Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

69451 Weinheim, Germany

Catalytic Enantioselective and Diastereoselective Addition of Aldehyde-Derived Enecarbamates to α-Keto Aldehydes

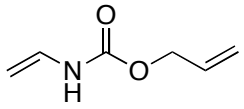
Ryosuku Matsubara, Nobuyuki Kawai, and Shū Kobayashi*

Graduate School of Pharmaceutical Sciences, The University of Tokyo, The HFRE Division, ERATO, Japan Science Technology Agency, JST, Hongo, Bunkyo-ku, Tokyo 113-0033, Japan.

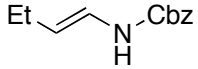
Experimental details and physical data of the products.

General. Melting points are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM-LA300, JNM-LA400, JNM-DE400, JNM-DE600 or JNM-LA500 spectrometer in $CDCl_3$ unless otherwise noted. Tetramethylsilane (TMS) served as internal standard ($\delta = 0$) for ^{1}H NMR, and $CDCl_3$ was used as internal standard ($\delta = 77.0$) for ^{13}C NMR. IR spectra were measured with a JASCO FT/IR-610 spectrometer. Optical rotations were measured with a JASCO P-1010 polarimeter. High-performance liquid chromatography was carried out using following apparatuses; SHIMADZU LC-10AT (liquid chromatograph), SHIMADZU SPD-10A (UV detector), and SHIMADZU C-R6A Chromatopac. Mass spectrometry analysis was carried out using following apparatuses; JEOL The MStation, JMS-700. Melting points of crystalline materials are uncorrected and were determined on YAZAWA BY-1 apparatus. Column chromatography was conducted on Silica gel 60 (Merck) and preparative thin-layer chromatography was carried out using Wakogel B-5F. All reactions were carried out under an argon atmosphere in dried glassware. All solvents were dried and distilled using standard procedures. Ethyl glyoxylate was freshly

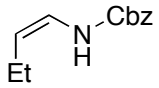
distilled before use according to the previously reported method.[1] Enecarbamates **2a**-**b**[2] and **2c**-**f**[3] were prepared in accordance with literature methods. Enecarbamate **2g** was synthesized by methylation of enecarbamate **2a** using NaH and MeI in THF. As enecarbamates **2b**, **2d**-**f** are new compounds, they were fully characterized in this report.



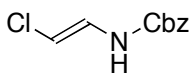
Allyl vinylcarbamate (**2b**): ^{1}H NMR (C_6D_6) δ = 4.09 (d, 1H, *J* = 8.7 Hz), 4.24 (d, 1H, *J* = 15. 6 Hz), 4.43 (d, 1H, *J* = 5.5 Hz), 4.96 (d, 1H, *J* = 10.5 Hz), 5.08 (d, 1H, *J* = 17.4 Hz), 5.62-5.78 (m, 1H), 6.35-6.70 (brs, 1H), 6.78-6.94 (m, 1H); ^{13}C NMR (C_6D_6) δ = 65.8, 93.0, 117.6, 130.4, 132.9, 153.6; IR (neat) 3316, 3170, 3085, 3026, 2954, 1704, 1650, 1517, 1399, 1328, 1254, 1091, 976, 930, 847, 773, 670 cm^{-1}; Anal. Calcd for $C_6H_9NO_2$: C, 56.68; H, 7.13; N, 11.02. Found: C, 56.78; H, 7.10; N, 11.10.



Benzyl (*E*)-but-1-enylcarbamate ((*E*)-2d): Mp. 34.5-35.0 °C; ^{1}H NMR (DMSO-d6) δ = 0.91 (t, 3H, *J* = 7.6 Hz), 1.94 (dq, 2H, *J* = 6.9, 7.3 Hz), 5.04-5.12 (m, 3H), 6.33 (dd, 1H, *J* = 10.1, 14.2 Hz), 7.28-7.38 (m, 5H), 9.32 (d, 1H, *J* = 10.1 Hz); ^{13}C NMR (DMSO-d6) δ = 14.5, 22.5, 65.7, 117.1, 123.6, 127.9, 127.9, 128.4, 136.7, 153.6; IR (neat) 3309, 3034, 2962, 2876, 1706, 1679, 1524, 1455, 1402, 1330, 1290, 1228, 1136, 1042, 946, 734, 696 cm^{-1}; HRMS (FAB); Exact mass calcd for $C_{12}H_{16}NO_2$ [M+H]$^+$, 206.1181. Found 206.1189.



Benzyl (*Z*)-but-1-enylcarbamate ((*Z*)-2d): ^{1}H NMR (DMSO-d6) δ = 0.87 (t, 3H, *J* = 7.3 H z), 2.03 (dq, 2H, *J* = 7.4, 7.4 Hz), 4.50 (q, 1H, *J* = 8.1 Hz), 5.10 (s, 2H), 6.20-6.27 (m, 1H), 7.30-7.40 (m, 5H), 9.08 (d, 1H, *J* = 10.5); ^{13}C NMR (DMSO-d6) δ = 14.1, 18.4, 65.8, 111.3, 122.1, 128.0, 128.0, 128.4, 136.6, 154.2; IR (neat) 3424, 3326, 3033, 2966, 2876, 1711, 1672, 1508, 1454, 1399, 1329, 1228, 1105, 1024, 741, 696, 535 cm^{-1}; HRMS (FAB); Exact mass calcd for $C_{12}H_{16}NO_2$ [M+H]$^+$, 206.1181. Found 206.1174.

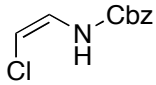


Benzyl (*E*)-2-chlorovinylcarbamate ((*E*)-2e): Mp. 110.0-111.0 °C; ^{1}H NMR (DMSO-d6) δ = 5.10 (s, 2H), 5.89 (d, 1H, *J* = 12.4 Hz), 6.75 (t, 1H, *J* = 10.8 Hz), 7.30-7.40 (m, 5H), 9.81 (d, 1H, *J* = 7.3 Hz); ^{13}C NMR (DMSO-d6) δ = 66.3, 100.8, 128.0, 12 8.1, 128.4, 136.2, 153.3; IR (neat) 3270, 3156, 3073, 3013, 2967, 1726, 1697, 1523, 1453, 1367, 1288, 1229, 930, 838, 768, 696, 556,

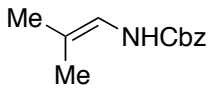
---

[1] Matsubara, R.; Vital, P.; Nakamura, Y.; Kiyohara, H.; Kobayashi, S. *Tetrahedron* **2004**, *60*, 9769.
[2] Kamatani, A.; Overman, L. E. *J. Org. Chem.* **1999**, *64*, 8743.

479 cm^{-1}; A single crystal suitable for X-ray diffraction analysis was obtained from CH_2Cl_2/hexane solvent system. CCDC-251938 contains the supplementary crystallographic data for this compound. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).

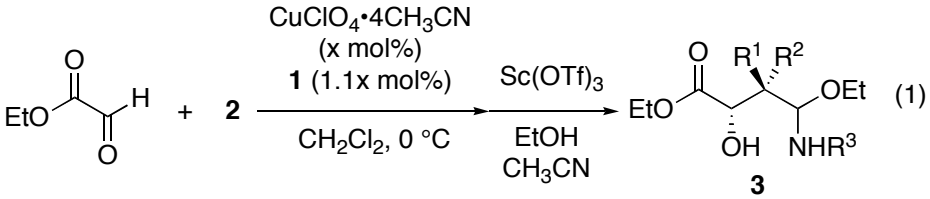


Benzyl (*Z*)-2-chlorovinylcarbamate ((*Z*)-2e): Mp. 25.0-26.0 °C; ^{1}H NMR (DMSO-d6) δ =5.15 (s, 2H), 5.54 (d, 1H, *J* = 6.0 Hz), 6.84 (dd, 1H, *J* = 6.0, 10.6 Hz), 7.30-7.45 (m, 5H), 9.46 (s, 1H); ^{13}C NMR (DMSO-d6) δ = 66.5, 97.7, 125.3, 127.9, 128.1, 128.4, 136.2, 153.9; IR (neat) 3422, 3312, 3085, 3039, 2960, 1729, 1666, 1478, 1321, 1213, 1132, 1047, 968, 909, 780, 696, 541 cm^{-1}; Anal. Calcd for $C_{10}H_{10}ClNO_2$: C, 56.75; H, 4.76; N, 6.62. Found: C, 56.65; H, 4.85; N, 6.37.



Benzyl 2-methylprop-1-enylcarbamate (2f): Mp. 38.5-39.0 °C; ^{1}H NMR (DMSO-d6) δ = 1.5 6 (s, 3H), 1.60 (s, 3H), 5.06 (s, 2H), 6.05-6.13 (m, 1H), 7.28 -7.40 (m, 5H), 8.80 (d, 1H, *J* = 10.1 Hz); ^{13}C NMR (DMSO-d6) δ = 16.6, 22.4, 65.7, 112.9, 118.6, 127.9, 128.0, 128.4, 136.8, 154.1; IR (neat) 3326, 3033, 2961, 2920, 1702, 1509, 1450, 1405, 1333, 1233, 1052, 837, 743, 698, 499 cm^{-1}; HRMS (FAB); Exact mass calcd for $C_{12}H_{16}NO_2$ $[M+H]^+$, 206.1181. Found 206.1178.

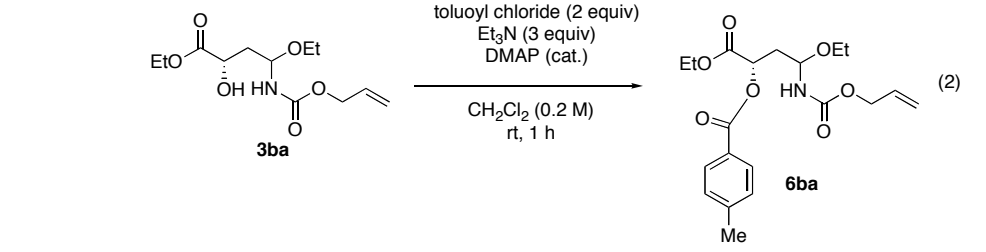
Typical experimental procedure for addition reactions of enecarbamates to aldehydes using a chiral copper catalyst prepared from $CuClO_4$•$4CH_3CN$ and chiral diimine ligand 1.

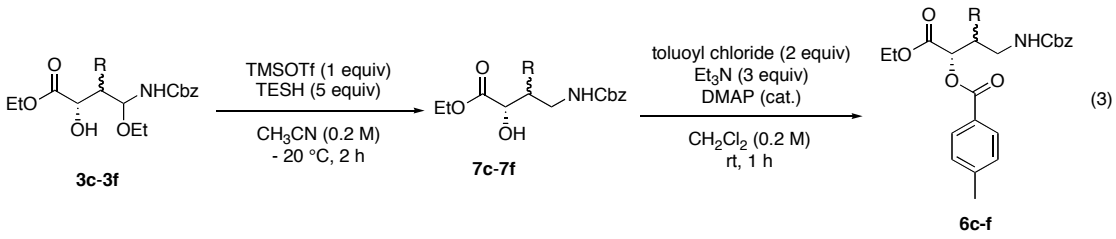


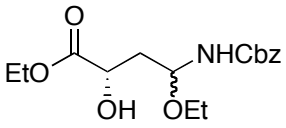
Ligand **1** (9.9 mg, 0.022 mmol) in CH_2Cl_2 (1.5 mL) was added to the $CuClO_4$•$4CH_3CN$ (6.5 mg, 0.020 mmol) flask under argon. The resultant yellow solution was stirred for 13 h, and took 0.15 mL of the solution (0.002 mmol of the catalyst) via gas-tight syringe to another dried flask. CH_2Cl_2 (1.35 mL) was added to

[3] For (*E*)-enecarbamate, see ref. 2. For (*Z*)-enecarbamate; Mecozzi, T.; Petrini, M. *Synlett* **2000**, 73.

that flask and cooled to 0 °C. Freshly distilled ethyl glyoxylate (40 μl, 0.40 mmol) in CH_2Cl_2 (0.8 mL) was added to the mixture, and then enecarbamate **2** (0.20 mmol) in CH_2Cl_2 (0.8 mL) was added in one portion. The reaction mixture was stirred at 0 °C, and was quenched by addition of saturated aqueous $NaHCO_3$. The reaction mixture was allowed to warm to rt, and was extracted with CH_2Cl_2. The organic layer was subsequently washed with brine and dried over anhydrous $MgSO_4$. After removal of solvent *in vacuo*, the residue was dissolved in CH_3CN (4 mL) and EtOH (2 mL). The solution was cooled to –10°C, $Sc(OTf)_3$ (9.8 mg, 0.02 mmol) was added and the mixture was stirred for 12 h. After this time, the reaction was quenched by addition of saturated aqueous $NaHCO_3$, extracted with ether, and the organic layer was washed with brine and dried over anhydrous Na_2SO_4. Evaporation of solvent gave the crude product, which was purified by chromatography on silica gel to afford the desired compound **3**. The enentioselectivities of **3aa**, **3ab**, **3bb** were determined by HPLC analysis. The enantioselectivity of **3ba** was determined after **3ba** was converted to the corresponding toloylated compound **6ba** (equation (2)). The diastereoselectivities of **3c**-**e** were determined after reduction of N,O-acetals. The enantioselectivities of **3c**-**f** were determined after conversion to toluoylated **6c**-**f** (equation (3)). Compound **3e** was reduced to **7e** directly without purification.

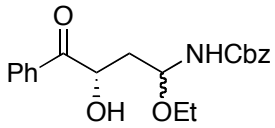




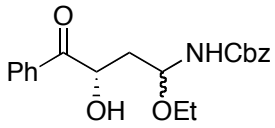


Benzyl (3*S*)-3-(ethoxycarbonyl)-1-ethoxy-3-hydroxypropylcarbamate (3aa, ***syn*/*anti*** **= ca. 1/1 mixture):** ^{1}H NMR ($CDCl_3$) δ = 1.16 (t, 3H, *J* = 6.9 Hz), 1.16 (t, 3H, *J* = 6.9 Hz), 1.26-1.31 (m, 6H), 1.90-2.22 (m, 4H), 3.26 (d, 1H, *J* = 4.1 Hz), 3.32 (d, 1H, *J* = 5.5 Hz), 3.46-3.54 (m, 2H), 3.63-3.73 (m, 2H), 4.16-4.26 (m, 4H), 4.32 (dt, 1H, *J* = 2.6, 5.3 Hz), 4.44 (m, 1H), 5.11 (m, 4H), 5.20-5.32 (m, 2H), 5.59 (d, 1H, *J* = 9.6 Hz), 5.83 (d, 1H, *J* = 9.6 Hz), 7.28-7.38 (m, 10H); ^{13}C NMR

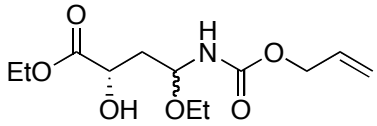
($CDCl_3$) δ = 14.1, 14.9, 15.0, 38.6, 61.7, 61.8, 63.5, 63.6, 66.8, 66.8, 67.2, 67.8, 78.9, 79.2, 128.1, 128.1, 128.1, 128.2, 128.5, 136.2, 136.2, 155.7, 155.9, 174.4, 174.5; HRMS (FAB); Exact mass calcd for $C_{14}H_{18}NO_5$ $[M\text{-}OEt]^+$, 280.1185. Found 280.1174.; HPLC, Daicel Chiralcel ASH, hexane/^{i}PrOH = 4/1, flow rate = 0.4 mL/min : t_R = 19.1 min (3*S*), t_R = 21.2 min (3*S*), t_R = 24.9 min (3*R*), t_R = 28.4 min (3*R*). (The peaks at 19.1 and 28.4 min are derived from the products which have the same relative configuration. The peaks at 21.2 and 24.9 min are derived from the products which have the same relative configuration.)



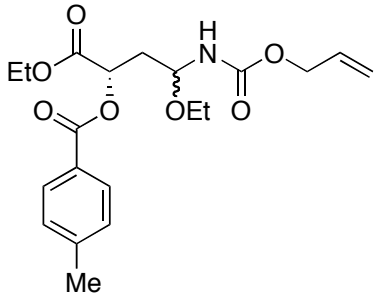
Benzyl (3*S*)-1-ethoxy-3-hydroxy-4-oxo-4-phenylbutylcarbamate (3ab-up): $[\alpha]^{23}_D$ -29.7 (88% ee, c 0.69, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ = 1.21 (t, 3H, *J* = 6.9 Hz), 1.76 (ddd, 1H, *J* = 4.1, 9.6, 13.8 Hz), 2.28 (ddd, 1H, *J* = 2.7, 4.8, 13.8 Hz), 3.55 (dq, 1H, *J* = 6.9, 8.9 Hz), 3.73 (dq, 1H, *J* = 6.9, 9.6 Hz), 4.00 (d, 1H, *J* = 5.5 Hz), 5.10 (s, 2H), 5.29 (dt, 1H, *J* = 4.1, 8.2 Hz), 5.45-5.53 (m, 1H), 6.29 (d, 1H, *J* = 9.6 Hz), 7.28 - 7.42 (m, 5H), 7.50 (t, 2H, *J* = 7.6 Hz), 7.61 (t, 1H, *J* = 7.2 Hz), 7.92 (d, 2H, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ = 15.1, 40.1, 63.5, 66.8, 69.8, 79.0, 128.1, 128.1, 128.5, 128.7, 128.9, 133.1, 134.0, 136.3, 156.1, 201.1; IR (neat) 3331, 2973, 2935, 1715, 1516, 1448, 1405, 1237, 1086, 983, 748, 697 cm^{-1}; HRMS (FAB); Exact mass calcd for $C_{18}H_{18}NO_4$ $[M\text{-}OEt]^+$, 312.1236. Found 312.1224.; HPLC, Daicel Chiralcel ADH, hexane/^{i}PrOH = 4/1, flow rate = 1.0 mL/min : t_R = 13.4 min (3R), t_R = 14.5 min (3*S*). Rf value 0.33 (benzene/AcOEt = 5/1).



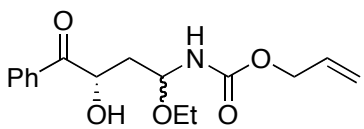
Benzyl (3*S*)-1-ethoxy-3-hydroxy-4-oxo-4-phenylbutylcarbamate (3ab-down): $[\alpha]^{23}_D$ -41.8 (88% ee, c 0.61, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ = 1.18 (t, 3H, *J* = 6.9 Hz), 1.84-1.92 (m, 1H), 2.16-2.27 (m, 1H), 3.54 (dq, 1H, *J* = 6.9, 8.9 Hz), 3.67-3.74 (m, 1H), 3.92 (brs, 1H), 5.08 (d, 1H, *J* = 12.4 Hz), 5.12 (d, 1H, *J* = 12.4 Hz), 5.22 (d, 1H, *J* = 7.6 Hz), 5.27-5.35 (m, 2H), 7.30-7.40 (m, 5H), 7.48 (t, 2H, *J* = 7.9 Hz), 7.60 (t, 1H, *J* = 7.6 Hz), 7.89 (d, 2H, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ = 15.0, 41.2, 63.9, 66.8, 70.7, 79.5, 128.1, 128.2, 128.5, 128.6, 128.9, 133.4, 133.9, 136.2, 155.7, 201.1; IR (neat) 3339, 3033, 2974, 2895, 1715, 1518, 1258, 1079, 1026, 994, 746, 696 cm^{-1}; HRMS (FAB); Exact mass calcd for $C_{18}H_{18}NO_4$ $[M\text{-}OEt]^+$, 312.1236. Found 312.1238.; HPLC, Daicel Chiralcel OJH, hexane/^{i}PrOH = 4/1, flow rate = 1.0 mL/min : t_R = 14.1 min (3R), t_R = 17.0 min (3*S*). Rf value 0.25 (benzene/AcOEt = 5/1).



Allyl (3*S*)-3-(ethoxycarbonyl)-1-ethoxy-3-hydroxypropylcarbamate (3ba, *syn*/*anti* = ca. 1/1 mixture): ^{1}H NMR ($CDCl_3$) δ = 1.17 (t, 6H, *J* = 7.1 Hz), 1.30 (t, 3H, *J* = 7.3 Hz), 1.31 (t, 3H, *J* = 7.1 Hz), 1.89-2.27 (m, 4H), 3.25 (brs, 2H), 3.45-3.58 (m, 2H), 3.63-3.75 (m, 2H), 4.20-4.30 (m, 4H), 4.33 (dd, 1H, *J* = 4.6, 6.0 Hz), 4.46 (dd, 1H, *J* = 3.2, 8.2 Hz), 4.57 (d, 4H, *J* = 5.5 Hz), 5.18-5.30 (m, 6H), 5.55 (d, 1H, *J* = 10.5 Hz), 5.80 (d, 1H, *J* = 10.1 Hz), 5.85-5.98 (m, 2H); ^{13}C NMR ($CDCl_3$) δ = 14.1, 15.0, 15.0, 38.7, 61.7, 61.8, 63.5, 63.6, 65.6, 67.3, 67.9, 78.9, 79.2, 117.8, 132.5, 132.6, 155.6, 155.8, 174.4, 174.6; HRMS (FAB); Exact mass calcd for $C_{10}H_{16}NO_5$ $[M\text{-}OEt]^+$, 230.1028. Found 230.1031.

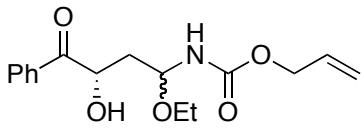


Allyl (3*S*)-3-(ethoxycarbonyl)-1-ethoxy-3-(*p*-toluoyloxy)propylcarbamate (6ba, *syn*/*anti* = ca. 1/1 mixture): ^{1}H NMR ($CDCl_3$) δ = 1.11 (t, 3H, *J* = 6.9 Hz), 1.16 (t, 3H, *J* = 6.9 Hz), 1.26 (t, 3H, *J* = 7.1 Hz), 1.27 (t, 3H, *J* = 7.1 Hz), 2.20-2.48 (m, 10H), 3.40-3.60 (m, 2H), 3.60-3.75 (m, 2H), 4.15-4.27 (m, 4H), 4.45-4.67 (m, 4H), 5.09-5.35 (m, 8H), 5.39 (dd, 1H, *J* = 5.0, 6.8 Hz), 5.44 (t, 1H, *J* = 6.4 Hz), 5.80-6.00 (m, 2H), 7.22-7.27 (m, 4H), 7.95-8.05 (m, 4H); ^{13}C NMR ($CDCl_3$) δ = 14.0, 14.9, 21.7, 37.0, 37.1, 61.4, 61.5, 63.7, 63.8, 65.7, 69.2, 69.4, 78.5, 78.7, 117.9, 126.4, 126.5, 129.1, 129.8, 129.9, 132.4, 144.1, 155.5, 165.7, 165.8, 169.5, 169.8; HRMS (FAB); Exact mass calcd for $C_{18}H_{22}NO_6$ $[M\text{-}OEt]^+$, 348.1447. Found 348.1461.; HPLC, Daicel Chiralcel ADH+ADH, hexane/^{i}PrOH = 9/1, flow rate = 0.65 mL/min : t_R = 37.0 min (3R), t_R = 41.2 min (3*S*). t_R = 45.1 min (3R), t_R = 53.9 min (3*S*). (The peaks at 37.0 and 41.2 min are derived from the products which have the same relative configuration. The peaks at 45.1 and 53.9 min are derived from the products which have the same relative configuration.)

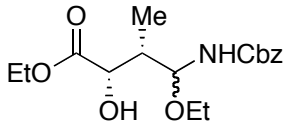


Allyl (3*S*)-1-ethoxy-3-hydroxy-4-oxo-4-phenylbutylcarbamate (3bb-up): $[\alpha]^{23}_D$ -32.5 (91% ee, c 0.65, $CHCl_3$); ^{1}H NMR (C_6D_6) δ = 1.09 (t, 3H, *J* = 6.9 Hz), 1.32-1.39 (m, 1H), 1.90-1.96 (m, 1H), 3.53 (dq, 1H, *J* = 6.9, 9.6 Hz), 3.74 (dq, 1H, *J* = 6.9, 9.6 Hz), 4.00 (d, 1H, *J* = 5.5 Hz), 4.49-4.58 (m, 2H), 4.96-5.00 (m, 1H), 5.13-5.18 (m, 1H), 5.30-5.40 (m, 2H), 5.73 (5.85 (m, 1H), 6.33 (d, 1H, *J* = 9.6 Hz), 6.90-7.00 (m, 2H), 7.00-7.05 (m, 1H), 7.70-7.78 (m, 2H); ^{13}C NMR (C_6D_6) δ =

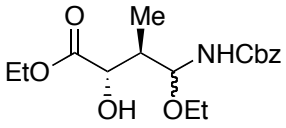
15.2, 40.0, 63.5, 65.5, 70.0, 79.3, 117.3, 128.3, 128.7, 128.9, 133.4, 133.5, 133.7, 156.0, 200.9; HRMS (FAB); Exact mass calcd for $C_{14}H_{16}NO_4$ [M-OEt]$^+$, 262.1079. Found 262.1070. HPLC, Daicel Chiralcel OJH, hexane/^{i}PrOH = 4/1, flow rate = 1.0 mL/min : t_R = 7.7 min (3R), t_R = 9.4 min (3*S*). Rf value 0.33 (benzene/AcOEt = 5/1).



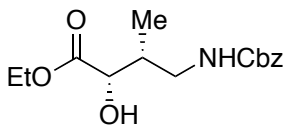
Allyl (3*S*)-1-ethoxy-3-hydroxy-4-oxo-4-phenylbutylcarbamate (3bb-down): $[\alpha]^{23}_D$ -47.2 (91% ee, c 0.48, $CHCl_3$); ^{1}H NMR (C_6D_6) δ = 1.05 (t, 3H, *J* = 7.2 Hz), 1.34-1.44 (m, 1H), 1.74-1.80 (m, 1H), 3.50 (dq, 1H, *J* = 6.9, 9.6 Hz), 3.68 (dq, 1H, *J* = 6.9, 9.6 Hz), 3.90 (d, 1H, *J* = 6.2 Hz), 4.46-4.56 (m, 2H), 4.88 (d, 1H, *J* = 10.3 Hz), 4.99 (d, 1H, *J* = 11.0 Hz), ;5.00-5.05 (m, 1H), 5.12-5.17 (m, 1H), 5.44-5.56 (m, 1H), 5.72-5.82 (m, 1H), 6.96 (t, 3H, *J* = 7.6 Hz), 7.03-7.08 (m, 1H), 7.73 (d, 2H, *J* = 7.6 Hz); ^{13}C NMR (C_6D_6) δ = 15.2, 41.4, 63.8, 65.5, 70.8, 79.6, 117.2, 128.7, 128.8, 133.4, 134.1, 155.5, 201.2; HRMS (FAB); Exact mass calcd for $C_{14}H_{16}NO_4$ [M-OEt]$^+$, 262.1079. Found 262.1072. HPLC, Daicel Chiralcel ADH+ADH, hexane/^{i}PrOH = 4/1, flow rate = 1.0 mL/min : t_R = 19.5 min (3*S*), t_R = 20.1 min (3*R*). Rf value 0.28 (benzene/AcOEt = 5/1).



Benzyl (2*S*,3*S*)-3-(ethoxycarbonyl)-1-ethoxy-3-hydroxy-2-methylpropylcarbamate (3c derived from (*Z*)-2c, 4 isomers mixture): ^{1}H NMR and ^{13} C NMR charts are shown in Figure. HRMS (FAB); Exact mass calcd for $C_{16}H_{22}NO_5$ [M-OEt]$^+$, 294.1341. Found 294.1353.

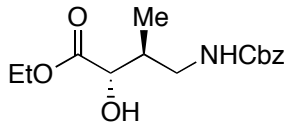


Benzyl (2*R*,3*S*)-3-(ethoxycarbonyl)-1-ethoxy-3-hydroxy-2-methylpropylcarbamate (3c derived from (*E*)-2c, 4 isomers mixture): ^{1}H NMR and ^{13} C NMR charts are shown in Figure. HRMS (FAB); Exact mass calcd for $C_{16}H_{22}NO_5$ [M-OEt]$^+$, 294.1341. Found 294.1342.

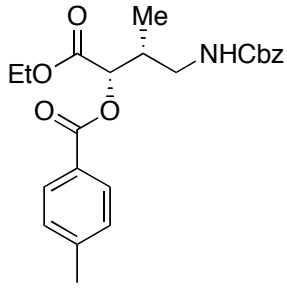


Benzyl (2*R*,3*S*)-3-(ethoxycarbonyl)-3-hydroxy-2-methylpropylcarbamate (*syn*-7c): ^{1}H NMR ($CDCl_3$) δ = 0.81 (d, 3H, *J* = 6.9 Hz), 1.28 (t, 3H, *J* = 7.2 Hz), 2.10-2.30 (m, 1H), 3.15-3.32 (m, 3H), 4.20-4.28 (m, 3H), 5.20 (s, 2H), 5.33-5.41 (m, 1H), 7.26-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 10.7, 14.1, 36.5, 43.8, 61.7, 66.7, 71.1,

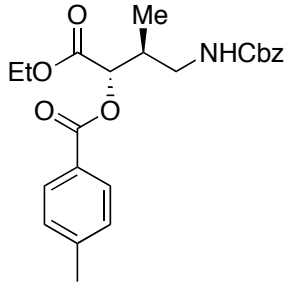
128.0, 128.4, 136.4, 156.8, 174.4; HRMS (FAB); Exact mass calcd for $C_{15}H_{22}NO_5$ $[M\text{-}OEt]^+$, 296.1498. Found 296.1490.



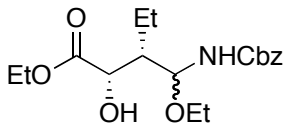
Benzyl (2S,3*S*)-3-(ethoxycarbonyl)-3-hydroxy-2-methylpropylcarbamate (*anti*-7c): ^{1}H NMR ($CDCl_3$) δ = 1.02 (d, 3H, *J* = 7.6 Hz), 1.24 (t, 3H, *J* = 7.2 Hz), 2.20-2.34 (m, 1H), 3.14-3.26 (m, 2H), 3.46 (d, 1H, *J* = 5.5 Hz), 4.05 (t, 1H, *J* = 4.1 Hz), 4.10-4.20 (m, 2H), 5.07 (s, 2H), 5.19 (m, 1H), 7.28-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 14.0, 14.1, 14.9, 37.2, 42.3, 61.6, 66.6, 73.0, 128.0, 128.4, 136.3, 156.6, 174.6; HRMS (FAB); Exact mass calcd for $C_{15}H_{22}NO_5$ $[M+H]^+$, 296.1498. Found 296.1490.



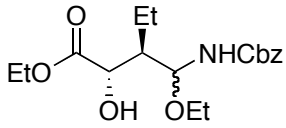
Benzyl (2*R*,3*S*)-3-(ethoxycarbonyl)-2-methyl-3-toluoyloxypropylcarbamate (*syn*-6c): ^{1}H NMR ($CDCl_3$) δ = 1.07 (d, 3H, *J* = 6.9 Hz), 1.27 (t, 3H, *J* = 7.2 Hz), 2.41 (s, 3H), 2.46-2.52 (m, 1H), 3.05-3.13 (m, 1H), 3.36 (dq, 1H, *J* = 6.9, 6.9 Hz), 4.20-4.28 (m, 2H), 5.07 (d, 1H, *J* = 12.4 Hz), 5.11 (d, 1H, *J* = 12.4 Hz), 5.21 (t, 1H, *J* = 5.8 Hz), 5.34 (d, 1H, *J* = 3.4 Hz), 7.22 (d, 1H, *J* = 8.2 Hz), 7.28-7.37 (m, 5H), 7.96 (d, 1H, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ = 12.5, 14.1, 14.1, 21.7, 35.9, 43.5, 61.5, 66.7, 73.1, 126.5, 128.1, 128.5, 129.2, 129.9, 136.4, 144.2, 156.4, 166.1, 169.4; HRMS (FAB); Exact mass calcd for $C_{23}H_{27}NO_6$ $[M+H]^+$, 414.1917.; Found 414.1915. HPLC, Daicel Chiralcel ODH, hexane/^{i}PrOH = 9/1, flow rate = 0.7 mL/min : t_R = 42.6 min (2*S*, 3*R*), t_R = 62.0 min (2*R*, 3*S*).



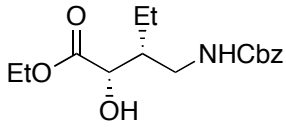
Benzyl (2S,3*S*)-3-(ethoxycarbonyl)-2-methyl-3-toluoyloxypropylcarbamate (*anti*-6c): ^{1}H NMR ($CDCl_3$) δ = 1.10 (d, 3H, *J* = 6.9 Hz), 1.24 (t, 3H, *J* = 7.2 Hz), 2.41 (s, 3H), 2.50-2.58 (m, 1H), 3.36 (t, 3H, *J* = 6.5 Hz), 4.14-4.26 (m, 2H), 5.09 (d, 1H, *J* = 12.4 Hz), 5.11 (d, 1H, *J* = 12.4 Hz), 7.22 (d, 2H, *J* = 8.2 Hz), 7.34-7.38 (m, 5H), 7.95 (d, 2H, *J* = 8.2 Hz); ^{13}C NMR ($CDCl_3$) δ = 14.1, 14.7, 21.7, 35.7, 42.8, 61.5, 66.7, 74.5, 126.5, 128.1, 128.5, 129.2, 129.8, 136.4, 144.2, 156.4, 166.0, 169.7; HRMS (FAB); Exact mass calcd for $C_{23}H_{27}NO_6$ $[M+H]^+$, 414.1917.; Found 414.1915. HPLC, Daicel Chiralcel ODH, hexane/^{i}PrOH = 9/1, flow rate = 0.7 mL/min : t_R = 29.9 min (2*R*, 3*R*), t_R = 32.8 min (2*S*, 3*S*).



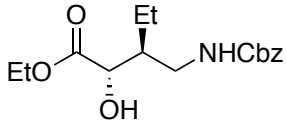
Benzyl (2*S*,3*S*)-3-(ethoxycarbonyl)-1-ethoxy-2-ethyl-3-hydroxypropylcarbamate (3d derived from (*Z*)-2d, a mixture of 4 isomers): ^{1}H NMR and ^{13} C NMR charts are shown in Figure. HRMS (FAB); Exact mass calcd for $C_{16}H_{22}NO_5$ $[M\text{-}OEt]^+$, 308.1498. Found 308.1497.



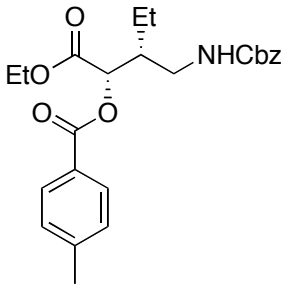
Benzyl (2*R*,3*S*)-3-(ethoxycarbonyl)-1-ethoxy-2-ethyl-3-hydroxypropylcarbamate (3d derived from (*E*)-2d, a mixture of 4 isomers): ^{1}H NMR and ^{13} C NMR charts are shown in Figure. HRMS (FAB); Exact mass calcd for $C_{16}H_{22}NO_5$ $[M\text{-}OEt]^+$, 308.1498. Found 308.1504.



Benzyl (2*R*,3*S*)-3-(ethoxycarbonyl)-2-ethyl-3-hydroxypropylcarbamate (*syn*-7d): ^{1}H NMR ($CDCl_3$) δ = 0.89 (t, 3H, *J* = 7.6 Hz), 1.27 (t, 3H, *J* = 7.2 Hz), 1.93-2.00 (m, 1H), 3.10-3.38 (m, 4H), 4.05-4.28 (m, 4H), 5.17 (s, 2H), 5.32 (brs, 1H), 7.25-7.36 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 11.7, 14.1, 19.0, 41.7, 43.2, 61.7, 66.6, 71.6, 128.0, 128.1, 128.4, 136.4, 156.8, 174.6; HRMS (FAB); Exact mass calcd for $C_{16}H_{24}NO_5$ $[M+H]^+$, 310.1654. Found 310.1669.

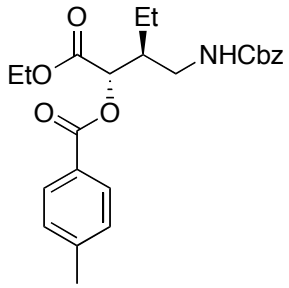


Benzyl (2*S*,3*S*)-3-(ethoxycarbonyl)-2-ethyl-3-hydroxypropylcarbamate (*anti*-7d): ^{1}H NMR ($CDCl_3$) δ = 0.97 (t, 3H, *J* = 7.6 Hz), 1.22 (t, 3H, *J* = 7.2 Hz), 1.30-1.55 (m, 2H), 2.00-2.10 (m, 1H), 3.05-3.35 (m, 4H), 4.08-4.28 (m, 4H), 5.00-5.05 (m, 1H), 5.05 (s, 2H), 7.25-7.35 (m, 5H); ^{13}C NMR ($CDCl_3$) δ =11.5, 14.0, 22.2, 40.4, 43.4, 61.7, 66.6, 71.0, 127.7, 128.0, 128.0, 128.3, 136.3, 156.5, 175.3; HRMS (FAB); Exact mass calcd for $C_{16}H_{24}NO_5$ $[M+H]^+$, 310.1654. Found 310.1669.

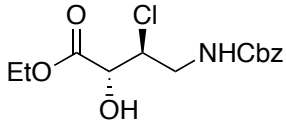


Benzyl (2*R*,3*S*)-3-(ethoxycarbonyl)-2-ethyl-3-toluoyloxypropylcarbamate (*syn*-6d): ^{1}H NMR ($CDCl_3$) δ = 1.04 (t, 3H, *J* = 7.6 Hz), 1.26 (t, 3H, *J* = 7.2 Hz), 1.44-1.62 (m, 2H), 2.23-2.32 (m, 1H), 2.40 (s, 3H), 3.03-3.11 (m, 1H), 3.46-3.54 (m, 1H), 4.20-4.26 (m, 2H), 5.06 (d, 1H, *J* = 12.4 Hz), 5.10 (d, 1H, *J* = 12.4 Hz), 5.24 (brs, 1H), 5.38 (d, 1H, *J* = 2.7 Hz), 7.20 (d, 1H, *J* = 8.2 Hz), 7.26-7.36 (m, 5H), 7.92-7.96 (m, 2H); ^{13}C NMR ($CDCl_3$) δ = 11.7, 14.1, 20.8, 21.6, 41.2, 42.5, 61.5,

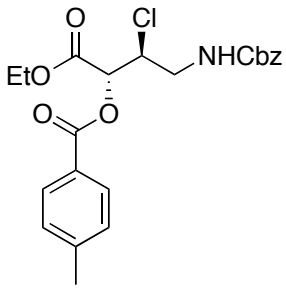
66.7, 73.0, 126.5, 128.0, 128.4, 129.1, 129.8, 136.4, 144.2, 156.4, 166.1, 169.7; HRMS (FAB); Exact mass calcd for $C_{24}H_{30}NO_6$ $[M+H]^+$, 428.2073.; Found 428.2088. HPLC, Daicel Chiralcel ADH, hexane/^{i}PrOH = 19/1, flow rate = 1.0 mL/min : t_R = 57.0 min (2*S*, 3*R*), t_R = 87.4 min (2*R*, 3*S*).



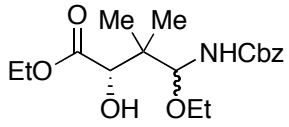
Benzyl (2S,3*S*)-3-(ethoxycarbonyl)-2-ethyl-3-toluoyloxypropylcarbamate (*anti*-6d): ^{1}H NMR ($CDCl_3$) δ = 1.02 (t, 3H, *J* = 7.2 Hz), 1.22 (t, 3H, *J* = 7.2 Hz), 1.40-1.54 (m, 2H), 2.24-2.34 (m, 1H), 2.40 (s, 3H), 3.36-3.42 (m, 2H), 4.10-4.24 (m, 2H), 5.08 (d, 1H, *J* = 12.4 Hz), 5.12 (d, 1H, *J* = 12.4 Hz), 5.33 (d, 1H, *J* = 2.7 Hz), 7.20 (d, 2H, *J* = 7.6 Hz), 7.29-7.36 (m, 5H), 7.94 (d, 2H, *J* = 8.2 Hz); ^{13}C NMR ($CDCl_3$) δ = 11.5, 14.0, 21.6, 22.2, 41.0, 42.3, 61.5, 66.6, 72.5, 126.5, 128.1, 128.4, 129.2, 129.8, 136.4, 144.2, 156.3, 165.8, 170.2; HRMS (FAB); Exact mass calcd for $C_{24}H_{30}NO_6$ $[M+H]^+$, 428.2073.; Found 428.2088. HPLC, Daicel Chiralcel ADH, hexane/^{i}PrOH = 19/1, flow rate = 1.0 mL/min : t_R = 44.1 min (2*R*, 3*R*), t_R = 68.7 min (2*S*, 3*S*).



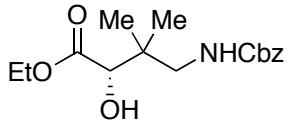
Benzyl (2*S*,3*R*)-3-(ethoxycarbonyl)-2-chloro-3-hydroxypropylcarbamate (*anti*-7e): ^{1}H NMR ($CDCl_3$) δ = 1.30 (t, 3H, *J* = 7.1 Hz), 3.50-3.74 (m, 2H), 3.84 (brs, 1H), 4.25 (q, 2H, *J* = 7.0 Hz), 4.30-4.48 (m, 2H), 5.12 (s, 2H), 5.25 (brs, 1H), 7.30-7.45 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 14.1, 43.1, 60.6, 62.3, 67.3, 72.4, 128.2, 128.3, 128.6, 135.9, 156.8, 170.8; HRMS (FAB); Exact mass calcd for $C_{14}H_{19}NClO_5$ $[M+H]^+$, 316.0952.; Found 316.0963.



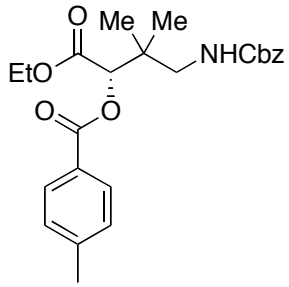
Benzyl (2S,3*S*)-3-(ethoxycarbonyl)-2-chloro-3-toluoyloxypropylcarbamate (*anti*-6e): ^{1}H NMR ($CDCl_3$) δ = 1.26 (t, 3H, *J* = 7.1 Hz), 2.42 (s, 3H), 3.60-3.70 (m, 1H), 3.75-3.85 (m, 1H), 4.23 (q, 2H, *J* = 7.0 Hz), 4.56-4.62 (m, 1H), 5.10 (s, 2H), 5.17-5.27 (m, 1H), 5.55 (d, 1H, *J* = 3.7 Hz), 7.23-7.27 (m, 2H), 7.30-7.37 (m, 5H), 7.97 (d, 2H, *J* = 7.8 Hz); ^{13}C NMR ($CDCl_3$) δ = 14.0, 21.7, 43.8, 57.9, 62.3, 67.1, 73.4, 126.0, 128.2, 128.3, 128.6, 129.3, 130.0, 136.1, 144.6, 156.1, 165.4, 166.8; HPLC, Daicel Chiralcel ADH, hexane/^{i}PrOH = 9/1, flow rate = 1.0 mL/min : t_R = 25.2 min (2*R*, 3*R*), t_R = 32.5 min (2*S*, 3*S*).



Benzyl (3*S*)-3-(ethoxycarbonyl)-1-ethoxy-3-hydroxy-2,2-dimethylpropylcarbamate (3f): ^{1}H NMR ($CDCl_3$) δ = 0.92 (s, 3H), 1.00 (s, 3H), 1.06 (s, 3H), 1.08 (s, 3H), 1.12-1.20 (m, 6H), 1.28-1.34 (m, 6H), 3.31 (d, 1H, *J* = 6.0 Hz), 3.42-3.80 (m, 4H), 3.98 (d, 1H, *J* = 7.3 Hz), 4.14-4.34 (m, 6H), 4.82 (d, 1H, *J* = 10.1 Hz), 4.95 (d, 1H, *J* = 10.1 Hz), 5.11 (s, 4H), 5.51 (d, 1H, *J* = 10.1 Hz), 6.11 (d, 1H, *J* = 10.1 Hz), 7.27-7.40 (m, 10H); ^{13}C NMR ($CDCl_3$) δ = 14.1, 14.9, 14.9, 18.0, 19.9, 21.2, 22.8, 41.8, 42.1, 61.4, 61.7, 63.8, 66.6, 66.8, 74.8, 86.2, 87.5, 127.9, 128.0, 128.0, 128.2 128.5, 128.5, 128.6, 136.2, 136.4, 156.3, 173.7, 174.3; HRMS (FAB); Exact mass calcd for $C_{16}H_{22}NO_5$ [M-OEt]$^+$, 308.1498.; Found 308.1501.

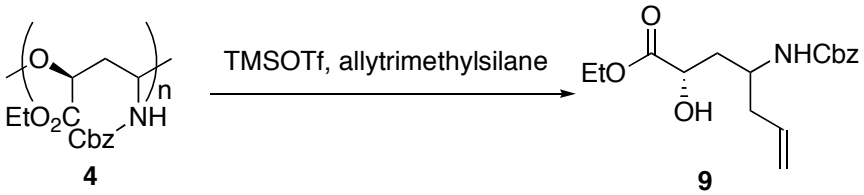


Benzyl (3*S*)-3-(ethoxycarbonyl)-3-hydroxy-2,2-dimethylpropylcarbamate (7f): $[\alpha]^{23}_D$ 8.2 (78% ee, c 0.96, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ = 0.91 (s, 3H), 1.01 (s, 3H), 1.30 (t, 3H, *J* = 7.2 Hz), 3.04 (dd, 1H, *J* = 6.2, 13.8 Hz), 3.33 (dd, 1H, *J* = 7.6, 13.8 Hz), 3.36 (d, 1H, *J* = 6.9 Hz), 3.93 (d, 1H, *J* = 6.2 Hz), 4.20-4.30 (m, 2H), 5.10 (s, 1H), 5.28 (brs, 1H), 7.30-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 14.2, 20.1, 22.5, 39.2, 49.2, 61.6, 66.8, 75.8, 128.1, 128.5, 136.4, 157.1, 173.7; ; IR (neat) 3382, 2971, 1721, 1527, 1461, 1367, 1240, 1143, 1088, 1029, 744, 701 cm^{-1};HRMS (FAB); Exact mass calcd for $C_{16}H_{24}NO_5$ [M+H]$^+$, 310.1654.; Found 310.1645.



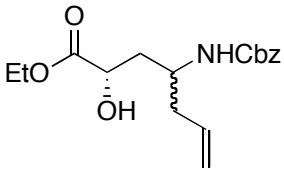
Benzyl (3*S*)-3-(ethoxycarbonyl)-3-toluoyloxy-2,2-dimethylpropylcarbamate (6f): $[\alpha]^{23}_D$ –2.6 (78% ee, c 0.885, $CHCl_3$);^{1}H NMR ($CDCl_3$) δ = 1.13 (t, 3H, *J* = 7.1 Hz), 1.27 (t, 3H, *J* = 7.2 Hz), 2.40 (s, 3H), 3.26 (dd, 1H, *J* = 6.2, 13.8 Hz), 4.19-4.29 (m, 2H), 4.90 (s, 1H), 5.09 (d, 1H, *J* = 12.4 Hz), 5.12 (d, 1H, *J* = 12.4 Hz), 5.28 (t, 1H, *J* = 6.5 Hz), 7.19 (d, 2H, *J* = 8.2 Hz), 7.26-7.39 (m, 5H), 7.94 (d, 1H, *J* = 8.2 Hz); ^{13}C NMR ($CDCl_3$) δ = 14.1, 21.7, 22.2, 22.4, 38.4, 48.4, 61.4, 66.8, 78.0, 126.4, 128.1, 128.5, 129.2, 129.8, 136.5, 144.3, 156.7, 166.1, 169.2; HRMS (FAB); Exact mass calcd for $C_{24}H_{30}NO_6$ [M+H]$^+$, 428.2073.; Found 429.2091.; HPLC, Daicel Chiralcel OD, hexane/^{i}PrOH = 19/1, flow rate = 1.0 mL/min : t_R = 19.4 min (3*R*), t_R = 24.4 min (3*S*).

Procedure of allylation of polymeric compound 4.



4 9 (4)

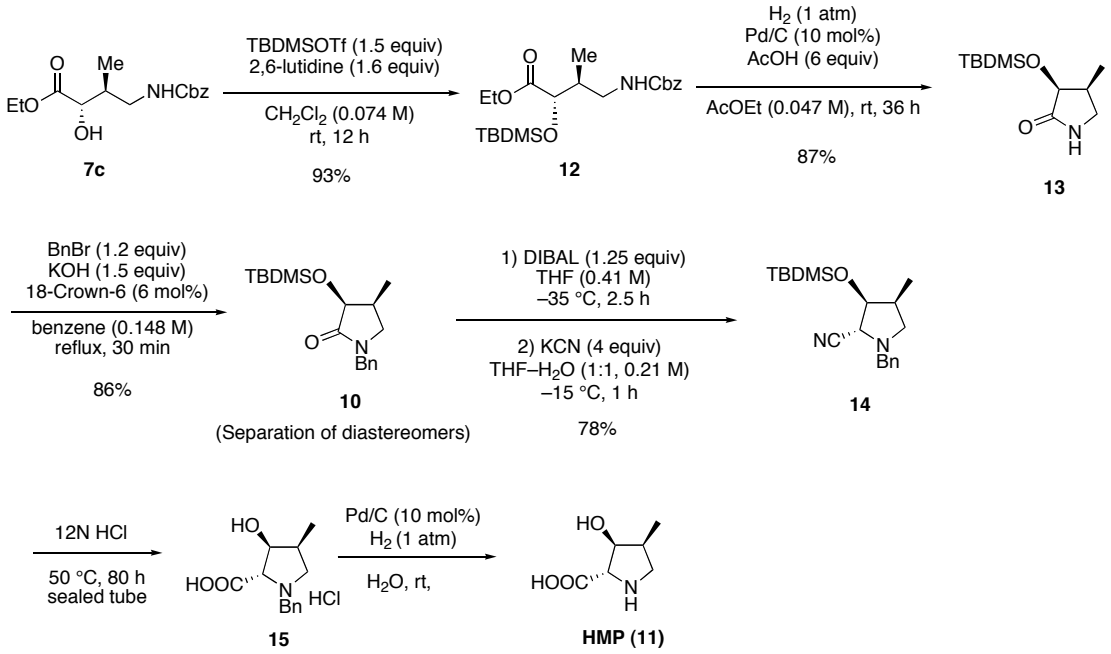
Ligand **1** (9.9 mg, 0.022 mmol) in CH_2Cl_2 (1.5 mL) was added to the $CuClO_4$•$4CH_3CN$ (6.5 mg, 0.020 mmol) flask under argon. The yellow solution thus obtained was stirred for 13 h, and took 0.15 mL of the solution (0.002 mmol of the catalyst) via gas-tight syringe to another dried flask. CH_2Cl_2 (1.35 mL) was added to that flask and cooled to 0 °C. Freshly distilled ethyl glyoxylate (40 μl, 0.40 mmol) in CH_2Cl_2 (0.8 mL) was added to the mixture, and then enecarbamate **2a** (0.20 mmol) in CH_2Cl_2 (0.8 mL) was added in one portion. The reaction mixture was stirred at 0 °C for 1 h, and was quenched by addition of saturated aqueous $NaHCO_3$. The reaction mixture was allowed to warm to rt, and was extracted with CH_2Cl_2. The organic layer was washed with brine and dried over anhydrous $MgSO_4$. After filtration and removal of solvent, CH_3CN (2.0 mL) was added to the crude product and cooled to 0 °C. Allyltrimethylsilane (95.3 μl, 0.6 mmol) and TMSOTf (72.4 μl, 0.4 mmol) were added successively. After stirring for 15.5 h, the reaction was quenched by addition of saturated aqueous $NaHCO_3$ and the reaction mixture was extracted with CH_2Cl_2. After the organic layer was dried over $MgSO_4$, the filtration and the solvent evaporation were done. The crude product was purified by chromatography on silica gel to provide compound **9** (41.7 mg, 65% yield, 95% ee).

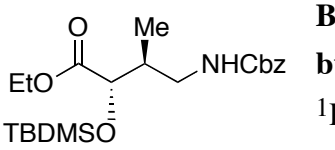


Benzyl (1*S*)-1-(ethoxycarbonyl)-1-hydroxyhex-5-en-3-ylcarbamate (8): ^{1}H NMR ($CDCl_3$) δ = 1.25 (t, 3H, *J* = 7.2 Hz), 1.28 (t, 3H, *J* = 7.2 Hz), 1.75-2.10 (m, 4H), 2.23-2.35 (m, 4H), 3.43 (d, 1H, *J* = 4.8 Hz), 3.66 (d, 1H, *J* = 4.1 Hz), 3.94-4.06 (m, 2H), 4.17 (q, 2H, *J* = 7.1 Hz), 4.22 (q, 2H, *J* = 6.6 Hz), 4.20-4.30 (m, 2H), 5.05-5.17 (m, 4H), 5.70-5.82 (m, 2H), 7.28-7.38 (m, 10H); ^{13}C NMR ($CDCl_3$) δ = 14.1, 14.1, 38.2, 38.8, 39.2, 39.3, 47.4, 47.6, 61.6, 61.7, 61.7, 62.2, 66.7, 66.8, 67.8, 68.3, 118.3, 118.3, 128.0, 128.1, 128.4, 128.5, 133.6, 133.7, 136.3. 136.4, 156.0, 156.6, 174.2, 174.7; HRMS (FAB); Exact mass calcd for $C_{17}H_{24}NO_5$ $[M+H]^+$, 322.1654.; Found 322.1664.; HPLC, Daicel Chiralcel ADH+ADH, hexane/^{i}PrOH = 4/1, flow rate = 0.6 mL/min : t_R = 27.5 min (1*R*), t_R = 28.5 min (1*S*), t_R = 31.0 min (1*S*), t_R = 34.8 min (1*R*). (The peaks at 27.5 and 28.5 min are derived from the

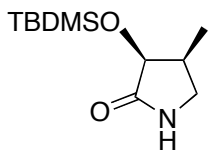
products which have the same relative configuration. The peaks at 31.0 and 34.8 min are derived from the products which have the same relative configuration.)

Synthesis of HMP (11).



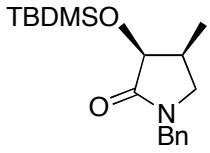


Benzyl (2S,3*S*)-3-(ethoxycarbonyl)-3-(*tert*-butyldimethylsilyl)oxy-2-methylpro pylcarbamate (12): ^{1}H NMR ($CDCl_3$) δ = 0.04 (s, 3H), 0.06 (s, 3H), 0.92 (s, 9H), 1.01 (d, 3H, *J* = 6.9 Hz), 1.23 (t, 3H, *J* = 7.1 Hz), 2.20-2.32 (m, 1H), 3.10-3.28 (m, 2H), 4.09 (d, 1H), *J* = 3.2 Hz), 4.13 (q, 2H, *J* = 7.1 Hz), 5.06 (s, 2H), 5.18-5.24 (m, 1H), 7.27-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = -5.7, -5.2, 14.0, 15.1, 18.0, 25.5, 37.2, 42.5, 60.7, 66.2, 75.1, 127.7, 127.8, 128.2, 136.6, 156.3, 173.2; HRMS (FAB); Exact mass calcd for $C_{21}H_{36}NSiO_5$ $[M+H]^+$, 410.2363.; Found 410.2382.;



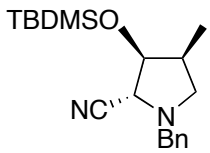
(3*S*,4*S*)-3-(*tert*-butyldimethylsilyl)oxy-4-methylpyrrolidin-2-one (13): ^{1}H NMR ($CDCl_3$) δ = 0.09 (s, 3H), 0.12 (s, 3H), 0.88 (s, 9H), 1.01 (d, 3H, *J* = 6.9 Hz), 2.34-2.46 (m, 1H), 2.34-2.45 (m, 1H), 2.97 (dd, 1H, *J* = 4.6, 9.6 Hz), 3.33 (dd, 1H, *J* = 6.4, 9.6 Hz), 4.10 (d, 1H, *J* = 6.4 Hz); ^{13}C NMR ($CDCl_3$) δ = -5.3, -4.7, 12.4, 18.3, 25.7, 35.3, 46.3, 72.7, 127.0.

Procedure of conversion of 13 to 10. To a solution of lactam **13** (183.6 mg, 0.800 mmol) and 18-crown-6 (12.7 mg, 6 mol%) in benzene (5.4 mL) was added powder KOH (67.4 mg, 1.2 mmol) at rt. The reaction mixture was heated to reflux, then BnBr (114.2 μl, 0.96 mmol) was added and stirred for 30 min. The reaction mixture was then cooled to rt and the precipitate was filtered off. The filtrate was concentrated *in vacuo* and the residue so obtained was purified by chromatography on silica gel to afford benzylated compound **10** (220.3 mg, 86%). At this stage, diastereomers could be separated by thin layer chromatography on silica gel (eluent: hexane/AcOEt = 2/1).



(3*S*,4*S*)-1-Benzyl-3-(*tert*-butyldimethylsilyl)oxy-4-methylpyrrolidin-2-one (10): $[\alpha]^{26}_{D}$ -43.8 (97% ee, c 3.995, $CHCl_3$);^{1}H NMR ($CDCl_3$) δ = 0.16 (s, 3H), 0.20 (s, 3H), 0.94 (s, 9H), 0.98 (d, 3H, *J* = 6.9 Hz), 2.25-2.40 (m, 1H), 2.87 (dd, 1H, *J* = 5.0, 9.6 Hz), 3.20 (dd, 1H, *J* = 6.9, 9.6 Hz), 4.23 (d, 1H, *J* = 6.4 Hz), 4.33 (d, 1H, *J* = 15.1 Hz), 4.53 (d, 1H, *J* = 15.1 Hz), 7.20-7.36 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = -5.3, -4.5, 12.4, 18.4, 25.8, 33.0, 46.6, 50.6, 73.4, 127.5, 128.0, 128.6, 136.3, 173.2; IR (neat) 2956, 2929, 2856, 1707, 1499, 1444, 1359, 1293, 1252, 1159, 1091, 1033, 934, 835, 780, 700, 524 cm^{-1}HRMS (FAB); Exact mass calcd for $C_{18}H_{30}NSiO_2$ $[M+H]^+$, 320.2046.; Found 320.2044.

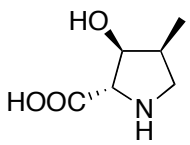
Procedure of conversion of 10 to 14. To a solution (1.4 mL) of lactam **10** (184.1 mg, 0.576 mmol) in THF (1.4 mL) was added DIBAL (hexane solution, 0.94M, 0.77 ml, 0.72 mmol) at –35 °C. The reaction mixture was stirred for 2.5 h, then allowed to warm to –15 °C. Aqueous solution of KCN (1.65 M, 1.4 mL, 2.3 mmol) was added to the reaction mixture and the reaction mixture was stirred at –15 °C for 1 h. The reaction was allowed to warm to rt and diluted by AcOEt and H_2O. The precipitate was filtered off and the mixture was extracted with AcOEt. The organic layer was dried over anhydrous Na_2SO_4. After filtration and solvent evaporation, the residue was purified by chromatography on silica gel to afford cyanated product **14** (149.2 mg, 78%).



(2*R*,3*S*,4*S*)-1-Benzyl-3-(*tert*-butyldimethylsilyl)oxy-4-methylpyrrolidine-2-carbonitrile (14): $[\alpha]^{26}_{D}$ -43.8 (97% ee, c 3.995, $CHCl_3$);^{1}H NMR ($CDCl_3$) δ = 0.08 (s, 3H), 0.10 (s, 3H), 0.90 (s, 9H), 0.96 (d, 3H), *J* = 6.0 Hz), 2.30-2.44 (m, 2H), 2.90-3.00 (m, 1H), 3.33 (d,

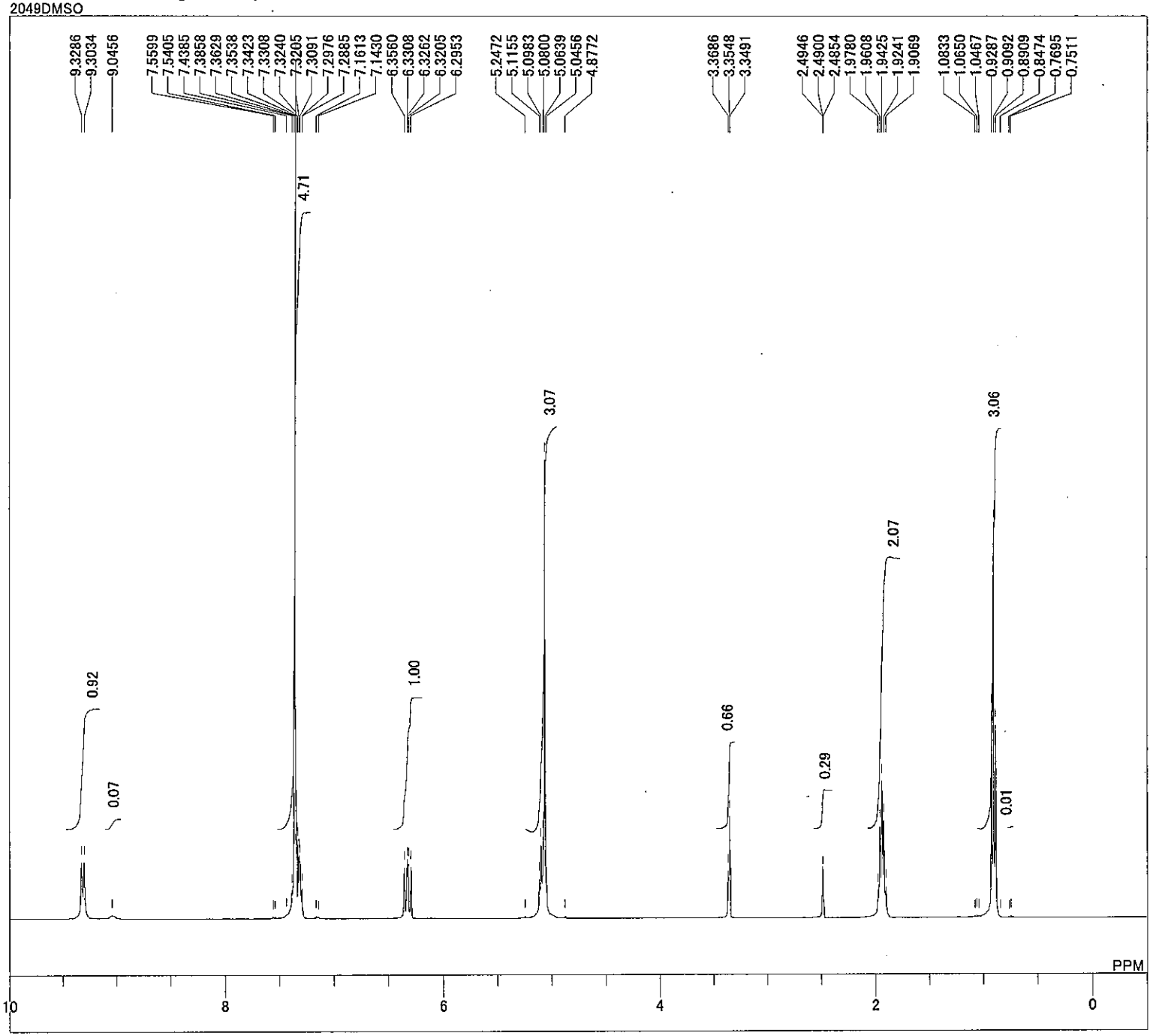
1H, *J* = 2.7 Hz), 3.61 (d, 1H, *J* = 13.3 Hz), 3.98 (d, 1H, *J* = 13.3 Hz), 4.37 (dd, 1H, *J* 2.7, 5.0 Hz), 7.23-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = -5.1, -4.8, 12.2, 18.0, 25.7, 37.4, 58.0, 58.3, 62.6, 77.8, 118.7, 127.4, 128.4, 128.7, 137.4; IR (neat) 3030, 2956, 2929, 2857, 2808, 1495, 1473, 1455, 1371, 1324, 1254, 1214, 1167, 1118, 1089, 1040, 1006, 915, 836, 778, 751, 700, 671, 473 cm^{-1}HRMS (FAB); Exact mass calcd for $C_{18}H_{30}NSiO$ $[M\text{-}CN]^+$, 304.2097.; Found 304.2099.

Procedure of conversion of 14 to HMP (11). Compound **14** (64 mg, 0.193 mmol) was placed into a reinforced flask fitted with a teflon screw cap. Concentrated HCl (1.5 mL) was added to the flask, and the flask was sealed. The reaction mixture was heated at 50 °C for 80 h then cooled to rt. The crude mixture was washed with AcOEt and extracted with H2O. The water layer was concentrated in vacuo to afford almost pure N-benzylated HMP N-benzylated HMP was dissolved in H_2O (2 mL) and Pd/C (wet, 10 mol%) was added. The mixture was vigorously stirred under 1 atm H2 atmosphere for 12 h at rt. After filtration, the filtrate was concentrated in vacuo. The residue was purified by ion exchange resin (Dowex 50W-X2, 50-100 mesh). The brown solid so obtained was washed with MeOH to afford HMP (**11**) as a white solid (24.7 mg, 88%).



(2*S*,3*S*,4*S*)-3-Hydroxy-4-methylproline (HMP (11)): $[\alpha]^{28}_D$ -27.8 (97% ee, c 0.8, H_2O) (lit. $[\alpha]^{25}_D$ -27 (c 0.8, H_2O). ;^{1}H NMR (D_2 O) δ = 1.06 (d, 3H, *J* = 6.4 Hz), 2.20-2.34 (m, 1H), 3.06 (t, 1H, *J* = 11.7 Hz), 3.60 (dd, 1H, *J* = 7.8, 11.7 Hz), 4.09 (s, 1H), 4.44 (d, 1H, *J* = 3.7 Hz); ^{13}C NMR (D_2 O) δ = 10.0, 37.2, 49.8, 70.0, 76.4, 172.4; IR (KBr) 3318, 3070, 2975, 2915, 2635, 2549, 1628, 1455, 1380, 1303, 1278, 1229, 1040, 1008, 879, 728, 652, 475 cm^{-1}HRMS (FAB); Exact mass calcd for $C_6H_{12}NO_3$ $[M+H]^+$, 146.0817.; Found 146.0821.

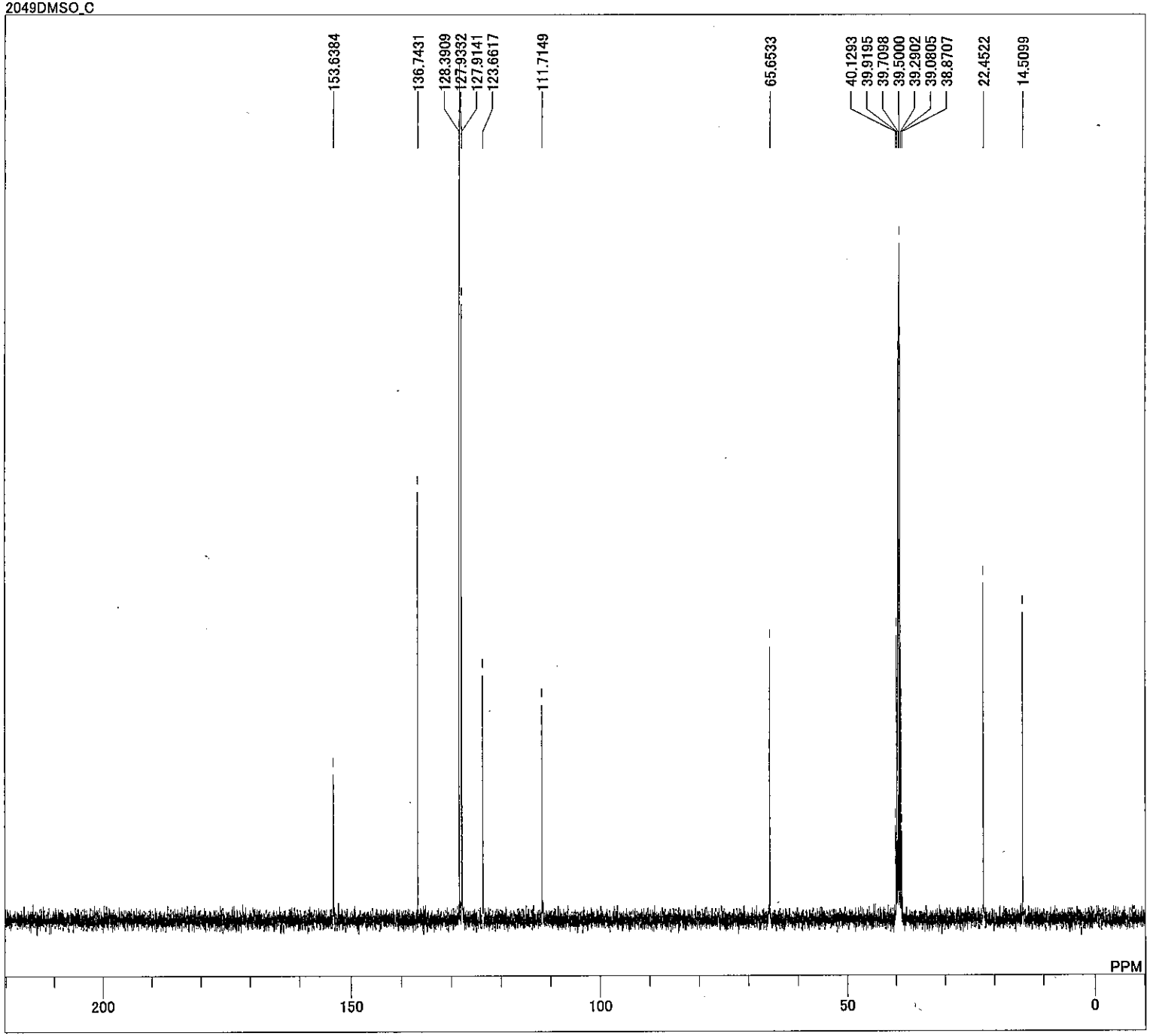
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2049DMSO.als
2049DMSO



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2049DMSO
DATIM	26-09-2005 10:48:39
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	DMSO
EXREF	2.49 ppm
BF	0.12 Hz
RGAIN	28

E-2d

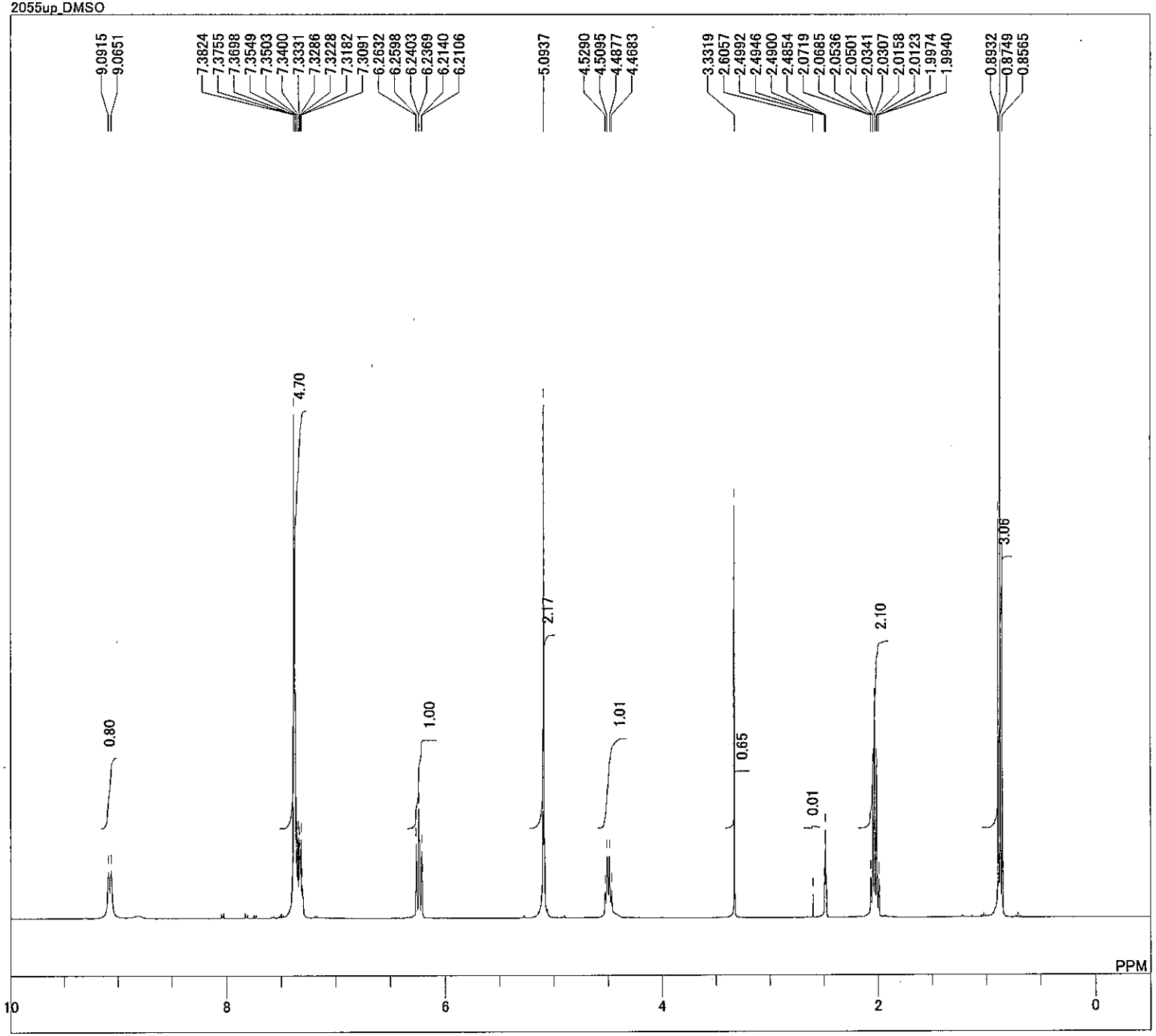
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2049DMSO_C.als
2049DMSO_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2049DMSO_C
DATIM	26-09-2005 10:57:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	117
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.1 c
SLVNT	DMSO
EXREF	39.50 ppm
BF	0.12 Hz
RGAIN	56

E-2d

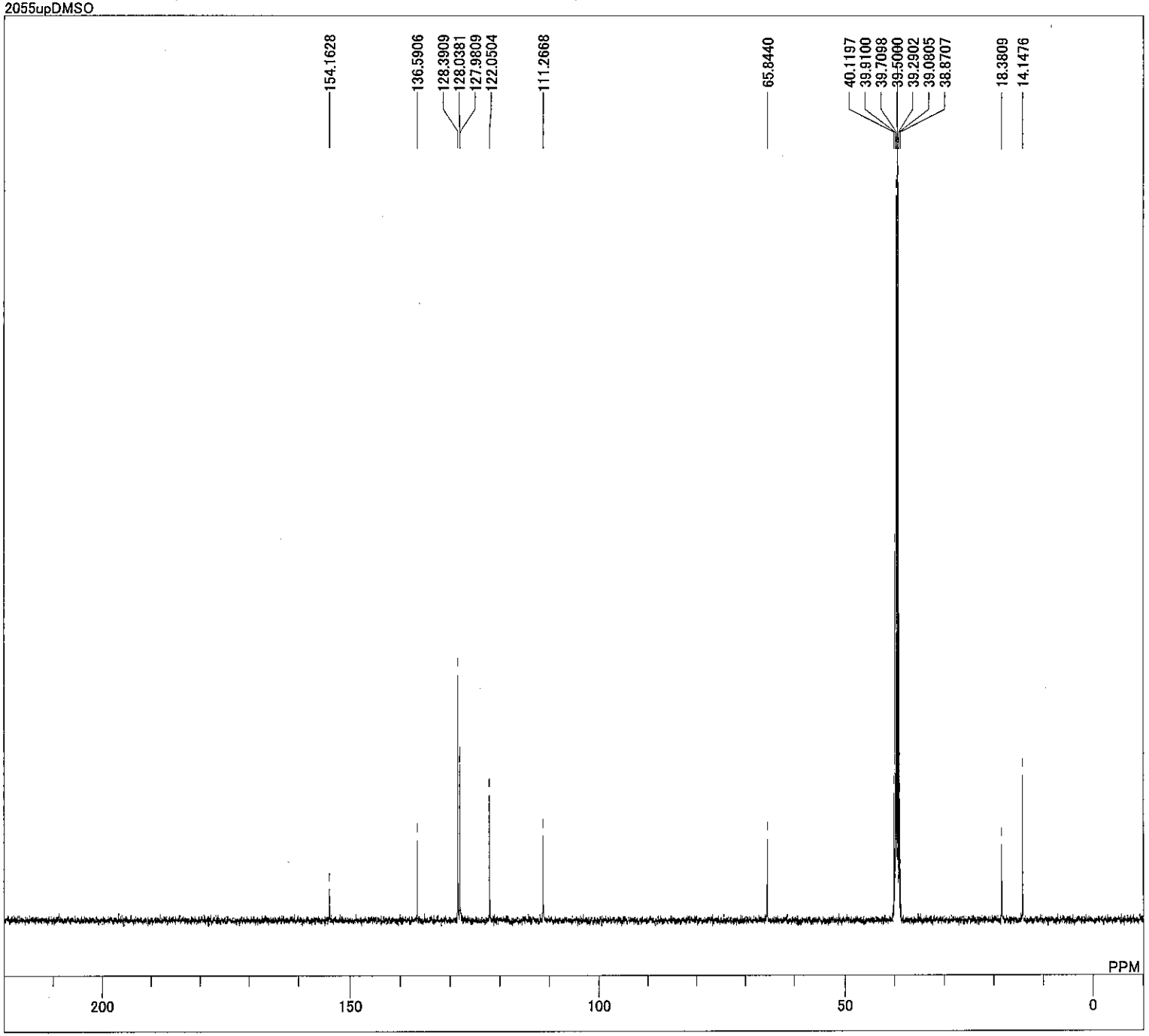
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2055up_DMSO.als
2055up_DMSO



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2055up_DMSO
DATIM	26-09-2005 14:26:07
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	DMSO
EXREF	2.49 ppm
BF	0.12 Hz
RGAIN	28

Z-2d

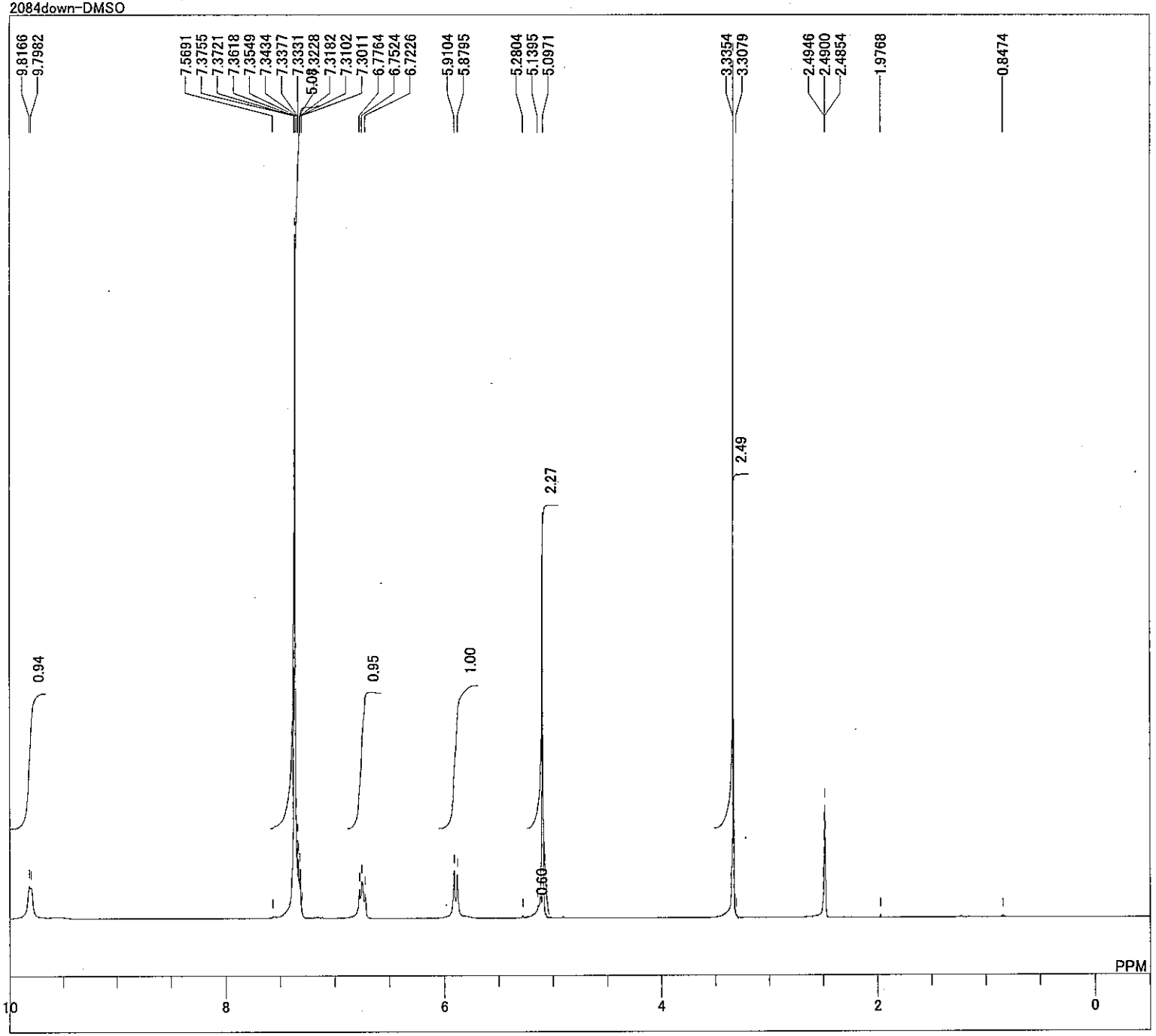
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2055upDMSO-C.als
2055upDMSO



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2055upDMSO
DATIM	24-11-2005 11:22:17
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	446
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.9 c
SLVNT	DMSO
EXREF	39.50 ppm
BF	1.20 Hz
RGAIN	56

Z-20

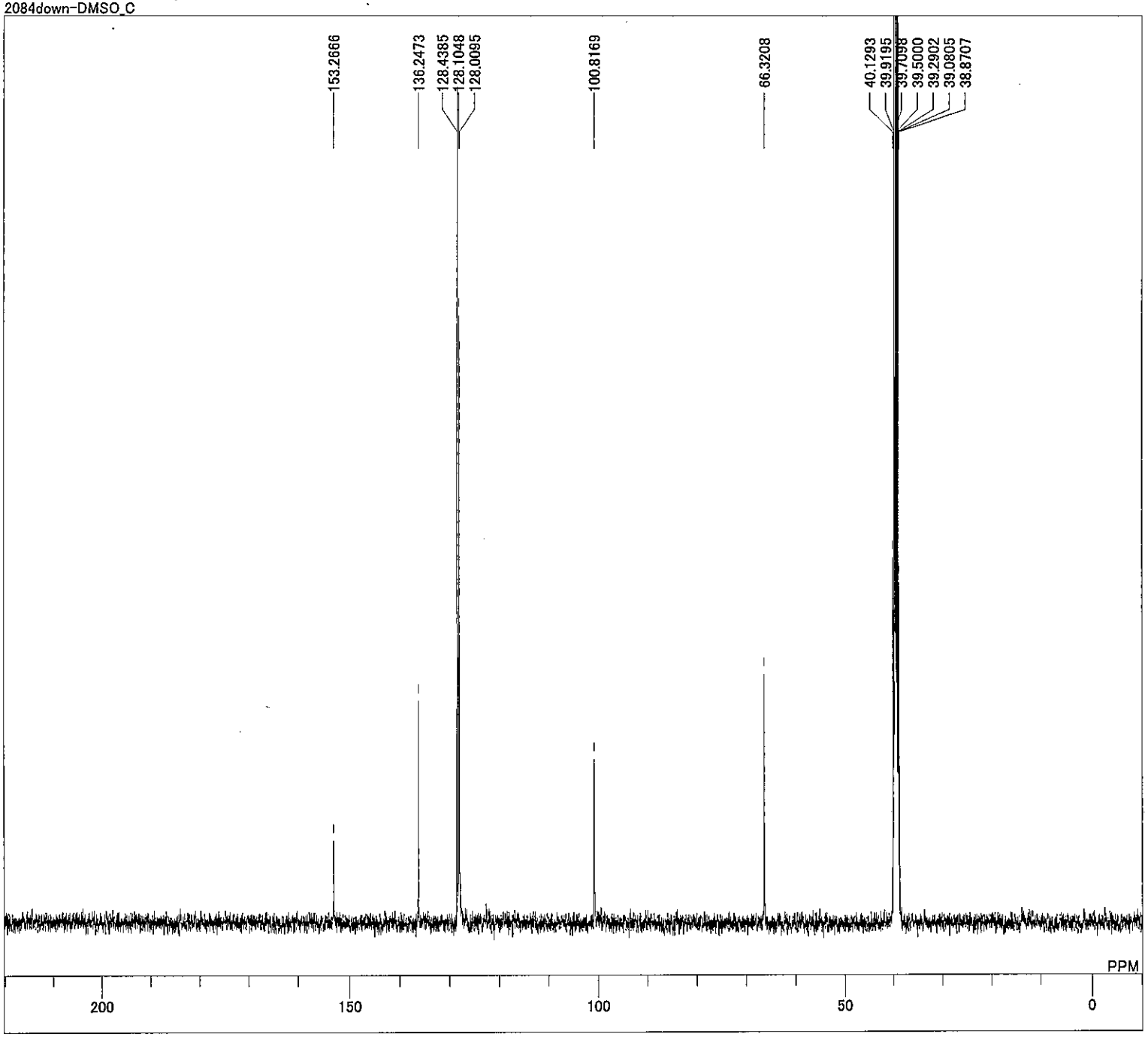
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2084down-DMSO.als
2084down-DMSO



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2084down-DMSO
DATIM	26-09-2005 13:38:27
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.1 c
SLVNT	DMSO
EXREF	2.49 ppm
BF	0.12 Hz
RGAIN	34

E-2e

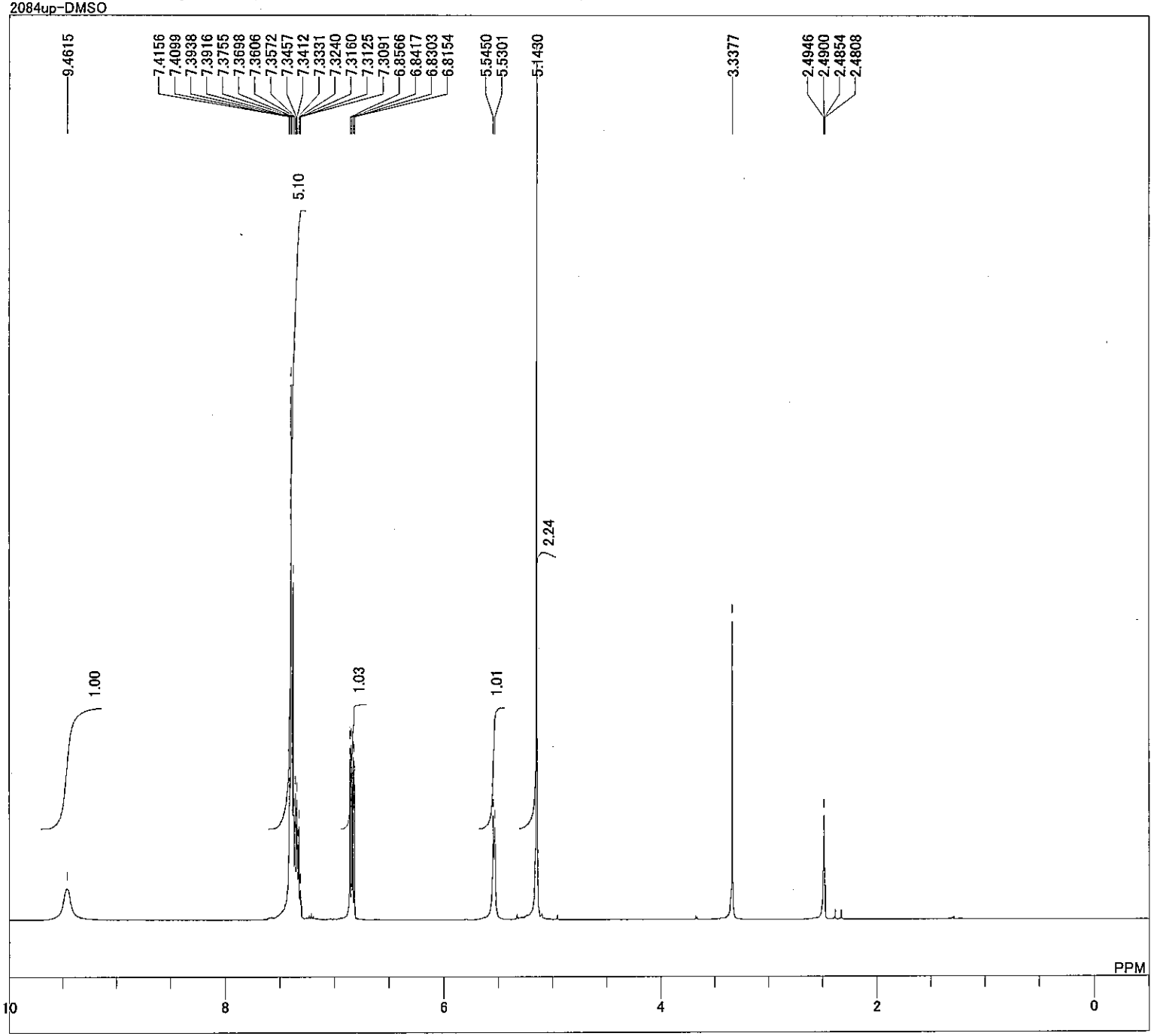
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2084down-DMSO_C.als
2084down-DMSO_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2084down-DMSO_C
DATIM	26-09-2005 14:03:23
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	470
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	DMSO
EXREF	39.50 ppm
BF	0.12 Hz
RGAIN	50

E-2e

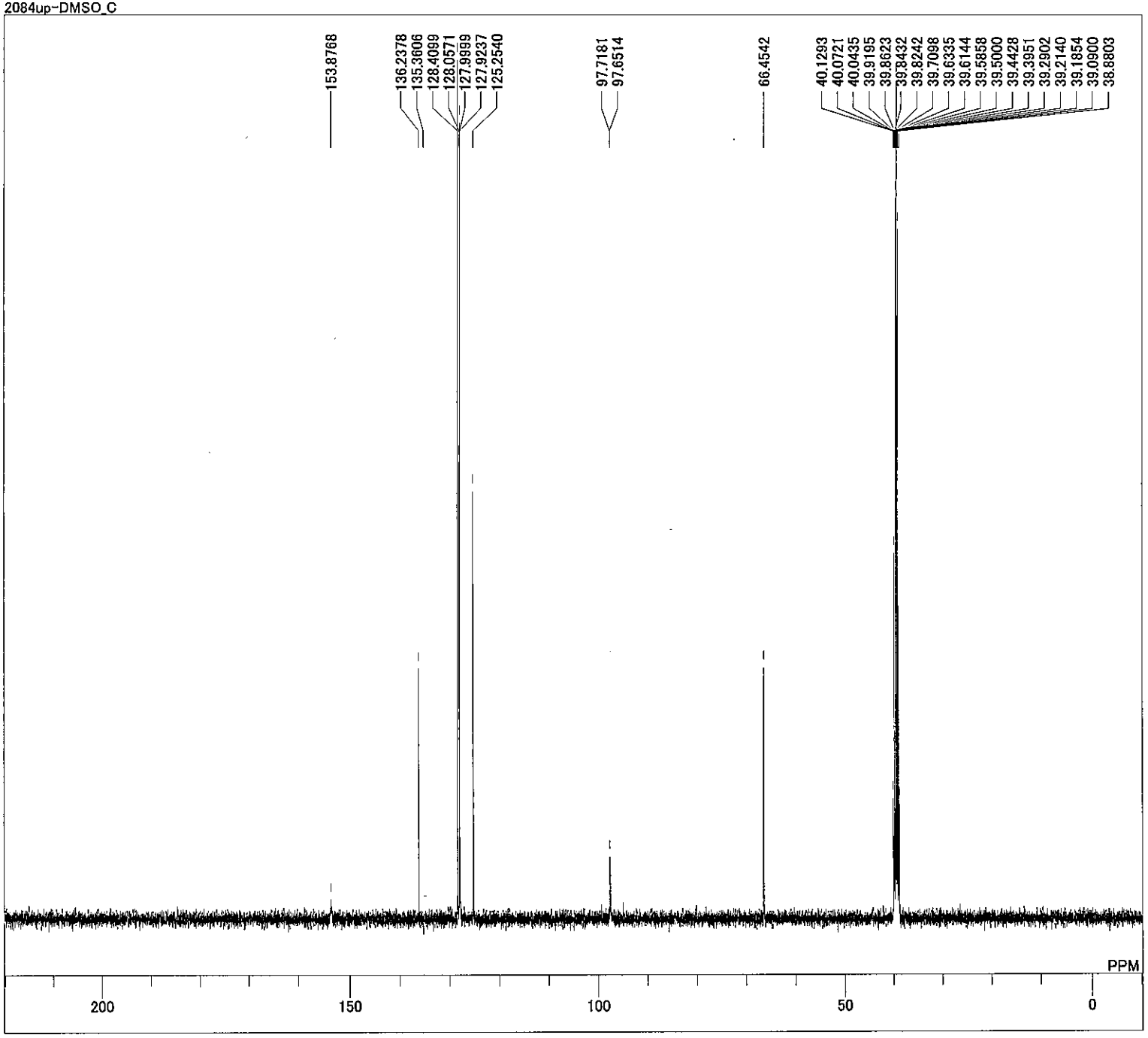
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2084up-DMSO.als
2084up-DMSO



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2084up-DMSO
DATIM	26-09-2005 14:07:14
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	DMSO
EXREF	2.49 ppm
BF	0.12 Hz
RGAIN	32

2-2e

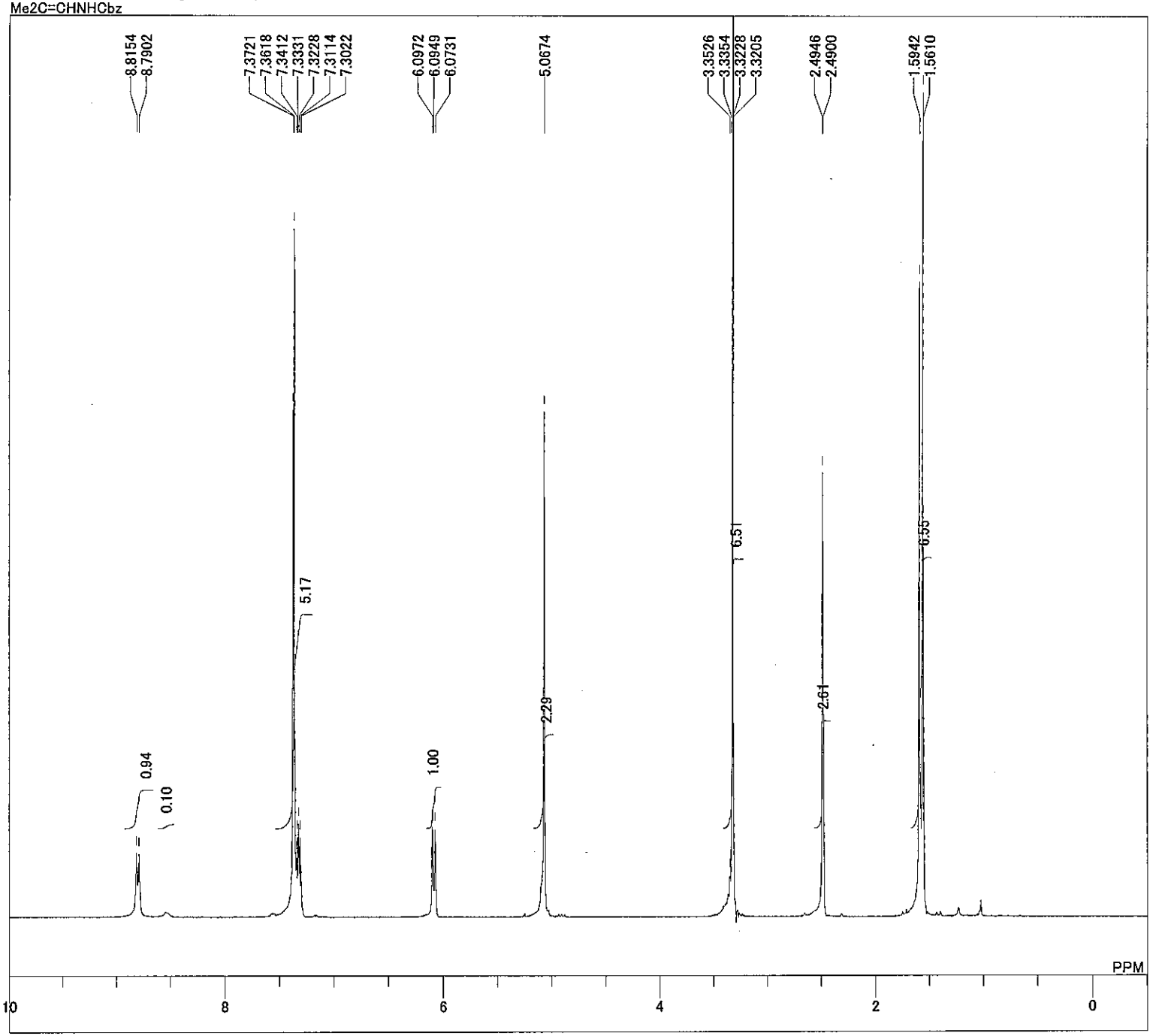
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2084up-DMSO_C.als
2084up-DMSO_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥
COMNT	2084up-DMSO_C
DATIM	26-09-2005 14:23:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	303
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	DMSO
EXREF	39.50 ppm
BF	0.12 Hz
RGAIN	50

Z-2e

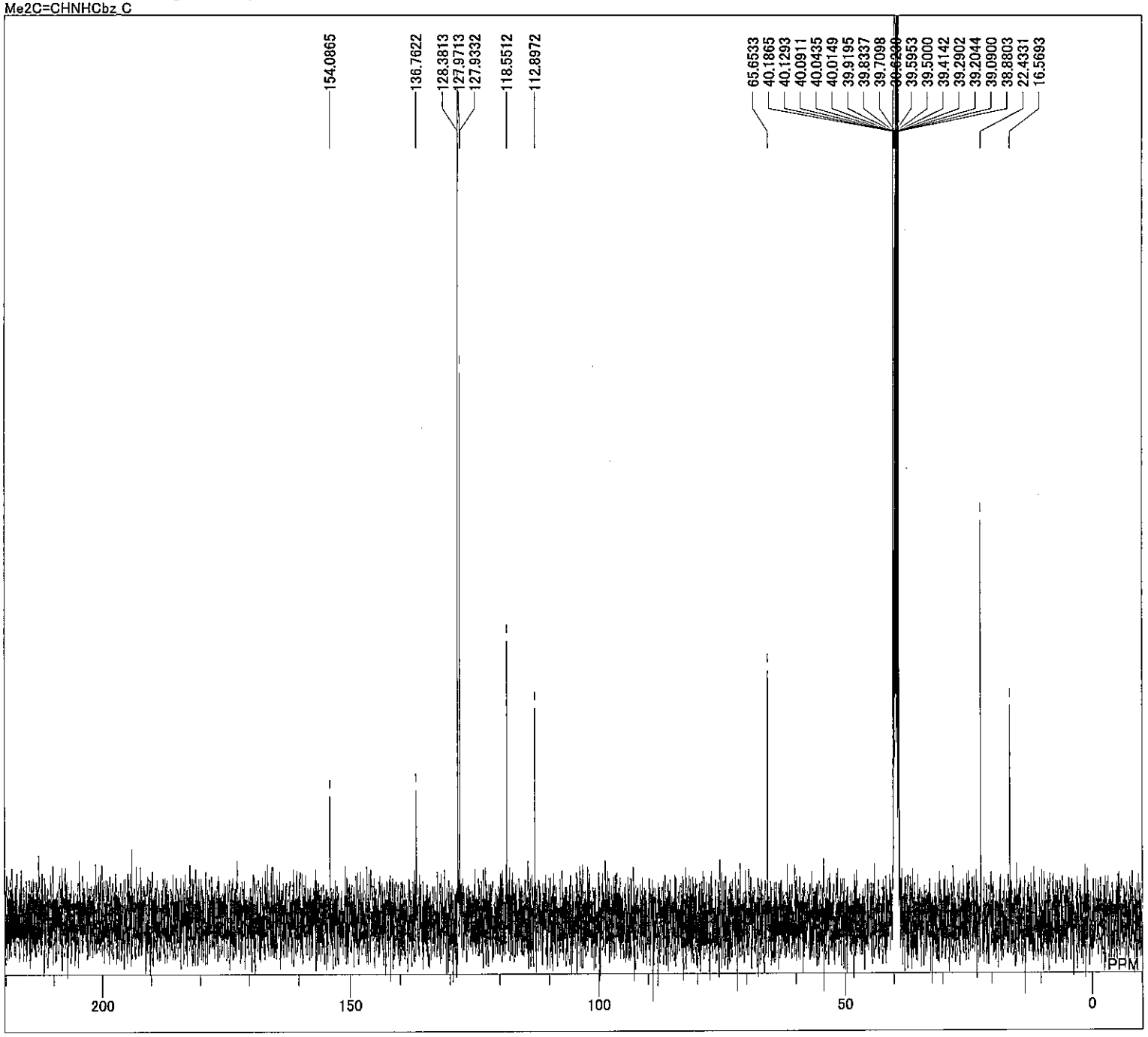
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥Me2C=CHNHCbz.als
Me2C=CHNHCbz



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	Me2C=CHNHCbz
DATIM	26-09-2005 14:35:19
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	DMSO
EXREF	2.49 ppm
BF	0.12 Hz
RGAIN	34

25

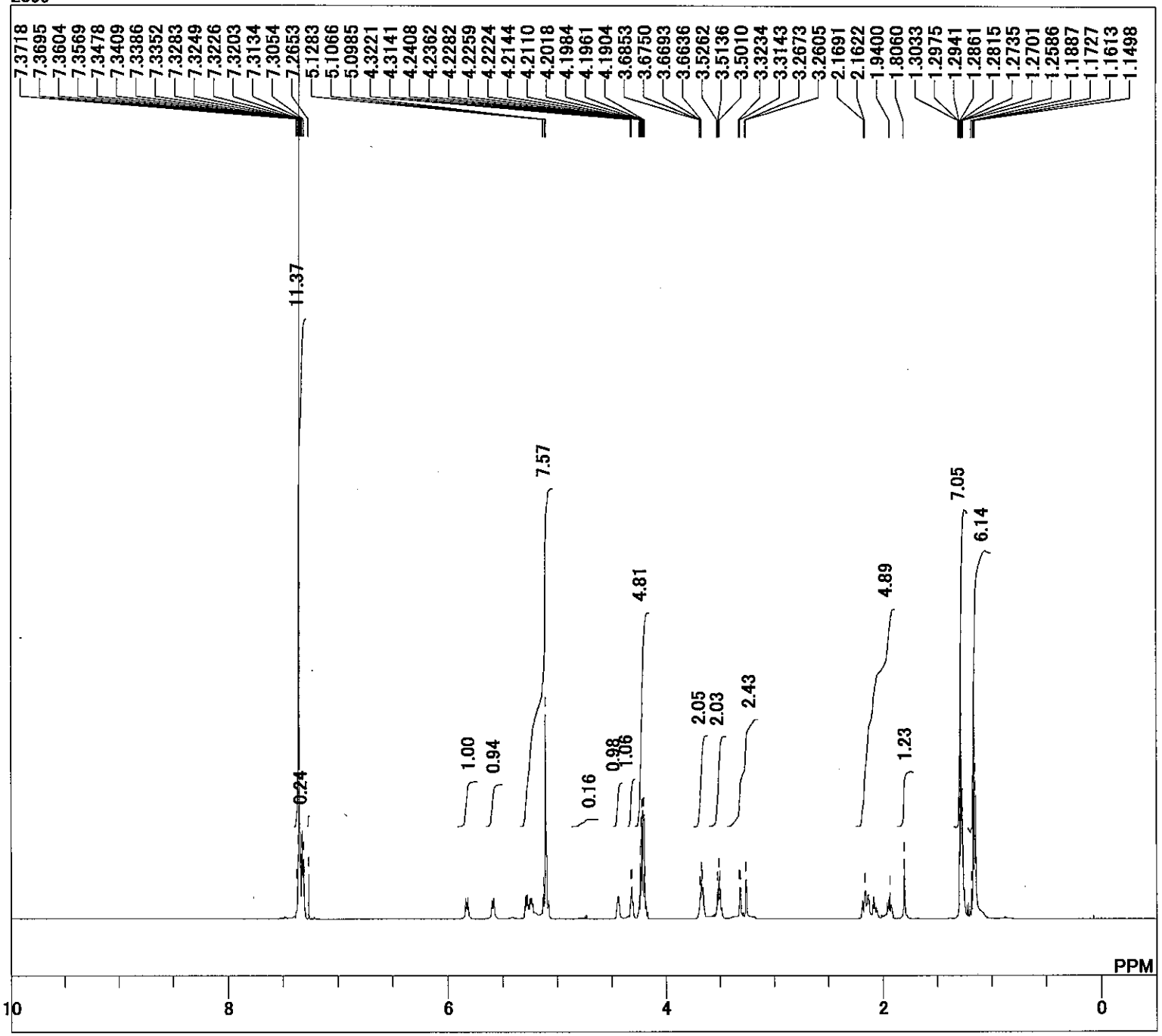
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥Me2C=CHNHCbz_C.als
Me2C=CHNHCbz_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	Me2C=CHNHCbz_C
DATIM	26-09-2005 15:02:21
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	517
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	DMSO
EXREF	39.50 ppm
BF	0.12 Hz
RGAIN	54

2f

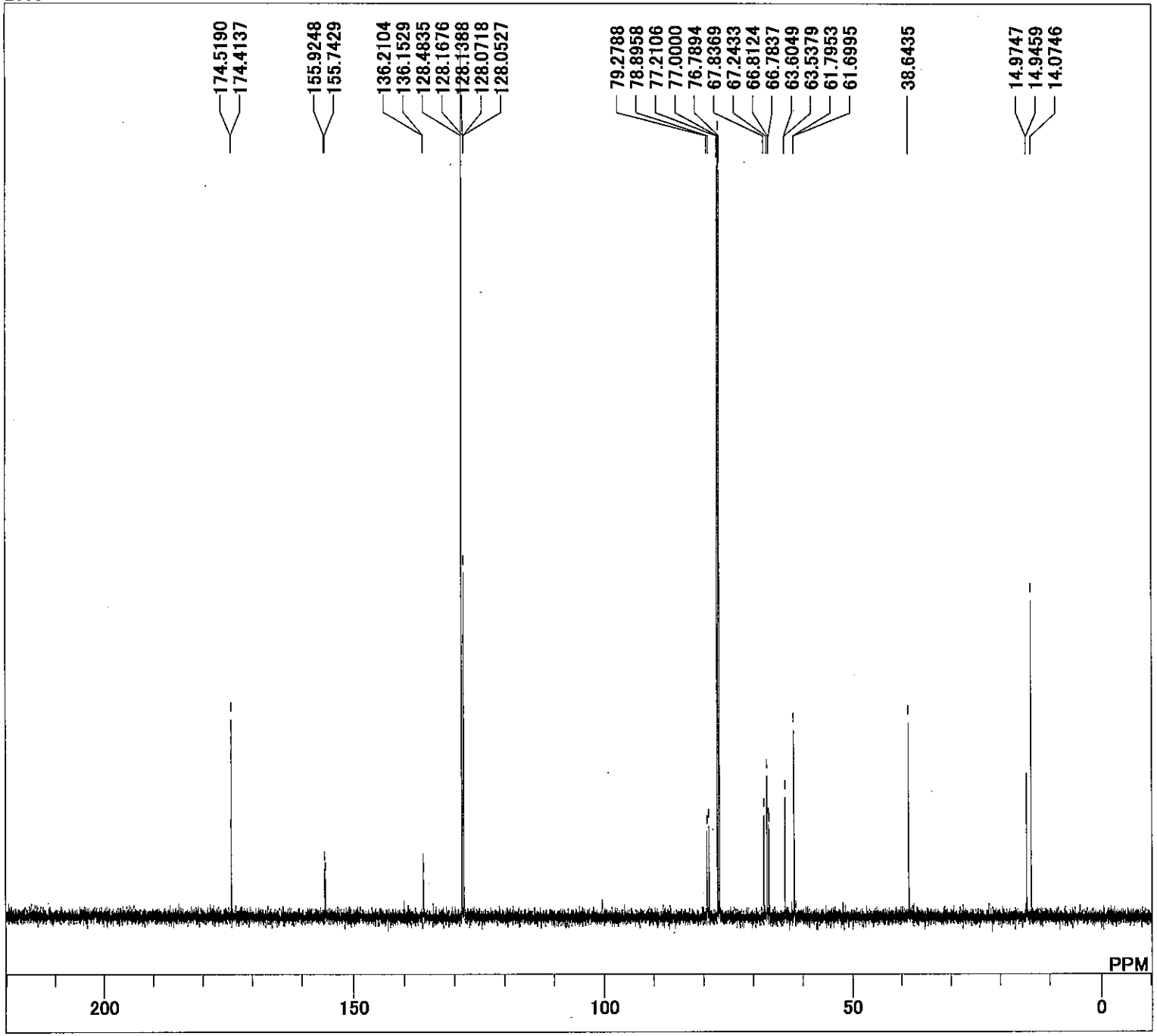
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2099_PROTON.als
2099



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2099
DATIM	24-08-2005 12:20:02
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	13107
FREQU	9008.87 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	23.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

3aa

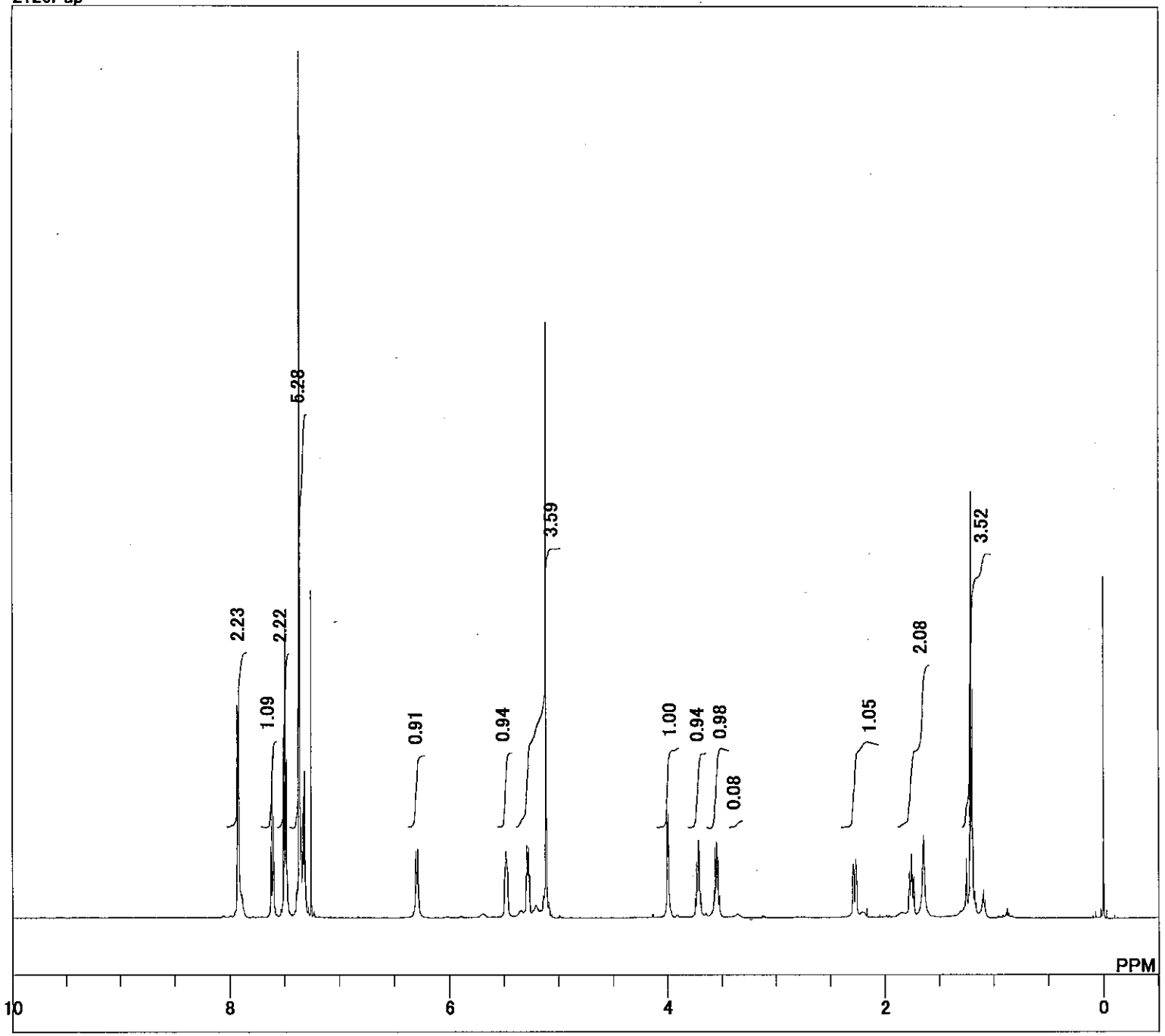
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2099_CARBON.als
2099



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2099
DATIM	24-08-2005 12:32:16
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	24.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

3aa

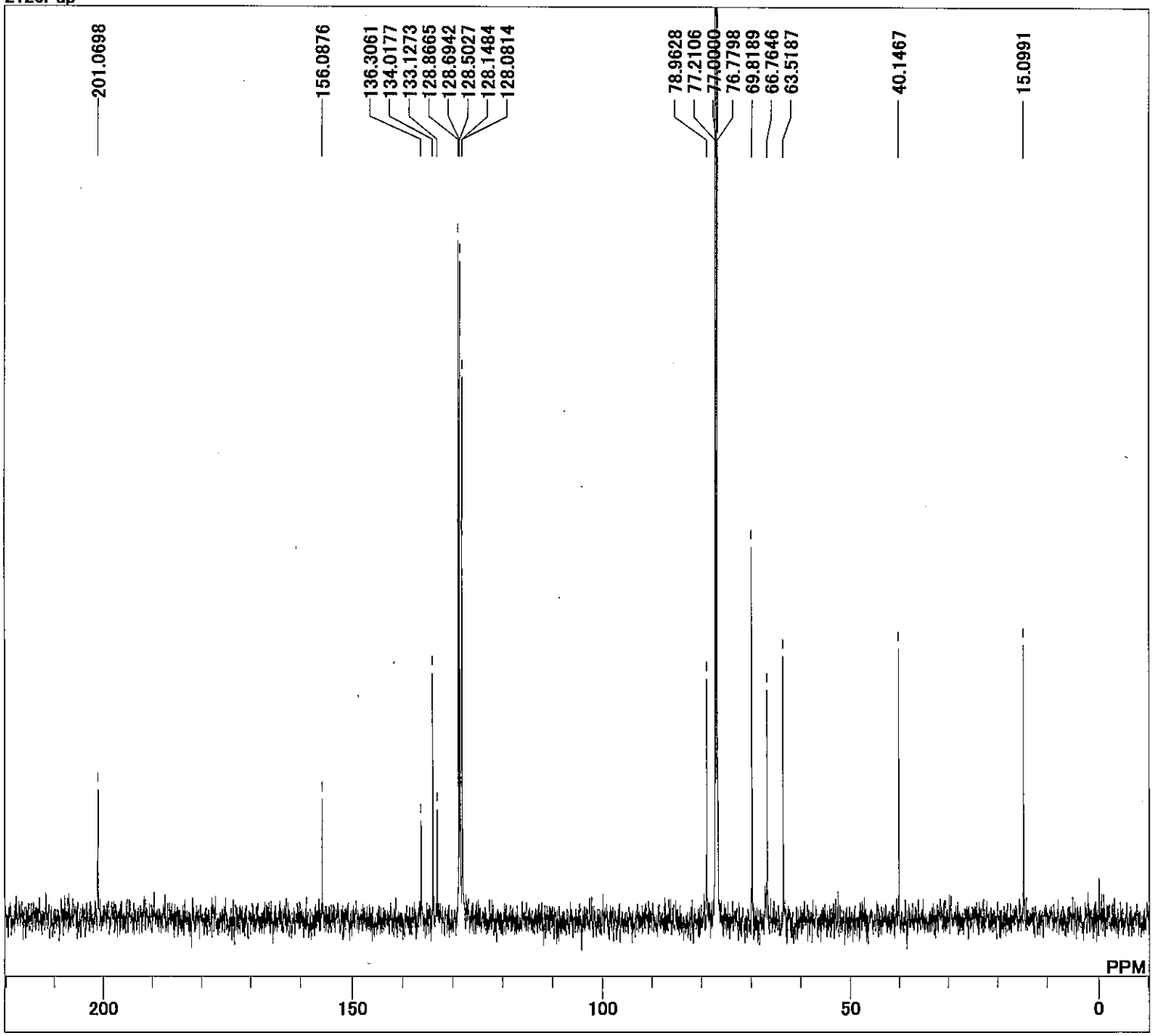
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2126Pup_PROTON.als
2126Pup



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2126Pup
DATIM	20-09-2005 13:31:01
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44

3ab-up

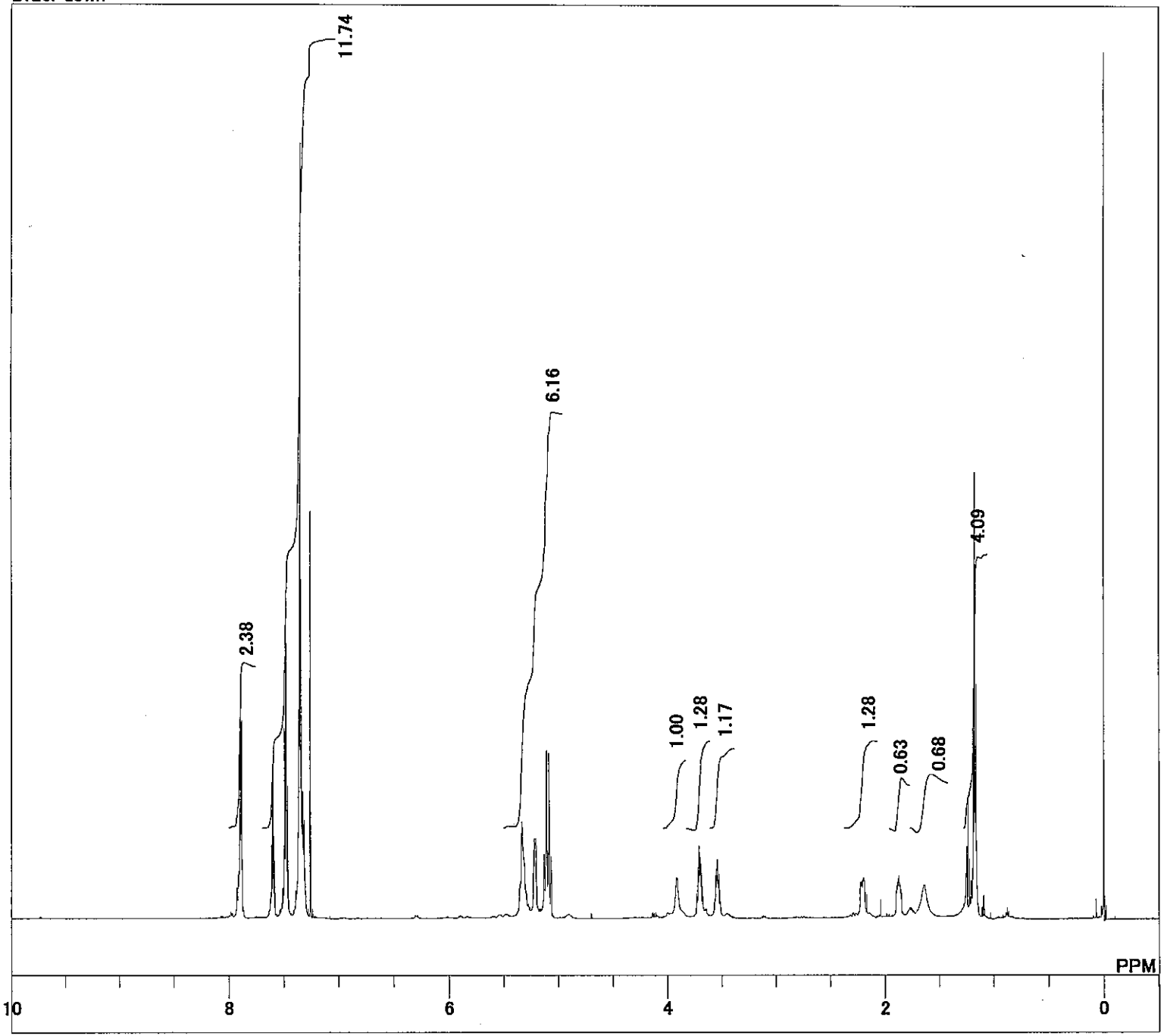
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2126Pup_CARBON.als
2126Pup



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2126Pup
DATIM	20-09-2005 13:45:13
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	300
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

3ab-up

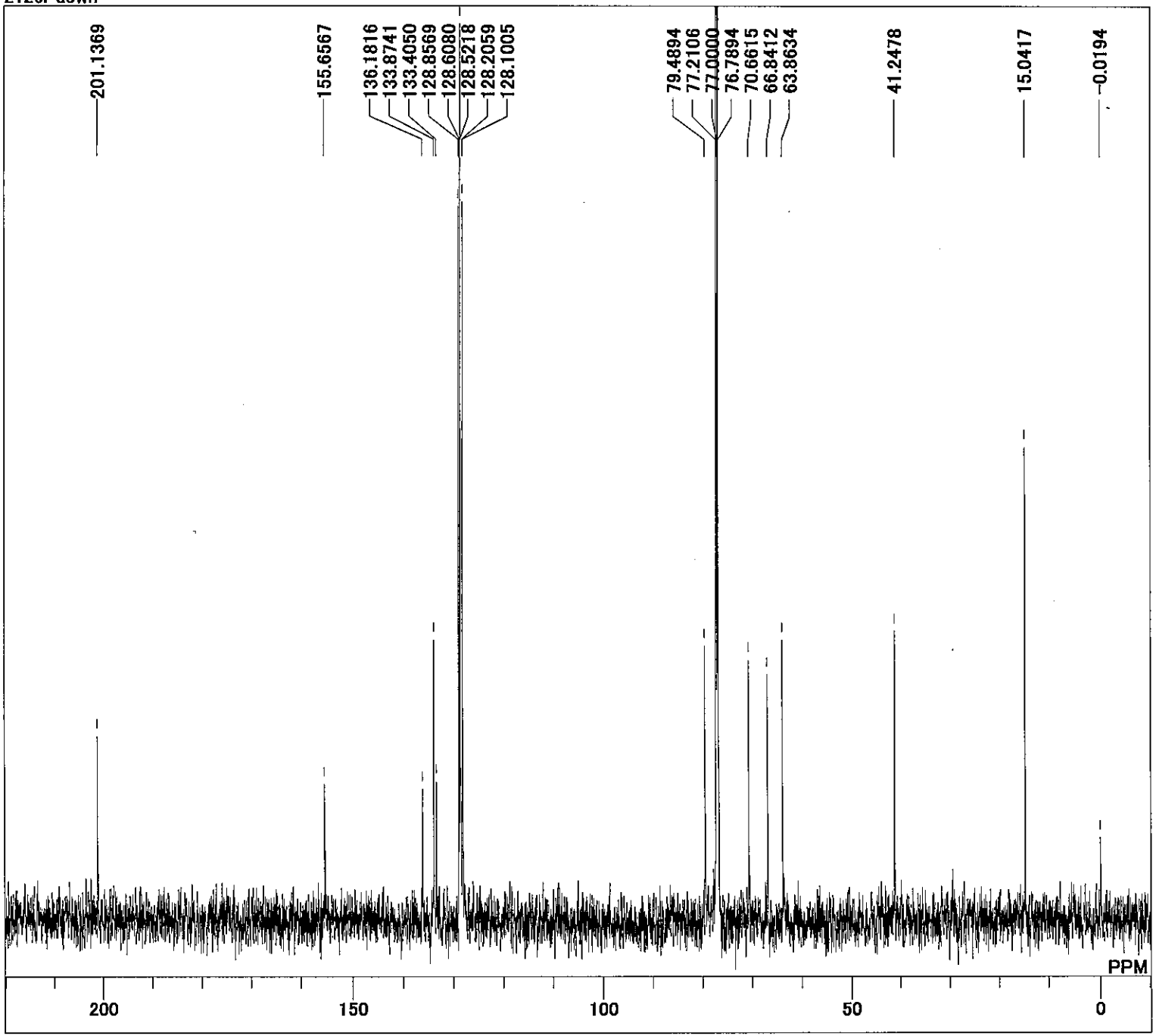
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2126Pdown_PROTON.als
2126Pdown



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2126Pdown
DATIM	20-09-2005 13:56:03
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44

3ab-down

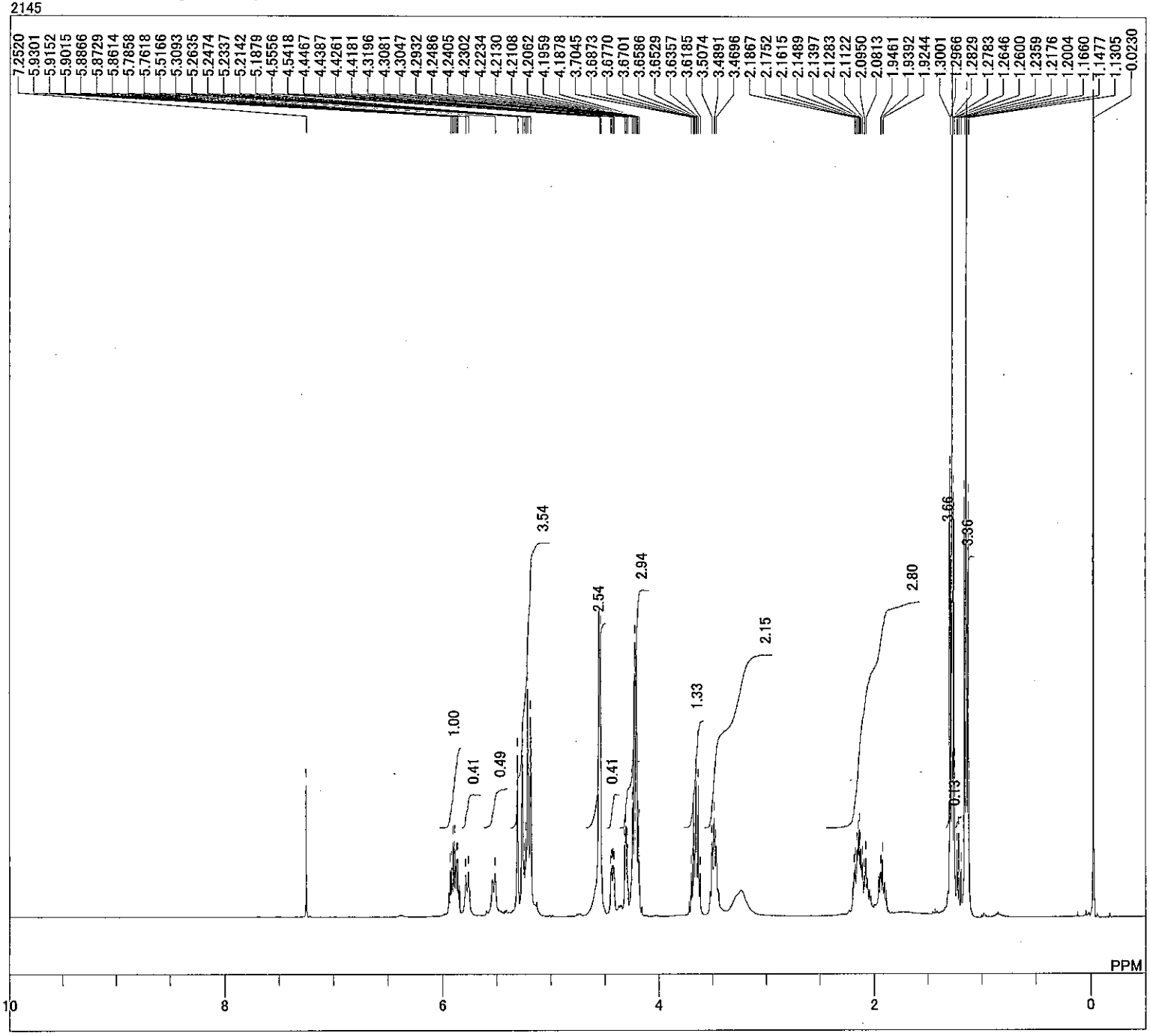
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2126Pdown_CARBON.als
2126Pdown



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2126Pdown
DATIM	20-09-2005 14:10:15
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	300
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

3ab- down

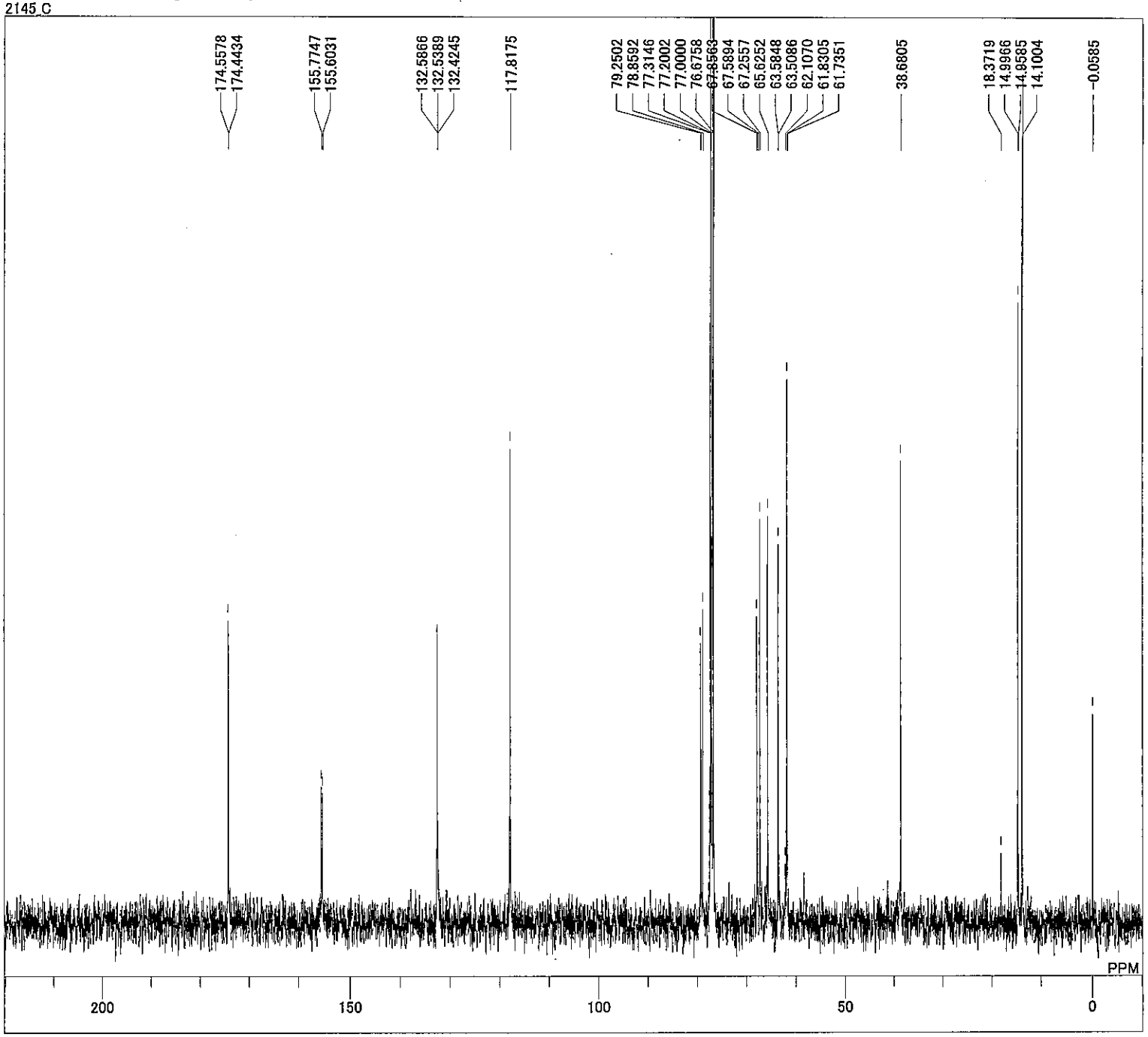
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2145.als
2145



DFILE	C:¥Documents and Settings¥delta¥My Documents¥
COMNT	2145
DATIM	15-10-2005 11:21:40
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.20 Hz
RGAIN	30

3ba

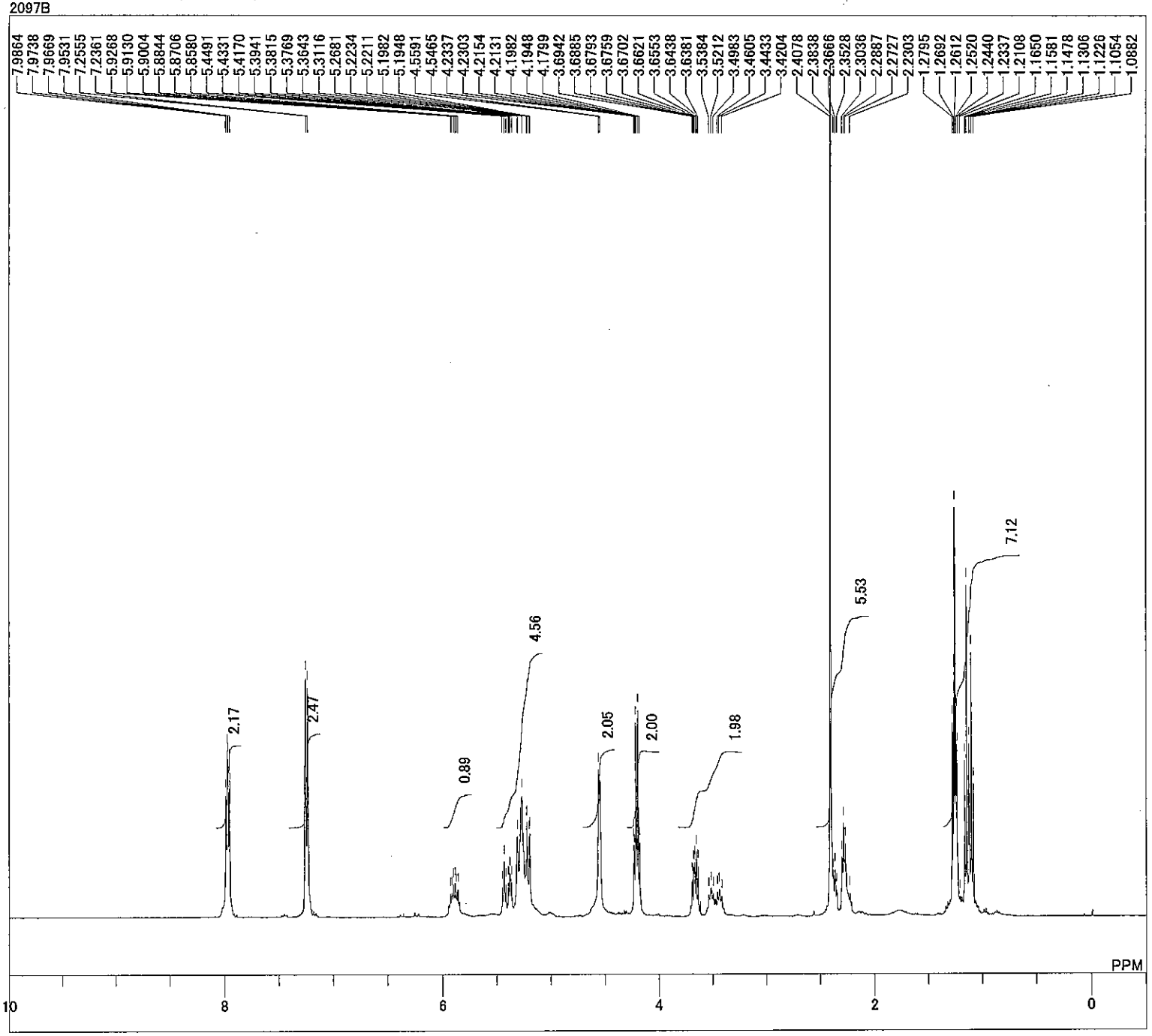
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2145C.als
2145_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2145_C
DATIM	15-10-2005 11:44:19
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	425
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	50

36a

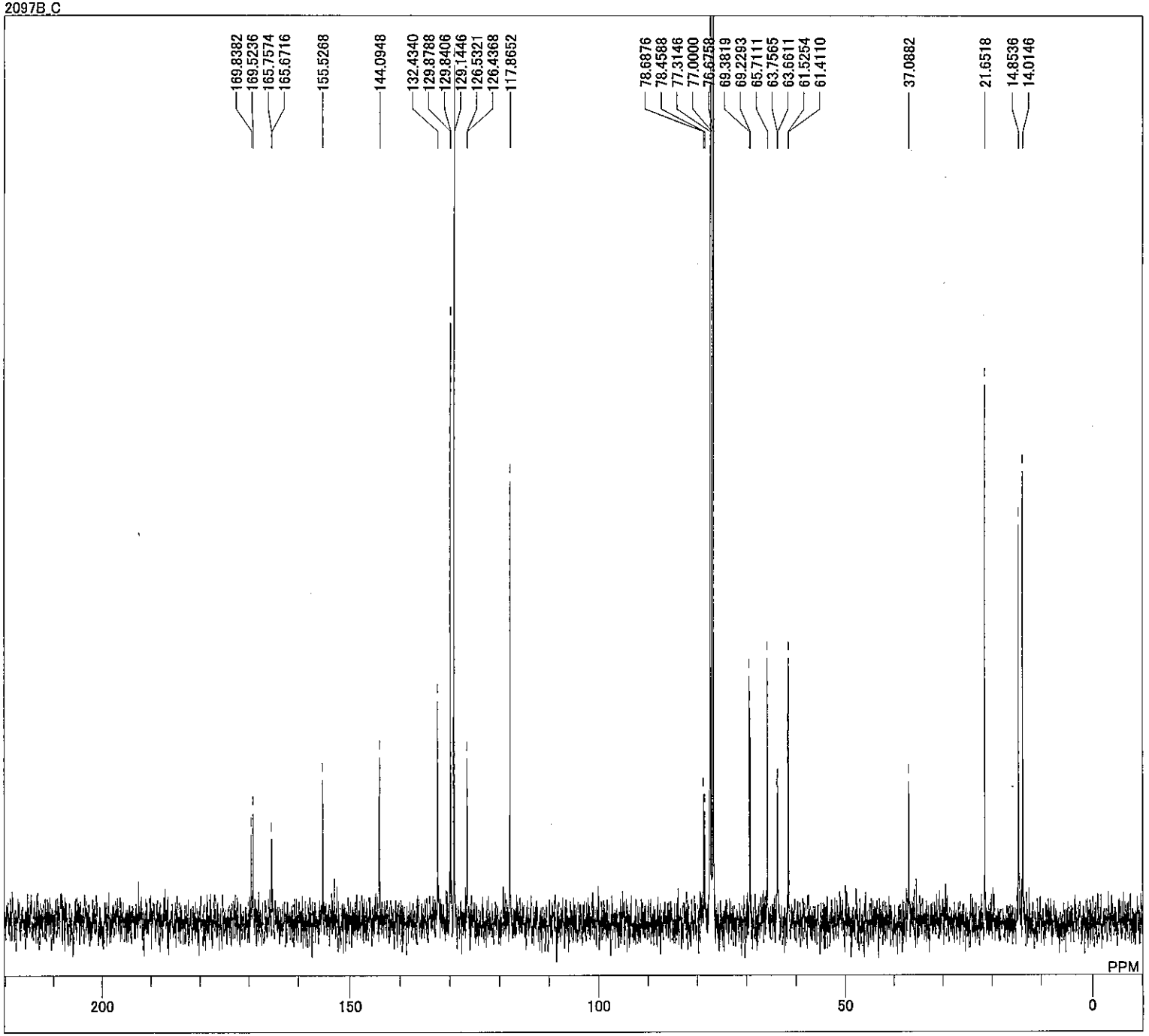
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2097B.als
2097B



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2097B
DATIM	05-08-2005 22:32:06
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	32

6ba

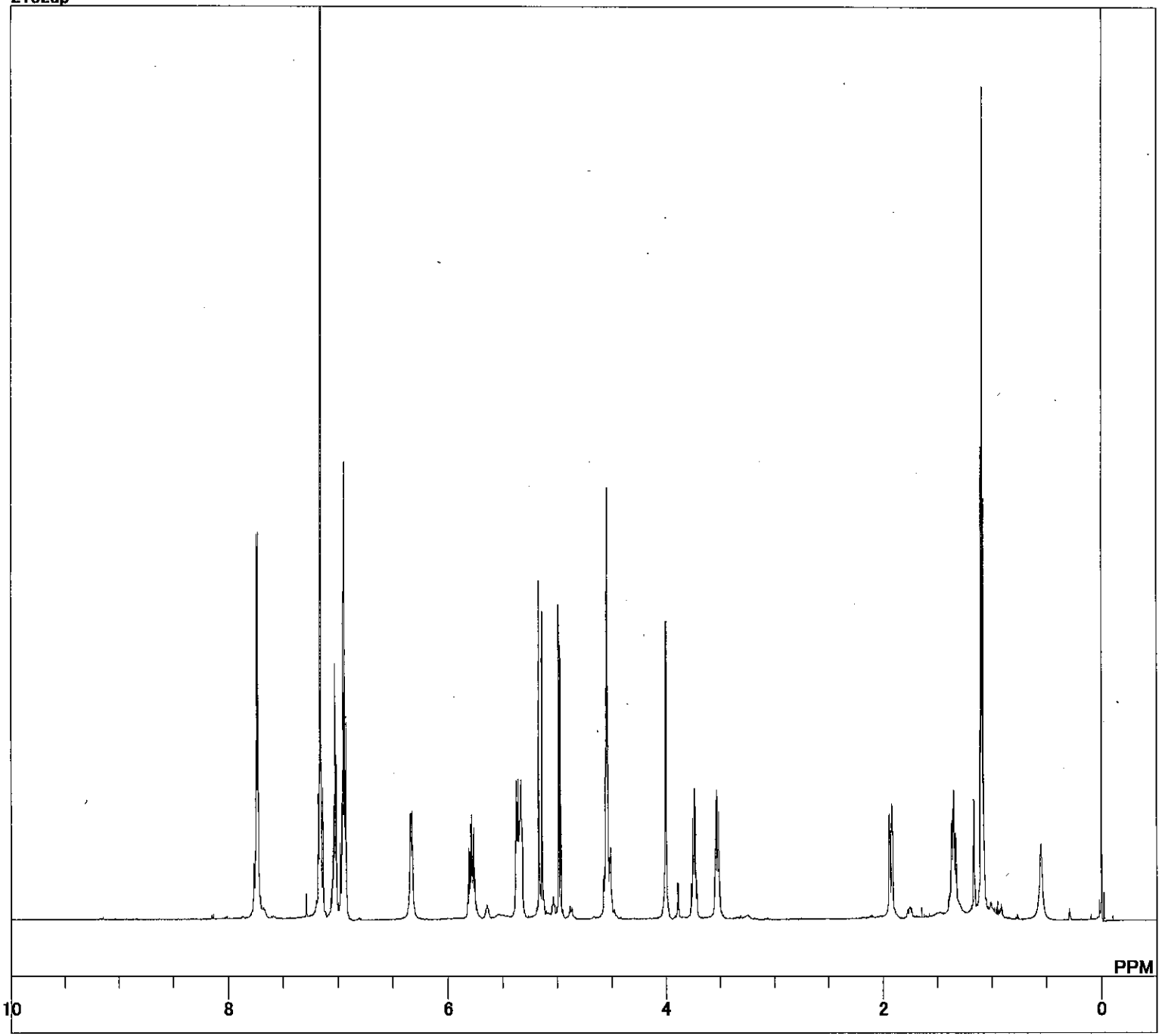
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2097B_C.als
2097B_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2097B_C
DATIM	05-08-2005 22:47:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	266
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	25.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

6ba

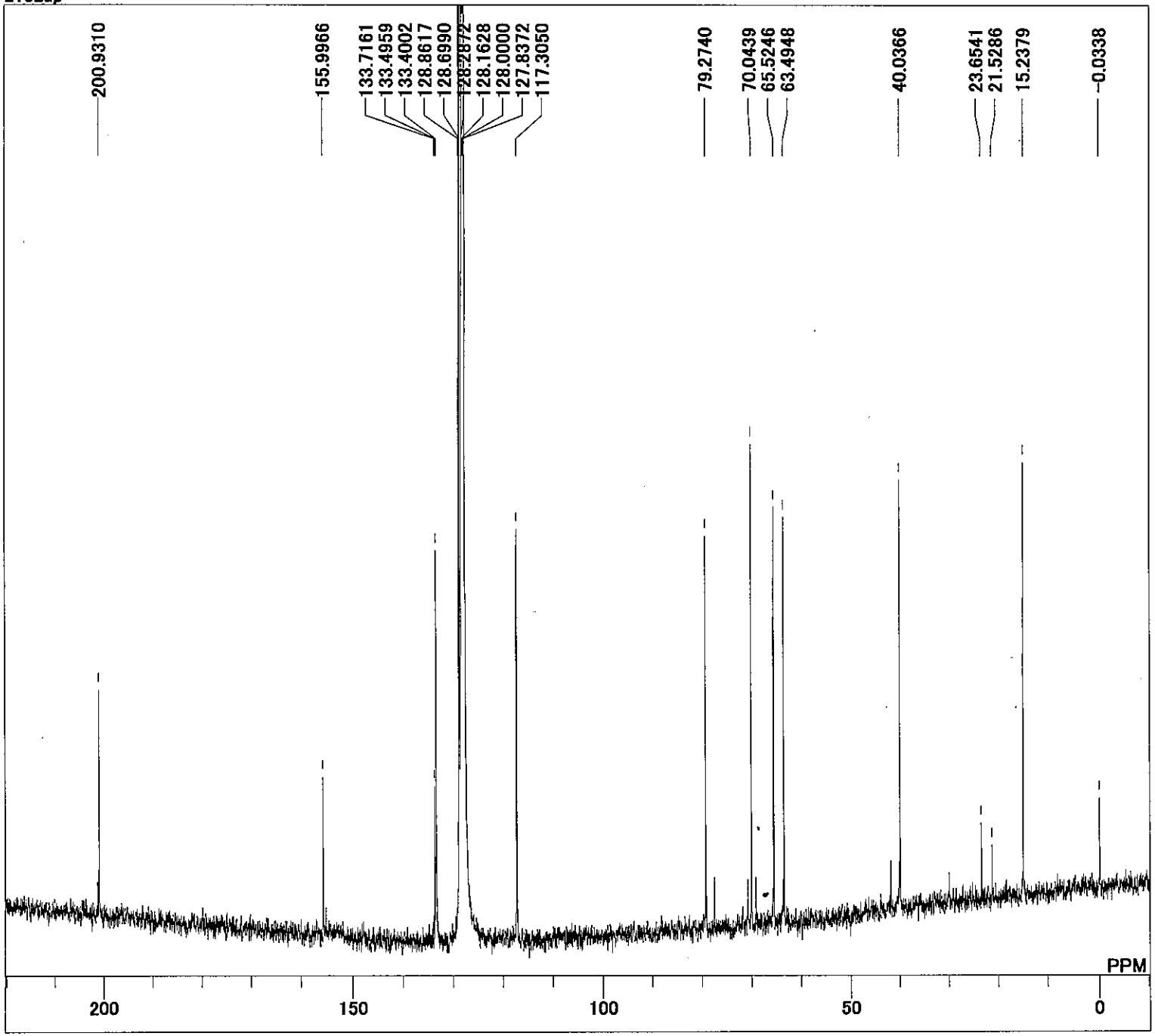
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2182up_PROTON-2.als
2182up



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2182up
DATIM	14-11-2005 23:20:06
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	C6D6
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44

3bb-up

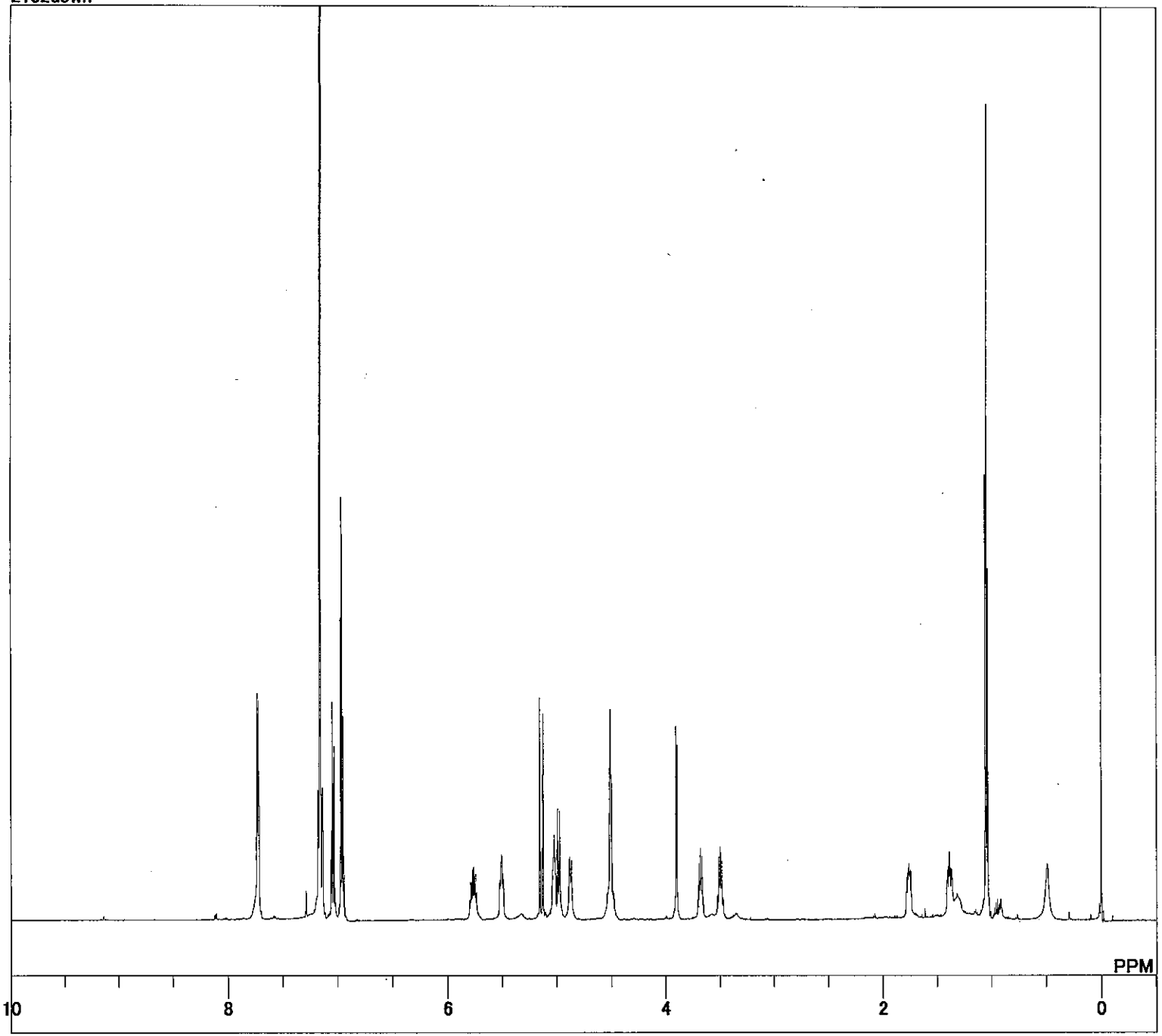
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2182up_CARBONinC6D6.als
2182up



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2182up
DATIM	15-11-2005 03:05:06
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	5000
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	C6D6
EXREF	128.00 ppm
BF	0.12 Hz
RGAIN	56

3bb-up

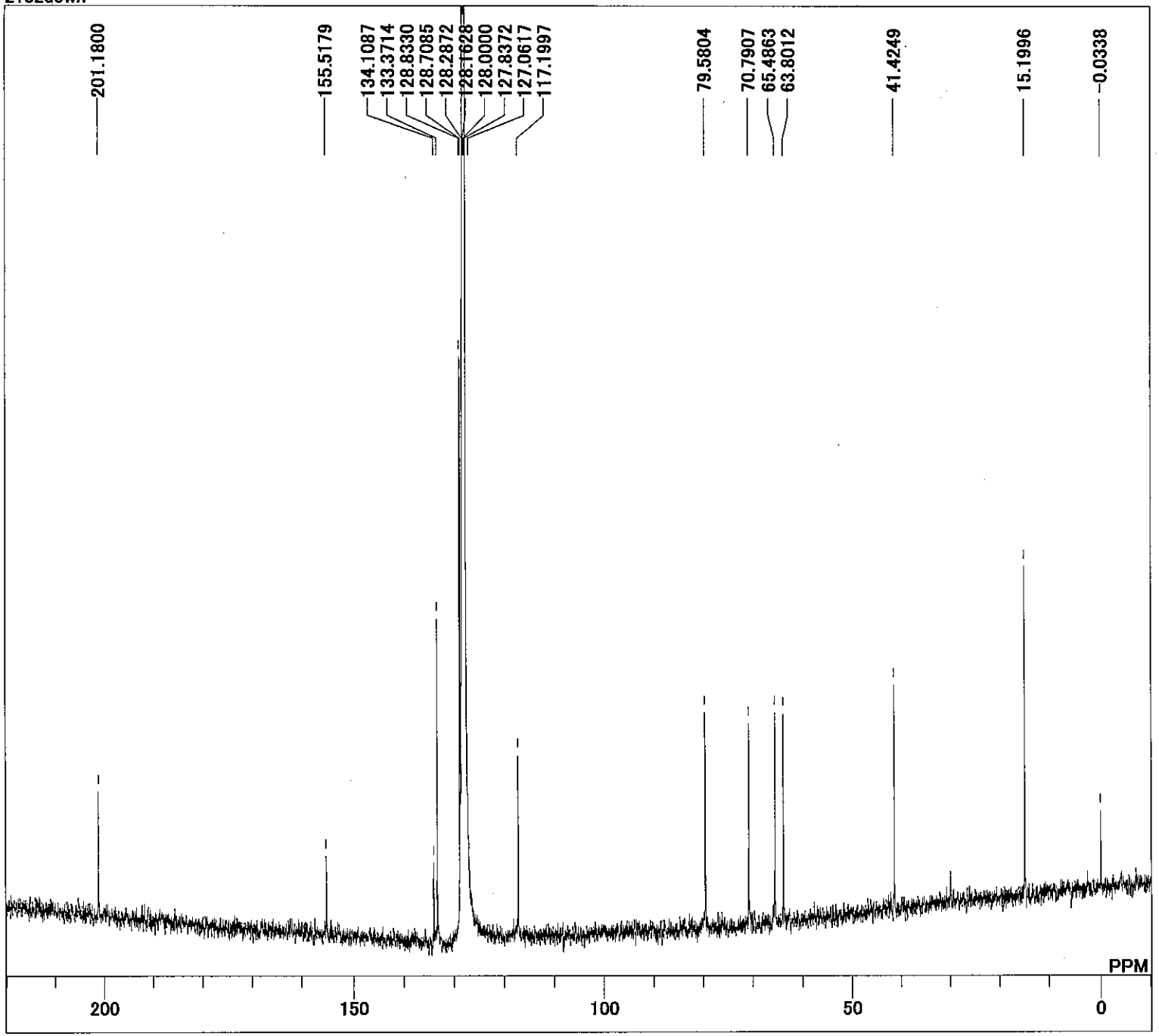
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2182down_PROTONinC6D6.als
2182down



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2182down
DATIM	15-11-2005 03:15:05
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	C6D6
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	46

3hb-down

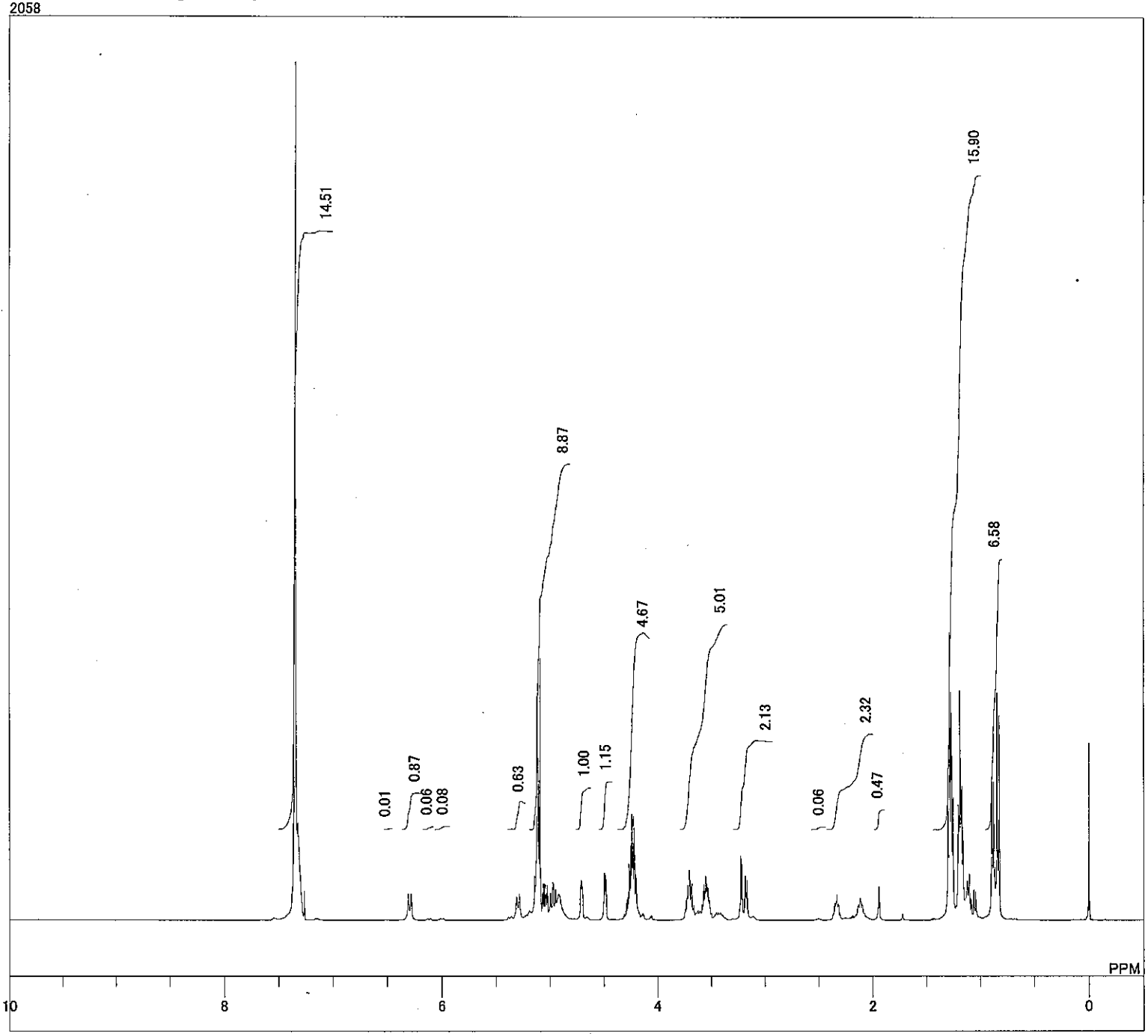
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2182down_CARBON.als
2182down



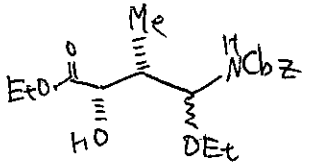
DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2182down
DATIM	15-11-2005 07:22:30
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	5500
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	C6D6
EXREF	128.00 ppm
BF	0.12 Hz
RGAIN	56

3bb-down

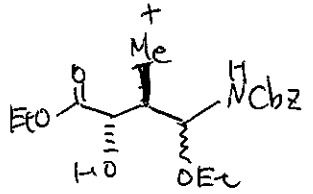
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2058.als
2058



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2058
DATIM	23-06-2005 09:47:16
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28

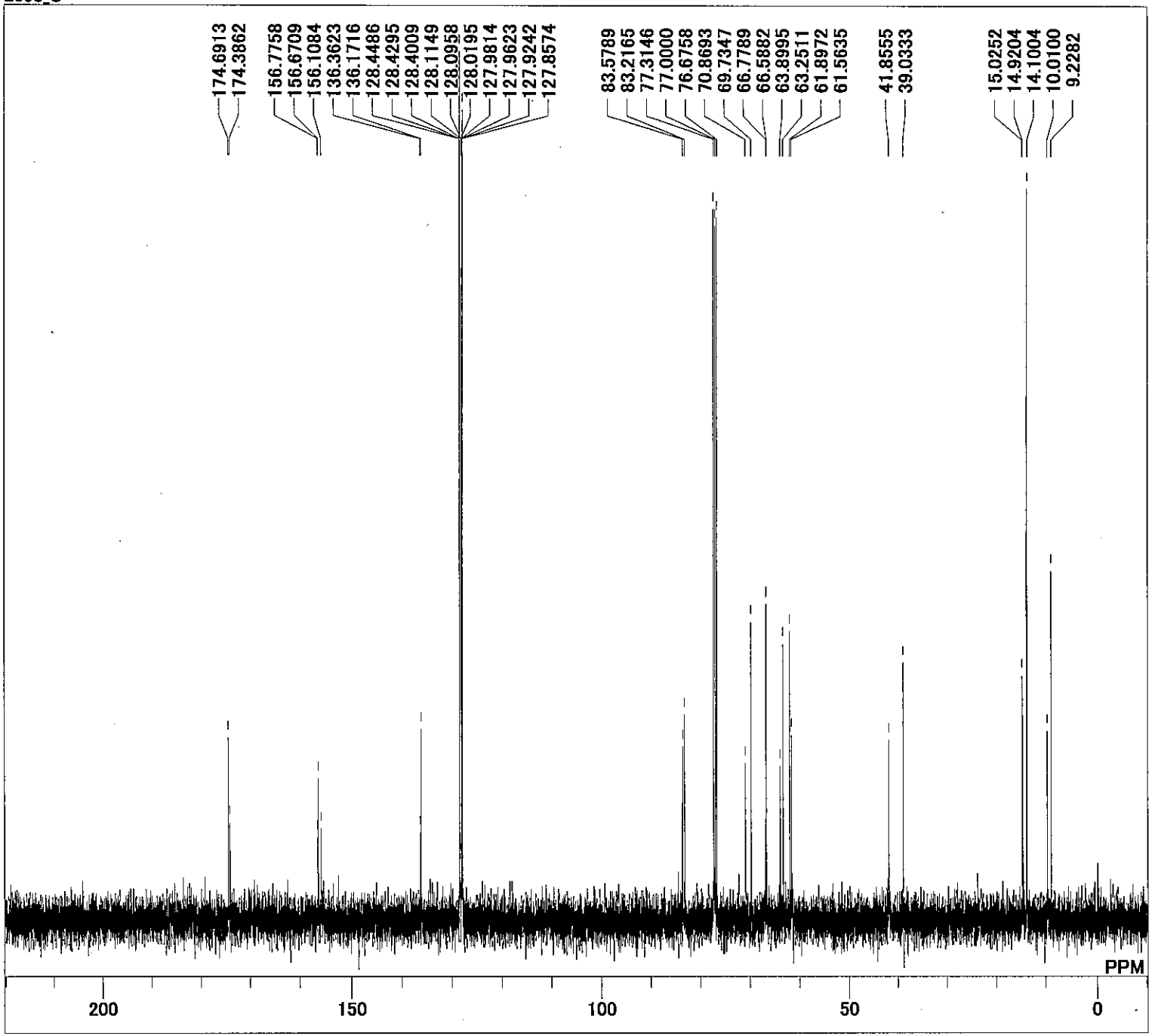


major

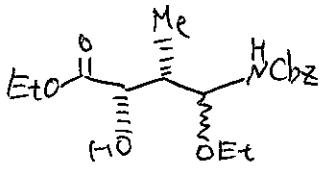


minor

E:¥Matsubara¥2058_C.als
2058_C

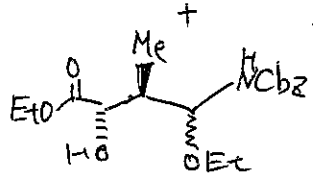


DFILE	E:¥Matsubara¥2058_C.als
COMNT	2058_C
DATIM	23-06-2005 09:52:21
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	84
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54



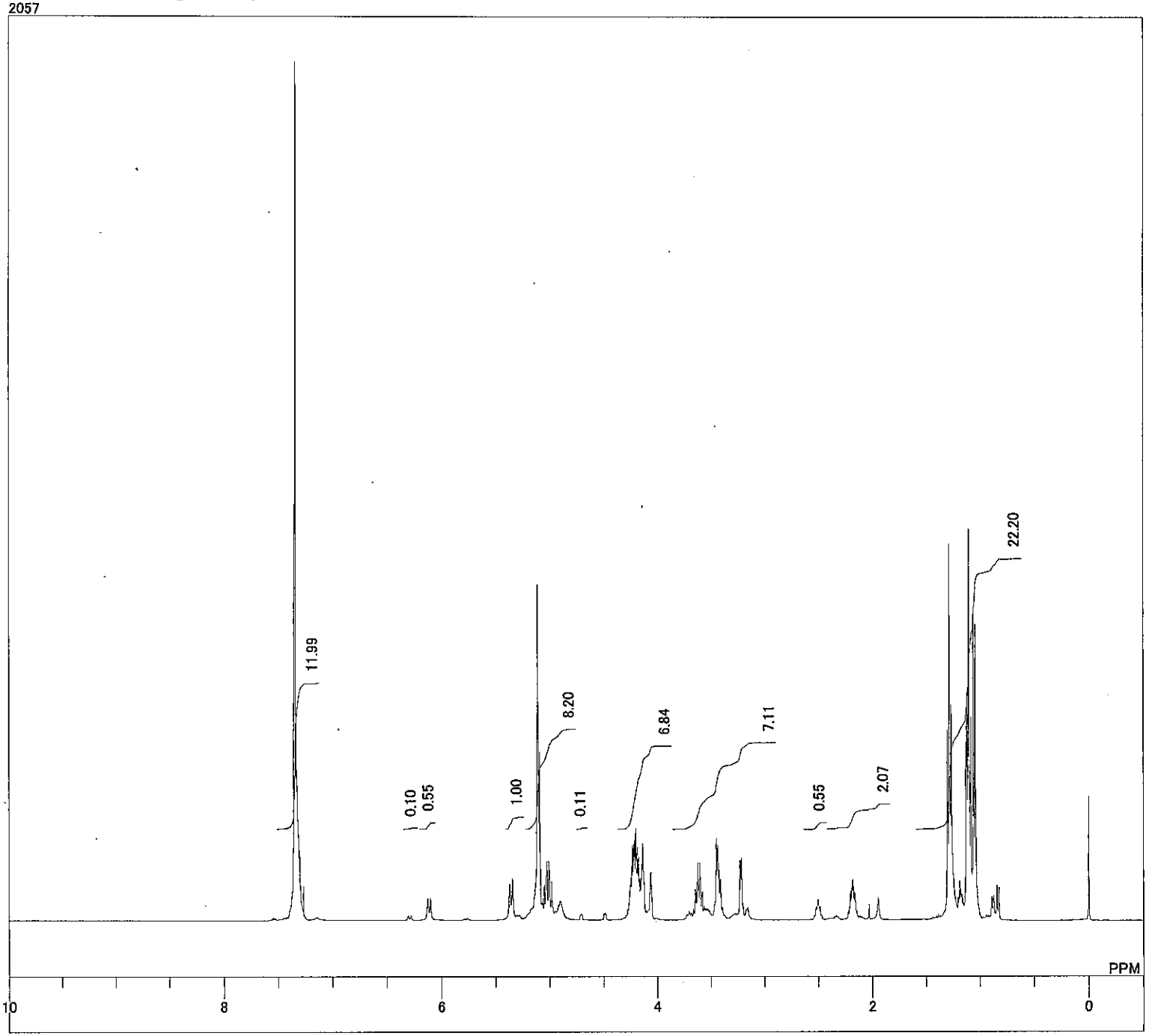
major

+

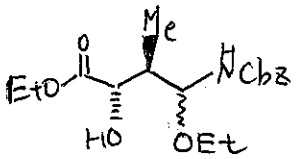


minor

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2057.als
2057

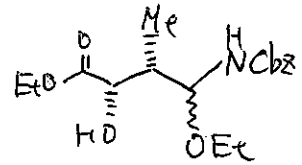


DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2057
DATIM	23-06-2005 09:17:01
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28



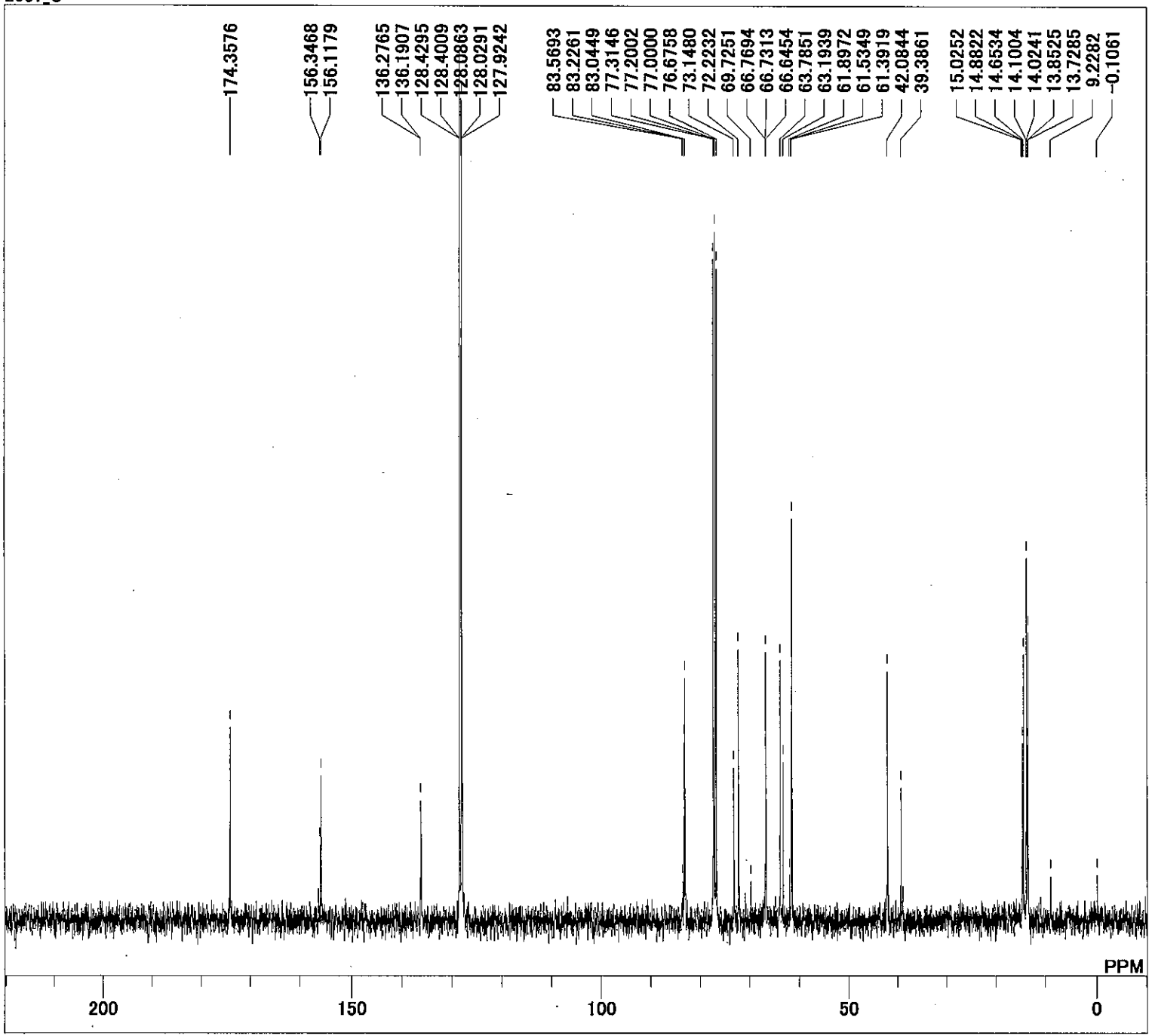
major

+

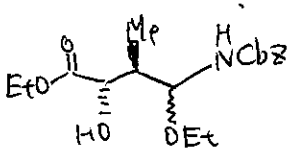


minor

E:¥Matsubara¥2057_C.als
2057_C

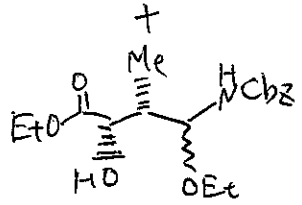


DFILE	E:¥Matsubara¥2057_C.als
COMNT	2057_C
DATIM	23-06-2005 09:25:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	74
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56



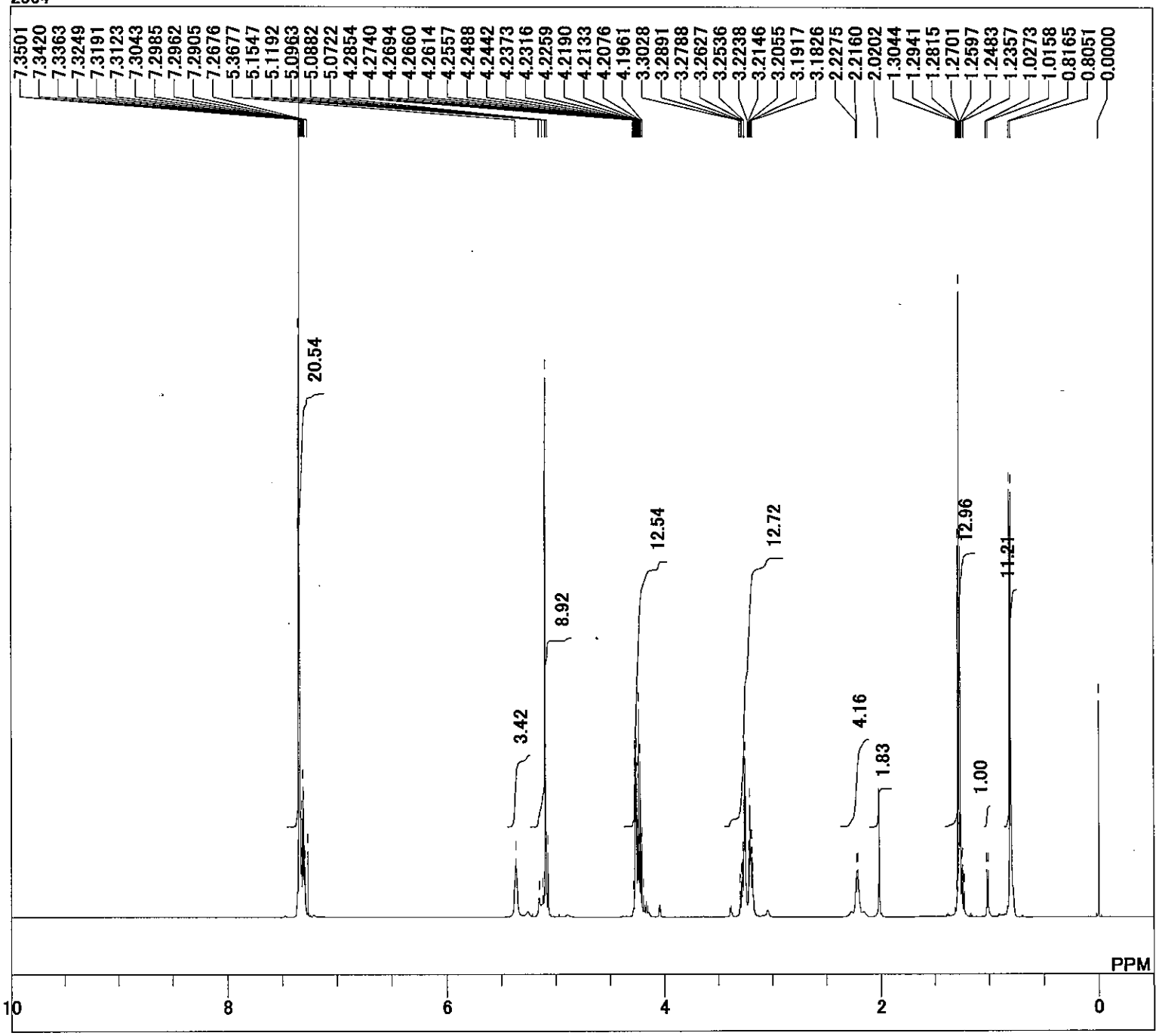
major

+



minor

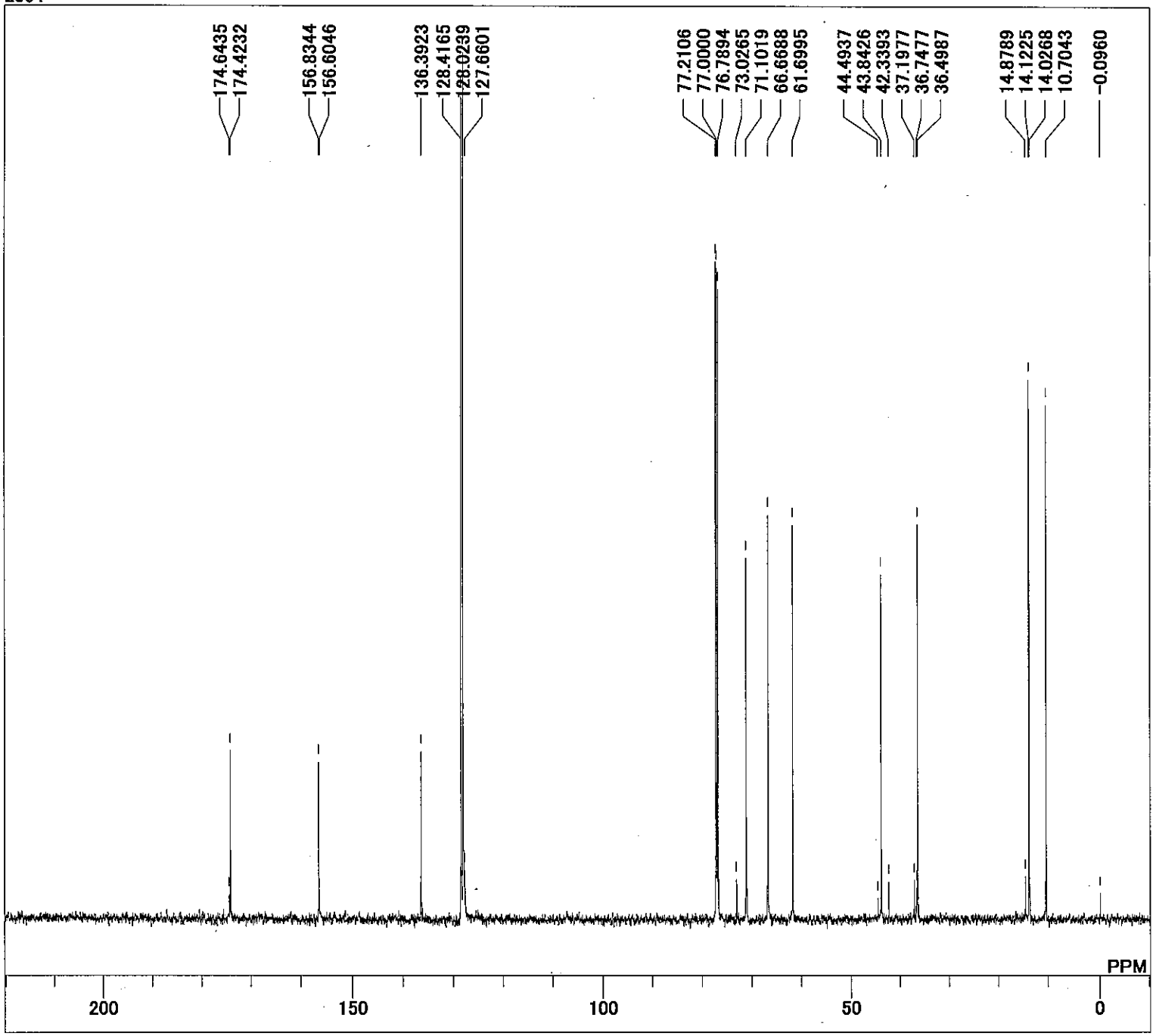
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2064_PROTON.als
2064



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2064
DATIM	27-06-2005 14:31:30
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34

syn - 7c

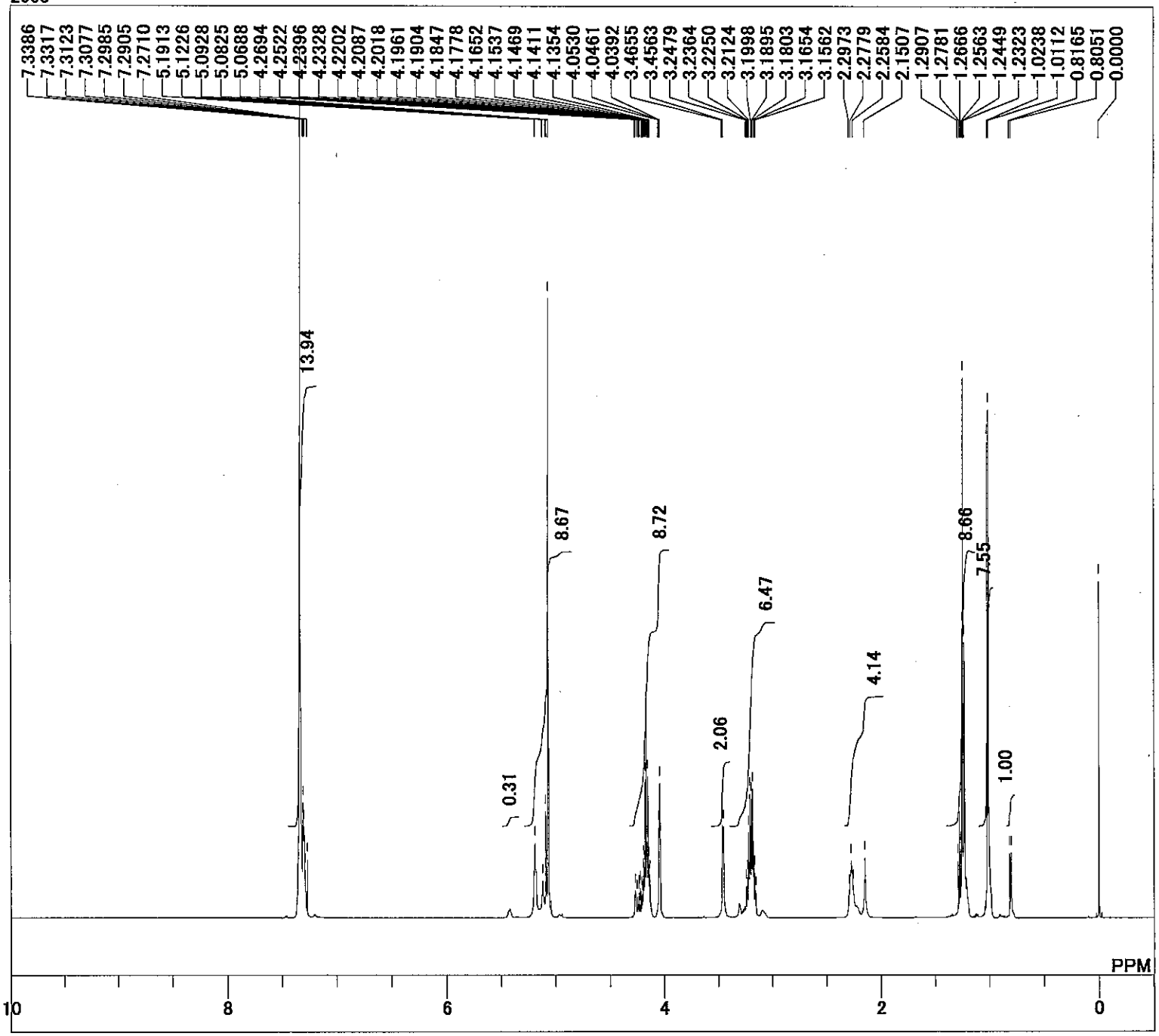
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2064_CARBON.als
2064



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2064
DATIM	27-06-2005 14:43:43
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

syn-7c

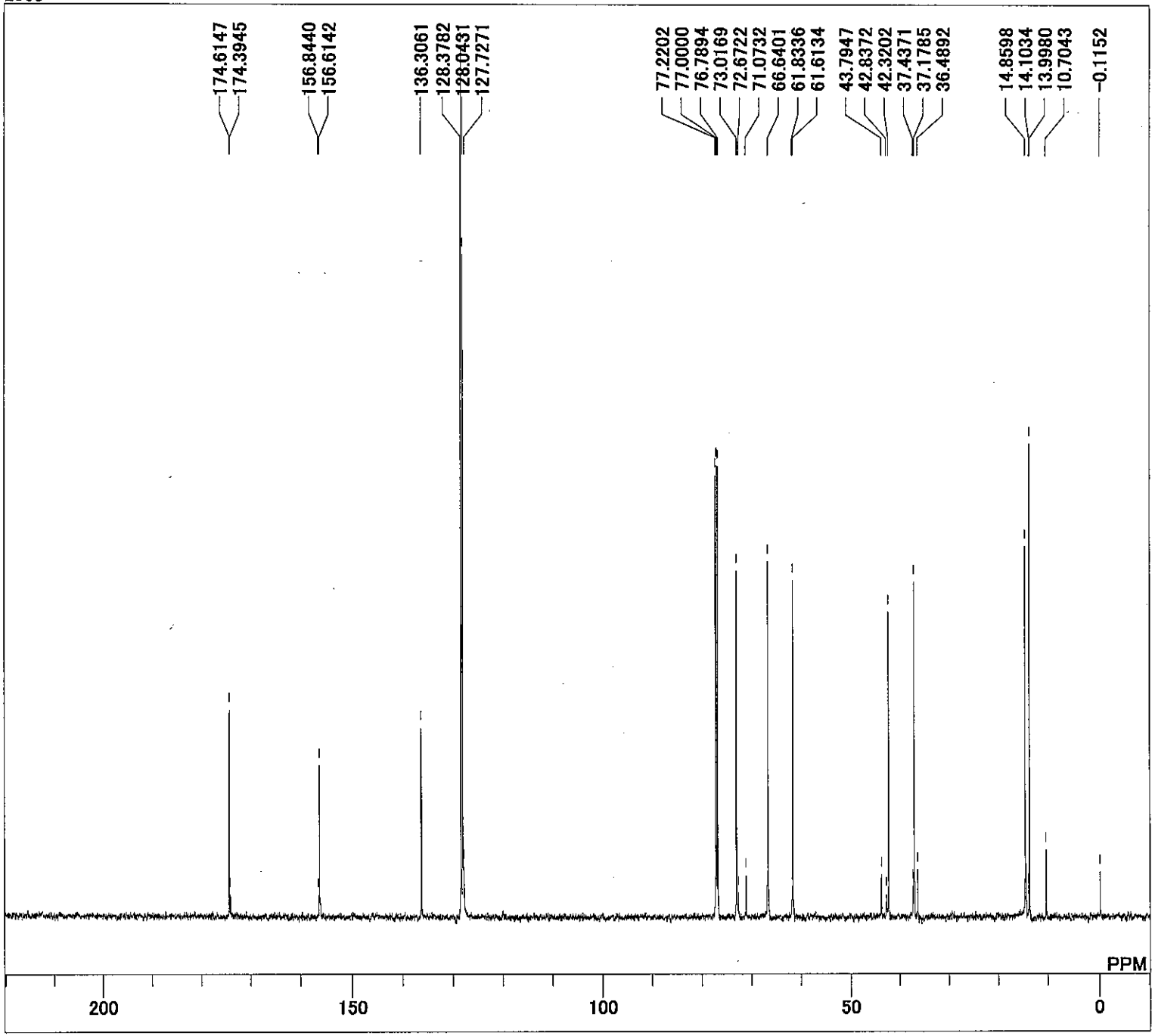
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2063_PROTON.als
2063



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2063
DATIM	27-06-2005 14:07:48
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34

anti - 7c

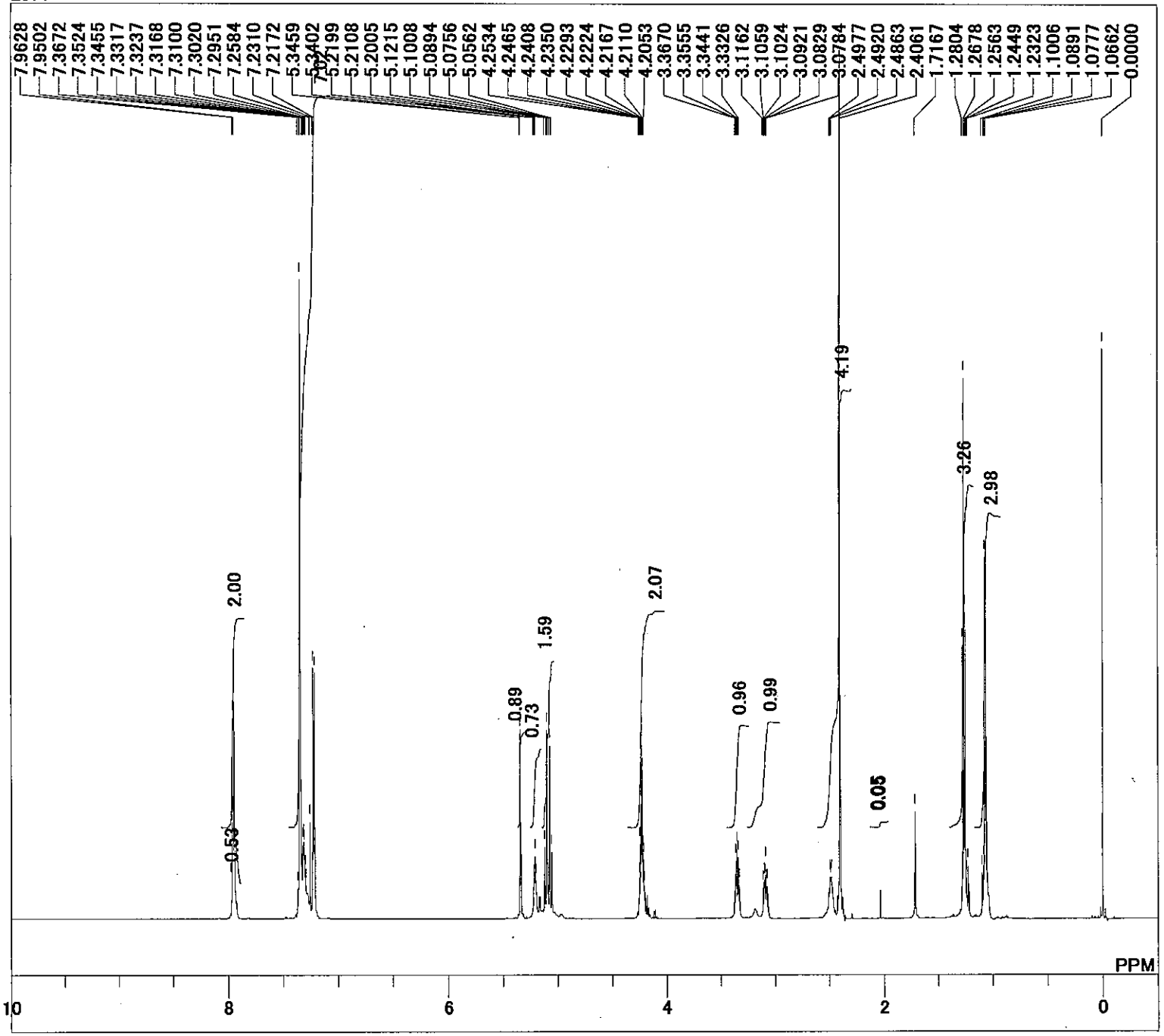
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2063_CARBON.als
2063



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2063
DATIM	27-06-2005 14:21:13
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

anti-7c

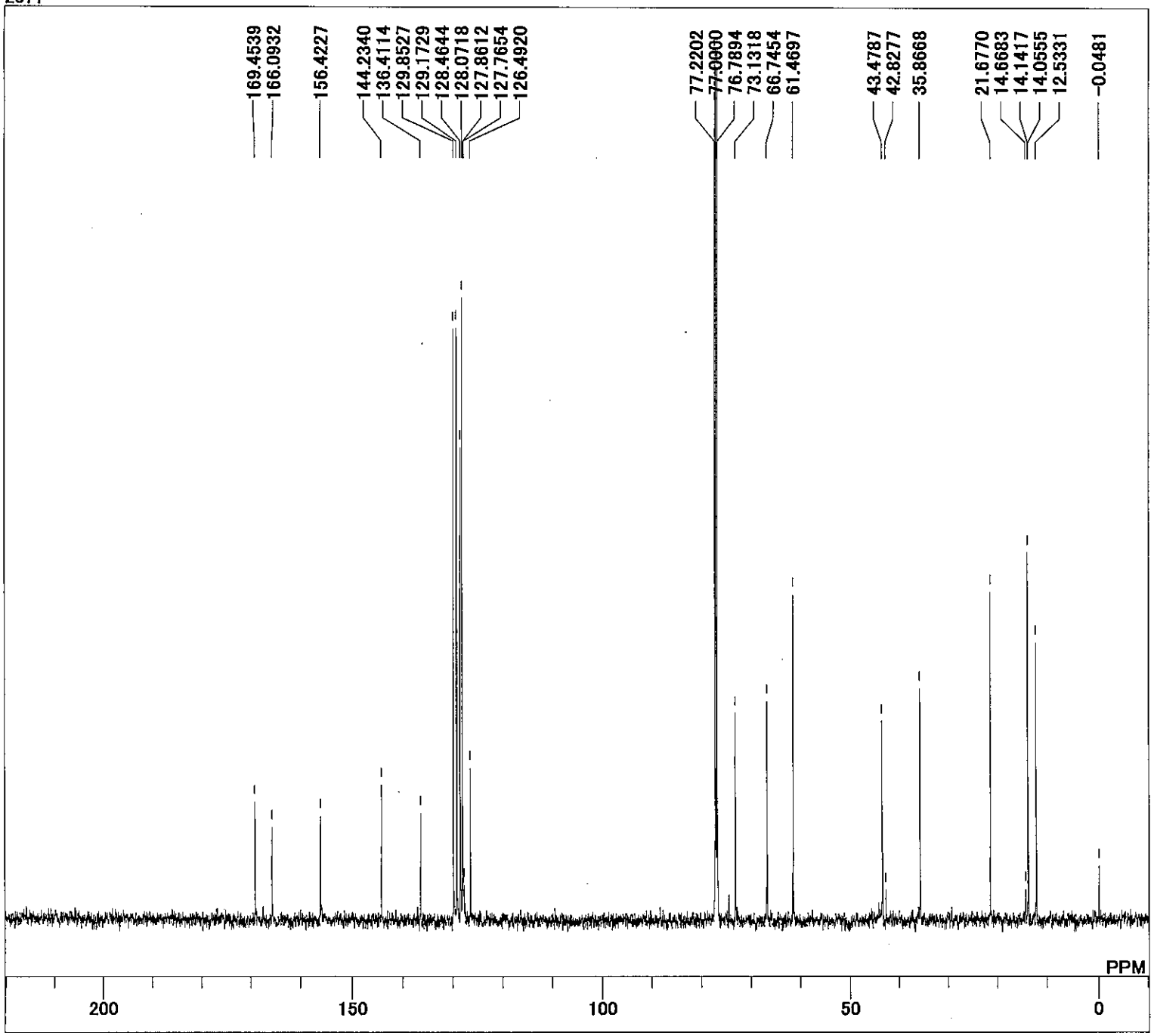
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2071_PROTON.als
2071



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2071
DATIM	28-06-2005 20:08:12
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

syn-6c

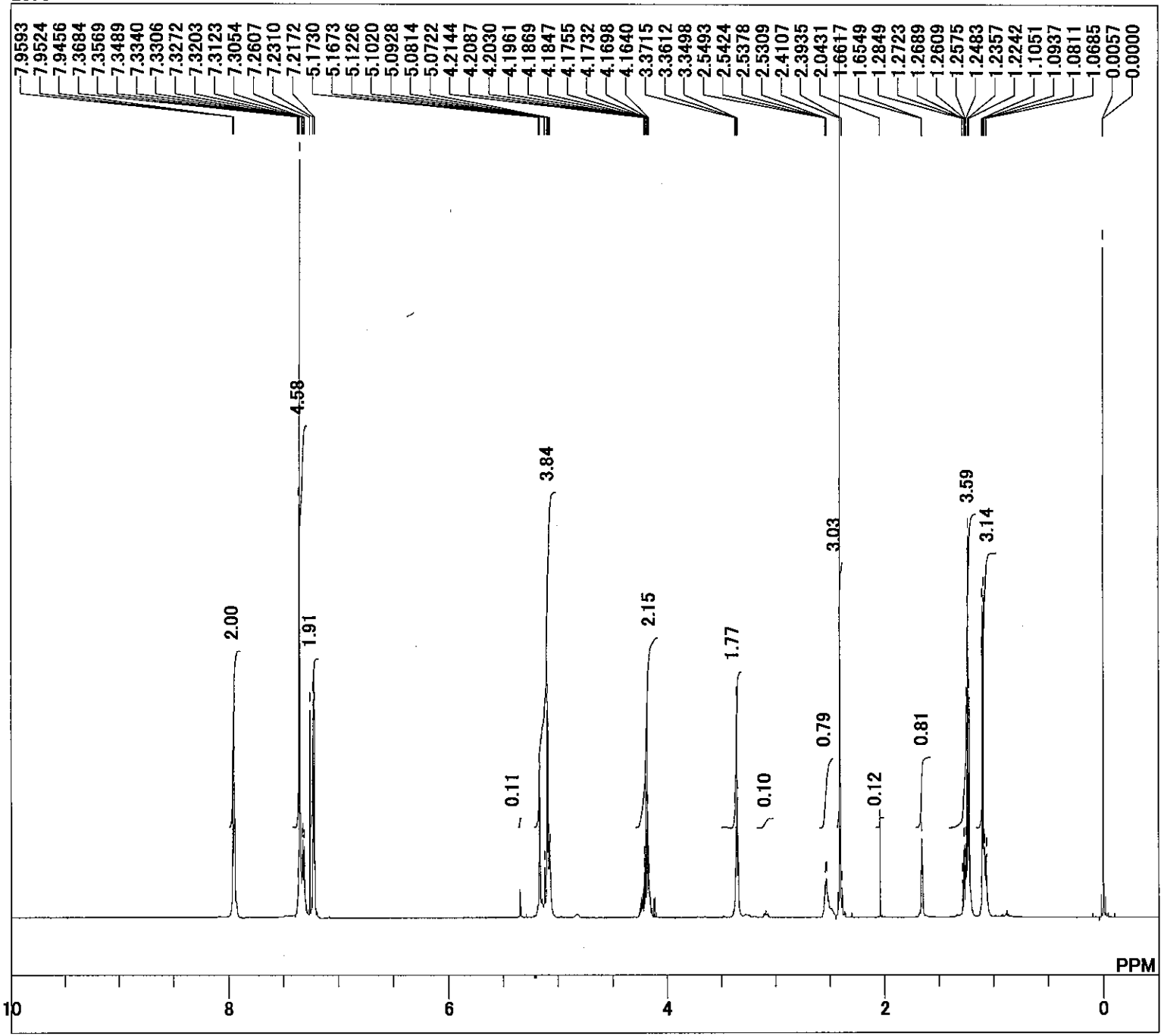
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2071_CARBON.als
2071



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2071
DATIM	28-06-2005 20:20:27
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

syn-6c

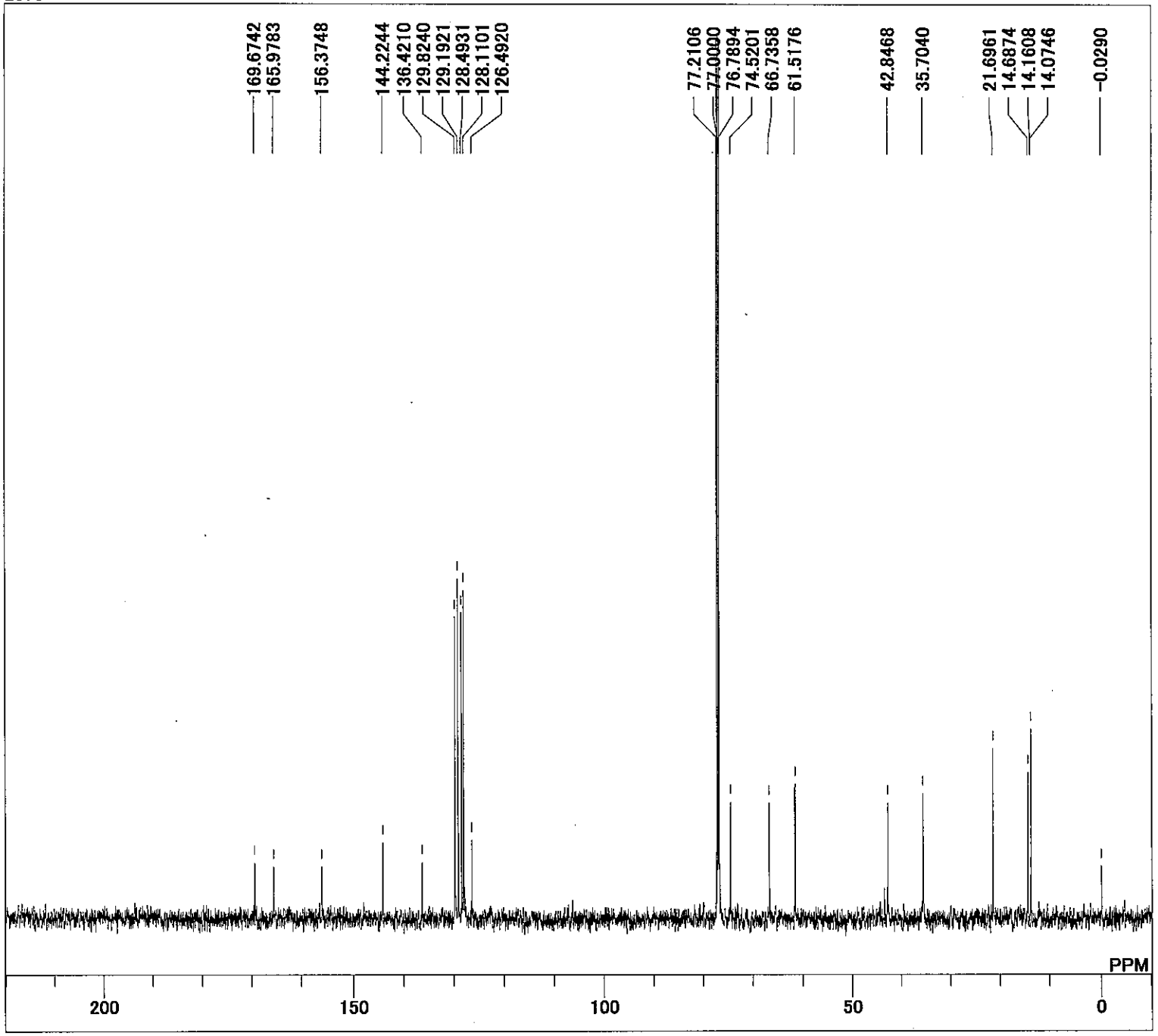
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2070_PROTON.als
2070



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2070
DATIM	28-06-2005 19:51:42
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	13107
FREQU	9008.87 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

anti-6c

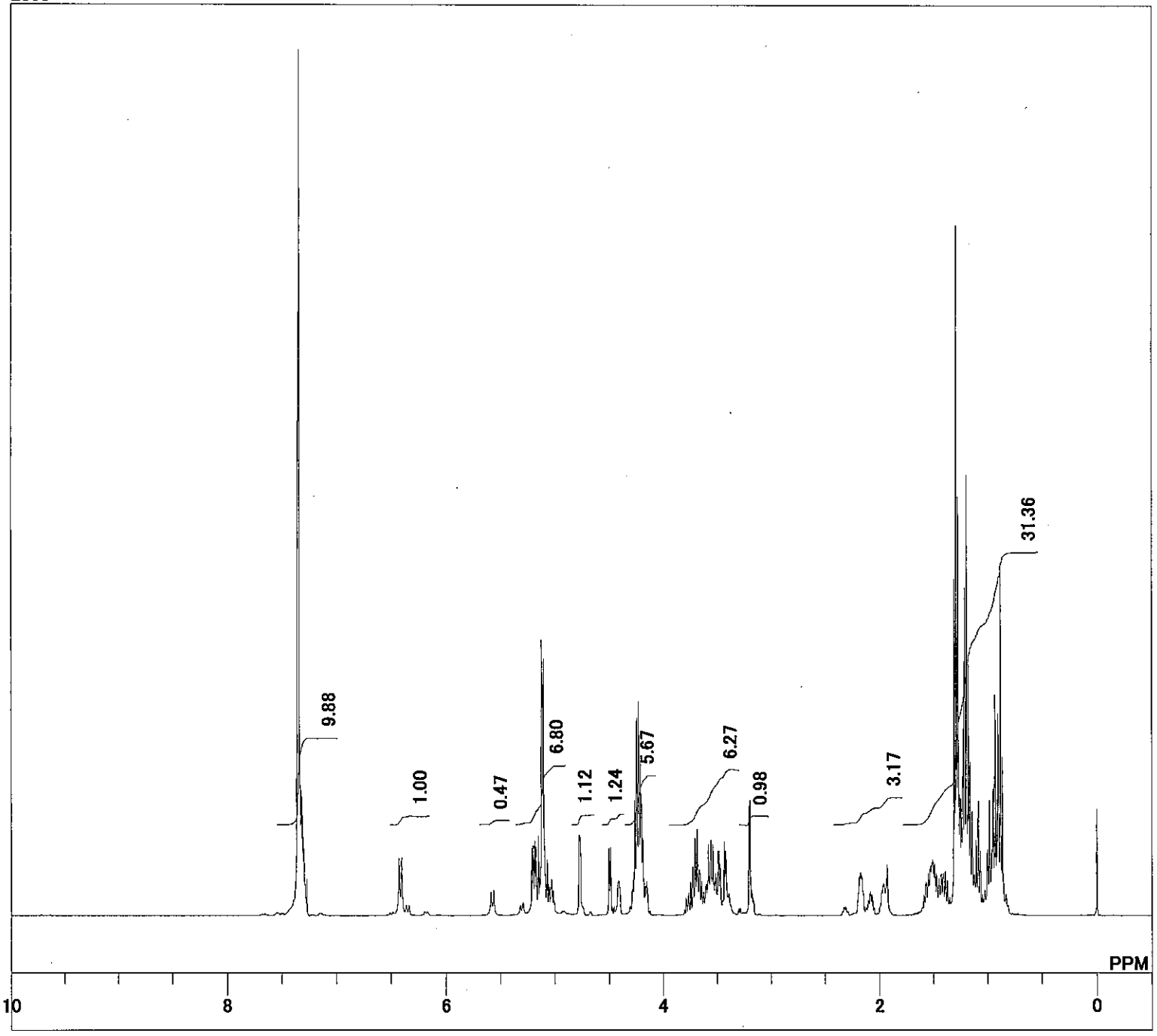
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2070_CARBON.als
2070



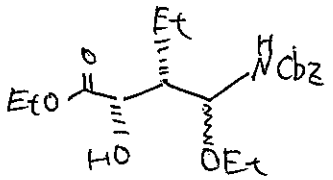
DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2070
DATIM	28-06-2005 19:58:13
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

anti-6c

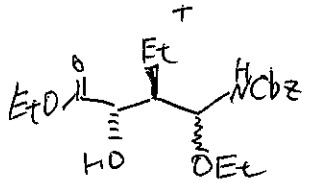
E:¥Matsubara¥2060.als
2060



DFILE	E:¥Matsubara¥2060.als
COMNT	2060
DATIM	23-06-2005 10:13:08
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28

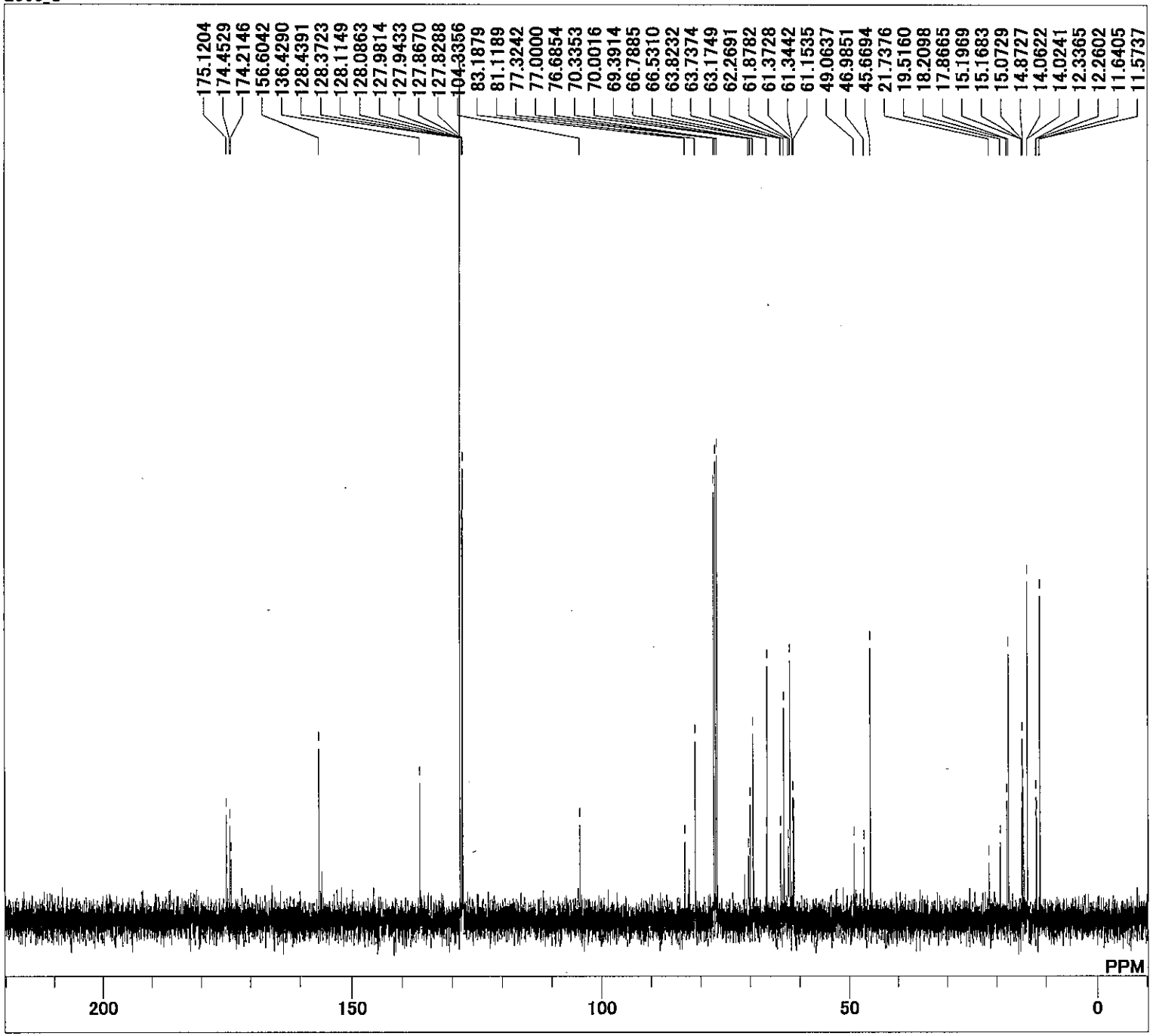


major

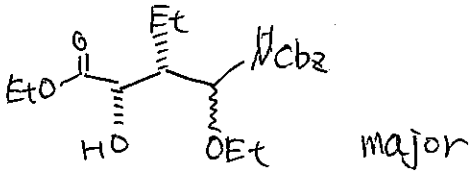


minor

E:¥Matsubara¥2060_C.als
2060_C

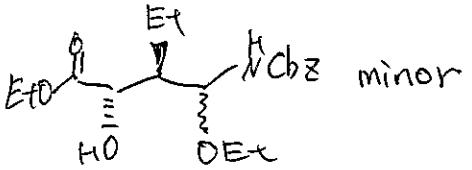


DFILE	E:¥Matsubara¥2060_C.als
COMNT	2060_C
DATIM	23-06-2005 10:16:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	42
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56



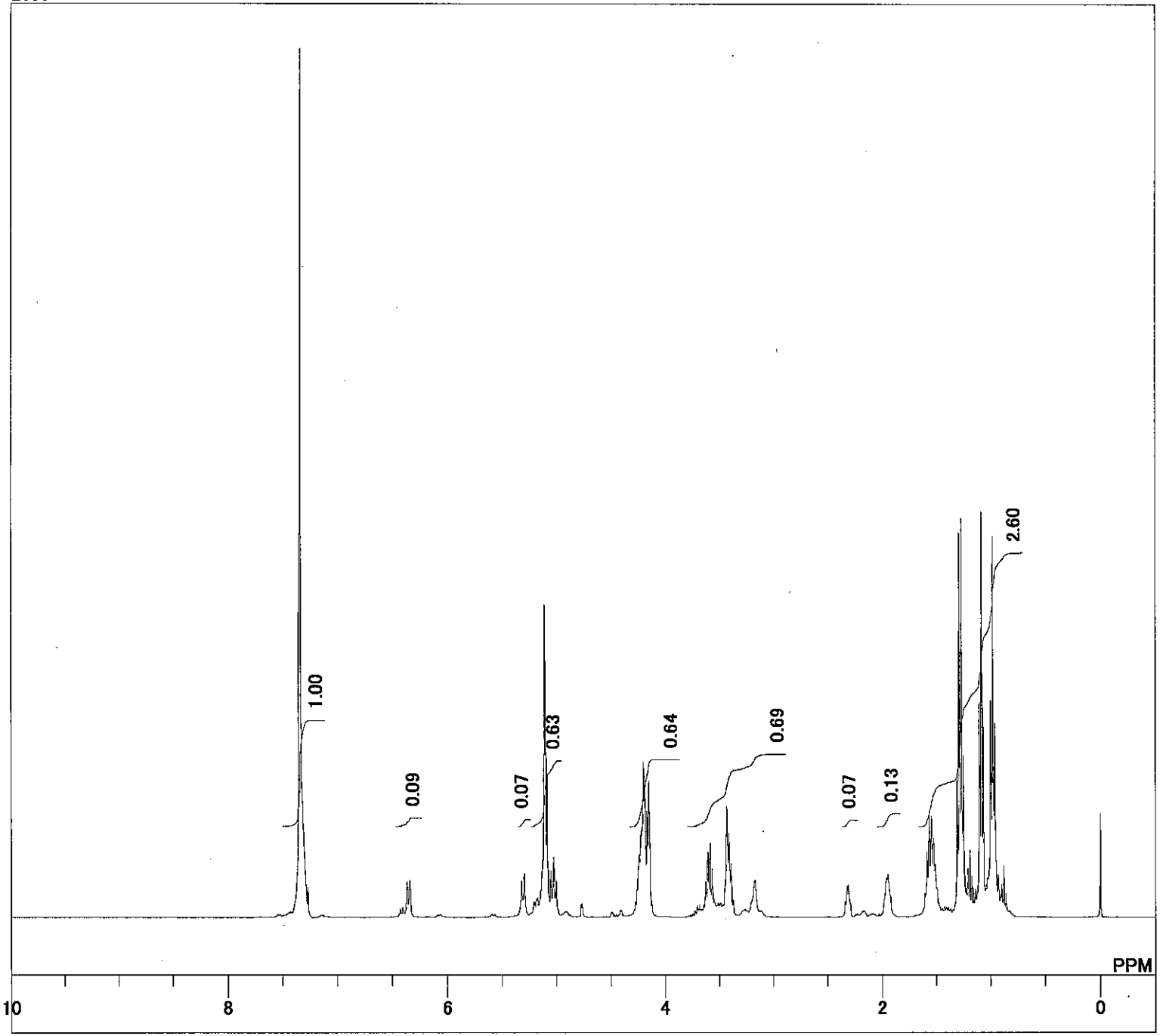
major

+

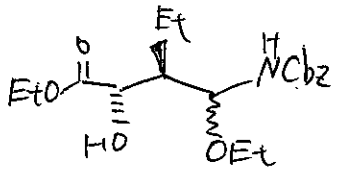


minor

E:¥Matsubara¥2059.als
2059

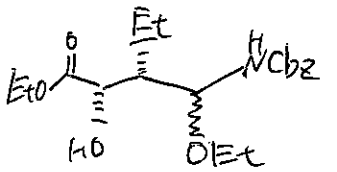


DFILE	E:¥Matsubara¥2059.als
COMNT	2059
DATIM	23-06-2005 09:28:40
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28



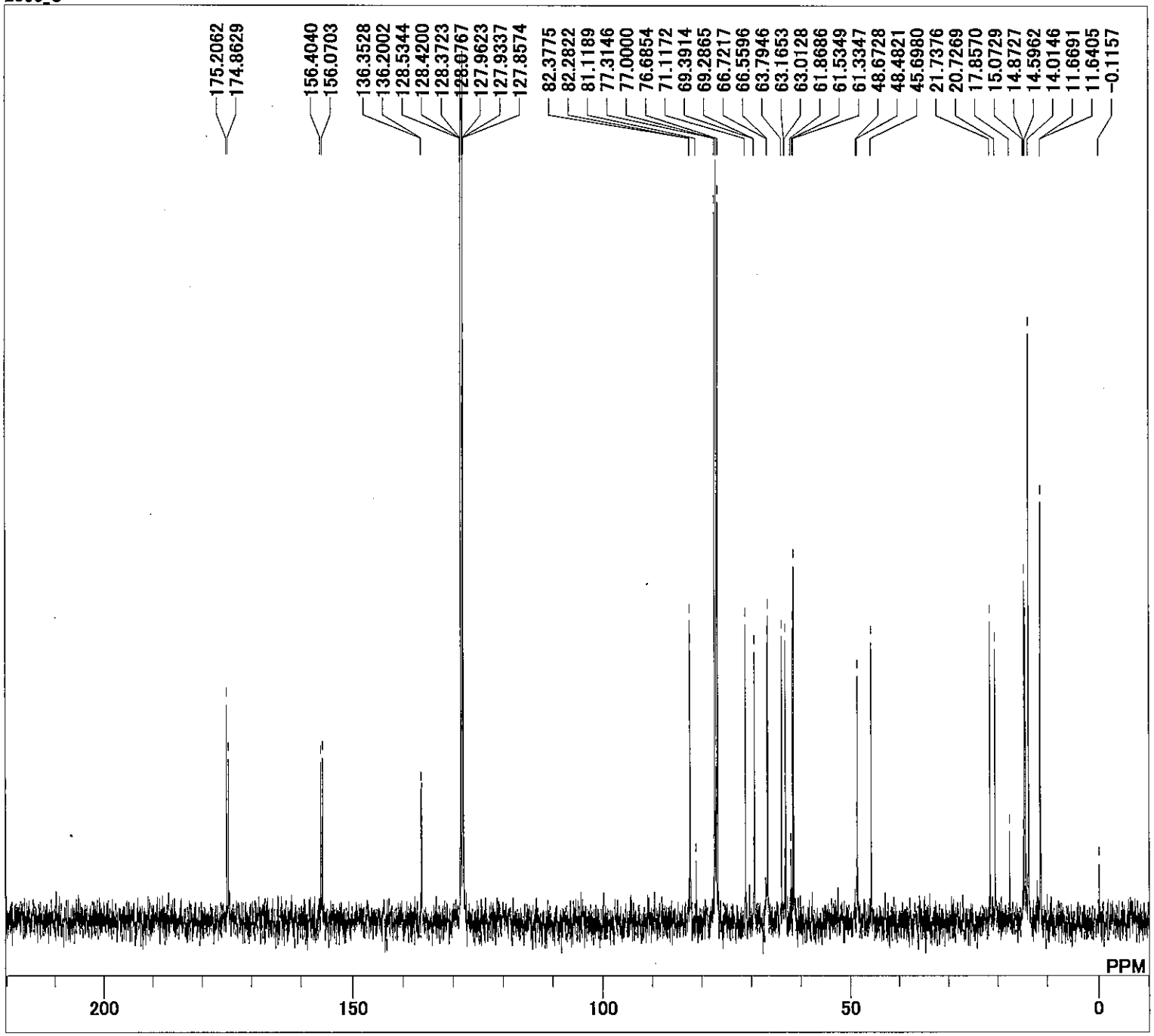
major

+

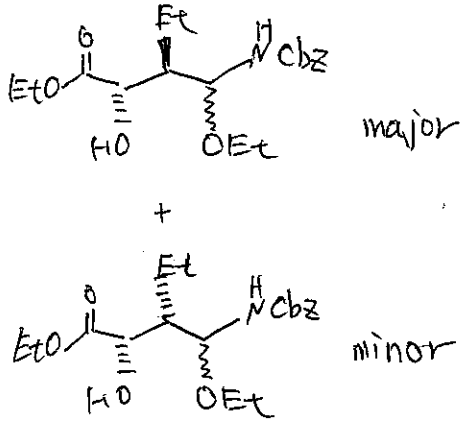


minor

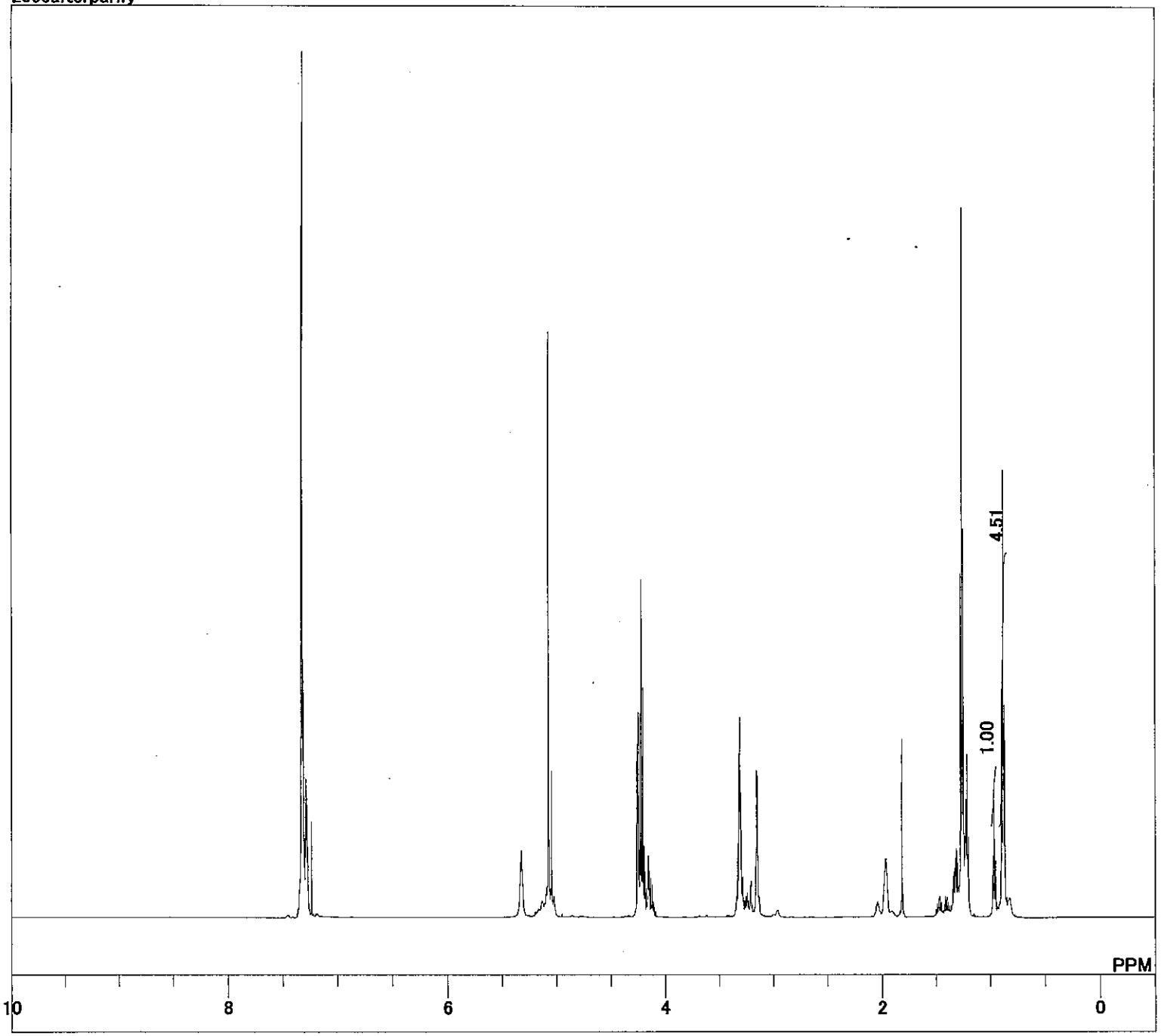
E:¥Matsubara¥2059_C.als
2059_C



DFILE	E:¥Matsubara¥2059_C.als
COMNT	2059_C
DATIM	23-06-2005 09:32:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	60
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	52



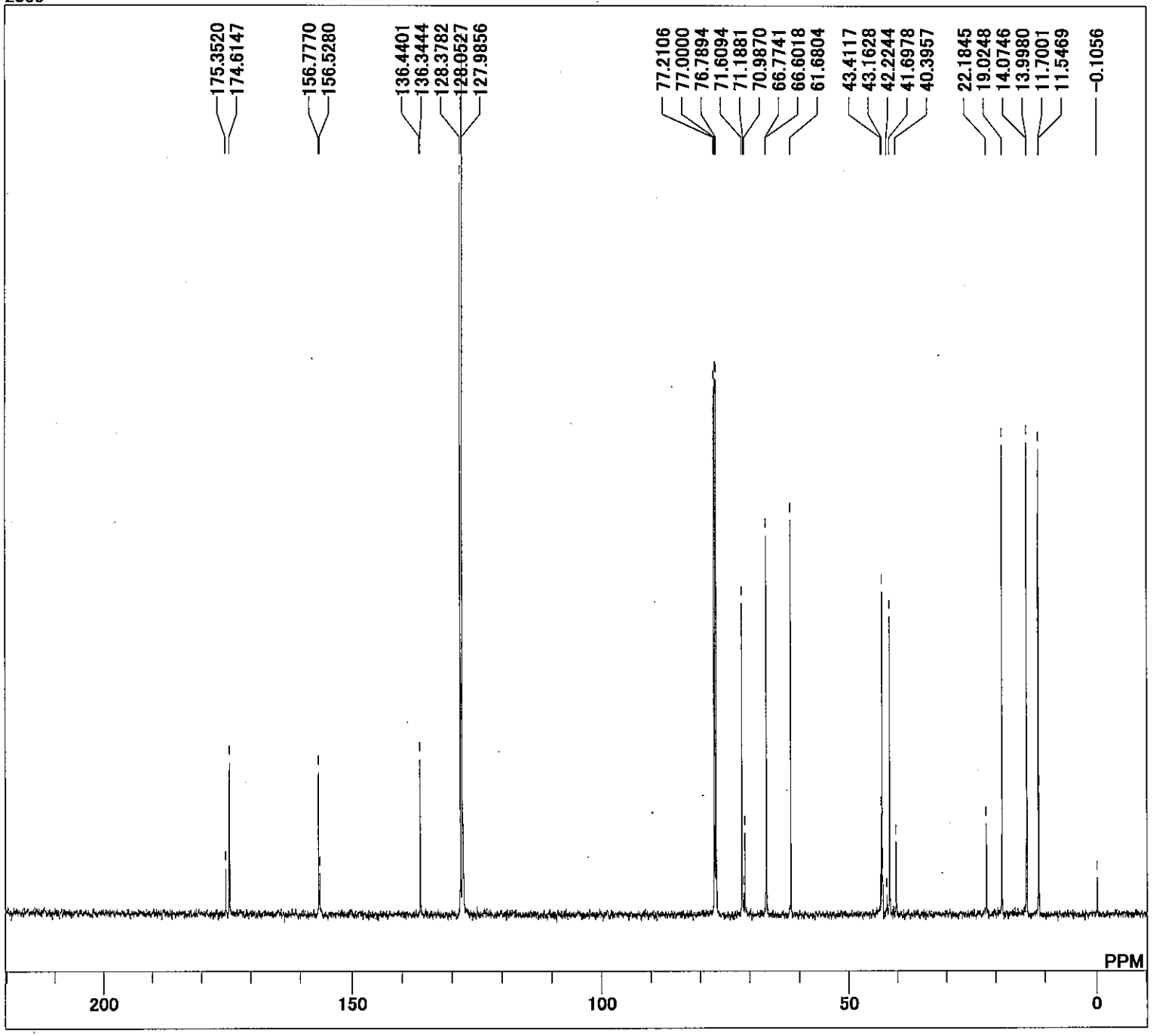
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2066afterpurify_PROTON.als
2066afterpurify



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2066afterpurify
DATIM	28-06-2005 19:18:43
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	12.50 ppm
BF	0.12 Hz
RGAIN	38

syn-7d

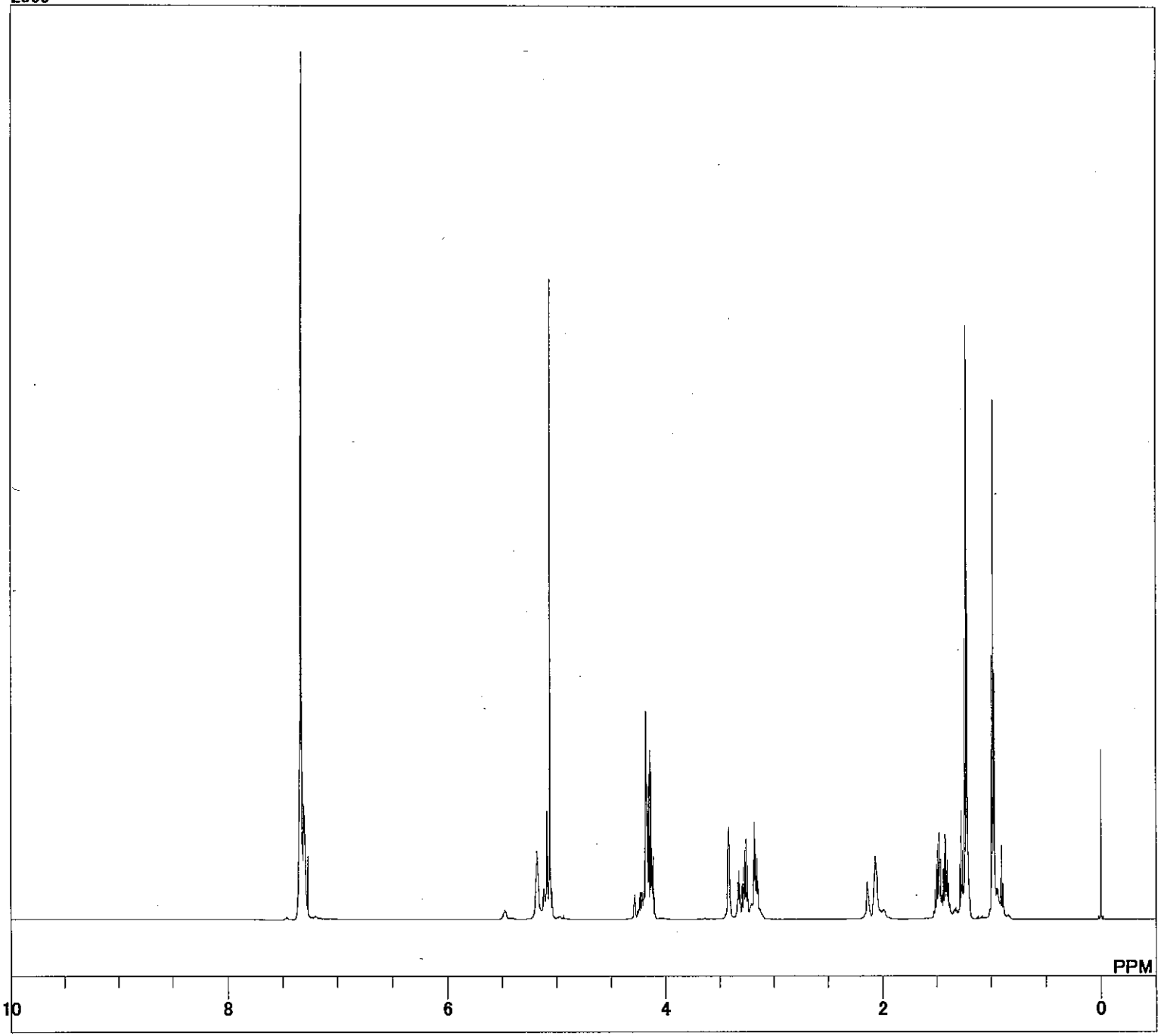
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2066_CARBON.als
2066



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2066
DATIM	27-06-2005 15:29:10
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

syn-7d

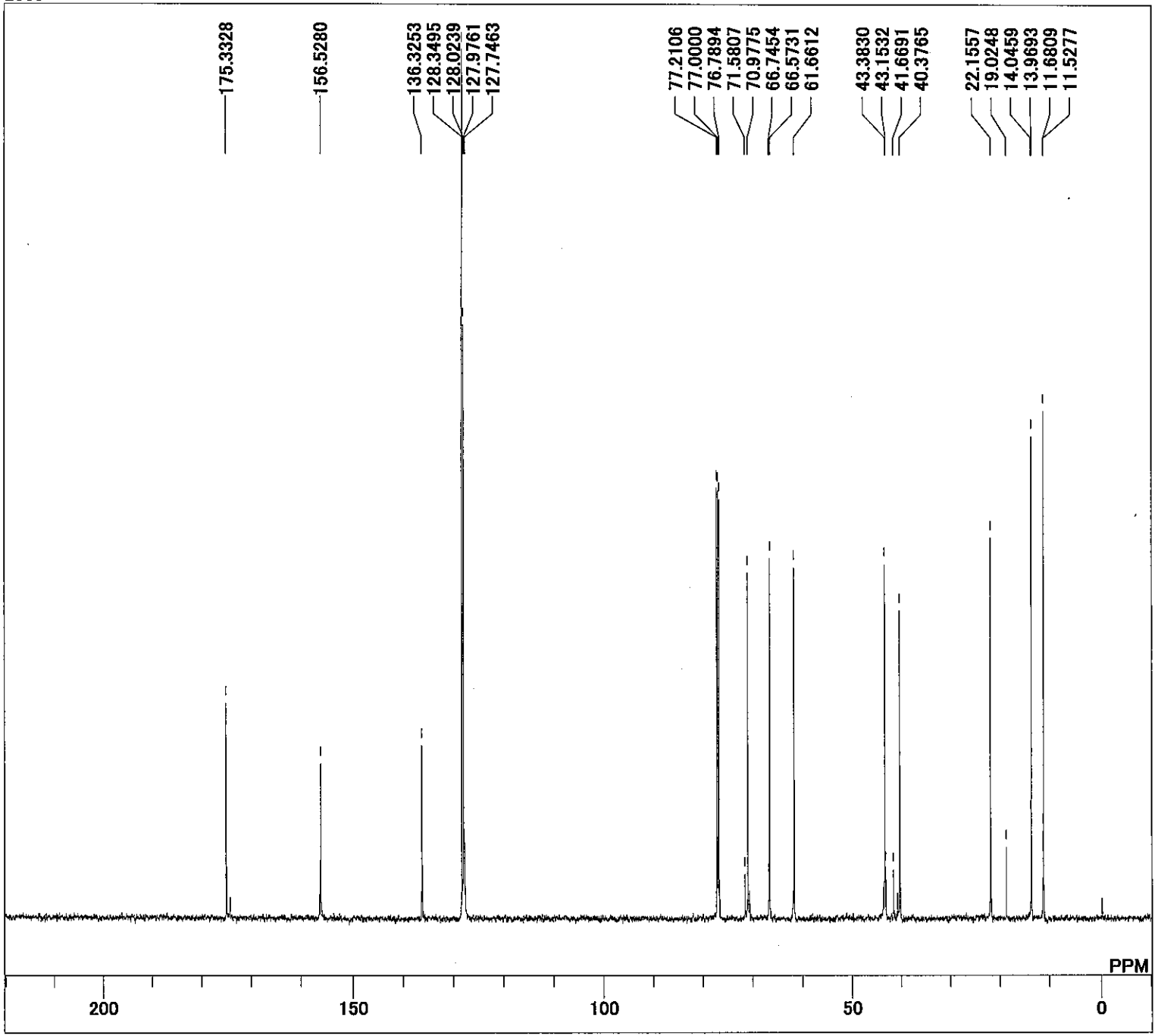
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2065_PROTON.als
2065



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2065
DATIM	27-06-2005 14:54:10
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30

anti-7d

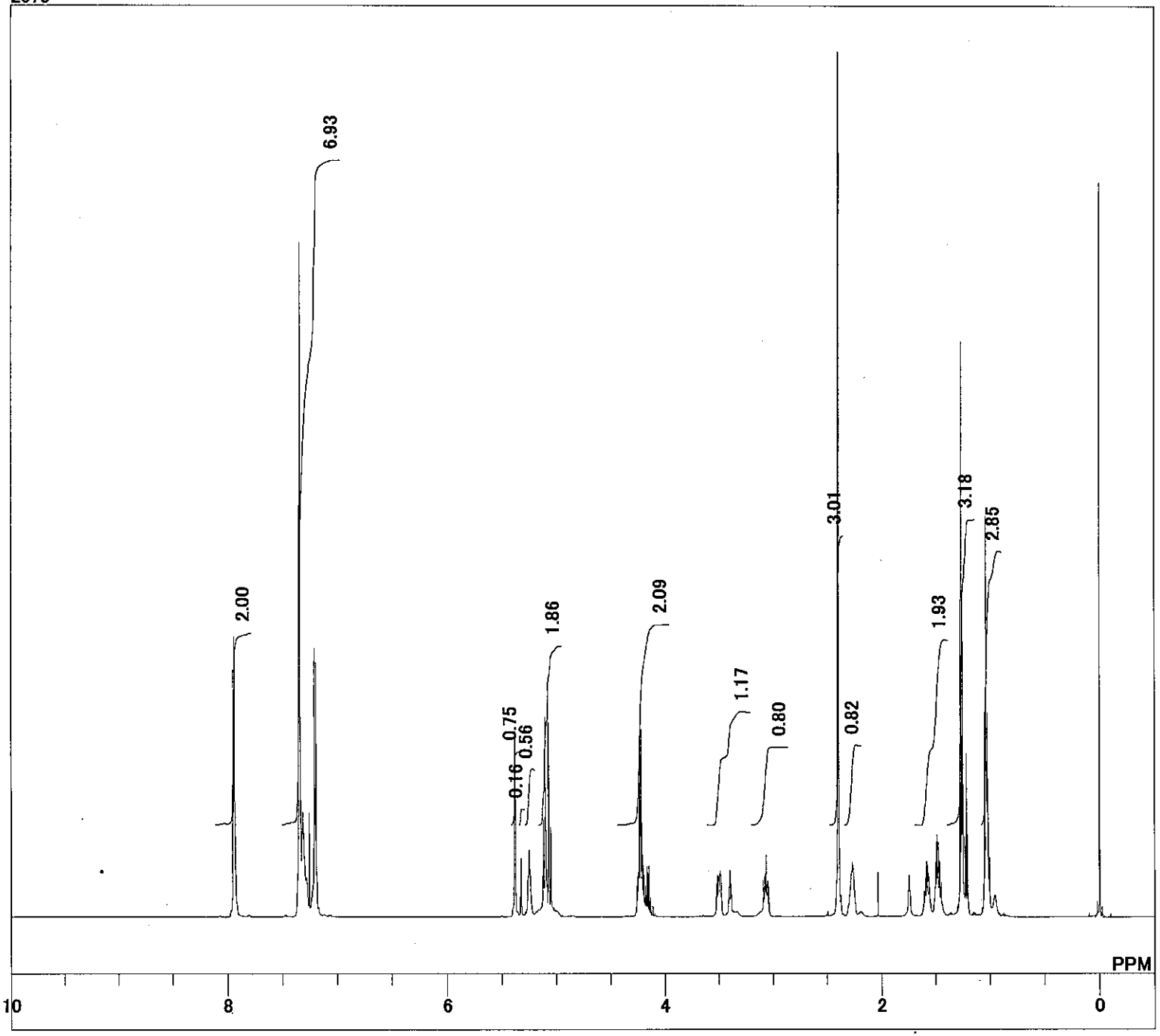
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2065_CARBON.als
2065



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2065
DATIM	27-06-2005 15:06:24
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

anti-7d

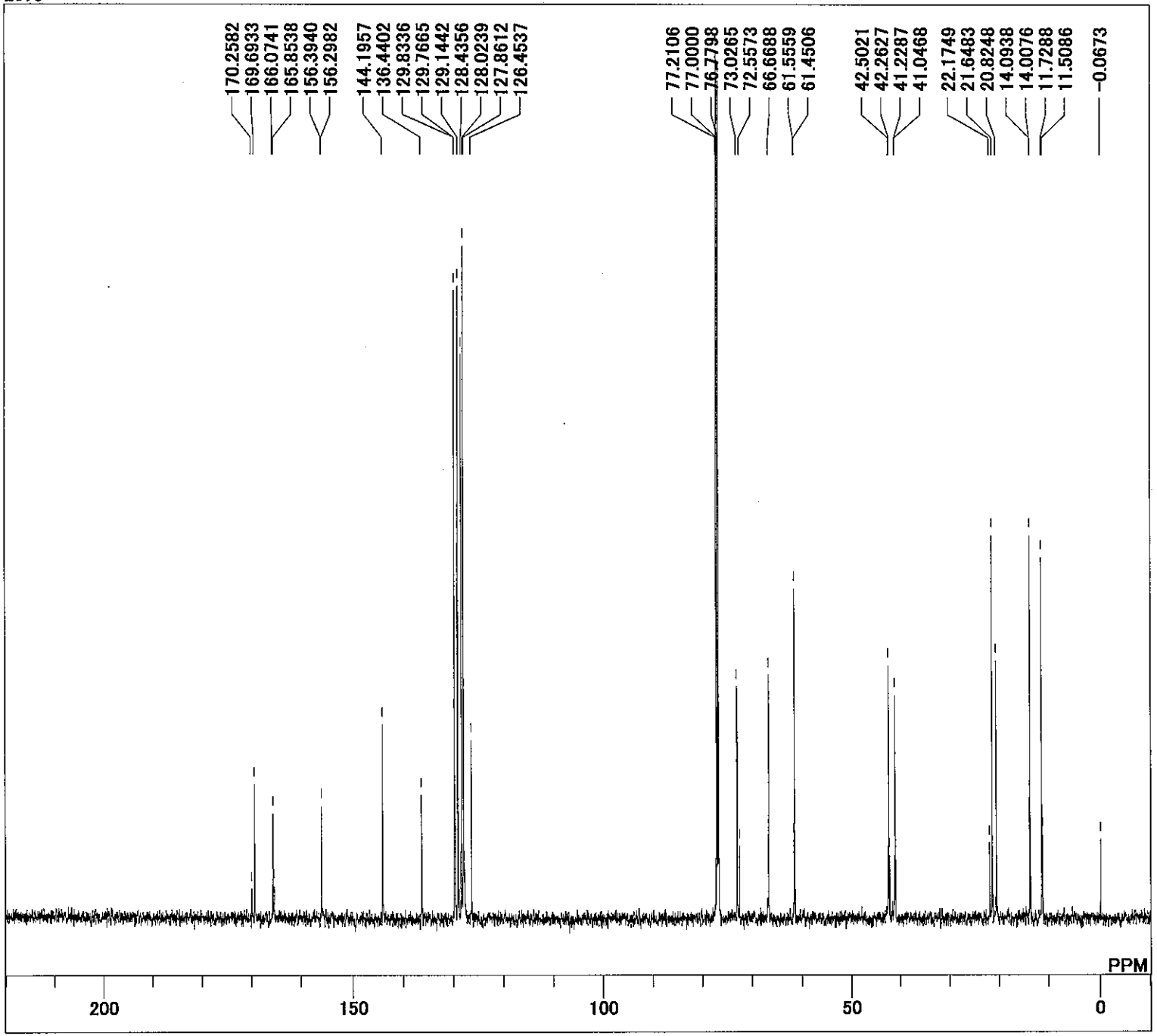
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2073_PROTON.als
2073



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2073
DATIM	28-06-2005 20:39:39
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	36

syn-6d

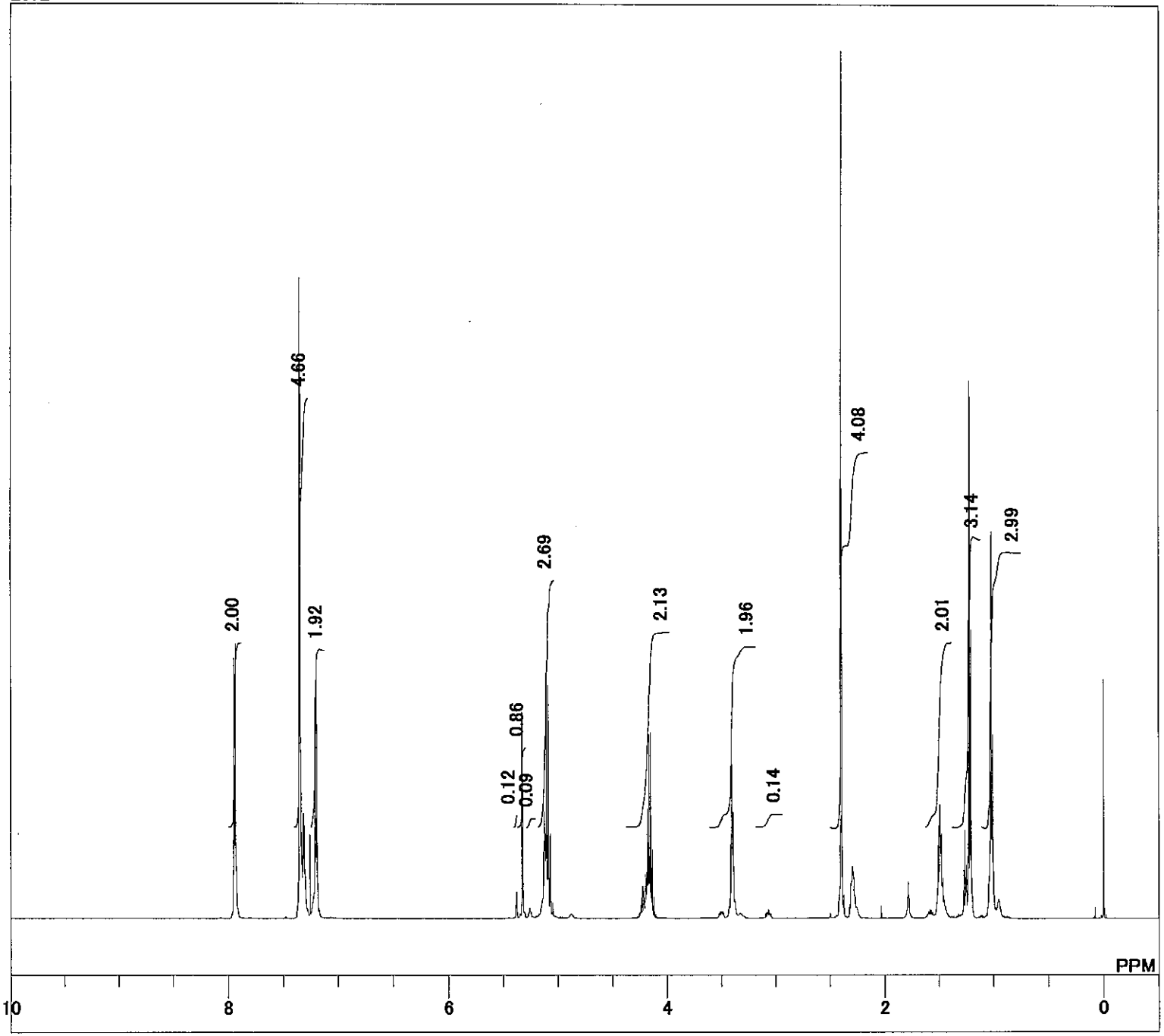
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2073_CARBON.als
2073



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2073
DATIM	28-06-2005 20:51:53
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

syn-6d

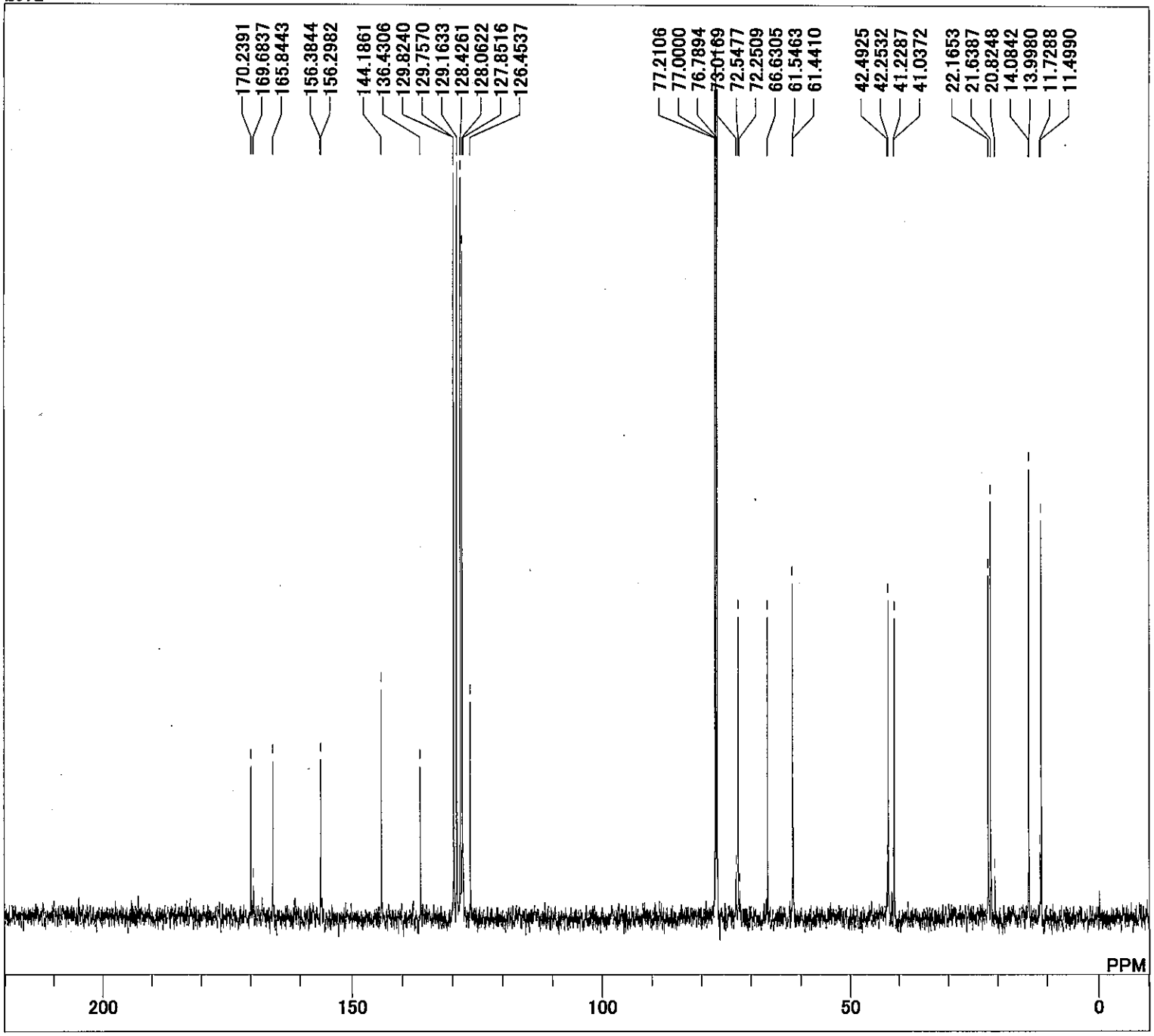
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2072_PROTON.als
2072



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2072
DATIM	28-06-2005 21:19:52
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

anti-6d

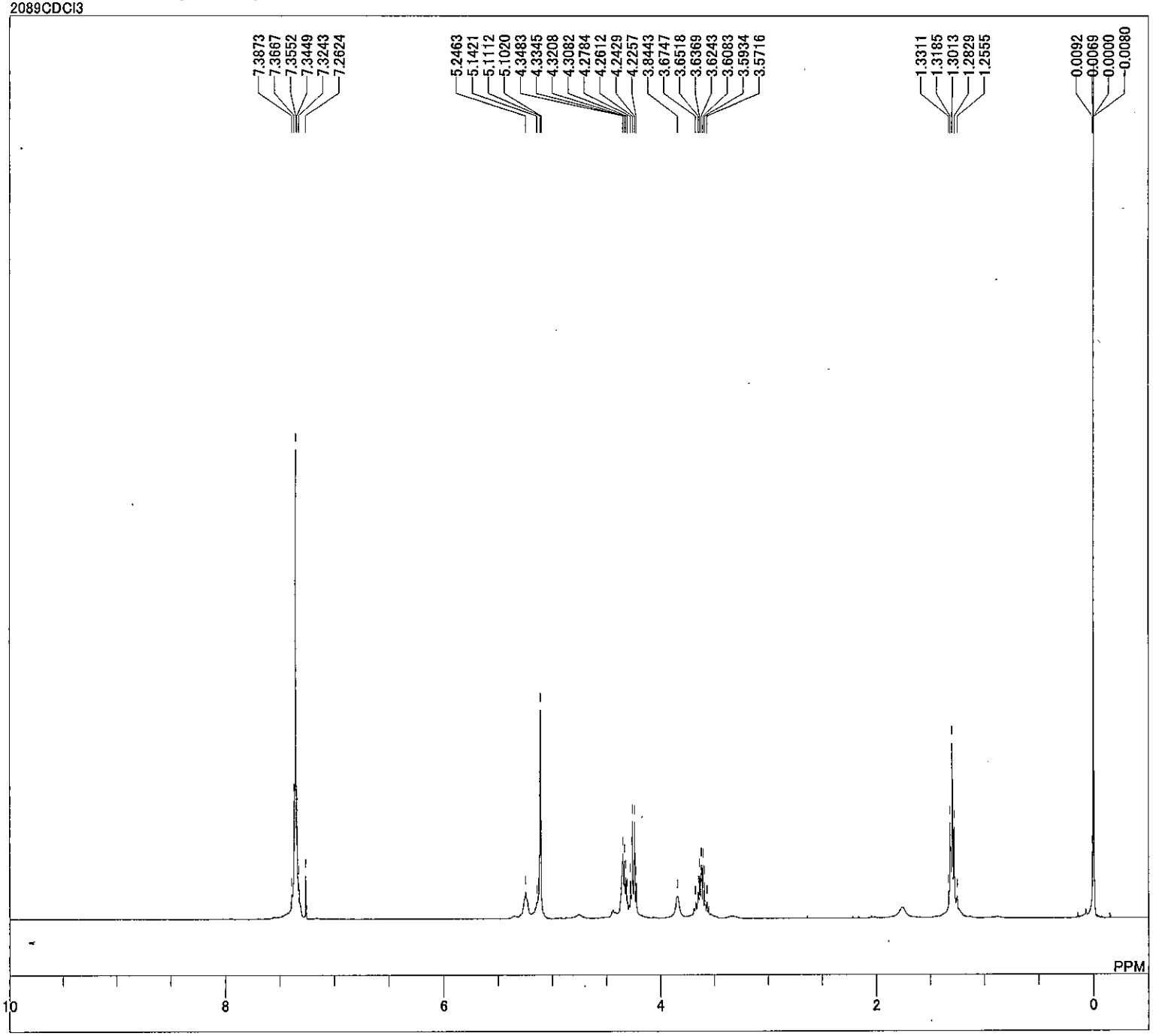
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2072_CARBON.als
2072



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2072
DATIM	28-06-2005 21:24:57
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	97
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

anti-6d

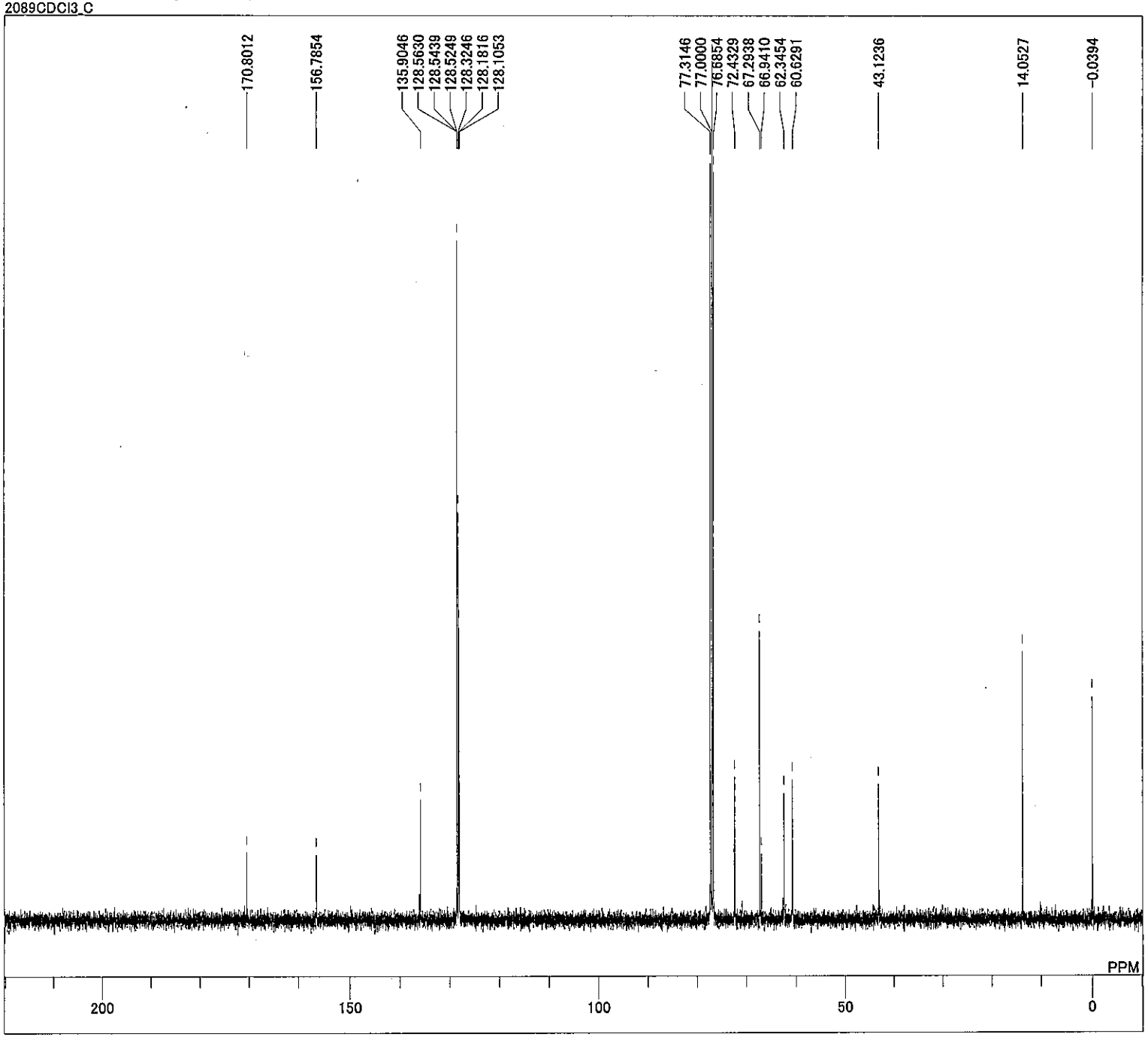
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2089CDCl3.als
2089CDCl3



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2089CDCl3
DATIM	26-09-2005 17:47:24
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	32

anti-7e

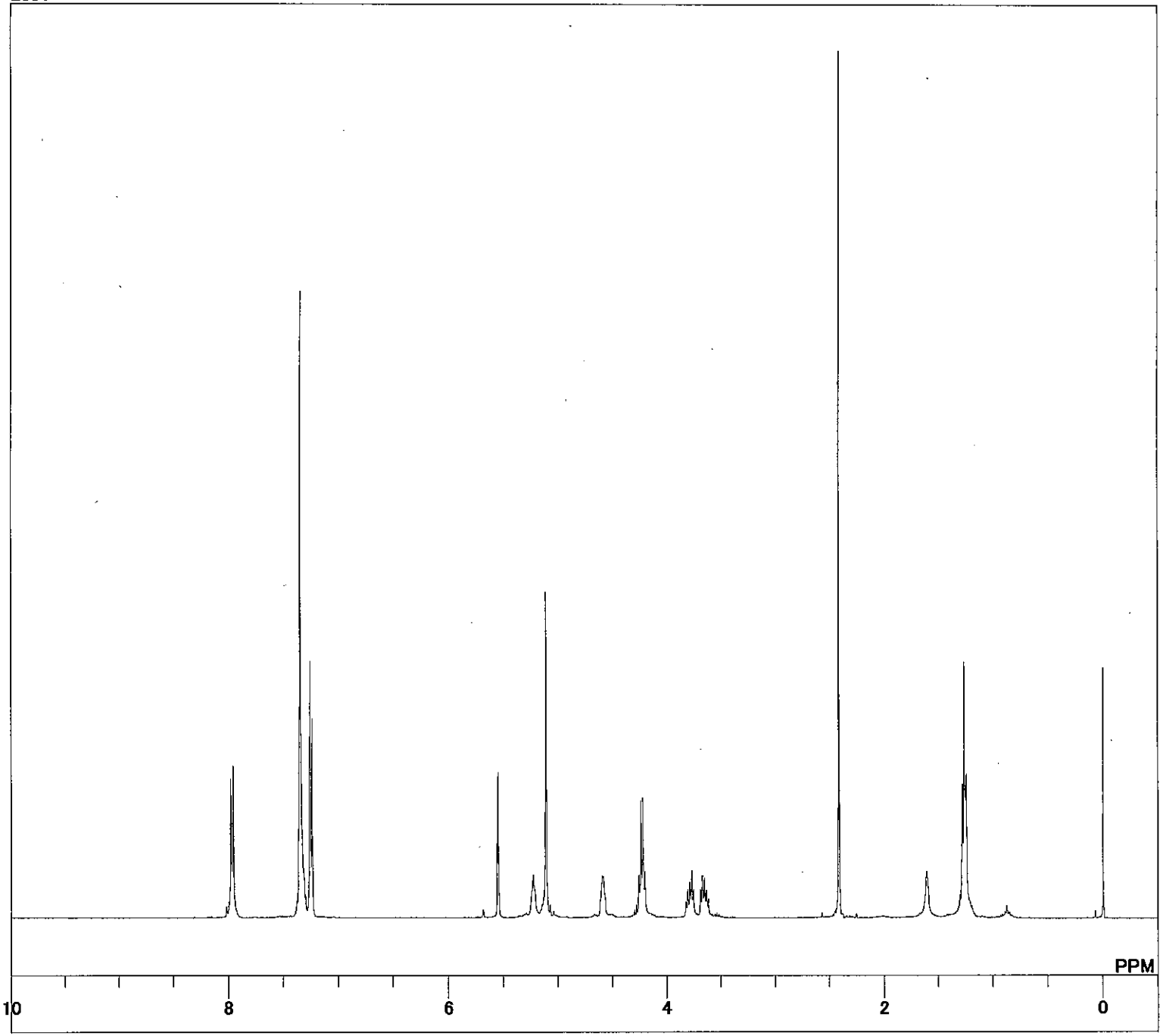
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2089CDCl3_C.als
2089CDCl3_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2089CDCl3_C
DATIM	26-09-2005 18:24:01
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	706
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

anti-7e

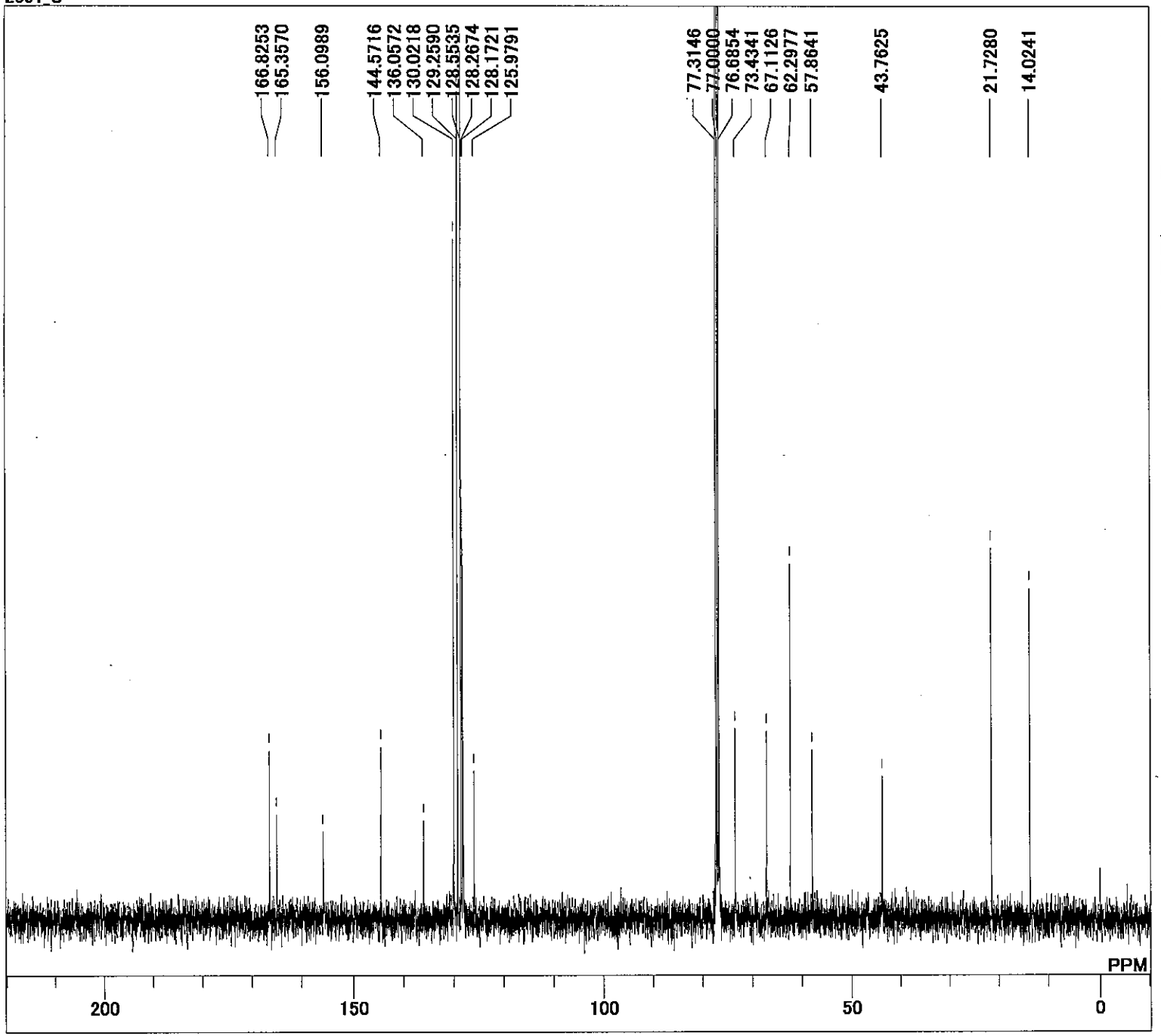
E:¥Matsubara¥2092_H.als
2091



DFILE	E:¥Matsubara¥2092_H.als
COMNT	2091
DATIM	28-07-2005 12:13:13
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	26.1 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.20 Hz
RGAIN	38

anti-6e

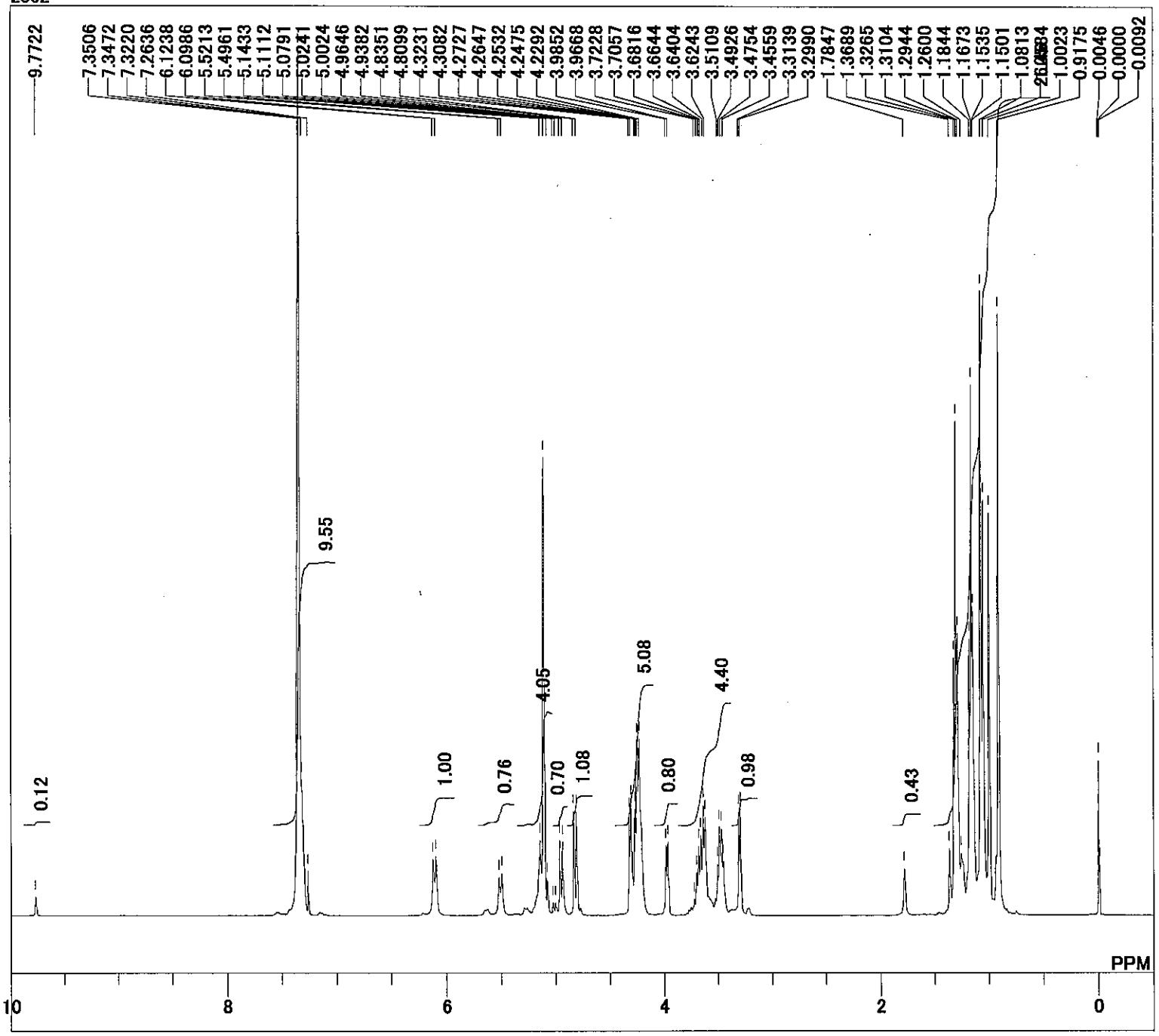
E:¥Matsubara¥2092_C.als
2091_C



DFILE	E:¥Matsubara¥2092_C.als
COMNT	2091_C
DATIM	28-07-2005 12:41:28
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	543
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	26.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	56

anti-6e

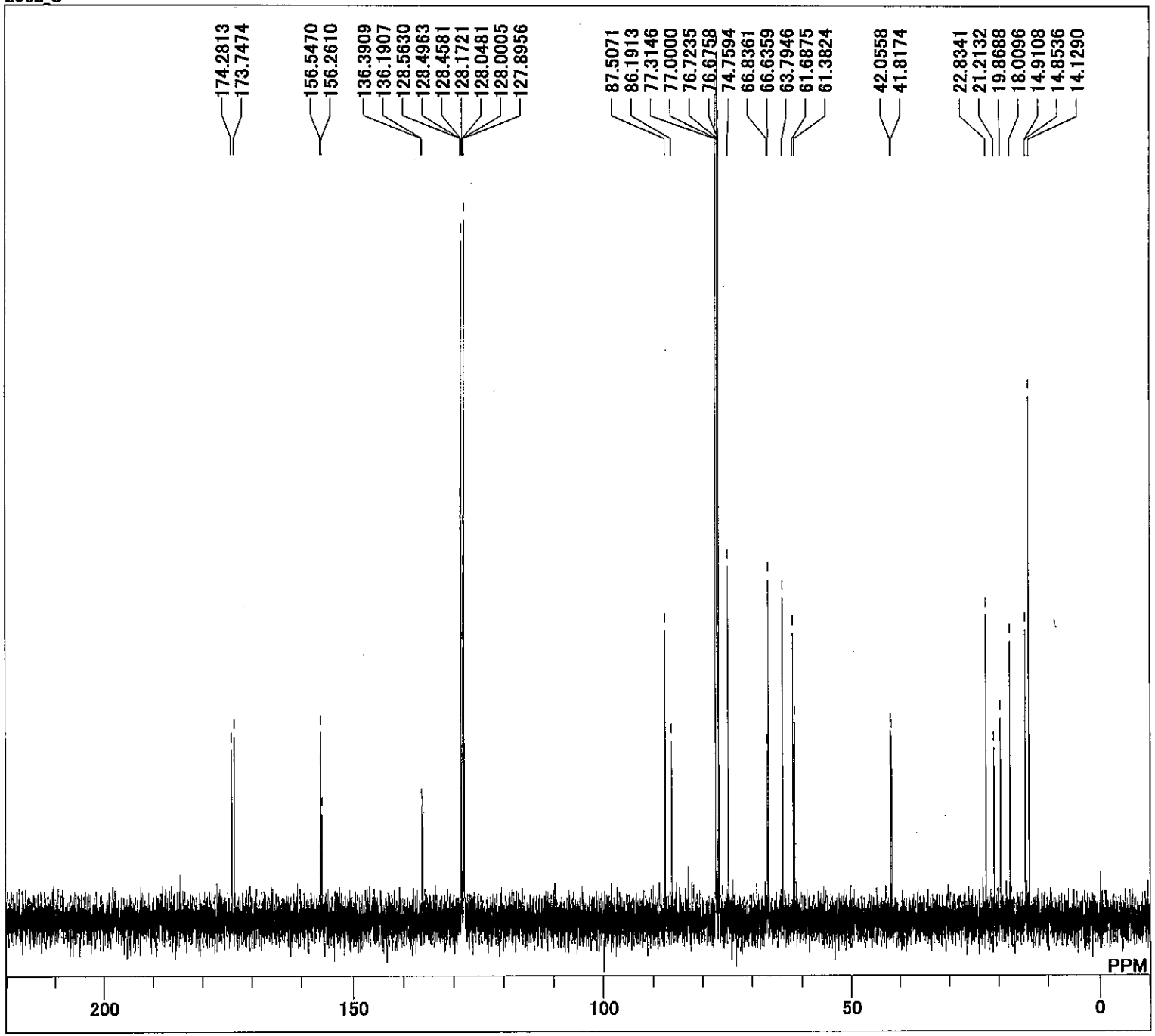
E:¥Matsubara¥2062.als
2062



DFILE	E:¥Matsubara¥2062.als
COMNT	2062
DATIM	23-06-2005 09:36:33
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30

3 f

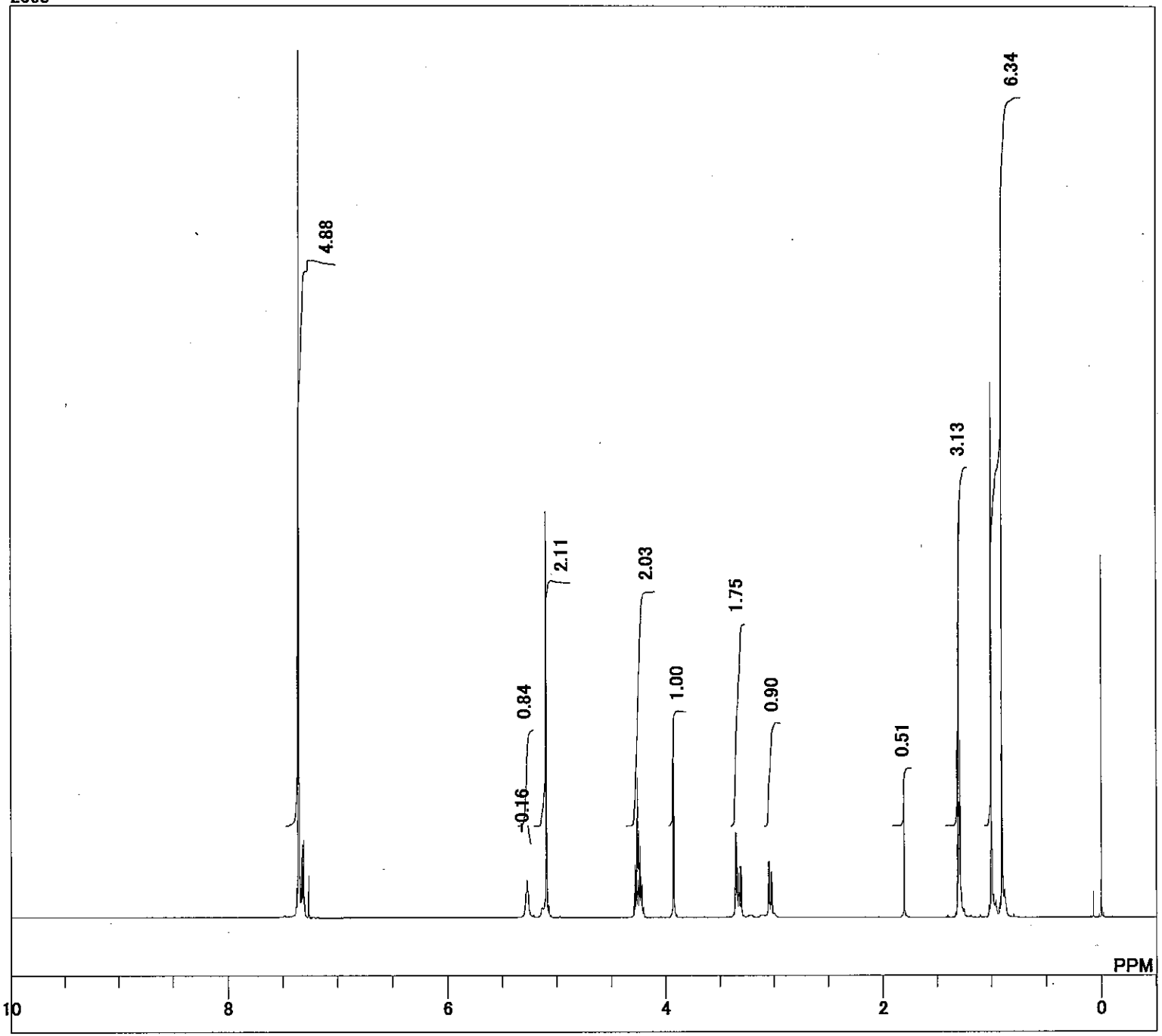
E:¥Matsubara¥2062_C.als
2062_C



DFILE	E:¥Matsubara¥2062_C.als
COMNT	2062_C
DATIM	23-06-2005 09:44:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	138
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

3f

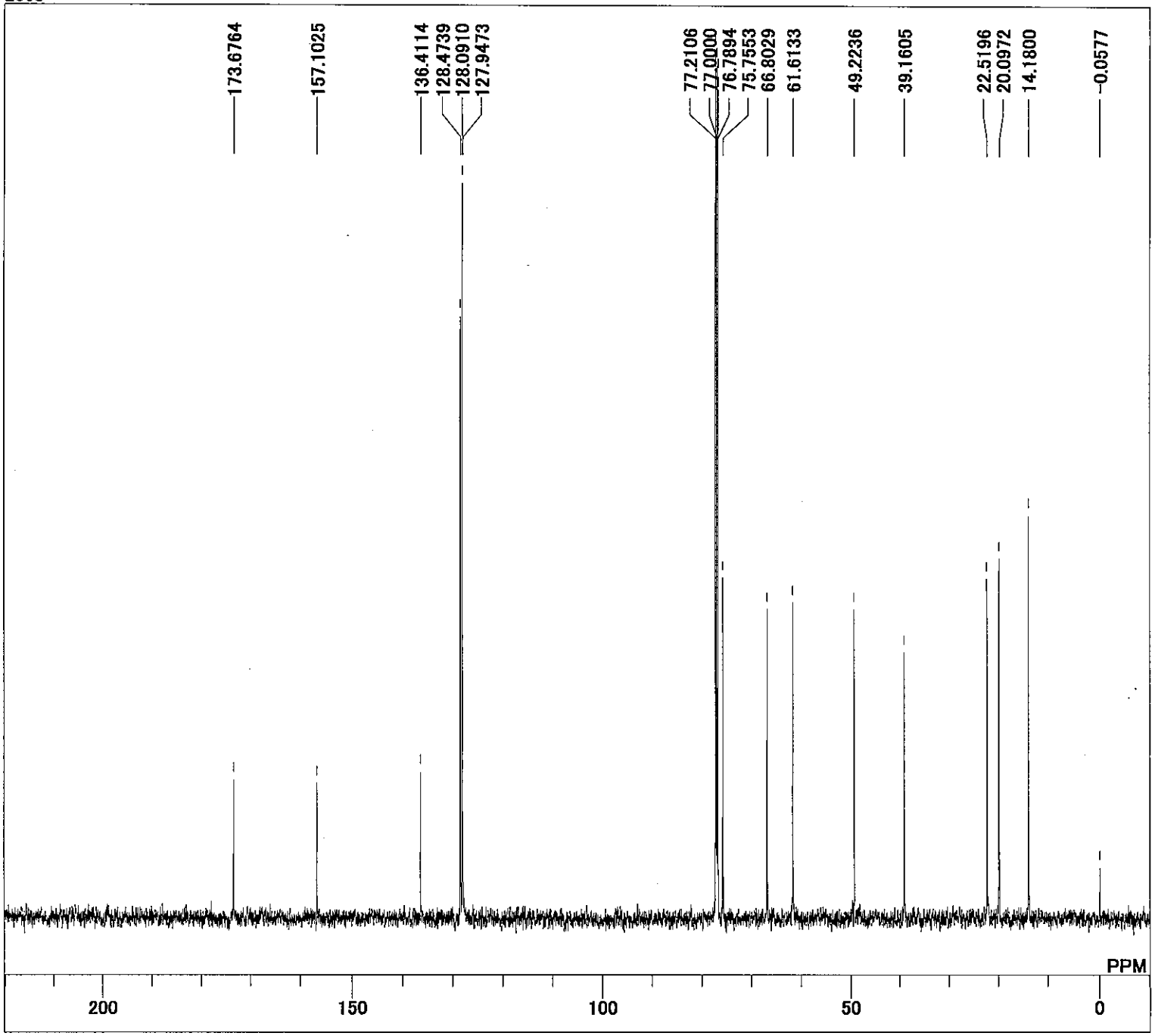
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2068_PROTON.als
2068



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2068
DATIM	27-06-2005 17:59:23
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	13107
FREQU	9008.87 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

7f

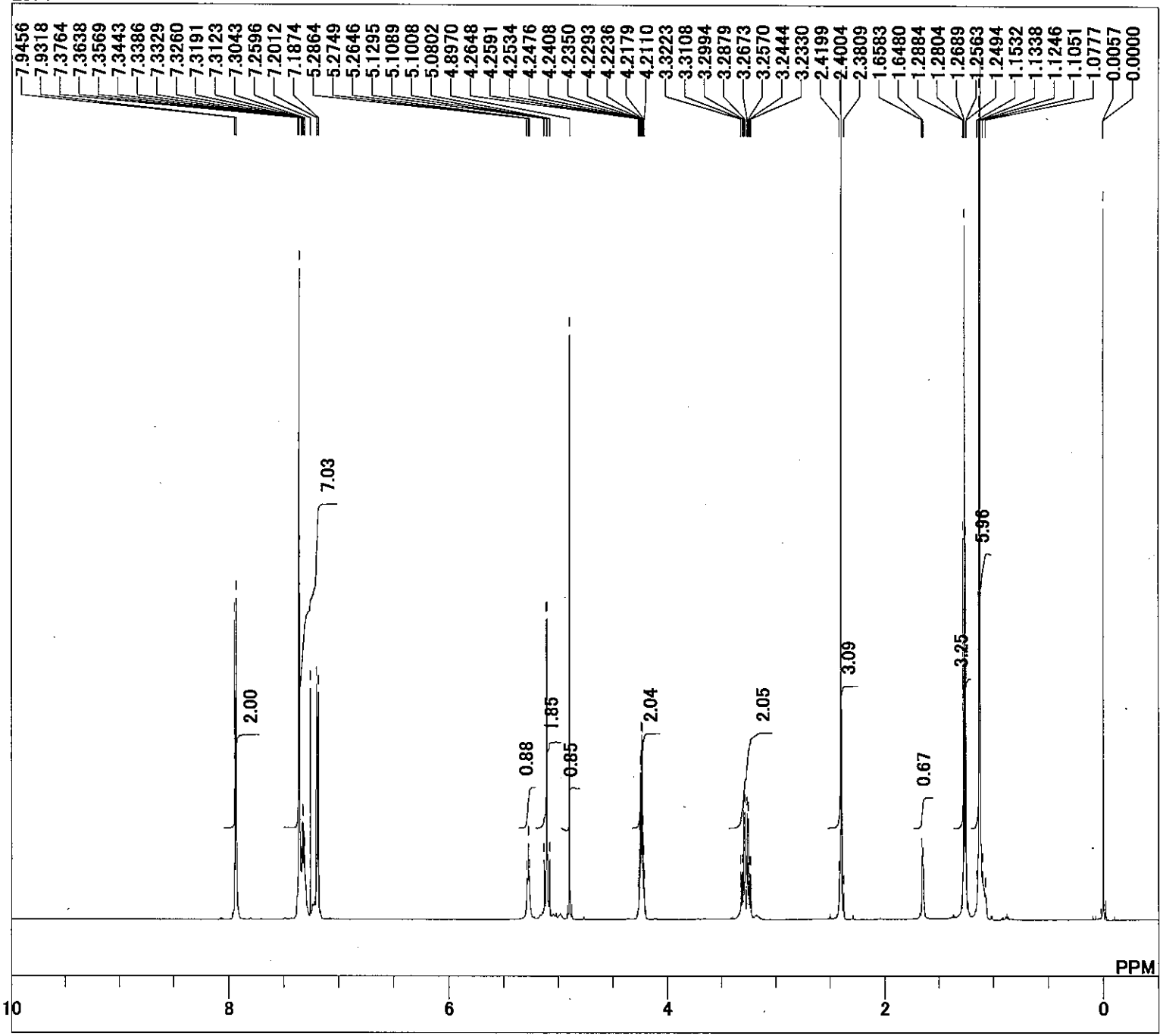
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2068_CARBON.als
2068



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2068
DATIM	27-06-2005 18:06:10
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	135
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

7f

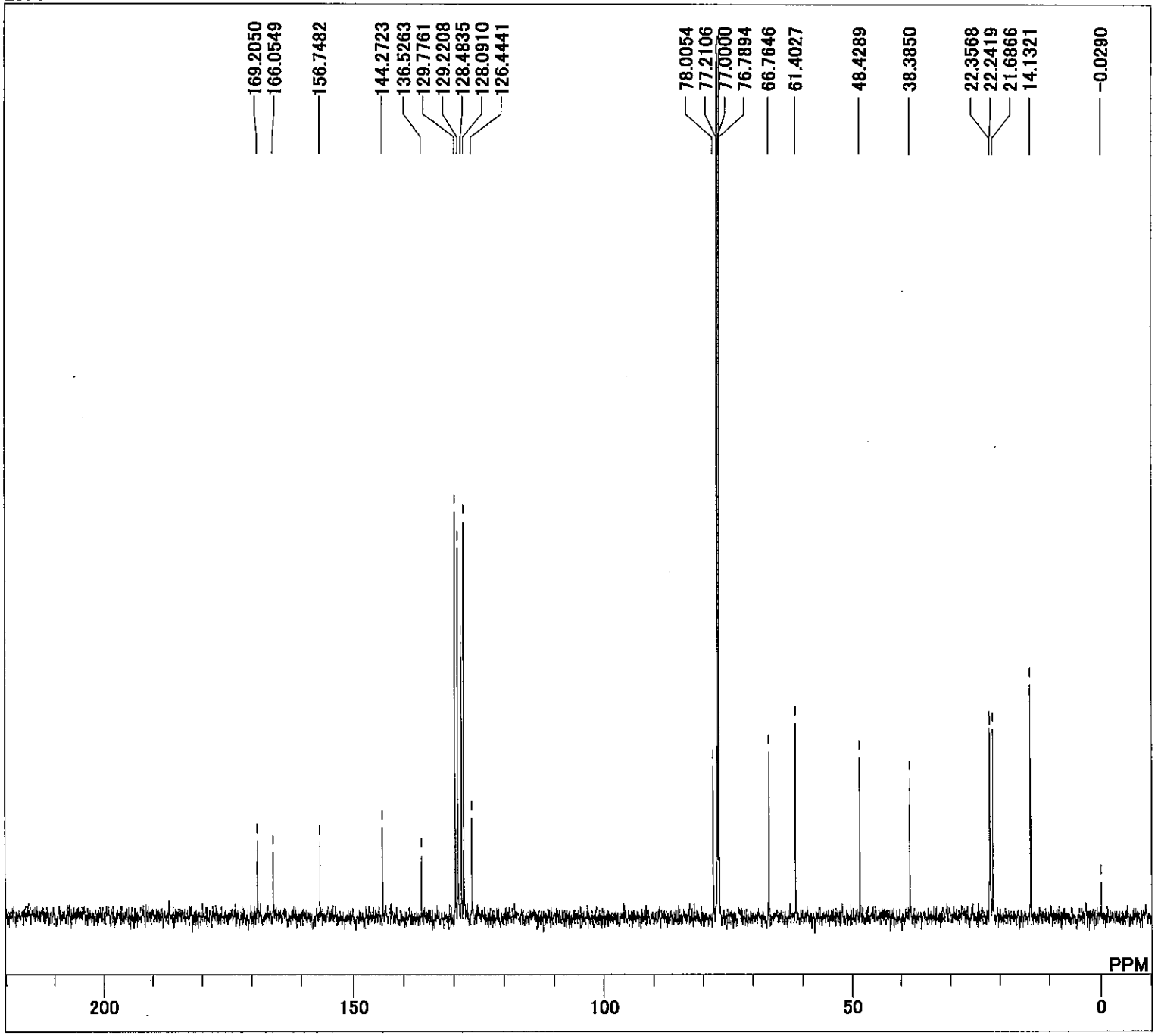
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2074_PROTON.als
2074



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2074
DATIM	28-06-2005 21:01:31
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

6f

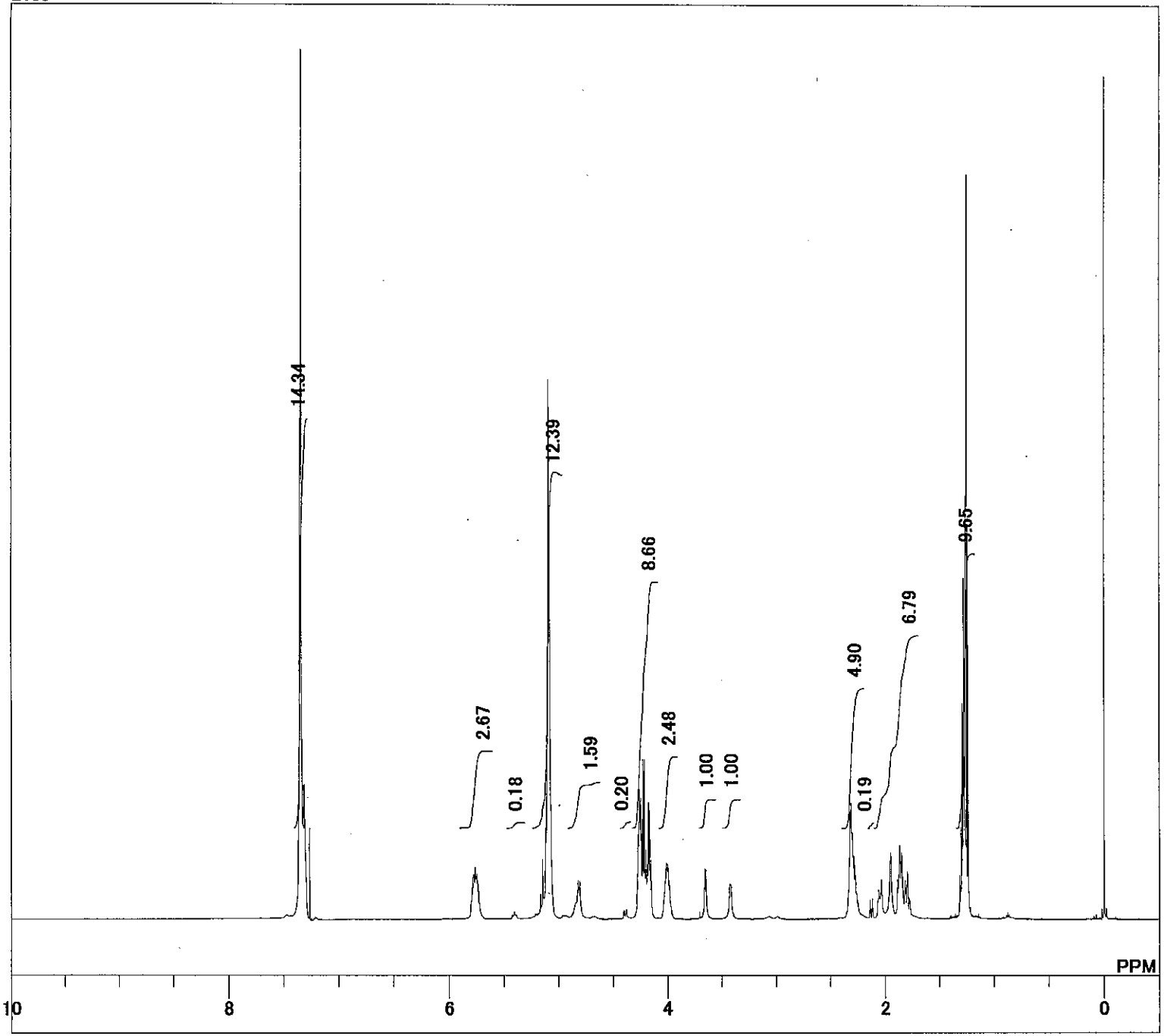
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2001-2100¥2074_CARBON.als
2074



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2074
DATIM	28-06-2005 21:09:56
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	171
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

6f

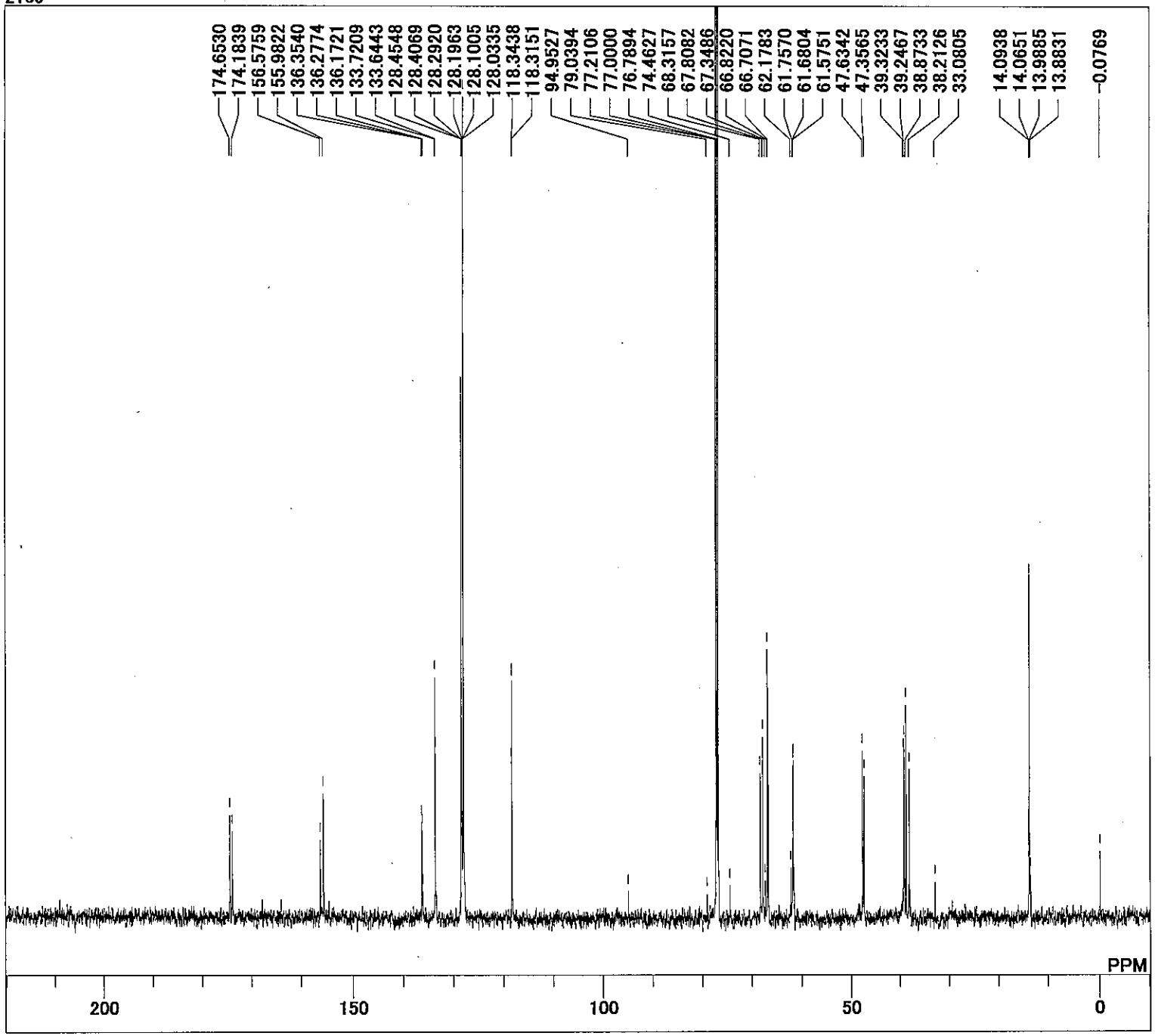
C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2109_PROTON.als
2109



DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2109
DATIM	06-09-2005 09:50:43
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	13107
FREQU	9008.87 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

8

C:¥Documents and Settings¥All Users¥Documents¥Matsubara¥2101-2200¥2109_CARBON.als
2109

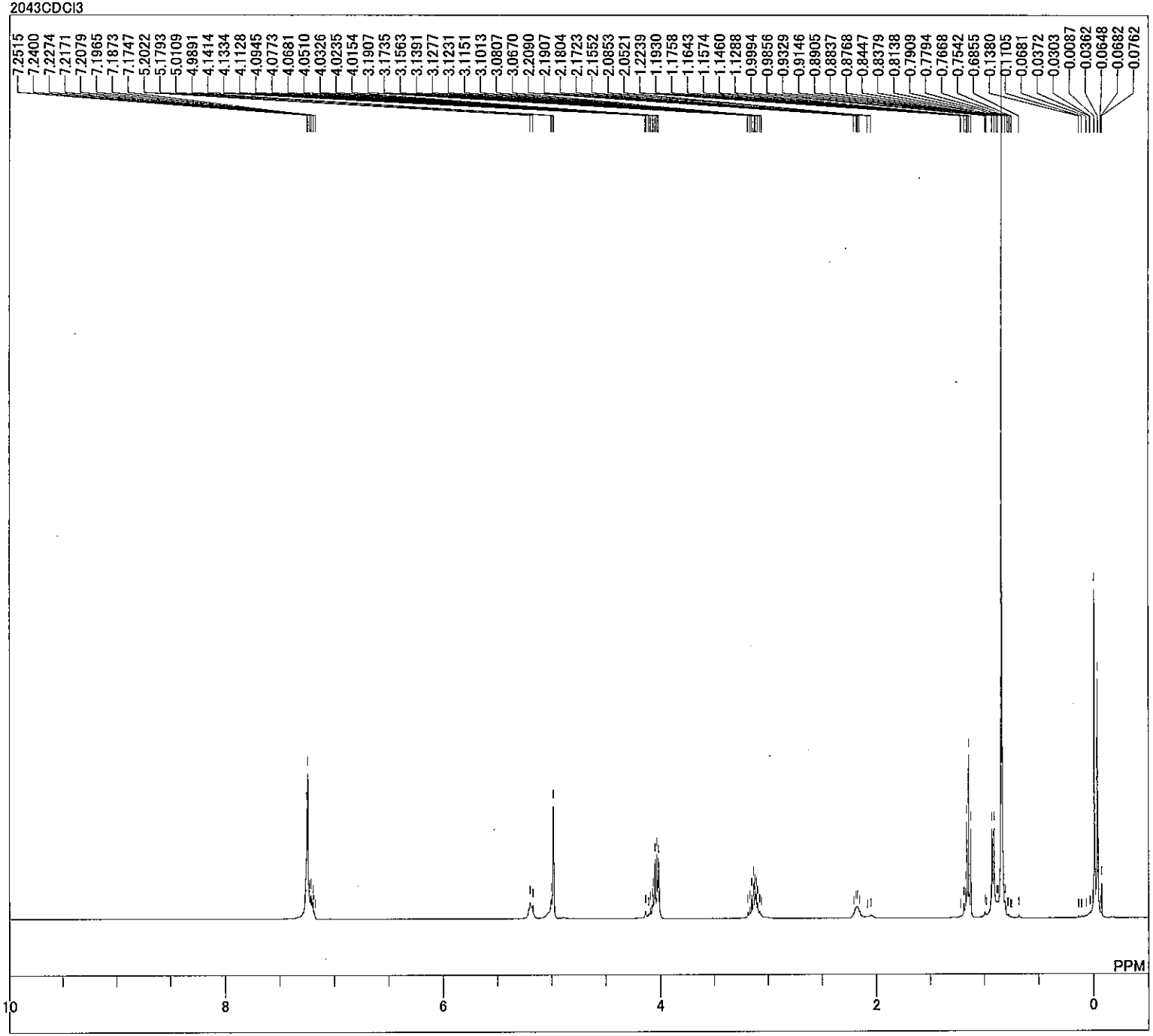


DFILE	C:¥Documents and Settings¥All Users¥
COMNT	2109
DATIM	06-09-2005 10:02:56
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	256
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	24.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

8

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2043CDCl3.als

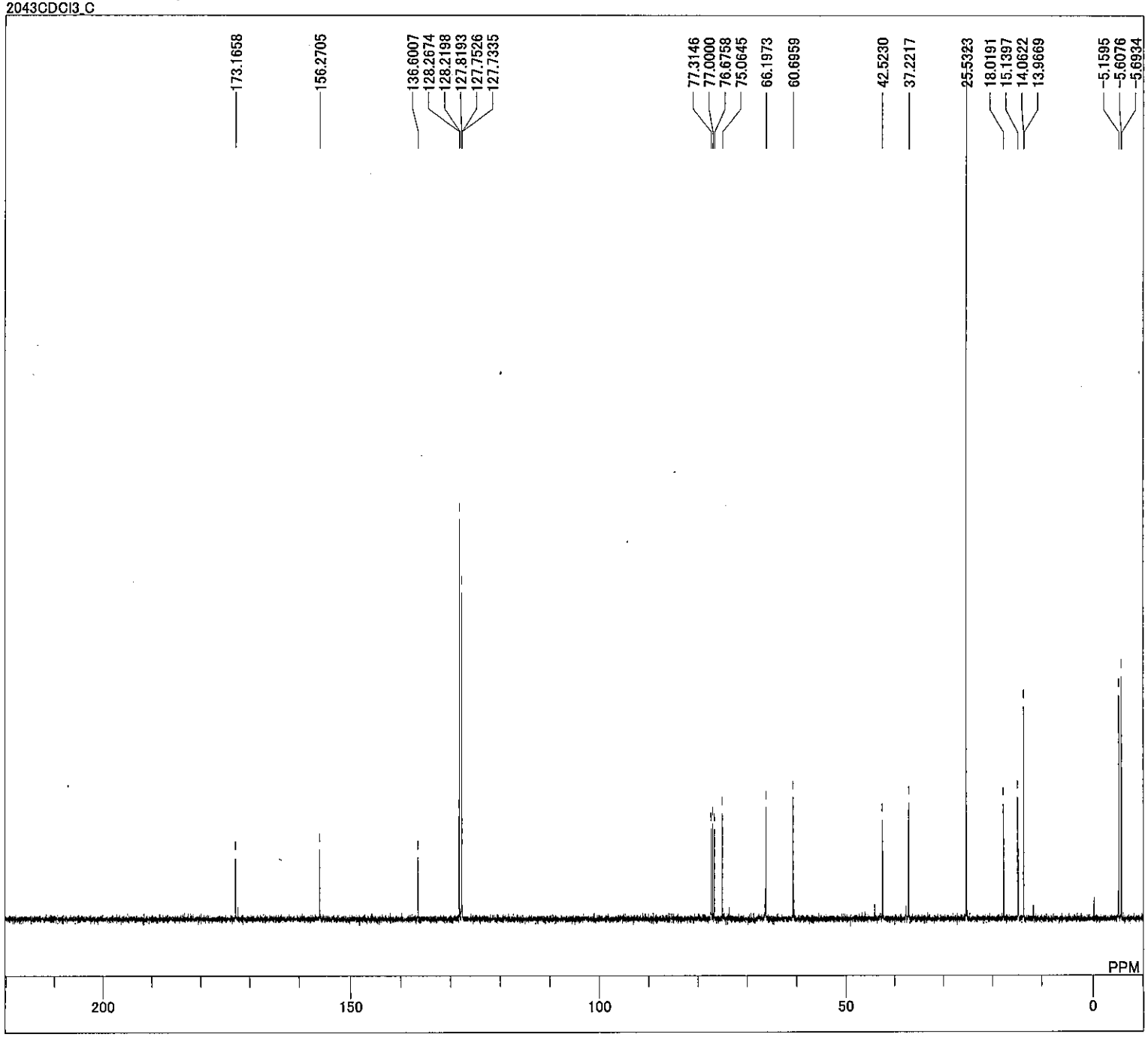
2043CDCl3



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2043CDCl3
DATIM	26-09-2005 18:26:45
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	16

12

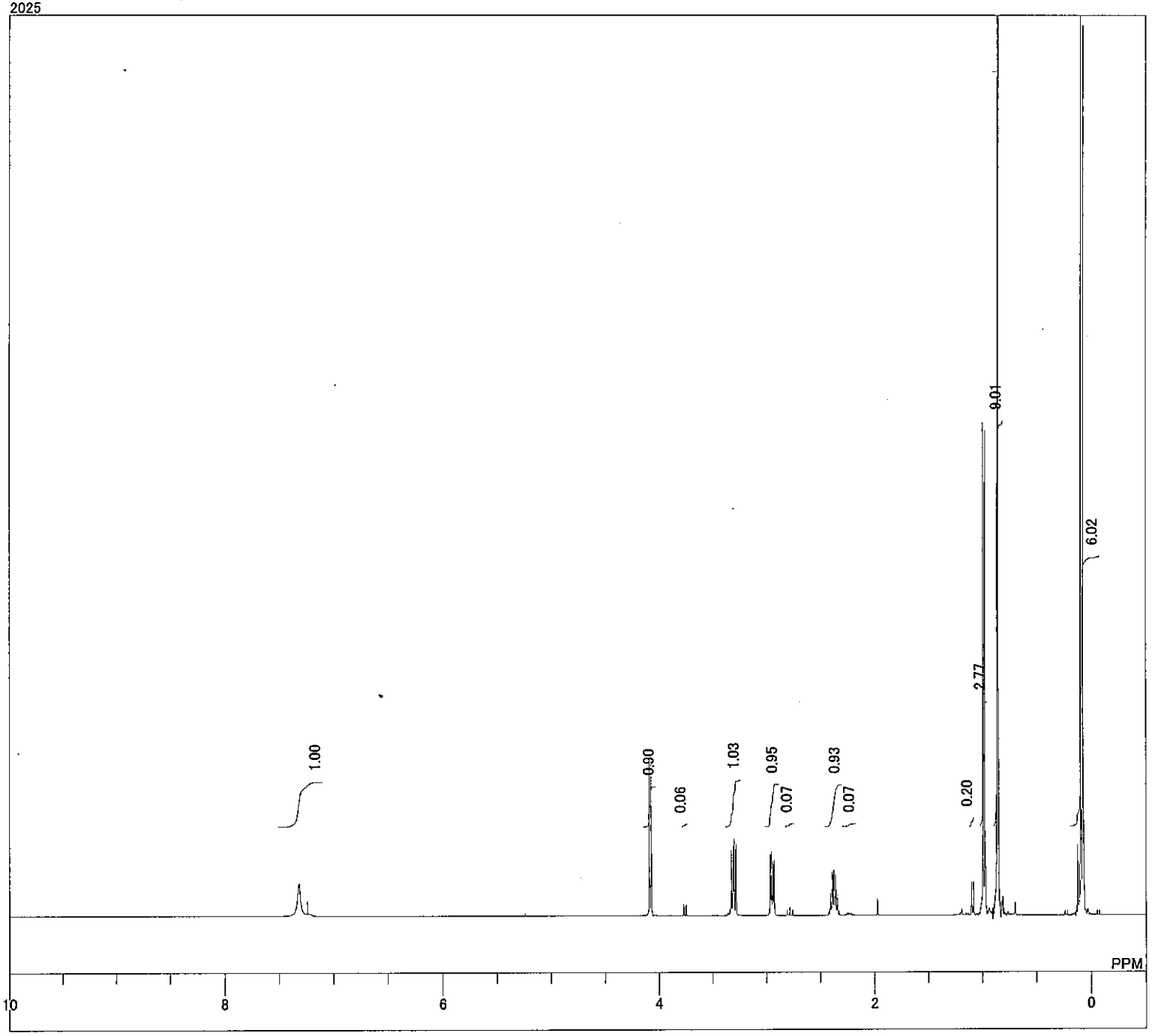
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2043CDCl3_C.als
2043CDCl3_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2043CDCl3_C
DATIM	26-09-2005 18:30:01
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	52
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

12

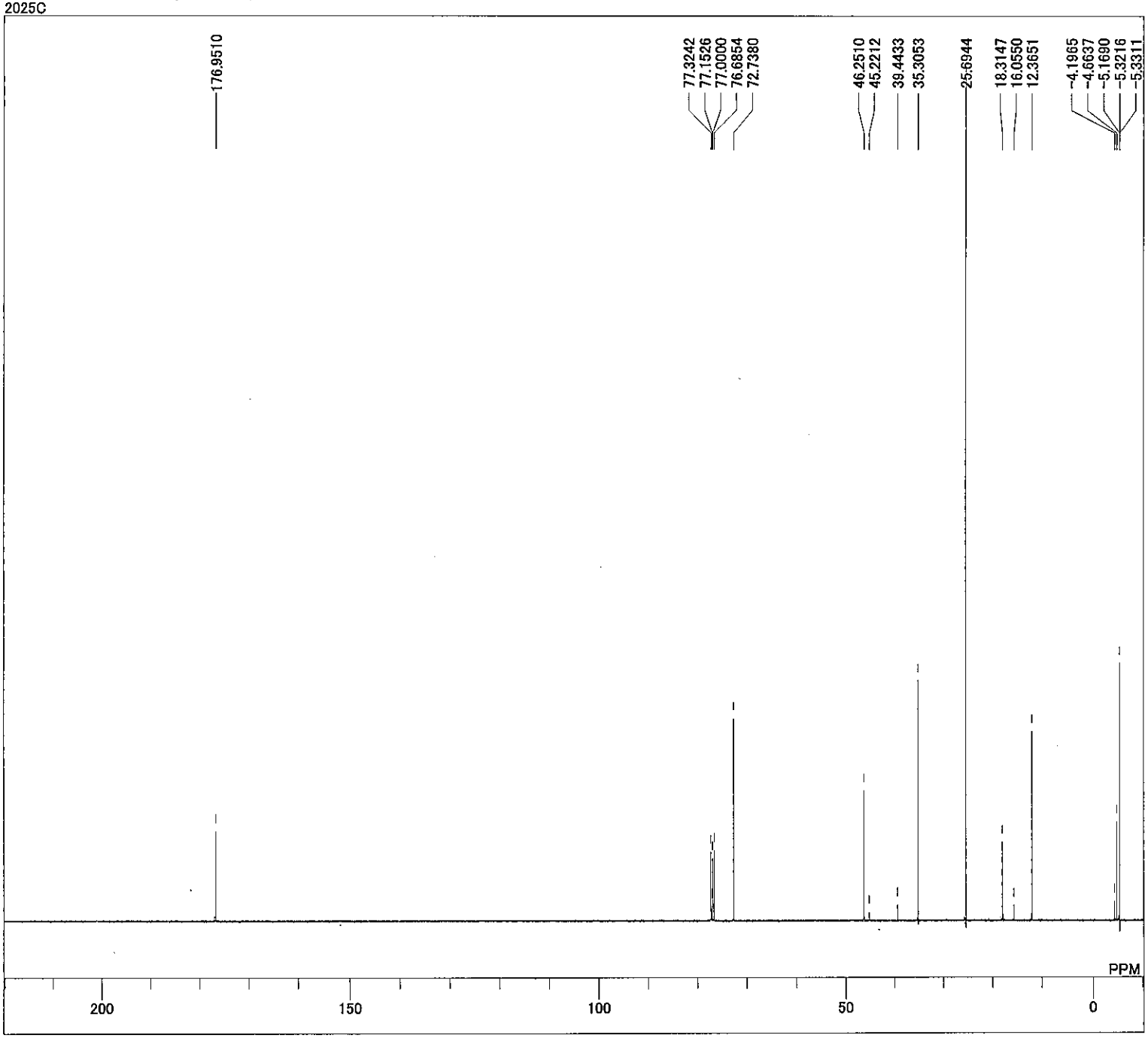
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2025.als
2025



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2025
DATIM	21-04-2005 22:54:47
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	22

13

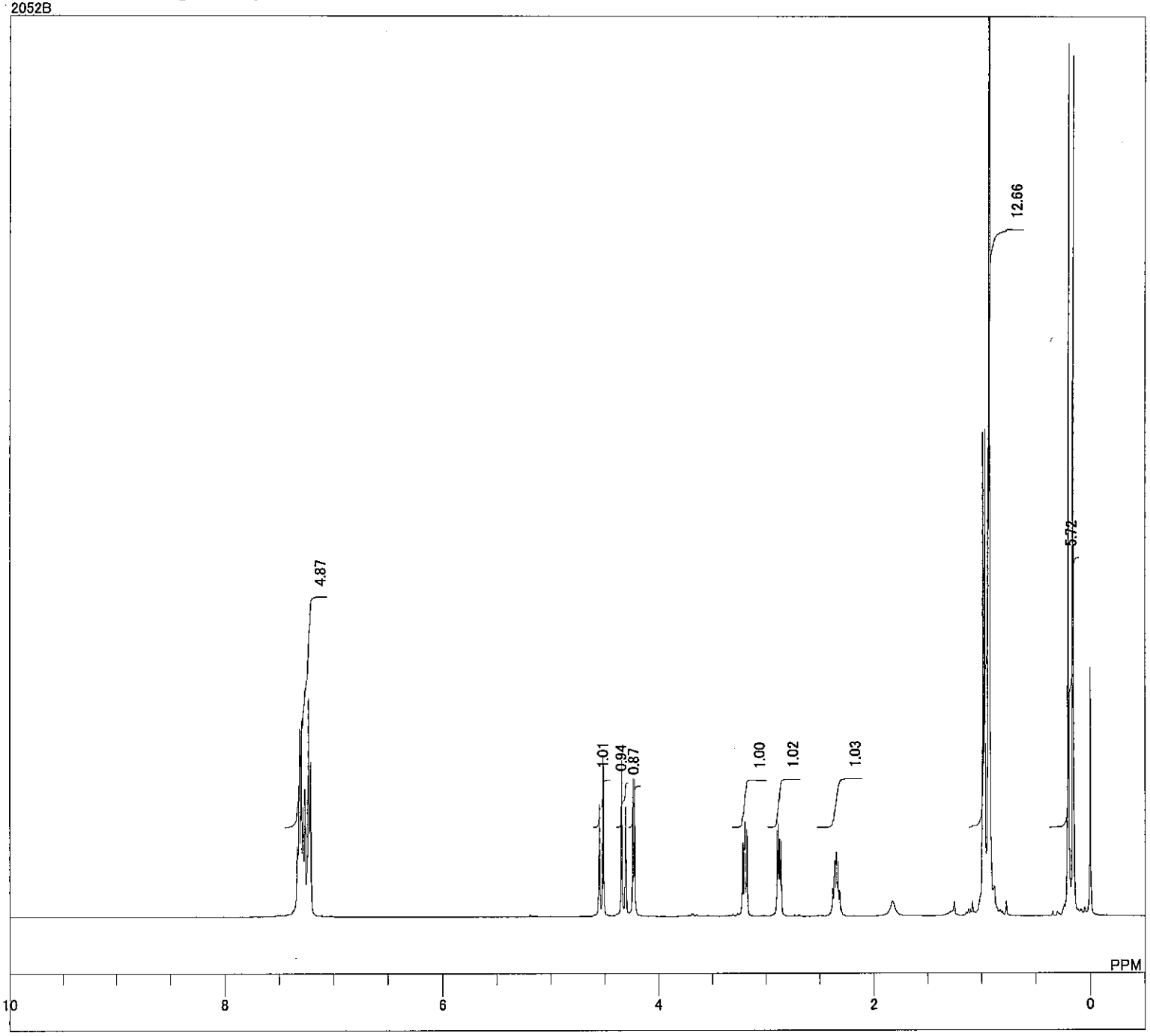
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2025C.als
2025C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2025C
DATIM	21-04-2005 23:11:58
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	325
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

13

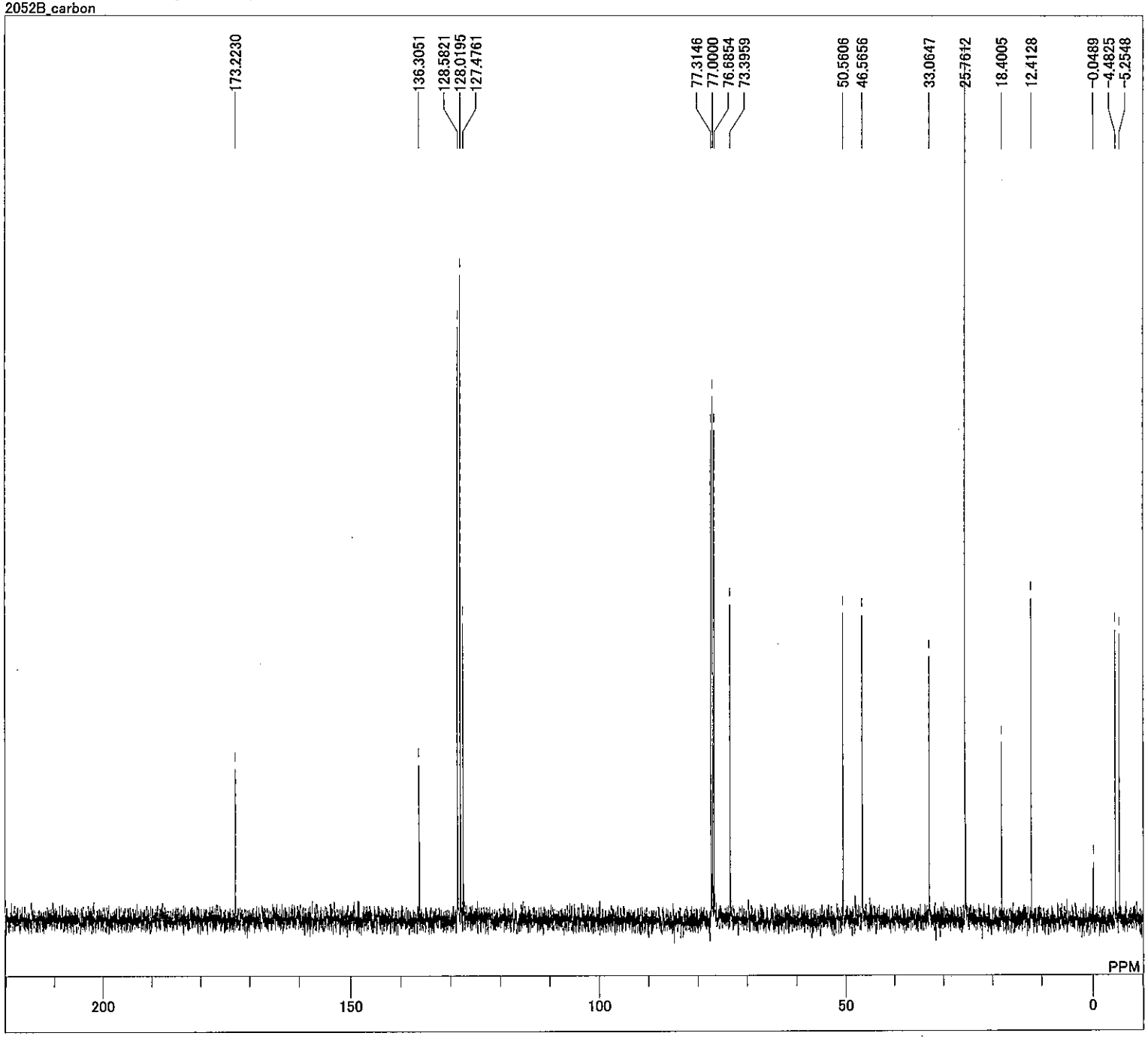
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2052B.als
2052B



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2052B
DATIM	10-06-2005 18:41:25
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	25.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.20 Hz
RGAIN	32

10

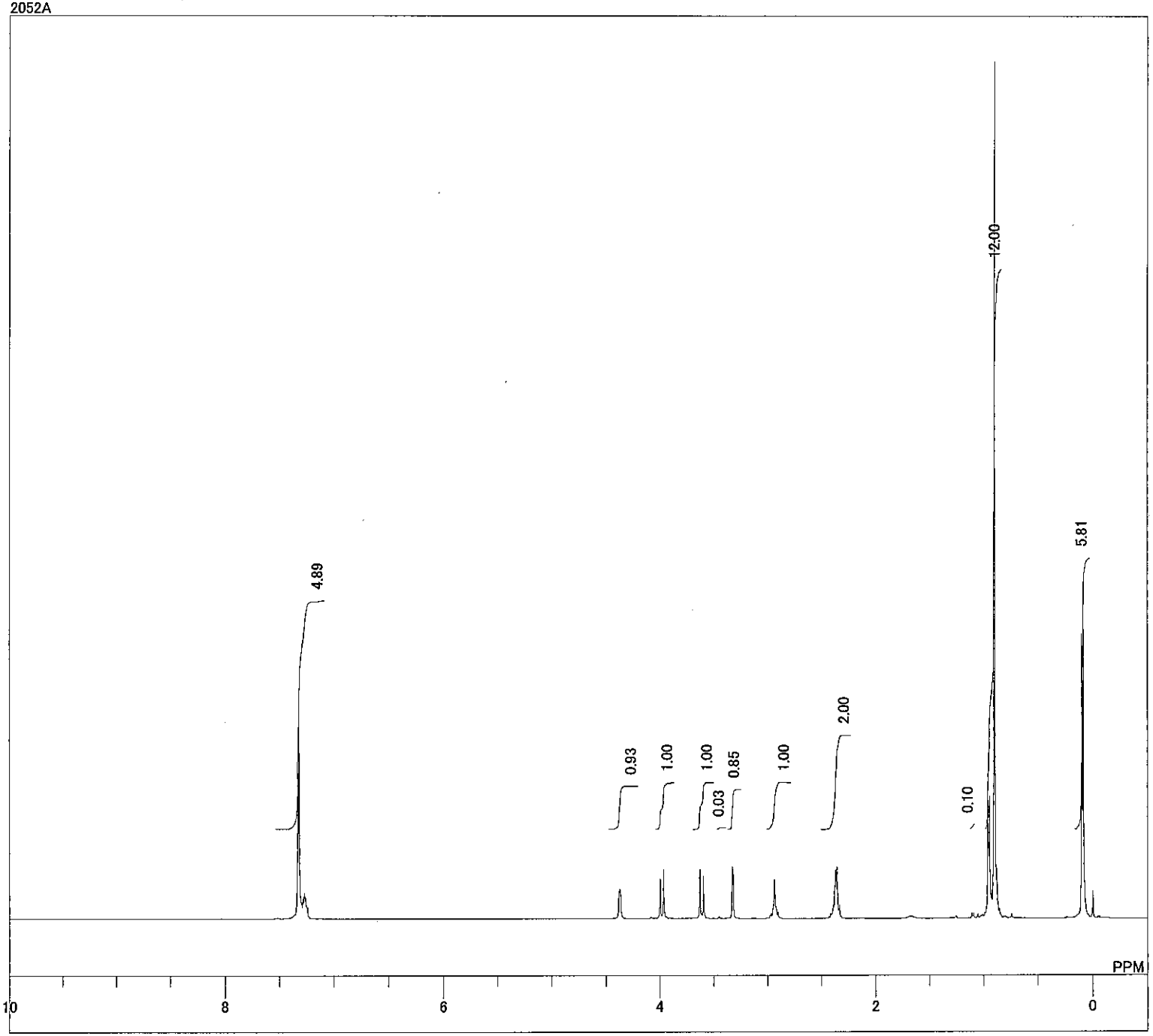
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2052B_carbon.als
2052B_carbon



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2052B_carbon
DATIM	10-06-2005 18:45:01
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	59
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	50

10

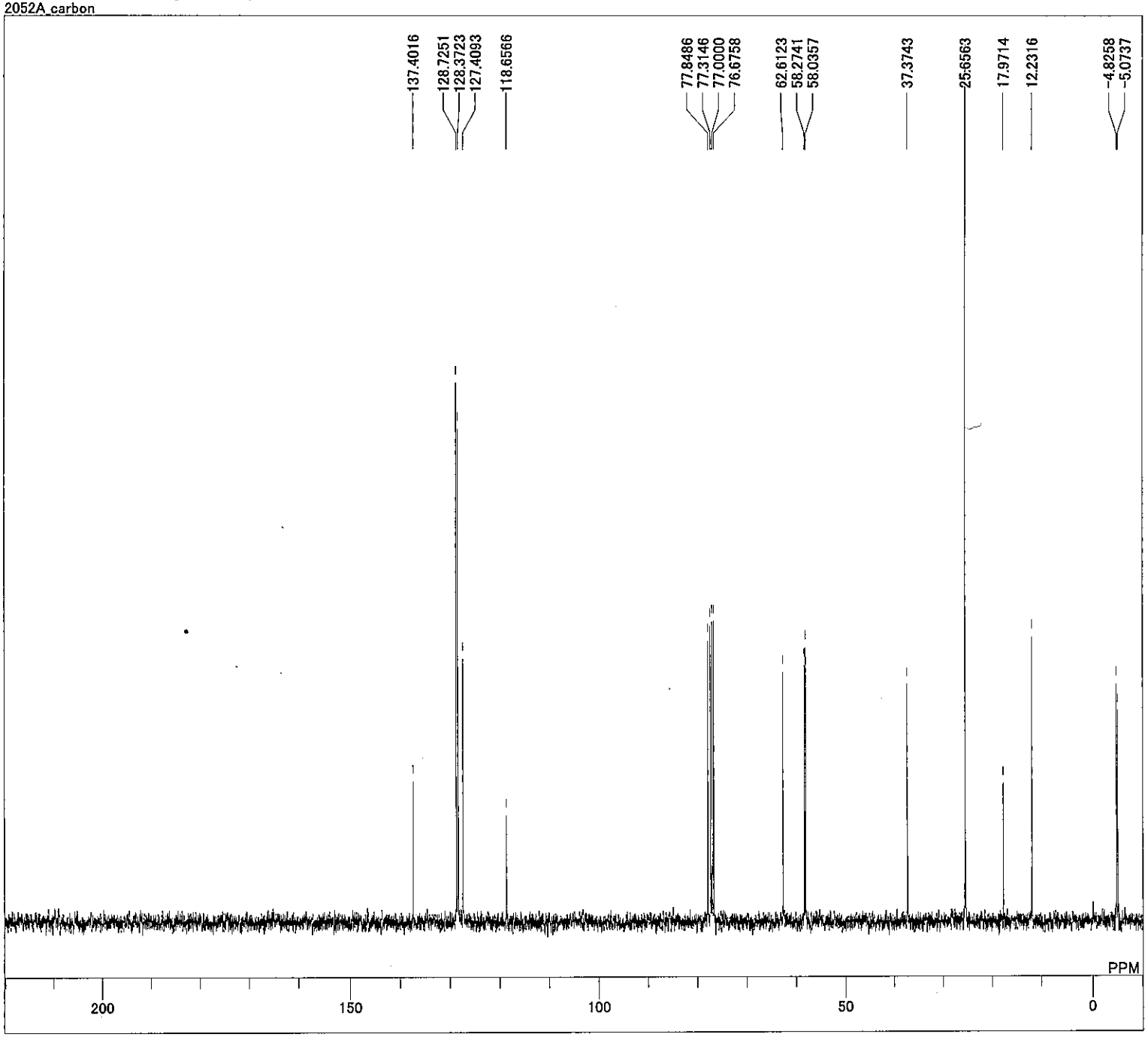
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2052A.als
2052A



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2052A
DATIM	10-06-2005 18:35:08
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	25.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.20 Hz
RGAIN	28

14

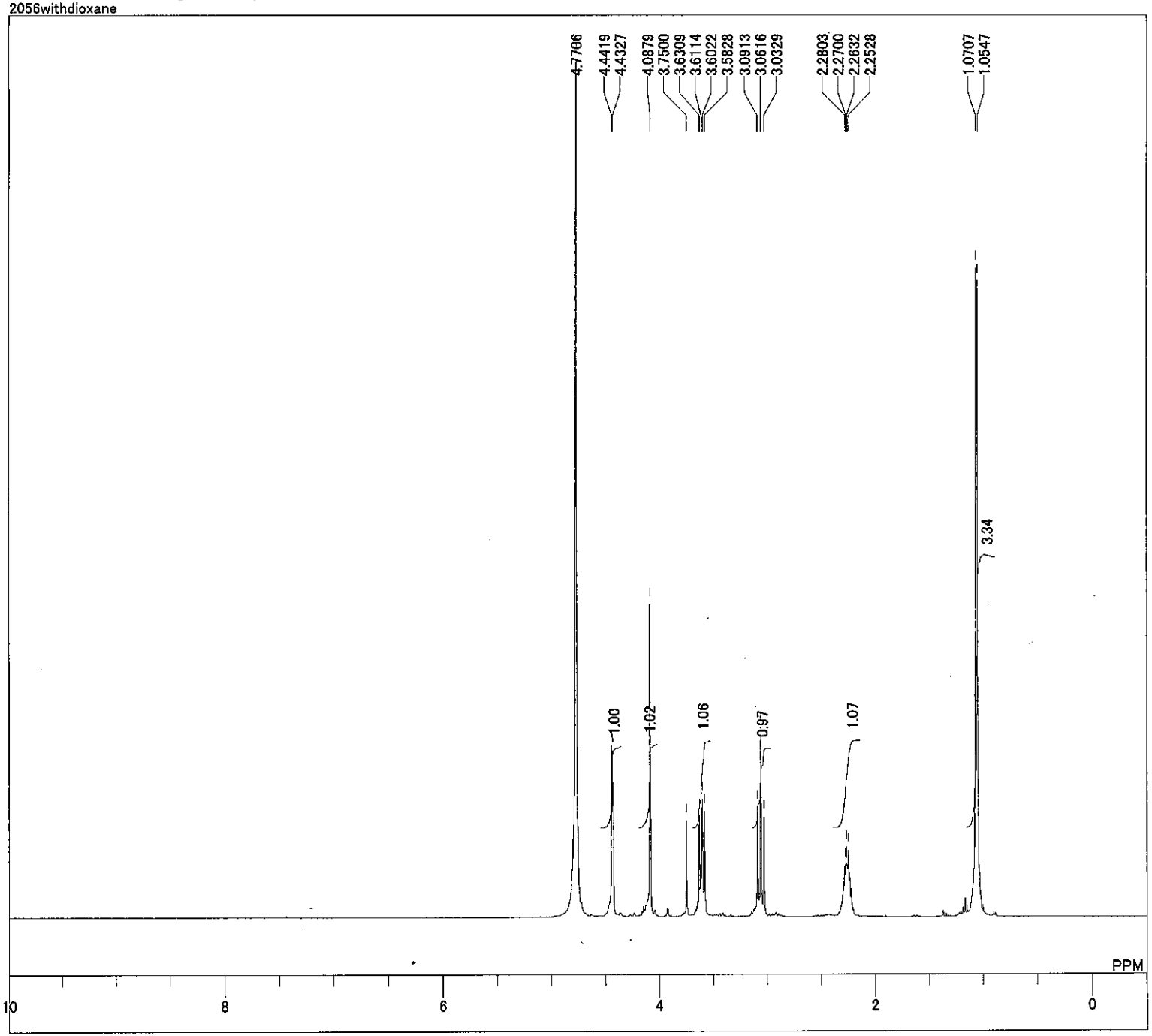
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2052A_carbon.als
2052A_carbon



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2052A_carbon
DATIM	10-06-2005 18:38:25
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	45
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	50

14

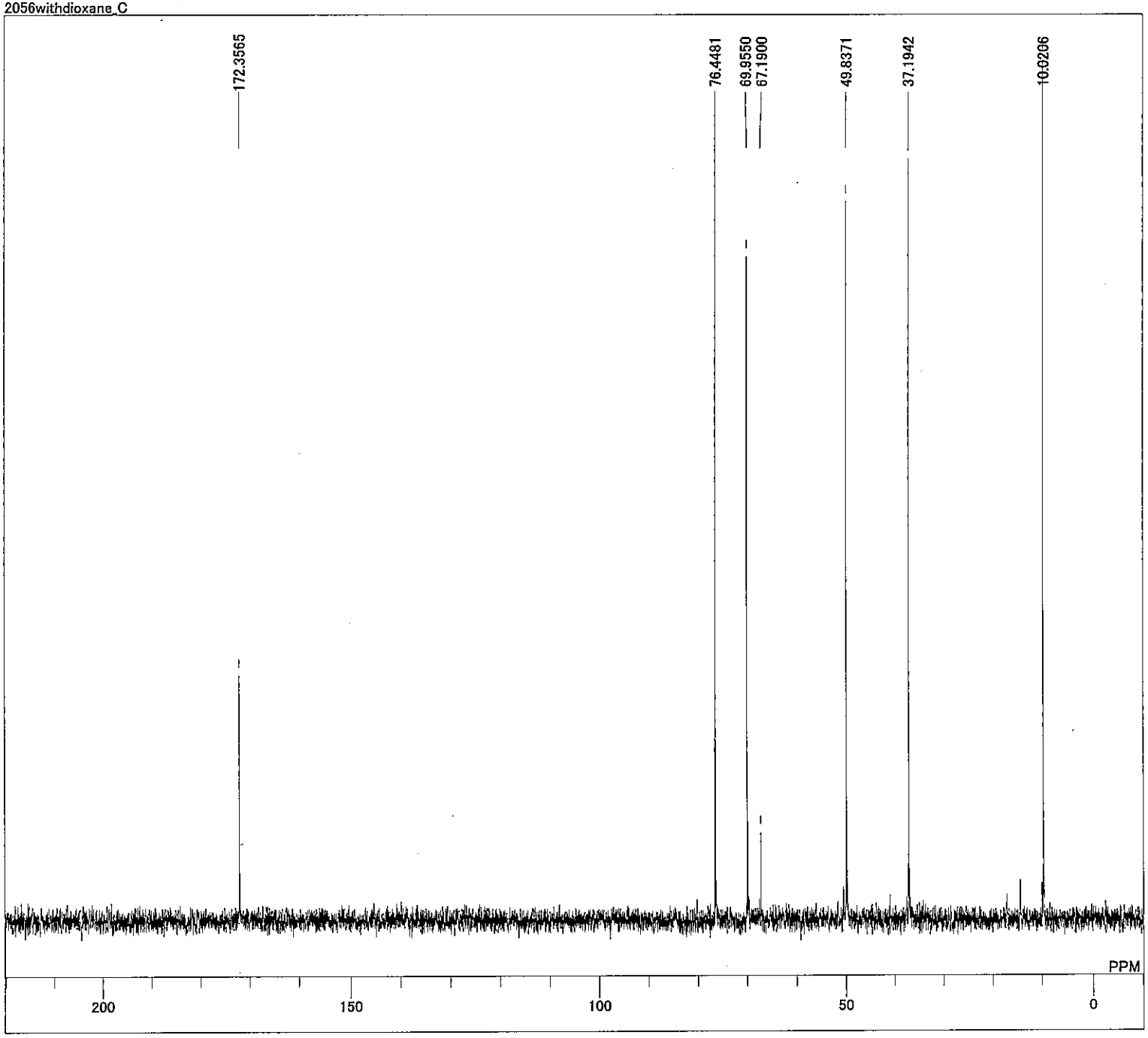
C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2056withdioxane.als
2056withdioxane



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2056withdioxane
DATIM	20-06-2005 13:35:46
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	26.7 c
SLVNT	D2O
EXREF	3.75 ppm
BF	0.12 Hz
RGAIN	34

HMP

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥Matsubara¥2056withdioxane_C.als
2056withdioxane_C



DFILE	C:¥Documents and Settings¥delta¥My Documents¥I
COMNT	2056withdioxane_C
DATIM	20-06-2005 13:55:02
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	362
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	27.1 c
SLVNT	D2O
EXREF	67.19 ppm
BF	0.12 Hz
RGAIN	58

HMP