Supporting Information

Enantioselective Total Synthesis of (+)-Rubrobramide, (+)-Talaramide A, and (–)-Berkeleyamide D by a Skeletal Diversification Strategy

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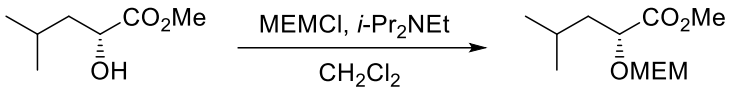
Materials and Methods: ^{1}H NMR spectra were measured at 300 MHz (JNM-AL300, JEOL) or 400 MHz (JNM-AL400, ECZ400S, JEOL). Chemical shifts are expressed in ppm relative to tetramethylsilane ($\delta = 0$) as an internal standard ($CDCl_3$ or DMSO-d_6). Splitting patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; sept, septet; m, multiplet; br, broad peak. ^{13}C NMR spectra were measured at 100 MHz (JNM-AL400, JEOL). The chemical shifts are reported in ppm, relative to the central line of the triplet at 77.0 ppm for $CDCl_3$ or the septet at 39.6 ppm for DMSO-d_6. Infrared spectra (IR) were measured on an IR spectrometer (VALOR-III, JASCO) and are reported in wavenumbers (cm^{-1}). High-resolution mass spectra (HRMS) were obtained using a mass spectrometer (JMS 700, JEOL) with a direct inlet system. Optical rotations were measured on a polarimeter (P-2200, JASCO) using a 100 mm pathlength cell. Melting points (m.p.) were measured on a Micro Melting Point system (Yanaco). Column chromatography was carried out on silica gel (40−100 mesh). Analytical thin-layer chromatography (TLC) was performed using 0.25 mm silica gel 60-F plates.

Experimental Procedures and Characterization Data

Methyl (*R*)-2-((2-methoxyethoxy)methoxy)-4-methylpentanoate (**S2**)

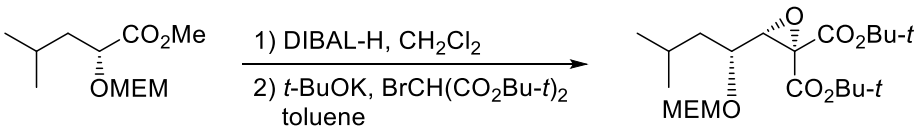


S1 **S2**

MEMCl (14.6 mL, 123 mmol) was slowly added to a stirred solution of secondary alcohol **S1** (7.02 g, 48.0 mmol) and *i*-Pr_2NEt (50 mL, 287 mmol) in CH_2Cl_2 (400 mL) over 5 min at 0 °C. The reaction mixture was heated to 40 °C (oil bath). After stirring for 48 h, the reaction was cooled to room temperature and quenched by addition of 1 N HCl. The organic layers were washed with saturated aqueous $NaHCO_3$ solution and dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (5:1 hexane/EtOAc) to afford MEM-protected ester **S2** (7.40 g, 66% yield) as a colorless liquid.

$[\alpha]_D^{26}$ +87.3 (*c* 1.05, $CHCl_3$); IR ($CHCl_3$) 2955, 2873, 1753, 1176, 1114, 1028 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.76 (d, *J* = 1.0 Hz, 2H), 4.19 (dd, *J* = 8.8, 4.4 Hz, 1H), 3.74-3.71 (m, 2H), 3.54-3.51 (m, 2H), 3.38 (s, 3H), 1.84-1.74 (m, 1H), 1.70 (ddd, *J* = 14.2, 8.8, 5.4 Hz, 1H), 1.51 (ddd, *J* = 13.6, 8.8, 4.4 Hz, 1H), 0.93 (d, *J* = 6.8 Hz, 3H), 0.93 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 95.3, 74.4, 71.6, 67.6, 59.0, 51.8, 41.8, 24.4, 23.1, 21.6; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{11}H_{23}O_5$ 235.1545; Found 235.1538.

Di-*tert*-butyl (*S*)-3-((*R*)-1-((2-methoxyethoxy)methoxy)-3-methylbutyl)oxirane-2,2-dicarboxylate (**13**)



S2 **13**

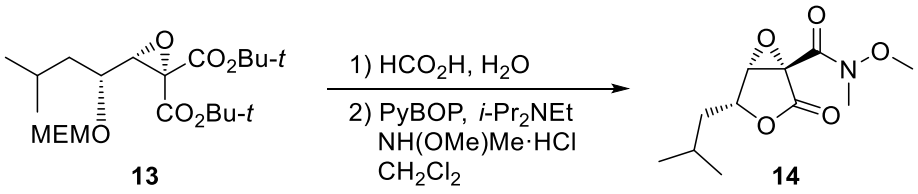
DIBAL-H (1.02 M solution in 31 mL hexane, 31 mmol) was slowly added to a stirred solution of ester **S2** (4.80 g, 20.5 mmol) in CH_2Cl_2 (200 mL) at –78 °C via dropping funnel over 20 min. After stirring for 1 h, the reaction was quenched by addition of MeOH and saturated aqueous Rochelle salt, and vigorously stirred for 1 h. The mixture was extracted with EtOAc three times. The combined organic layers were washed with water

and brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was used in the next reaction without further purification.

A suspension of *t*-BuOK (2.40 g, 21.1 mmol) in toluene (50 mL) was added dropwise to di-*tert*-butyl bromomalonate (6.70 g, 22.7 mmol) in toluene (110 mL) at –78 °C. After stirring for 30 min, the crude aldehyde in toluene (50 mL) was added dropwise. After complete addition, the reaction mixture was allowed to warm to –45 °C gradually and was stirred for 12 h. The reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted three times with EtOAc. The combined organic layers were washed with water and brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (5:1 hexane/EtOAc) to afford epoxide **13** (5.23 g, 61% yield) as a colorless oil.

$[\alpha]_D^{22}$ +33.6 (*c* 1.23, $CHCl_3$); IR ($CHCl_3$) 2983, 2960, 1740, 1371, 1255, 1165, 1124, 1046, 1028 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.96 (d, *J* = 6.8 Hz, 1H), 4.75 (d, *J* = 6.8 Hz, 1H), 3.79-3.74 (m, 1H), 3.71-3.65 (m, 1H), 3.55-3.53 (m, 2H), 3.45 (dt, *J* = 8.6, 2.4 Hz, 1H), 3.41 (d, *J* = 8.6 Hz, 1H), 3.37 (s, 3H), 1.89-1.79 (m, 1H), 1.62 (ddd, *J* = 14.0, 10.0, 4.0 Hz, 1H), 1.50 (s, 9H), 1.48 (s, 9H), 1.26 (m, 1H), 0.93 (d, *J* = 6.3 Hz, 3H), 0.86 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 163.5, 94.8, 83.7, 83.5, 73.0, 71.7, 67.4, 64.3, 59.8, 59.0, 40.5, 27.9 (3C), 27.8 (3C), 23.8, 23.6, 21.2; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{21}H_{39}O_8$ 419.2645; Found 419.2638.

(1*R*,4*R*,5*S*)-4-Isobutyl-*N*-methoxy-*N*-methyl-2-oxo-3,6-dioxabicyclo[3.1.0]hexane-1-carboxamide (**14**)



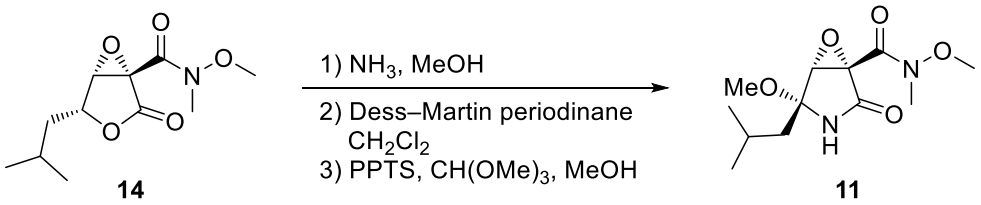
13 **14**

Epoxide **13** (2.00 g, 4.78 mmol) was dissolved in HCO_2H (30 mL) and H_2O (10 mL), and the solution was stirred at 35 °C for 12 h. The reaction mixture was concentrated *in vacuo*. The crude product was used in the next reaction without further purification.

N,*O*-dimethylhydroxylamine hydrochloride (1.40 g, 14.4 mmol), *i*-Pr_2NEt (5.0 mL, 34.4 mmol), and PyBOP (5.0 g, 9.60 mmol) were added successively to a stirred solution of the crude mono carboxylic acid **12** in CH_2Cl_2 (100 mL) at 0 °C. After stirring at room temperature for 2 h, the reaction was quenched by addition of 1 N HCl. The mixture was extracted with CH_2Cl_2 three times and washed with saturated aqueous $NaHCO_3$. The

combined organic layers were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (1:1 hexane/EtOAc) to afford Weinreb amide **14** (1.00 g, 87% yield over two steps) as a colorless solid.
m.p. 67.5-69.5 °C; $[\alpha]_D^{24}$ +61.4 (*c* 0.61, $CHCl_3$); IR ($CHCl_3$) 3019, 2963, 1787, 1687, 1682, 1468, 1223, 1216, 1072, 938 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.61 (dd, *J* = 6.8, 5.9 Hz, 1H), 4.21 (s, 1H), 3.74 (s, 3H), 3.28 (s, 3H), 1.89-1.75 (m, 2H), 1.63-1.56 (m, 1H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.99 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 161.5, 77.2, 62.0, 61.3, 58.9, 38.2, 32.2, 24.8, 22.9, 22.1; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{11}H_{18}NO_5$ 244.1185; Found 244.1184.

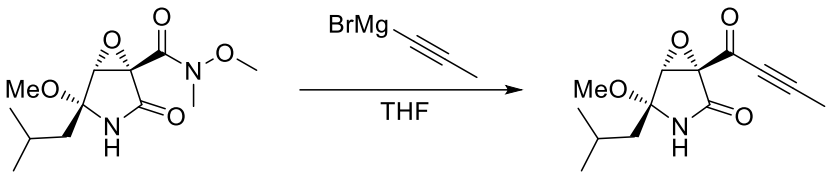
(1*S*,4*R*,5*R*)-4-Isobutyl-*N*,4-dimethoxy-*N*-methyl-2-oxo-6-oxa-3-azabicyclo[3.1.0]hexane-1-carboxamide (**11**)



NH_3 (21 mmol, 7 M solution in 3 mL MeOH) was added to a stirred solution of Weinreb amide **14** (770 mg, 3.16 mmol) in MeOH (20 mL) at 0 °C. After stirring at 0 °C for 30 min, additional NH_3 (14 mmol, 7 M solution in 2 mL MeOH) was added. After 30 min, the reaction was concentrated *in vacuo*. The crude product was used in the next reaction without further purification.
Dess–Martin periodinane (1.95 g, 4.60 mmol) was added to a solution of the crude product in CH_2Cl_2 (30 mL) and the mixture was stirred at room temperature for 1 h. The reaction was quenched by addition of saturated aqueous $NaHCO_3$ and $Na_2S_2O_3$ solution and stirred vigorously for 10 min. The mixture was extracted with $CHCl_3$ three times. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was used in the next reaction without further purification.
$CH(OMe)_3$ (3 mL) and PPTS (800 mg, 3.18 mmol) were added to a stirred solution of the crude product in MeOH (40 mL). The reaction was heated to reflux (oil bath) and stirred for 48 h. After cooling to room temperature, the reaction was quenched by addition of Et_3N (4.4 mL) and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 to 1:1 hexane/EtOAc) to afford lactam **11** (620 mg, 72% yield in three steps) as a colorless oil.
$[\alpha]_D^{25}$ –45.6 (*c* 8.2, $CHCl_3$); IR (neat) 3503, 3272, 2957, 1725, 1673, 1464, 1062, 763 cm^{-1};

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.03 (br s, 1H), 3.99 (d, *J* = 2.6 Hz, 1H), 3.70 (s, 3H), 3.27 (s, 3H), 3.23 (s, 3H), 1.87 (sept, *J* = 6.5 Hz, 1H), 1.73 (dd, *J* = 14.5, 5.1 Hz, 1H), 1.58 (dd, *J* = 14.5, 7.9 Hz, 1H), 0.97 (d, *J* = 6.5 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.3, 162.4, 89.6, 61.9, 61.4, 60.3, 49.8, 41.5, 32.2, 24.1, 23.9, 23.4; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{12}H_{21}N_2O_5$ 273.1450; Found 273.1446.

(1*R*,4*R*,5*R*)-1-(But-2-ynoyl)-4-isobutyl-4-methoxy-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**9**)

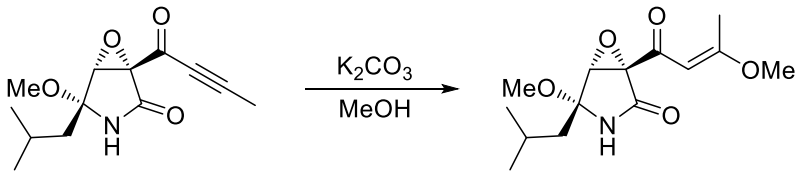


11 **9**

1-Propynyl magnesium bromide (4.5 mmol, 0.5 M in 9 mL THF) was added to a stirred solution of lactam **11** (200 mg, 0.734 mmol) in THF (7 mL) at –55 °C. The reaction was heated to –40 °C, stirred for 1 h, and quenched by addition of saturated aqueous NH_4Cl solution. The reaction mixture was extracted with EtOAc three times. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford alkynyl ketone **9** (182 mg, 99% yield) as a yellow oil.

$[\alpha]_D^{25}$ –166.1 (*c* 4.5, $CHCl_3$); IR (neat) 3280, 2959, 2215, 1732, 1666, 1399, 763 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.88 (br s, 1H), 4.06 (d, *J* = 2.9 Hz, 1H), 3.25 (s, 3H), 2.07 (s, 3H), 1.93 (sept, *J* = 6.3 Hz, 1H), 1.75 (dd, *J* = 14.6, 5.4 Hz, 1H), 1.64 (dd, *J* = 14.6, 7.8 Hz, 1H), 1.01 (d, *J* = 6.3 Hz, 3H), 0.97 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.5, 167.7, 96.3, 88.8, 77.6, 63.7, 61.9, 49.6, 42.3, 24.0, 23.8, 23.5, 4.5; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{13}H_{18}NO_4$ 252.1236; Found 252.1245.

(1*R*,4*R*,5*R*)-4-Isobutyl-4-methoxy-1-(3-methoxybut-2-enoyl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**15**)

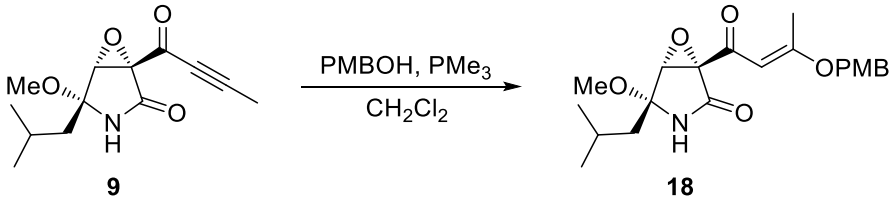


9 **15**

A solution of alkynyl ketone **9** (32.5 mg, 0.129 mmol) in MeOH (1.5 mL) was added to a suspension of K_2CO_3 (18 mg, 0.13 mmol) in MeOH (12 mL) over 5 min at 0 °C. After stirring for 10 min, the reaction was concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford methoxy vinylogous ester **15** (34 mg, 93% yield) as a yellow oil.

$[\alpha]_D^{26}$ –60.9 (*c* 1.5, $CHCl_3$); IR (neat) 3284, 2957, 1728, 1577, 1060, 810 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.41 (br s, 1H),5.80 (s, 1H), 3.91 (d, *J* = 2.4 Hz, 1H), 3.71 (s, 3H), 3.27 (s, 3H), 2.37 (s, 3H), 1.94 (sept, *J* = 6.8 Hz, 1H), 1.72 (dd, *J* = 14.6, 5.9 Hz, 1H), 1.67 (dd, *J* = 14.6, 7.8 Hz, 1H), 1.02 (d, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.5, 178.0, 169.6, 94.5, 88.7, 63.4, 61.7, 56.1, 49.7, 42.8, 24.1, 23.9, 23.5, 20.7; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{14}H_{21}NO_5$ 284.1498; Found 284.1506.

(1*R*,4*R*,5*R*)-4-Isobutyl-4-methoxy-1-(3-((4-methoxybenzyl)oxy)but-2-enoyl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**18**)

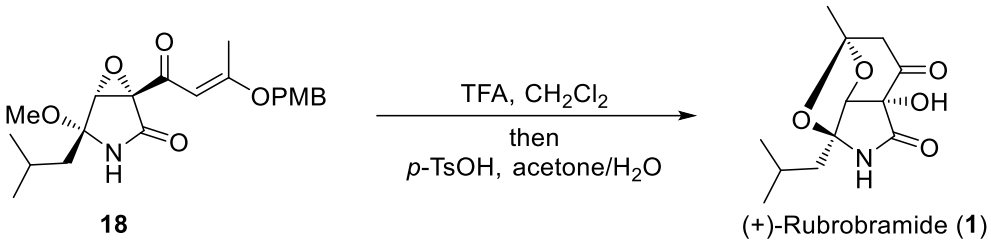


9 **18**

A solution of **9** (73 mg, 0.29 mmol) in CH_2Cl_2 (9 mL) was slowly added to a stirred solution of PMBOH (120 mg, 0.868 mmol) and PMe_3 (1.0 M in THF 0.06 mL, 0.06 mmol) in CH_2Cl_2 (20 mL) over 5 min at –20 °C. After stirring for 1 h, the reaction mixture was diluted with *n*-hexane and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford **18** (101 mg, 89% yield) as a yellow oil.

$[\alpha]_D^{23}$ –66.4 (*c* 2.1, $CHCl_3$); IR (neat) 3284, 2957, 1726, 1573, 1252, 1033, 824, 758 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 5.96 (s, 1H), 5.92 (br s, 1H), 4.86 (s, 2H), 3.91 (d, *J* = 2.7 Hz, 1H), 3.82 (s, 3H), 3.27 (s, 3H), 2.42 (s, 3H), 1.95 (sept, *J* = 6.8 Hz, 1H), 1.71 (dd, *J* = 6.8, 1.6 Hz, 2H), 1.04 (d, *J* = 6.8 Hz, 3H), 0.99 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.4, 177.0, 169.4, 159.9, 129.7 (2C), 126.8, 114.1 (2C), 95.6, 88.6, 70.9, 63.4, 61.6, 55.3, 49.7, 43.0, 24.1, 23.9, 23.6, 21.0; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{21}H_{27}NO_6$ 390.1917; Found 390.1910.

(+)-Rubrobramide (**1**)



18 (+)-Rubrobramide (**1**)

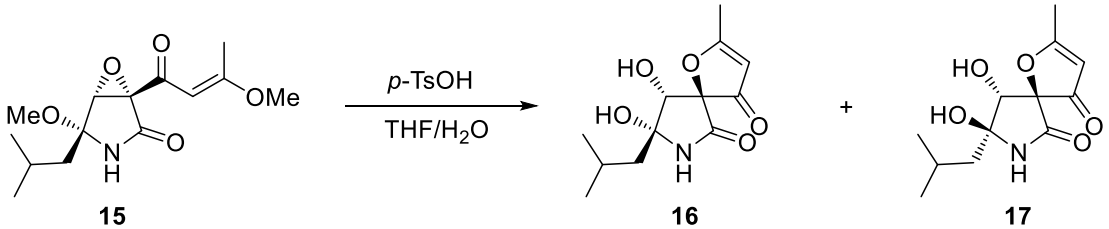
TFA (0.1 mL) was added to a stirred solution of *p*-methoxy benzyloxy vinylogous ester **18** (44 mg, 0.11 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred at room temperature for 18 h and concentrated with toluene *in vacuo*. The crude product was used in the next reaction without further purification.

p-TsOH (33 mg, 0.17 mmol) was added to a stirred solution of the crude product in acetone (4.5 mL) and H_2O (1.5 mL). The reaction mixture was heated to reflux (oil bath) and stirred for 6 days. The reaction was quenched by addition of saturated aqueous $NaHCO_3$. The mixture was extracted with $CHCl_3$ three times, and the organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford **1** (14.6 mg, 51% yield) as a colorless solid.

m.p. 131-132.5 °C; $[\alpha]_D^{24}$ +224.1 (*c* 0.63, $CHCl_3$); IR ($CHCl_3$) 3275, 2959, 1748, 1717, 1389, 1230, 1191, 757 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.58 (br s, 1H), 4.88 (s, 1H), 4.23 (s, 1H), 2.97 (d, *J* = 18.5 Hz, 1H), 2.85 (d, *J* = 18.5 Hz, 1H), 1.92-1.87 (m, 1H), 1.86-1.81 (m, 2H), 1.63 (s, 3H), 1.03 (d, *J* = 6.5 Hz, 3H), 1.02 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.2, 168.4, 109.1, 92.8, 86.5, 82.4, 48.6, 45.5, 25.2, 24.3, 23.7, 23.5; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{12}H_{18}NO_5$ 256.1185; Found 256.1184.

(5*S*,8*R*,9*R*)-8,9-Dihydroxy-8-isobutyl-2-methyl-1-oxa-7-azaspiro[4.4]non-2-ene-4,6-dione (**16**)

(5*S*,8*S*,9*R*)-8,9-Dihydroxy-8-isobutyl-2-methyl-1-oxa-7-azaspiro[4.4]non-2-ene-4,6-dione (**17**)



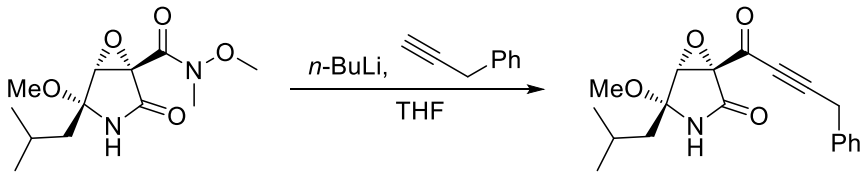
15 **16** **17**

p-TsOH (19.5 mg, 0.103 mmol) was added to a stirred solution of methoxy vinylogous ester **15** (29.1 mg, 0.103 mmol) in THF (9 mL) and H_2O (1 mL). The reaction mixture was stirred at 40 °C for 5 days. The reaction was diluted with $CHCl_3$ and H_2O and extracted with $CHCl_3$ three times. The organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (1:1 to 1:3 hexane/EtOAc) to afford **16** (19.0 mg, 72% yield) and **17** (7.1 mg, 27% yield) as colorless solids.

Compound **16**: m.p. 136-139.5 °C; $[\alpha]_D^{25}$ –27.8 (*c* 0.17, $CHCl_3$); IR ($CHCl_3$) 3281, 2958, 1731, 1686, 1596, 1336, 1162, 1124, 756 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 9.33 (br s, 1H), 6.05 (d, *J* = 5.9 Hz, 1H), 5.70 (s, 1H), 5.35 (s, 1H), 4.35 (d, *J* = 5.9 Hz, 1H), 2.37 (s, 3H), 1.85 (sept, *J* = 6.8 Hz, 1H), 1.62 (dd, *J* = 14.4, 6.3 Hz, 1H), 1.58 (dd, *J* = 14.4, 6.3 Hz, 1H), 0.92 (d, *J* = 6.8 Hz, 3H), 0.92 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.8, 164.1, 103.8, 95.9, 85.3, 73.9, 45.4, 24.3, 23.9, 23.4, 16.6; HRMS (FAB-DFMS) *m*/*z*: $[M+Na]^+$ Calcd for $C_{12}H_{17}NO_5Na$ 278.1004; Found 278.1004.

Compound **17**: m.p. 146.5 °C (decomp.); $[\alpha]_D^{25}$ +25.3 (*c* 0.17, MeOH); IR ($CHCl_3$) 3410, 3243, 2952, 2959, 2867, 1732, 1680, 1584, 1338, 1157, 1127 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 8.95 (br s, 1H), 5.97 (d, *J* = 5.9 Hz, 1H), 5.78 (s, 1H), 5.44 (s, 1H), 4.38 (d, *J* = 5.9 Hz, 1H), 2.27 (s, 3H), 2.02 (dd, *J* = 14.1, 4.9 Hz, 1H), 1.94 (sept, *J* = 6.8 Hz, 1H), 1.59 (dd, *J* = 14.1, 6.8 Hz, 1H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.92 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 197.1, 190.9, 163.7, 103.8, 94.8, 87.6, 81.8, 43.8, 25.0, 24.8, 22.5, 16.3; HRMS (FAB-DFMS) *m*/*z*: $[M+Na]^+$ Calcd for $C_{12}H_{17}NO_5Na$ 278.1004; Found 278.1000.

(1*R*,4*R*,5*R*)-4-Isobutyl-4-methoxy-1-(4-phenylbut-2-ynoyl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**10**)

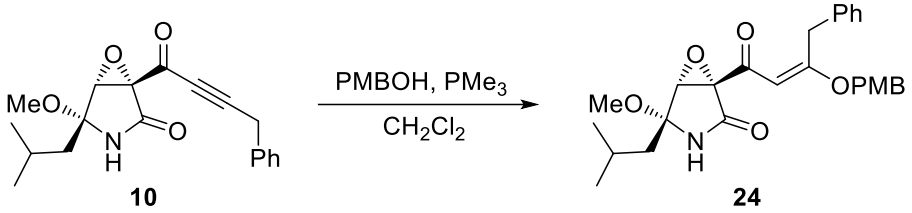


11 **10**

n-BuLi (0.93 mmol, 2.65 M in 0.35 mL *n*-hexane) was added to a stirred solution of 3-phenyl-1-propyne (74 mg, 0.27 mmol) in THF (5 mL) at –78 °C. After stirring for 30 min, lactam **11** (74 mg, 0.27 mmol) in THF (2 mL) was added. The reaction mixture was heated to –45 °C and stirred for 2 h. The reaction was quenched by addition of AcOH (0.05 mL, 0.29 mmol) in THF (1 mL) and H_2O, and the mixture was extracted with EtOAc three

times. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford alkynyl ketone **10** (87 mg, 98% yield) as a yellow oil.
$[\alpha]_D^{23}$ +6.5 (*c* 5.6, $CHCl_3$); IR (neat) 3259, 2958, 2213, 1728, 1668, 1398, 761, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.37-7.28 (m, 5H), 5.68 (br s, 1H), 4.07 (d, *J* = 2.7 Hz, 1H), 3.84 (s, 2H), 3.22 (s, 3H), 1.94 (sept, *J* = 6.6 Hz, 1H), 1.71 (d, *J* = 6.6 Hz, 2H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.99 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.4, 167.7, 133.4, 128.8 (2C), 127.9 (2C), 127.3, 97.2, 88.8, 79.6, 63.8, 62.1, 49.6, 42.3, 25.5, 24.0, 23.8, 23.4; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{19}H_{22}NO_4$ 328.1549; Found 328.1555.

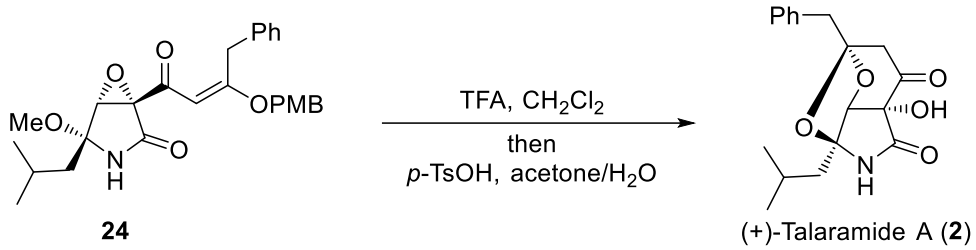
(1*R*,4*R*,5*R*)-4-Isobutyl-4-methoxy-1-(3-((4-methoxybenzyl)oxy)-4-phenylbut-2-enoyl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**24**)



10 **24**

A solution of alkynyl ketone **10** (44.0 mg, 0.134 mmol) in CH_2Cl_2 (3 mL) was slowly added to a stirred solution of PMBOH (56 mg, 0.41 mmol) and PMe_3 (1.0 M in THF, 0.05 mL, 0.05 mmol) in CH_2Cl_2 (10 mL) over 8 min at –20 °C. After stirring for 2 h, the reaction mixture was diluted with *n*-hexane and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford *p*-methoxy benzyloxy vinylogous ester **24** (20.1 mg, 32% yield) as a yellow oil.
$[\alpha]_D^{22}$ –52.5 (*c* 1.2, $CHCl_3$); IR (neat) 3275, 2956, 2937, 1726, 1570, 1251, 822 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.38-7.20 (m, 5H), 7.19 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.05 (br s, 1H), 5.96 (s, 1H), 4.86 (s, 2H), 4.25 (d, *J* = 13.7 Hz, 1H), 4.12 (d, *J* = 13.7 Hz, 1H), 3.90 (d, *J* = 2.4 Hz, 1H), 3.81 (s, 3H), 3.24 (s, 3H), 1.94 (sept, *J* = 6.8 Hz, 1H), 1.71 (s, 1H), 1.70 (d, *J* = 2.4 Hz, 1H), 1.03 (d, *J* = 6.8 Hz, 3H), 0.99 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.1, 177.1, 169.2, 159.8, 136.9, 129.4 (2C), 129.3 (2C), 128.3 (2C), 126.8, 126.6, 114.0 (2C), 95.8, 88.6, 70.9, 63.5, 61.6, 55.3, 49.6, 42.9, 38.7, 24.1, 23.9, 23.5; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{27}H_{32}NO_6$ 466.2230; Found 466.2235.

(–)-Talaramide (**2**)



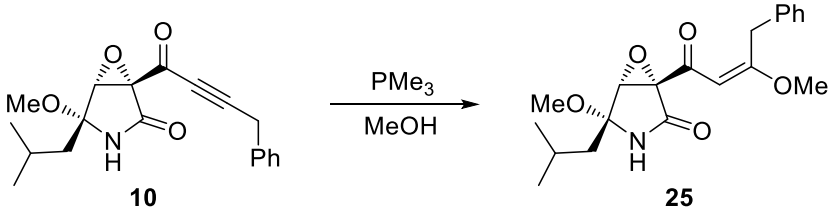
24 (+)-Talaramide A (**2**)

TFA (0.05 mL) was added to a stirred solution of **24** (28 mg, 0.060 mmol) in CH_2Cl_2 (5 mL). The reaction mixture was stirred at room temperature for 19 h and concentrated with toluene *in vacuo*. The crude product was used in the next reaction without further purification.

p-TsOH (11 mg, 0.058 mmol) was added to a stirred solution of the crude product in acetone (3 mL) and H_2O (1 mL). The reaction mixture was heated to reflux (oil bath) and stirred for 5 days. The reaction was quenched by addition of saturated aqueous $NaHCO_3$. The mixture was extracted with $CHCl_3$ three times, the organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (5:1 to 3:1 hexane/EtOAc) to afford talaramide (**2**) (12 mg, 62% yield) as a colorless solid.

m.p. 168-173 °C; $[\alpha]_D^{24}$ +163.1 (*c* 0.82, $CHCl_3$); IR ($CHCl_3$) 3312, 3216, 2964, 2930, 1744, 1720, 760 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 9.37 (s, 1H), 7.33-7.23 (m, 5H), 6.58 (s, 1H), 4.76 (s, 1H), 3.11 (s, 2H), 2.79 (d, J = 18.5 Hz, 1H), 2.68 (d, J = 18.5 Hz, 1H), 1.71-1.62 (m, 1H), 1.58-1.51 (m, 2H), 0.84 (d, J = 7.3 Hz, 3H), 0.83 (d, J = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 200.3, 168.7, 134.4, 130.7 (2C), 127.9 (2C), 126.7, 108.7, 93.0, 86.3, 83.2, 47.5, 44.2, 43.1, 23.8, 23.4, 23.0; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{18}H_{22}NO_5$ 332.1498; Found 332.1500.

(1*R*,4*R*,5*R*)-4-Isobutyl-4-methoxy-1-(3-methoxy-4-phenylbut-2-enoyl)-6-oxa-3-azabicyclo[3.1.0]hexan-2-one (**25**)



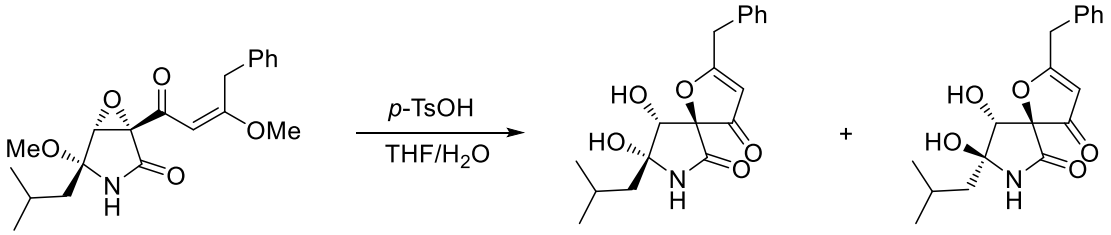
10 **25**

A solution of alkynyl ketone **10** (38.1 mg, 0.122 mmol) in CH_2Cl_2 (2 mL) was slowly added to a stirred solution of MeOH (0.05 mL, 1.2 mmol) and PMe_3 (1.0 M in THF, 0.02

mL, 0.02 mmol) in CH_2Cl_2 (10 mL) over 8 min at –20 °C. Additional PMe_3 (1.0 M in THF, 0.02 mL, 0.02 mmol) was added after stirring for 50 min and for 1.5 h. After stirring for 30 min, the reaction mixture was diluted with *n*-hexane and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 hexane/EtOAc) to afford vinylogous ester **25** (25.2 mg, 60% yield) as a red oil.

$[\alpha]_D^{26}$ –94.1 (*c* 1.35, $CHCl_3$); IR (neat) 3273, 2957, 1727, 1574, 1150, 812, 759, 701 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.18 (m, 5H), 6.39 (br s, 1H), 5.85 (s, 1H), 4.24 (d, *J* = 13.7 Hz, 1H), 4.09 (d, *J* = 13.7 Hz, 1H), 3.94 (d, *J* = 2.4 Hz, 1H), 3.71 (s, 3H), 3.27 (s, 3H), 1.94 (sept, *J* = 6.3 Hz, 1H), 1.73 (dd, *J* = 14.6, 5.9 Hz, 1H), 1.68 (dd, *J* = 14.6, 7.3 Hz, 1H), 1.03 (d, *J* = 6.3 Hz, 3H), 0.99 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.5, 178.2, 169.5, 136.8, 129.2 (2C), 128.3 (2C), 126.6, 94.7, 88.7, 63.4, 61.7, 56.4, 49.7, 42.7, 38.5, 24.1, 23.9, 23.5; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{20}H_{25}NO_5$ 360.1811; Found 360.1809.

(–)-Berkeleyamide (**3**) and 14-*epi*-**3**



25 (–)-Berkeleyamide D (**3**) 14-*epi*-**3**

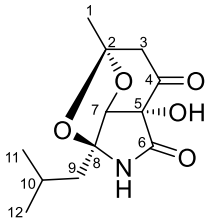
p-TsOH (7.4 mg, 0.039 mmol) was added to a stirred solution of **25** (14 mg, 0.039 mmol) in THF (2 mL) and H_2O (0.2 mL). The reaction mixture was stirred at 40 °C for 3 days. The reaction was diluted with $CHCl_3$ and H_2O and extracted with $CHCl_3$ three times. The organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (3:1 to 1:1 hexane/EtOAc) to afford berkeleyamide D (**3**) (6.3 mg, 49% yield) and 14-*epi*-**3** (5.4 mg, 42% yield) as colorless solids.

Berkeleyamide D (**3**): m.p. 114-121 °C; $[\alpha]_D^{26}$ –61.1 (*c* 0.54, MeOH); IR ($CHCl_3$) 3286, 2959, 1732, 1685, 1583, 758 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.39-7.29 (m, 5H), 6.72 (br s, 1H), 5.49 (s, 1H), 5.37 (s, 1H), 4.43 (d, *J* = 9.3 Hz, 1H), 4.02 (d, *J* = 17.6 Hz, 1H), 3.96 (d, *J* = 17.6 Hz, 1H), 3.03 (d, *J* = 10.2 Hz, 1H), 1.94 (sept, *J* = 6.3 Hz, 1H), 1.88 (dd, *J* = 14.6, 6.3 Hz, 1H), 1.62 (dd, *J* = 7.3, 6.3 Hz, 1H), 1.02 (d, *J* = 6.3 Hz, 3H), 1.01 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.4, 197.8, 163.9, 133.2, 129.2 (2C), 129.0 (2C), 127.8, 104.4, 95.3, 84.9, 75.2, 45.6, 37.4, 24.0, 23.9, 23.8; HRMS (FAB-

DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{18}H_{22}NO_5$ 332.1498; Found 332.1497.

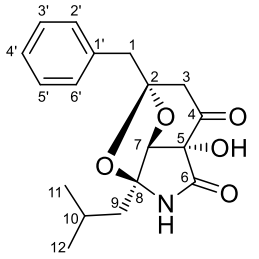
Compound 14-*epi*-**3**: m.p. 126.5-130.5 °C; $[\alpha]_D^{23}$ –18.1 (*c* 0.67, $CHCl_3$); IR ($CHCl_3$) 3474, 3363, 3019, 1744, 1710, 1675, 1572, 669 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 8.96 (br s, 1H), 7.41-7.22 (m, 5H), 5.99 (d, *J* = 5.9 Hz, 1H), 5.75 (s, 1H), 5.33 (s, 1H), 4.39 (d, *J* = 5.9 Hz, 1H), 3.96 (s, 2H), 1.99 (d, *J* = 14.1 Hz, 1H), 1.94 (sept, *J* = 6.8 Hz, 1H), 1.59 (dd, *J* = 14.1, 6.8 Hz, 1H), 0.94 (d, *J* = 6.8 Hz, 3H), 0.92 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 196.9, 192.5, 163.6, 134.8, 129.0 (2C), 128.6 (2C), 127.0, 104.0, 95.0, 87.6, 81.7, 43.8, 35.9, 25.0, 24.7, 22.5; HRMS (FAB-DFMS) *m*/*z*: $[M+H]^+$ Calcd for $C_{18}H_{22}NO_5$ 332.1498; Found 332.1520.

NMR chemical shifts of natural products and synthetic compounds



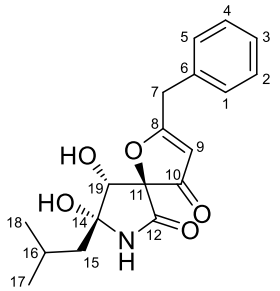
(+)-Rubrobramide (**1**)

	Natural ($CDCl_3$)		Synthetic ($CDCl_3$)	
	^{1}H (500 MHz)	^{13}C (125 MHz)	^{1}H (400 MHz)	^{13}C (100 MHz)
1	1.61 (s)	25.2	1.63 (s)	25.2
2		109.0		109.1
3a	2.98 (d, *J* = 18.4 Hz)	48.6	2.97 (d, *J* = 18.5 Hz)	48.6
3b	2.83 (d, *J* = 18.4 Hz)		2.85 (d, *J* = 18.5 Hz)	
4		199.4		199.2
5		82.6		82.4
6		169.2		168.4
7	4.87 (s)	86.4	4.88 (s)	86.5
8		93.1		92.8
9	1.84 (m)	45.3	1.86-1.81 (m)	45.5
10	1.87 (m)	24.2	1.92-1.87 (m)	24.3
11	1.01 (d, *J* = 6.3 Hz)	23.4	1.02 (d, *J* = 6.5 Hz)	23.5
12	1.01 (d, *J* = 6.3 Hz)	23.8	1.03 (d, *J* = 6.5 Hz)	23.7
5-OH	4.42 (br s)		4.23 (s)	
6-NH	7.38 (br s)		6.58 (br s)	



(+)-Talaramide A (**2**)

	Natural (DMSO-d_6)		Synthetic (DMSO-d_6)	
	^{1}H (500 MHz)	^{13}C (125 MHz)	^{1}H (400 MHz)	^{13}C (100 MHz)
1	3.10 (s)	43.1	3.11 (s)	43.1
2		108.7		108.7
3a	2.68 (d, J = 18.6 Hz)	47.5	2.68 (d, J = 18.5 Hz)	47.5
3b	2.78 (d, J = 18.6 Hz)		2.79 (d, J = 18.5 Hz)	
4		200.4		200.3
5		83.3		83.2
6		168.8		168.7
7	4.76 (s)	86.3	4.76 (s)	86.3
8		93.0		93.0
9	1.55 (m)	44.3	1.58-1.51 (m)	44.2
10	1.86 (m)	23.0	1.71-1.62 (m)	23.0
11	0.83 (d, J = 6.6 Hz)	23.4	0.84 (d, J = 7.3 Hz)	23.4
12	0.82 (d, J = 6.6 Hz)	23.8	0.83 (d, J = 7.3 Hz)	23.8
1’		134.4		134.4
2’/6’	7.29 (m)	130.7	7.33-7.23 (m)	130.7
3’/5’	7.29 (m)	127.9	7.33-7.23 (m)	127.9
4’	7.25 (m)	126.8	7.33-7.23 (m)	126.7
5-OH	6.59 (br s)		6.58 (s)	
6-NH	9.37 (s)		9.37 (s)	



(–)-Berkeleyamide D (**3**)

	Natural ($CDCl_3$)		Synthetic ($CDCl_3$)	
	^{1}H (300 MHz)	^{13}C (75 MHz)	^{1}H (400 MHz)	^{13}C (100 MHz)
1, 5	7.33 (m)	129.2	7.39-7.29 (m)	129.2
2, 4	7.33 (m)	129.0	7.39-7.29 (m)	129.0
3	7.33 (m)	127.0	7.39-7.29 (m)	127.8
6		133.2		133.2
7	3.98 (d, J = 17.4 Hz), 3.96 (d, J = 17.4 Hz)	37.4	4.02 (d, J = 17.6 Hz), 3.96 (d, J = 17.6 Hz)	37.4
8		197.8		197.8
9	5.35 (br s)	104.4	5.37 (s)	104.4
10		199.4		199.4
11		95.3		95.3
12		164.1		163.9
13	6.78 (br s)		6.72 (br s)	
14		84.9		84.9
15	1.88 (m, 2H)	45.5	1.88 (dd, J = 14.6, 6.3 Hz), 1.62 (dd, J = 7.3, 6.3 Hz)	45.6
16	1.92 (m)	24.0	1.94 (sept, J = 6.3 Hz)	24.0
17	1.00 (d, J = 5.2 Hz, 3H)	23.9	1.02 (d, J = 6.3 Hz)	23.9
18	0.98 (d, J = 5.2 Hz, 3H)	23.8	1.01 (d, J = 6.3 Hz)	23.8
19	4.41 (d, J = 10.0 Hz)	75.1	4.43 (d, J = 9.3 Hz)	75.2
OH, C-19	3.04 (d, J = 10.0 Hz)		3.03 (d, J = 10.2 Hz)	
OH, C-14	5.48 (br s)		5.49 (s)	

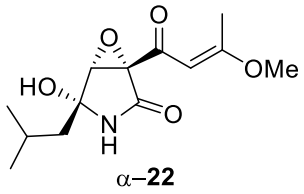
B3LYP 6-311G+ (d, p) Calculated Cartesian Coordinates

In the previous studies by Tsubaki's group, **3** was obtained as a single diastereomer at the hemiaminal position (C-14), which was attributed to the large energy difference between **3** and 14-*epi*-**3** (7.2 kcal/mol, see ref 9a). However, we obtained both **3** and 14-*epi*-**3** in a 1.2:1 ratio. In addition, structurally related molecules **16** and **17** (14-*epi*-**16**) were formed in a 2.7:1 ratio.

As our results cannot be explained by the relative thermodynamic stability of the spiro-cyclic products, we calculated the energy difference between the plausible intermediates α-**22** and β-**22** with a methyl substituent, which would lead to **16** and **17**, respectively (see Scheme 4 in the manuscript). The calculations revealed that the conformation of α-**22** with the lowest energy was more stable than that of β-**22** by 0.67 kcal/mol (see S17-S20), meaning that they would be present in a ratio of 2:1~3:1. We speculate that the mixture of α-**22** and β-**22** initially gave **16** and **17** in an almost 1:1 ratio, and over the long reaction time (3 days) for conversion of **15** to **16** and **17**, there was gradual isomerization from **17** to the more stable **16**, resulting in the 2.7:1 ratio. On the other hand, the similar reaction of **25** generated **3** and 14-*epi*-**3** in a 1.2:1 ratio, which can be rationalized by the shorter reaction time (2 days) for conversion of **25** to **3** and 14-*epi*-**3**.

Comparison of stability between α-**22** and β-**22** by DFT Calculations. Conformational analyses were performed using conformational search with MMFF and energy calculation with B3LYP/6-31G* level implemented in version 1.4.8 of the Spartan 18 software (Wavefunction, Inc., Irvine CA, America). The lower energy conformers of each compound, which differed from the most stable confer by less than 2 kcal/mol, were optimized using DFT calculations at the B3LYP/6-311+G(d,p) level, that were implemented in the Gaussian 09 program package (Gaussian, Inc., Wallingford, CT, USA). The lowest energy conformations were determined by comparing the energies of each conformer.

α-**22**



α–**22**

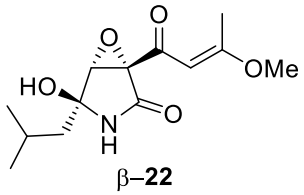
-937.665733 Hartree

Gibbs Free Energy: -588394.6241 kcal/mol

N	-1.450426	-0.214785	1.209393
C	-1.960267	-0.623023	-0.067659
C	-0.788129	-1.295605	-0.736744
C	0.376747	-1.255416	0.227204
C	-0.157658	-0.611407	1.452888
O	0.453216	-0.475795	2.498356
O	-2.968759	-1.572157	0.180400
O	-0.298737	-2.491846	-0.120165
C	1.743160	-1.083136	-0.329310
O	2.175701	-1.860847	-1.172159
C	2.529327	0.088231	0.159371
C	3.609907	0.598404	-0.465881
O	4.362960	1.661052	0.000894
C	4.131358	0.093213	-1.785221
C	4.073225	2.201743	1.285023
H	-0.663347	-1.159877	-1.797960

C	-2.485553	0.555222	-0.899739
C	-3.517692	1.483654	-0.219264
C	-4.810886	0.773132	0.178486
C	-3.846387	2.650342	-1.157887
H	-2.050267	0.076770	1.968504
H	-2.519309	-2.360868	0.533523
H	2.187550	0.518751	1.090259
H	4.801751	0.829165	-2.243722
H	4.700872	-0.830596	-1.644257
H	3.323520	-0.076696	-2.503581
H	4.803824	2.989663	1.490614
H	3.076701	2.654374	1.301327
H	4.171777	1.438366	2.063539
H	-2.927719	0.160932	-1.825136
H	-1.629999	1.173934	-1.205615
H	-3.069923	1.911438	0.686152
H	-5.239100	0.222894	-0.665889
H	-4.643324	0.072115	1.000927
H	-5.560072	1.493219	0.525763
H	-4.537074	3.353006	-0.679542
H	-2.940083	3.205457	-1.422305
H	-4.310762	2.296076	-2.084565

β-**22**



β–**22**

-937.664661Hartree

Gibbs Free Energy: -588393.9514 kcal/mol

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C	0.113742	-1.515835	-0.115715
N	-1.174398	-1.733111	0.297686
C	-2.001742	-0.552665	0.545448
C	-1.041267	0.571640	0.131988
O	0.936619	-2.377745	-0.357424
O	-0.627506	0.517786	-1.221604
C	-3.320393	-0.646272	-0.235107
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C	2.800745	0.043156	-0.101552
C	4.032100	0.608825	0.058305
O	5.173335	-0.092984	-0.034321
C	4.309188	2.050753	0.347162
C	5.125221	-1.495686	-0.325232
H	-1.564670	-2.665486	0.269434
H	-1.128666	1.554885	0.576440
H	-3.731283	-1.638733	-0.015634
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H	-4.901087	2.490996	-0.250222
H	-3.196606	2.232230	0.089715
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H	2.703321	-1.012251	-0.308500
H	3.746930	2.384711	1.220393
H	3.971055	2.675253	-0.483055
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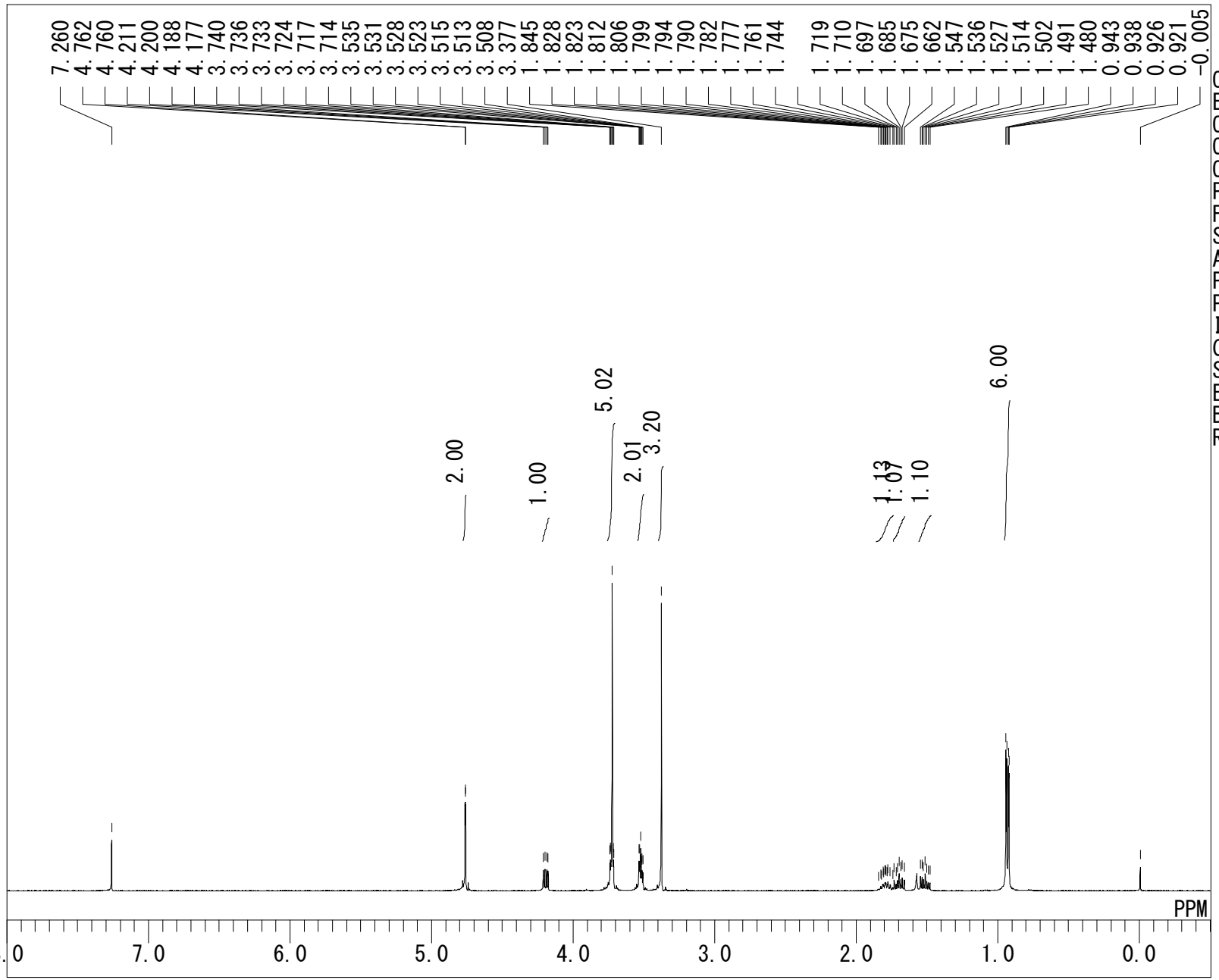
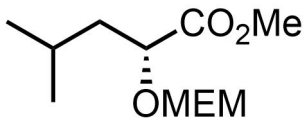
H	6.162866	-1.821159	-0.350066
H	4.581442	-2.035049	0.453991
H	4.652582	-1.674153	-1.293996

Computations were carried out with the SPARTAN' 18 series of programs:Y. Shao, L. Y. Molnar, Y. Jung, J. Kussmann, C. Ochsenfeld, S. T. Brown, A. T. B. Gilbert, L. V. Slipchenko, S. V. Levchnko, D. P. O'Neill, R. A DiStasio Jr., R. C. Lochan, T. Wang, G. J. O. Beran, N. A. Besley, J. M. Herbert, C. Y. Lin, T. Van Voorhis, S. H. Chien, A. Sodt, R. P. Steele, V. A. Rassolov, P. E. Maslen, P. P. Korambath, R. D. Adamson, B. Austin, J. Baker, E. F. C. Byrd, H. Dachsel, R. J. Doerksen, A. Dreuw, B. D. Dunietz, A. D. Dutoi, T. R. Furlani, S. R. Gwaltney, A. Heyden, S. Hirata, C-P. Hsu, G. Kedziora, R. Z. Khalliulin, P. Klunzinger, A. M. Lee, M. S. Lee, W. Z. Liang, I. Lotan, N. Nair, B. Peters, E. I. Proynov, P. A. Pieniazek, Y. M. Rhee, J. Ritchie, E. Rosta, C. D. Sherrill, A. C. Simmonett, J. E. Subotnik, H. L. Woodcock III, W. Zhang, A. T. Bell, A. K. Chakraborty, D. M. Chipman, F. J. Keil, A. Warshel, W. J. Hehre, H. F. Schaefer, J. Kong, A. I. Krylov, P. M. W. Gill and M. Head-Gordon, *Phys. Chem. Chem. Phys.* **8**, 3172 (2006).

Computations were carried out with the GAUSSIAN 09 series of programs: Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Mennucci, B., Petersson, G. A., Nakatsuji, H., Caricato, M., Li, X., Hratchian, H. P., Izmaylov, A. F., Bloino, J., Zheng, G., Sonnenberg, J. L., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T., Montgomery, J. A., Jr., Peralta, J. E., Ogliaro, F., Bearpark, M., Heyd, J. J., Brothers, E., Kudin, K. N., Staroverov, V. N., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Rega, N., Millam, J. M., Klene, M., Knox, J. E., Cross, J. B., Bakken, V., Adamo, C.,

Jaramillo, J., Gomperts, R., Stratmann, R. E., Yazyev, O., Austin, A. J., Cammi, R., Pomelli, C., Ochterski, J. W., Martin, R. L., Morokuma, K., Zakrzewski, V. G., Voth, G. A., Salvador, P., Dannenberg, J. J., Dapprich, S., Daniels, A. D., Farkas, Ö., Foresman, J. B., Ortiz, J. V., Cioslowski, J., Fox, D. J. Gaussian 09 Revision D.01, Gaussian, Inc.: Wallingford, CT, 2009.

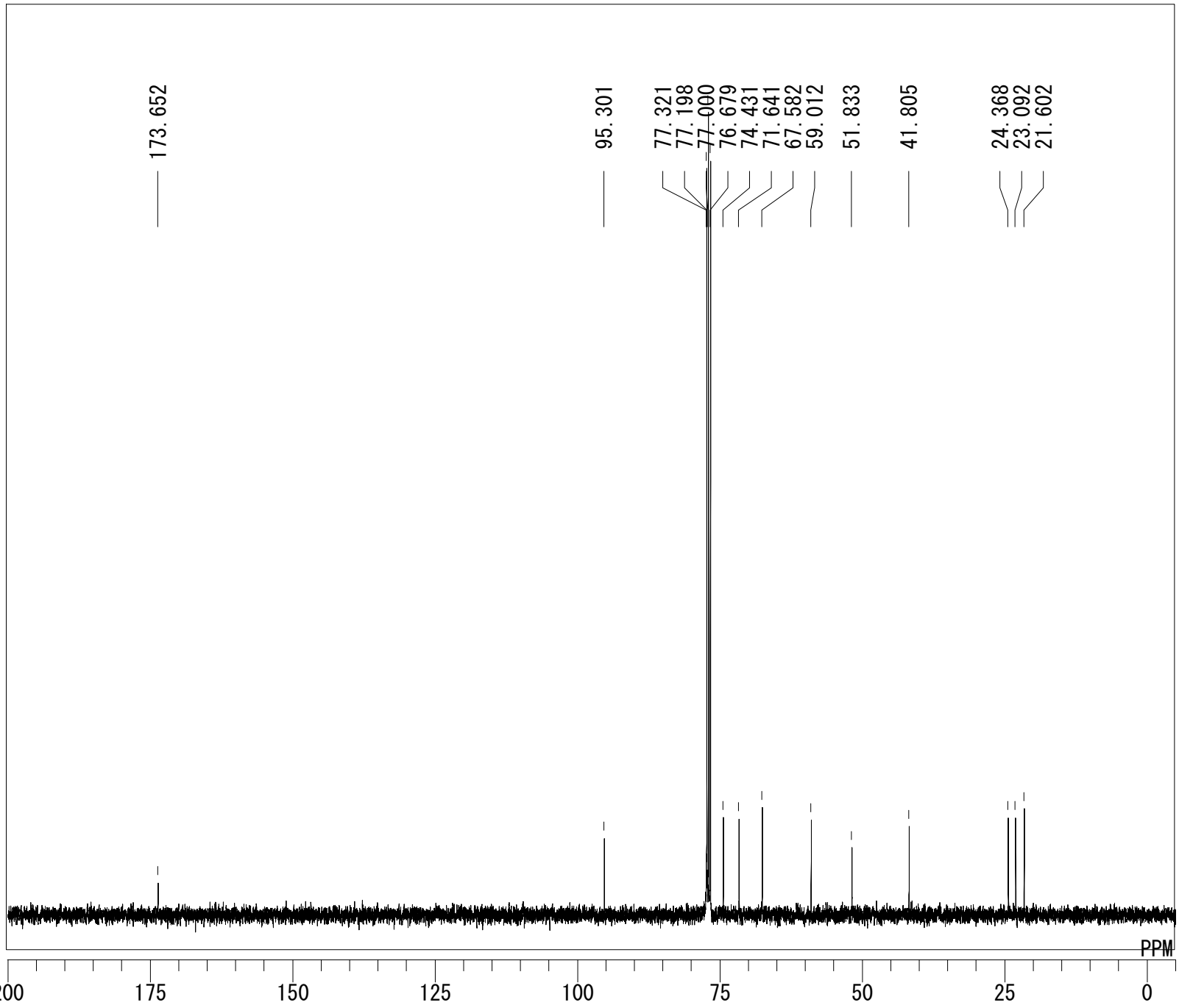




S2

^{1}H NMR

$CDCl_3$ (400 MHz)



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EXMOD	BCM	
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	316	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.4	c
SLVNT	CDCL3	
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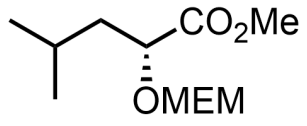
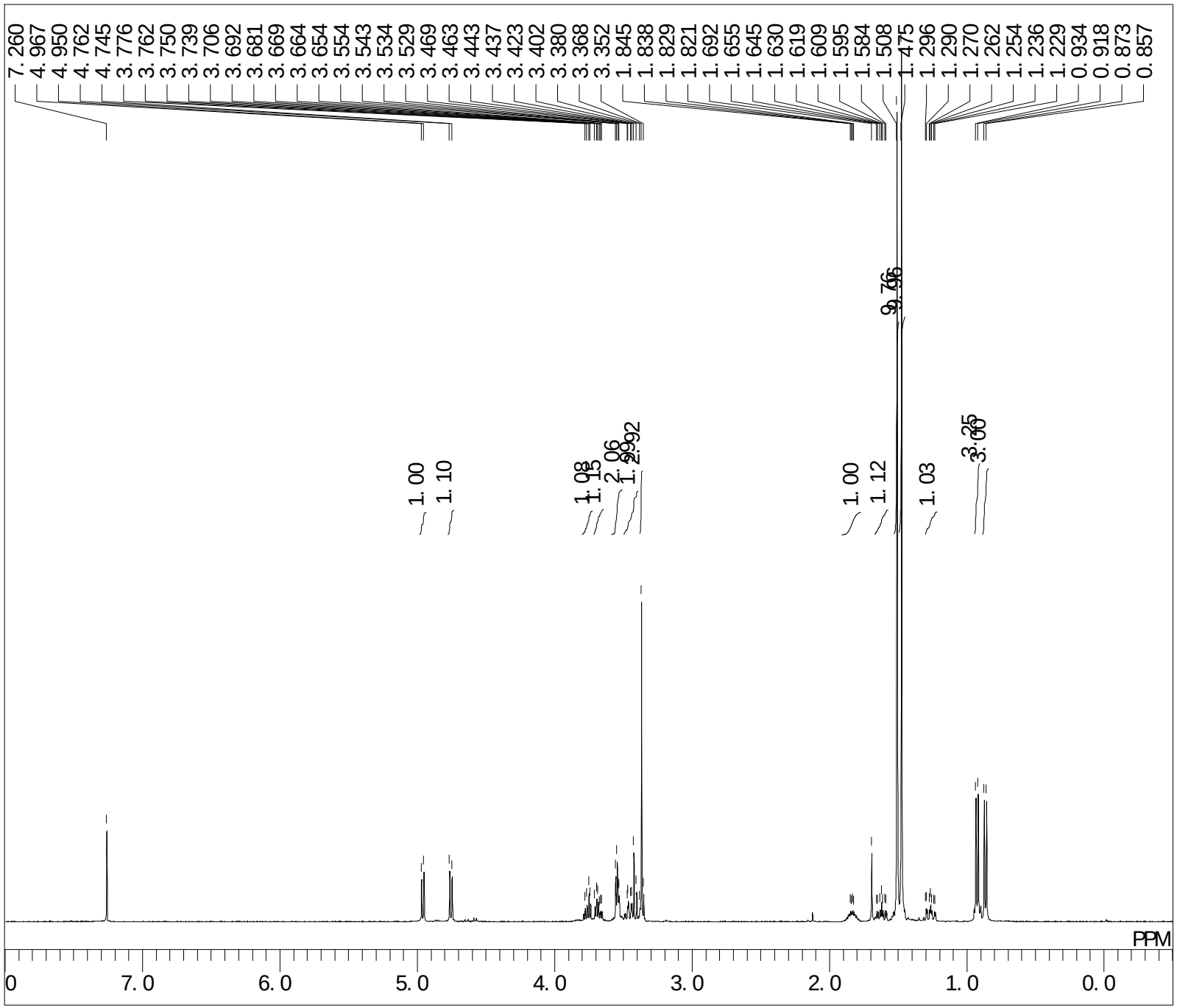
BF	1.20	Hz
RGAIN	25	



S2

^{13}C NMR

$CDCl_3$ (100 MHz)



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OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
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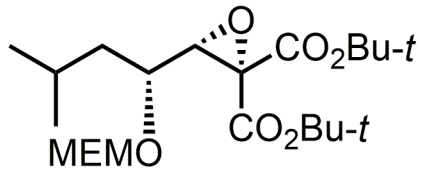
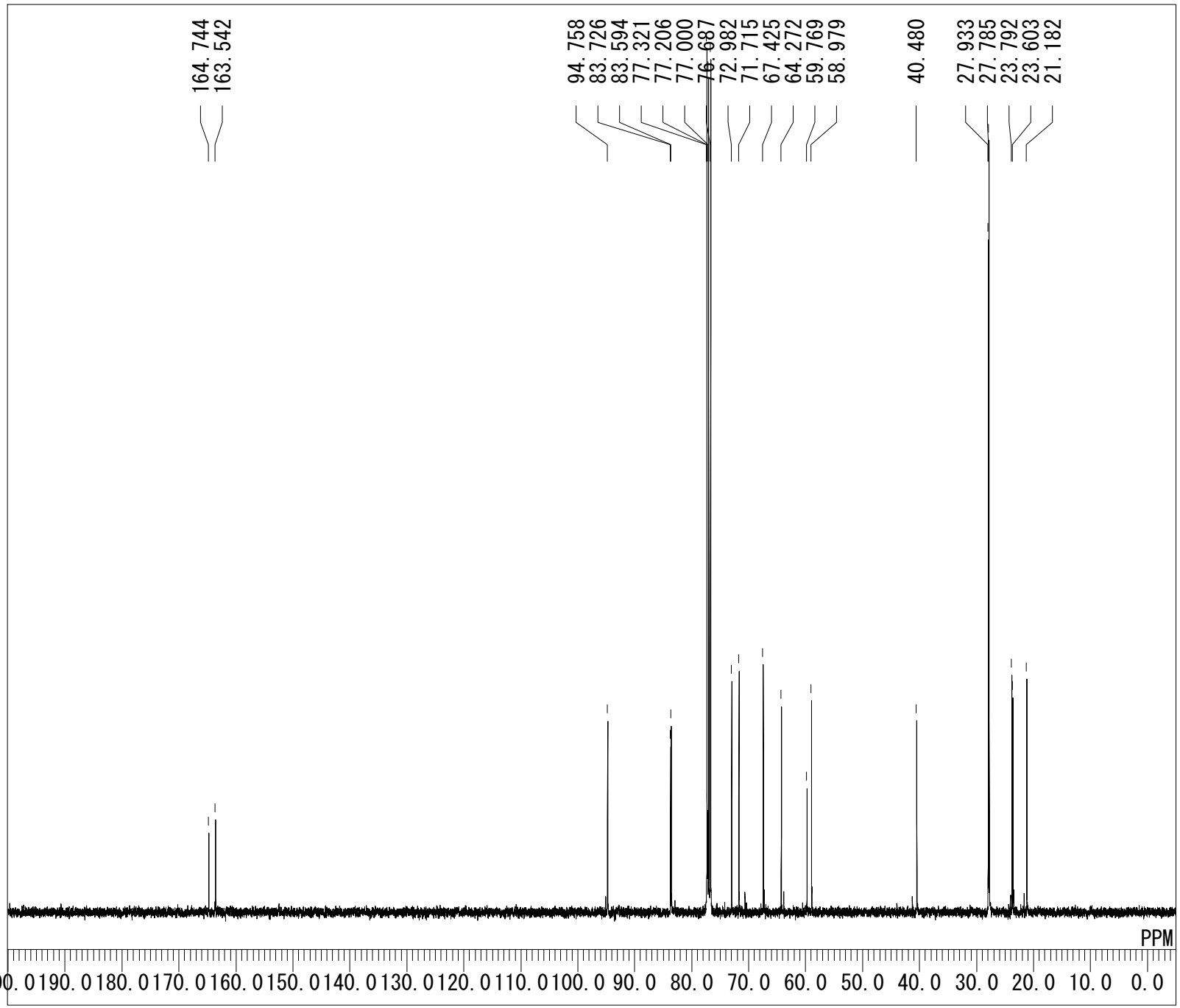
PD	4.9500	sec
PW1	5.60	usec
IRNUC	1H	
CTEMP	23.9	c
SLVNT	CDCL3	
EXREF	7.26	ppm
BF	0.12	Hz
RGAIN	13	



13

^{1}H NMR

$CDCl_3$ (400 MHz)



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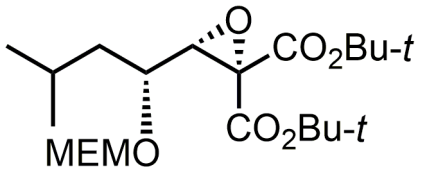
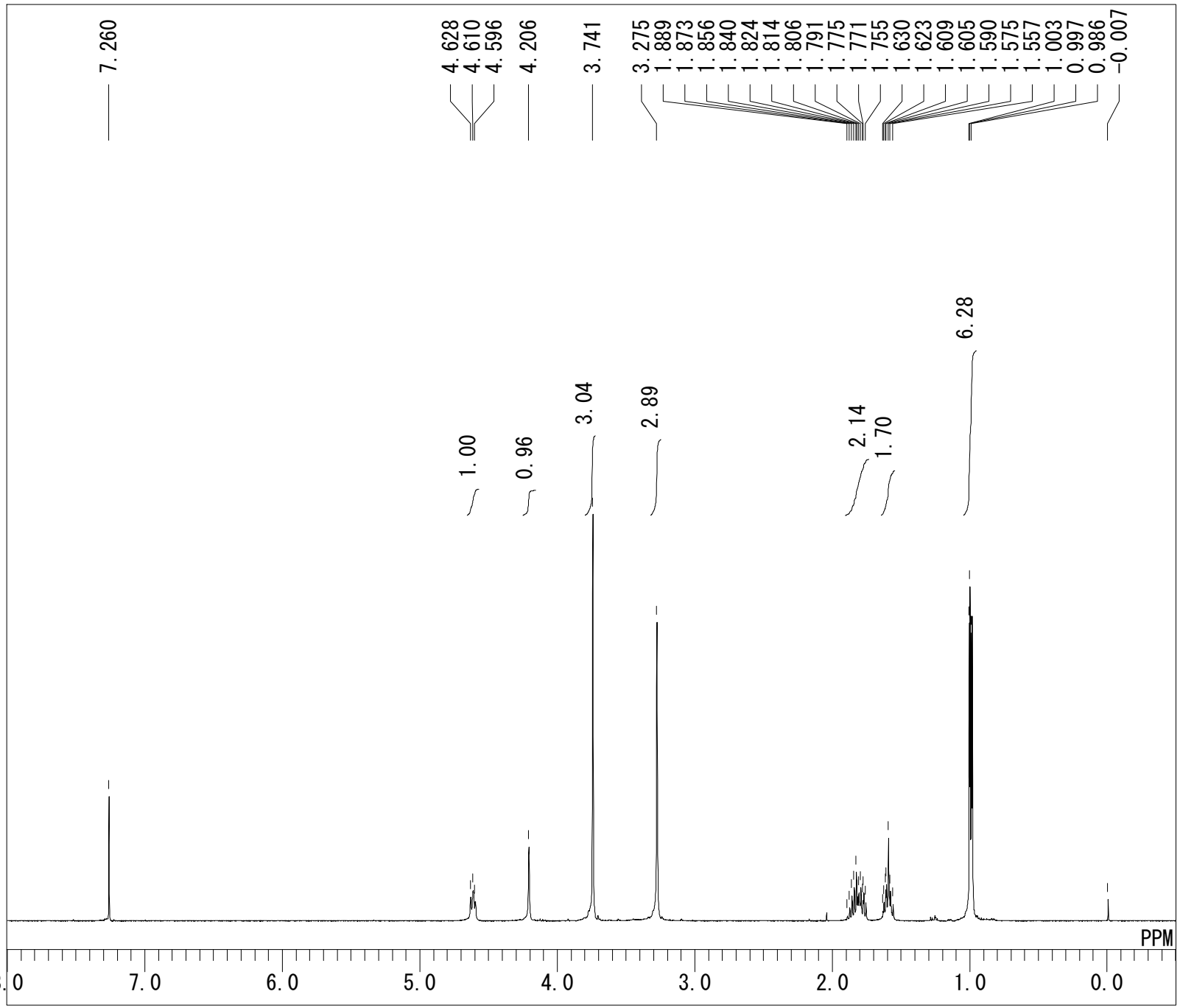
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	1000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.7	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	1.20	Hz
RGAIN	25	



13

^{13}C NMR

$CDCl_3$ (100 MHz)



OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
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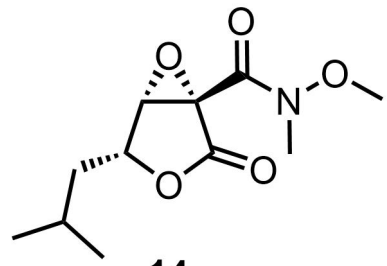
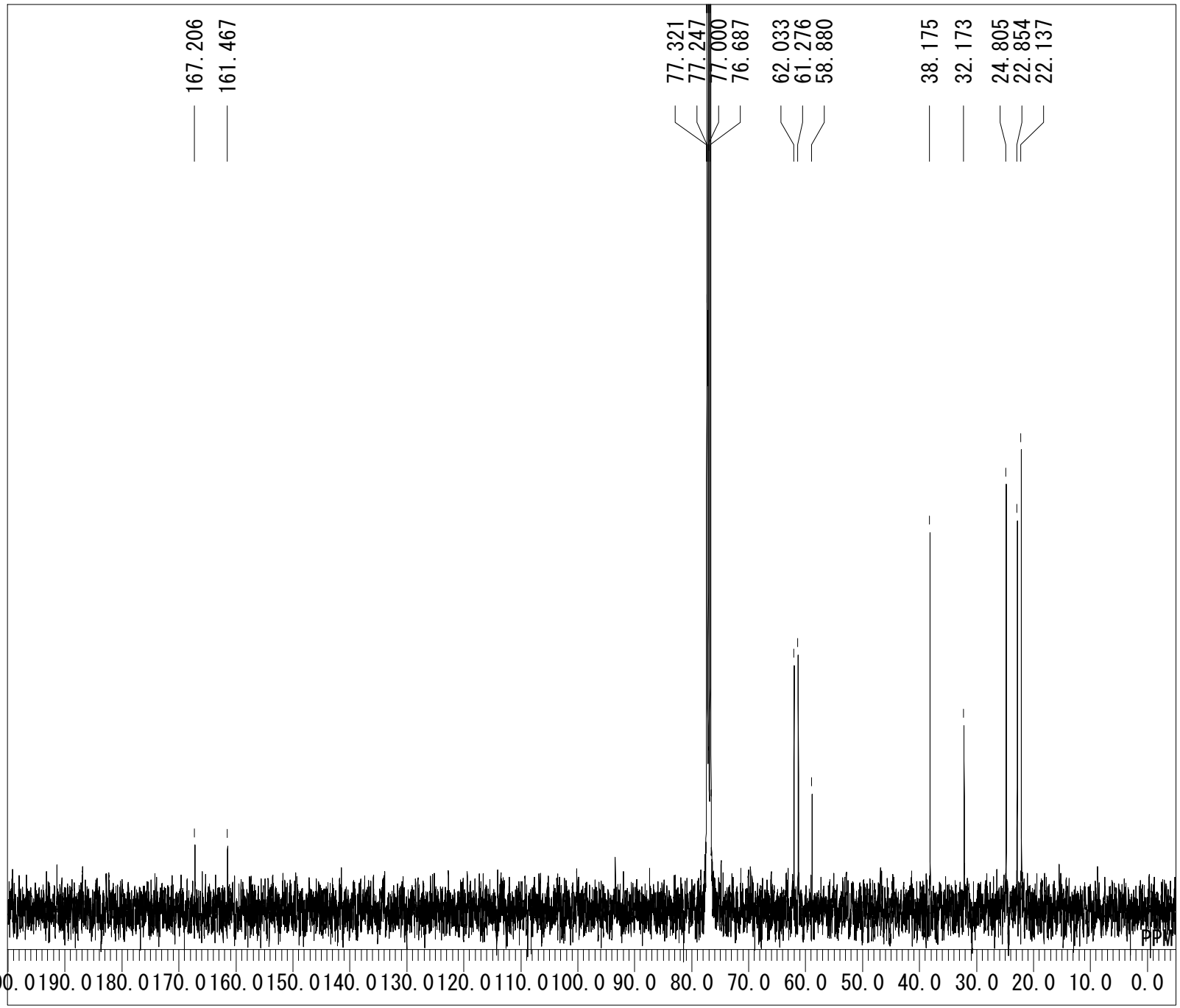
PD	4.9500	sec
PW1	5.60	usec
IRNUC	1H	
CTEMP	21.8	c
SLVNT	CDCL3	
EXREF	7.26	ppm
BF	0.09	Hz
RGAIN	18	



14

^{1}H NMR

$CDCl_3$ (400 MHz)



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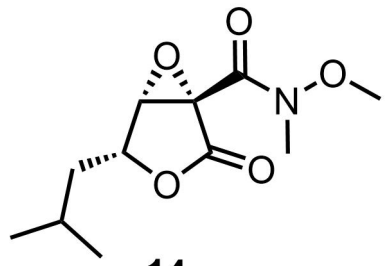
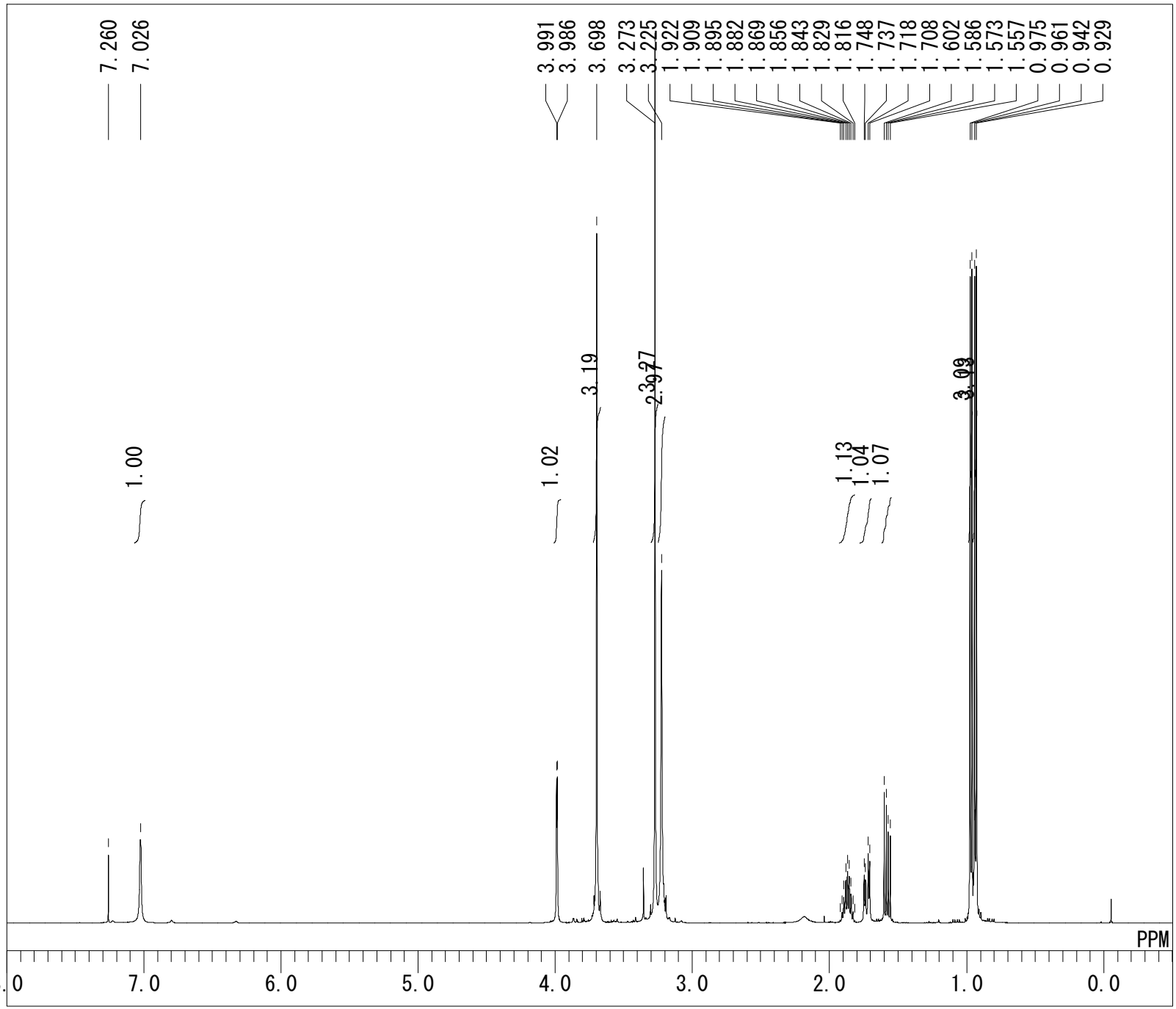
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	22.6	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	2.00	Hz
RGAIN	25	



14

^{13}C NMR

$CDCl_3$ (100 MHz)



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OBSET		4.38 KHz
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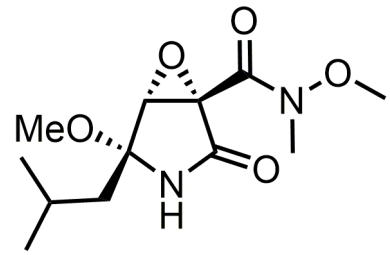
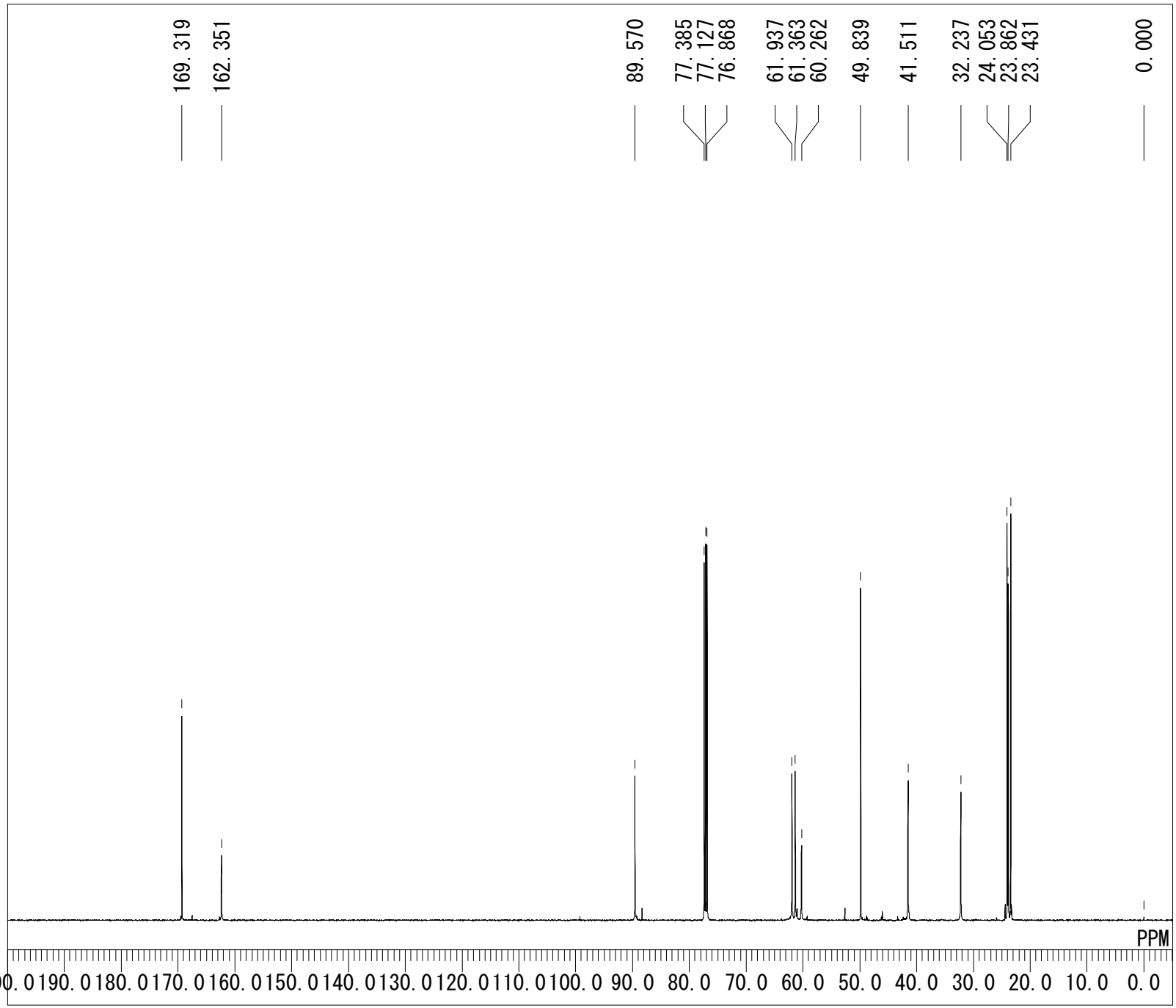
POINT		26214
FREQU		7429.42 Hz
SCANS		16
ACQTM		3.5285 sec
PD		5.0000 sec
PW1		6.25 usec
IRNUC	1H	
CTEMP		23.8 c
SLVNT	CDCL3	
EXREF		7.26 ppm
BF		0.09 Hz
RGAIN		24



11

^{1}H NMR

$CDCl_3$ (500 MHz)



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OBFIN	6.00	Hz
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PD	2.0000	sec
PW1	3.42	usec
IRNUC	1H	
CTEMP	23.9	c
SLVNT	CDCL3	
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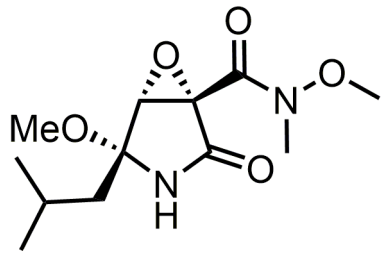
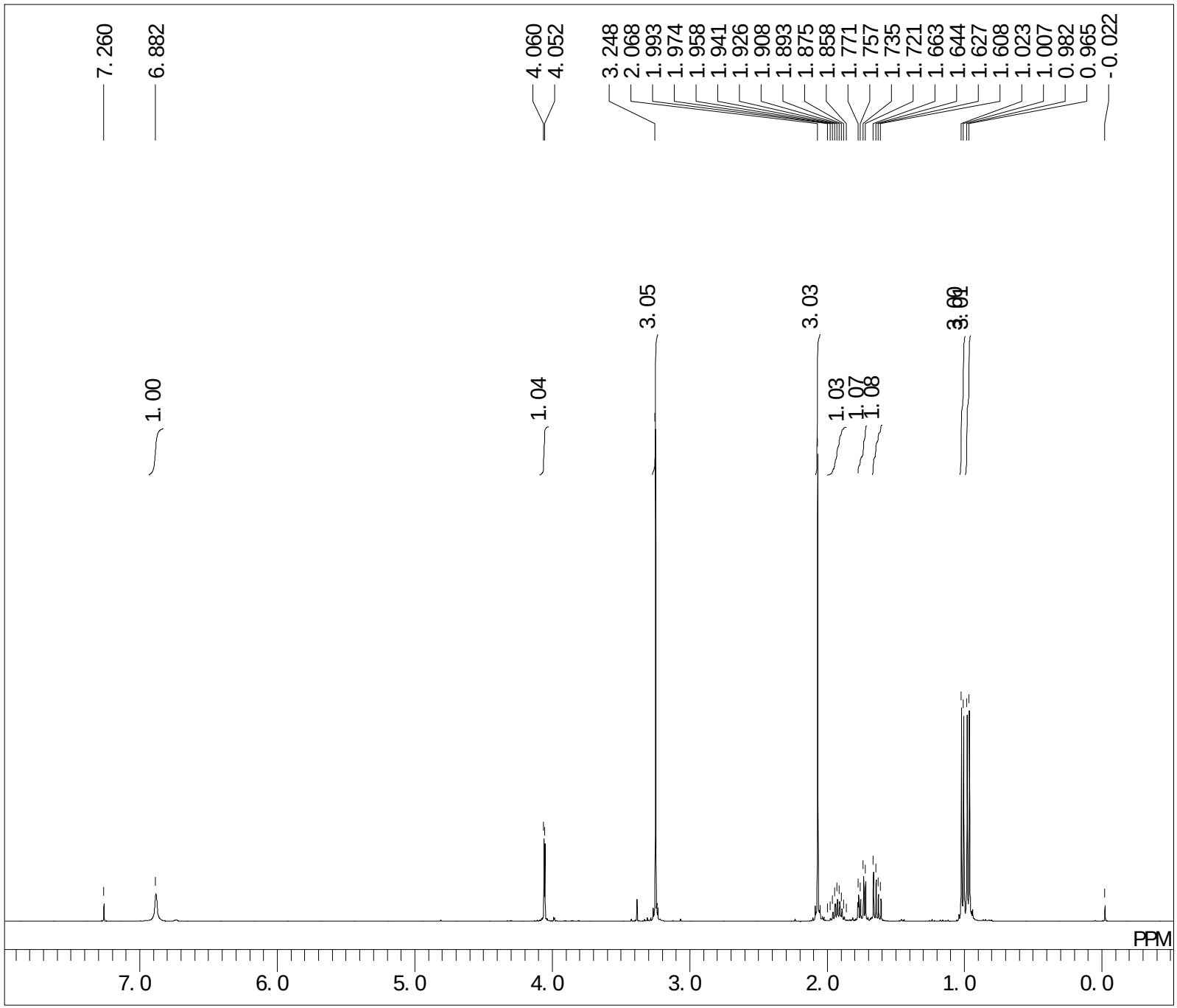
BF	0.09	Hz
RGAIN	60	



11

^{13}C NMR

$CDCl_3$ (125 MHz)



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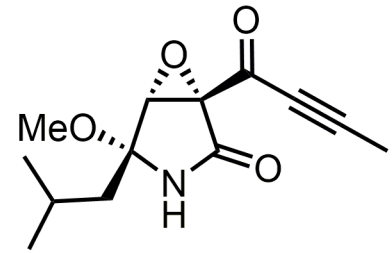
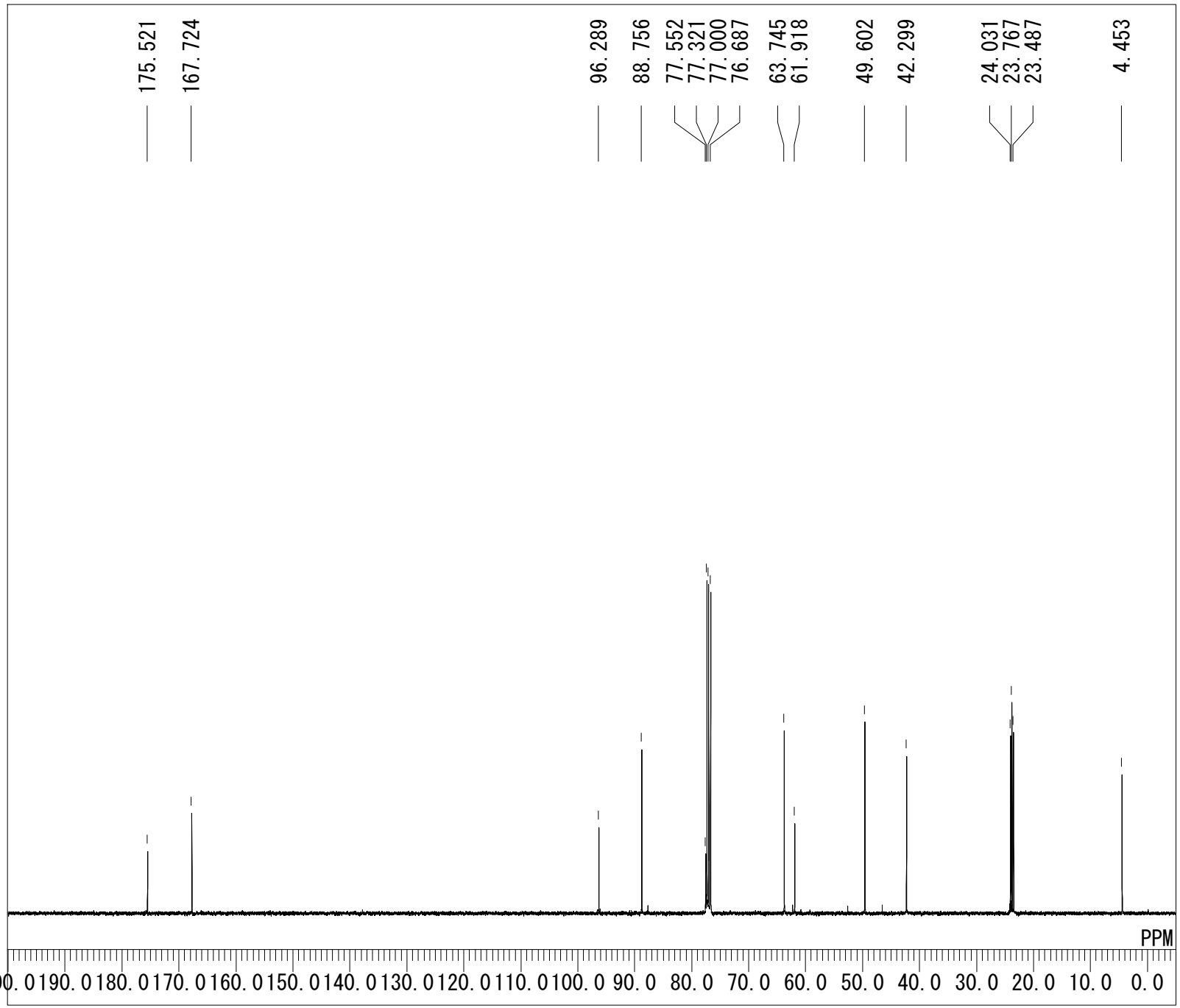
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.60	usec
INUC	1H	
CTEMP	24.8	c
SLVNT	CDCL3	
EXREF	7.26	ppm
BF	0.12	Hz
RGAIN	14	



9

^{1}H NMR

$CDCl_3$ (400 MHz)



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SCANS	1000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	25.3	c
SLVNT	CDCL3	
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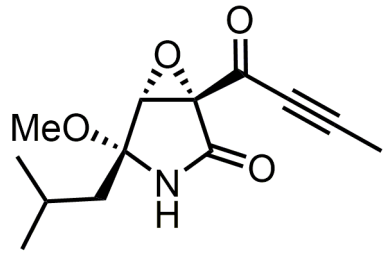
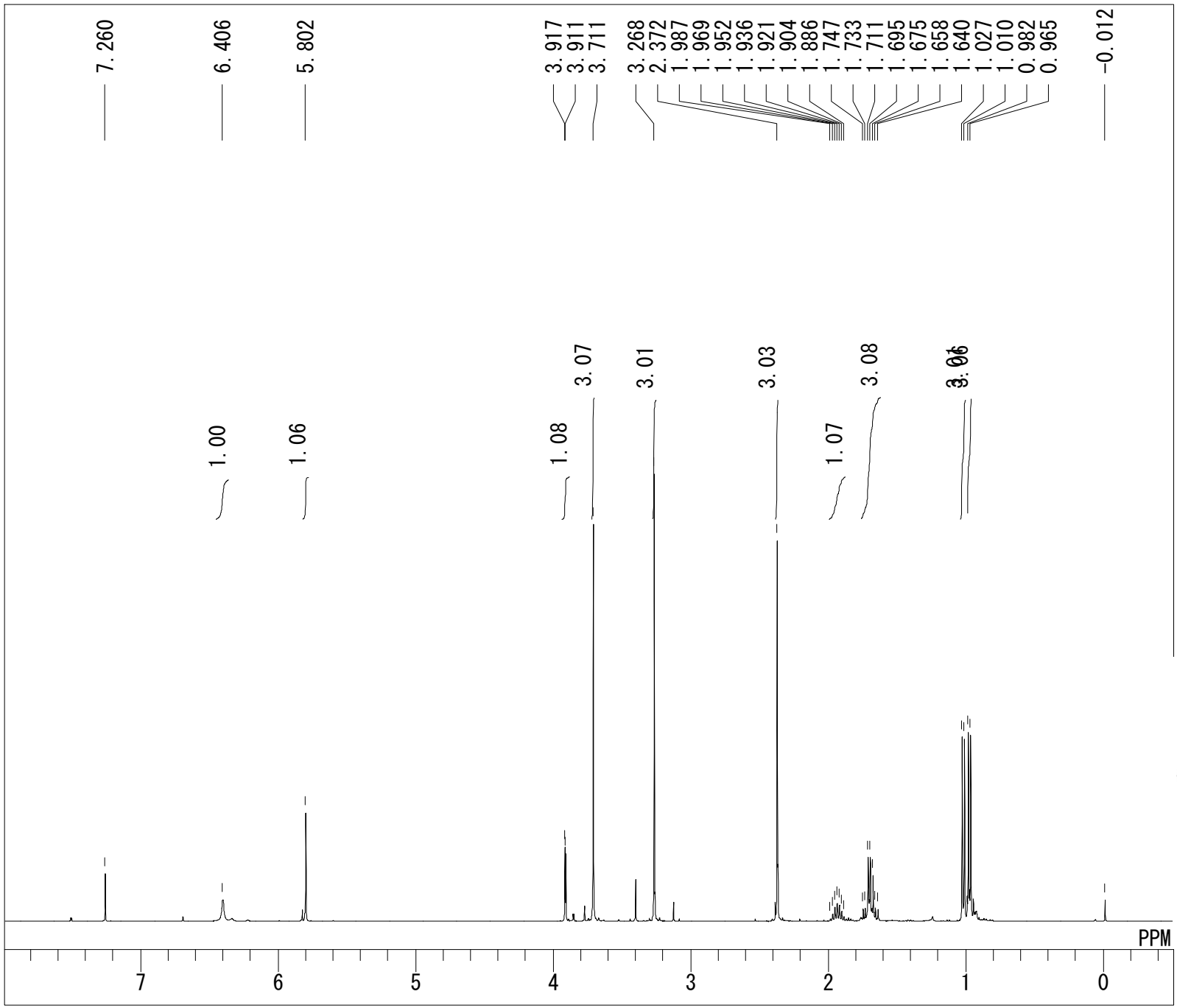
RGAIN	25	



9

^{13}C NMR

$CDCl_3$ (100 MHz)



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OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
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PD	4.9500	sec
PW1	5.60	usec
IRNUC	1H	
CTEMP	23.7	c
SLVNT	CDCL3	
EXREF	7.26	ppm
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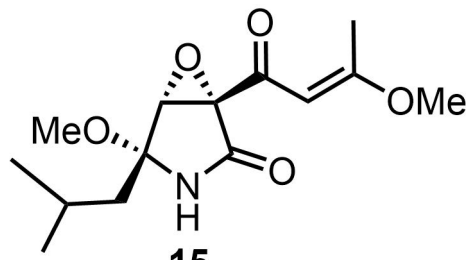
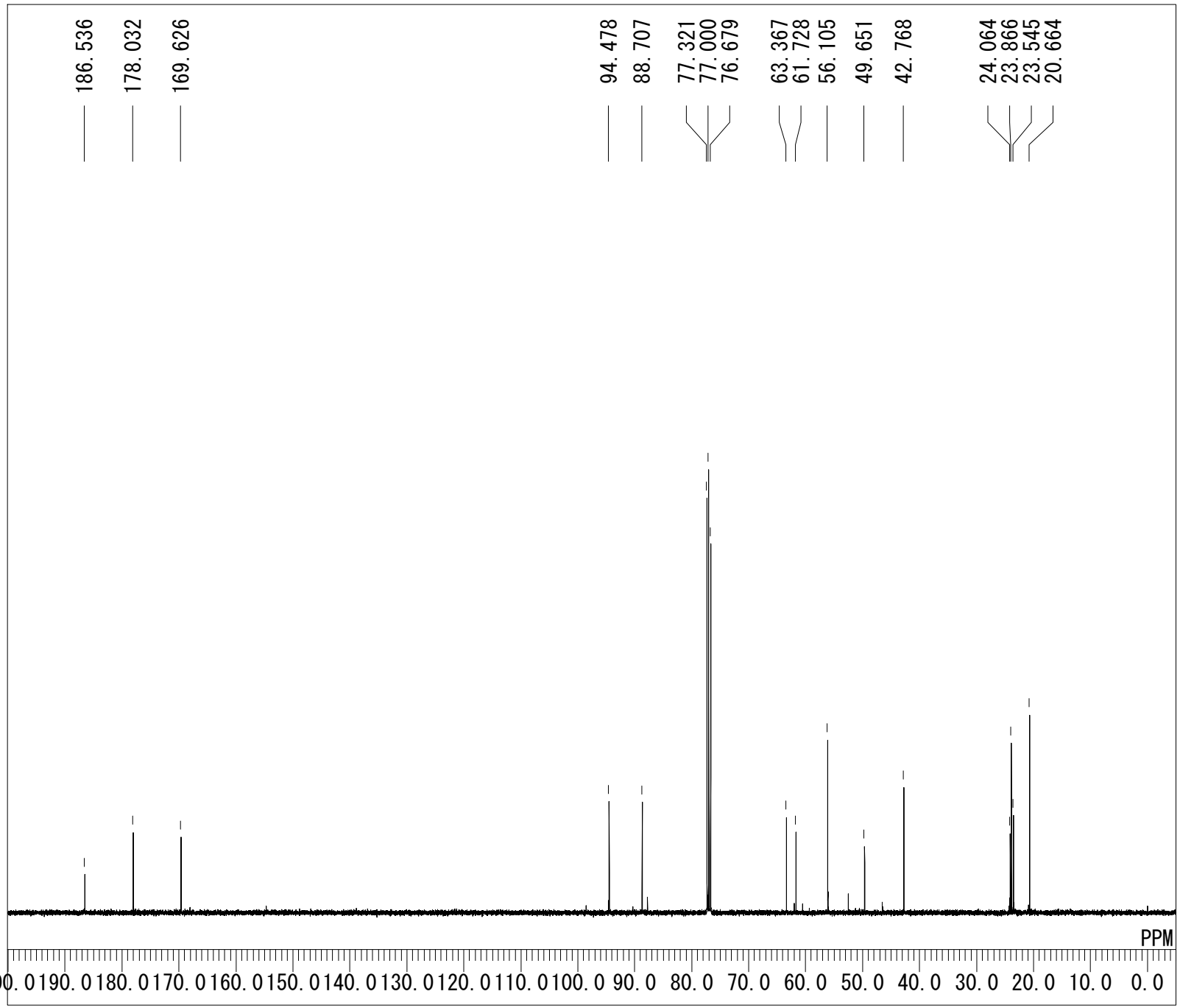
RGAIN	16	



15

^{1}H NMR

$CDCl_3$ (400 MHz)



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OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
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SCANS	2000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.4	c
SLVNT	CDCL3	
EXREF	77.00	ppm
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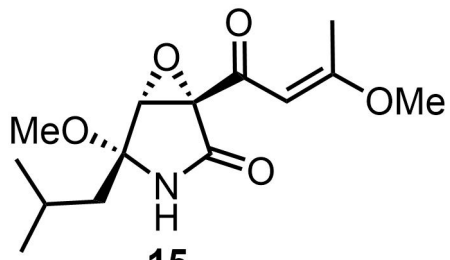
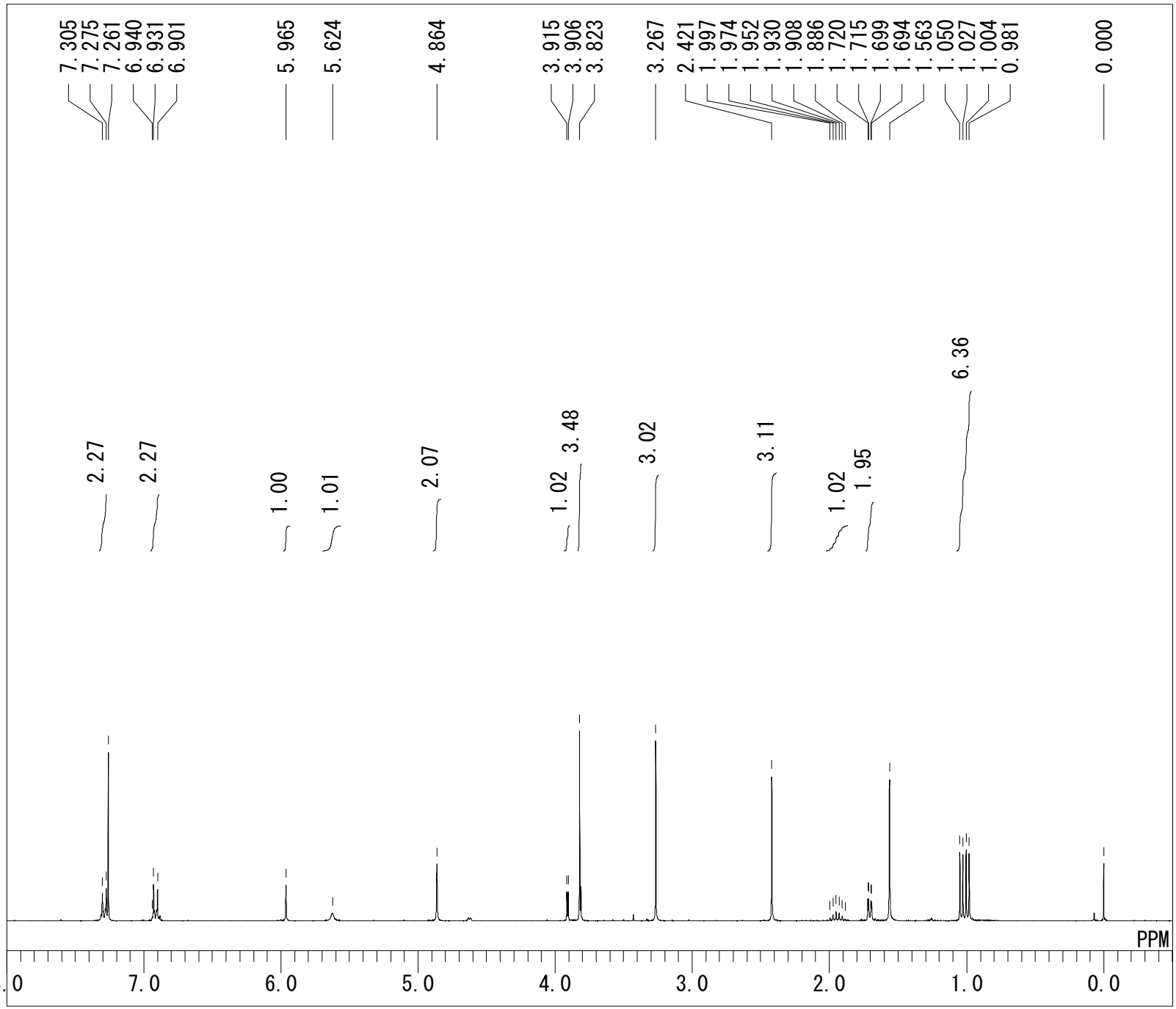
RGAIN	25	



15

^{13}C NMR

$CDCl_3$ (100 MHz)



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PD	1.5440	sec
PW1	5.30	usec
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CTEMP	26.2	c
SLVNT	CDCL3	
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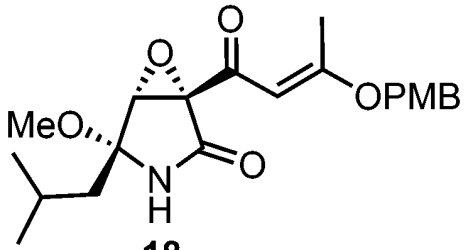
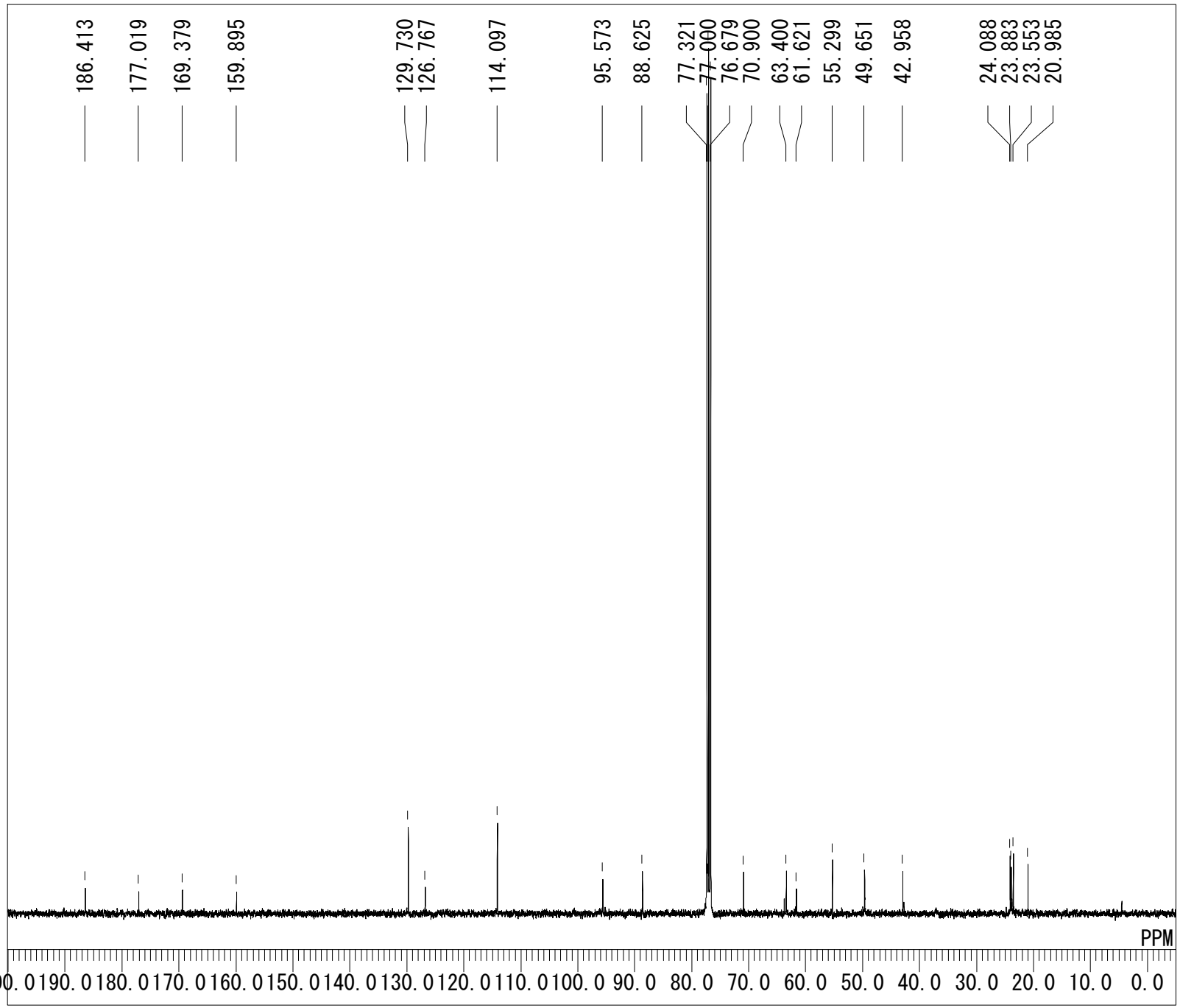


18

^{1}H NMR

$CDCl_3$ (300 MHz)



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ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	23.5	c
SLVNT	CDCL3	
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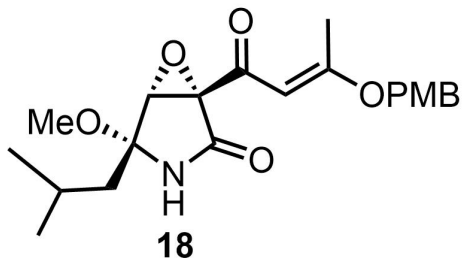
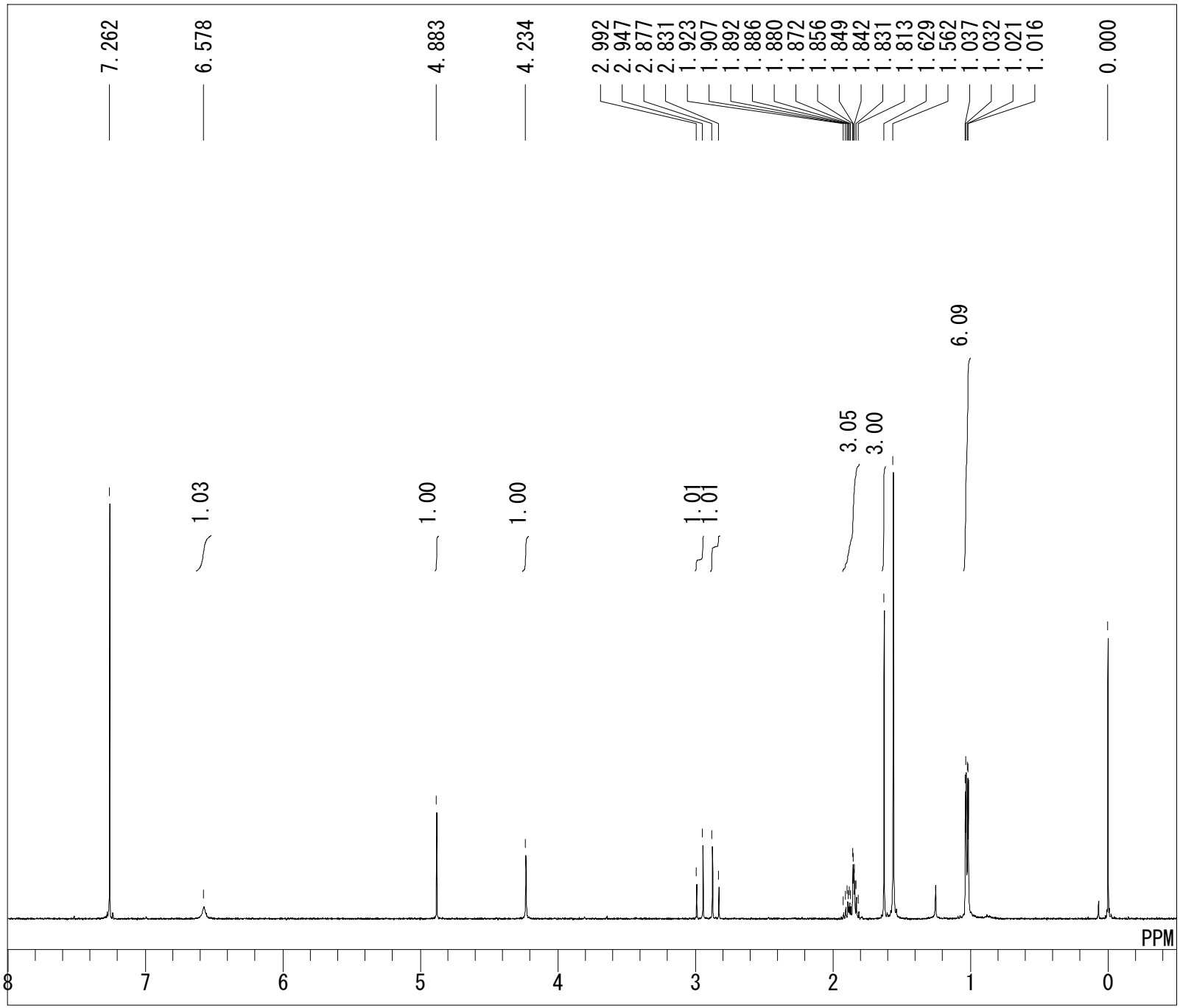
RGAIN	25	



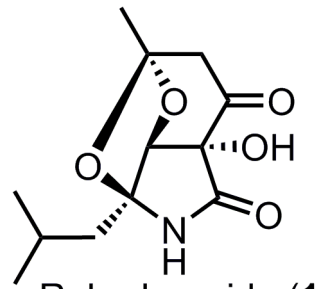
18

^{13}C NMR

$CDCl_3$ (100 MHz)



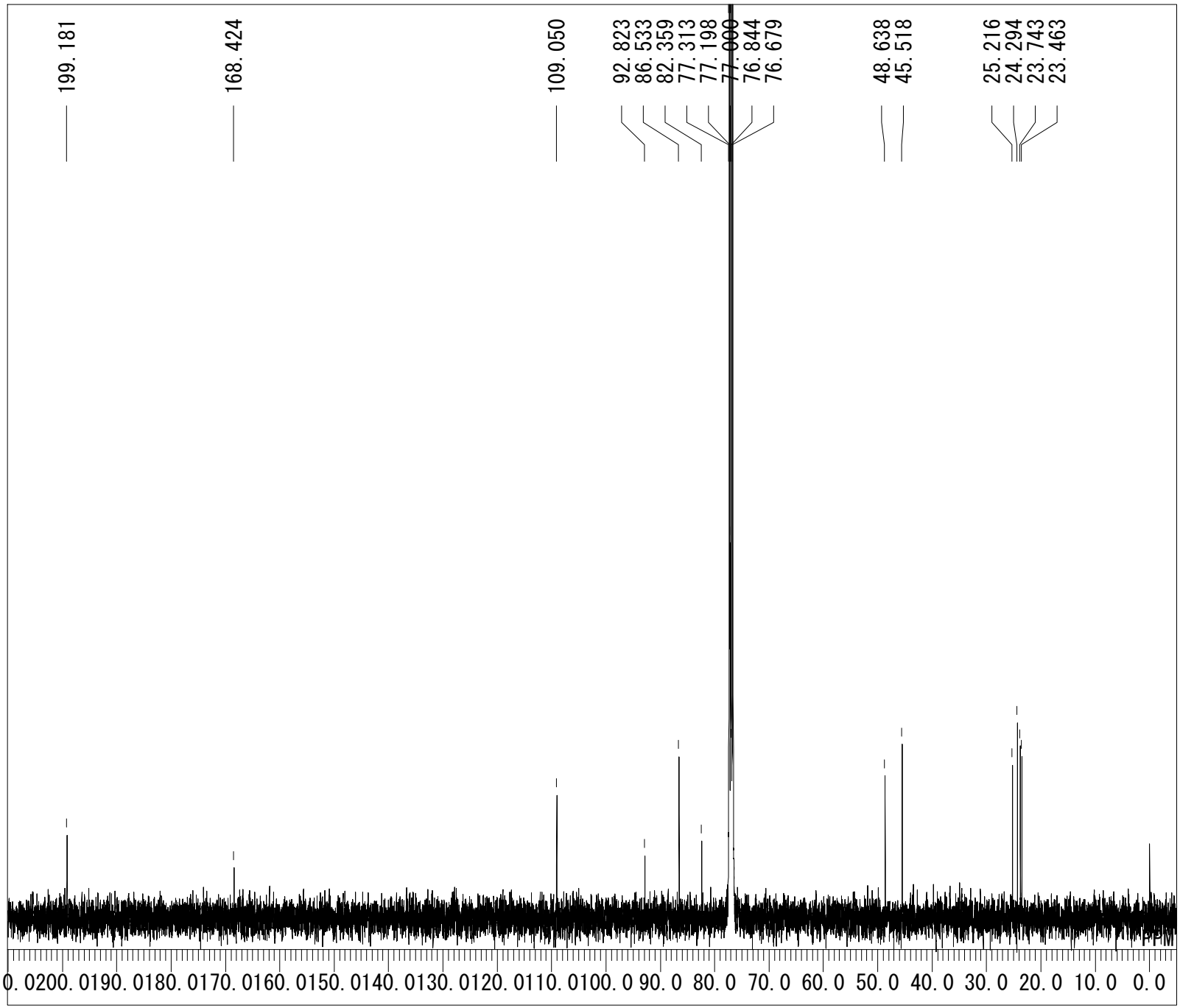
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.60	usec
IRNUC	1H	
CTEMP	23.6	c
SLVNT	CDCL3	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	23	



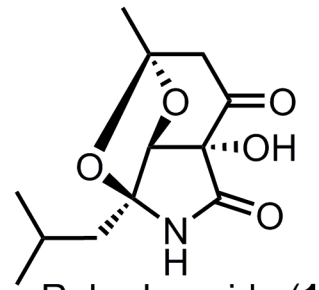
Rubrobramide (**1**)

^{1}H NMR

$CDCl_3$ (400 MHz)



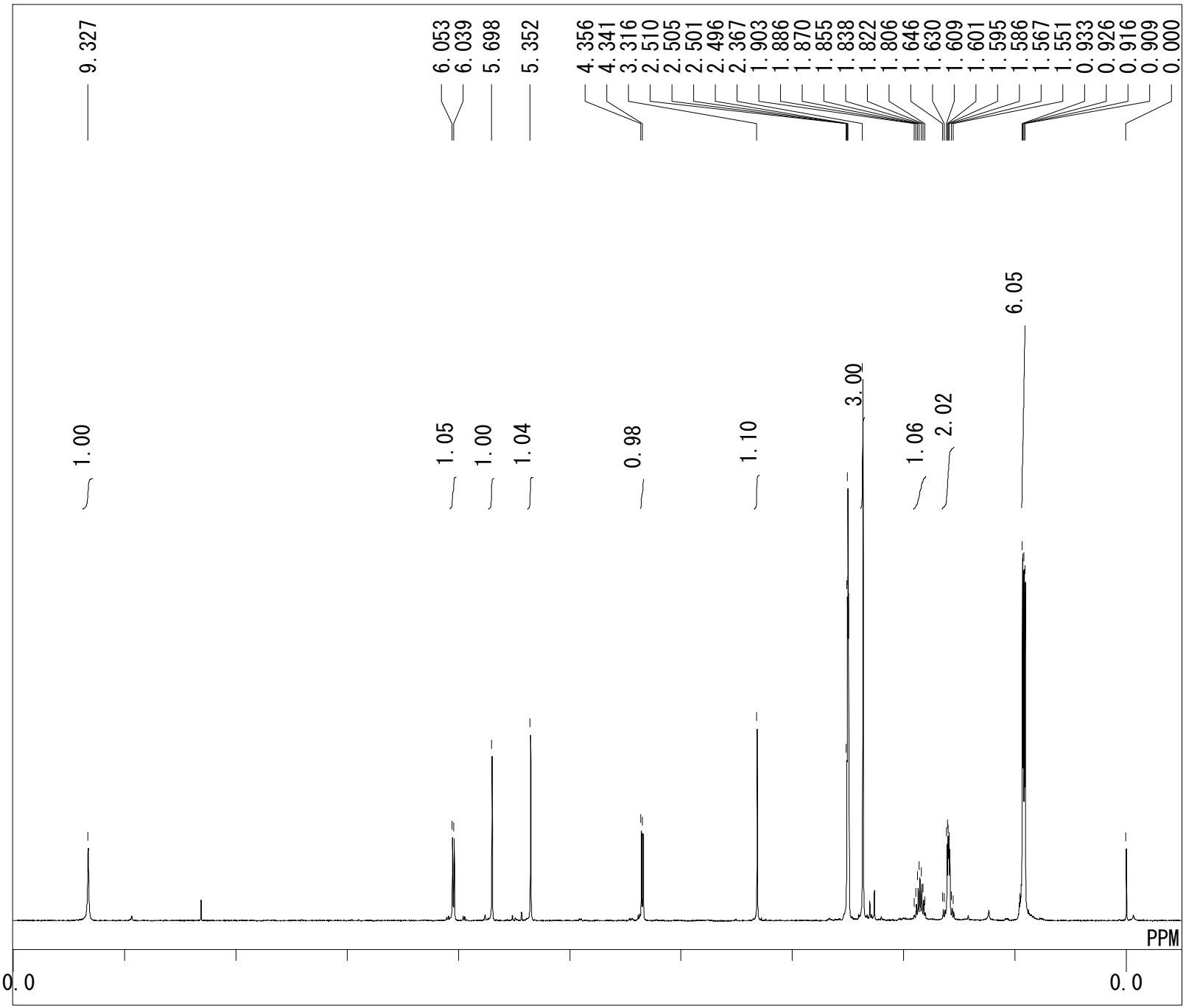
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	5000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.1	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	1.20	Hz
RGAIN	25	



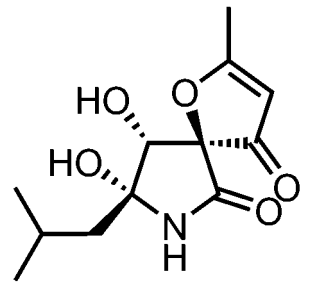
Rubrobramide (**1**)

^{13}C NMR

$CDCl_3$ (100 MHz)



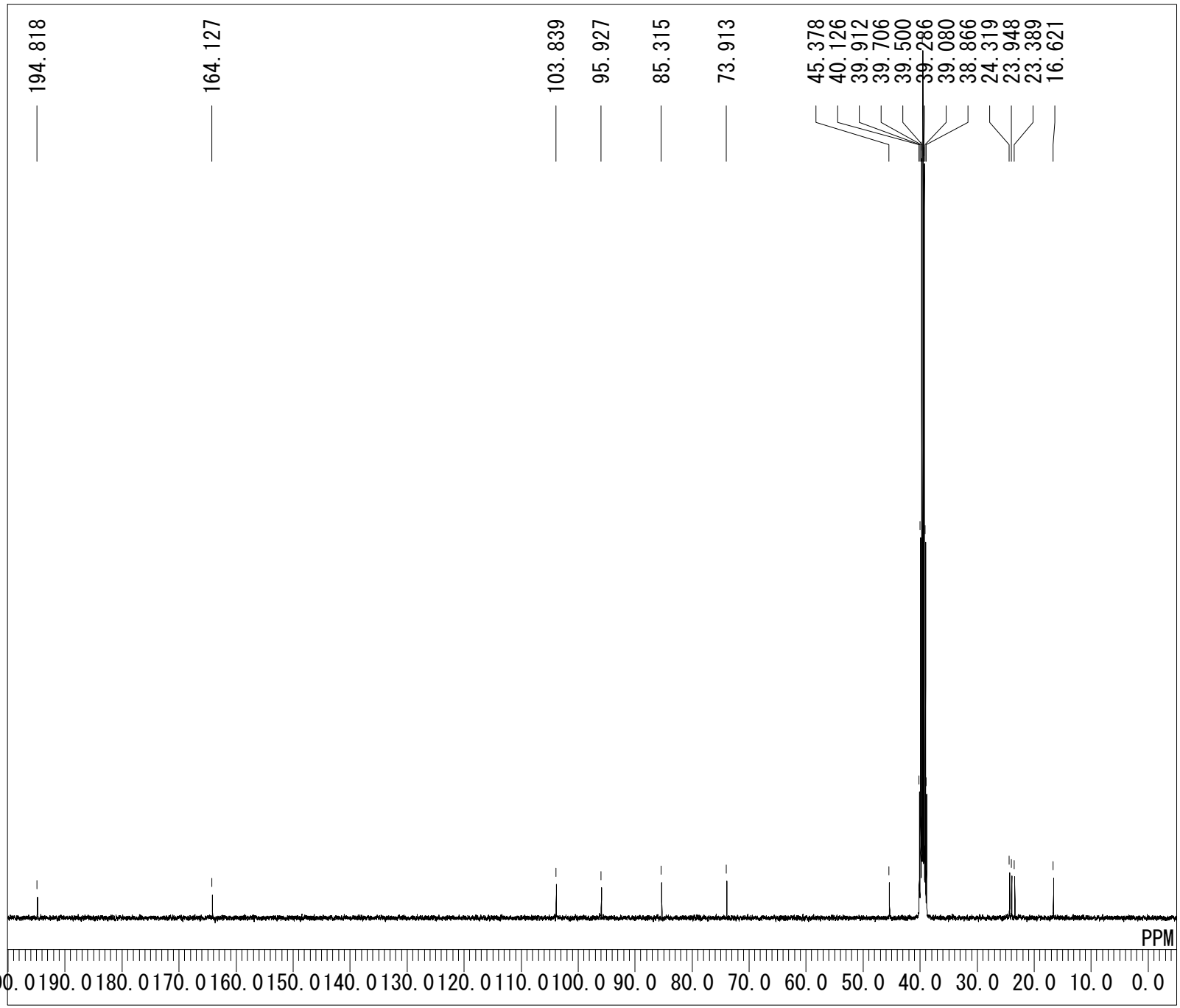
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.2	c
SLVNT	DMSO	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	19	



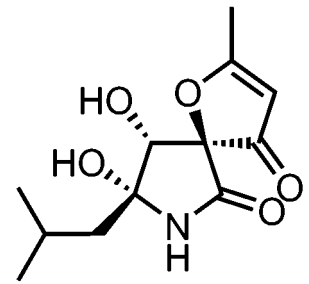
16

^{1}H NMR

DMSO-d_6 (400 MHz)



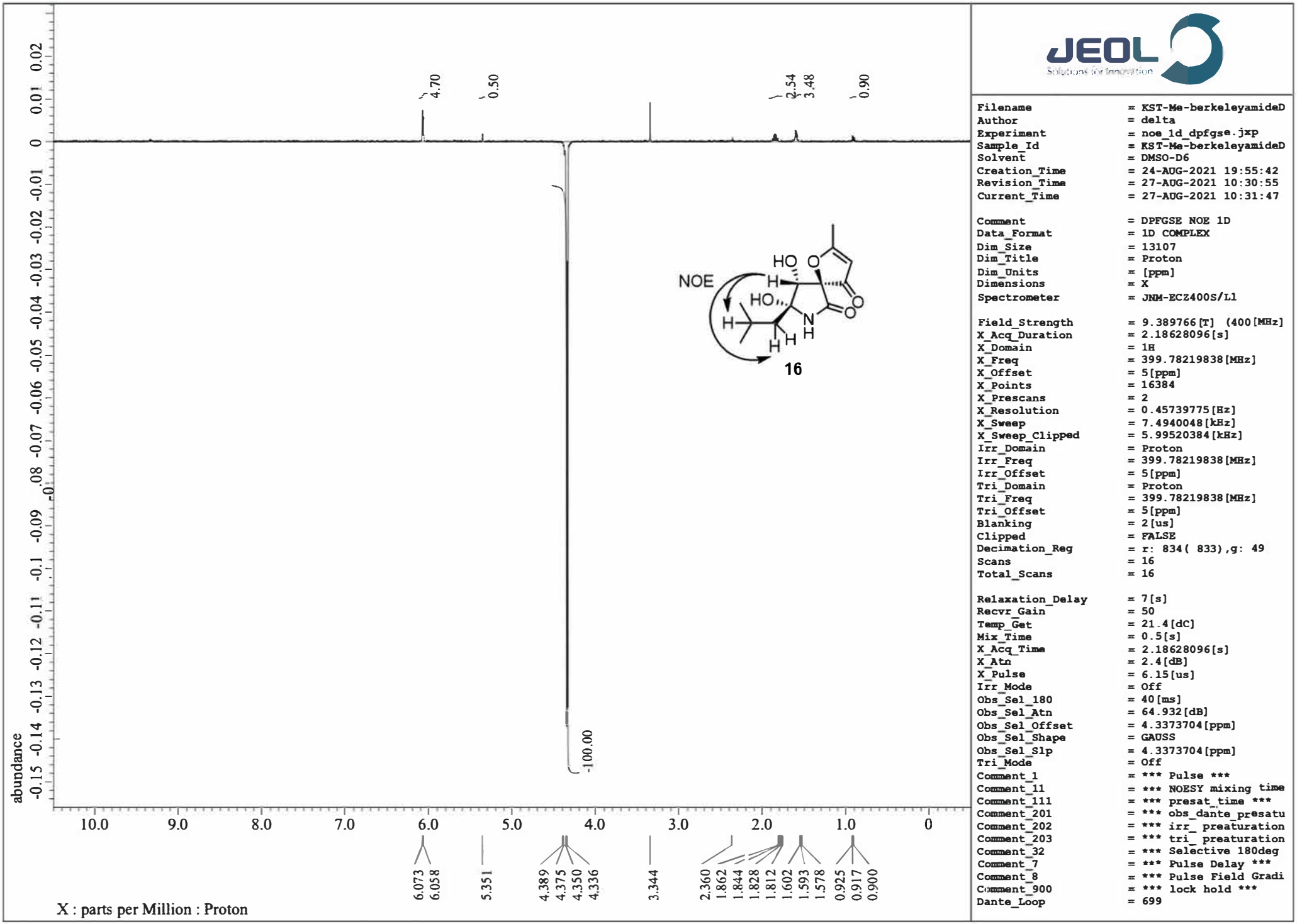
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	995	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.2	c
SLVNT	DMSO	
EXREF	39.50	ppm
BF	2.00	Hz
RGAIN	24	



16

^{13}C NMR

DMSO-d_6 (100 MHz)



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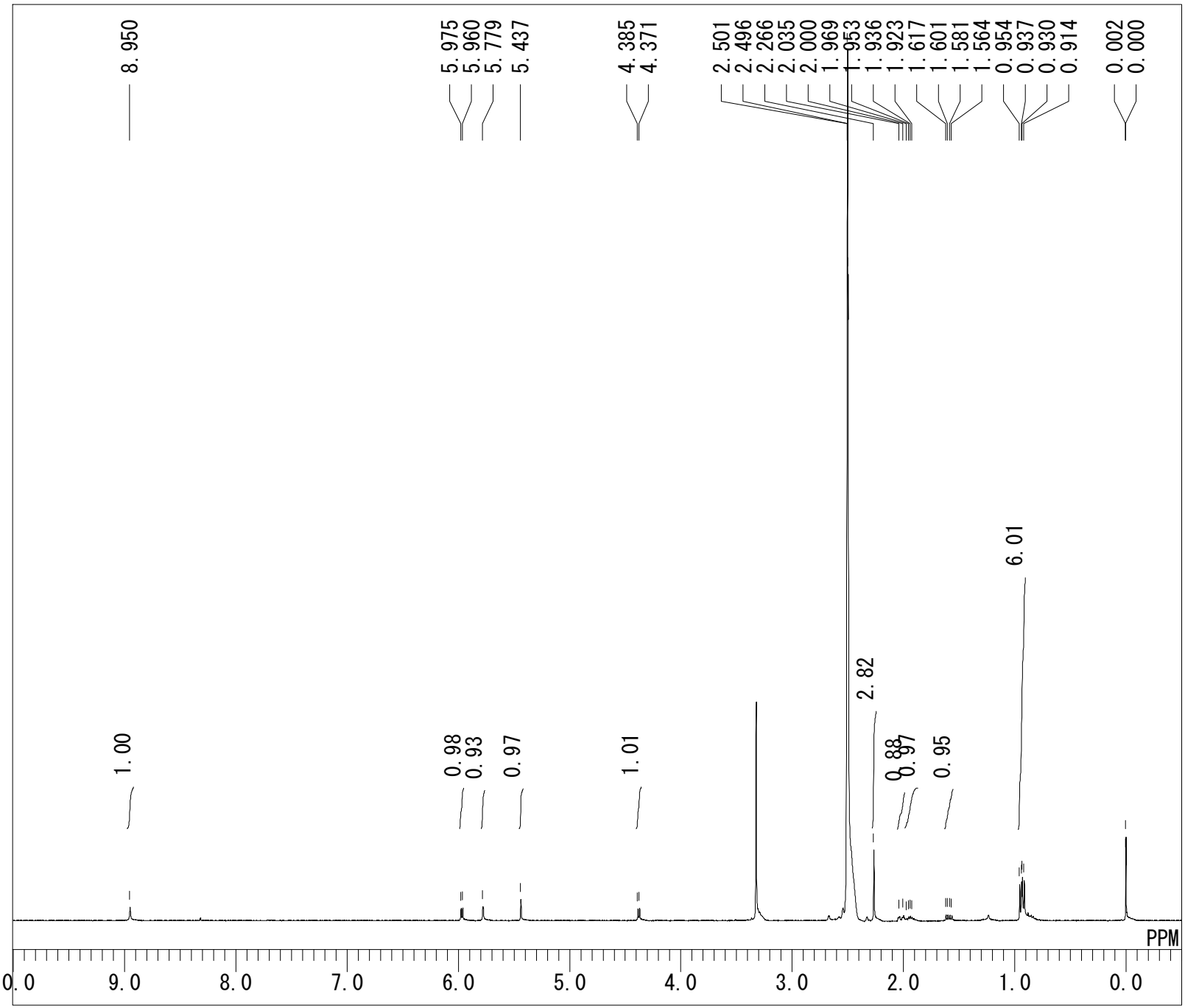
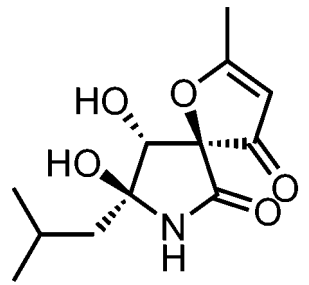
Author	= delta
Experiment	= noe_1d_dpfgse.jxp
Sample_Id	= KST-Me-berkeleyamideD
Solvent	= DMSO-D6
Creation_Time	= 24-AUG-2021 19:55:42
Revision_Time	= 27-AUG-2021 10:30:55
Current_Time	= 27-AUG-2021 10:31:47
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Decimation_Reg	= r: 834(833),g: 49
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.4[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 2.4[dB]
X_Pulse	= 6.15[us]
Irr_Mode	= Off
Obs_Sel_180	= 40[ms]
Obs_Sel_Atn	= 64.932[dB]
Obs_Sel_Offset	= 4.3373704[ppm]
Obs_Sel_Shape	= GAUSS
Obs_Sel_Slp	= 4.3373704[ppm]
Tri_Mode	= Off
Comment_1	= *** Pulse ***
Comment_11	= *** NOESY mixing time
Comment_111	= *** presat_time ***
Comment_201	= *** obs_dante_presatu
Comment_202	= *** irr_ preaturation
Comment_203	= *** tri_ preaturation
Comment_32	= *** Selective 180deg
Comment_7	= *** Pulse Delay ***
Comment_8	= *** Pulse Field Gradi
Comment_900	= *** lock hold ***
Dante_Loop	= 699



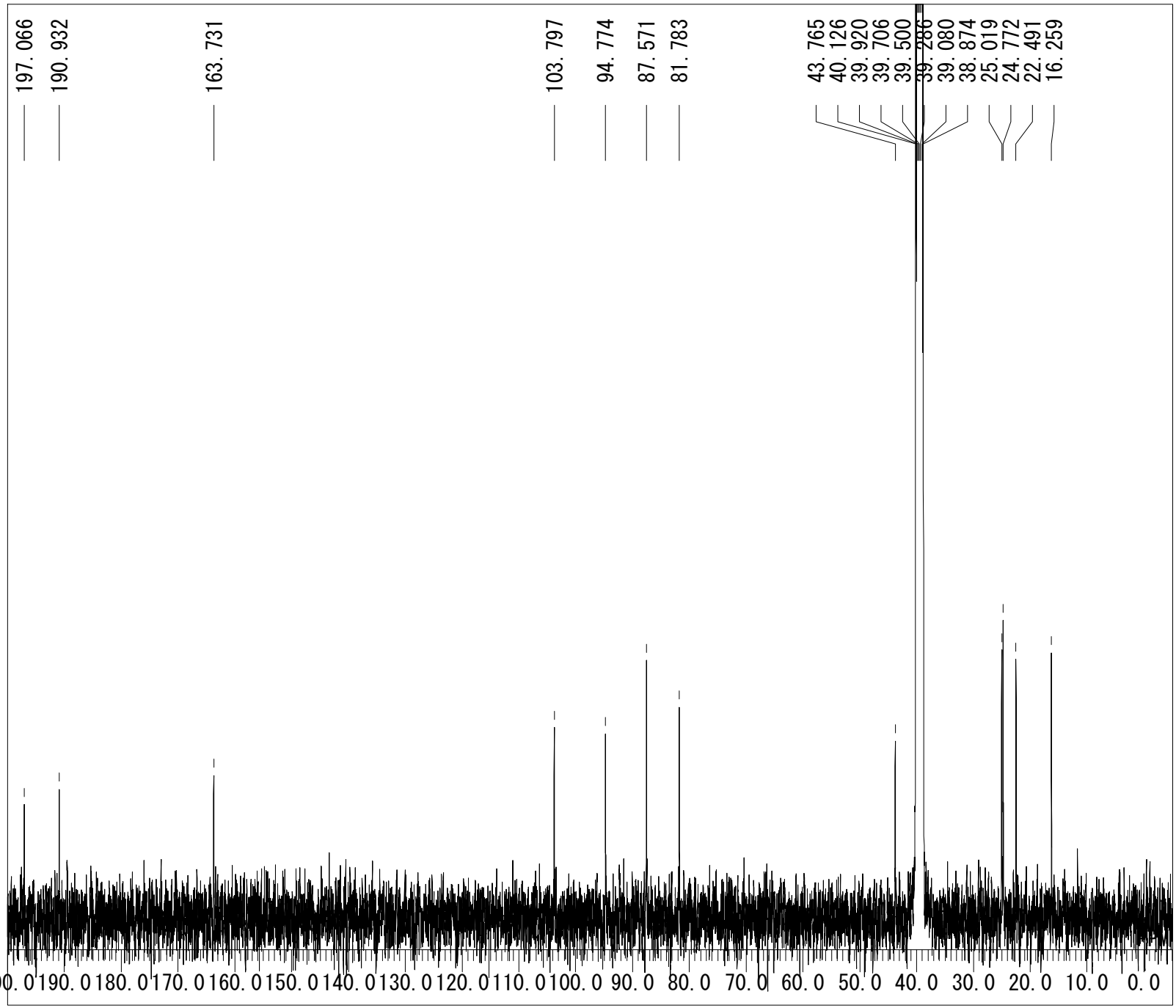
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	32	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	22.7	c
SLVNT	DMSO	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	21	



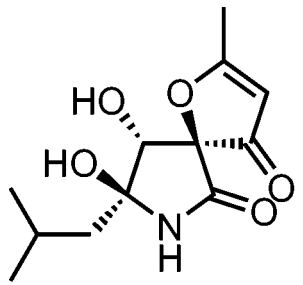
17

^{1}H NMR

DMSO-d_6 (400 MHz)



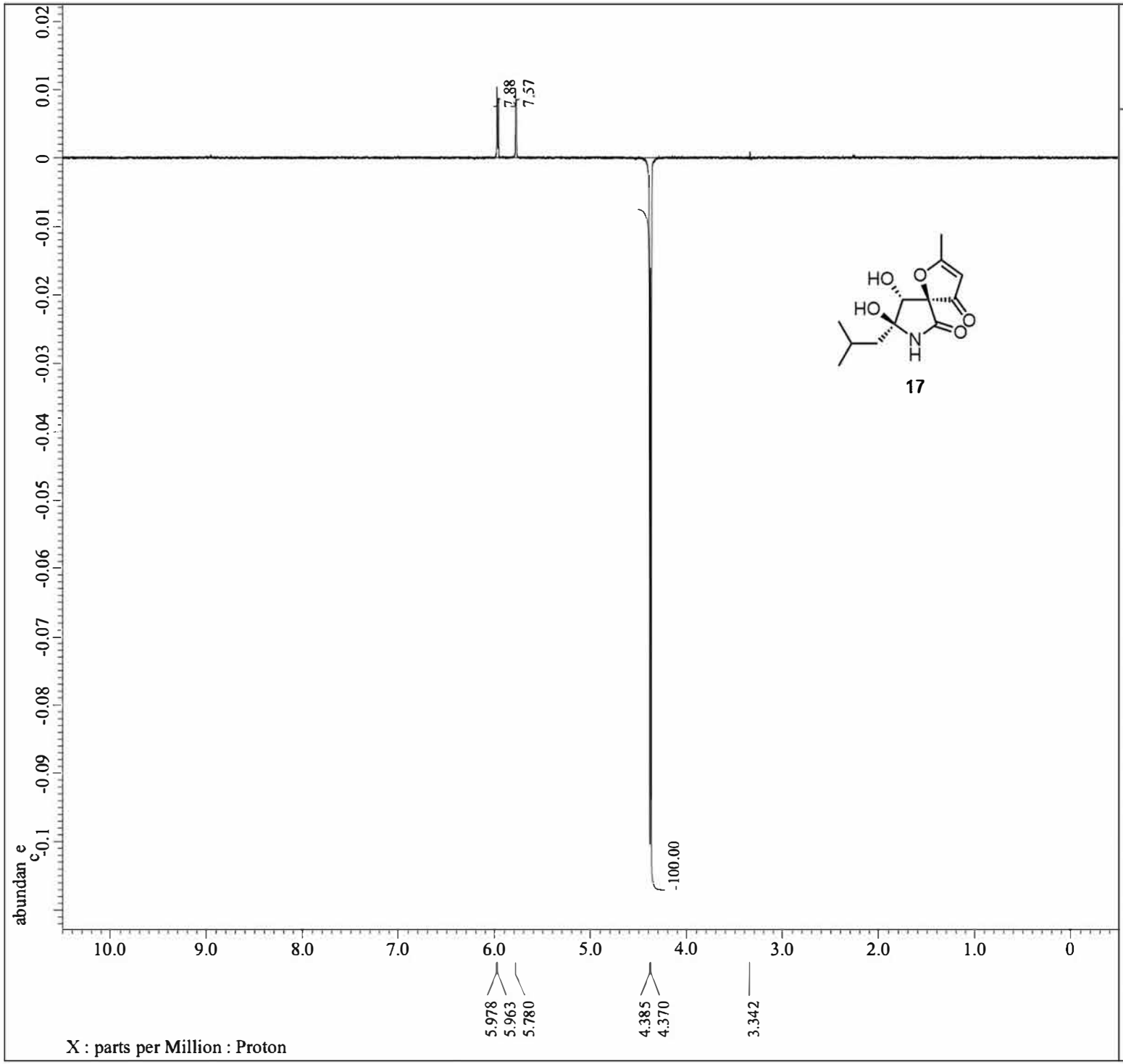
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	2000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	23.4	c
SLVNT	DMSO	
EXREF	39.50	ppm
BF	2.00	Hz
RGAIN	24	



17

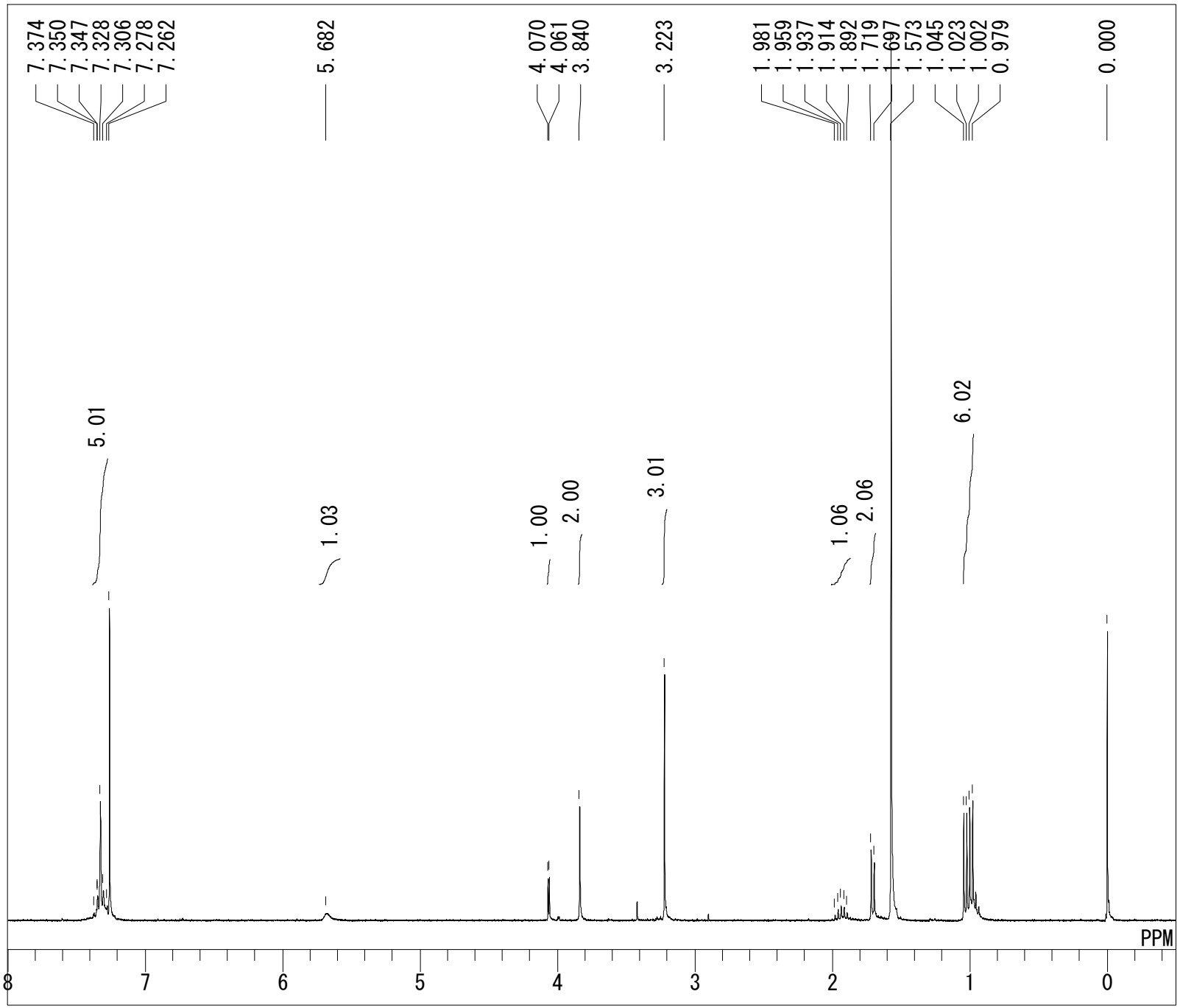
^{13}C NMR

DMSO-d_6 (100 MHz)



JEOL Solutions for Innovation

Filename	= KST-Me-berkeleyamideD
Author	= delta
Experiment	= noe_ld_dpfgse.jxp
Sample_Id	= KST-Me-berkeleyamideD
Solvent	= DMSO-D6
Creation_Time	= 25-AUG-2021 19:03:15
Revision_Time	= 27-AUG-2021 10:46:56
Current_Time	= 27-AUG-2021 10:47:19
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
SPectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Decimation_Reg	= r: 834(833),g: 49
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
TemP_Get	= 21.5[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 2.4[dB]
X_Pulse	= 6.15[us]
Irr_Mode	= Off
Obs_Sel_180	= 40[ms]
Obs_Sel_Atn	= 64.932[dB]
Obs_Sel_Offset	= 4.36573172[ppm]
Obs_Sel_Shape	= GAUSS
Obs_Sel_Slp	= 4.36573172[ppm]
Tri_Mode	= Off
Comment_1	= *** Pulse ***
Comment_11	= *** NOESY mixing time
Comment_111	= *** presat_time ***
Comment_201	= *** obs_dante_presatu
Comment_202	= *** irr_ preaturation
Comment_203	= *** tri_ preaturation
Comment_32	= *** Selective 180deg
Comment_7	= *** Pulse Delay ***
Comment_8	= *** Pulse Field Gradi
Comment_900	= *** lock hold ***
Dante_Loop	= 699



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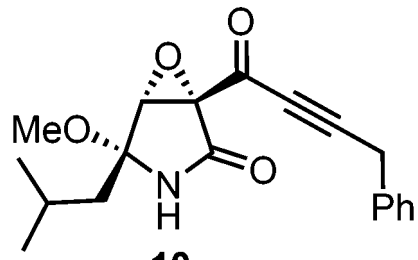
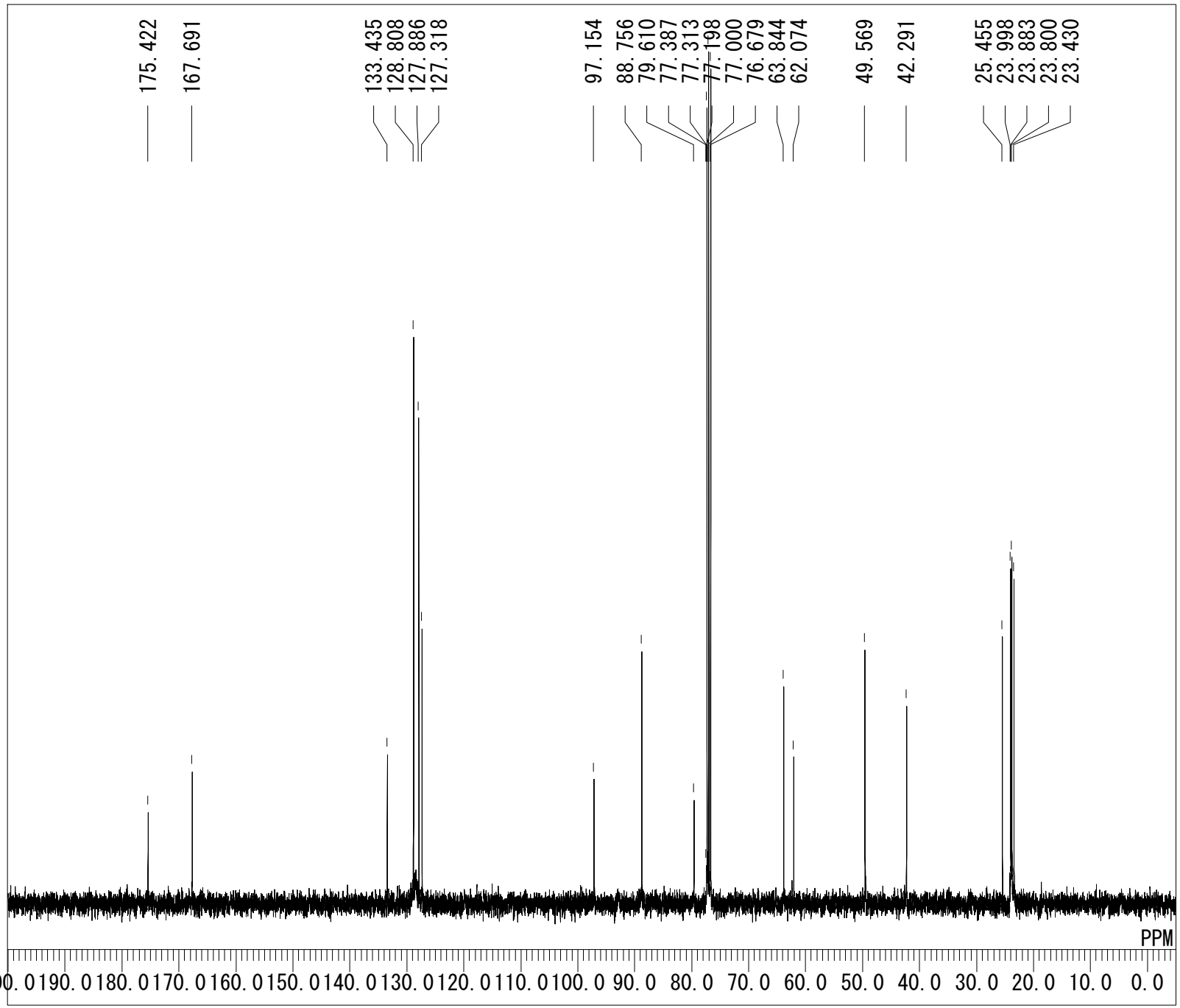
OBSET	130.00	KHz
OBFIN	1150.00	Hz
POINT	32768	
FREQU	6006.01	Hz
SCANS	16	
ACQTM	5.4559	sec
PD	1.5440	sec
PW1	5.20	usec
IRNUC	1H	
CTEMP	25.3	c
SLVNT	CDCL3	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	22	



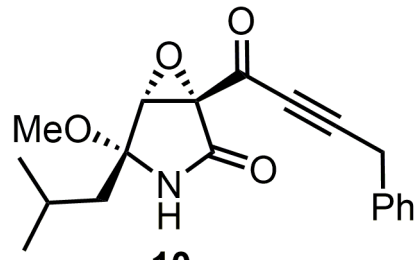
10

^{1}H NMR

$CDCl_3$ (300 MHz)



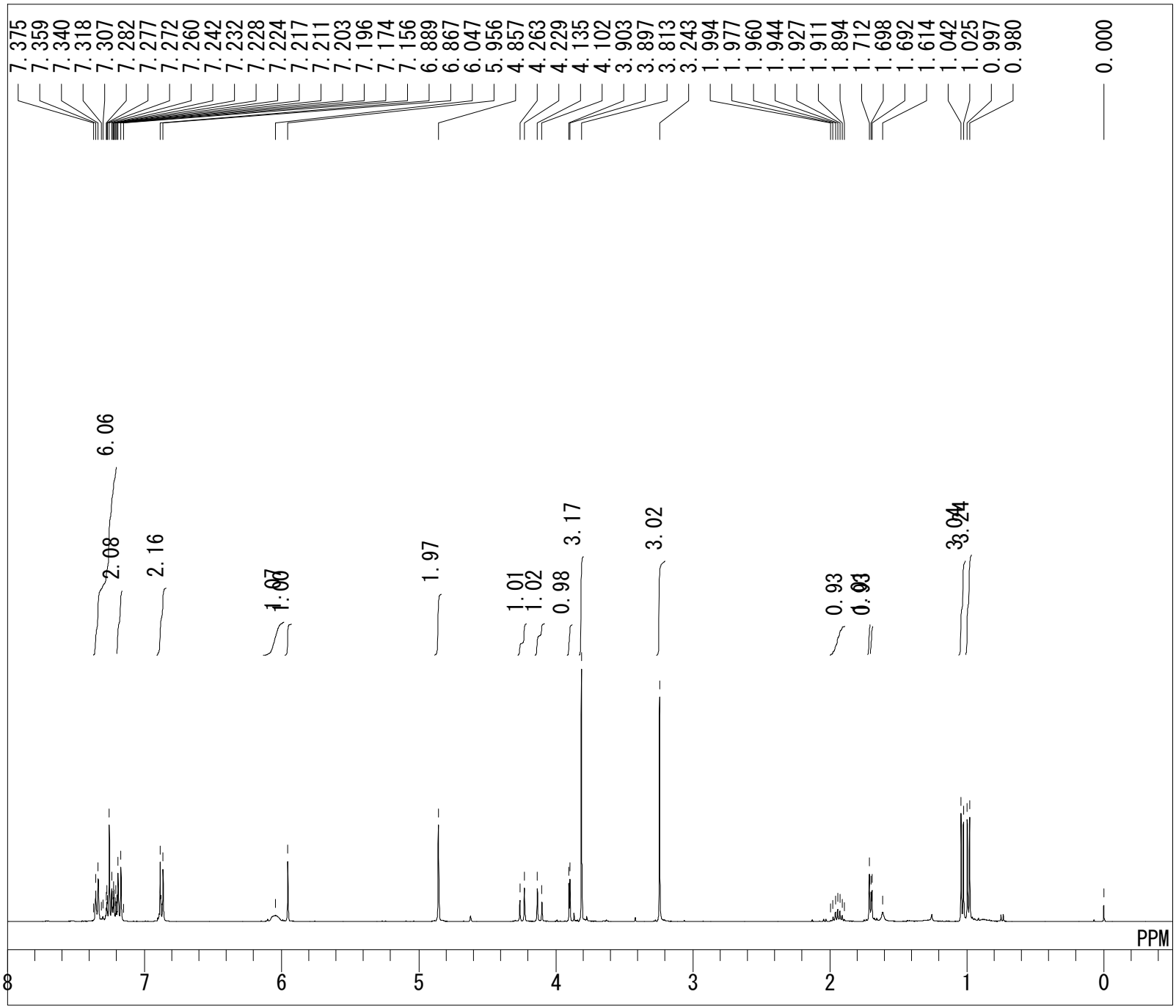
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	150	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	26.6	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	1.20	Hz
RGAIN	25	



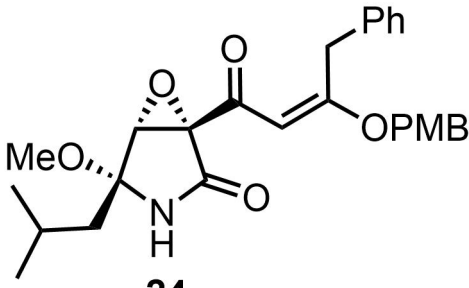
10

^{13}C NMR

$CDCl_3$ (100 MHz)



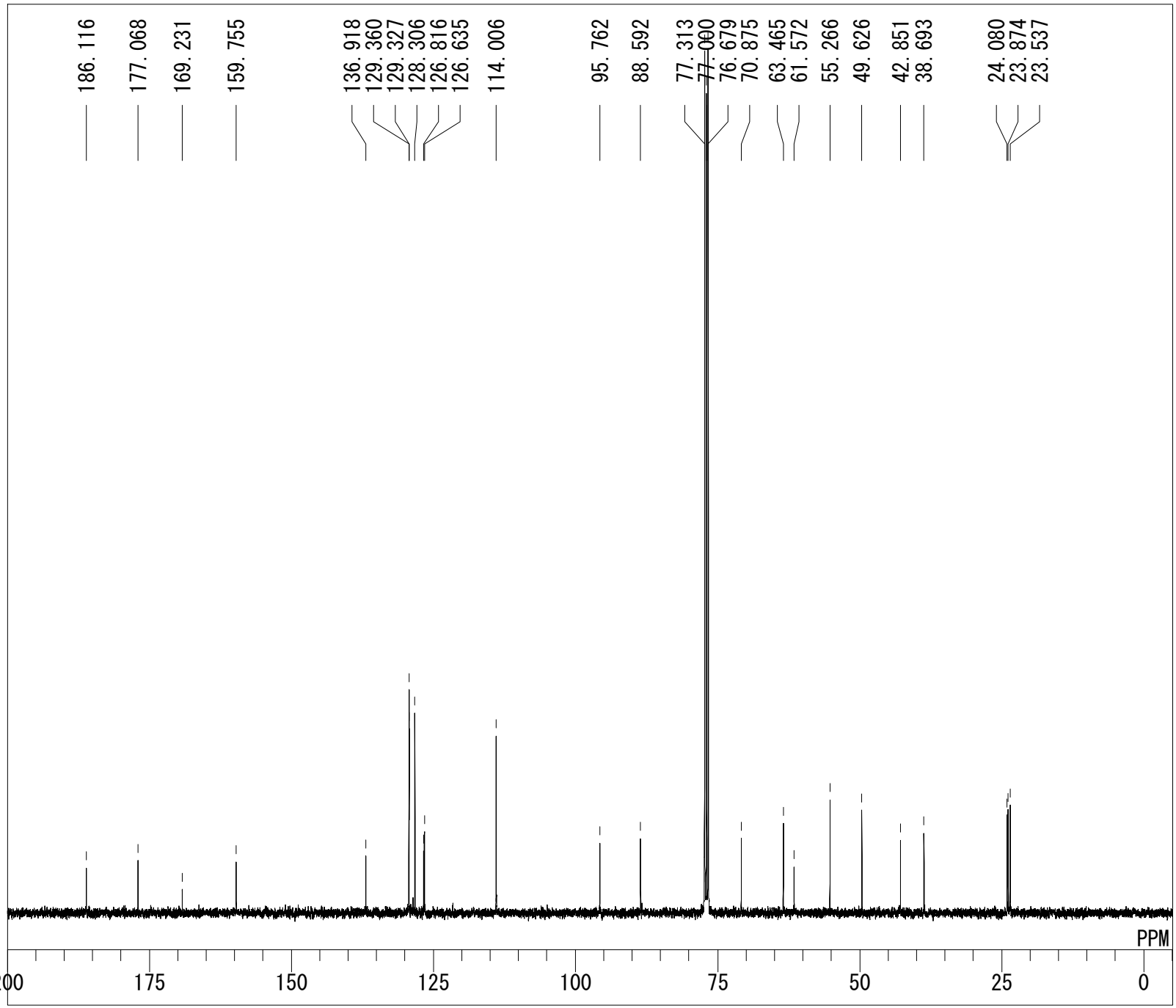
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.0	c
SLVNT	CDCL3	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	18	



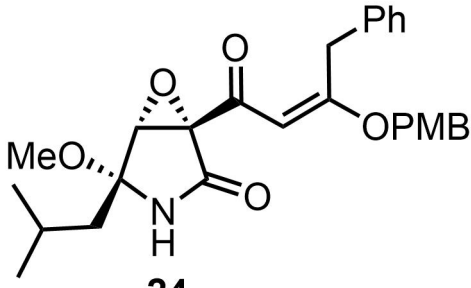
24

^{1}H NMR

$CDCl_3$ (400 MHz)



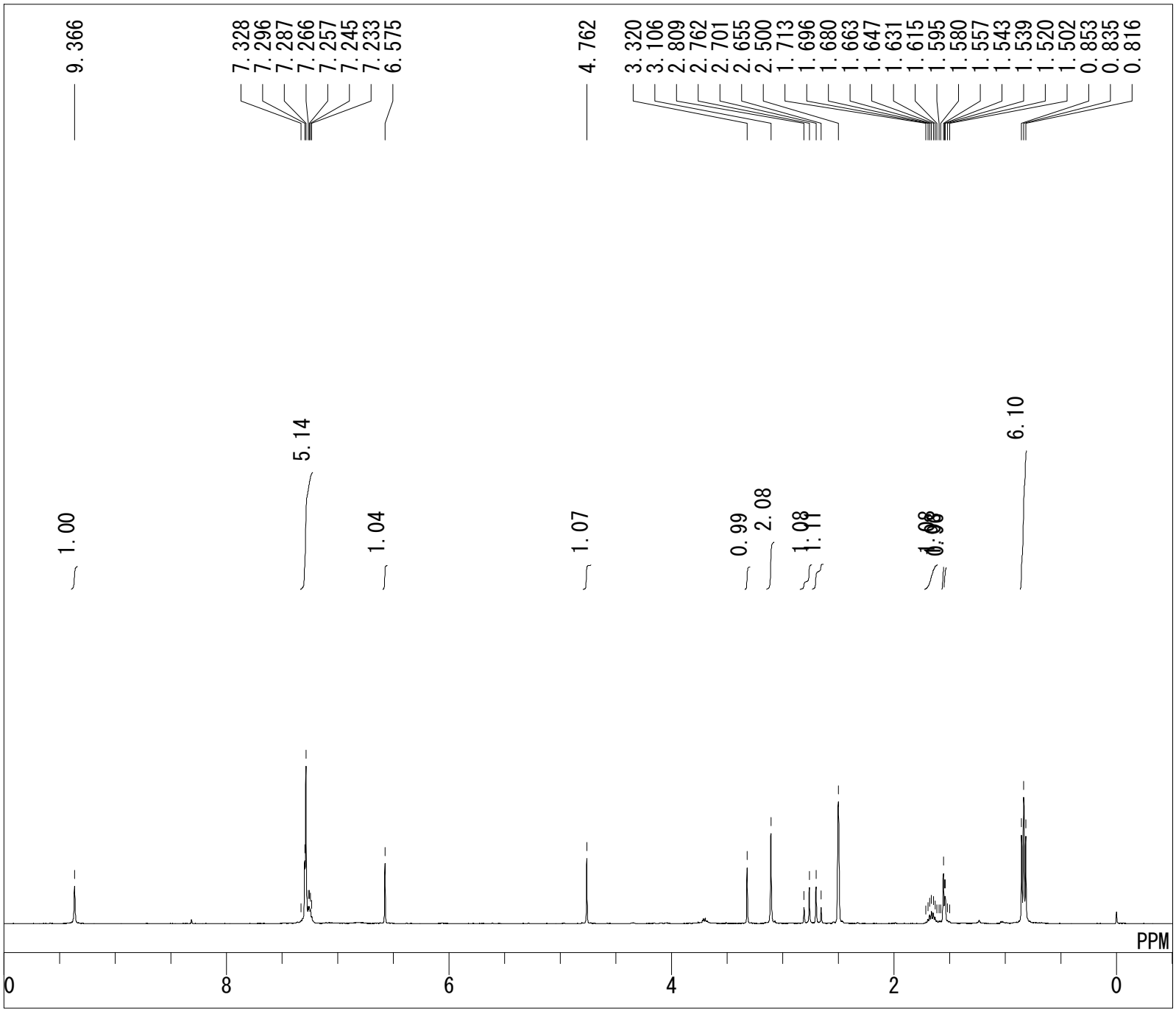
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	1000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.3	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	1.20	Hz
RGAIN	25	



24

^{13}C NMR

$CDCl_3$ (100 MHz)



OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
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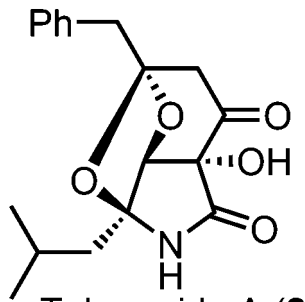
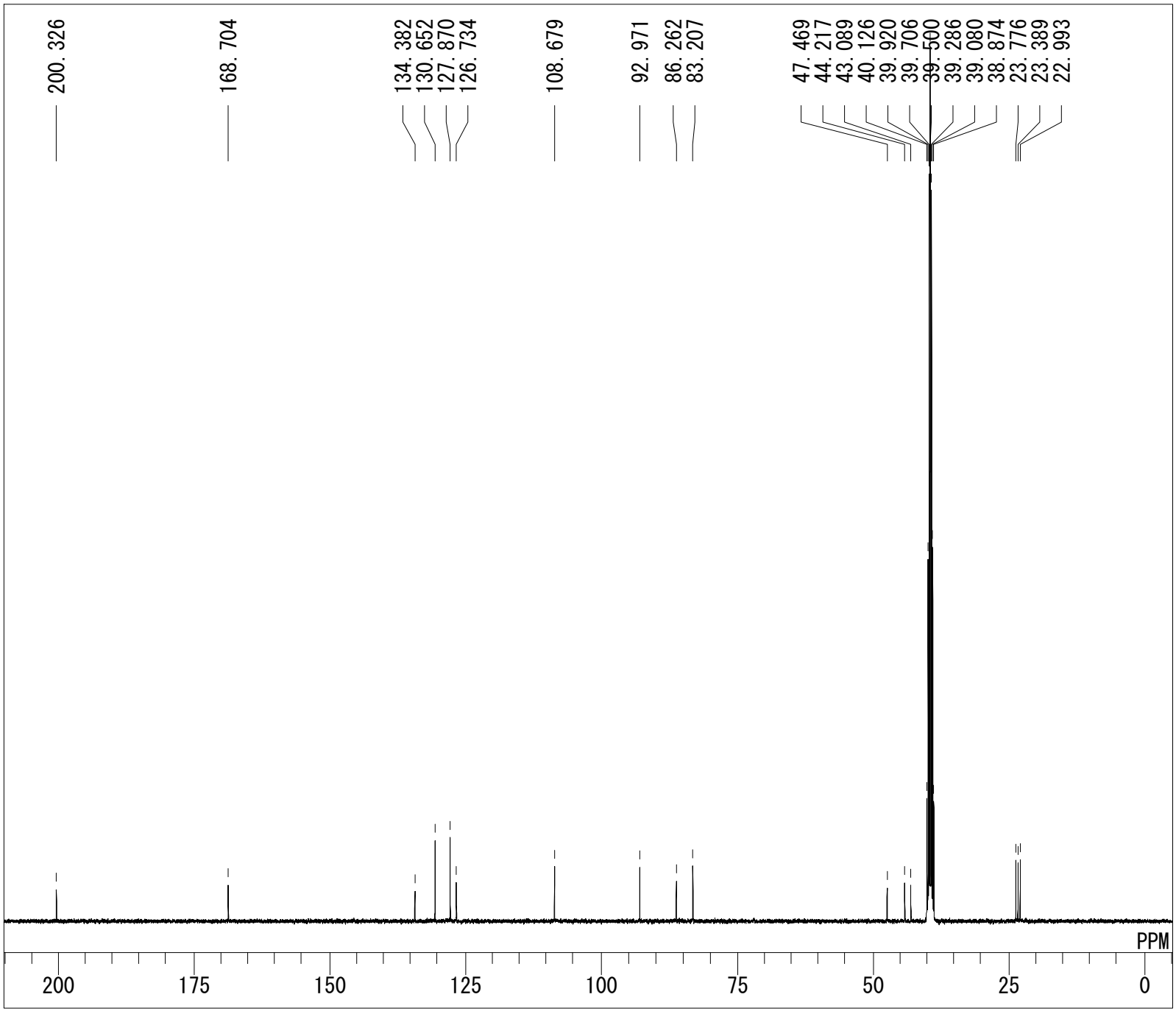
PD	4.9500	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.2	c
SLVNT	DMSO	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	18	



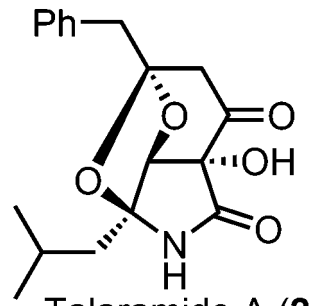
Talaramide A (**2**)

^{1}H NMR

DMSO-d_6 (400 MHz)



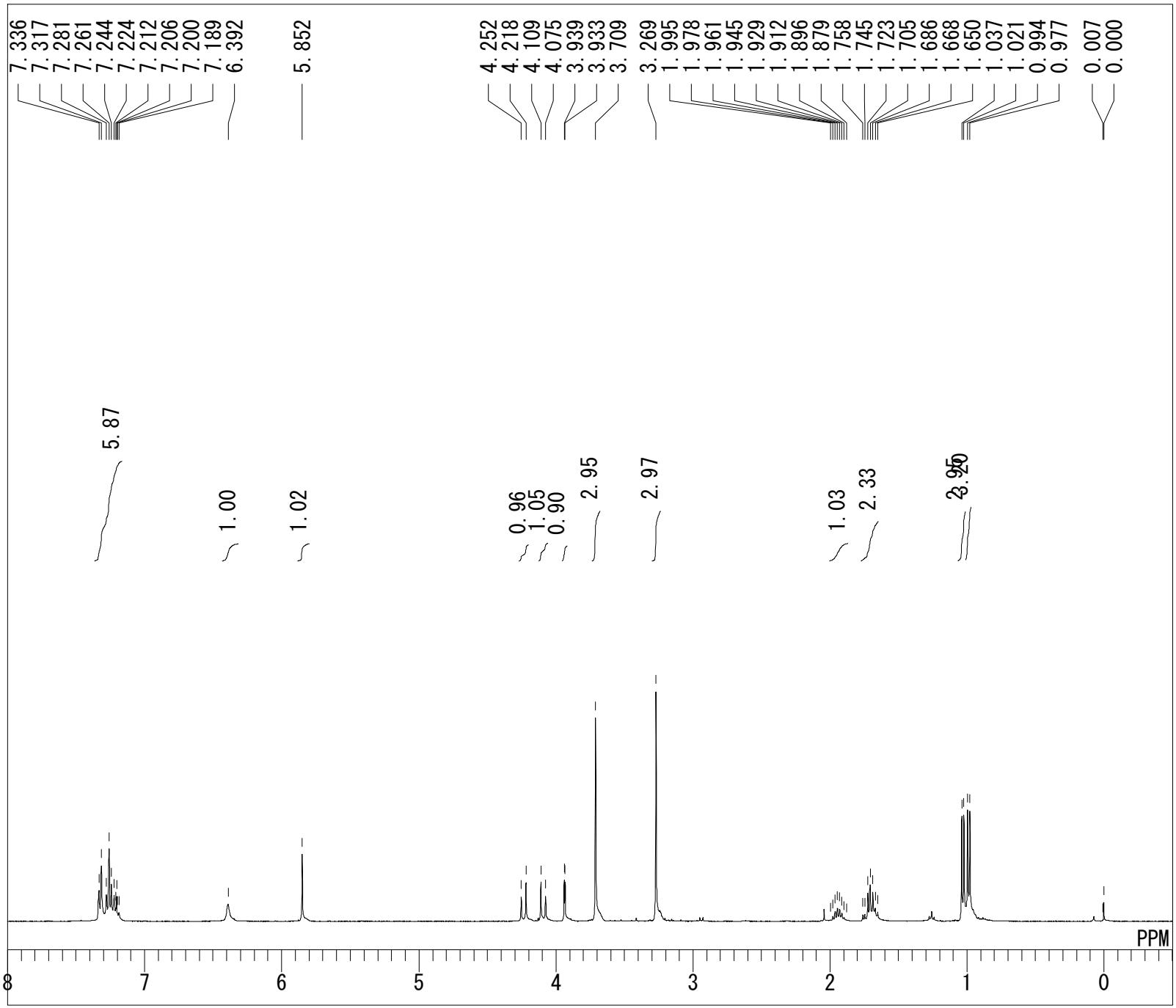
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	1972	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.3	c
SLVNT	DMSO	
EXREF	39.50	ppm
BF	1.20	Hz
RGAIN	24	



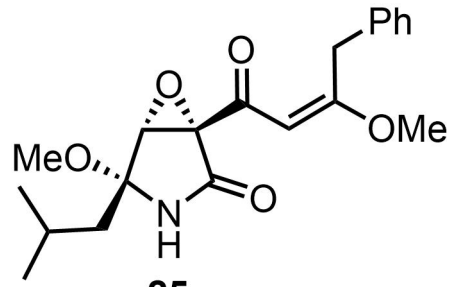
Talaramide A (**2**)

^{13}C NMR

DMSO-d_6 (100 MHz)



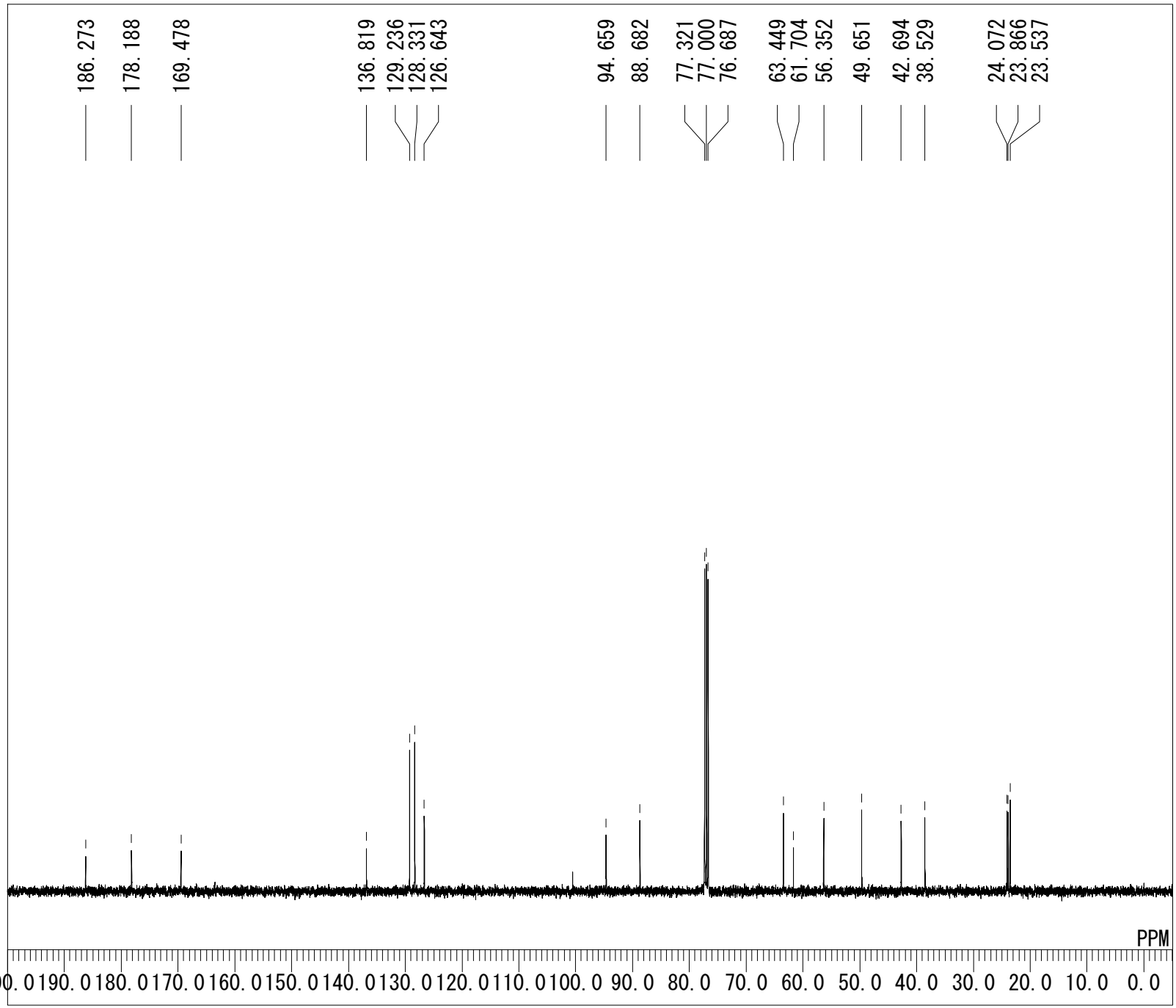
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.0	c
SLVNT	CDCL3	
EXREF	0.00	ppm
BF	0.10	Hz
RGAIN	16	



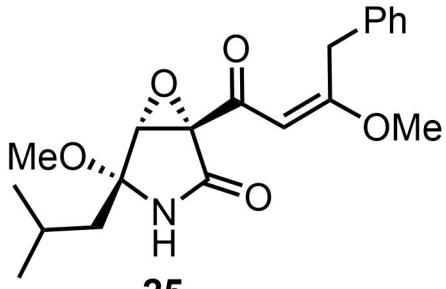
25

^{1}H NMR

$CDCl_3$ (400 MHz)



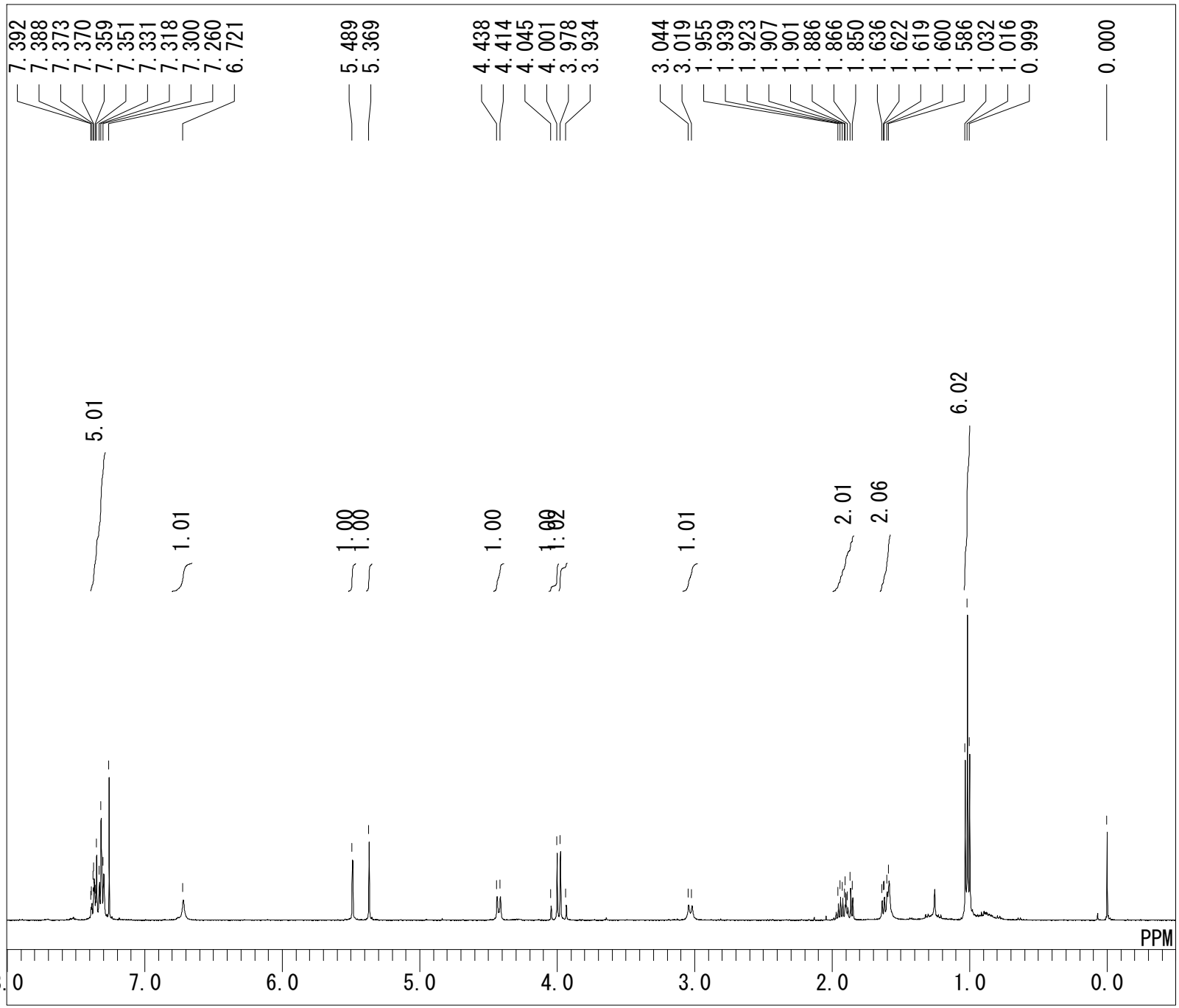
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	200	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.3	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	1.20	Hz
RGAIN	25	



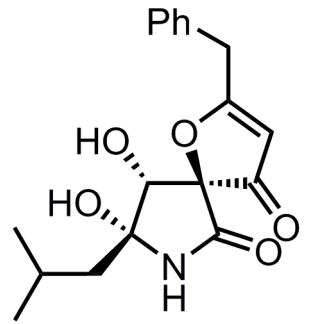
25

^{13}C NMR

$CDCl_3$ (100 MHz)



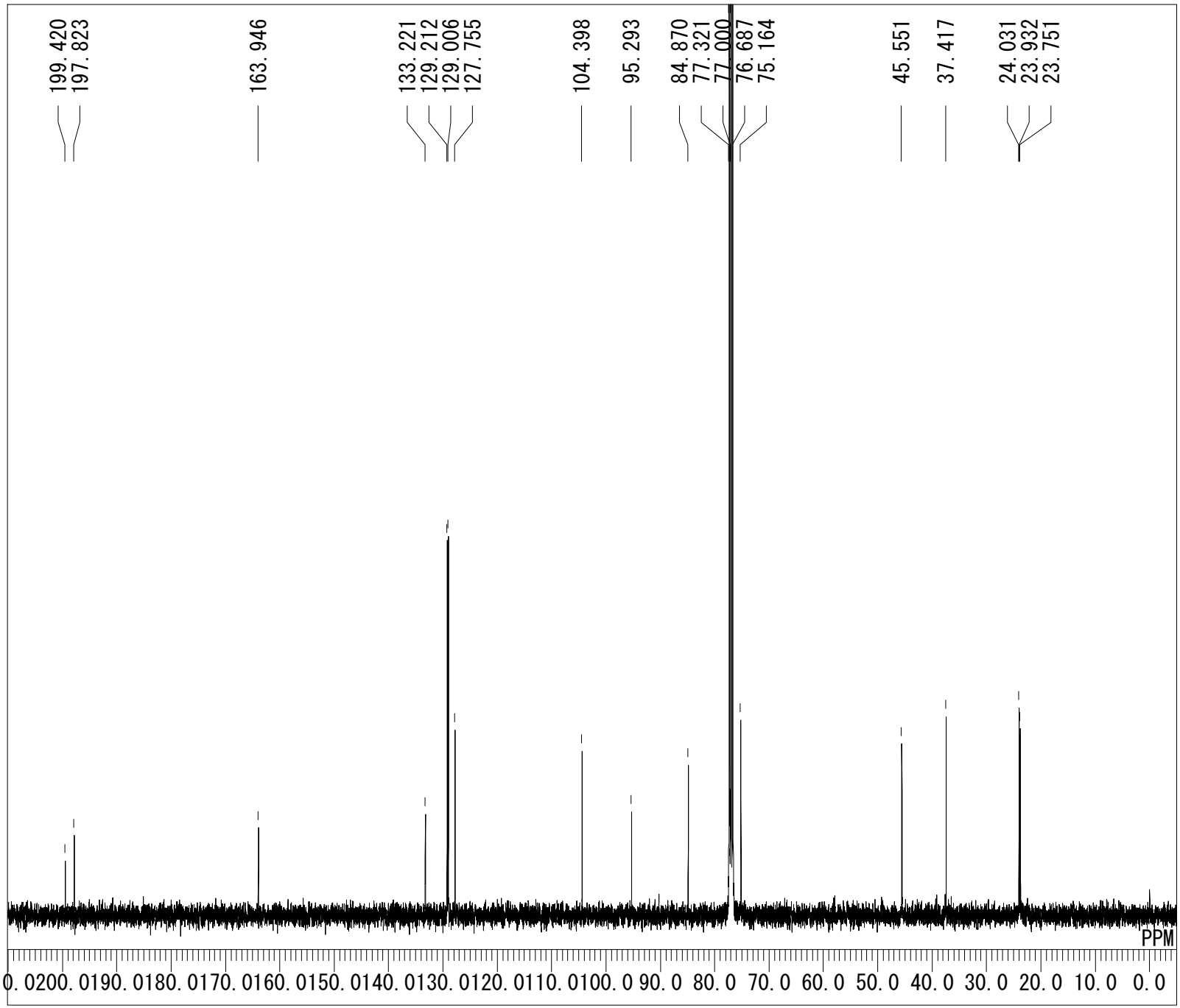
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	8	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.60	usec
IRNUC	1H	
CTEMP	24.0	c
SLVNT	CDCL3	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	20	



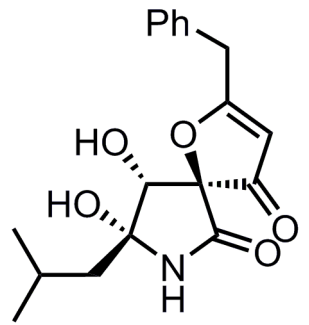
Berkeleyamide D (**3**)

^{1}H NMR

$CDCl_3$ (400 MHz)



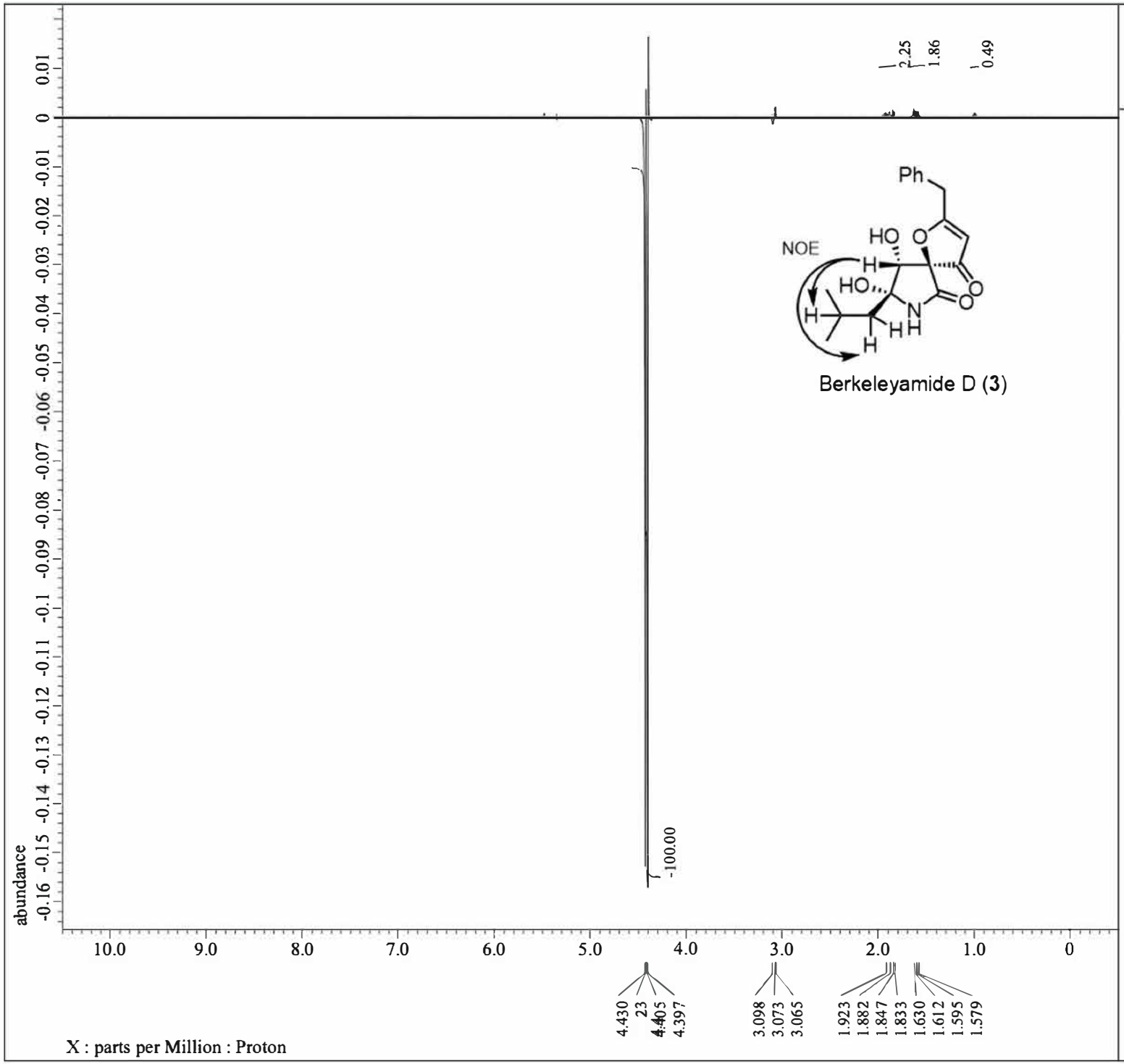
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	2000	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.6	c
SLVNT	CDCL3	
EXREF	77.00	ppm
BF	1.20	Hz
RGAIN	25	



Berkeleyamide D (**3**)

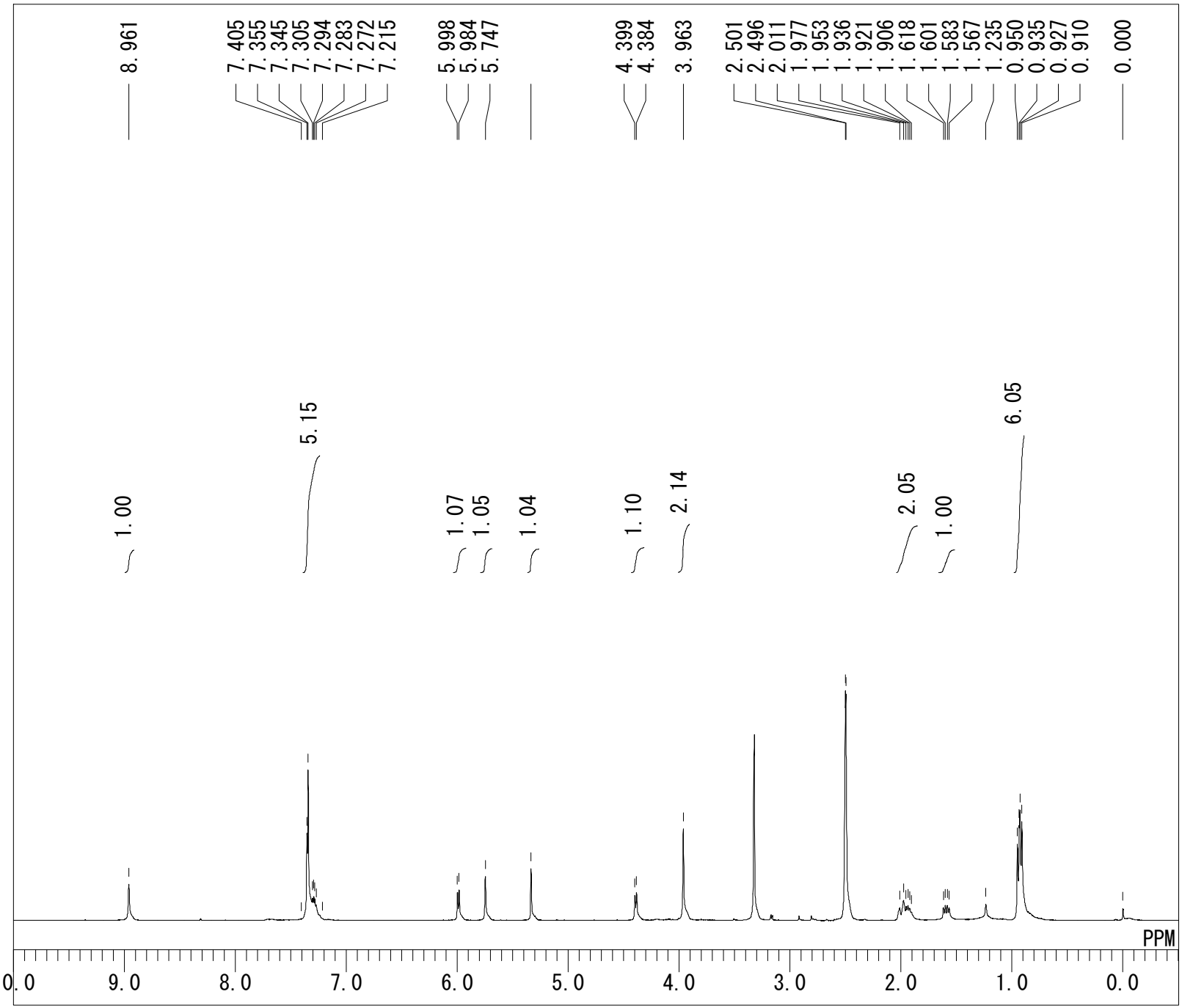
^{13}C NMR

$CDCl_3$ (100 MHz)

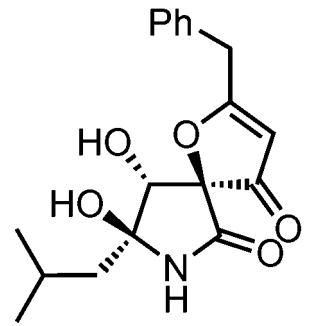


JEOL
Solutions for Innovation

Filename	= KST-berkeleyamideD_no
Author	= delta
Experiment	= noe_ld_dpfgse.jxp
Sample_Id	= KST-berkeleyamideD
Solvent	= CHLOROFORM-D
Creation_Time	= 23-AUG-2021 17:10:32
Revision_Time	= 27-AUG-2021 10:40:25
Current_Time	= 27-AUG-2021 10:40:57
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
SPectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Decimation_Reg	= r: 834(833),g: 49
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
TemP_Get	= 21.6[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 2.4[dB]
X_Pulse	= 6.15[us]
Irr_Mode	= Off
Obs_Sel_180	= 40[ms]
Obs_Sel_Atn	= 64.932[dB]
Obs_Sel_Offset	= 4.42566776[ppm]
Obs_Sel_Shape	= GAUSS
Obs_Sel_Slp	= 4.42566776[ppm]
Tri_Mode	= Off
Comment_1	= *** Pulse ***
Comment_11	= *** NOESY mixing time
Comment_111	= *** presat_time ***
Comment_201	= *** obs_dante_presatu
Comment_202	= *** irr_ preaturation
Comment_203	= *** tri_ preaturation
Comment_32	= *** Selective 180deg
Comment_7	= *** Pulse Delay ***
Comment_8	= *** Pulse Field Gradi
Comment_900	= *** lock hold ***
Dante_Loop	= 699



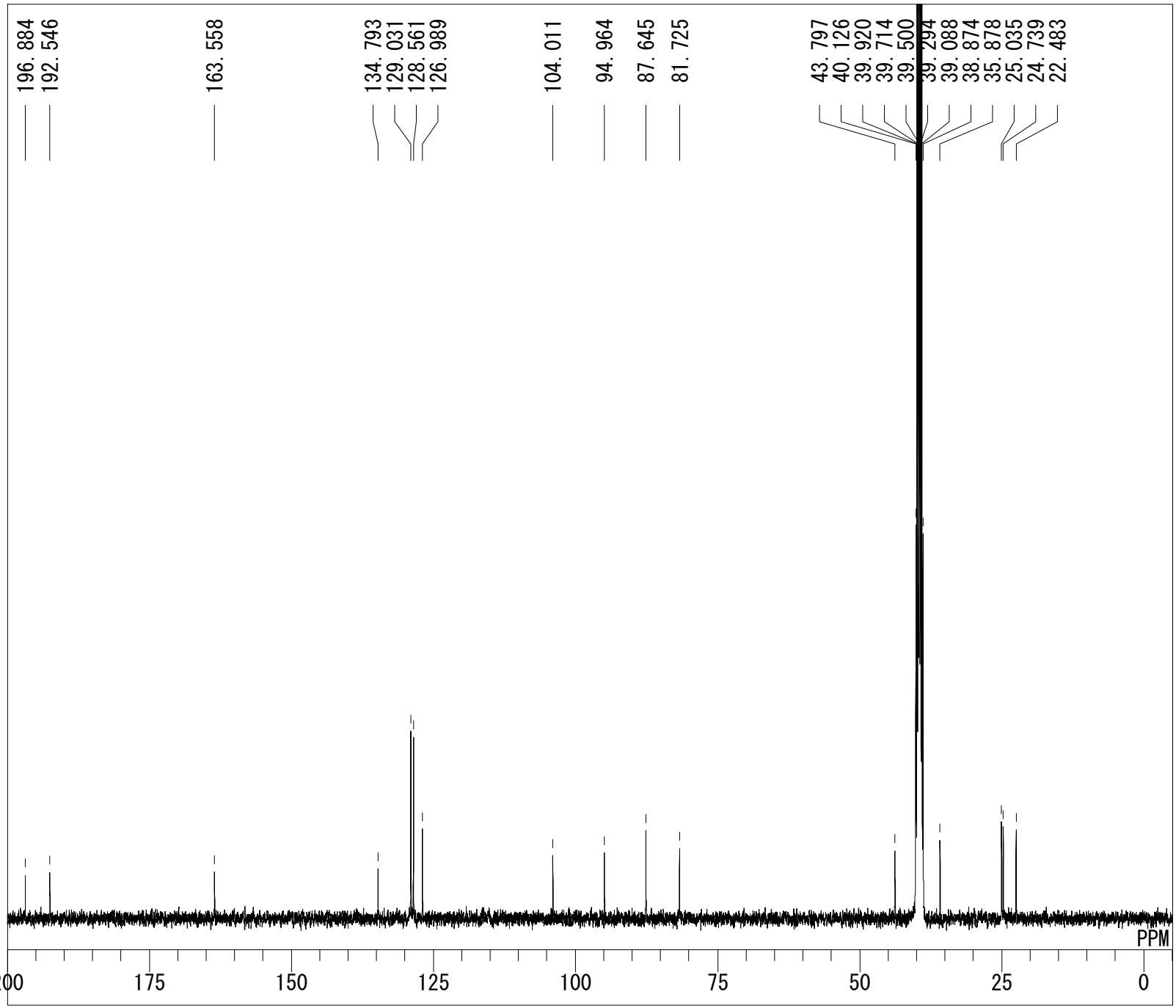
OBFRQ	399.65	MHz
OBSET	124.00	KHz
OBFIN	10500.00	Hz
POINT	16384	
FREQU	7992.01	Hz
SCANS	32	
ACQTM	2.0500	sec
PD	4.9500	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	23.6	c
SLVNT	DMSO	
EXREF	0.00	ppm
BF	0.12	Hz
RGAIN	19	



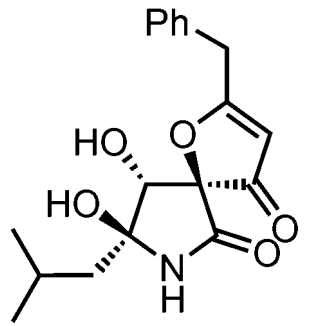
14-*epi*-**3**

^{1}H NMR

DMSO-d_6 (400 MHz)



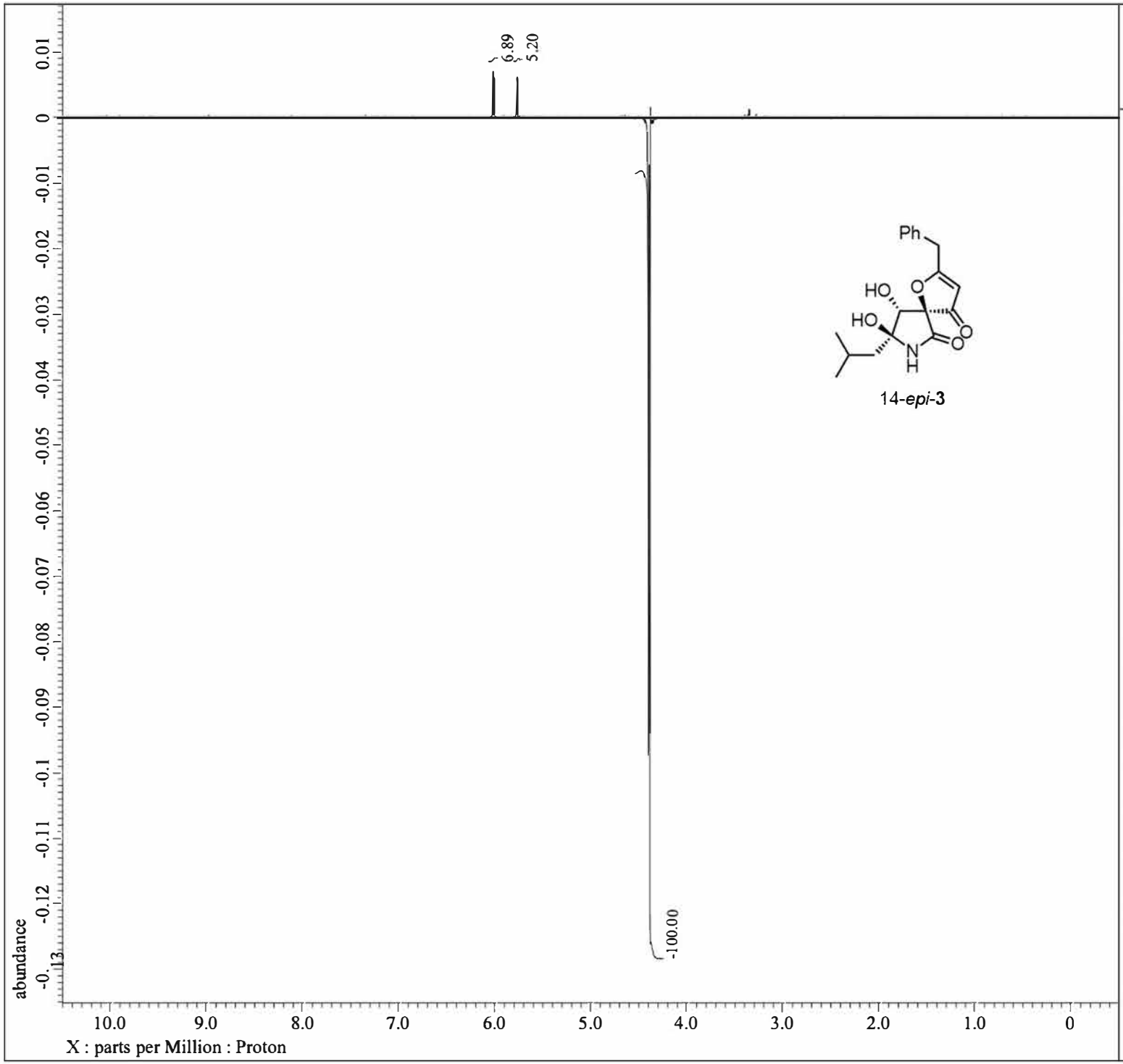
OBFRQ	100.40	MHz
OBSET	125.00	KHz
OBFIN	10500.00	Hz
POINT	32768	
FREQU	27118.64	Hz
SCANS	1500	
ACQTM	1.2083	sec
PD	1.7920	sec
PW1	5.80	usec
IRNUC	1H	
CTEMP	24.2	c
SLVNT	DMSO	
EXREF	39.50	ppm
BF	2.00	Hz
RGAIN	25	



14-*epi*-**3**

^{13}C NMR

DMSO-d_6 (100 MHz)



JEOL
Solutions for Innovation

Filename	= KST-berkeleyamideD-is
Author	= delta
Experiment	= noe_ld_dpfgse.jxp
Sample_Id	= KST-berkeleyamideD-is
Solvent	= DMSO-D6
Creation_Time	= 23-AUG-2021 18:13:17
Revision_Time	= 27-AUG-2021 10:27:26
Current_Time	= 27-AUG-2021 10:28:49
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
SPectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Decimation_Reg	= r: 834(833),g: 49
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.4[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 2.4[dB]
X_Pulse	= 6.15[us]
Irr_Mode	= Off
Obs_Sel_180	= 40[ms]
Obs_Sel_Atn	= 64.932[dB]
Obs_Sel_Offset	= 4.37970352[ppm]
Obs_Sel_Shape	= GAUSS
Obs_Sel_Slp	= 4.37970352[ppm]
Tri_Mode	= Off
Comment_1	= *** Pulse ***
Comment_11	= *** NOESY mixing time
Comment_111	= *** Presat_time ***
Comment_201	= *** obs_dante_presatu
Comment_202	= *** irr_ preaturation
Comment_203	= *** tri_ preaturation
Comment_32	= *** Selective 180deg
Comment_7	= *** Pulse Delay ***
Comment_8	= *** Pulse Field Gradi
Comment_900	= *** lock hold ***
Dante_Loop	= 699