

Supporting information

(7S)-Kaitocephalin: A Highly NMDA Receptor Selective Ligand

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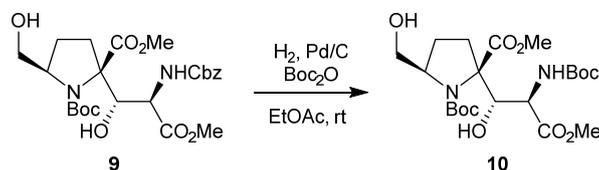
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General.

All reagents and solvents were purchased from either Aldrich Chemical Company, Inc., Kanto Kagaku Co., Inc., Merck KGaA, Inc., Nacalai Tesque Company, Ltd., Peptide Institute, Tokyo Kasei Kogyo Co., Ltd., or Wako Pure Chemical Industries, Ltd., and used without further purification unless otherwise indicated. Dichloromethane (CH₂Cl₂) was distilled from phosphorus pentoxide (P₂O₅). Tetrahydrofuran (THF) and toluene of anhydrous grade were used. [³H]CGP 39653, [³H]AMPA, and [³H]KA were purchased from PerkinElmer, Inc.

Optical rotations were taken on a JASCO P-1030 polarimeter with a sodium lamp (D line). Melting points were determined with a Yanaco MP-21 melting point apparatus and were uncorrected. FTIR spectra were measured on a JASCO FT/IR-6200 infrared spectrophotometer. ¹H NMR spectra were recorded on an either Bruker AVANCE 300 (300 MHz) or JEOL JNM-LA 400 (400 MHz) spectrometer. Chemical shifts of ¹H NMR were reported in parts per million (ppm, δ) relative to CHCl₃ (δ = 7.26) in CDCl₃ or HDO (δ = 4.79) in D₂O. ¹³C NMR spectra were recorded on an either Bruker AVANCE 300 (75 MHz) or JEOL JNM-LA 400 (100 MHz) spectrometer. Chemical shifts of ¹³C NMR were reported in ppm (δ) relative to CHCl₃ (δ = 77.0) in CDCl₃ or CD₂HOD (δ = 49.0) in D₂O. Low resolution mass spectra (LRMS) and high resolution mass spectra (HRMS) were obtained on a JEOL JMS-AX500 for fast atom bombardment ionization (FAB). All reactions were monitored by thin layer chromatography (TLC), which was performed with precoated plates (silica gel 60 F-254, 0.25 mm thickness, manufactured by Merck). TLC visualization was accompanied using UV lamp (254 nm) or a charring solution (ethanoic phosphomolybdic acid, aqueous potassium permanganate and butanoic ninhydrin). Daisogel IR-60 1002W (40/63 μm) was used for flash column chromatography on silica gel.

(2*R*,5*R*)-1-*tert*-Butyl 2-methyl 2-((1*S*,2*R*)-2-(*tert*-butoxycarbonylamino)-1-hydroxy-3-methoxy-3-oxopropyl)-5-(hydroxymethyl)pyrrolidine-1,2-dicarboxylate (**10**)



To a solution of **9** (315 mg, 617 μmol) in EtOAc (3.1 mL) were added Boc₂O (0.43 mL, 1.85 mmol) and 10% Pd/C (31.5 mg, 10 wt%). The mixture was stirred for 21 h under hydrogen at room temperature and filtered through a thin Celite[®] pad. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5:1 to 2:1) to give **10** (264 mg, 90%);

Viscous oil;

$[\alpha]_D^{25} = +1.4$ (c 1.34, CHCl_3);

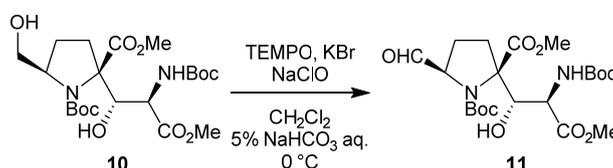
FTIR (neat) 3336, 2979, 1747, 1716, 1676, 1506, 1394, 1369, 1255, 1165 cm^{-1} ;

^1H NMR (300 MHz, CDCl_3) δ 6.43 (brd, $J = 9.3$ Hz, 1 H), 5.59 (brd, $J = 8.1$ Hz, 1 H), 4.39 (m, 1 H), 4.30 (m, 1 H), 4.09–3.98 (m, 2 H), 3.83 (s, 3 H), 3.74 (s, 3 H), 3.51 (m, 1 H), 2.69 (brs, 1 H), 2.45 (m, 1 H), 2.36–2.20 (m, 2 H), 1.91 (m, 1 H), 1.51 (s, 9 H), 1.43 (s, 9 H);

^{13}C NMR (75 MHz, CDCl_3) δ 174.2, 170.7, 156.5, 155.1, 82.4, 80.3, 77.9, 73.8, 64.5, 63.5, 54.8, 53.3, 52.3, 36.1, 28.4, 28.3, 26.3;

HRMS (FAB) calcd for $\text{C}_{21}\text{H}_{37}\text{N}_2\text{O}_{10}$ m/z 477.2448 $[\text{M}+\text{H}]^+$, found 477.2458.

(2*R*,5*R*)-1-*tert*-Butyl 2-methyl 2-((1*S*,2*R*)-2-(*tert*-butoxycarbonylamino)-1-hydroxy-3-methoxy-3-oxopropyl)-5-formylpyrrolidine-1,2-dicarboxylate (11**)**



To a mixture of **10** (102 mg, 214 μmol) and TEMPO (1.0 mg, 6.43 μmol) in CH_2Cl_2 (2.0 mL) and KBr in H_2O (43 μL , 21.4 μmol , 0.5 M solution) was added the mixture of aq. NaOCl solution/5% NaHCO_3 aq. (1:1) at 0 $^\circ\text{C}$ with vigorous stirring until the starting material was consumed (monitored by TLC). The organic layer was separated and the aqueous layer was extracted with EtOAc (5 mL \times 2). The combined organic layers were washed with brine (10 mL), dried over anhydrous MgSO_4 , and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5:1 to 3:1) to give **11** (89.9 mg, 89%);

Colorless viscous oil;

$[\alpha]_D^{23} = +1.0$ (c 2.58, CHCl_3);

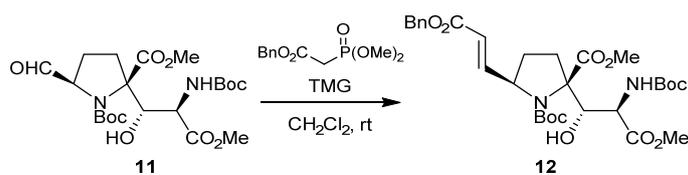
FTIR (neat) 3359, 2979, 1738, 1714, 1687, 1504, 1456, 1437, 1390, 1369, 1257, 1163 cm^{-1} ;

^1H NMR (300 MHz, CDCl_3) δ 9.60 (d, $J = 3.6$ Hz, 1 H), 6.23 (brd, $J = 11.1$ Hz, 1 H), 5.49 (brd, $J = 8.4$ Hz, 1 H), 4.49 (brdd, $J = 8.4, 6.0$ Hz, 1 H), 4.31 (dd, $J = 11.1, 6.0$ Hz, 1 H), 4.21 (dt, $J = 7.2, 3.6$ Hz, 1 H), 3.81 (s, 3 H), 3.74 (s, 3 H), 2.40 (m, 1 H), 2.39 (m, 1 H), 2.14 (m, 1 H), 1.76 (m, 1 H), 1.49 (s, 9 H), 1.43 (s, 9 H);

^{13}C NMR (75 MHz, CDCl_3) δ 199.8, 171.7, 170.9, 155.7, 154.7, 83.8, 80.4, 75.0, 74.9, 68.1, 55.1, 53.2, 52.3, 34.1, 28.3, 28.1, 24.1;

HRMS (FAB) calcd for $\text{C}_{21}\text{H}_{35}\text{N}_2\text{O}_{10}$ m/z 475.2292 $[\text{M}+\text{H}]^+$, found 475.2293.

(2*R*,5*R*)-1-*tert*-Butyl 2-methyl 5-((*E*)-3-(benzyloxy)-3-oxoprop-1-enyl)-2-((1*S*,2*R*)-2-(*tert*-butoxycarbonylamino)-1-hydroxy-3-methoxy-3-oxopropyl)pyrrolidine-1,2-dicarboxylate (12**)**



To a solution of benzyl dimethylphosphonoacetate (300 mg, 1.16 mmol) in CH_2Cl_2 (2.0 mL) was added TMG (146 mL, 1.16 mmol) at 0°C under argon and stirred for 10 min. To the mixture was added a solution of **11** (184 mg, 388 μmol) in CH_2Cl_2 (1.9 mL) at 0°C . The mixture was stirred for 23 h at room temperature and quenched with sat. NH_4Cl (5 mL). The organic layer was separated and the aqueous layer was extracted with EtOAc (5 mL \times 2). The combined organic layers were washed with brine (10 mL), dried over anhydrous MgSO_4 , and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 3:1) to give **12** (209 mg, 89%);

Colorless oil;

$[\alpha]_D^{25} = -16.8$ (c 3.4, CHCl_3);

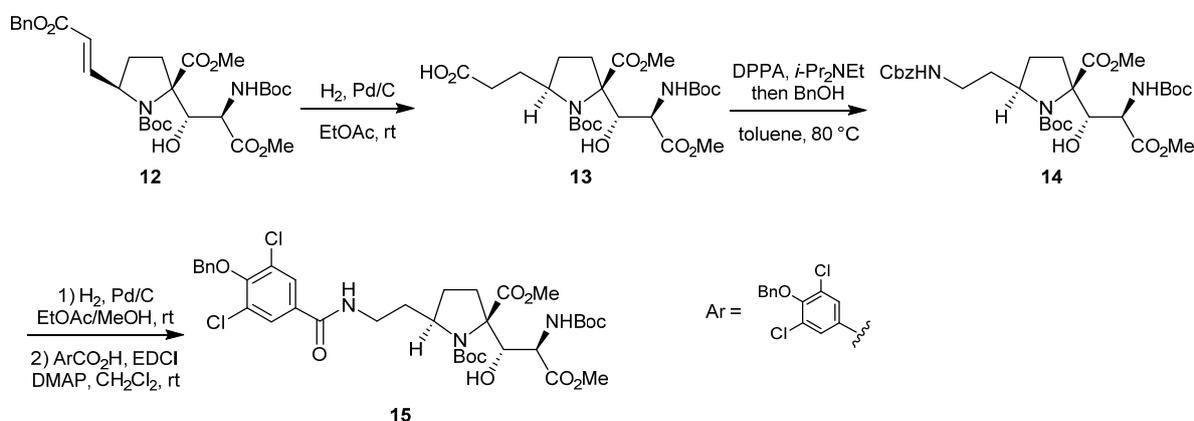
FTIR (neat) 3342, 2979, 1747, 1716, 1678, 1498, 1456, 1389, 1369, 1257, 1163 cm^{-1} ;

^1H NMR (300 MHz, CDCl_3) δ 7.38–7.33 (m, 5 H), 6.97 (dd, $J = 15.8, 7.7$ Hz, 1 H), 6.26 (brs, 1 H), 6.00 (dd, $J = 15.8, 0.8$ Hz, 1 H), 5.57 (brd, $J = 8.4$ Hz, 1 H), 5.23 (d, $J = 12.5$ Hz, 1 H), 5.18 (d, $J = 12.5$ Hz, 1 H), 4.51–4.41 (m, 2 H), 4.30 (dd, $J = 10.2, 4.5$ Hz, 1 H), 3.78 (s, 3 H), 3.72 (s, 3 H), 2.36–2.27 (m, 3 H), 1.68 (m, 1 H), 1.43 (s, 9 H), 1.42 (s, 9 H);

^{13}C NMR (75 MHz, CDCl_3) δ 171.9, 170.7, 166.0, 156.2, 155.0, 148.3, 136.0, 128.5, 128.2, 128.1, 121.0, 82.5, 80.2, 77.2, 73.5, 66.1, 62.2, 55.2, 52.9, 52.2, 34.0, 28.9, 28.2, 28.1;

HRMS (FAB) calcd for $\text{C}_{30}\text{H}_{43}\text{N}_2\text{O}_{11}$ m/z 607.2867 $[\text{M}+\text{H}]^+$, found 607.2867.

(2*R*,5*R*)-1-*tert*-Butyl 2-methyl 5-(2-(4-(benzyloxy)-3,5-dichlorobenzamido)ethyl)-2-((1*S*,2*R*)-2-(*tert*-butoxycarbonylamino)-1-hydroxy-3-methoxy-3-oxopropyl)pyrrolidine-1,2-dicarboxylate (15**)**



To a solution of **12** (90.5 mg, 149 μmol) in EtOAc (1.5 mL) was added 10% Pd/C (9.1 mg, 10 wt%). The mixture was stirred for 24 h under hydrogen at room temperature and filtered through a thin

Celite[®] pad. The filtrate was concentrated under reduced pressure to give corresponding carboxylic acid **13** and the product was used without further purification. To a solution of the crude **13** in toluene (1.5 mL) were added *i*-Pr₂NEt (25.6 mL, 149 μmol) and DPPA (37.2 mL, 164 μmol, 95% purity) under argon. The mixture was stirred for 30 min at room temperature, then for 30 min at 80 °C. To the mixture was added BnOH (17.0 μL, 164 μmol) and additionally stirred for 16 h at 80 °C. After cooling to room temperature, the mixture was quenched with sat. NH₄Cl (5 mL) and extracted with EtOAc (5 mL × 3). The combined organic layers were washed with brine (15 mL), dried over anhydrous MgSO₄, and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5:1 to 2:1) to give crude **14** (67 mg, R_f value = 0.3, hexane/AcOEt = 1:1). The crude **14** contained a byproduct that showed the same R_f value with **14**. Since these were inseparable by further silica gel column chromatography, the mixture was subjected to the next step without further purification. To a solution of the crude **14** (67.0 mg) in EtOAc/MeOH (1:1, 2.0 mL) was added 10% Pd/C (6.7 mg, 10 wt%). The mixture was stirred for 14 h under hydrogen at room temperature and filtered. The filtrate was concentrated under reduced pressure to give corresponding amine and the product was used without further purification. To a solution of the crude amine in CH₂Cl₂ (1.0 mL) were added 3,5-dichloro-4-benzyloxybenzoic acid (31.8 mg, 107 μmol), EDCI (20.5 mg, 107 μmol) and DMAP (13.1 mg, 107 μmol) under argon. The mixture was stirred for 5 h at room temperature and quenched with sat. NH₄Cl (5 mL). The organic layer was separated and the aqueous layer was extracted with EtOAc (5 mL × 2). The combined organic layers were washed with brine (15 mL), dried over anhydrous MgSO₄, and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by PLC (silica gel 60 F-254, 0.5 mm thickness, manufactured by Merck)(hexane/EtOAc = 1:1) to give **15** (43.9 mg, 38% over 4 steps) as a 1:1 mixture of rotamers;

Colorless amorphous solid;

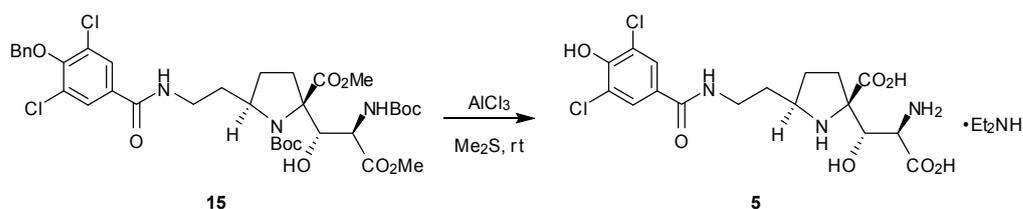
$[\alpha]_D^{24} = -27.3$ (*c* 1.3, CHCl₃);

FTIR (neat) 3336, 2978, 1745, 1716, 1668, 1550, 1456, 1392, 1369, 1259, 1167 cm⁻¹;

The NMR signals of **15** are broaden and complicated because of observation of rotamers. Therefore, NMR data assignments are not given for **15**. The actual spectra are shown in P23–24.

HRMS (FAB) calcd for C₃₆H₄₈Cl₂N₃O₁₁ *m/z* 768.2666 [M+H]⁺, found 768.2653.

(2*R*,5*R*)-2-((1*S*,2*R*)-2-Amino-2-carboxy-1-hydroxyethyl)-5-(2-(3,5-dichloro-4-hydroxybenzamido)ethyl)pyrrolidine-2-carboxylic acid (5)



15

5

To a solution of **15** (15.8 mg, 20.6 μmol) in Me_2S (1.0 mL) was added AlCl_3 (54.9 mg, 412 μmol) at 0 $^\circ\text{C}$ under argon. The mixture was stirred for 15 h at room temperature and quenched with water (1 mL) at 0 $^\circ\text{C}$. The mixture was stirred for 1 h at room temperature and concentrated under reduced pressure. The residue was purified by Dowex[®] 50WX4 (elution with 1N NH_4OH) and reversed-phase column chromatography (elution with water) to give roughly purified product. The product was purified by HPLC (COSMOSIL[®] 5C₁₈-PAQ Packed Column, $\phi 20 \times 250$ mm, elution with 10% $\text{MeOH}/20$ mM $\text{Et}_2\text{NH}\text{-CO}_2$ buffer pH 7, 6.0 mL/min) to give **5** (7.4 mg, 69%) as a diethylamine salt;

White solid;

$[\alpha]_{\text{D}}^{25} = -24.7$ (c 0.11, H_2O);

FTIR (neat) 3057, 1628, 1468, 1298, 1095 cm^{-1} ;

^1H NMR (300 MHz, D_2O) δ 7.68 (s, 2 H), 4.51 (s, 1 H), 4.24 (s, 1 H), 3.73 (m, 1 H), 3.49 (m, 2 H), 3.06 (t, $J = 7.3$ Hz, 4 H, Et_2NH), 2.41 (m, 1 H), 2.26–1.99 (m, 4 H), 1.70 (m, 1 H), 1.27 (t, $J = 7.3$ Hz, 6 H, Et_2NH);

^{13}C NMR (75 MHz, D_2O) δ 174.4, 170.9, 169.2, 162.2, 127.4, 124.2, 117.7, 76.5, 70.8, 59.1, 55.6, 42.4 (Et_2NH), 37.0, 32.1, 31.9, 29.4, 10.6 (Et_2NH);

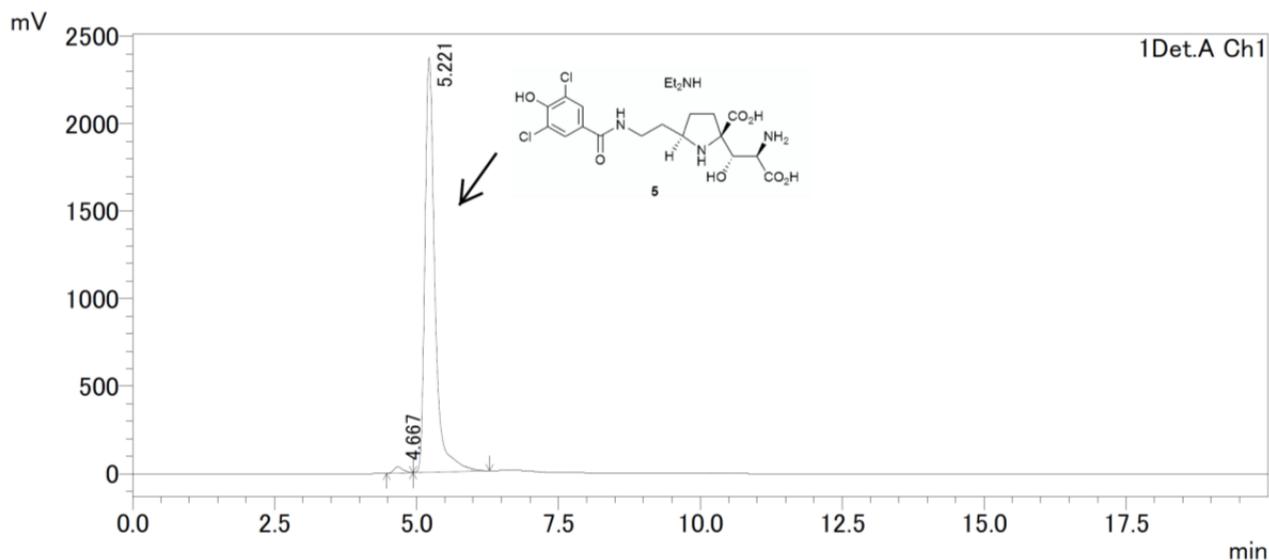
HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{22}\text{Cl}_2\text{N}_3\text{O}_7$ m/z 450.0835 $[\text{M}+\text{H}]^+$, found 450.0840.

HPLC data of **5** after purification:

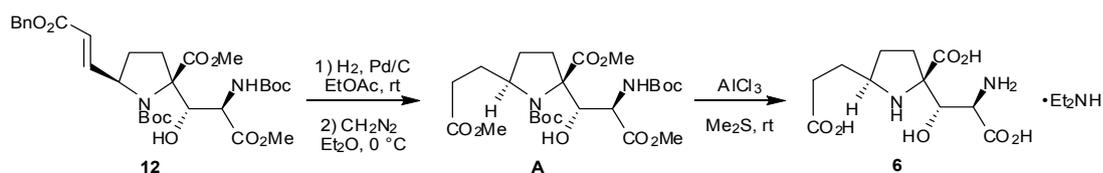
column: COSMOSIL[®] 5C₁₈-PAQ Packed Column, $\phi 4.6 \times 250$ mm

elution: 10% $\text{MeOH}/20$ mM $\text{Et}_2\text{NH}\text{-CO}_2$ buffer pH 7

flow rate: 1 mL/min, detect: 300 nm



(2*R*,5*R*)-2-((1*S*,2*R*)-2-Amino-2-carboxy-1-hydroxyethyl)-5-(2-carboxyethyl)pyrrolidine-2-carboxylic acid (6**)**



To a solution of **12** (38.2 mg, 62.9 μmol) in EtOAc (1.0 mL) was added 10% Pd/C (3.8 mg, 10 wt%). The mixture was stirred for 24 h under hydrogen at room temperature and filtered through a thin Celite[®] pad. The filtrate was concentrated under reduced pressure to give the crude carboxylic acid and the product was used without further purification. To a solution of the crude in Et₂O (1.0 mL) was added excess amount of the solution of CH₂N₂ in Et₂O at 0 °C with stirring until the starting material was consumed (monitored by TLC). The mixture was warm to 40 °C to remove residual CH₂N₂ and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5:1 to 3:1) to give **A** (29.5 mg, 88%). The data of **A** are as follows;

Colorless oil;

$[\alpha]_D^{27} = -22.7$ (*c* 1.45, CHCl₃);

FTIR (neat) 3317, 2978, 1743, 1714, 1672, 1506, 1436, 1392, 1369, 1255, 1163 cm⁻¹;

¹H NMR (300 MHz, CDCl₃) δ 6.47 (brs, 1 H), 5.53 (brd, *J* = 5.4 Hz, 1 H), 4.38 (m, 1 H), 4.26 (dd, *J* = 10.4, 4.4 Hz, 1 H), 3.87 (m, 1 H), 3.78 (s, 3 H), 3.72 (s, 3 H), 3.67 (s, 3 H), 2.34–2.13 (m, 6 H), 1.65–1.55 (m, 2 H), 1.51 (s, 9 H), 1.43 (s, 9 H);

¹³C NMR (75 MHz, CDCl₃) δ 173.4, 172.5, 170.8, 156.4, 155.2, 82.1, 80.3, 77.3, 72.7, 61.6, 55.1, 52.9, 52.3, 51.7, 34.5, 31.8, 28.43, 28.36, 28.0, 26.8;

HRMS (FAB) calcd for C₂₄H₄₁N₂O₁₁ *m/z* 533.2710 [M+H]⁺, found 533.2711.

To a solution of methyl ester **A** (15.7 mg, 29.5 μmol) in Me_2S (1.0 mL) was added AlCl_3 (78.7 mg, 590 μmol) at 0 $^\circ\text{C}$ under argon. The mixture was stirred for 20 h at room temperature, quenched with water (1 mL) at 0 $^\circ\text{C}$. The mixture was stirred for 1 h and concentrated under reduced pressure. The residue was purified by Dowex[®] 50WX4 (elution with 1N NH_4OH) and reversed-phase column chromatography (elution with water) to give roughly purified product. The product was purified by HPLC (COSMOSIL[®] 5C₁₈-PAQ Packed Column, $\phi 20 \times 250$ mm, elution with 0.5% MeOH/20 mM $\text{Et}_2\text{NH}\text{-CO}_2$ buffer pH 7, 6.0 mL/min) to give **6** (5.0 mg, 47%) as a diethylamine salt;

White solid;

$[\alpha]_{\text{D}}^{25} = -35.7$ (*c* 0.41, H_2O);

FTIR (neat) 2989, 1631, 1552, 1452, 1358, 1308, 1090, 1065 cm^{-1} ;

^1H NMR (300 MHz, D_2O) δ 4.50 (s, 1 H), 4.26 (s, 1 H), 3.65 (m, 1 H), 3.06 (t, $J = 7.3$ Hz, 4 H, Et_2NH), 2.43–2.30 (m, 3 H), 2.28–2.05 (m, 3 H), 1.96 (m, 1 H), 1.64 (m, 1 H), 1.26 (t, $J = 7.3$ Hz, 6 H, Et_2NH);

^{13}C NMR (75 MHz, D_2O) δ 181.4, 174.5, 171.4, 76.6, 71.0, 61.3, 55.7, 42.4 (Et_2NH), 34.4, 32.2, 29.3, 28.9, 10.7 (Et_2NH);

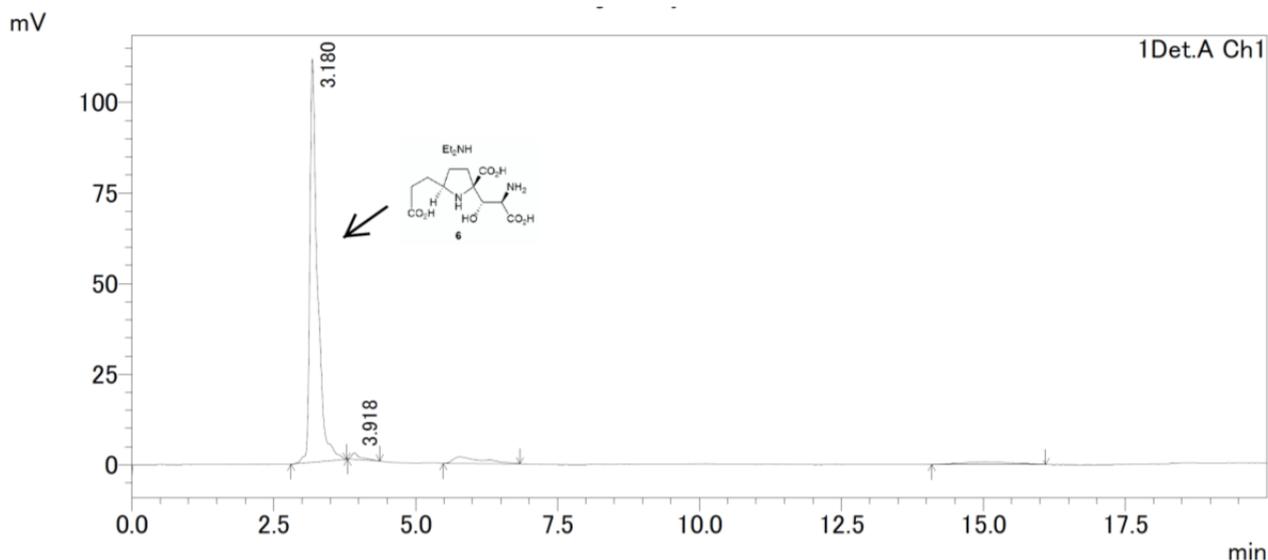
HRMS (FAB) calcd for $\text{C}_{11}\text{H}_{17}\text{N}_2\text{O}_7$ m/z 289.1036 $[\text{M}-\text{H}]^-$, found 289.1052.

HPLC data of **6** after purification:

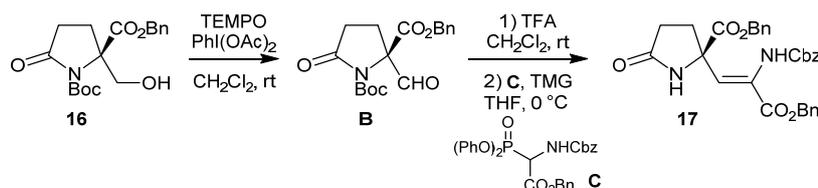
column: COSMOSIL[®] 5C₁₈-PAQ Packed Column, $\phi 4.6 \times 250$ mm

elution: 0.5% MeOH/20 mM $\text{Et}_2\text{NH}\text{-CO}_2$ buffer pH 7

flow rate: 1 mL/min, detect: 210 nm



Synthesis of 17 from 16



To a solution of **16**¹ (972 mg, 2.78 mmol) in CH₂Cl₂ (5.6 mL) were added TEMPO (43.4 mg, 0.278 mmol) and PhI(OAc)₂ (1.08 g, 3.34 mmol) at 0 °C. The mixture was stirred for 19 h at room temperature and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5:1 to 2:1) to give **B** (896 mg, 94%). The data of **B** are as follows;

Colorless oil;

$[\alpha]_D^{25} = +23.1$ (*c* 1.4, CHCl₃);

FTIR (neat) 3469, 2979, 2931, 2354, 1795, 1731, 1455, 1371, 1288, 1259, 1151, 1072, 1025 cm⁻¹;

¹H NMR (400 MHz, CDCl₃) δ 10.2 (s, 1 H), 7.36 (m, 5 H), 2.58–2.53 (m, 2 H), 2.26 (dt, *J* = 13.5, 8.1 Hz, 1 H), 2.13 (m, 1 H), 1.38 (s, 9 H);

¹³C NMR (75 MHz, CDCl₃) δ 193.4, 171.9, 168.6, 148.2, 134.1, 128.5, 128.4, 128.3, 84.3, 71.8, 67.7, 29.9, 27.2, 24.5;

HRMS (FAB) calcd for C₁₈H₂₂NO₆ *m/z* 348.1447 [M+H]⁺, found 348.1447.

To a solution of **B** (152 mg, 442 μmol) in CH₂Cl₂ (4.4 mL) was added TFA (0.34 mL, 4.42 mmol) at 0 °C. The mixture was stirred for 1 h under argon at room temperature and concentrated under reduced pressure. The residual TFA was removed by azeotropic distillation with toluene (3 mL × 3). The resulting aldehyde was subjected to the next olefination without further purification. To a solution of **C** (707 mg, 1.33 mmol) (Synthetic procedure of **C** was described in the next column) in THF (4.4 mL) was added TMG (180 μL, 1.46 mmol) at 0 °C. The mixture was stirred for 15 min under argon, then the crude aldehyde in THF (2.2 mL) was added to the mixture. The mixture was stirred for 30 min at 0 °C, quenched by sat. NH₄Cl (10 mL), and extracted with EtOAc (10 mL × 3). The combined organic layers were washed brine (30 mL), dried over anhydrous MgSO₄, and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 7:1 to 1:2) to give **17** (142 mg, 61%);

Colorless amorphous solid;

$[\alpha]_D^{29} = -35.0$ (*c* 1.0, CHCl₃);

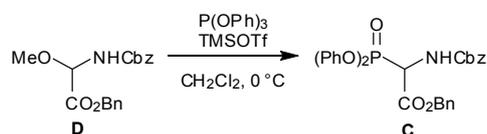
FTIR (neat) 3262, 3032, 1718, 1498, 1455, 1379, 1215, 1182, 1163, 1127, 1064 cm⁻¹;

¹H NMR (400 MHz, CDCl₃) δ 7.36–7.27 (m, 15 H), 6.92 (brs, 1 H), 6.65 (brs, 1 H), 6.57 (s, 1 H), 5.18–5.05 (m, 6 H), 2.54 (m, 1 H), 2.39–2.28 (m, 3 H);

¹³C NMR (100 MHz, CDCl₃) δ 177.0, 171.4, 163.8, 154.5, 135.4, 135.0, 134.8, 131.7, 128.63, 128.60,

128.57, 128.5, 128.40, 128.36, 128.30, 128.28, 67.9, 64.2, 33.4, 29.1;
HRMS (FAB) calcd for C₃₀H₂₉N₂O₇ *m/z* 529.1975 [M+H]⁺, found 529.1972.

Benzyl 2-(benzyloxycarbonylamino)-2-(diphenoxyphosphoryl)acetate (C)



To a solution of **D**² (1.90 g, 5.75 mmol) in CH₂Cl₂ (12 mL) was added triphenyl phosphite (2.3 mL, 8.63 mmol) and TMSOTf (1.2 mL, 6.90 mmol) at 0 °C under argon. The mixture was stirred for 3 h, and quenched with sat. NaHCO₃ (15 mL). The organic layer was separated and the aqueous layer was extracted with EtOAc (15 mL × 2). The combined organic layers were washed with brine (45 mL), dried over anhydrous MgSO₄, and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 10:1 to 4:1), and recrystallized from EtOAc/hexane to give **C** (2.23 g, 73%);

White solid;

mp 102–105 °C;

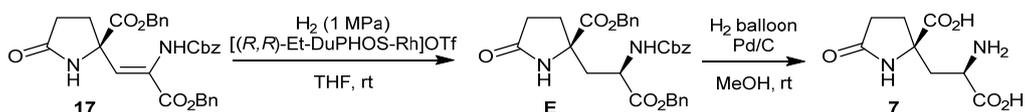
FTIR (neat) 3295, 3064, 2952, 1723, 1591, 1490, 1456, 1379, 1286, 1207, 1185, 1163, 1049, 1027, 1006 cm⁻¹;

¹H NMR (400 MHz, CDCl₃) δ 7.34–7.23 (m, 14 H), 7.18–7.14 (m, 2 H), 7.07 (d, *J* = 7.8 Hz, 4 H), 5.76 (brd, *J* = 9.5 Hz, 1 H), 5.32 (dd, *J* = 23.2, 9.5 Hz, 1 H), 5.27 (d, *J* = 12.2 Hz, 1 H), 5.23 (d, *J* = 12.2 Hz, 1 H), 5.15 (d, *J* = 12.1 Hz, 1 H), 5.11 (d, *J* = 12.1 Hz, 1 H);

¹³C NMR (100 MHz, CDCl₃) δ 166.0, 165.9, 155.53, 155.46, 150.0, 149.9, 135.7, 134.5, 129.8, 128.6, 128.53, 128.52, 128.3, 128.2, 125.6, 120.39, 120.35, 120.29, 120.25, 68.5, 67.8, 53.8, 52.3;

HRMS (FAB) calcd for C₂₉H₂₇NO₇P *m/z* 532.1525 [M+H]⁺, found 532.1526.

(*R*)-2-((*R*)-2-Amino-2-carboxyethyl)-5-oxopyrrolidine-2-carboxylic acid (7)



In a glove box, [Rh(I)(COD)-(*R,R*)-Et-DuPHOS]OTf (28.4 mg, 39.3 μmol) and **17** (208 mg, 394 μmol) was dissolved in THF (2.6 mL). The mixture was placed in a high-pressure hydrogen tube under argon. The tube was sealed cooled to –78 °C. The tube was vacuumed and then hydrogen was introduced into the tube. After repeating of the gas-exchange process for 3 times, the mixture was stirred for 72 h at room temperature under 1.0 MPa of hydrogen atmosphere. The hydrogenation was carefully leaked from the

tube, and the mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 3:1 to 1:2) to give **E** (161 mg, 77%, dr = >25:1). The data of **E** are as follows;

Colorless amorphous solid;

$[\alpha]_D^{21} = -21.2$ (*c* 1.11, CHCl₃);

FTIR (neat) 3355, 3018, 1721, 1498, 1456, 1378, 1340, 1261, 1215, 1167, 1081, 1048, 1028 cm⁻¹;

¹H NMR (400 MHz, CDCl₃) δ 7.36–7.29 (m, 15 H), 6.33 (brs, 1 H), 5.43 (brs, 1 H), 5.17–5.04 (m, 6 H), 4.48 (brs, 1 H), 2.61 (brd, *J* = 11.0 Hz, 1 H), 2.40–2.30 (m, 3 H), 2.14–2.04 (m, 2 H);

¹³C NMR (75 MHz, CDCl₃) δ 177.1, 172.7, 171.5, 156.1, 136.1, 135.0, 128.8, 128.7, 128.6, 128.5, 128.4, 67.9, 67.4, 64.1, 51.5, 40.8, 32.1, 29.2;

HRMS (FAB) calcd for C₃₀H₃₁N₂O₇ *m/z* 531.2131 [M+H]⁺, found 531.2156.

To a solution of **E** (139 mg, 0.262 mmol, dr = >25:1) in MeOH (2.6 mL) was added 10% Pd/C (13.9 mg, 10 wt%). The mixture was stirred under hydrogen for 3.5 h at room temperature and filtration through a thin Celite[®] pad. The filtrate was concentrated under reduced pressure. The residue was purified by Dowex[®] 50WX4 (elution with 1N NH₄OH) to give **7** (51.5 mg, 84%, dr = >25:1) as an ammonium salt;

Brown solid;

mp 170–173 °C;

$[\alpha]_D^{29} = -3.6$ (*c* 1.21, H₂O);

FTIR (H₂O) 2927, 1655, 1597, 1458, 1396 cm⁻¹;

¹H NMR (300MHz, D₂O) δ 3.77 (dd, *J* = 8.9, 3.5 Hz, 1 H), 2.48–2.42 (m, 2 H), 2.40–2.16 (m, 4 H);

¹³C NMR (100 MHz, D₂O) δ 181.5, 181.0, 175.2, 67.4, 52.7, 39.9, 31.6, 30.7;

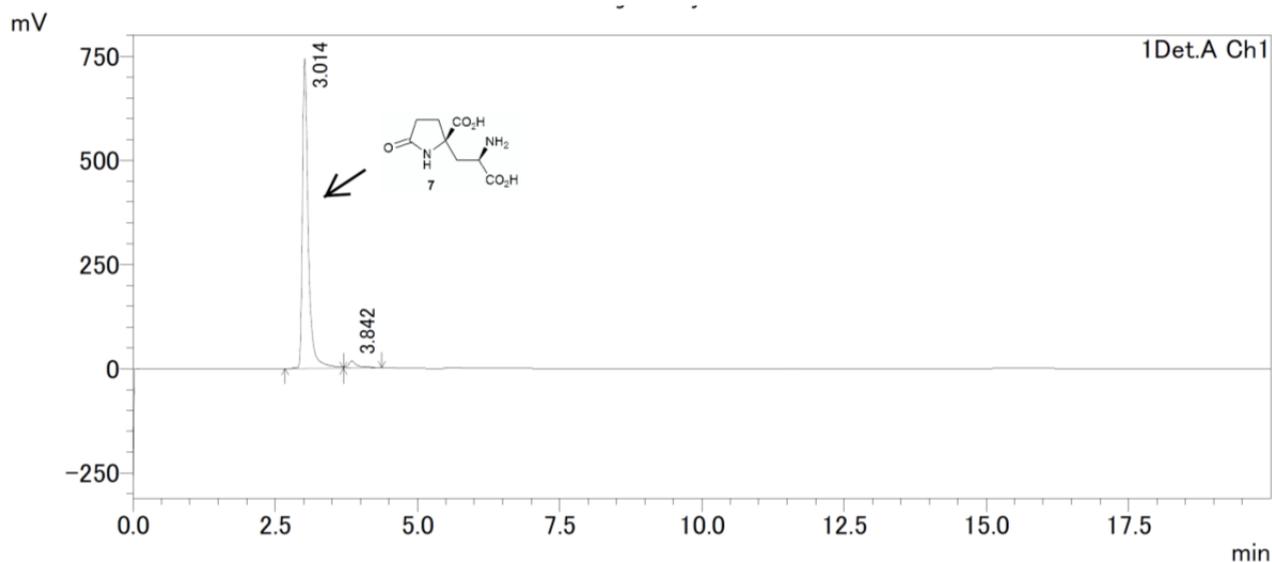
HRMS (FAB) calcd for C₈H₁₃N₂O₅ *m/z* 217.0824 [M+H]⁺, found 217.0816.

HPLC data of **7** after purification:

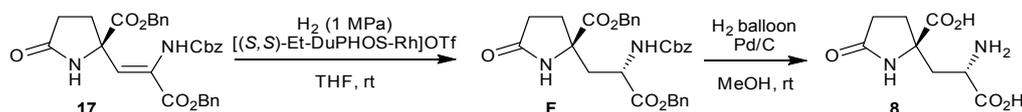
column: COSMOSIL[®] 5C₁₈-PAQ Packed Column, φ4.6 × 250 mm

elution: 0.5% MeOH/20 mM Et₂NH-CO₂ buffer pH 7

flow rate: 1 mL/min, detect: 210 nm



(R)-2-((S)-2-Amino-2-carboxyethyl)-5-oxopyrrolidine-2-carboxylic acid (8**)**



According to the experimental procedure of **7** from **17**, A mixture of **17** (211 mg, 399 μmol) and $[\text{Rh}(\text{I})(\text{COD})-(S,S)\text{-Et-DuPHOS}]\text{OTf}$ (29 mg, 40.0 μmol) in THF (2.7 mL) was placed in a hydrogenation tube and pressurized to 1.0 MPa with hydrogen at room temperature for 72 h. The mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 3:1 to 1:2), followed by preparative TLC (hexane/EtOAc = 1:2) to give **F** (168 mg, 79%, dr = 25:1). The data of **F** are as follows;

Colorless amorphous solid;

$[\alpha]_{\text{D}}^{21} = -3.4$ (c 1.22, CHCl_3);

FTIR (neat) 3320, 3033, 2952, 1737, 1700, 1518, 1499, 1456, 1378, 1218, 1178, 1072 cm^{-1} ;

^1H NMR (400 MHz, CDCl_3) δ 7.38–7.30 (m, 15 H), 6.98 (s, 1 H), 5.19–4.89 (m, 7 H), 4.59 (m, 1 H), 2.53–2.06 (m, 6 H);

^{13}C NMR (75 MHz, CDCl_3) δ 176.9, 173.6, 171.2, 156.4, 135.8, 135.1, 134.9, 128.8, 128.7, 128.63, 128.55, 128.51, 128.47, 128.1, 67.9, 67.8, 67.5, 63.4, 51.0, 41.3, 33.0, 29.2;

HRMS (FAB) calcd for $\text{C}_{30}\text{H}_{31}\text{N}_2\text{O}_7$ m/z 531.2131 $[\text{M}+\text{H}]^+$, found 531.2126.

To a solution of **F** (142 mg, 0.268 mmol, dr = 25:1) in MeOH (2.7 mL) was added 10% Pd/C (14.2 mg, 10 wt%), the mixture was stirred under hydrogen for 3 h at room temperature and filtration through a thin Celite[®] pad. The filtrate was concentrated under reduced pressure. The residue was purified by Dowex[®] 50WX4 (elution with 1N NH_4OH) to give **8** (40.7 mg, 65%, dr = 25:1) as an ammonium salt;

Brown solid;

mp 171–176 °C;

$[\alpha]_D^{21} = +10.4$ (c 1.04, H₂O);

FTIR (H₂O) 3031, 1652, 1591, 1459, 1444, 1395 cm⁻¹;

¹H NMR (300MHz, D₂O) δ 3.70 (brt, $J = 6.2$ Hz, 1 H), 2.49–2.31 (m, 5 H), 2.15 (m, 1 H);

¹³C NMR (75 MHz, D₂O) δ 181.9, 180.7, 175.3, 67.8, 53.1, 40.7, 33.7, 30.5;

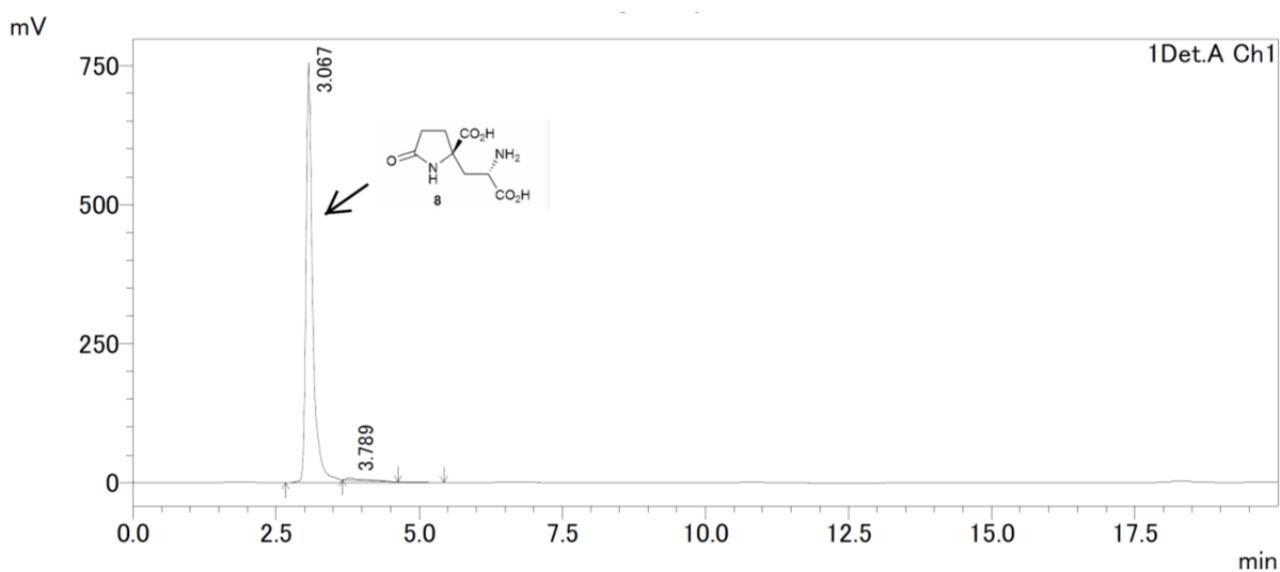
HRMS (FAB) calcd for C₈H₁₁N₂O₅ m/z 215.0668 [M-H]⁻, found 215.0663.

HPLC data of **8** after purification:

column: COSMOSIL[®] 5C₁₈-PAQ Packed Column, ϕ 4.6 × 250 mm

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flow rate: 1 mL/min, detect: 210 nm

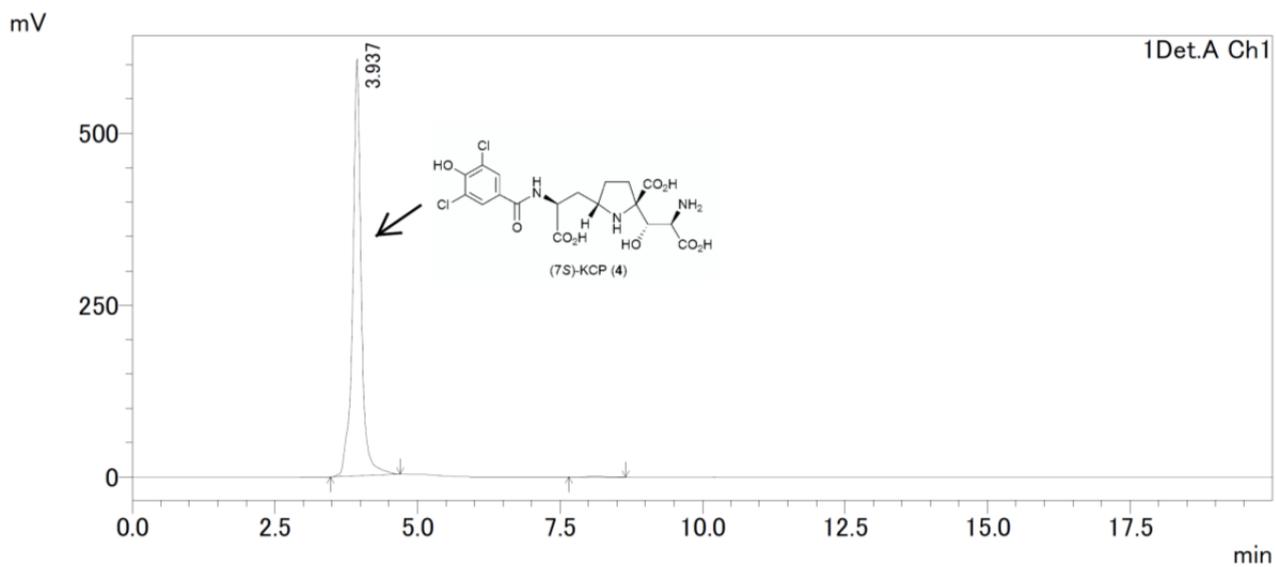
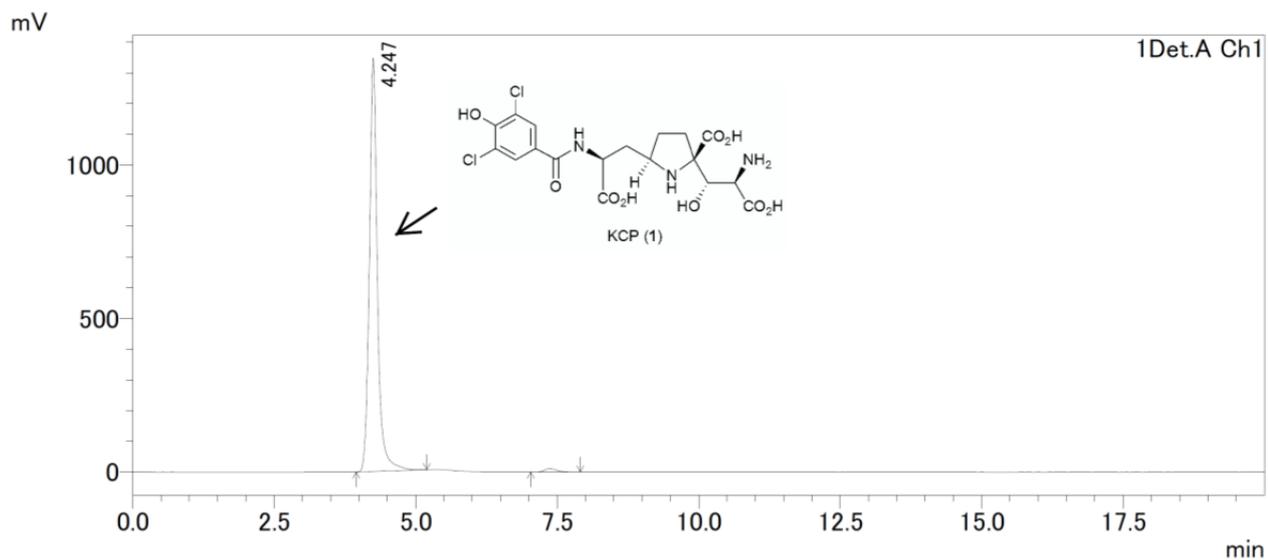


HPLC data of KCP (1) and (7S)-KCP (4)

column: COSMOSIL[®] 5C₁₈-PAQ Packed Column, $\phi 4.6 \times 250$ mm

elution: 5% MeOH/20 mM Et₂NH-CO₂ buffer pH 7

flow rate: 1 mL/min, detect: 300 nm



Receptor binding assay.

Rat brain synaptic membranes were prepared³ and modified⁴ as described previously and stored at $-78\text{ }^{\circ}\text{C}$ until use. On the day of the assay, the membrane suspension was incubated in a buffer containing 0.04% Triton X-100 at $37\text{ }^{\circ}\text{C}$ for 15 min. Triton X-100 was removed by centrifugation, and the pellet was washed three times with an assay buffer. Binding assays were performed according to published methods.⁵ Incubation conditions were as follows (ligand, ligand concentration, temperature, time, buffers); for NMDA receptors: [³H]CGP 39653, 2 nM, $4\text{ }^{\circ}\text{C}$, 1 h, 50 mM Tris-HCl buffer pH 7.6; for AMPA receptors: [³H]AMPA, 5 nM, $4\text{ }^{\circ}\text{C}$, 1 h, 50 mM Tris-HCl buffer pH 7.6 containing 100 mM KSCN; for KA receptors: [³H]KA, 1 nM, $4\text{ }^{\circ}\text{C}$, 1 h, 50 mM Tris-HCl buffer pH 7.6.

Calculation of K_i values.

K_i values were calculated from the equation $K_i = \text{IC}_{50}/(1 + [\text{H-labelled ligand}]/K_d)$. The K_d values were used 19 nM for [³H]CGP 39653, 10 nM for [³H]AMPA, and 3.8 nM for [³H]KA. Each IC_{50} value for **1** or **4–8** was determined based on the concentration-inhibition curve (Fig. 4 and Fig. S1) using GraphPad Prism 3.03. K_i values are indicated as the mean \pm SE for three determinations.

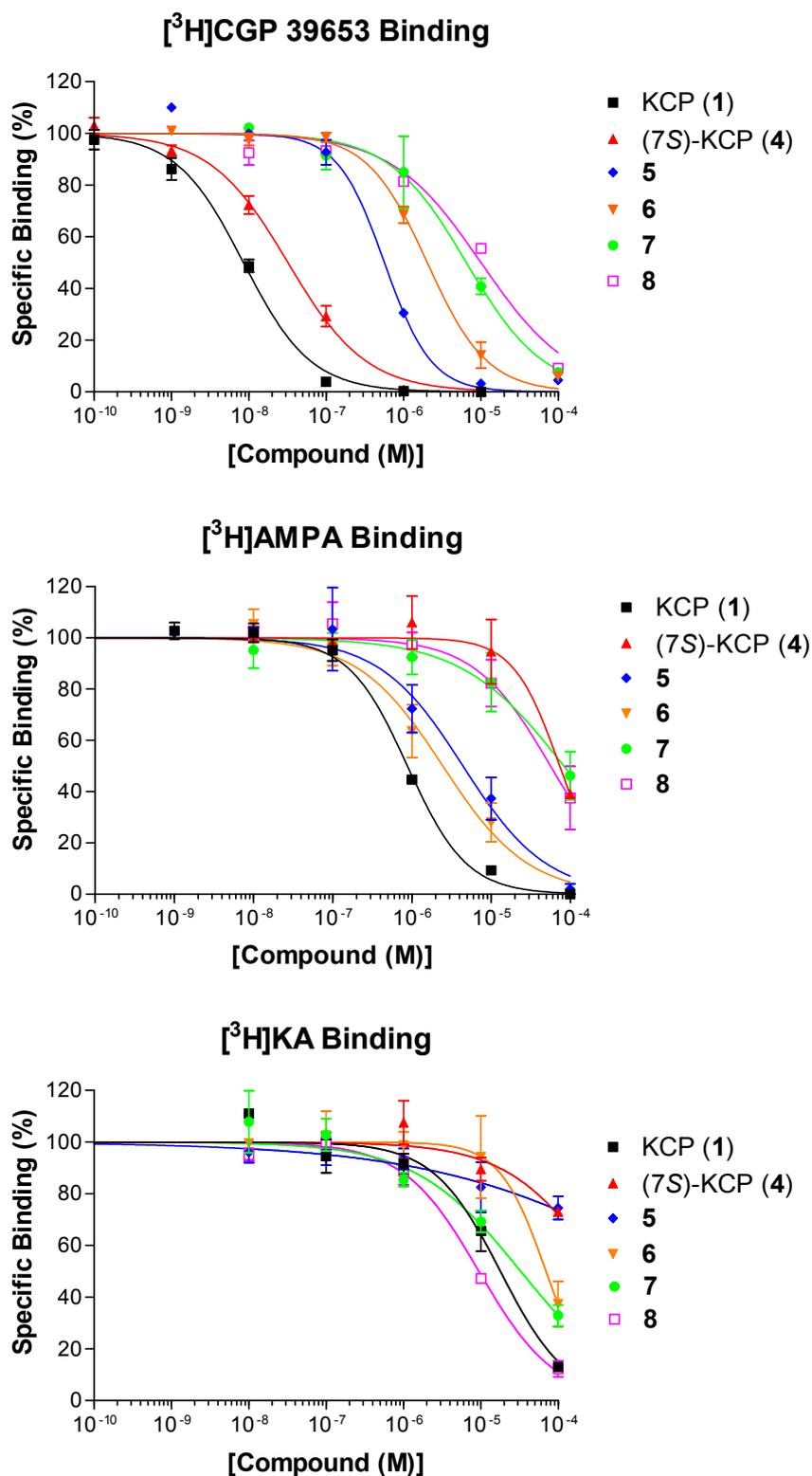


Fig. S1 Displacement of the specific ³H-labelled ligand binding (NMDA, AMPA, and KA receptors) to rat synaptic membranes by increasing concentrations of **1** and **4–8**. Each point is the mean of triplicate determinations.

References

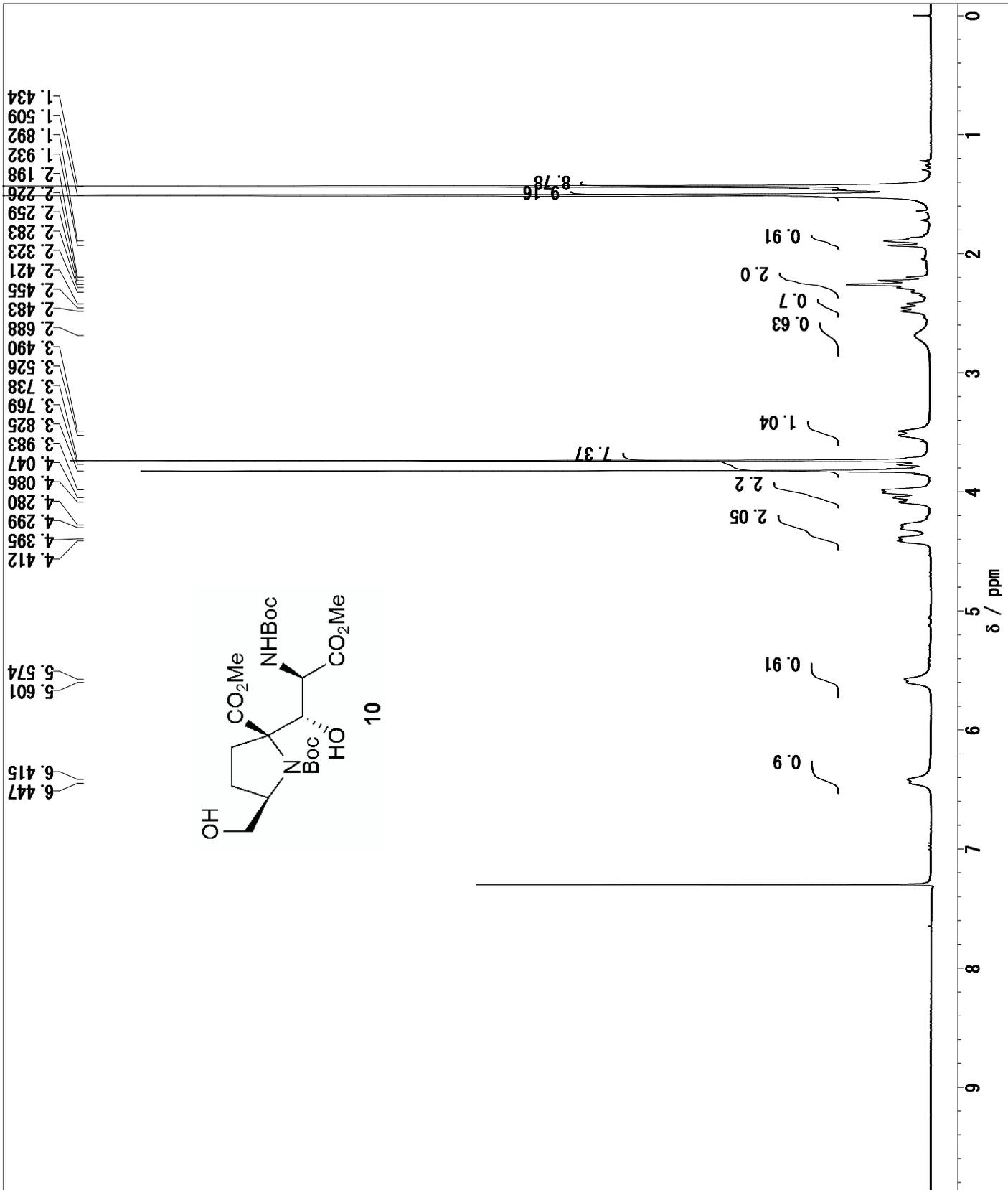
1. T. Shinada, H. Yoshida, Y. Ohfuné, *Synthesis* **2009**, 3751.
2. Y. Harayama, M. Yoshida, D. Kamimura, Y. Wada, Y. Kita, *Chem. Eur. J.* **2006**, *12*, 4893.
3. S. J. Enna, S. H. Snyder, *Mol. Pharmacol.* **1977**, *13*, 422.
4. D. E. Murphy, A. J. Hutchison, S. D. Hurt, M. Williams, M. A. Sills, *Br. J. Pharmacol.* **1988**, *95*, 932.
5. (a) M. Kawai, Y. Horikawa, T. Ishihara, K. Shimamoto, Y. Ohfuné, *Eur. J. Pharmacol.* **1992**, *211*, 195. (b) D. E. Murphy, E. W. Snowhill, M. Williams, *Neurochem. Res.* **1987**, *12*, 775. (c) E. D. London, J. T. Coyle, *Mol. Pharmacol.* **1979**, *15*, 492.

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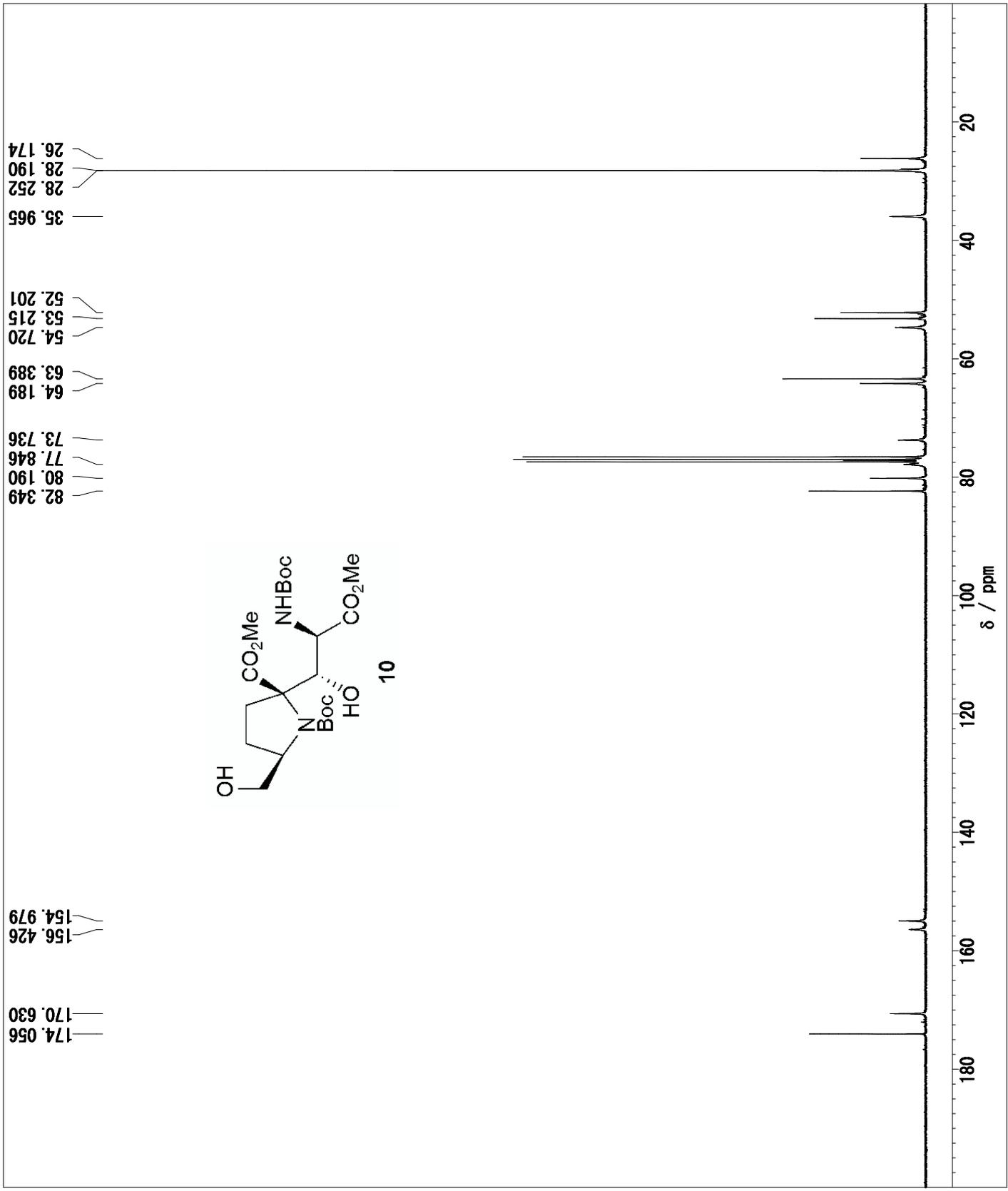
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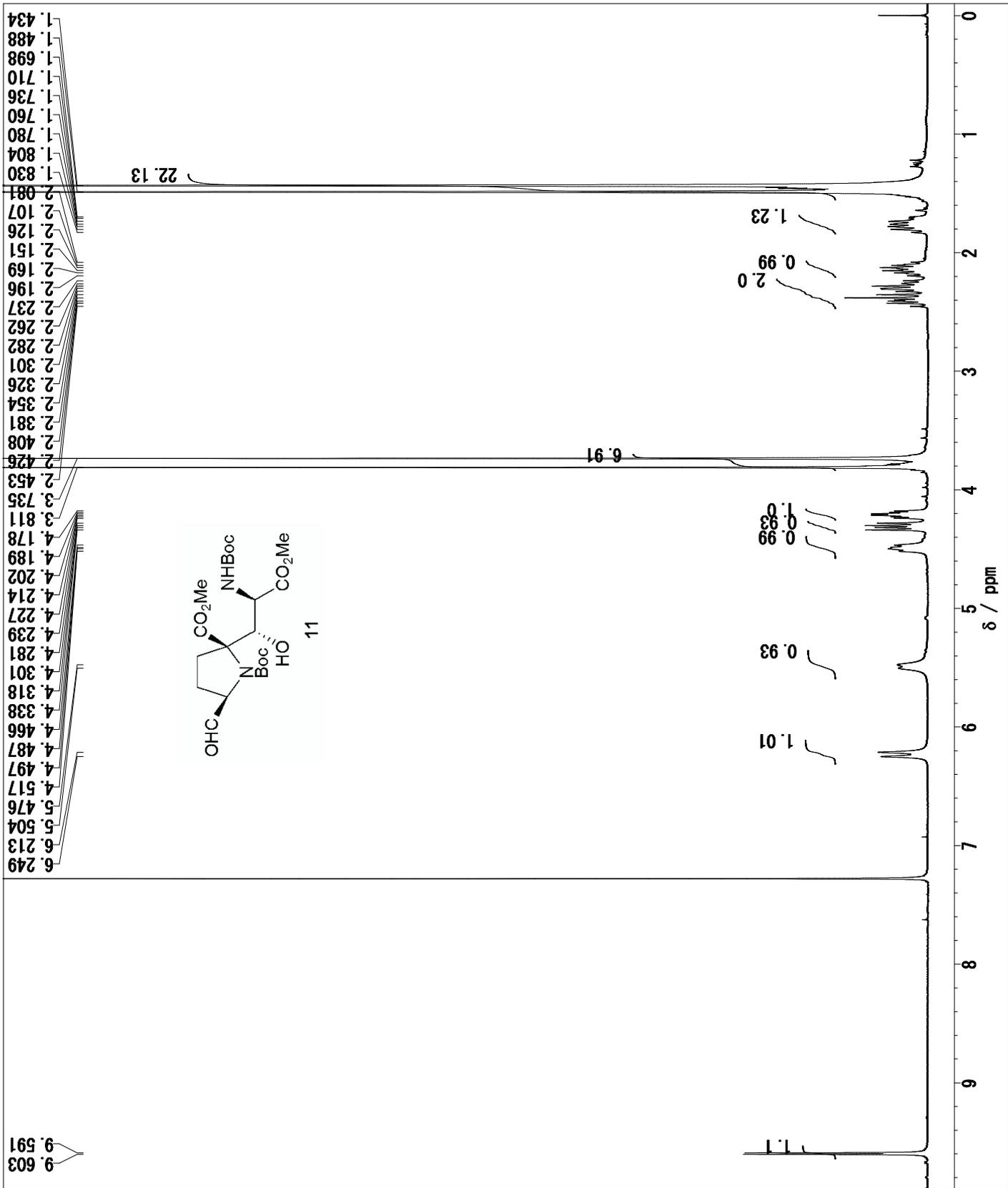
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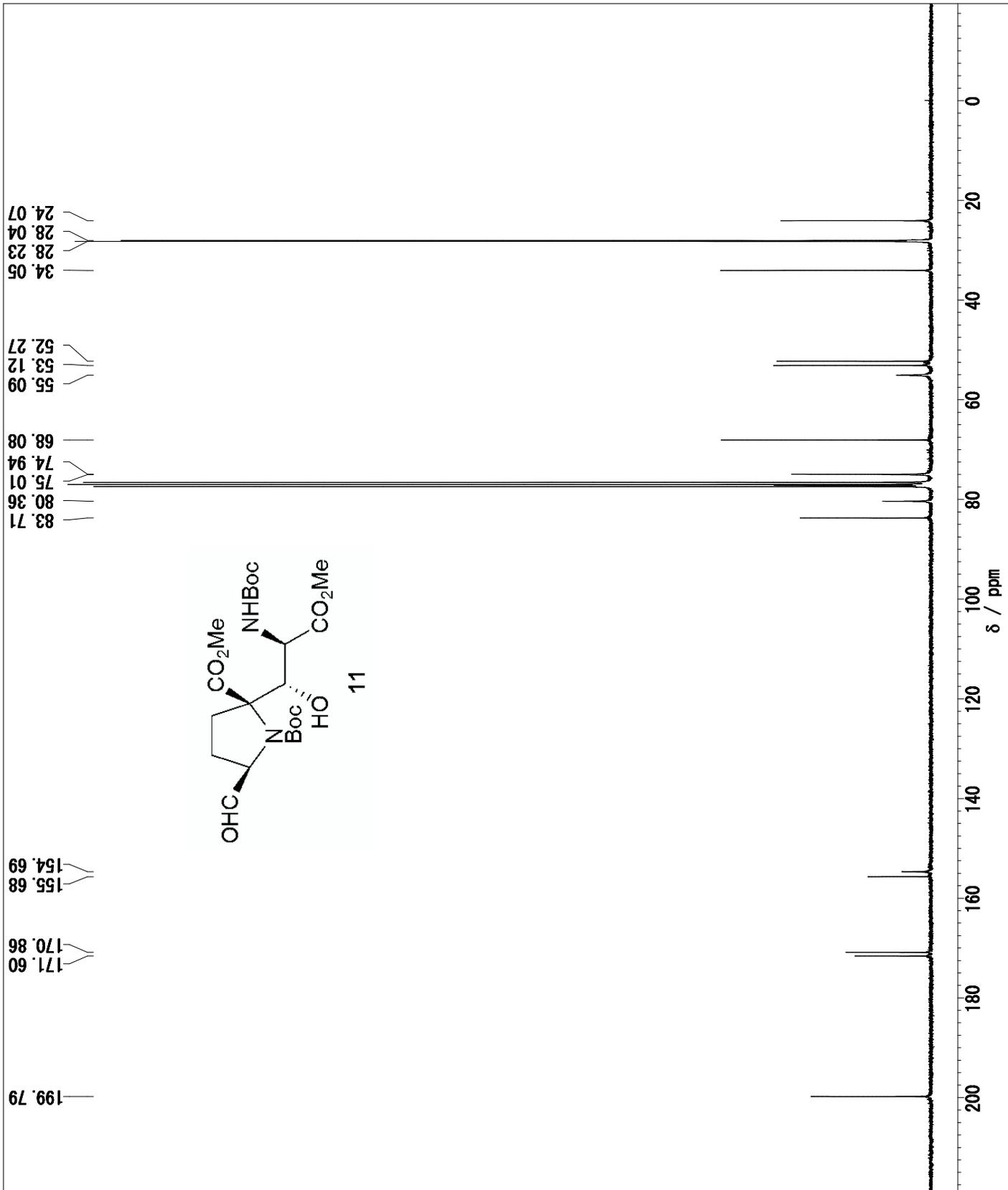
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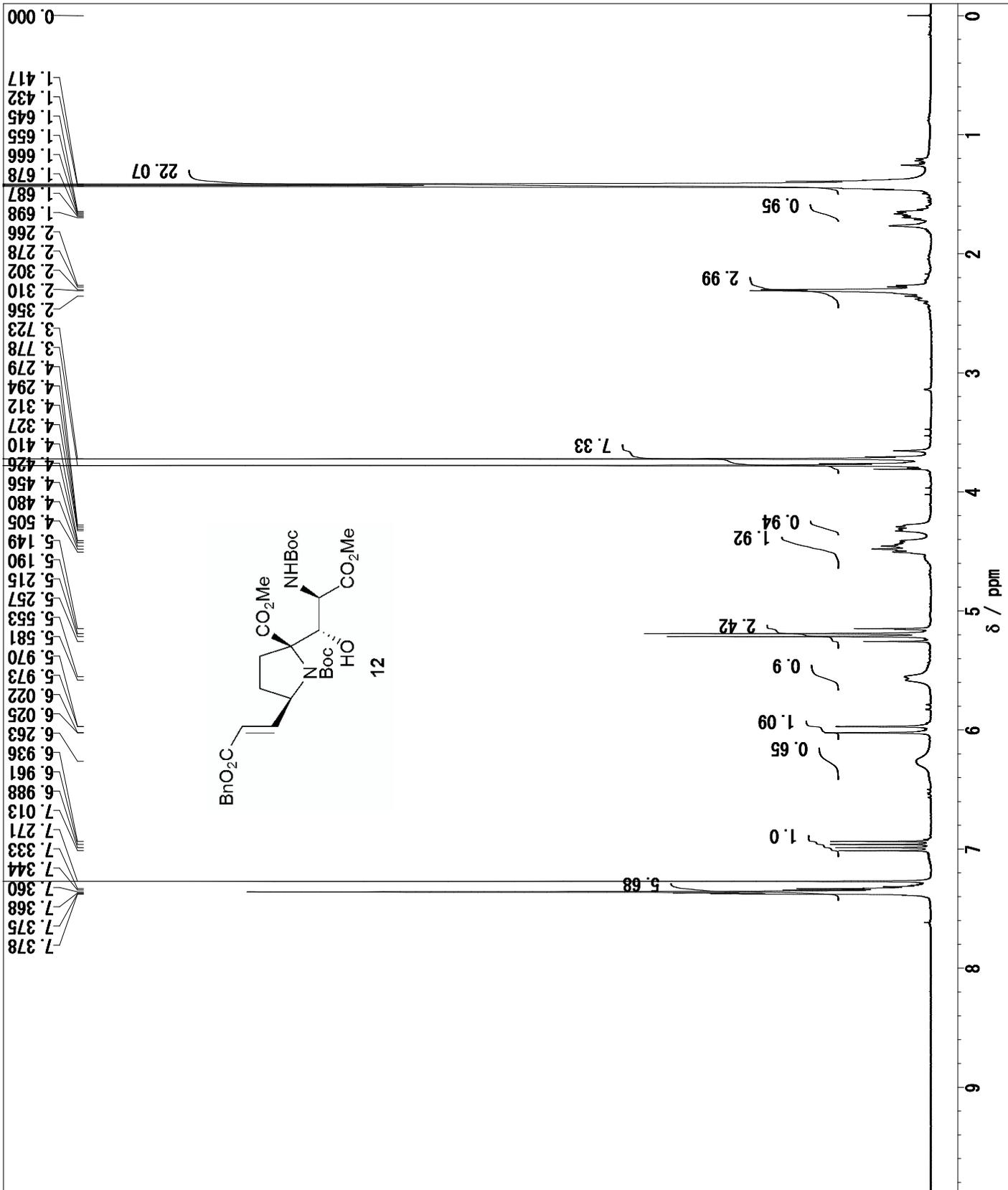


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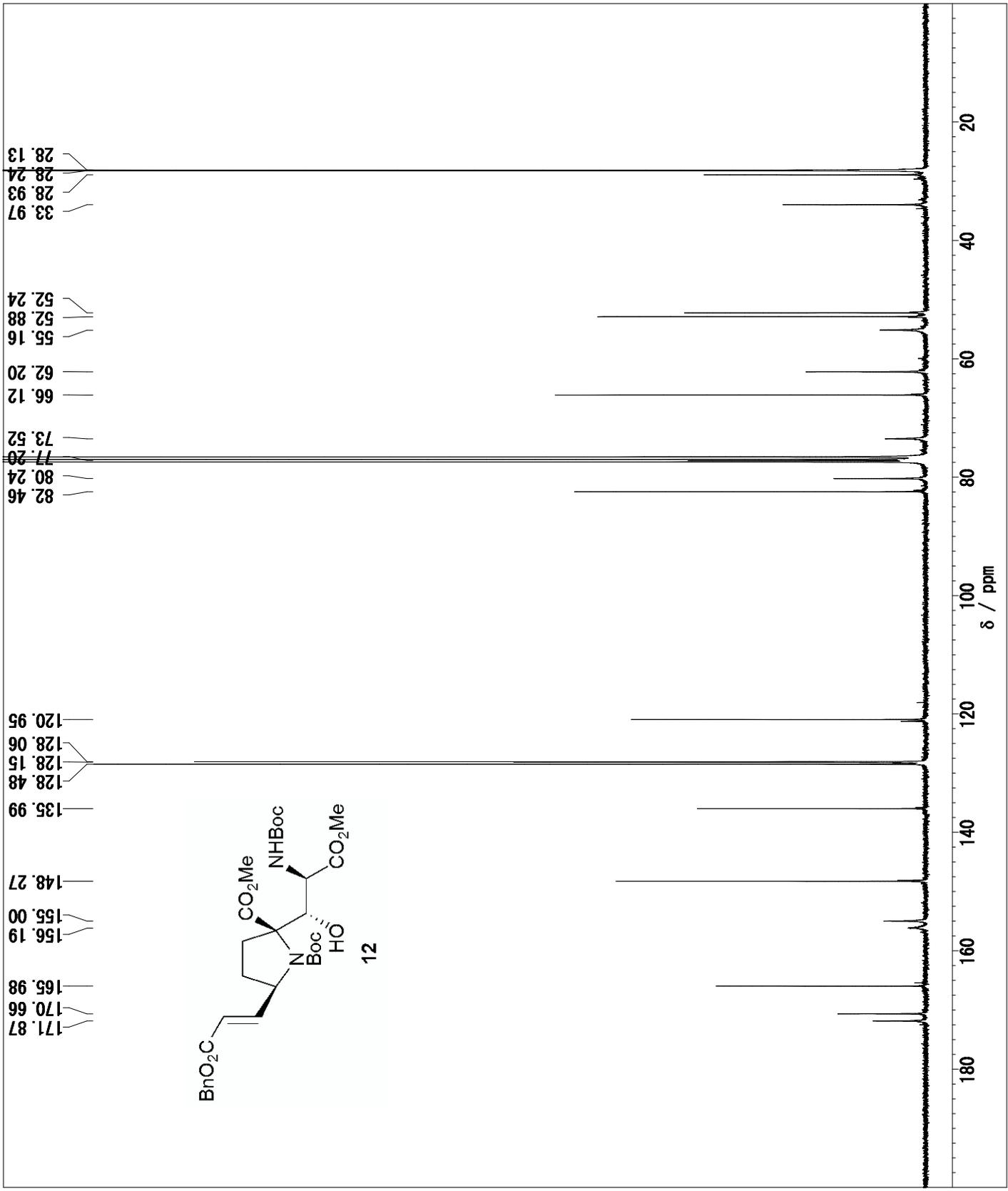
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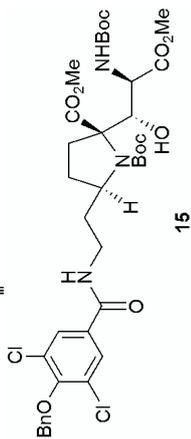


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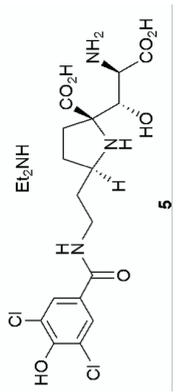
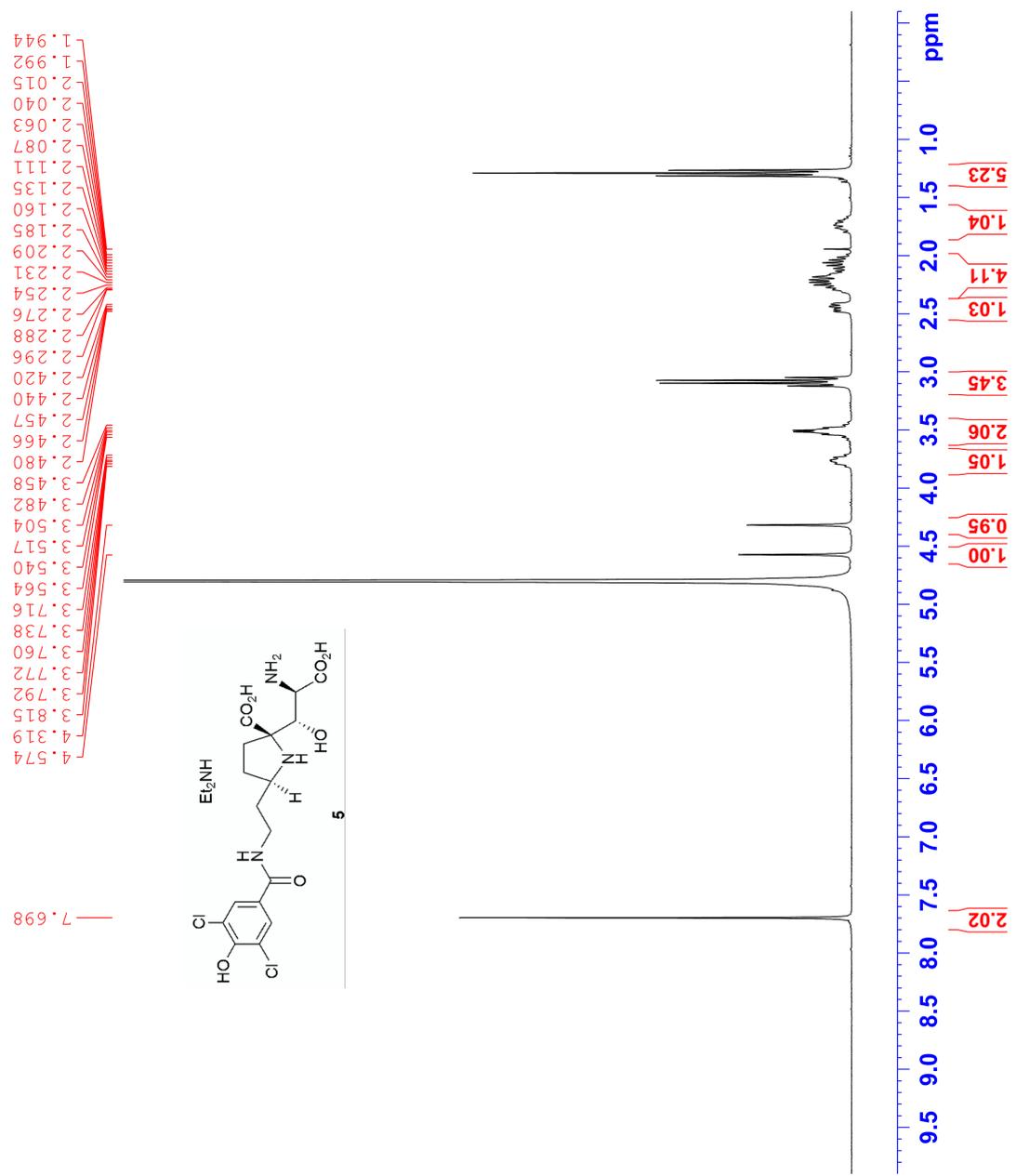


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 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

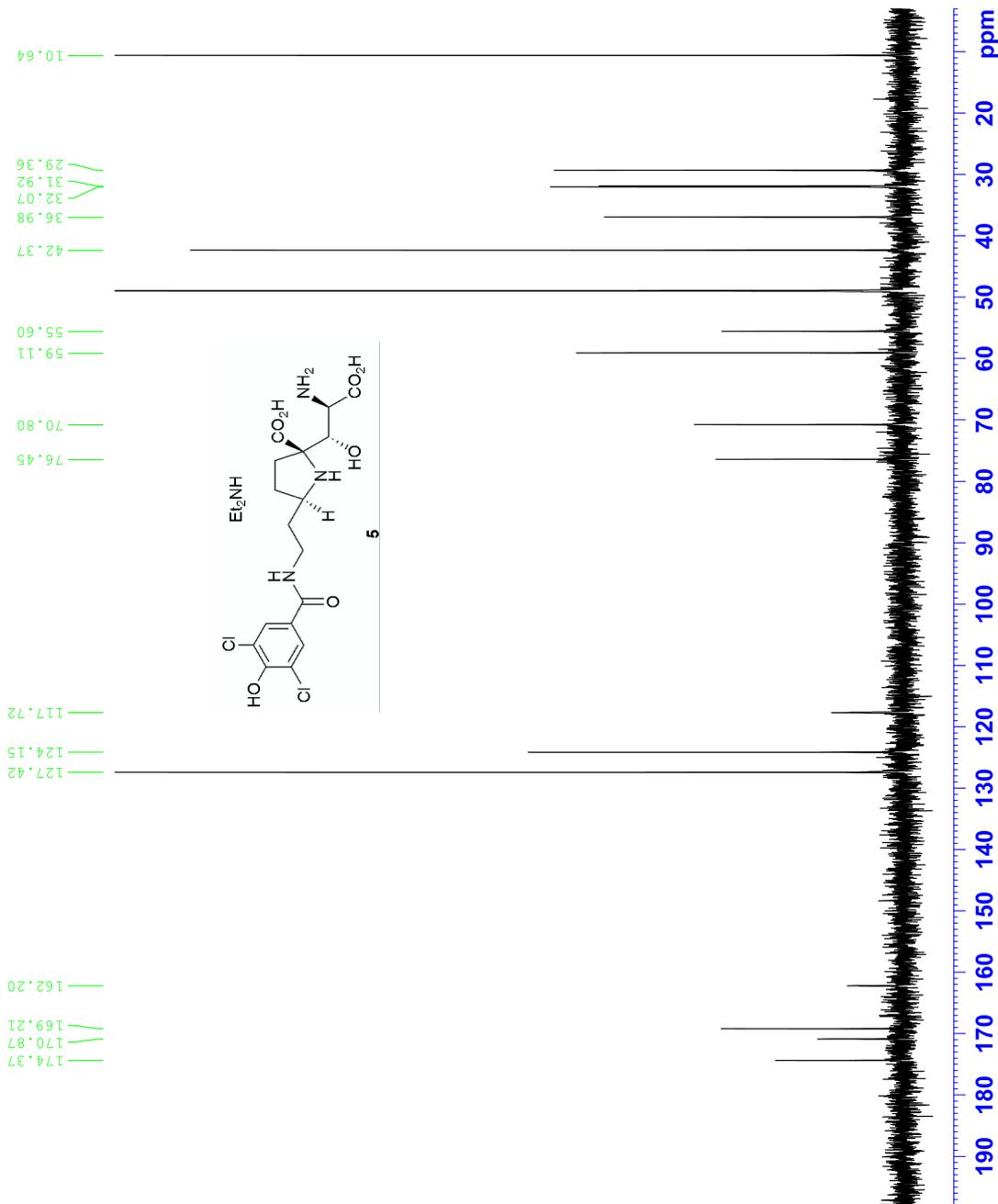




NAME 09182009-HM1495
EXPNO 20
PROCNO 1
Date_ 20090919
Time_ 9.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 12200
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 406
DW 27.733 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

=====
CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -0.80 dB
PL1W 38.05139160 W
SFO1 75.4752953 MHz

=====
CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PLI2 16.50 dB
PLI3 16.50 dB
PLI2W 7.25881100 W
PLI2W 0.25755233 W
PLI3W 0.25755233 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677369 MHz
EM 0
WDW 1.00 Hz
SSB 0
GB 0
PC 1.40



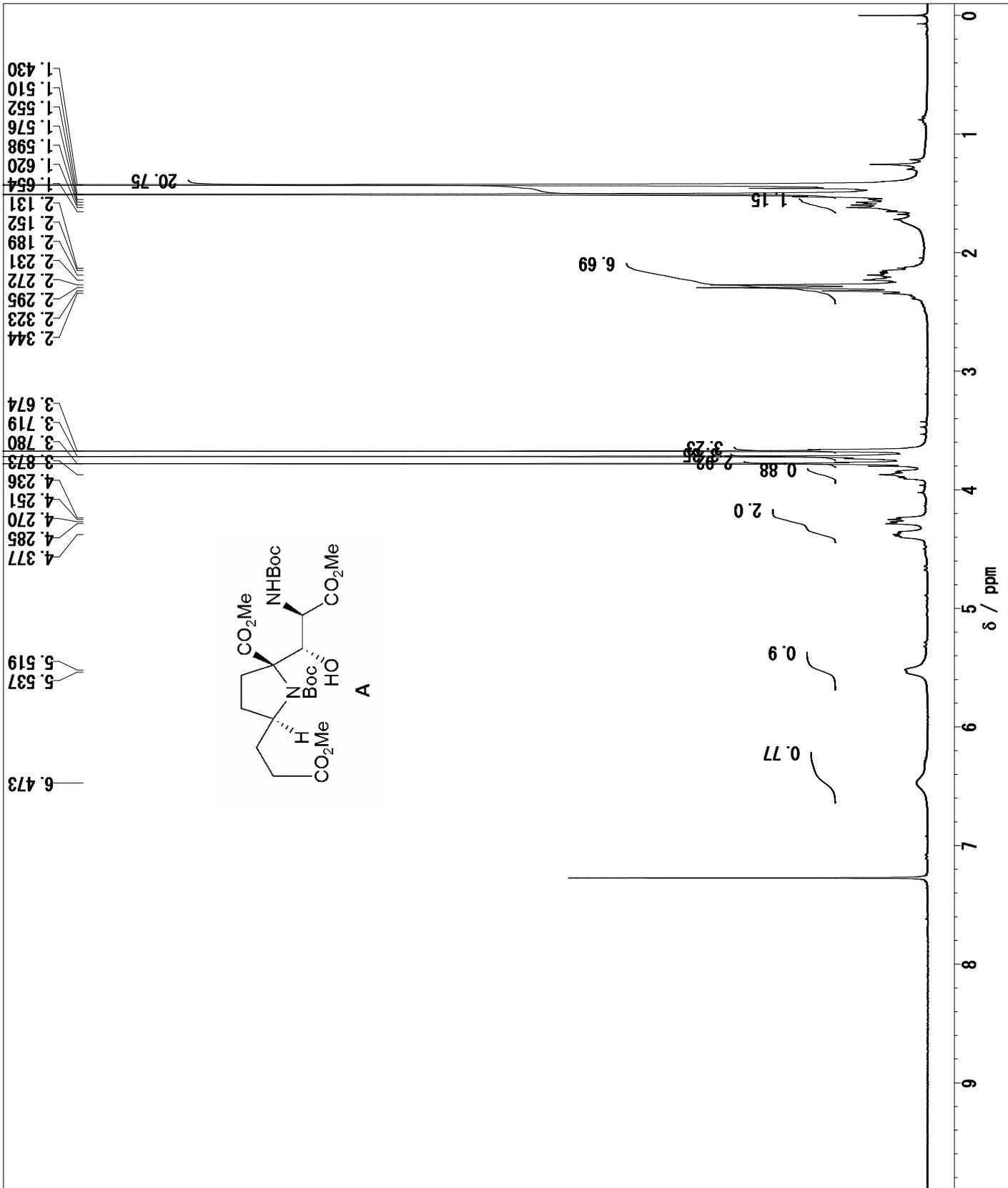
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 業生フォルダ\2010卒業生\HAMAMA
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 CM\NM\08182009-PRO\DAKC\10\PP
 DATA\1\1R

Original File: C:\USERS\YOCU\DOC
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 ER\DATA\MCM\NM\08182009-PRO
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Date 18/Aug/2009 20:13:41

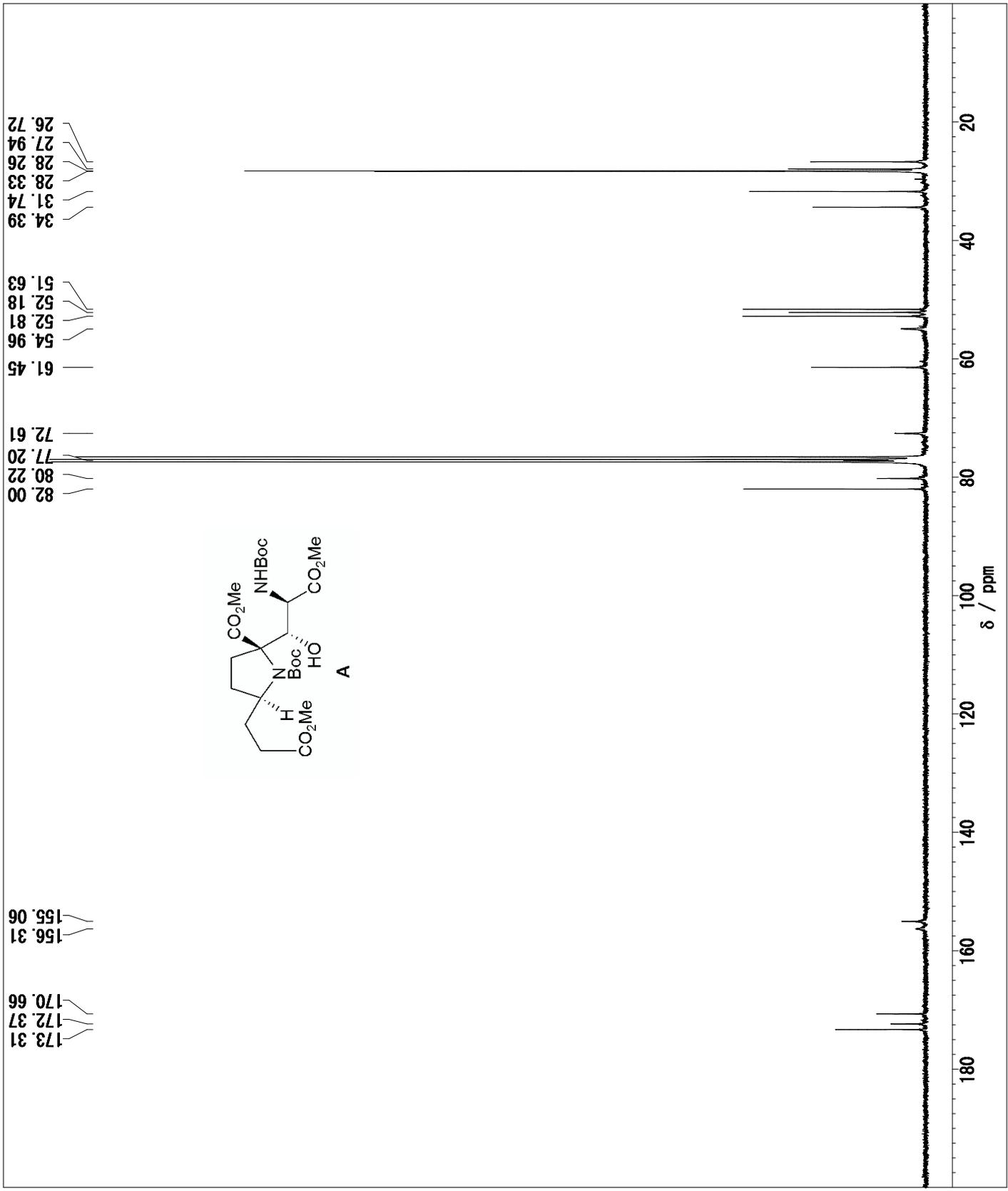
Comment:

ObsNuc ¹H
 ObsFreq 300.13 MHz
 ObsSet 0.0 kHz
 ObsFine 9996.736 Hz
 Pulse1 15.0 μs
 Pulse2 15.0 μs
 Pulse3 30.0 μs
 P11 1.0 ms
 P12 0.0 ms
 P13 0.0 ms
 Loop1 0
 Point 32768
 Scan 16
 DummyScan 2
 Frequency (Span) 6188.119 Hz
 AcqTime 5.2953 s
 PD 1.0 s
 RGain 72
 Broad. Factor 0.25 Hz
 ExMode ZG30
 IrrNuc OFF
 IrrFreq 0.0 MHz
 IrrSet 0.0 kHz
 IrrFine 0.0 Hz
 IrrPulse 0 μs
 IrrAttn 0
 Spinning 8.0 Hz
 Temperature 23.11 °C
 Printed 2015/Nov/23 16:10:06



File C:\USERS\YOCU\DOCUMENTS\卒
 業生フォルダ\2010卒業生\HAMAMA
 DA\NMR\BRUKER2\BRUKER\DATA\M
 CM\NMR\08182009-PRO\DAK\11\PP
 DATA\11R
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 業生\HAMADA\NMR\BRUKER2\BRUK
 ER\DATA\MCM\NMR\08182009-PRO
 DAK\11\PP\DATA\11R

Date 19/Aug/2009 09:11:35
 Comment:
¹³C
 ObsNuc 13C
 ObsFreq 75.47 MHz
 ObsSet 0.0 kHz
 ObsPine 10001.42 Hz
 Pulse1 10.0 μs
 Pulse2 10.0 μs
 Pulse3 20.0 μs
 P11 2.0 ms
 P12 0.0035 m
 P13 0.0 ms
 Loop1 0
 Point 32768
 Scan 12000
 DummyScan 4
 Frequency (Span) 18028.85 Hz
 AcqTime 1.8175 s
 PD 2.0 s
 RGain 322
 Broad.Factor 0.25 Hz
 ExMode ZGPC30
 IrrNuc OFF
 IrrFreq 0.0 MHz
 IrrSet 0.0 kHz
 IrrPine 0.0 Hz
 IrrPulse 0 μs
 IrrAttn 0
 Spinning 6.0 Hz
 Temperature 24.31 °C
 Printed 2015/Nov/23 16:13:10



File C:\USERS\YOCU\DOCUMENTS\本
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 DA\NM\BRUKER2\BRUKER\DATA\M
 CM\NM\05012009-DEAC\YLKC\10\
 PDATA\1\1R

Original File: C:\USERS\YOCU\DOC
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 業生\HAMADA\NM\BRUKER2\BRUK
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 C\YLKC\10\PDATA\1\1R

Date 01/May/2009 20:21:19

Comment:

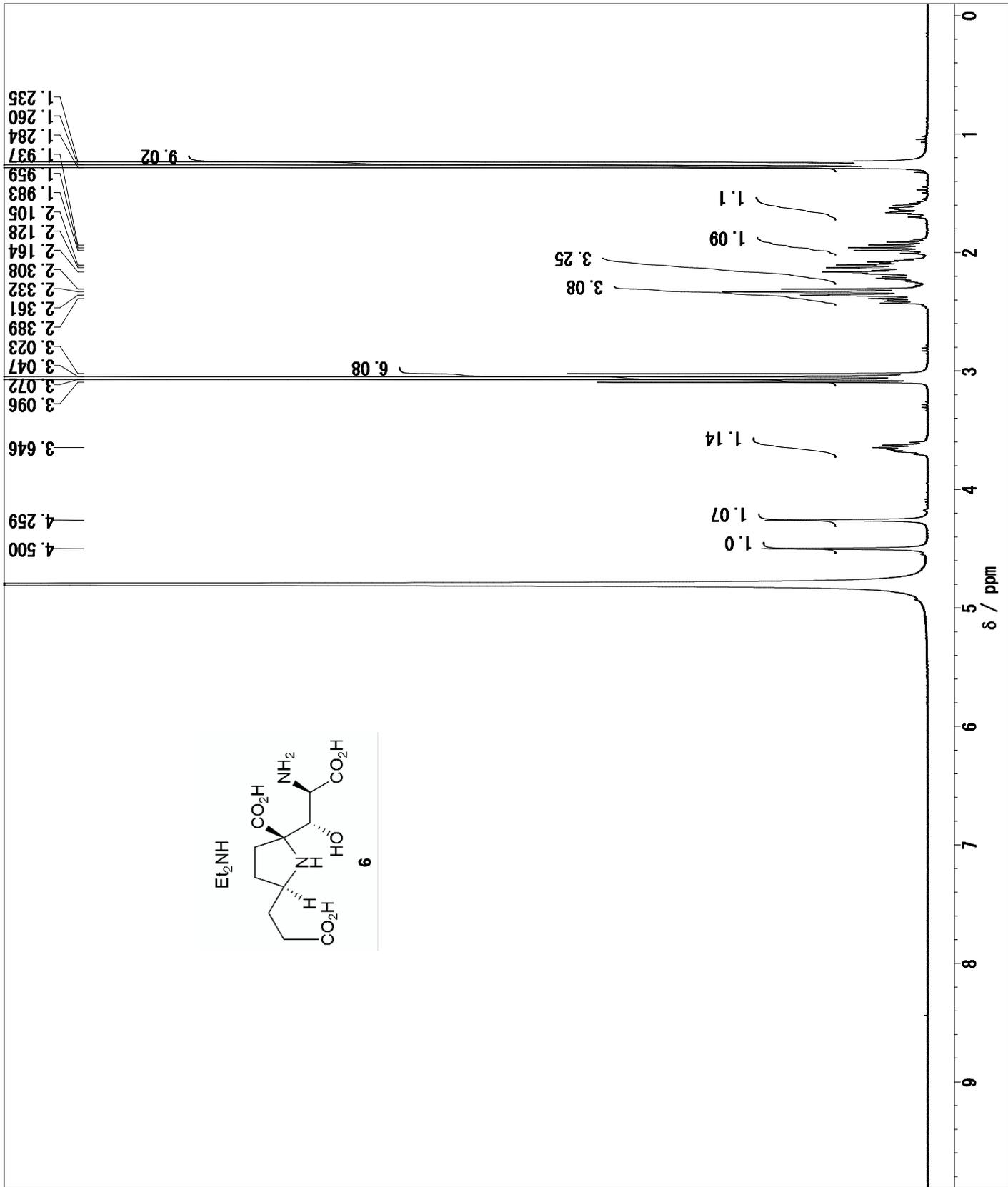
ObsNuc ¹H
 ObsFreq 300.13 MHz
 ObsSet 0.0 kHz
 ObsFine 9996.736 Hz
 Pulse1 15.0 μs
 Pulse2 15.0 μs
 Pulse3 30.0 μs
 P11 1.0 ms
 P12 0.0 ms
 P13 0.0 ms
 Loop1 0
 Point 32768
 Scan 16

DummyScan 2
 Frequency (Span) 6188.119 Hz
 AcqTime 5.2953 s
 PD 1.0 s

RGain 114
 Broad. Factor 0.25 Hz
 ExMode ZG30

IrrNuc OFF
 IrrFreq 0.0 MHz
 IrrSet 0.0 kHz
 IrrFine 0.0 Hz
 IrrPulse 0 μs
 IrrAttn 0

Spinning 20.0 Hz
 Temperature 19.71 °C
 Printed 2015/Nov/21 20:25:42





NAME 05012009--deacy1KC

EXPNO 21

PROCNO 1

Date_ 20090502

Time 9.20

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT D2O

NS 12000

DS 4

SWH 18028.846 Hz

FIDRES 0.275098 Hz

AQ 1.8175818 sec

RG 456

DW 27.733 usec

DE 6.50 usec

TE 294.0 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

==== CHANNEL f1 =====

NUC1 13C

P1 10.00 usec

PL1 -0.80 dB

PL1W 38.05139160 W

SFO1 75.4752953 MHz

==== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 80.00 usec

PL2 2.00 dB

PL12 16.50 dB

PL13 16.50 dB

PL12W 7.25881100 W

PL12W 0.25755233 W

PL13W 0.25755233 W

SFO2 300.1312005 MHz

SI 32768

SF 75.4677326 MHz

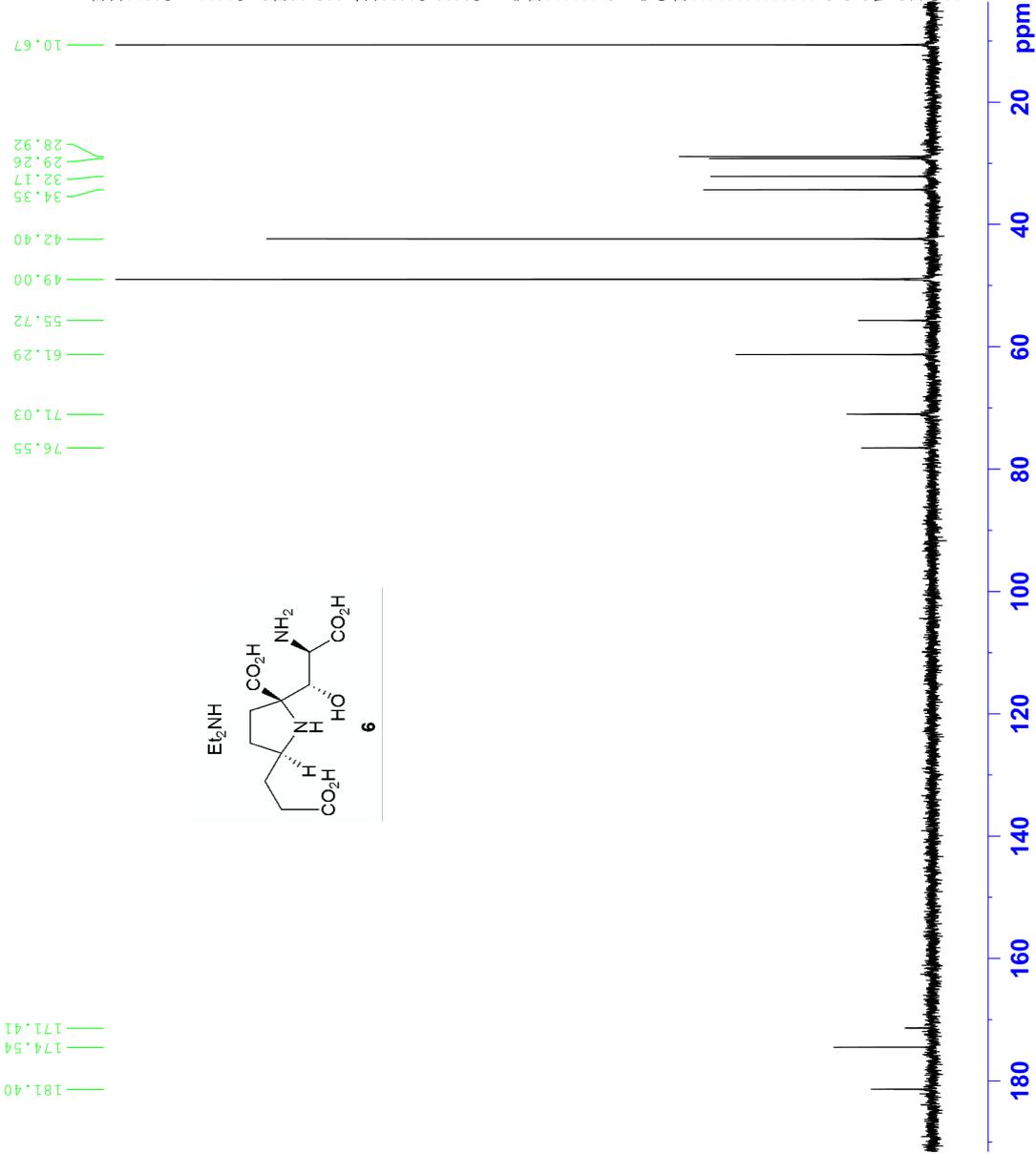
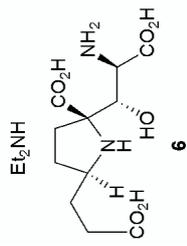
WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40



File F:\vol.1\090715 14.als
 Original File: F:\vol.1\090715
 14.als

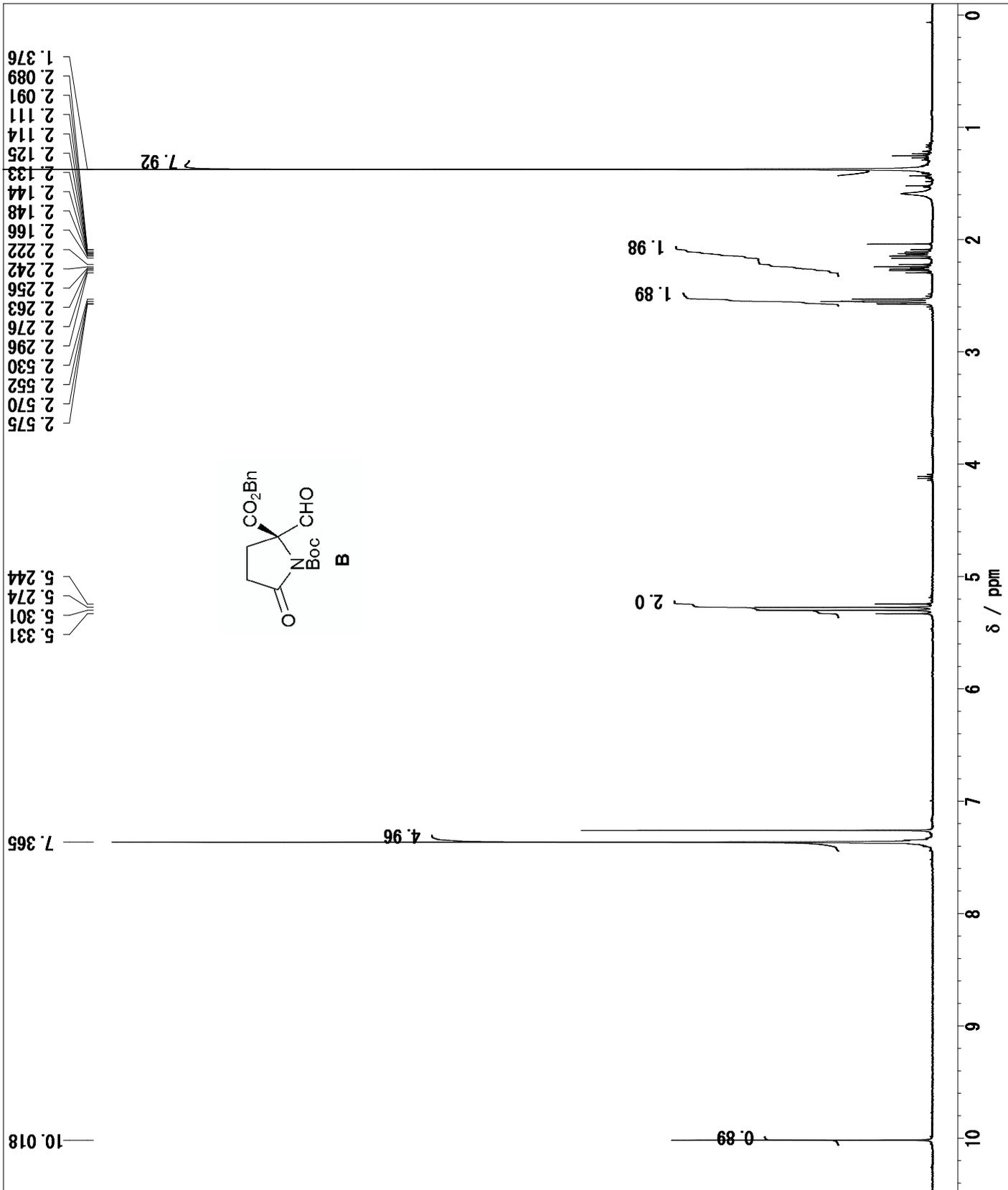
Date 15/Jul/2009 17:07:50

Comment:

ObsNuc ¹H
 ObsFreq 399.65 MHz
 ObsSet 0.0 kHz
 ObsFine 134300.0 Hz
 Pulse1 6.25 μs
 Pulse2 9.0 μs
 Pulse3 10.0 μs
 P11 0.01 ms
 P12 0.25 ms
 P13 1.0 ms
 Loop1 1
 Point 131072(ZeroFi)

11:×4)

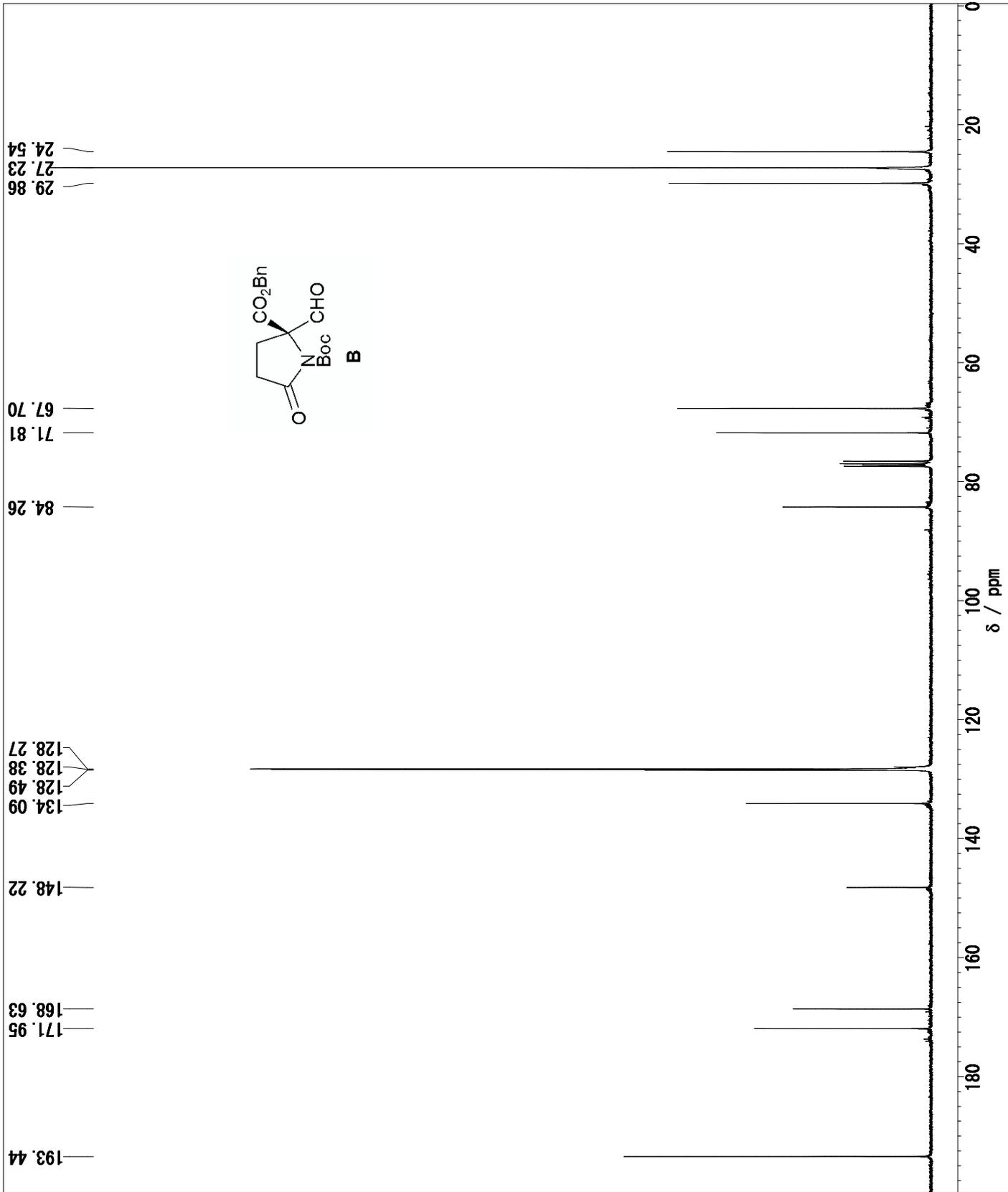
Scan 4
 DummyScan 1
 Frequency(Span) 7993.605 Hz
 AcqTime 4.0993 s
 PD 2.901 s
 RGain 17
 Broad.Factor 0.122 Hz
 ExMode non
 IrrNuc ¹H
 IrrFreq 399.65 MHz
 IrrSet 0.0 kHz
 IrrFine 134500.0 Hz
 IrrPulse 45 μs
 IrrAttn 511
 Spinning 14.0 Hz
 Temperature 26.7 °C
 Printed 2015/Nov/23 17:40:47



File F:\DATA\MCM\MR\PYROGLU-AL
 DEHYDE 13C\20\PDAT\A\1\1R
 Original File: F:\DATA\MCM\MR\PY
 PYROGLU-ALDEHYDE 13C\20\PDAT
 A\1\1R

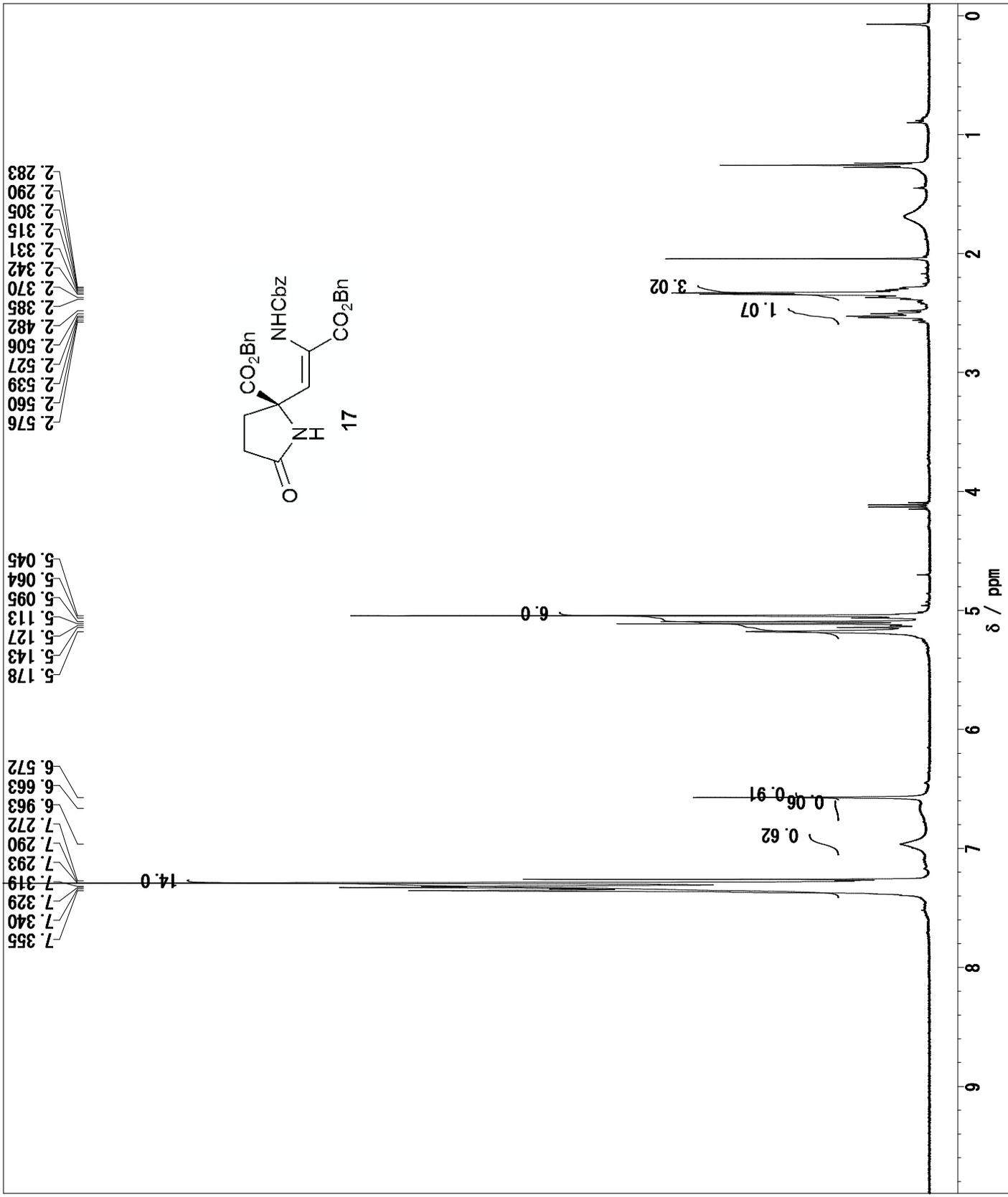
Date 24/Nov/2015 12:37:51
 Comment:

ObsNuc	¹³ C
ObsFreq	75.47 MHz
ObsSet	0.0 kHz
ObsFine	10001.42 Hz
Pulse1	10.0 μs
Pulse2	10.0 μs
Pulse3	20.0 μs
PI1	2.0 ms
PI2	0.0035 m
PI3	0.0 ms
Loop1	0
Point	32768
Scan	800
DummyScan	2
Frequency(Span)	18028.85 Hz
AcqTime	1.8175 s
PD	2.0 s
RGain	203
Broad. Factor	0.25 Hz
ExMode	ZGPC30
IrrNuc	OFF
IrrFreq	0.0 MHz
IrrSet	0.0 kHz
IrrFine	0.0 Hz
IrrPulse	0 μs
IrrAttn	0
Spinning	0.0 Hz
Temperature	22.31 °C
Printed	2015/Nov/24 20:46:23



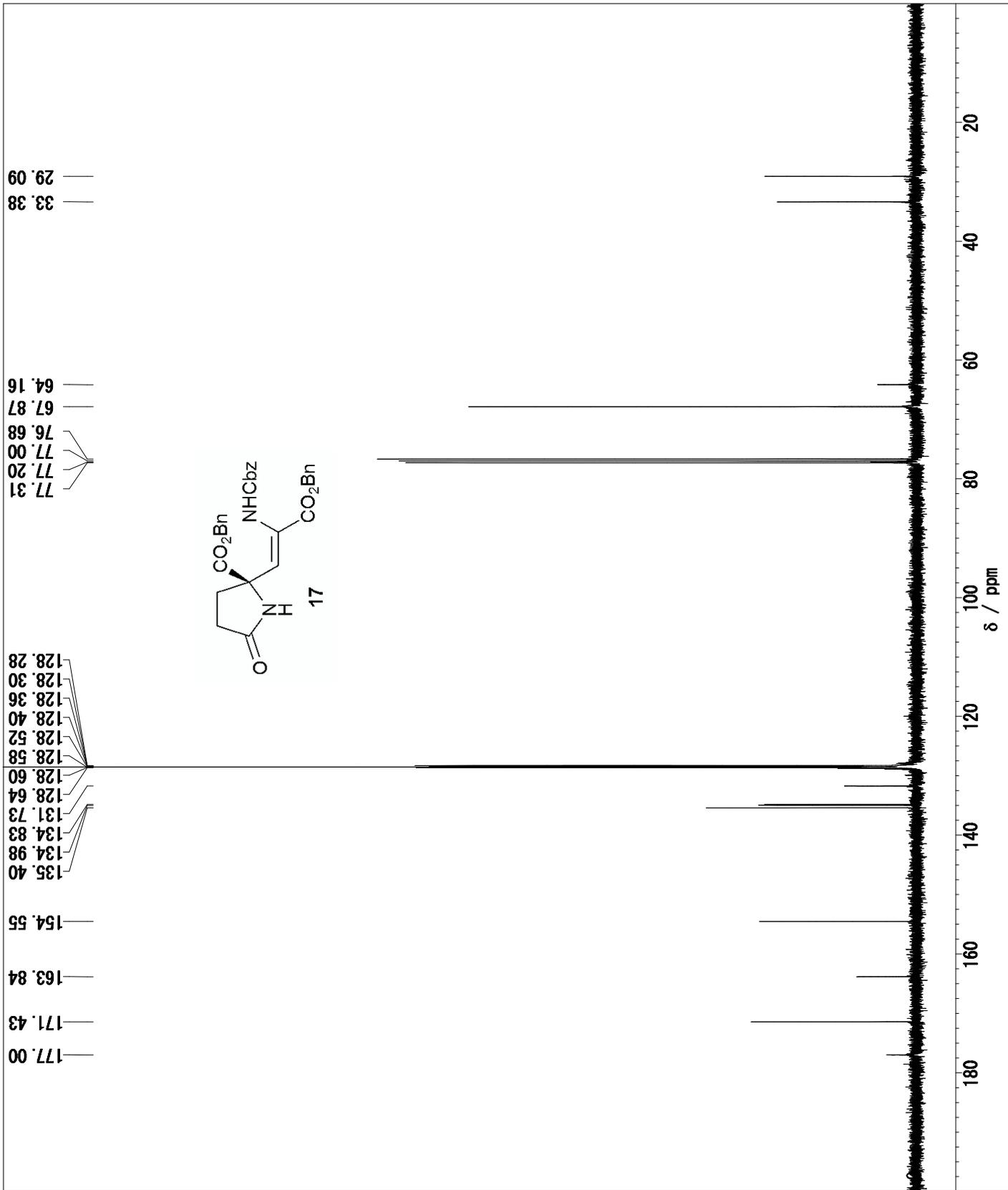
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 Date 10/May/2010 18:43:54

Comment:
 ObsNuc ¹H
 ObsFreq 399.65 MHz
 ObsSet 0.0 kHz
 ObsFine 134300.0 Hz
 Pulse1 6.25 μs
 Pulse2 9.0 μs
 Pulse3 10.0 μs
 P11 0.01 ms
 P12 0.25 ms
 P13 1.0 ms
 Loop1 1
 Point 32768
 Scan 4
 DummyScan 1
 Frequency(Span) 7993.605 Hz
 AcqTime 4.0993 s
 PD 2.901 s
 RGain 17
 Broad.Factor 0.122 Hz
 ExMode non
 IrrNuc ¹H
 IrrFreq 399.65 MHz
 IrrSet 0.0 kHz
 IrrFine 134500.0 Hz
 IrrPulse 45 μs
 IrrAttn 511
 Spinning 11.0 Hz
 Temperature 25.3 °C
 Printed 2015/Nov/26 13:22:58



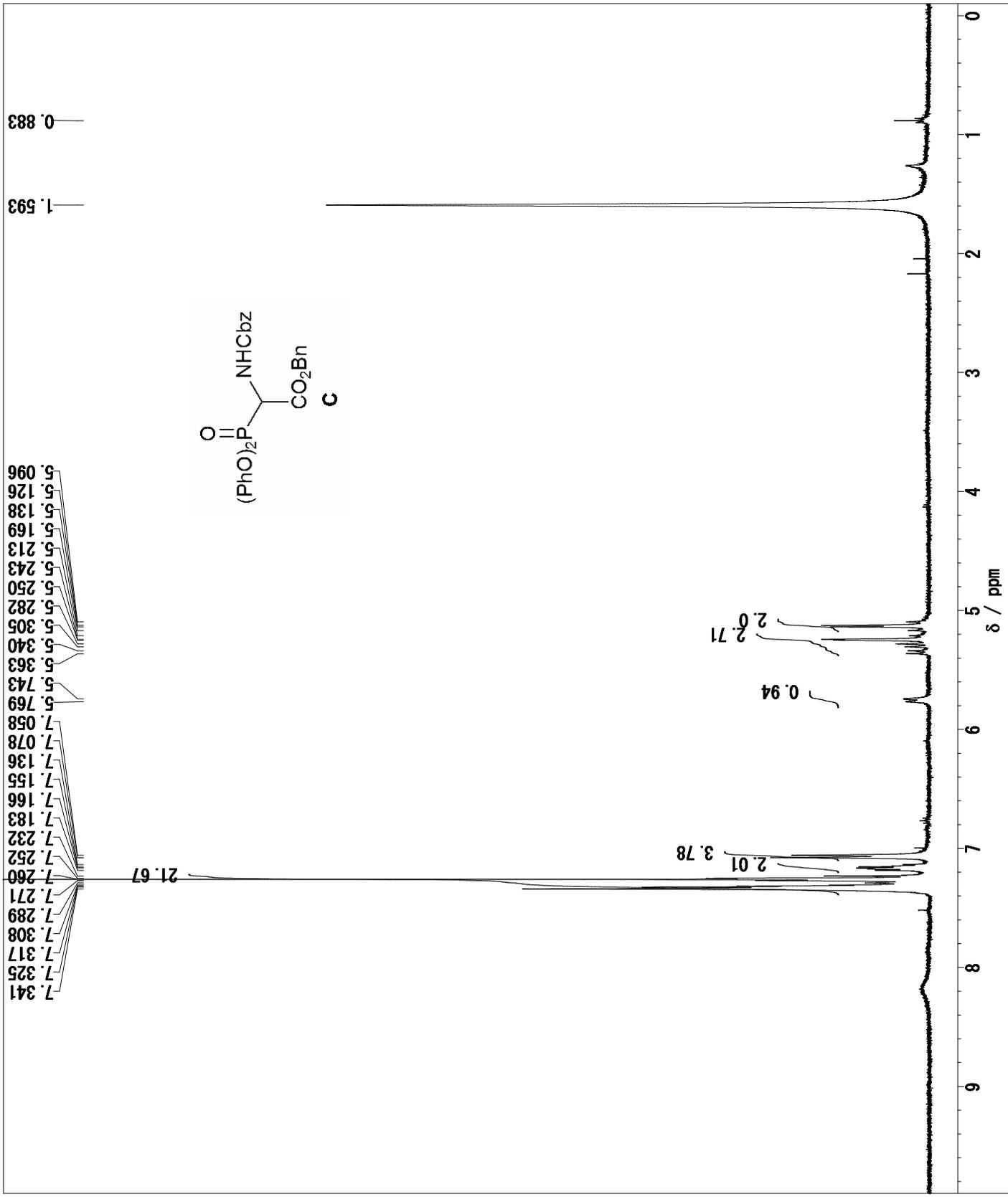
File F:\Date\#(R)-isomer\#13C-NMR
 #100818 Olefine-Bnester.als
 Original File: Date\#(R)-isomer\#
 100818 Olefine-Bnester.als
 Date 19/Aug/2010 20:29:43

Comment: 0
¹³C
 ObsNuc 100.4 MHz
 ObsFreq 0.0 kHz
 ObsSet 135500.0 Hz
 ObsFine
 Pulse1 5.0 μs
 Pulse2 9.0 μs
 Pulse3 10.0 μs
 PI1 0.01 ms
 PI2 0.25 ms
 PI3 1.0 ms
 Loop1 1
 Point 32768
 Scan 1024
 DummyScan 1
 Frequency(Span) 27100.27 Hz
 AcqTime 1.2091 s
 PD 1.791 s
 RGain 26
 Broad.Factor 0.4135 H
 z
 ExMode bcm
 IrrNuc ¹H
 IrrFreq 399.65 MHz
 IrrSet 0.0 kHz
 IrrFine 134500.0 Hz
 IrrPulse 50 μs
 IrrAttn 511
 Spinning 14.0 Hz
 Temperature 27.6 °C
 Printed 2015/Nov/26 13:10:46



File F:\vol.4\1-50\100726 23.a1
 Original File: :%vol.4\1-50\100
 726 23.a1s
 Date 26/Jul/2010 17:21:37

Comment:
 ObsNuc ¹H 399.65 MHz
 ObsFreq 0.0 kHz
 ObsSet 134300.0 Hz
 ObsFine
 Pulse1 6.25 μs
 Pulse2 9.0 μs
 Pulse3 10.0 μs
 P11 0.01 ms
 P12 0.25 ms
 P13 1.0 ms
 Loop1 1
 Point 32768
 Scan 8
 DummyScan 1
 Frequency(Span) 7993.605 Hz
 AcqTime 4.0993 s
 PD 2.901 s
 RGain 24
 Broad.Factor 0.122 Hz
 ExMode non
 IrrNuc ¹H 399.65 MHz
 IrrFreq 0.0 kHz
 IrrFine 134500.0 Hz
 IrrPulse 45 μs
 IrrAttn 511
 Spinning 13.0 Hz
 Temperature 26.3 °C
 Printed 2015/Nov/23 18:41:48

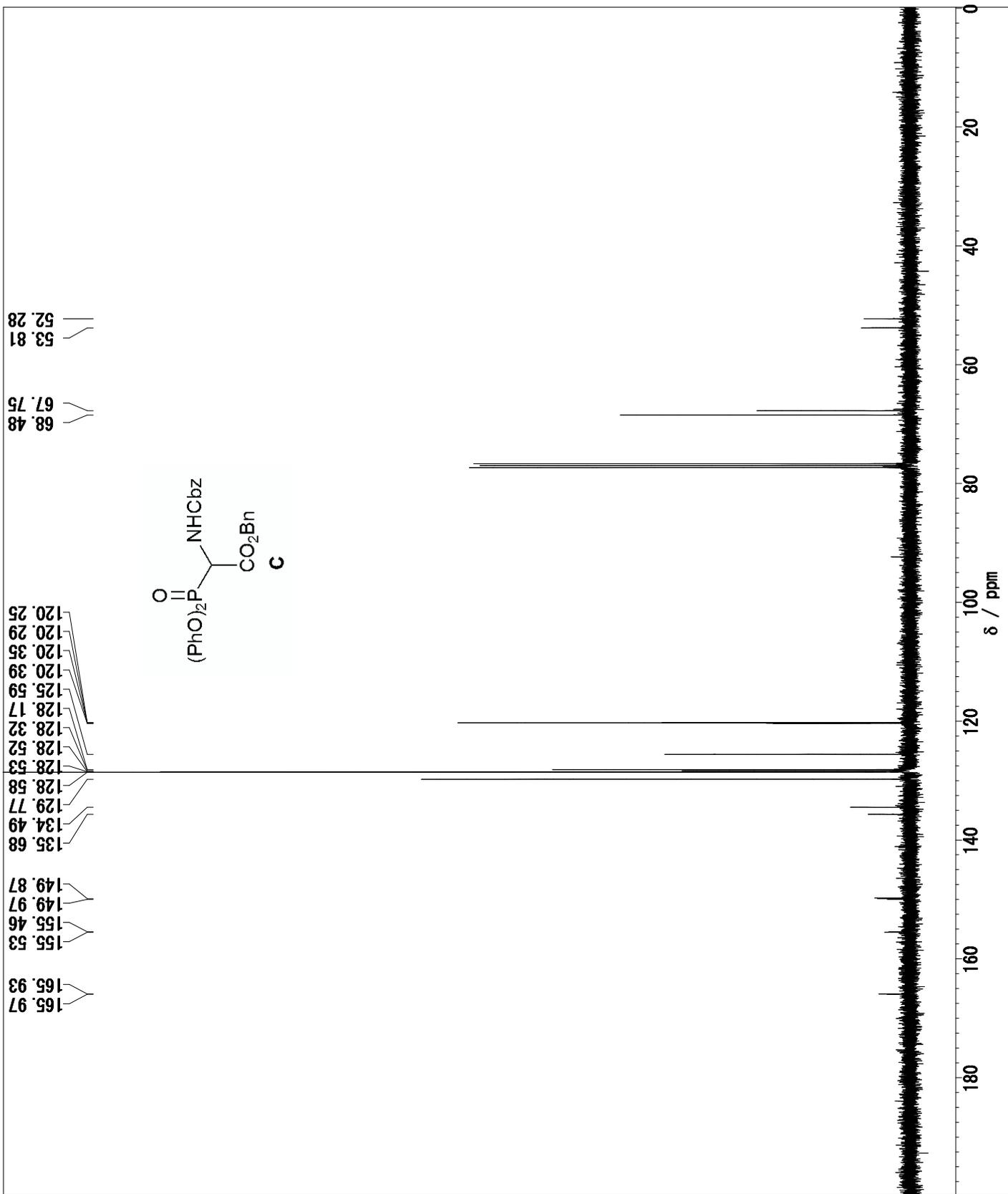


File F:\Date\101008 phosphite-B
 nester-2.als
 Original File: \Date\101008 ph
 ospHITE-Bnester-2.als
 Date 08/Oct/2010 15:59:27

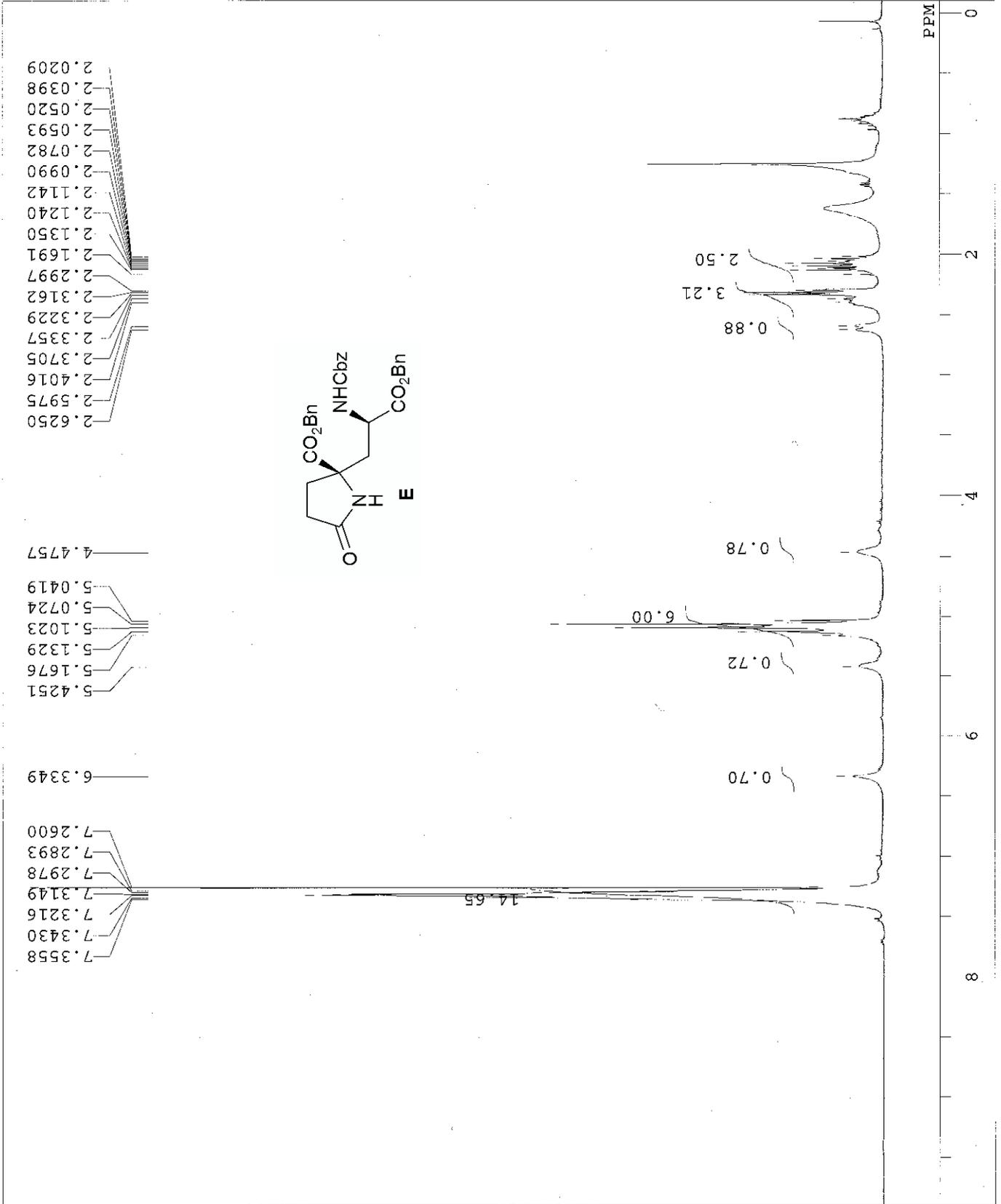
Comment:

¹³C
 ObsNuc 100.4 MHz
 ObsFreq 0.0 kHz
 ObsSet 135500.0 Hz
 ObsFine
 Pulse1 5.0 μs
 Pulse2 9.0 μs
 Pulse3 10.0 μs
 P11 0.01 ms
 P12 0.25 ms
 P13 1.0 ms
 Loop1 1
 Point 32768
 Scan 321
 DummyScan 1
 Frequency(Span) 27100.27 Hz
 AcqTime 1.2091 s
 PD 1.791 s
 RGain 26
 Broad. Factor 0.4135 H

z
 ExMode bcm
 IrrNuc ¹H
 IrrFreq 399.65 MHz
 IrrSet 0.0 kHz
 IrrFine 134500.0 Hz
 IrrPulse 50 μs
 IrrAttn 511
 Spinning 14.0 Hz
 Temperature 27.3 °C
 Printed 2015/Nov/23 19:05:41



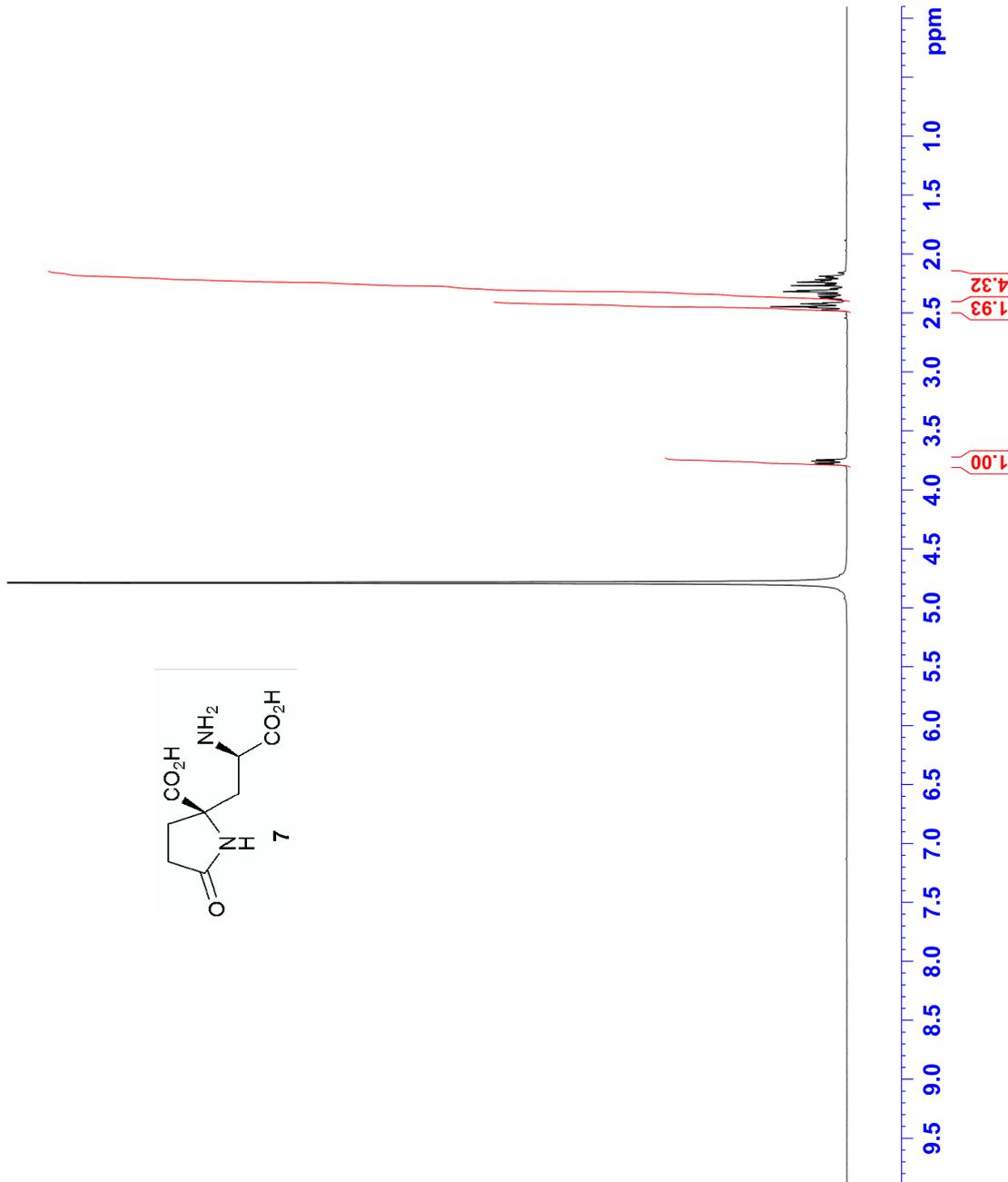
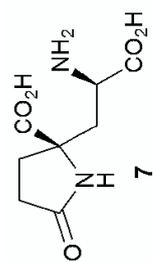
DFILE E:\5_51-100\100828
 COMNT Sat Aug 28 15:51:10
 DATIM 1H
 OBNUC non
 EXMOD 399.65 MHz
 OBFRQ 0.00 KHz
 OBSET 134300.0 Hz
 OBFIN 32768
 POINT 7993.6 Hz
 FREQU 36
 SCANS 4.099 sec
 ACQTM 2.901 sec
 PD 6.2 us
 PW1 1H
 IRNUC 25.7 C
 CTEMP CDCL3
 SLVNT 7.26 ppm
 EXREF 0.12 Hz
 BF 20
 RGAIN





NAME 71
EXPNO 10
PROCNO 1
Date_ 20110210
Time_ 16.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT D2O
NS 64
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 80.6
DW 80.800 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.00 dB
PL1W 7.25881100 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299677 MHz
WDW EM
SSE 0
LB 0.30 Hz
GB 0
PC 1.00



C:\Documents and Settings\henkan\My Documents\NMR\yasuno\Data\ (R)-isomer\13C\100922 2R-aminoacid+MeOH-d.als

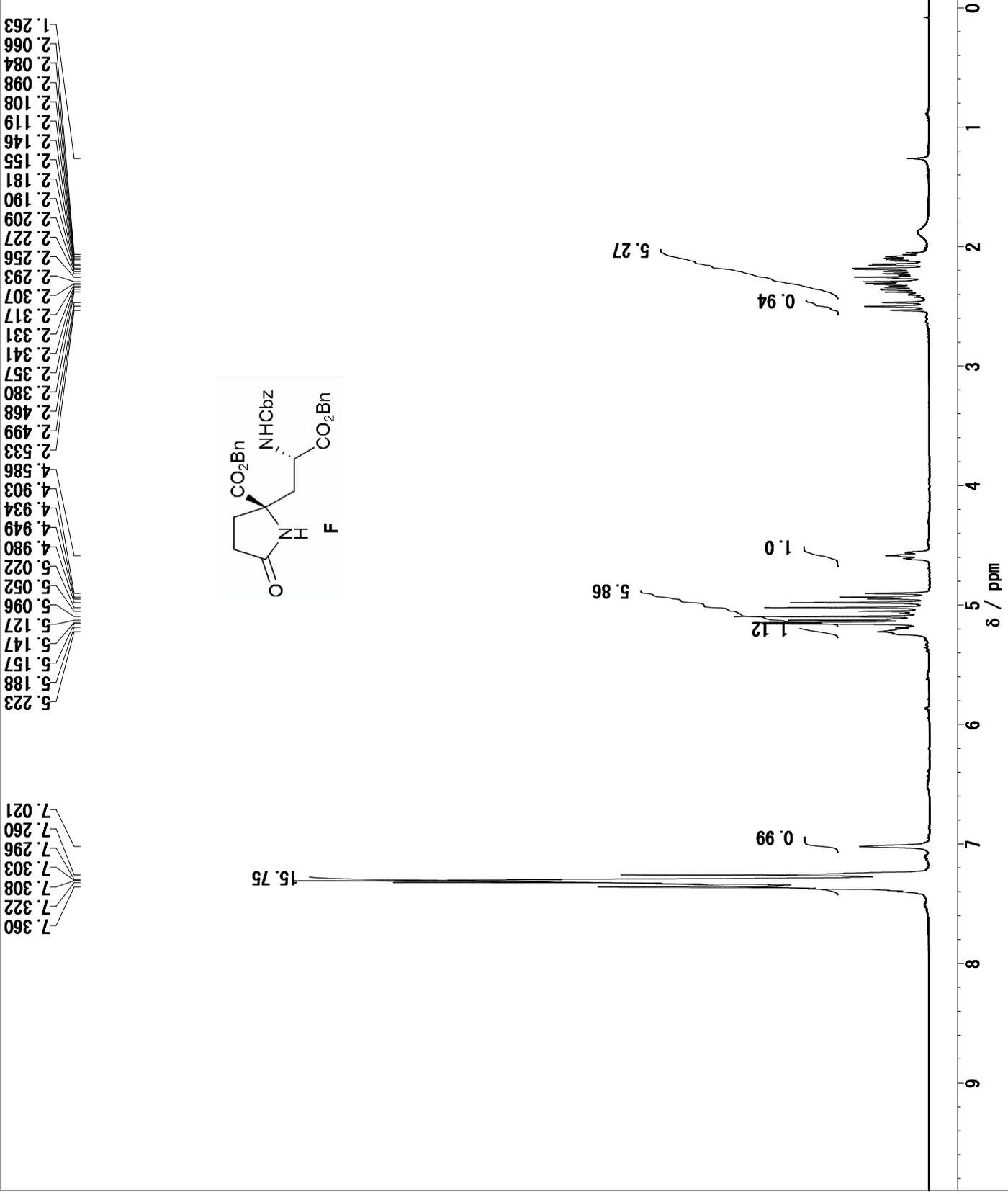
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\Documents and Settir
Wed Sep 22 08:56:16 201
13C
bcm
100.40 MHz
0.00 KHz
135500.0 Hz
32768
27100.3 Hz
14861
1.209 sec
1.791 sec
5.0 us
1H
27.3 c
D2O
49.00 ppm
0.12 Hz
27



File F:\vol.4\51-100\100909 51-
 2sai.als
 Original File: :#vol.4\51-100\1
 00909 51-2sai.als
 Date 09/Sep/2010 19:47:03

Comment:
 ObsNuc ¹H
 ObsFreq 399.65 MHz
 ObsSet 0.0 kHz
 ObsFine 134300.0 Hz
 Pulse1 6.25 μs
 Pulse2 9.0 μs
 Pulse3 10.0 μs
 P11 0.01 ms
 P12 0.25 ms
 P13 1.0 ms
 Loop1 1
 Point 32768
 Scan 32
 DummyScan 1
 Frequency(Span) 7993.605 Hz
 AcqTime 4.0993 s
 PD 2.901 s
 RGain 13
 Broad.Factor 0.122 Hz
 ExMode non
 IrrNuc ¹H
 IrrFreq 399.65 MHz
 IrrSet 0.0 kHz
 IrrFine 134500.0 Hz
 IrrPulse 45 μs
 IrrAttn 511
 Spinning 11.0 Hz
 Temperature 25.9 °C
 Printed 2015/Nov/24 11:11:46





NAME R-S-13C
 EXPNO 10
 PROCNO 1
 Date 20101208
 Time 18.56
 INSTRUM spect
 PROBD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 720
 DS 4
 SWH 18028.846 Hz
 FIDRES 0.275098 Hz
 AQ 1.8175818 sec
 RG 256
 DW 27.733 usec
 DE 6.50 usec
 TE 293.9 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -0.80 dB
 PL1W 38.05139160 W
 SFO1 75.4752953 MHz

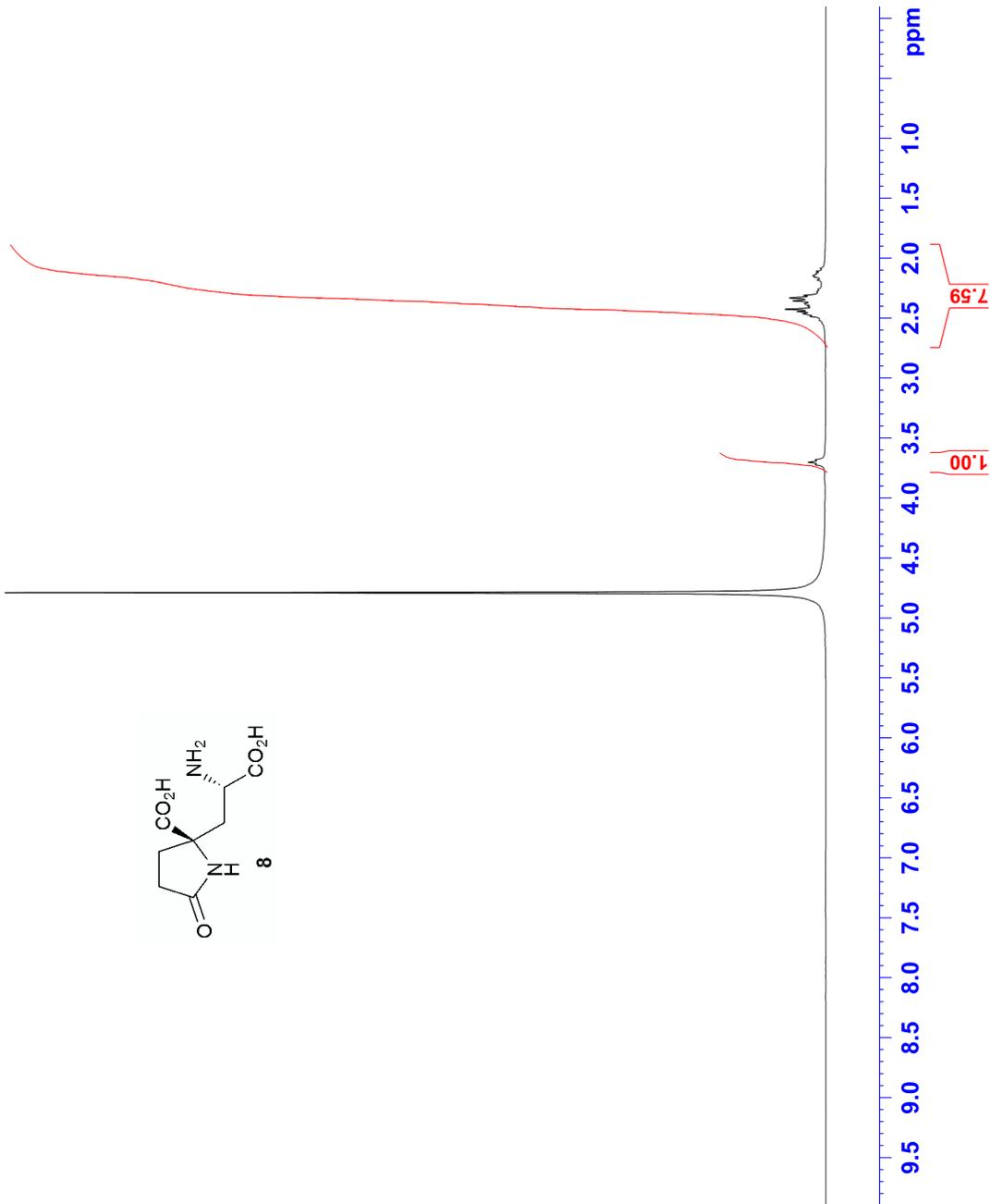
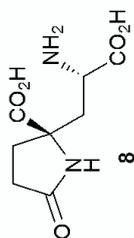
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 16.50 dB
 PL13 16.50 dB
 PL2W 7.25881100 W
 PL12W 0.25755233 W
 PL13W 0.25755233 W
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677415 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

67.98
 67.96
 67.94
 67.92
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NAME R-Samino acid
EXPNO 10
PROCNO 1
Date_ 20110310
Time_ 10.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT D2O
NS 128
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 294.4 K
D1 1.0000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.00 dB
PL1W 7.2588100 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299685 MHz
WDW EM
SSE 0
LB 0.30 Hz
GB 0
PC 1.00





NAME R-S-amino acid
 EXPNO 10
 PROCNO 1
 Date_ 20110305
 Time_ 9.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 11200
 DS 4
 SWH 18028.846 Hz
 FIDRES 0.275098 Hz
 AQ 1.8175818 sec
 RG 203
 DW 27.733 usec
 DE 6.50 usec
 TE 294.4 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -0.80 dB
 PL1W 38.05139160 W
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 16.50 dB
 PL13 16.50 dB
 PL12W 7.2588100 W
 PL12W 0.25755233 W
 PL13W 0.25755233 W
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4676686 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

