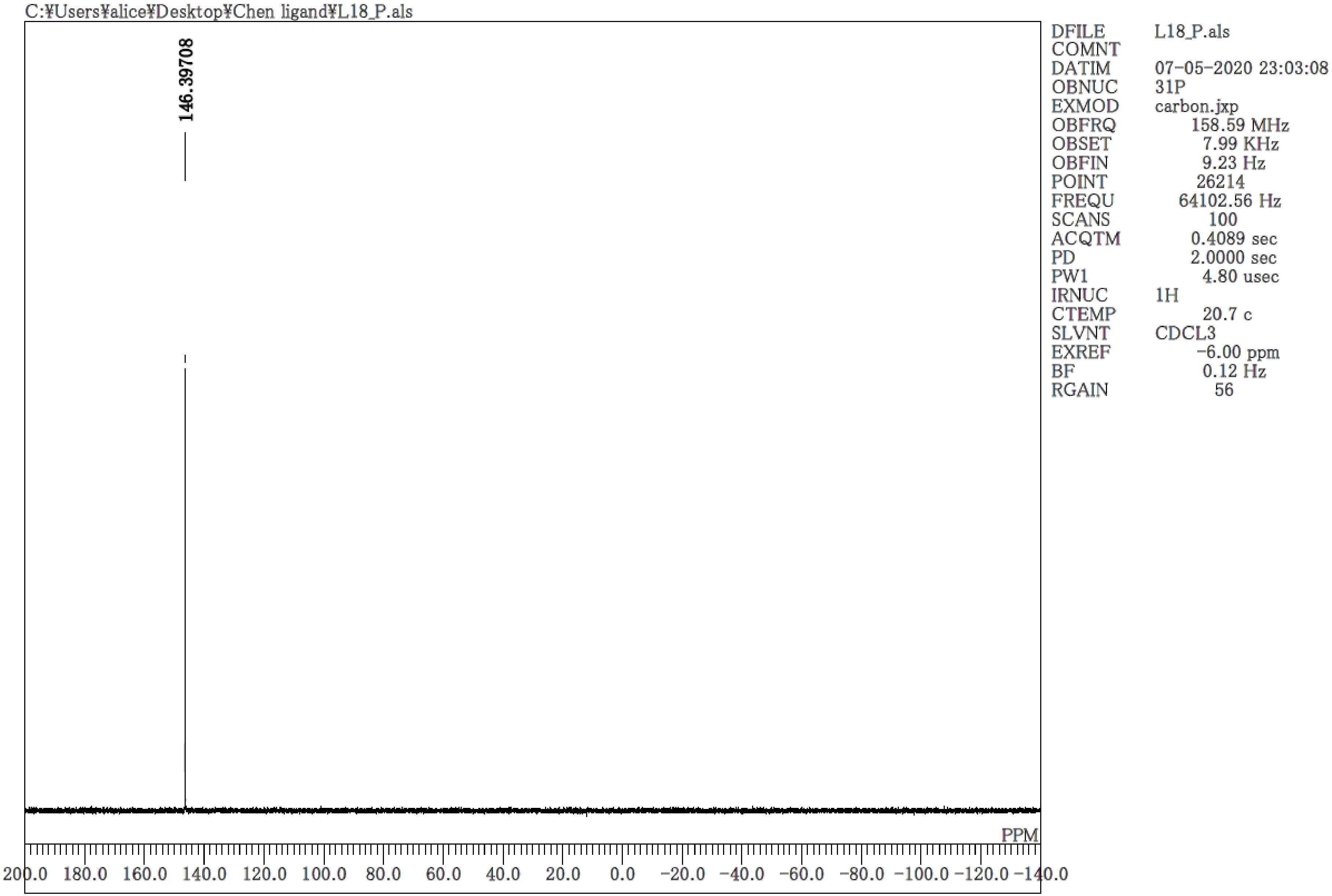


L18

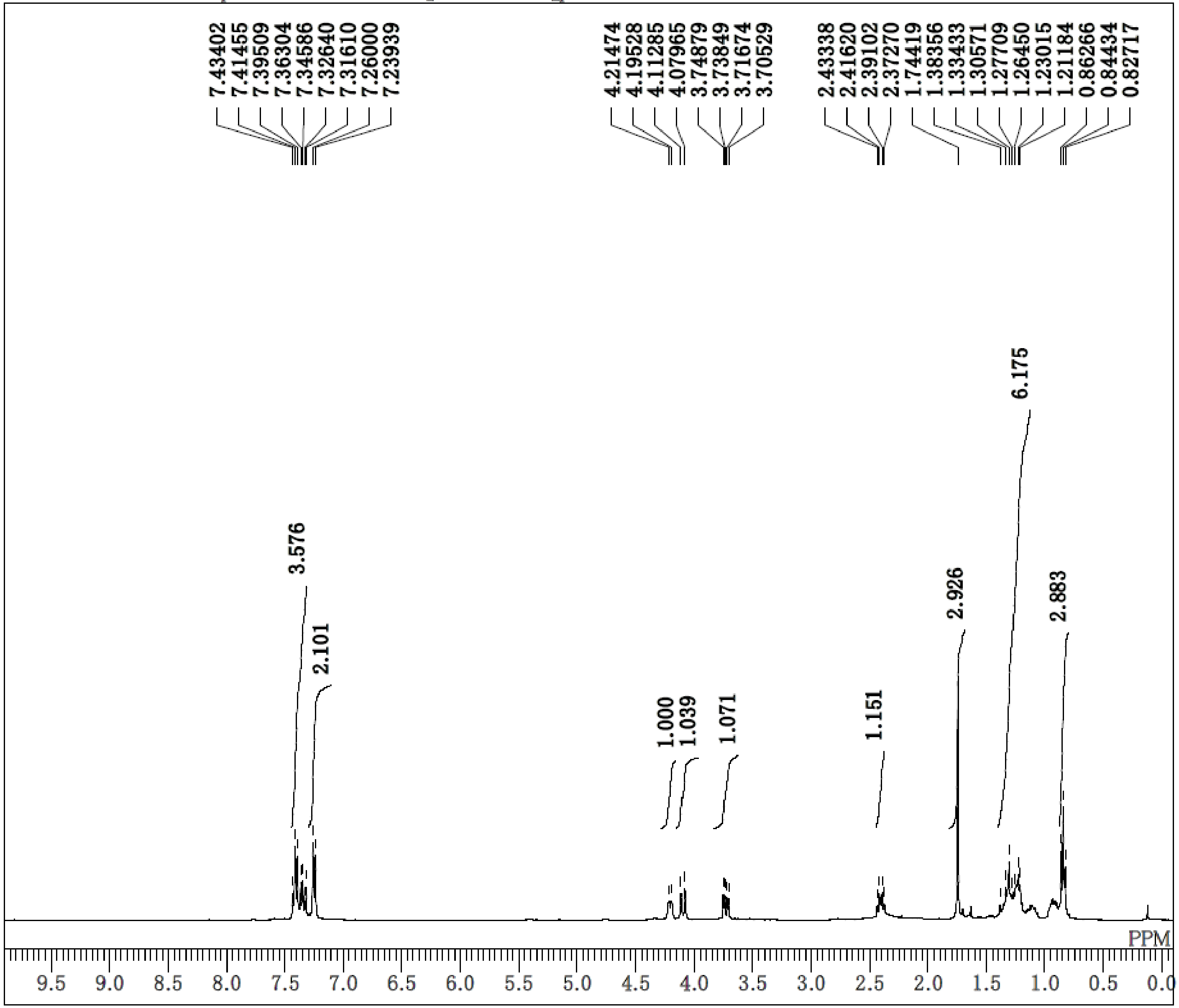
C:¥Users¥alice¥Desktop¥Chen ligand¥L18_carbon.als



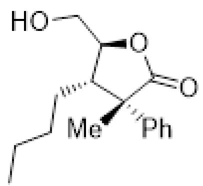
DFILE	L18_carbon.als
COMNT	
DATIM	2020-04-18 21:35:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	221
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60


C:¥Users¥alice¥Desktop¥Chen ligand¥L18_P.als
146.39708
PPM
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0.0 −20.0 −40.0 −60.0 −80.0 −100.0 −120.0 −140.0
DFILE L18_P.als
COMNT
DATIM 07-05-2020 23:03:08
OBNUC 31P
EXMOD carbon.jxp
OBFRQ 158.59 MHz
OBSET 7.99 KHz
OBFIN 9.23 Hz
POINT 26214
FREQU 64102.56 Hz
SCANS 100
ACQTM 0.4089 sec
PD 2.0000 sec
PW1 4.80 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF −6.00 ppm
BF 0.12 Hz
RGAIN 56

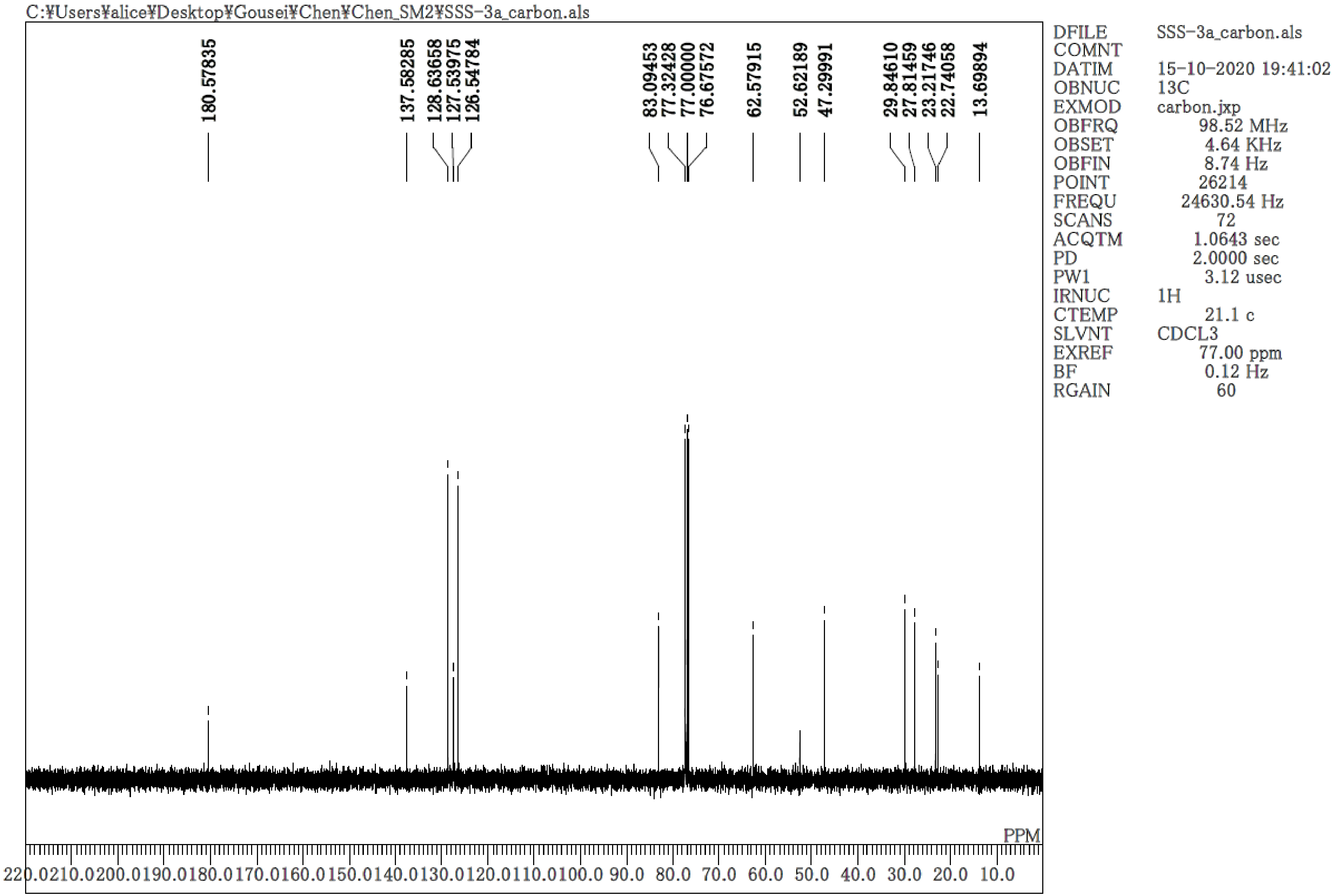
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥SSS-3a_proton.als



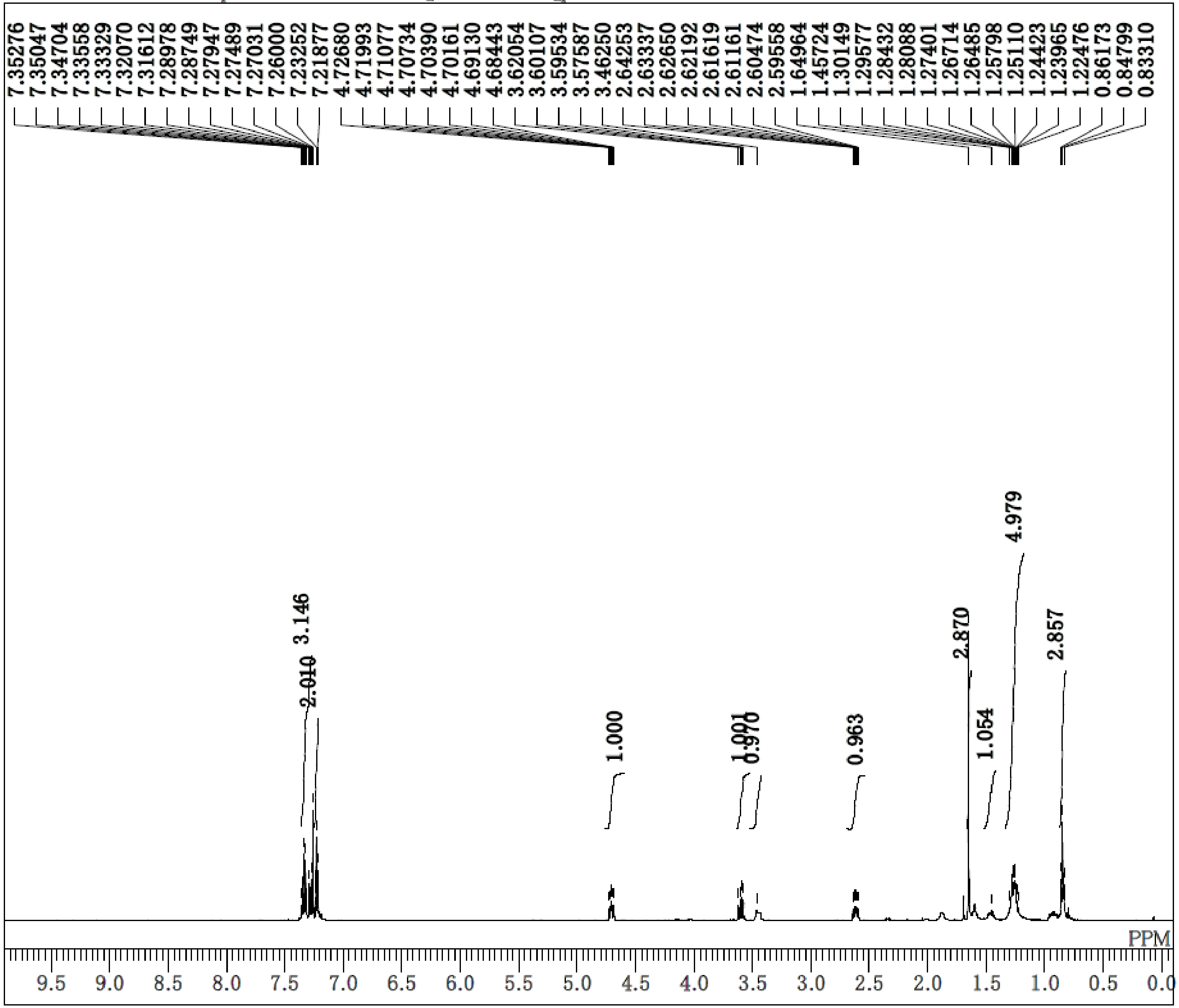
DFILE	SSS-3a_proton.als
COMNT	
DATIM	15-10-2020 19:38:13
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30



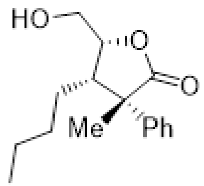
(3*S*,4*S*,5*S*)-**3a**


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥SSS-3a_carbon.als
180.57835
137.58285
128.63658
127.53975
126.54784
83.09453
77.32428
77.00000
76.67572
62.57915
52.62189
47.29991
29.84610
27.81459
23.21746
22.74058
13.69894
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE SSS-3a_carbon.als
COMNT
DATIM 15-10-2020 19:41:02
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 72
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

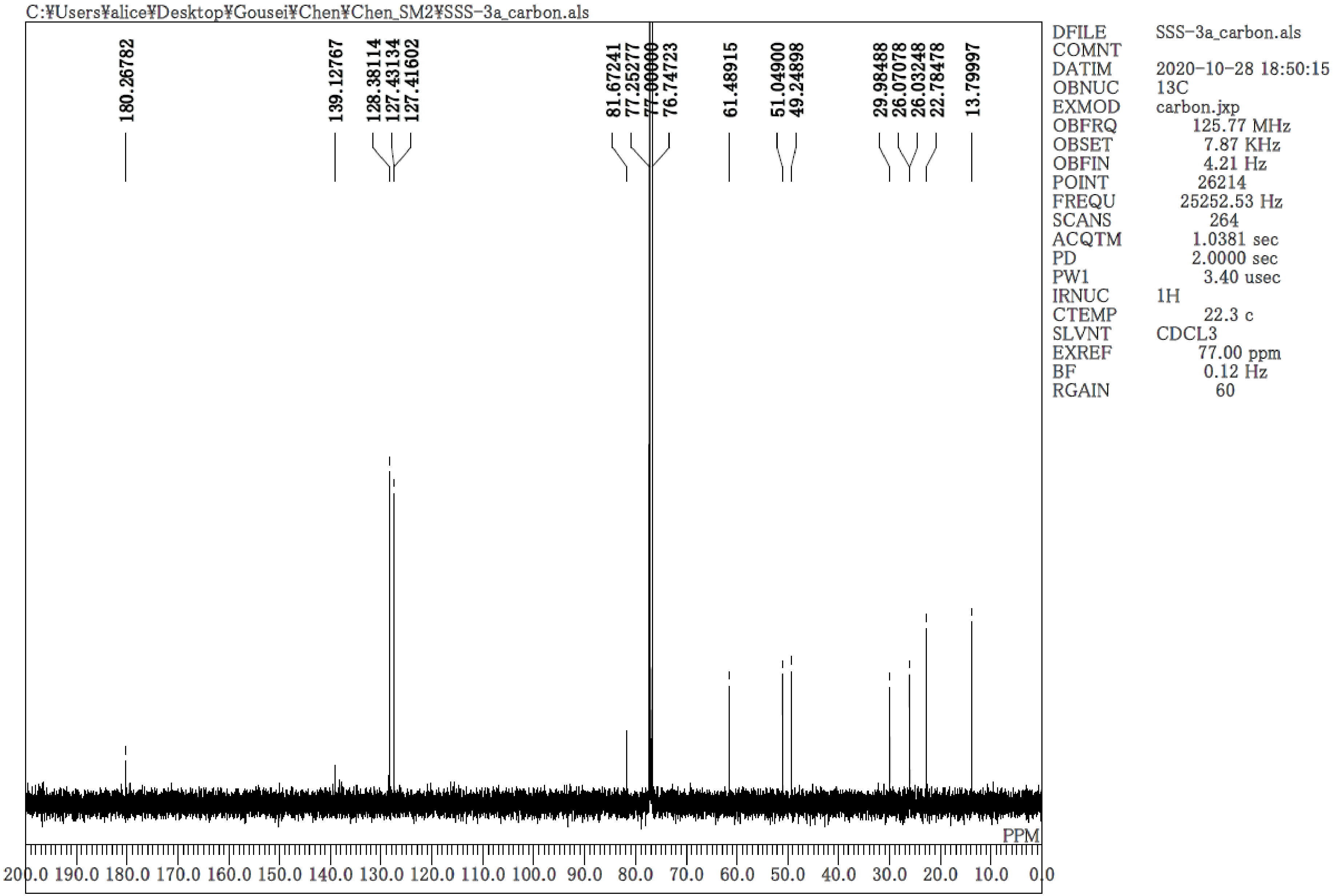
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥SSR-3a_proton.als



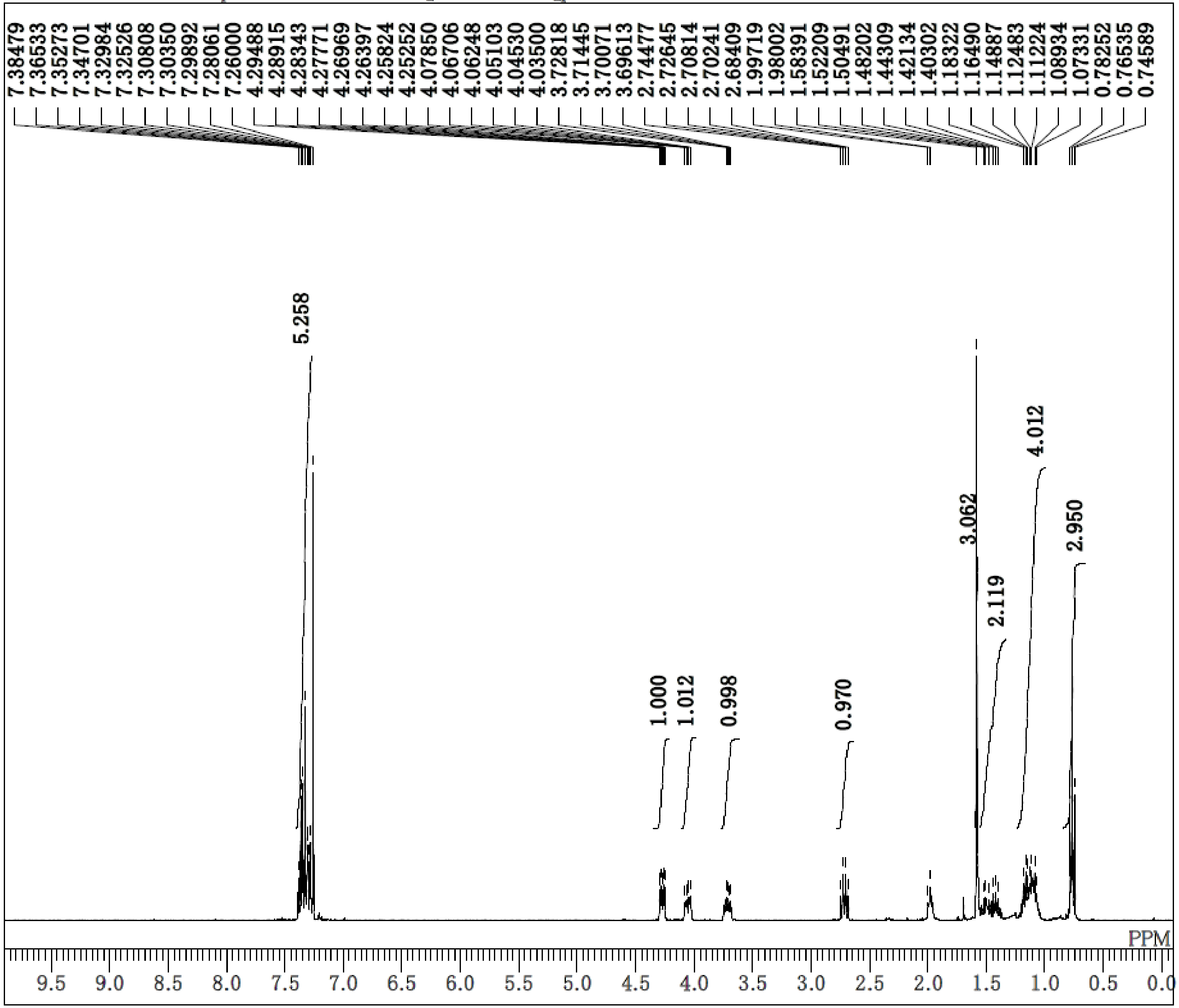
DFILE SSR-3a_proton.als
COMNT
DATIM 2020-10-28 18:40:32
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40



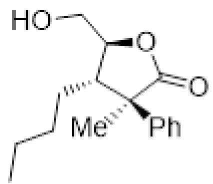
(3*S*,4*S*,5*R*)-3a


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥SSS-3a_carbon.als
180.26782
139.12767
128.38114
127.43134
127.41602
81.67241
77.25277
77.00000
76.74723
61.48915
51.04900
49.24898
29.98488
26.07078
26.03248
22.78478
13.79997
PPM
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
DFILE SSS-3a_carbon.als
COMNT
DATIM 2020-10-28 18:50:15
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 264
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

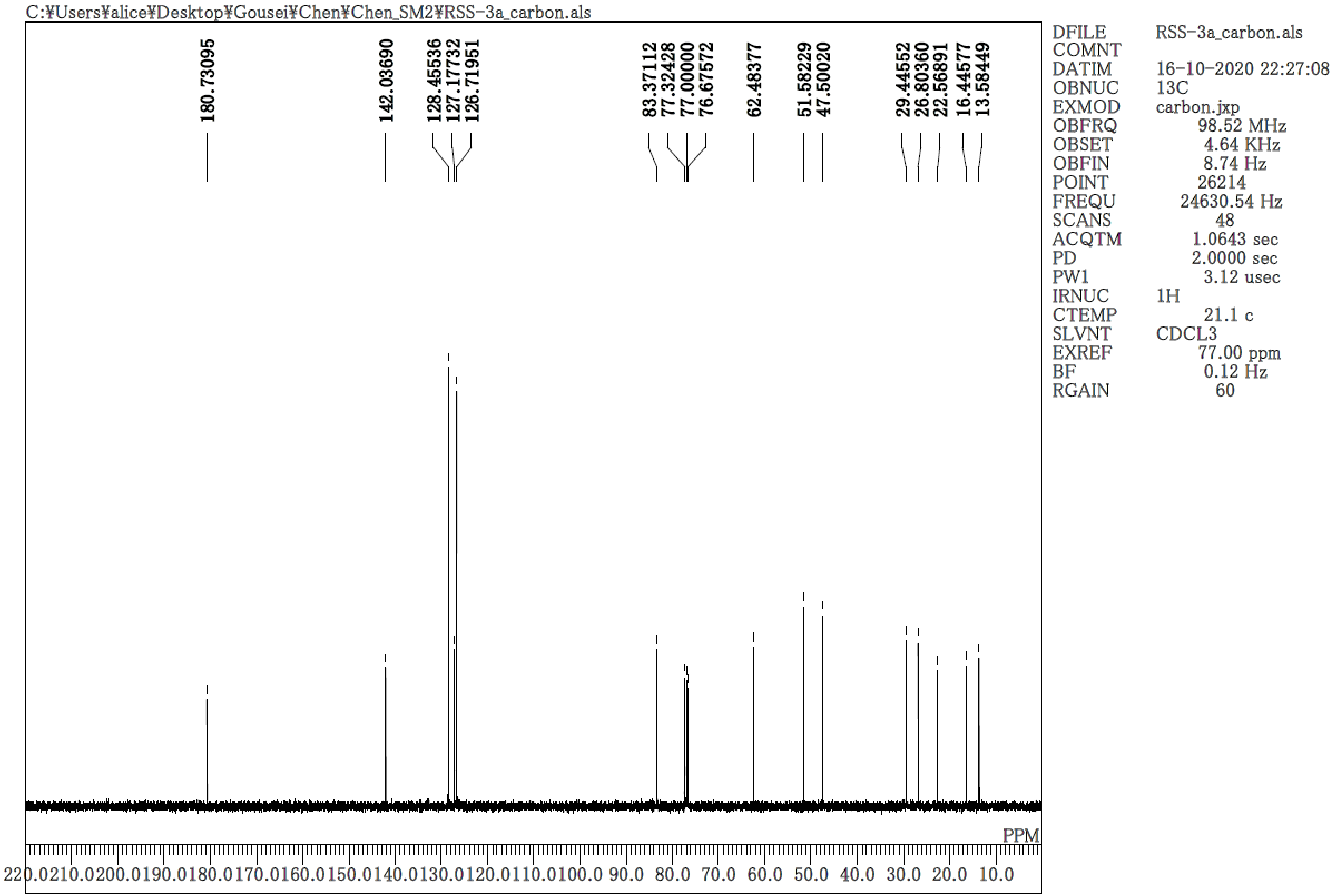
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥RSS-3a_proton.als



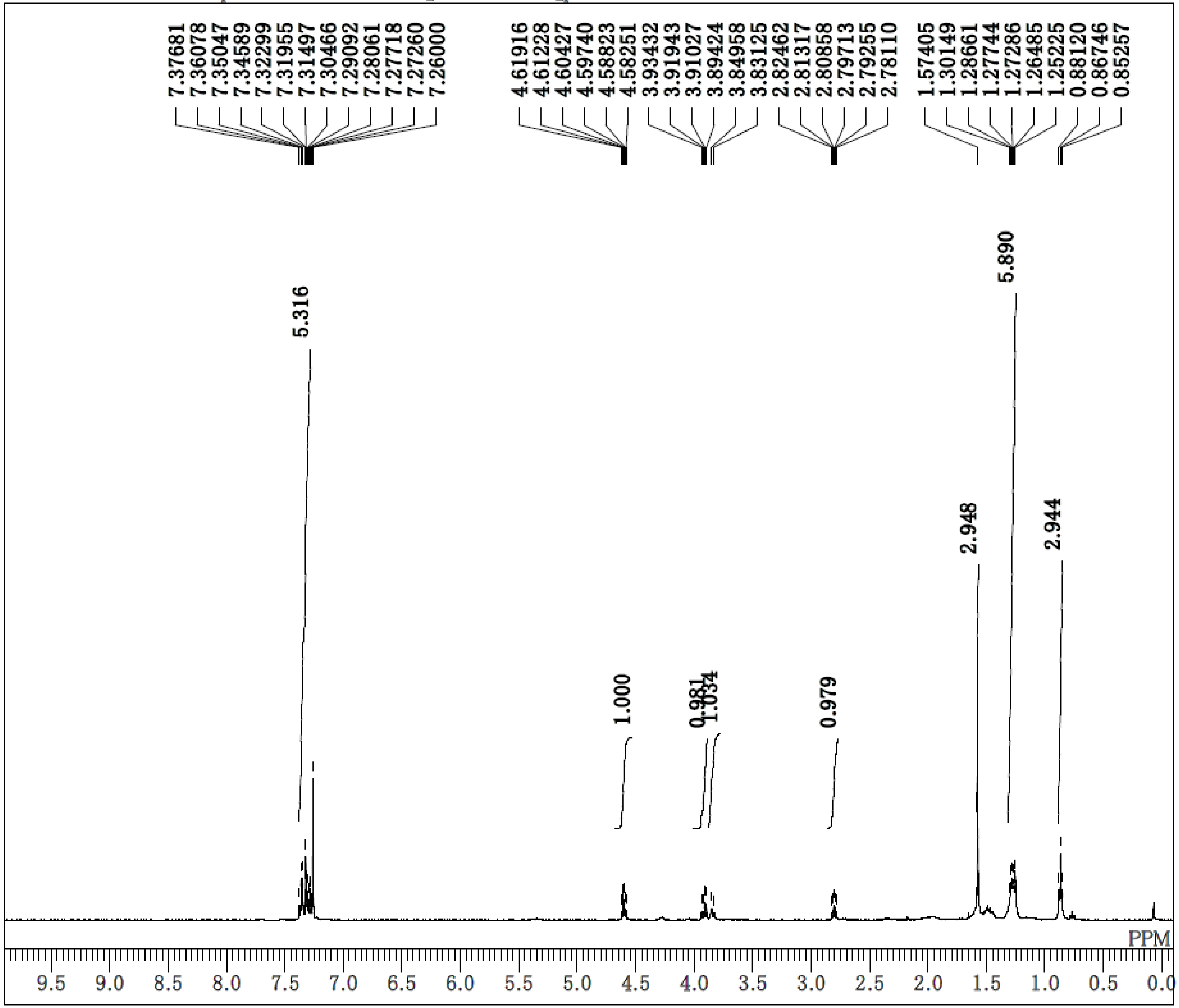
DFILE	RSS-3a_proton.als
COMNT	
DATIM	16-10-2020 22:19:34
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	46



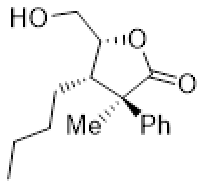
(3*R*,4*S*,5*S*)-3a


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥RSS-3a_carbon.als
180.73095
142.03690
128.45536
127.17732
126.71951
83.37112
77.32428
77.00000
76.67572
62.48377
51.58229
47.50020
29.44552
26.80360
22.56891
16.44577
13.58449
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
DFILE RSS-3a_carbon.als
COMNT
DATIM 16-10-2020 22:27:08
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 48
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

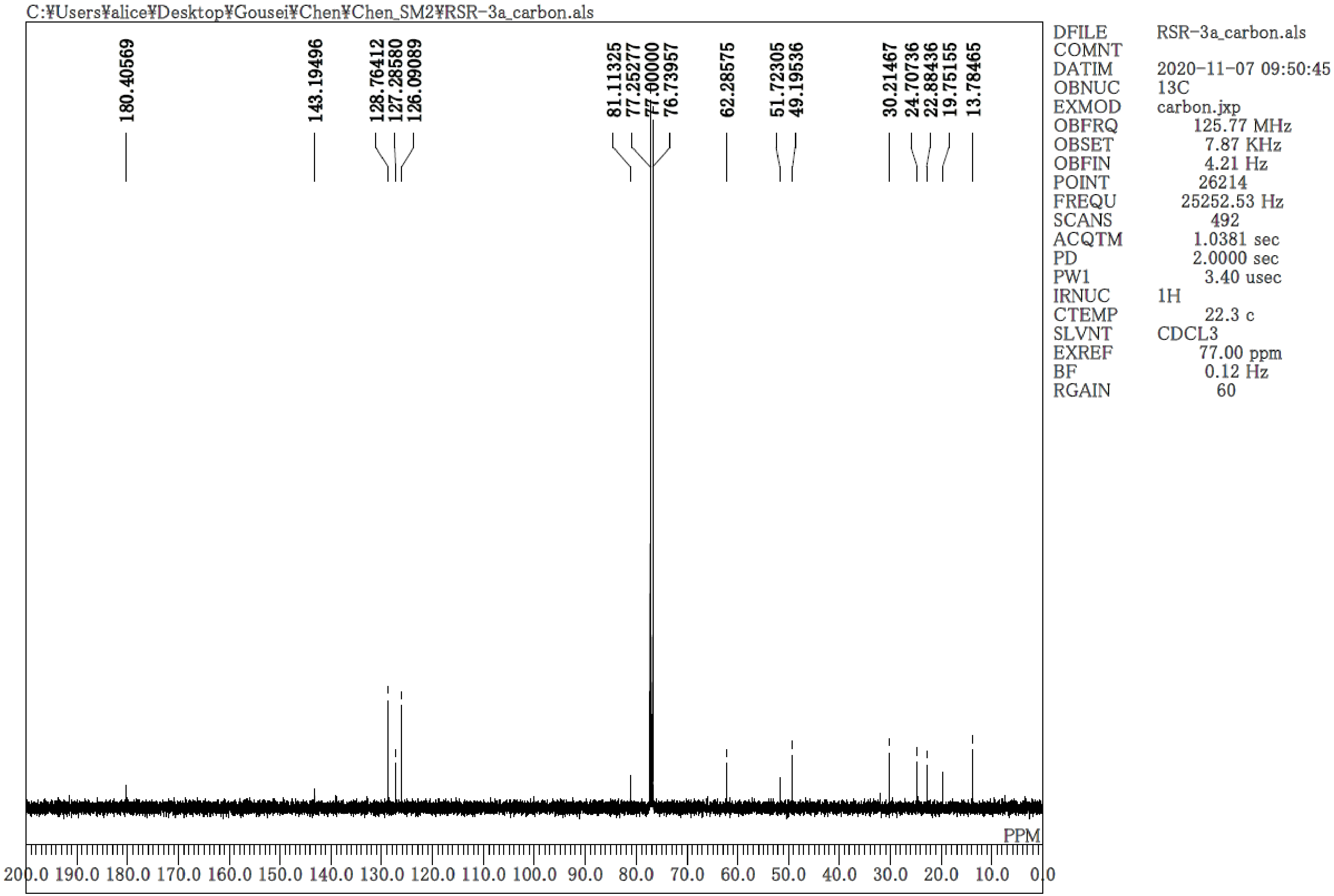
C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥RSR-3a_proton.als

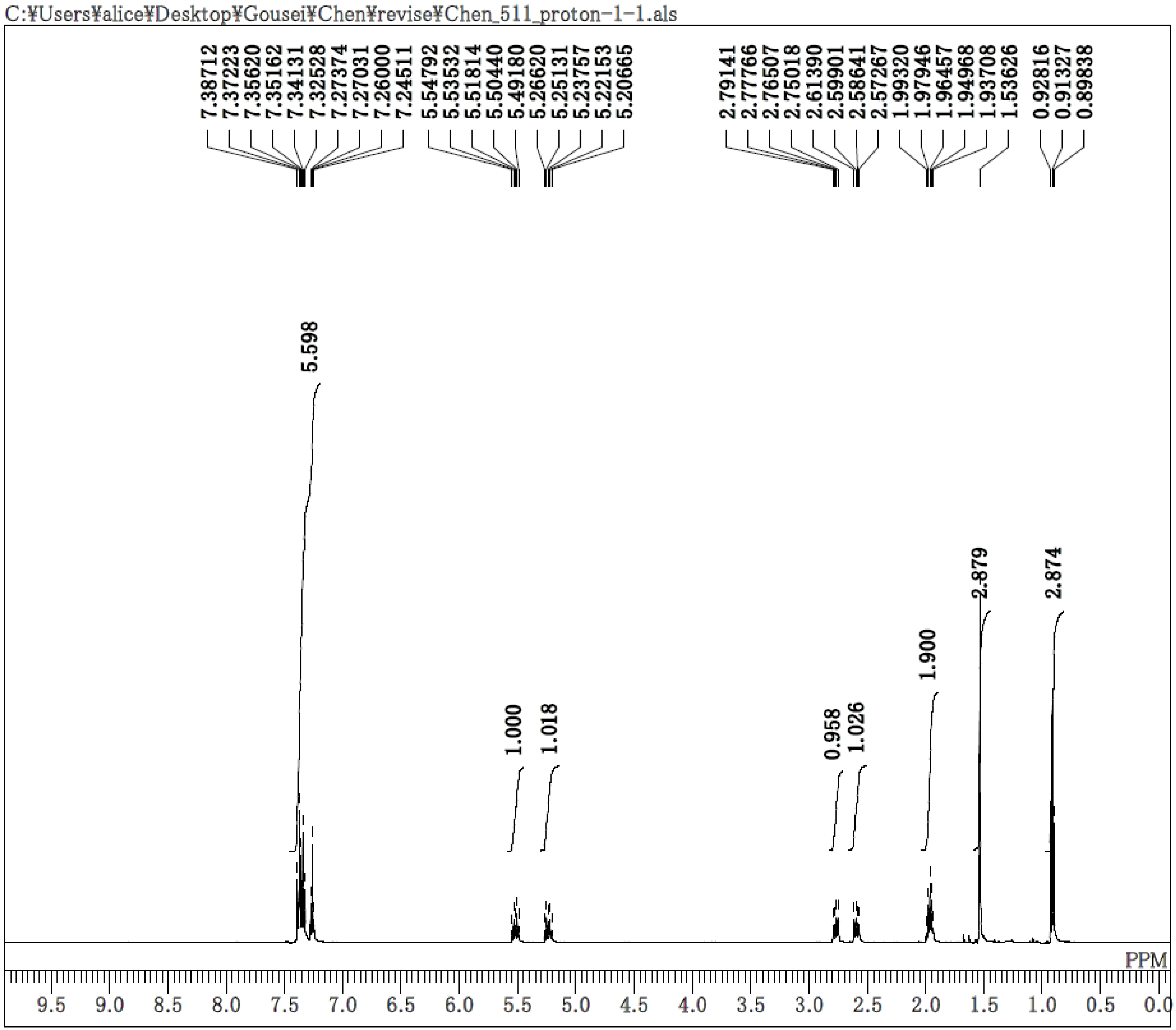


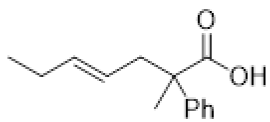
DFILE	RSR-3a_proton.als
COMNT	
DATIM	2020-11-06 18:15:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

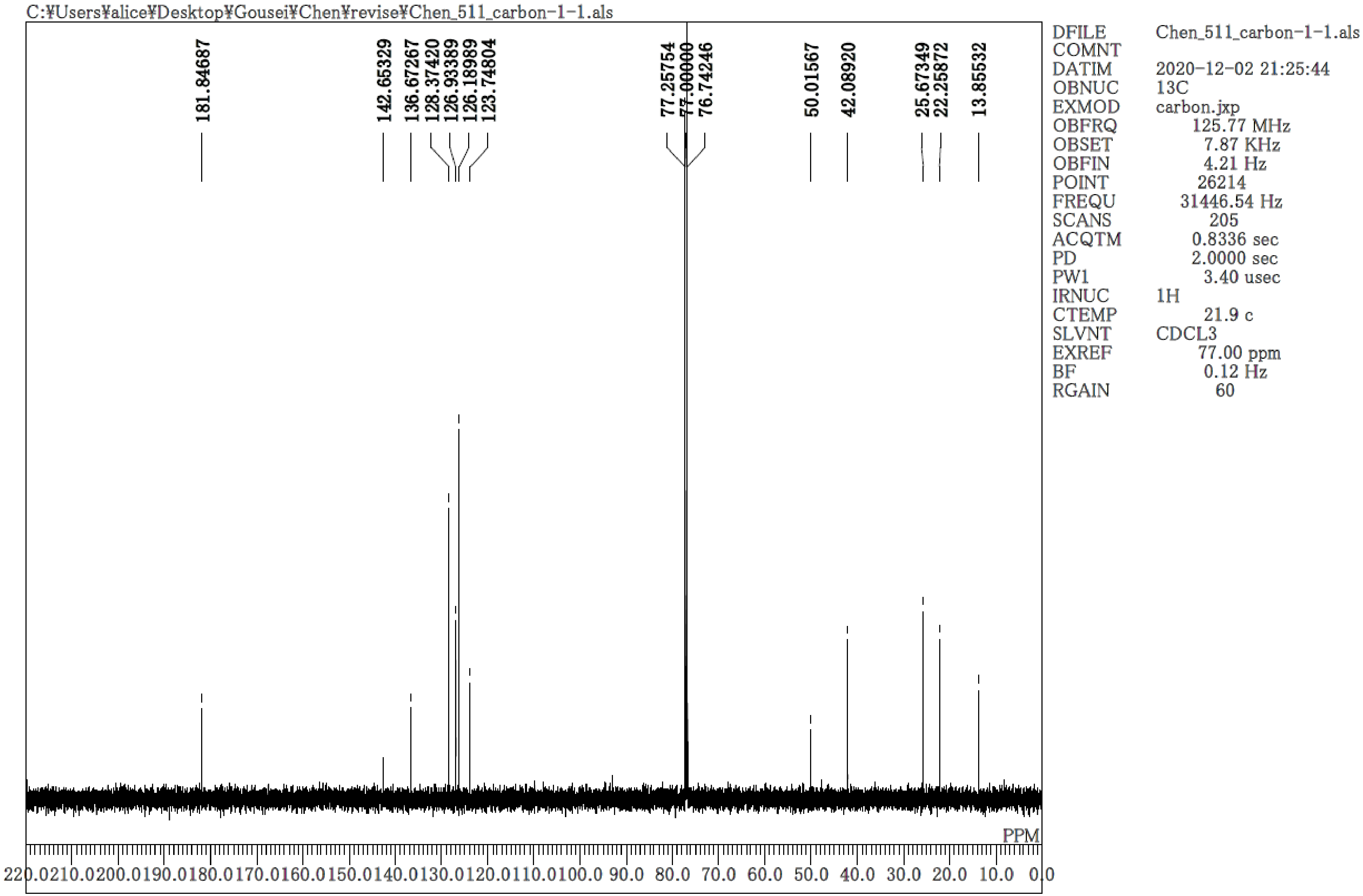


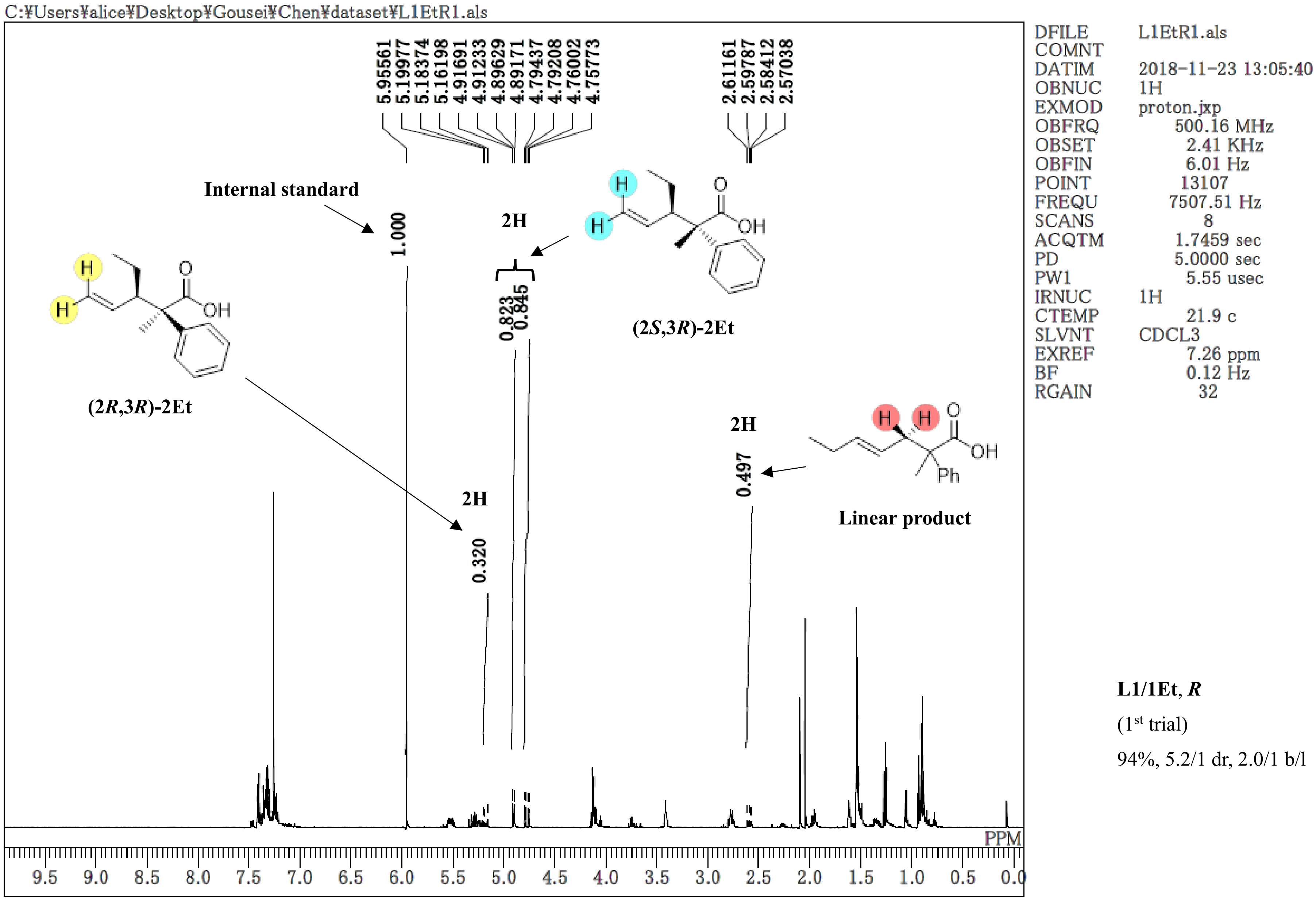
(*3R*,*4S*,*5R*)-**3a**


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥Chen_SM2¥RSR-3a_carbon.als
180.40569
143.19496
128.76412
127.28580
126.09089
81.11325
77.25277
77.00000
76.73957
62.28575
51.72305
49.19536
30.21467
24.70736
22.88436
19.75155
13.78465
PPM
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
DFILE RSR-3a_carbon.als
COMNT
DATIM 2020-11-07 09:50:45
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 492
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60


C:¥Users¥alice¥Desktop¥Gousei¥Chen¥revise¥Chen_511_proton-1-1.als
7.38712
7.37223
7.35620
7.35162
7.34131
7.32528
7.27374
7.27031
7.26000
7.24511
5.54792
5.53532
5.51814
5.50440
5.49180
5.26620
5.25131
5.23757
5.22153
5.20665
2.79141
2.77766
2.76507
2.75018
2.61390
2.59901
2.58641
2.57267
1.99320
1.97946
1.96457
1.94968
1.93708
1.53626
0.92816
0.91327
0.89838
5.598
1.000
1.018
0.958
1.026
1.900
2.879
2.874
PPM
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
DFILE Chen_511_proton-1-1.als
COMNT
DATIM 2020-10-24 21:32:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38


O
OH
Ph

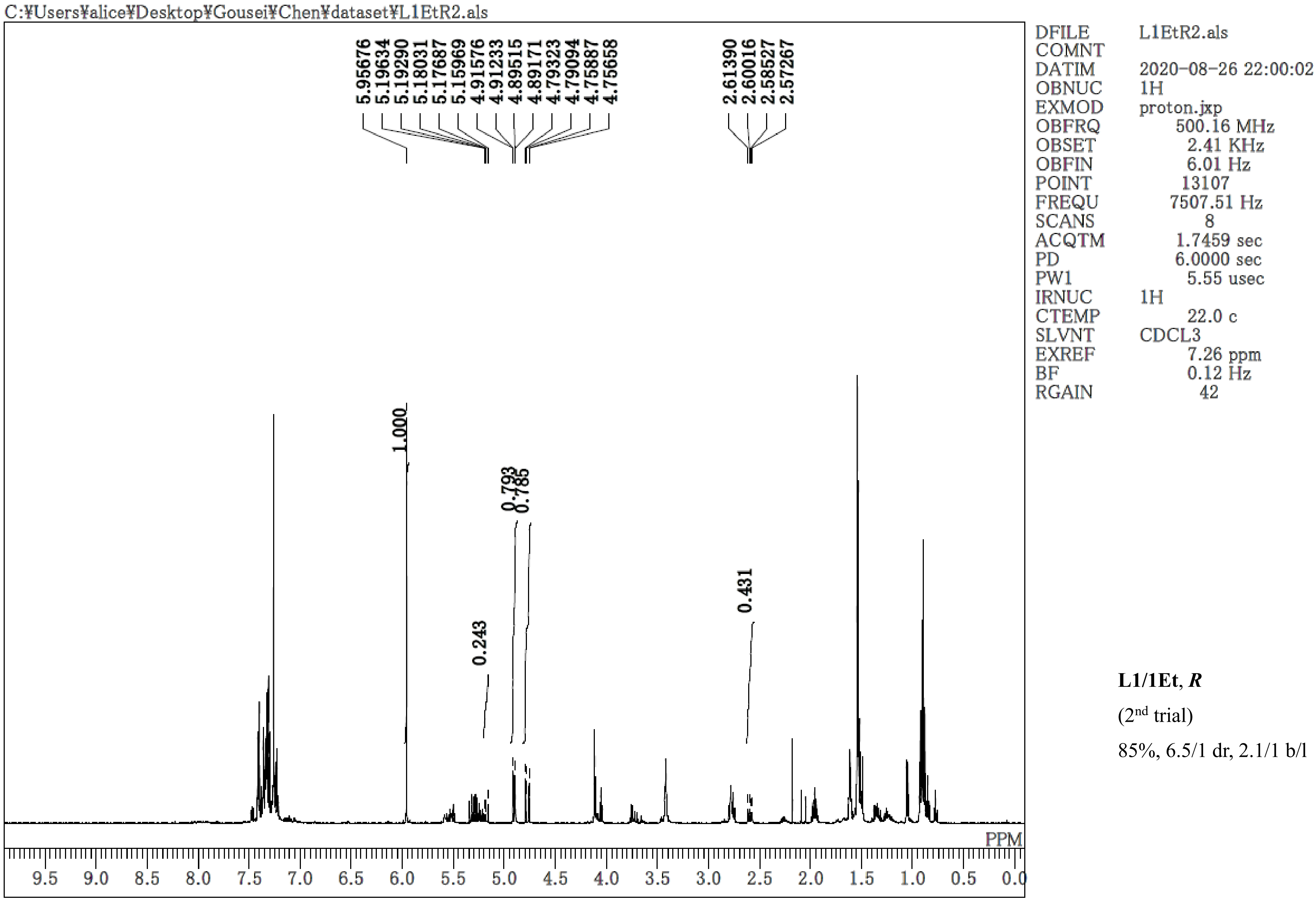

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥revise¥Chen_511_carbon-1-1.als
181.84687
142.65329
136.67267
128.37420
126.93389
126.18989
123.74804
77.25754
77.00000
76.74246
50.01567
42.08920
25.67349
22.25872
13.85532
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
DFILE Chen_511_carbon-1-1.als
COMNT
DATIM 2020-12-02 21:25:44
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 205
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



L1/1Et, *R*

(1st trial)

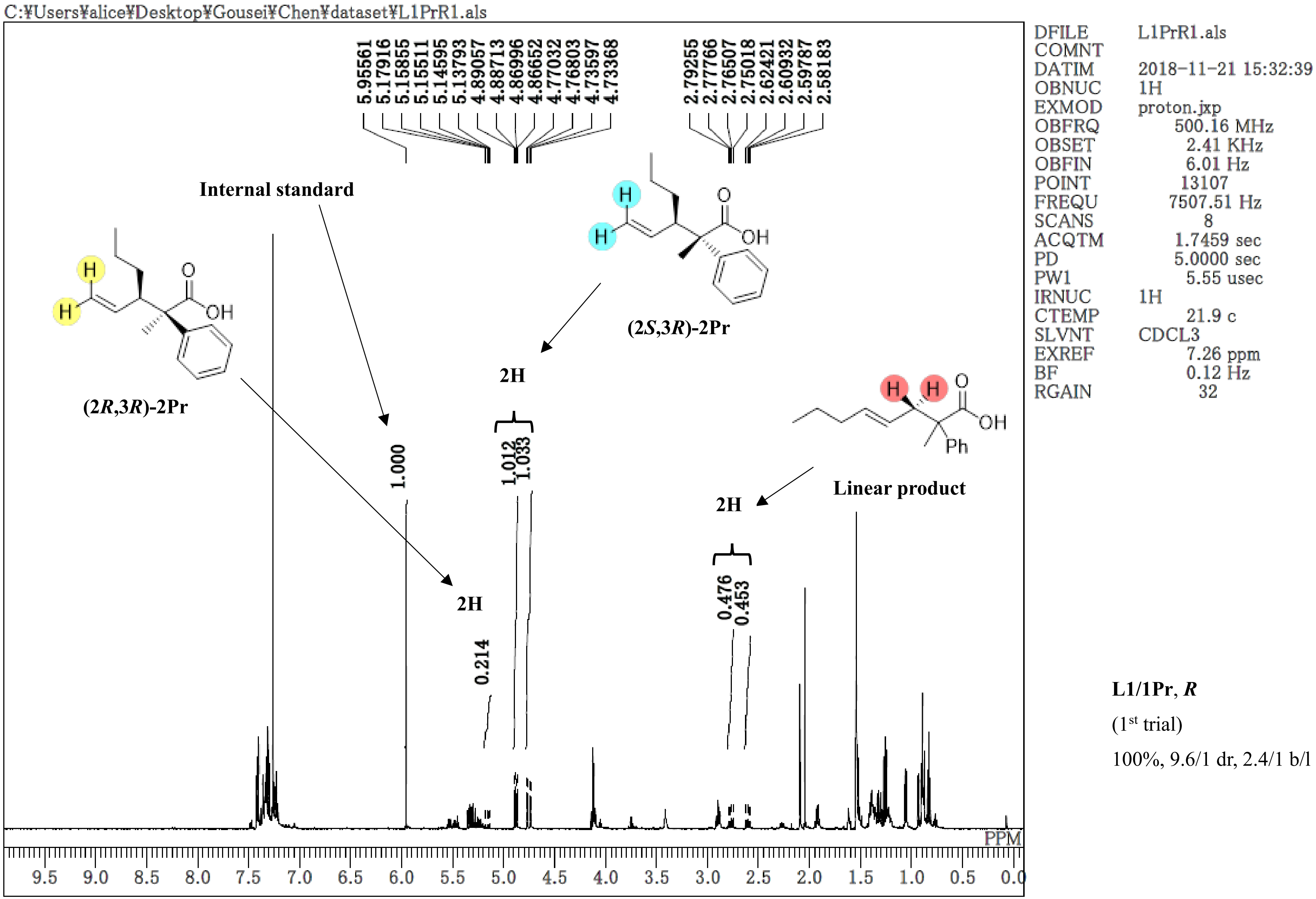
94%, 5.2/1 dr, 2.0/1 b/l



L1/1Et, ***R***

(2nd trial)

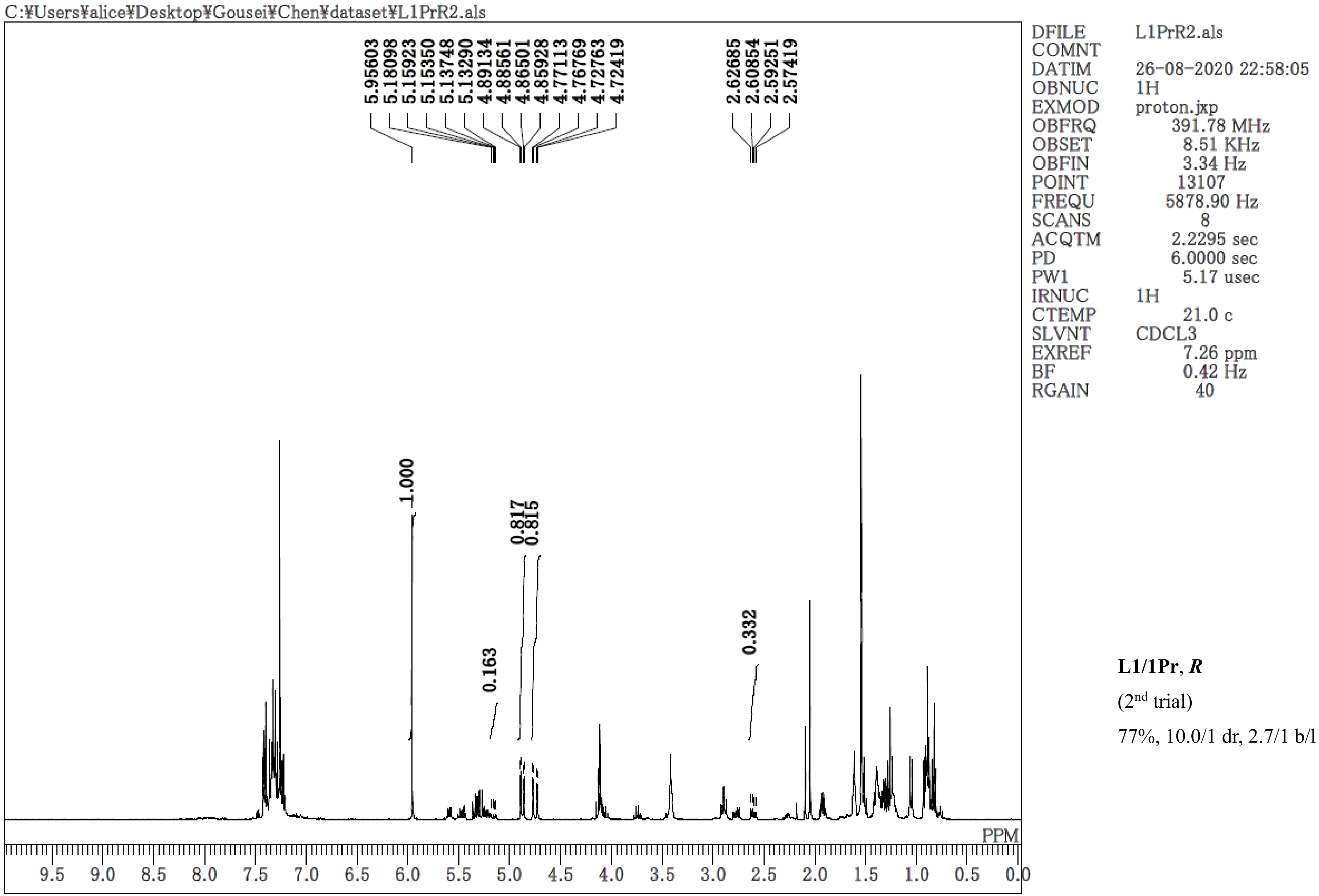
85%, 6.5/1 dr, 2.1/1 b/l



L1/1Pr, *R*

(1st trial)

100%, 9.6/1 dr, 2.4/1 b/l

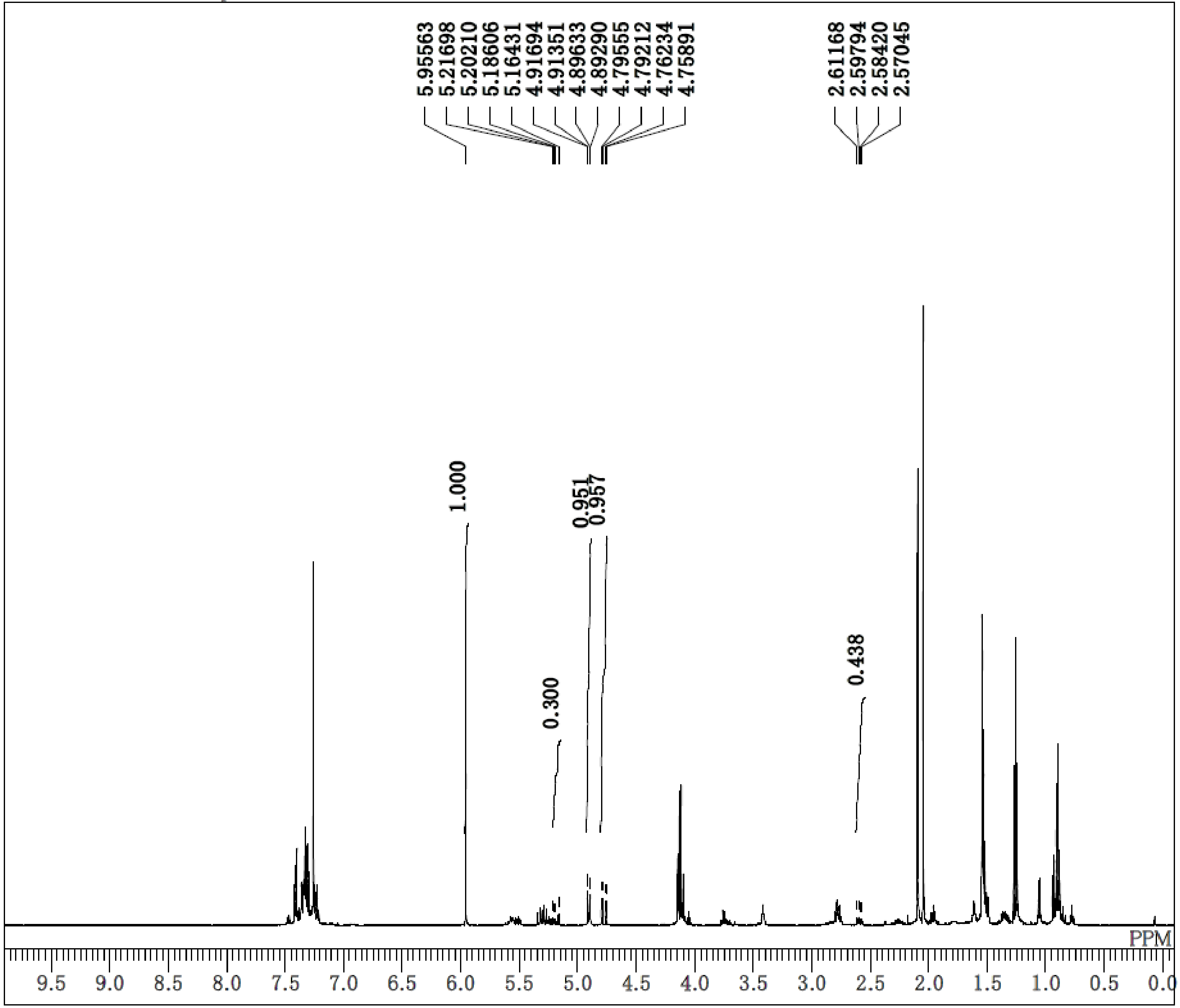


L1/1Pr, ***R***

(2nd trial)

77%, 10.0/1 dr, 2.7/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L2EtR1.als



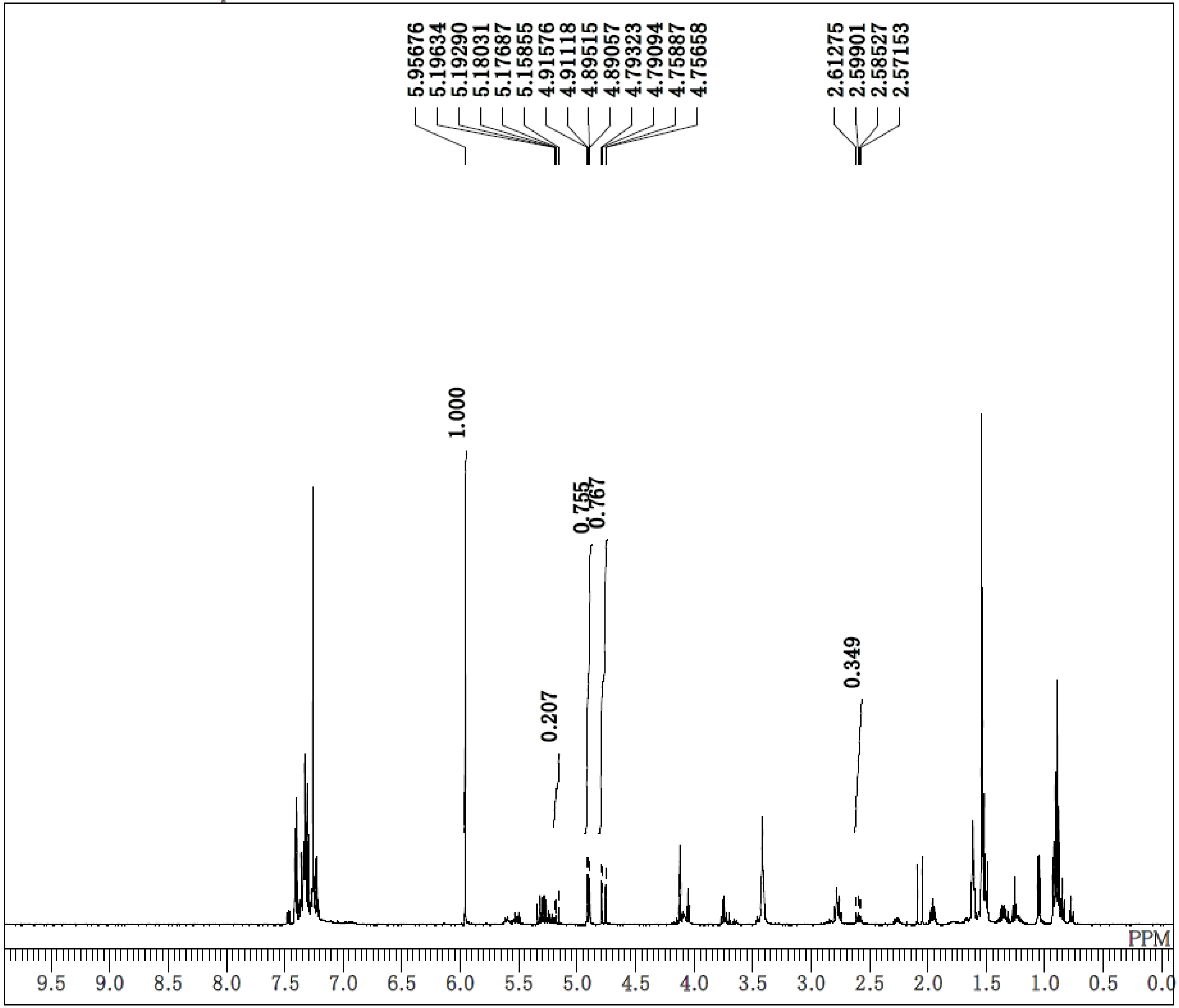
DFILE	L2EtR1.als
COMNT	
DATIM	2018-11-13 16:21:37
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L2/1Et, ***R***

(1st trial)

97%, 6.1/1 dr, 2.4/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L2EtR2.als

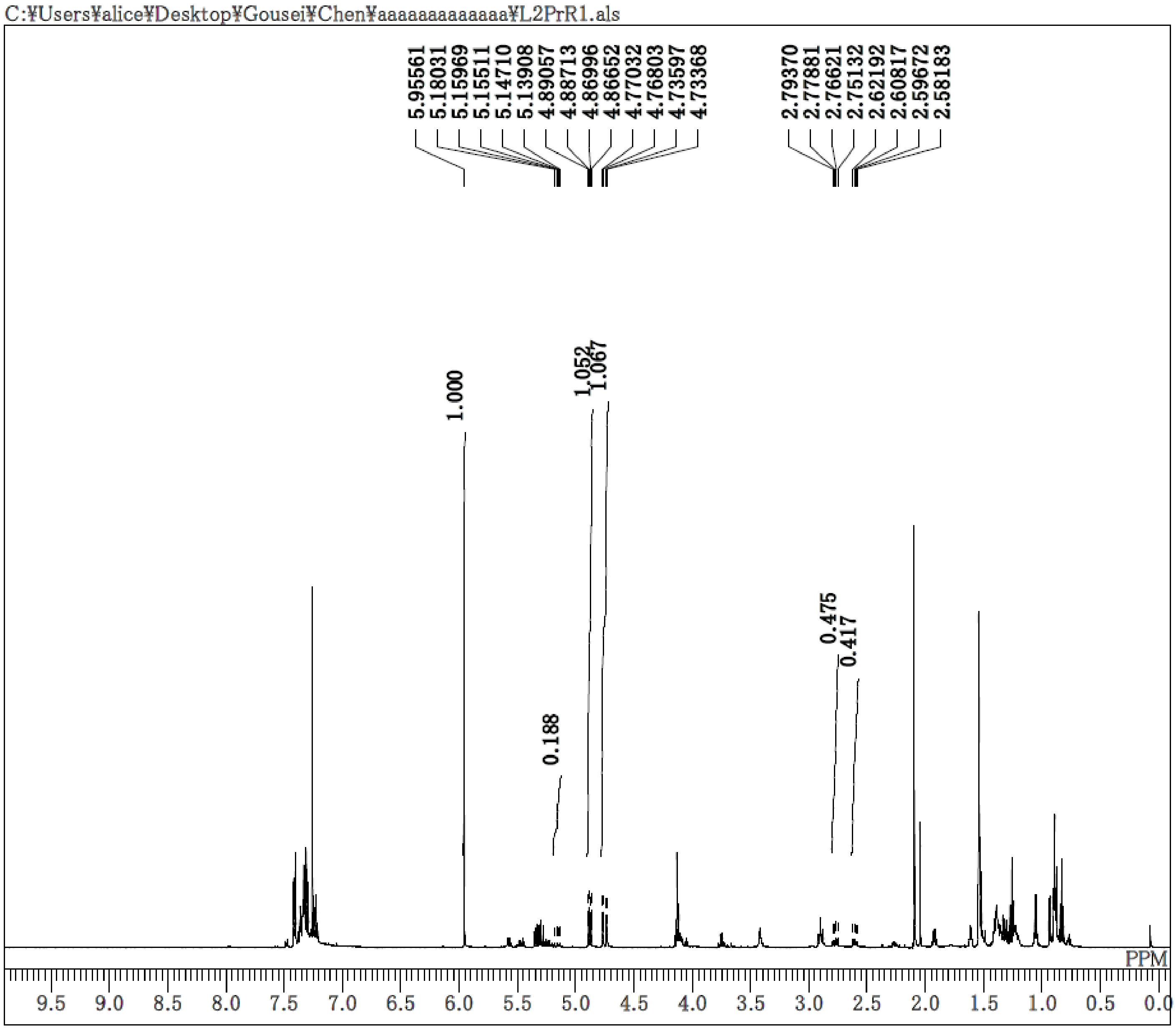


DFILE	L2EtR2.als
COMNT	
DATIM	2020-08-28 00:42:14
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L2/1Et, ***R***

(2^{nd} trial)

76%, 7.4/1 dr, 2.5/1 b/l

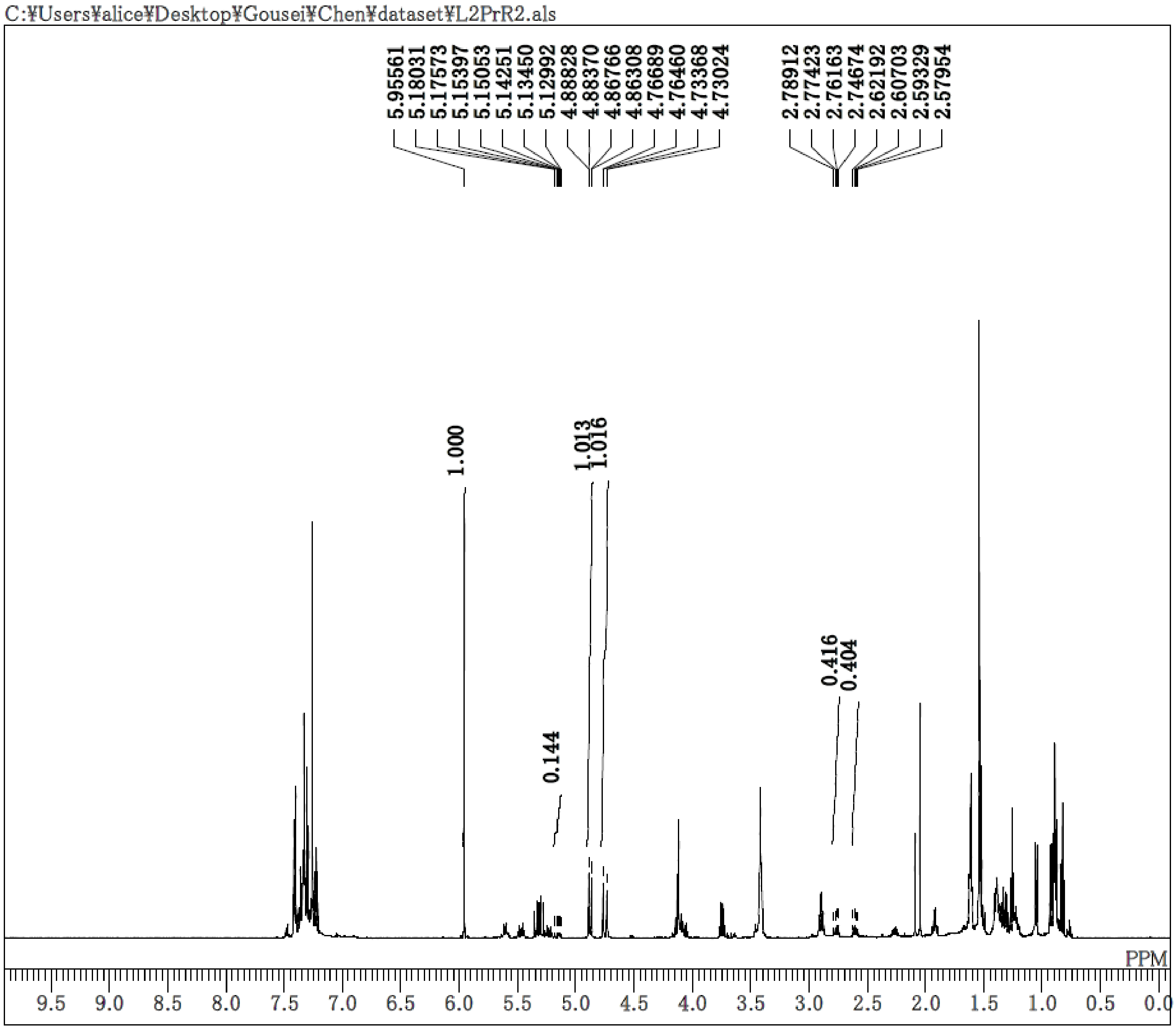


DFILE	L2PrR1.als
COMNT	
DATIM	2018-11-11 23:00:26
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L2/1Pr, ***R***

(1st trial)

100%, 11.3/1 dr, 2.6/1 b/l

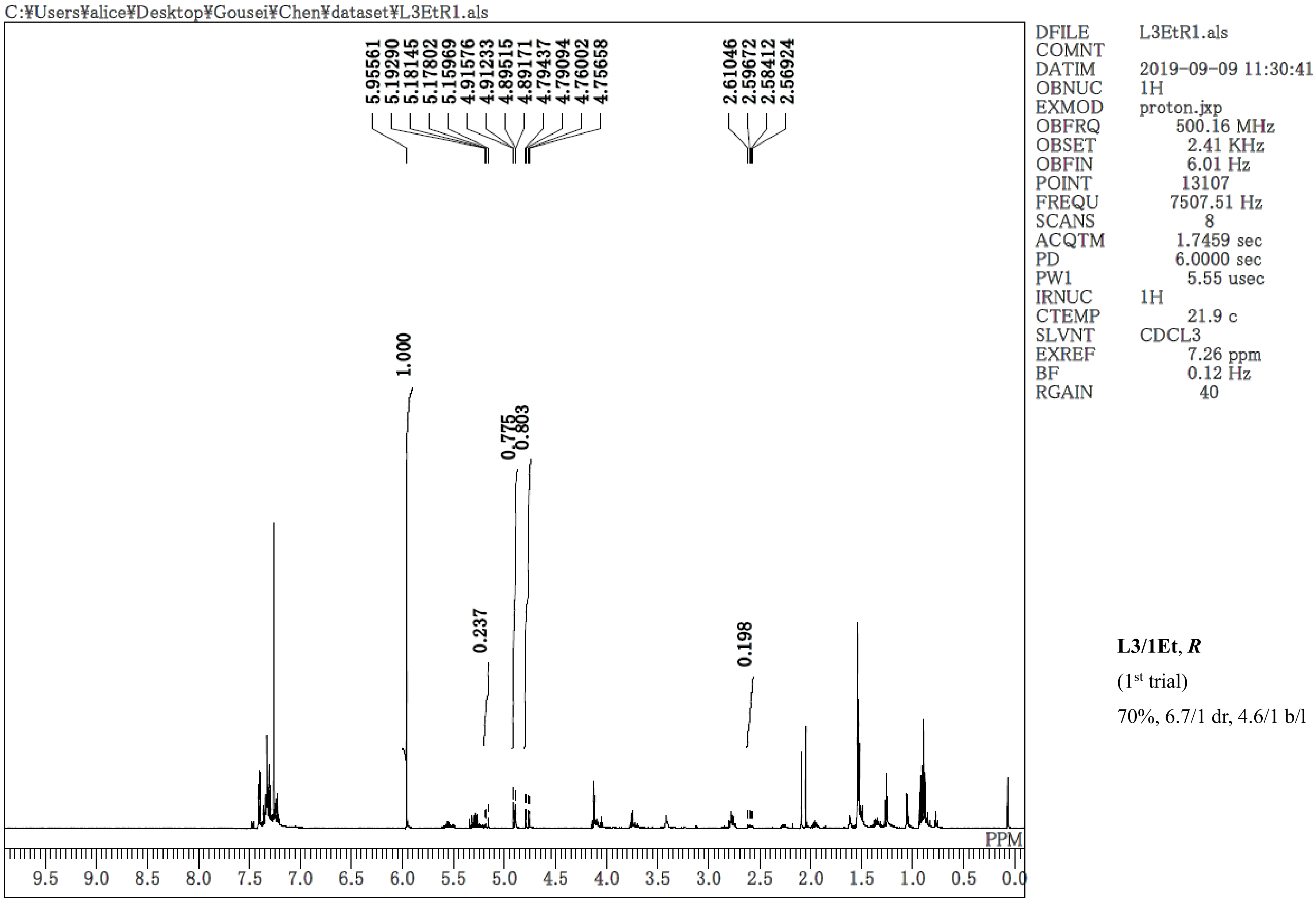


DFILE	L2PrR2.als
COMNT	
DATIM	2020-08-28 00:49:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L2/1Pr, ***R***

(2nd trial)

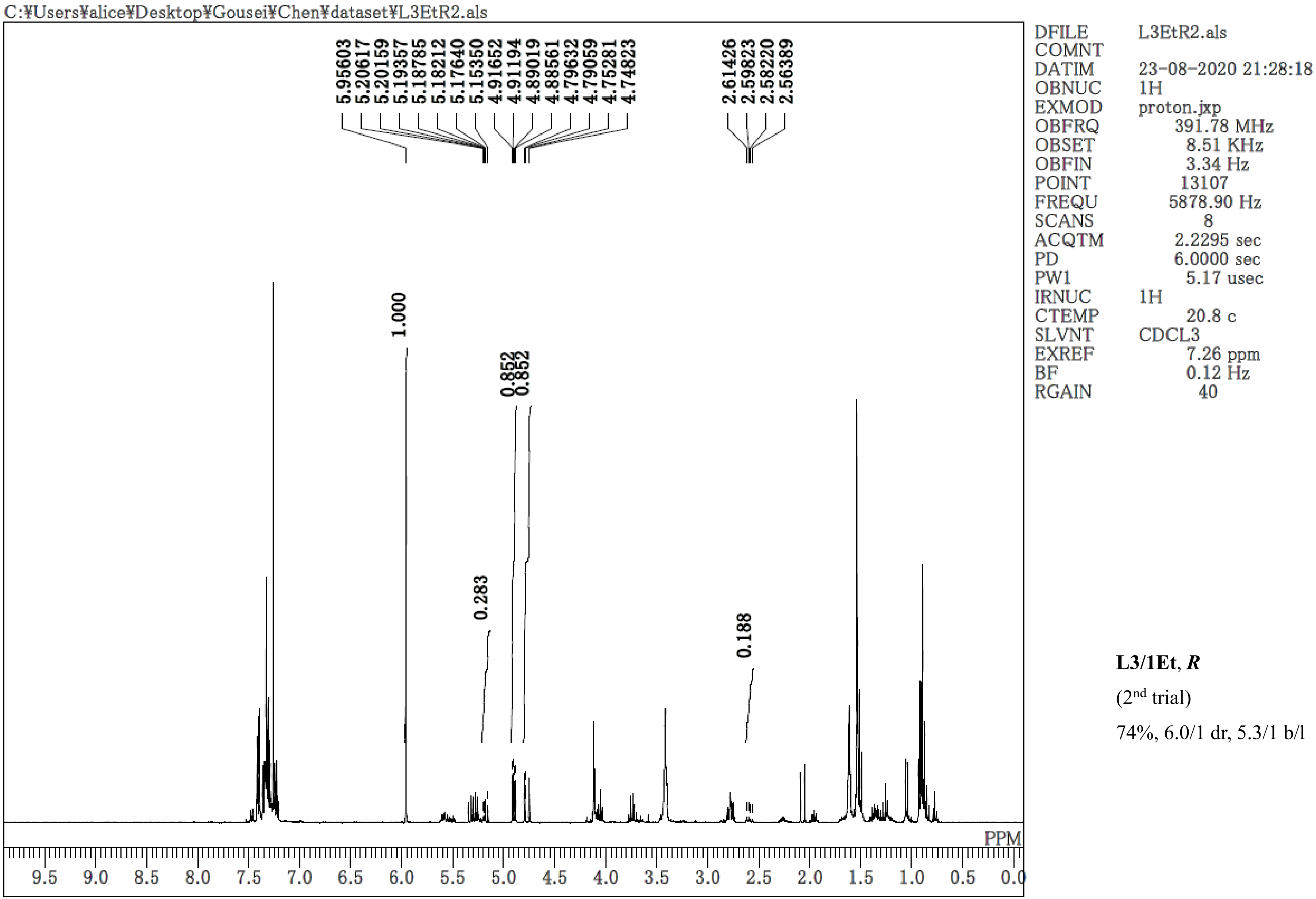
94%, 14.1/1 dr, 2.7/1 b/l



L3/1Et, ***R***

(1st trial)

70%, 6.7/1 dr, 4.6/1 b/l

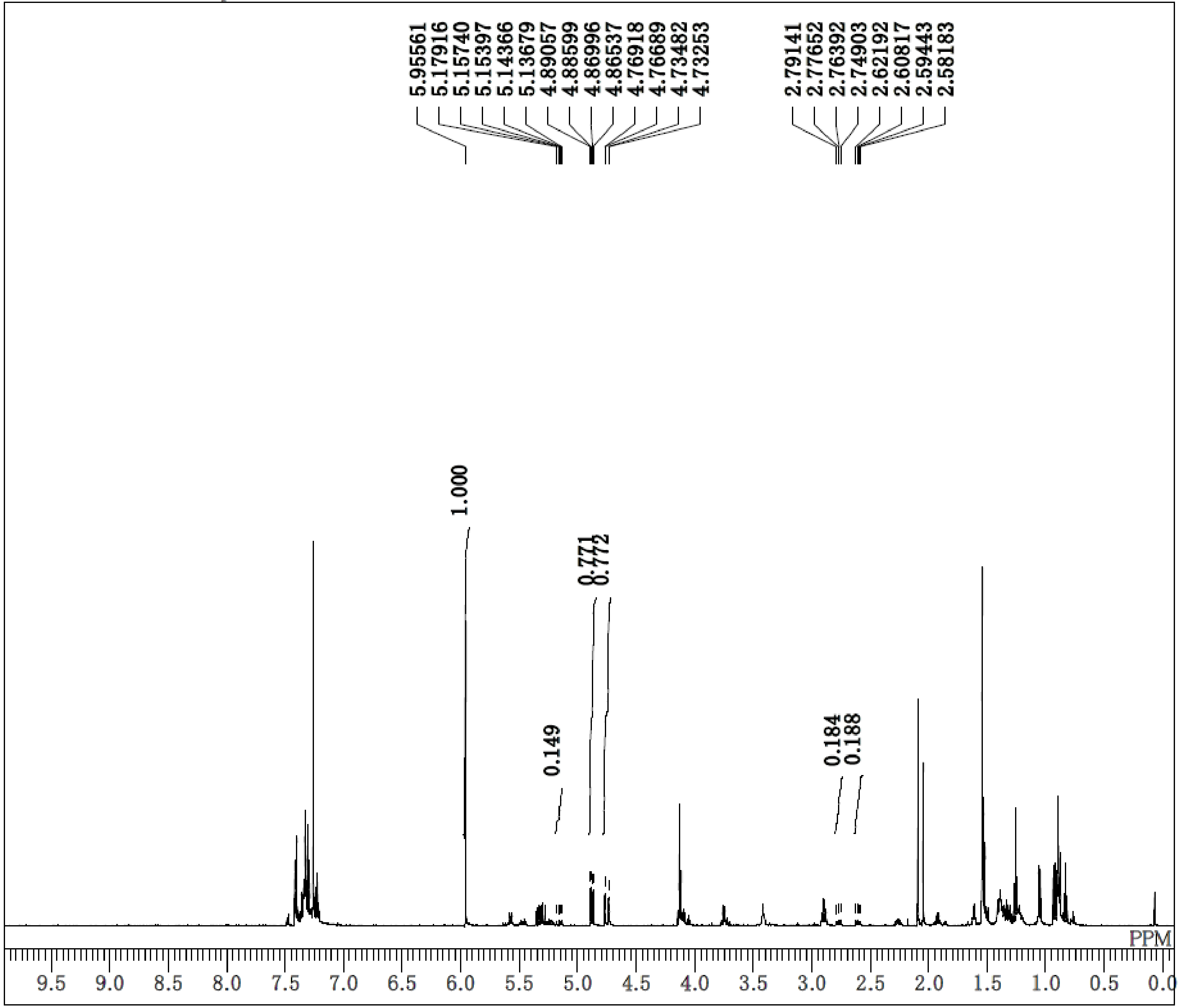


L3/1Et, ***R***

(2nd trial)

74%, 6.0/1 dr, 5.3/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L3PrR1.als

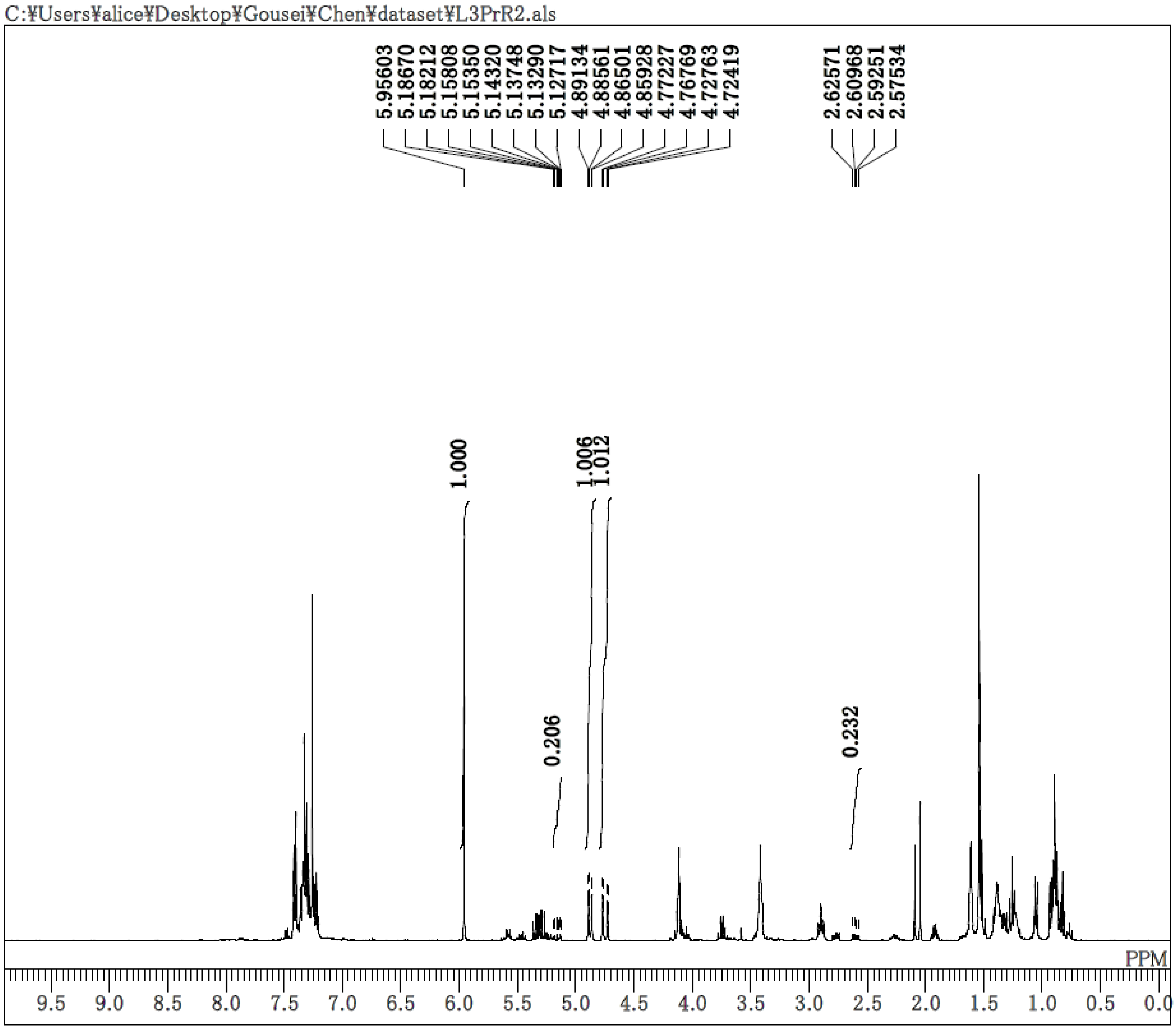


DFILE	L3PrR1.als
COMNT	
DATIM	2019-09-09 11:37:36
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L3/1Pr, ***R***

(1st trial)

65%, 10.4/1 dr, 4.5/1 b/l



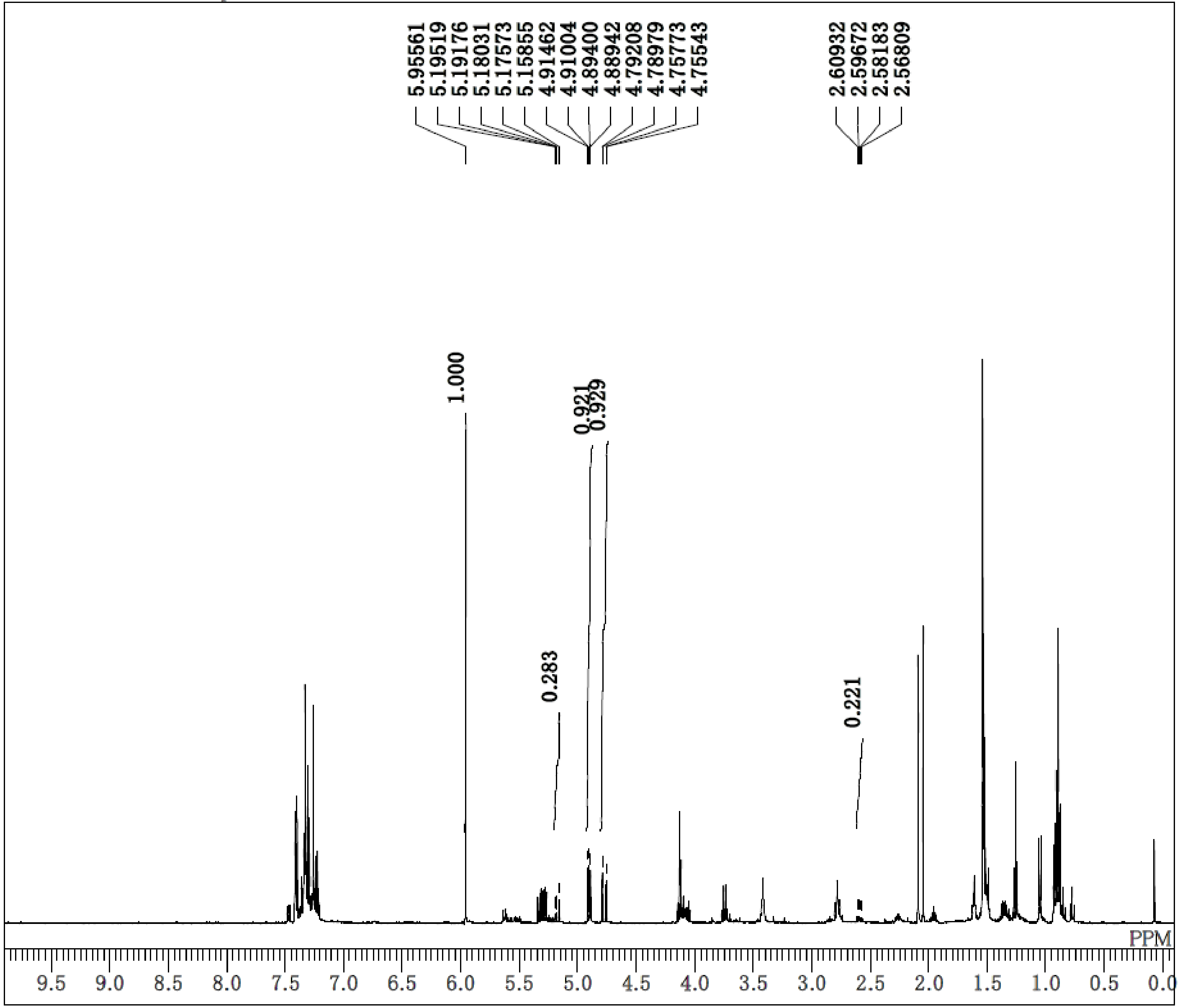
DFILE	L3PrR2.als
COMNT	
DATIM	22-08-2020 13:57:30
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.62 Hz
RGAIN	40

L3/1Pr, ***R***

(2nd trial)

85%, 9.8/1 dr, 4.8/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L4EtR1.als

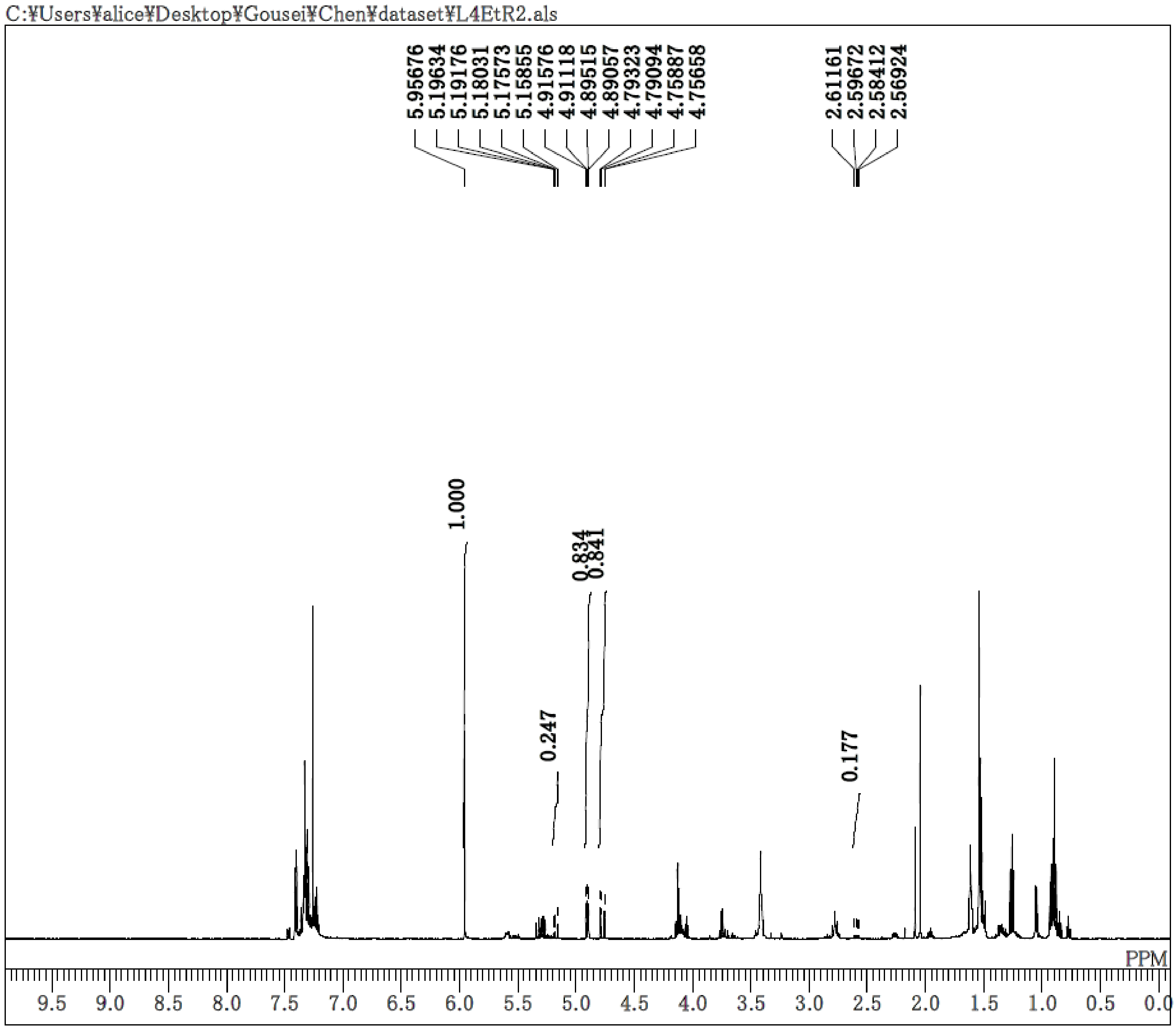


DFILE	L4EtR1.als
COMNT	
DATIM	2019-09-28 15:03:15
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L4/1Et, *R*

(1st trial)

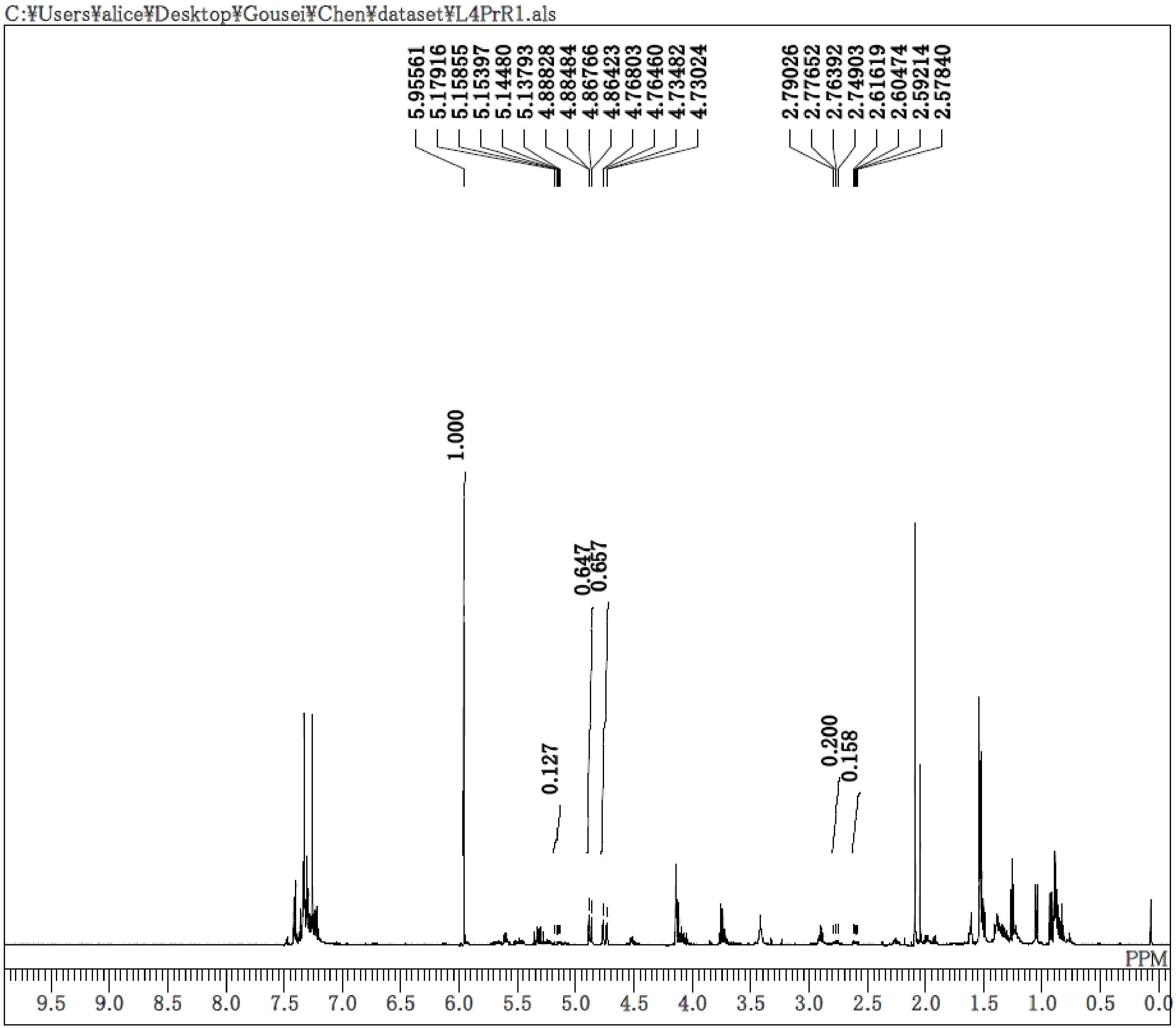
81%, 6.5/1 dr, 4.8/1 b/l



L4/1Et, *R*

(2nd trial)

72%, 6.8/1 dr, 5.4/1 b/l

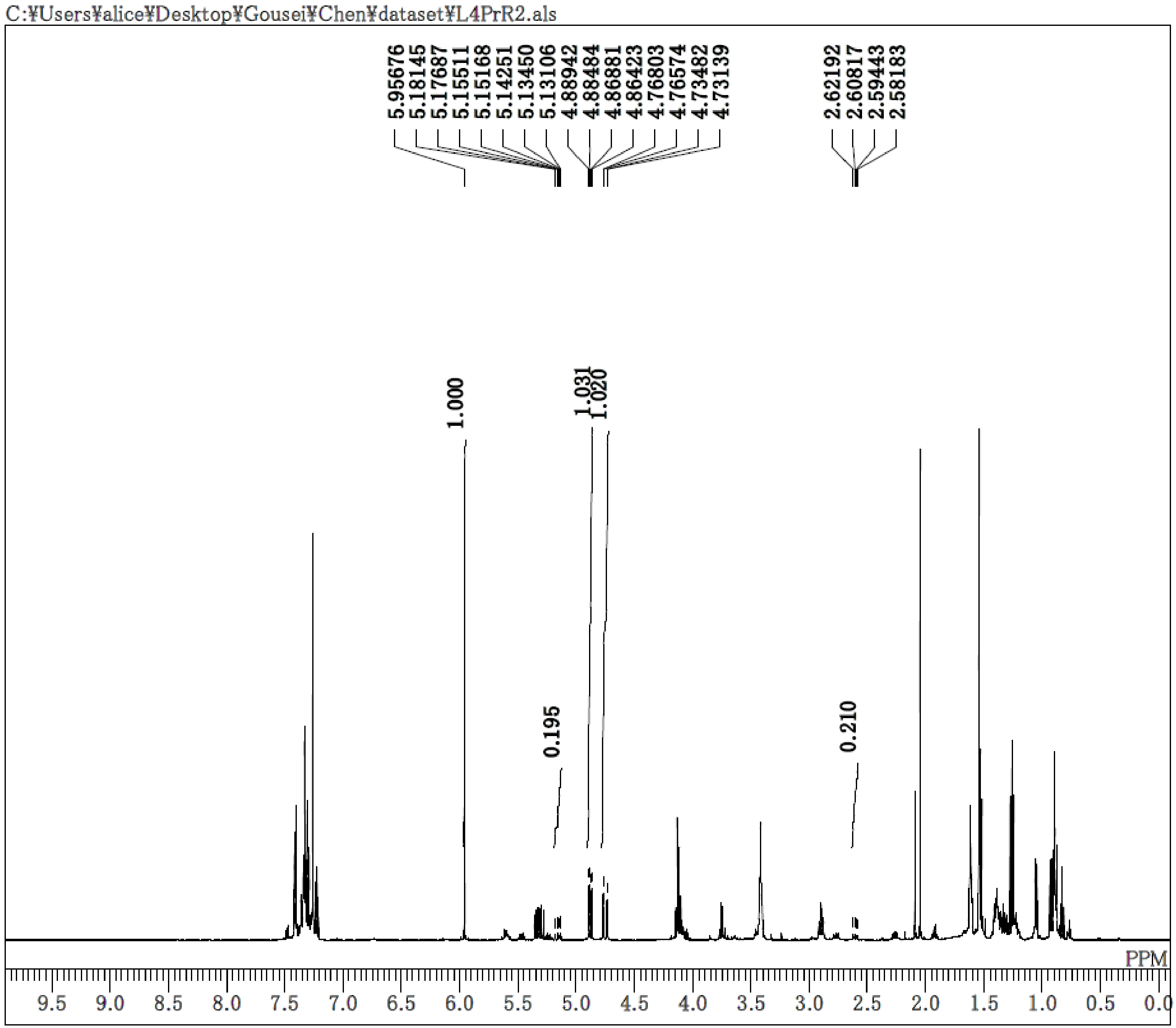


DFILE	L4PrR1.als
COMNT	
DATIM	2019-09-28 15:19:42
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L4/1Pr, ***R***

(1st trial)

56%, 10.3/1 dr, 4.0/1 b/l



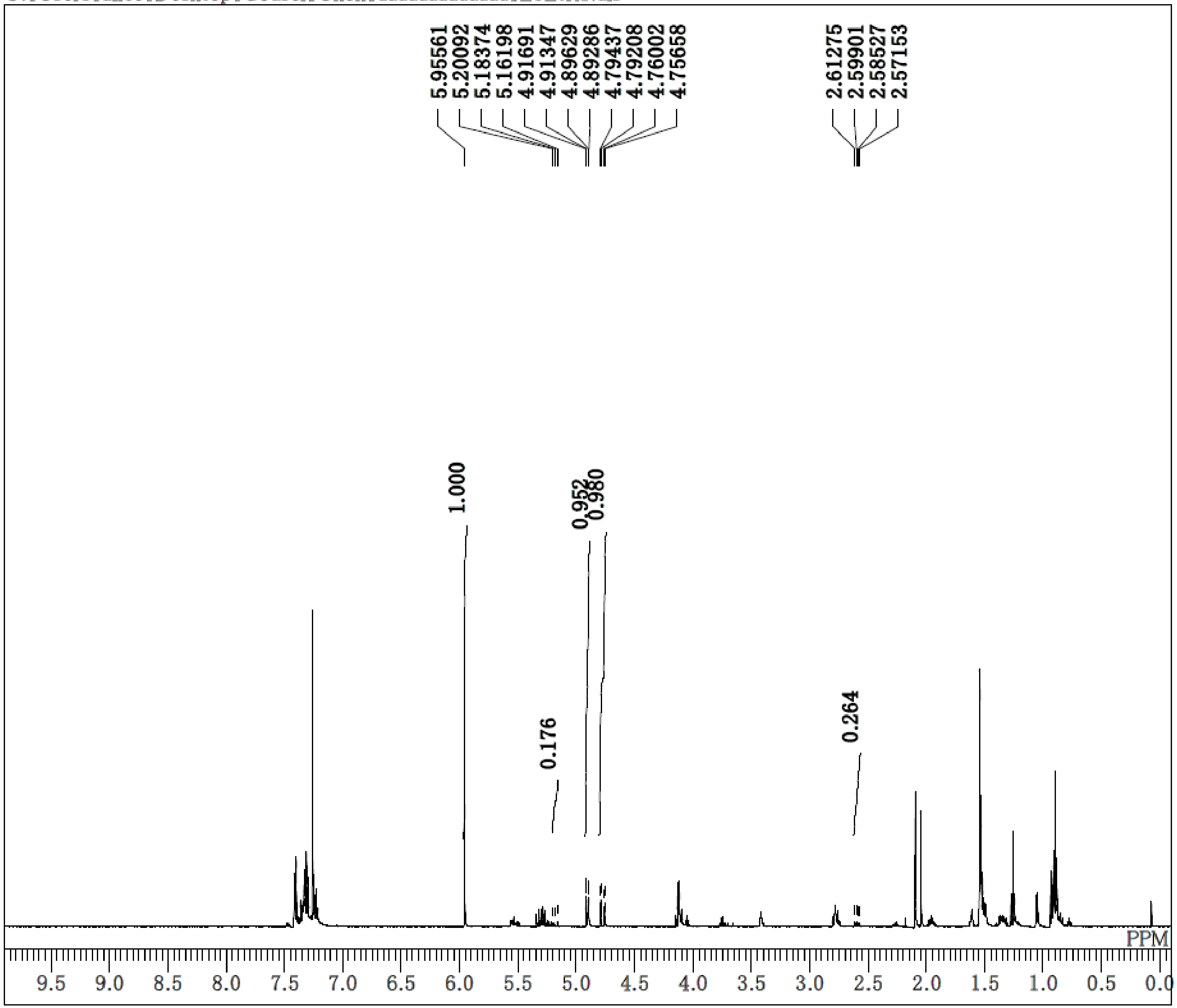
DFILE	L4PrR2.als
COMNT	
DATIM	2020-08-28 01:19:43
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L4/1Pr, ***R***

(2nd trial)

84%, 10.5/1 dr, 5.3/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥aaaaaaaaaaaaa¥L5EtR1.als

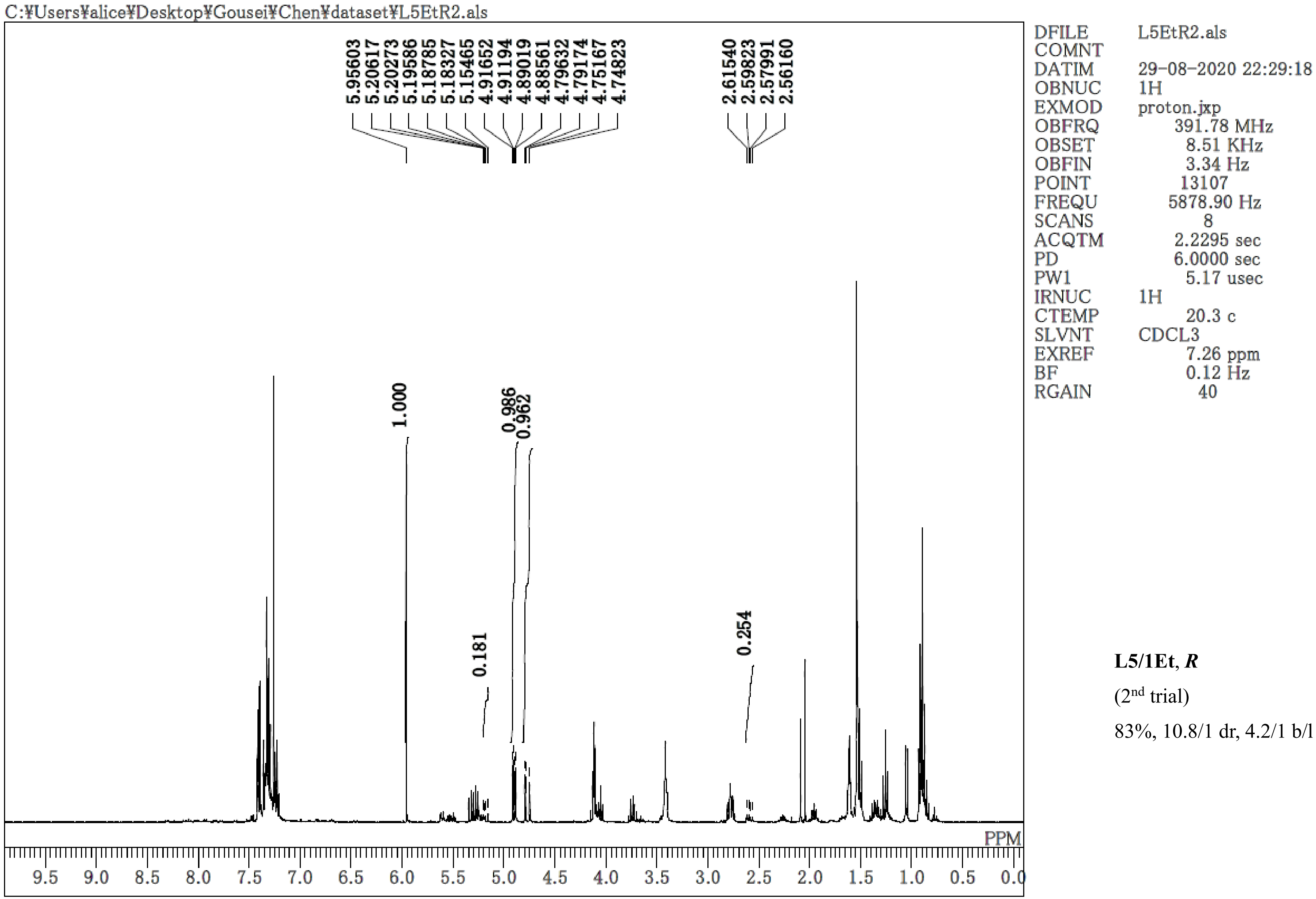


DFILE	L5EtR1.als
COMNT	
DATIM	2018-11-13 15:49:47
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L5/1Et, ***R***

(1st trial)

83%, 11.0/1 dr, 4.0/1 b/l

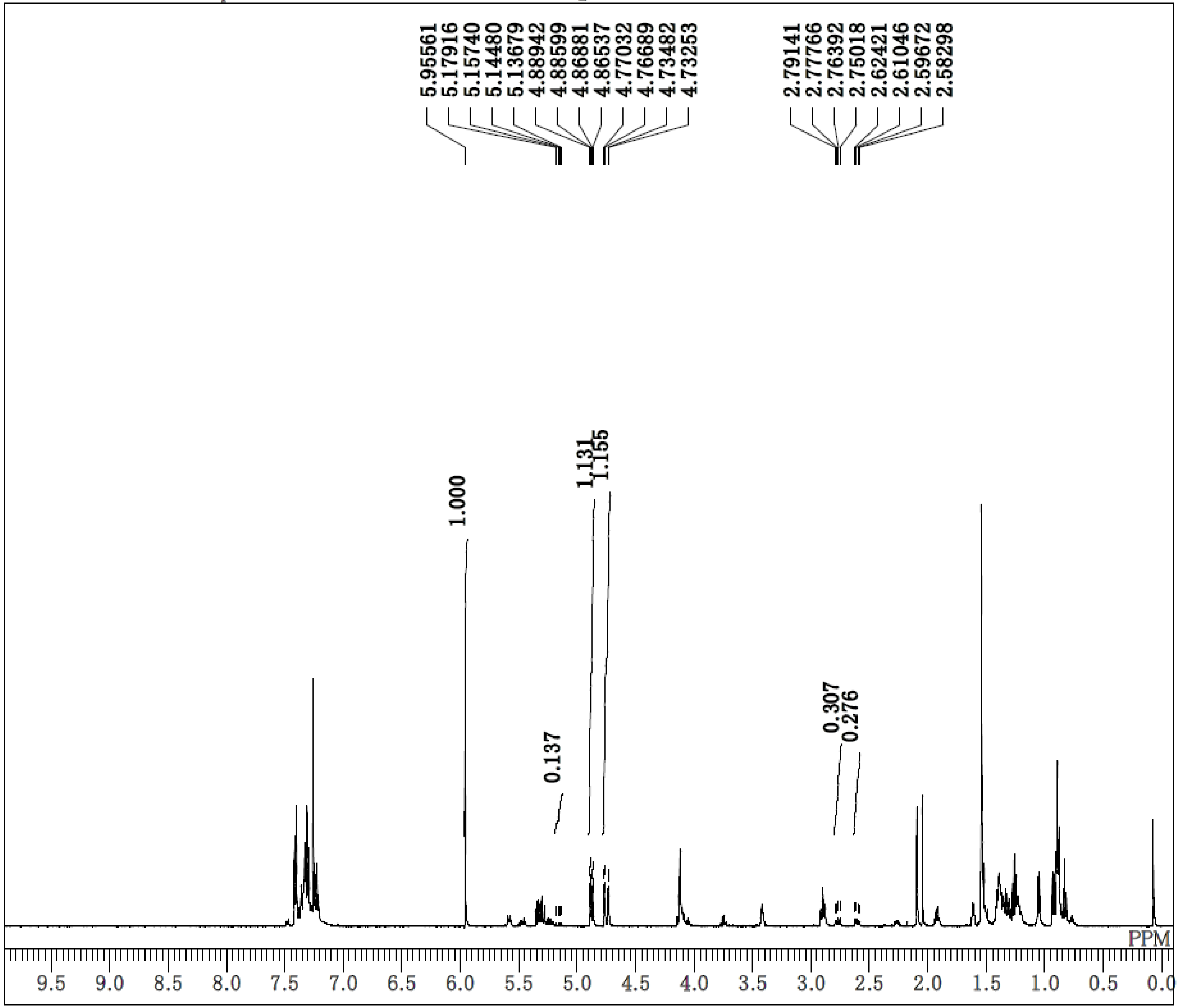


L5/1Et, ***R***

(2nd trial)

83%, 10.8/1 dr, 4.2/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥aaaaaaaaaaaaa¥L5PrR_1.als

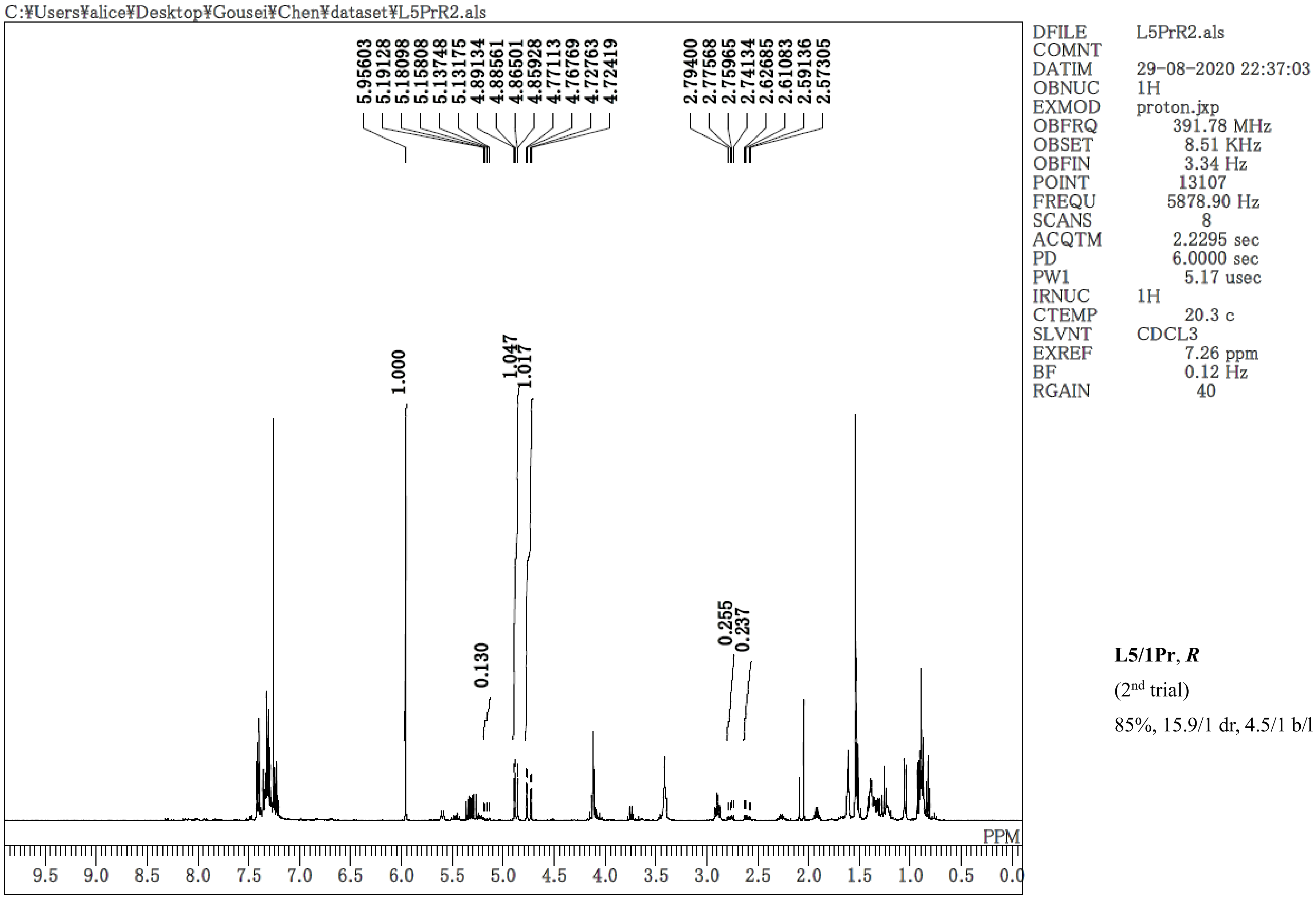


DFILE	L5PrR_1.als
COMNT	
DATIM	2018-11-11 23:26:52
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L5/1Pr, ***R***

(1st trial)

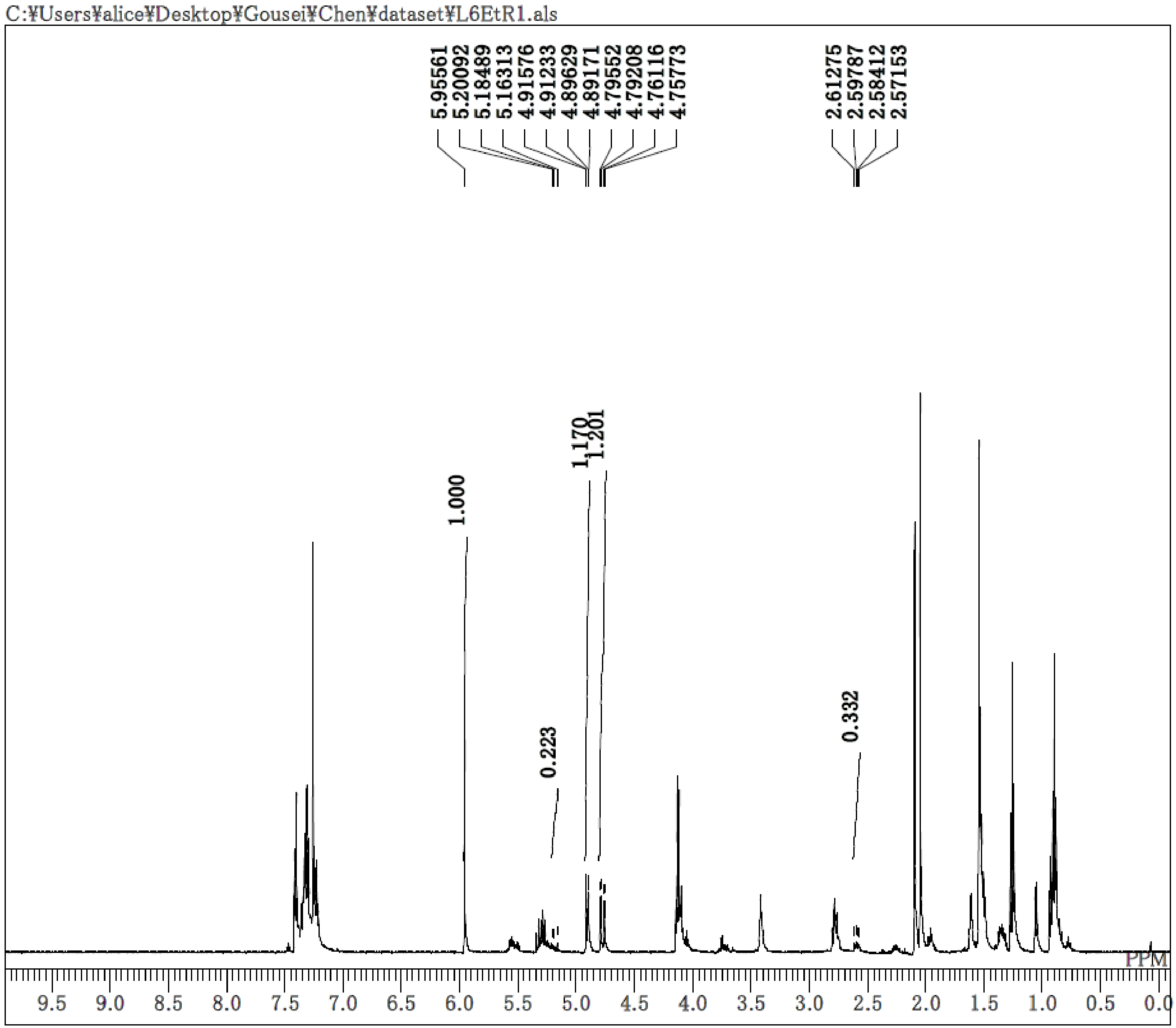
95%, 16.7/1 dr, 4.2/1 b/l



L5/1Pr, ***R***

(2nd trial)

85%, 15.9/1 dr, 4.5/1 b/l

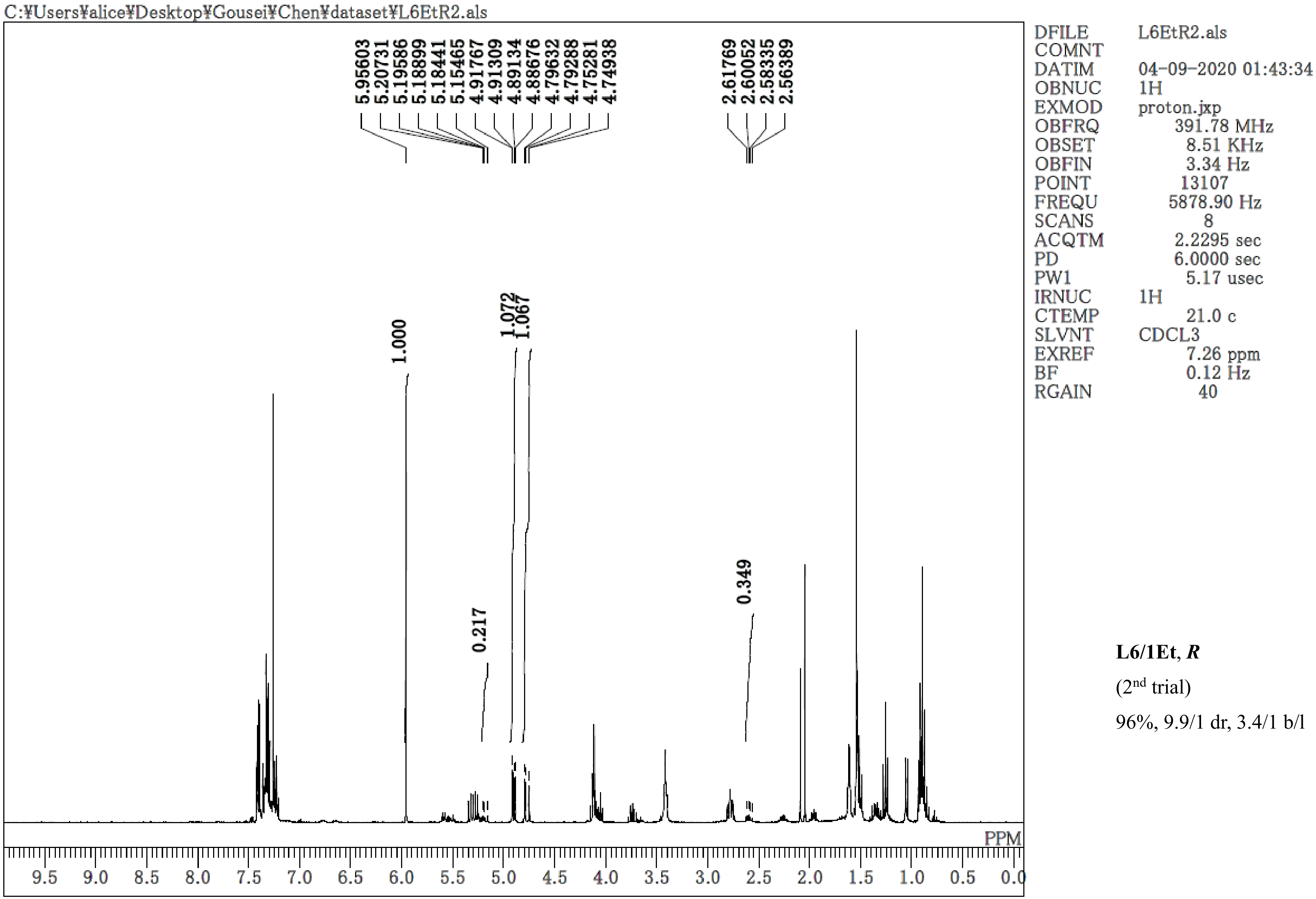


DFILE	L6EtR1.als
COMNT	
DATIM	2018-11-23 13:19:34
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L6/1Et, *R*

(1st trial)

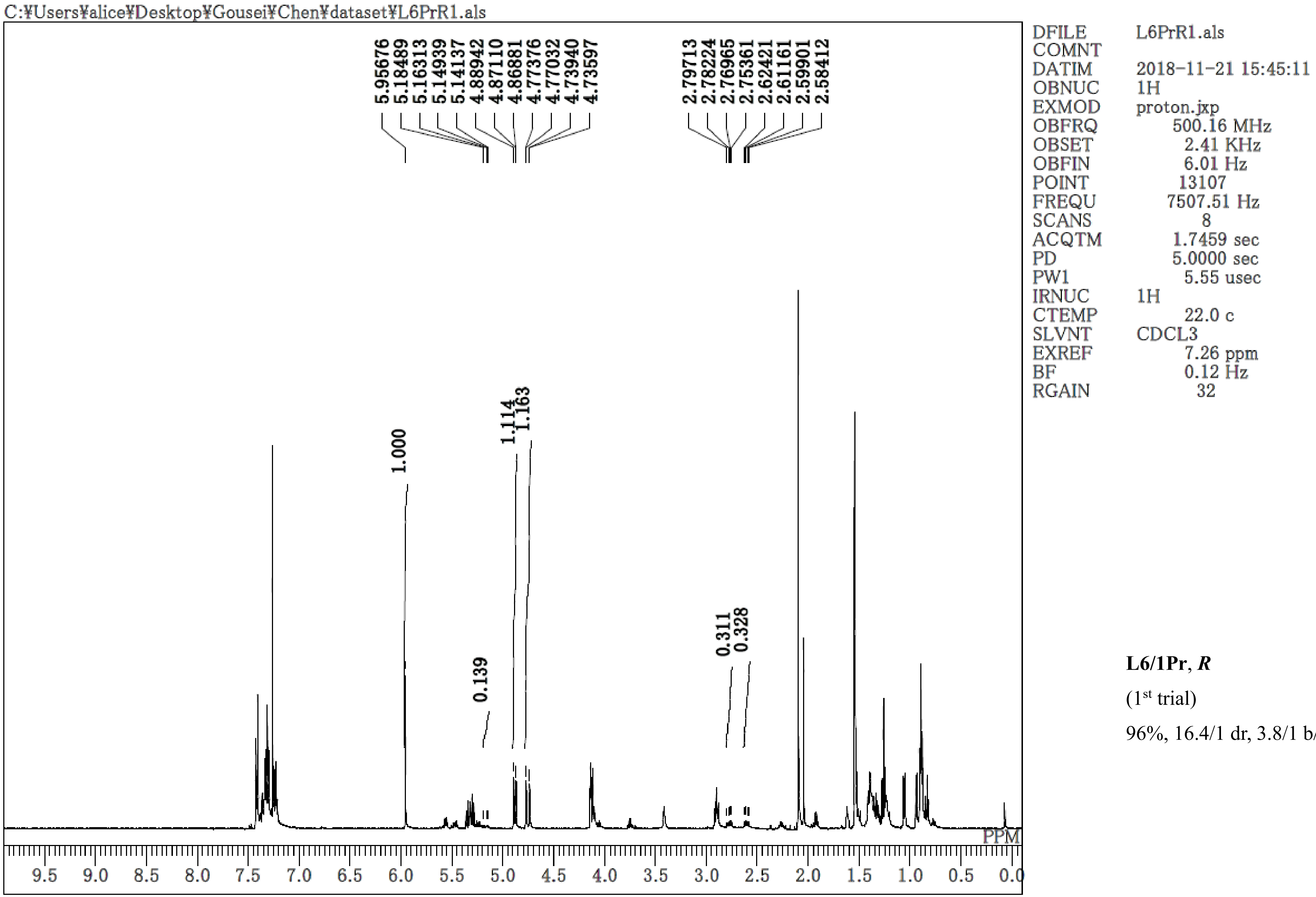
100%, 10.6/1 dr, 3.9/1 b/l



L6/1Et, ***R***

(2nd trial)

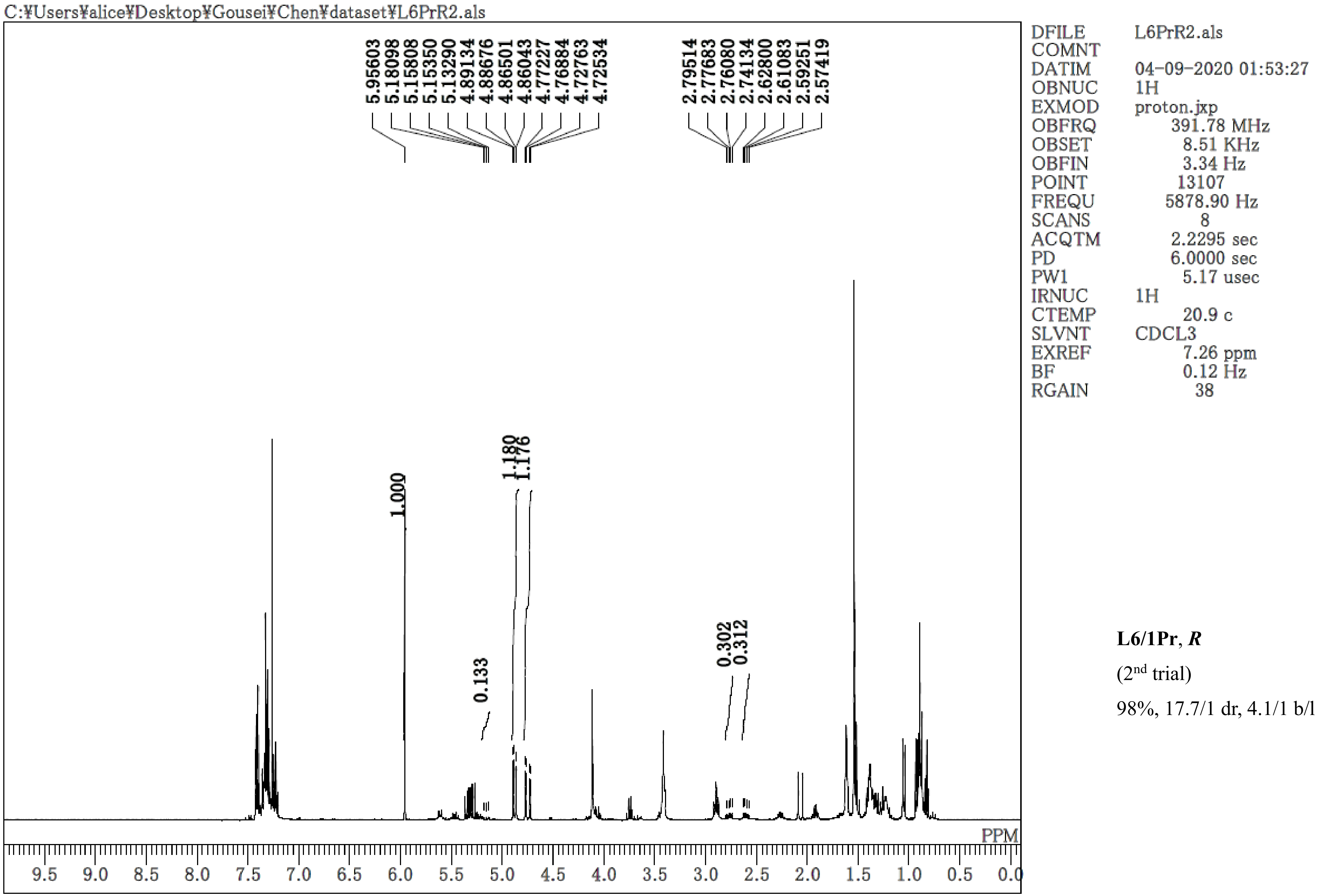
96%, 9.9/1 dr, 3.4/1 b/l



L6/1Pr, ***R***

(1st trial)

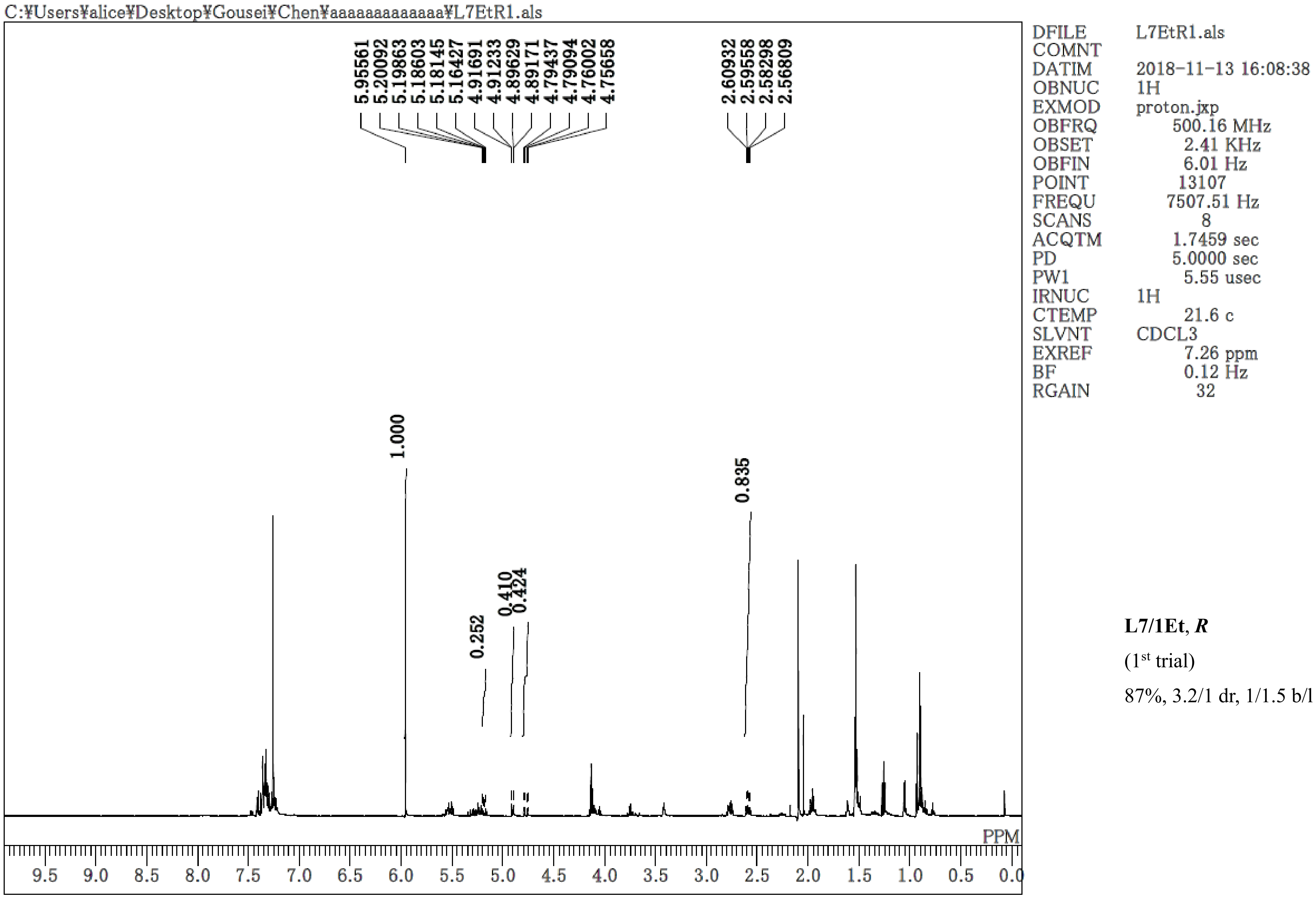
96%, 16.4/1 dr, 3.8/1 b/l



L6/1Pr, ***R***

(2nd trial)

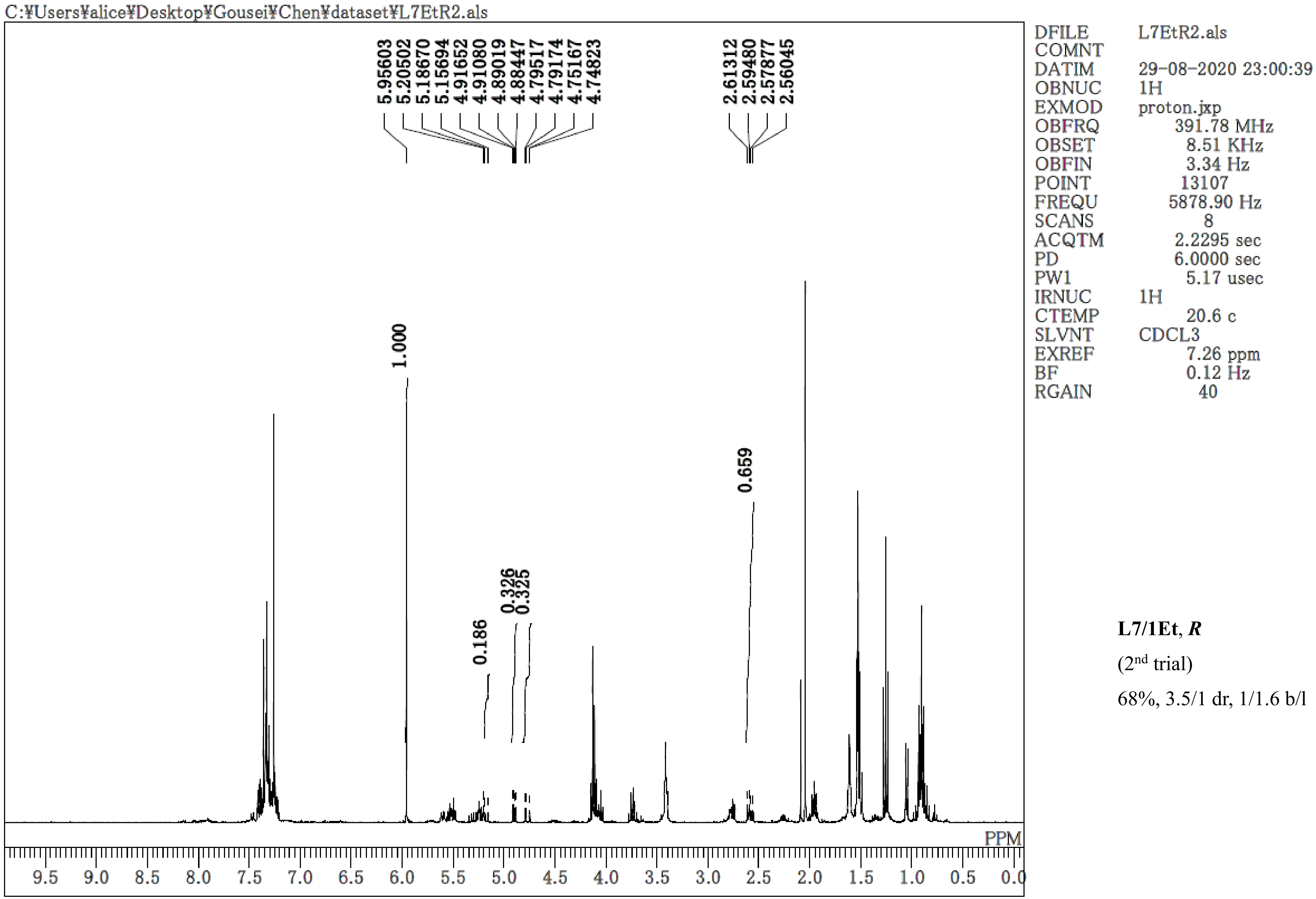
98%, 17.7/1 dr, 4.1/1 b/l



L7/1Et, ***R***

(1st trial)

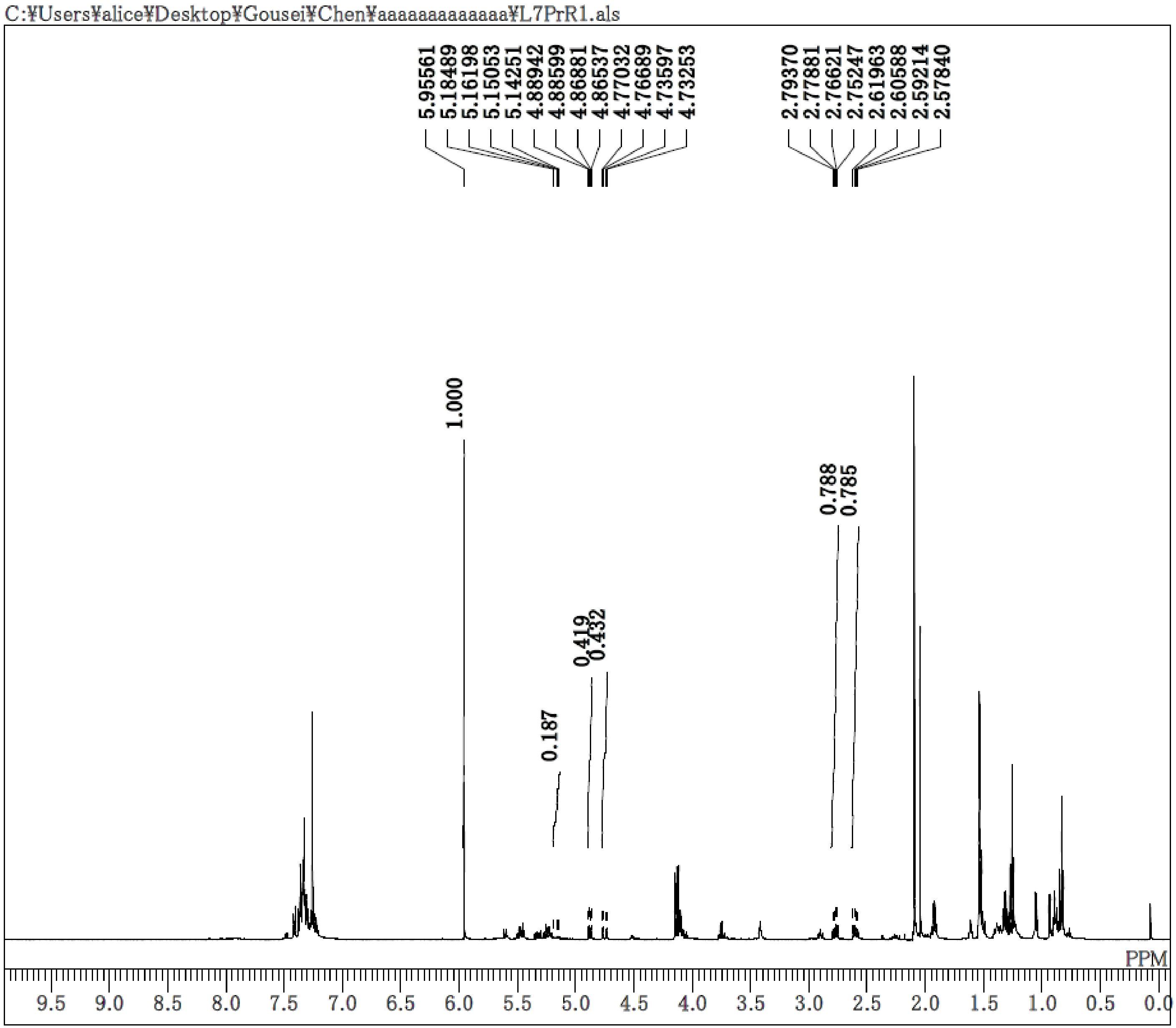
87%, 3.2/1 dr, 1/1.5 b/l



L7/1Et, ***R***

(2nd trial)

68%, 3.5/1 dr, 1/1.6 b/l

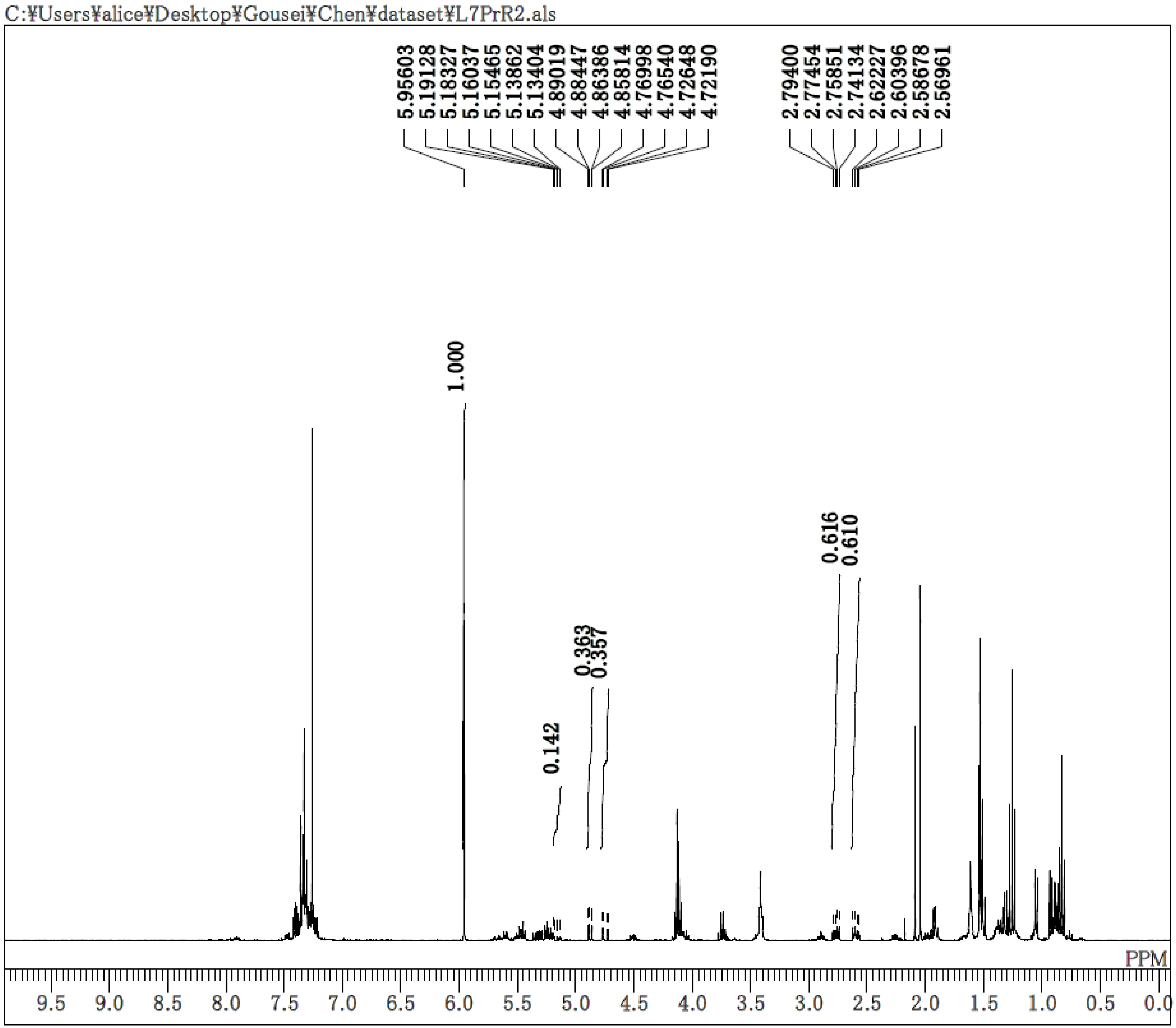


DFILE	L7PrR1.als
COMNT	
DATIM	2018-11-11 23:09:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L7/1Pr, *R*

(1st trial)

82%, 4.6/1 dr, 1/1.5 b/l



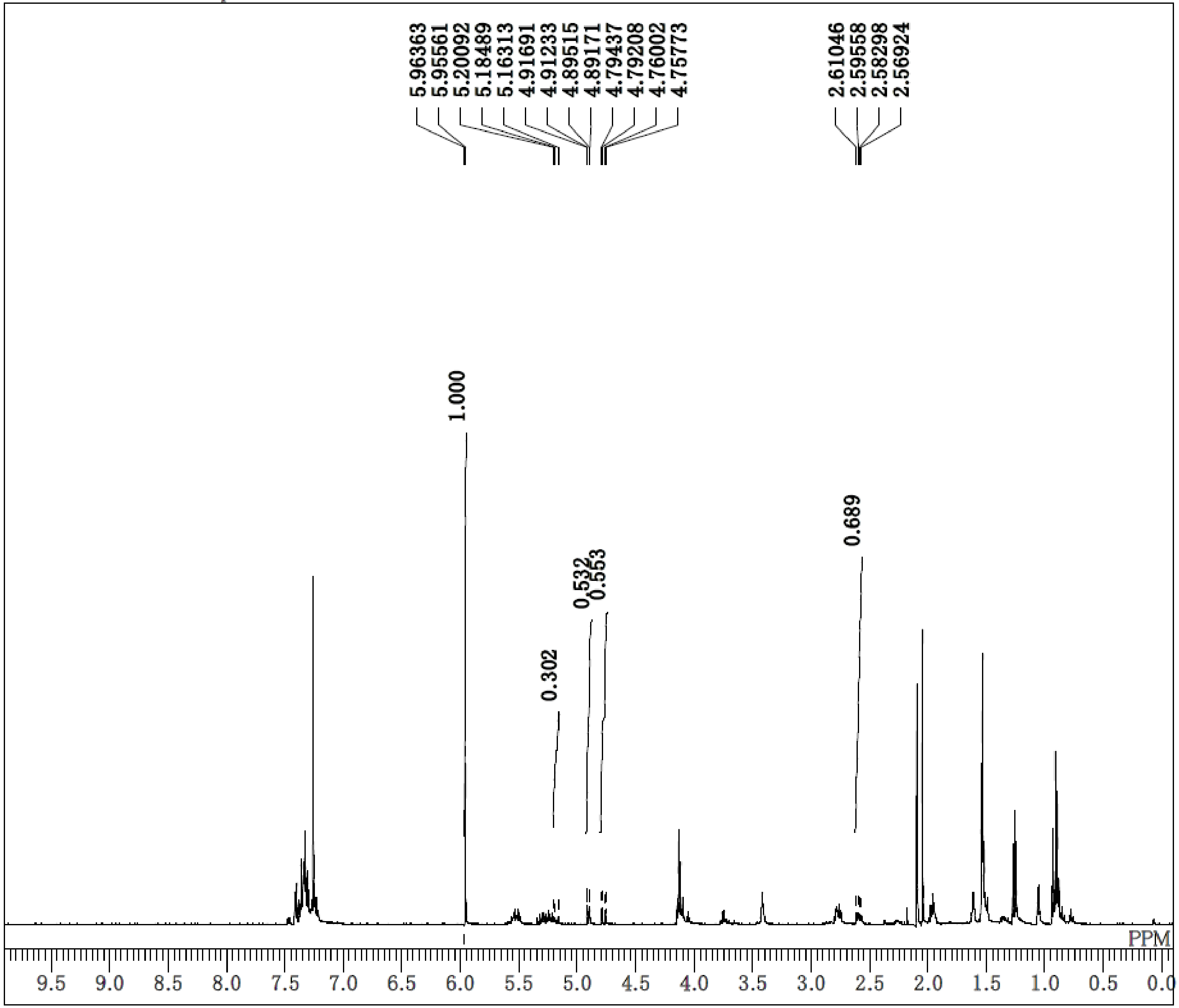
DFILE	L7PrR2.als
COMNT	
DATIM	29-08-2020 23:08:22
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L7/1Pr, ***R***

(2nd trial)

66%, 5.1/1 dr, 1/1.4 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L8EtR1.als

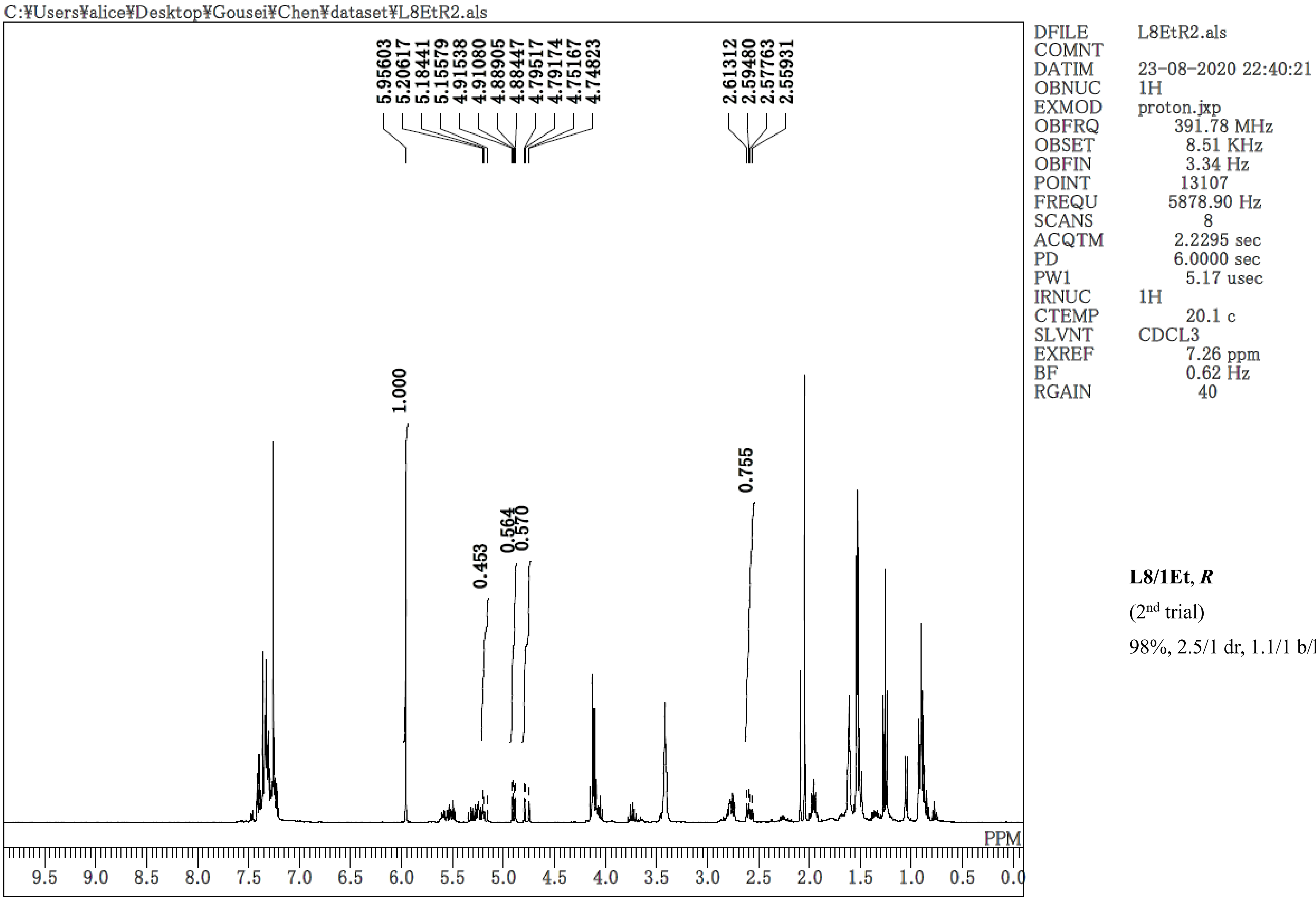


DFILE	L8EtR1.als
COMNT	
DATIM	2018-11-23 13:32:36
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L8/1Et, ***R***

(1st trial)

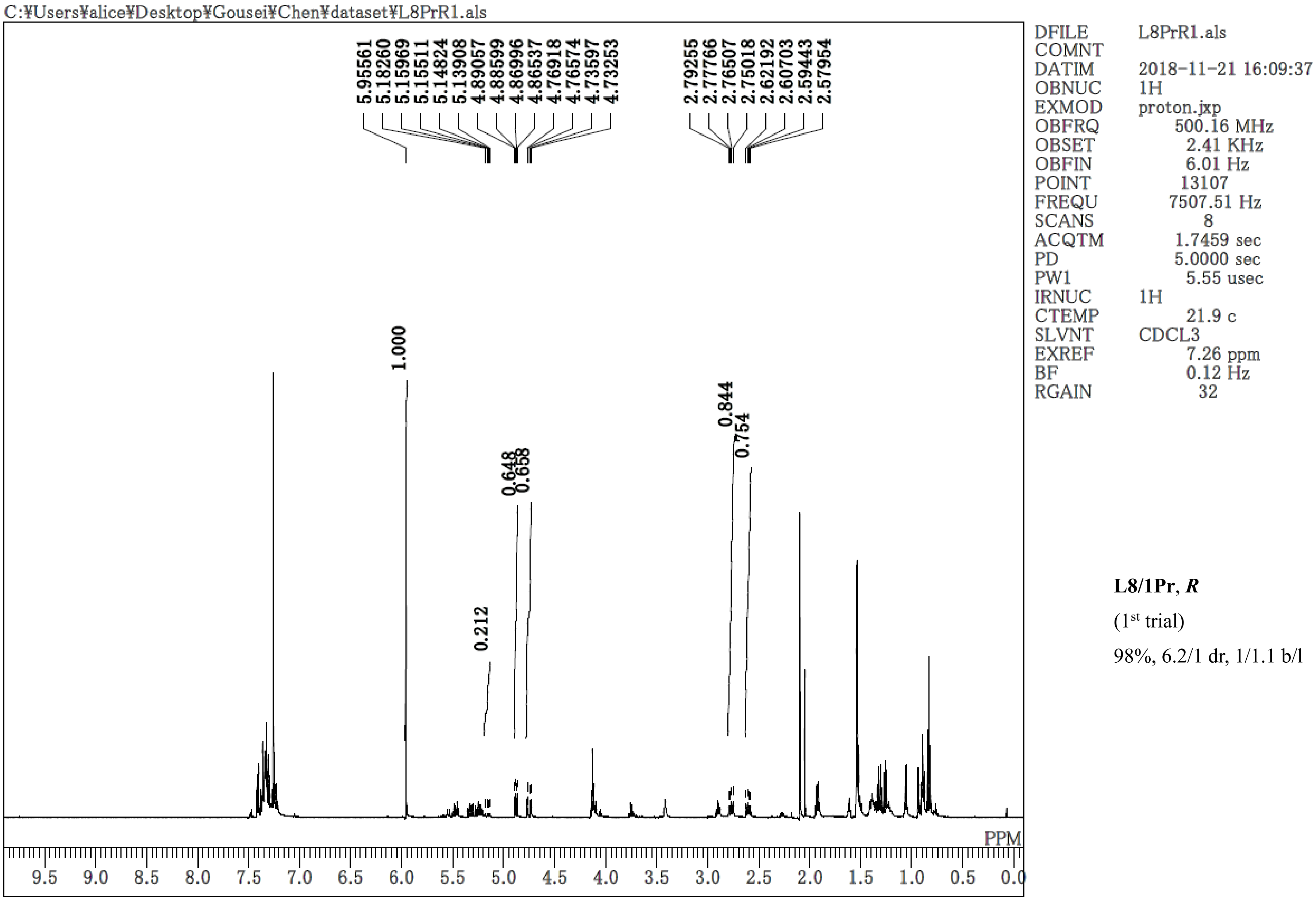
87%, 3.6/1 dr, 1.0/1 b/l



L8/1Et, ***R***

(2nd trial)

98%, 2.5/1 dr, 1.1/1 b/l

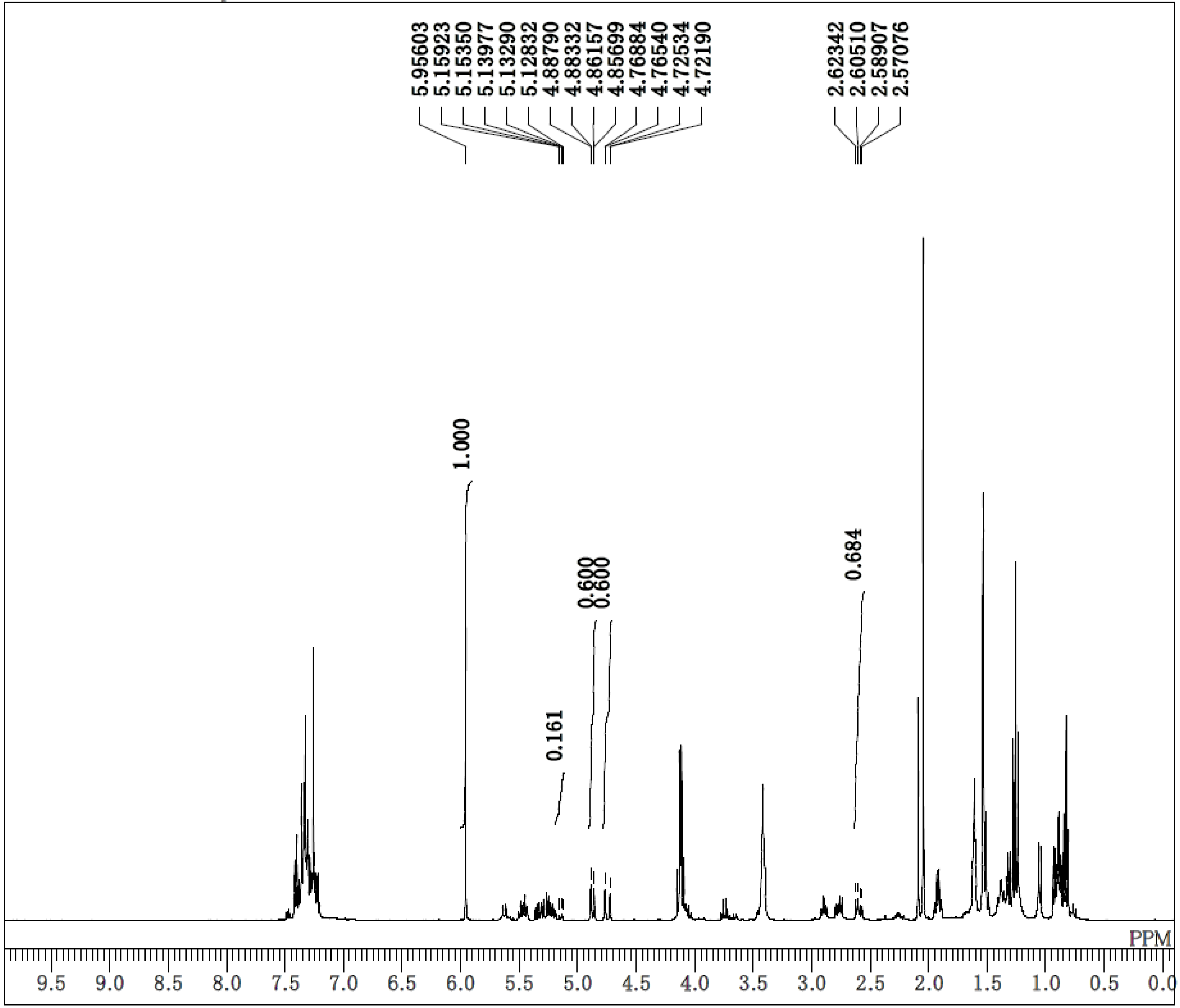


L8/1Pr, ***R***

(1st trial)

98%, 6.2/1 dr, 1/1.1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L8PrR2.als



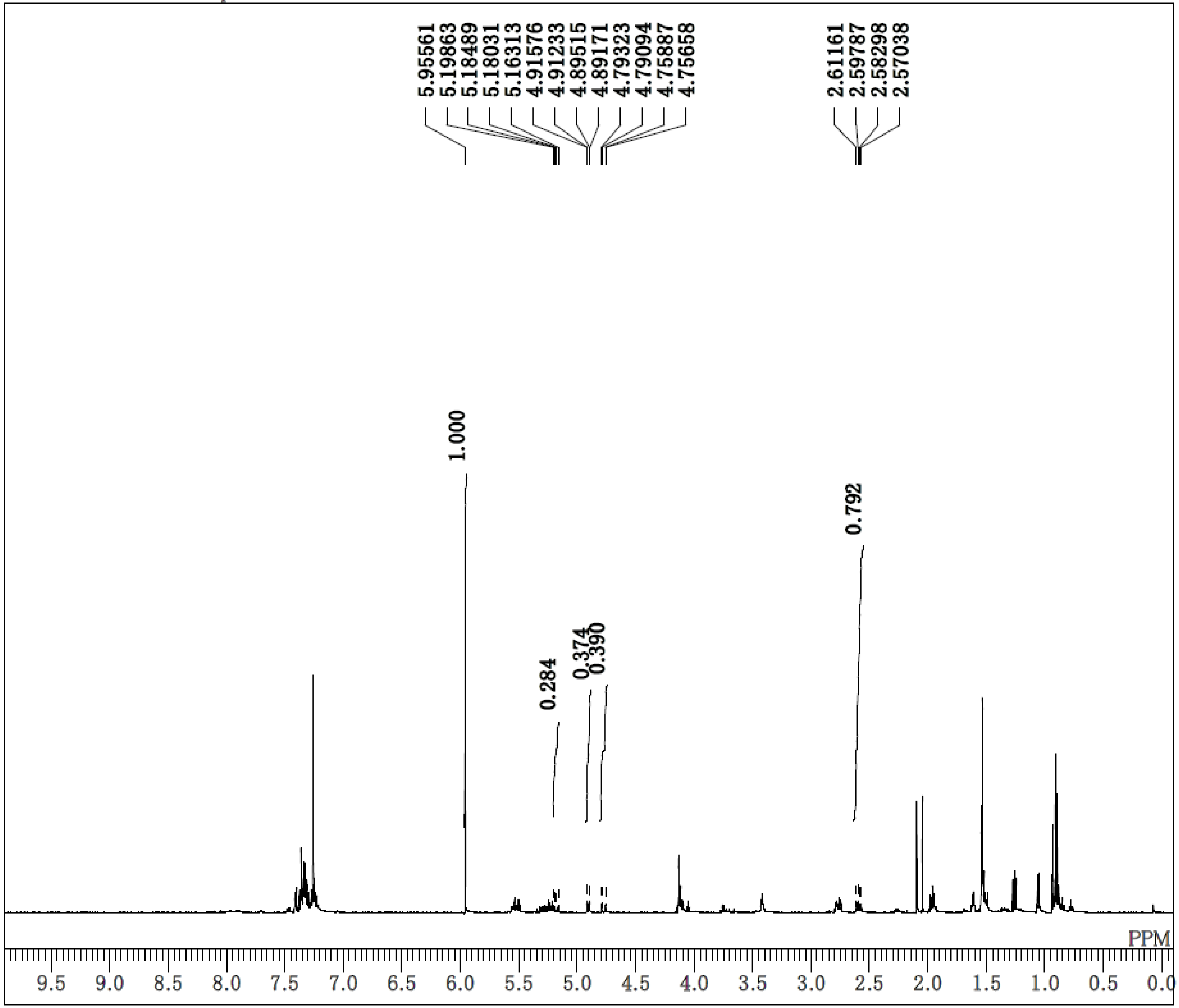
DFILE	L8PrR2.als
COMNT	
DATIM	23-08-2020 22:52:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.62 Hz
RGAIN	36

L8/1Pr, ***R***

(2nd trial)

86%, 7.5/1 dr, 1/1.0 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L9EtR1.als



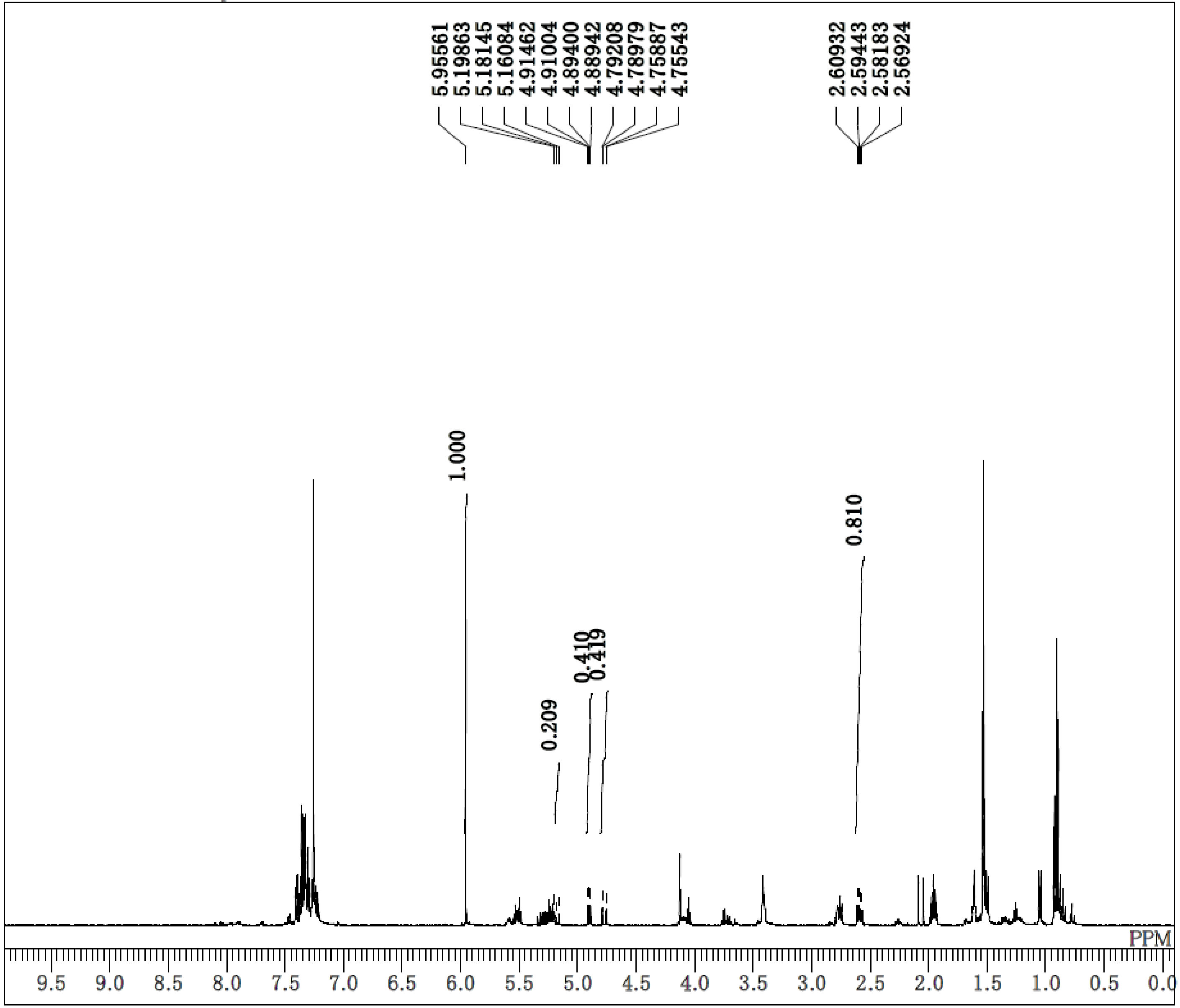
DFILE	L9EtR1.als
COMNT	
DATIM	2018-12-06 14:35:29
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L9/1Et, ***R***

(1st trial)

83%, 2.7/1 dr, 1/1.5 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L9EtR2.als

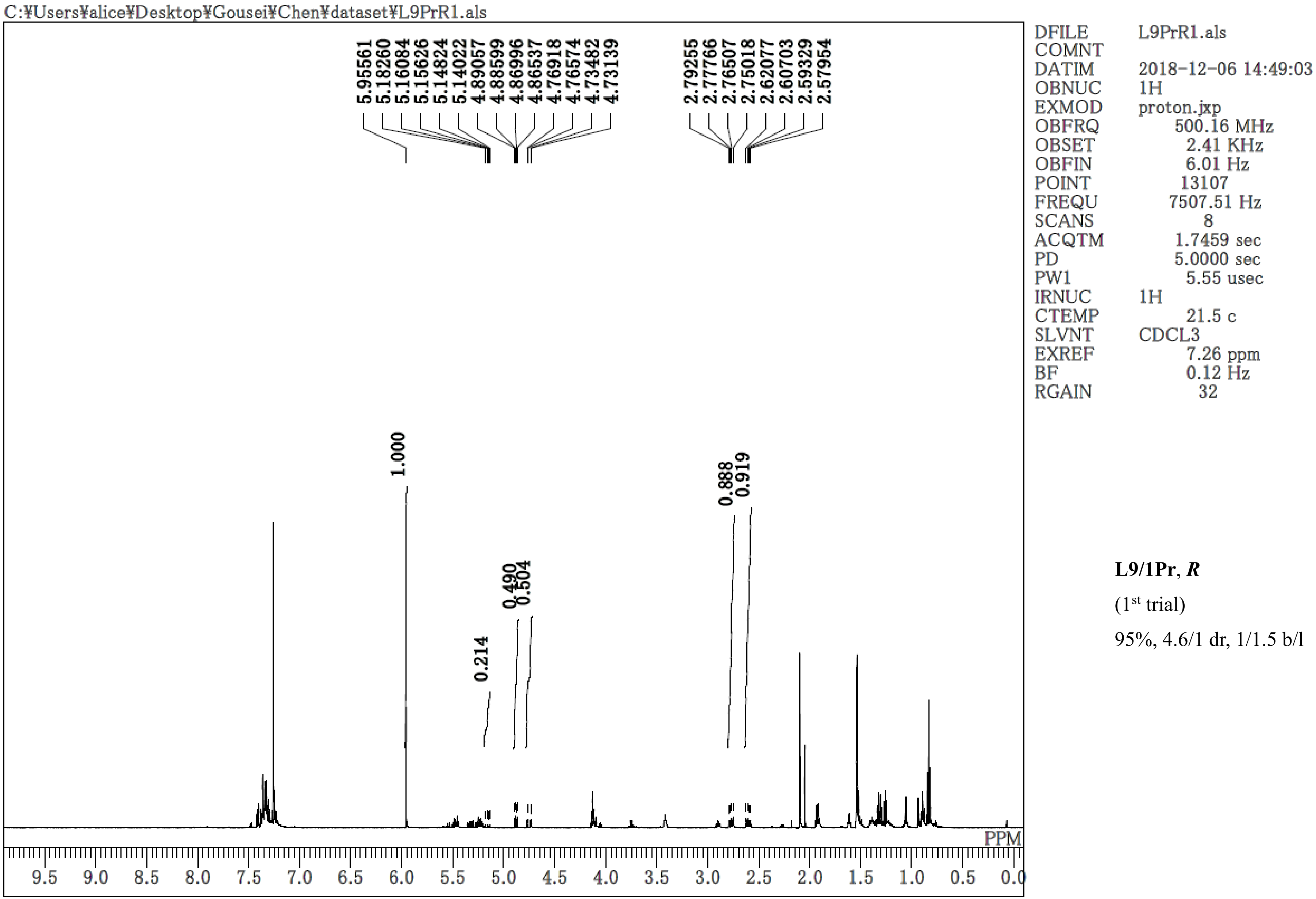


DFILE	L9EtR2.als
COMNT	
DATIM	2020-09-01 22:37:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L9/1Et, ***R***

(2nd trial)

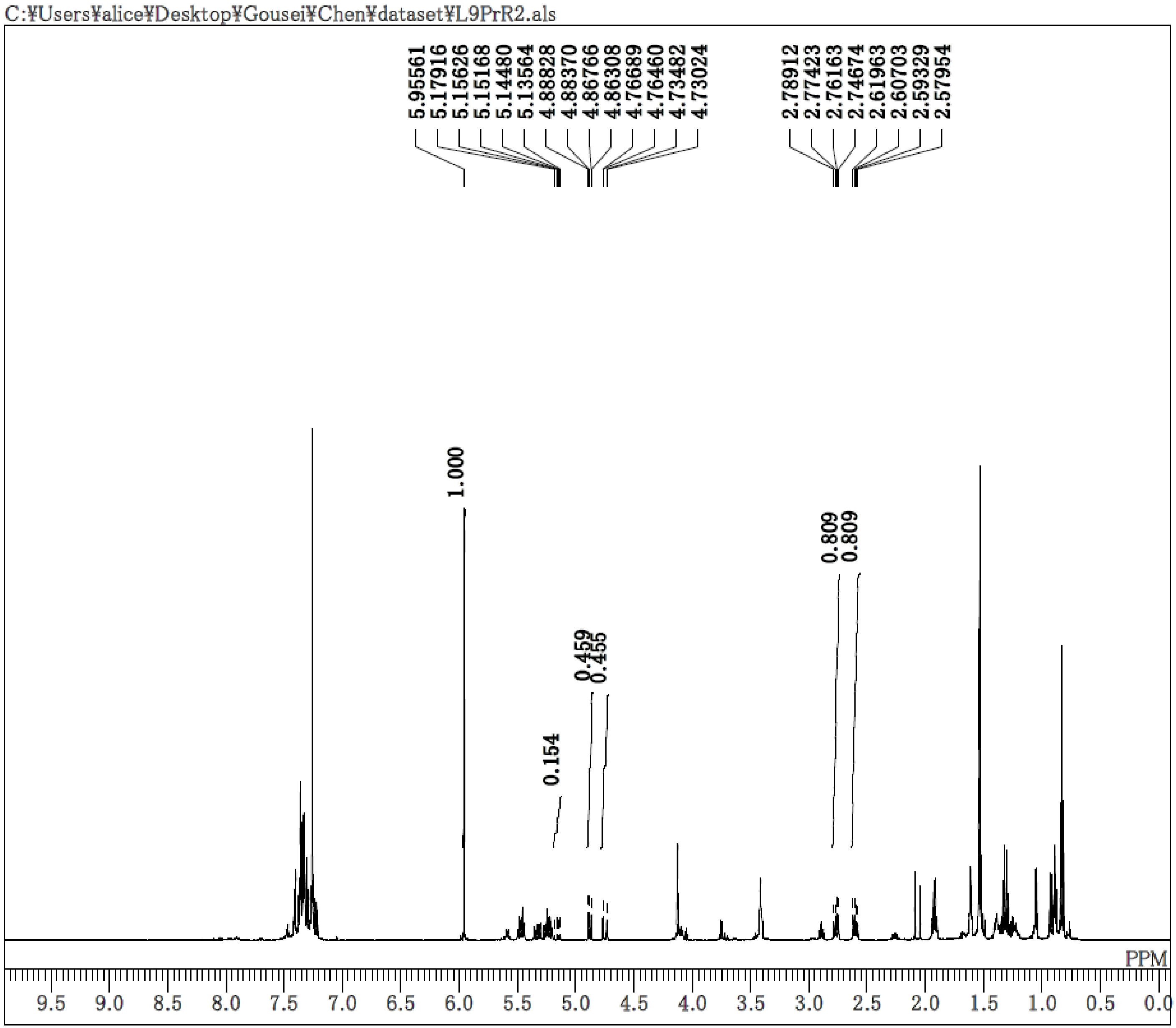
84%, 4.0/1 dr, 1/1.6 b/l



L9/1Pr, ***R***

(1st trial)

95%, 4.6/1 dr, 1/1.5 b/l



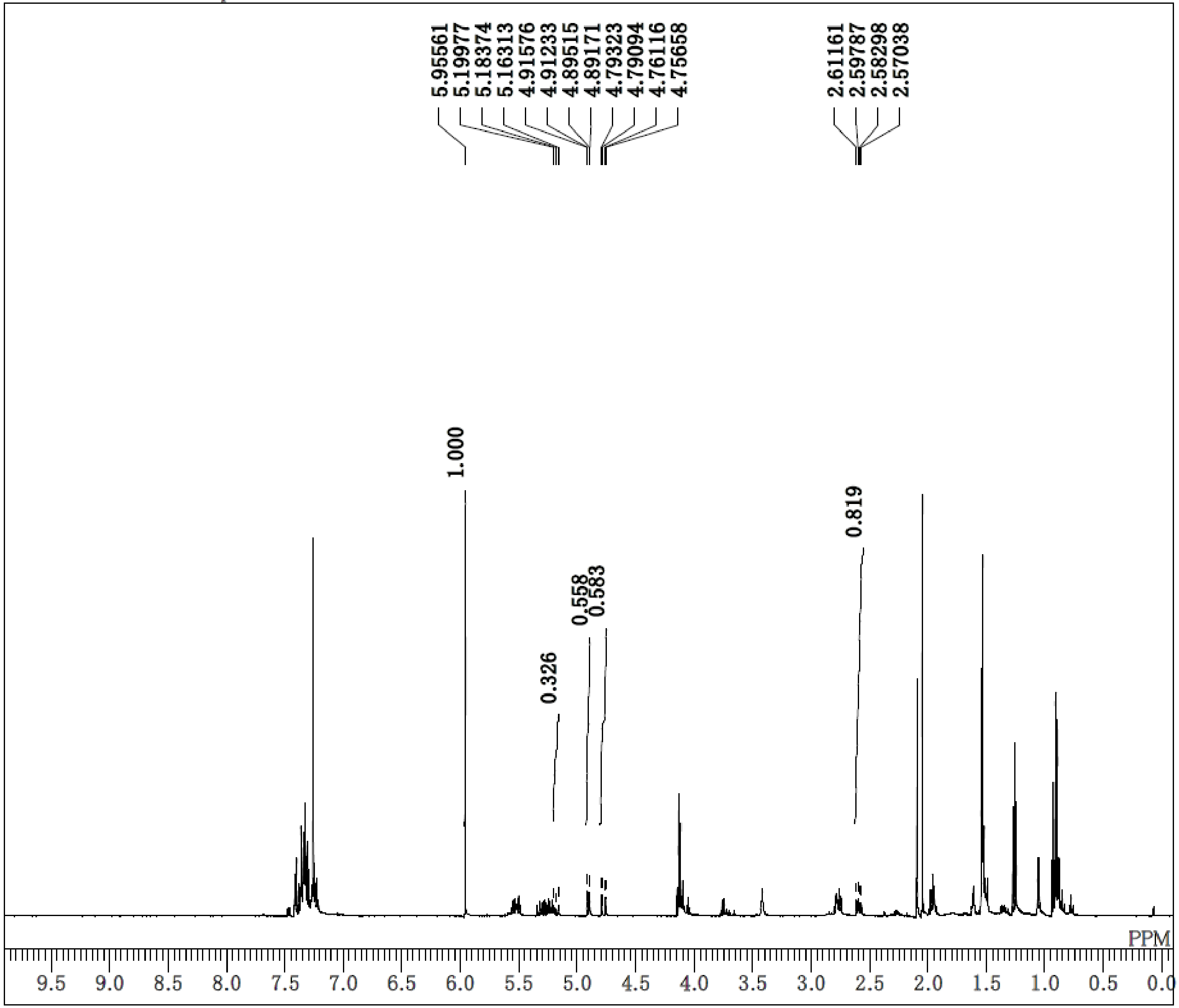
DFILE	L9PrR2.als
COMNT	
DATIM	2020-09-01 22:46:44
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L9/1Pr, ***R***

(2^{nd} trial)

85%, 5.9/1 dr, 1/1.5 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L10EtR1.als



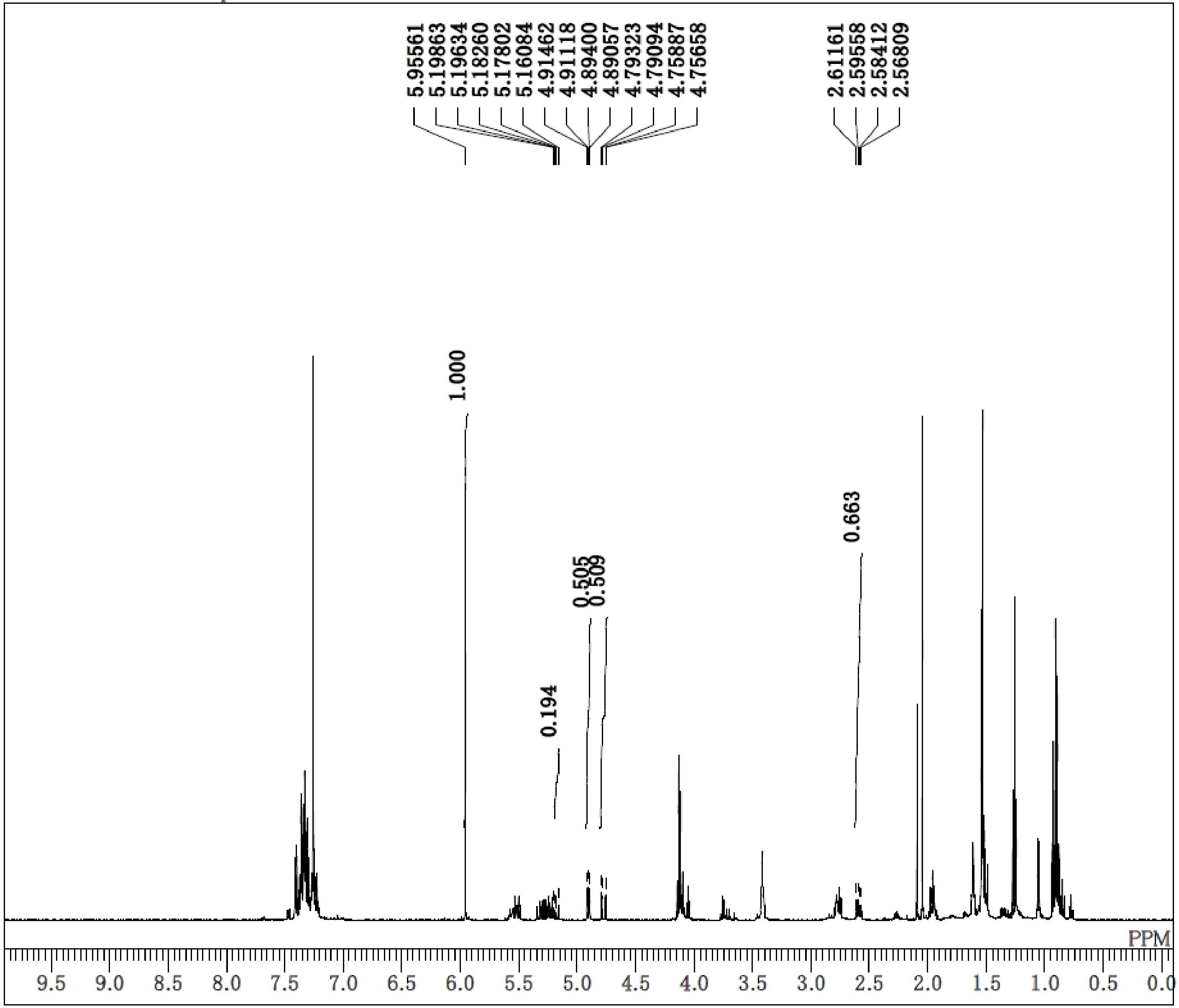
DFILE	L10EtR1.als
COMNT	
DATIM	2018-12-05 09:21:32
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L10/1Et, ***R***

(1st trial)

88%, 3.8/1 dr, 1/1.1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L10EtR2.als

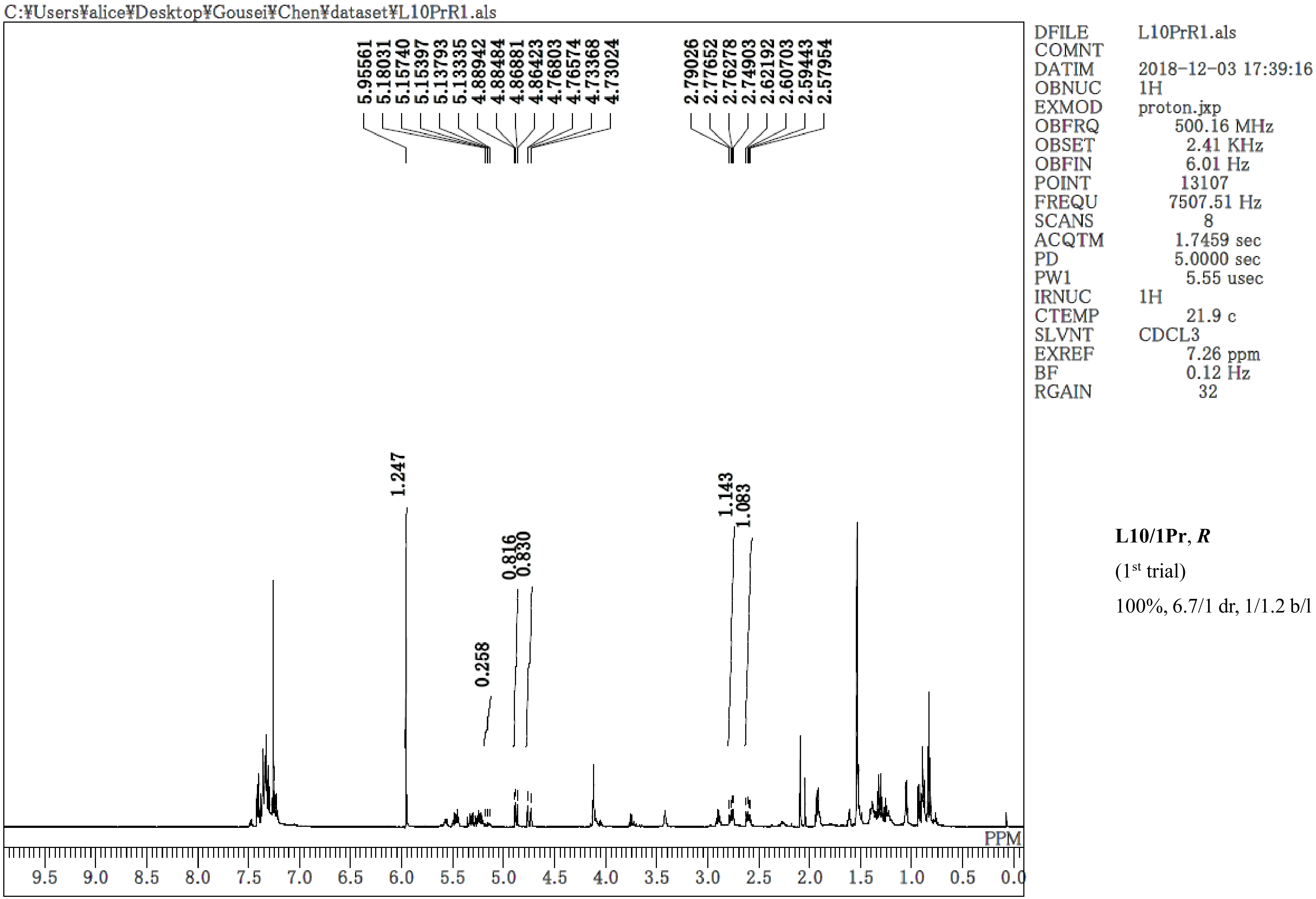


DFILE	L10EtR2.als
COMNT	
DATIM	2020-09-01 23:22:47
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L10/1Et, ***R***

(2nd trial)

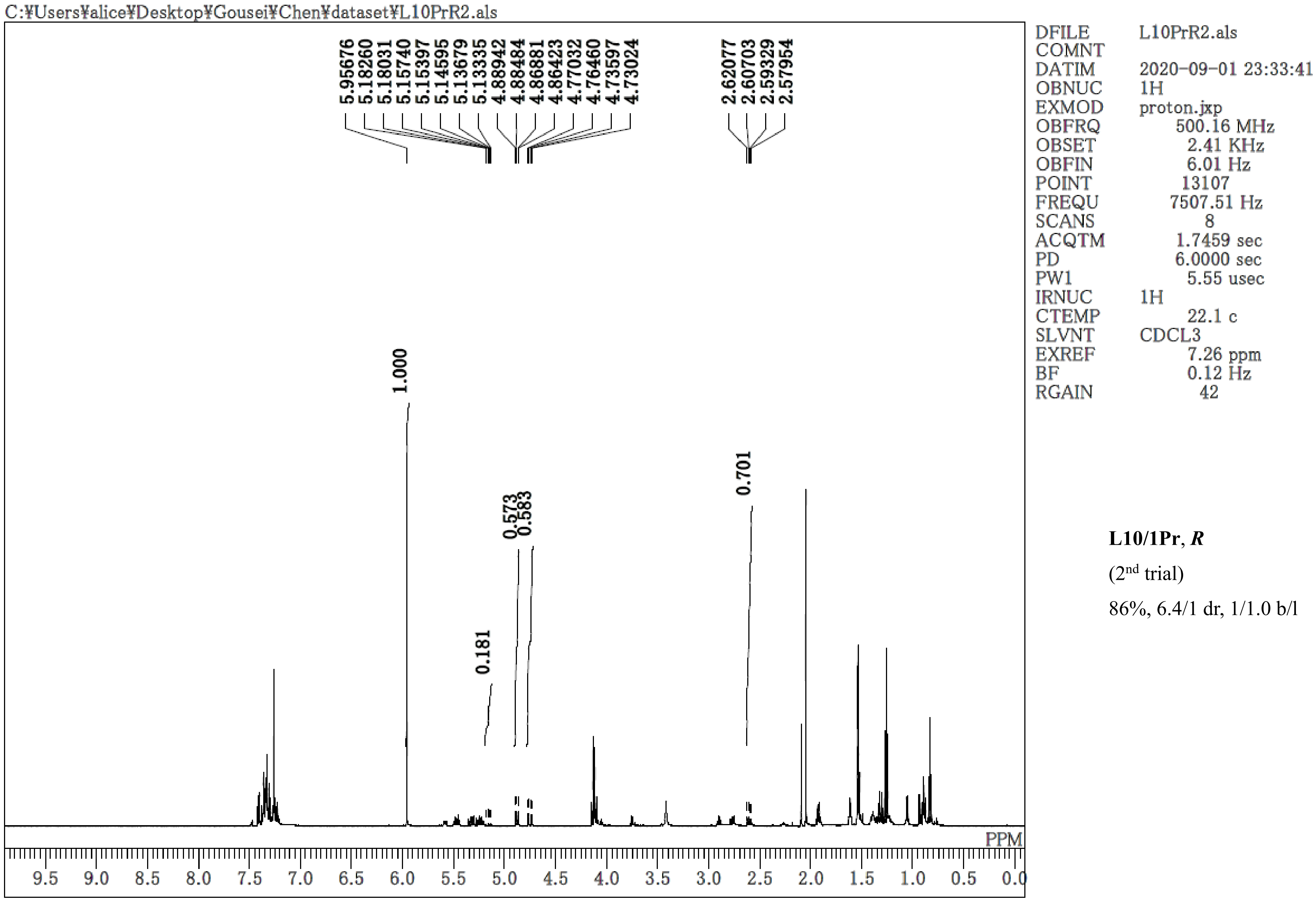
80%, 5.2/1 dr, 1/1.1 b/l



L10/1Pr, ***R***

(1st trial)

100%, 6.7/1 dr, 1/1.2 b/l

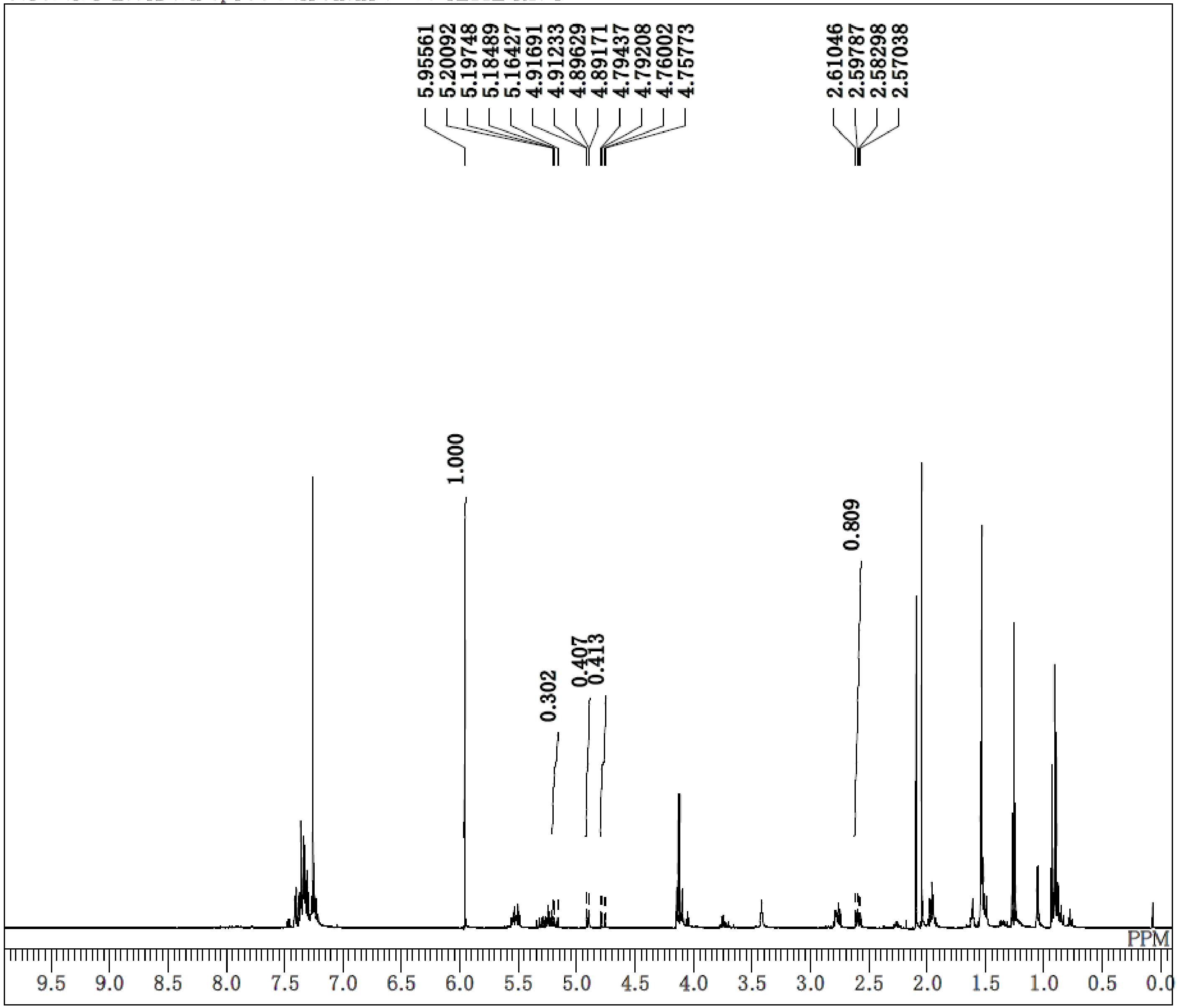


L10/1Pr, ***R***

(2nd trial)

86%, 6.4/1 dr, 1/1.0 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L11EtR1.als

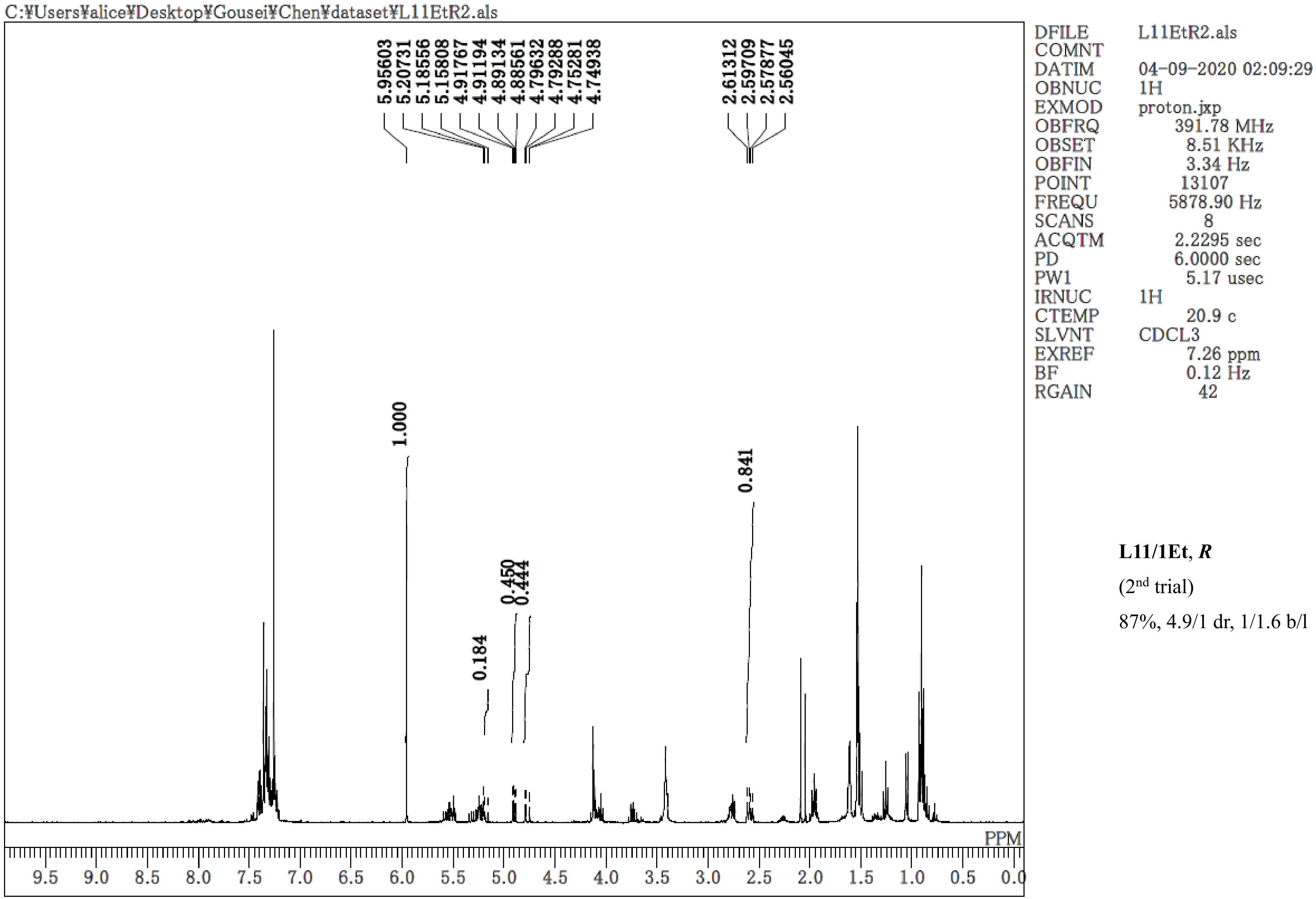


DFILE	L11EtR1.als
COMNT	
DATIM	2018-12-05 09:34:08
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L11/1Et, ***R***

(1st trial)

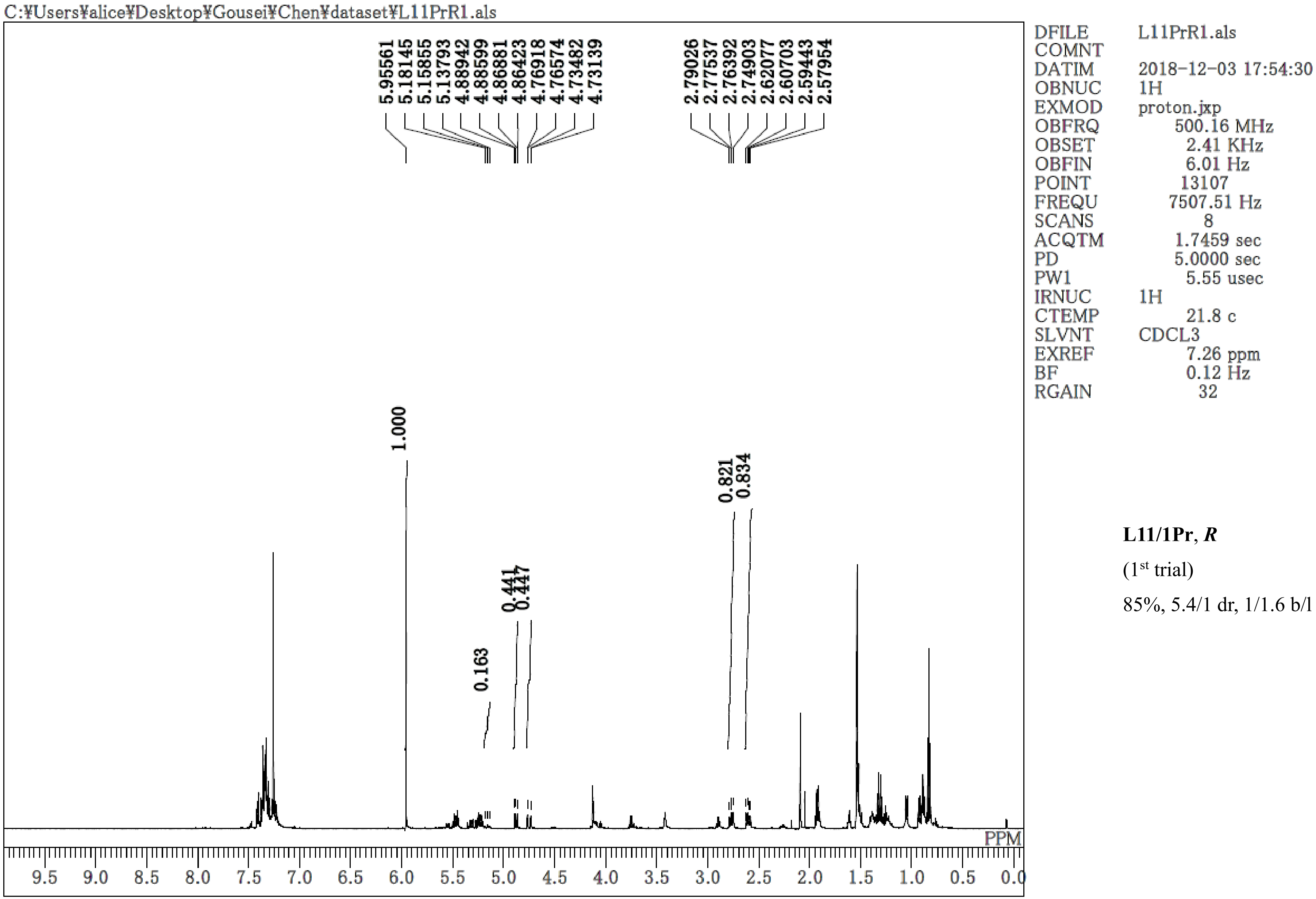
86%, 2.7/1 dr, 1/1.4 b/l



L11/1Et, ***R***

(2nd trial)

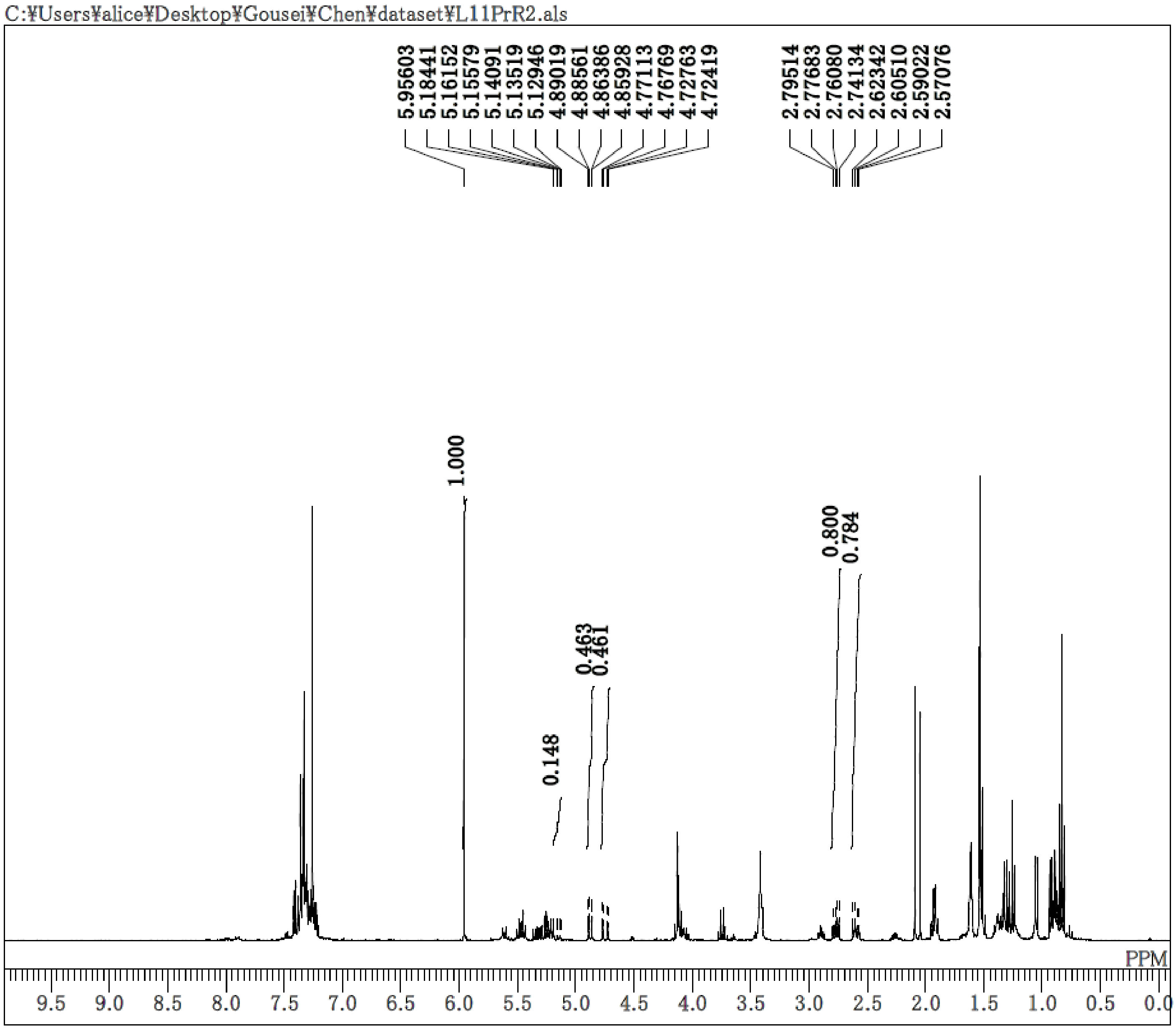
87%, 4.9/1 dr, 1/1.6 b/l



L11/1Pr, ***R***

(1st trial)

85%, 5.4/1 dr, 1/1.6 b/l

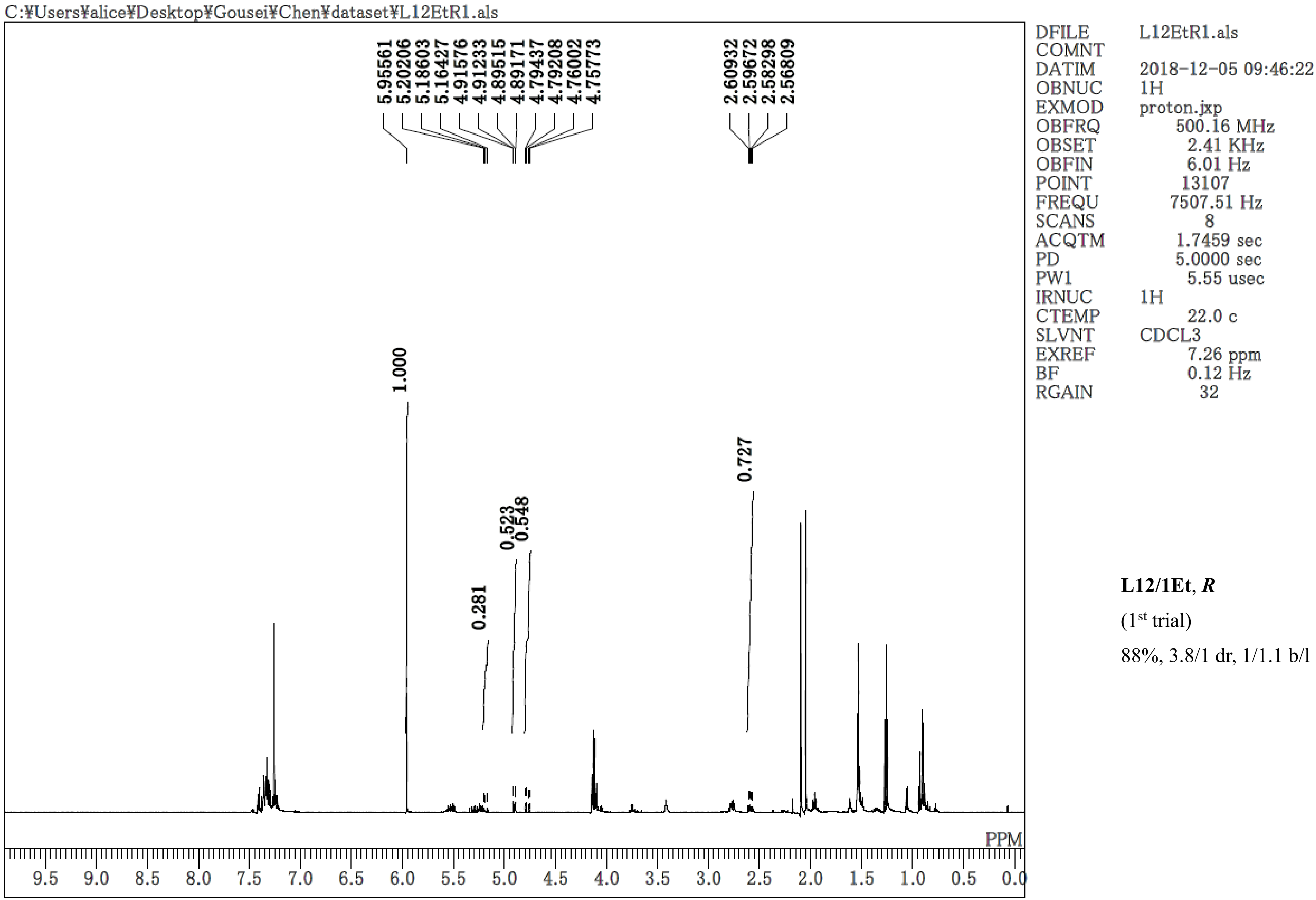


DFILE L11PrR2.als
COMNT
DATIM 04-09-2020 02:18:19
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

L11/1Pr, ***R***

(2nd trial)

84%, 6.2/1 dr, 1/1.5 b/l

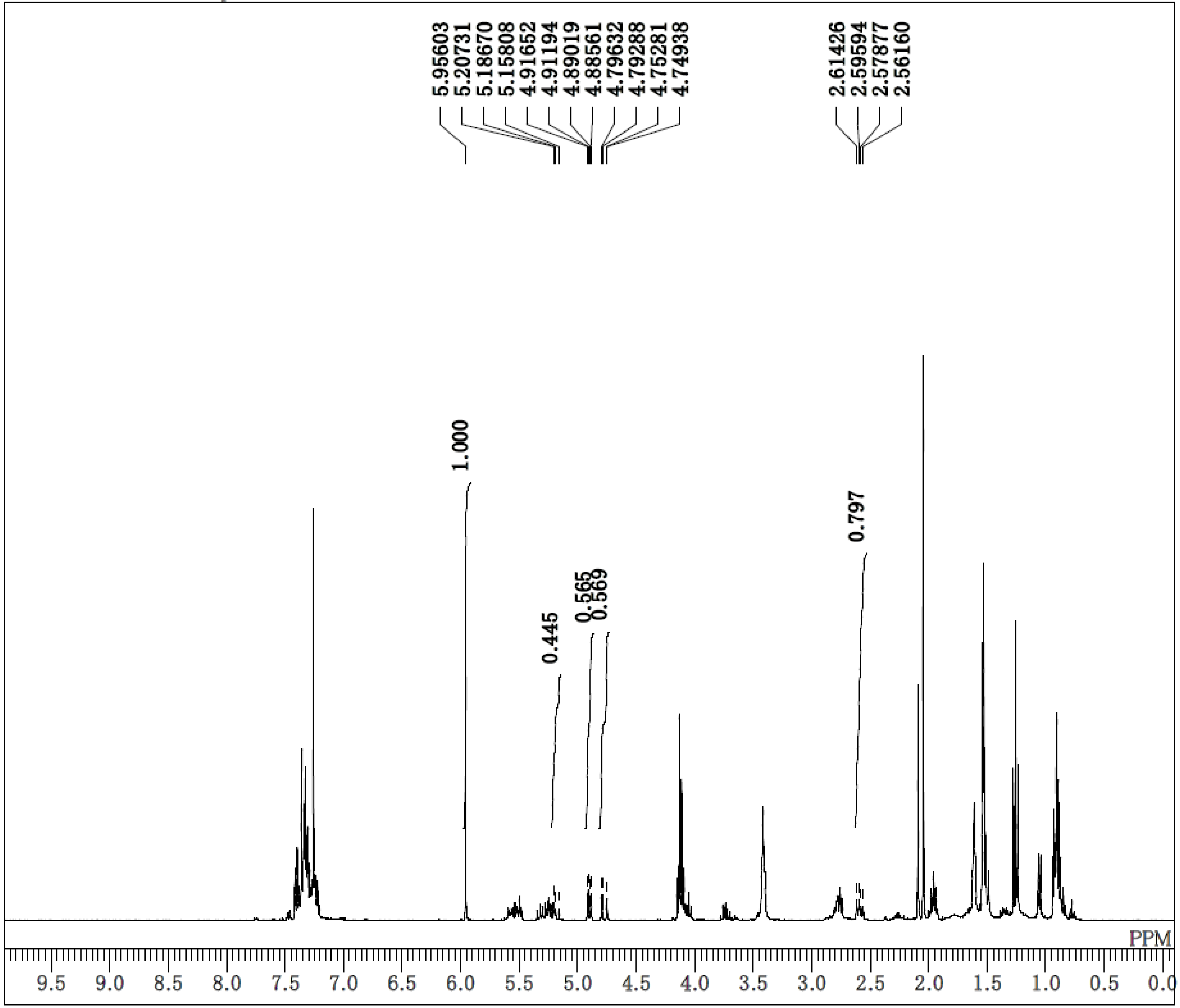


L12/1Et, ***R***

(1st trial)

88%, 3.8/1 dr, 1/1.1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L12EtR2.als

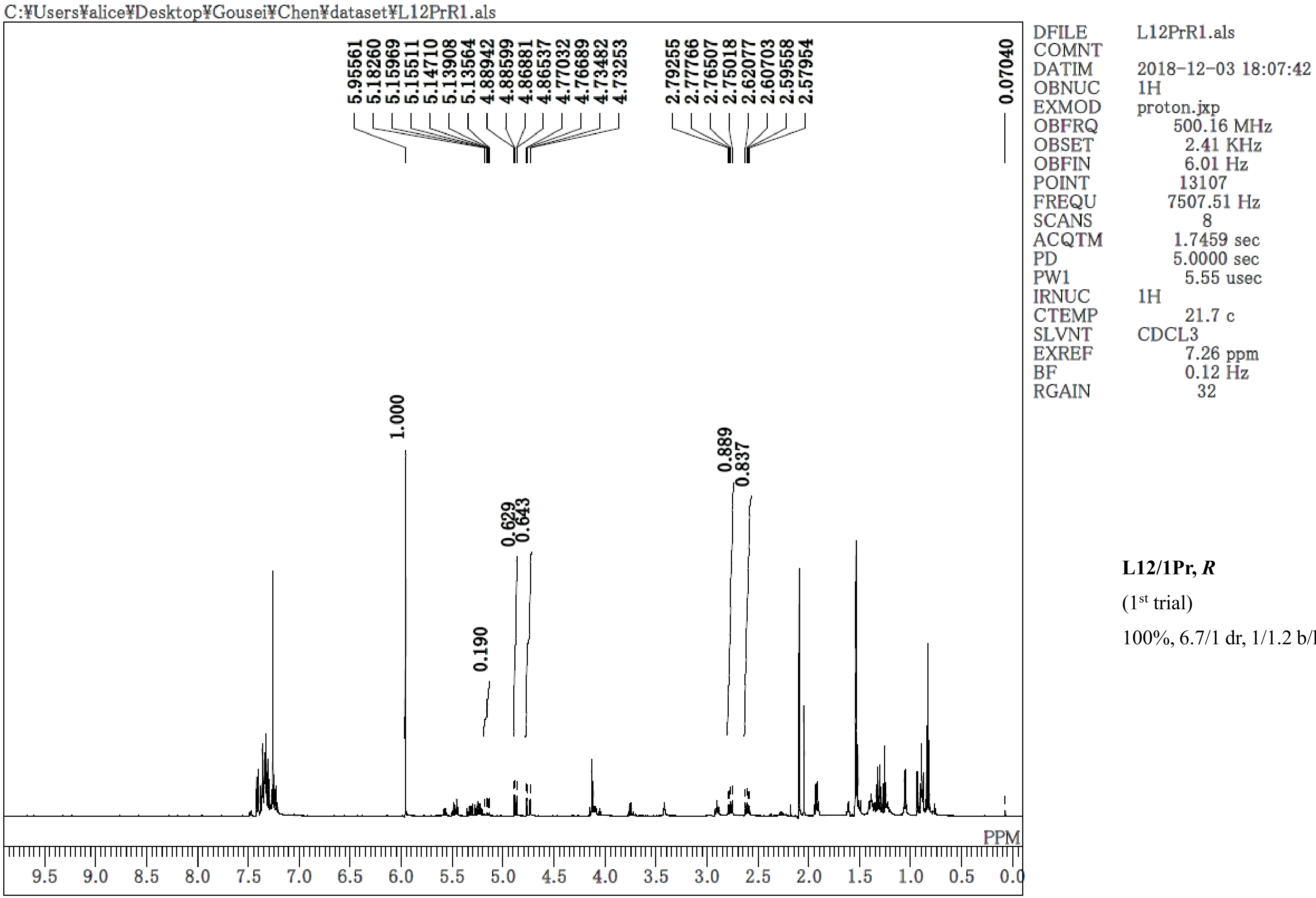


DFILE	L12EtR2.als
COMNT	
DATIM	22-08-2020 14:13:03
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.62 Hz
RGAIN	42

L12/1Et, ***R***

(2nd trial)

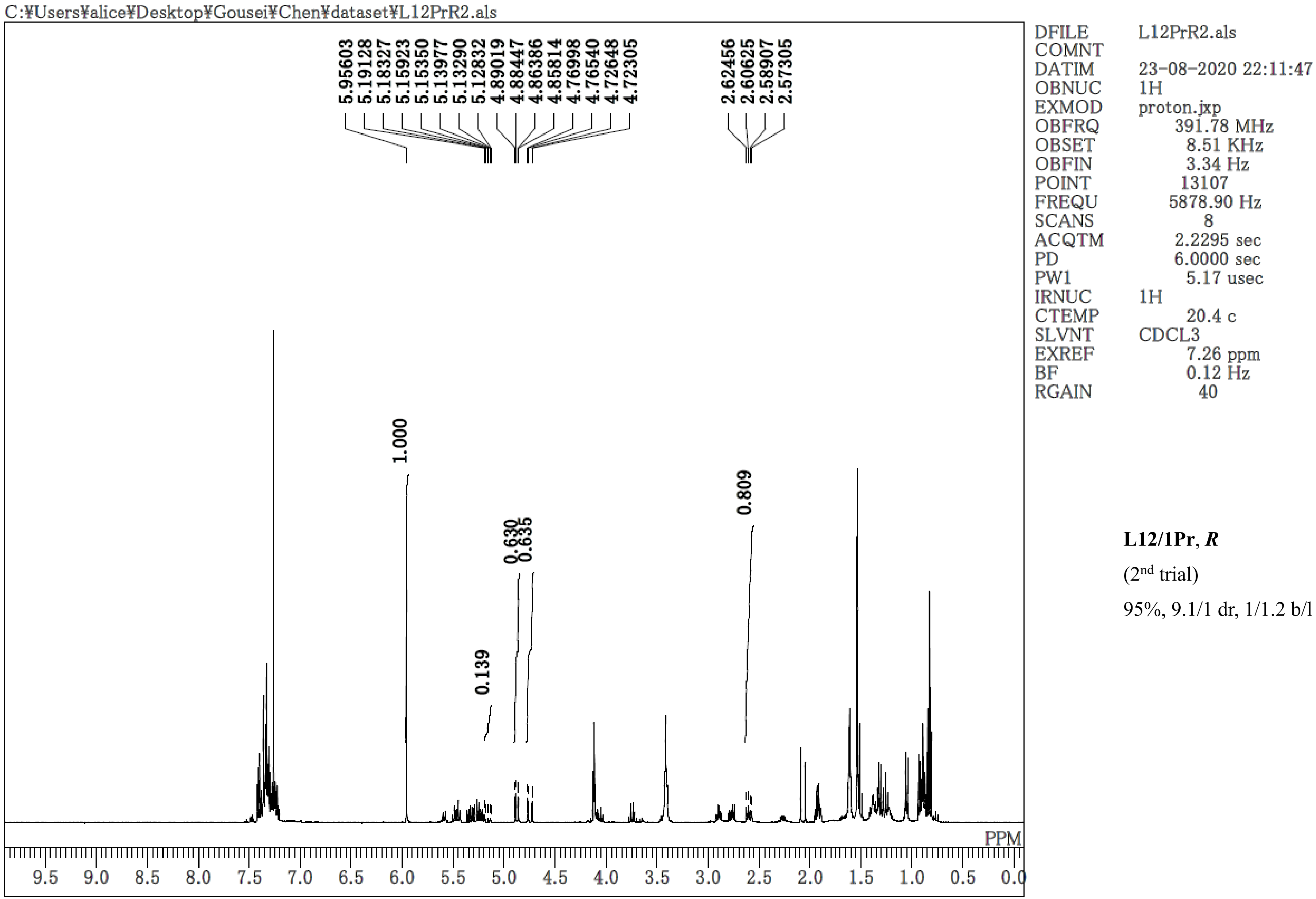
100%, 2.5/1 dr, 1.0/1 b/l



L12/1Pr, *R*

(1st trial)

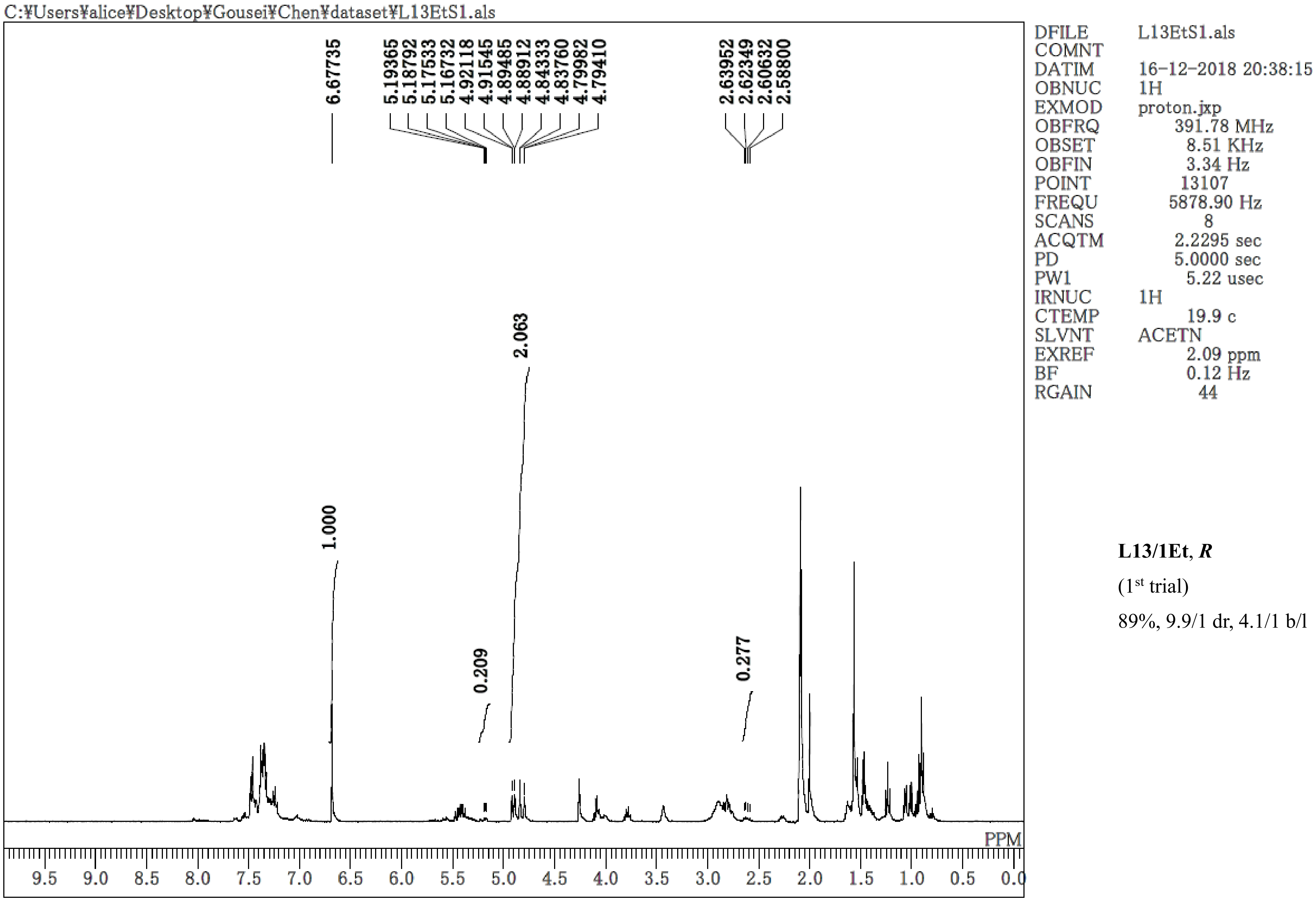
100%, 6.7/1 dr, 1/1.2 b/l



L12/1Pr, ***R***

(2nd trial)

95%, 9.1/1 dr, 1/1.2 b/l

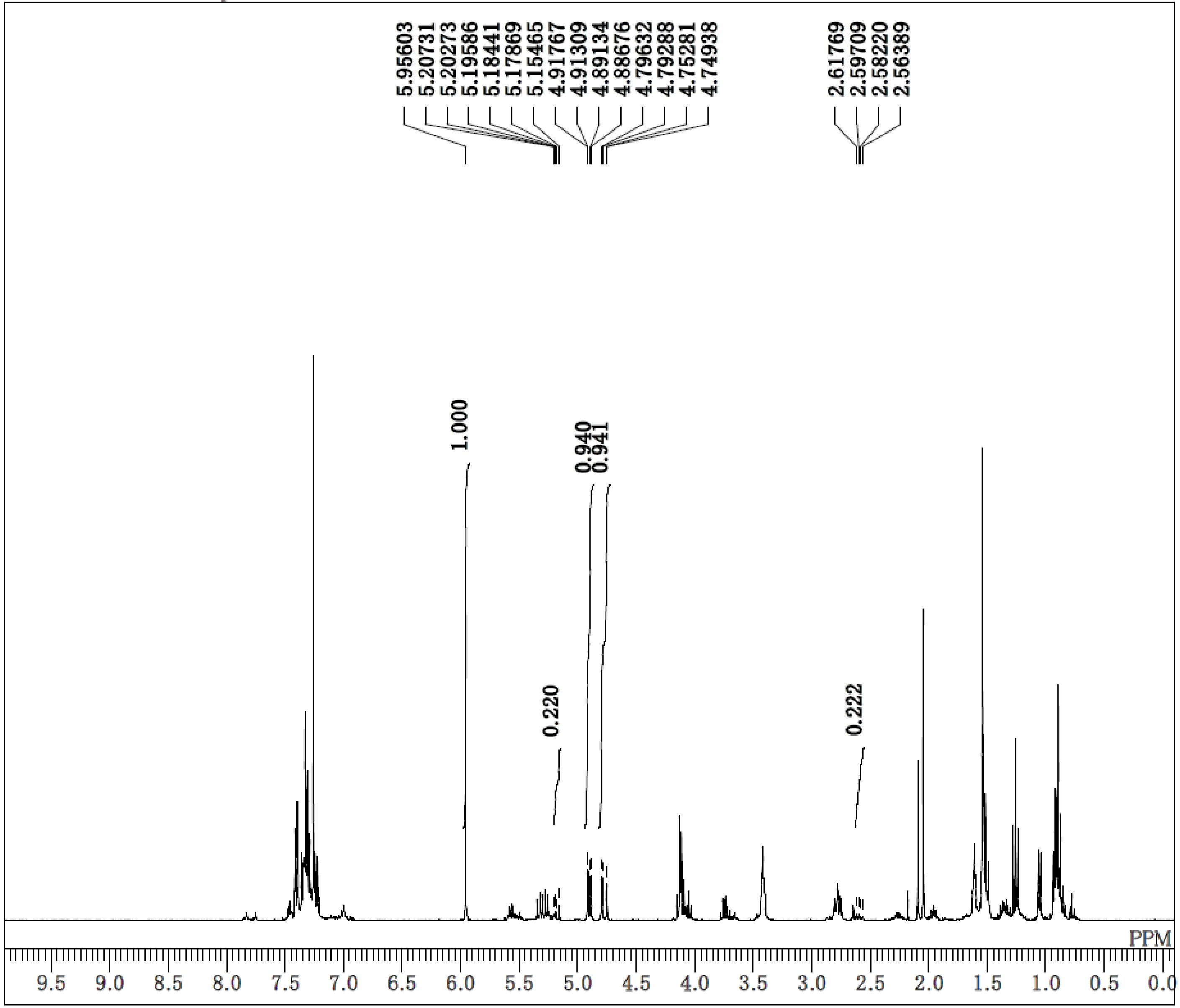


L13/1Et, ***R***

(1st trial)

89%, 9.9/1 dr, 4.1/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L13EtR2.als

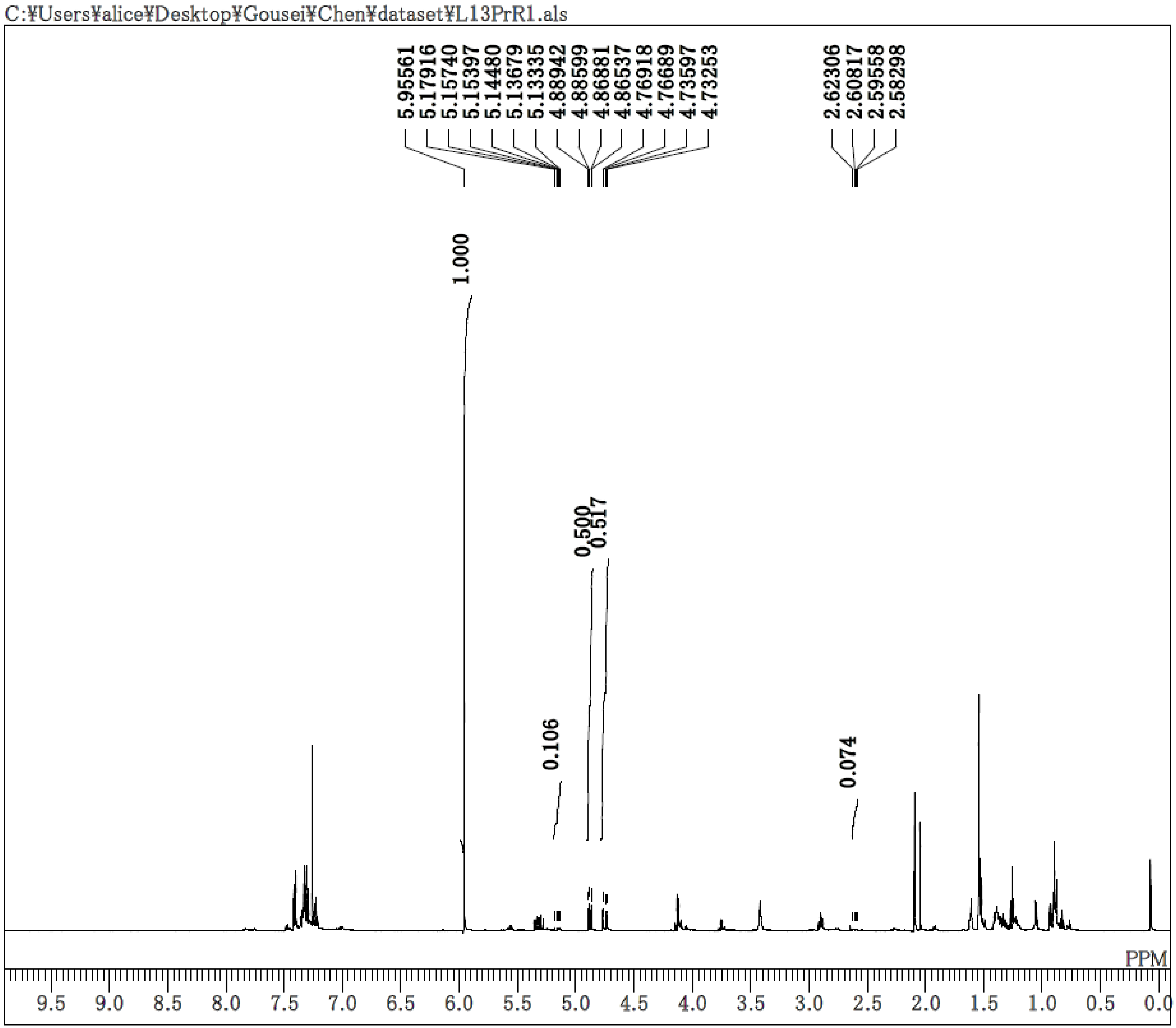


DFILE	L13EtR2.als
COMNT	
DATIM	26-08-2020 23:08:20
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.42 Hz
RGAIN	44

L13/1Et, ***R***

(2nd trial)

80%, 8.5/1 dr, 4.7/1 b/l



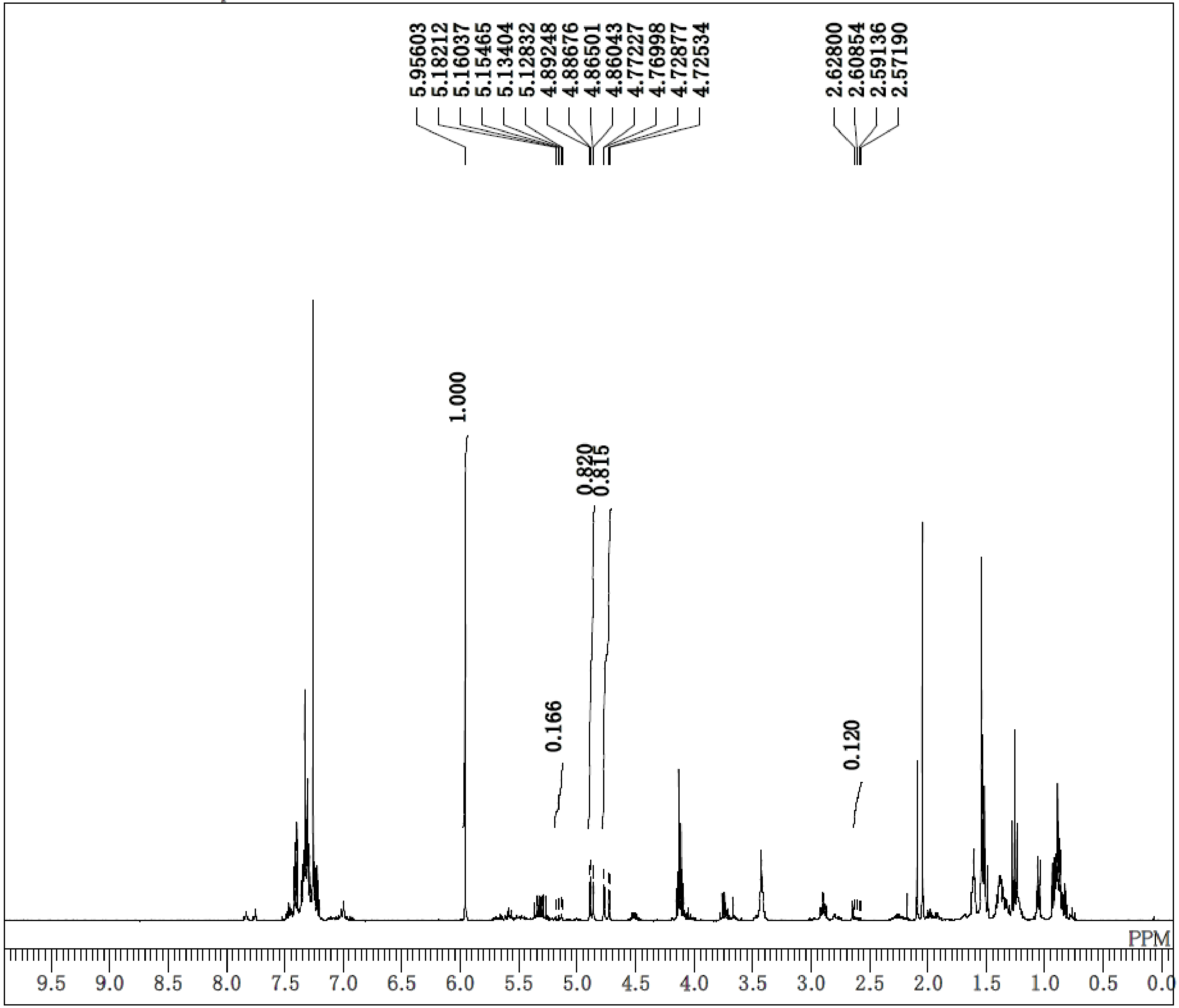
DFILE	L13PrR1.als
COMNT	
DATIM	2018-04-27 20:08:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	17
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30

L13/1Pr, ***R***

(1st trial)

81%, 10.0/1 dr, 9.9/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L13PrR2.als

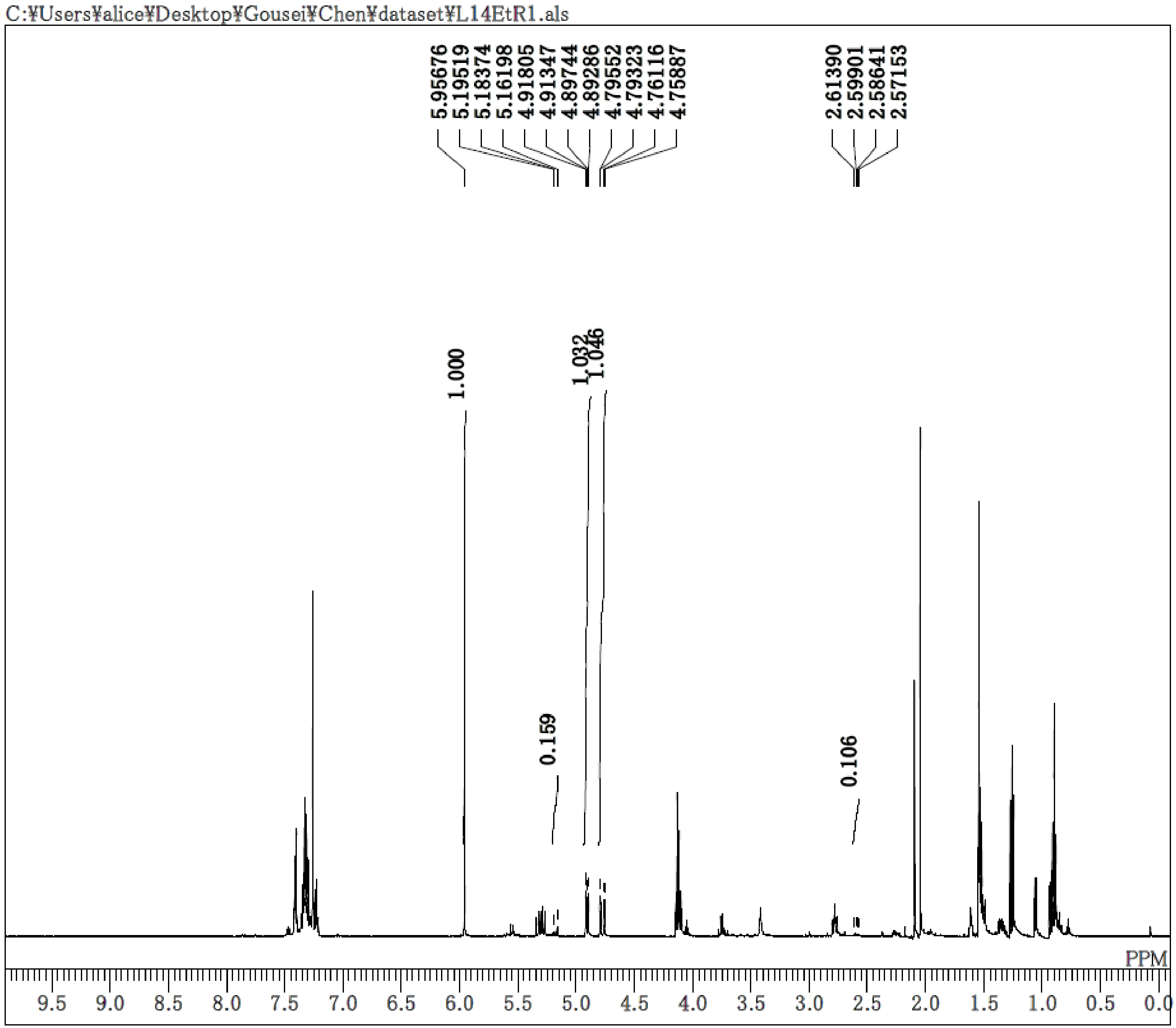


DFILE	L13PrR2.als
COMNT	
DATIM	26-08-2020 23:16:22
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.42 Hz
RGAIN	44

L13/1Pr, ***R***

(2^{nd} trial)

64%, 9.8/1 dr, 7.5/1 b/l

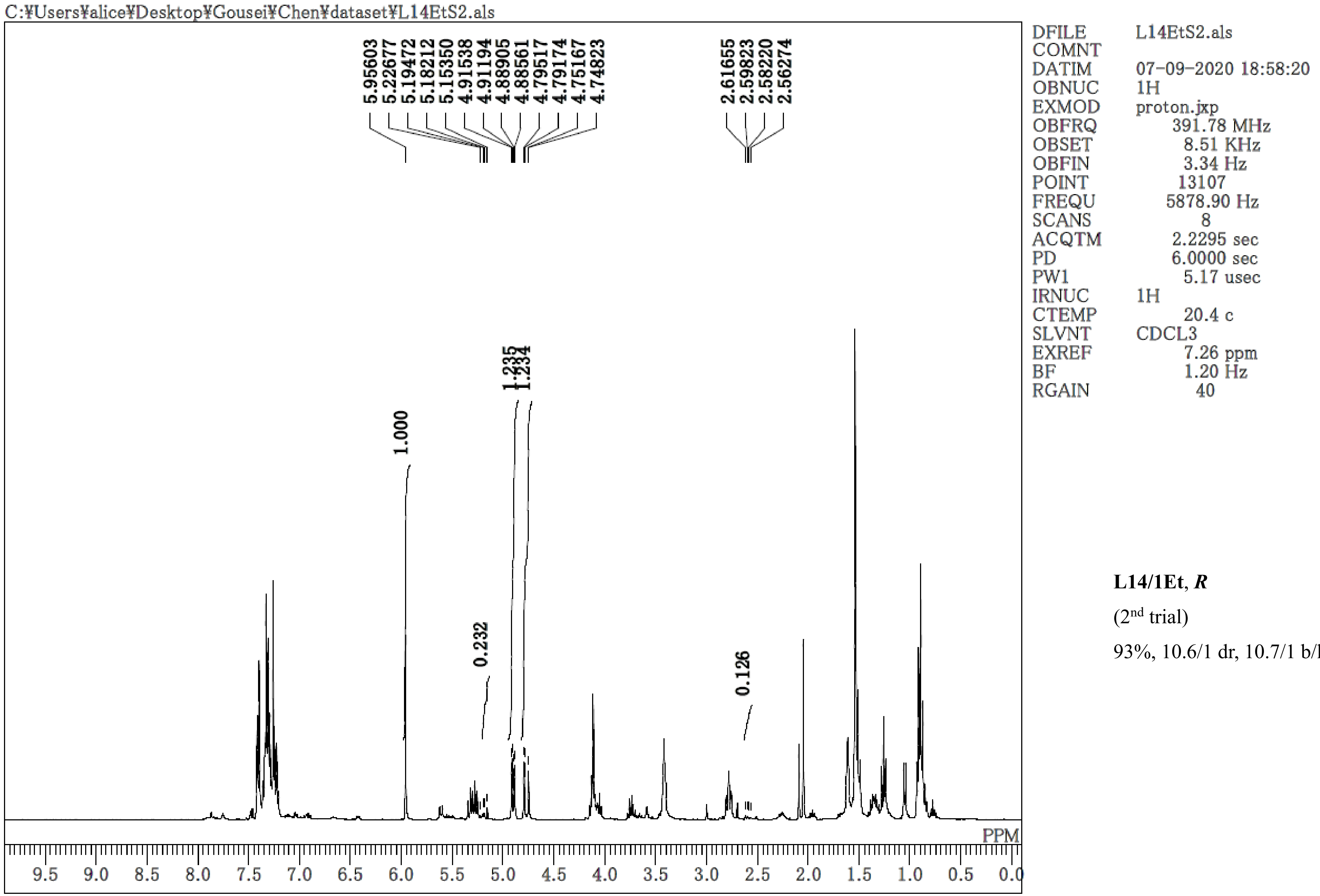


DFILE	L14EtR1.als
COMNT	
DATIM	2019-09-19 14:07:33
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L14/1Et, ***R***

(1st trial)

77%, 13.1/1 dr, 11.0/1 b/l

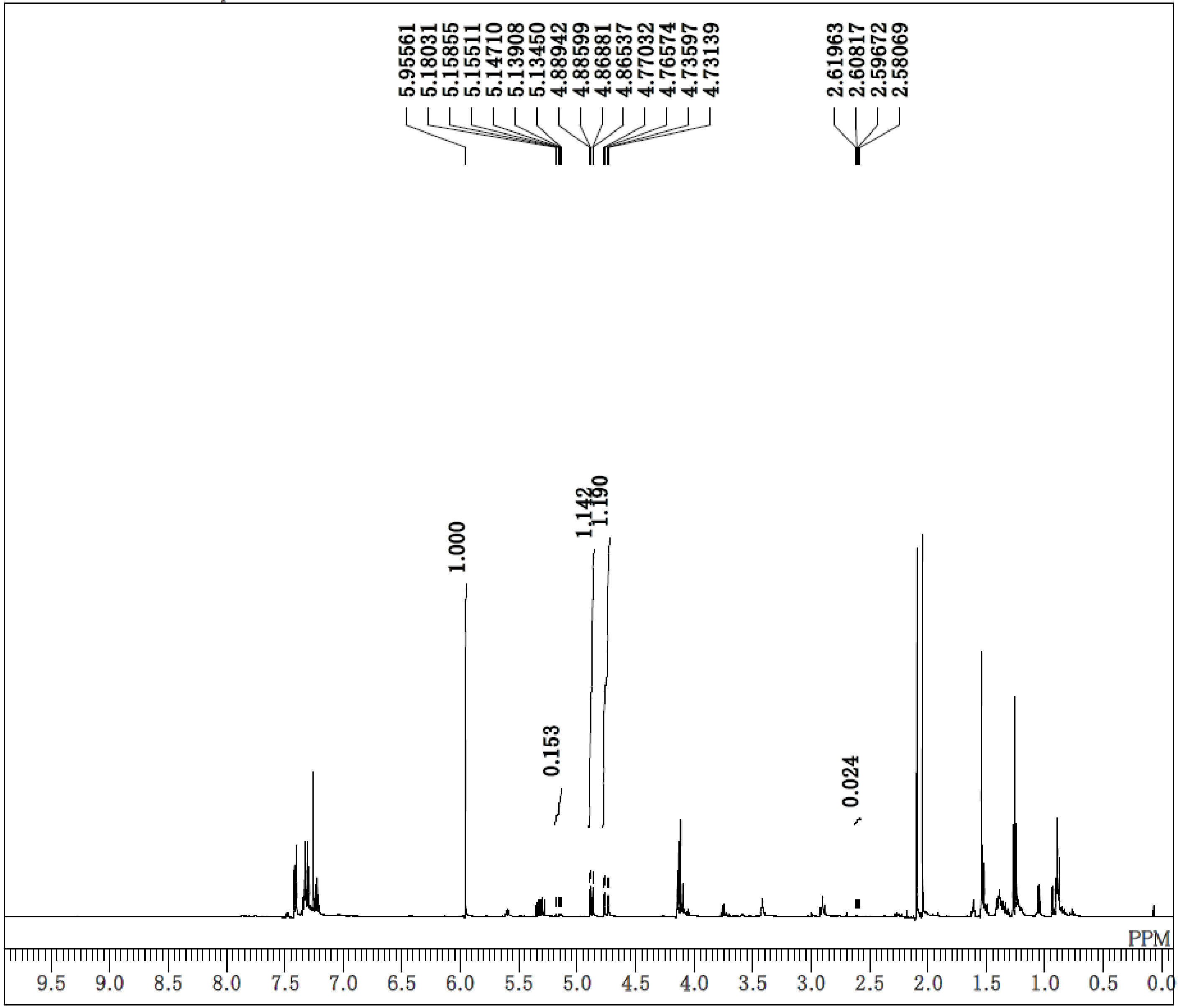


L14/1Et, ***R***

(2nd trial)

93%, 10.6/1 dr, 10.7/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L14PrR1.als



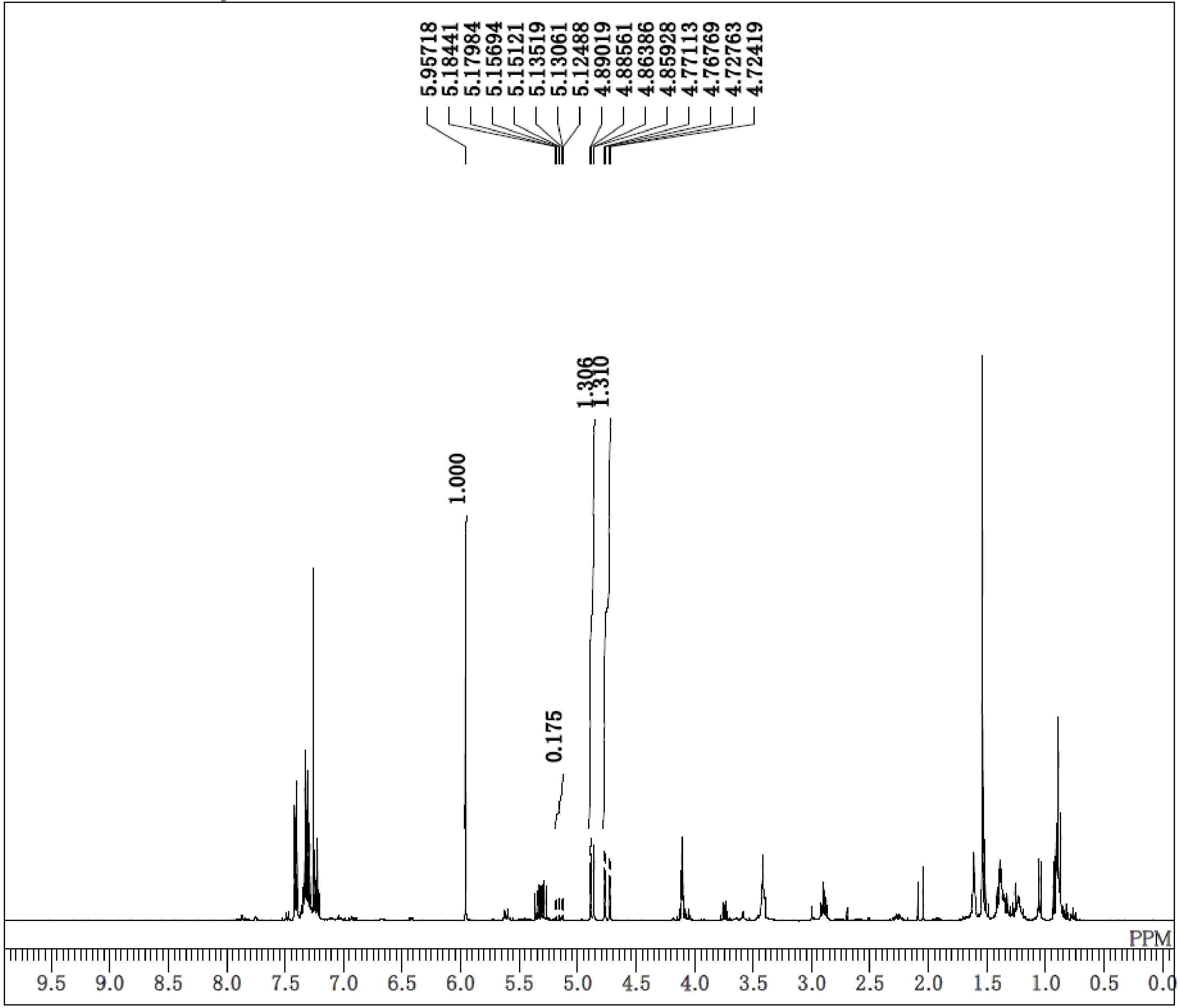
DFILE	L14PrR1.als
COMNT	
DATIM	2019-09-19 14:23:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L14/1Pr, ***R***

(1st trial)

80%, 15.2/1 dr, >50/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L14PrS2.als

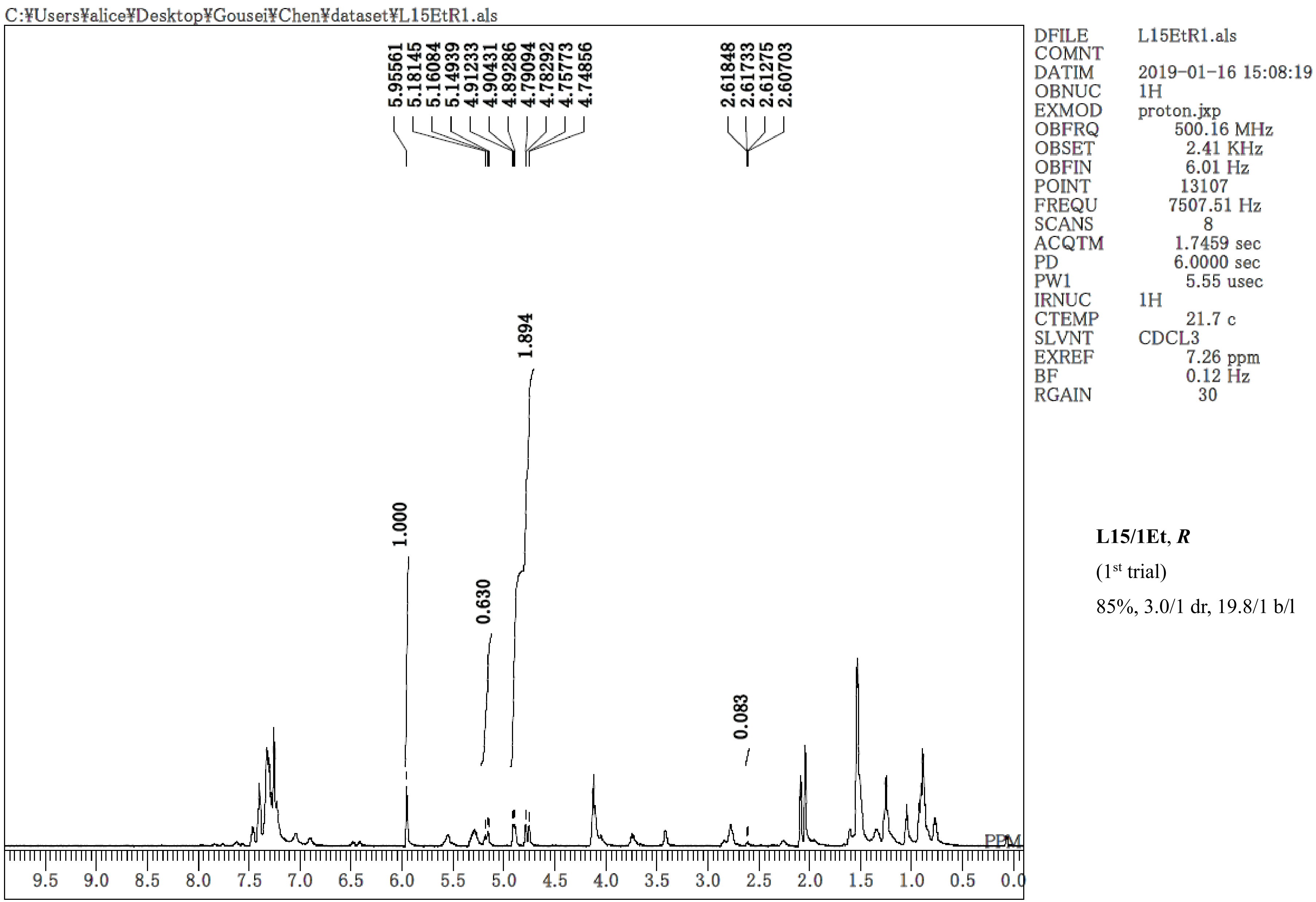


DFILE	L14PrS2.als
COMNT	
DATIM	07-09-2020 22:56:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L14/1Pr, ***R***

(2nd trial)

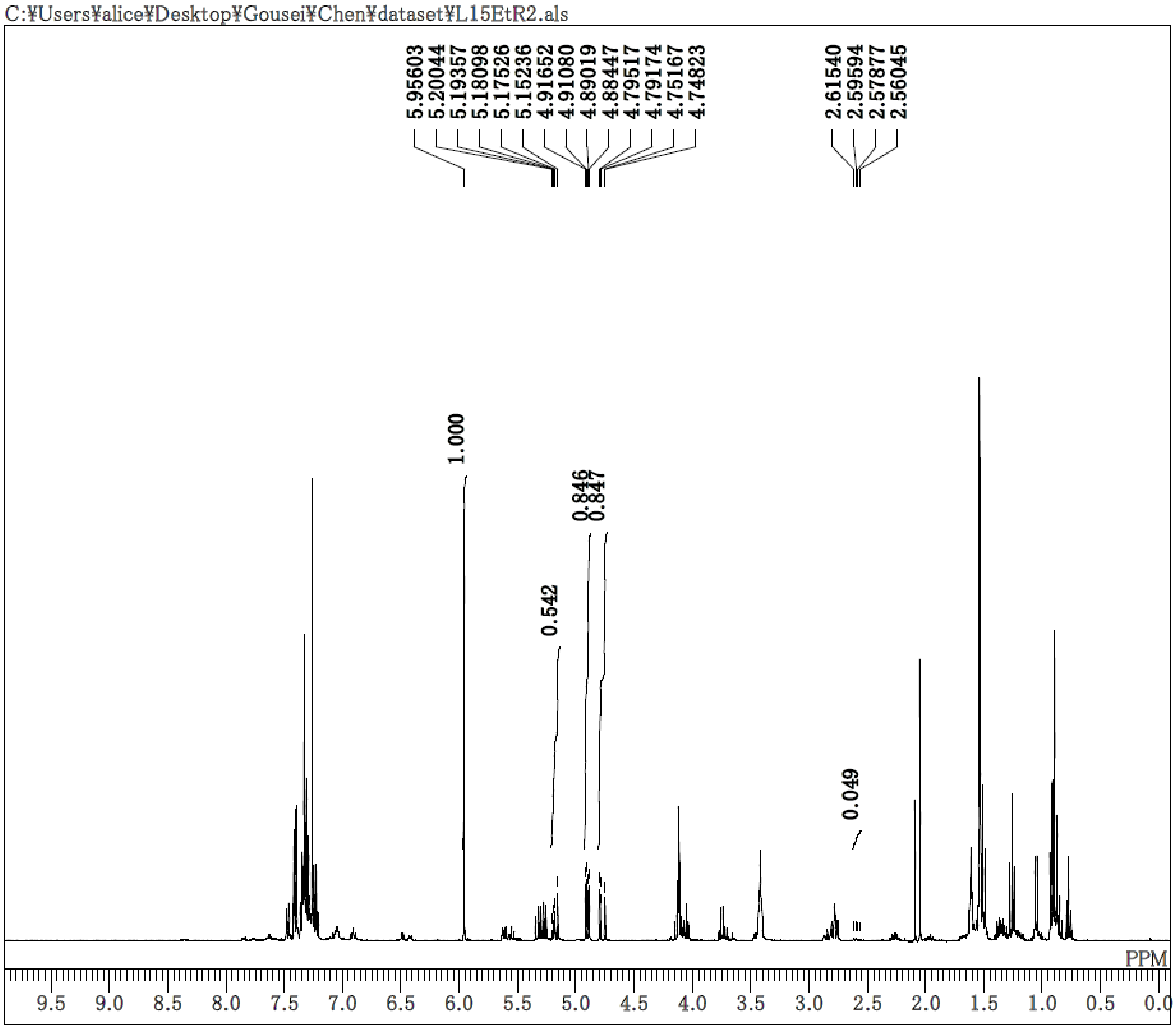
88%, 14.9/1 dr, >50/1 b/l



L15/1Et, ***R***

(1st trial)

85%, 3.0/1 dr, 19.8/1 b/l

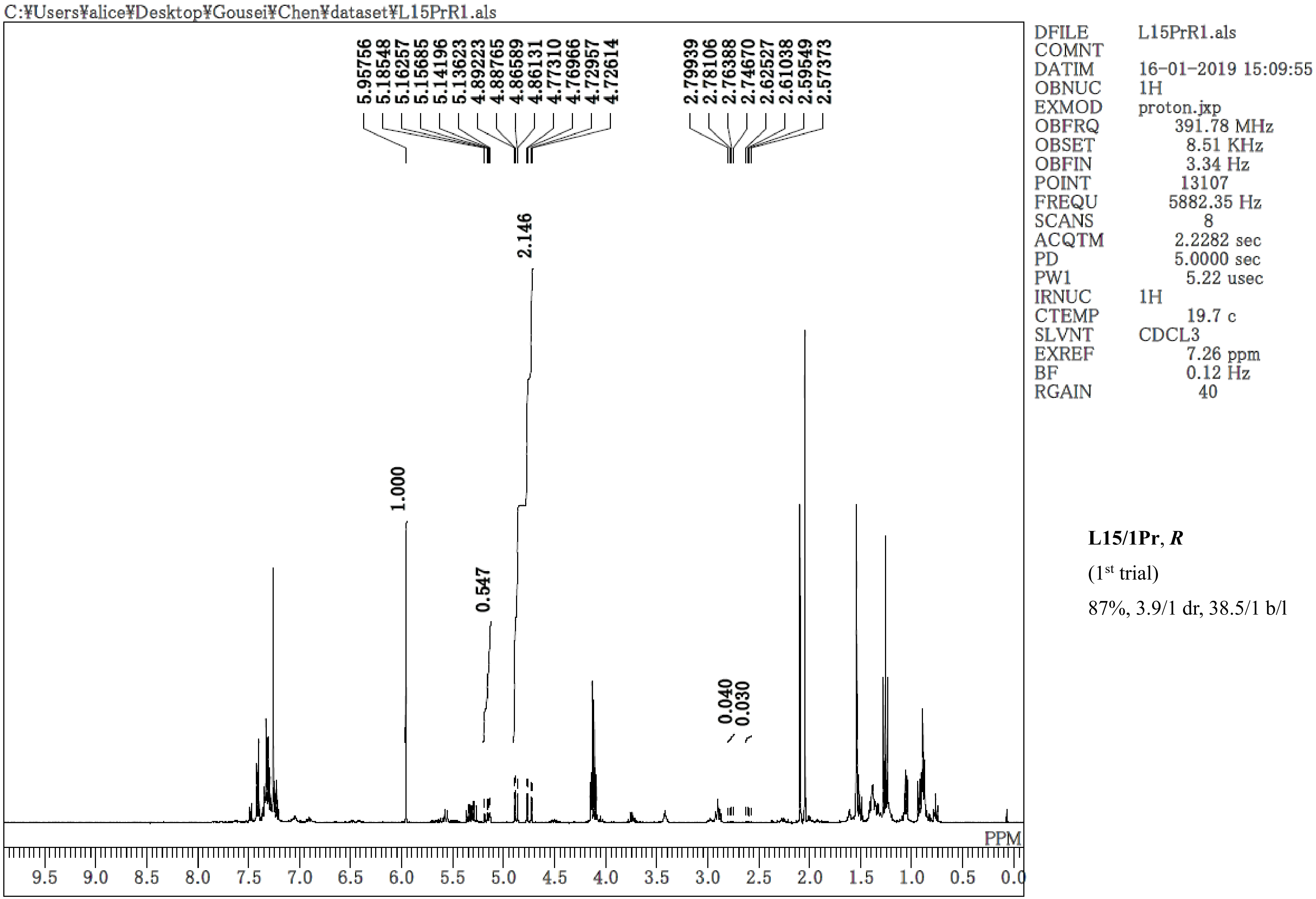


DFILE	L15EtR2.als
COMNT	
DATIM	07-09-2020 22:39:16
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38

L15/1Et, ***R***

(2nd trial)

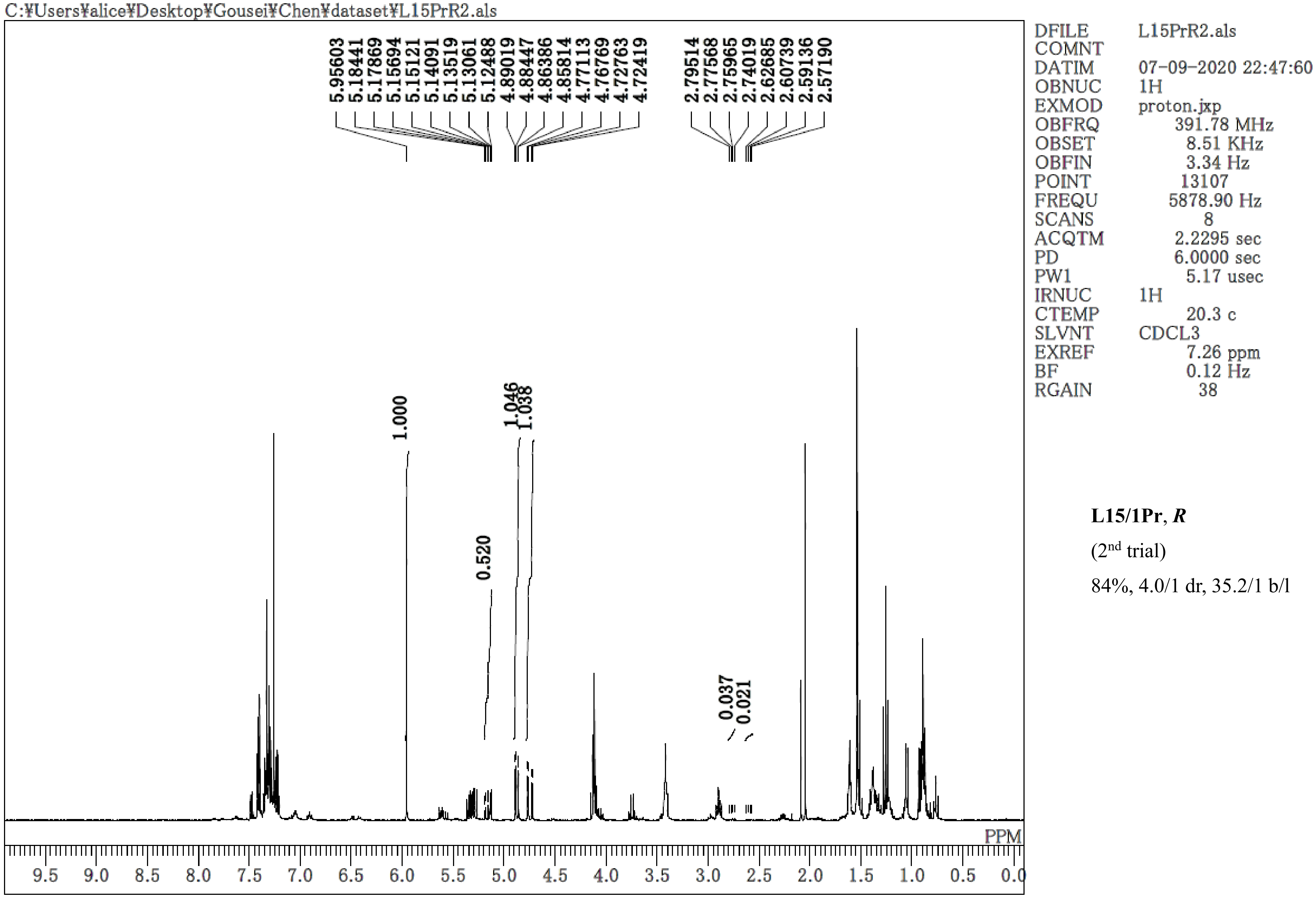
73%, 3.1/1 dr, 22.8/1 b/l



L15/1Pr, ***R***

(1st trial)

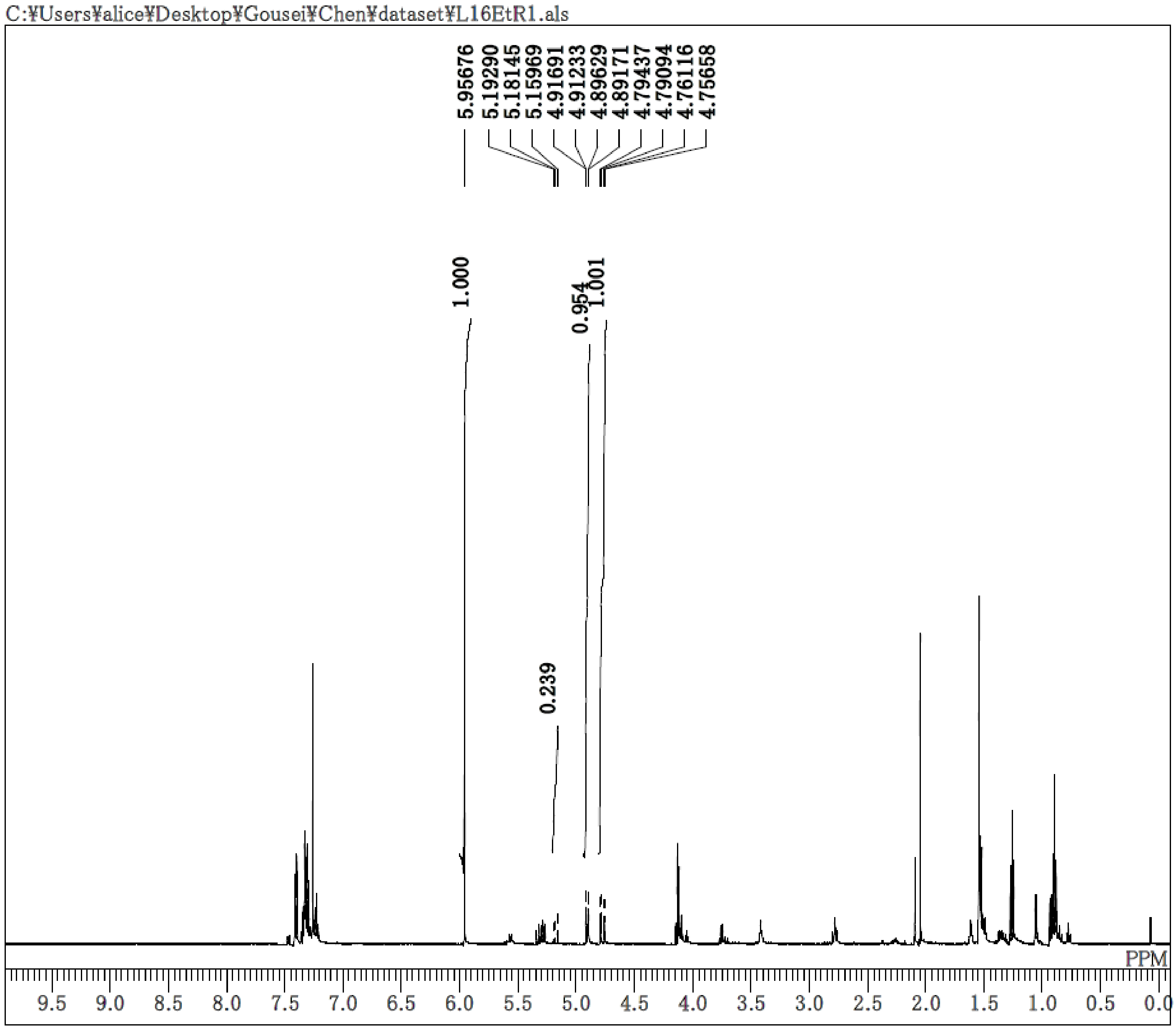
87%, 3.9/1 dr, 38.5/1 b/l



L15/1Pr, ***R***

(2nd trial)

84%, 4.0/1 dr, 35.2/1 b/l



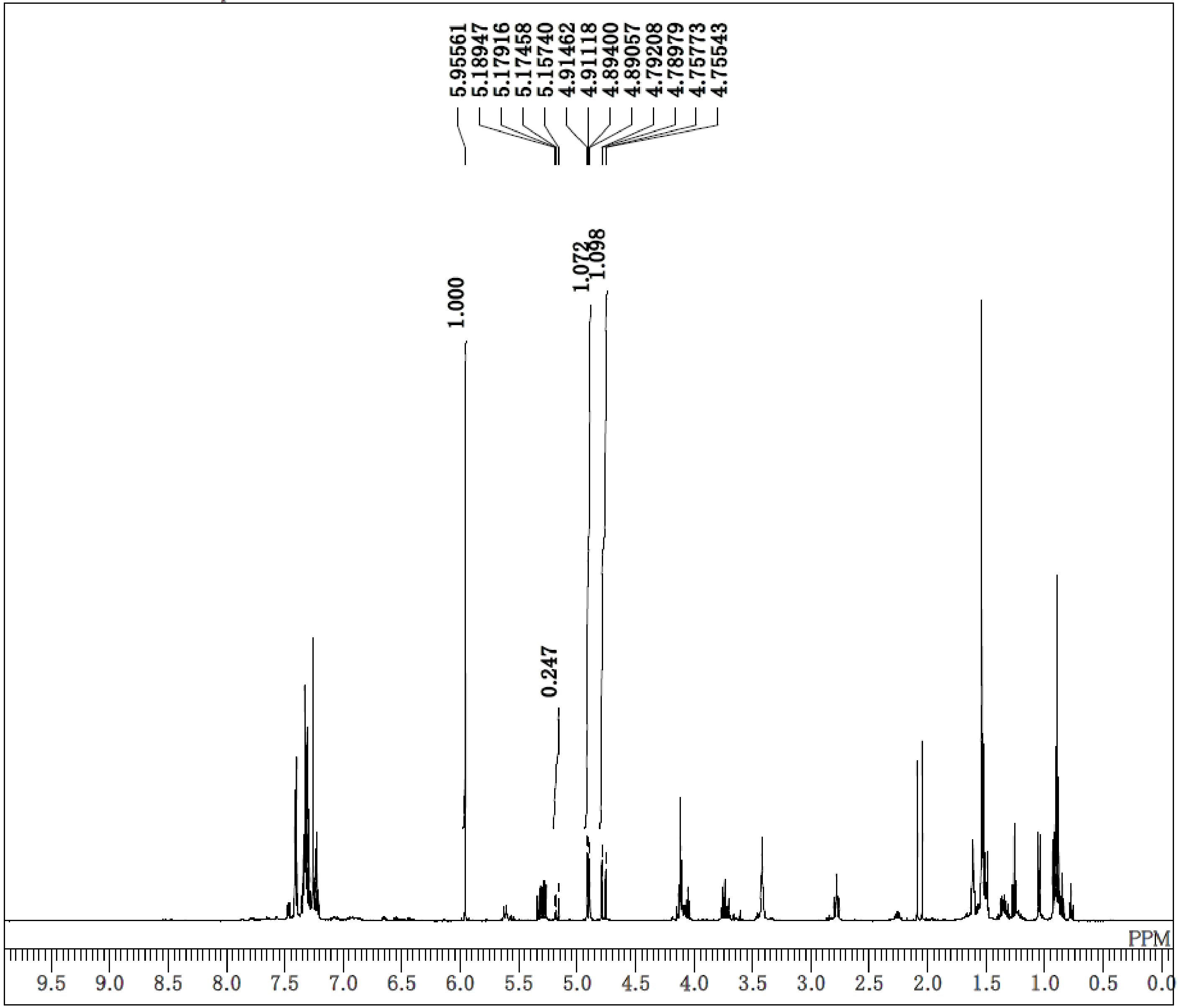
DFILE	L16EtR1.als
COMNT	
DATIM	2019-09-06 14:45:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L16/1Et, ***R***

(1st trial)

69%, 8.2/1 dr, >50/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L16EtR2.als



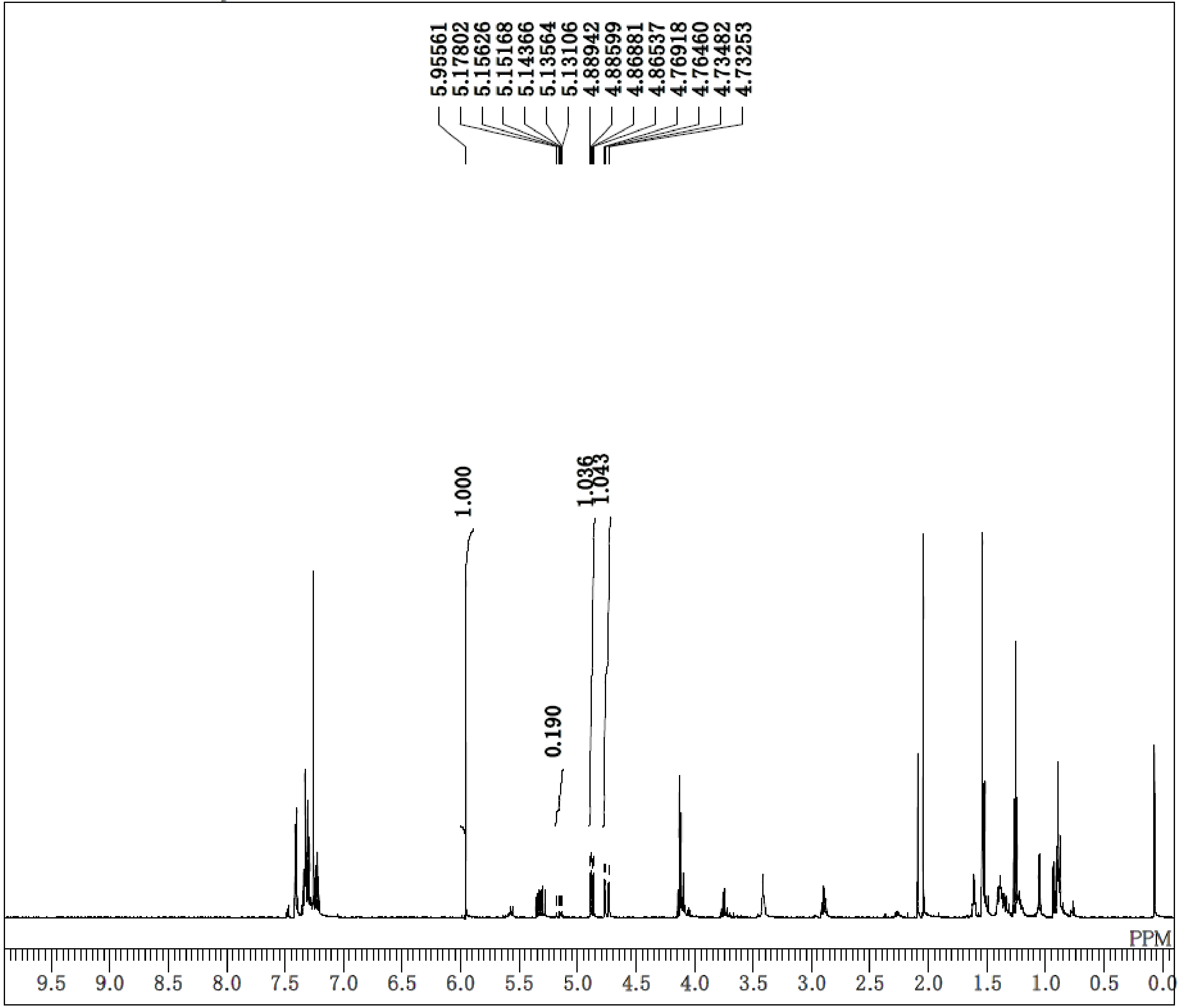
DFILE	L16EtR2.als
COMNT	
DATIM	2020-09-05 16:00:22
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	16
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L16/1Et, ***R***

(2nd trial)

76%, 8.8/1 dr, >50/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L16PrR1.als



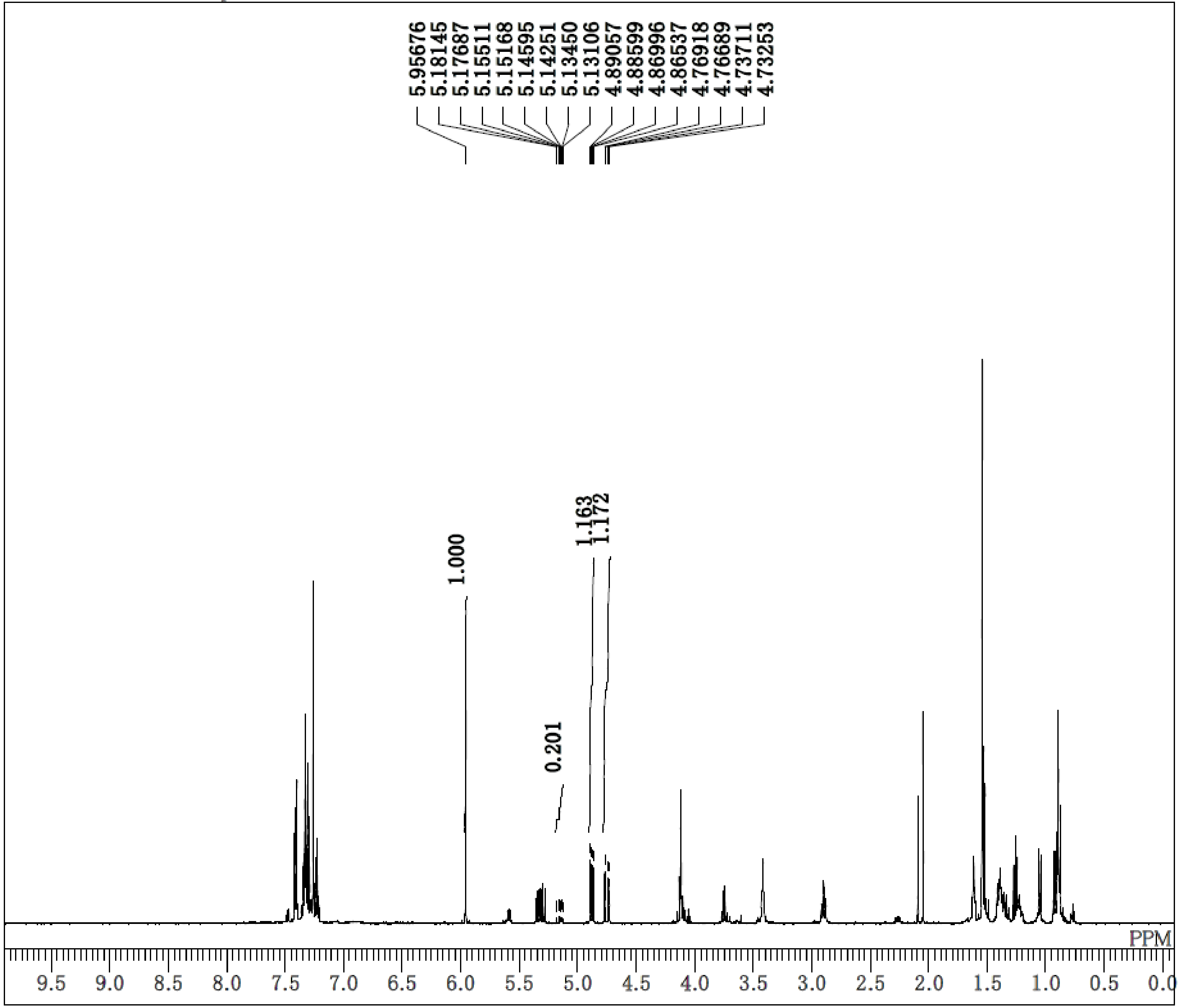
DFILE	L16PrR1.als
COMNT	
DATIM	2019-09-04 10:22:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L16/1Pr, ***R***

(1st trial)

71%, 10.9/1 dr, >50/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L16PrR2.als

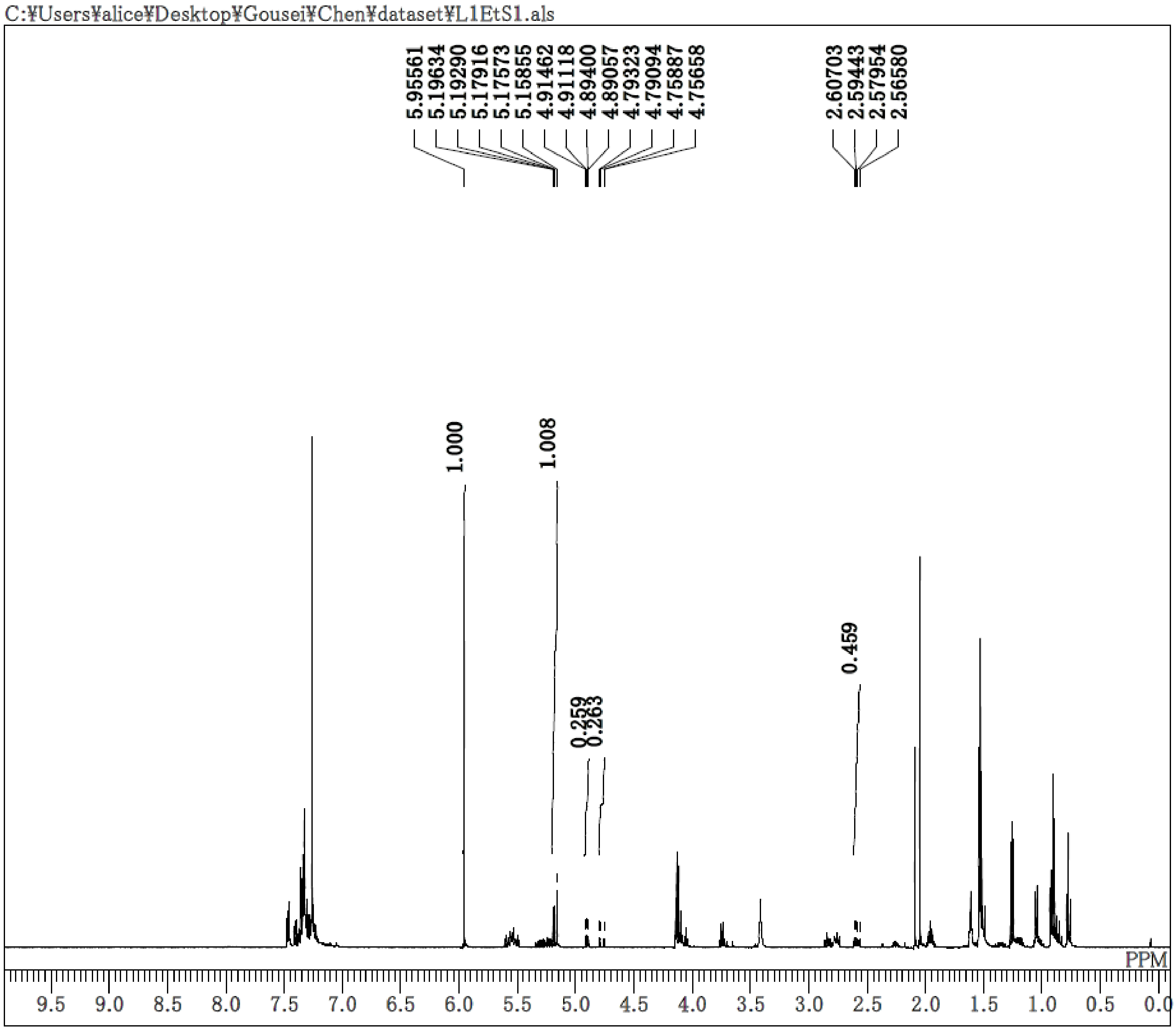


DFILE	L16PrR2.als
COMNT	
DATIM	2020-09-05 16:08:04
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	16
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L16/1Pr, ***R***

(2nd trial)

80%, 11.6/1 dr, >50/1 b/l

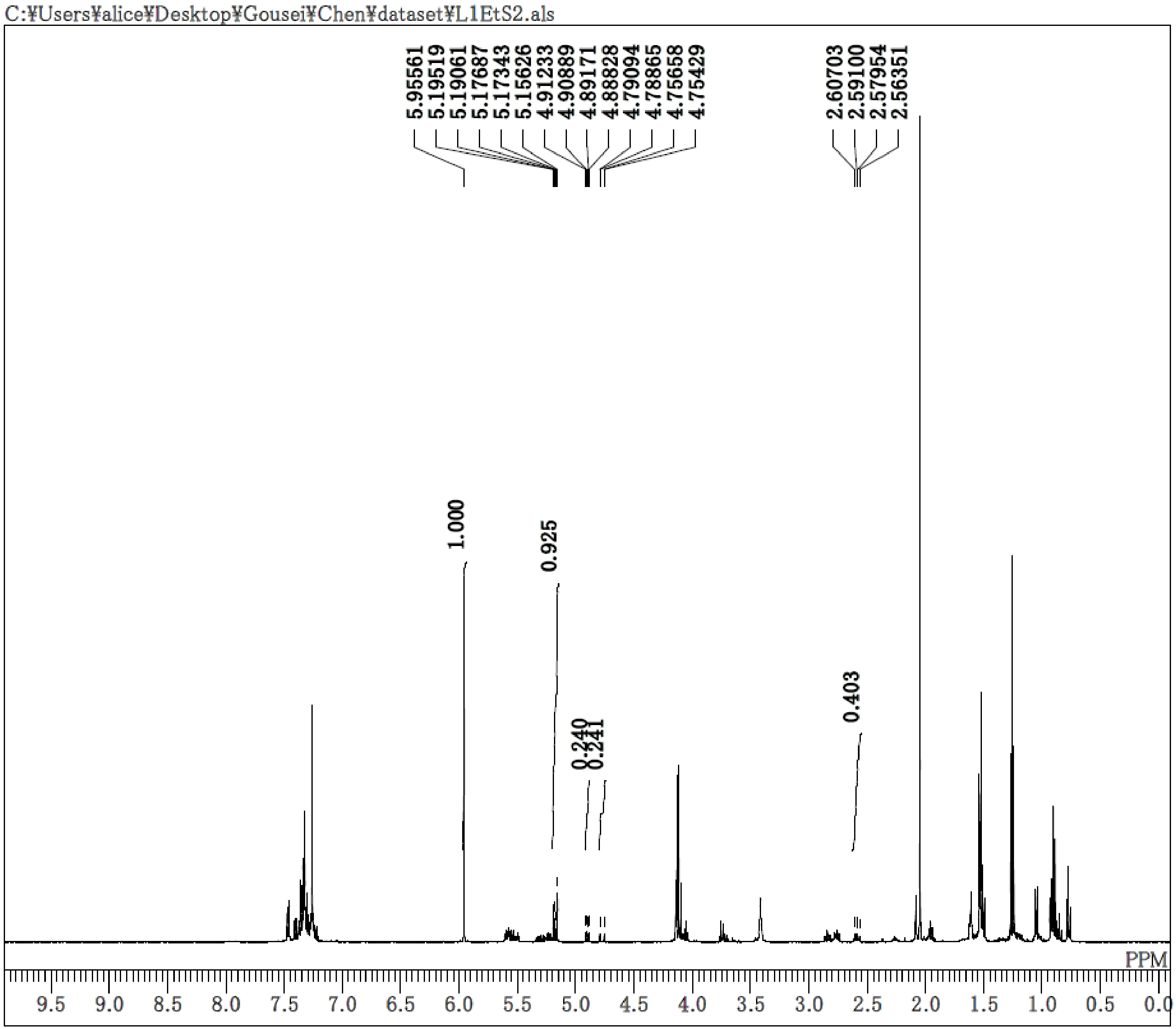


DFILE	L1EtS1.als
COMNT	
DATIM	2018-11-23 12:59:23
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L1/1Et, ***S***

(1st trial)

77%, 1.9/1 dr, 1.7/1 b/l

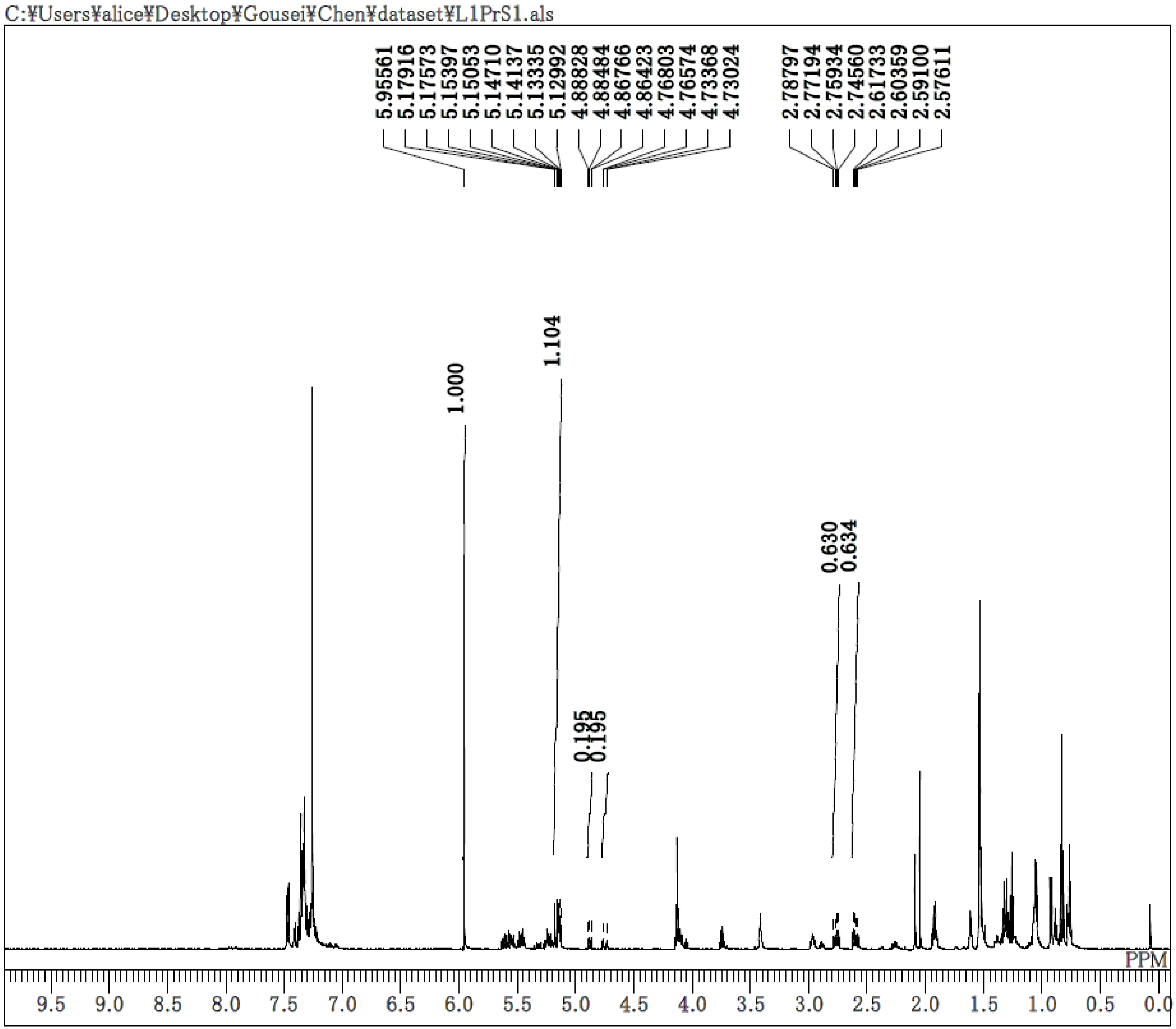


DFILE	L1EtS2.als
COMNT	
DATIM	2020-08-26 15:30:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L1/1Et, ***S***

(2nd trial)

70%, 1.9/1 dr, 1.7/1 b/l

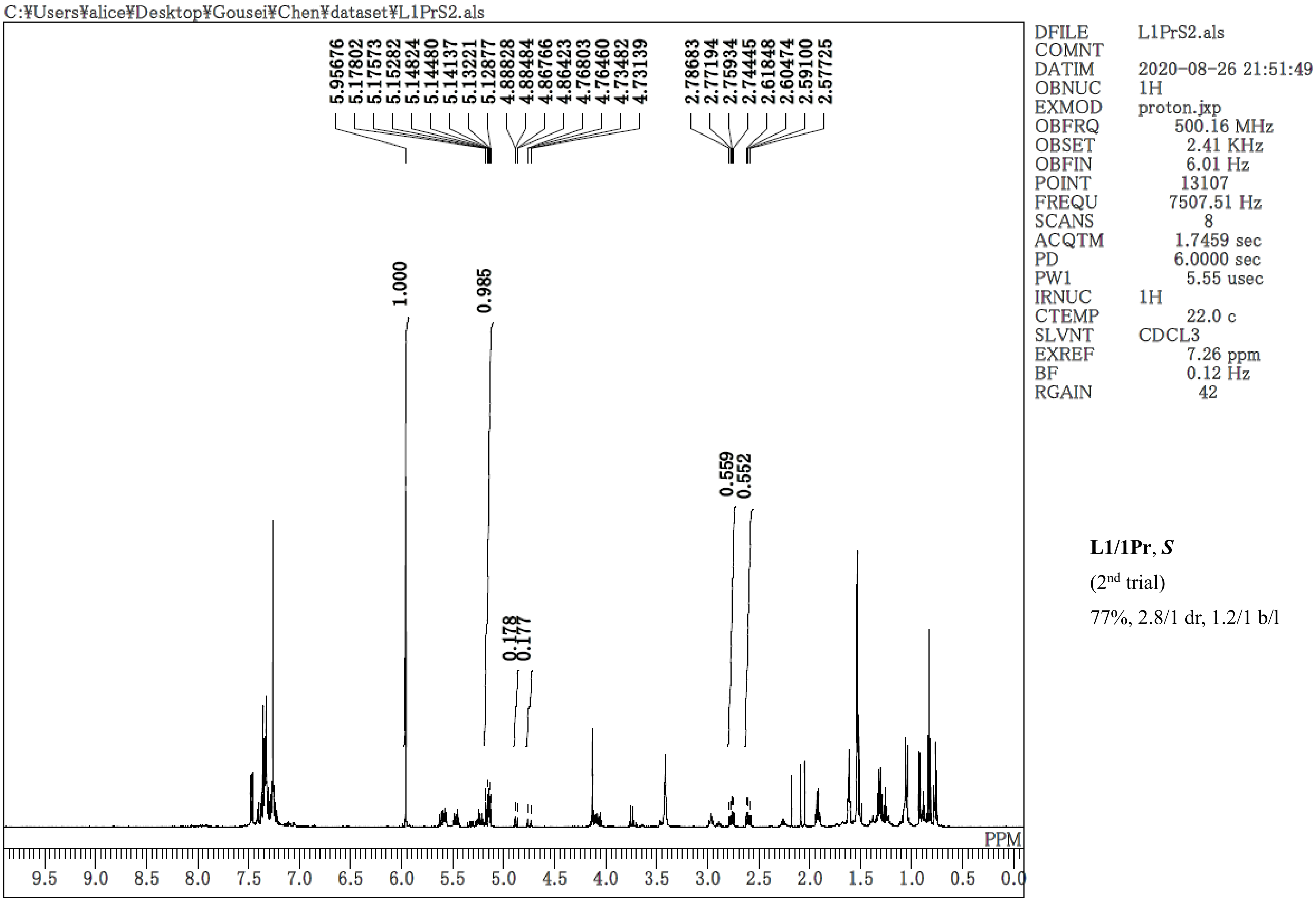


DFILE	L1PrS1.als
COMNT	
DATIM	2018-11-21 15:26:37
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L1/1Pr, ***S***

(1st trial)

87%, 2.8/1 dr, 1.2/1 b/l

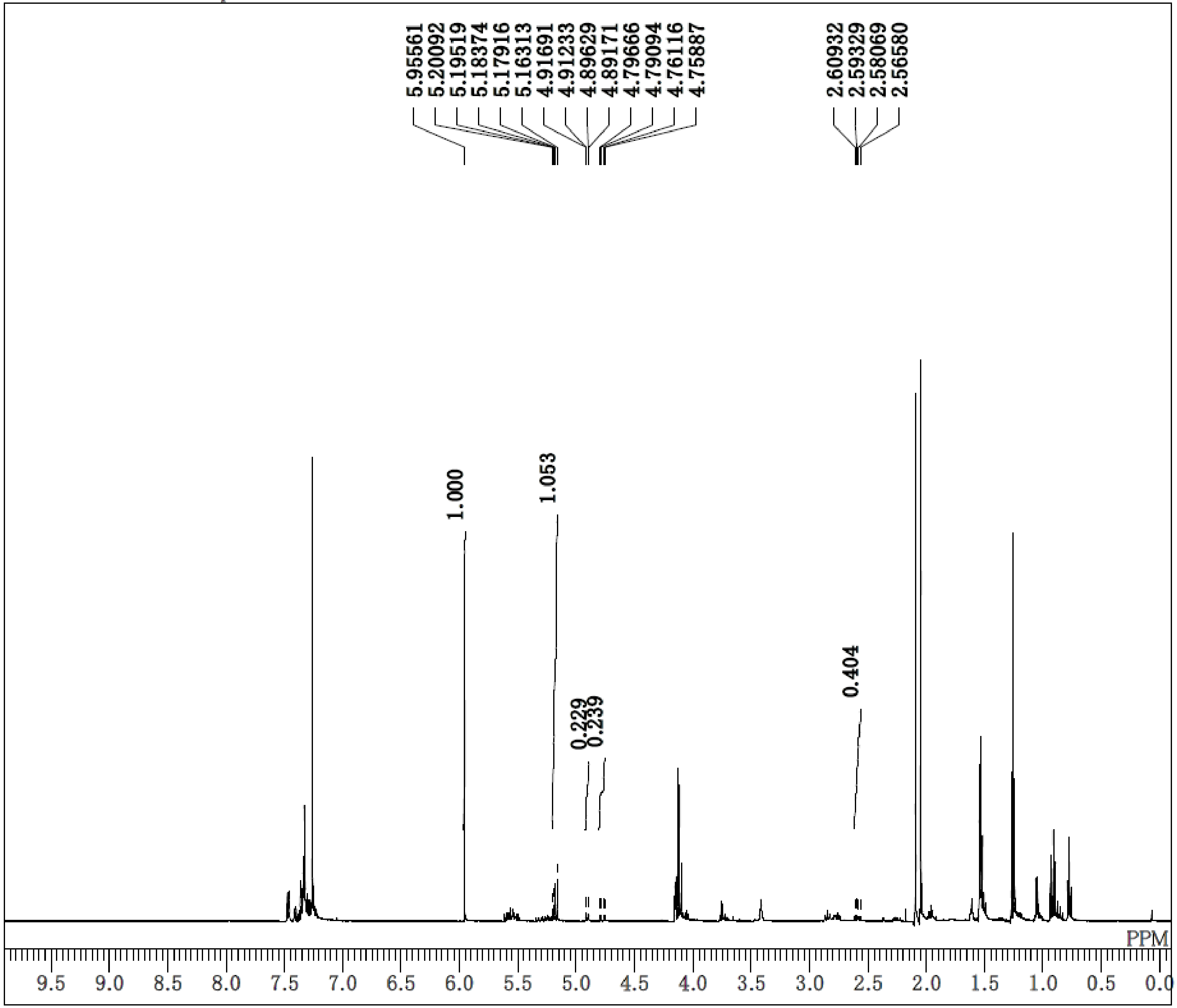


L1/1Pr, ***S***

(2nd trial)

77%, 2.8/1 dr, 1.2/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L2EtS1.als



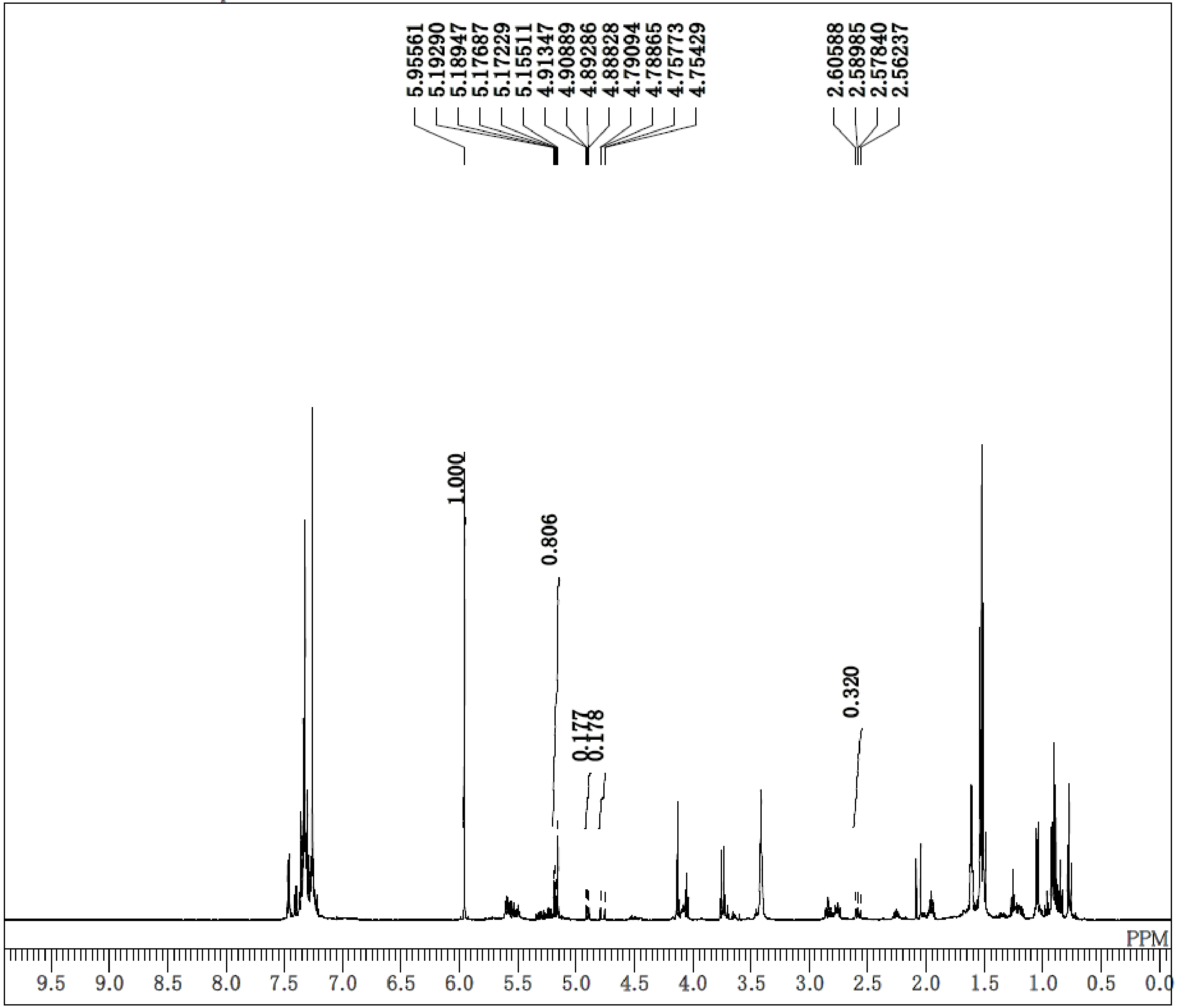
DFILE	L2EtS1.als
COMNT	
DATIM	2018-11-13 16:15:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L2/1Et, ***S***

(1st trial)

73%, 2.3/1 dr, 1.9/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L2EtS2.als



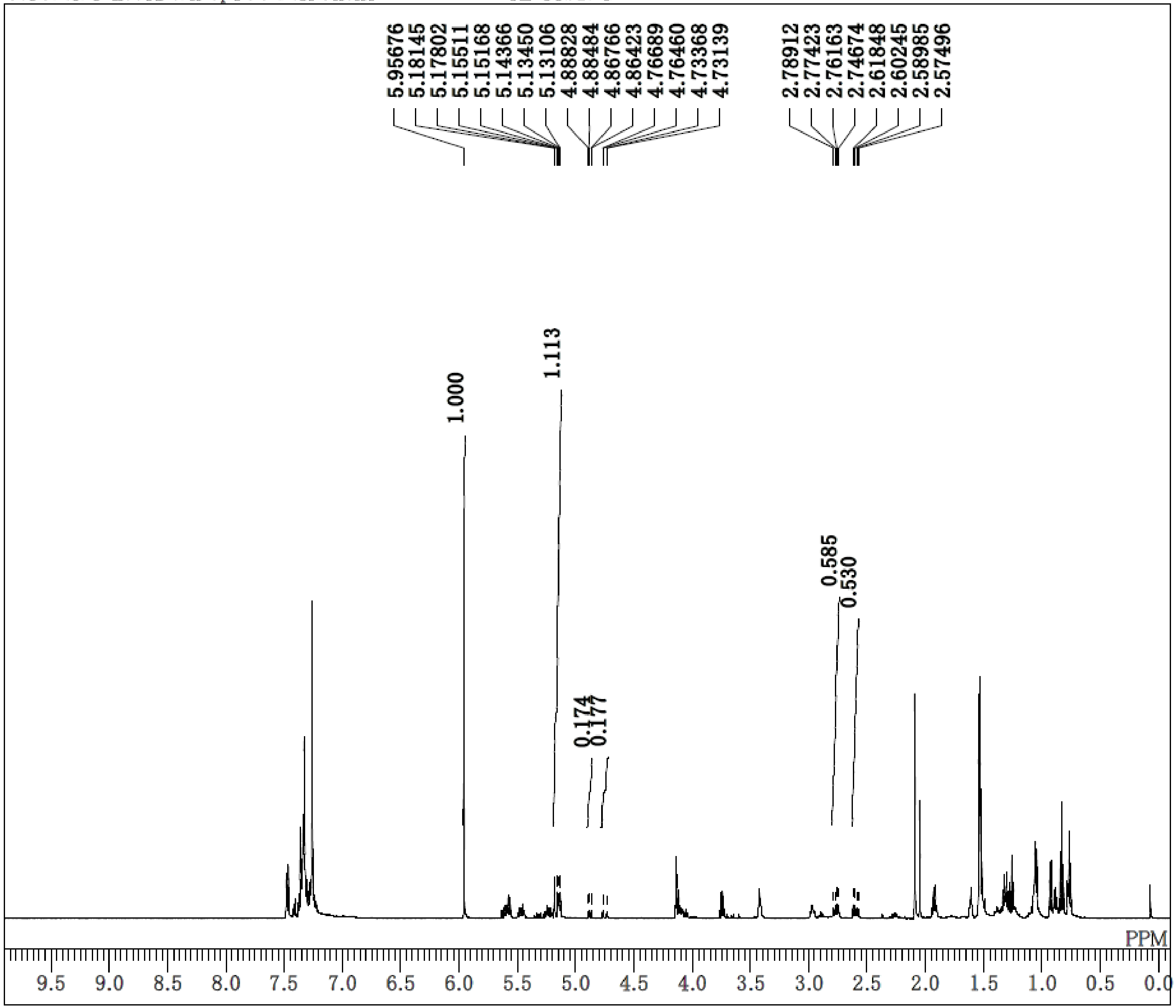
DFILE	L2EtS2.als
COMNT	
DATIM	2020-08-28 00:28:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L2/1Et, ***S***

(2nd trial)

57%, 2.3/1 dr, 1.8/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥aaaaaaaaaaaaa¥L2PrS1.als

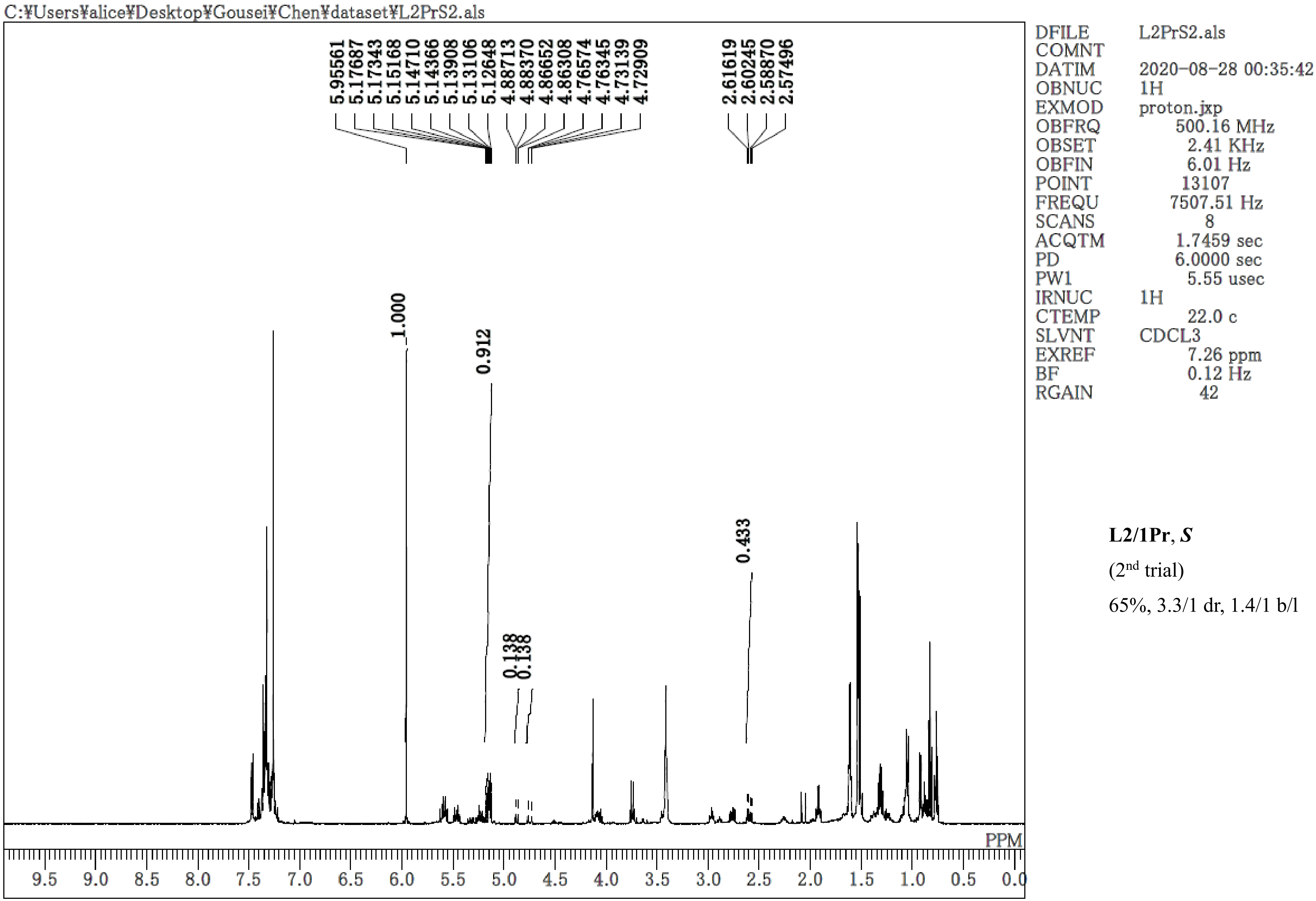


DFILE	L2PrS1.als
COMNT	
DATIM	2018-11-11 22:53:41
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L2/1Pr, ***S***

(1st trial)

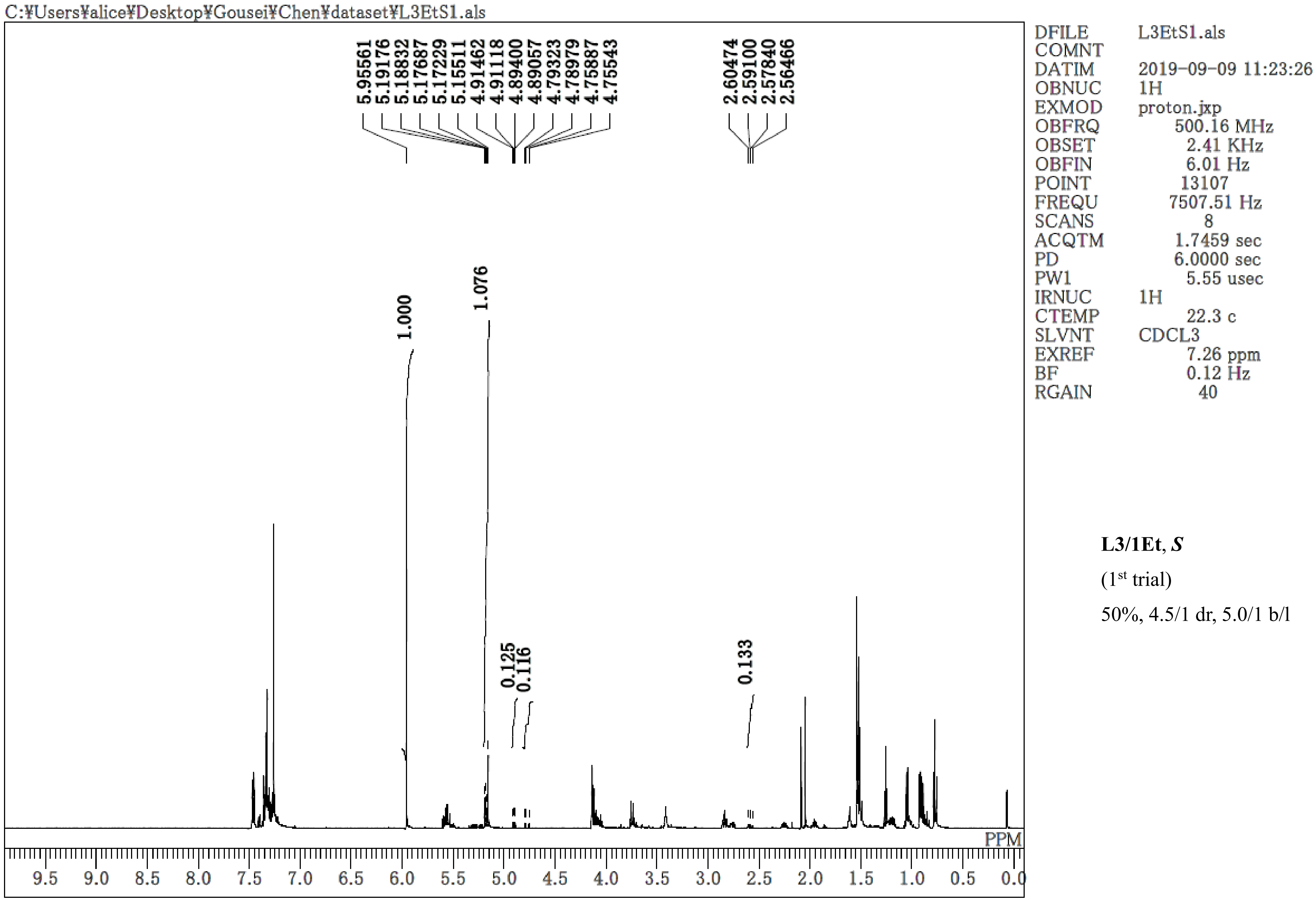
81%, 3.2/1 dr, 1.3/1 b/l



L2/1Pr, ***S***

(2nd trial)

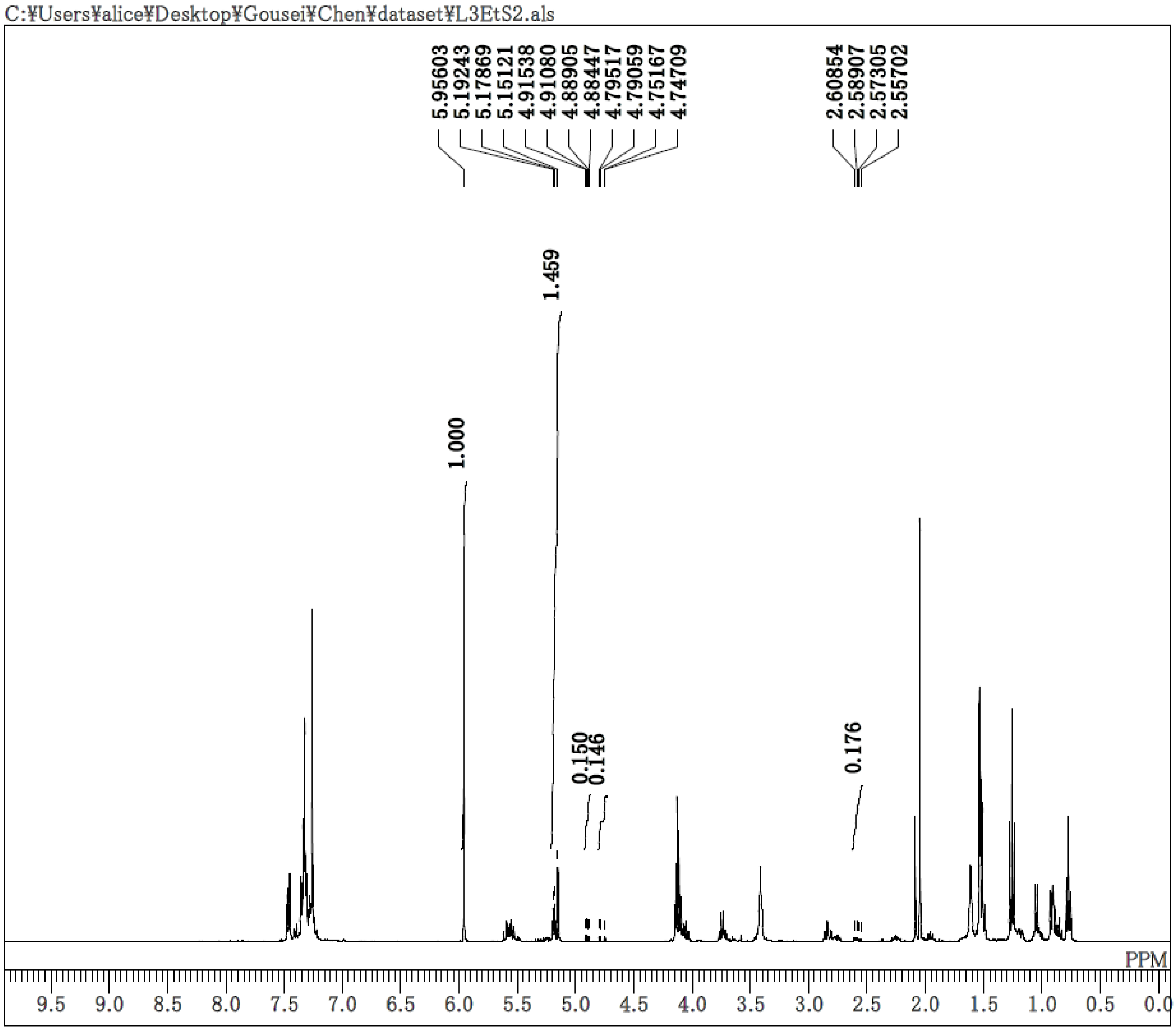
65%, 3.3/1 dr, 1.4/1 b/l



L3/1Et, ***S***

(1st trial)

50%, 4.5/1 dr, 5.0/1 b/l

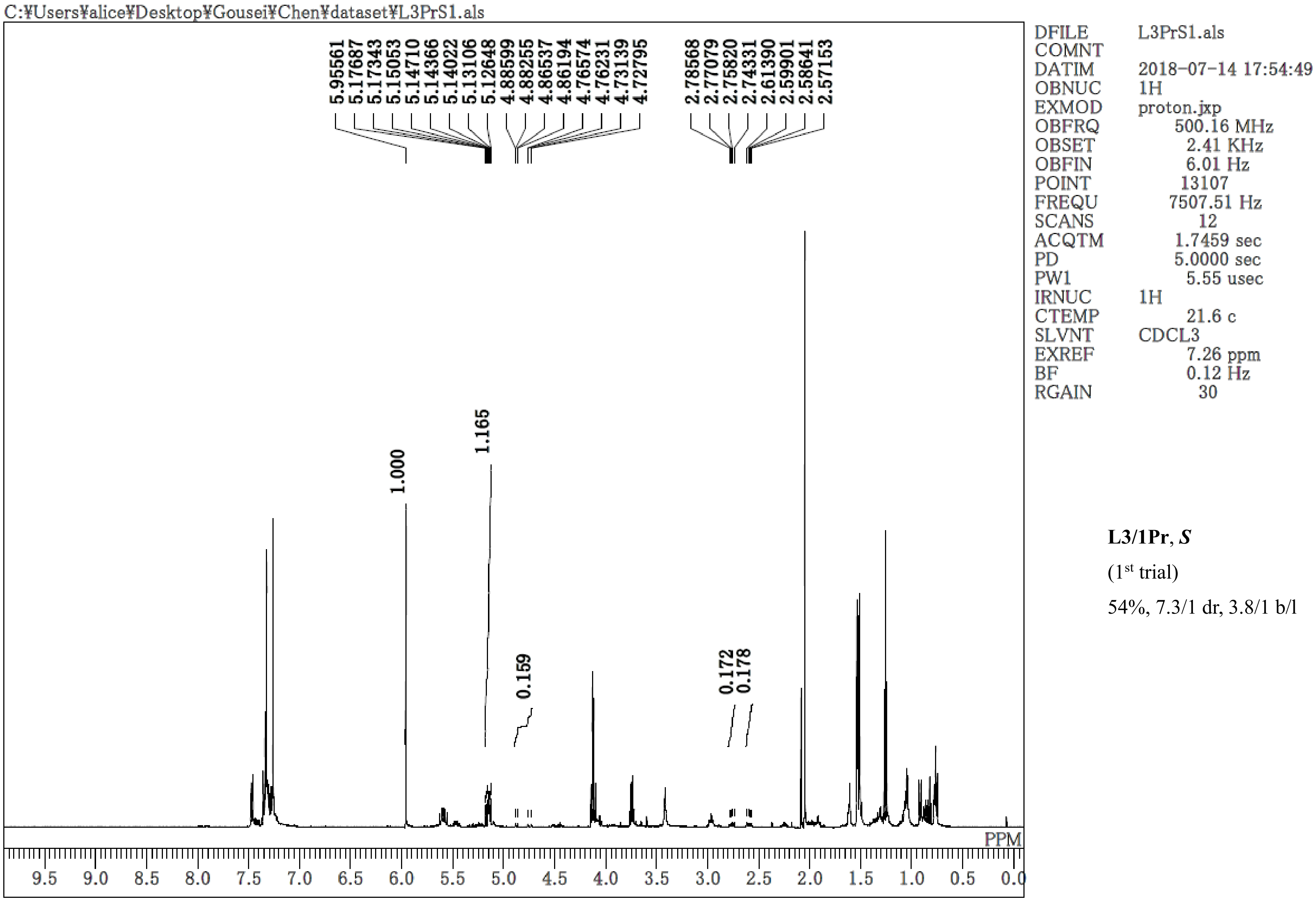


DFILE	L3EtS2.als
COMNT	
DATIM	22-08-2020 11:24:59
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.62 Hz
RGAIN	40

L3/1Et, *S*

(2nd trial)

66%, 4.9/1 dr, 5.0/1 b/l

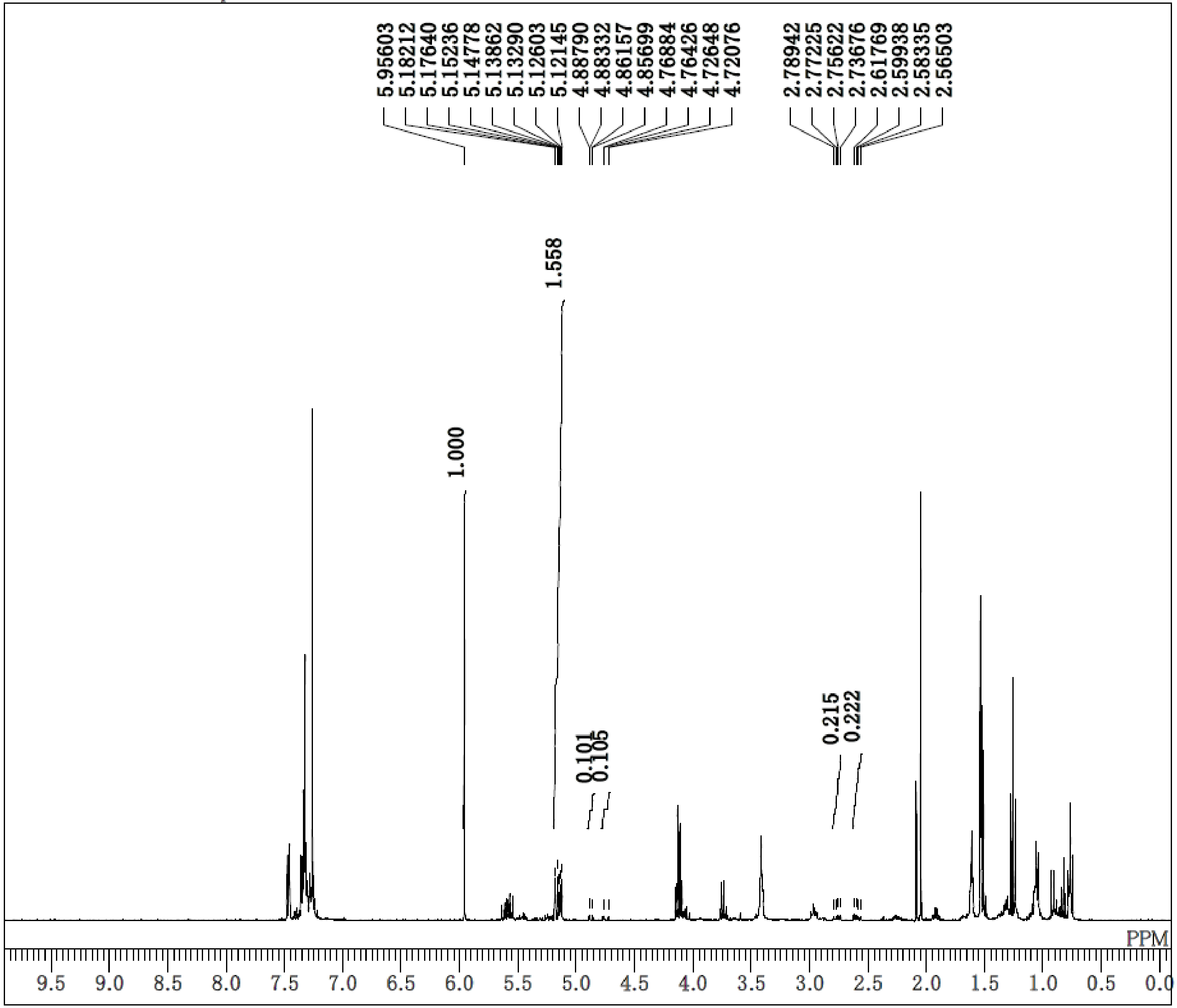


L3/1Pr, ***S***

(1st trial)

54%, 7.3/1 dr, 3.8/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L3PrS2.als



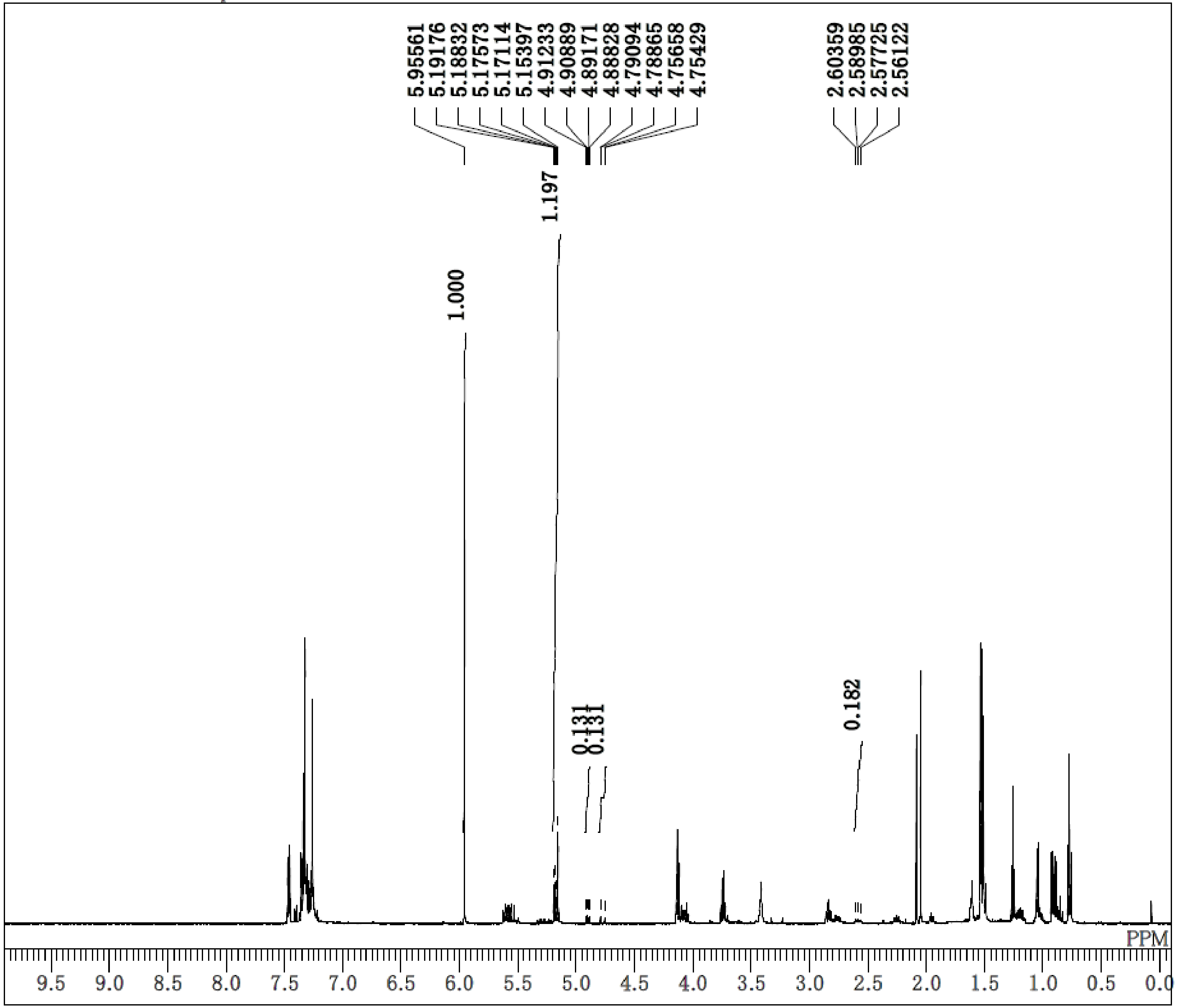
DFILE	L3PrS2.als
COMNT	
DATIM	23-08-2020 16:55:12
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L3/1Pr, ***S***

(2nd trial)

69%, 7.6/1 dr, 4.0/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L4EtS1.als

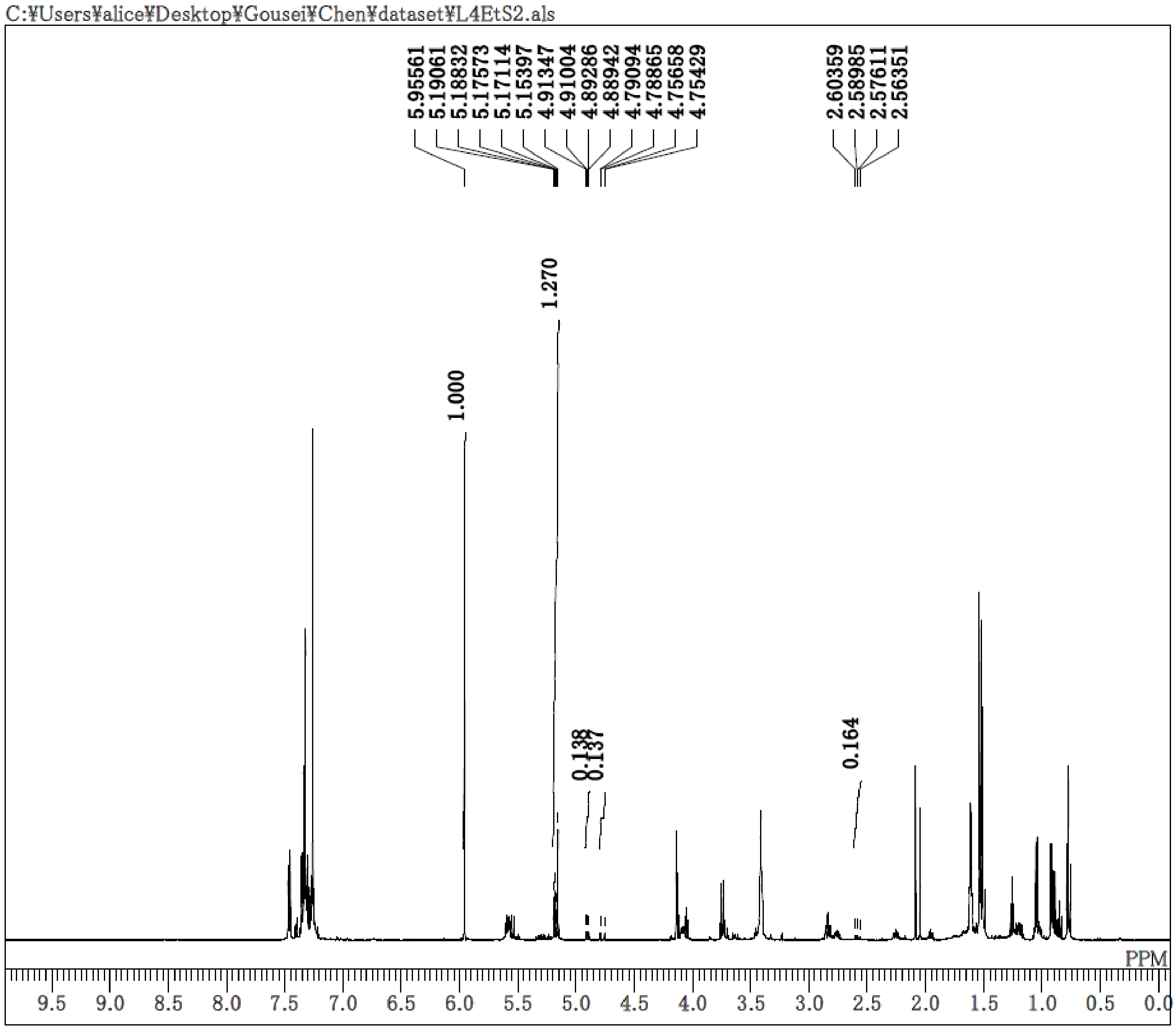


DFILE	L4EtS1.als
COMNT	
DATIM	2019-09-28 14:55:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L4/1Et, ***S***

(1st trial)

57%, 4.6/1 dr, 4.0/1 b/l

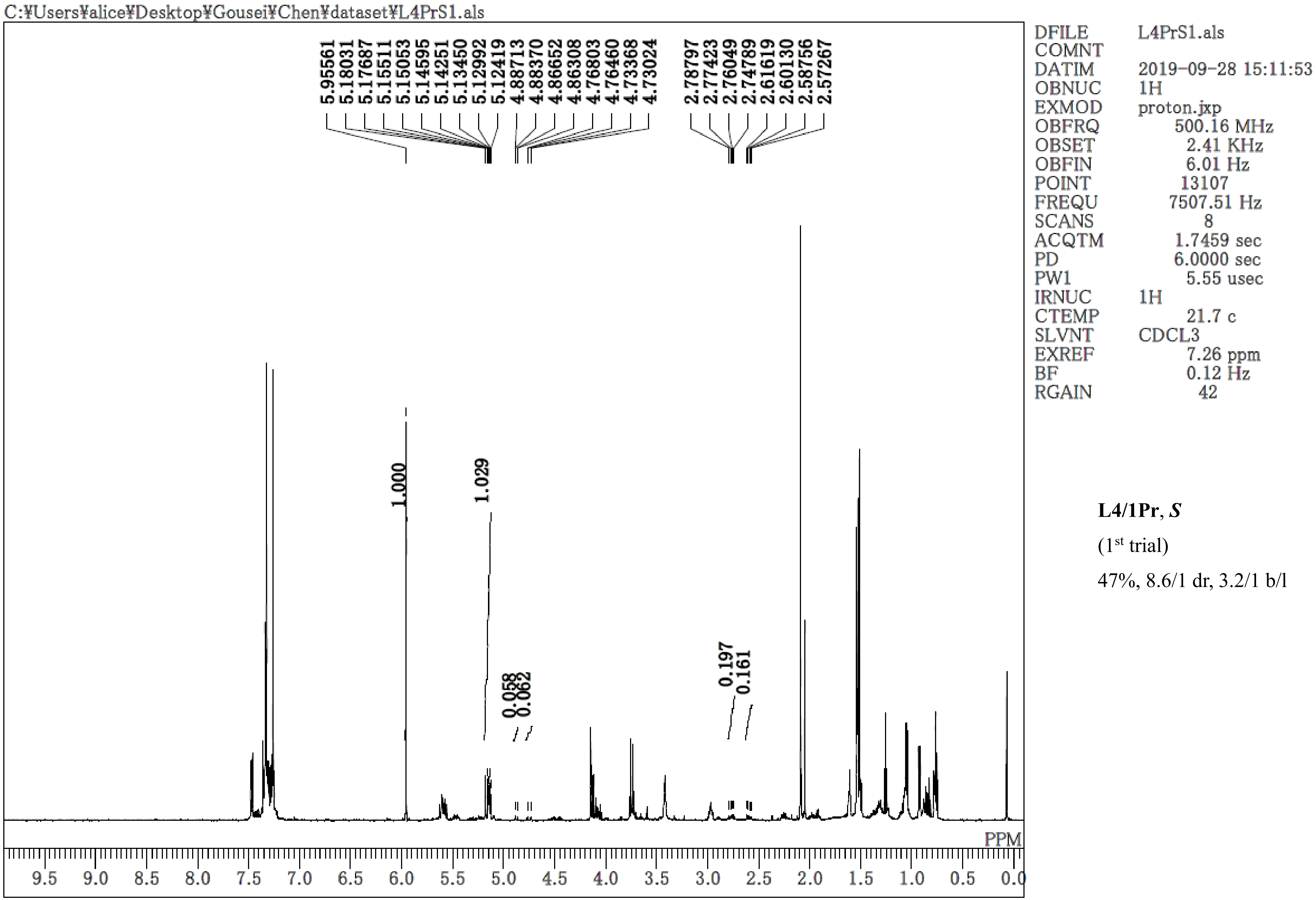


DFILE	L4EtS2.als
COMNT	
DATIM	2020-08-28 00:56:06
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L4/1Et, ***S***

(2nd trial)

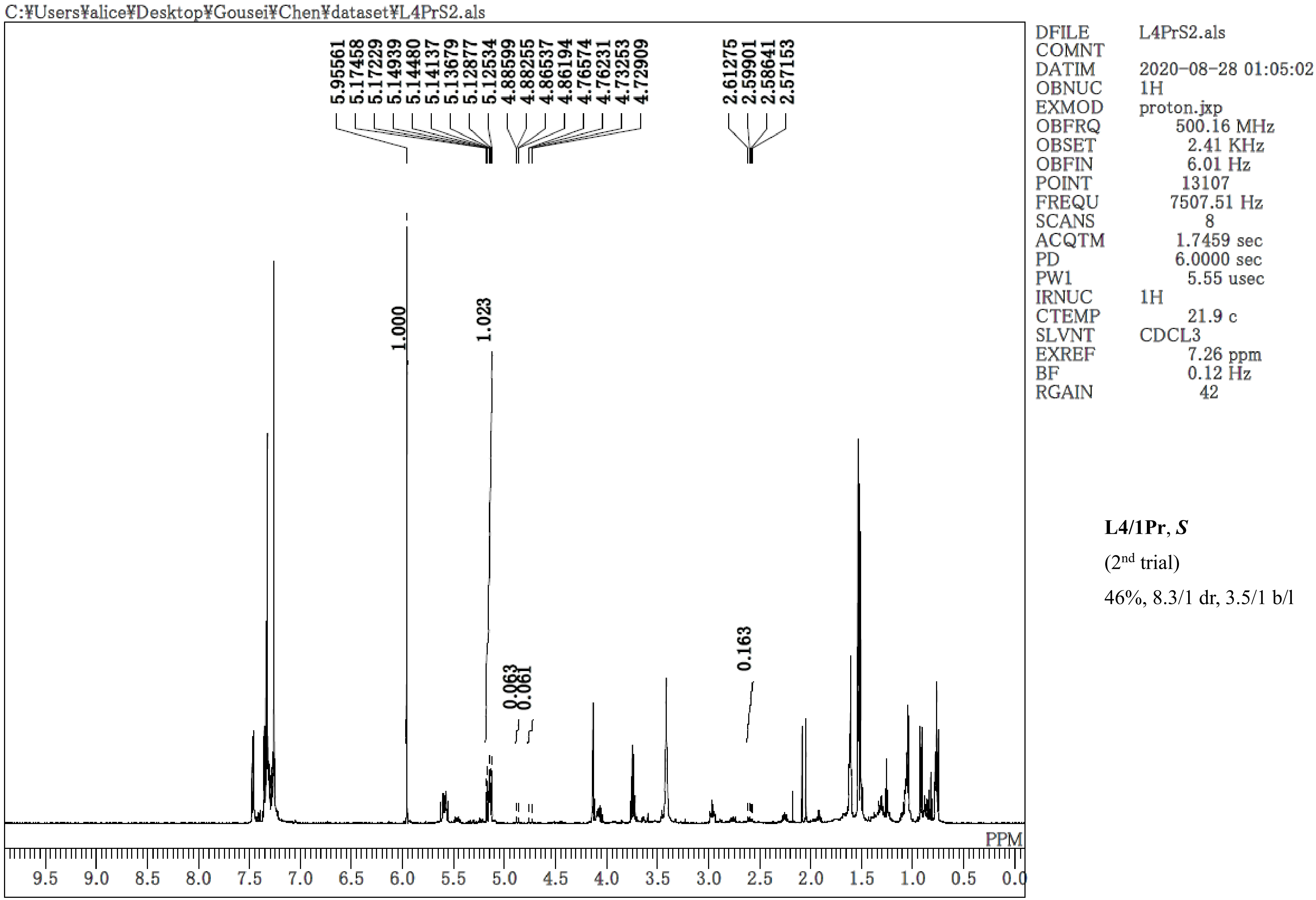
59%, 4.6/1 dr, 4.7/1 b/l



L4/1Pr, ***S***

(1st trial)

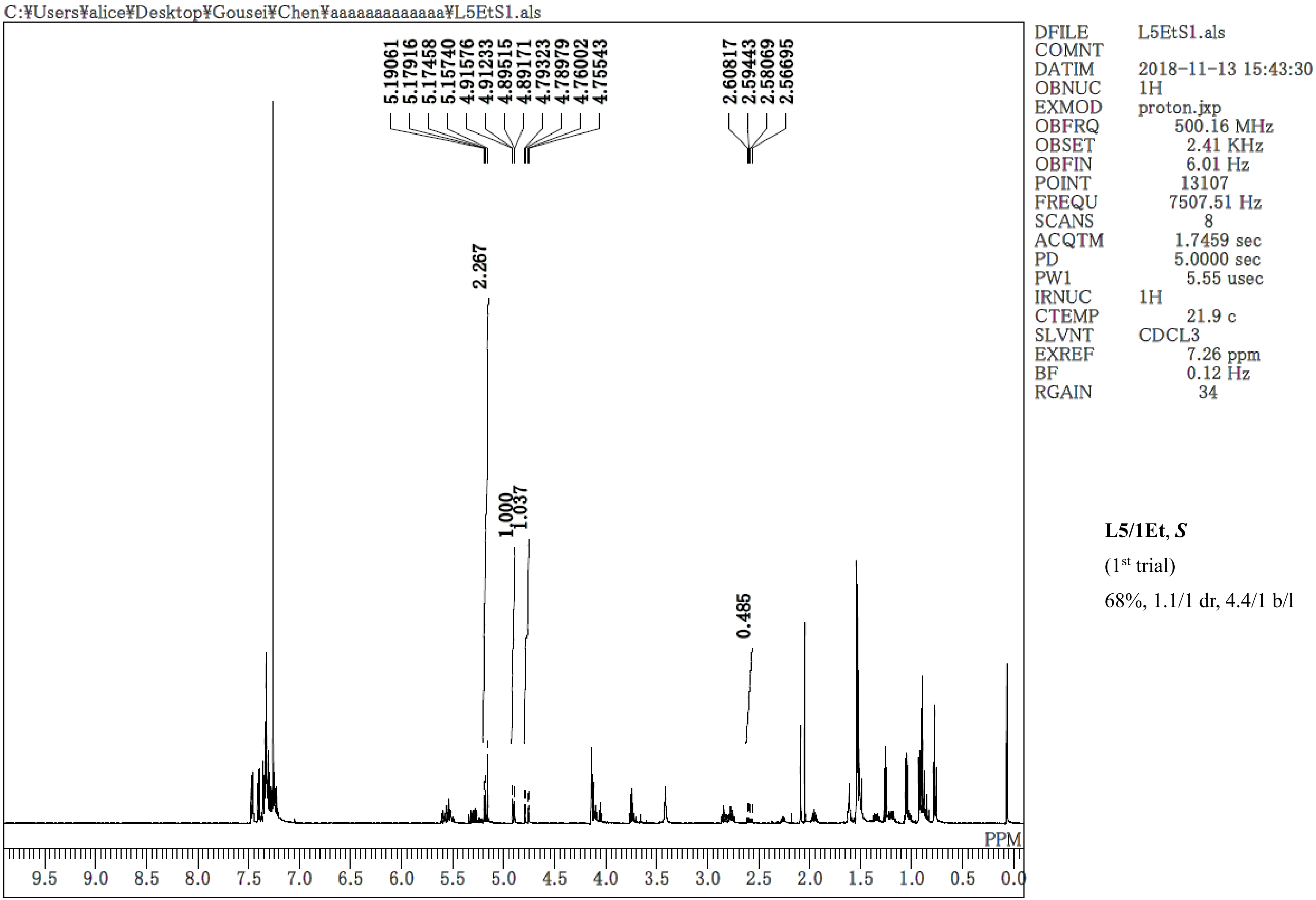
47%, 8.6/1 dr, 3.2/1 b/l



L4/1Pr, *S*

(2nd trial)

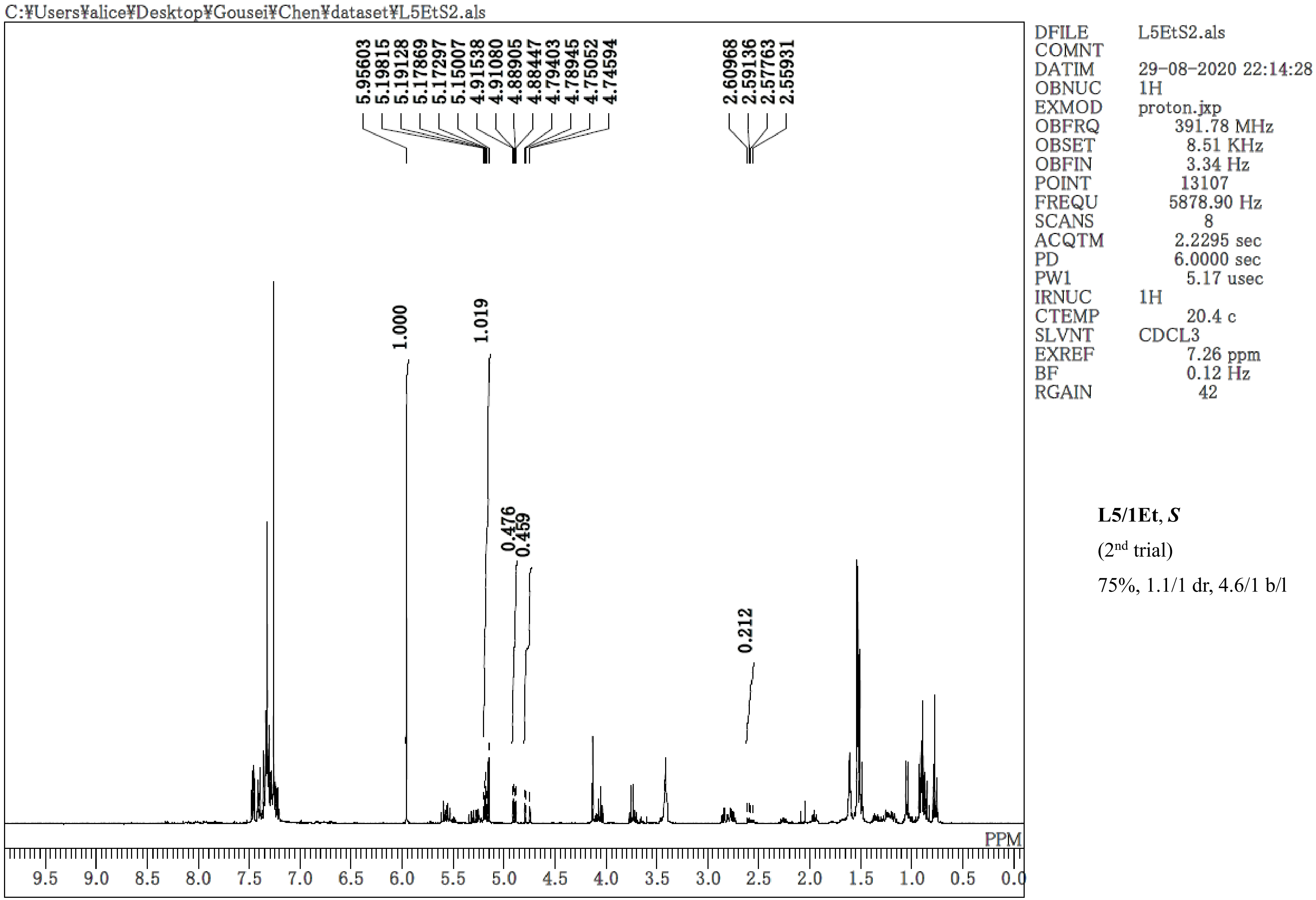
46%, 8.3/1 dr, 3.5/1 b/l



L5/1Et, *S*

(1st trial)

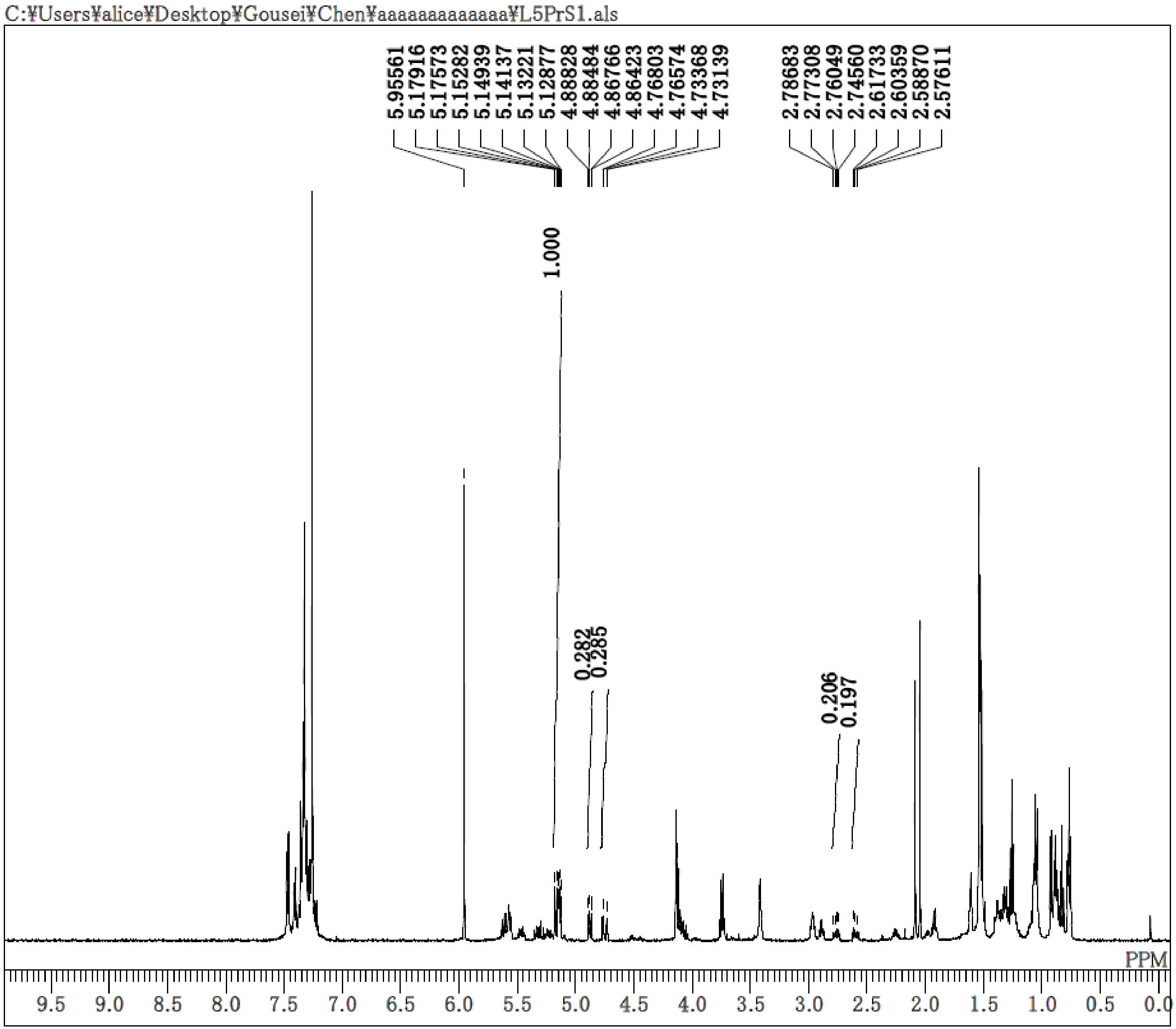
68%, 1.1/1 dr, 4.4/1 b/l



L5/1Et, ***S***

(2nd trial)

75%, 1.1/1 dr, 4.6/1 b/l

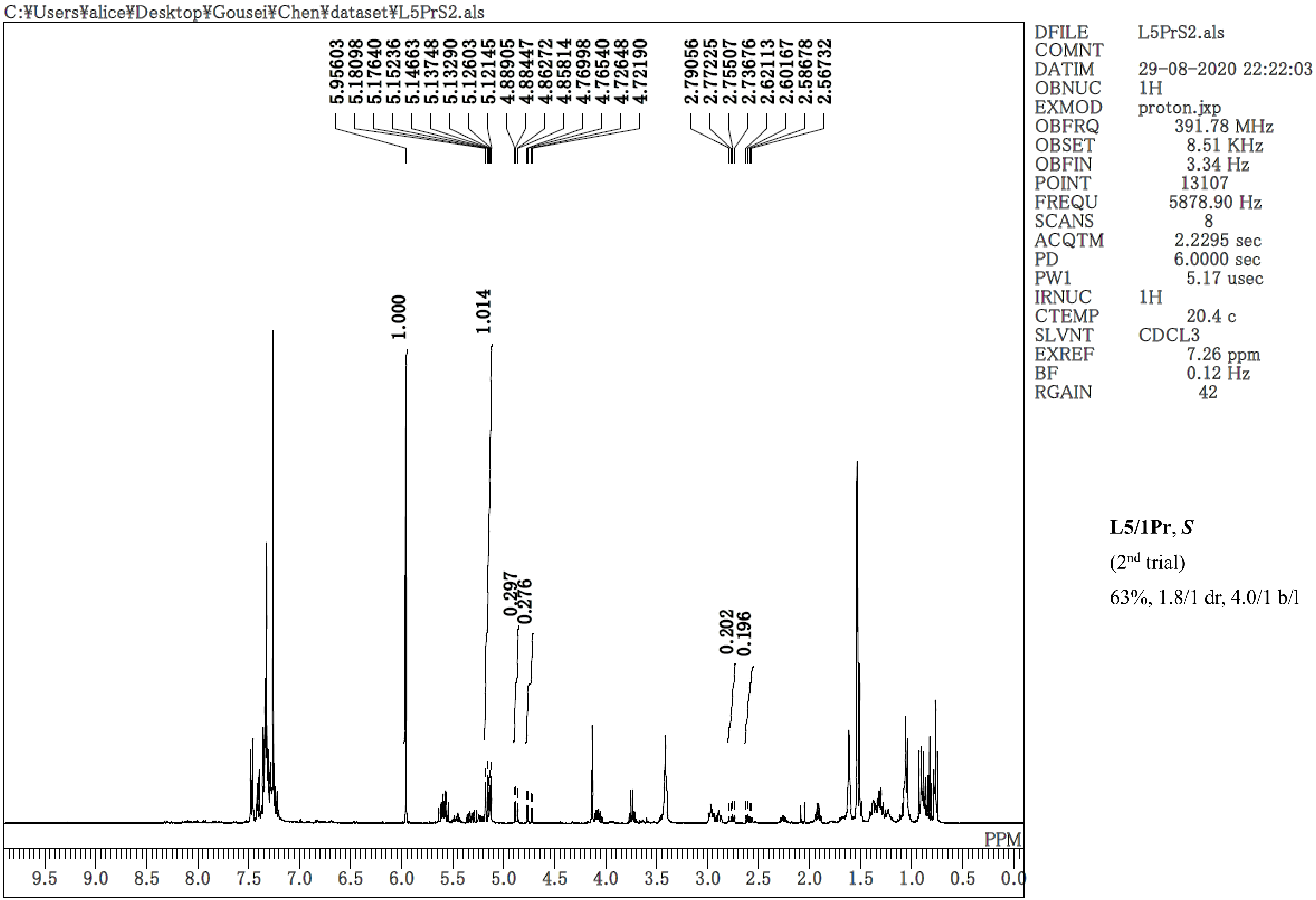


DFILE	L5PrS1.als
COMNT	
DATIM	2018-11-11 23:32:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L5/1Pr, ***S***

(1st trial)

66%, 1.7/1 dr, 3.7/1 b/l

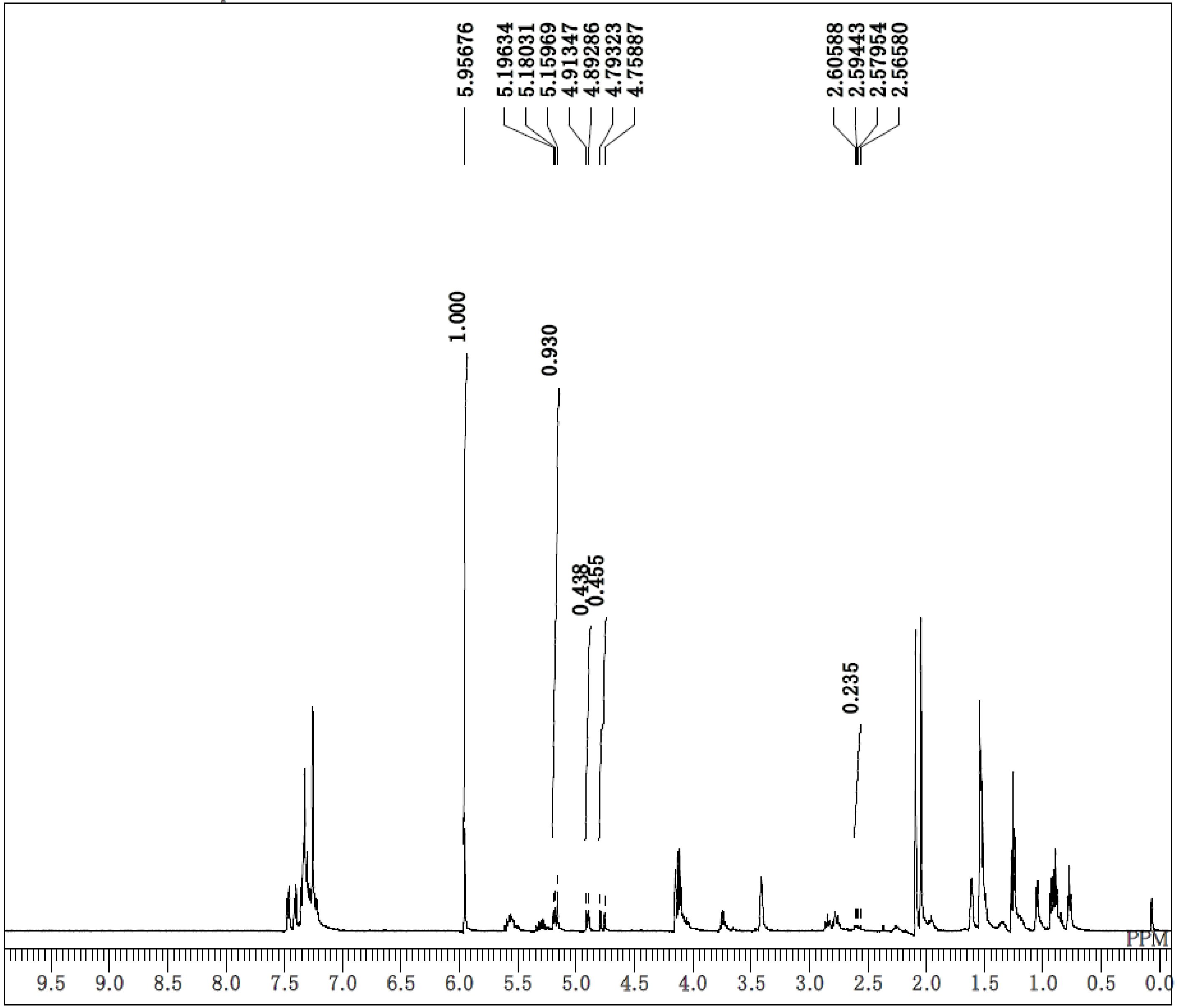


L5/1Pr, ***S***

(2nd trial)

63%, 1.8/1 dr, 4.0/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L6EtS1.als

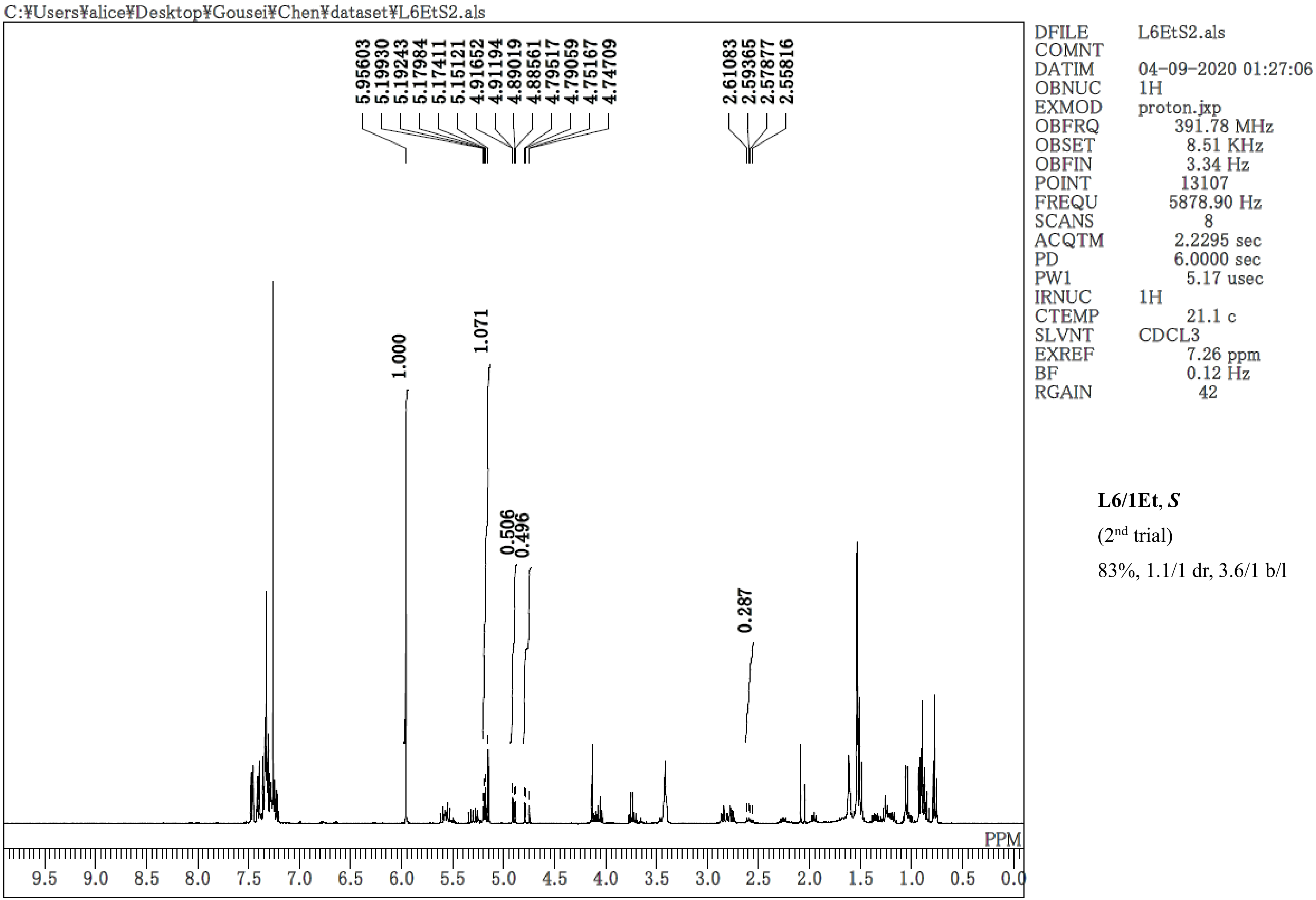


DFILE	L6EtS1.als
COMNT	
DATIM	2018-11-23 13:11:50
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L6/1Et, ***S***

(1st trial)

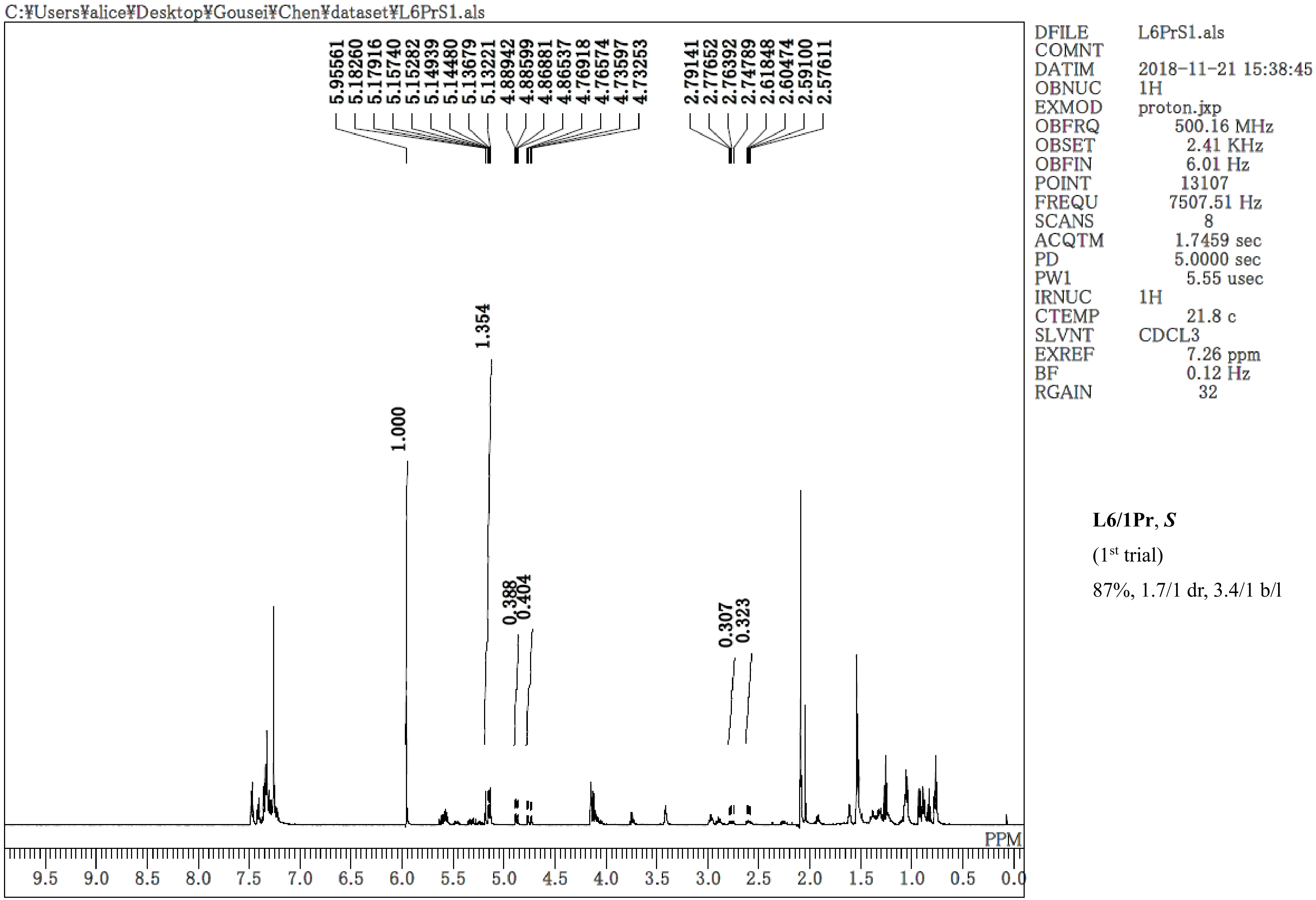
72%, 1.0/1 dr, 3.9/1 b/l



L6/1Et, ***S***

(2nd trial)

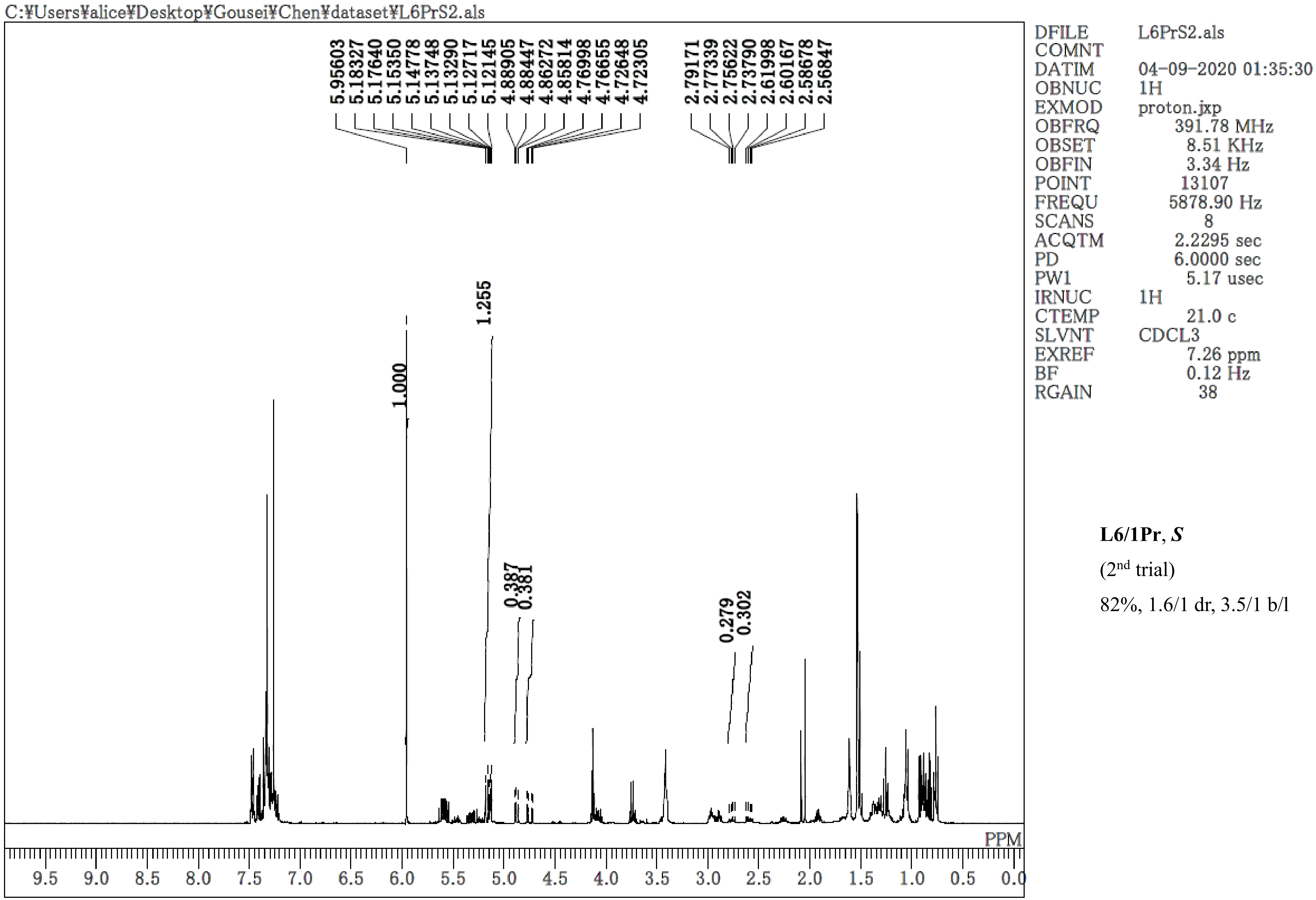
83%, 1.1/1 dr, 3.6/1 b/l



L6/1Pr, ***S***

(1st trial)

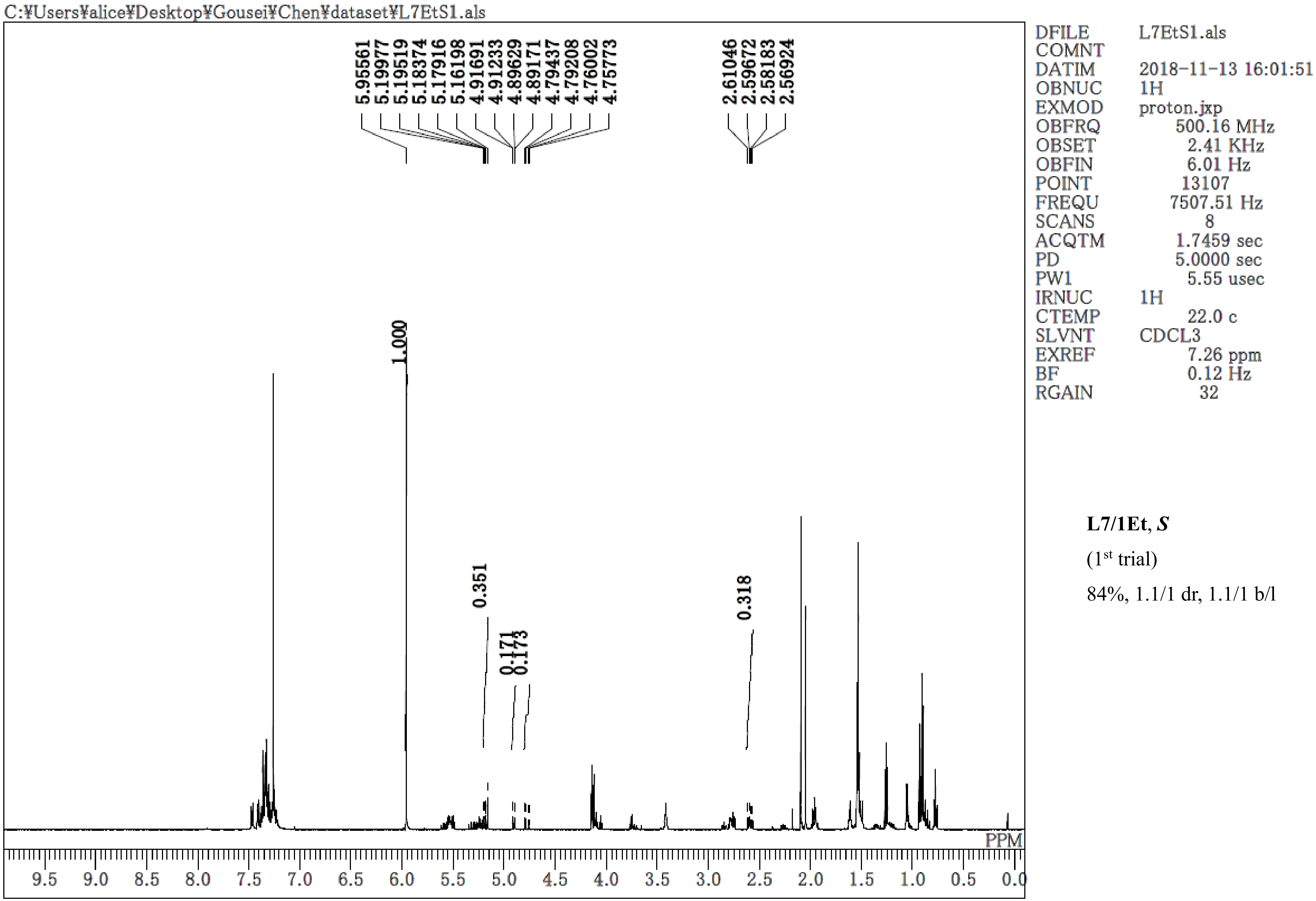
87%, 1.7/1 dr, 3.4/1 b/l



L6/1Pr, ***S***

(2^{nd} trial)

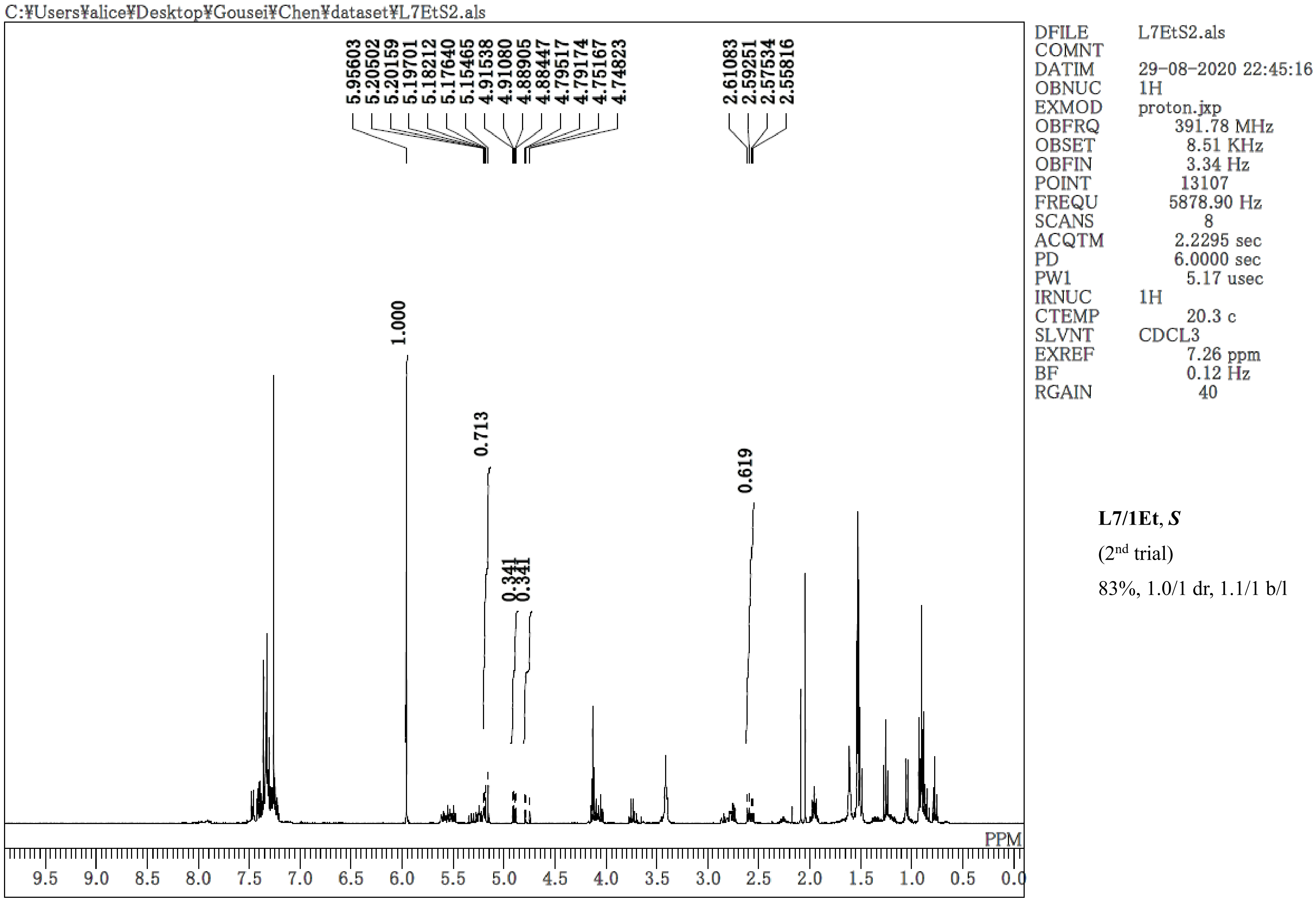
82%, 1.6/1 dr, 3.5/1 b/l



L7/1Et, ***S***

(1st trial)

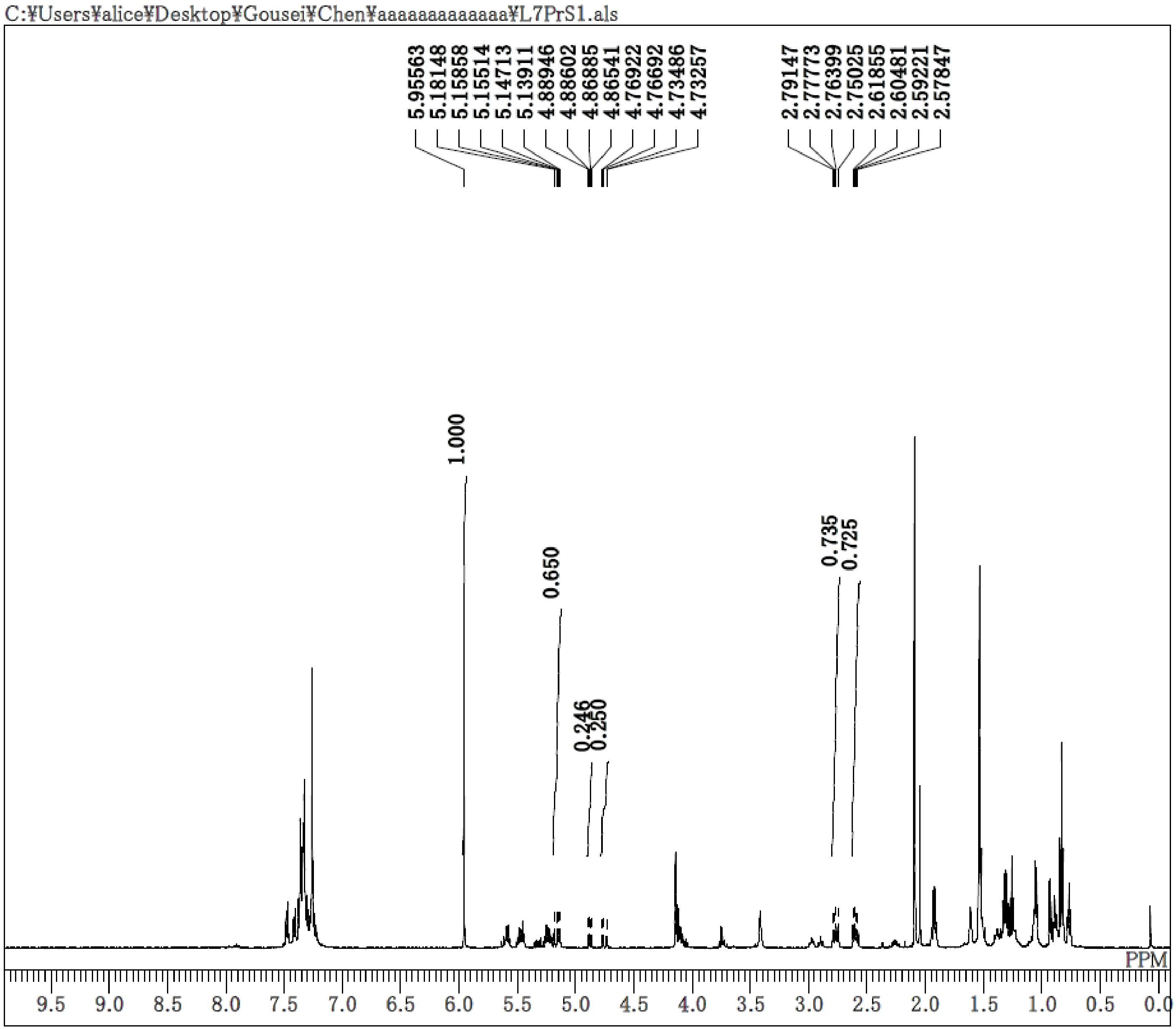
84%, 1.1/1 dr, 1.1/1 b/l



L7/1Et, ***S***

(2nd trial)

83%, 1.0/1 dr, 1.1/1 b/l



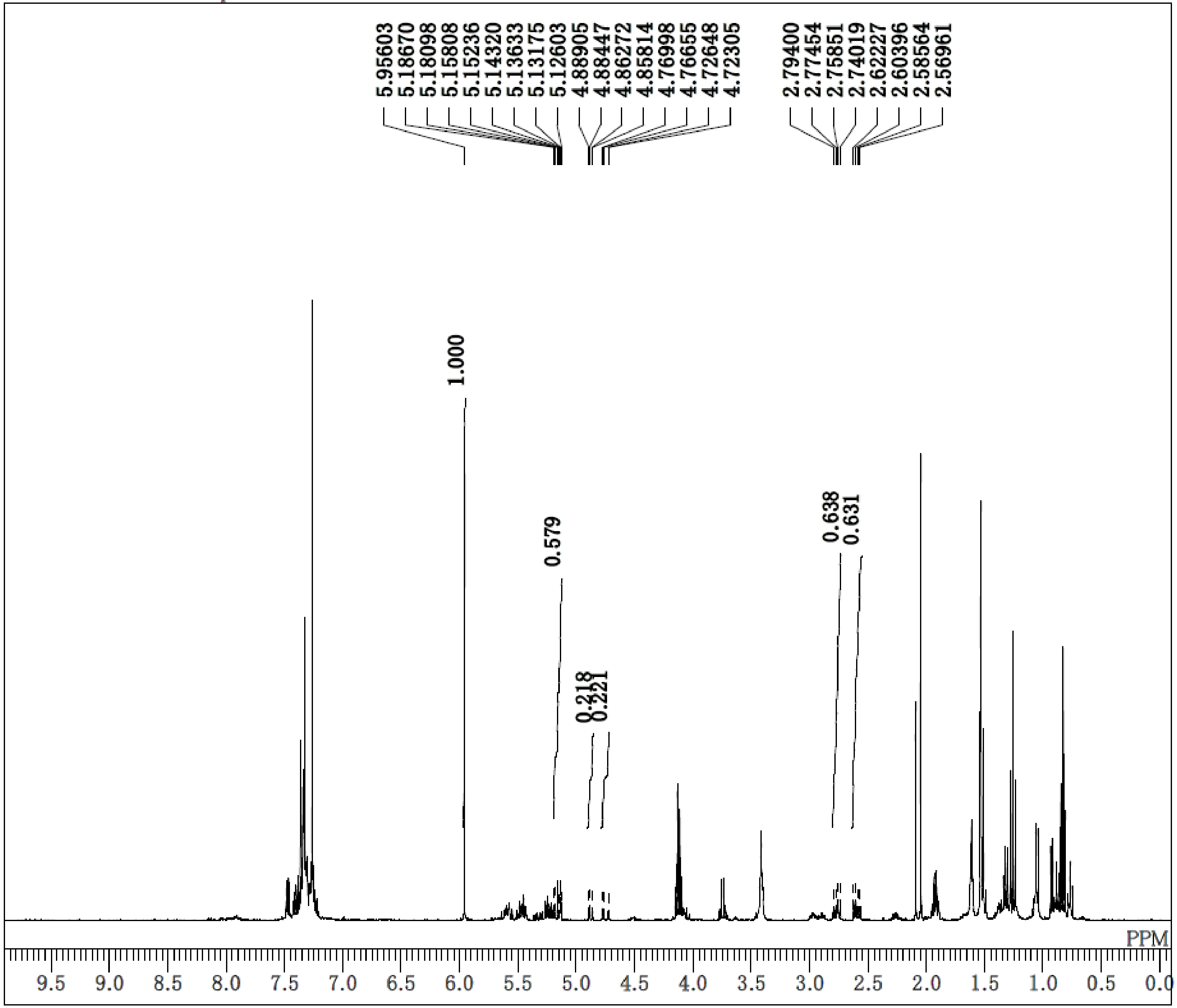
DFILE	L7PrS1.als
COMNT	
DATIM	2018-11-11 23:20:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L7/1Pr, *S*

(1st trial)

83%, 1.4/1 dr, 1/1.2 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L7PrS2.als

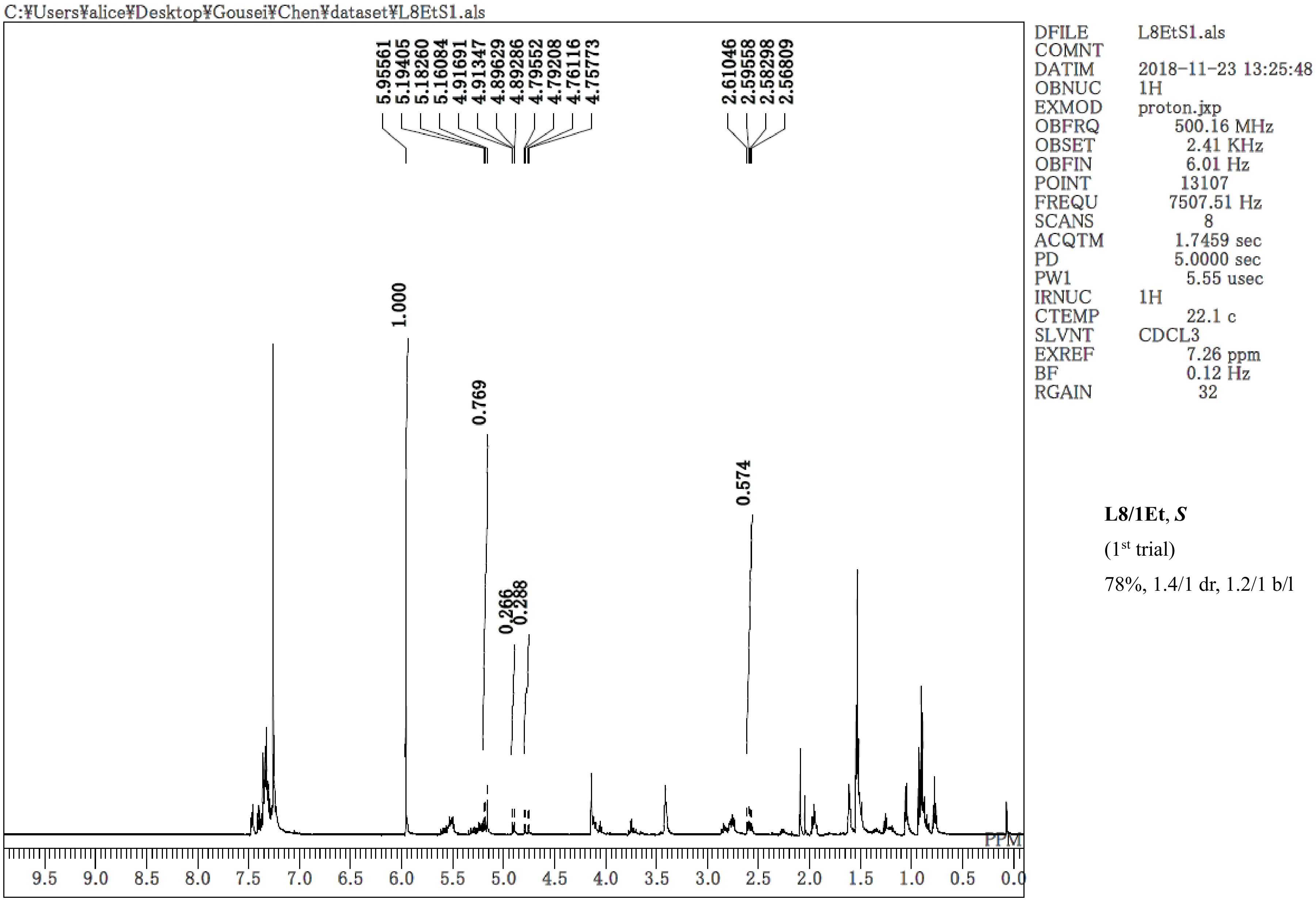


DFILE	L7PrS2.als
COMNT	
DATIM	29-08-2020 22:52:48
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L7/1Pr, ***S***

(2nd trial)

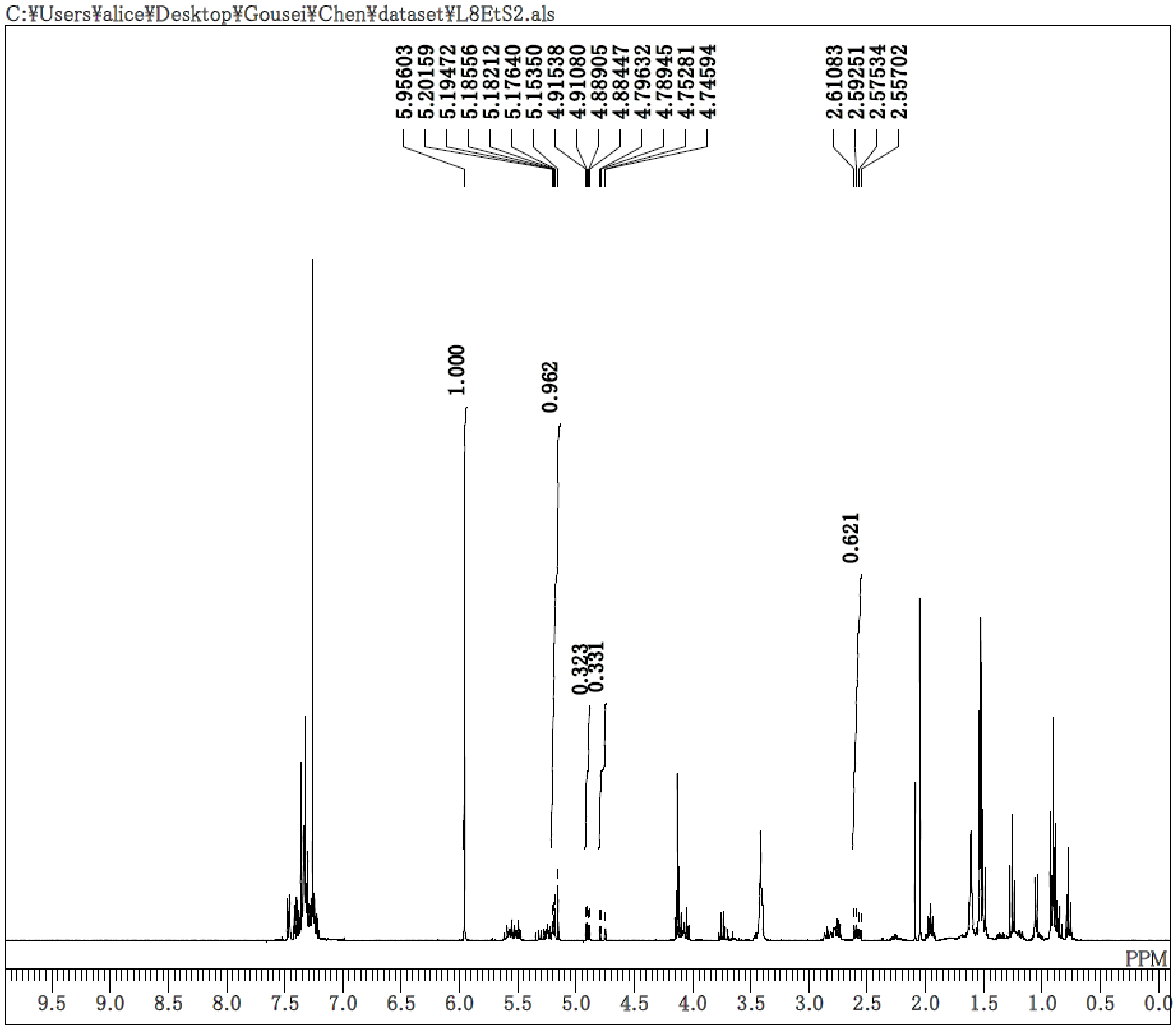
72%, 1.3/1 dr, 1/1.2 b/l



L8/1Et, ***S***

(1st trial)

78%, 1.4/1 dr, 1.2/1 b/l

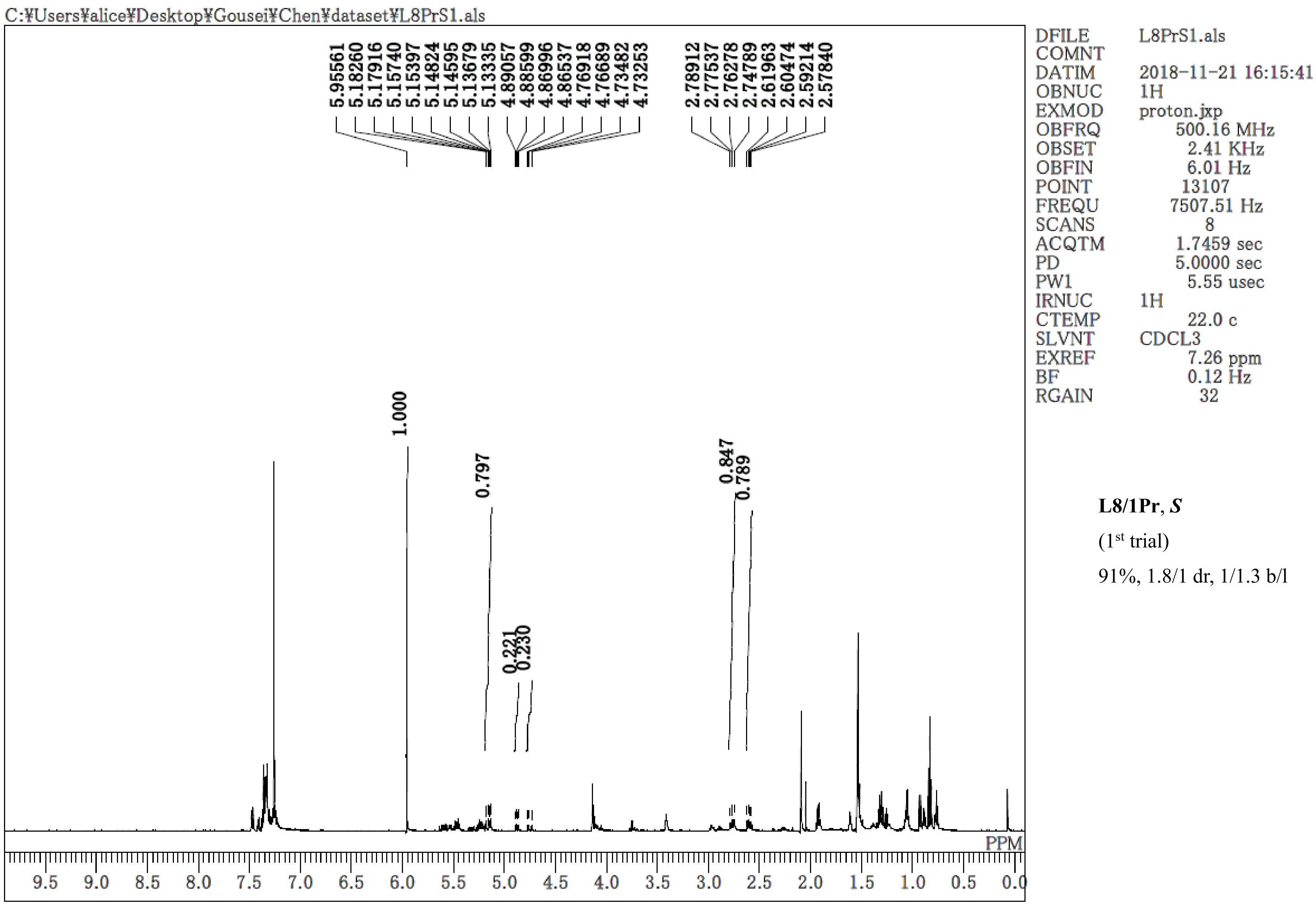


DFILE	L8EtS2.als
COMNT	
DATIM	23-08-2020 22:21:26
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L8/1Et, ***S***

(2^{nd} trial)

90%, 1.5/1 dr, 1.3/1 b/l

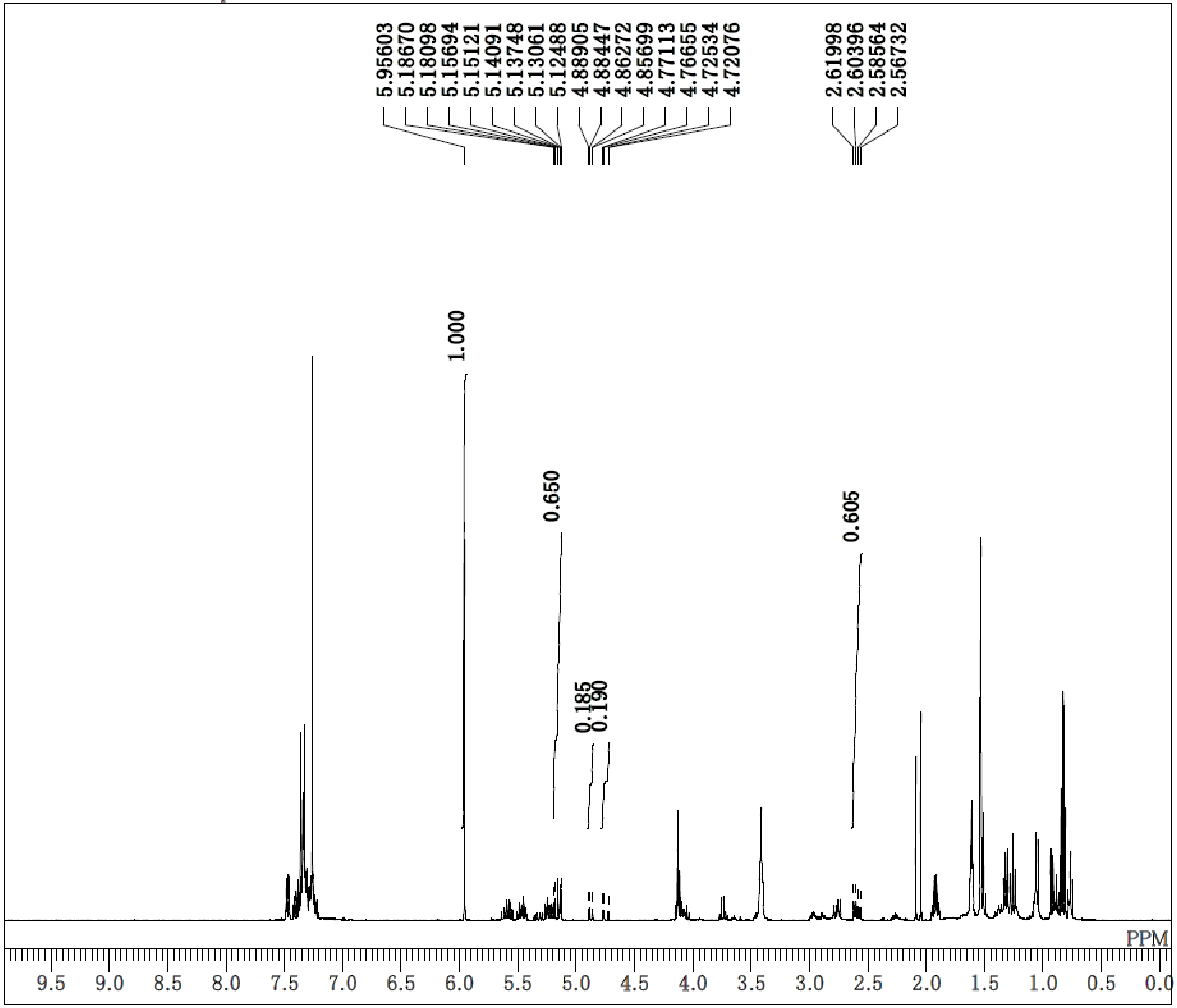


L8/1Pr, ***S***

(1st trial)

91%, 1.8/1 dr, 1/1.3 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L8PrS2.als

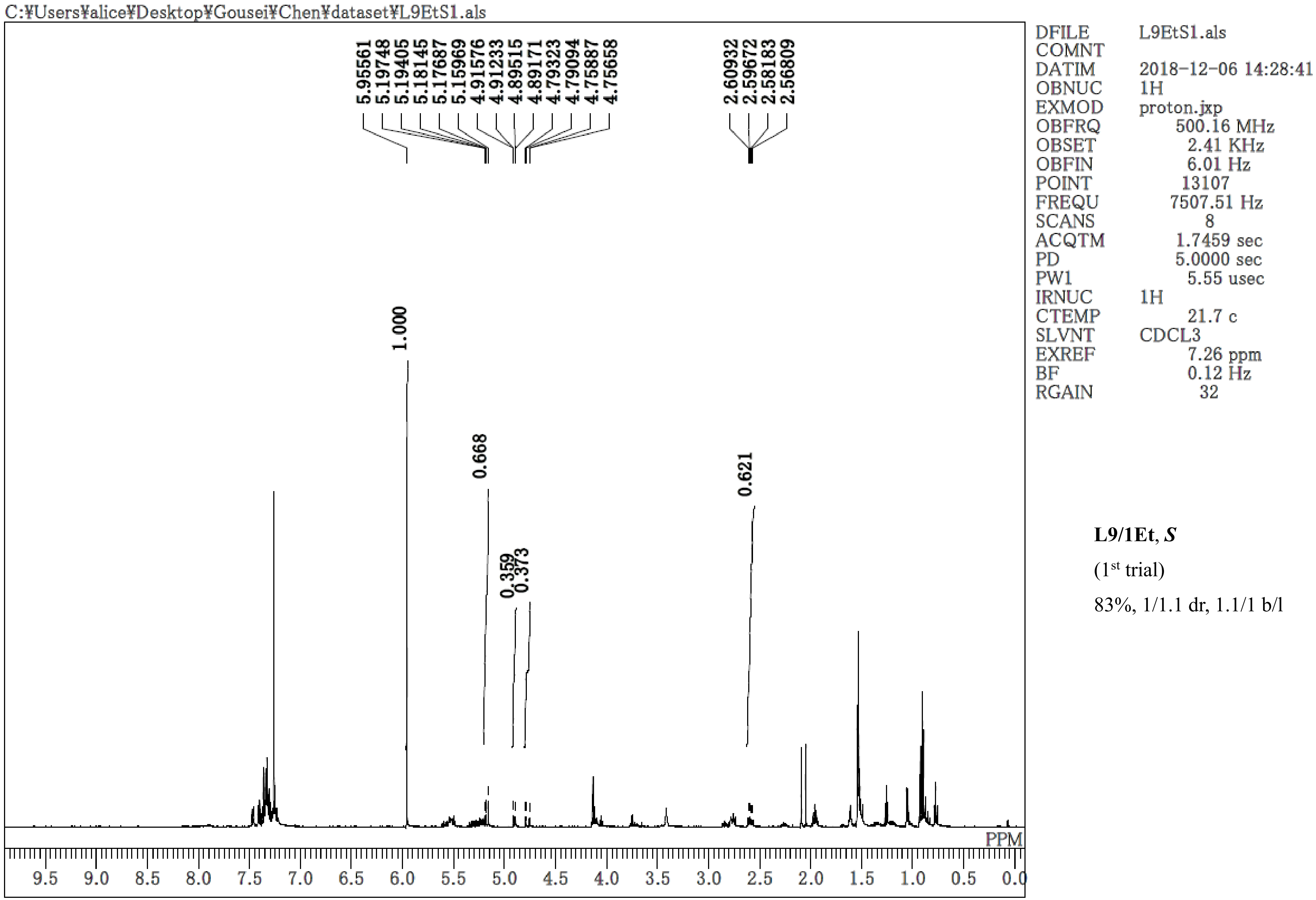


DFILE	L8PrS2.als
COMNT	
DATIM	23-08-2020 22:32:38
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L8/1Pr, ***S***

(2^{nd} trial)

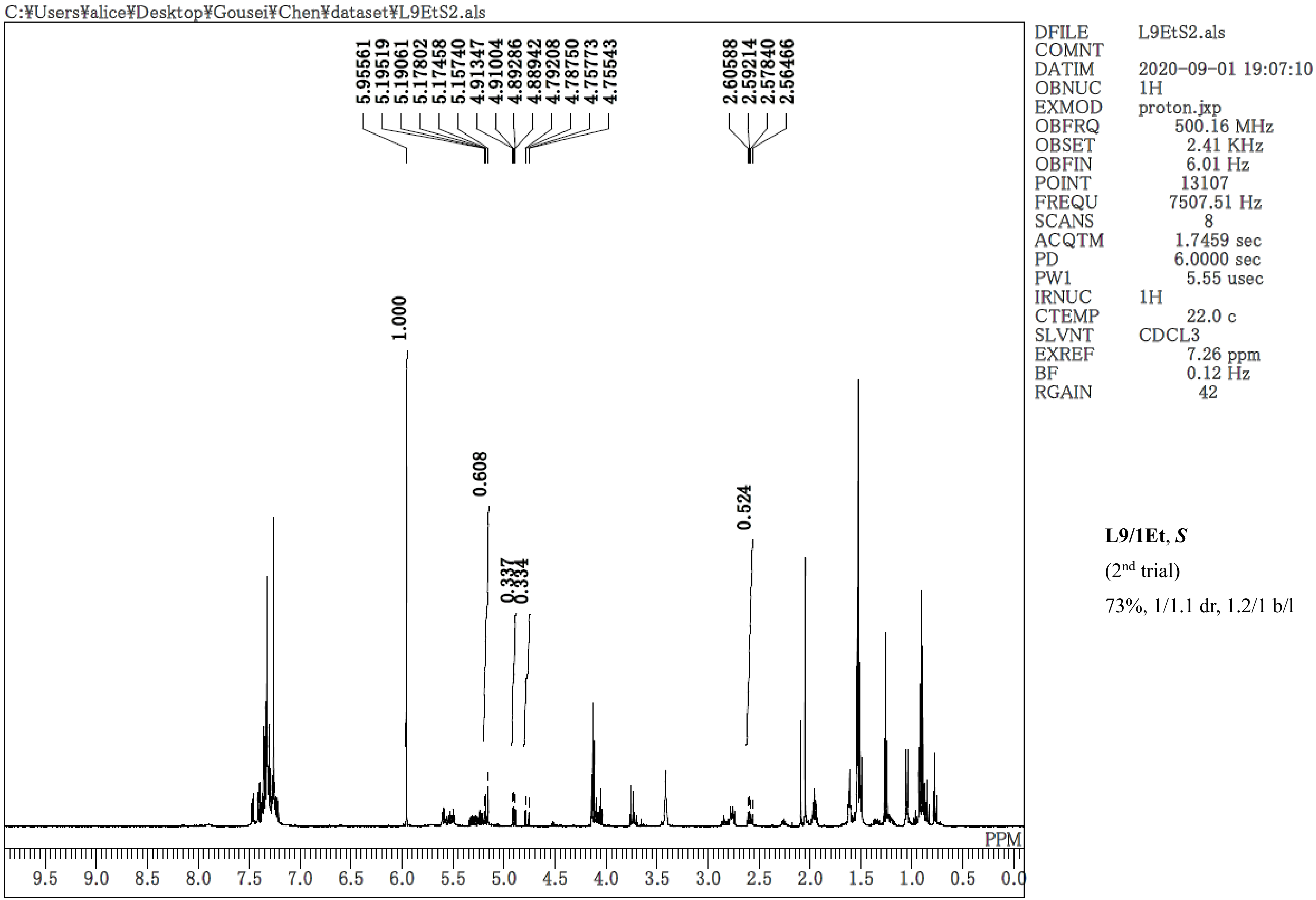
70%, 1.7/1 dr, 1/1.2 b/l



L9/1Et, ***S***

(1st trial)

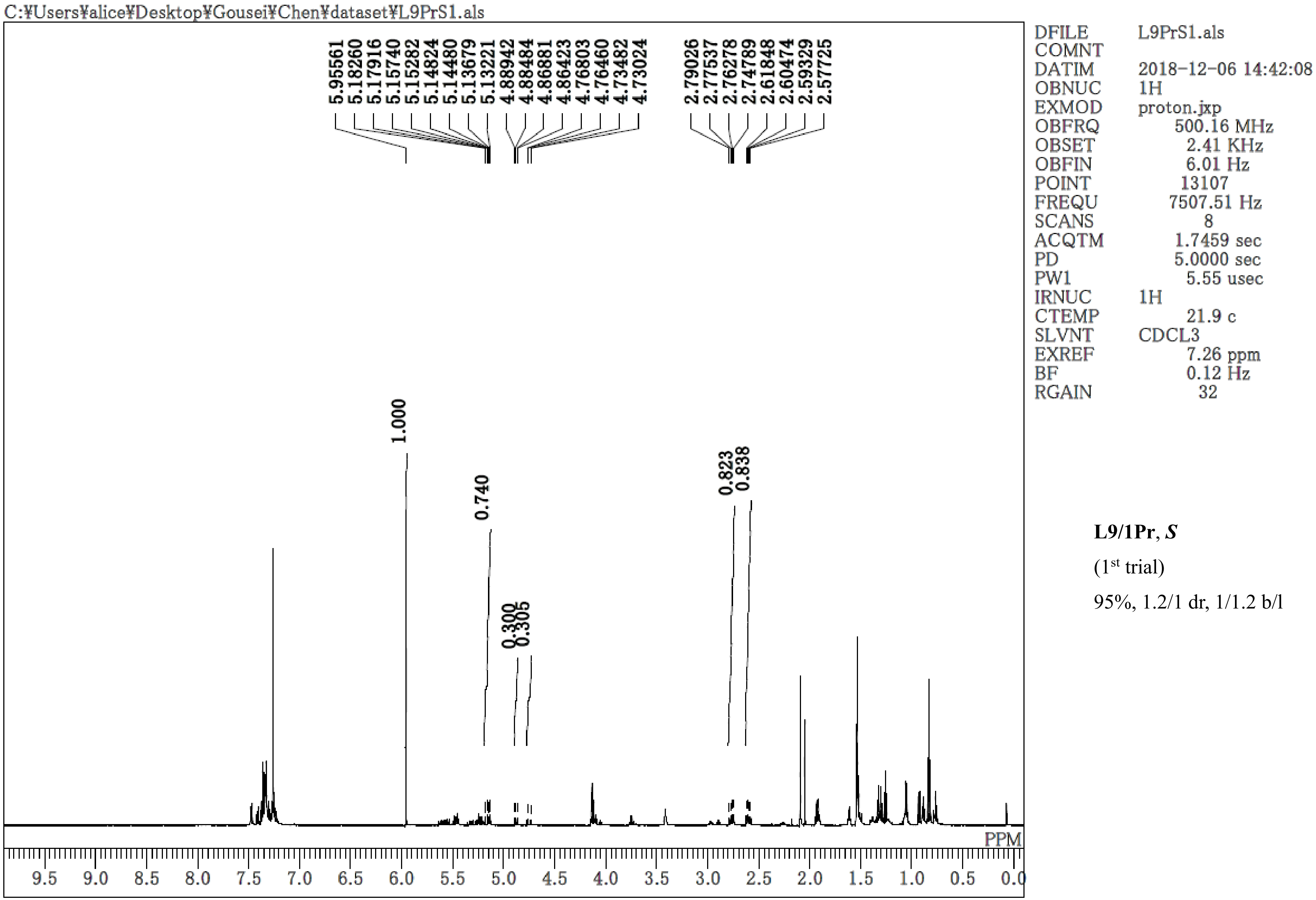
83%, 1/1.1 dr, 1.1/1 b/l



L9/1Et, *S*

(2nd trial)

73%, 1/1.1 dr, 1.2/1 b/l

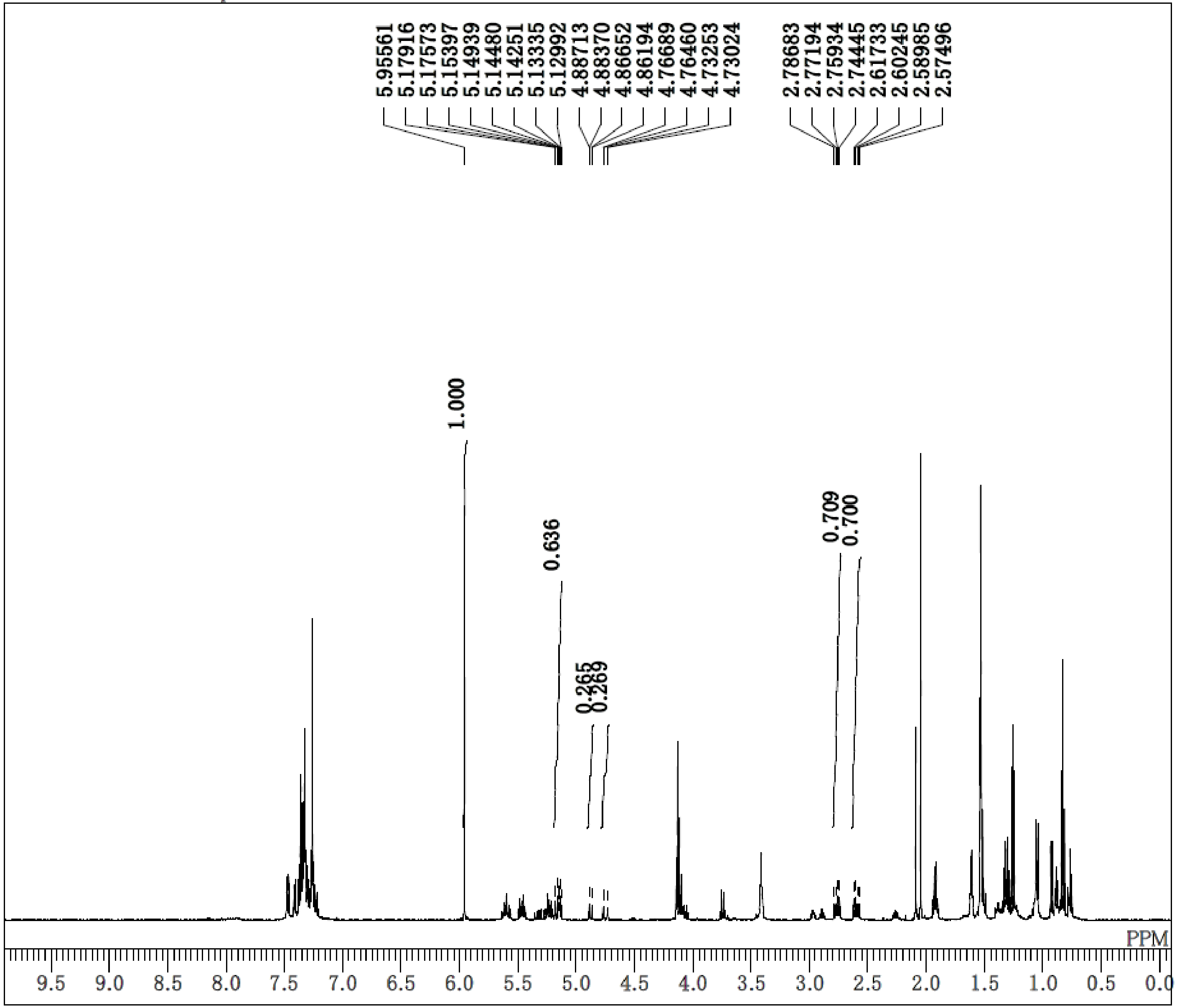


L9/1Pr, ***S***

(1st trial)

95%, 1.2/1 dr, 1/1.2 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L9PrS2.als

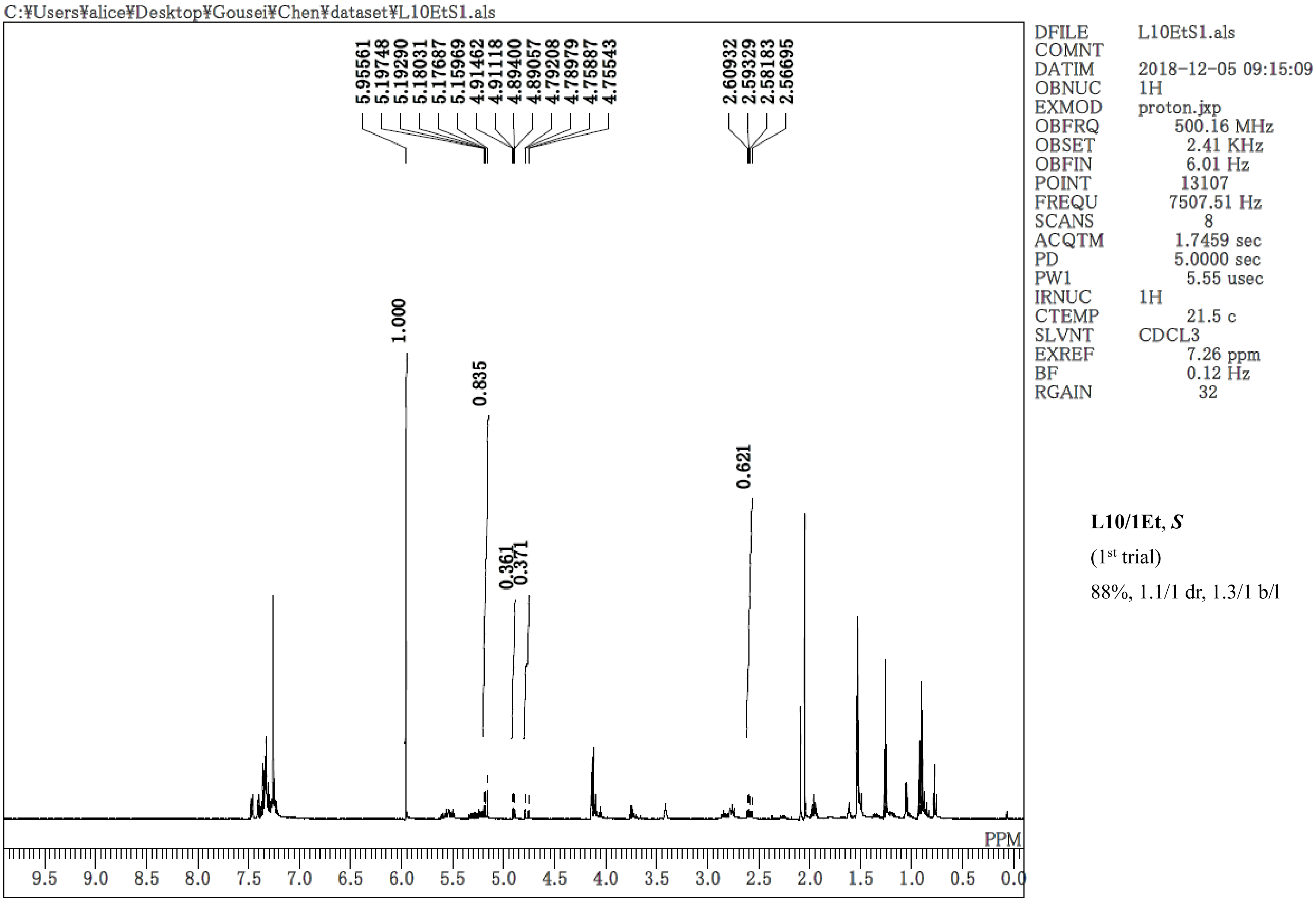


DFILE	L9PrS2.als
COMNT	
DATIM	2020-09-01 19:14:20
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L9/1Pr, ***S***

(2nd trial)

81%, 1.2/1 dr, 1/1.2 b/l

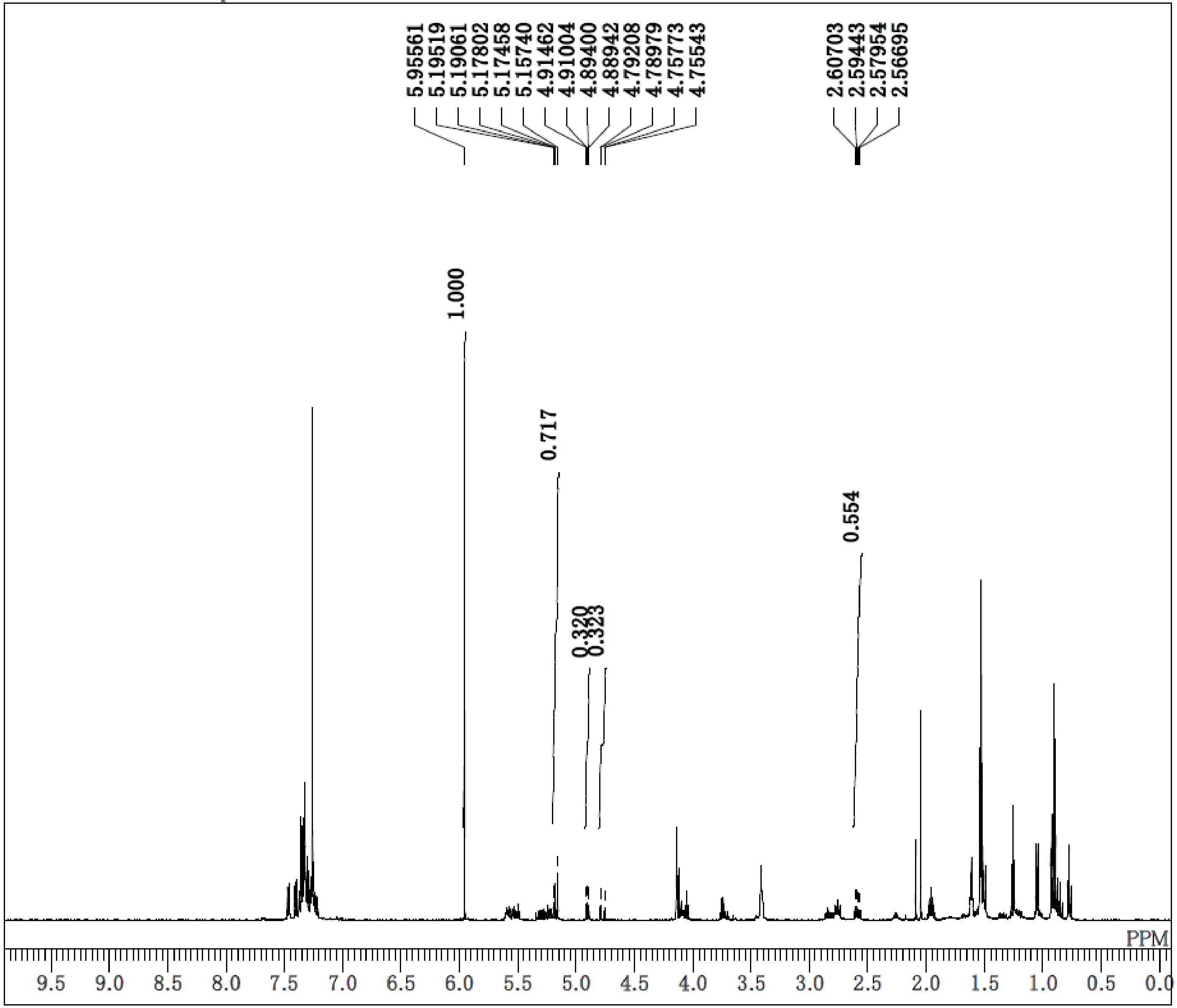


L10/1Et, ***S***

(1st trial)

88%, 1.1/1 dr, 1.3/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L10EtS2.als



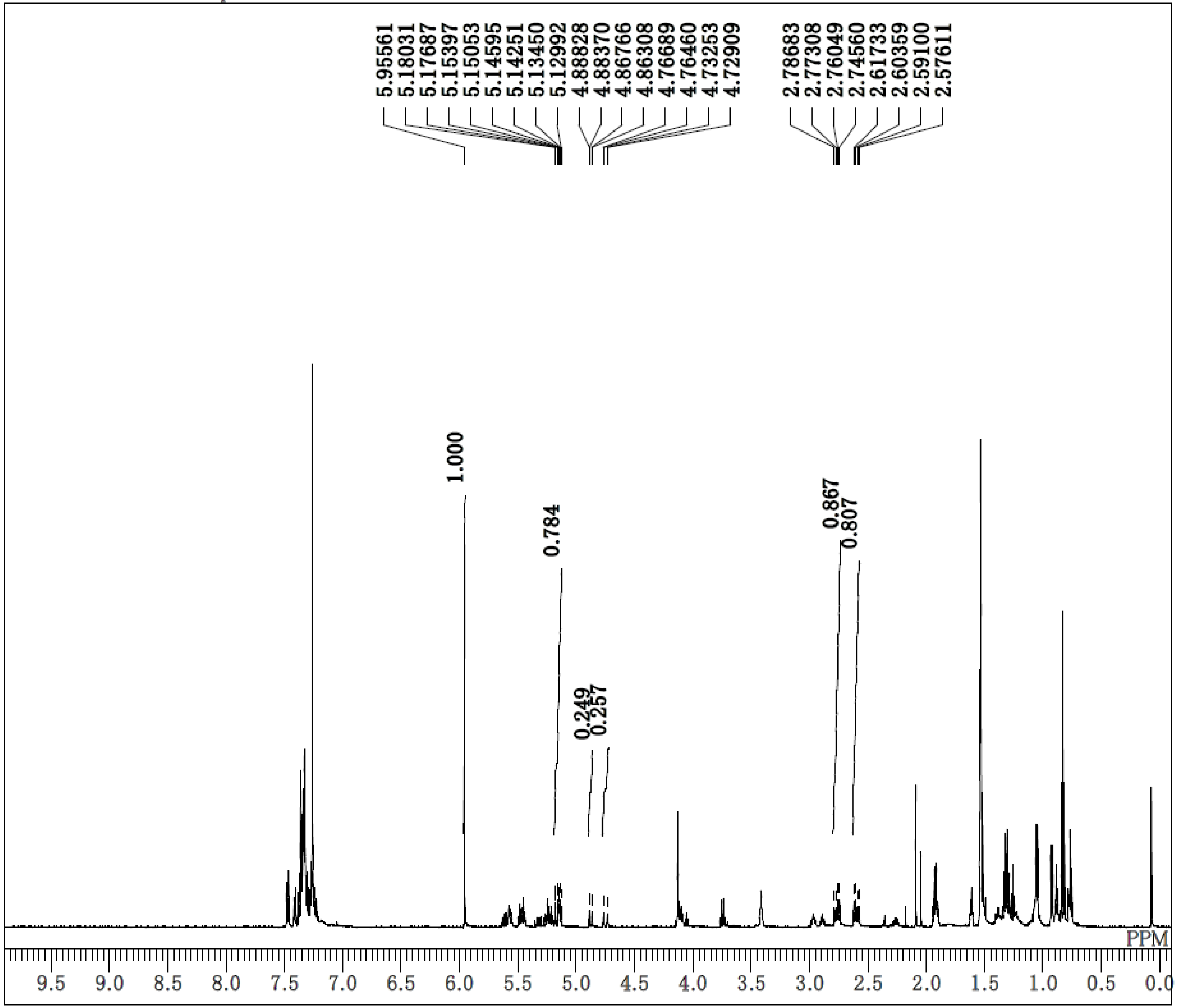
DFILE	L10EtS2.als
COMNT	
DATIM	2020-09-01 23:04:01
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L10/1Et, ***S***

(2nd trial)

78%, 1.1/1 dr, 1.2/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L10PrS1.als

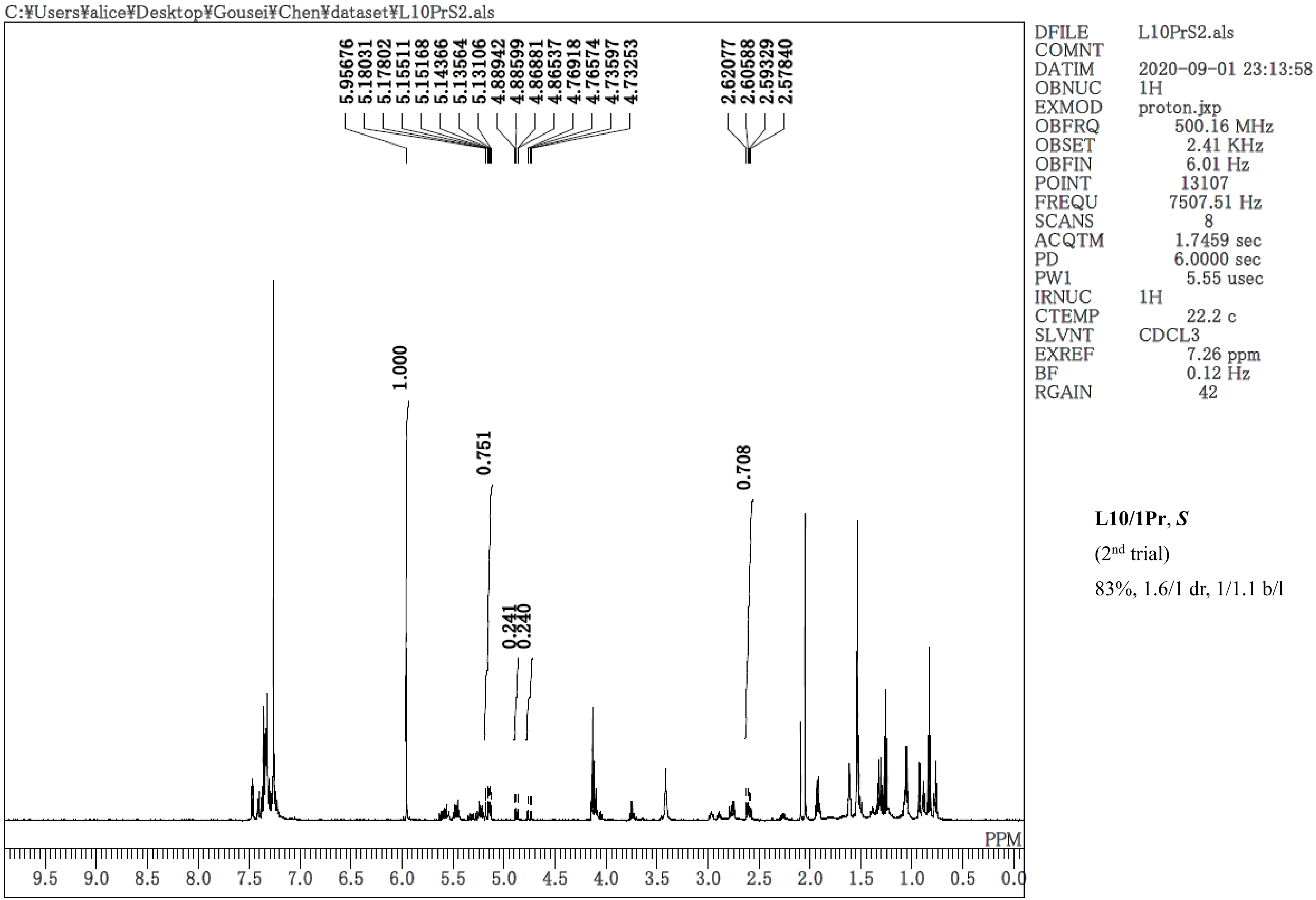


DFILE	L10PrS1.als
COMNT	
DATIM	2018-12-03 17:32:44
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L10/1Pr, ***S***

(1st trial)

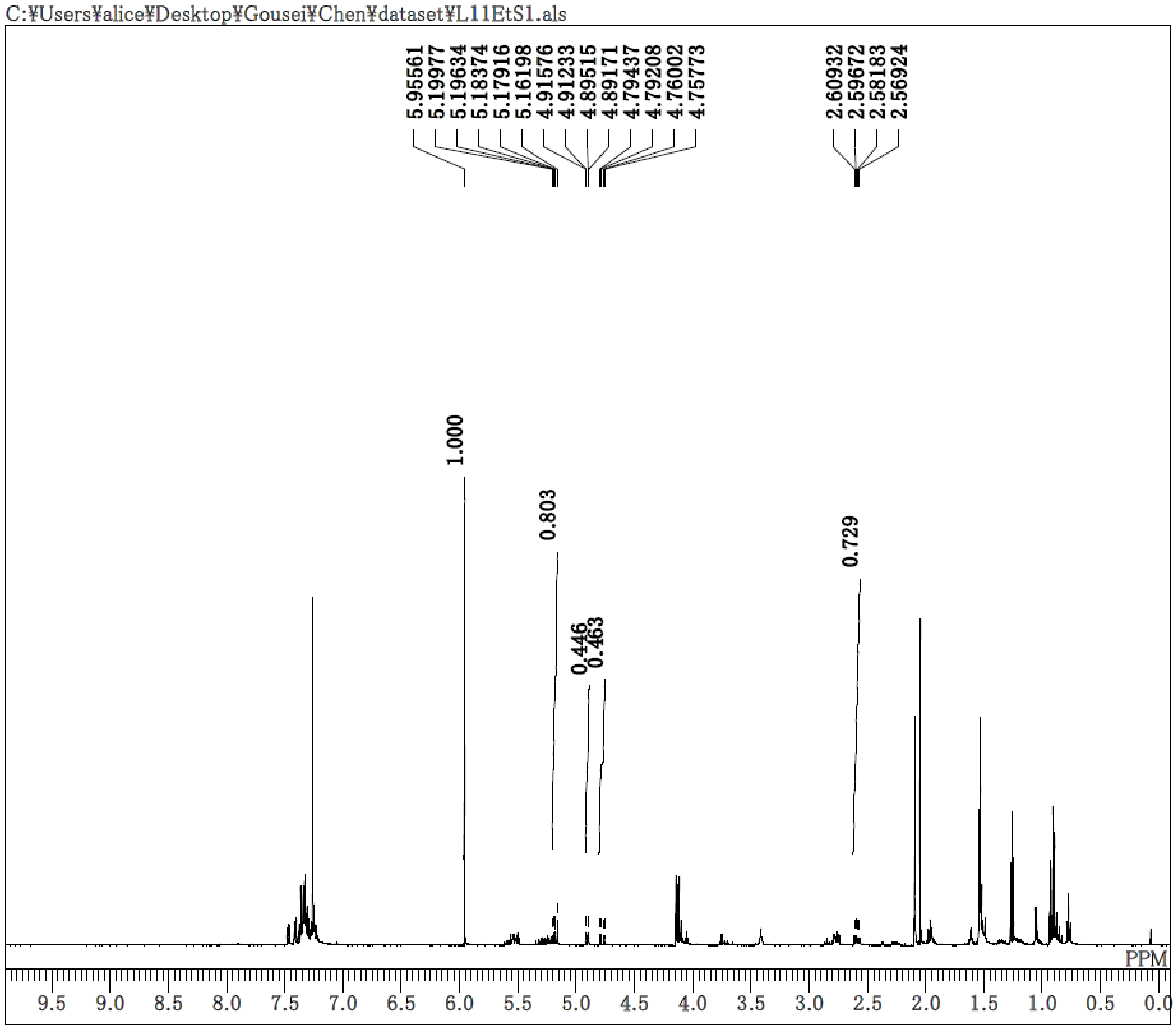
93%, 1.5/1 dr, 1/1.3 b/l



L10/1Pr, ***S***

(2nd trial)

83%, 1.6/1 dr, 1/1.1 b/l

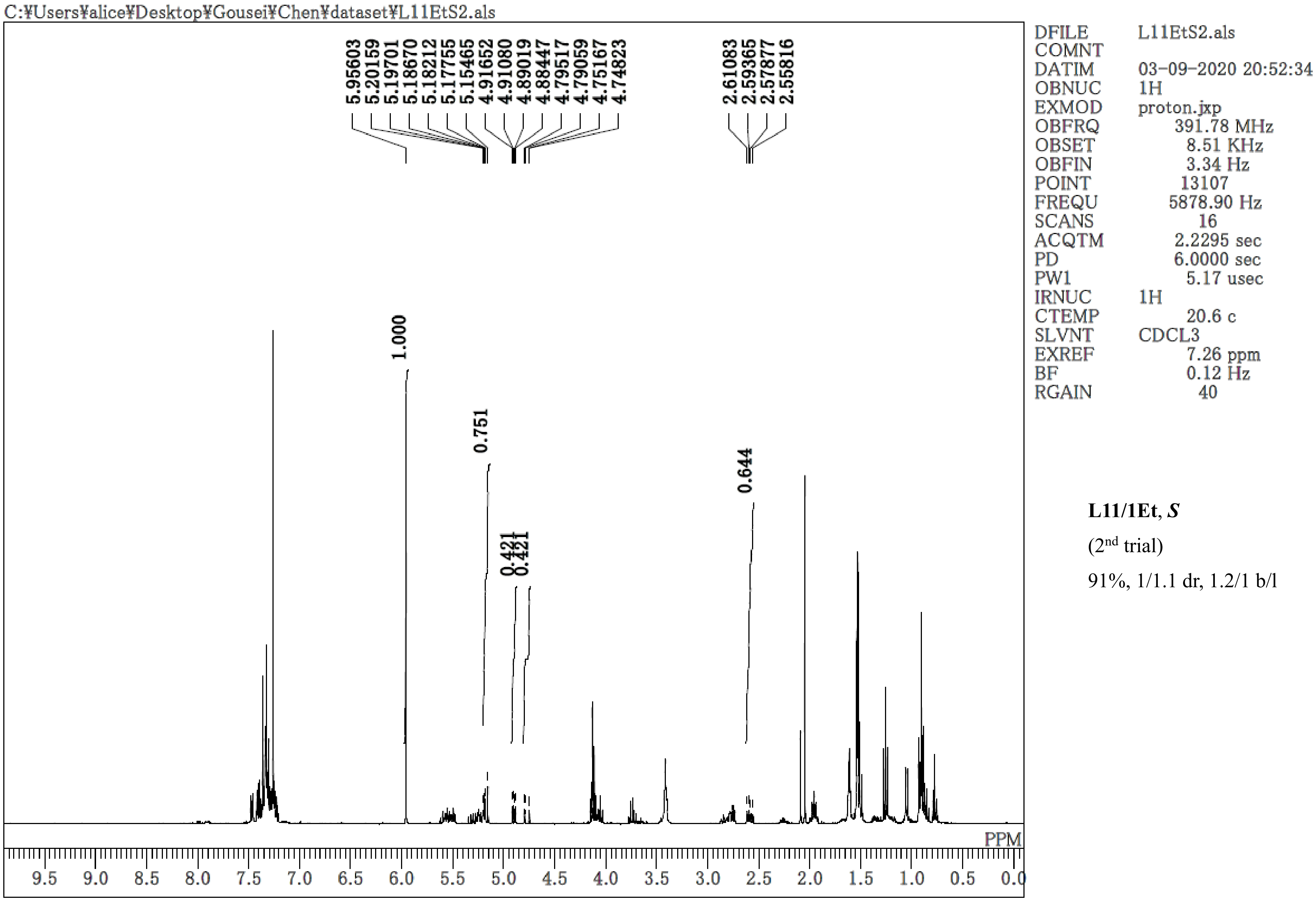


DFILE	L11EtS1.als
COMNT	
DATIM	2018-12-05 09:27:38
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L11/1Et, ***S***

(1^{st} trial)

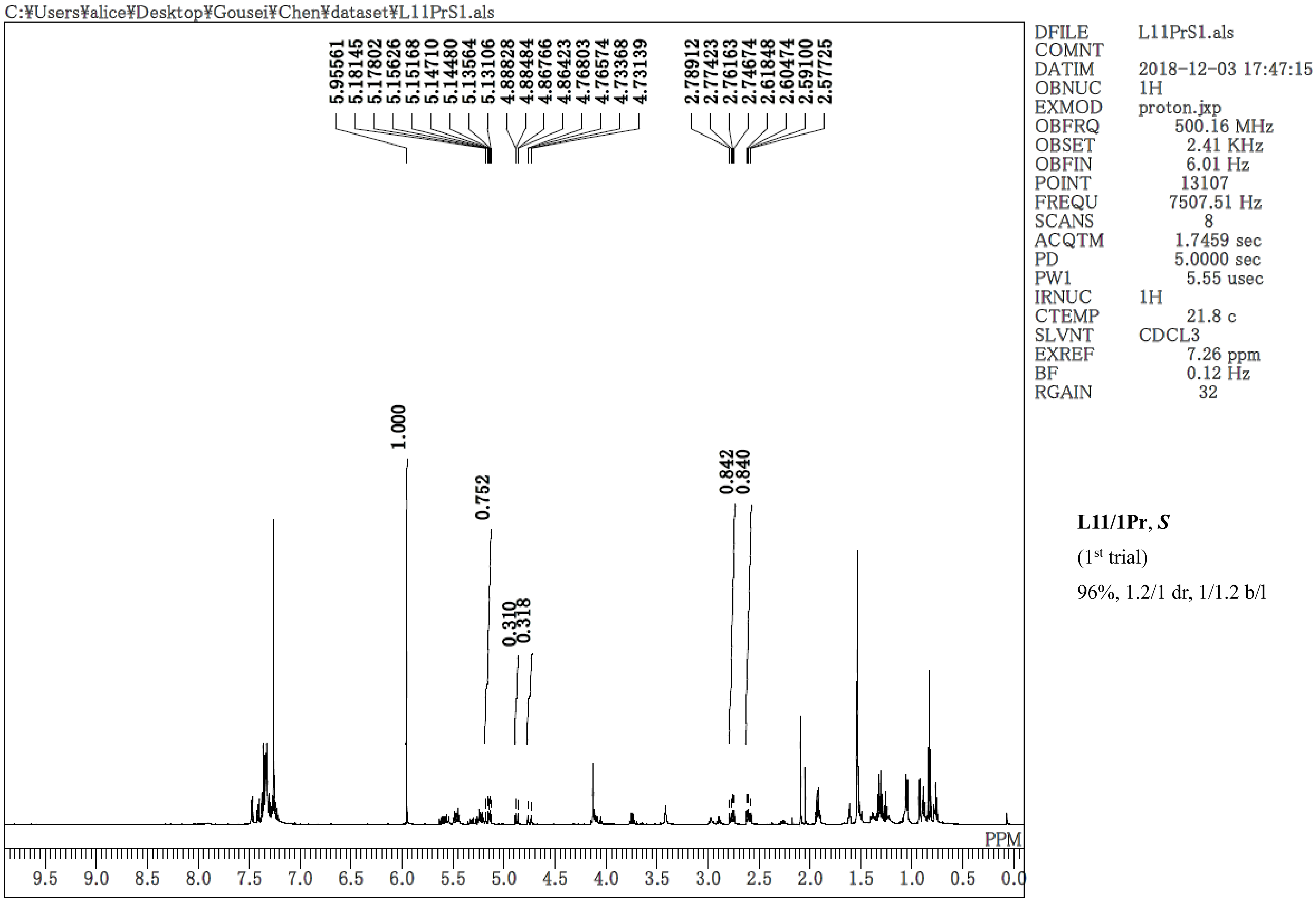
100%, 1/1.1 dr, 1.2/1 b/l



L11/1Et, ***S***

(2nd trial)

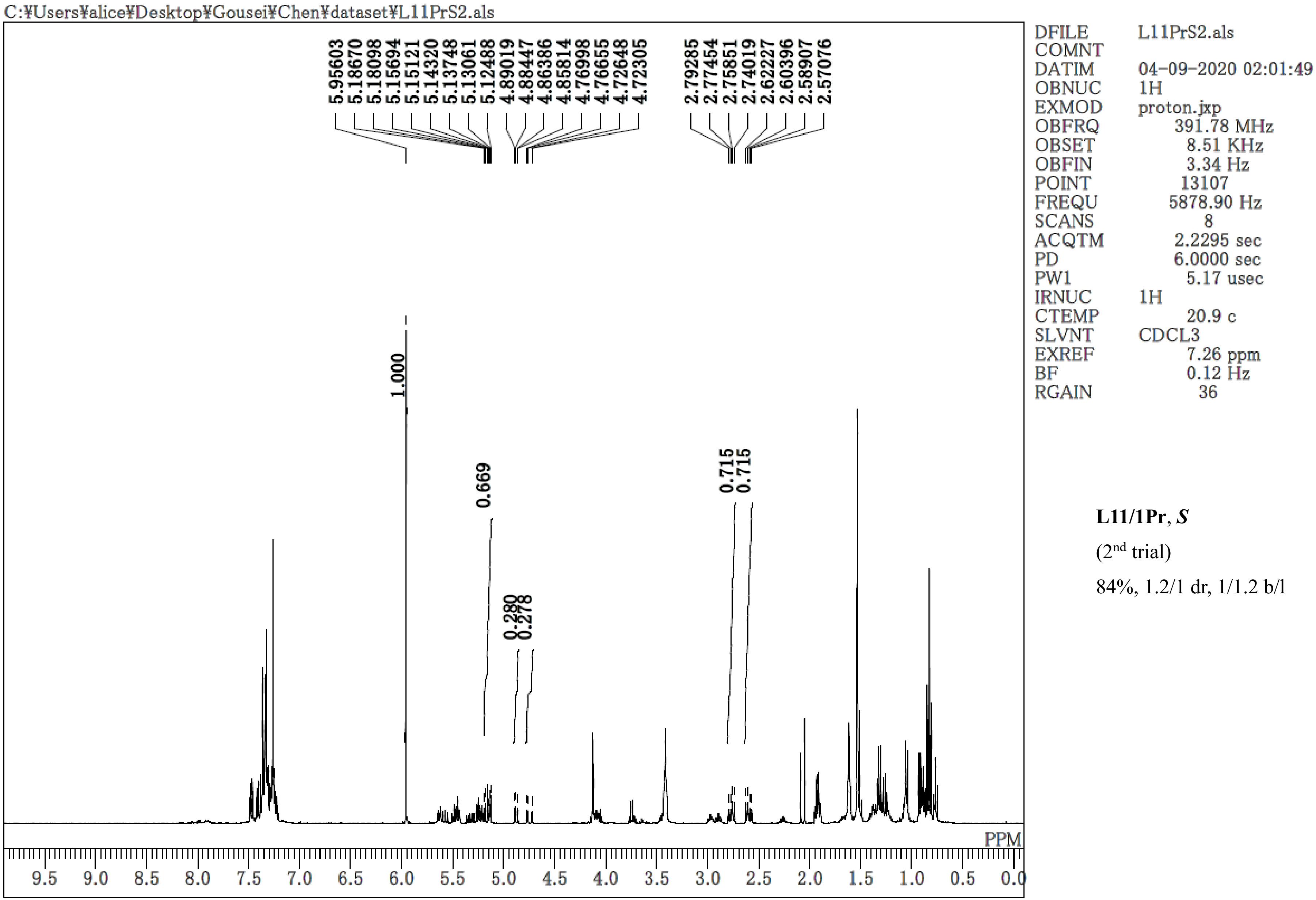
91%, 1/1.1 dr, 1.2/1 b/l



L11/1Pr, ***S***

(1st trial)

96%, 1.2/1 dr, 1/1.2 b/l

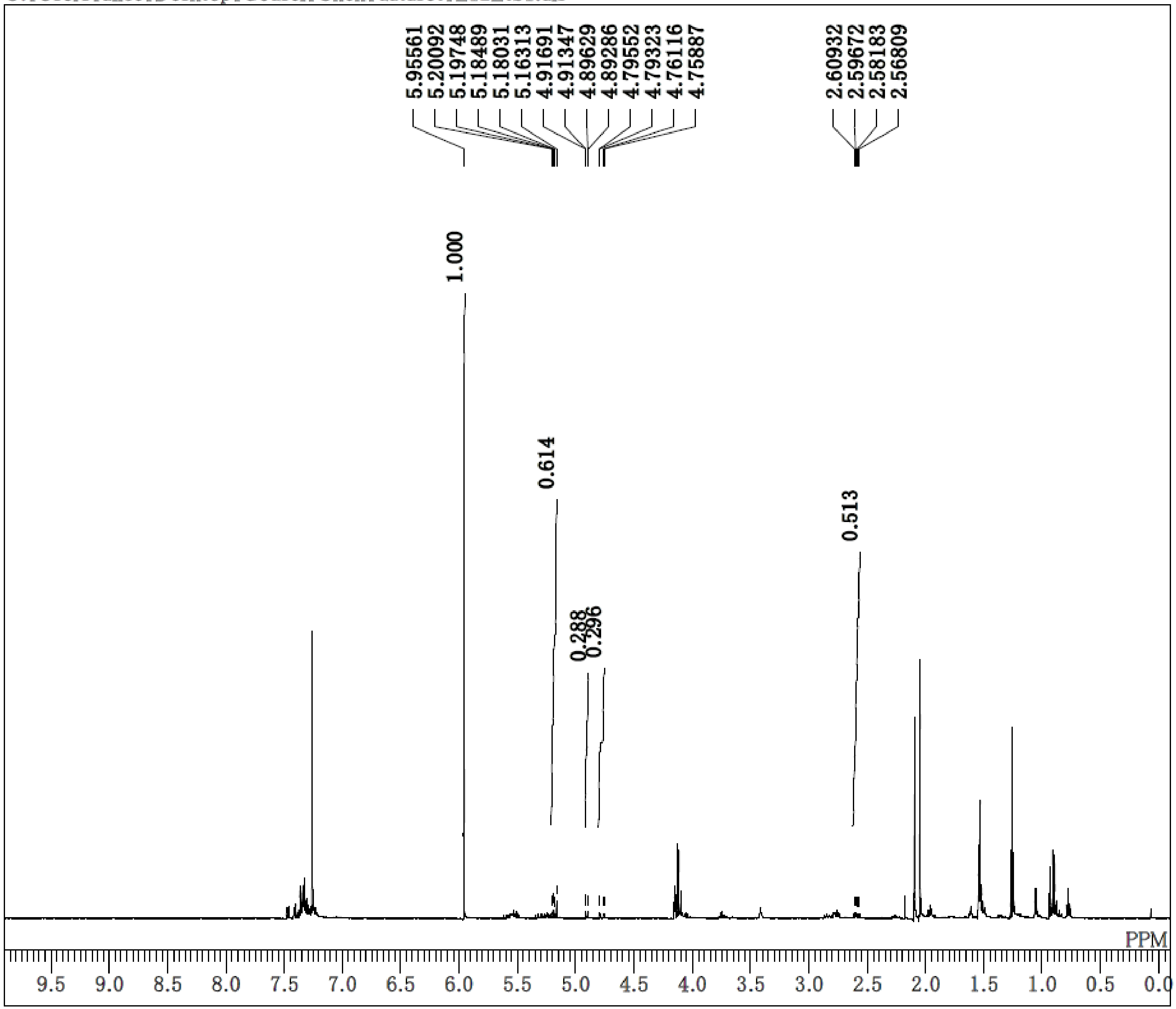


L11/1Pr, ***S***

(2nd trial)

84%, 1.2/1 dr, 1/1.2 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L12EtS1.als

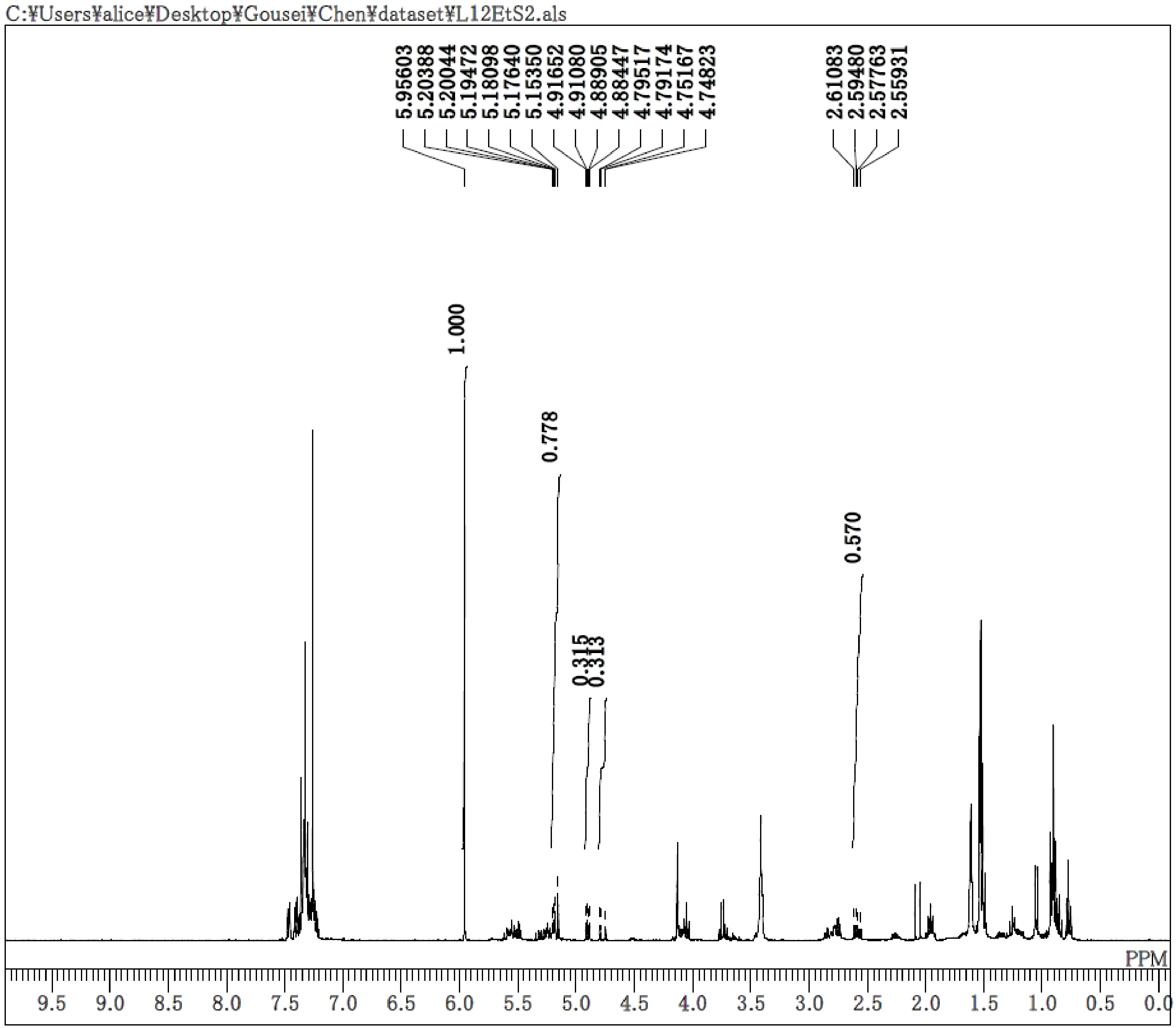


DFILE L12EtS1.als
COMNT
DATIM 2018-12-05 09:40:24
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 32

L12/1Et, ***S***

(1st trial)

84%, 1.1/1 dr, 1.2/1 b/l



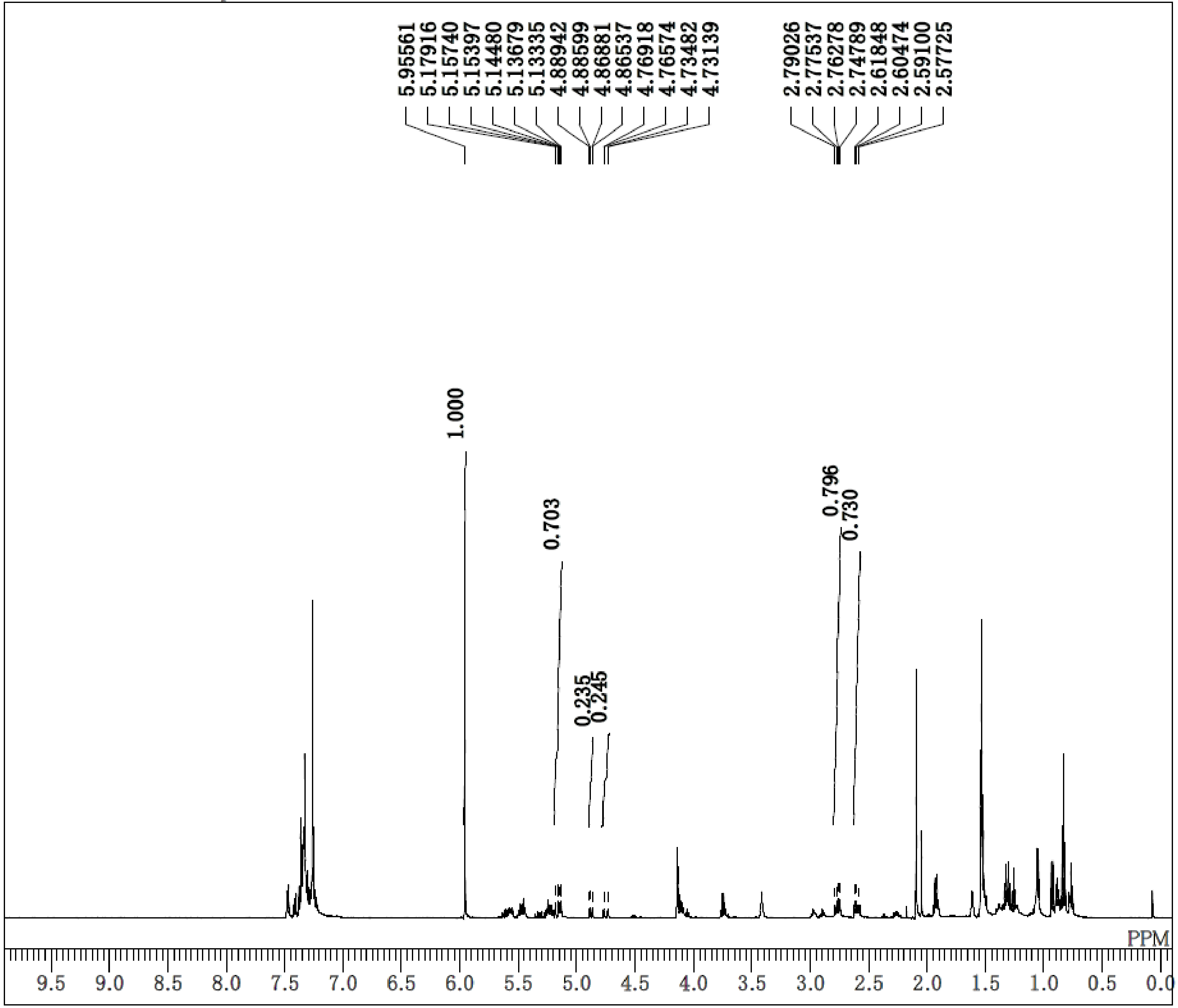
DFILE	L12EtS2.als
COMNT	
DATIM	23-08-2020 21:36:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L12/1Et, ***S***

(2nd trial)

80%, 1.2/1 dr, 1.2/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L12PrS1.als

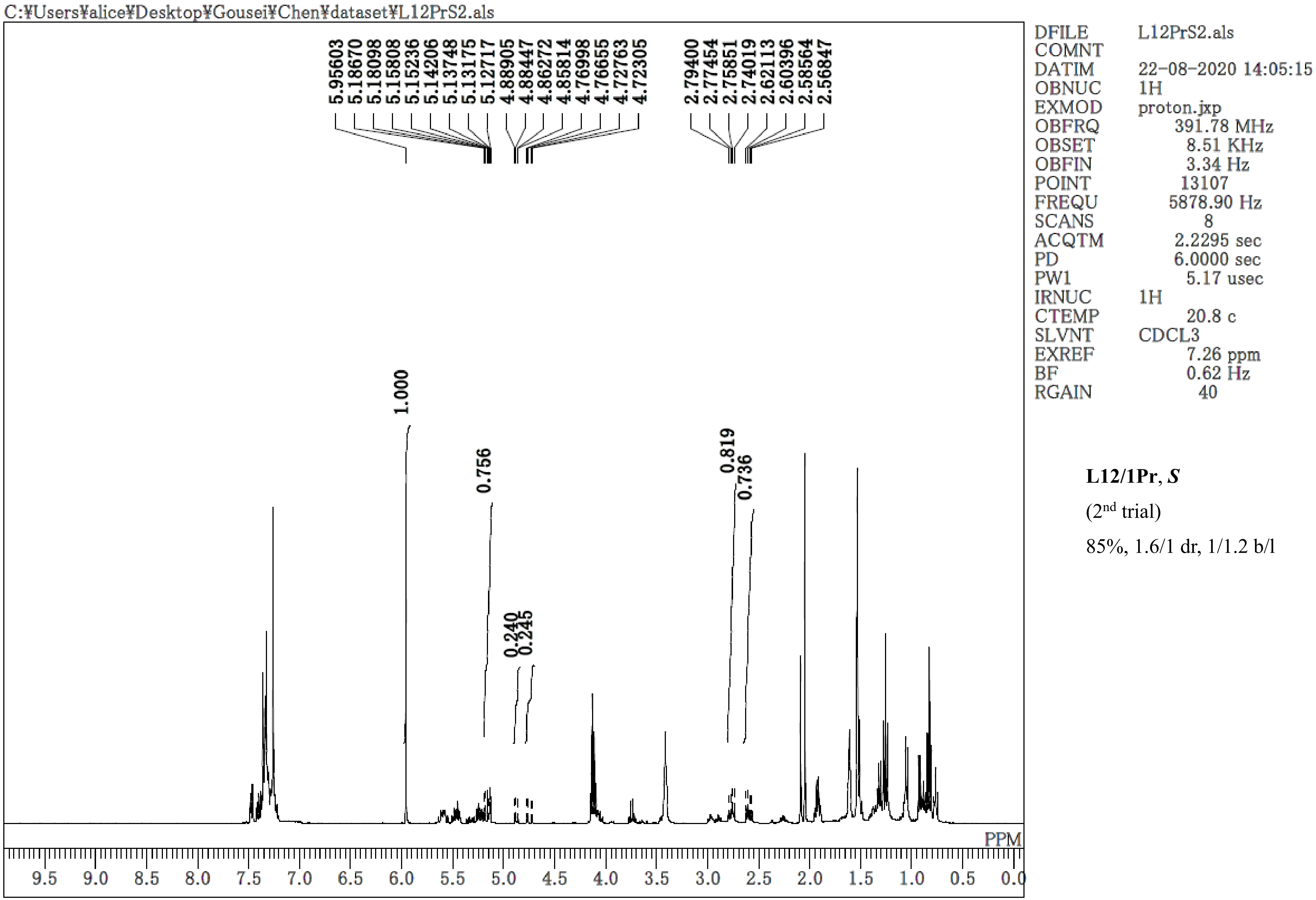


DFILE	L12PrS1.als
COMNT	
DATIM	2018-12-03 18:00:56
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	32

L12/1Pr, ***S***

(1st trial)

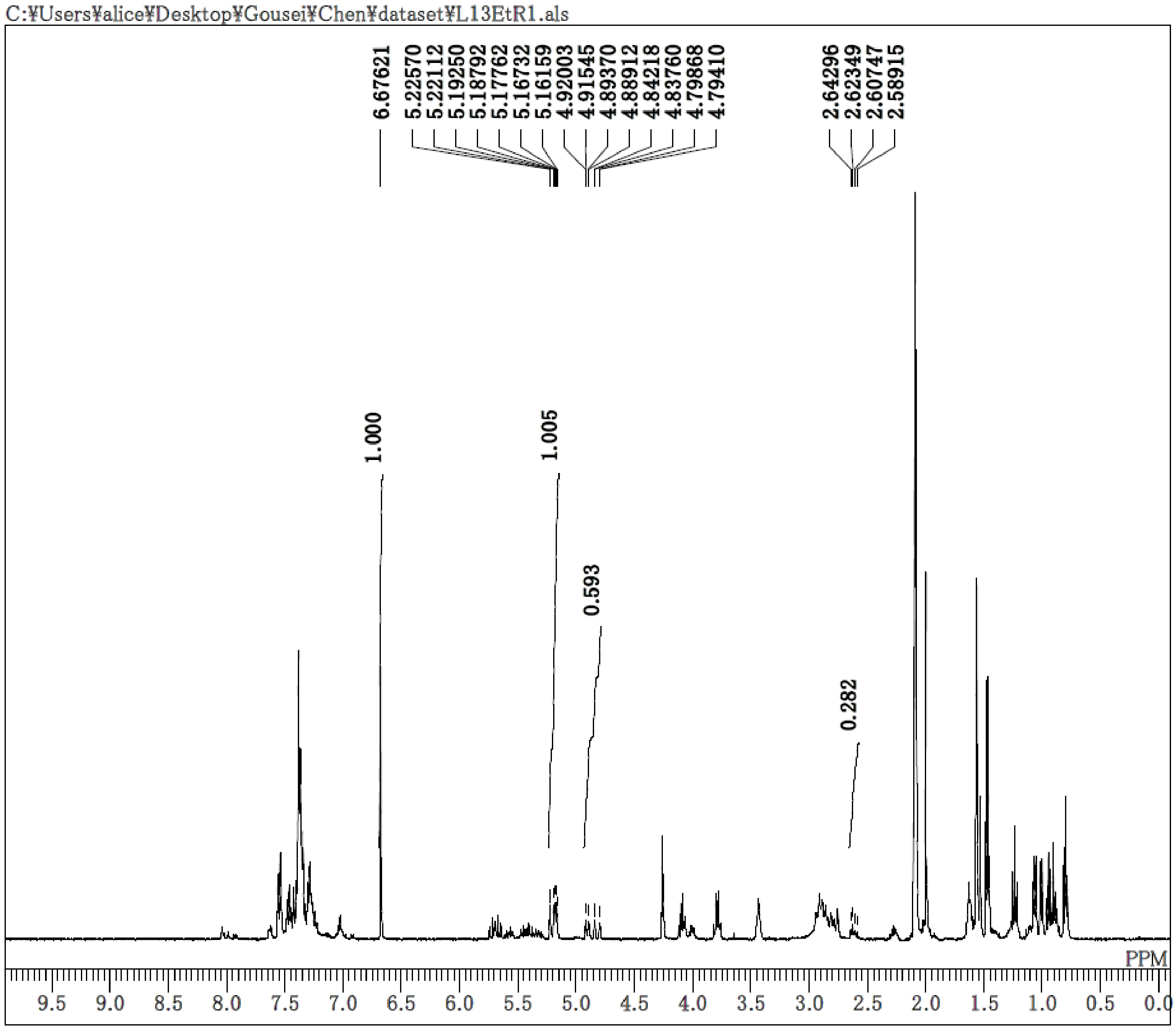
85%, 1.5/1 dr, 1/1.3 b/l



L12/1Pr, ***S***

(2nd trial)

85%, 1.6/1 dr, 1/1.2 b/l



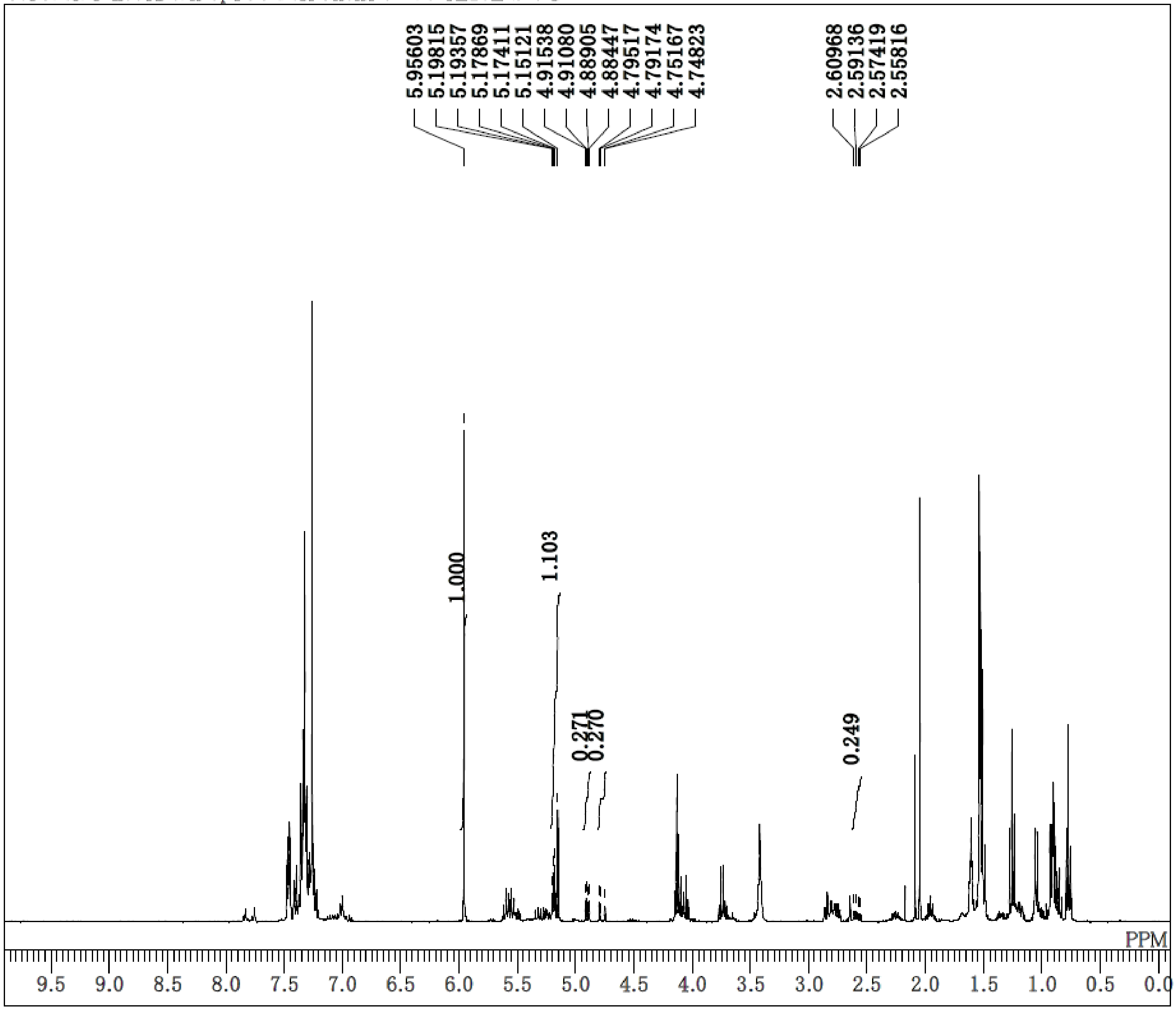
DFILE	L13EtR1.als
COMNT	
DATIM	16-12-2018 20:51:53
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	5.0000 sec
PW1	5.22 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	ACETN
EXREF	2.09 ppm
BF	0.12 Hz
RGAIN	44

L13/1Et, ***S***

(1st trial)

68%, 1.7/1 dr, 2.8/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L13EtS2.als

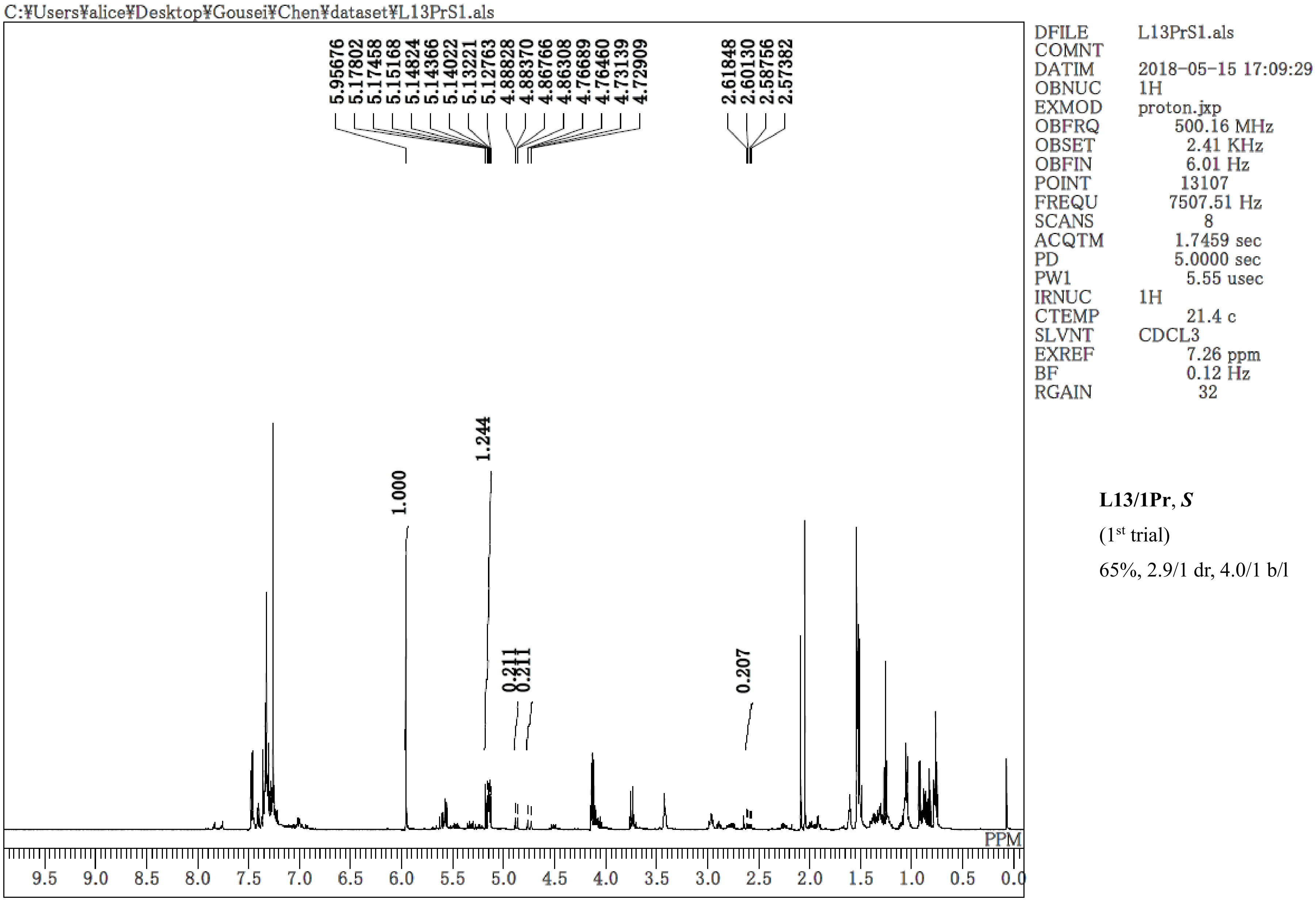


DFILE	L13EtS2.als
COMNT	
DATIM	26-08-2020 23:24:12
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.42 Hz
RGAIN	40

L13/1Et, ***S***

(2nd trial)

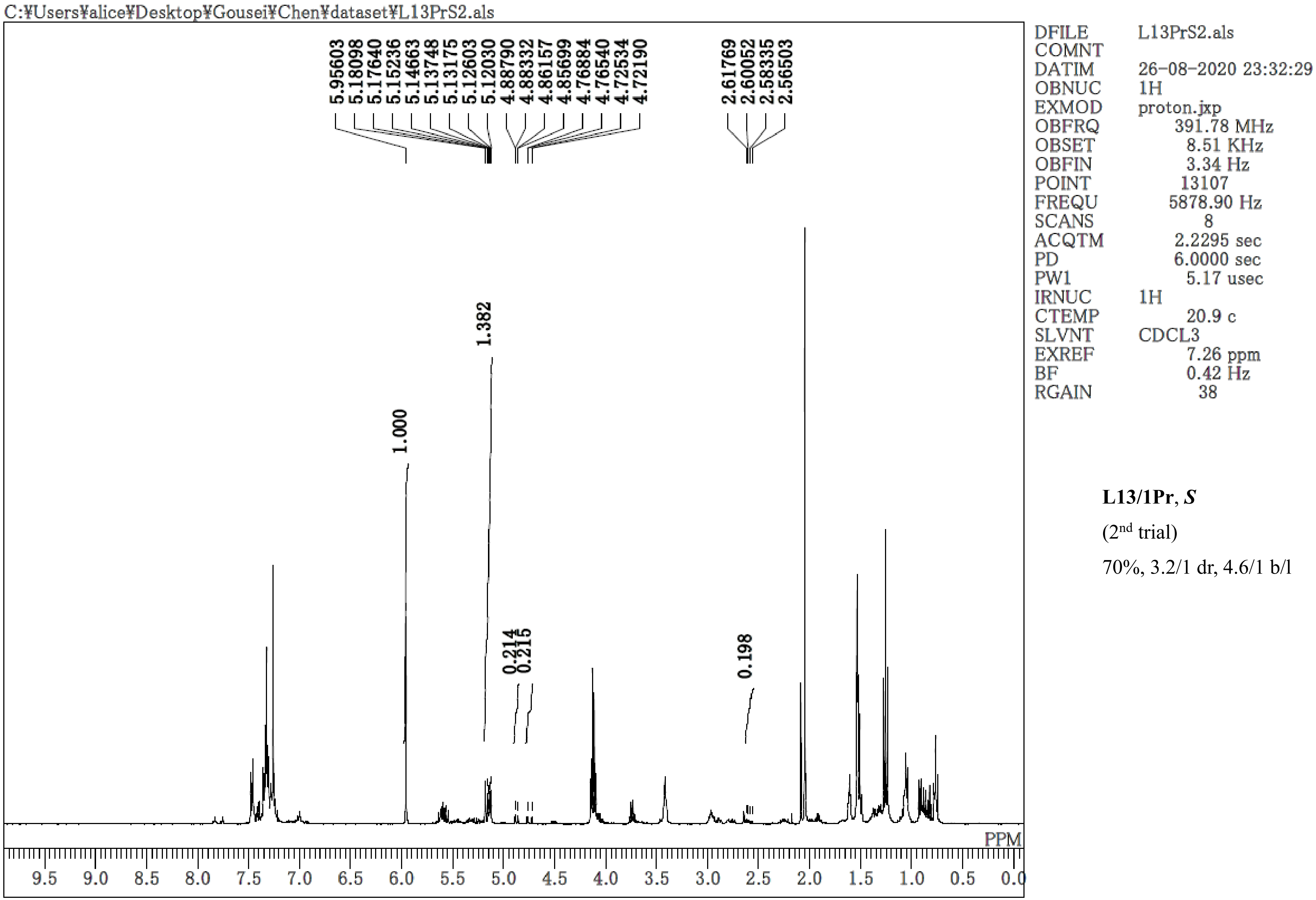
67%, 2.0/1 dr, 3.3/1 b/l



L13/1Pr, ***S***

(1st trial)

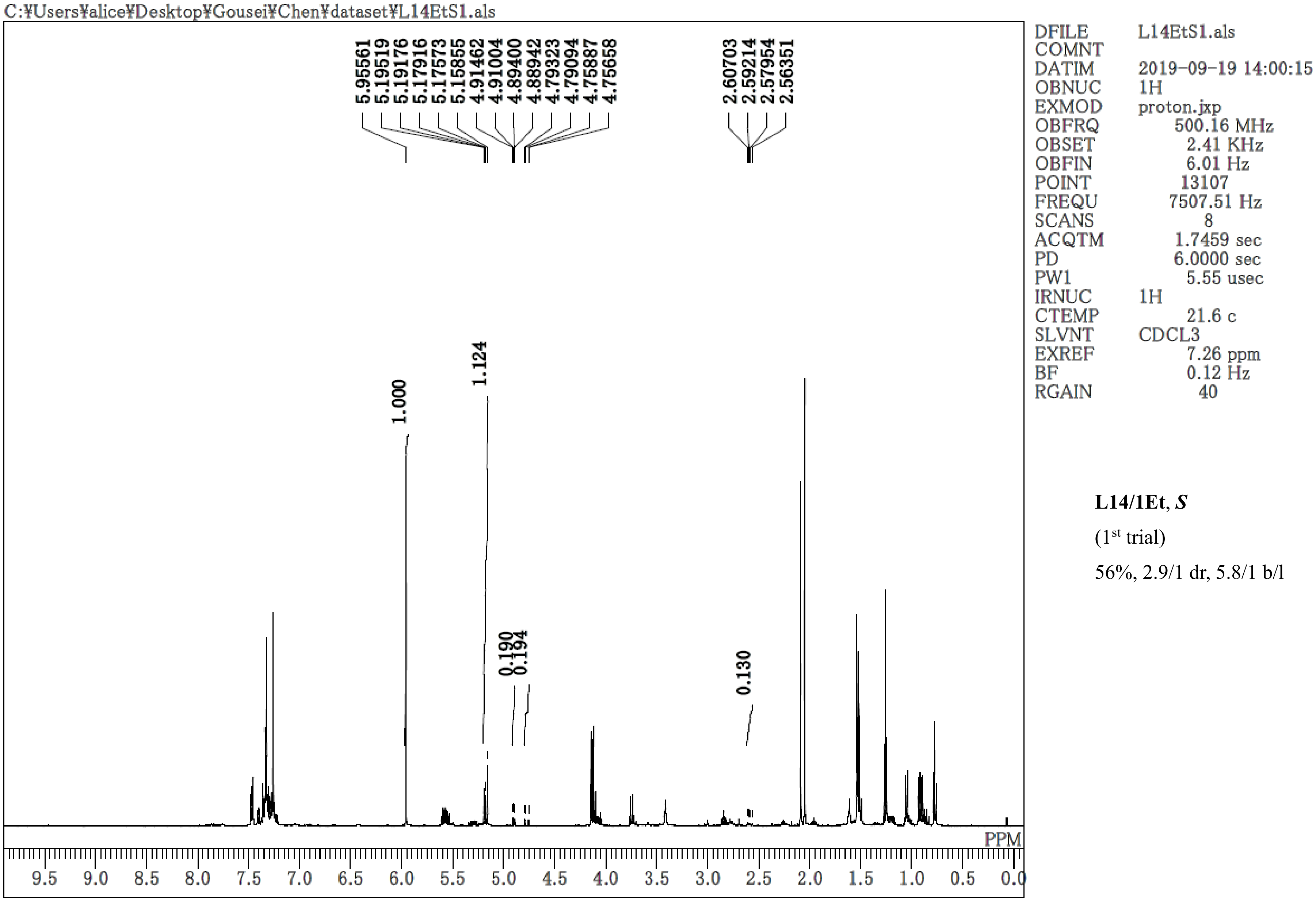
65%, 2.9/1 dr, 4.0/1 b/l



L13/1Pr, ***S***

(2nd trial)

70%, 3.2/1 dr, 4.6/1 b/l

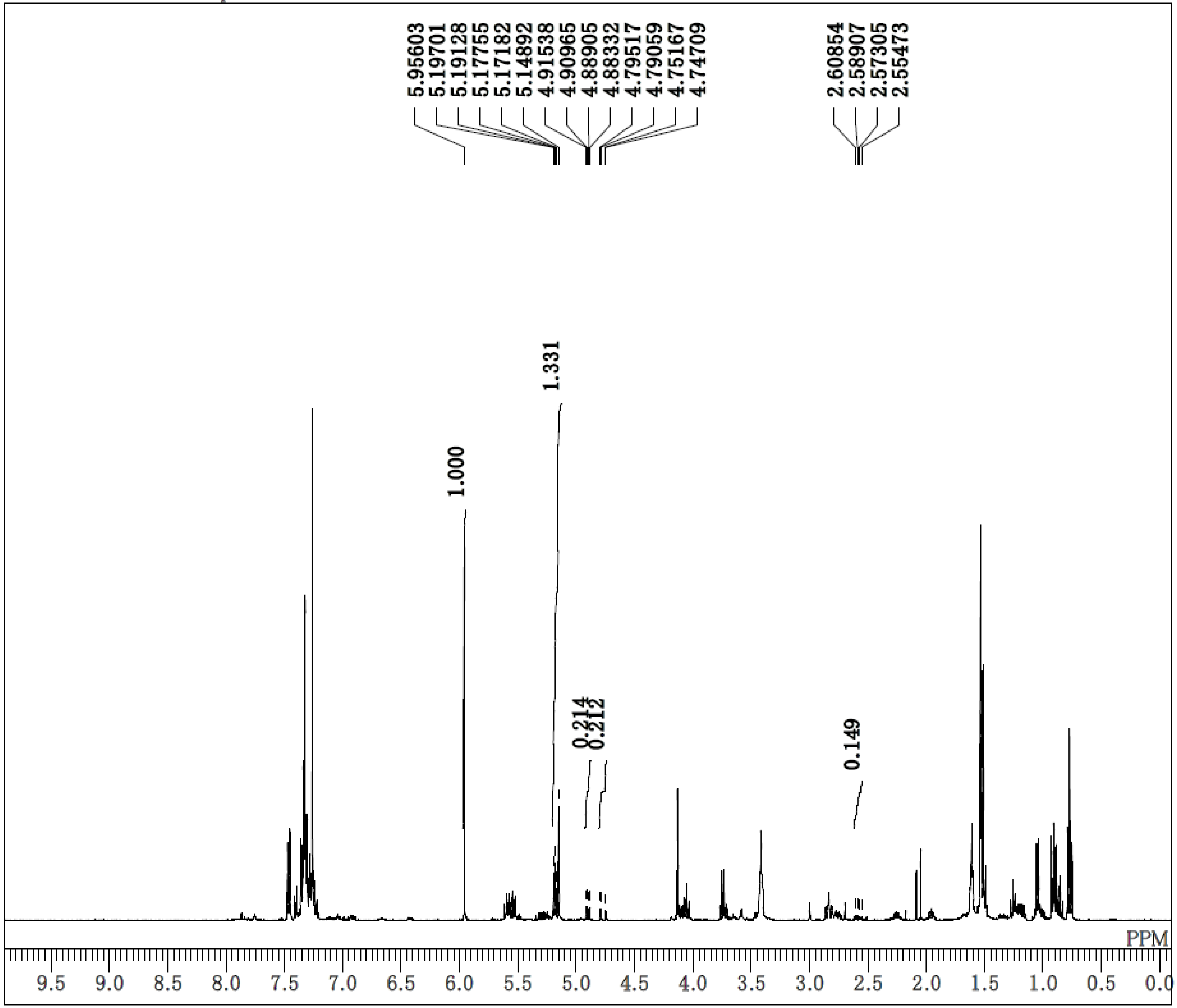


L14/1Et, ***S***

(1st trial)

56%, 2.9/1 dr, 5.8/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L14EtR2.als



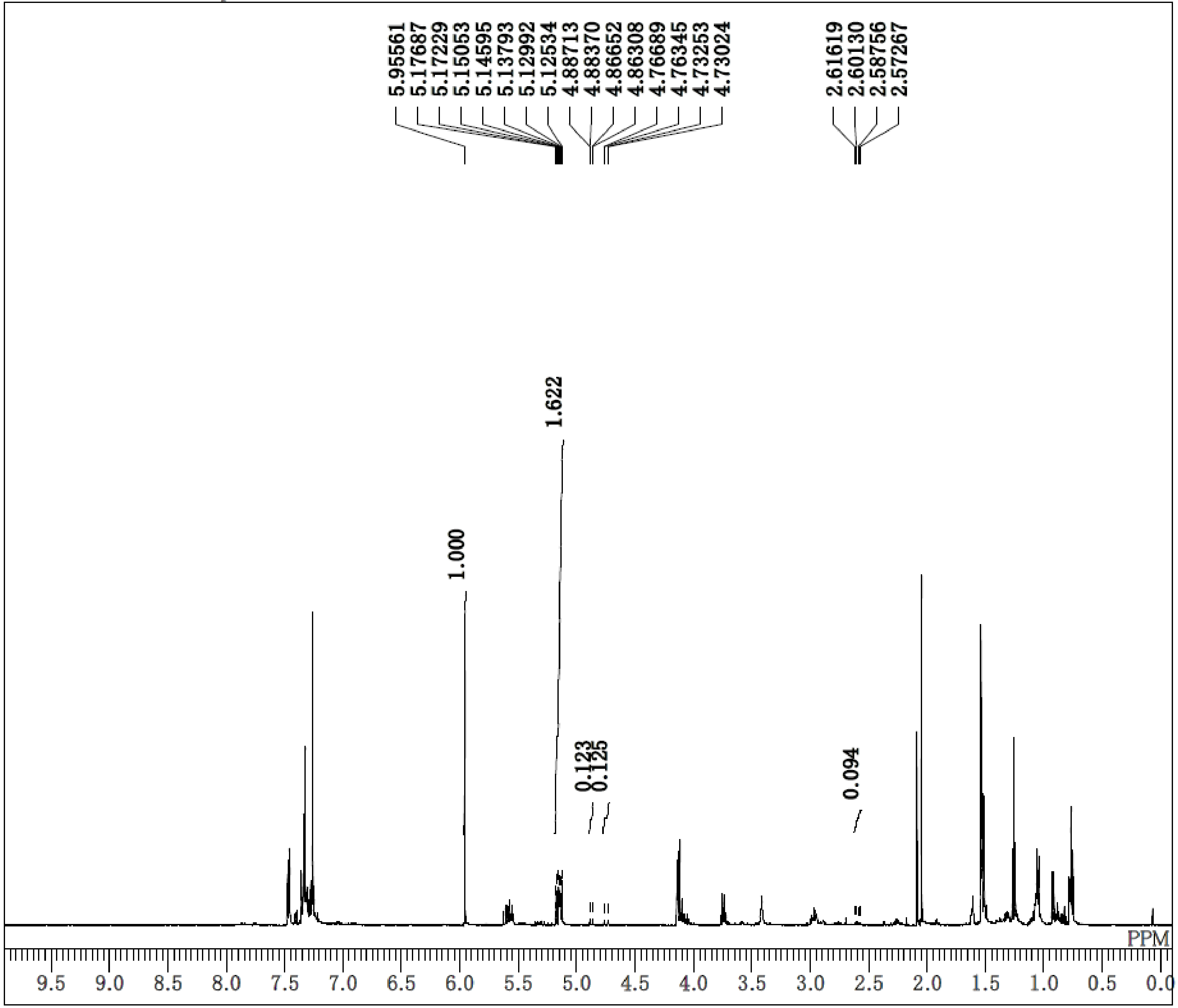
DFILE	L14EtR2.als
COMNT	
DATIM	07-09-2020 23:05:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

L14/1Et, ***S***

(2nd trial)

65%, 3.1/1 dr, 5.9/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L14PrS1.als

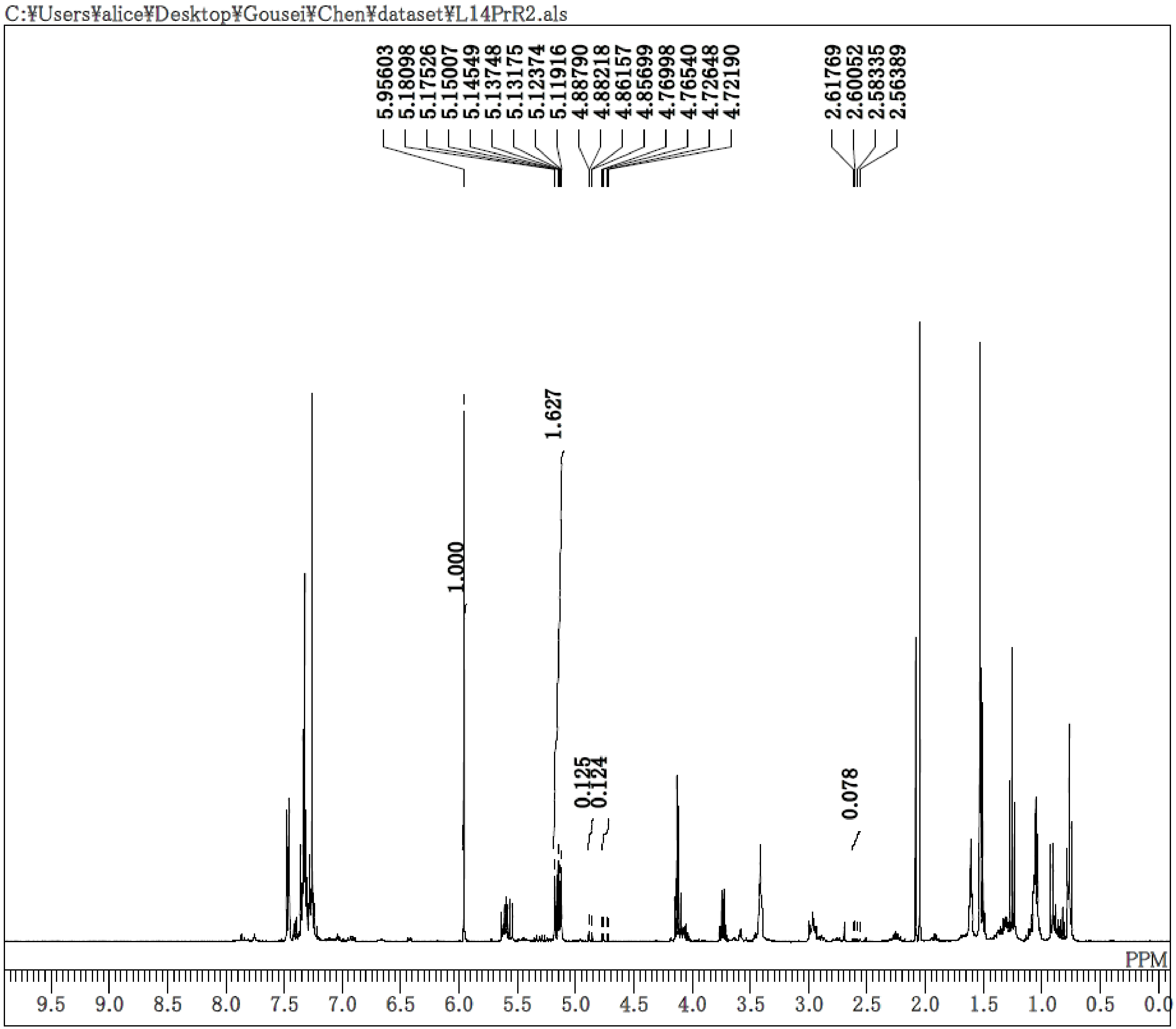


DFILE	L14PrS1.als
COMNT	
DATIM	2019-09-19 14:15:03
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42

L14/1Pr, ***S***

(1st trial)

65%, 6.5/1 dr, 11/1 b/l



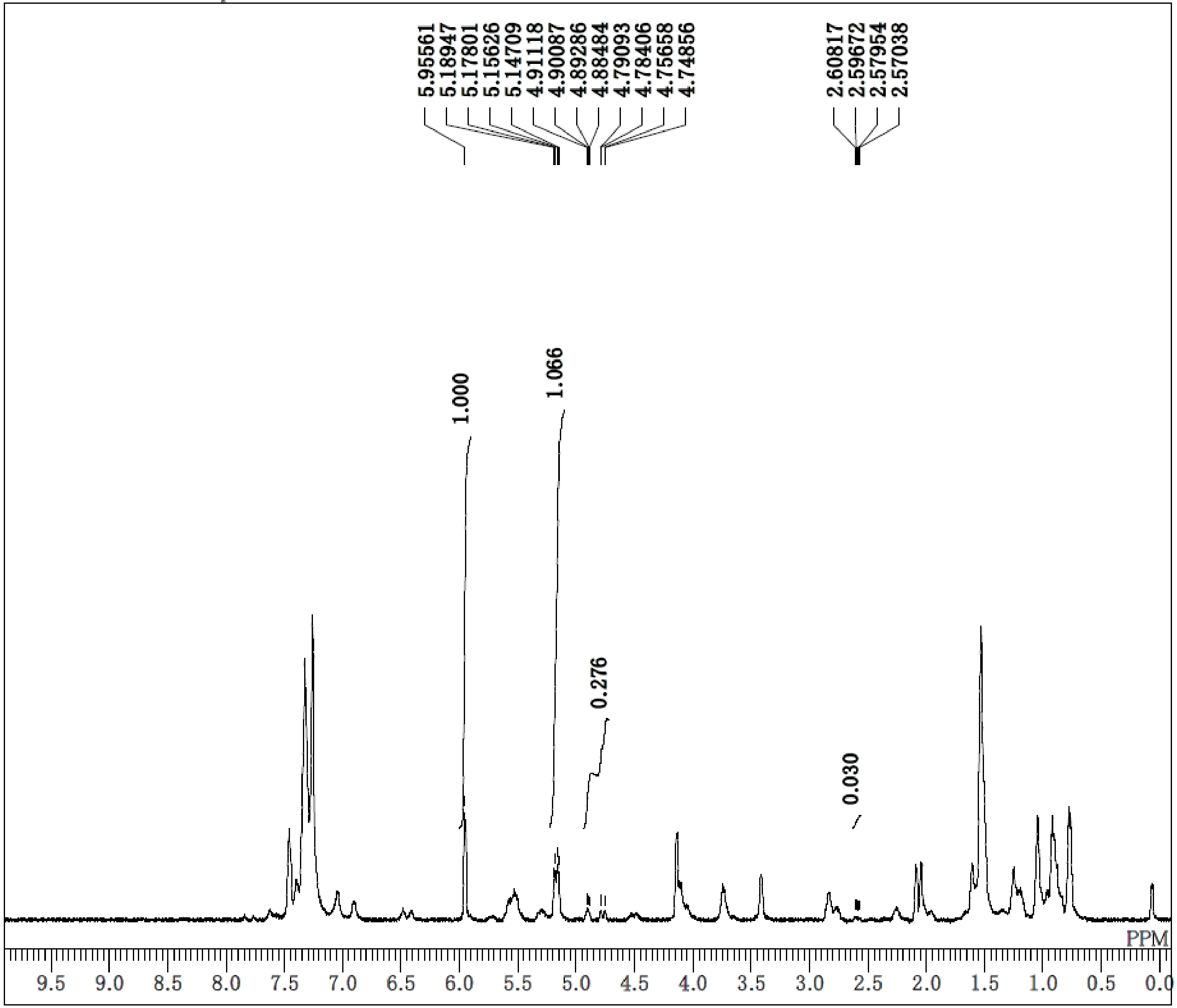
DFILE	L14PrR2.als
COMNT	
DATIM	07-09-2020 23:12:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38

L14/1Pr, ***S***

(2nd trial)

64%, 6.5/1 dr, 12/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L15EtS1.als



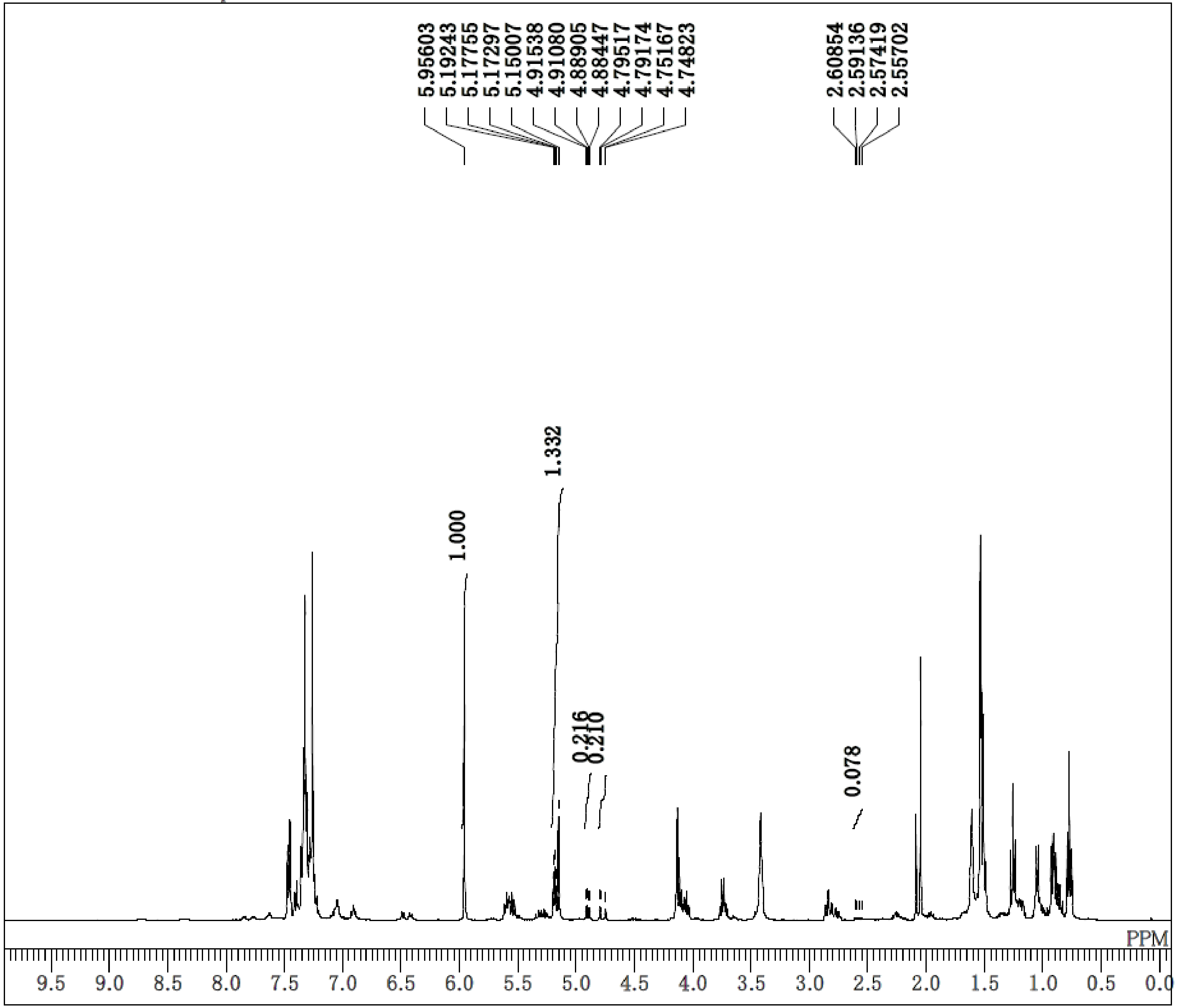
DFILE	L15EtS1.als
COMNT	
DATIM	2019-01-16 15:14:20
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30

L15/1Et, *S*

(1st trial)

44%, 3.9/1 dr, 22.4/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L15EtS2.als

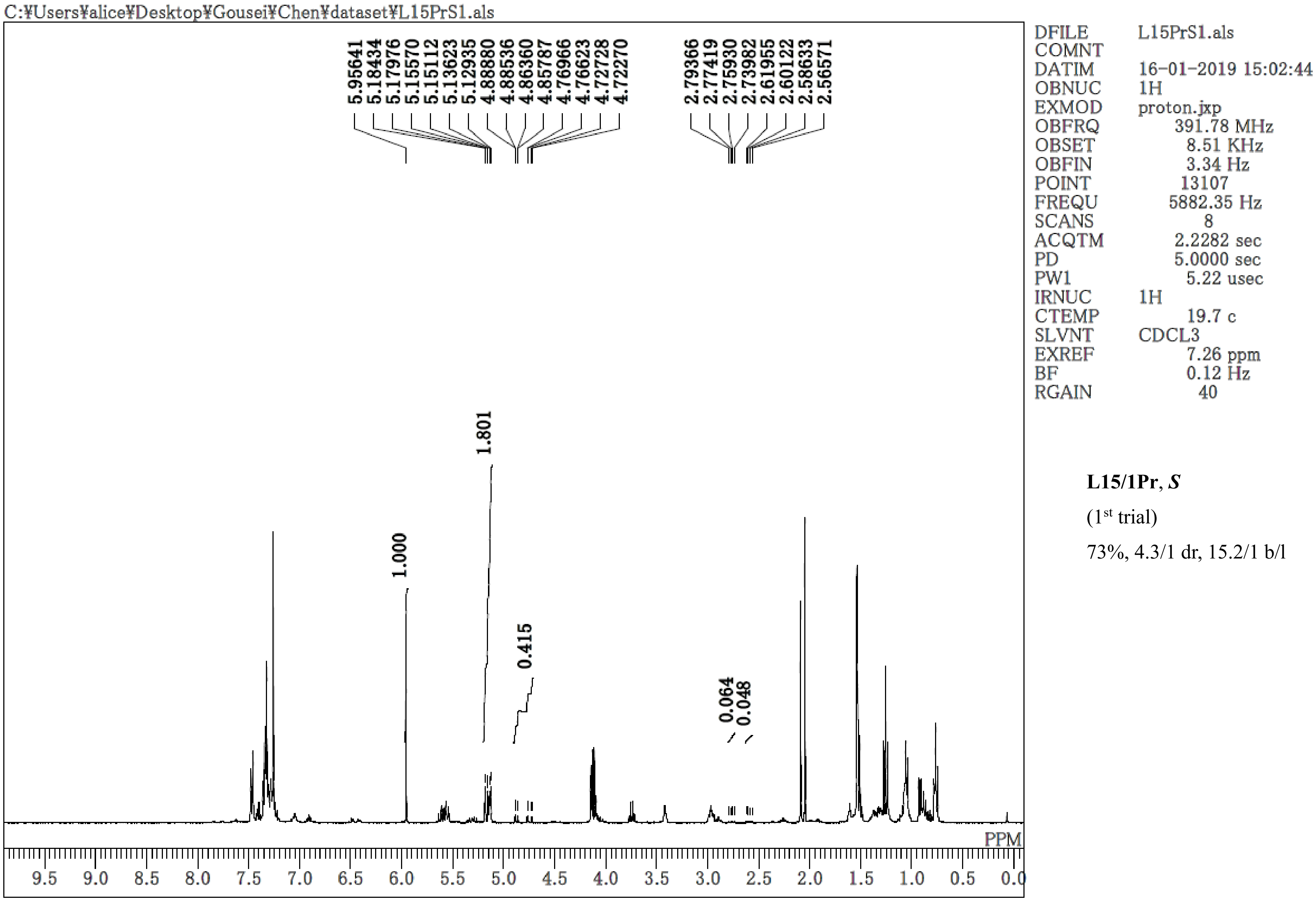


DFILE	L15EtS2.als
COMNT	
DATIM	07-09-2020 18:48:24
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5878.90 Hz
SCANS	8
ACQTM	2.2295 sec
PD	6.0000 sec
PW1	5.17 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	40

L15/1Et, ***S***

(2nd trial)

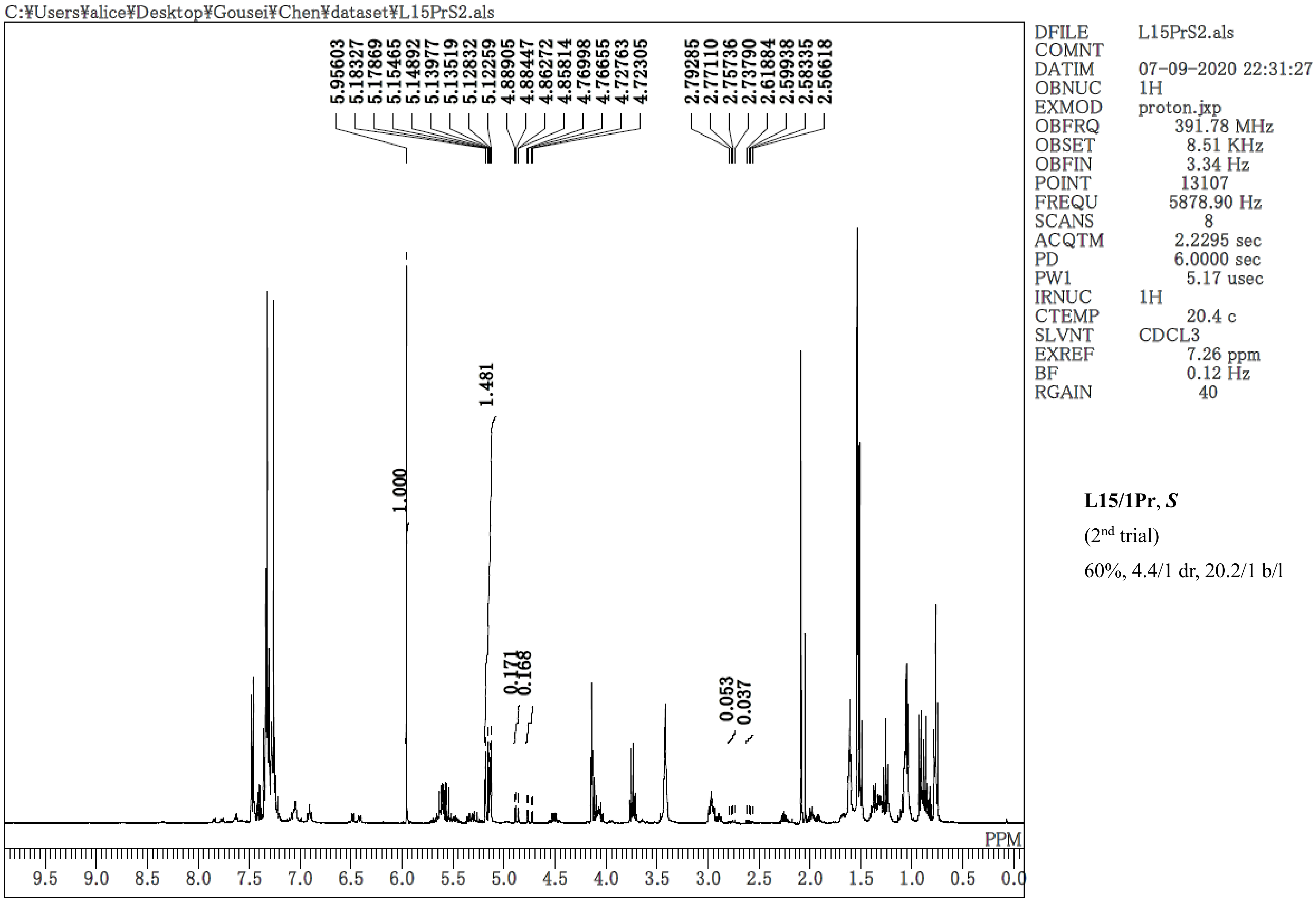
60%, 3.1/1 dr, 11.4/1 b/l



L15/1Pr, ***S***

(1st trial)

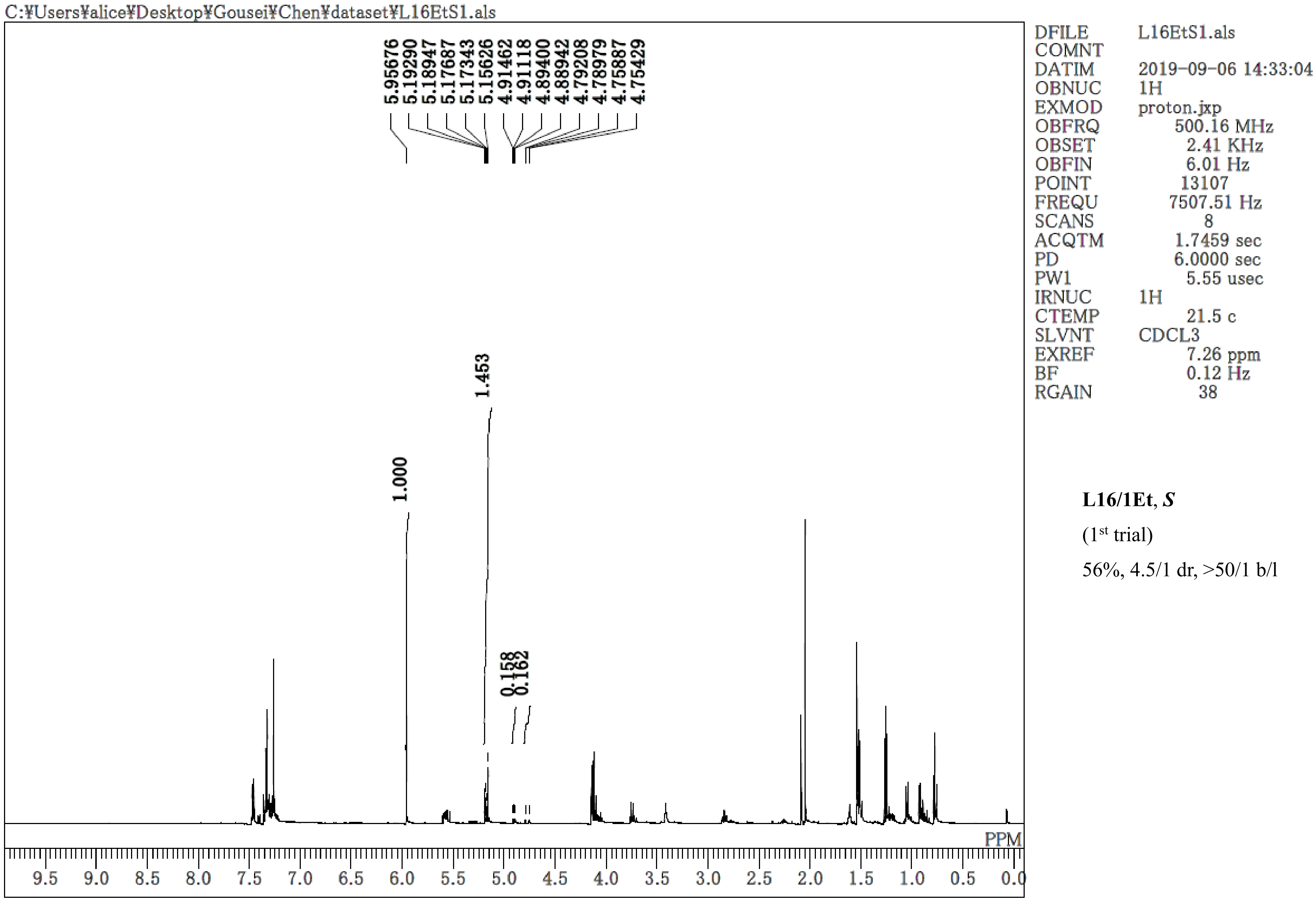
73%, 4.3/1 dr, 15.2/1 b/l



L15/1Pr, *S*

(2nd trial)

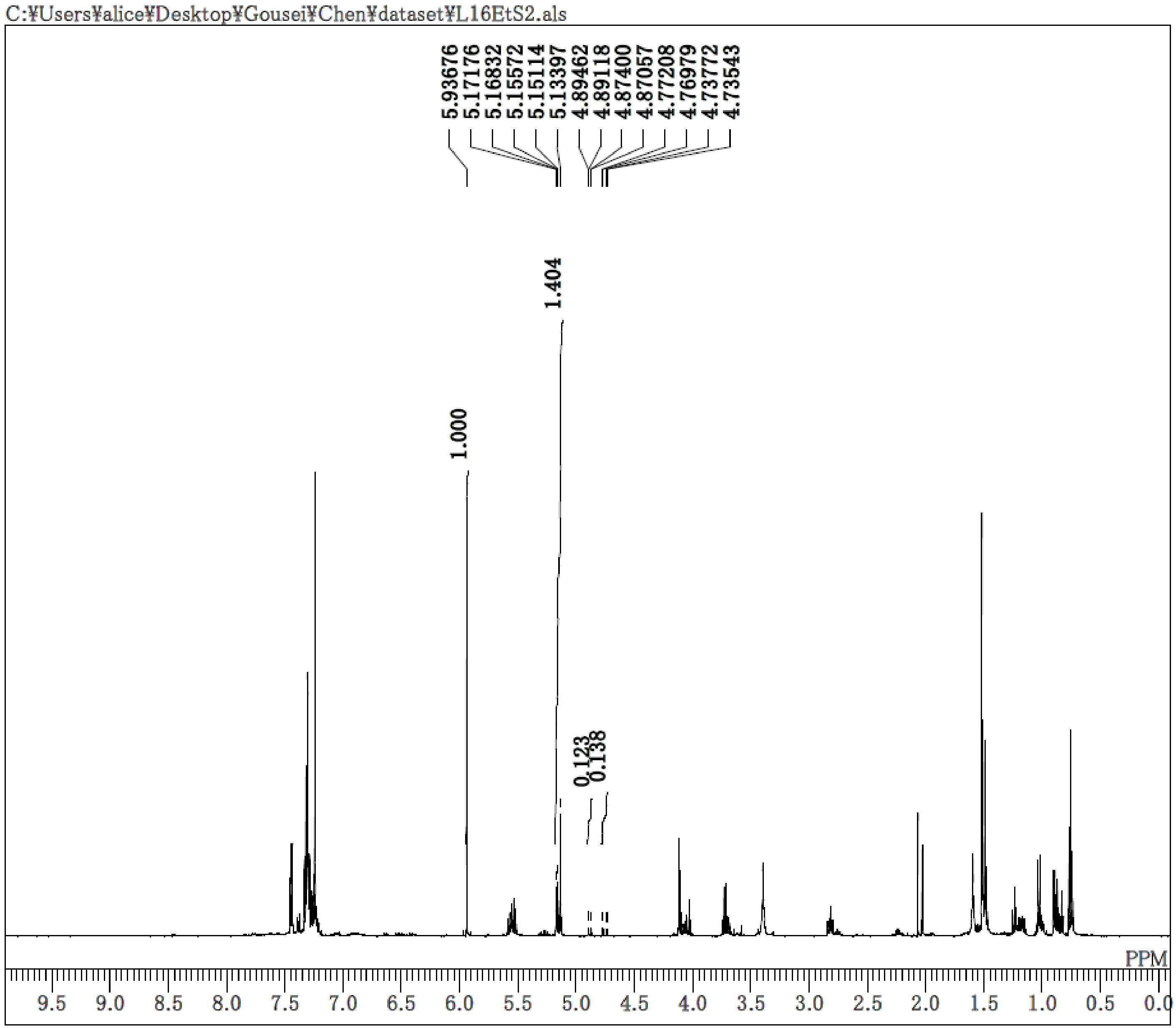
60%, 4.4/1 dr, 20.2/1 b/l



L16/1Et, ***S***

(1st trial)

56%, 4.5/1 dr, >50/1 b/l



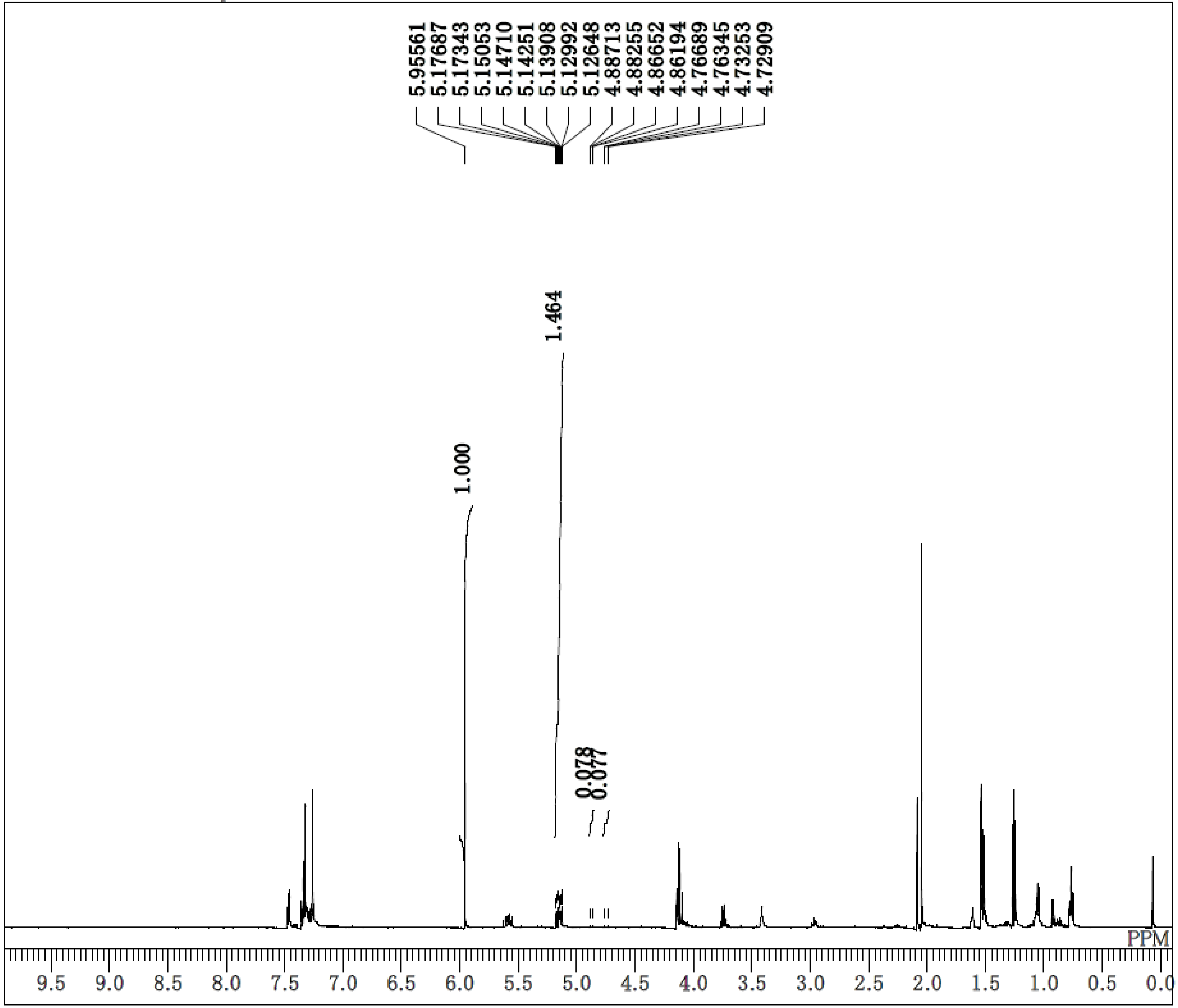
DFILE	L16EtS2.als
COMNT	
DATIM	2020-09-05 13:46:04
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	6.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	42

L16/1Et, ***S***

(2nd trial)

52%, 5.4/1 dr, >50/1 b/l

C:¥Users¥alice¥Desktop¥Gousei¥Chen¥dataset¥L16PrS1.als

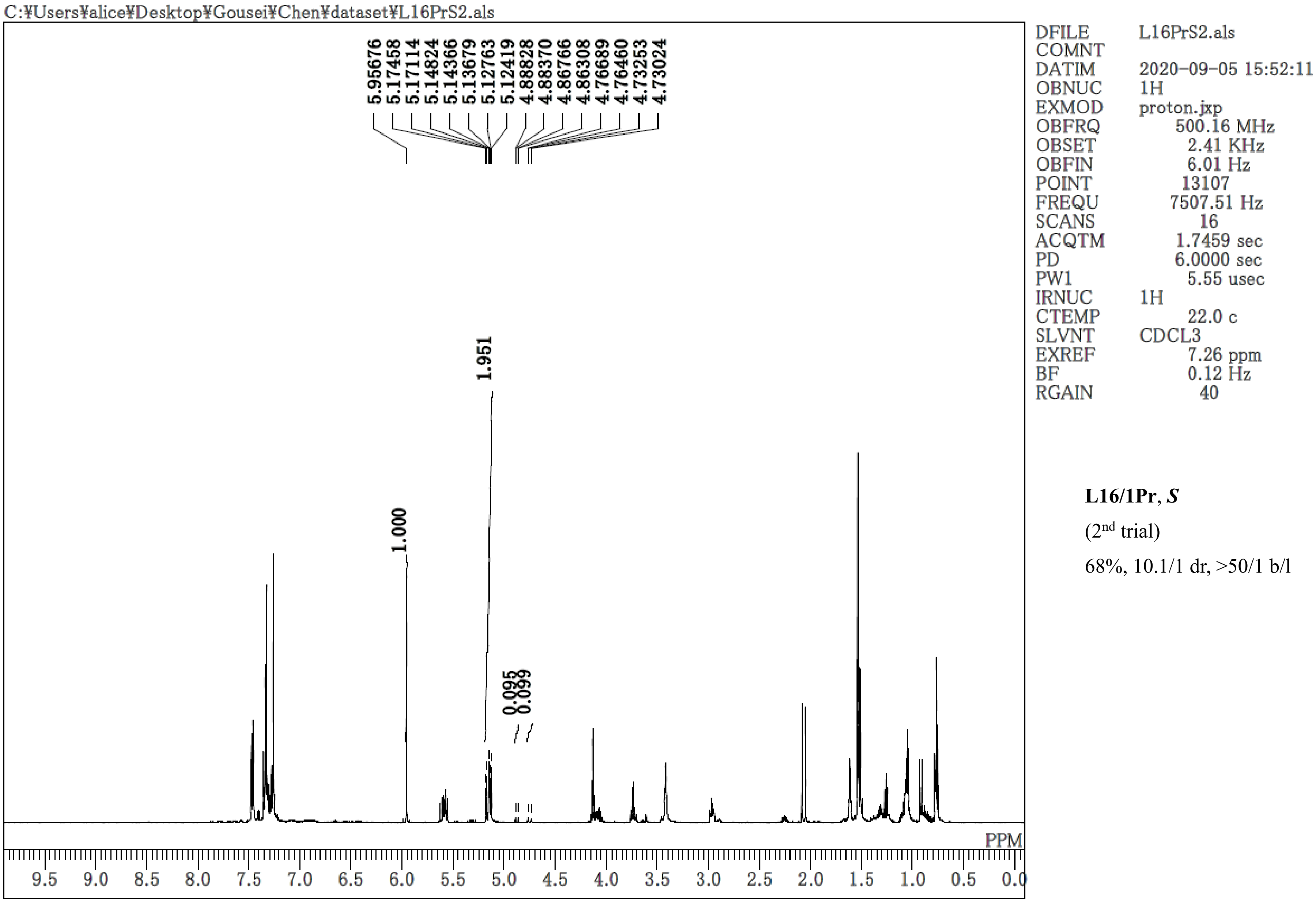


DFILE L16PrS1.als
COMNT
DATIM 2019-09-04 10:13:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

L16/1Pr, ***S***

(1st trial)

51%, 9.4/1 dr, >50/1 b/l



L16/1Pr, ***S***

(2nd trial)

68%, 10.1/1 dr, >50/1 b/l