

Sulfonylimides as Nucleophiles in Catalytic Addition Reactions

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Supporting Information

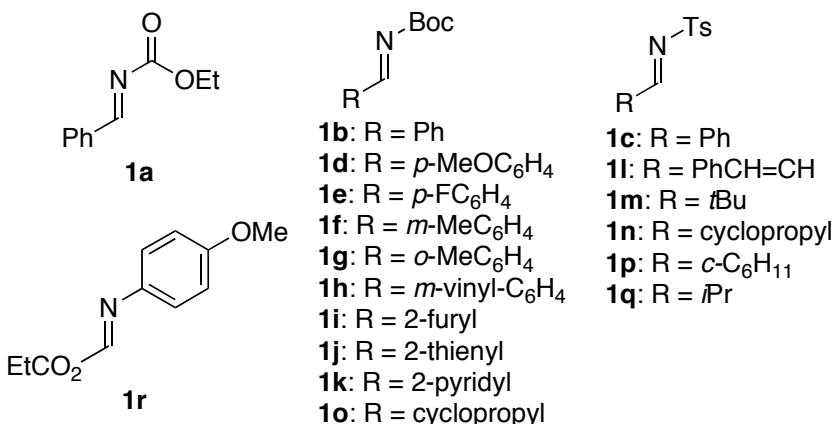
Experimental details and physical data of the products.

General. Melting points are uncorrected. ^1H and ^{13}C NMR spectra were recorded on a JEOL JNM-ECX-400, JNM-ECX-500 and JNM-ECX-600 spectrometer in CDCl_3 or C_6D_6 unless otherwise noted. Tetramethylsilane (TMS) served as internal standard ($\delta = 0$) for ^1H NMR, and CDCl_3 ($\delta = 77.0$) and C_6D_6 ($\delta = 128.0$) were used as internal standard for ^{13}C NMR. IR spectra were measured on a JASCO FT/IR-610 spectrometer. Column chromatography was conducted on Silica gel 60 (Merck) and preparative thin-layer chromatography was carried out using Wakogel B-5F. X-ray diffraction analysis was performed on a Rigaku-RAXIS-RAPID diffractometer. All reactions were carried out under argon atmosphere in dried glassware. All solvents were dried and distilled by standard procedures. N-alkoxycarbonyl imines **1a**, **1b**, **1d-k**, **1o**,¹ N-sulfonylimines **1c**, **1l-n**, **1p-q**,² and N-arylimino ester **1r**³ were prepared according to the reported method.

¹ Kanazawa, A. M.; Denis, J.-N.; Greene, A. E. *J. Am. Chem. Soc.* **1994**, *59*, 1238.

² (a) Vishwakarma, L. C.; Stringer, O. D.; Davis, F. A. *Org. Synth.* **1987**, *66*, 203. (b) Love, B. E.; Raje, P. S.; Williams, T. C., II. *Synlett* **1994**, 493. (c) Hodous, B. L.; Fu, G. C. *J. Am. Chem. Soc.* **2002**, *124*, 1578. and the references cited therein.

³ Hagiwara, E.; Fujii, A.; Sodeoka, M. *J. Am. Chem. Soc.* **1998**, *120*, 2474. and the references cited therein.



Preparation of sulfonylimine 2.⁴ To a mixture of acetophenone (3 ml, 25.7 mmol), tosyl amide (4.403 g, 25.7 mmol) and DCE (dichloroethane, 40 ml) were added a solution of TiCl₄ (4.879 g, 25.7 mmol) in DCE (10 ml) and Et₃N (7.89 ml, 56.6 mmol), successively at 0 °C. The mixture was stirred at room temperature (RT) for 18 h, and poured into saturated NaHCO₃ aqueous solution at 0 °C. After filtration, the filtrate was extracted by CH₂Cl₂ twice, and the organics were dried over anhydrous Na₂SO₄. Filtration and evaporation of the solvents afforded the crude product, which was purified by column chromatography on neutral SiO₂ and recrystallization (hexane/AcOEt) to give us sulfonylimine **2** (1 g, 15%). The reaction condition has not been optimized.

1-phenyl-N-tosylethanimine (2): Mp. 89-90 °C; ¹H NMR (CDCl₃) δ = 7.86-7.95 (m, 4H), 7.47-7.54 (m, 1H), 7.37-7.43 (m, 2H), 7.33 (d, 2H, *J* = 7.9 Hz), 2.98 (s, 3H), 2.43 (s, 3H); ¹³C NMR (CDCl₃) δ = 179.7, 143.5, 138.7, 137.5, 133.1, 129.4, 128.6, 128.2, 127.0, 21.5, 21.1; IR (neat) 1698, 1600, 1590, 1570, 1448, 1373, 1308, 1286, 1181, 1153, 1083, 1017, 984, 819, 806, 768, 724, 687, 611, 582, 546, 531 cm⁻¹; HRMS (FAB); Exact mass calcd for C₁₅H₁₆NO₂S [M+H]⁺, 274.0901. Found 274.0899.

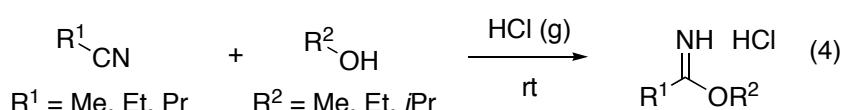
Preparation of sulfonylimidates. Sulfonylimidates **6a-6h** were synthesized by using the similar method to the reported one.⁵ General procedure is as follows (equations 4 and 5).

Imidate HCl salt formation.(eq. 4) HCl gas was bubbled into a mixture of nitrile (400 mmol) and alcohol (400 mmol) for 10-20 min (exothermic). In some cases

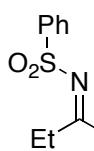
⁴ Matsubara, R.; Doko, T.; Uetake, R.; Kobayashi, S. *Angew. Chem. Int. Ed.* **2007**, *46*, 3047.

⁵ Kupfer, R.; Nagel, M.; Würthwein, E.-U.; Allmann, R. *Chem. Ber.* **1985**, *118*, 3089.

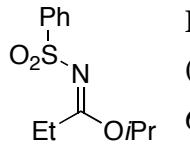
(especially when iPrOH was used as an alcohol), temperature higher than room temperature was necessary for the reaction, that is why the reaction was not cooled during the exothermic reaction. After completion of bubbling of HCl, the mixture was left for 3-10 h under Ar atmosphere. Removal of all the volatiles by evaporation gave us almost pure imidate HCl salt in 40-80% yield (moderate yield may be caused by evaporation of the volatile starting materials during the course of the exothermic reactions), which can be used in the next reaction without further purification. Further purification is possible by washing the solid with dry Et₂O. Imidate HCl salts are hygroscopic, but can be kept under inert gas atmosphere in the refrigerator for at least 1 year.



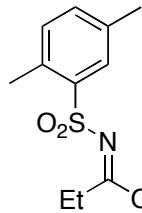
Representative procedure for the syntheses of sulfonylimidates. (eq. 5) To a solution of imidate HCl salt **A** (3.01 g, 19.85 mmol) in dichloromethane (DCM, 50 mL) was added Et₃N (8.3 mL, 59.55 mmol) dropwise at rt. To the resultant suspension was added TsCl (3.785 g, 19.85 mmol) and DMAP (242.5 mg, 1.985 mmol). The reaction mixture was stirred until TsCl was consumed (40 h). The mixture was poured into water, and extracted with DCM. The organics were dried over anhydrous Na₂SO₄. Filtration and evaporation of the solvents afforded the crude product, which was purified by column chromatography on SiO₂ to give us a pure sulfonylimidate **6g** (4.565 g, 85% yield).



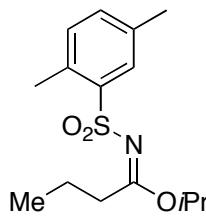
Methyl N-benzenesulfonyl-propionimidate (6a): ¹H NMR (CDCl₃) δ = 7.94-7.98 (m, 2H), 7.48-7.60 (m, 3H), 3.75 (s, 3H), 2.95 (q, 2H, J = 7.6 Hz), 1.24 (t, 3H, J = 7.7 Hz); ¹³C NMR (CDCl₃) δ = 177.6, 142.0, 132.4, 128.7, 126.5, 55.4, 27.5, 10.0; IR (neat) 3063, 2985, 2950, 2887, 1602, 1541, 1507, 1446, 1377, 1305, 1235, 1155, 1091, 1023, 945, 798, 758, 733, 690, 621, 585, 536 cm⁻¹; HRMS (FAB); Exact mass calcd for C₁₀H₁₄NO₃S [M+H]⁺, 228.0694. Found 228.0697.



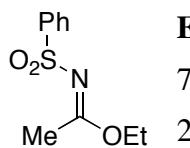
Isopropyl N-benzenesulfonyl-propionimidate (6b): ^1H NMR (CDCl_3); δ = 8.06-8.09 (m, 2H), 7.60-7.64 (m, 3H), 4.70 (sept, 1H, J = 6.3 Hz), 2.88 (qd, 2H, J = 7.3, 1.7 Hz), 1.05 (td, 3H, J = 7.3, 1.2 Hz), 0.83 (d, 6H, J = 6.3 Hz); ^{13}C NMR (CDCl_3) δ = 176.0, 143.6, 132.0, 128.8, 126.9, 71.6, 28.2, 20.9, 10.4; IR (neat) 3055, 2988, 1506, 1448, 1308, 1265, 1157, 1093, 896, 740, 705, 634, 459, 445, 413 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{12}\text{H}_{18}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 256.1007. Found 256.1010.



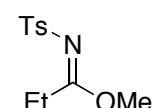
Isopropyl N-(2,5-xylylsulfonyl)-propionimidate (6c): Mp. 32-33 °C; ^1H NMR (CDCl_3); δ = 8.20 (s, 1H), 6.90 (d, 1H, J = 7.4 Hz), 6.84 (dd, 1H, J = 7.4, 1.4 Hz), 4.74 (sept, 1H, J = 6.3 Hz), 2.91 (q, 2H, J = 7.4 Hz), 2.77 (s, 3H), 1.94 (s, 3H), 1.07 (t, 3H, J = 7.4 Hz), 0.83 (d, 6H, J = 6.3 Hz); ^{13}C NMR (CDCl_3) δ = 176.2, 141.2, 135.9, 134.4, 133.0, 132.2, 128.9, 71.2, 28.4, 21.1, 20.6, 20.2, 10.4; IR (neat) 3055, 2988, 1590, 1458, 1308, 1265, 1154, 1092, 1066, 896, 740, 705, 642, 459, 413 cm^{-1} ; HRMS (ESI); Exact mass calcd for $\text{C}_{14}\text{H}_{22}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 284.1320. Found 284.1323.



Isopropyl N-(2,5-xylylsulfonyl)-butyrimidate (6d): Mp. 35-36 °C; ^1H NMR (CDCl_3); δ = 8.18 (s, 1H), 6.92 (d, 1H, J = 8.0 Hz), 6.84 (d, 1H, J = 8.0 Hz), 4.77 (sept, 1H, J = 6.2 Hz), 2.87-2.93 (m, 2H), 2.77 (s, 3H), 1.95 (s, 3H), 1.65 (sext, 2H, J = 7.4 Hz), 0.87 (d, 6H, J = 6.2 Hz), 0.82 (t, 3H, J = 7.4 Hz); ^{13}C NMR (CDCl_3) δ = 175.3, 141.2, 135.9, 134.4, 133.0, 132.2, 128.3, 71.2, 36.5, 21.1, 20.6, 20.2, 20.0, 13.8; IR (neat) 3055, 2987, 2933, 2878, 1590, 1457, 1373, 1303, 1219, 1154, 1102, 1067, 918, 896, 739, 706, 641, 459, 412 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{15}\text{H}_{24}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 298.1477. Found 298.1465.

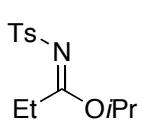


Ethyl N-benzenesulfonyl-acetimidate (6e): ^1H NMR (CDCl_3) δ = 7.94 (d, 2H, J = 7.9 Hz), 7.46-7.60 (m, 3H), 4.16 (q, 2H, J = 7.2 Hz), 2.49 (s, 3H), 1.27 (t, 3H, J = 7.1 Hz); ^{13}C NMR (CDCl_3) δ = 173.7, 141.7, 132.3, 128.6, 126.5, 64.6, 20.5, 13.5; IR (neat) 3066, 2986, 2940, 2904, 1748, 1606, 1474, 1447, 1396, 1376, 1306, 1158, 1118, 1093, 1046, 1121, 998, 869, 803, 735, 689, 633, 588, 567, 525 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{10}\text{H}_{14}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 228.0694. Found 228.0695.

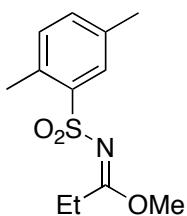


Methyl N-tosyl-propionimidate (6f): Mp. 46-46.5 °C; ^1H NMR (CDCl_3) δ = 7.82 (d, 2H, J = 8.2 Hz), 7.28 (d, 2H, J = 8.2 Hz), 3.72 (s,

3H), 2.92 (q, 2H, J = 7.6 Hz), 2.40 (s, 3H), 1.22 (t, 3H, J = 7.6 Hz); ^{13}C NMR (CDCl_3) δ = 177.4, 143.1, 139.1, 129.3, 126.6, 55.4, 27.4, 21.5, 10.0; IR (neat) 2983, 2949, 1605, 1496, 1464, 1442, 1378, 1314, 1235, 1190, 1155 1092, 1030, 1018, 944.9, 815, 709, 687, 593, 555 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 242.0851. Found 242.0856.



Isopropyl N-tosyl-propionimidate (6g): Mp. 38-39 °C; ^1H NMR (CDCl_3) δ = 7.82 (d, 2H, J = 8.5 Hz), 7.29 (d, 2H, J = 7.9 Hz), 4.97-5.10 (m, 1H), 2.88 (q, 2H, J = 7.6 Hz), 2.42 (s, 3H), 1.23 (d, 6H, J = 6.2 Hz), 1.21 (t, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 176.3, 142.9, 139.4, 129.3, 126.4, 71.9, 27.8, 21.5, 21.1, 10.1; IR (neat) 2983, 2942, 1597, 1496, 1465, 1383, 1356, 1312, 1235, 1183, 1157, 1093, 1032, 1017, 955, 909, 838, 814, 799, 692, 600, 555, 529 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{13}\text{H}_{20}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 270.1164. Found 270.1167.

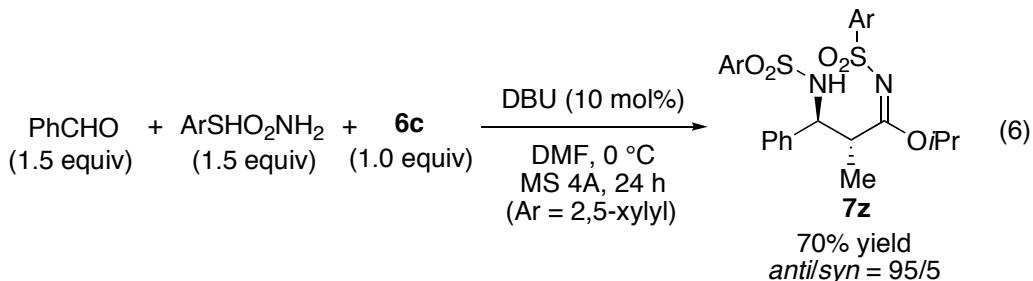


Methyl N-(2,5-xylylsulfonyl)-propionimidate (6h): Mp. 71-72 °C; ^1H NMR (C_6D_6) δ = 8.15-8.17 (m, 1H), 7.16-7.17 (m, 1H), 6.85-6.94 (m, 2H), 3.10 (s, 3H), 2.87 (q, 2H, J = 7.6 Hz), 2.72 (s, 3H), 1.95 (s, 3H), 1.02 (t, 3H, J = 7.4 Hz); ^{13}C NMR (C_6D_6) δ = 177.1, 140.9, 135.9, 134.5, 133.1, 132.3, 128.9, 54.6, 27.9, 20.5, 20.2, 10.0; IR (neat) 2982, 2949, 2884, 1602, 1491, 1461, 1442, 1378, 1307, 1234, 1191, 1154, 1142, 1094, 1065, 1028, 944, 884, 821, 798, 747, 705, 630, 579, 548, 503 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{12}\text{H}_{18}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$, 256.1007. Found 256.1000.

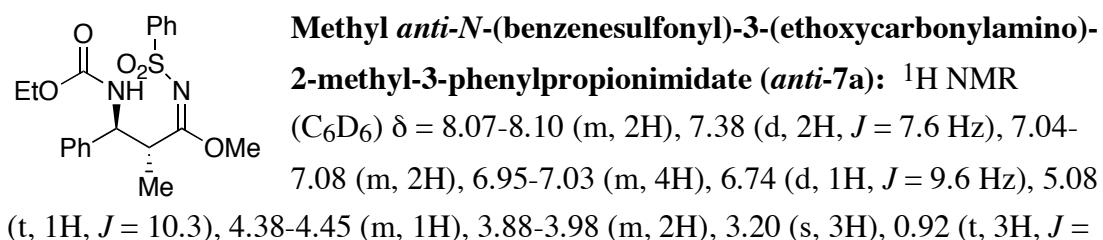
General procedure of addition reactions of sulfonylimidates to imines. To MS 4A (50 mg) was added a solution of imine (0.45 mmol) in DMF (0.5 ml) and sulfonylimidate (0.3 mmol). The mixture was cooled to 0 °C and a solution of DBU (1,8-diazabicyclo[5.4.0]undec-7-ene, 5 mol%) in DMF (100 μl) was added. The mixture was stirred for 24 h and then diluted by addition of Et_2O . The mixture obtained after filtration (for removal of MS 4A) was washed with water 3 times, then dried over anhydrous Na_2SO_4 . Filtration and removal of solvents afforded the crude product. Diastereo ratio was determined by ^1H NMR spectroscopy analysis of the crude product. Purification of the crude product was conducted by chromatography on SiO_2 , to afford the desired product **7**. When it was difficult to determine the

deastereo ratio at the crude product stage, the ratio was determined by the weight of the separated diastereomers after purification.

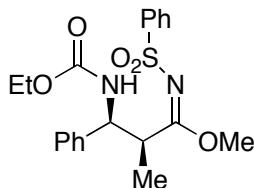
Procedure of addition reaction of sulfonylimidate **6e to *N*-Boc imine **1a** (Table 2, entry 3).** To MS 4A (50 mg) was added a solution of imine **1a** (53.2 mg, 0.3 mmol) in DMF (0.5 ml). The mixture was cooled to 0 °C, then sulfonylimidate **6e** (340.9 mg, 1.5 mmol) and a solution of DBU in DMF (10 mol%, 100 µl) were added successively. The mixture was stirred for 30 min at 0 °C, and then diluted by addition of Et₂O. The mixture obtained after filtration (for removal of MS 4A) was washed with water 3 times, then dried over anhydrous Na₂SO₄. Filtration and removal of solvents afforded the crude product. Purification of the crude product was conducted by chromatography on SiO₂, to afford the desired product **7f** (95.8 mg, 79%) and the recovered sulfonylimidate **6e** (275 mg, 1.21 mmol).



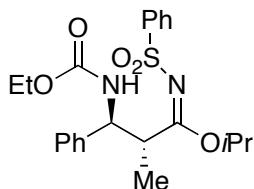
Procedure of 3 component reaction between benzaldehyde, sulfonamide, and sulfonylimidate **6c (equation 6).** To a suspension of MS 4A (100 mg) in DMF (0.5 ml) were added benzaldehyde (45.7 µl, 0.45 mmol) and 2,5-xylylsulfonamide (83.4 mg, 0.45 mmol). The mixture was stirred at RT for 20 min, and then cooled to 0 °C. To the suspension were added sulfonylimidate **6c** (85.0 mg, 0.3 mmol) and a solution of DBU in DMF (10 mol%, 100 µl) successively. The mixture was stirred for 23 h at 0 °C, and then diluted by addition of Et₂O. The mixture obtained after filtration (for removal of MS 4A) was washed with water 3 times, then dried over anhydrous Na₂SO₄. Filtration and removal of solvents afforded the crude product. Diastereo ratio was determined by ¹H NMR spectroscopy analysis of the crude product (*anti/syn* = 95/5). Purification of the crude product was conducted by chromatography on SiO₂, to afford the desired product **7z** (116.8 mg, 70% yield).



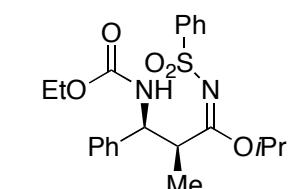
6.9 Hz), 0.88 (t, 3H, J = 7.2 Hz); ^{13}C NMR (C_6D_6) δ = 14.6, 14.9, 45.6, 55.2, 59.4, 60.8, 127.0, 127.6, 128.8, 129.1, 132.4, 140.9, 142.6, 155.8, 177.9; IR (neat) 3375, 3064, 3030, 2982, 2951, 1722, 1602, 1518, 1447, 1369, 1303, 1245, 1222, 1152, 1090, 1059, 1025, 999, 950, 907, 842, 772, 758, 734, 701, 689, 623, 525, 426 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_5\text{S} [\text{M}+\text{H}]^+$, 405.1484. Found 405.1494.



Methyl *syn*-N-(benzenesulfonyl)-3-(ethoxycarbonylamino)-2-methyl-3-phenylpropionimidate (*syn*-7a): Mp. 130-131 °C; ^1H NMR (C_6D_6) δ = 7.93-7.96 (m, 2H), 7.48 (d, 2H, J = 6.9 Hz), 7.13-7.18 (m, 3H), 7.02 (t, 1H, J = 7.2 Hz), 6.90-6.95 (m, 3H), 5.38 (t, 1H, J = 10 Hz), 4.76 (d, 1H, J = 9.6 Hz), 4.30-4.39 (m, 1H), 3.88-4.06 (m, 2H), 2.88 (s, 3H), 1.47 (d, 3H, J = 6.9 Hz), 0.94 (t, 3H, J = 6.9 Hz); ^{13}C NMR (C_6D_6) δ = 176.4, 156.3, 143.0, 141.2, 132.1, 128.8, 128.7, 126.9, 60.8, 60.1, 57.3, 54.6, 44.5, 20.5, 15.5, 14.6, 14.2; IR (neat) 3343, 3063, 2924, 2853, 1715, 1697, 1607, 1532, 1448, 1375, 1305, 1287, 1261, 1237, 1155, 1084, 1035, 951, 927, 905, 733, 701, 689, 622, 586 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_5\text{S} [\text{M}+\text{H}]^+$, 405.1484. Found 405.1502.

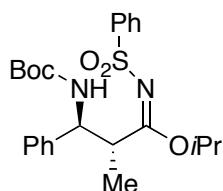


***iso*-Propyl (ethoxycarbonylamino)-2-methyl-3-phenylpropionimidate (*anti*-7b):** Mp. 78 °C; ^1H NMR (CDCl_3); δ = 8.07-8.13 (m, 2H), 7.40 (d, 2H, J = 6.9 Hz), 7.07 (t, 2H, J = 6.9 Hz), 6.98-7.04 (m, 4H), 6.77 (d, 1H, J = 9.6 Hz), 5.09 (t, 1H, J = 11.0 Hz), 4.83 (quint, 1H, J = 6.2 Hz), 4.41 (qd, 1H, J = 11.0, 6.9 Hz), 3.85-4.02 (m, 2H), 1.08 (d, 3H, J = 6.2 Hz), 0.98 (d, 3H, J = 6.2 Hz), 0.88-94 (m, 6H); ^{13}C NMR (CDCl_3) δ = 176.8, 155.7, 143.0, 141.1, 132.4, 129.0, 128.9, 128.2, 127.7, 126.9, 72.5, 60.7, 59.2, 45.7, 20.8, 20.4, 14.9, 14.6; IR (neat) 3063, 3033, 2983, 2938, 2880, 1724, 1593, 1520, 1481, 1456, 1448, 1385, 1374, 1361, 1302, 1245, 1222, 1154, 1091, 1056, 1026, 1000, 974, 929, 909, 852, 829, 769, 758, 738, 702, 689, 671, 636, 597, 566, 537, 504, 470, 418 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{22}\text{H}_{29}\text{N}_2\text{O}_5\text{S} [\text{M}+\text{H}]^+$, 433.1797. Found 433.1803.

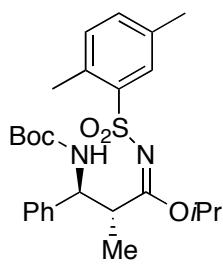


***iso*-Propyl (ethoxycarbonylamino)-2-methyl-3-phenylpropionimidate (*syn*-7b):** Mp. 145-147 °C; ^1H NMR (CDCl_3); δ = 7.97-8.02 (m, 2H), 7.54 (d, 2H, J = 6.9 Hz), 7.13-7.18 (m, 3H), 7.01 (t, 1H, J = 7.5 Hz), 6.93-6.98 (m, 2H), 5.29 (t, 1H, J = 9.7 Hz), 4.52 (d, 1H, J = 9.7 Hz), 4.46 (quint, 1H, J = 6.2 Hz), 4.34 (dq, 1H, J = 9.7, 7.7 Hz), 3.86-4.04 (m, 2H), 1.54 (d,

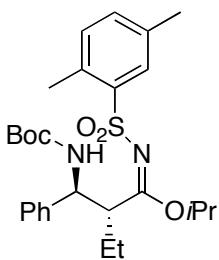
3H, $J = 6.9$ Hz), 0.95 (t, 3H, $J = 6.9$ Hz), 0.71 (d, 3H, $J = 6.2$ Hz), 0.45 (d, 3H, $J = 5.5$ Hz); ^{13}C NMR (CDCl_3) $\delta = 175.2, 156.3, 143.4, 141.4, 132.0, 128.8, 128.7, 128.3, 126.8, 71.8, 60.8, 57.5, 44.2, 20.6, 20.0, 16.0, 14.5$; IR (neat) 3063, 3033, 2982, 2936, 2876, 1698, 1596, 1532, 1481, 1447, 1448, 1382, 1355, 1305, 1286, 1262, 1236, 1157, 1093, 1082, 1034, 1001, 980, 929, 908, 844, 766, 737, 702, 689, 638, 587, 540, 418 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{22}\text{H}_{29}\text{N}_2\text{O}_5\text{S} [\text{M}+\text{H}]^+$, 433.1797. Found 433.1779.



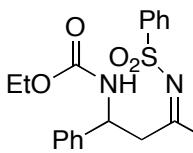
iso-Propyl anti-N-(benzenesulfonyl)-3-(tert-butoxycarbonylamino)-2-methyl-3-phenylpropionimidate (anti-7c): ^{1}H NMR (C_6D_6) $\delta = 8.14$ (d, 2H, $J = 6.8$ Hz), 7.38 (d, 2H, $J = 7.4$ Hz), 6.95-7.07 (m, 6H), 6.66 (d, 1H, $J = 9.6$ Hz), 5.06 (t, 1H, $J = 10.5$ Hz), 4.80-4.86 (m, 1H), 4.32-4.43 (m, 1H), 1.34 (s, 9H), 1.12 (d, 3H, $J = 6.2$ Hz), 0.95 (d, 3H, $J = 6.8$ Hz), 0.91 (d, 3H, $J = 6.2$ Hz); ^{13}C NMR (C_6D_6) $\delta = 17.67, 154.9, 143.2, 132.3, 129.0, 128.8, 127.6, 126.9, 78.7, 72.5, 58.8, 45.8, 28.4, 20.8, 20.6, 14.9$; IR (neat) 3387, 2979, 2938, 1717, 1592, 1508, 1455, 1386, 1364, 1302, 1247, 1155, 1090, 1054, 1002, 973, 909, 851, 757, 737, 700, 688, 635, 590, 538 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{24}\text{H}_{33}\text{N}_2\text{O}_5\text{S} [\text{M}+\text{H}]^+$, 461.2110. Found 461.2099.



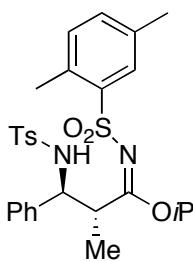
iso-Propyl anti-3-(tert-butoxycarbonylamino)-2-methyl-3-phenyl-N-(2,5-xylylsulfonyl)propionimidate (anti-7d): Mp. 122-123 °C; ^{1}H NMR (CDCl_3); $\delta = 8.19$ (s, 1H), 7.36 (d, 2H, $J = 6.8$ Hz), 6.93-7.05 (m, 4H), 6.89 (d, 1H, $J = 7.3$ Hz), 6.62 (d, 1H, $J = 9.6$ Hz), 5.03 (t, 1H, $J = 10.5$ Hz), 4.87 (quint, 1H, $J = 6.2$ Hz), 4.48 (sext, 1H, $J = 5.9$ Hz), 2.86 (s, 3H), 1.96 (s, 3H), 1.34 (s, 9H), 1.15 (d, 3H, $J = 5.7$ Hz), 0.98 (d, 3H, $J = 6.2$ Hz), 0.91 (d, 3H, $J = 6.2$ Hz); ^{13}C NMR (CDCl_3) $\delta = 177.5, 154.7, 141.4, 140.5, 136.1, 134.5, 133.4, 132.4, 128.9, 128.3, 127.9, 127.7, 78.6, 72.1, 58.9, 46.0, 28.4, 21.0, 21.0, 20.6, 20.6, 14.9$; IR (neat) 3060, 3032, 2841, 2936, 2881, 1715, 1588, 1513, 1495, 1456, 1389, 1365, 1299, 1267, 1248, 1225, 1155, 1102, 1066, 1053, 1004, 973, 930, 910, 884, 851, 824, 738, 707, 644, 599, 553, 499, 465 cm^{-1} ; HRMS (ESI); Exact mass calcd for $\text{C}_{26}\text{H}_{37}\text{N}_2\text{O}_5\text{S} [\text{M}+\text{H}]^+$, 489.2423. Found 489.2417.



iso-Propyl anti-3-(tert-butoxycarbonylamino)-2-ethyl-3-phenyl-N-(2,5-xylylsulfonyl)propionimidate (anti-7e): Mp. 95-96 °C; ^1H NMR (CDCl_3) δ = 8.18 (s, 1H), 7.44 (d, 2H, J = 7.4 Hz), 6.93-7.08 (m, 4H), 6.80-6.92 (m, 2H), 5.08 (t, 1H, J = 10.5 Hz), 4.93 (quint, 1H, J = 6.2 Hz), 4.32 (td, 1H, J = 11.4, 4.0 Hz), 2.87 (s, 3H), 1.95 (s, 3H), 1.59-1.65 (m, 1H), 1.34 (s, 9H), 1.20-1.40 (m, 1H), 1.16 (d, 3H, J = 6.2 Hz), 0.88-0.95 (m, 6H); ^{13}C NMR (CDCl_3) δ = 176.9, 153.8, 141.9, 140.6, 136.1, 134.5, 133.4, 53.7, 58.4, 132.4, 129.0, 128.9, 128.3, 127.7, 78.6, 72.0, 28.4, 23.9, 21.1, 21.0, 20.7, 20.6, 11.8; IR (neat) 3062, 3031, 2978, 2934, 2878, 2717, 1717, 1586, 1509, 1496, 1458, 1390, 1365, 1297, 1251, 1202, 1154, 1100, 1065, 1011, 991, 915, 885, 865, 837, 822, 774, 747, 707, 642, 599, 553, 532, 499, 464, 437, 419 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{27}\text{H}_{39}\text{N}_2\text{O}_5\text{S}$ [M+H] $^+$, 503.2580. Found 503.2554.

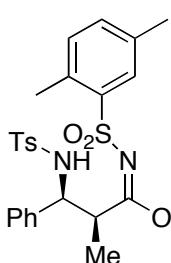


Ethyl N-(benzenesulfonyl)-3-(ethoxycarbonylamino)-3-phenylpropionimidate (7f): Mp. 80-81 °C; ^1H NMR (C_6D_6) δ = 8.05-8.10 (m, 2H), 7.26-7.30 (m, 2H), 7.00-7.10 (m, 6H), 6.26 (d, 1H, J = 9.1 Hz), 5.47-5.57 (m, 1H), 3.92-4.03 (m, 2H), 3.59-3.81 (m, 3H), 2.95 (dd, 1H, J = 5.1, 14.2 Hz), 0.95 (t, 3H, J = 7.1 Hz), 0.82 (t, 3H, J = 7.1 Hz); ^{13}C NMR (C_6D_6) δ = 172.8, 155.9, 142.7, 142.0, 132.4, 129.0, 128.8, 127.0, 126.6, 64.8, 60.8, 53.3, 41.1, 14.6, 13.2; IR (neat) 3360, 3065, 2982, 1719, 1599, 1523, 1473, 1446, 1397, 1374, 1305, 1243, 1155, 1091, 1041, 888, 755, 734, 700, 689, 631 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_5\text{S}$ [M+H] $^+$, 405.1484. Found 405.1487.

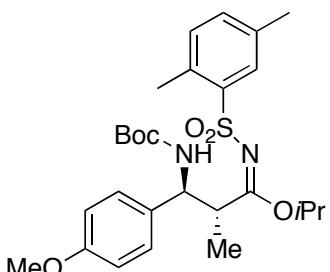


iso-Propyl anti-2-methyl-3-phenyl-3-(p-toluenesulfonylamino)-N-(2,5-xylylsulfonyl)propionimidate (anti-7g): Mp. 140-142 °C; ^1H NMR (CDCl_3) δ = 8.21 (s, 1H), 7.34 (d, 2H, J = 7.9 Hz), 6.98 (d, 1H, J = 7.9 Hz), 6.86-6.95 (m, 3H), 6.70-6.86 (m, 4H), 6.48 (d, 2H, J = 8.5 Hz), 5.02 (quint, 1H, J = 6.2 Hz), 4.69 (t, 1H, J = 10.2 Hz), 4.30 (qd, 1H, J = 10.7, 6.8 Hz), 3.11 (s, 3H), 1.97 (s, 3H), 1.81 (s, 3H), 1.40 (d, 3H, J = 5.2 Hz), 1.00 (d, 3H, J = 6.2 Hz), 0.93 (d, 3H, J = 6.8 Hz); ^{13}C NMR (CDCl_3) δ = 176.9, 141.8, 140.2, 139.3, 138.6, 136.1, 135.0, 133.6, 132.6, 128.9, 128.8, 128.6, 127.6, 127.0, 73.0, 62.1, 46.7, 21.4, 21.1, 20.9, 20.6, 20.5, 14.6; IR (neat) 3056, 2986, 2938, 2880, 1589, 1494, 1457, 1438, 1423, 1386, 1355, 1333, 1305, 1291, 1266, 1162, 1101, 1092, 1063, 975, 910, 896, 856, 822, 814, 738, 706,

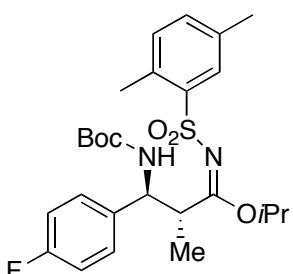
668, 645, 581, 565, 549, 500, 463, 418 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₈H₃₅N₂O₅S₂ [M+H]⁺, 543.1987. Found 543.1990.



***iso*-Propyl *syn*-2-methyl-3-phenyl-3-(*p*-toluenesulfonylamino)-*N*-(2,5-xylsulfonyl)propionimidate (*syn*-7g):** Mp. 160-161 °C; ¹H NMR (CDCl₃) δ = 7.80 (s, 1H), 7.46 (d, 2H, *J* = 8.5 Hz), 7.21 (d, 1H, *J* = 7.4 Hz), 7.11 (d, 1H, *J* = 7.9 Hz), 7.00-7.07 (m, 7H), 5.17 (d, 1H, *J* = 9.6 Hz), 4.68-4.77 (m, 1H), 4.52 (t, 1H, *J* = 9.4 Hz), 4.10-4.17 (m, 1H), 2.39 (s, 3H), 2.35 (s, 3H), 2.31 (s, 3H), 1.44 (d, 3H, *J* = 6.8 Hz), 1.14 (d, 3H, *J* = 6.2 Hz), 0.78 (d, 3H, *J* = 6.2 Hz); ¹³C NMR (CDCl₃) δ = 17.46, 142.9, 139.5, 138.5, 137.8, 135.6, 134.2, 133.1, 132.0, 129.2, 128.1, 128.0, 127.6, 127.4, 127.0, 72.1, 60.3, 44.6, 21.4, 21.1, 20.8, 20.5, 19.6, 15.6; IR (neat) 3276, 3032, 2983, 2877, 1592, 1492, 1455, 1381, 1331, 1303, 1184, 1159, 1096, 1062, 981, 909, 813, 764, 733, 704, 669, 646, 607, 562, 511 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₈H₃₅N₂O₅S₂ [M+H]⁺, 543.1987. Found 543.1971.

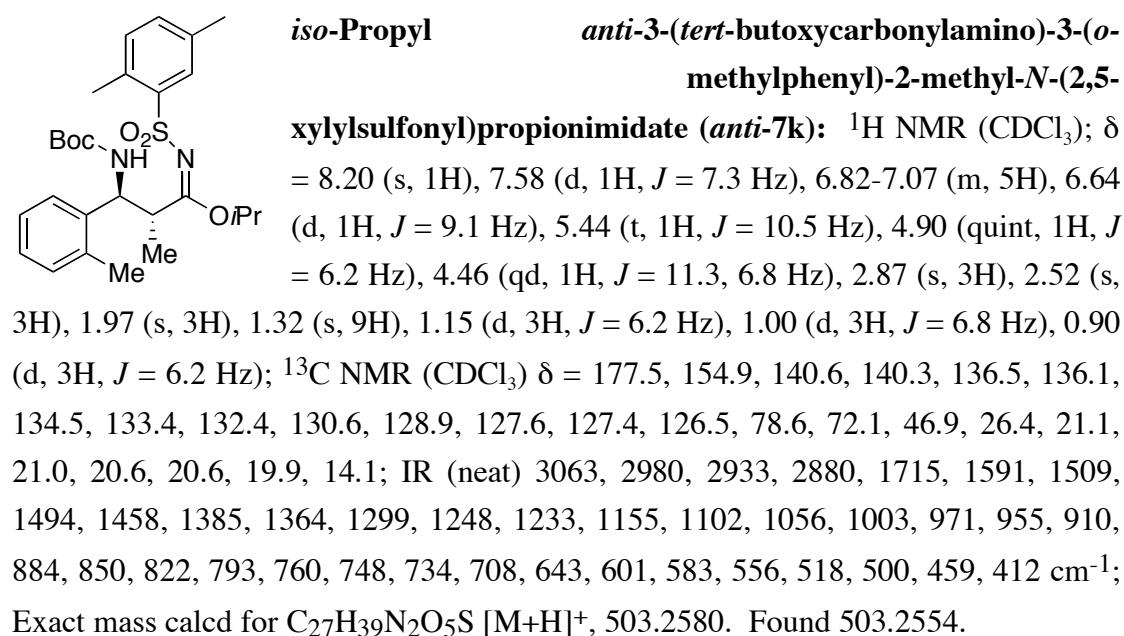
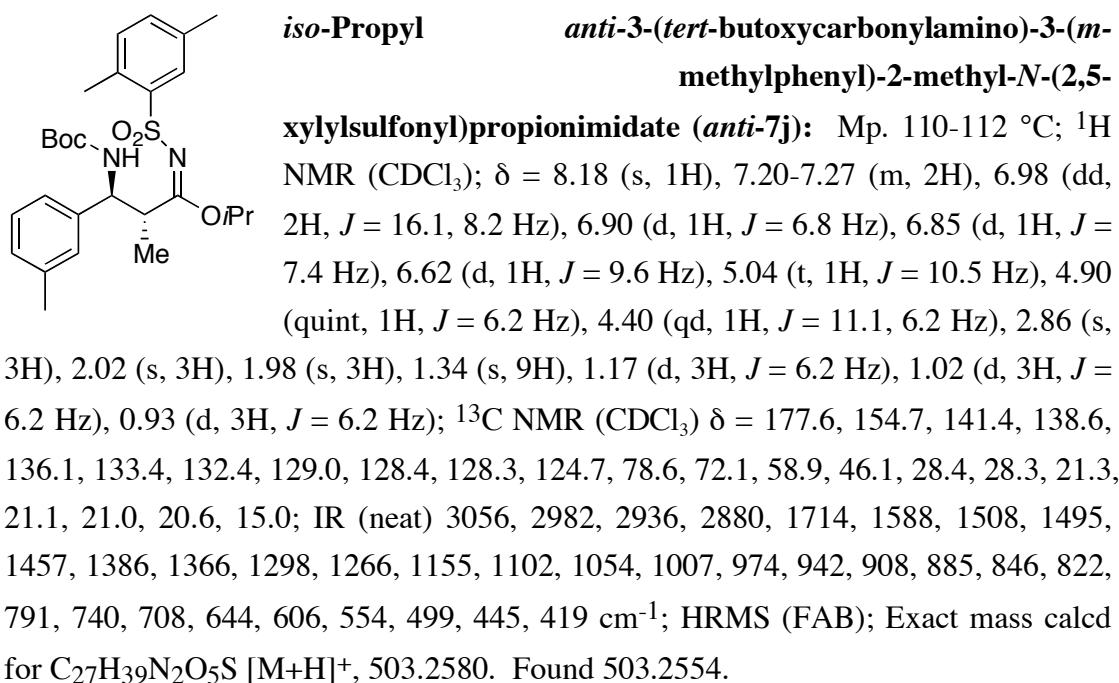


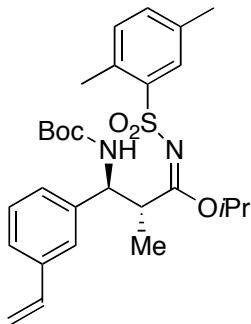
***iso*-Propyl *anti*-3-(*tert*-butoxycarbonylamino)-3-(*p*-methoxyphenyl)-2-methyl-*N*-(2,5-xylsulfonyl)propionimidate (*anti*-7h):** Mp. 142-145 °C; ¹H NMR (CDCl₃); δ = 8.20 (s, 1H), 7.29 (d, 2H, *J* = 9.1 Hz), 6.98 (d, 1H, *J* = 7.4 Hz), 6.89 (d, 1H, *J* = 7.4 Hz), 6.55-6.65 (m, 3H), 5.02 (t, 1H, *J* = 9.5 Hz), 4.91 (quint, 1H, *J* = 6.2 Hz), 4.38 (qd, 1H, *J* = 11.3, 6.2 Hz), 3.27 (s, 3H), 2.87 (s, 3H), 1.97 (s, 3H), 1.36 (s, 9H), 1.16 (d, 3H, *J* = 6.2 Hz), 1.04 (d, 3H, *J* = 6.8 Hz), 0.93 (d, 3H, *J* = 6.2 Hz); ¹³C NMR (CDCl₃) δ = 177.7, 159.6, 154.7, 140.6, 136.1, 134.6, 133.4, 132.4, 131.8, 128.9, 128.7, 114.5, 78.6, 72.1, 58.4, 54.7, 46.2, 28.4, 21.0, 21.0, 20.6, 20.6, 15.0; IR (neat) 3055, 2984, 2937, 1713, 1586, 1513, 1457, 1422, 1386, 1365, 1299, 1265, 1155, 1101, 1054, 1036, 910, 832, 740, 706, 644, 609, 419 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₇H₃₉N₂O₆S [M+H]⁺, 519.2529. Found 519.2512.



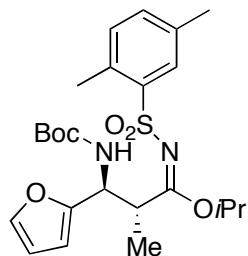
***iso*-Propyl *anti*-3-(*tert*-butoxycarbonylamino)-3-(*p*-fluorophenyl)-2-methyl-*N*-(2,5-xylsulfonyl)propionimidate (*anti*-7i):** Mp. 131-133 °C; ¹H NMR (CDCl₃); δ = 8.19 (s, 1H), 7.12-7.21 (m, 2H), 6.96 (d, 1H, *J* = 7.4 Hz), 6.88 (d, 1H, *J* = 7.4 Hz), 6.59-6.69 (m, 2H), 4.95 (t, 1H, *J* = 10.2 Hz), 4.87 (quint, 1H, *J* = 6.0 Hz),

4.29 (sext, 1H, J = 6.4 Hz), 2.85 (s, 3H), 1.95 (s, 3H), 1.35 (s, 9H), 1.14 (d, 3H, J = 5.6 Hz), 0.95 (d, 3H, J = 6.2 Hz), 0.91 (d, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 177.2, 160.6 (d, $J_{\text{C}-\text{F}}$ = 244.3 Hz), 154.7, 140.4, 137.1, 136.2, 134.5, 133.5, 132.4, 129.3 (d, $J_{\text{C}-\text{F}}$ = 8.4 Hz), 128.9, 115.7 (d, $J_{\text{C}-\text{F}}$ = 21.5 Hz), 78.8, 72.1, 58.2, 46.0, 28.4, 21.0, 21.0, 20.6, 14.8; IR (neat) 3056, 2984, 2933, 1714, 1588, 1542, 1457, 1366, 1298, 1266, 1226, 1156, 1096, 1055, 837, 741, 706, 644, 507, 527, 499 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{26}\text{H}_{36}\text{N}_2\text{O}_5\text{SF}$ [$\text{M}+\text{H}]^+$, 507.2329. Found 507.2326.

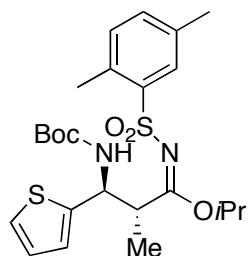




iso-Propyl anti-3-(tert-butoxycarbonylamino)-3-(*m*-vinylphenyl)-2-methyl-*N*-(2,5-xylylsulfonyl)propionimidate (anti-7l): Mp. 87-91 °C; ^1H NMR (CDCl_3); δ = 8.19 (s, 1H), 7.52 (s, 1H), 7.28 (d, 1H, J = 7.9 Hz), 7.10 (d, 1H, J = 7.4 Hz), 6.98 (t, 1H, J = 7.9 Hz), 6.96 (d, 1H, J = 7.4 Hz), 6.88 (d, 1H, J = 7.9 Hz), 6.67 (d, 1H, J = 9.6 Hz), 6.46 (dd, 1H, J = 17.6, 10.7 Hz), 5.57 (d, 1H, J = 17.6 Hz), 5.07 (t, 1H, J = 10.2 Hz), 5.00 (d, 1H, J = 10.7 Hz), 4.88 (quint, 1H, J = 6.2 Hz), 4.41 (qd, 1H, J = 11.3, 6.8 Hz), 2.86 (s, 3H), 1.96 (s, 3H), 1.34 (s, 9H), 1.16 (d, 3H, J = 6.2 Hz), 0.98 (d, 3H, J = 6.8 Hz), 0.92 (d, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 177.4, 154.7, 141.8, 140.5, 138.4, 136.9, 136.2, 134.5, 133.4, 132.4, 129.4, 128.9, 126.8, 126.1, 125.7, 114.1, 78.7, 72.1, 58.9, 46.1, 28.4, 21.0, 20.6, 20.6, 14.9; IR (neat) 3087, 3048, 2981, 2933, 2879, 1718, 1602, 1507, 1457, 1389, 1363, 1309, 1250, 1140, 1092, 1055, 1004, 973, 908, 857, 844, 804, 784, 748, 708, 682, 643, 600, 552, 503, 460, 412 cm⁻¹; HRMS (FAB); Exact mass calcd for $\text{C}_{28}\text{H}_{39}\text{N}_2\text{O}_5\text{S}$ [M+H]⁺, 515.2580. Found 515.2570.

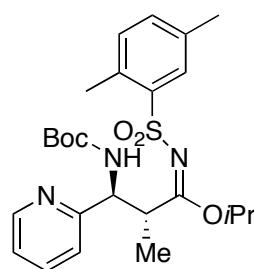


iso-Propyl anti-3-(tert-butoxycarbonylamino)-3-(2-furyl)-2-methyl-*N*-(2,5-xylylsulfonyl)propionimidate (anti-7m): Mp. 84 °C; ^1H NMR (CDCl_3); δ = 8.17 (s, 1H), 6.90-6.97 (m, 2H), 6.87 (d, 1H, J = 7.4 Hz), 6.18 (d, 1H, J = 9.6 Hz), 6.09 (d, 1H, J = 2.7 Hz), 5.90-5.94 (m, 1H), 5.21 (t, 1H, J = 10.2 Hz), 4.87 (quint, 1H, J = 6.2 Hz), 4.59 (qd, 1H, J = 11.3, 6.8 Hz), 2.82 (s, 3H), 1.95 (s, 3H), 1.34 (s, 9H), 1.13 (d, 3H, J = 6.2 Hz), 1.06 (d, 3H, J = 6.7 Hz), 0.89 (d, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 176.6, 154.6, 153.1, 140.6, 136.1, 134.6, 133.3, 132.4, 128.9, 110.3, 108.1, 78.8, 72.1, 52.1, 43.7, 28.3, 21.0, 21.0, 20.6, 20.5, 14.8; IR (neat) 3139, 3121, 3056, 2980, 2936, 2879, 1715, 1583, 1509, 1456, 1390, 1367, 1299, 1256, 1200, 1155, 1137, 1103, 1064, 1051, 1010, 971, 910, 884, 860, 848, 822, 798, 754, 731, 709, 680, 644, 592, 556, 498, 424 cm⁻¹; HRMS (FAB); Exact mass calcd for $\text{C}_{24}\text{H}_{35}\text{N}_2\text{O}_6\text{S}$ [M+H]⁺, 479.2216. Found 479.2225.

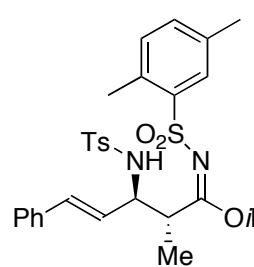


iso-Propyl anti-3-(tert-butoxycarbonylamino)-2-methyl-3-(2-thienyl)-*N*-(2,5-xylylsulfonyl)propionimidate (anti-7n): Mp. 104-107 °C; ^1H NMR (CDCl_3); δ = 8.17 (s, 1H), 6.85-7.00 (m, 3H), 6.76 (d, 1H, J = 4.6 Hz), 6.61 (dd, 1H, J = 5.1, 3.4 Hz), 6.32 (d, 1H, J = 9.1 Hz), 5.35 (t, 1H, J = 10.5 Hz), 4.87 (quint, 1H, J = 6.2 Hz), 4.59 (qd, 1H, J = 10.8, 6.8 Hz), 2.81 (s, 3H),

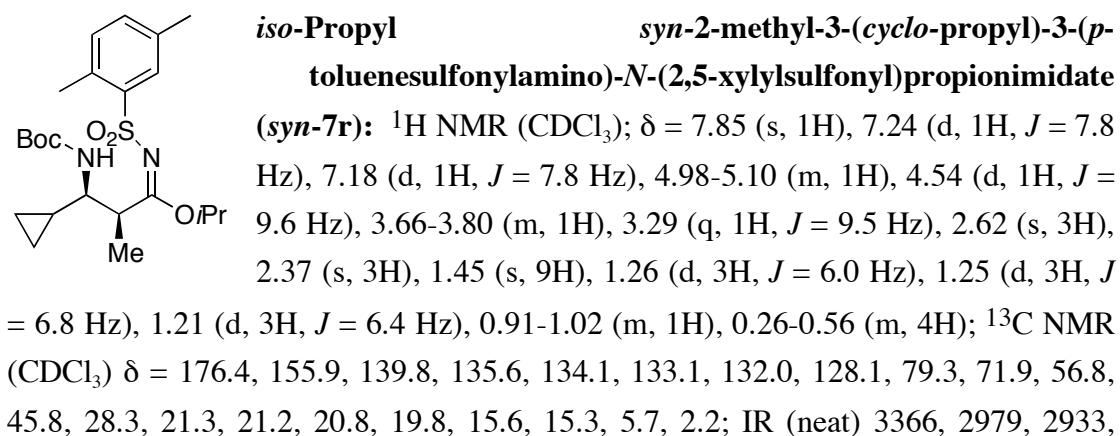
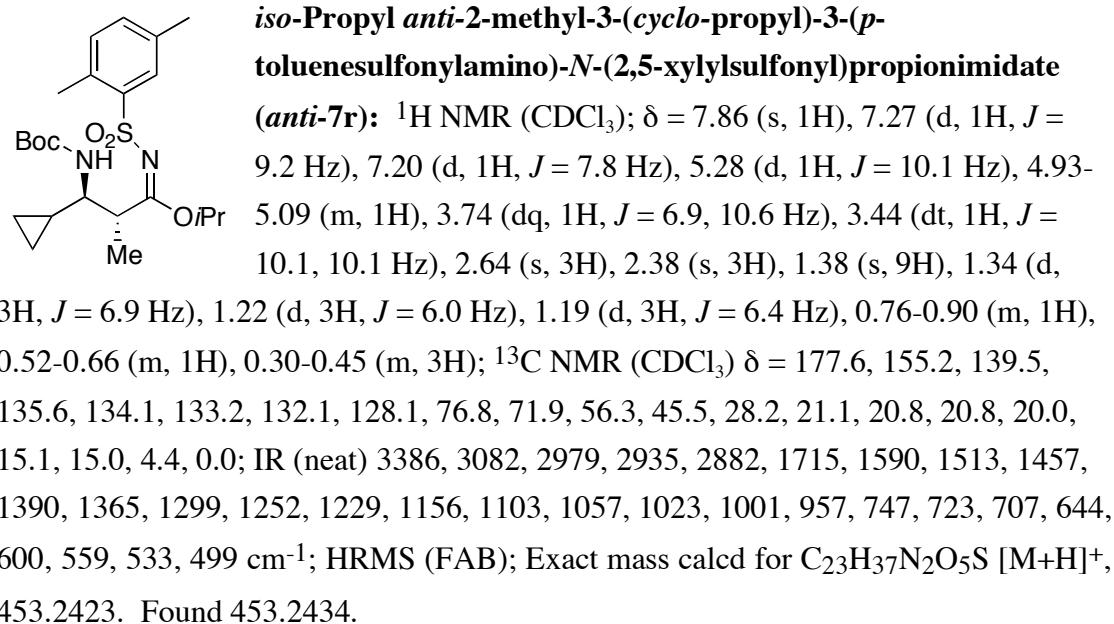
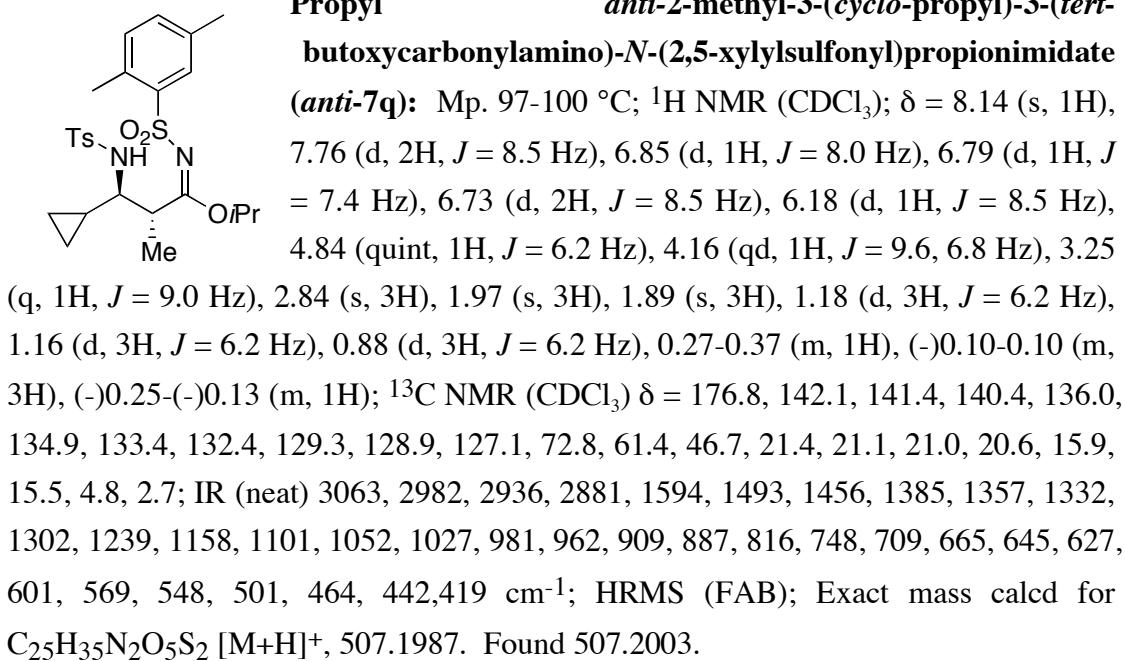
1.96 (s, 3H), 1.33 (s, 9H), 1.10 (d, 6H, J = 6.8 Hz), 0.88 (d, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 176.7, 154.6, 144.7, 140.5, 136.1, 134.5, 133.4, 132.4, 128.9, 127.1, 125.6, 124.8, 78.9, 72.1, 54.0, 46.3, 26.4, 21.0, 20.6, 20.5, 15.2; IR (neat) 3056, 2983, 2934, 2880, 1714, 1588, 1507, 1494, 1456, 1420, 1387, 1366, 1299, 1265, 1233, 1155, 1101, 1054, 1003, 973, 909, 850, 822, 739, 707, 644, 607, 553, 503, 412 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{24}\text{H}_{35}\text{N}_2\text{O}_5\text{S}_2$ [M+H] $^+$, 495.1987. Found 495.1980.



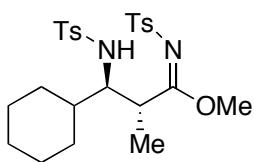
***iso*-Propyl *anti*-3-(*tert*-butoxycarbonylamino)-2-methyl-3-(2-pyridyl)-N-(2,5-xylylsulfonyl)propionimidate (*anti*-7o):** Mp. 113-115 °C; ^1H NMR (CDCl_3) δ = 8.79 (s, 1H), 8.35-8.45 (m, 1H), 8.17 (s, 1H), 7.46 (d, 1H, J = 7.9 Hz), 6.96 (d, 1H, J = 7.4 Hz), 6.87 (d, 1H, J = 7.4 Hz), 6.60-6.73 (m, 2H), 5.00 (t, 1H, J = 9.9 Hz), 4.84 (quint, 1H, J = 6.2 Hz), 4.27 (qd, 1H, J = 11.3, 6.8 Hz), 2.83 (s, 3H), 1.96 (s, 3H), 1.33 (s, 9H), 1.11 (d, 3H, J = 6.2 Hz), 0.88 (d, 3H, J = 6.2 Hz), 0.87 (d, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 176.9, 154.7, 150.0, 149.7, 140.3, 136.4, 136.2, 134.5, 133.8, 133.6, 132.4, 128.9, 123.9, 79.0, 72.3, 56.6, 45.6, 28.4, 21.0, 20.9, 20.6, 14.7; IR (neat) 3059, 2980, 2934, 2880, 1715, 1591, 1507, 1495, 1456, 1429, 1390, 1366, 1298, 1255, 1154, 1101, 1054, 1027, 1005, 973, 910, 883, 852, 820, 779, 735, 708, 643, 602, 554, 499, 460, 412 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{25}\text{H}_{36}\text{N}_3\text{O}_5\text{S}$ [M+H] $^+$, 490.2376. Found 490.2356.



***iso*-Propyl *anti*-2-methyl-5-phenyl-3-(p-toluenesulfonylamino)-N-(2,5-xylylsulfonyl)pent-4-enimide (*anti*-7p):** Mp. 125-128 °C; ^1H NMR (CDCl_3) δ = 8.22 (s, 1H), 7.67 (d, 2H, J = 7.9 Hz), 6.98-7.04 (m, 3H), 6.95 (d, 1H, J = 7.9 Hz), 6.88 (dd, 1H, J = 7.4, 1.1 Hz), 6.73 (dd, 2H, J = 7.6, 2.0 Hz), 6.58 (d, 2H, J = 7.9 Hz), 6.37 (d, 1H, J = 9.1 Hz), 5.84 (d, 1H, J = 15.9 Hz), 5.24 (dd, 1H, J = 15.9, 9.1 Hz), 5.00 (quint, 1H, J = 6.2 Hz), 4.27 (q, 1H, J = 9.8 Hz), 4.30 (qd, 1H, J = 10.8, 6.8 Hz), 2.97 (s, 3H), 1.97 (s, 3H), 1.70 (s, 3H), 1.37 (d, 3H, J = 6.2 Hz), 1.12 (d, 3H, J = 6.2 Hz), 0.98 (d, 3H, J = 6.2 Hz); ^{13}C NMR (CDCl_3) δ = 176.7, 142.4, 140.3, 139.8, 136.2, 136.0, 134.9, 134.3, 133.5, 132.5, 129.6, 129.3, 128.8, 128.3, 127.6, 126.8, 126.2, 72.9, 60.7, 44.7, 21.4, 21.1, 20.9, 20.6, 20.6, 14.8; IR (neat) 3060, 3029, 2982, 2935, 2877, 1595, 1493, 1457, 1385, 1338, 1303, 1212, 1183, 1153, 1104, 1050, 971, 909, 887, 815, 752, 708, 668, 646, 625, 599, 573, 545, 517, 501, 465, 437, 419 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{30}\text{H}_{37}\text{N}_2\text{O}_5\text{S}_2$ [M+H] $^+$, 569.2144. Found 569.2150. ***iso*-**

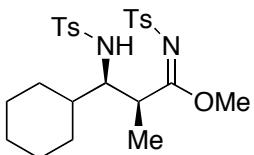


1698, 1591, 1519, 1455, 1365, 1286, 1155, 1103, 1060, 1018, 956, 910, 824, 748, 706, 644, 500 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₃H₃₇N₂O₅S [M+H]⁺, 453.2423. Found 453.2400.



Methyl *anti*-2-methyl-3-(cyclohexyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate

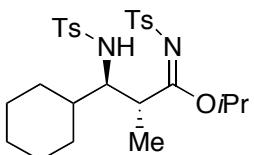
(anti-7s): ¹H NMR (CDCl₃) δ = 7.86 (apparent d, 2H, J = 8.5 Hz), 7.70 (d, 2H, J = 7.9 Hz), 7.32 (d, 2H, J = 7.9 Hz), 7.19 (d, 2H, J = 7.9 Hz), 5.60 (d, 1H, J = 9.6 Hz), 3.87 (dq, 1H, J = 6.2, 10.2 Hz), 3.56 (dt, 1H, J = 2.3, 9.6 Hz), 3.50 (s, 3H), 2.44 (s, 3H), 2.37 (s, 3H), 1.72-1.80 (m, 1H), 1.54-1.68 (m, 3H), 1.36-1.52 (m, 2H), 1.20 (d, 3H, J = 6.8 Hz), 1.08-1.18 (m, 2H), 0.84 (m, 3H); ¹³C NMR (CDCl₃) δ = 177.5, 143.3, 142.6, 139.6, 129.3, 129.2, 126.6, 61.9, 55.5, 41.7, 39.8, 30.9, 26.3, 26.1, 25.4, 21.5, 21.4, 15.5; IR (neat) 3306, 2928, 2854, 1600, 1495, 1447, 1380, 1329, 1303, 1289, 1182, 1158, 1091, 1039, 1011, 951, 913, 887, 862, 814, 735, 706, 689, 596, 567, 547 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₅H₃₅N₂O₅S₂ [M+H]⁺, 507.1987. Found 507.1970.



Methyl *syn*-2-methyl-3-(cyclohexyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate

(syn-7s): ¹H NMR (CDCl₃) δ = 7.85 (d, 2H, J = 8.2 Hz), 7.73 (d, 2H, J = 8.2 Hz), 7.30 (d, 2H, J = 8.2 Hz), 7.27 (d, 2H, J = 8.2 Hz), 4.43 (d, 1H, J = 9.6 Hz), 3.79-3.85 (m, 1H), 3.67-3.73 (m, 4H), 2.43 (s, 3H), 2.41 (s, 3H), 1.46-1.70 (m, 5H), 0.93-1.20 (m, 8H), 0.55-0.65 (m, 1H); ¹³C NMR (CDCl₃) δ = 176.6, 143.1, 143.1, 139.2, 138.7, 129.4, 129.3, 127.0, 126.6, 60.3, 55.7, 41.2, 40.9, 30.3, 27.6, 26.2, 26.0, 25.9, 21.5, 21.5, 14.2, 13.5; IR (neat) 3293, 2928, 2854, 1599, 1496, 1448, 1319, 1300, 1185, 1157, 862, 814, 737, 687, 606, 570, 553 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₅H₃₅N₂O₅S₂ [M+H]⁺, 507.1987.

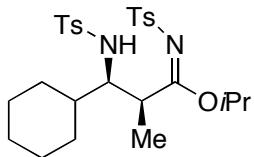
Found 507.1970.



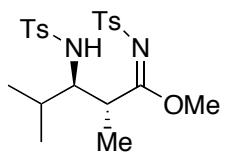
iso-Propyl *anti*-2-methyl-3-(cyclohexyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate

(anti-7t): ¹H NMR (CDCl₃) δ = 7.85 (d, 2H, J = 8.2 Hz), 7.71 (d, 2H, J = 8.2 Hz), 7.30 (d, 2H, J = 8.2 Hz), 7.20 (d, 2H, J = 7.8 Hz), 5.68 (d, 1H, J = 9.6 Hz), 4.76-4.88 (m, 1H), 3.84 (dq, 1H, J = 6.9, 10.0 Hz), 3.59 (dt, 1H, J = 2.7, 9.2 Hz), 2.43 (s, 3H), 2.37 (s, 3H), 0.75-1.75 (m, 11H), 1.30 (d, 3H, J = 6.4 Hz), 1.22 (d, 3H, J = 6.0 Hz), 1.17 (d, 3H, J = 6.4 Hz); ¹³C NMR (CDCl₃) δ = 176.4, 143.1, 142.5, 139.8, 129.3, 129.1, 126.5, 73.1, 61.9, 41.8, 39.8, 30.8,

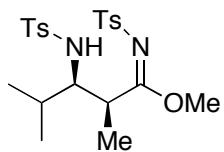
26.3, 26.1, 25.8, 21.5, 21.4, 21.2, 20.8, 15.7; IR (neat) 3277, 2982, 2929, 2854, 2308, 1593, 1495, 1449, 1385, 1374, 1331, 1302, 1182, 1158, 1091, 1020, 968, 909, 887, 862, 840, 814, 735, 694, 671, 605, 582, 547 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₇H₃₉N₂O₅S₂ [M+H]⁺, 535.2300. Found 535.2297.



***iso*-Propyl *syn*-2-methyl-3-(cyclo-hexyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate (*syn*-7t):** ¹H NMR (CDCl₃) δ = 7.81 (d, 2H, *J* = 8.2 Hz), 7.75 (d, 2H, *J* = 8.2 Hz), 7.25-7.33 (m, 4H), 4.97-5.08 (m, 1H), 4.39 (d, 1H, *J* = 8.7 Hz), 3.71 (dq, 1H, *J* = 6.4, 9.2 Hz), 3.62 (dt, 1H, *J* = 3.2, 9.2 Hz), 2.42 (s, 6H), 0.65-1.71 (m, 11H), 1.25 (d, 3H, *J* = 6.4 Hz), 1.22 (d, 3H, *J* = 6.4 Hz), 1.06 (d, 3H, *J* = 6.9 Hz); ¹³C NMR (CDCl₃) δ = 175.5, 143.1, 143.0, 139.3, 138.9, 129.4, 129.3, 127.0, 126.5, 72.3, 60.8, 41.6, 41.4, 31.0, 26.6, 26.4, 26.1, 26.0, 21.5, 21.5, 21.0, 20.9, 15.1; IR (neat) 3295, 2981, 2928, 2854, 1596, 1496, 1449, 1381, 1300, 1184, 1159, 1093, 1053, 1019, 982, 960, 909, 839, 814, 718, 693, 609, 569, 547 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₇H₃₉N₂O₅S₂ [M+H]⁺, 535.2300. Found 535.2297.

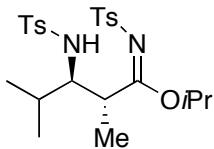


Methyl *anti*-2-methyl-3-(*iso*-propyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate (*anti*-7u): Mp. 147-148 °C; ¹H NMR (CDCl₃) δ = 7.88 (d, 2H, *J* = 8.5 Hz), 7.71 (d, 2H, *J* = 8.5 Hz), 7.32 (d, 2H, *J* = 7.9 Hz), 7.19 (d, 2H, *J* = 7.9 Hz), 5.63 (d, 1H, *J* = 9.6 Hz), 3.82 (dq, 1H, *J* = 6.8, 10.2 Hz), 3.62 (dt, 1H, *J* = 2.8, 10.2 Hz), 3.57 (s, 3H), 2.44 (s, 3H), 2.37 (s, 3H), 1.83-1.93 (m, 1H), 1.21 (d, 3H, *J* = 6.2 Hz), 0.83 (d, 3H, *J* = 6.8 Hz), 0.71 (d, 3H, *J* = 6.8 Hz); ¹³C NMR (CDCl₃) δ = 177.4, 143.3, 142.6, 139.6, 138.7, 129.3, 129.2, 126.7, 126.6, 62.2, 55.6, 42.5, 28.9, 21.5, 21.4, 20.6, 15.3, 14.8; IR (neat) 3307, 2965, 1601, 1540, 1496, 1455, 1329, 1304, 1287, 1157, 1090, 1044, 950, 814, 734, 688, 666, 596, 584, 547 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₂H₃₁N₂O₅S₂ [M+H]⁺, 467.1674. Found 467.1673.

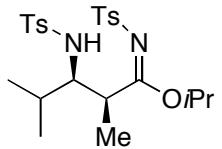


Methyl *syn*-2-methyl-3-(*iso*-propyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate (*syn*-7u): ¹H NMR (CDCl₃) δ = 7.83 (d, 2H, *J* = 8.2 Hz), 7.73 (d, 2H, *J* = 8.2 Hz), 7.29 (d, 2H, *J* = 8.2 Hz), 7.25 (d, 2H, *J* = 8.2 Hz), 4.51 (d, 1H, *J* = 10.1 Hz), 3.71 (s, 3H), 3.66-3.80 (m, 2H), 2.41 (s, 3H), 2.39 (s, 3H), 1.49-1.63 (m, 1H), 1.13 (d, 3H, *J* = 6.9 Hz), 0.76 (d, 3H, *J* = 6.9 Hz), 0.71 (d, 3H, *J* =

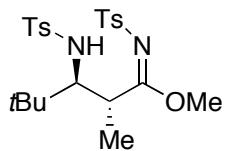
6.9 Hz); ^{13}C NMR (CDCl_3) δ = 176.6, 143.2, 143.1, 139.1, 138.8, 129.5, 129.4, 126.9, 126.5, 60.9, 55.6, 41.8, 31.2, 21.5, 21.5, 20.4, 16.7, 14.2; IR (neat) 3295, 2965, 2882, 1599, 1495, 1455, 1436, 1325, 1290, 1157, 1092, 1039, 952, 909, 814, 688, 666, 603, 573, 548 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{22}\text{H}_{31}\text{N}_2\text{O}_5\text{S}_2$ [$\text{M}+\text{H}]^+$, 467.1674. Found 467.1682.



iso-Propyl anti-2-methyl-3-(iso-propyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate (anti-7v): ^1H NMR (CDCl_3) δ = 7.87 (d, 2H, J = 8.2 Hz), 7.72 (d, 2H, J = 8.2 Hz), 7.31 (d, 2H, J = 7.7 Hz), 7.20 (d, 2H, J = 8.2 Hz), 5.74 (d, 1H, J = 9.1 Hz), 4.83-4.91 (m, 1H), 3.79 (dq, 1H, J = 6.3, 10.0 Hz), 3.67 (dt, 1H, J = 2.3, 9.0 Hz), 2.43 (s, 3H), 2.38 (s, 3H), 1.80-1.90 (m, 1H), 1.33 (d, 3H, J = 6.3 Hz), 1.23 (d, 3H, J = 6.3 Hz), 1.19 (d, 3H, J = 6.8 Hz), 0.80 (d, 3H, J = 6.8 Hz), 0.69 (d, 3H, J = 6.8 Hz); ^{13}C NMR (CDCl_3) δ = 176.4, 143.1, 142.5, 139.9, 139.0, 129.3, 129.2, 126.5, 126.5, 73.2, 62.3, 42.6, 29.2, 21.5, 21.4, 21.2, 20.9, 20.6, 15.6, 14.9; IR (neat) 3647, 3273, 2968, 2936, 2877, 1918, 1736, 1592, 1496, 1455, 1387, 1373, 1331, 1286, 1241, 1184, 1155, 1089, 1042, 968, 909, 883, 849, 814, 727, 694, 666, 609, 579, 547 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{24}\text{H}_{35}\text{N}_2\text{O}_5\text{S}_2$ [$\text{M}+\text{H}]^+$, 495.1987. Found 495.1969.

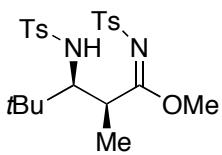


iso-Propyl syn-2-methyl-3-(iso-propyl)-3-(*p*-toluenesulfonylamino)-*N*-(*p*-toluenesulfonyl)propionimidate (syn-7v): ^1H NMR (CDCl_3) δ = 7.80 (d, 2H, J = 8.5 Hz), 7.75 (d, 2H, J = 7.9 Hz), 7.26-7.31 (m, 4H), 4.96-5.06 (m, 1H), 4.35-4.42 (m, 1H), 3.62-3.71 (m, 2H), 2.42 (s, 3H), 2.41 (s, 3H), 1.55-1.65 (m, 1H), 1.25 (d, 3H, J = 6.2 Hz), 1.22 (d, 3H, J = 6.2 Hz), 1.11 (d, 3H, J = 6.2 Hz), 0.79 (d, 3H, J = 6.8 Hz), 0.73 (d, 3H, J = 6.8 Hz); ^{13}C NMR (CDCl_3) δ = 175.4, 143.1, 143.1, 139.3, 139.0, 129.5, 129.3, 126.9, 126.5, 72.3, 61.1, 42.4, 31.0, 21.5, 21.5, 21.0, 20.9, 20.8, 15.9, 15.5; IR (neat) 3295, 2977, 2933, 2878, 1594, 1456, 1374, 1317, 1290, 1159, 1092, 1040, 909, 839, 814, 718, 693, 666, 608, 572, 548, 458 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{24}\text{H}_{35}\text{N}_2\text{O}_5\text{S}_2$ [$\text{M}+\text{H}]^+$, 495.1987. Found 495.1969.

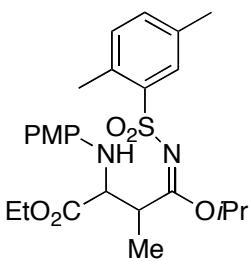


Methyl anti-3-(*p*-toluenesulfonylamino)-2,4,4-trimethyl-*N*-(*p*-toluenesulfonyl)pentanimidate (anti-7w): ^1H NMR (CDCl_3) δ = 7.83 (d, 2H, J = 8.2 Hz), 7.73 (d, 2H, J = 8.2 Hz), 7.31 (d, 2H, J = 8.2 Hz), 7.25 (d, 2H, J = 8.2 Hz), 5.47 (d, 1H, J = 9.6 Hz), 4.03 (dq, 1H, J = 6.8, 6.9 Hz), 3.68 (s, 3H), 3.43 (dd, 1H, J = 5.5, 10.4 Hz), 2.43 (s, 3H), 2.40 (s, 3H), 1.25 (d,

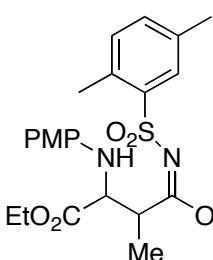
3H, $J = 6.9$ Hz), 0.87 (s, 9H); ^{13}C NMR (CDCl_3) $\delta = 177.4, 143.4, 142.8, 139.6, 138.8, 129.4, 126.6, 66.6, 55.5, 39.4, 36.4, 27.5, 21.5, 21.5, 18.4$; IR (neat) 3325, 2954, 1601, 1540, 1455, 1315, 1259, 1185, 1157, 1091, 1025, 927, 814, 675, 594, 572, 547 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{23}\text{H}_{34}\text{N}_3\text{O}_5\text{S}_2$ [$\text{M}+\text{H}]^+$, 481.1831. Found 481.1832.



Methyl *syn*-3-(*p*-toluenesulfonylamino)-2,4,4-trimethyl-*N*(*p*-toluenesulfonyl)pentanimidate (*syn*-7w): Mp. 189-190 °C; ^1H NMR (CDCl_3) $\delta = 7.84$ (d, 2H, $J = 8.2$ Hz), 7.74 (d, 2H, $J = 8.2$ Hz), 7.30 (d, 2H, $J = 8.2$ Hz), 7.26 (d, 2H, $J = 8.2$ Hz), 4.65-4.72 (m, 1H), 3.80-3.88 (m, 2H), 3.71 (s, 3H), 2.42 (s, 3H), 2.40 (s, 3H), 1.18 (d, 3H, $J = 6.2$ Hz), 0.82 (s, 9H); ^{13}C NMR (CDCl_3) $\delta = 177.7, 143.1, 142.9, 139.2, 139.1, 129.4, 129.3, 126.8, 126.5, 63.1, 55.4, 40.0, 36.4, 26.8, 21.5, 21.4, 16.4$; IR (neat) 3303, 2952, 1600, 1540, 1456, 1436, 1315, 1301, 1286, 1155, 1093, 1071, 953, 916, 814, 688, 605, 574 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{23}\text{H}_{34}\text{N}_3\text{O}_5\text{S}_2$ [$\text{M}+\text{H}]^+$, 481.1831. Found 481.1832.

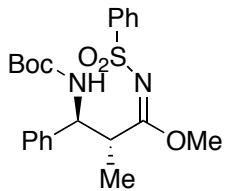


iso-Propyl 3-ethoxycarbonyl-3-(*p*-methoxyphenylamino)-2-methyl-*N*(2,5-xylylsulfonyl)propionimidate (major-7x): Mp. 99-100 °C; ^1H NMR (CDCl_3) $\delta = 8.04-8.09$ (m, 2H), 6.95-7.02 (m, 3H), 6.67-6.74 (m, 4H), 4.90 (d, 1H, $J = 11.9$ Hz), 4.56 (qd, 1H, $J = 10.2, 6.8$ Hz), 4.37 (t, 1H, $J = 10.8$ Hz), 3.78-3.93 (m, 2H), 3.29 (s, 3H), 3.08 (s, 3H), 1.29 (d, 3H, $J = 6.2$ Hz), 0.82 (t, 3H, $J = 7.1$ Hz); ^{13}C NMR (CDCl_3) $\delta = 176.5, 171.8, 153.9, 142.8, 141.0, 132.4, 128.8, 127.0, 116.2, 115.1, 62.1, 61.1, 55.1, 42.6, 14.4, 14.1$; IR (neat) 3055, 2986, 2953, 2836, 1735, 1607, 1514, 1457, 1447, 1421, 1305, 1265, 1242, 1191, 1156, 1092, 1034, 953, 896, 825, 737, 705, 624, 603, 527, 451, 419 cm^{-1} ; HRMS (FAB); Exact mass calcd for $\text{C}_{25}\text{H}_{35}\text{N}_2\text{O}_6\text{S}$ [$\text{M}+\text{H}]^+$, 491.2216. Found 491.2198.

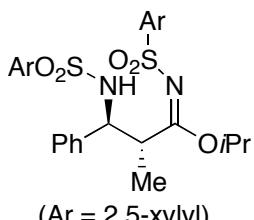


iso-Propyl 3-ethoxycarbonyl-3-(*p*-methoxyphenylamino)-2-methyl-*N*(2,5-xylylsulfonyl)propionimidate (minor-7x): ^1H NMR (CDCl_3) $\delta = 8.05-8.14$ (m, 2H), 6.95-7.05 (m, 3H), 6.74 (d, 2H, $J = 9.1$ Hz), 6.62 (d, 2H, $J = 9.1$ Hz), 4.50 (t, 1H, $J = 9.4$ Hz), 4.40 (qd, 1H, $J = 9.0, 6.8$ Hz), 3.92-4.07 (m, 2H), 3.84 (d, 1H, $J = 10.2$ Hz), 3.35 (s, 3H), 3.21 (s, 3H), 1.35 (d, 3H, $J = 8.0$ Hz), 0.94 (t, 3H, $J = 7.1$ Hz); ^{13}C NMR (CDCl_3) $\delta = 176.3, 171.7, 143.1, 141.6, 132.3, 128.8, 127.2, 115.9, 115.1, 61.5, 60.7, 55.2, 55.0, 42.9, 14.8, 14.1$; IR (neat) 3063,

3033, 2984, 2951, 2907, 2834, 1735, 1608, 1511, 1446, 1369, 1306, 1241, 1198, 1156, 1092, 1077, 1060, 1027, 953, 852, 824, 758, 734, 690, 624, 587, 519, 446, 418 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₅H₃₅N₂O₆S [M+H]⁺, 491.2216. Found 491.2198.



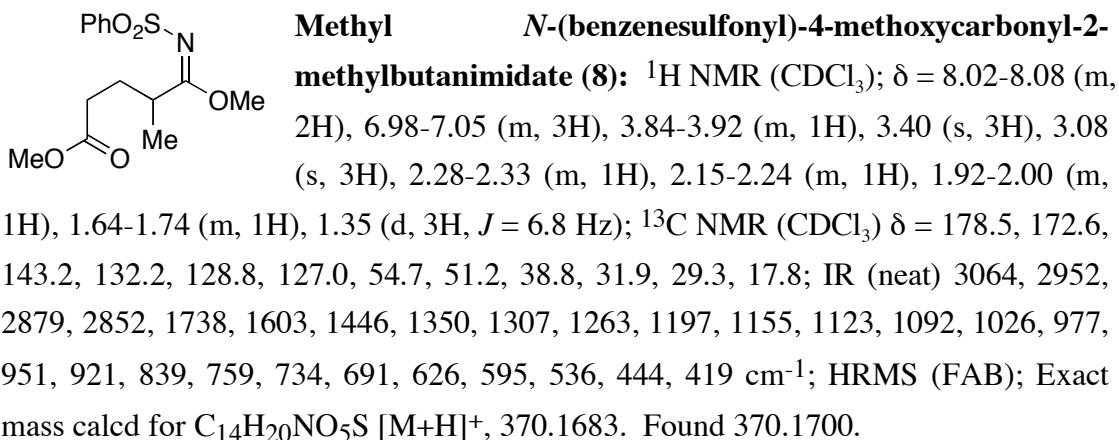
Methyl anti-N-(benzenesulfonyl)-3-(tert-butoxycarbonylamino)-2-methyl-3-phenylpropionimidate (7y): Mp. 99-100 °C; ¹H NMR (CDCl₃) δ = 8.01 (d, 2H, J = 7.4 Hz), 7.59 (t, 1H, J = 7.4 Hz), 7.53 (t, 2H, J = 7.7 Hz), 7.25-7.35 (m, 5H), 6.01 (d, 1H, J = 9.6 Hz), 4.75 (t, 1H, J = 10.2 Hz), 3.96-4.07 (m, 1H), 3.75 (s, 3H), 1.35 (s, 9H), 1.03 (d, 3H, J = 6.8 Hz); ¹³C NMR (CDCl₃) δ = 177.6, 154.6, 141.8, 140.2, 132.6, 128.7, 128.7, 127.8, 127.1, 126.5, 79.2, 58.2, 55.5, 45.1, 28.2, 14.8; IR (neat) 3381, 2977, 1716, 1602, 1508, 1455, 1392, 1365, 1304, 1246, 1154, 1090, 1057, 1002, 950, 881, 757, 734, 700, 688, 623 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₂H₂₉N₂O₅S [M+H]⁺, 433.1797. Found 433.1811.



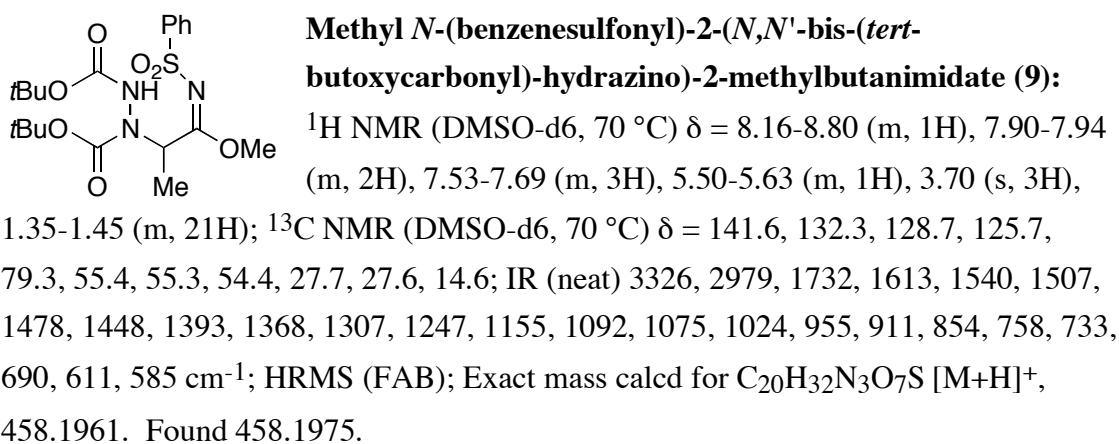
iso-Propyl anti-2-methyl-3-phenyl-N-(2,5-xylylsulfonyl)-3-(2,5-xylylsulfonylamino)propionimidate (anti-7z): Mp. 99-100 °C; ¹H NMR (CDCl₃) δ = 8.23 (s, 1H), 7.39 (s, 1H), 7.12 (d, 1H, J = 9.1 Hz), 6.90-6.97 (m, 3H), 6.87 (dd, 1H, J = 7.6, 1.4 Hz), 6.65-6.73 (m, 3H), 6.53 (qd, 2H, J = 7.6, 1.4 Hz), 5.03 (quint, 1H, J = 6.2 Hz), 4.68 (dd, 1H, J = 11.0, 9.3 Hz), 4.48 (qd, 1H, J = 11.0, 6.5 Hz), 3.00 (s, 3H), 2.62 (s, 3H), 1.95 (s, 3H), 1.78 (s, 3H), 1.40 (d, 3H, J = 6.2 Hz), 0.96 (d, 3H, J = 6.2 Hz), 0.91 (d, 3H, J = 6.2 Hz); ¹³C NMR (CDCl₃) δ = 177.0, 140.1, 140.0, 138.0, 136.1, 135.2, 135.0, 133.6, 133.2, 132.6, 132.2, 131.9, 129.7, 128.8, 128.3, 127.3, 73.1, 62.5, 46.4, 21.4, 21.1, 20.8, 20.5, 20.4, 20.0, 14.5; IR (neat) 3060, 3029, 2982, 2936, 2878, 1592, 1578, 1493, 1458, 1386, 1359, 1331, 1292, 1226, 1208, 1162, 1102, 1065, 1030, 973, 910, 855, 824, 768, 747, 725, 707, 645, 607, 580, 548, 516, 501, 464, 436, 419 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₉H₃₇N₂O₅S₂ [M+H]⁺, 557.2144. Found 557.2151.

Procedure of addition reaction of sulfonylimide **6a to methyl acrylate.** To MS 4A (100 mg) were added a solution of sulfonylimide **6a** (68.1 mg, 0.3 mmol) in DMF (0.5 mL), methyl acrylate (38.7 mg, 0.45 mmol), and a solution of DBU in DMF (10 mol%, 100 μL) at RT. The reaction mixture was stirred for 20 h at RT, and then diluted by addition of Et₂O. The mixture obtained after filtration (for removal of

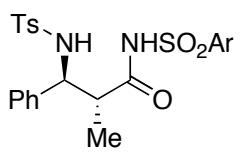
MS 4A) was washed with water 3 times, then dried over anhydrous Na_2SO_4 . Filtration and removal of solvents afforded the crude product. Purification of the crude product was conducted by chromatography on SiO_2 , to afford the desired product **8** (70.4 mg, 75%).



Procedure of addition reaction of sulfonylimide **6a to di-*tert*-butyl azodicarboxylate.** To MS 4A (100 mg) were added a solution of sulfonylimide **6a** (136.4 mg, 0.6 mmol) in DMF (1.1 ml), di-*tert*-butyl azodicarboxylate (152 mg, 0.66 mmol), and a solution of DBU in DMF (5 mol%, 100 μl) at RT. The reaction mixture was stirred for 1.5 h at RT, and then diluted by addition of Et_2O . The mixture obtained after filtration (for removal of MS 4A) was washed with water 3 times, then dried over anhydrous Na_2SO_4 . Filtration and removal of solvents afforded the crude product. Purification of the crude product was conducted by chromatography on SiO_2 , to afford the desired product **9** (236.3 mg, 86%).

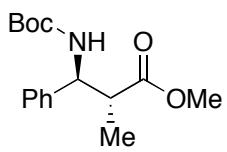


Procedure for the conversion of sulfonylimidate 7g to compound 10. To sulfonylimidate **7g** (50 mg, 0.092 mmol) were added a mixture of *iso*-propanol and H₂O (95/5, 0.5 ml) and concentrated H₂SO₄ (66 mg, ca. 7.0 equiv). The reaction mixture was stirred at 80 °C for 40 min. TLC analysis revealed that no reaction had occurred, then the mixture was heated to 100 °C and stirred for 3 h. After cooling to RT, the mixture was diluted by addition of CH₂Cl₂. A saturated aqueous solution of NaHCO₃ was added, and the mixture was extracted with CH₂Cl₂ three times. The organics were dried over anhydrous MgSO₄. Filtration and removal of solvents afforded compound **10** (46.1 mg, quant). The obtained compound **10** was found to be pure by ¹H NMR spectroscopy analysis in DMSO-d6 (**10** was very hard to dissolve in any usual organic solvents except polar solvents such as DMSO).



anti-2-methyl-3-phenyl-3-(*p*-toluenesulfonylamino)-*N*-(2,5-xylsulfonyl)propionamide (10**):** Mp. 218-219 °C; ¹H NMR (DMSO-d6) δ = 12.08 (s, 1H), 8.67 (d, 1H, *J* = 9.6 Hz), 7.76 (s, 1H), 7.33-7.37 (m, 1H), 7.20-7.25 (m, 3H), 6.87-7.03 (m, 7H), 4.41 (t, 1H, *J* = 9.9 Hz), 2.60-2.72 (m, 1H), 2.50 (s, 3H), 2.34 (s, 3H), 2.18 (s, 3H), 0.59 (d, 3H, *J* = 7.4 Hz); ¹³C NMR (DMSO-d6) δ = 172.2, 141.5, 138.3, 137.0, 135.4, 134.0, 133.9, 132.2, 13.3, 128.6, 127.8, 126.8, 126.3, 58.9, 46.6, 20.8, 20.3, 19.1, 14.7; IR (neat) 3251, 3063, 3032, 2979, 2926, 2878, 1693, 1655, 1647, 1638, 1617, 1599, 1560, 1542, 1493, 1457, 1382, 1341, 1288, 1223, 1201, 1163, 1123, 1087, 1062, 898, 849, 815, 766, 702, 667 cm⁻¹; HRMS (FAB); Exact mass calcd for C₂₅H₂₉N₂O₅S₂ [M+H]⁺, 501.1518. Found 501.1538.

Procedure for the conversion of sulfonylimidate 7y to ester 11. To sulfonylimidate **7y** (82.3 mg, 0.190 mmol) were added a mixture of DMF and H₂O (95/5, 0.38 ml), and a solution of DBU in DMF (10 mol%, 30 µl). The mixture was stirred at RT for 33 h, and then diluted by addition of Et₂O. The mixture was washed with water 3 times, then dried over anhydrous Na₂SO₄. Filtration and removal of solvents afforded the crude product. Purification of the crude product was conducted by chromatography on SiO₂, to afford the desired ester **11** (50.4 mg, 90% yield).

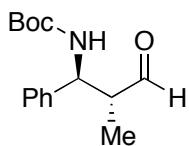


tert-Butyl anti-2-(methoxycarbonyl)-1-phenylpropylcarbamate (11**):** Mp. 89-90 °C; ¹H NMR (CDCl₃) δ = 7.28-7.34 (m, 2H), 7.20-7.27 (m, 3H), 5.78 (s, 1H), 4.83 (s, 1H), 3.58 (s, 3H), 2.92 (s, 1H), 1.42 (s, 9H), 1.23 (d, 3H, *J* = 6.2 Hz); ¹³C NMR (CDCl₃) δ = 175.3, 155.4, 127.3, 126.6, 126.2, 79.4, 51.6, 45.2, 28.3, 15.3; IR (neat) 3428, 3360, 2977, 2356, 1716, 1497, 1455, 1434, 1365, 1289, 1245, 1168, 1085, 1051, 1005, 879, 756, 701,

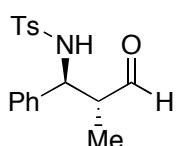
585 cm⁻¹; HRMS (FAB); Exact mass calcd for C₁₆H₂₄NO₄ [M+H]⁺, 294.1705.

Found 294.1696.

Procedure for Red-Al reduction of sulfonylimides 7d and 7g to aldehydes 12a and 12b. The procedure for the conversion of **7d** to **12a** is as follows. A solution of **7d** (100 mg, 0.205 mmol) in 2 ml of THF was cooled to -70 °C, and then Red-Al (65% w/w toluene solution, 385 µl, 7.0 equiv) was added slowly lest the temperature should increase. The mixture was stirred at -70 °C for 18 h, and the reaction was quenched by addition of MeOH (0.1 ml) at -70 °C. The mixture was stirred for 5 min at that temperature, and H₂O was added. The temperature was allowed to increase to RT, and AcOEt and a saturated aqueous solution of NH₄Cl were added. After filtration, the obtained filtrate was extracted with AcOEt three times, and the organics were dried over anhydrous Na₂SO₄. Filtration and removal of solvents afforded the crude product. Purification of the crude product was conducted by chromatography on neutral SiO₂, to afford the aldehyde **12a** (46.9 mg, 87% yield). Aldehyde **12b** was obtained by following the same procedure mentioned above (83% yield).

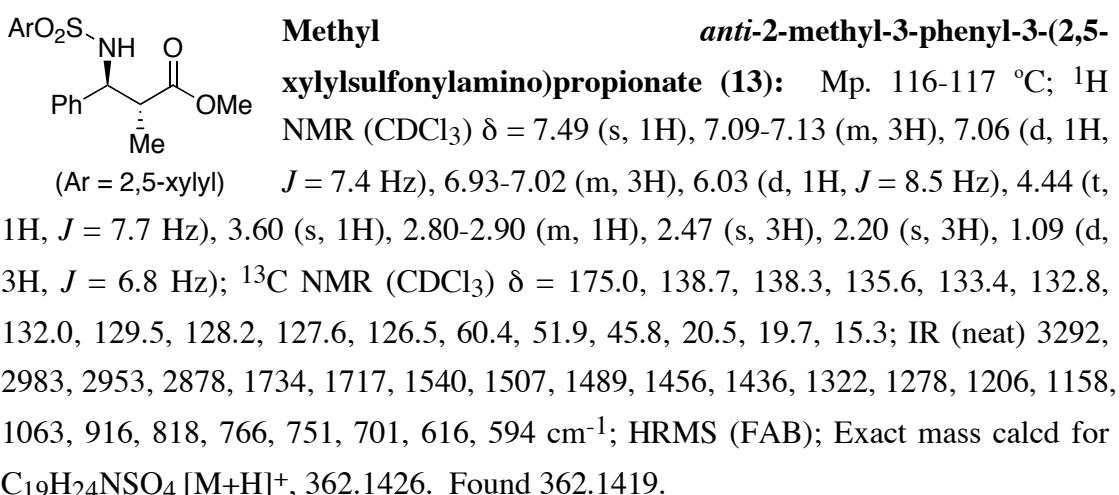


tert-butyl anti-2-formyl-1-phenylpropylcarbamate (12a): Mp. 82-83 °C; ¹H NMR (CDCl₃) δ = 9.66 (d, 1H, J = 2.3 Hz), 7.22-7.37 (m, 5H), 5.28 (s, 1H), 4.78 (s, 1H), 2.81 (s, 1H), 1.39 (s, 9H), 1.02 (d, 3H, J = 6.8 Hz); ¹³C NMR (CDCl₃) δ = 203.3, 155.2, 139.9, 128.7, 127.7, 126.8, 79.9, 52.1, 28.2, 11.8; IR (neat) 3343, 2978, 2933, 2817, 2723, 1716, 1699, 1519, 1507, 1455, 1392, 1366, 1288, 1249, 1169, 1086, 1002, 755, 701 cm⁻¹; HRMS (FAB); Exact mass calcd for C₁₅H₂₂NO₃ [M+H]⁺, 264.1599. Found 264.1606.



anti-2-methyl-3-phenyl-3-(tosylamino)propanal (12b): Mp. 137-138 °C; ¹H NMR (CDCl₃) δ = 9.64 (d, 1H, J = 2.8 Hz), 7.47 (d, 2H, J = 7.9 Hz), 6.96-7.15 (m, 7H), 5.90 (d, 1H, J = 8.5 Hz), 4.54 (t, 1H, J = 8.5 Hz), 2.75-2.84 (m, 1H), 2.30 (s, 3H), 0.96 (d, 3H, J = 6.8 Hz); ¹³C NMR (CDCl₃) δ = 203.2, 143.0, 137.8, 137.2, 129.2, 128.4, 127.6, 127.0, 127.0, 59.1, 51.9, 21.3, 11.8; IR (neat) 3268, 3068, 3030, 2972, 2926, 2872, 2727, 1724, 1598, 1495, 1456, 1325, 1185, 1160, 1090, 1045, 914, 812, 761, 702, 670, 566, 547 cm⁻¹; HRMS (FAB); Exact mass calcd for C₁₇H₂₀NSO₃ [M+H]⁺, 318.1184. Found 318.1169.

Procedure for direct formation of ester **13 from benzaldehyde and sulfonylimide **6h**.** To a solution of benzaldehyde (60.9 μ l, 0.6 mmol) and DBU (18.3 mg, 0.12 mmol) in 0.2 ml of DMF was added 2,5-xylylsulfonylamide (11.1 mg, 0.06 mmol). To the mixture was slowly added over 32 h a solution of sulfonylimide **6h** (183.8 mg, 0.72 mmol) in 1 ml of DMF. After completion of slow addition, the mixture was stirred at RT for further 46 h (total 78 h), and then diluted by addition of Et₂O. The mixture was washed with water 3 times, then dried over anhydrous Na₂SO₄. Filtration and removal of solvents afforded the crude product. Purification of the crude product was conducted by chromatography on SiO₂, to afford the desired ester **13** (diastereomers mixture, 196.2 mg, 90% yield, *anti/syn* = 87/13).



Origin of high *anti*-selectivity

Single crystal X-ray diffraction analysis of a sulfonylimide (**6a**) and some of the Mannich adducts (*syn*-**7a**, *anti*-**7d**, *anti*-**7g**, and *syn*-**7g**), not only provided information about the relative configuration of the products, but also characteristic structure of the sulfonylimide group. In every case the imine adopts an *E* configuration and the oxygen at the carbon of the C=N bond is sp² hybridised, and displays an s-*cis* geometry about the N-C-O-C bonds.⁶ This phenomena can be rationalized by considering n- σ^* interaction between oxygen lone electron pair and σ^* anti-bonding orbital of C=N group. Predominant *E*-configuration of the imine may stem from the steric repulsion of the alkyl group on oxygen and the sulfonyl group.

The proposed mechanism for *anti*-selectivity is demonstrated in Figure 1. Based on the experimental information that the imine adopts an *E* configuration, the geometry of the generated aza-enolate is expected to be Z (Scheme 4). Considering the steric repulsion between the methyl substituent and the Boc group (repulsion *c*), and that between the SO₂Ar group and the R group (repulsion *b*) in TS-2 led us to the conclusion that TS-1 may be favored to give *anti* product. When pivalaldehyde-

⁶ Neilson, D. G. In *The Chemistry of Amidines and Imidates*; Patai, S., Ed.; John Wiley & Sons: London, UK, 1975; Chapter 8.

derived Ts imine was used (Table 2, entry 14), *syn*-selectivity was observed, which may be rationalized by considering that the repulsion between *t*Bu group and Me substituent (repulsion *a* in TS-1) is large in Ts-1. In **14** the formation of *s-trans* configuration about the S-N-C-C bonds would be suppressed by steric repulsion between the *iPr* group and the methyl substituent (repulsion *d*), while the *s-trans* formation may be possible in the case of a sulfonylimidate bearing less sterically demanding methoxy group, which may be the reason for the observed low *anti*-selectivity (Table 1, entry 4).

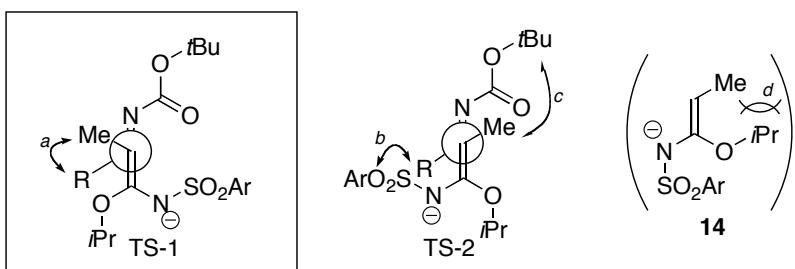
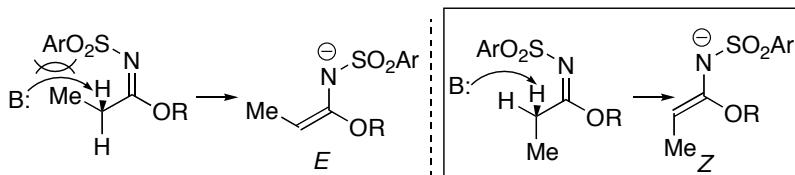
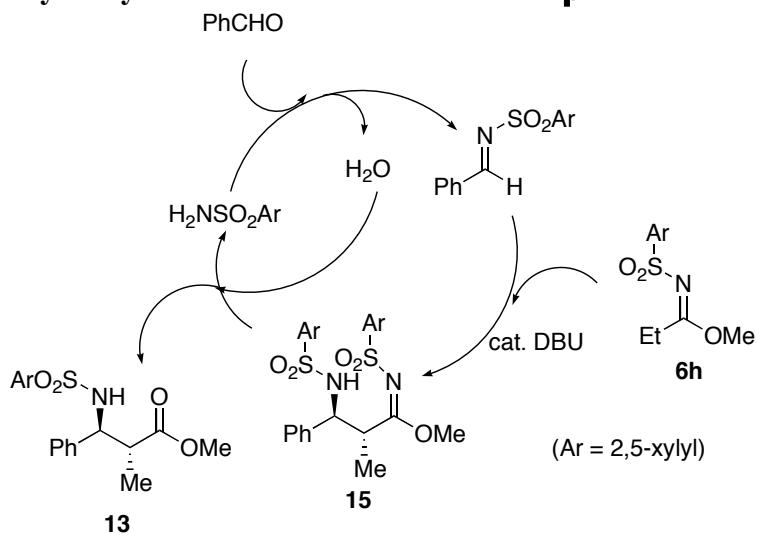


Figure 1. Proposed Transition State Model.

Scheme 4. Proposed Geometry of Aza-enolate Derived from Sulfonylimidate.

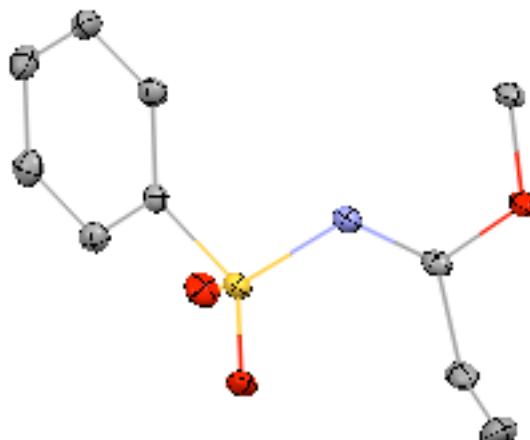


Proposed Catalytic Cycle for the Direct Formation of β -Amino Acid Derivative.



The fact that intermediate **15** could be isolated if slow addition of **6h** was not conducted, supports this proposed reaction mechanism. The structure of product **13** was unambiguously determined by X-ray diffraction analysis.

X-ray Diffraction Analysis



6a

A. Crystal Data

Empirical Formula	C ₁₀ H ₁₃ NSO ₃
Formula Weight	227.28
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.40 X 0.30 X 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 11.0472(4) Å b = 8.8900(3) Å c = 12.0126(4) Å β = 109.5182(10) ° V = 1111.96(6) Å ³

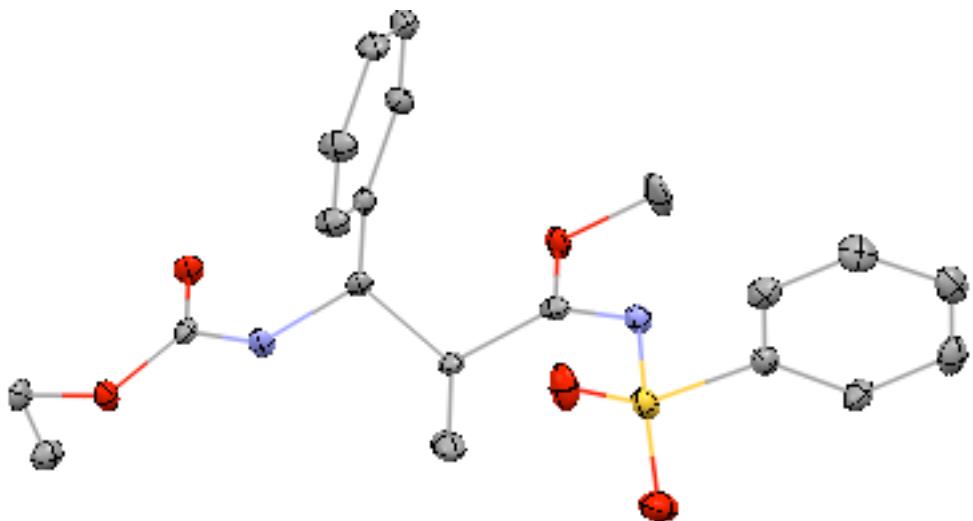
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.358 g/cm ³
F ₀₀₀	480.00
μ (MoK α)	2.776 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	30.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	30.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 10581 Unique: 2544 ($R_{\text{int}} = 0.029$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.807 - 0.946)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0010Fo^2 + 3.0000s(Fo^2) + 0.5000]/(4Fo^2)$
$2\theta_{\text{max}}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2544
No. Variables	188
Reflection/Parameter Ratio	13.53
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.0294
Residuals: R (All reflections)	0.0314
Residuals: wR2 (All reflections)	0.0874
Goodness of Fit Indicator	0.805
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.30 e/Å³
Minimum peak in Final Diff. Map	-0.47 e/Å³



syn-7a

A. Crystal Data

Empirical Formula	C ₂₀ H ₂₄ N ₂ O ₅ S
Formula Weight	404.48
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.30 X 0.30 X 0.30 mm
Crystal System	triclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 9.6790(10) \text{ \AA}$ $b = 9.7118(9) \text{ \AA}$ $c = 22.524(3) \text{ \AA}$ $\alpha = 87.724(3)^\circ$ $\beta = 77.352(3)^\circ$ $\gamma = 79.122(3)^\circ$ $V = 2028.8(4) \text{ \AA}^3$

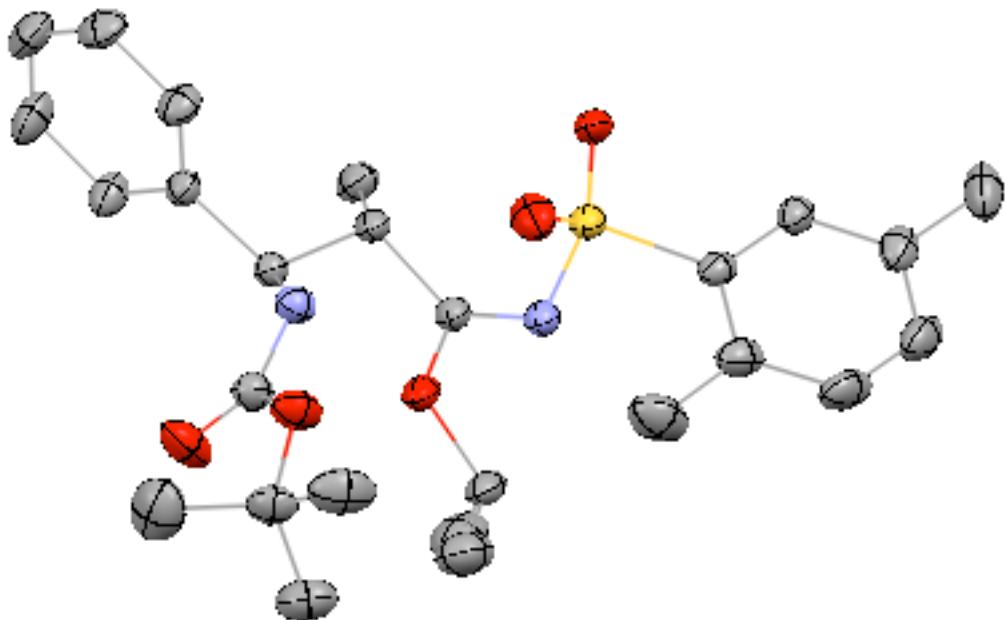
Space Group	P-1 (#2)
Z value	4
D _{calc}	1.324 g/cm ³
F ₀₀₀	856.00
μ (MoK α)	1.928 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=30.0$)	130.0 - 190.0°
Exposure Rate	60.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=210.0$)	0.0 - 160.0°
Exposure Rate	60.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 19196 Unique: 9141 ($R_{\text{int}} = 0.111$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.936 - 0.944)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/[0.0078F_o^2 + 1.0000s(F_o^2)]/(4F_o^2)$
$2\theta_{\text{max}}$ cutoff	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	9141
No. Variables	553
Reflection/Parameter Ratio	16.53
Residuals: R1 ($I > 2.00\sigma(I)$)	0.1204
Residuals: R (All reflections)	0.1445
Residuals: wR2 (All reflections)	0.3145
Goodness of Fit Indicator	0.987
Max Shift/Error in Final Cycle	0.004
Maximum peak in Final Diff. Map	1.98 e/Å³
Minimum peak in Final Diff. Map	-1.44 e/Å³



anti-7d

A. Crystal Data

Empirical Formula	C ₂₆ H ₃₆ N ₂ O ₅ S
Formula Weight	488.64
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.20 X 0.10 X 0.10 mm
Crystal System	triclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 8.6250(6)$ Å $b = 10.2643(7)$ Å $c = 17.2442(13)$ Å $\alpha = 85.8573(19)$ ° $\beta = 85.7269(18)$ ° $\gamma = 65.8507(16)$ °

$$V = 1387.69(17) \text{ \AA}^3$$

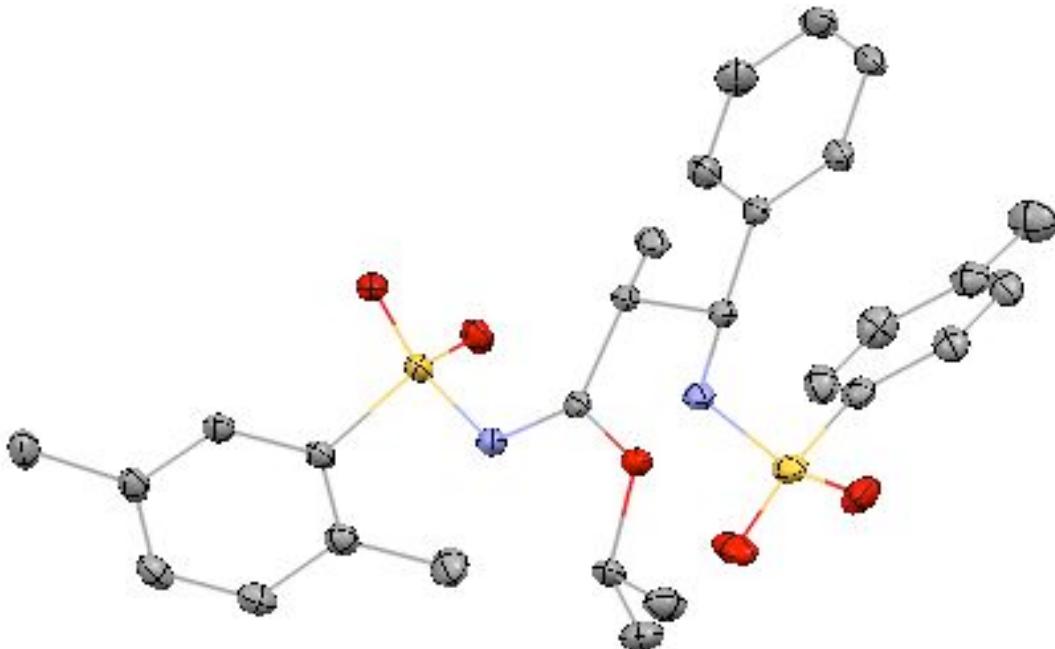
Space Group	P-1 (#2)
Z value	2
D _{calc}	1.169 g/cm ³
F ₀₀₀	524.00
$\mu(\text{MoK}\alpha)$	1.519 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=30.0$)	130.0 - 190.0°
Exposure Rate	60.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	60.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 13534 Unique: 6242 ($R_{\text{int}} = 0.028$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.913 - 0.985)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (SHELXS86)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(Fo^2) + (0.1229 \cdot P)^2 + 0.0000 \cdot P]$ where $P = (\text{Max}(Fo^2, 0) + 2Fc^2)/3$
$2\theta_{\text{max}}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	6242
No. Variables	312
Reflection/Parameter Ratio	20.01
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.0600
Residuals: R (All reflections)	0.1065
Residuals: wR2 (All reflections)	0.2098
Goodness of Fit Indicator	1.047
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.39 e/Å³
Minimum peak in Final Diff. Map	-0.37 e/Å³



anti-7g

A. Crystal Data

Empirical Formula	C ₂₈ H ₃₄ N ₂ S ₂ O ₅
Formula Weight	542.71
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 9.2941(8) \text{ \AA}$ $b = 19.0359(17) \text{ \AA}$ $c = 16.2694(14) \text{ \AA}$ $\beta = 102.4056(18)^\circ$ $V = 2811.2(4) \text{ \AA}^3$

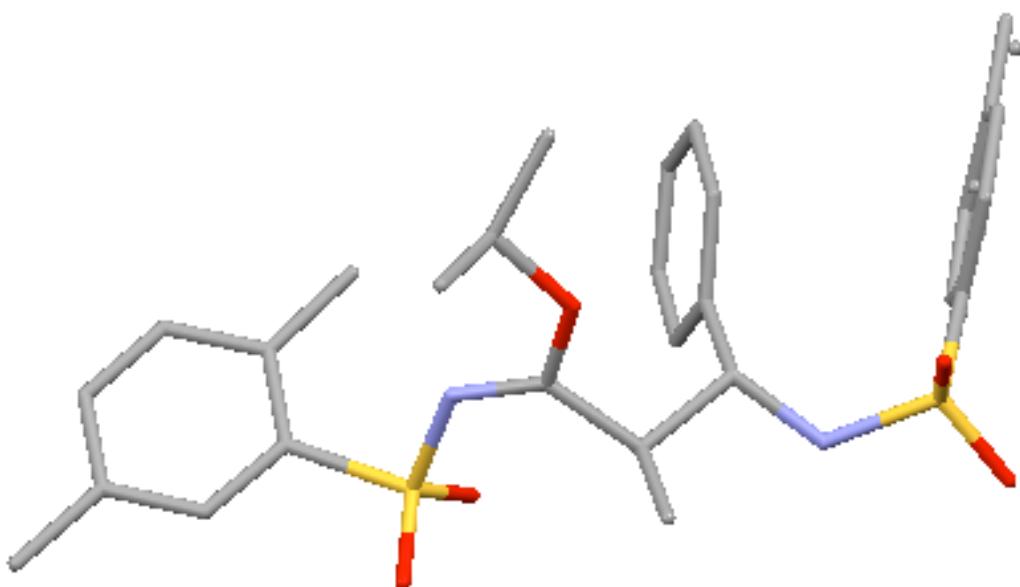
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.282 g/cm ³
F ₀₀₀	1152.00
μ (MoK α)	2.288 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	30.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	30.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 26494 Unique: 6381 ($R_{\text{int}} = 0.051$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.905 - 0.955)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(Fo^2) + (0.0625 \cdot P)^2 + 0.6498 \cdot P]$ where $P = (\text{Max}(Fo^2, 0) + 2Fc^2)/3$
$2\theta_{\text{max}}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	6381
No. Variables	341
Reflection/Parameter Ratio	18.71
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.0429
Residuals: R (All reflections)	0.0514
Residuals: wR2 (All reflections)	0.1207
Goodness of Fit Indicator	1.084
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.30 e/Å³
Minimum peak in Final Diff. Map	-0.54 e/Å³



syn-7g

A. Crystal Data

Empirical Formula	C ₂₈ H ₂₉ N ₂ S ₂ O ₅
Formula Weight	537.67
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	triclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 9.4702(5)$ Å $b = 12.0832(7)$ Å $c = 14.1583(8)$ Å $\alpha = 105.7265(16)$ ° $\beta = 106.9474(15)$ ° $\gamma = 99.1637(17)$ ° $V = 1440.95(14)$ Å ³

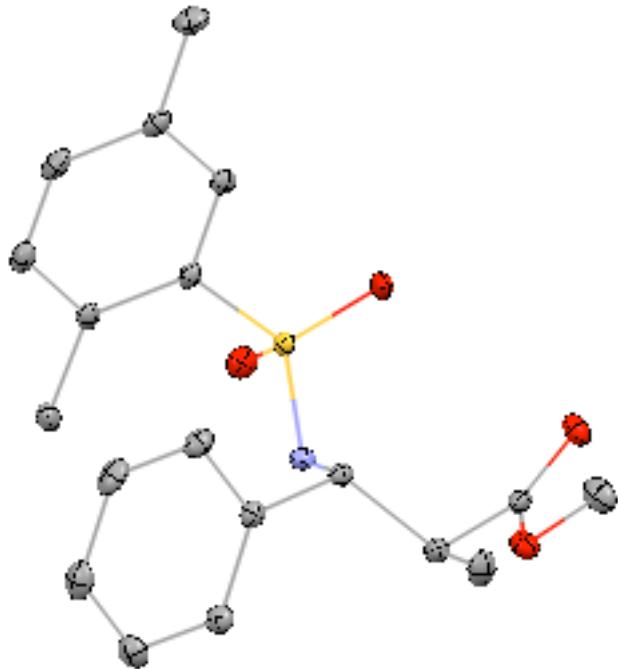
Space Group	P-1 (#2)
Z value	2
D _{calc}	1.239 g/cm ³
F ₀₀₀	566.00
μ (MoK α)	2.227 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	120.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	120.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 14126 Unique: 6518 ($R_{\text{int}} = 0.020$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.739 - 0.956)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(Fo^2) + (0.0818 \cdot P)^2 + 0.3155 \cdot P]$ where $P = (\text{Max}(Fo^2, 0) + 2Fc^2)/3$
$2\theta_{\text{max}}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	6518
No. Variables	302
Reflection/Parameter Ratio	21.58
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.0508
Residuals: R (All reflections)	0.0610
Residuals: wR2 (All reflections)	0.1552
Goodness of Fit Indicator	1.100
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.41 e/Å³
Minimum peak in Final Diff. Map	-0.41 e/Å³



13

A. Crystal Data

Empirical Formula	C ₁₉ H ₂₃ NO ₄ S
Formula Weight	361.45
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.60 X 0.60 X 0.30 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 9.5765(3) Å b = 9.6154(3) Å

$$\begin{aligned}c &= 19.9940(7) \text{ \AA} \\ \beta &= 100.6976(12)^\circ \\ V &= 1809.08(10) \text{ \AA}^3\end{aligned}$$

Space Group $P2_1/c$ (#14)

Z value 4

D_{calc} 1.327 g/cm³

F₀₀₀ 768.00

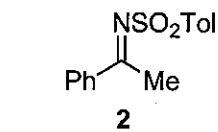
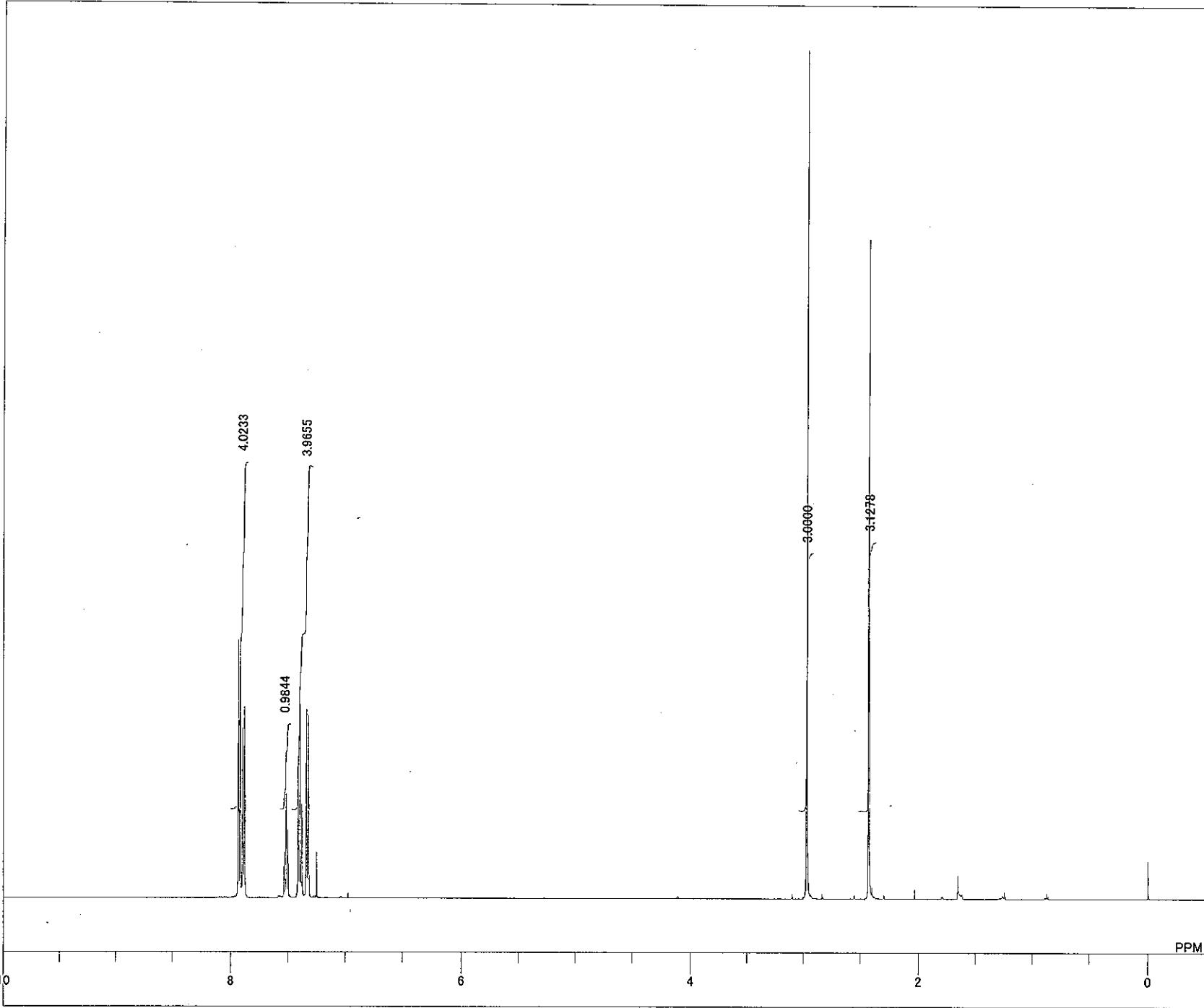
$\mu(\text{MoK}\alpha)$ 2.020 cm⁻¹

B. Intensity Measurements

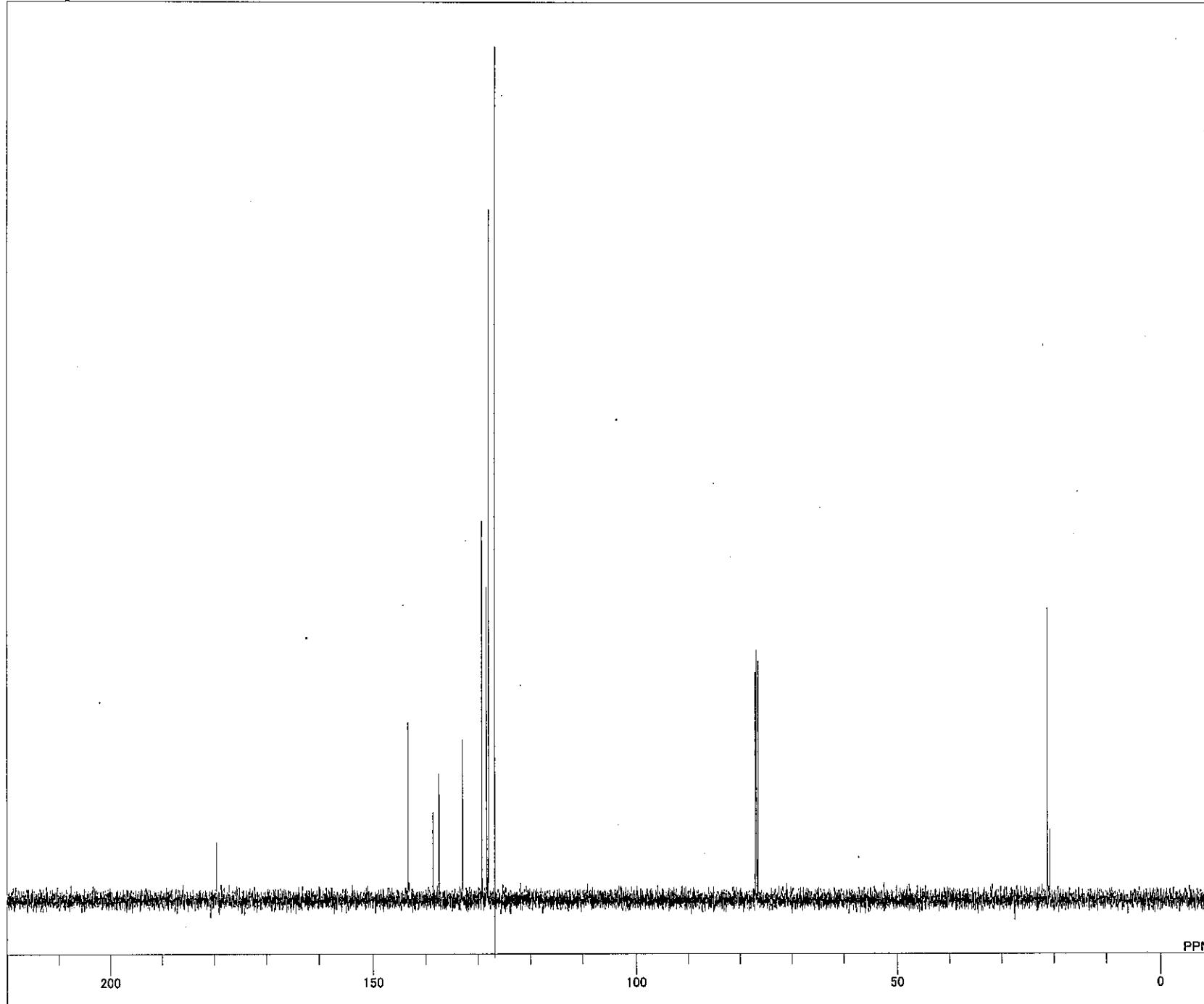
Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	30.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	30.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.8°
No. of Reflections Measured	Total: 17274 Unique: 4124 ($R_{\text{int}} = 0.017$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.788 - 0.941)

C. Structure Solution and Refinement

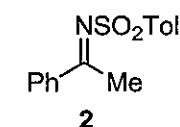
Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0016Fo^2 + 1.0000s(Fo^2)]/(4Fo^2)$
$2\theta_{\text{max}}$ cutoff	54.8°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4124
No. Variables	318
Reflection/Parameter Ratio	12.97
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.0287
Residuals: R (All reflections)	0.0315
Residuals: wR2 (All reflections)	0.0970
Goodness of Fit Indicator	1.004
Max Shift/Error in Final Cycle	0.002
Maximum peak in Final Diff. Map	0.35 e/Å³
Minimum peak in Final Diff. Map	-0.42 e/Å³

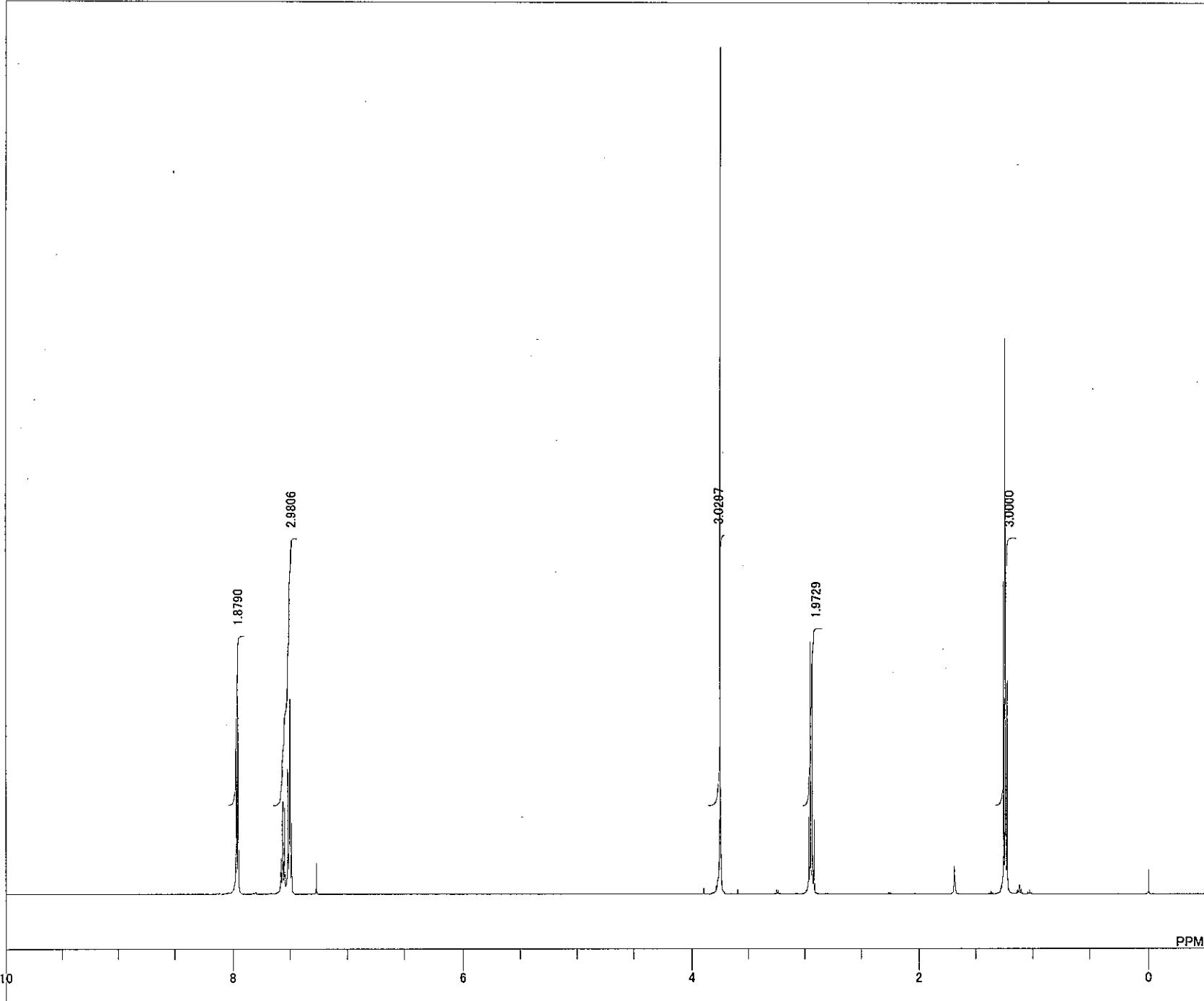


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OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
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SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

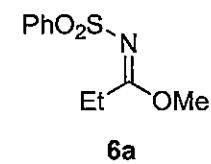


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OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
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PD 2.0000 sec
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CTEMP 29.4 °C
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 54

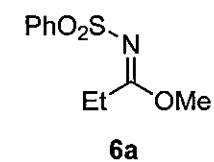
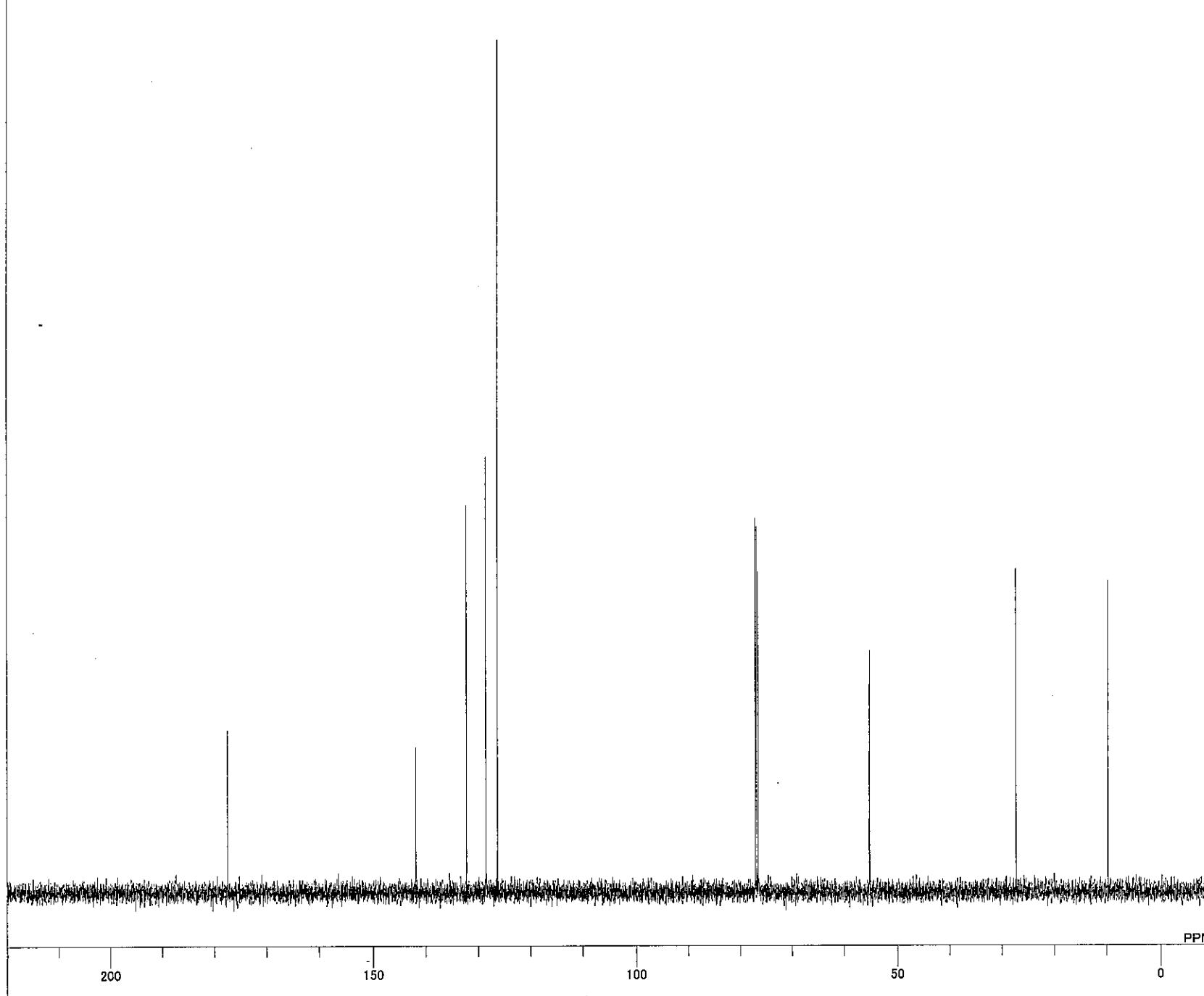


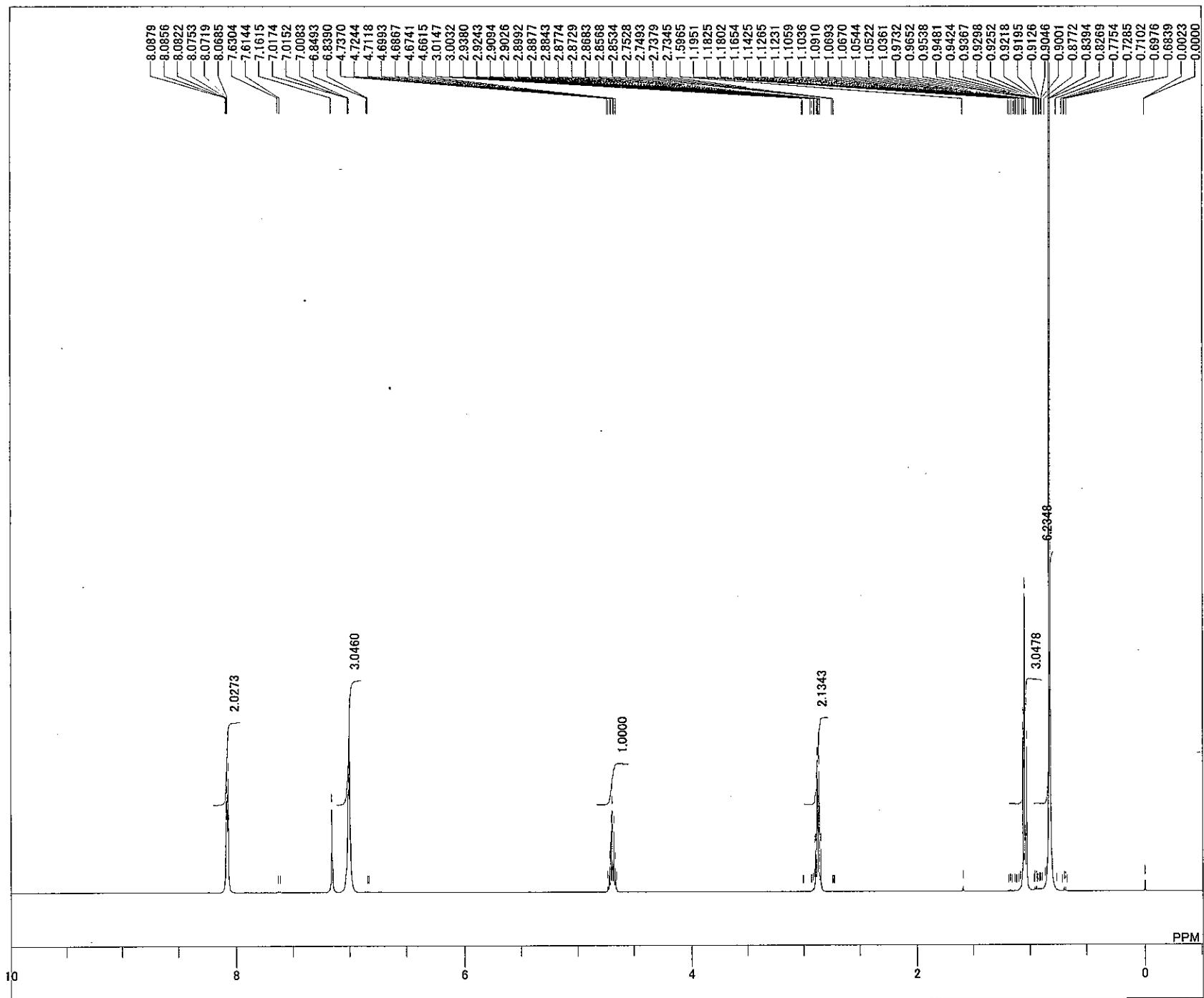


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4.38 KHz
9.64 Hz
13107
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
29.2 c
CDCL₃
0.00 ppm
0.12 Hz
36



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POINT 26214
FREQU 31249.52 Hz
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.1H 29.5 c
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EXREF RGAIN 56

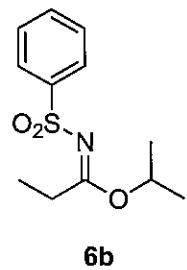


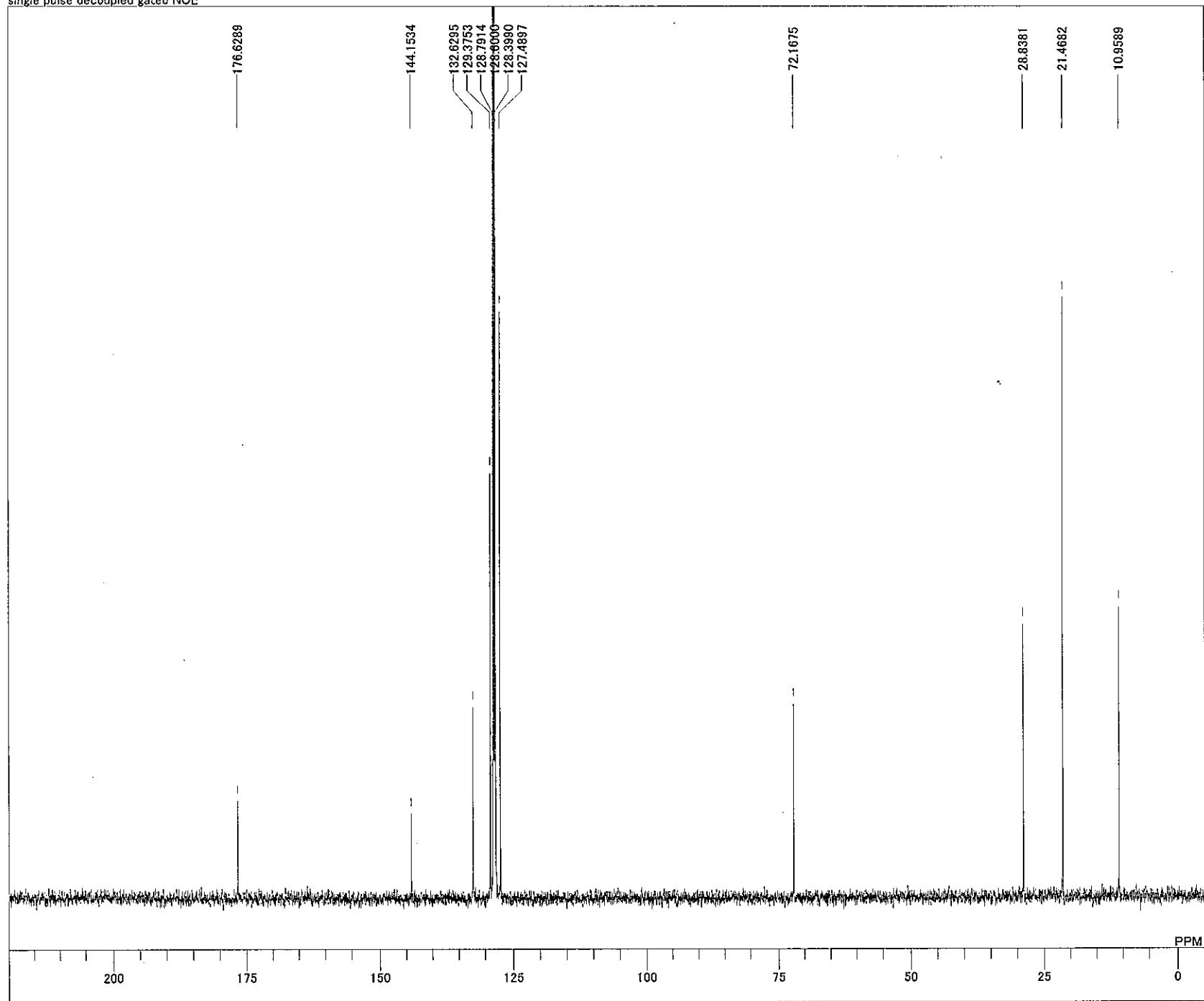


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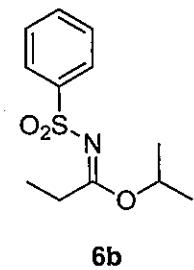
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EXMOD
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OBSET
OBFIN
POINT    16400
FREQU    9286.78 Hz
SCANS     8
ACQTM    1.7642 sec
PD        5.0000 sec
PW1
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CTEMP    24.4 c
SLVNT
EXREF
BF        0.00 ppm
RGAIN    0.12 Hz
            30
1H
C6D6

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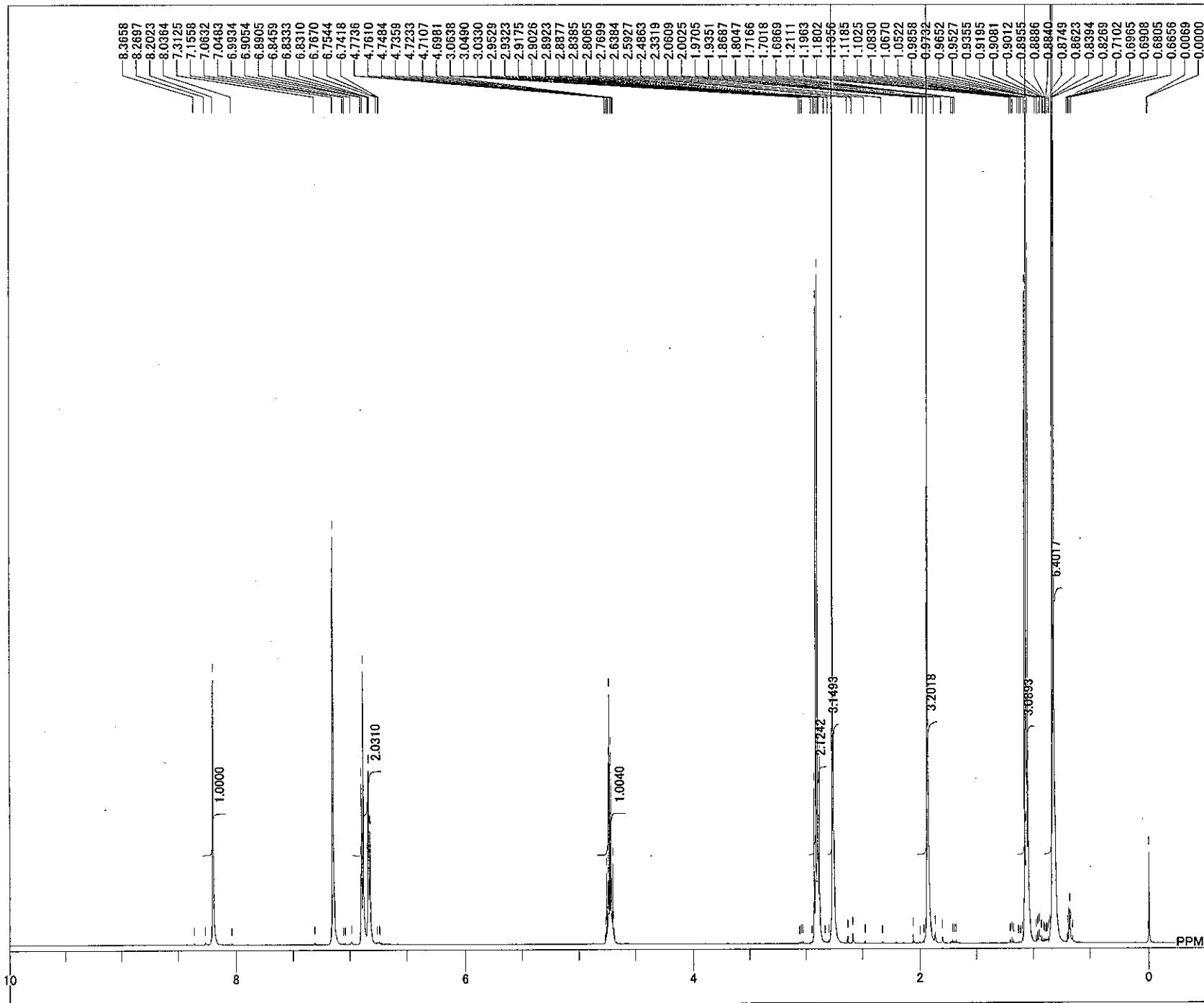




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EXMOD
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OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
10-07-2007 17:59:10
13C
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124.51 MHz
3.45 KHz
6.00 Hz
26224
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24.7 c
C6D6
128.60 ppm
0.12 Hz
50



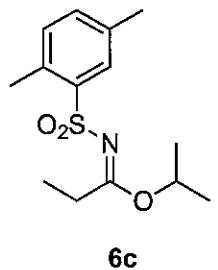
6b



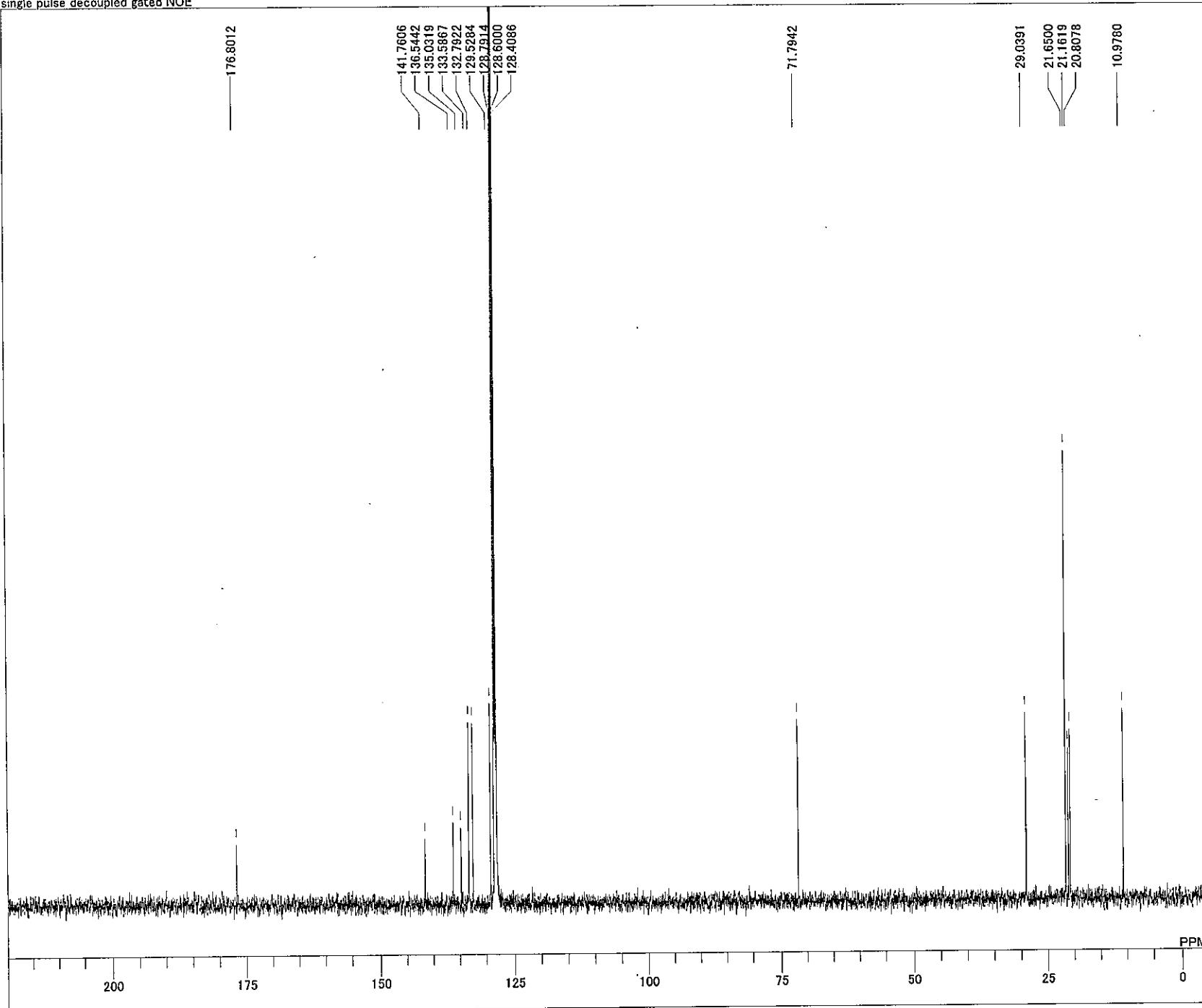
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POINT   16400
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SCANS   8
ACQTM   1.7642 sec
PD      5.0000 sec
PW1     6.50 usec
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RGAIN   34

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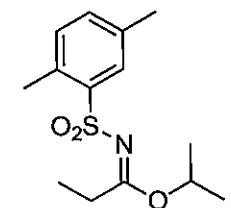


6c

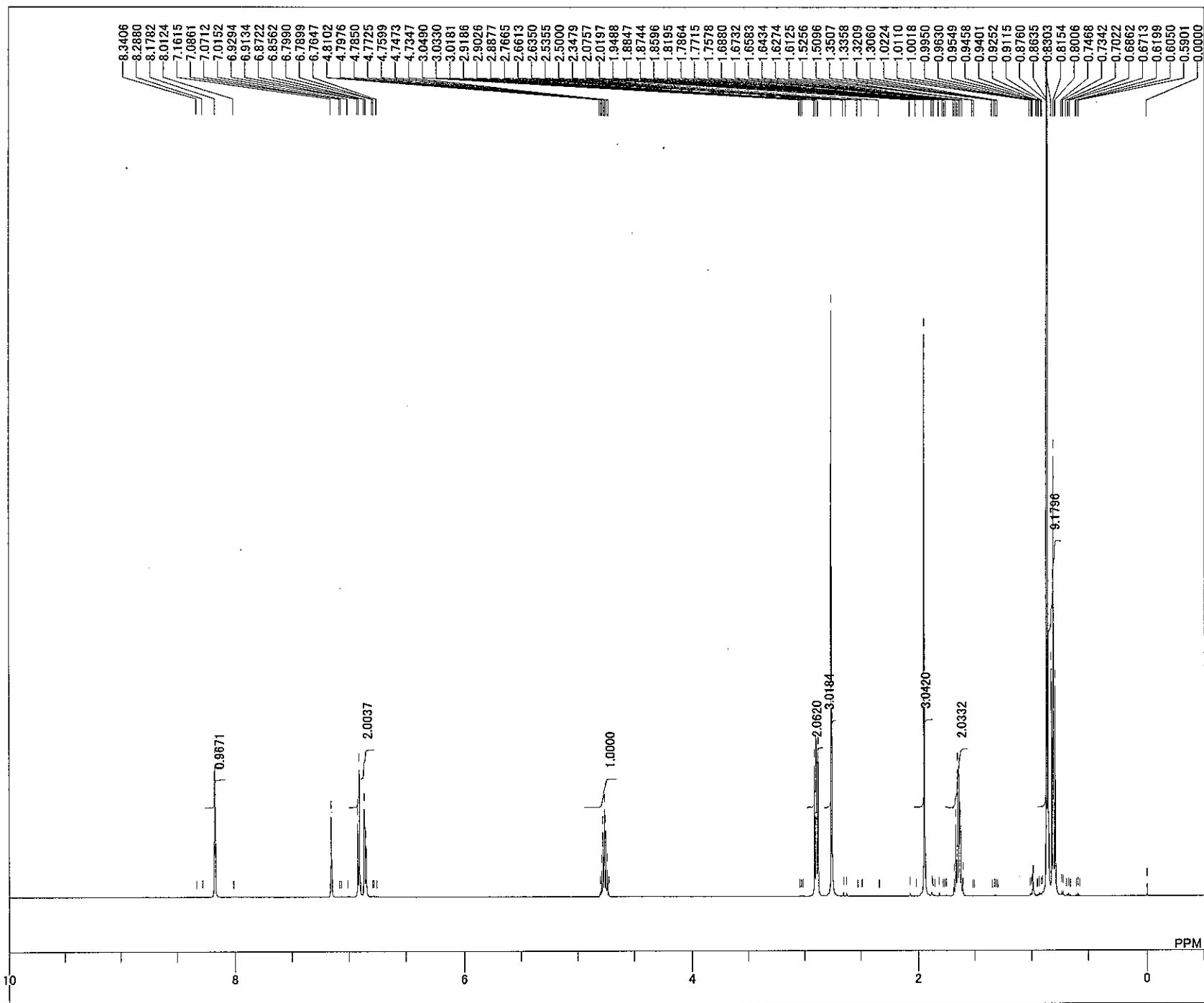


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OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

1H
C6D6
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
2.0000 sec
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24.8 c
128.60 ppm
0.12 Hz
50

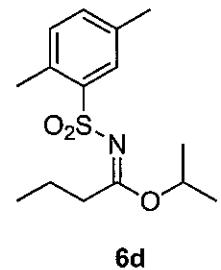


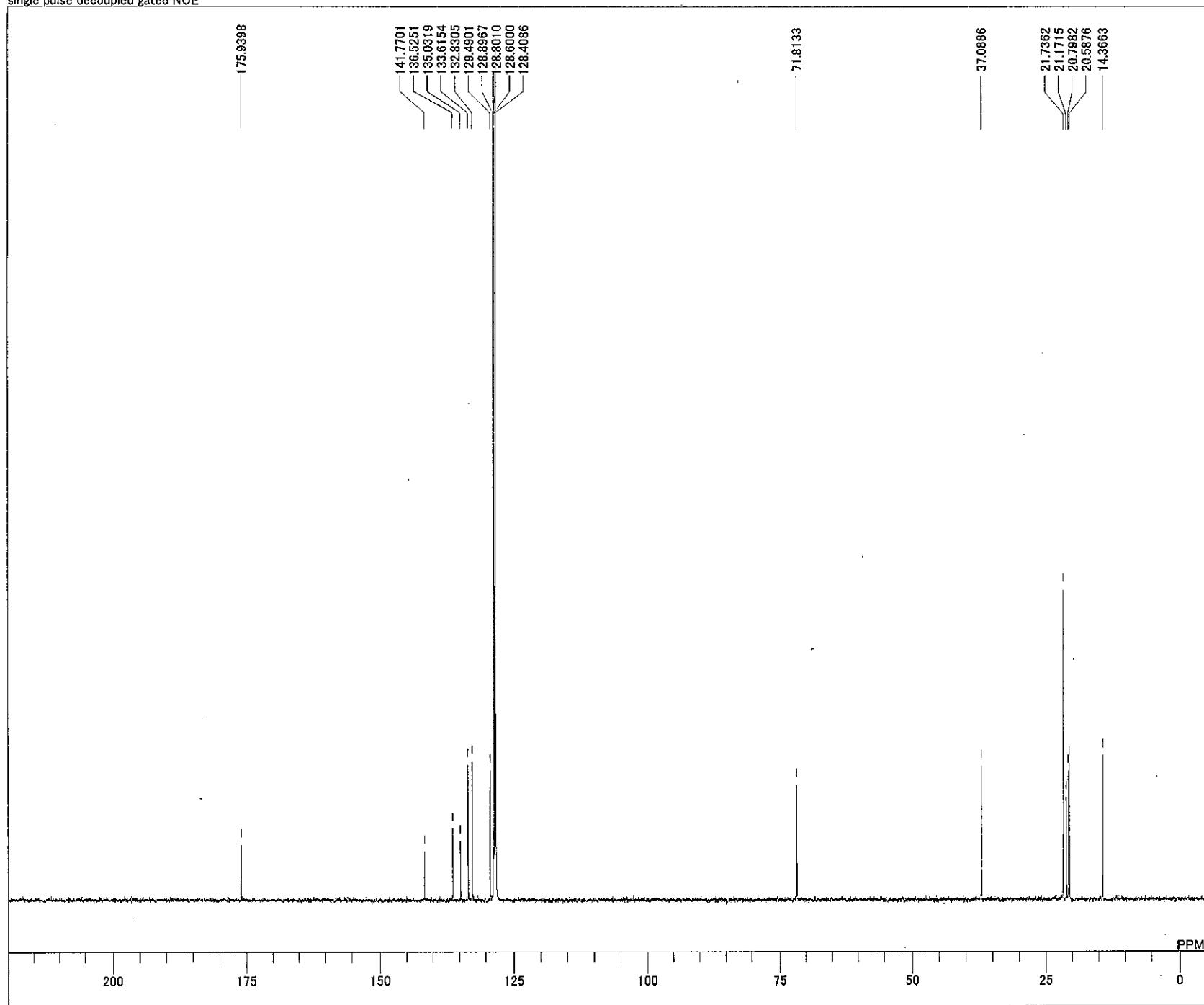
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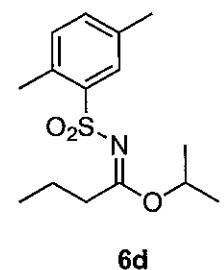
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PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Ecadata**florian**fb366pp2-1.jdf
10-07-2007 20:13:04
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16400
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5.0000 sec
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1H
24.5 c
C6D6
0.00 ppm
0.12 Hz
26



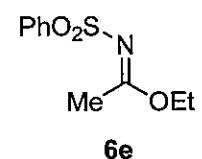
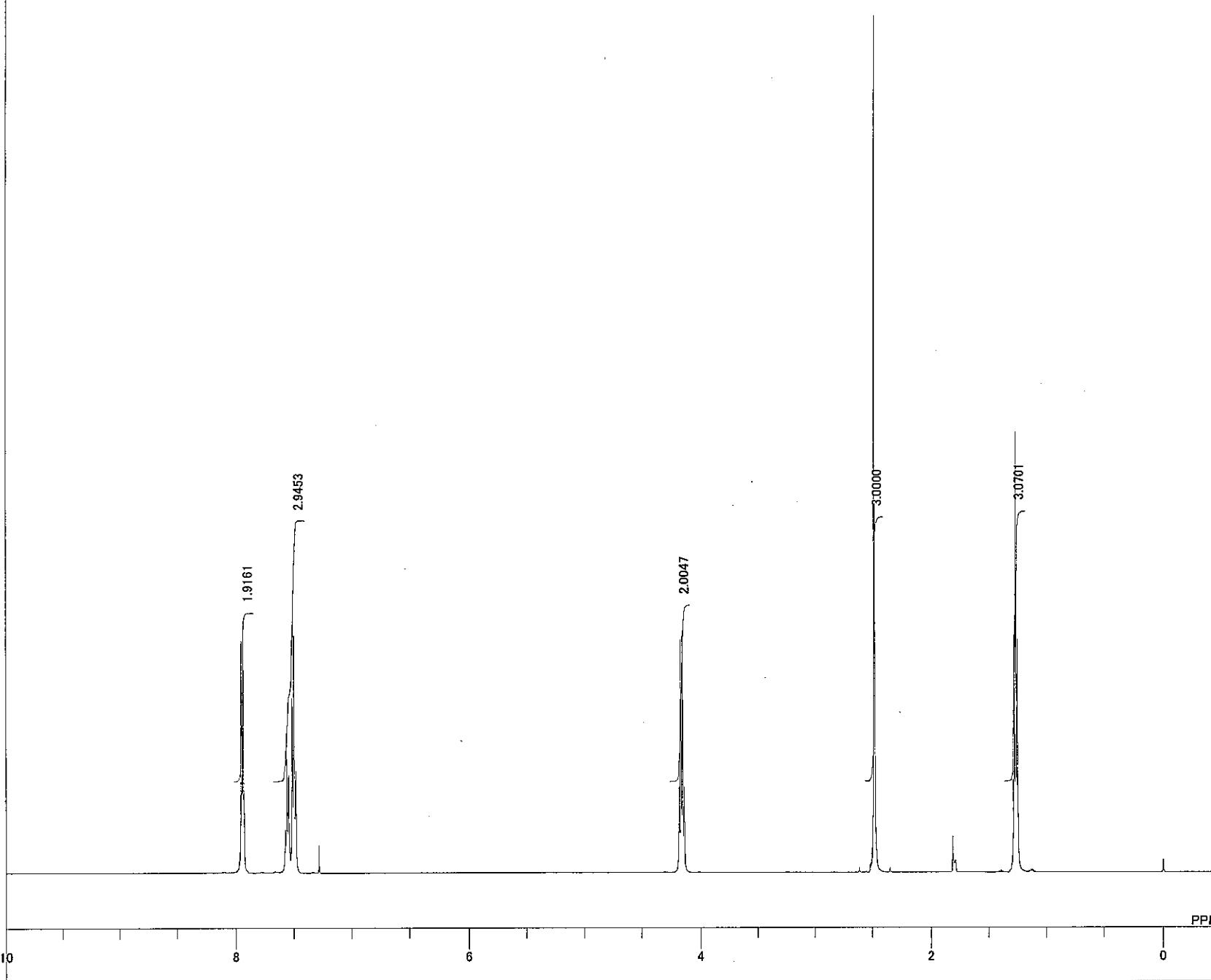


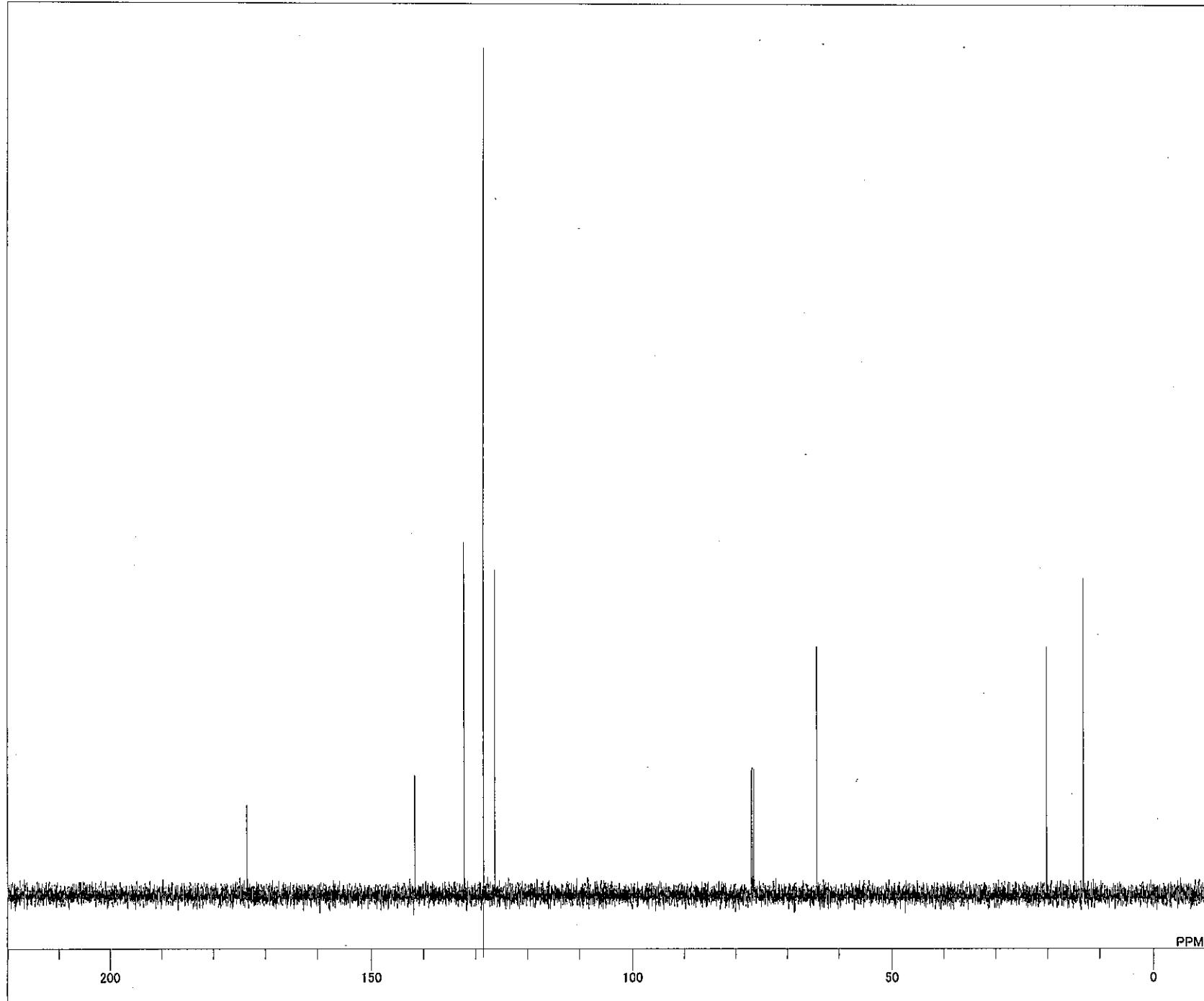
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
10-07-2007 20:17:01
13C
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OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
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CTEMP
SLVNT
EXREF
BF
RGAIN
1H
C6D6
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
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24.9 c
128.60 ppm
0.12 Hz
50



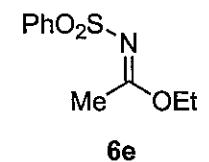
6d

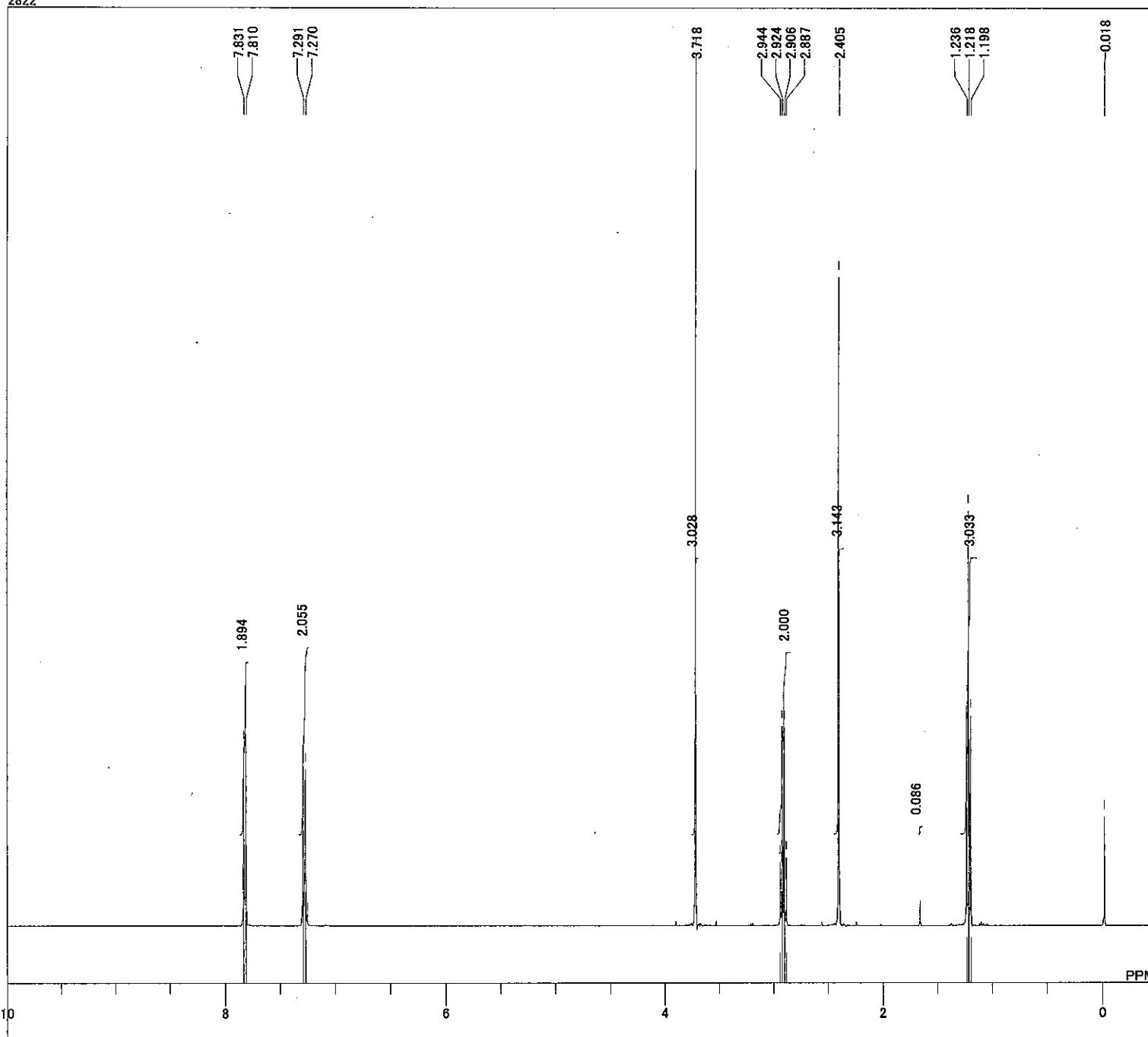
C:\Documents and Settings\All Users\Docu
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13107
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1H
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0.00 ppm
0.12 Hz
30





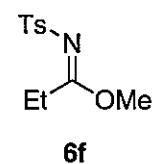
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OBFIN 6.00 Hz
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FREQU 31249.52 Hz
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ACQTM 0.8389 sec
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CTEMP 29.4 c
SLVNT CDCl₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56

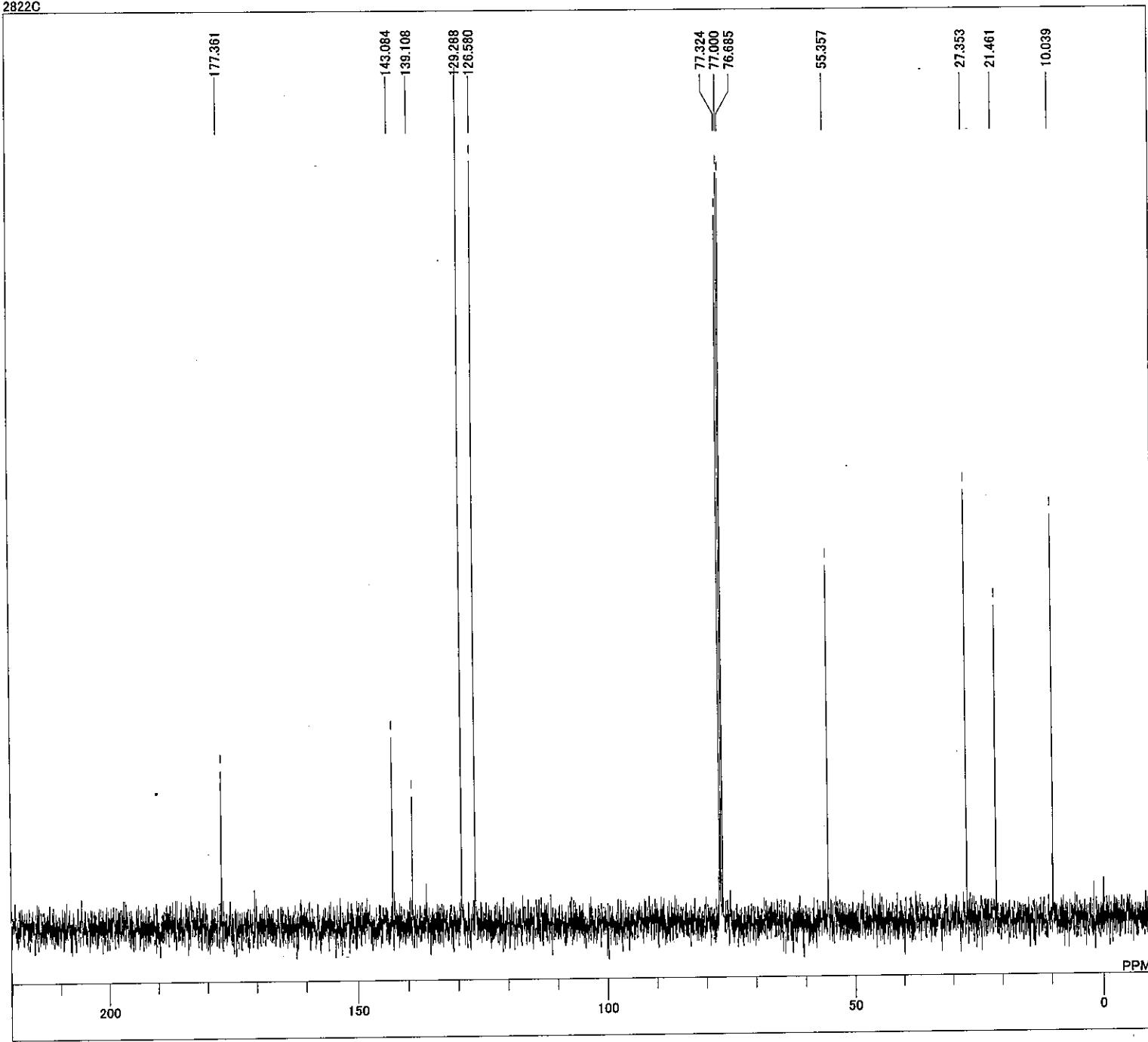




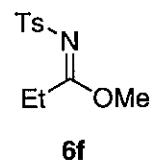
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POINT 7503.00 Hz
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SCANS 2.1837 sec
ACQTM 2.0000 sec
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RGAIN 28

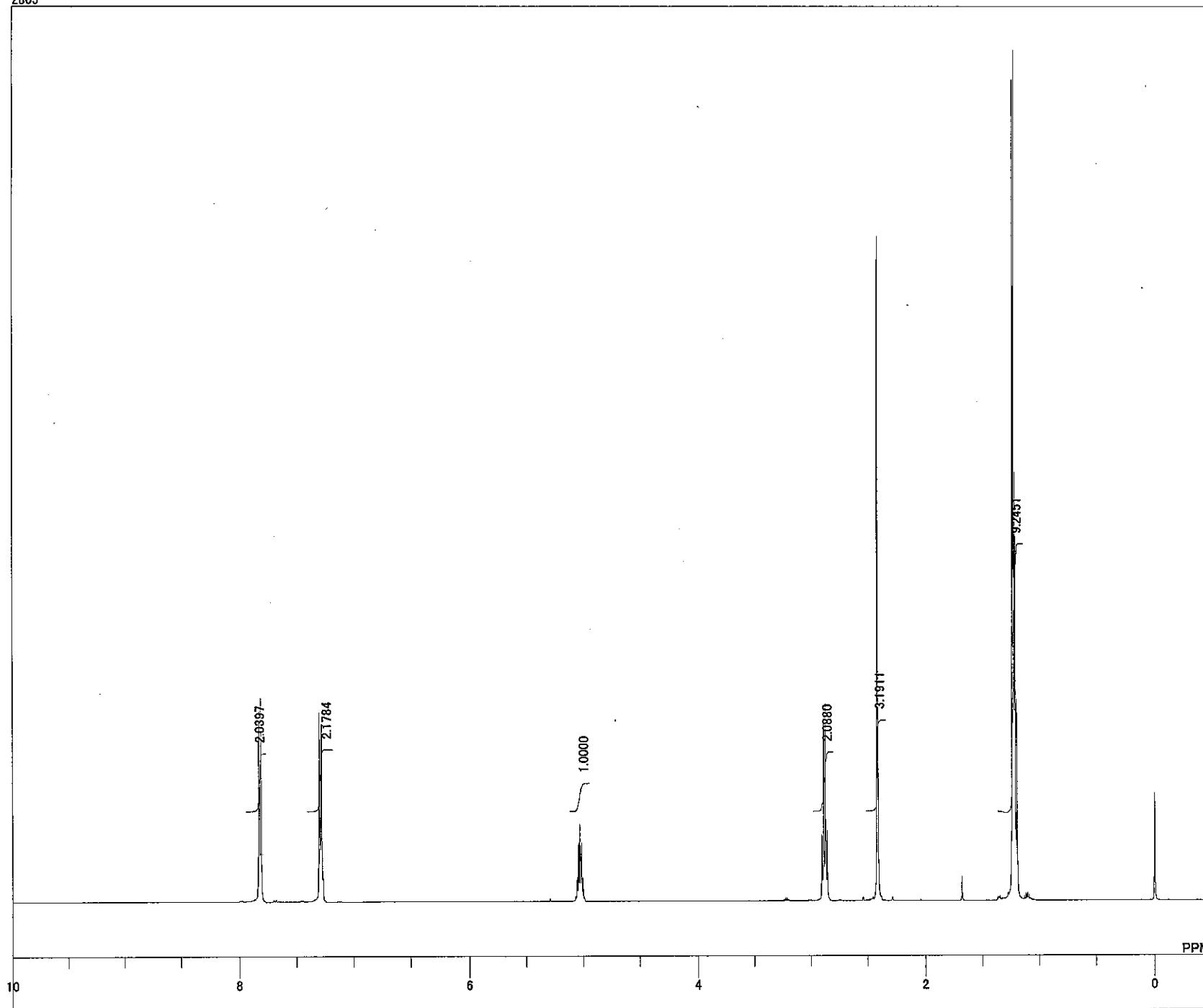
1H -NMR (CDCl₃) δ :
7.82 (2H, d, J = 8.2 Hz),
7.28 (2H, d, J = 8.2 Hz),
2.92 (2H, q, J = 7.6 Hz),
1.22 (3H, t, J = 7.6 Hz).



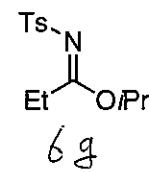


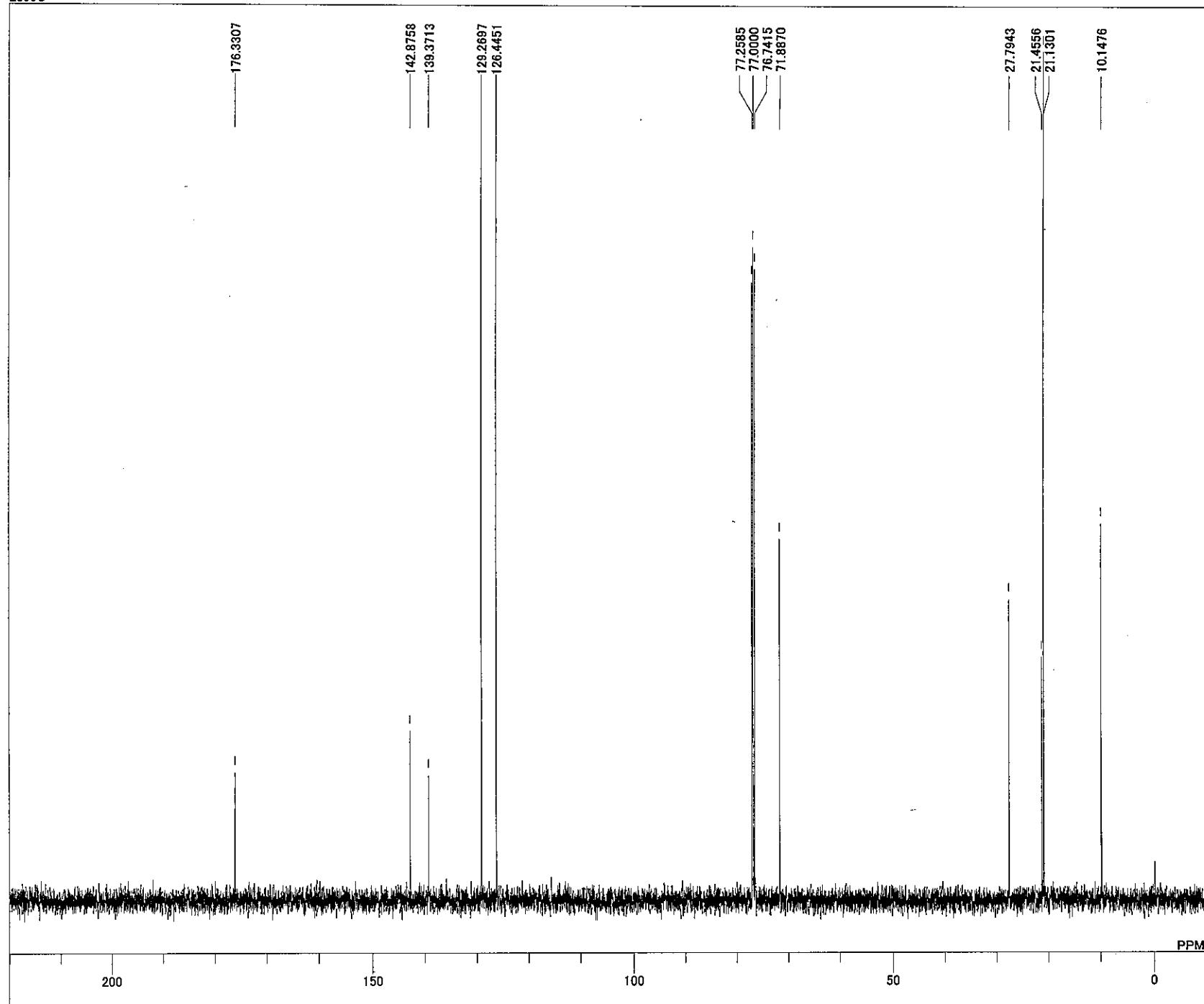
C:\Documents and Settings\deltaelta\My Documents\Personal Folder\Matsubara\2801-2900\2822C.als
2822C
12-12-2007 15:15:09
13C
single_pulse dec
100.53 MHz
5.35 KHz
5.86 Hz
32768
31407.04 Hz
21
1.0433 sec
2.0000 sec
2.83 usec
1H
24.9 c
CDCL₃
77.00 ppm
0.12 Hz
50
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



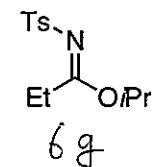


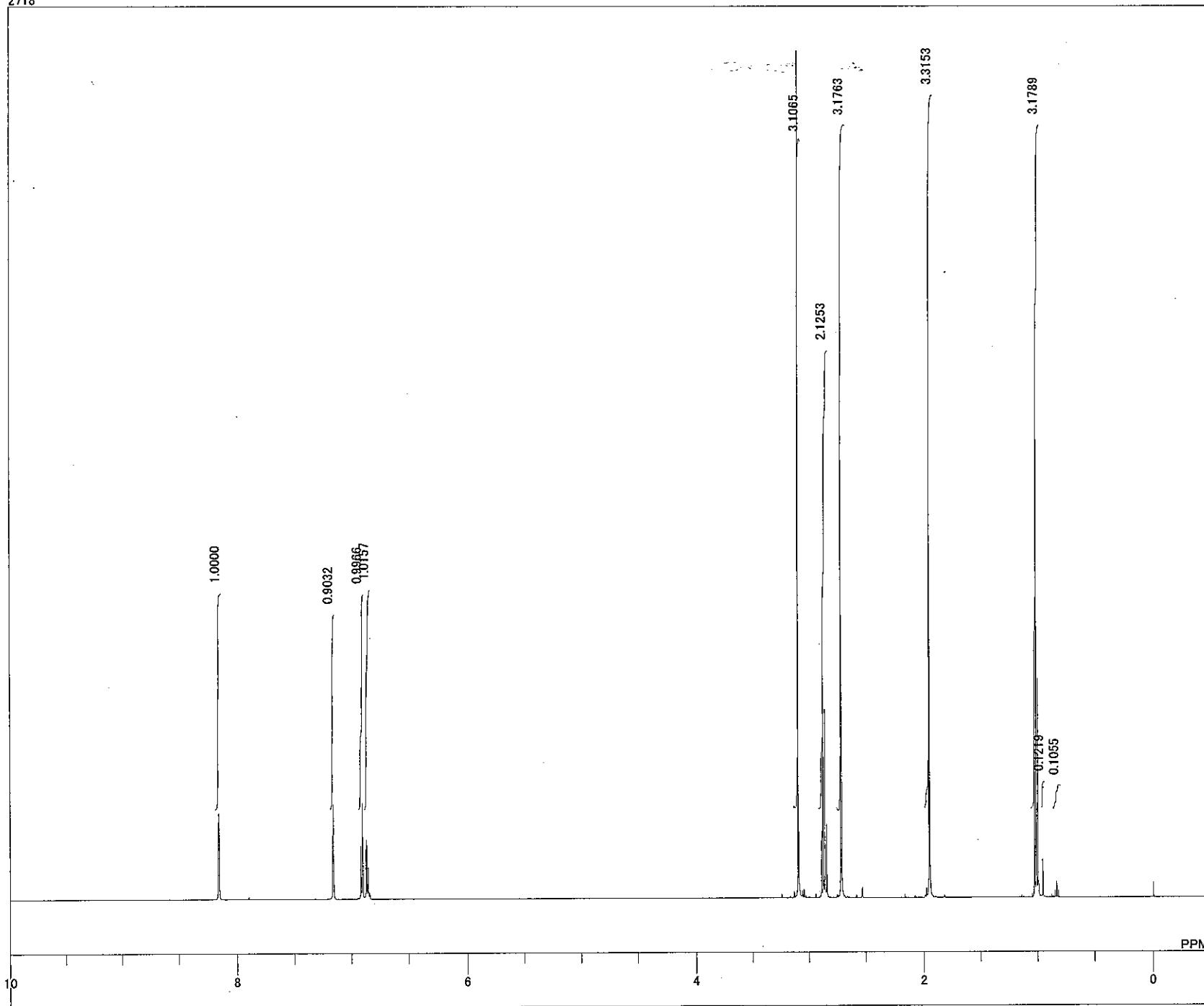
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2805
DATIM 30-11-2007 20:27:48
OBNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 32



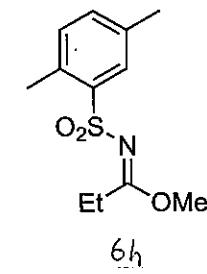


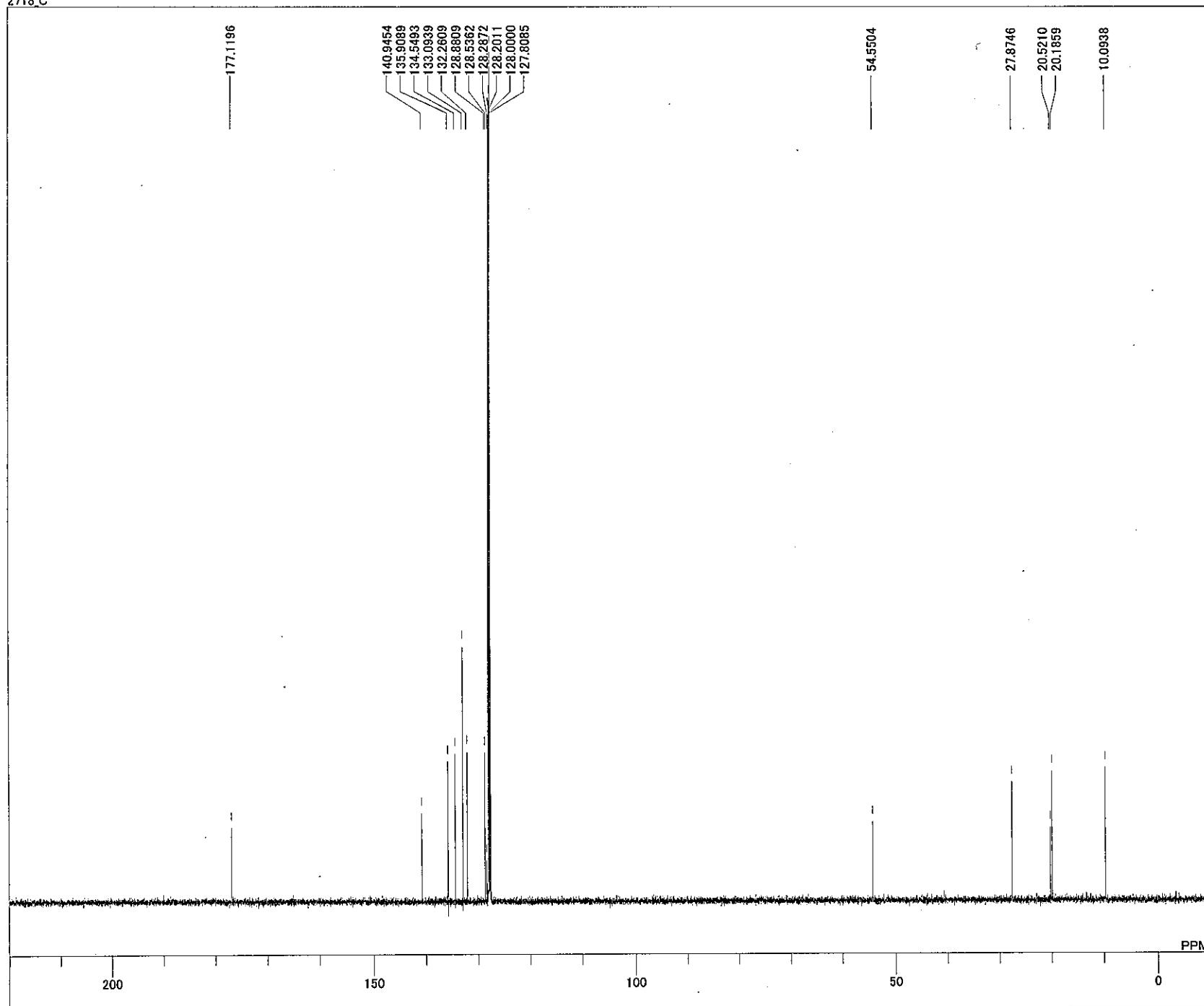
C:\Documents and Settings\All Users\Docu
2805C
30-11-2007 20:30:23
13C
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26214
31249.52 Hz
43
0.8389 sec
2.0000 sec
3.67 usec
1H
22.5 c
CDCL₃
77.00 ppm
1.20 Hz
50



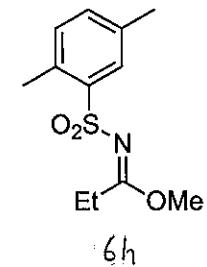


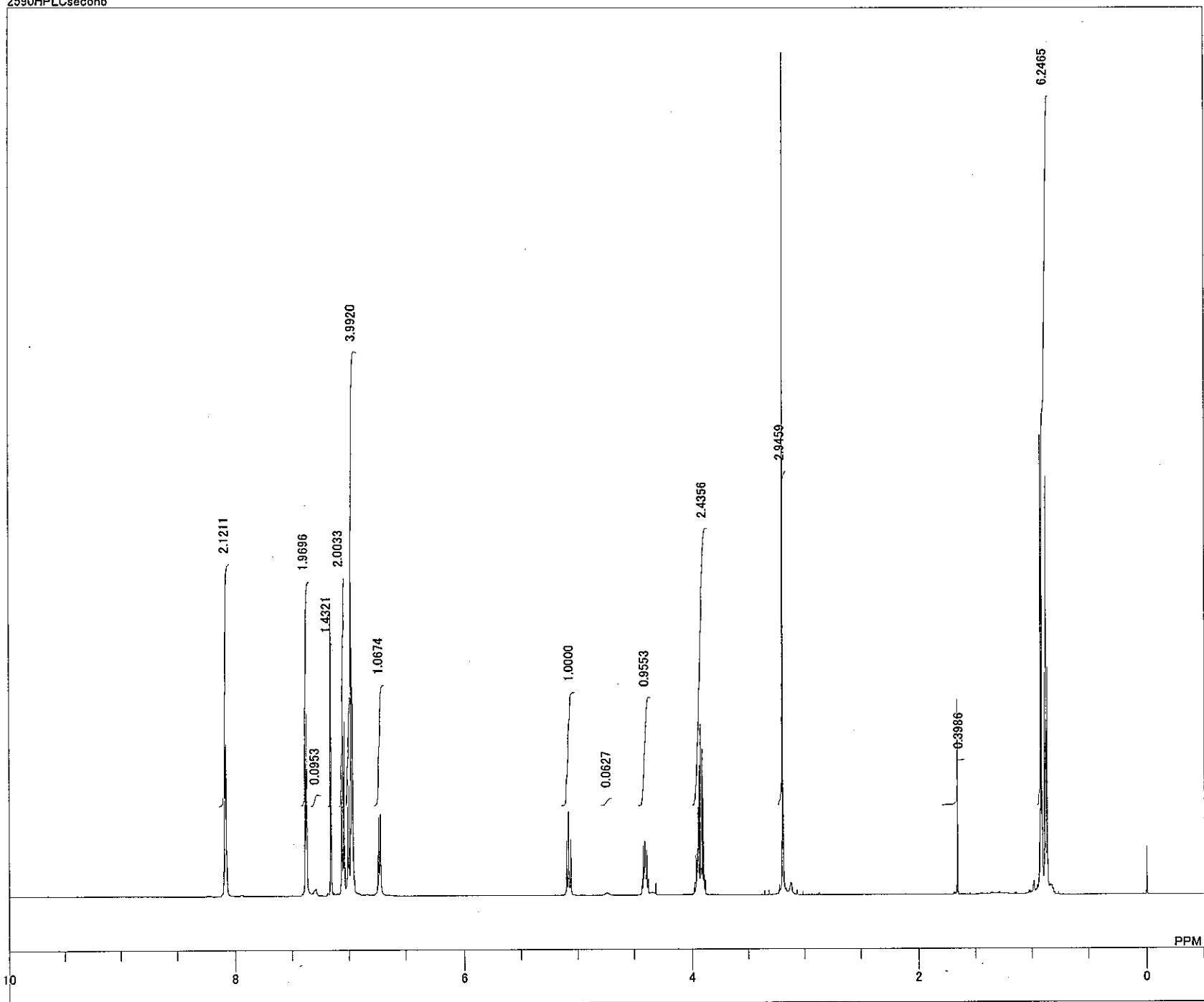
FILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
2718
02-08-2007 09:30:12
1H
1H NMR.ex2
495.13 MHz
4.38 KHz
9.64 Hz
13107
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
26.6 c
C6D6
0.00 ppm
0.12 Hz
30



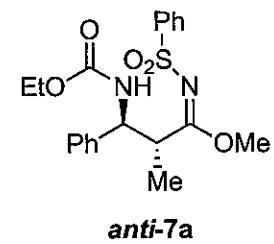


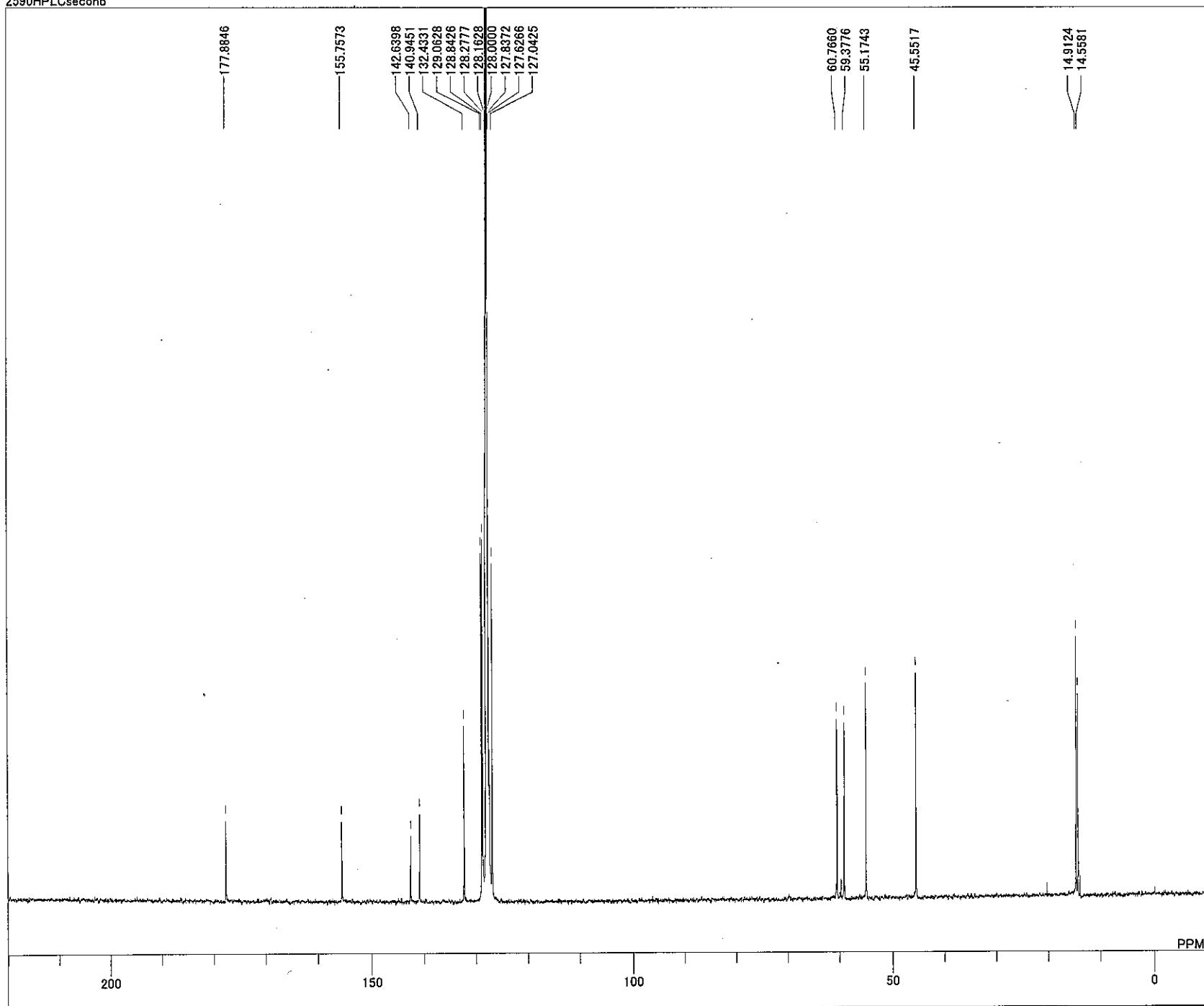
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2718_C
DATIM 02-08-2007 09:35:51
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 kHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 79
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 27.0 °C
SLVNT C6D6
EXREF 128.00 ppm
BF 0.12 Hz
RGAIN 50





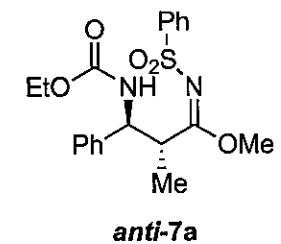
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2590HPLCsecond
DATIM 24-02-2007 03:42:17
1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 kHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT C6D6
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38



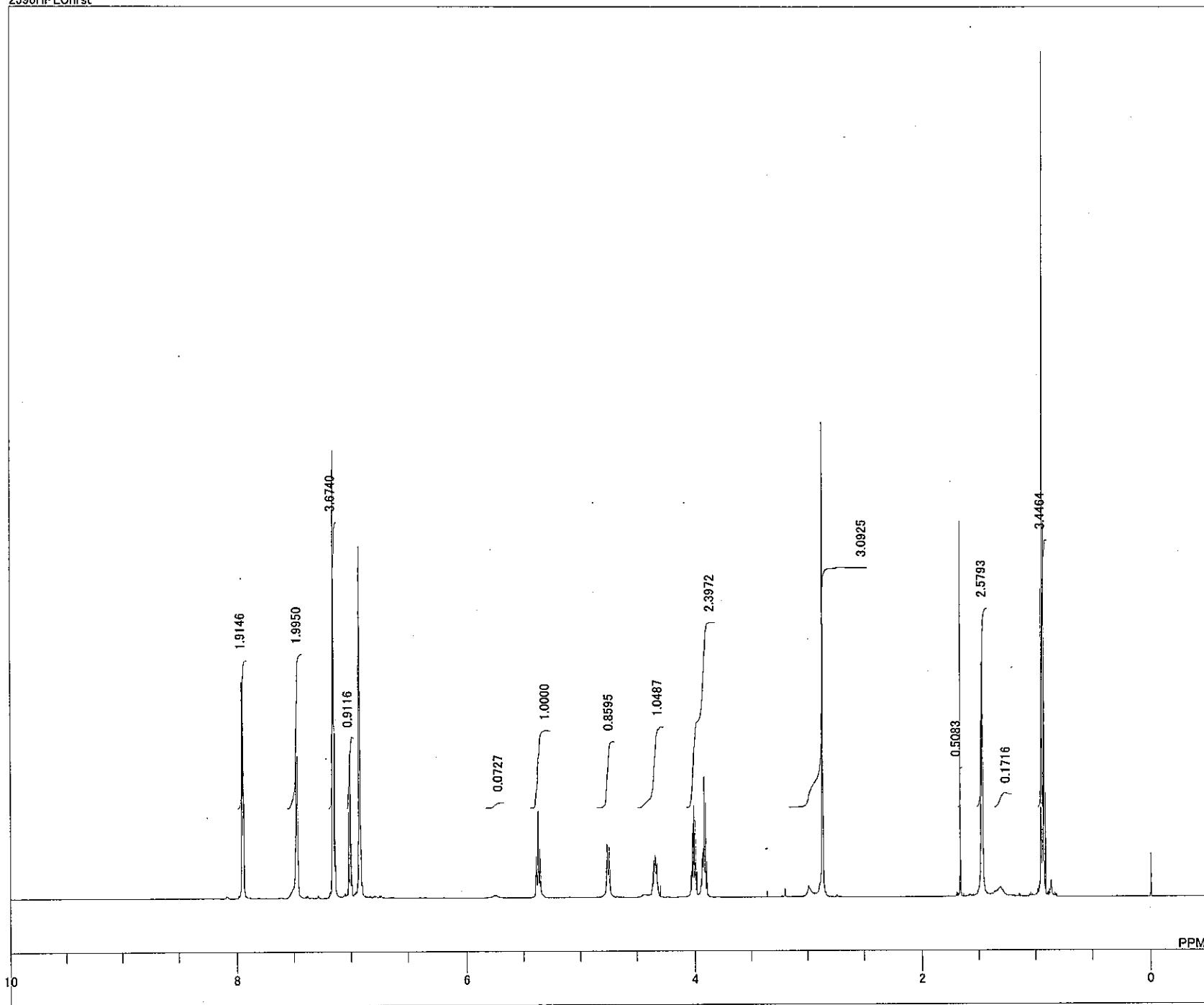


FILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

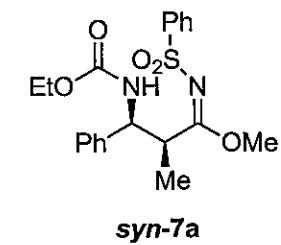
2590HPLCsecond
24-02-2007 04:28:53
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
1024
0.6921 sec
2.0000 sec
3.45 usec
1H
C6D6
128.00 ppm
0.12 Hz
56

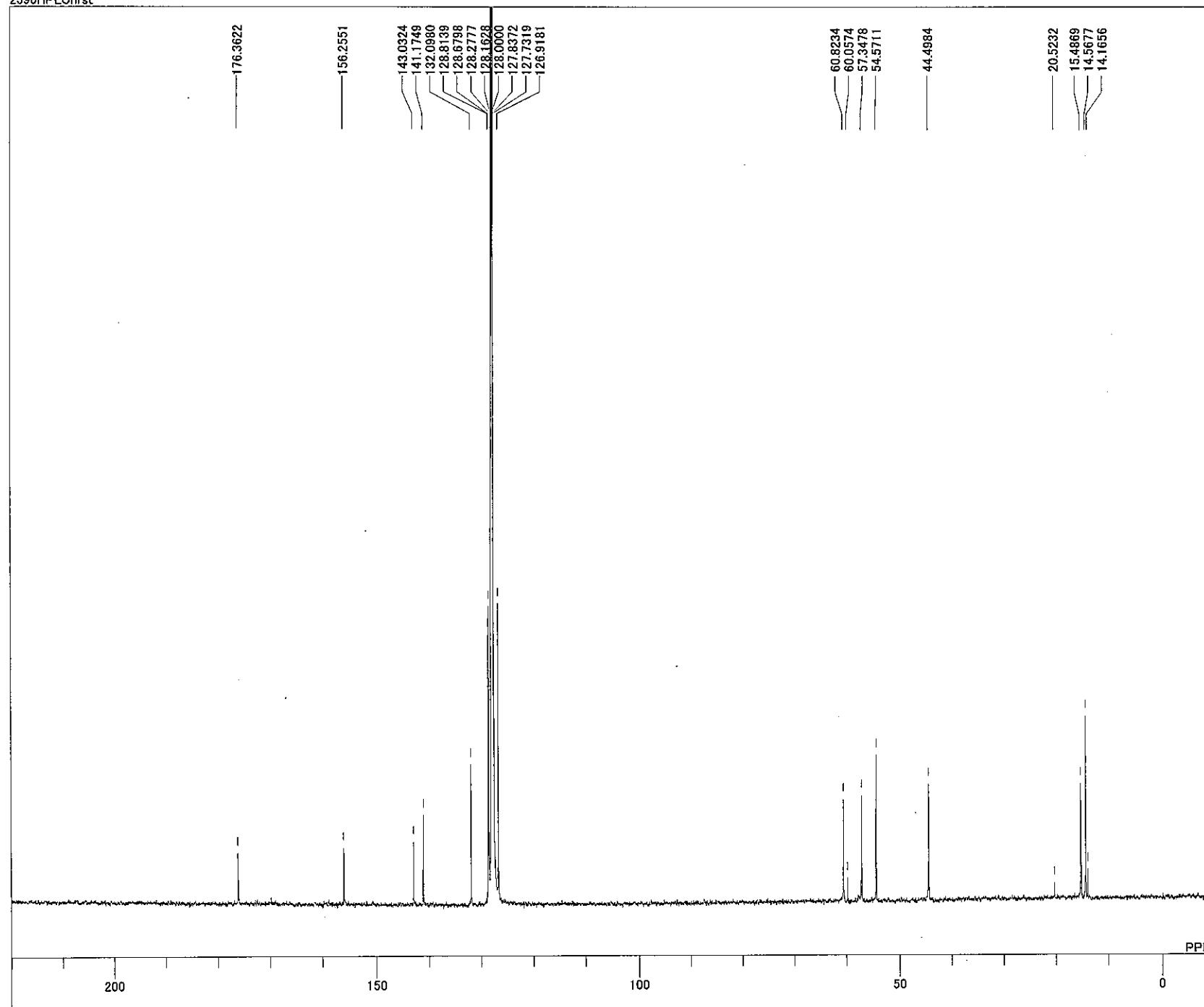


anti-7a

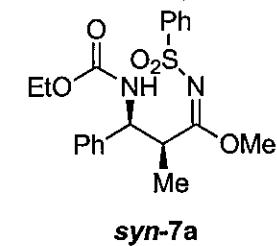


DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\Documents and Settings\All Users\Docu
2590HPLCfirst
24-02-2007 02:45:04
1H
single_pulse.ex2
600.17 MHz
5.30 kHz
5.47 Hz
16384
11261.26 Hz,
16
1.4549 sec
4.0000 sec
7.30 usec
1H
21.6 c
C6D6
0.00 ppm
0.12 Hz
40

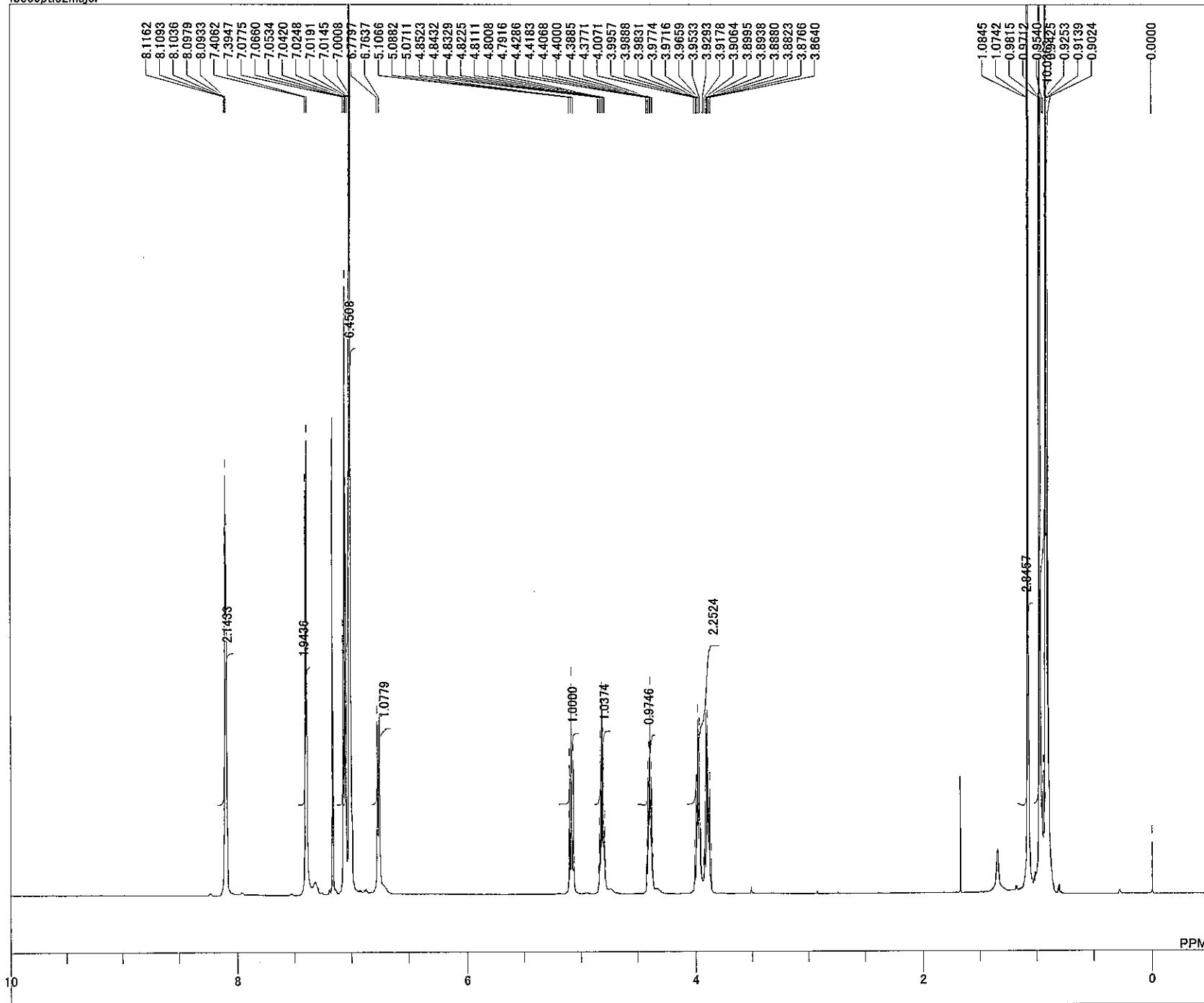




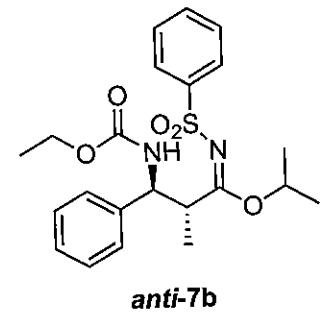
C:\Documents and Settings\All Users\Docu
2590HPLCfirst
24-02-2007 03:31:40
13C
single_pulse_dec
150.92 MHz
8.52 kHz
1.74 Hz
32768
47348.48 Hz
1024
0.6921 sec
2.0000 sec
3.45 usec
1H
23.0 c
C6D6
128.00 ppm
0.12 Hz
56

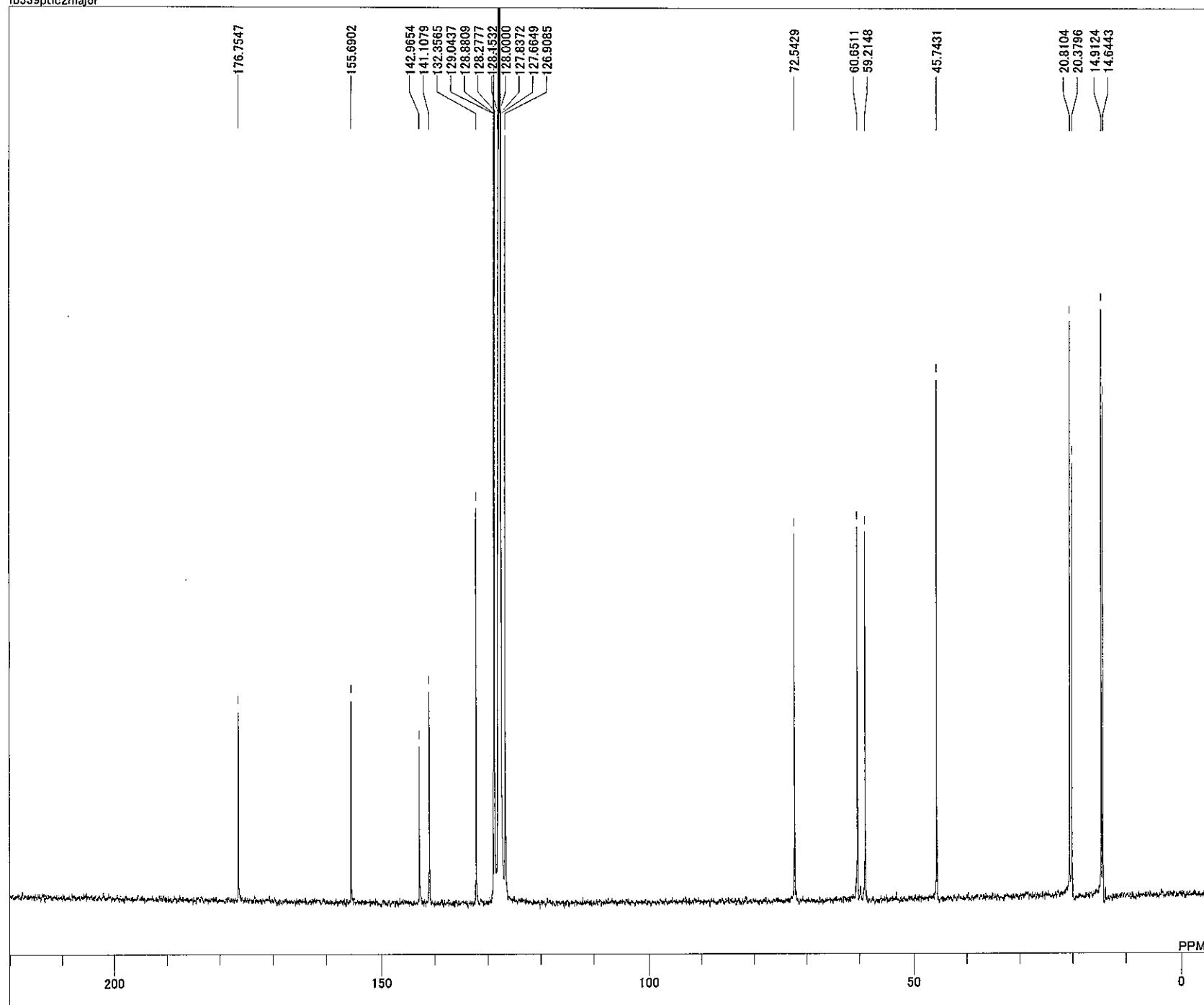


syn-7a

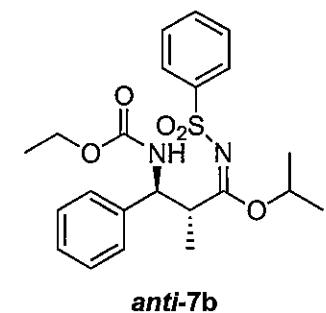


C:\Documents and Settings\All Users\Docu
fb339ptlc2major
24-08-2007 19:34:52
1H
single_pulse.ex2
600.17 MHz
5.30 kHz
5.47 Hz
16384
11261.26 Hz
16
1.4549 sec
4.0000 sec
9.10 usec
1H
29.7 c
C6D6
0.00 ppm
0.12 Hz
30

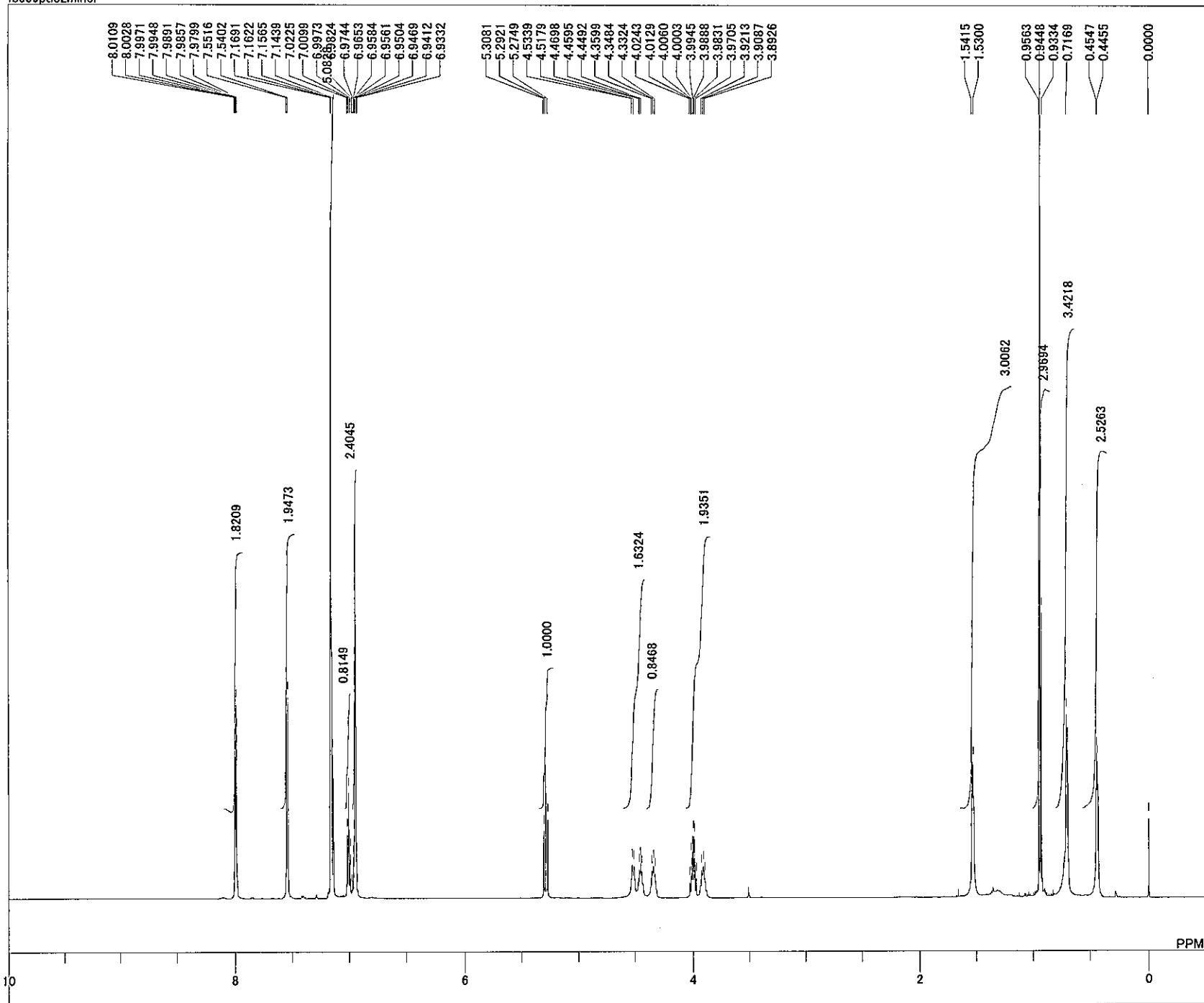




DFILE C:\Documents and Settings\All Users\Docu
COMNT fb339ptlc2major
DATIM 24-08-2007 20:21:34
13C single_pulse_dec
EXMOD 150.92 MHz
OBFRQ 8.52 KHz
OBSET 1.74 Hz
OBFIN 32768
POINT 47348.48 Hz
FREQU 1024
SCANS 0.6921 sec
ACQTM 2.0000 sec
PD 3.45 usec
PW1
IRNUC
CTEMP 30.6 c
SLVNT C6D6
EXREF 128.00 ppm
BF 0.12 Hz
RGAIN 56



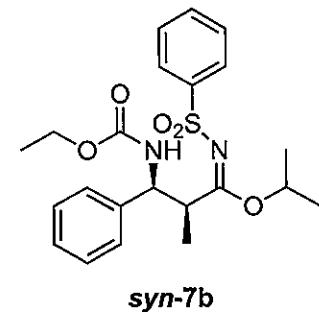
anti-7b



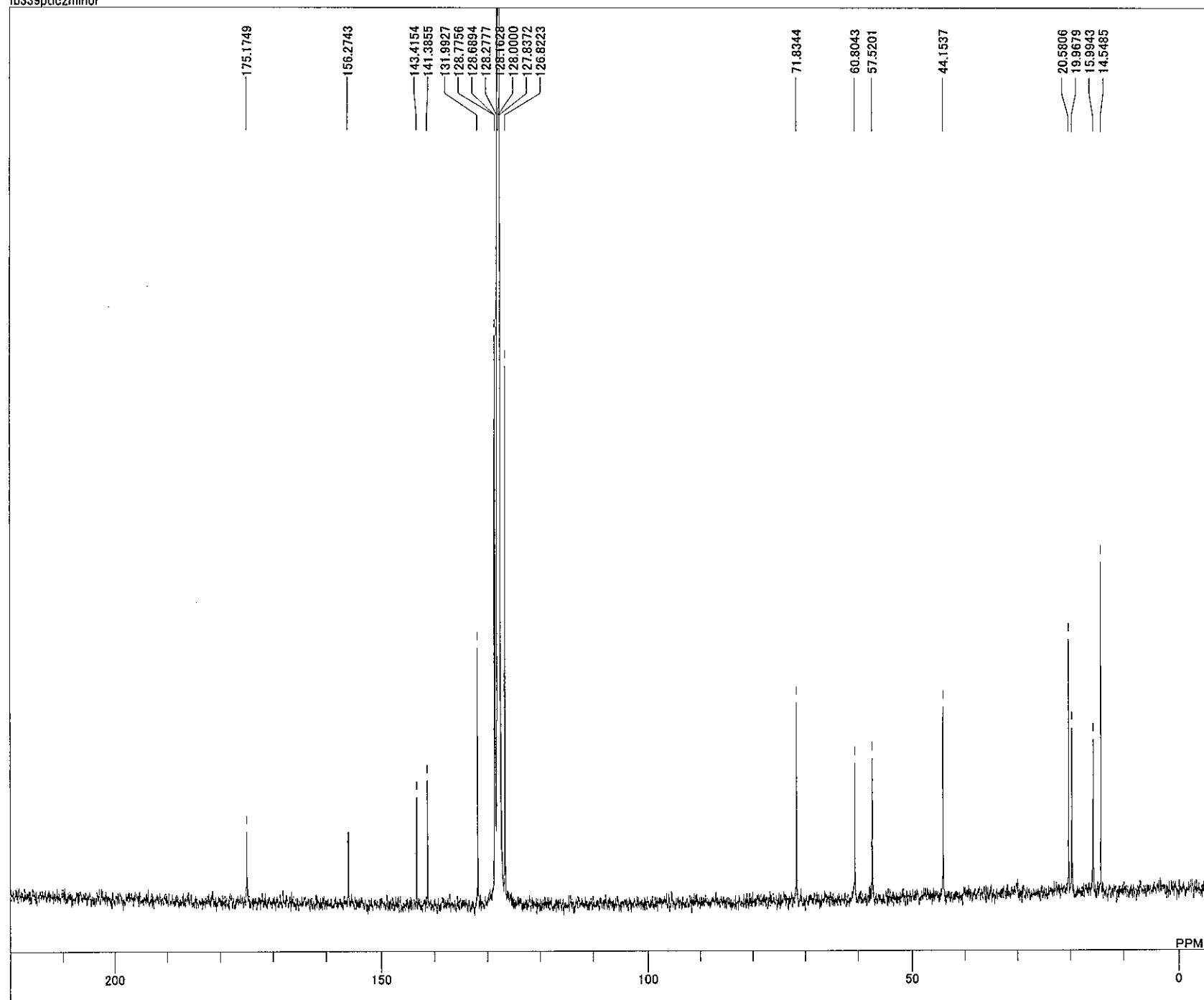
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DFILE C:\Documents and Settings\All Users\Docu
COMNT fb339ptlc2minor
DATIM 24-08-2007 20:31:39
OBNUC 1H
EXMOD single_pulse.ex2
CBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 9.10 usec
IRNUC 1H
CTEMP 29.9 c
SLVNT C6D6
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38

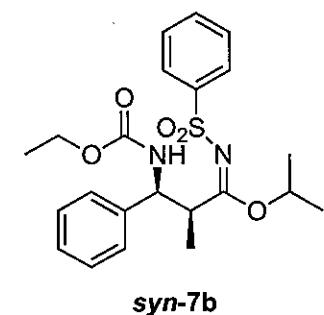
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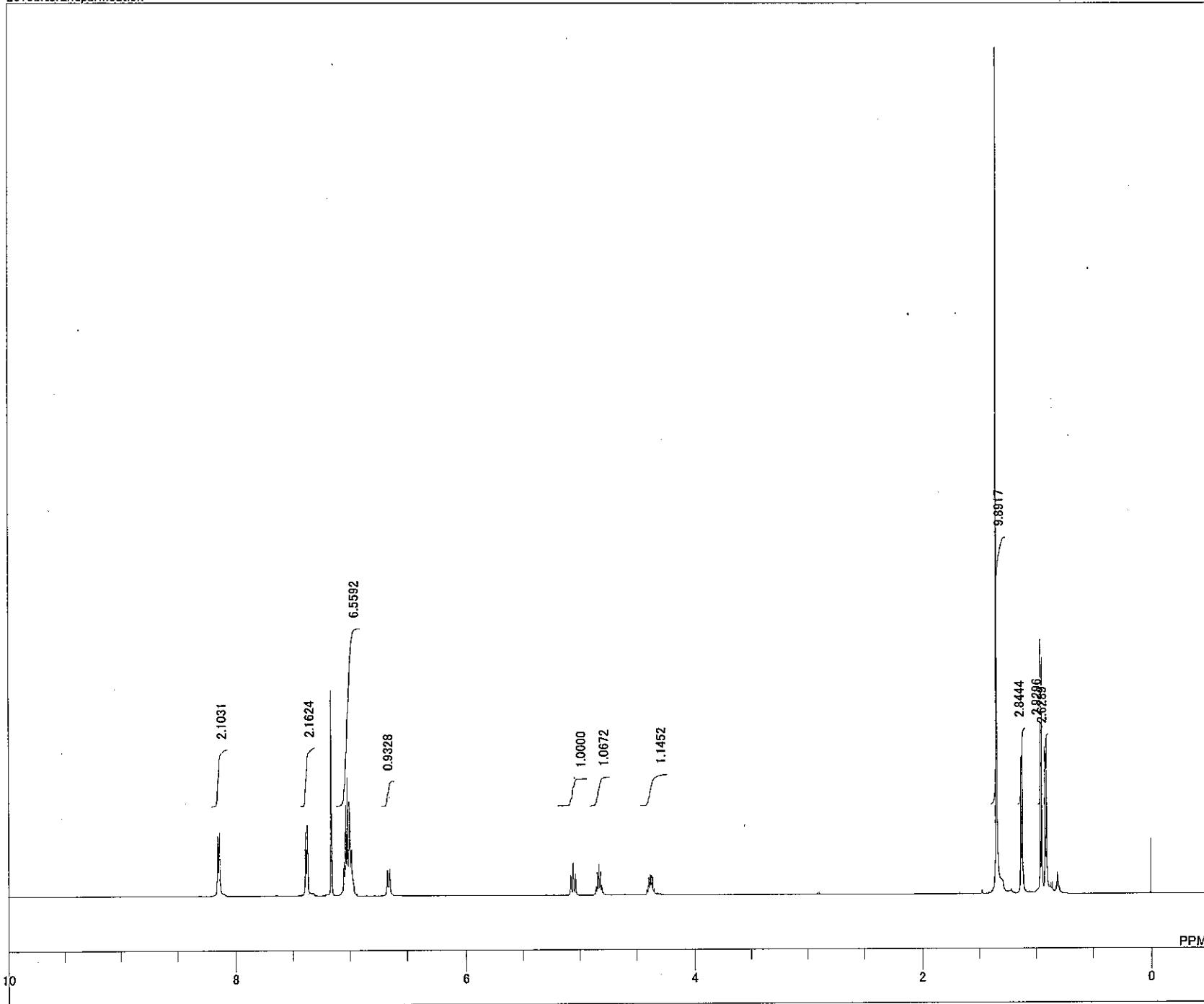


syn-7b

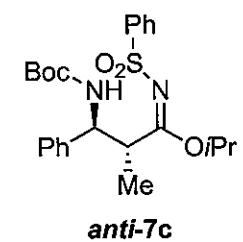


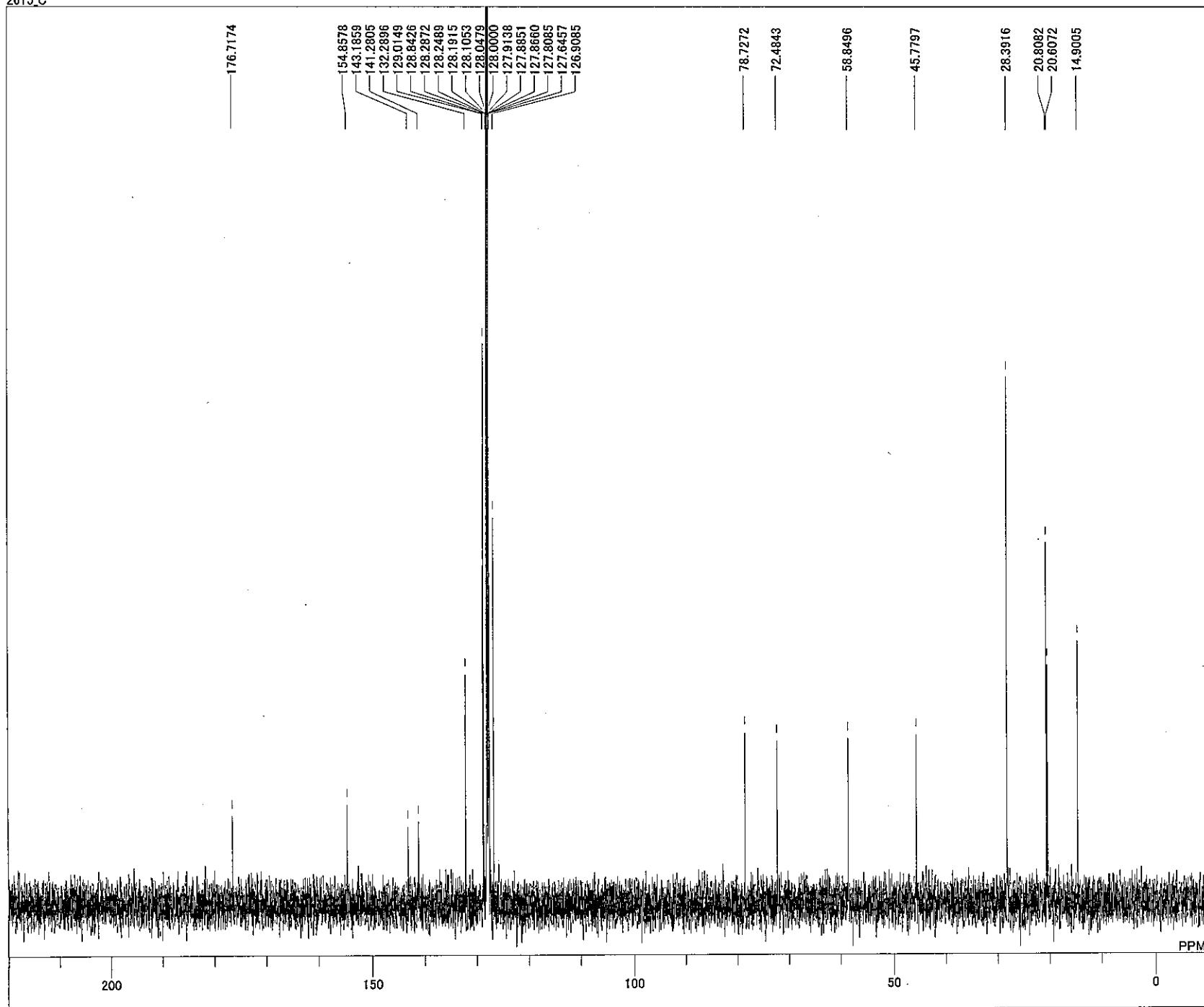
C:\Documents and Settings\All Users\Docu
fb339ptlc2minor
24-08-2007 21:18:15
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
1024
0.6921 sec
2.0000 sec
3.45 usec
1H
30.6 c
C6D6
128.00 ppm
0.12 Hz
56



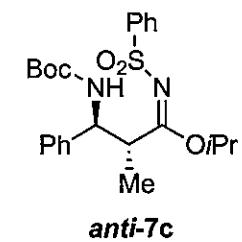


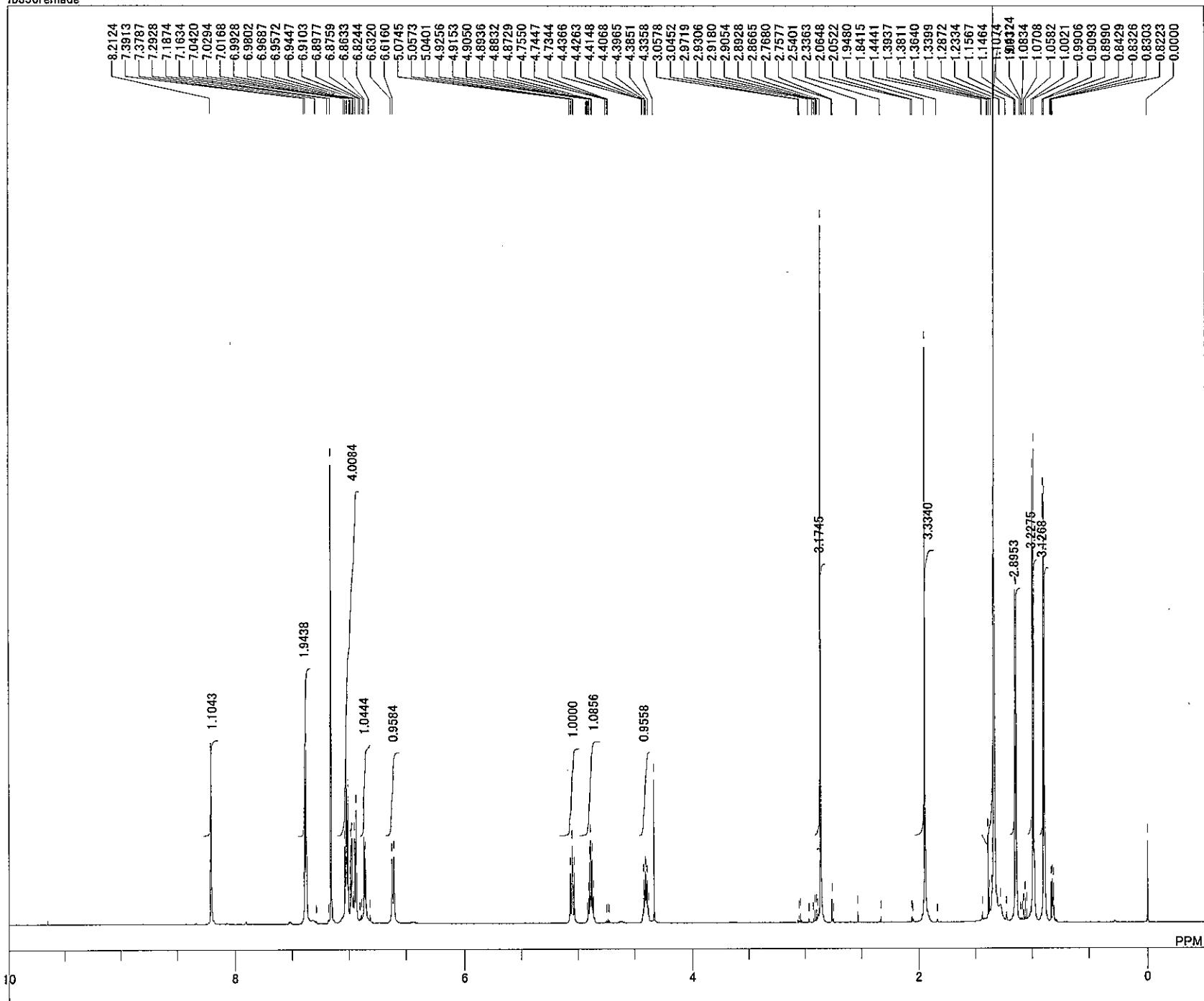
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2615after2ndpurification
DATIM 12-07-2007 20:57:31
1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 26.1 °C
SLVNT C6D6
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34



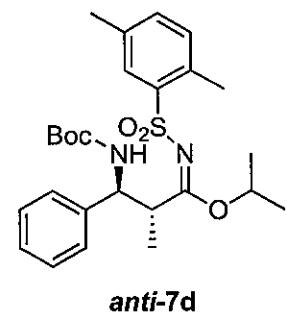


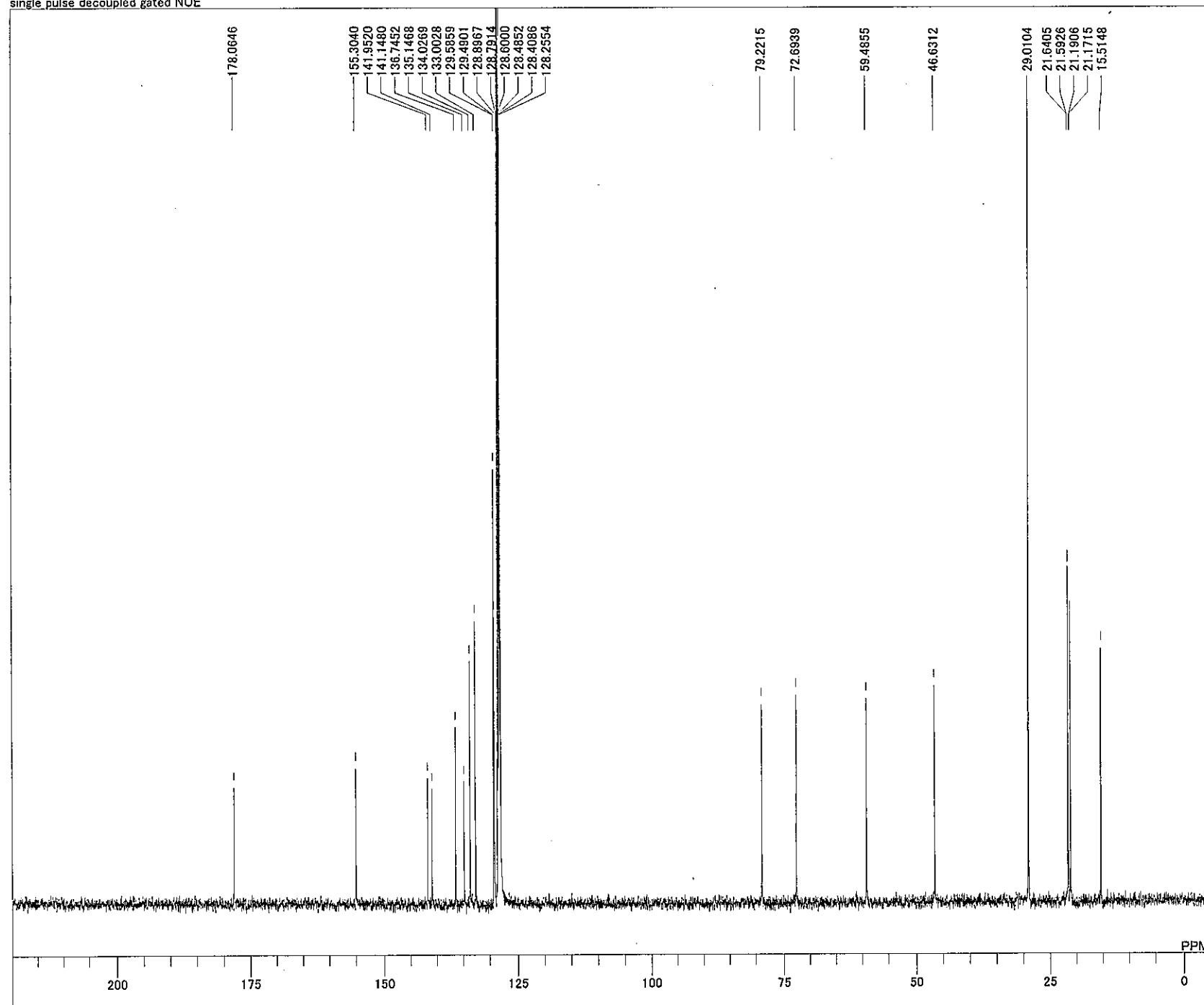
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2615.C
DATIM 12-07-2007 21:03:27
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 kHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 111
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 26.5 c
SLVNT C6D6
EXREF 128.00 ppm
BF 0.12 Hz
RGAIN 52



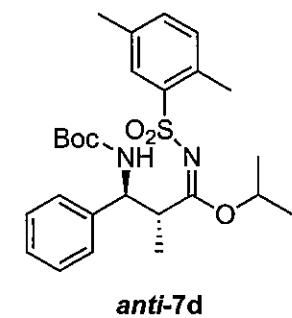


C:\Documents and Settings\ALICE2\Desktop
fb356remade
28-08-2007 21:09:29
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
20480
14076.79 Hz
16
1.4549 sec
4.0000 sec
9.10 usec
1H
29.4 c
C6D6
0.00 ppm
0.12 Hz
48

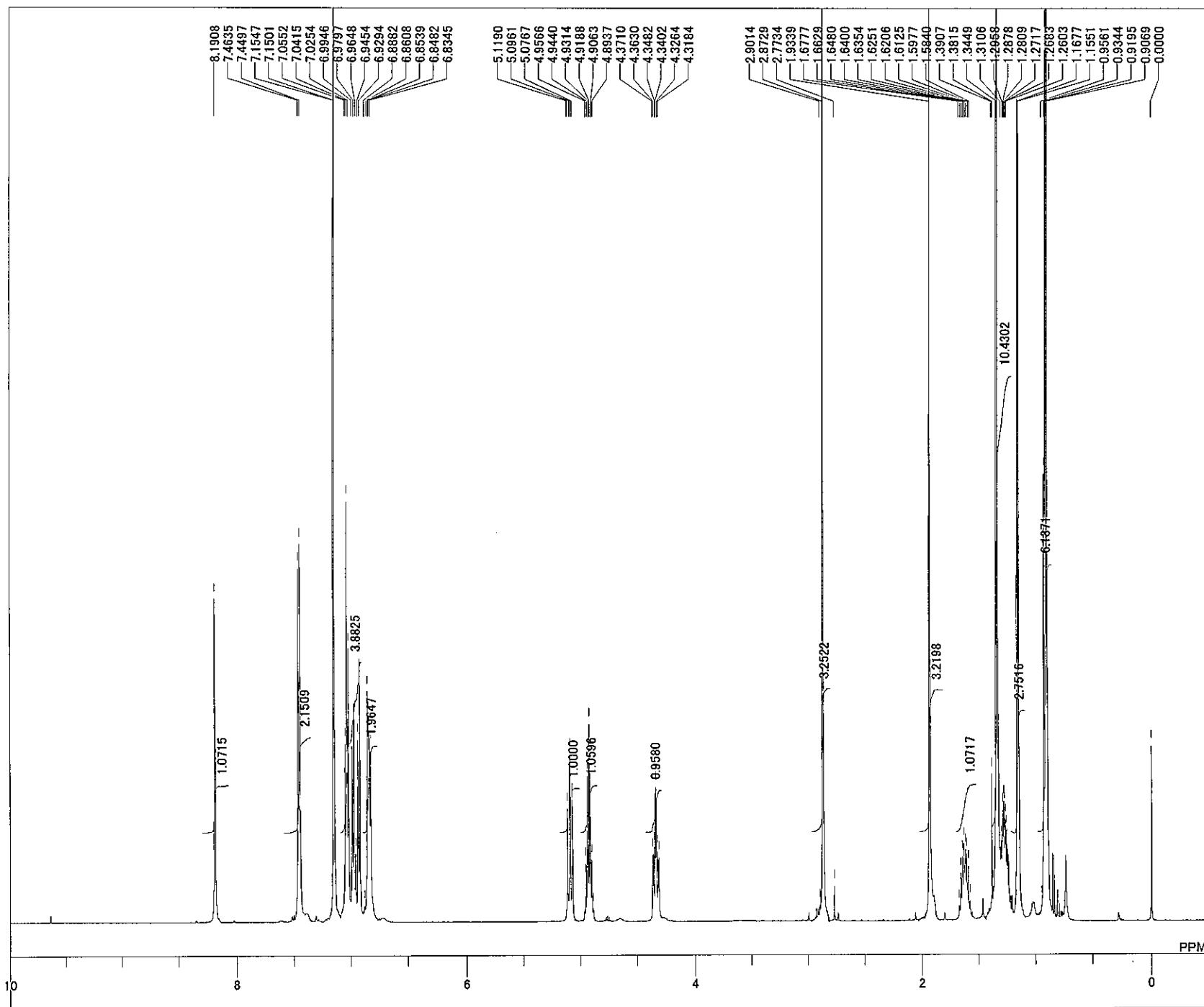




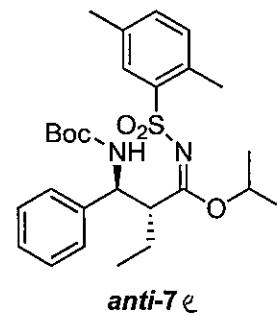
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
01-07-2007 16:24:51
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26224
31249.52 Hz
116
0.8389 sec
PD
2.0000 sec
3.67 usec
1H
25.5 c
C6D6
128.60 ppm
BF
1.20 Hz
RGAIN
50

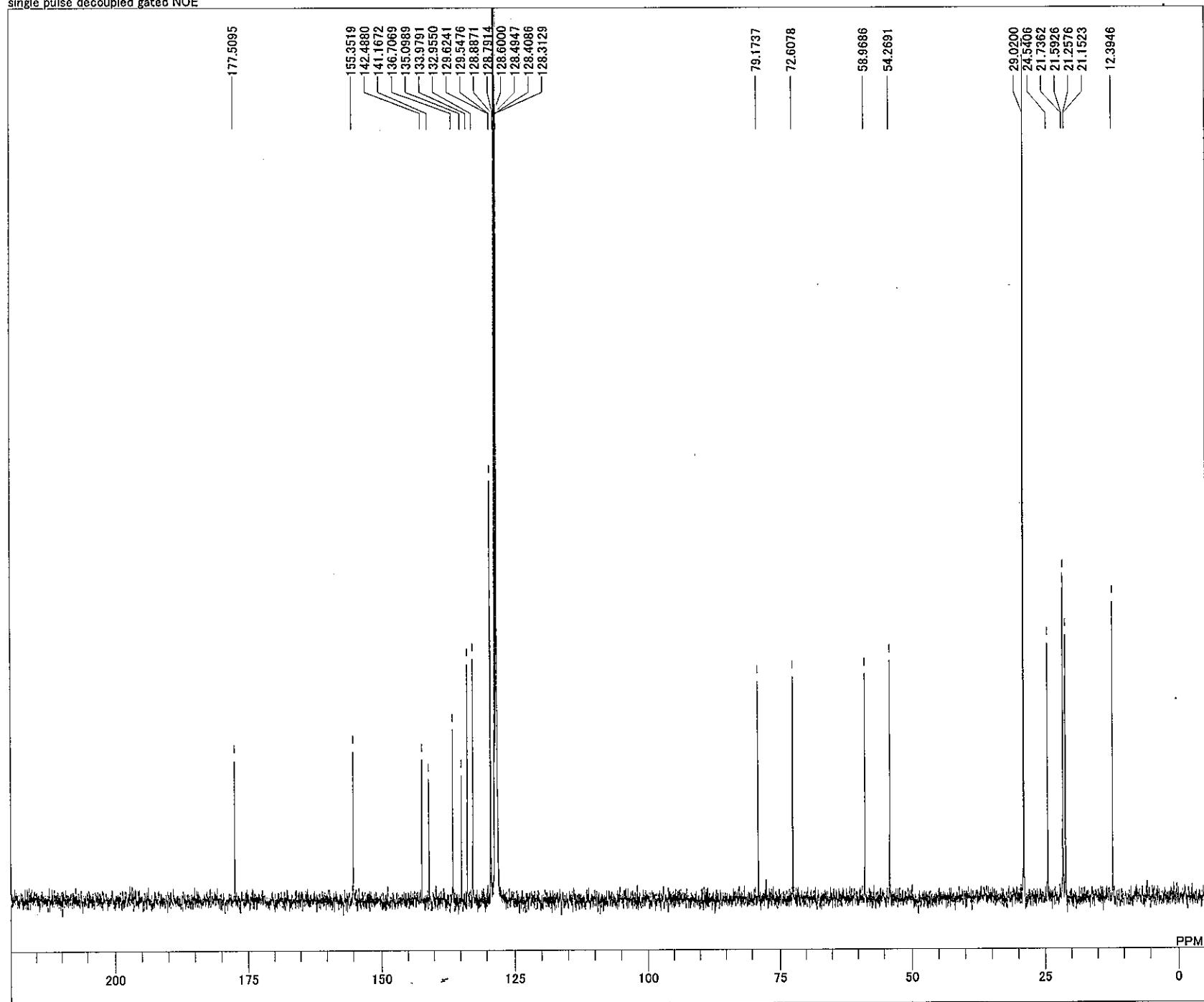


anti-7d

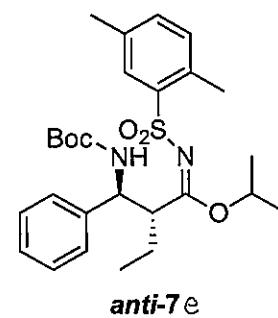


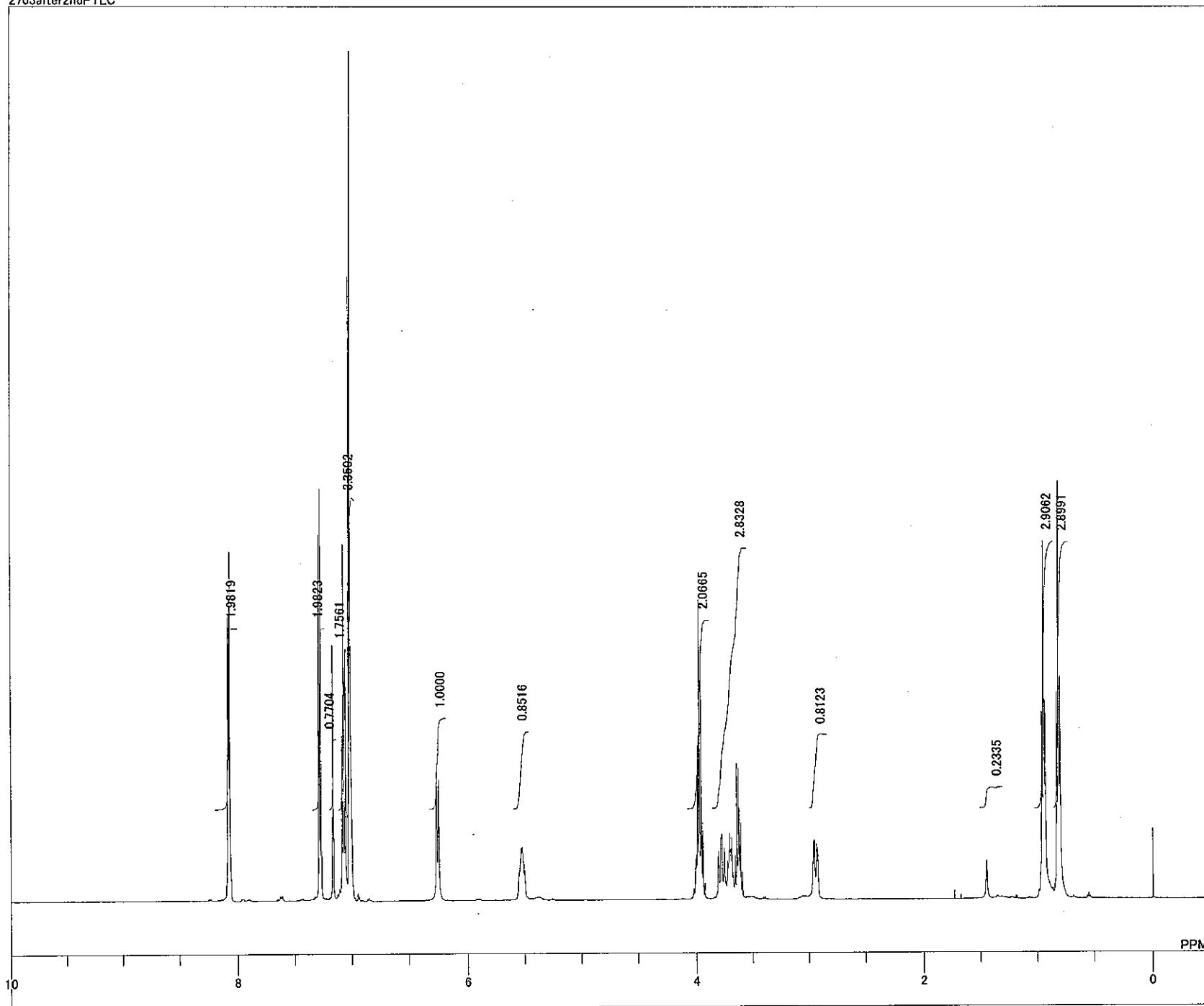
¥¥Eca¥data¥florian¥fb406-1.jdf
 DFILE
 COMNT
 DATIM 30-08-2007 21:09:25
 1H
 1H NMR.ex2
 495.13 MHz
 4.38 KHz
 9.64 Hz
 16400
 9286.78 Hz
 8
 1.7642 sec
 5.0000 sec
 6.50 usec
 1H 27.6 c
 C6D6 0.00 ppm
 0.12 Hz
 34



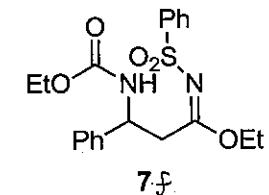


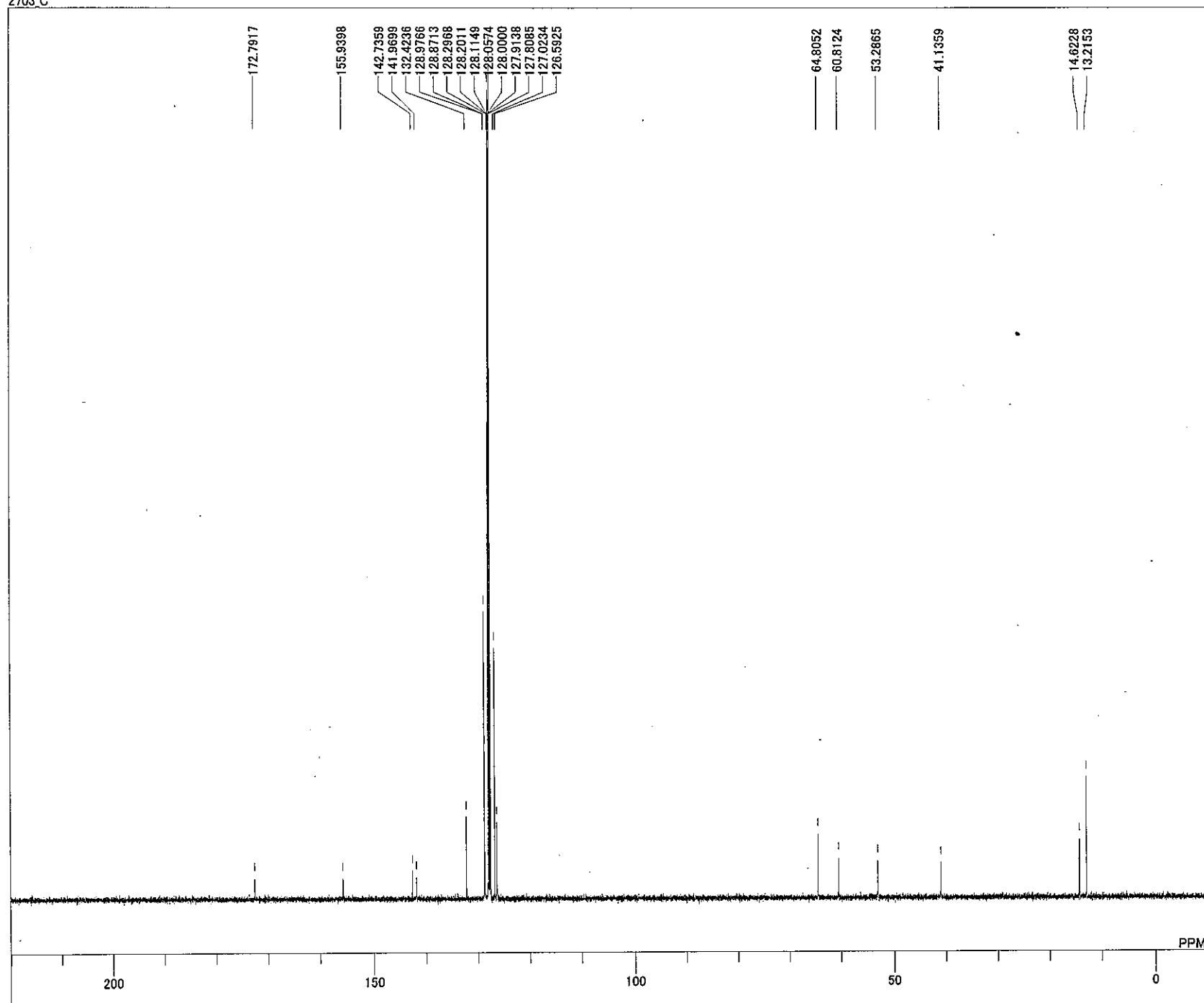
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\
single pulse decoupled gated NOE
01-07-2007 17:42:49
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
2.0000 sec
3.67 usec
1H
25.7 c
C6D6
128.60 ppm
1.20 Hz
52



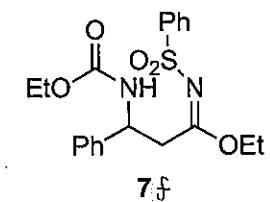


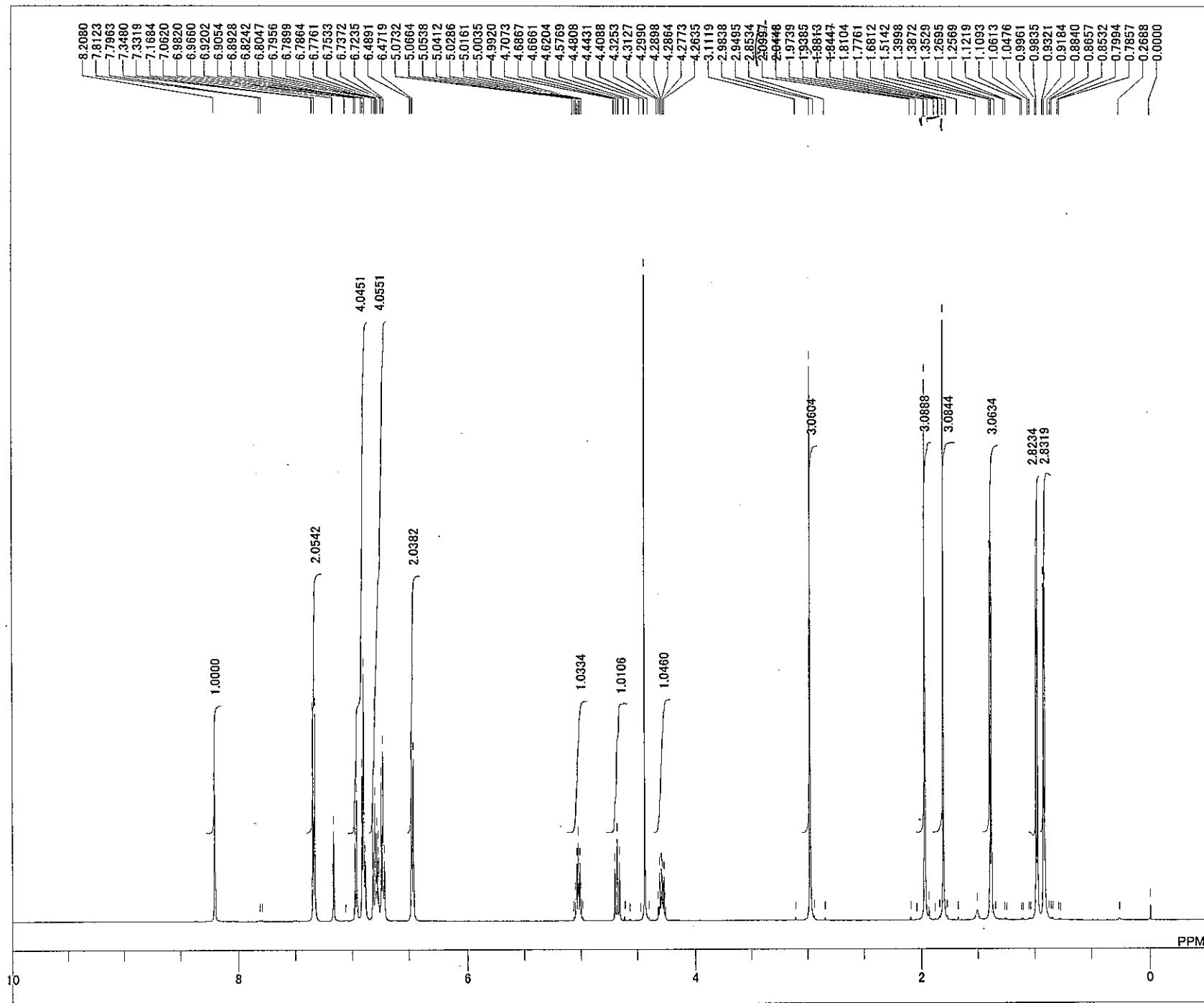
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2703after2ndPTLC
DATIM 10-07-2007 19:22:04
OBNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT C6D6
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28





DFILE C:\Documents and Settings\All Users\Docu
COMNT 2703_C
DATIM 10-07-2007 19:27:44
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 106
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 24.9 c
SLVNT C6D6
EXREF 128.00 ppm
BF 0.12 Hz
RGAIN 50

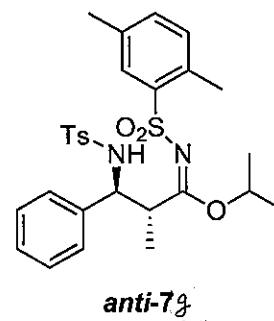


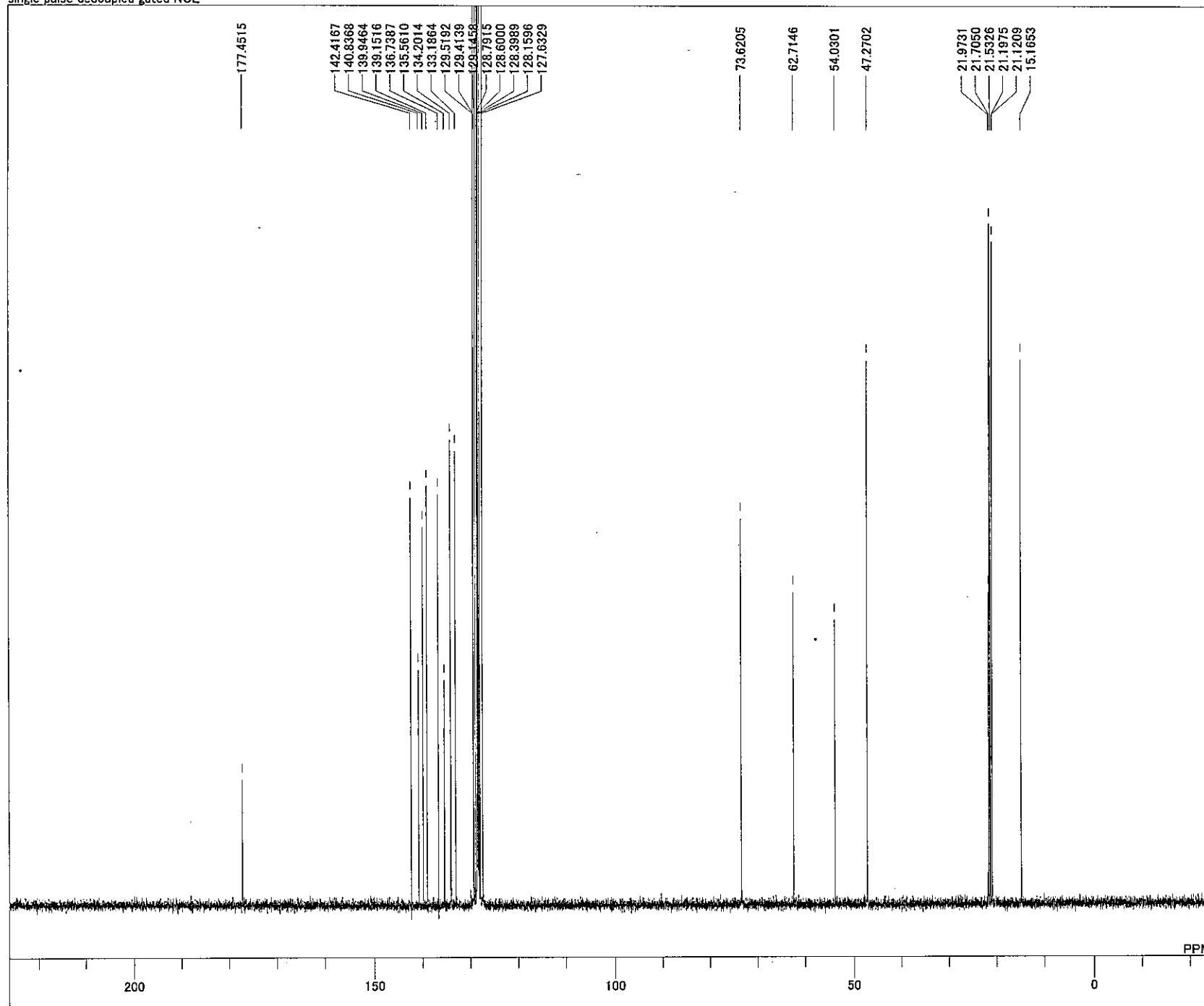


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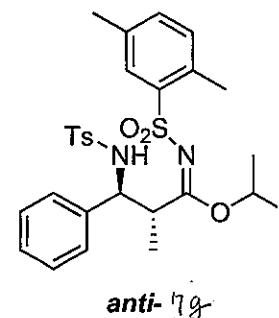
DFILE    ¥¥Eca¥data¥floria
COMNT
DATIM   24-08-2007 08:2
OBNUC   1H
EXMOD   1H NMR.ex2
OBFRQ   495.13 MHz
OBSET   4.38 kHz
OBFIN   9.64 Hz
POINT   16400
FREQU   9286.78 Hz
SCANS   8
ACQT M 1.7642 sec
PD      5.0000 sec
PW1     6.50 usec
IRNUC   1H
CTEMP   27.9 c
SLVNT   C6D6
EXREF   0.00 ppm
BF      0.12 Hz
RGAIN   26

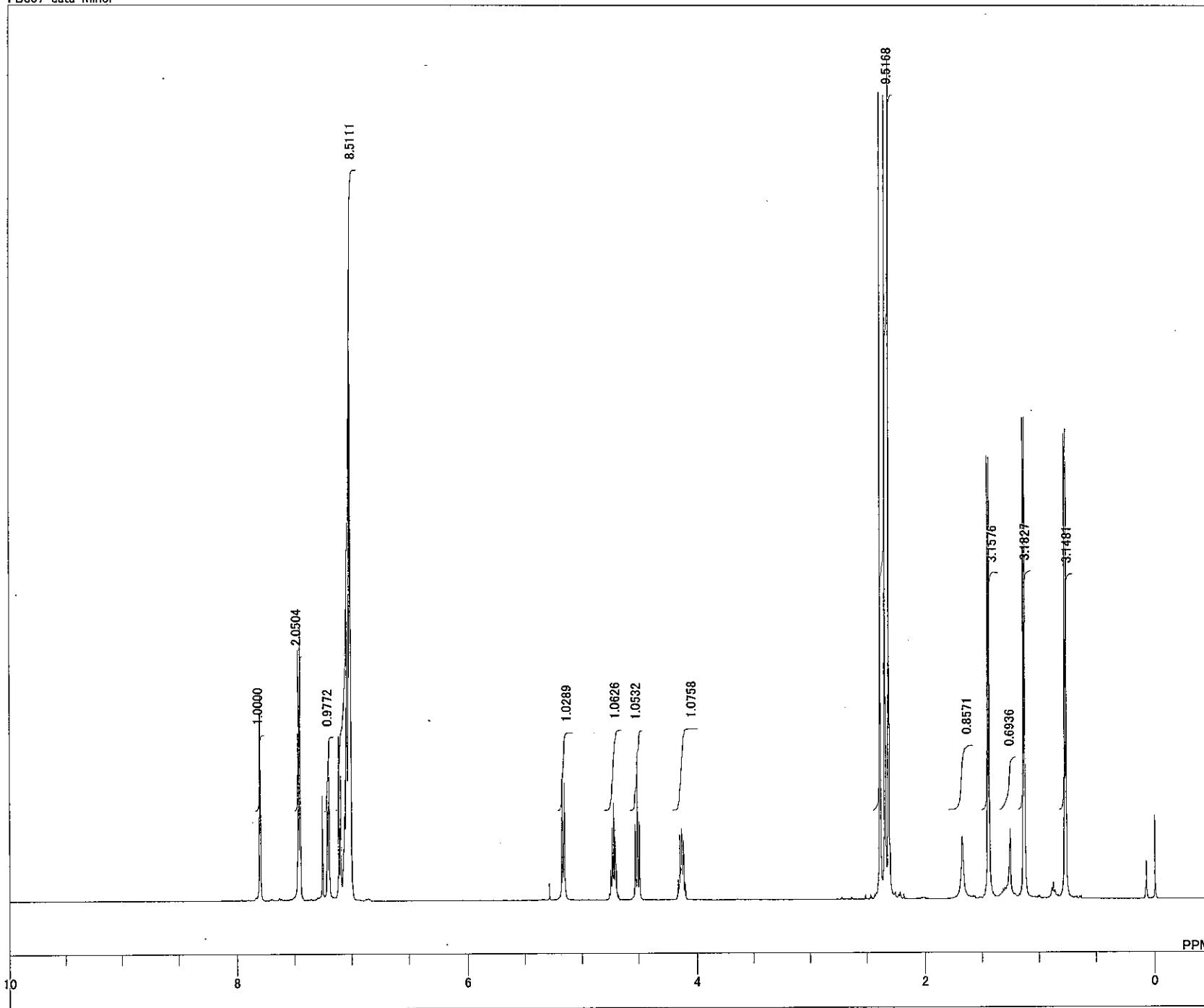
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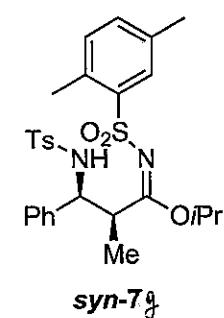


C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
24-08-2007 08:52:59
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26214
31249.52 Hz
442
0.8389 sec
PD
2.0000 sec
3.67 usec
PW1
IRNUC
28.5 c
CTEMP
C6D6
SLVNT
EXREF
BF
RGAIN
1H
128.60 ppm
0.12 Hz
52

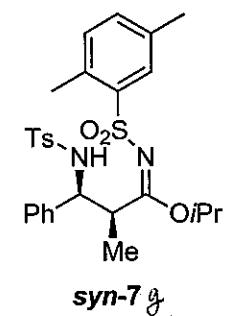
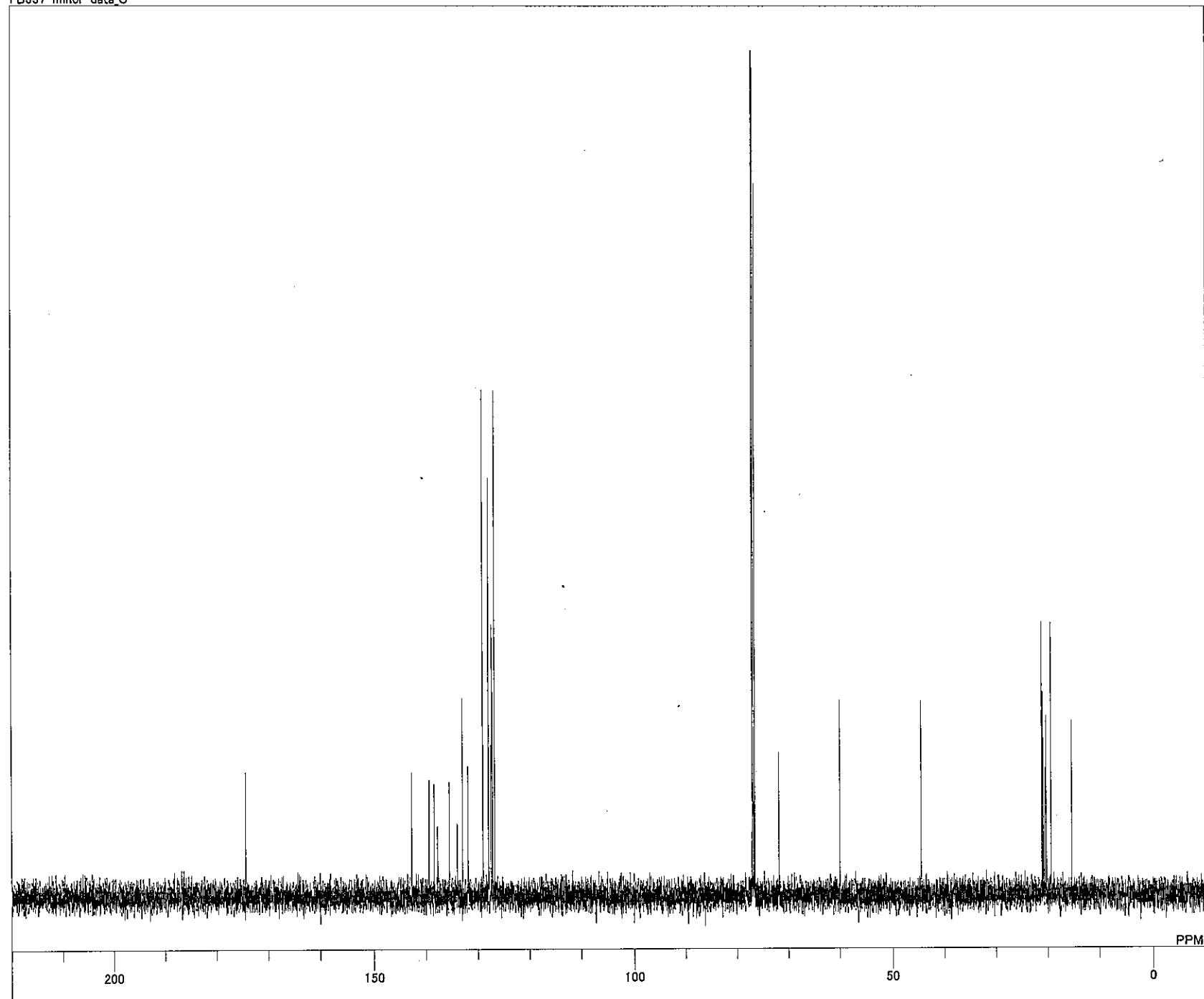


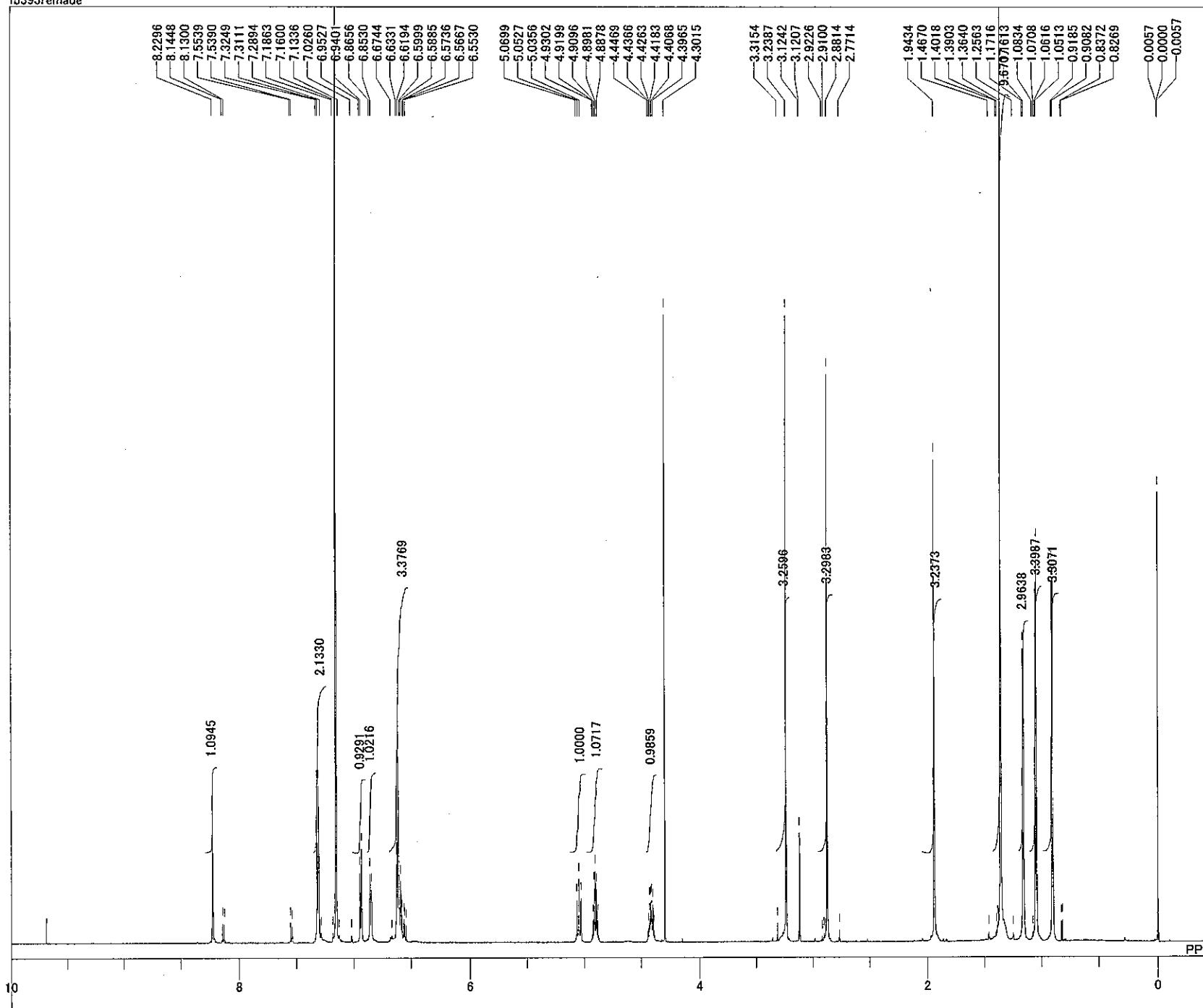


DFILE C:\Documents and Settings\All Users\Docu
COMNT FB357-data-minor
DATIM 11-08-2007 13:36:53
OBNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 29.7 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38

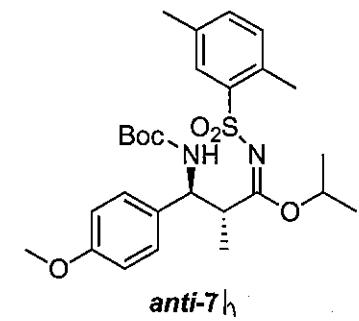


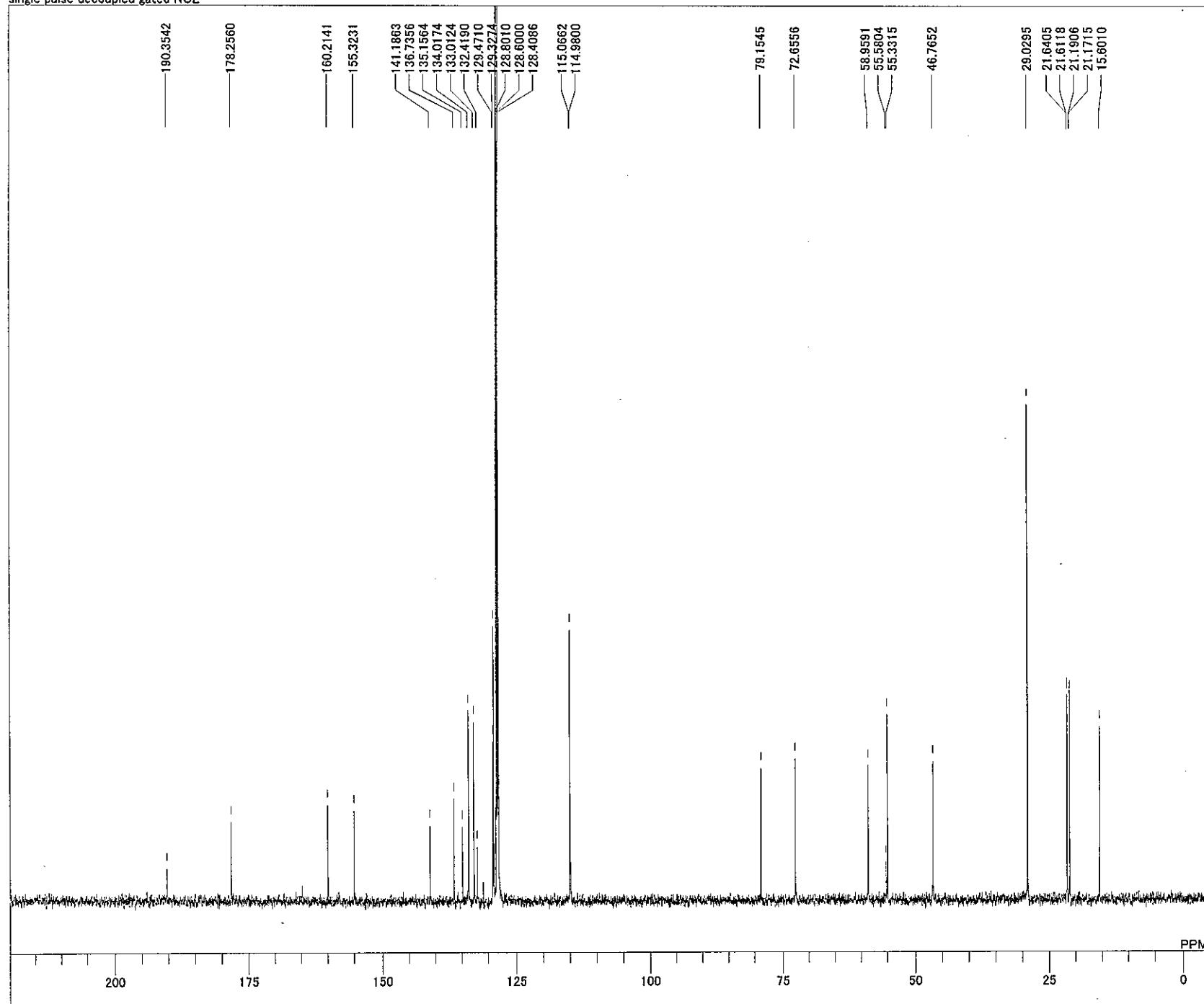
syn-7g



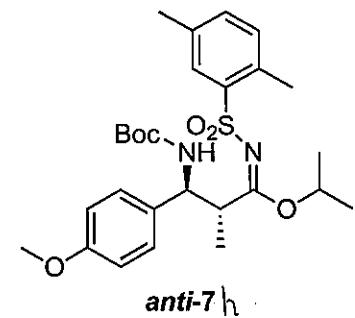


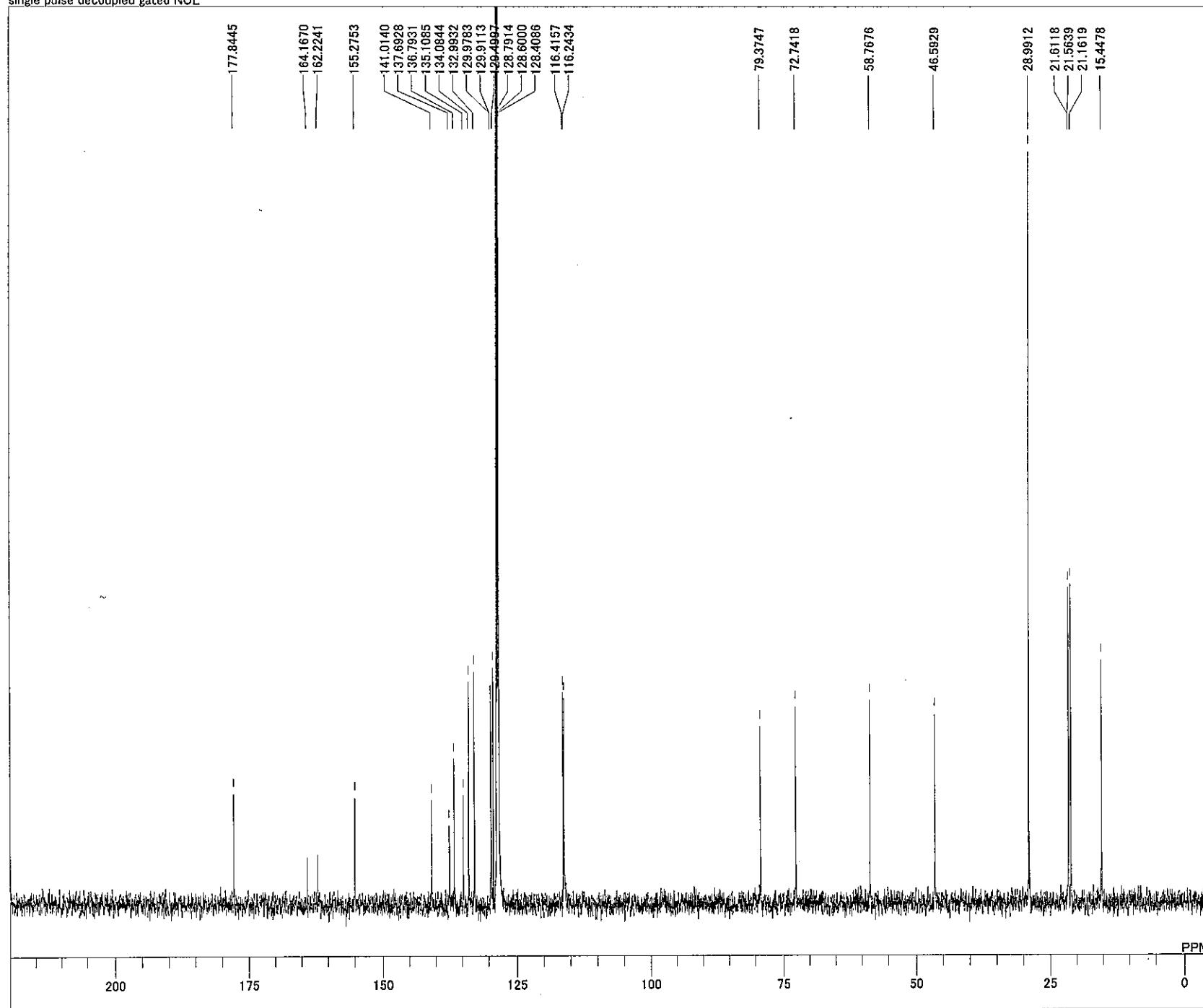
C:\Documents and Settings\ALICE2\デスク
fb393remade
28-08-2007 21:19:46
1H
single_pulse.ex2
600.17 MHz
5.30 kHz
5.47 Hz
20480
14076.79 Hz
16
1.4549 sec
4.0000 sec
9.10 usec
1H
29.4 c
C6D6
0.00 ppm
0.12 Hz
52



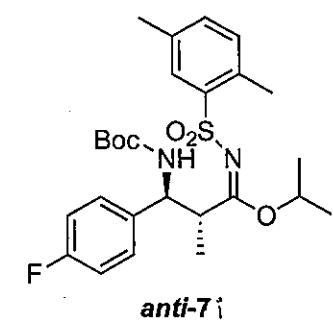


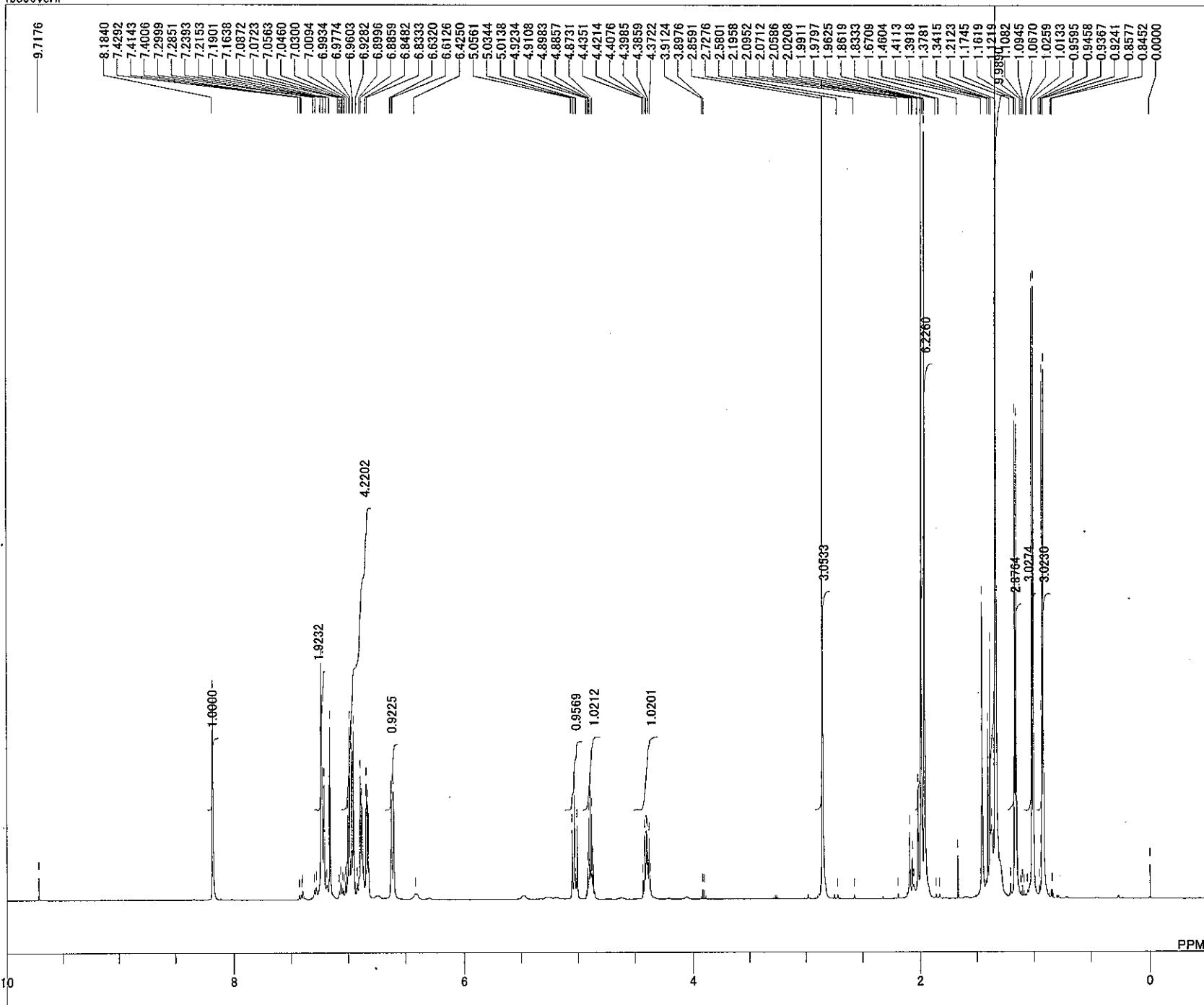
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
01-07-2007 16:57:05
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26224
31249.52 Hz
62
0.8388 sec
2.0000 sec
3.67 usec
1H
25.5 c
C6D6
128.60 ppm
1.20 Hz
52



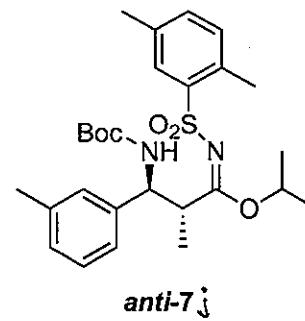


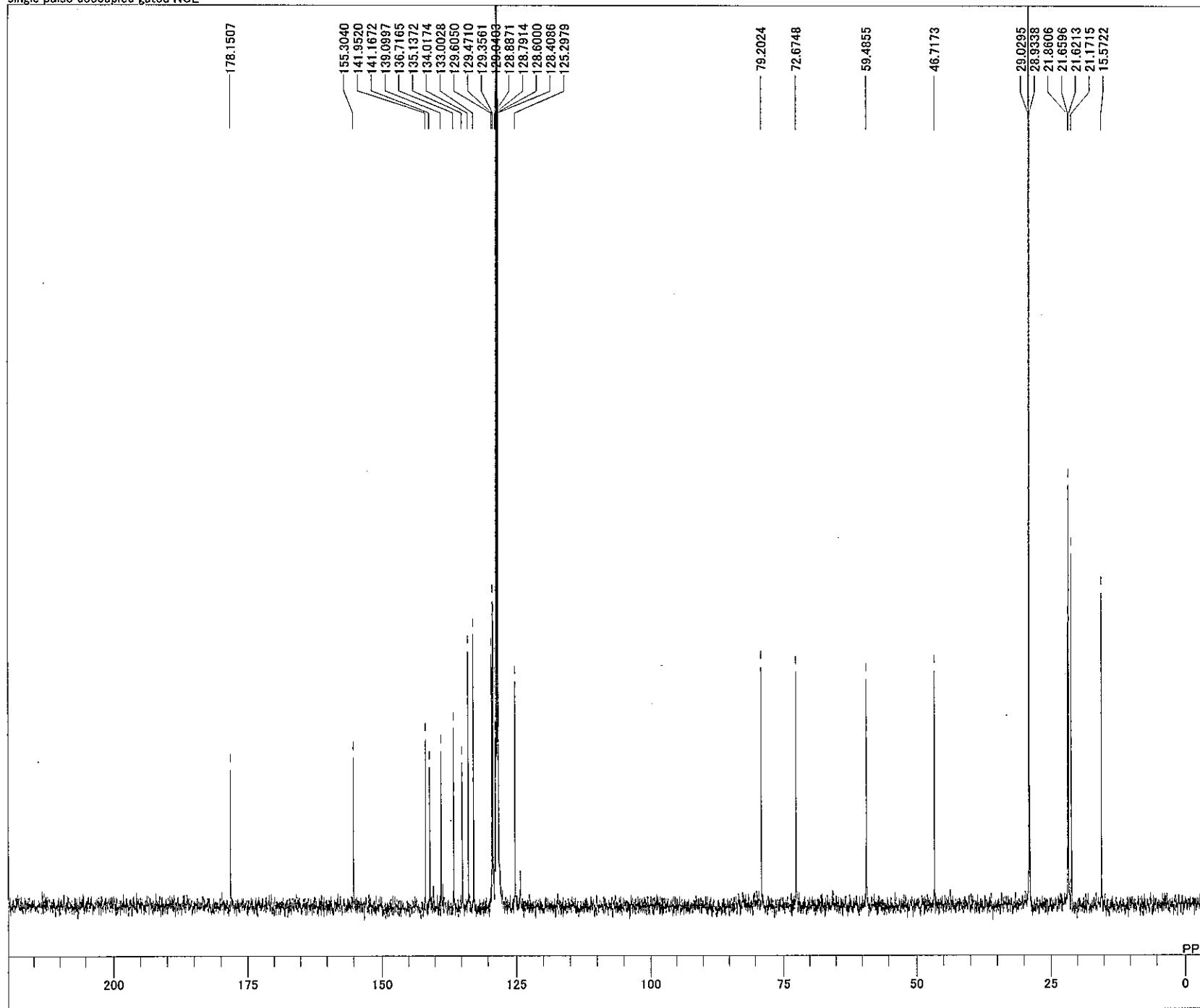
DFILE C:\Documents and Settings\All Users\Docu
COMNT single pulse decoupled gated NOE
DATIM 01-07-2007 16:35:07
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.48 KHz
OBFIN 6.00 Hz
POINT 26224
FREQU 31249.52 Hz
SCANS 62
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT C6D6
EXREF 128.60 ppm
BF 1.20 Hz
RGAIN 54



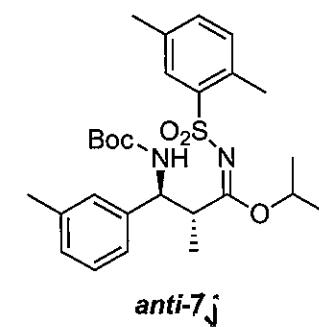


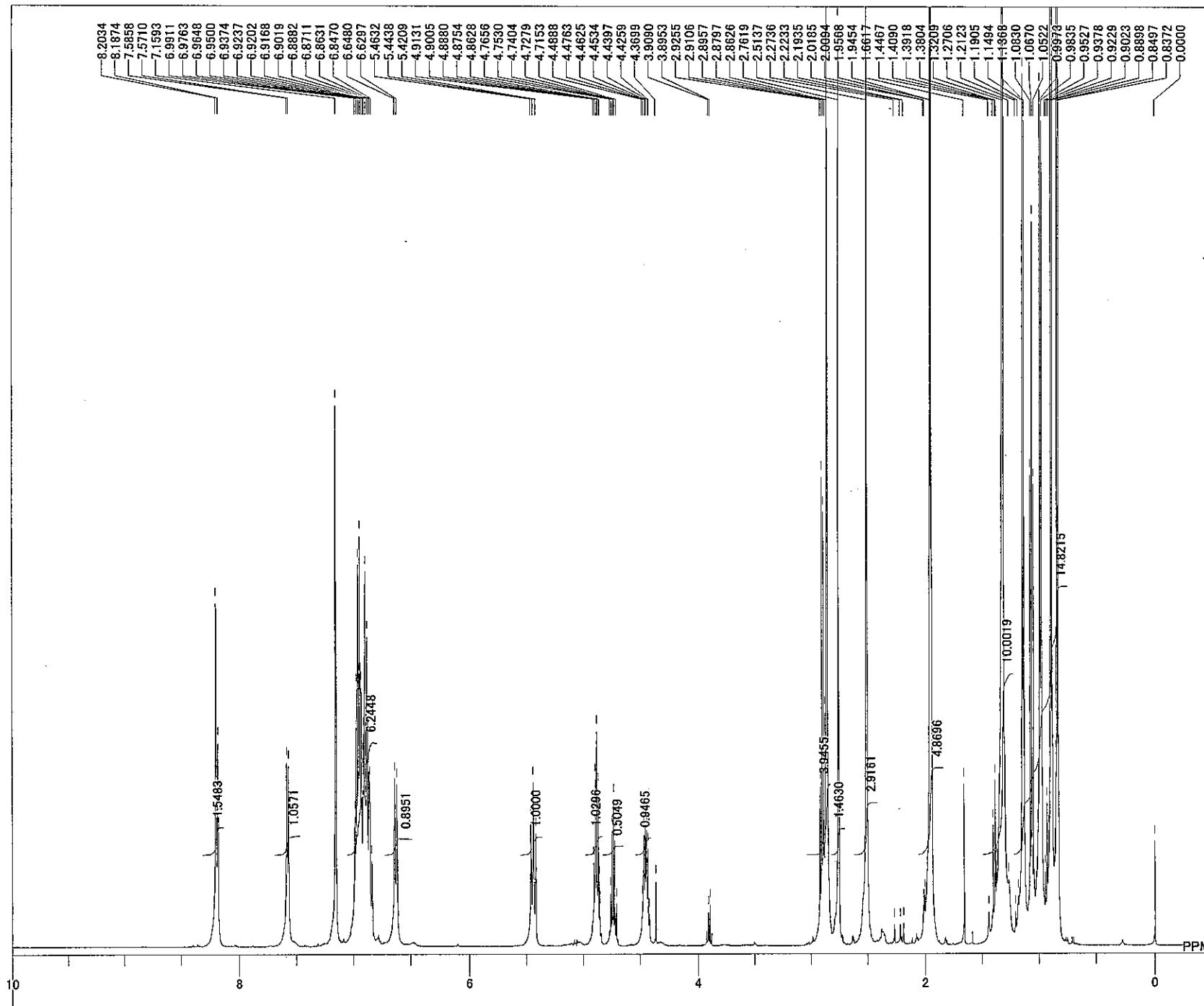
¥Eca¥data¥florian¥fb396verif-1.jdf
fb396verif
02-07-2007 18:23:42
1H
1H NMR.ex2
495.13 MHz
4.38 kHz
9.64 Hz
16400
9286.78 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
25.7 c
C6D6
0.00 ppm
0.12 Hz
22



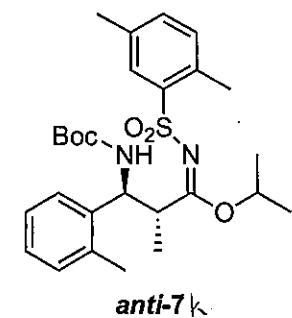


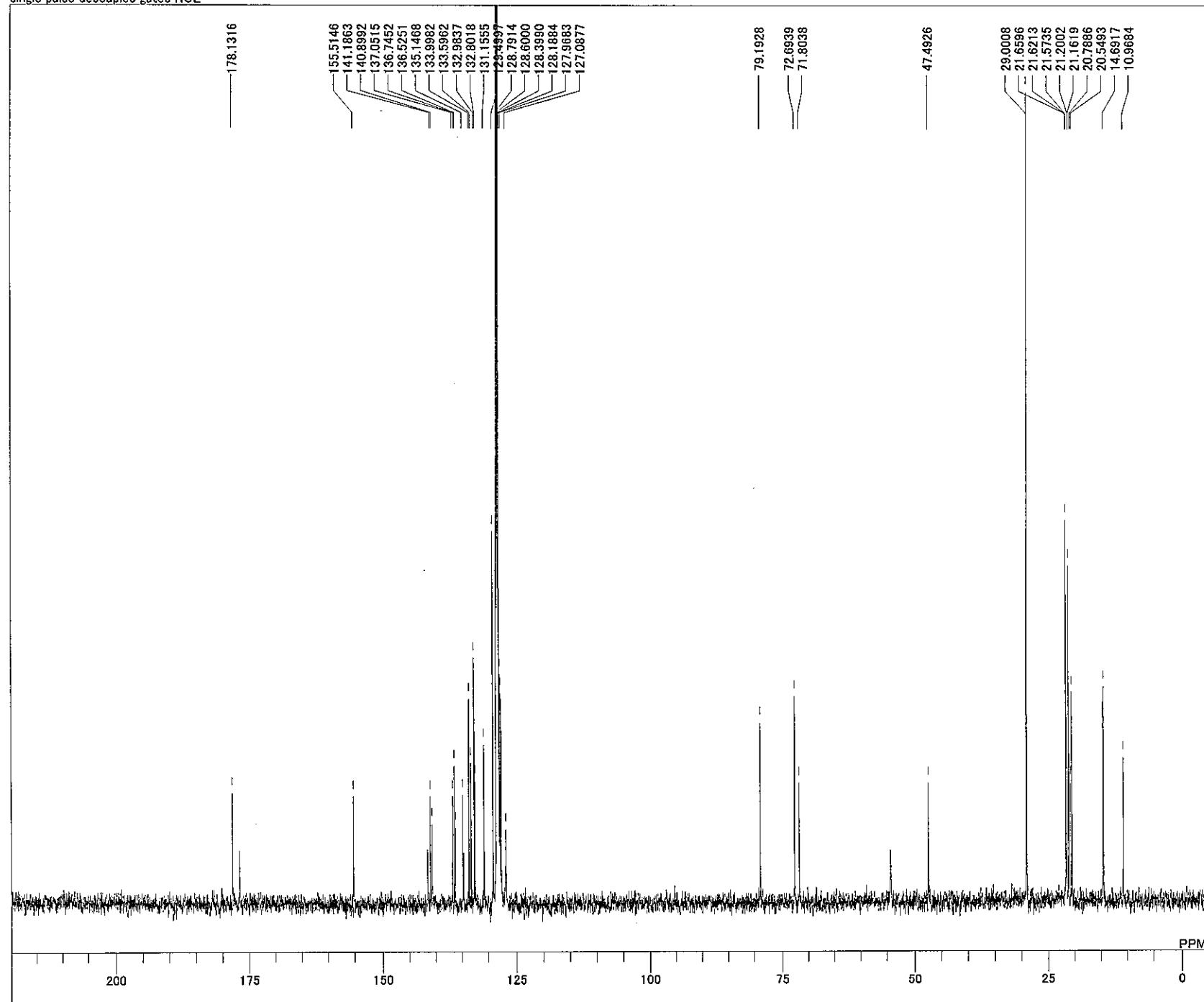
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
02-07-2007 18:28:36
13C
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
2.0000 sec
3.67 usec
1H
26.0 c
C6D6
128.60 ppm
0.12 Hz
50





¥¥Eca¥data¥florian¥fb394finalptlcdown-1.jdf
 30-07-2007 22:06:24
 1H
 1H NMR.ex2
 495.13 MHz
 4.38 KHz
 9.64 Hz
 16400
 9286.78 Hz
 8
 1.7642 sec
 5.0000 sec
 6.50 usec
 1H
 26.3 c
 C6D6
 0.00 ppm
 0.12 Hz
 28

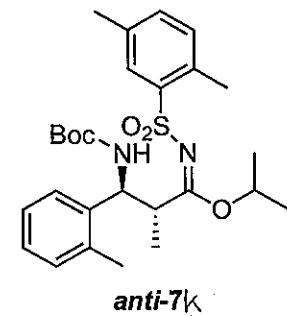


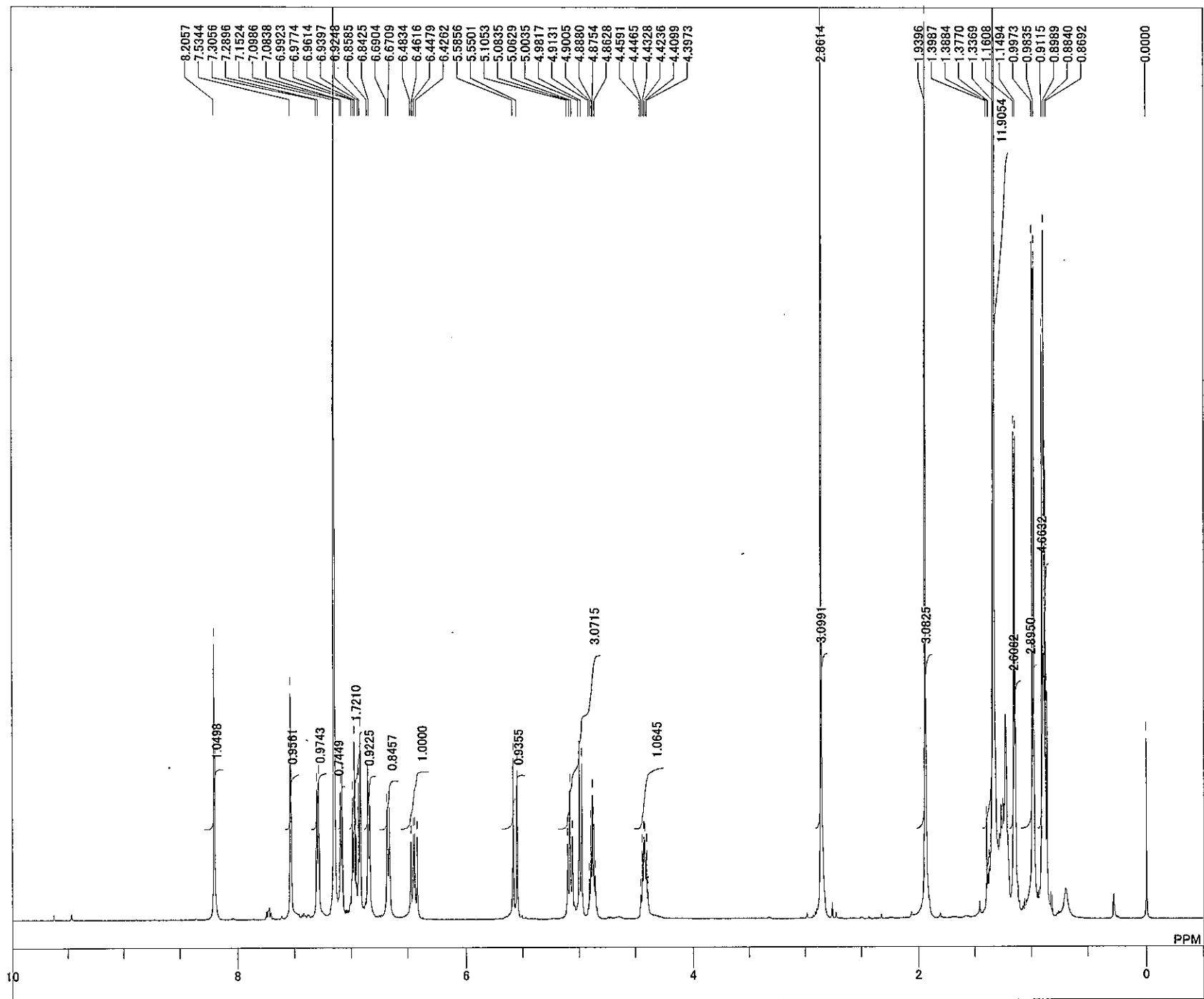


DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

single pulse decoupled gated NOE
30-07-2007 22:16:51
13C
¹³C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
139
0.8389 sec
2.0000 sec
3.87 usec

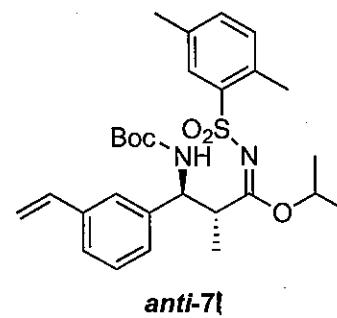
¹H
26.7 c
C6D6
128.80 ppm
0.12 Hz
50

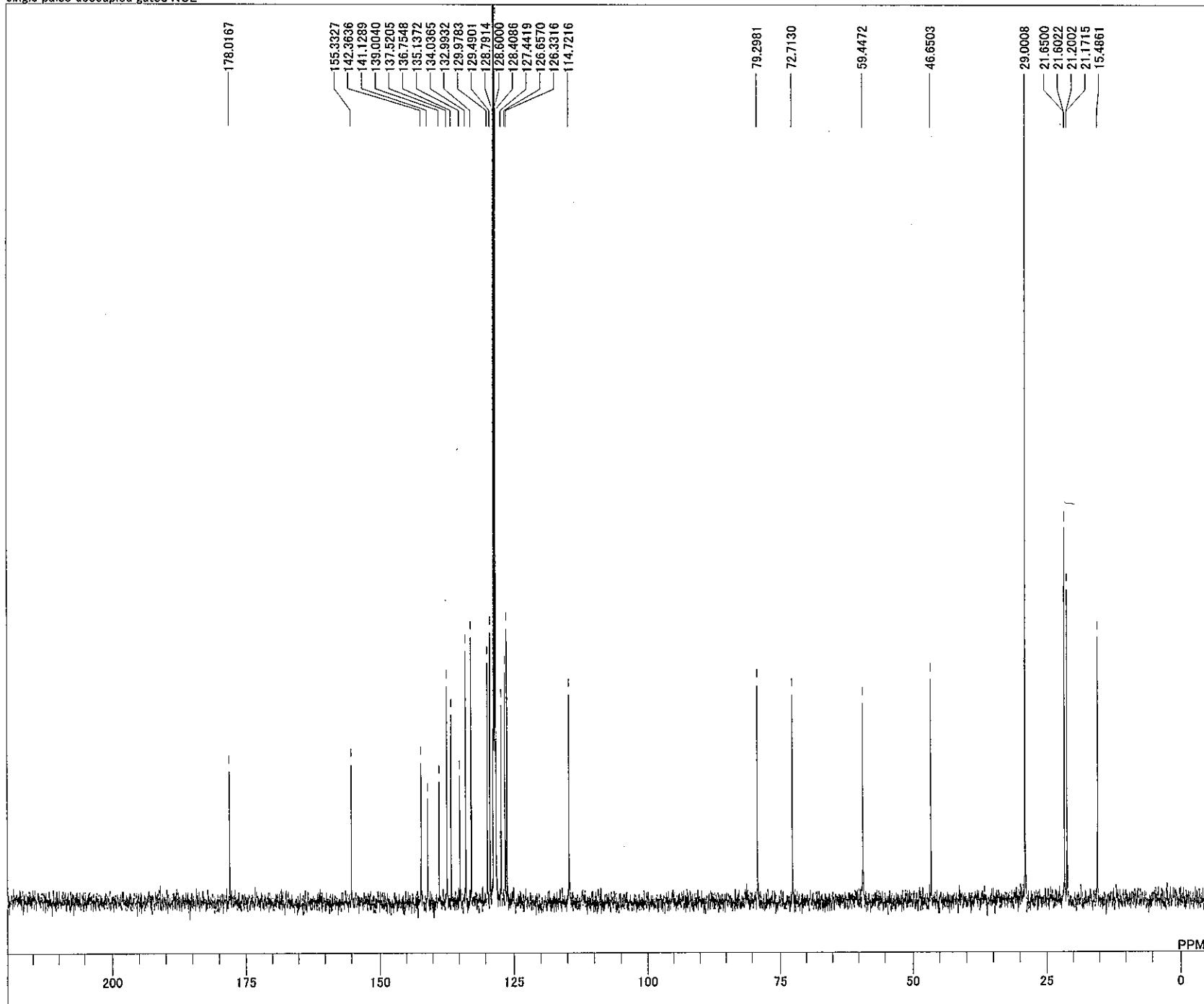




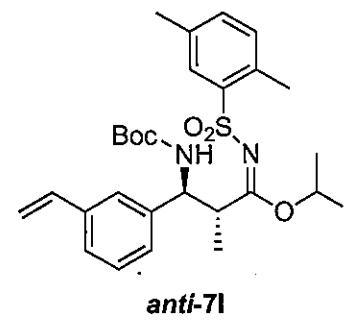
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

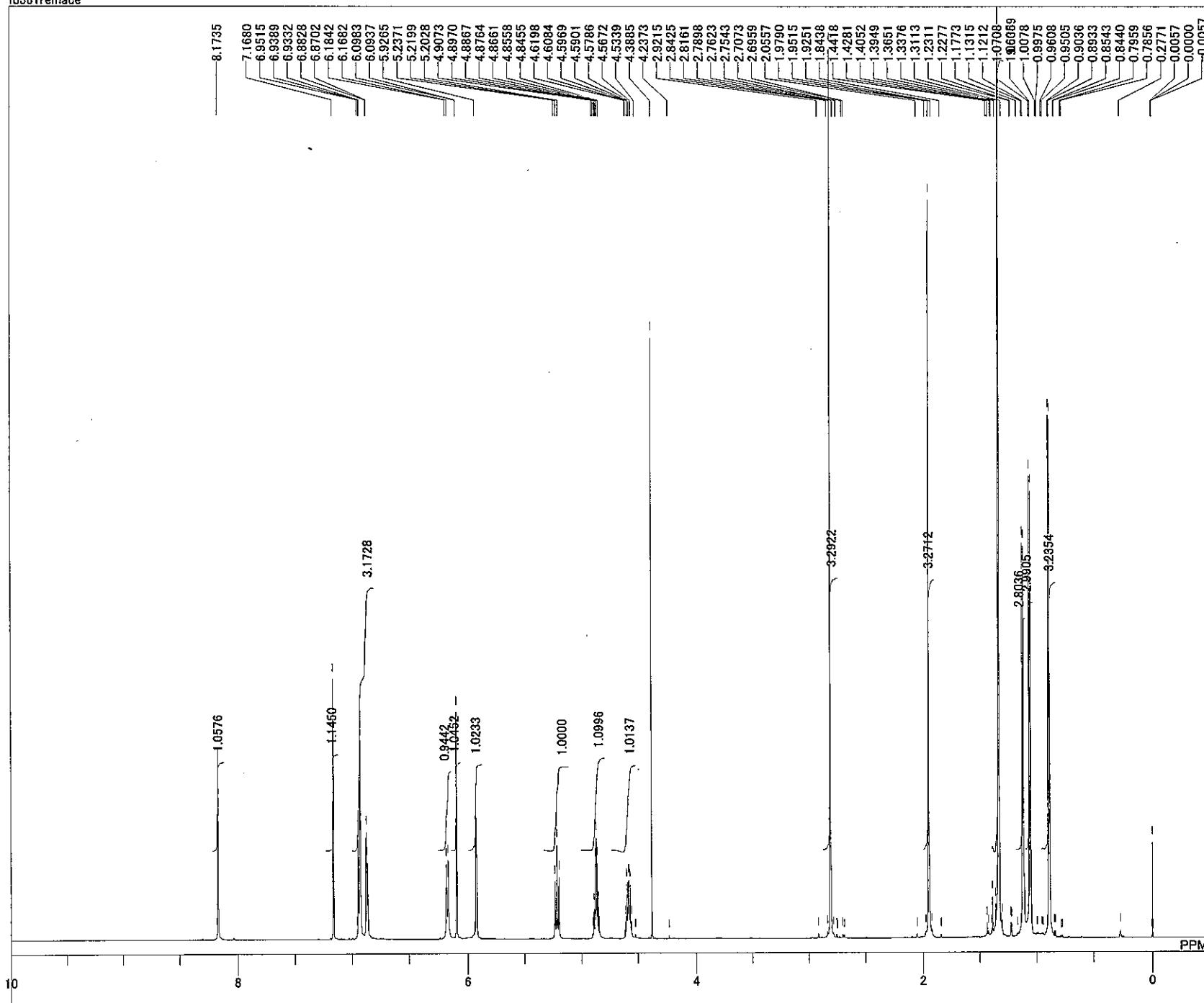
Ecadata\florian\fb383remade-3.jdf
29-08-2007 17:31:47
1H
1H NMR.ex2
495.13 MHz
4.38 kHz
9.64 Hz
16400
9286.78 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
28.4 c
C6D6
0.00 ppm
0.12 Hz
36



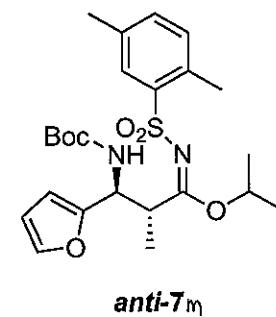


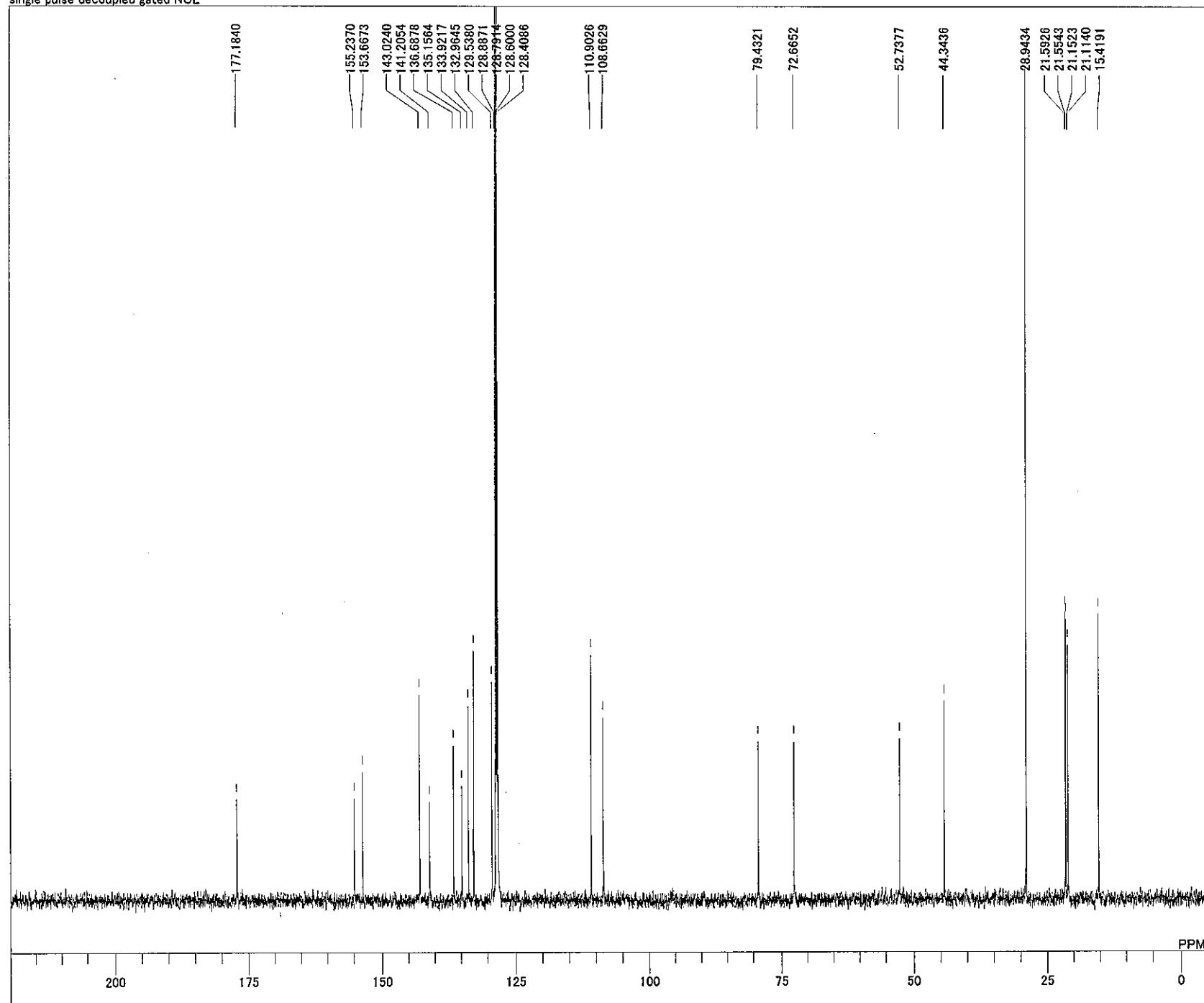
DFILE C:\Documents and Settings\All Users\Docu
COMNT single pulse decoupled gated NOE
DATIM 01-07-2007 17:07:14
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 kHz
OBFIN 6.00 Hz
POINT 26224
FREQU 31249.52 Hz
SCANS 65
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
1H 25.6 c
C6D6 128.60 ppm
BF 1.20 Hz
RGAIN 54



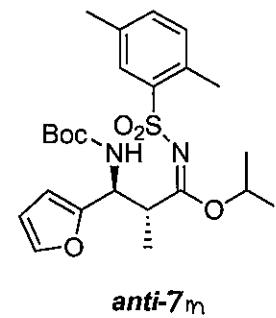


C:\Documents and Settings\ALICE2\デスク
fb381remade
28-08-2007 21:39:43
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
20480
14076.79 Hz
16
1.4549 sec
4.0000 sec
9.10 usec
1H
29.4 c
C6D6
0.00 ppm
0.12 Hz
32

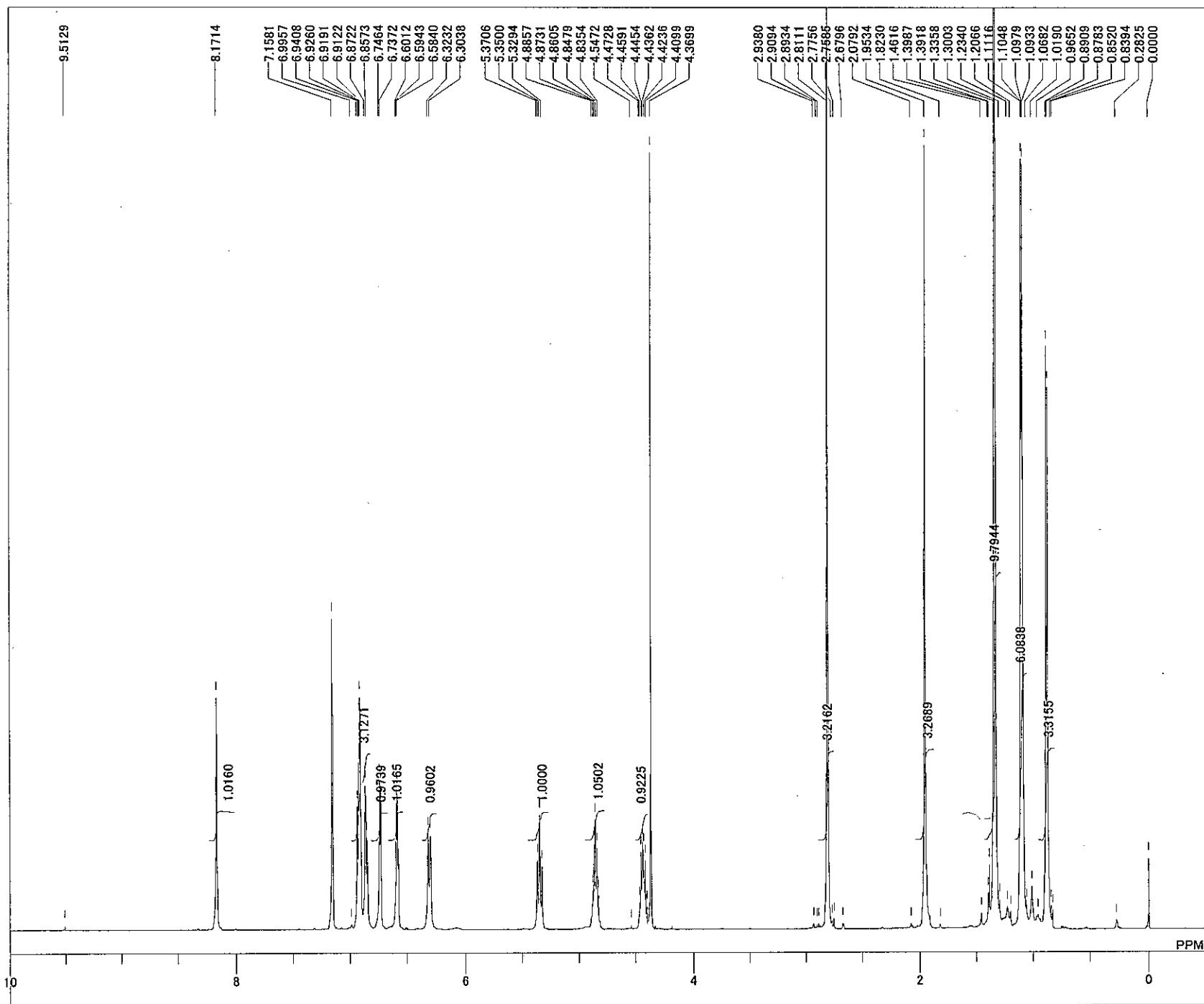




C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
01-07-2007 16:46:07
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
2.0000 sec
3.67 usec
1H
25.5 c
C6D6
128.60 ppm
1.20 Hz
54

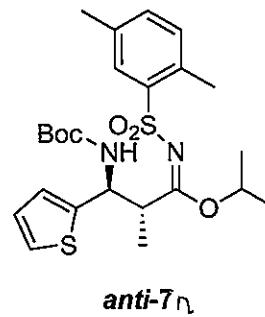


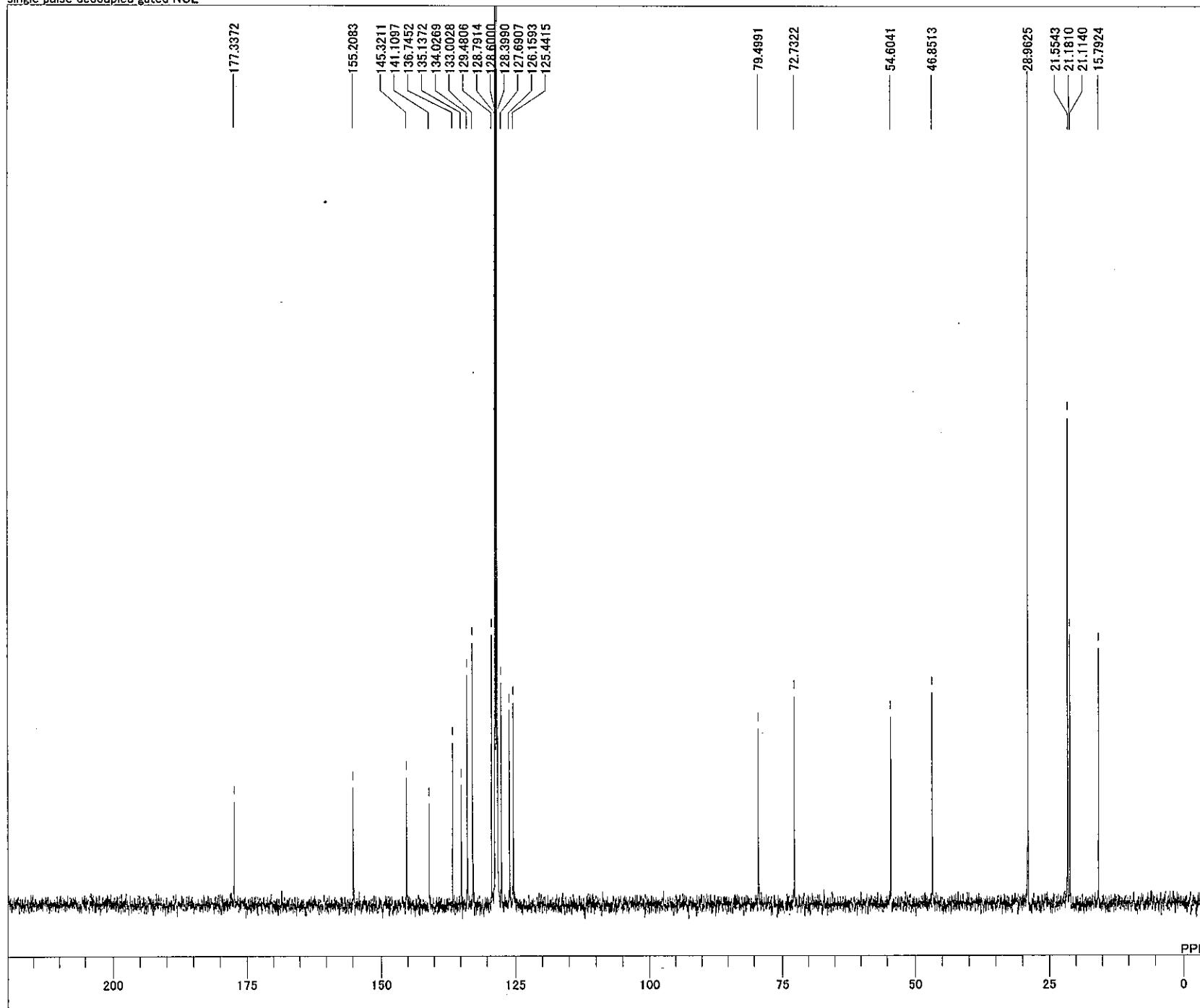
anti-7m



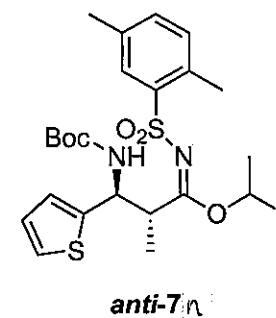
DFILE
COMM_T
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

29-08-2007 20:51:07
1H
1H NMR.ex2
495.13 MHz
4.38 KHz
9.64 Hz
16400
9286.78 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
28.2 c
C6D6
0.00 ppm
0.12 Hz
30

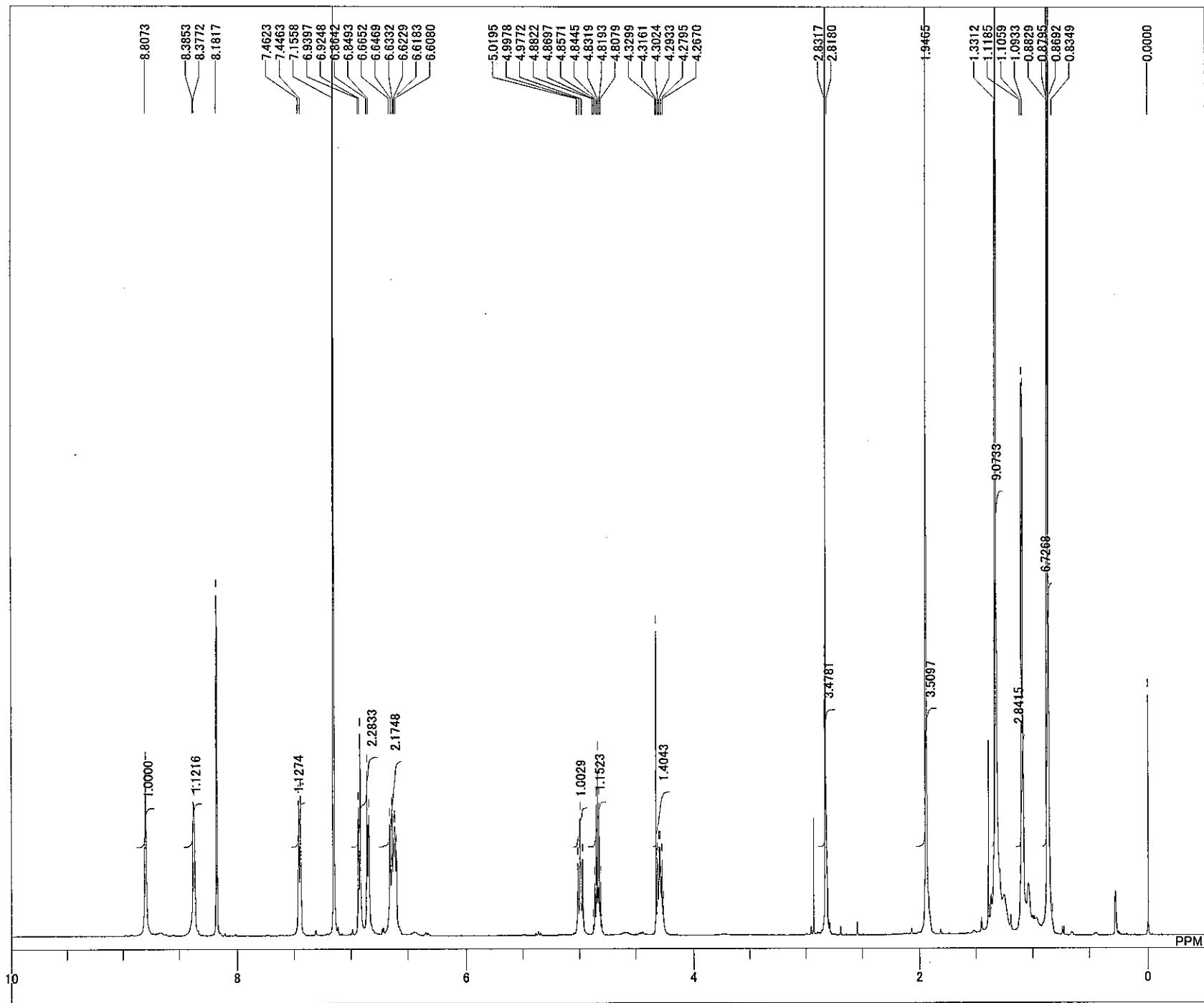




DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
01-07-2007 17:22:27
13C
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
64
0.8389 sec
2.0000 sec
3.67 usec
1H
25.6 c
C6D6
128.60 ppm
1.20 Hz
52



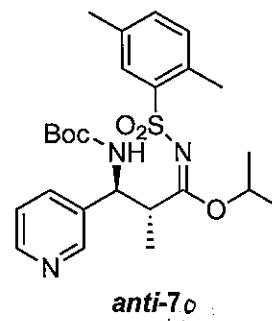
anti-7n

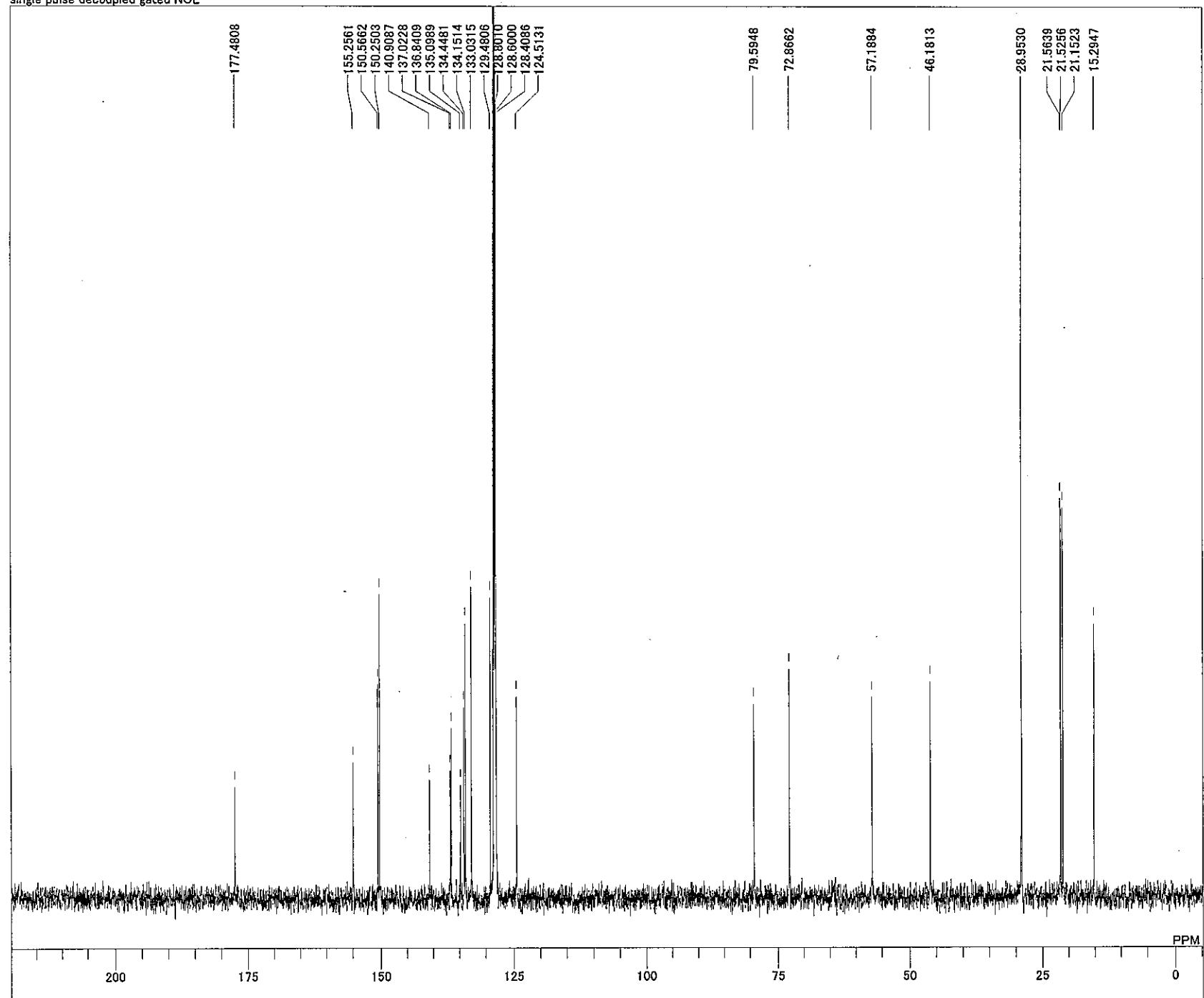


FILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

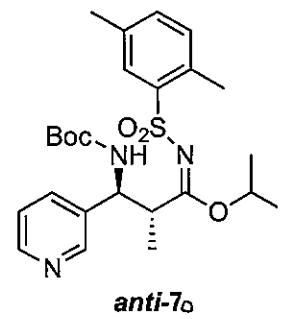
1H NMR.ex2
495.13 MHz
4.38 kHz
9.64 Hz
16400
9286.78 Hz
8
1.7642 sec
5.0000 sec
6.50 usec

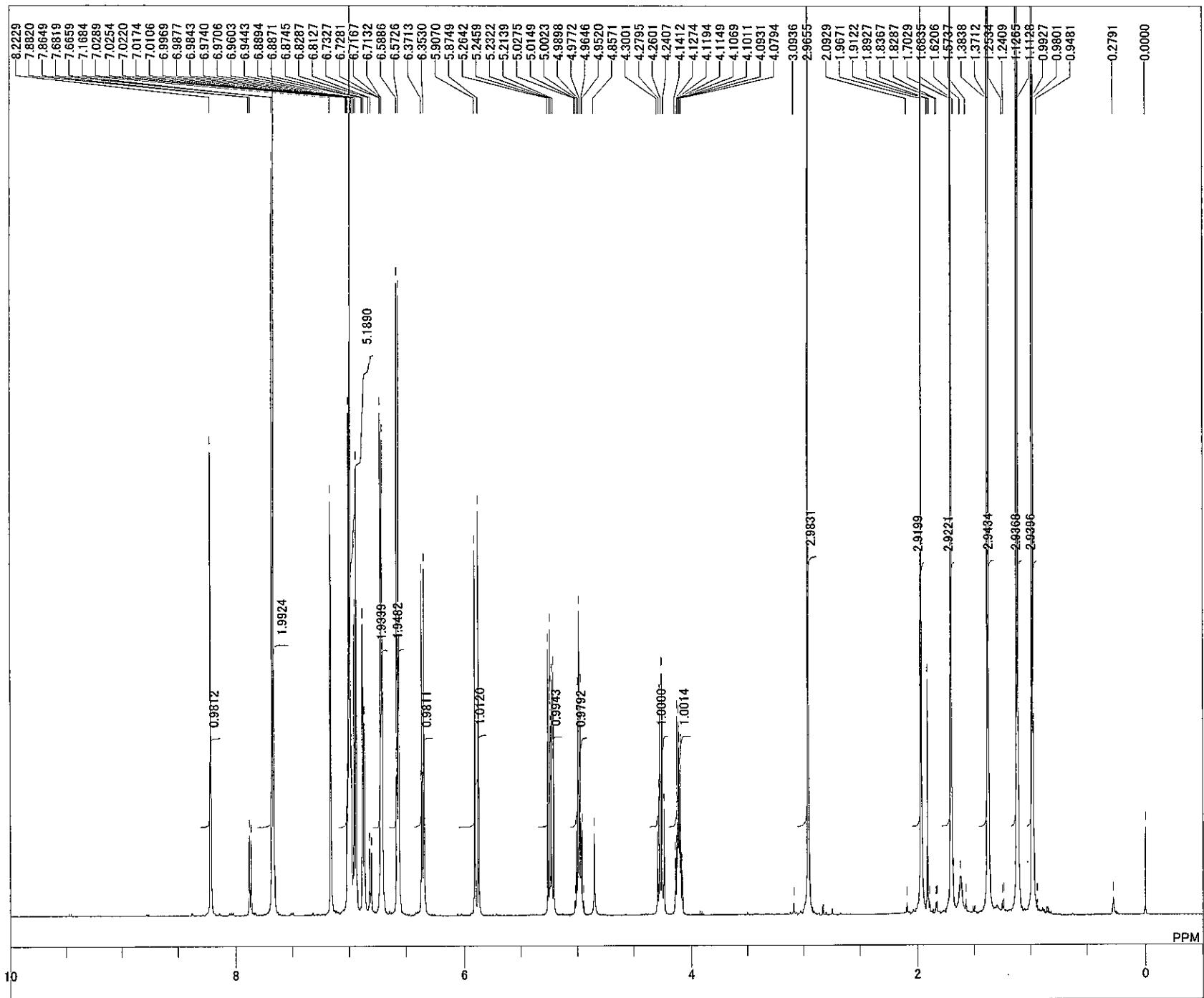
1H
28.5 c
C6D6
0.00 ppm
0.12 Hz
36





C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
01-07-2007 17:32:33
13C
13C NMR.ex2
124.51 MHz
3.45 KHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
2.0000 sec
3.67 usec
1H
25.6 c
C6D6
128.60 ppm
1.20 Hz
54

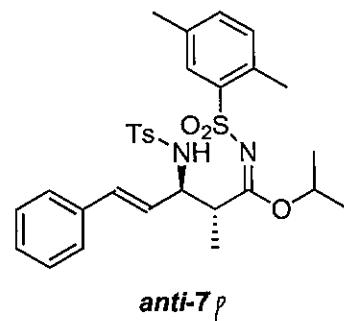


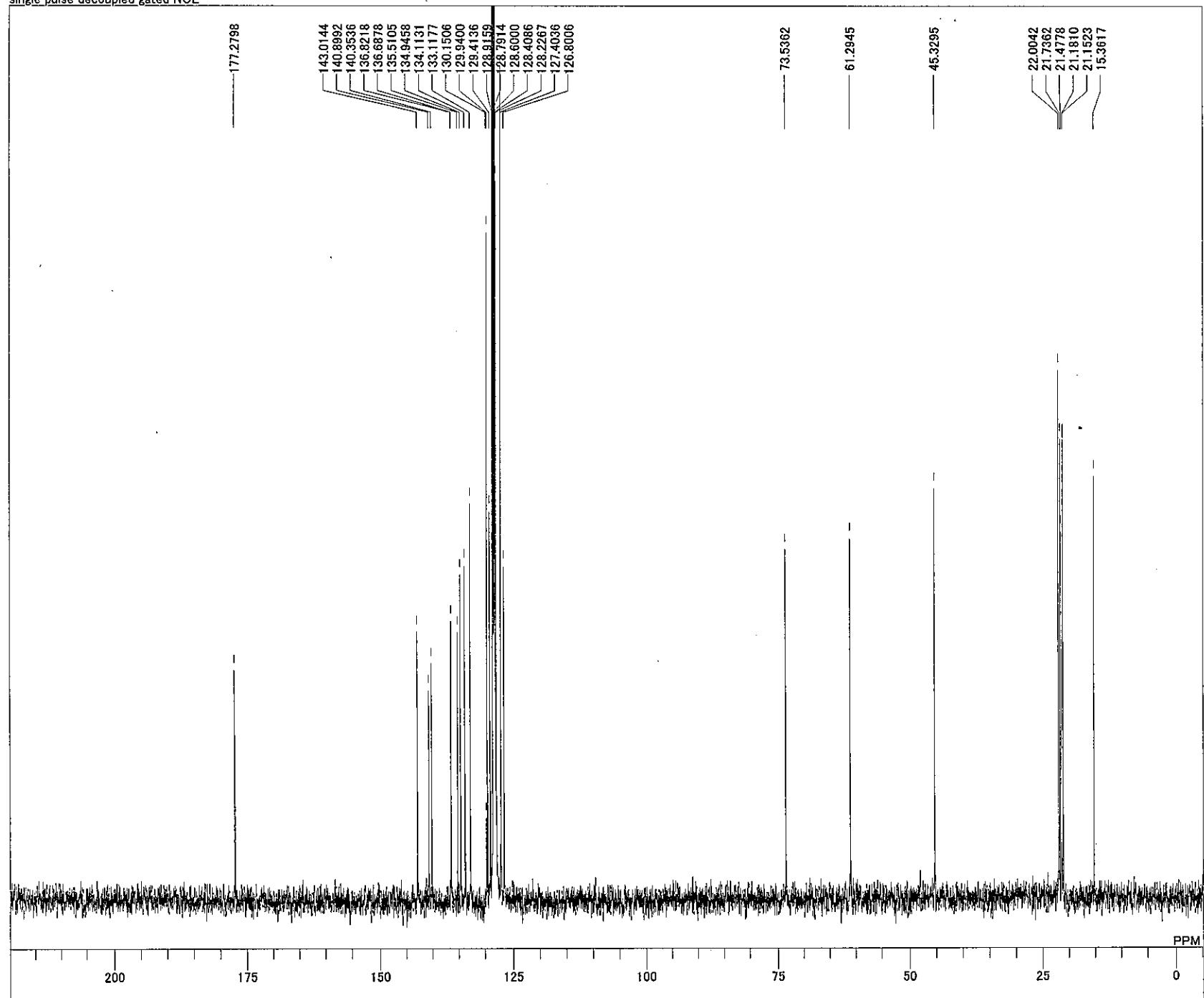


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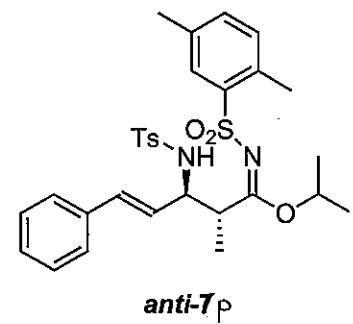
DFILE      ¥¥Eca¥data¥florian¥fb442prana-1.jdf
COMNT
DATIM    08-08-2007 20:29:43
OBNUC    1H
EXMOD   1H NMR.ex2
OBFRQ   49.13 MHz
OBSET   4.38 KHz
OBFIN   9.64 Hz
POINT   16400
FREQU   9286.78 Hz
SCANS    8
ACQTM   1.7642 sec
PD      5.0000 sec
PW1     6.50 usec
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
1H      27.9 c
C6D6   0.00 ppm
          0.12 Hz
          26

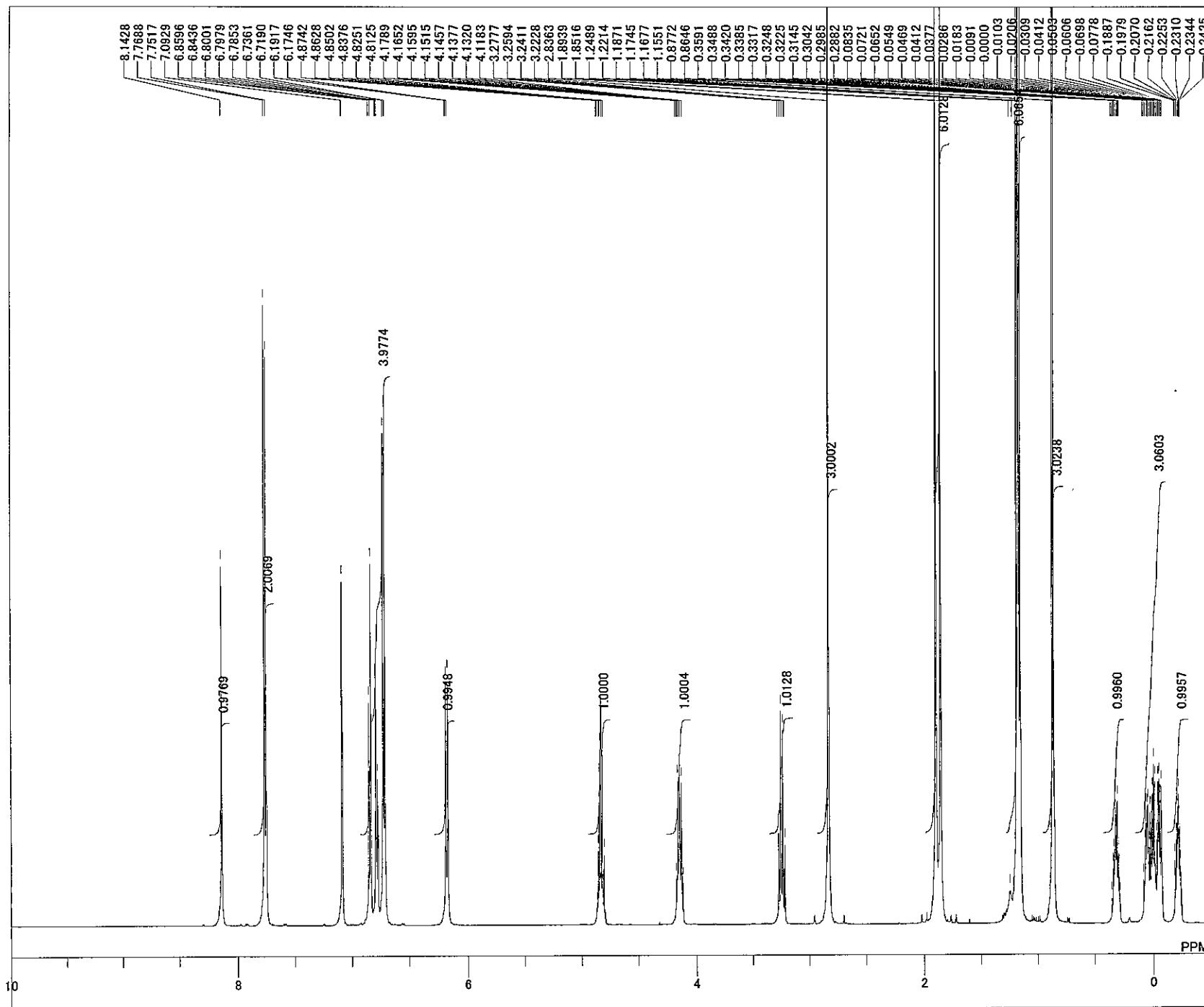
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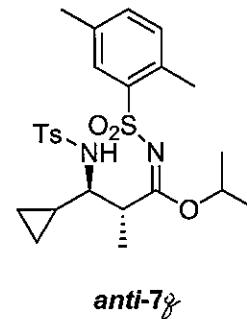


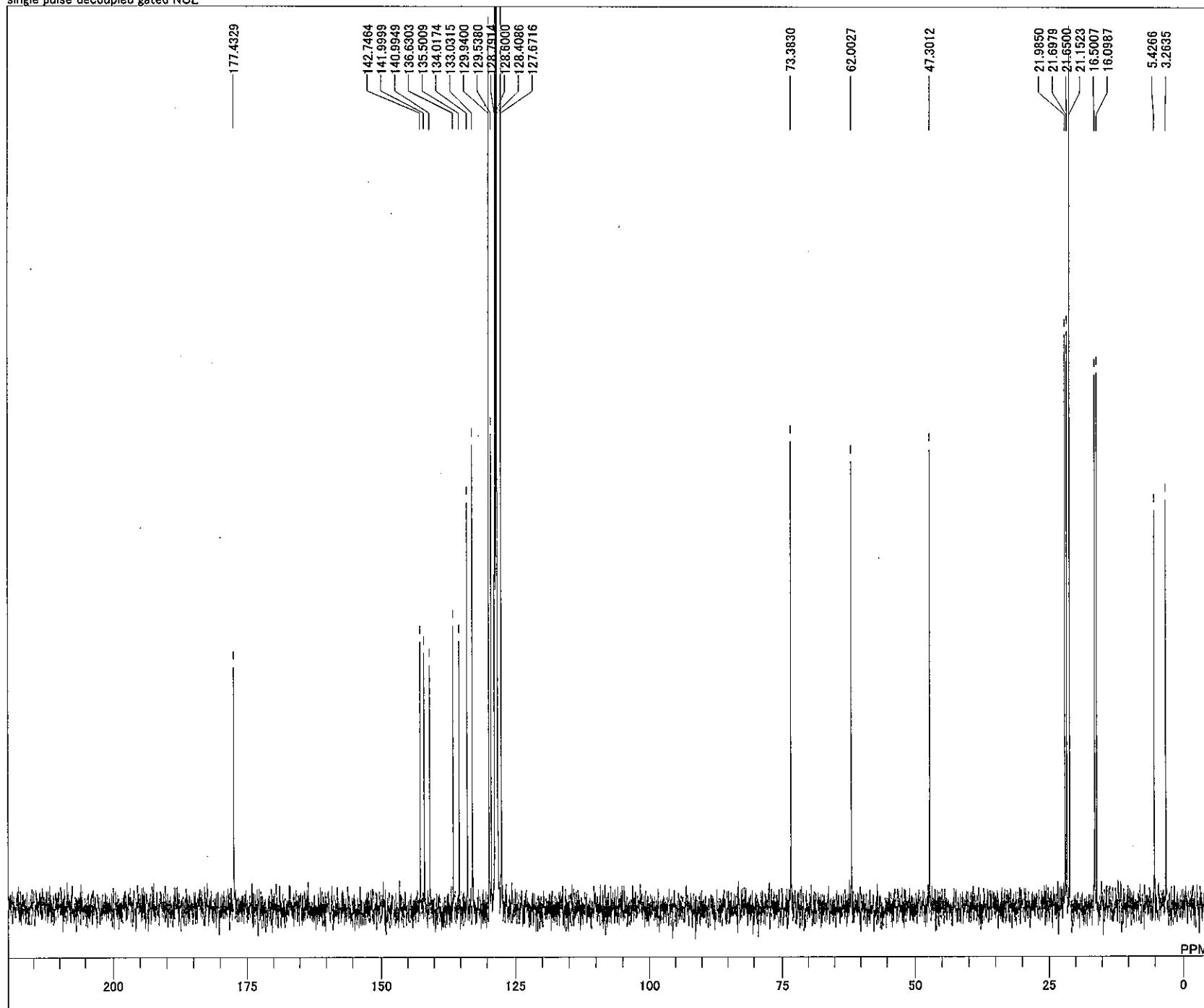
DFILE C:\Documents and Settings\All Users\Docu
COMNT single pulse decoupled gated NOE
DATIM 08-08-2007 20:33:55
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26224
FREQU 31249.52 Hz
SCANS 64
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 28.2 c
SLVNT C6D6
EXREF 128.60 ppm
BF 0.12 Hz
RGAIN 54



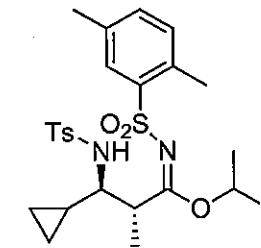


C:\Documents and Settings\All Users\Docu
08-08-2007 21:22:35
1H
1H NMR.ex2
495.13 MHz
4.38 KHz
9.64 Hz
13120
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
28.0 c
C6D6
0.00 ppm
0.12 Hz
28

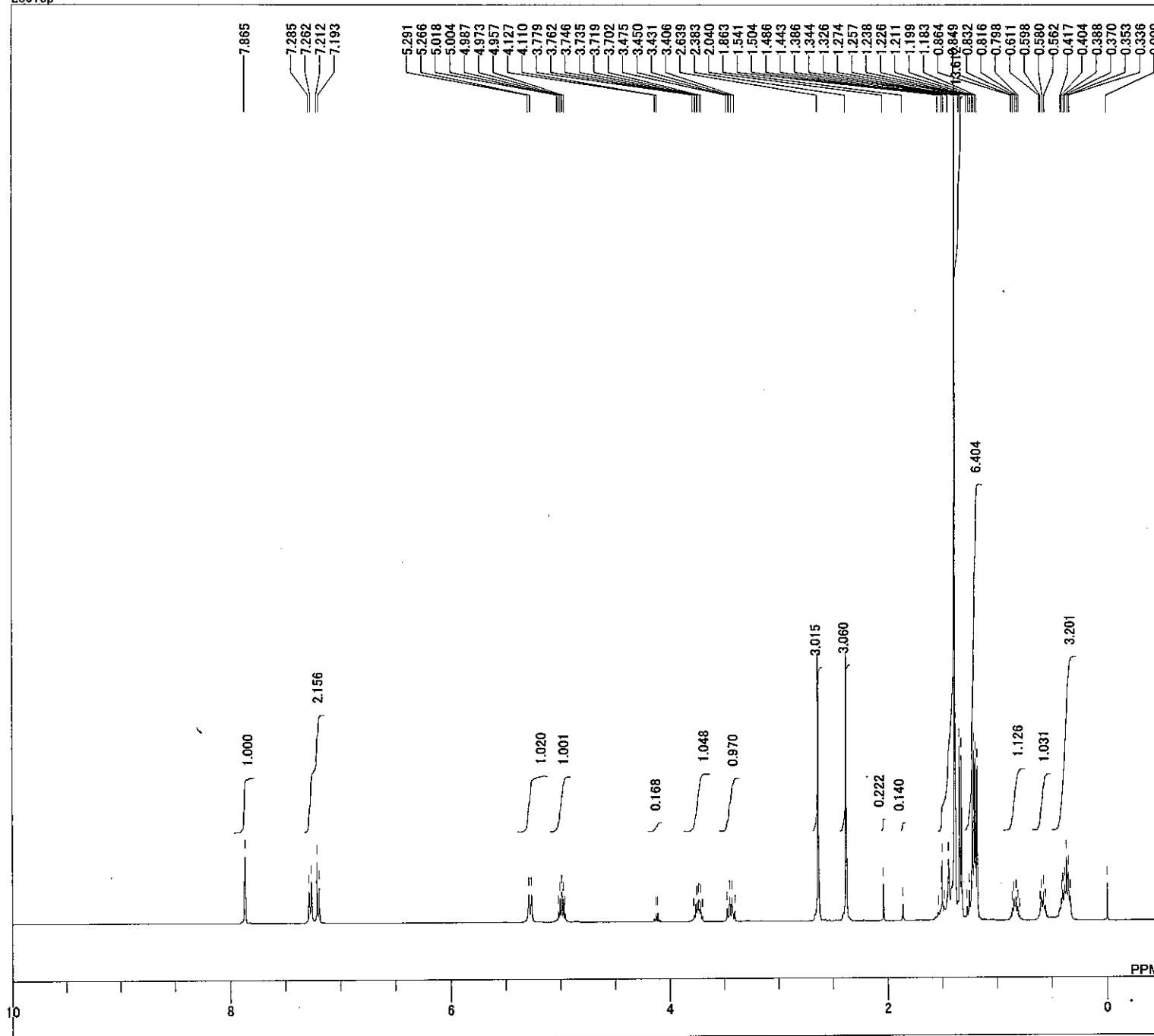




C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
08-08-2007 21:26:32
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
62
0.8389 sec
2.0000 sec
3.67 usec
1H
28.3 c
C6D6
128.60 ppm
0.12 Hz
50



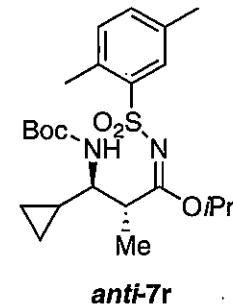
anti-7b



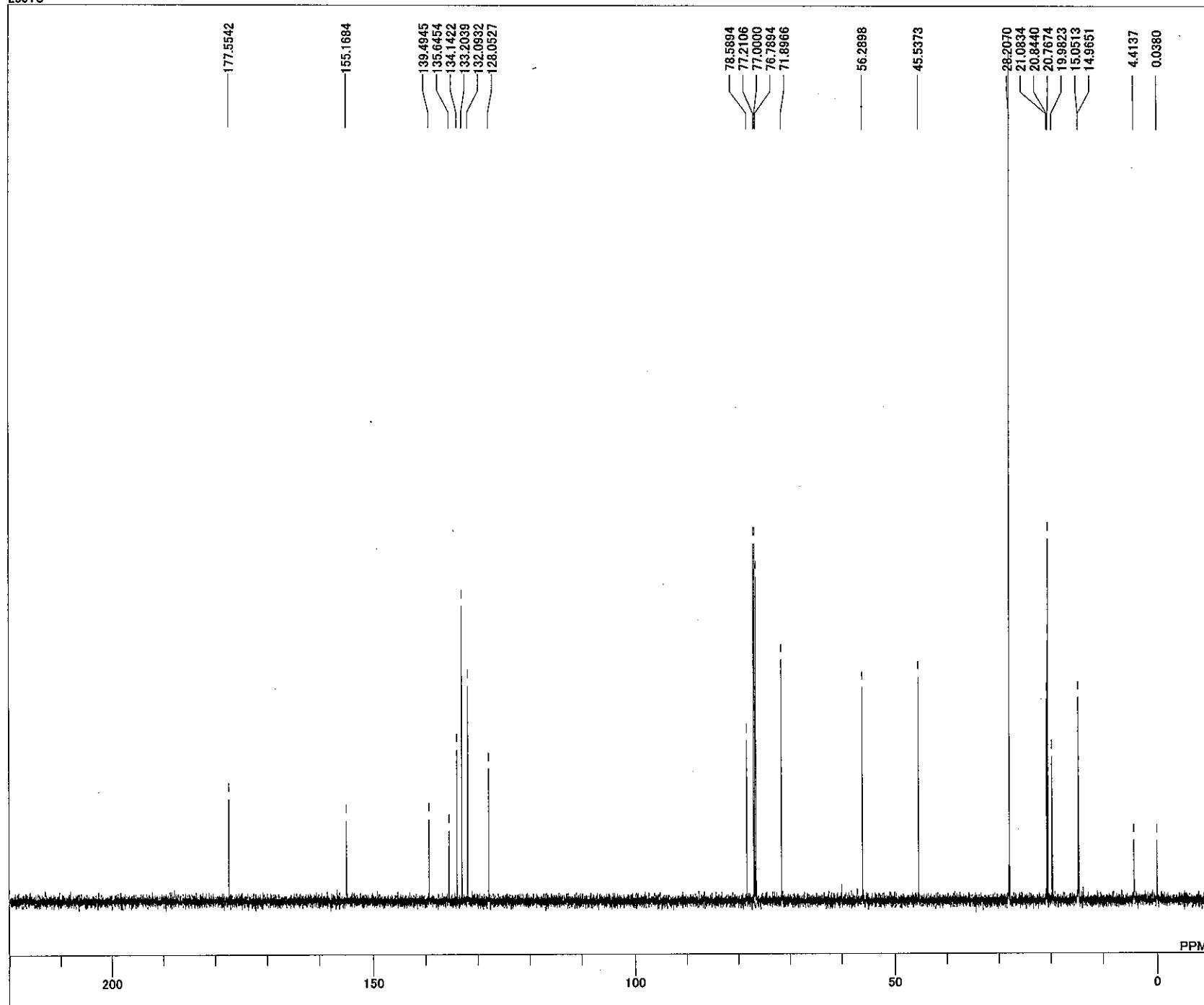
DFILE
COMNT
DATIM
OBNUC

EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

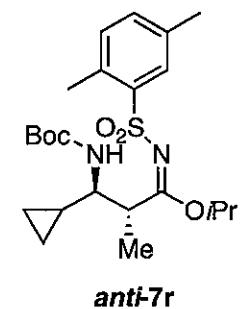
single_pulse.ex2
399.78 MHz
4.19 kHz
7.29 Hz
16384
7503.00 Hz
8
2.1837 sec
2.0000 sec
5.90 usec
1H
25.0 c
CDCL3
0.00 ppm
0.12 Hz
22



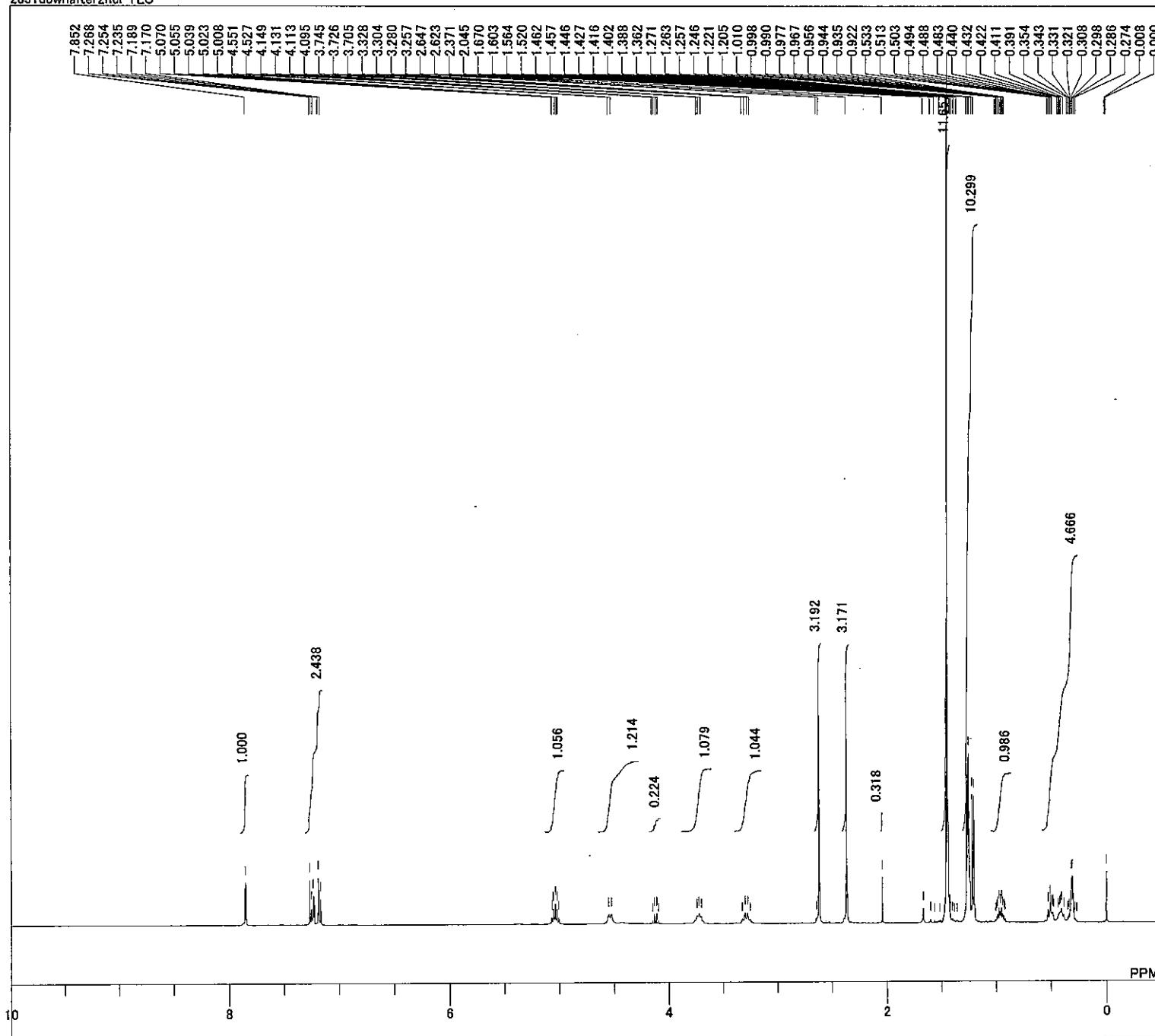
anti-7r



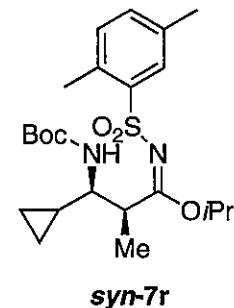
DFILE
COMNT
DATIM
OBNUC
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\Documents and Settings\All Users\Docu
2831C
13-12-2007 20:55:28
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
26214
37878.21 Hz
61
0.6921 sec
2.0000 sec
2.83 usec
1H
22.6 c
CDCL3
77.00 ppm
0.12 Hz
58

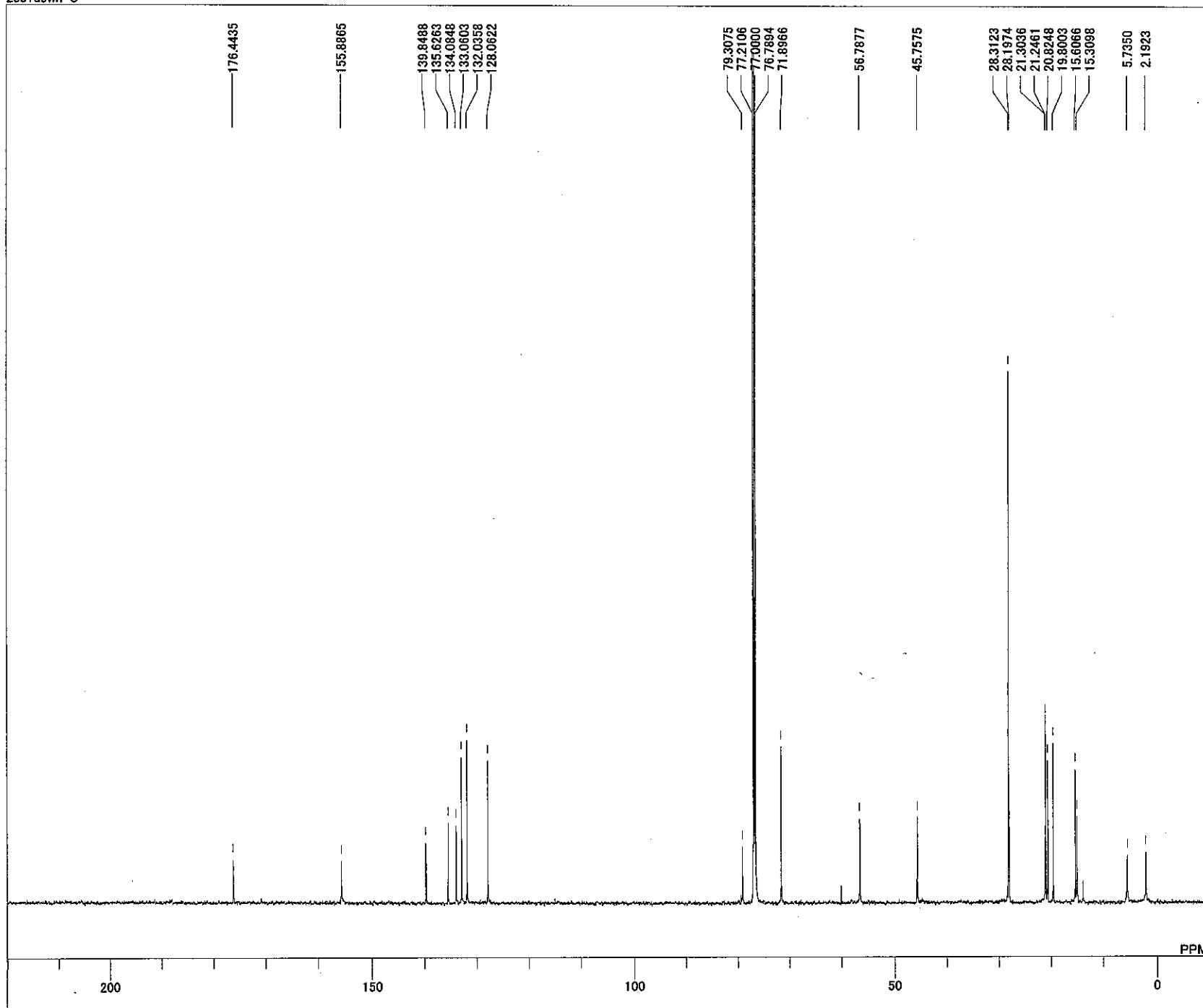


anti-7r



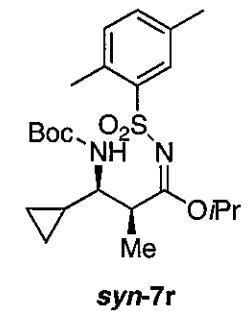
C:\Documents and Settings\deltaelta\My Documents\Person:
2831downafter2ndPTLC
14-12-2007 19:11:56
1H
single_pulse.ex2
399.78 MHz
4.19 KHz
7.29 Hz
13107
6002.31 Hz
8
2.1837 sec
2.0000 sec
5.90 usec
1H
25.0 c
CDCL3
0.00 ppm
0.12 Hz
32





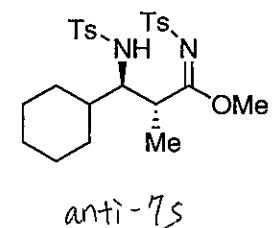
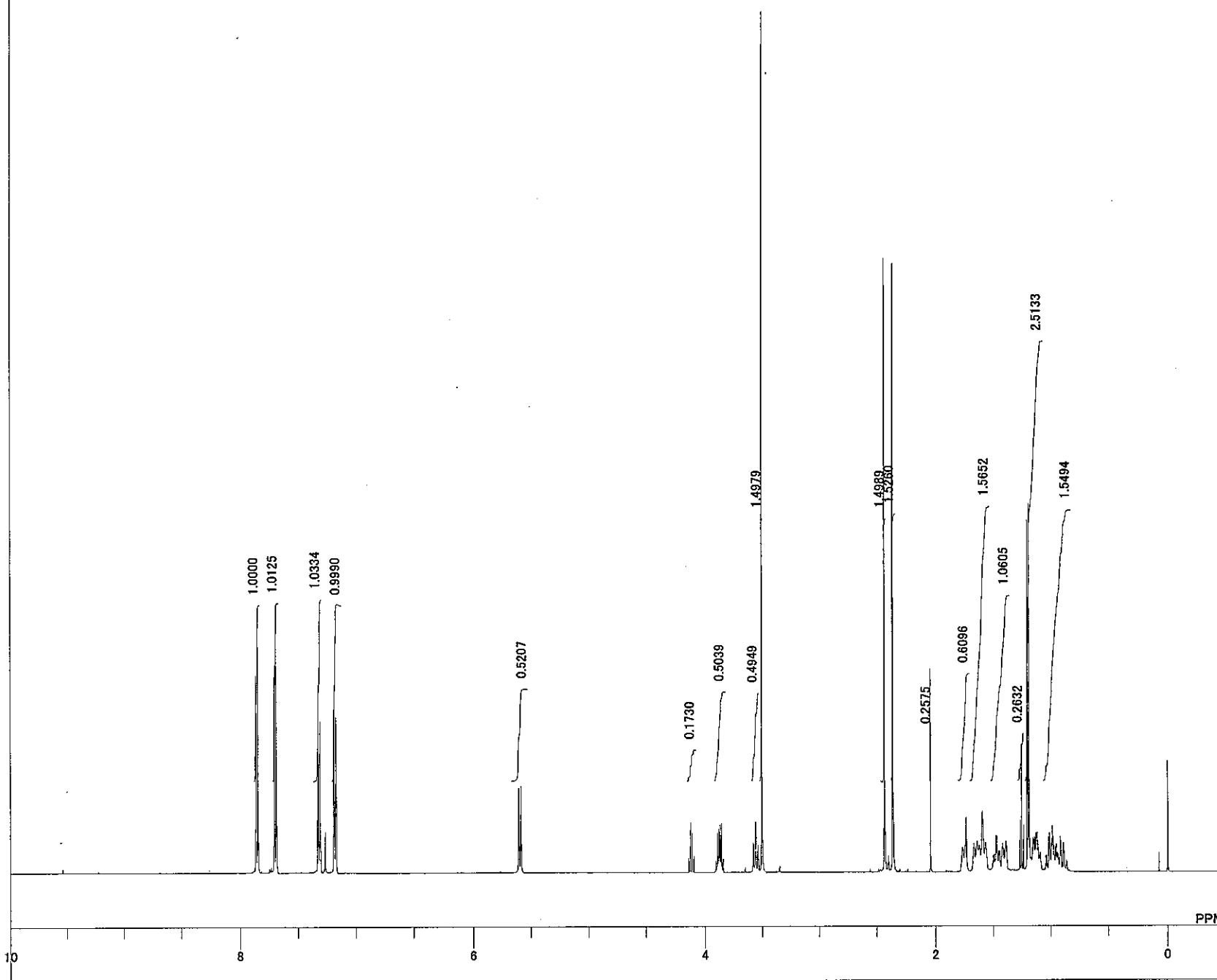
DFILE
COMNT
DATIM
OBNUC
OBSET
OBFIN
POINT
EXMOD
OBFRQ
OBSET
OBFIN
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

2831down-C
14-12-2007 10:27:37
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
40961
59186.51 Hz
3000
0.6921 sec
2.0000 sec
2.83 usec
1H
23.6 c
CDCL₃
77.00 ppm
0.12 Hz
58

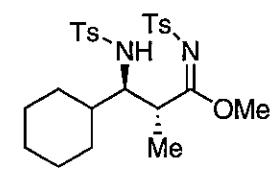
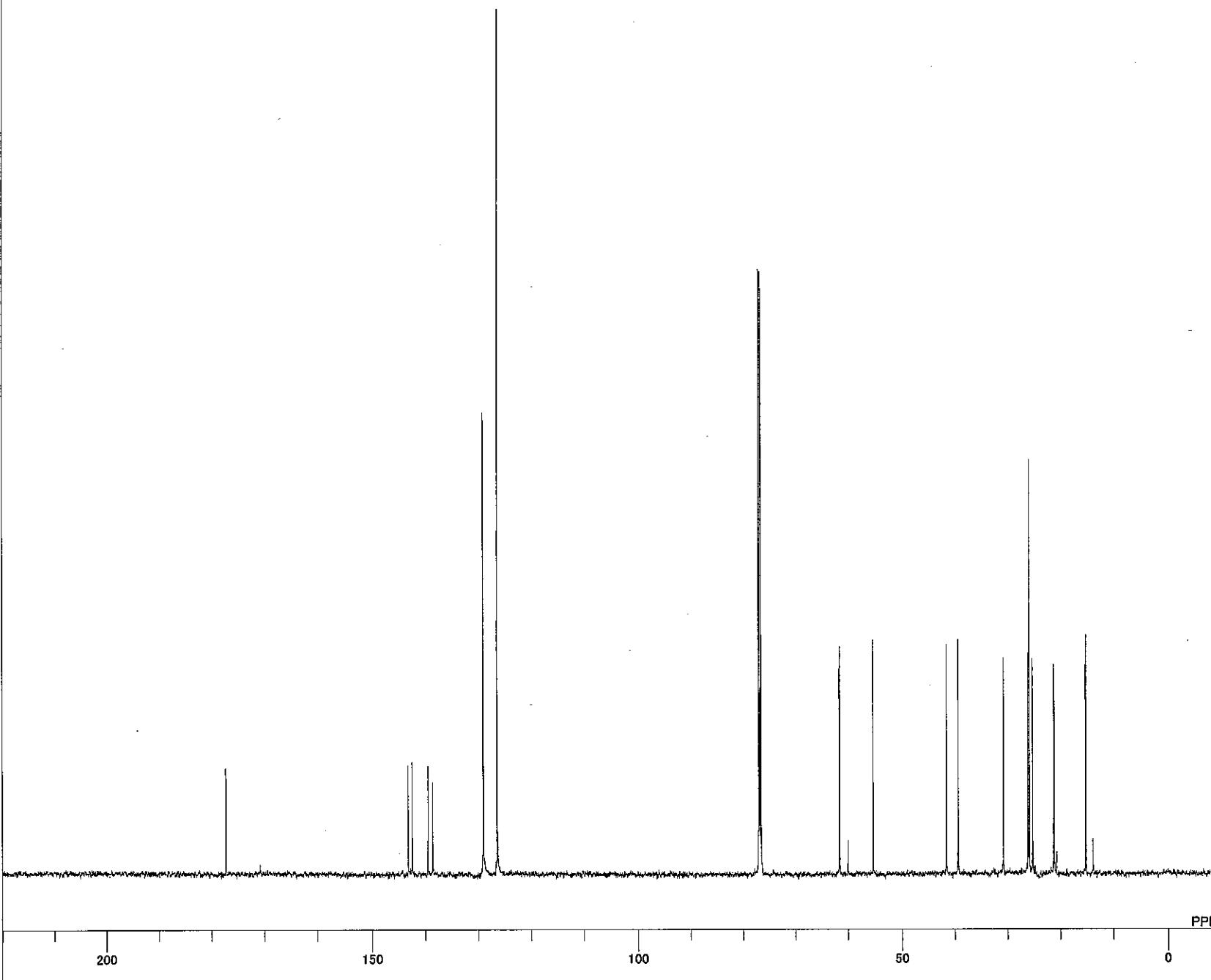


syn-7r

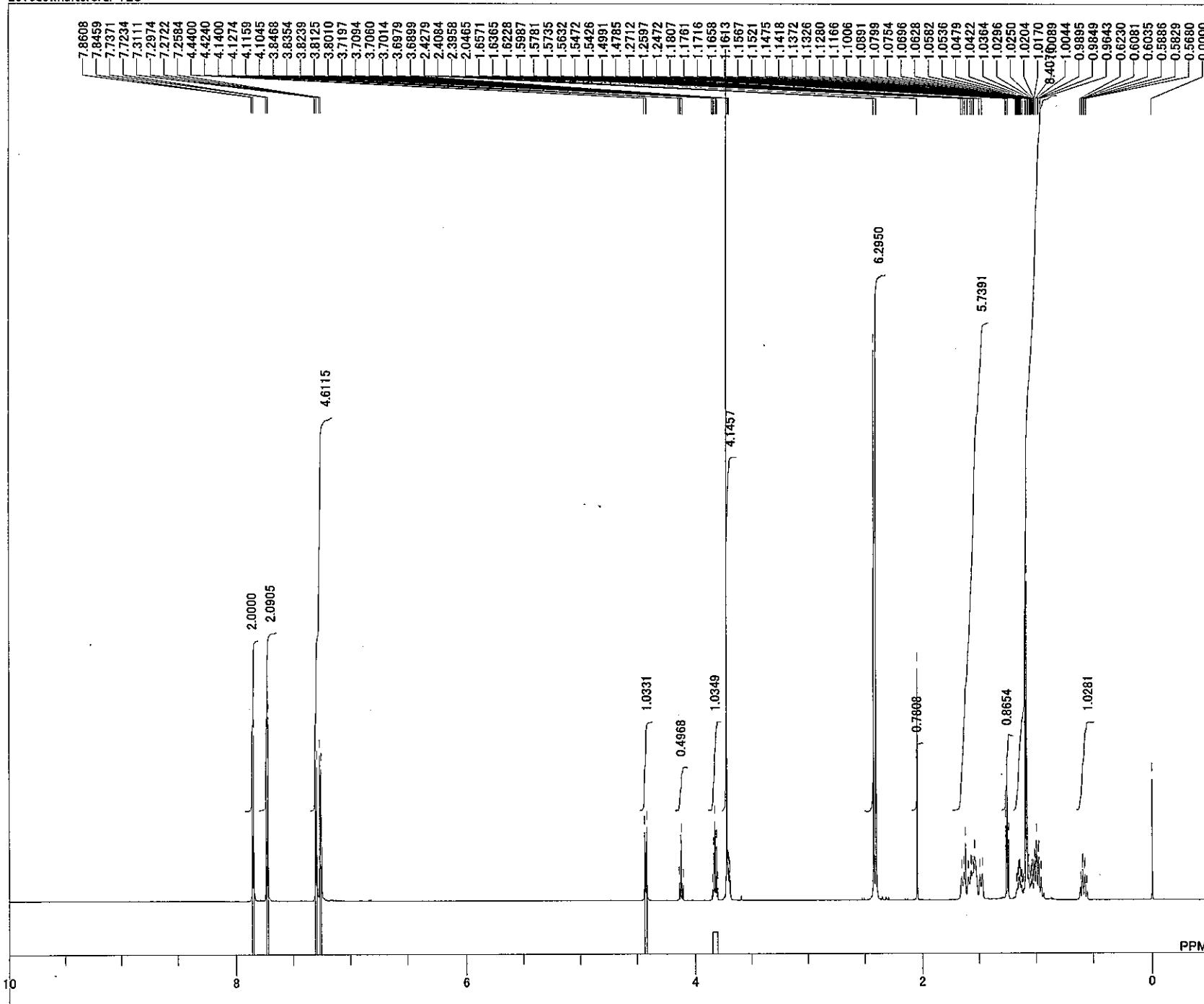
C:\Documents and Settings\All Users\Docu
2810upup
07-12-2007 12:23:26
1H
1H NMR.ex2
495.13 MHz
4.38 KHz
9.64 Hz
13107
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
21.7 °C
CDCL₃
0.00 ppm
0.12 Hz
30



DFILE C:\Documents and Settings\All Users\Docu
COMNT 2810upup600
DATIM 07-12-2007 03:26:35
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 40961
FREQU 59186.51 Hz
SCANS 512
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 2.83 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58



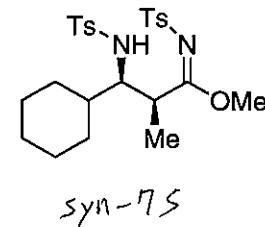
anti-7s

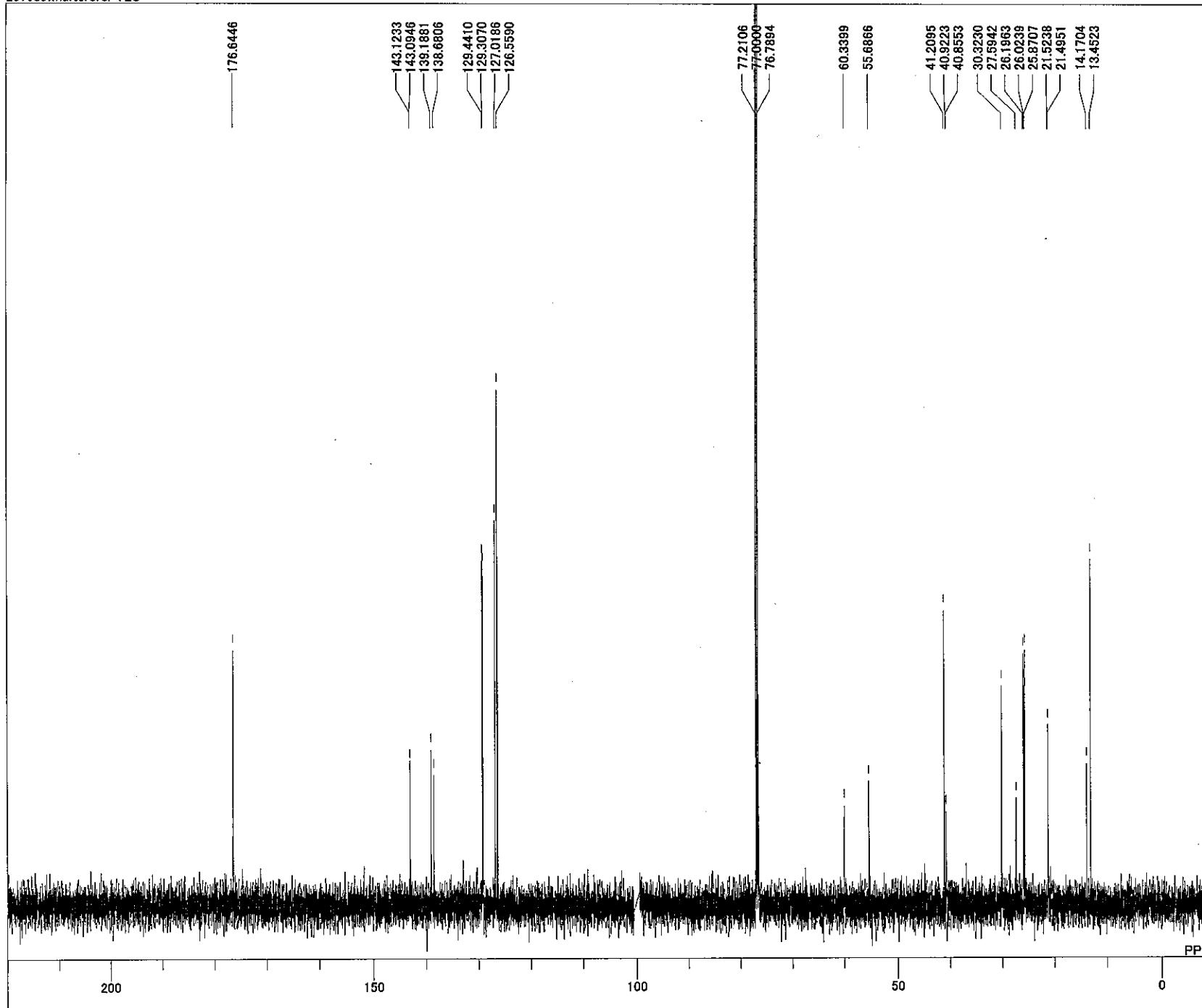


DFILE
COMT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

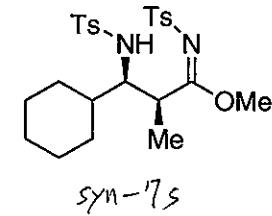
single_pulse.ex2
600.17 MHz
5.30 kHz
5.47 Hz
20480
14076.79 Hz
8
1.4549 sec
4.0000 sec
6.75 usec
1H
21.3 c
CDCL3
0.00 ppm
BF
1.20 Hz
RGAIN
40

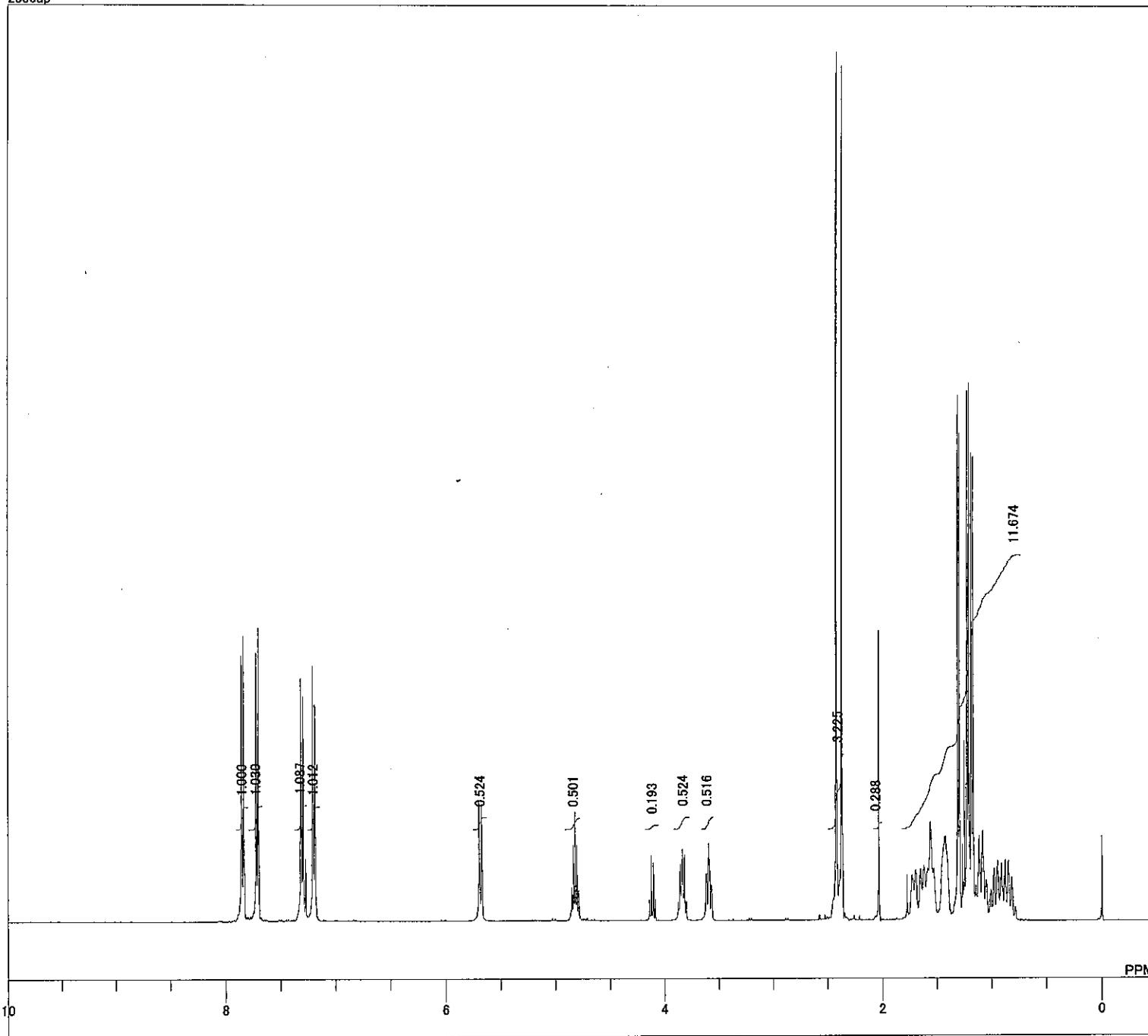
¹H -NMR (CDCl₃) δ:
7.85 (2H, d, J = 8.9 Hz),
7.73 (2H, d, J = 8.2 Hz),
7.30 (5H, d, J = 8.2 Hz),
7.27 (5H, d, J = 8.2 Hz),
4.43 (1H, d, J = 9.6 Hz),
3.85–3.80 (1H, m).



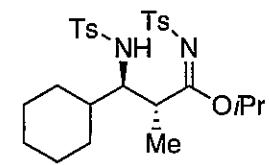


DFILE C:\Documents and Settings\All Users\Docu
COMNT 2810downafter3rdPTLC
DATIM 21-12-2007 06:51:52
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 26214
FREQU 37878.21 Hz
SCANS 512
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 2.83 usec
IRNUC 1H
CTEMP 22.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56

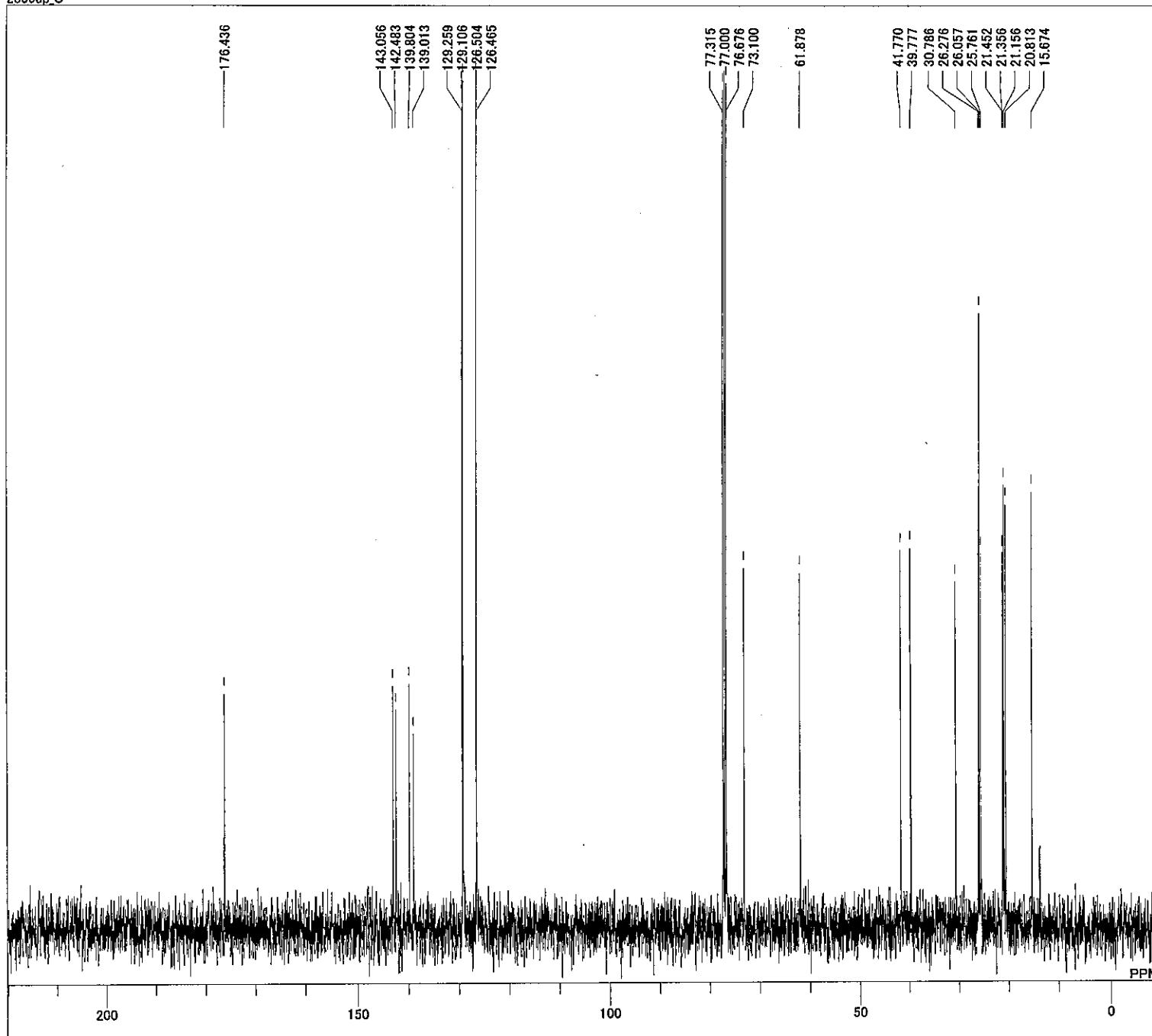




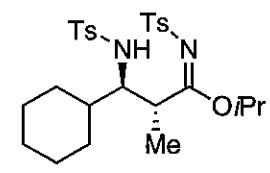
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
1H
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
2830up
14-12-2007 19:05:05
1H
single_pulse.ex2
399.78 MHz
4.19 KHz
7.29 Hz
16384
7503.00 Hz
8
2.1837 sec
2.0000 sec
5.90 usec
25.0 c
CDCL3
0.00 ppm
0.12 Hz
24



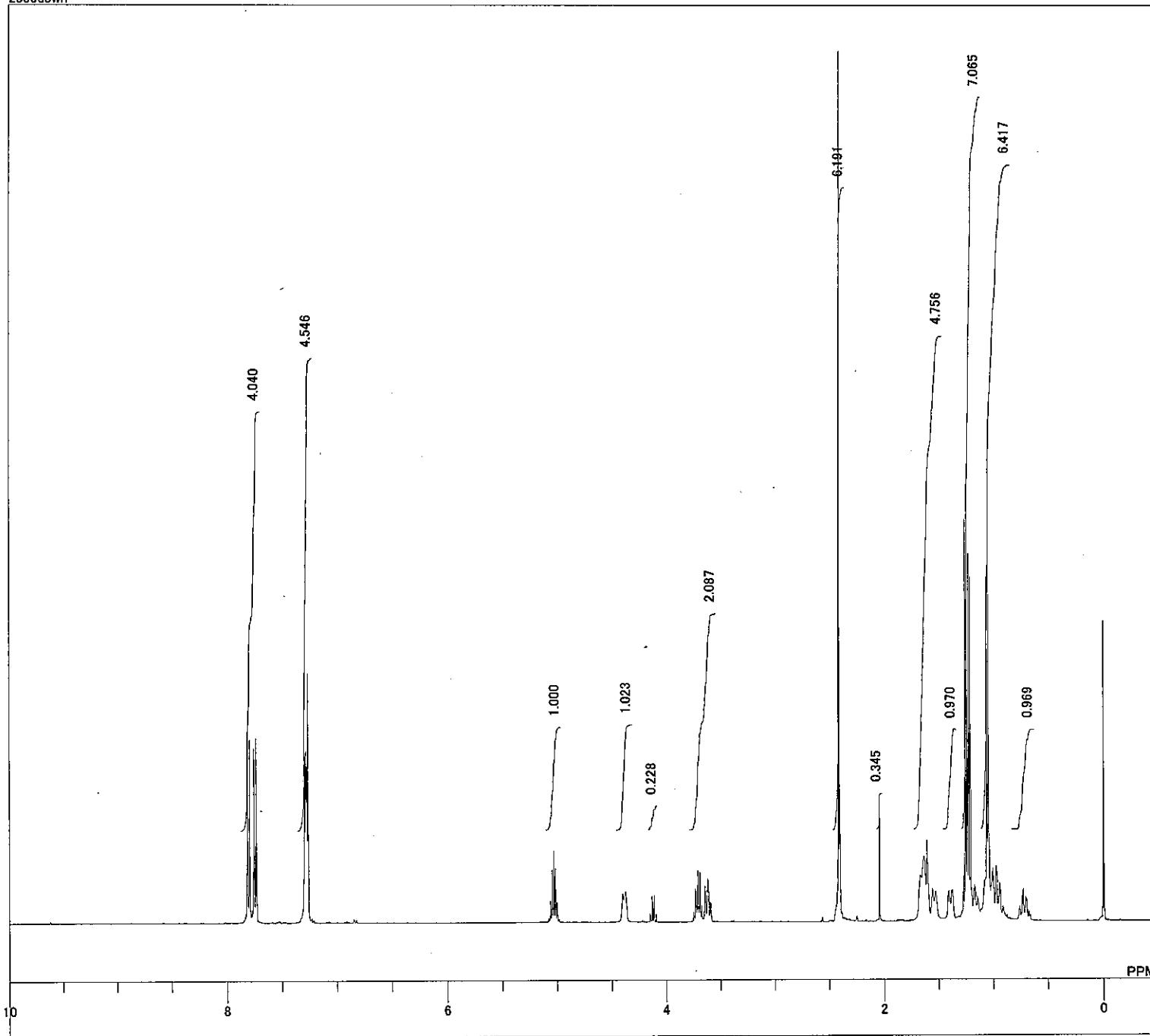
anti-7t



C:\Documents and Settings\deltaelta\My Documents\Personal Folder
2830up_C
14-12-2007 19:08:41
13C
single_pulse_dec
100.53 MHz
5.35 KHz
5.86 Hz
40961
39259.39 Hz
15
1.0433 sec
2.0000 sec
2.83 usec
1H
25.0 c
CDCL3
77.00 ppm
0.12 Hz
56

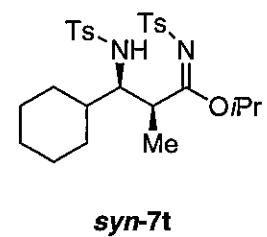


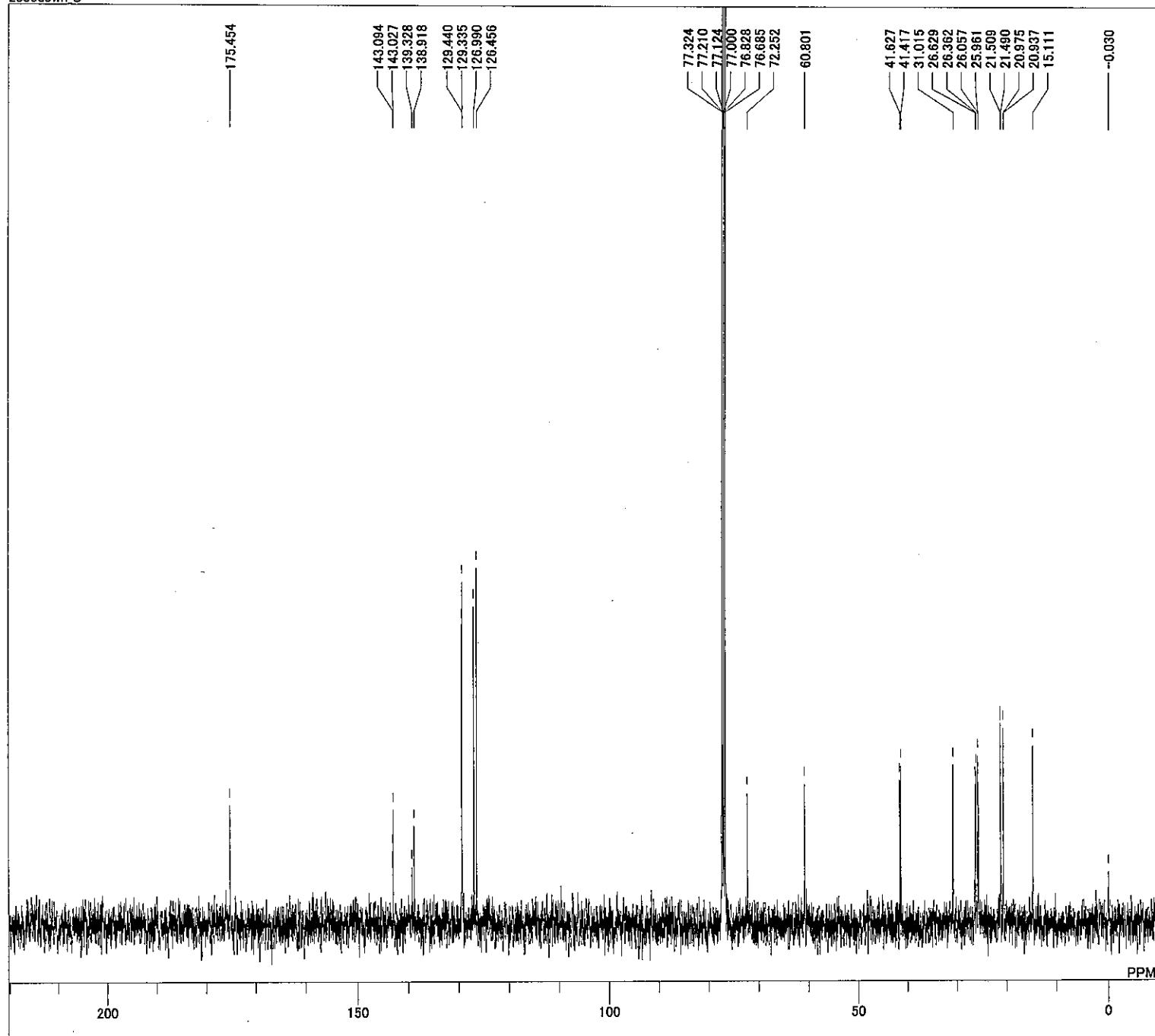
anti-7t



DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

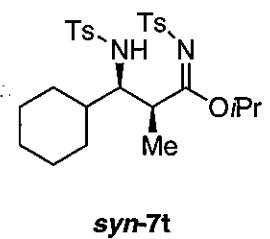
C:\Documents and Settings\deltaelta\My Documents\Person:
2830down
14-12-2007 18:53:58
1H
single_pulse.ex2
399.78 MHz
4.19 KHz
7.29 Hz
13107
6002.31 Hz
8
2.1837 sec
2.0000 sec
5.90 usec
1H
25.1 c
CDCL3
0.00 ppm
0.12 Hz
34

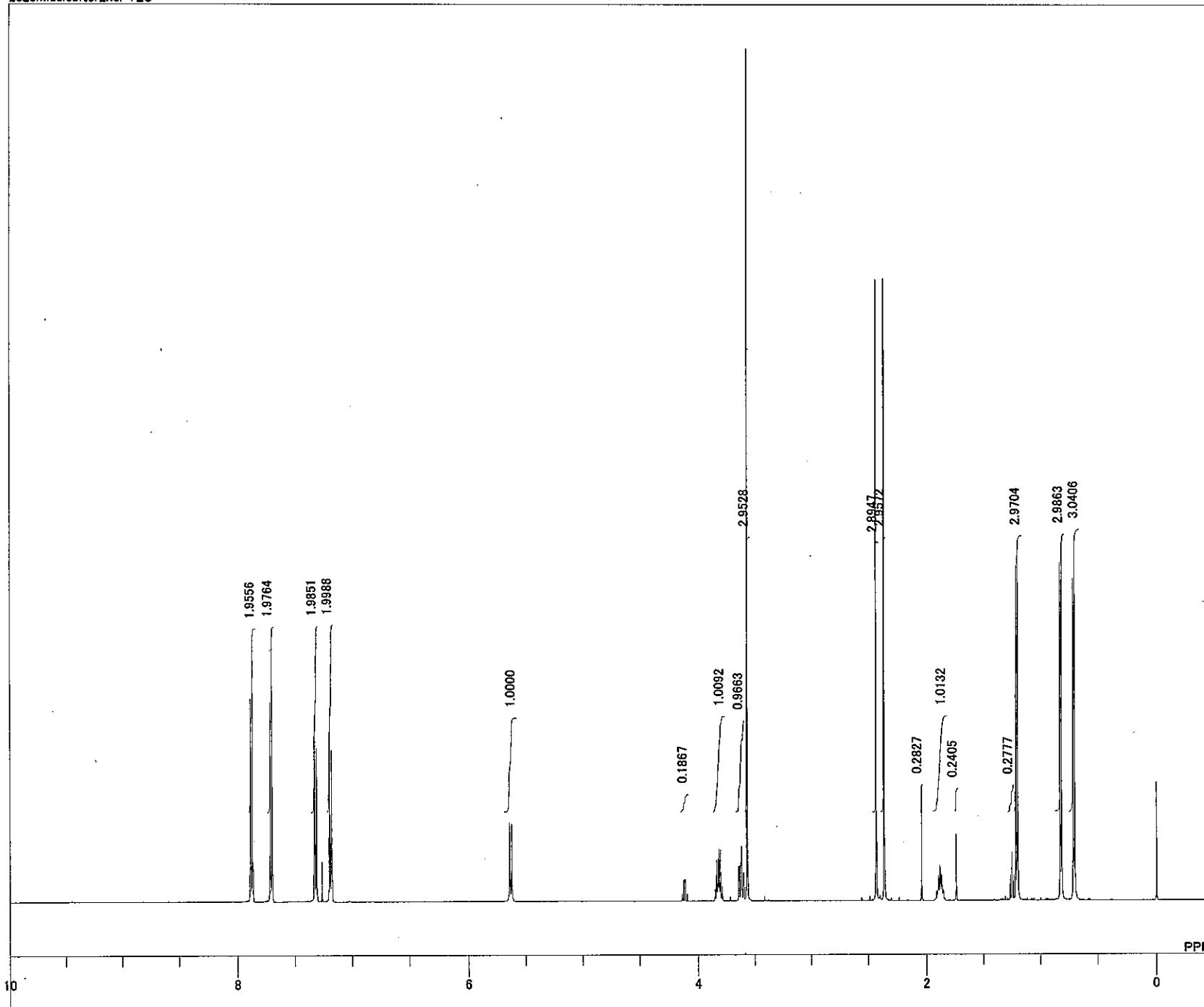




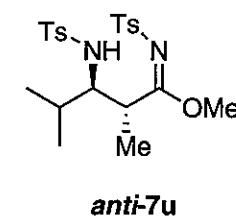
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

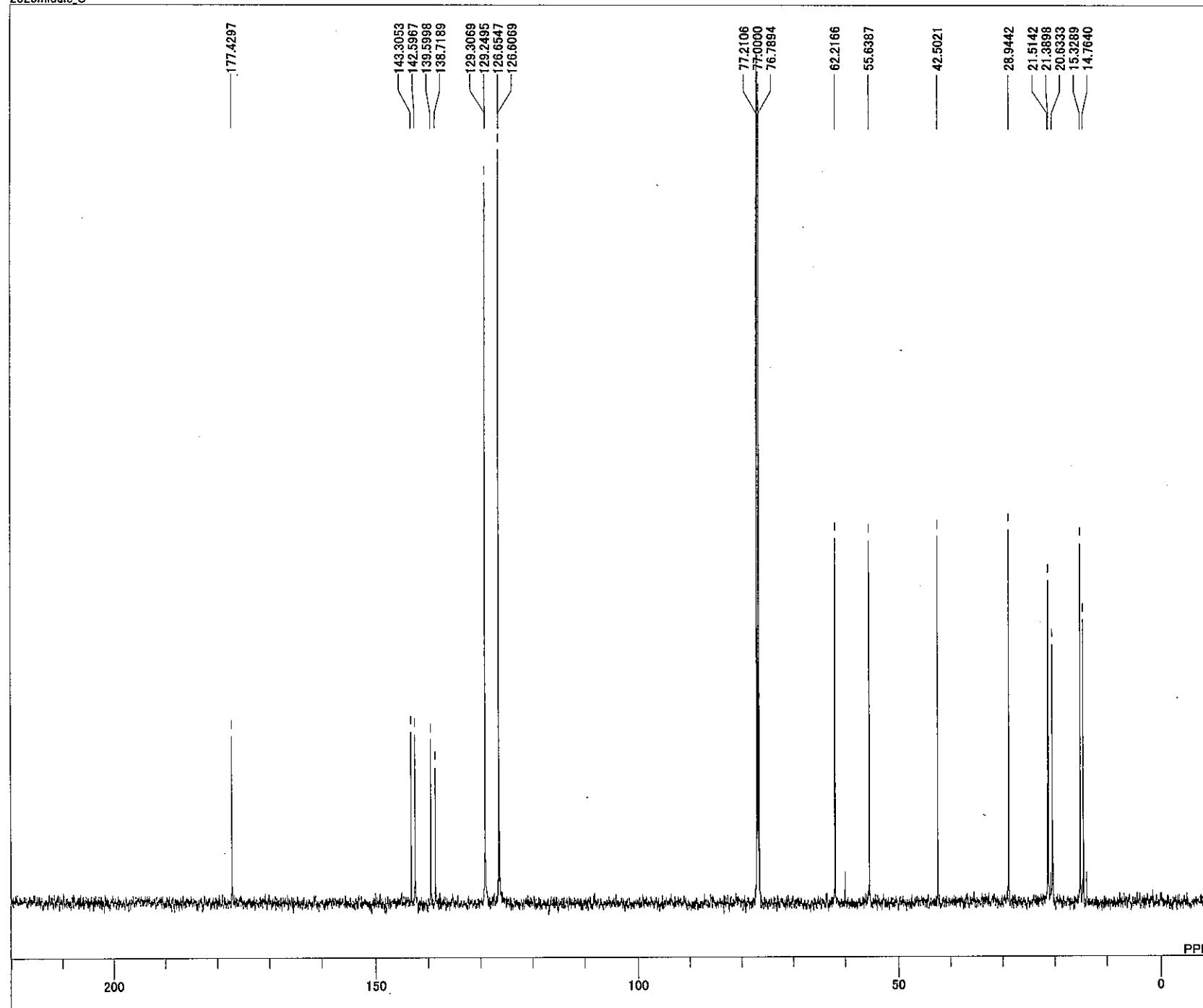
2830down_C
14-12-2007 19:01:32
13C
single_pulse_dec
100.53 MHz
5.35 kHz
5.86 Hz
32768
31407.04 Hz
90
1.0433 sec
2.0000 sec
2.83 usec
1H
25.0 c
CDCL3
77.00 ppm
0.12 Hz
58





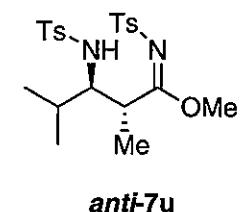
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
1H
1H NMR.ex2
495.13 MHz
4.38 KHz
9.64 Hz
13107
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
22.3 c
CDCL3
0.00 ppm
0.12 Hz
30



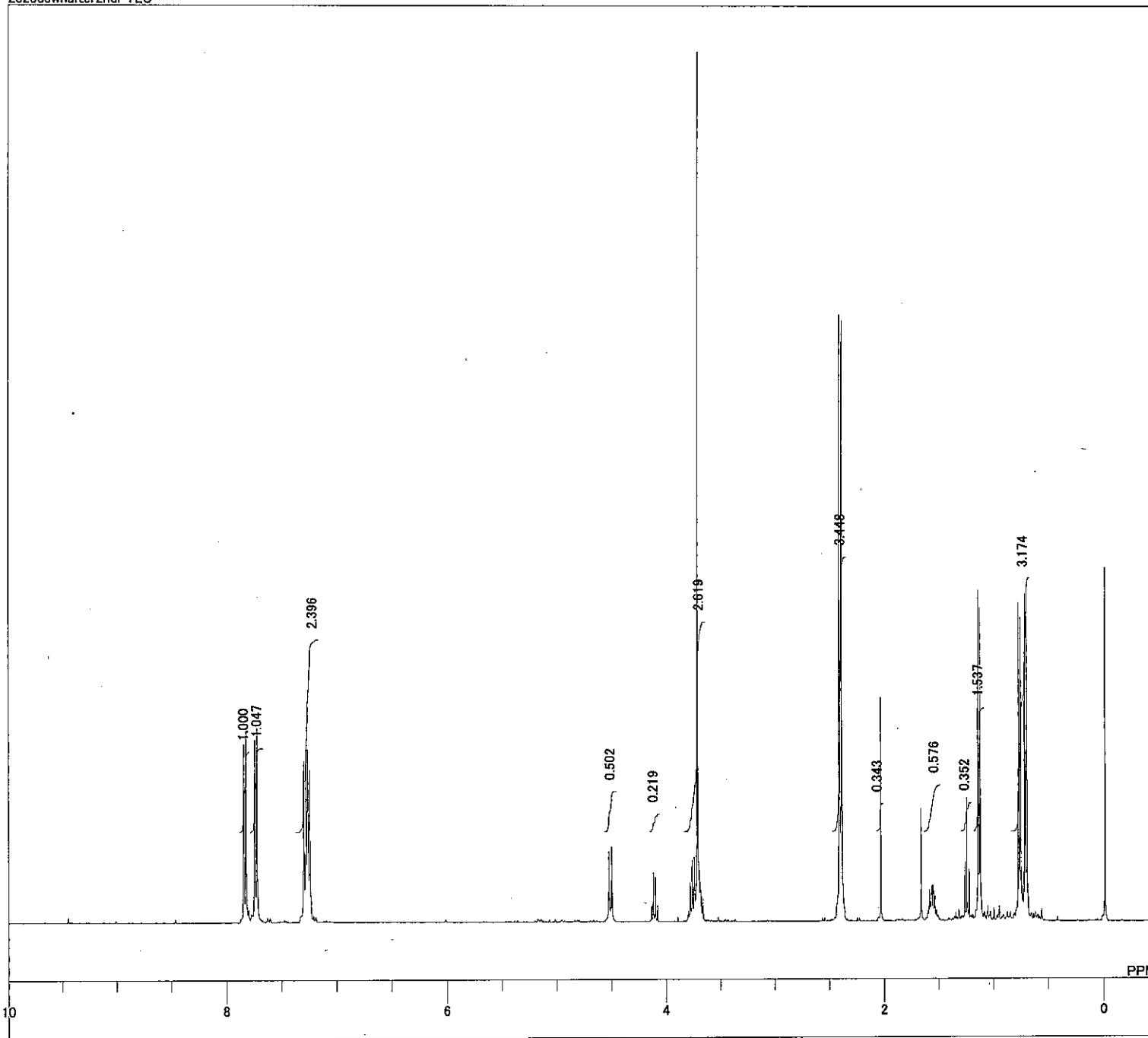


DFILE
COMNT
DATIM
OBNUC
OBSET
OBFRQ
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

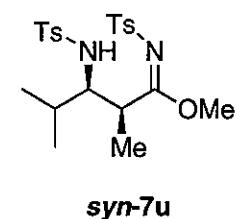
2828middle_C
13-12-2007 19:25:31
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
40961
59186.51 Hz
256
0.6921 sec
2.0000 sec
2.83 usec
1H
22.0 c
CDCL3
77.00 ppm
0.12 Hz
58



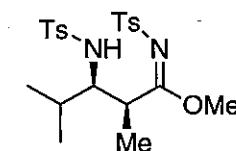
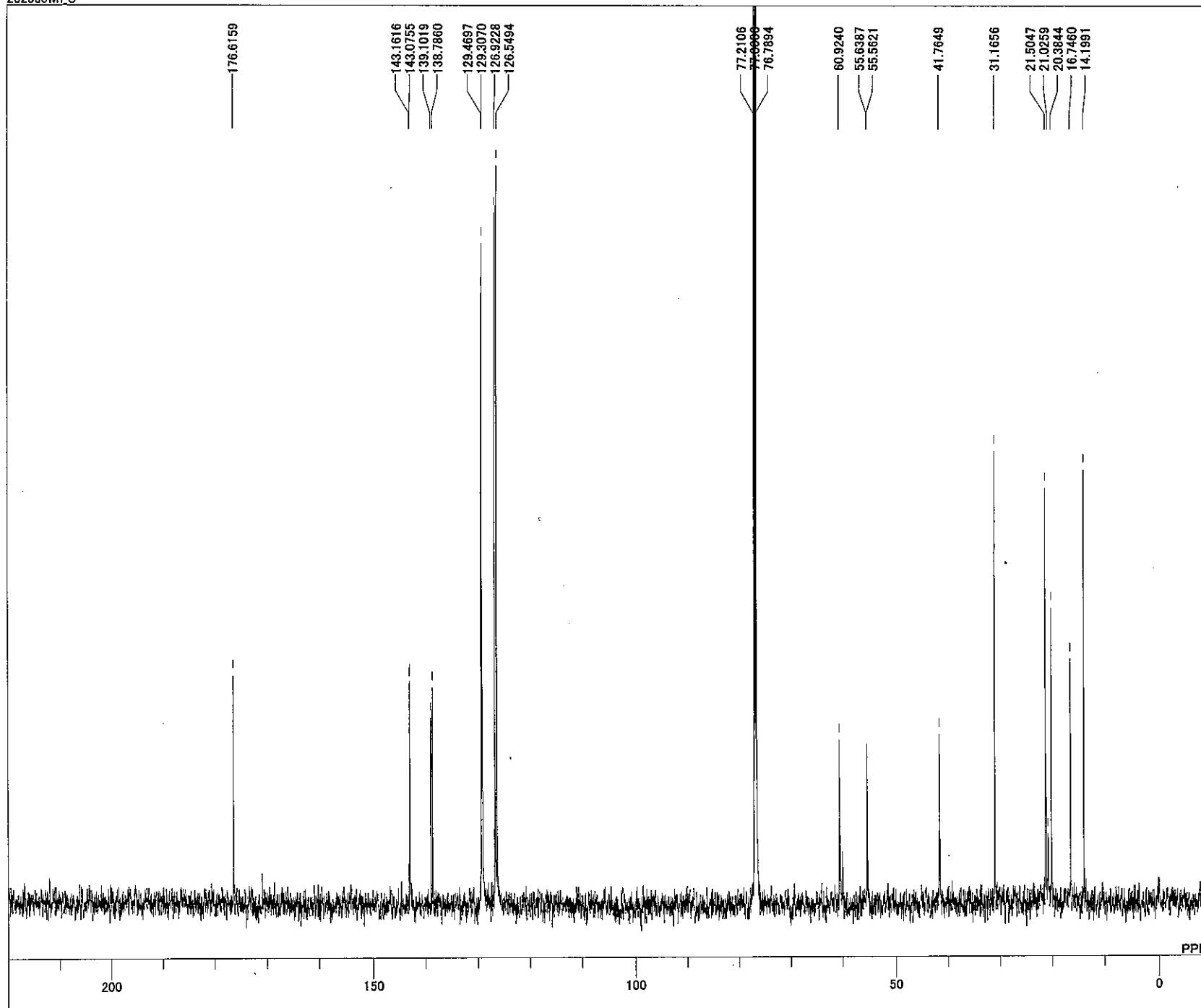
120



C:\Documents and Settings\deltaelta\My Documents\Personal
2828downafter2ndPTLC
13-12-2007 20:27:11
1H
single_pulse.ex2
399.78 MHz
4.19 KHz
7.29 Hz
16384
7503.00 Hz
8
2.1837 sec
2.0000 sec
5.90 usec
1H
25.1 c
CDCL3
12.51 ppm
0.12 Hz
32

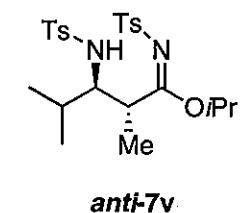
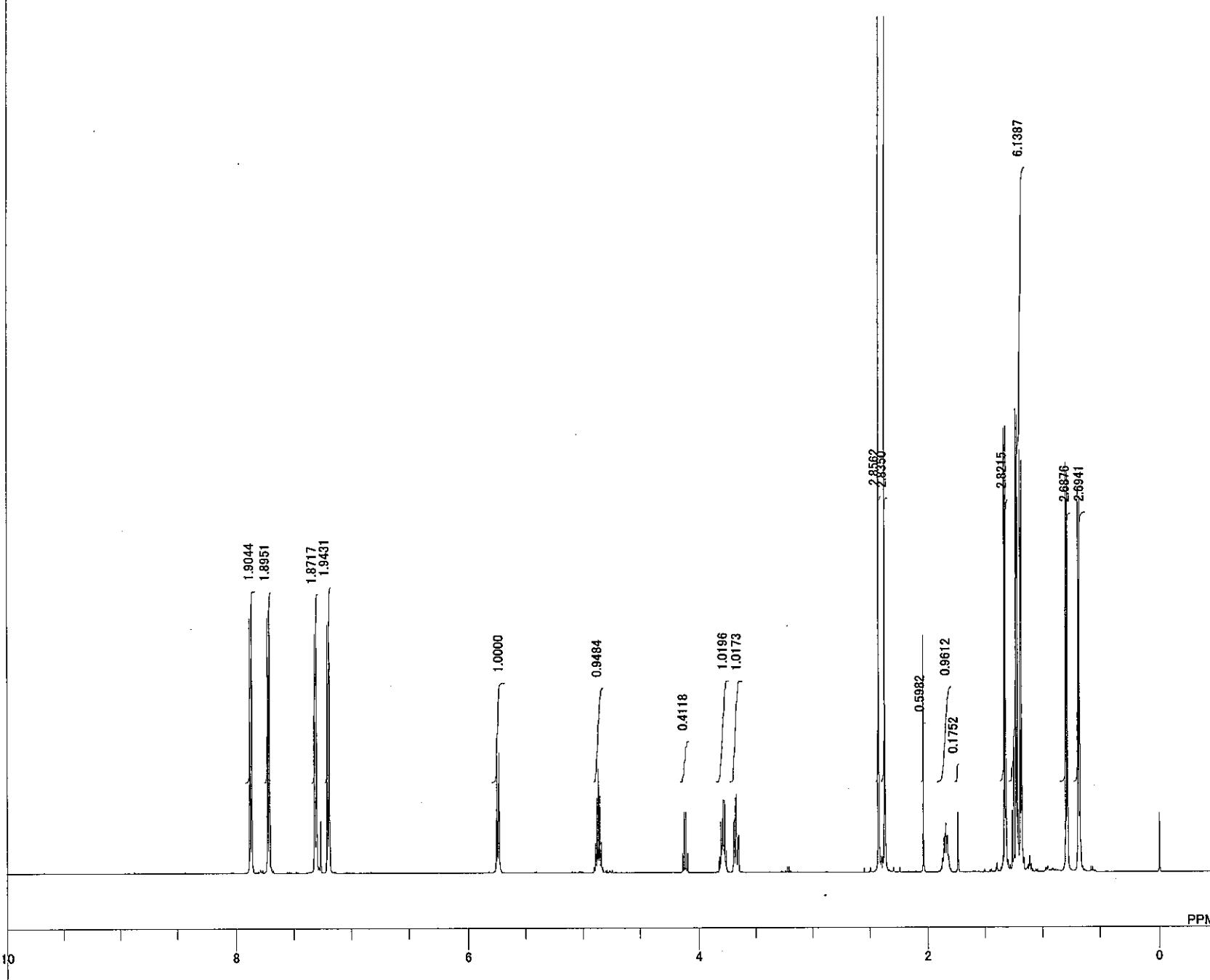


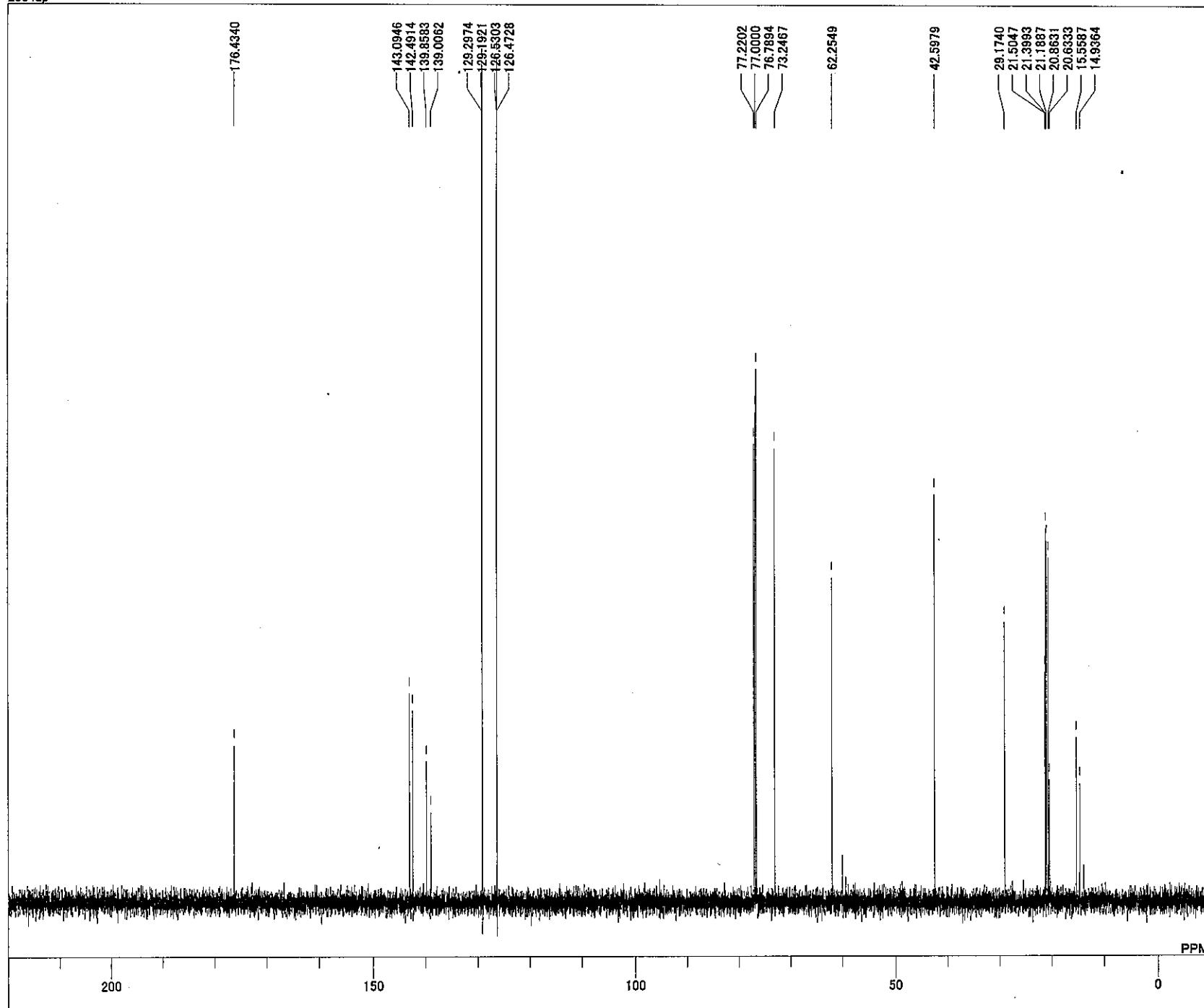
syn-7u



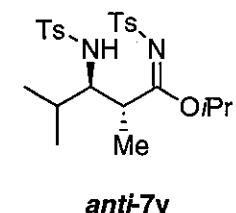
syn-7u

C:\Documents and Settings\All Users\Docu
2834up
20-12-2007 19:46:34
DFILE
COMNT
DATIM
OBNUC
1H
EXMOD
OBFRQ
495.13 MHz
OBSET
4.38 KHz
OBFIN
9.64 Hz
POINT
16384
FREQU
7427.21 Hz
SCANS
8
ACQTM
2.2059 sec
PD
5.0000 sec
PW1
6.50 usec
IRNUC
1H
CTEMP
21.7 °C
SLVNT
CDCL₃
EXREF
0.00 ppm
BF
0.12 Hz
RGAIN
34



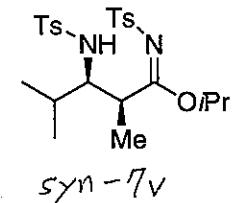
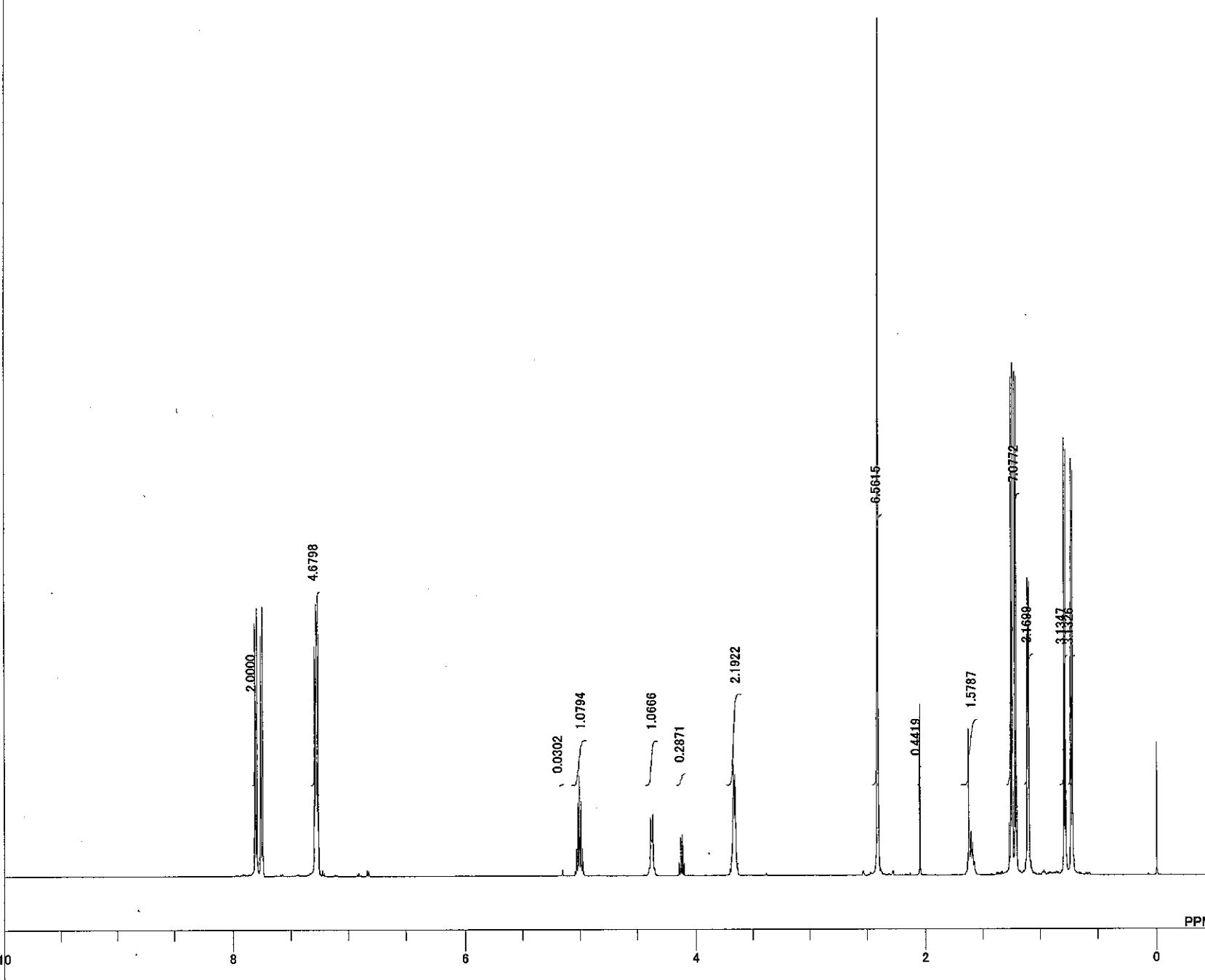


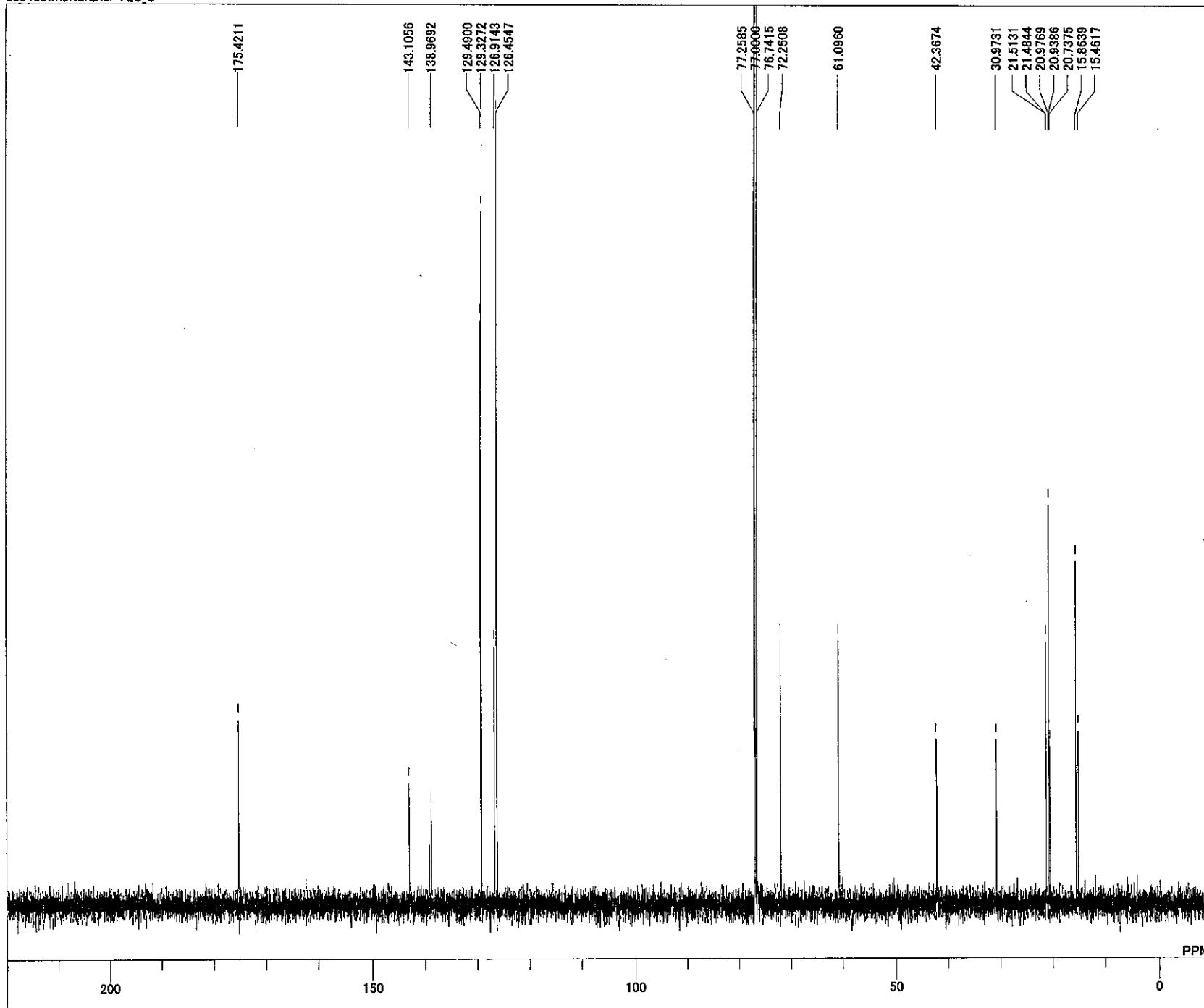
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2834up
DATIM 20-12-2007 06:05:56
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 26214
FREQU 37878.21 Hz
SCANS 44
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 2.83 usec
IRNUC 1H
CTEMP 23.0 °C
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58



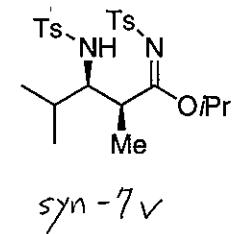
anti-7v

DFILE C:\Documents and Settings\All Users\Docu
COMNT 2834downafter2ndPTLC
DATIM 21-12-2007 13:50:53
1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

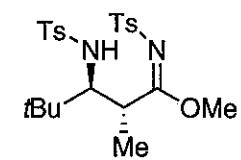
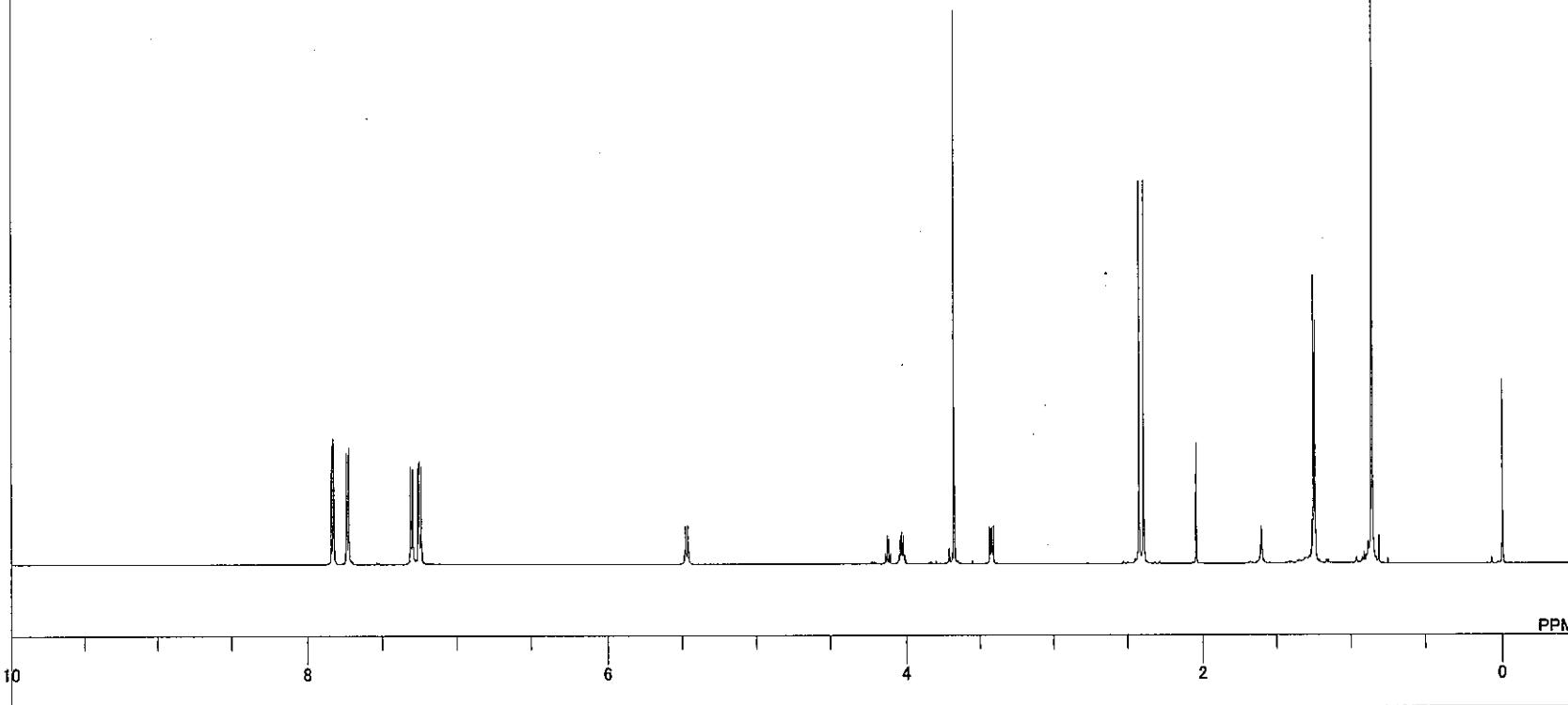




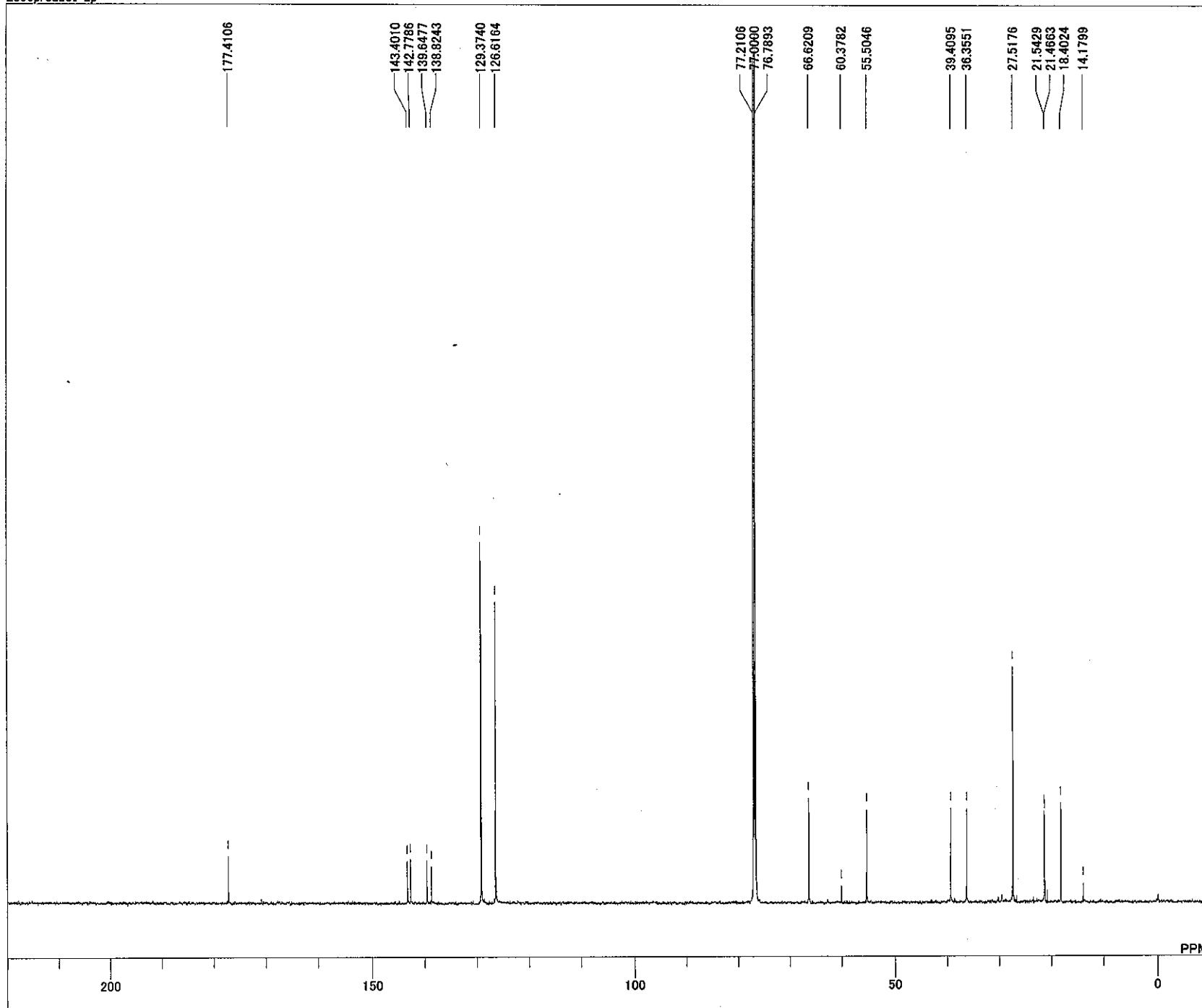
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2834downafter2ndPTLC_C
DATIM 21-12-2007 14:14:54
13C
OBNUC 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 496
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



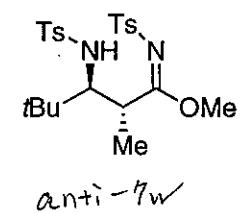
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2800product-up
DATIM 30-11-2007 08:11:00
1H
OBNUC single_pulse.ex2
EXMOD 600.17 MHz
OBFRQ 5.30 KHz
OBSET 5.47 Hz
OBFIN 20480
POINT 14076.79 Hz
FREQU 16
SCANS 1.4549 sec
ACQTM PD 4.0000 sec
PW1 PW1 6.75 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40



anti -7w

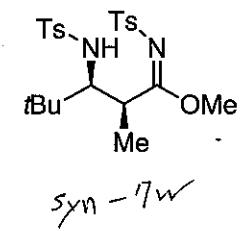
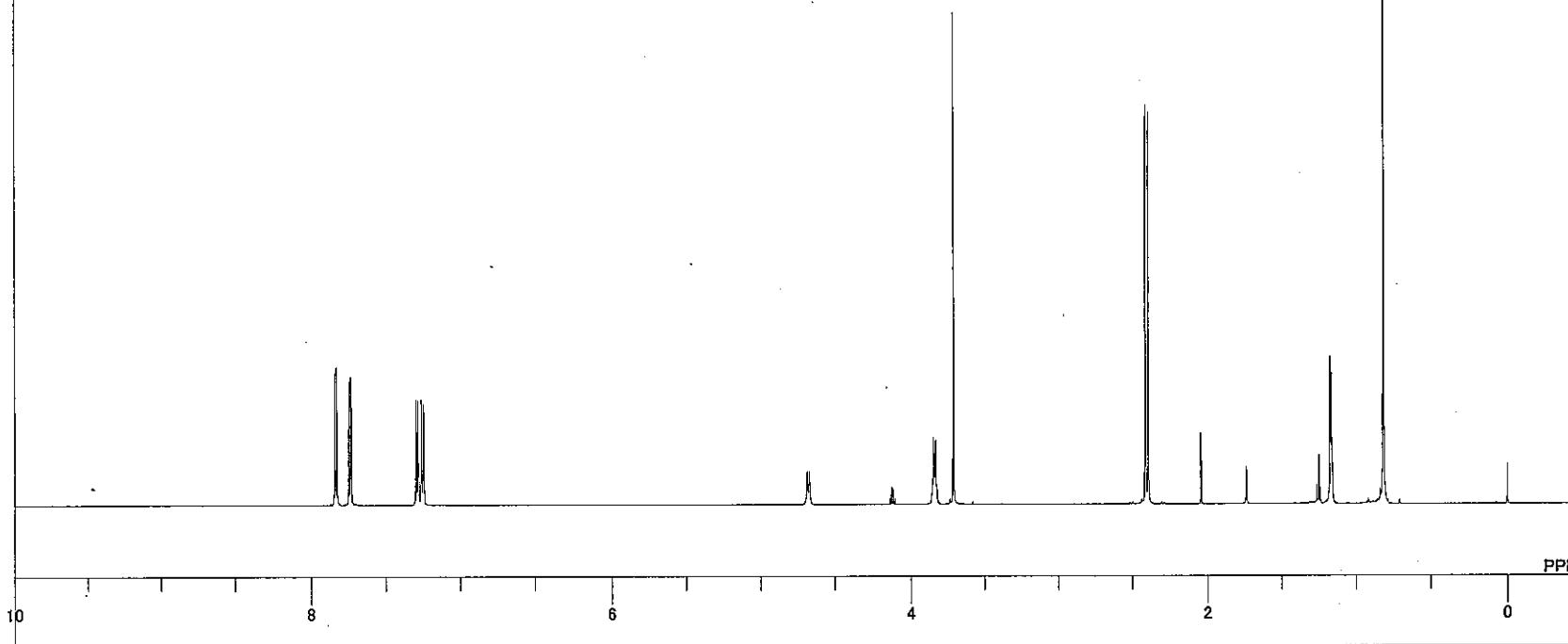


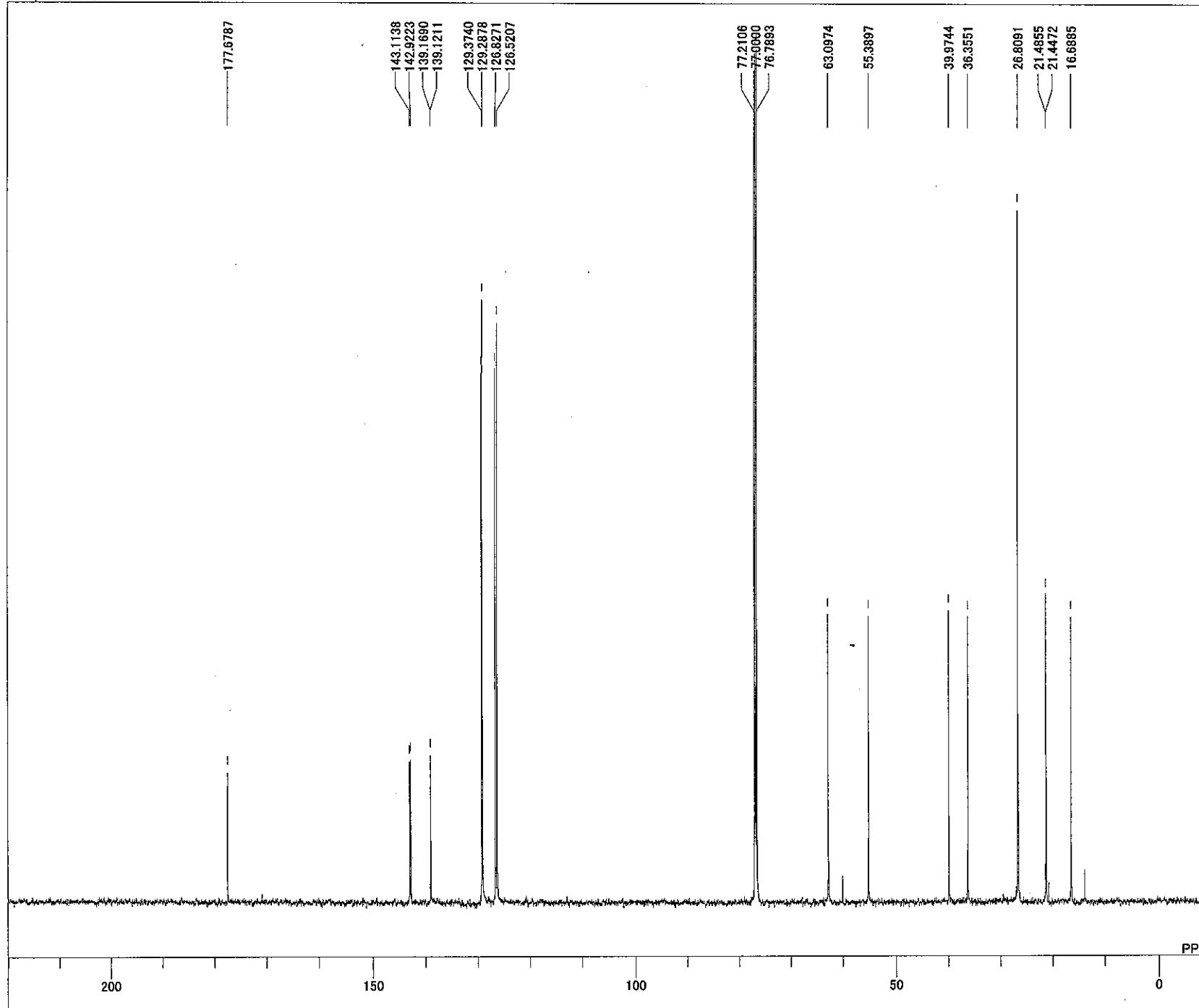
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2800product-up
DATIM 30-11-2007 11:12:32
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 kHz
OBFIN 1.74 Hz
POINT 40961
FREQU 59186.51 Hz
SCANS 4000
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 2.83 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL₃
EXREF 225.39 ppm
BF 0.12 Hz
RGAIN 58



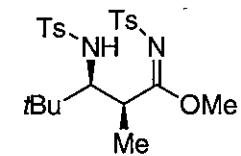
anti-7w

DFILE C:\Documents and Settings\All Users\Docu
COMNT 2800product-down
DATIM 30-11-2007 11:23:52
1H
OBNUC single_pulse.ex2
EXMOD 600.17 MHz
OBFRQ 5.30 kHz
OBSET 5.47 Hz
OBFIN 5.47 Hz
POINT 20480
FREQU 14076.79 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 6.75 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48

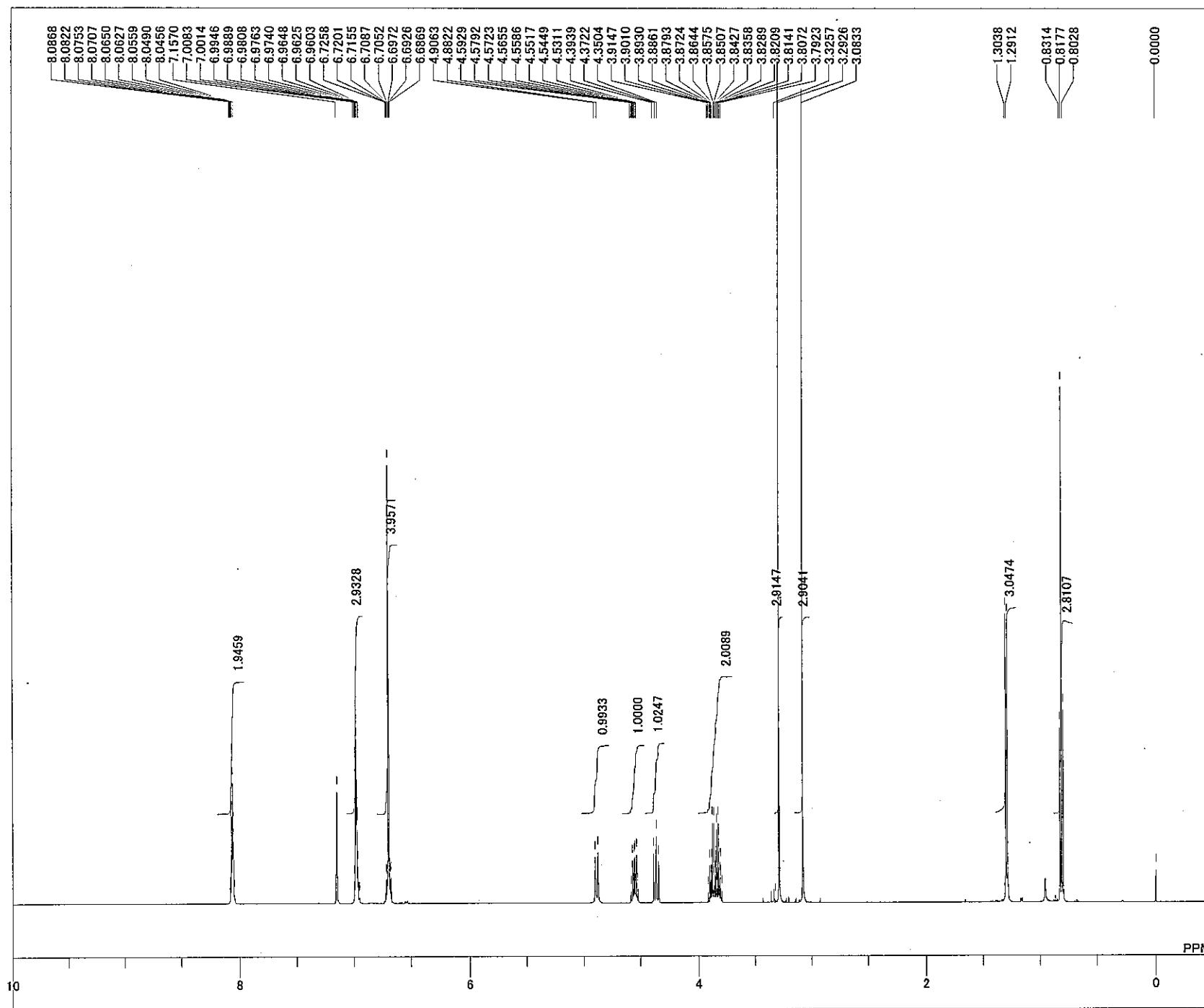




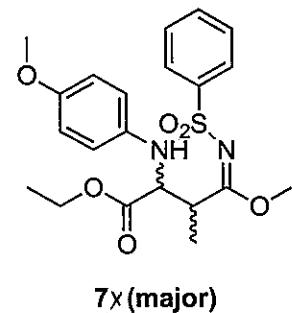
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2800product-down
DATIM 30-11-2007 12:12:40
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 40961
FREQU 59186.51 Hz
SCANS 1024
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 2.83 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL₃
EXREF 225.36 ppm
BF 0.12 Hz
RGAIN 58



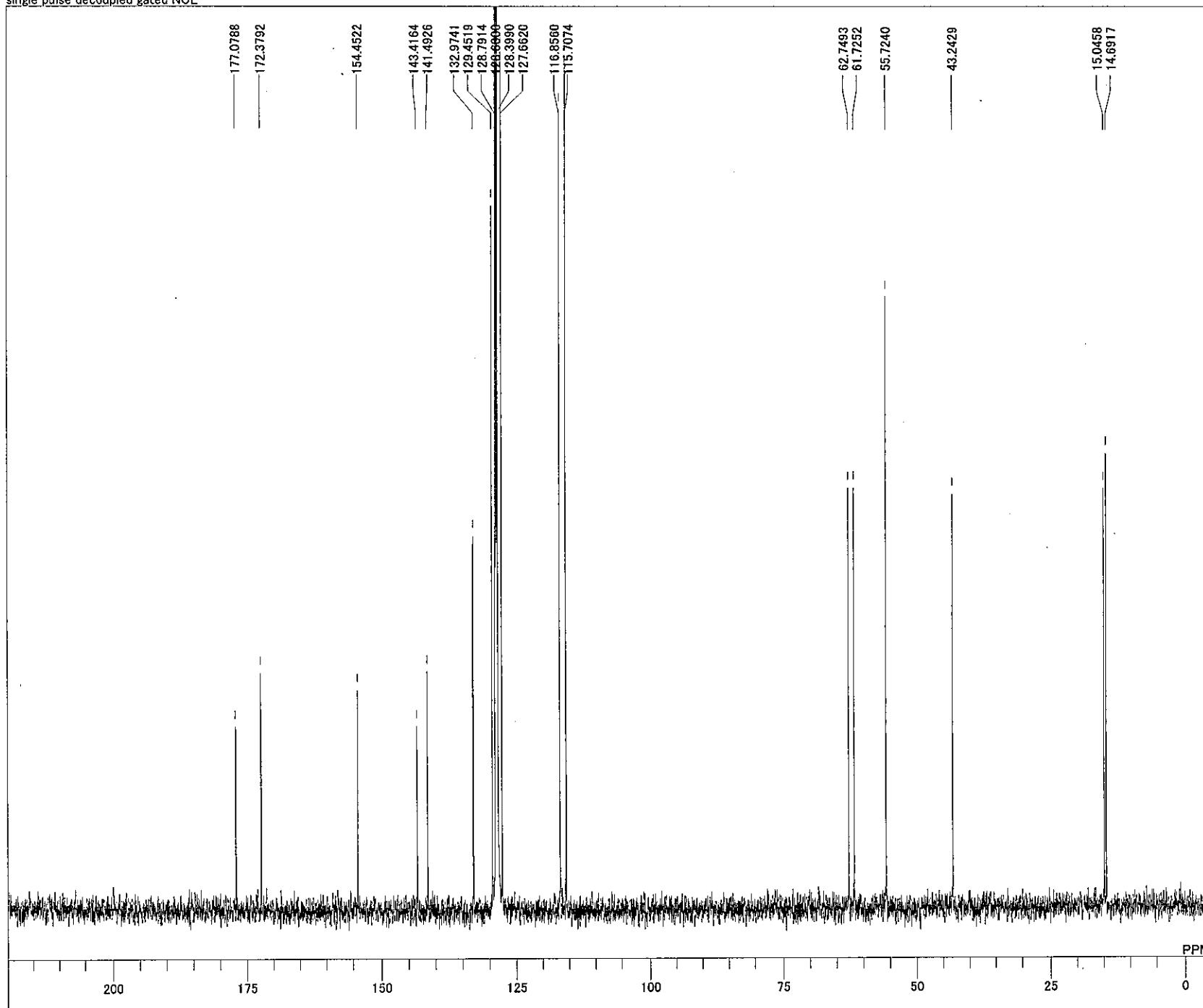
syn-17w



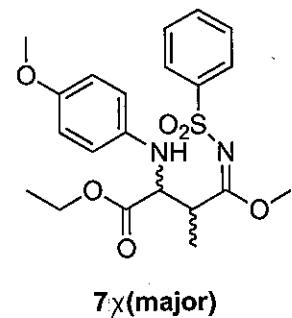
DFILE C:\Documents and Settings\All Users\Docu
COMNT
DATIM 05-07-2007 14:52:06
OBNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13120
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.1 c
SLVNT C6D6
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30



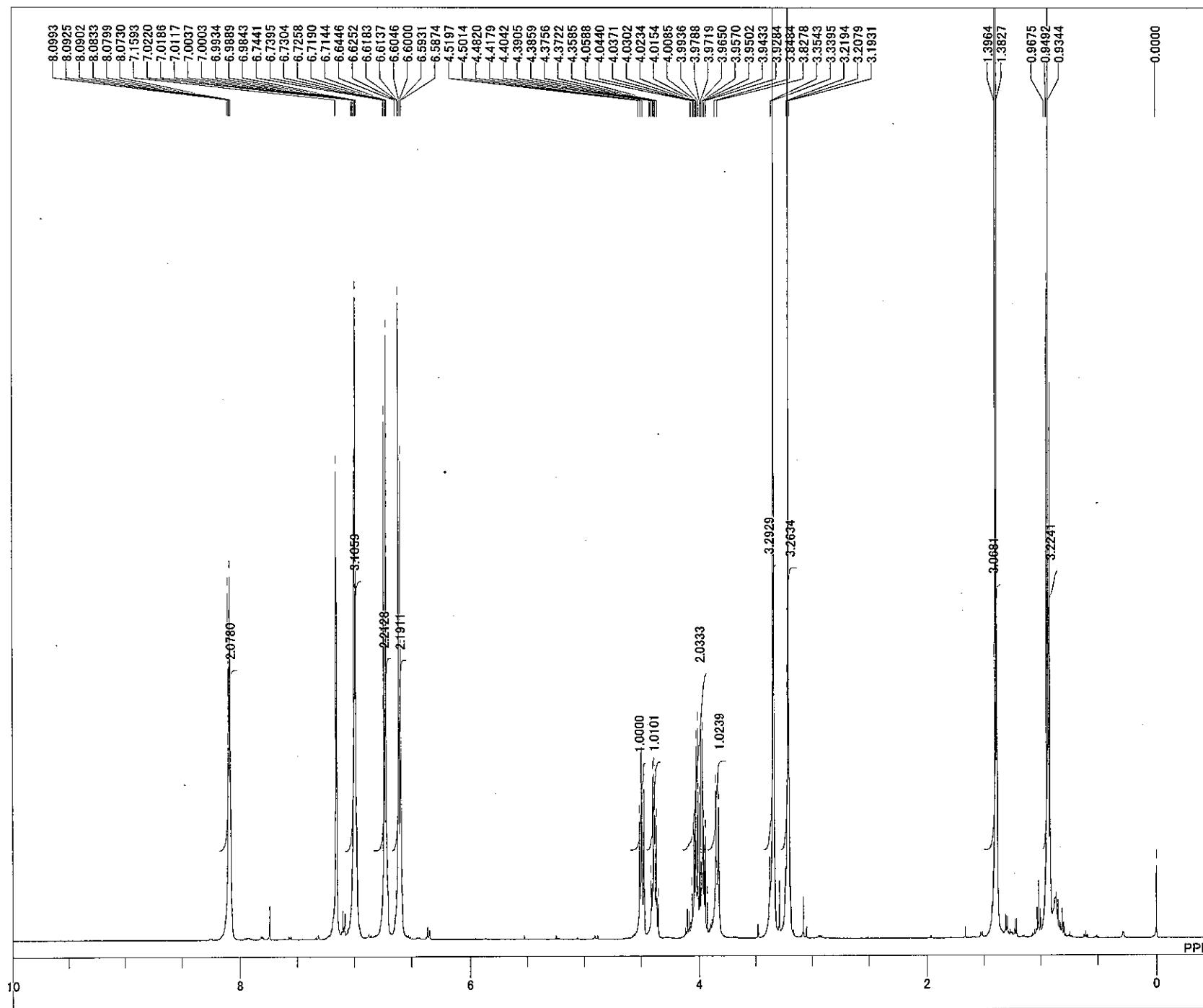
7x (major)



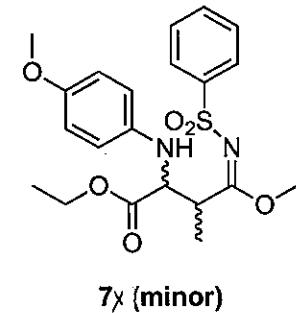
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
05-07-2007 16:02:13
¹³C
¹³C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
152
0.8389 sec
2.0000 sec
3.67 usec
¹H
25.7 c
¹³C
128.60 ppm
0.12 Hz
50



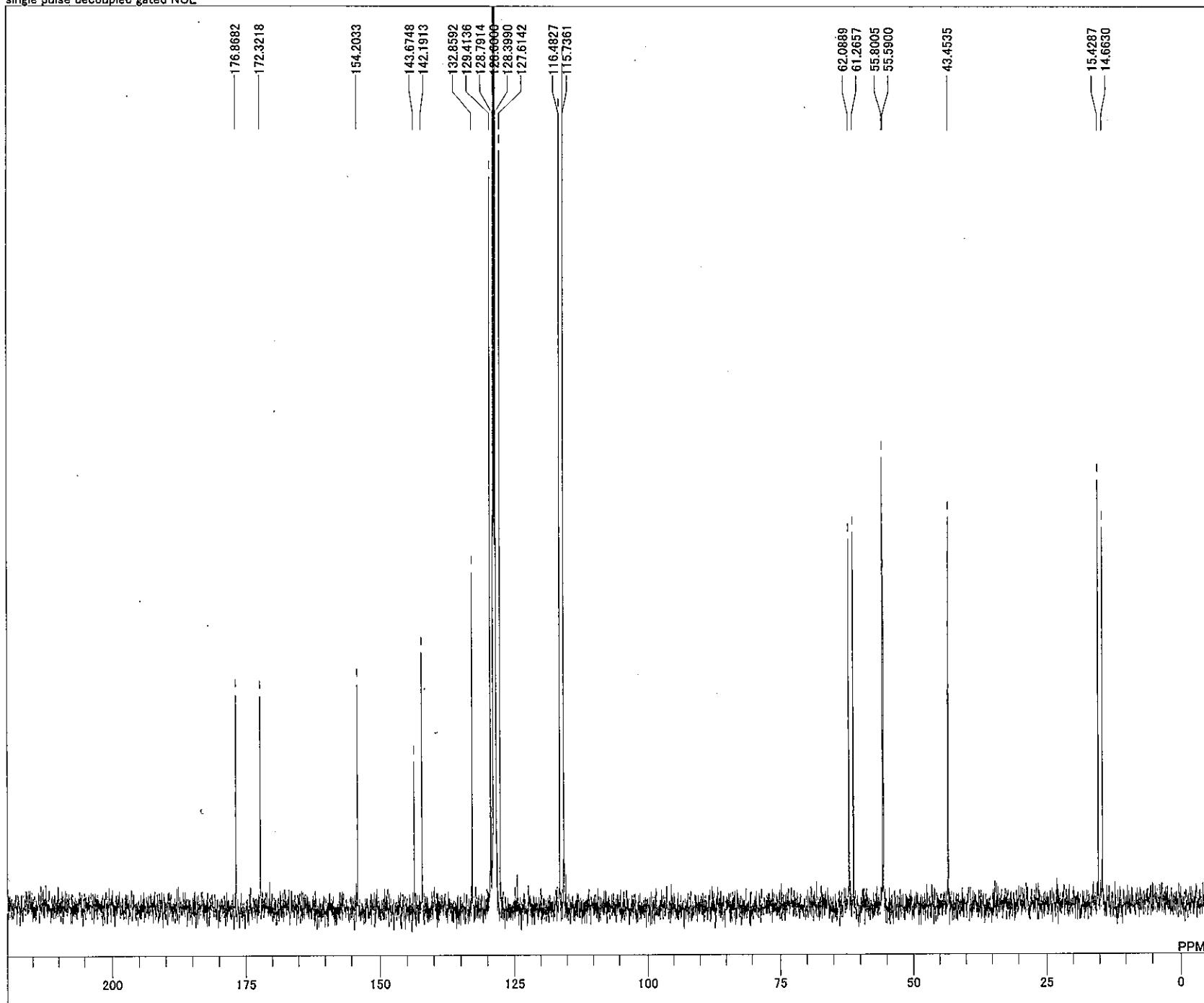
7x(major)



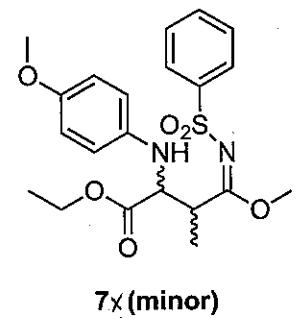
C:\Documents and Settings\All Users\Docu
05-07-2007 14:57:53
1H
1H NMR.ex2
495.13 MHz
4.38 kHz
9.64 Hz
13120
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
1H
25.1 c
C6D6
0.00 ppm
0.12 Hz
32



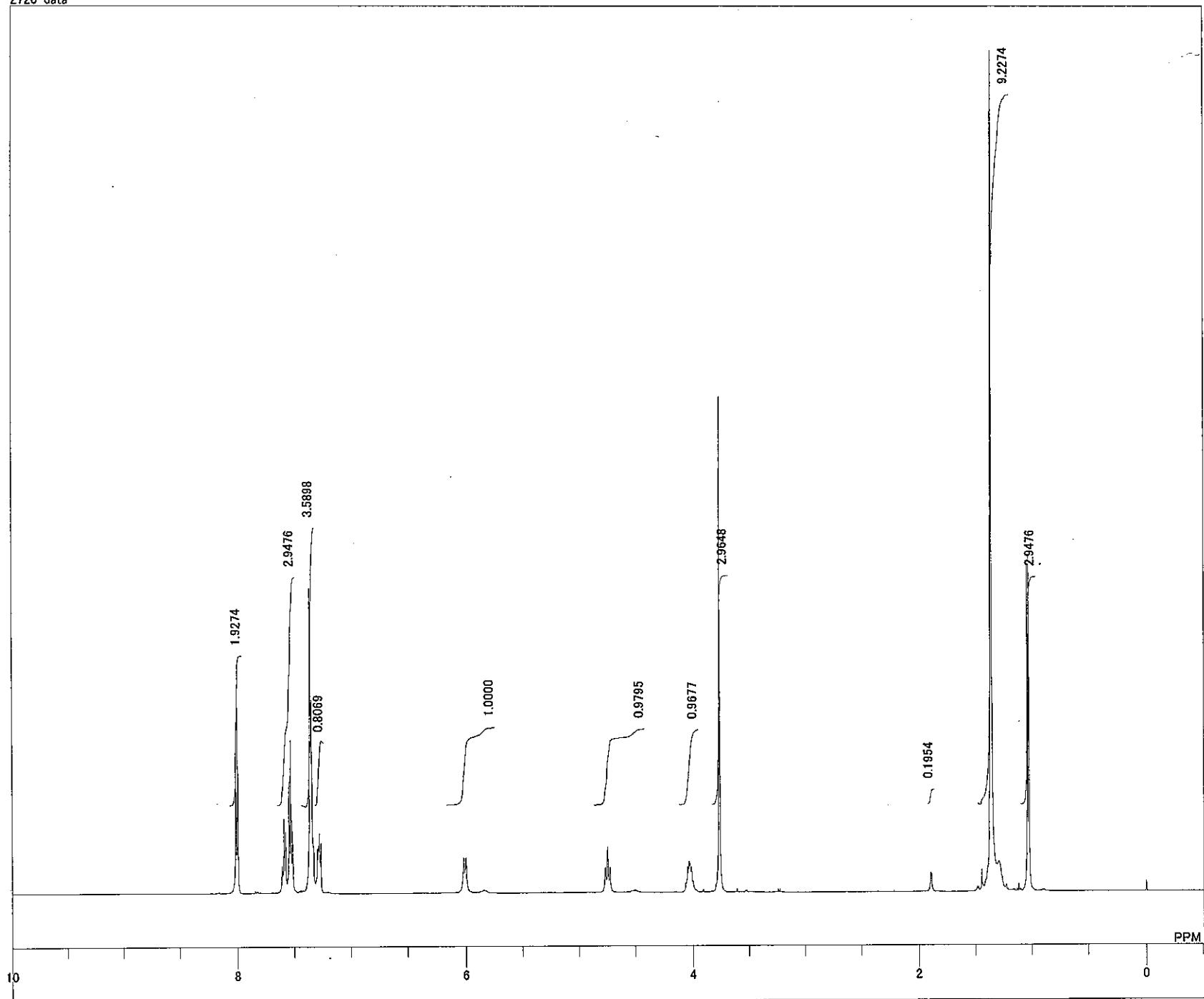
7x (minor)



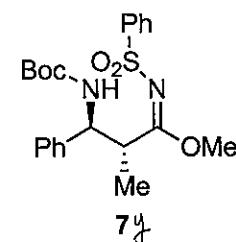
DFILE C:\Documents and Settings\All Users\Docu
COMINT single pulse decoupled gated NOE
DATIM 05-07-2007 16:13:47
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 kHz
OBFIN 6.00 Hz
POINT 26224
FREQU 31249.52 Hz
SCANS 149
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 25.8 c
SLVNT C6D6
EXREF 128.60 ppm
BF 0.12 Hz
RGAIN 50

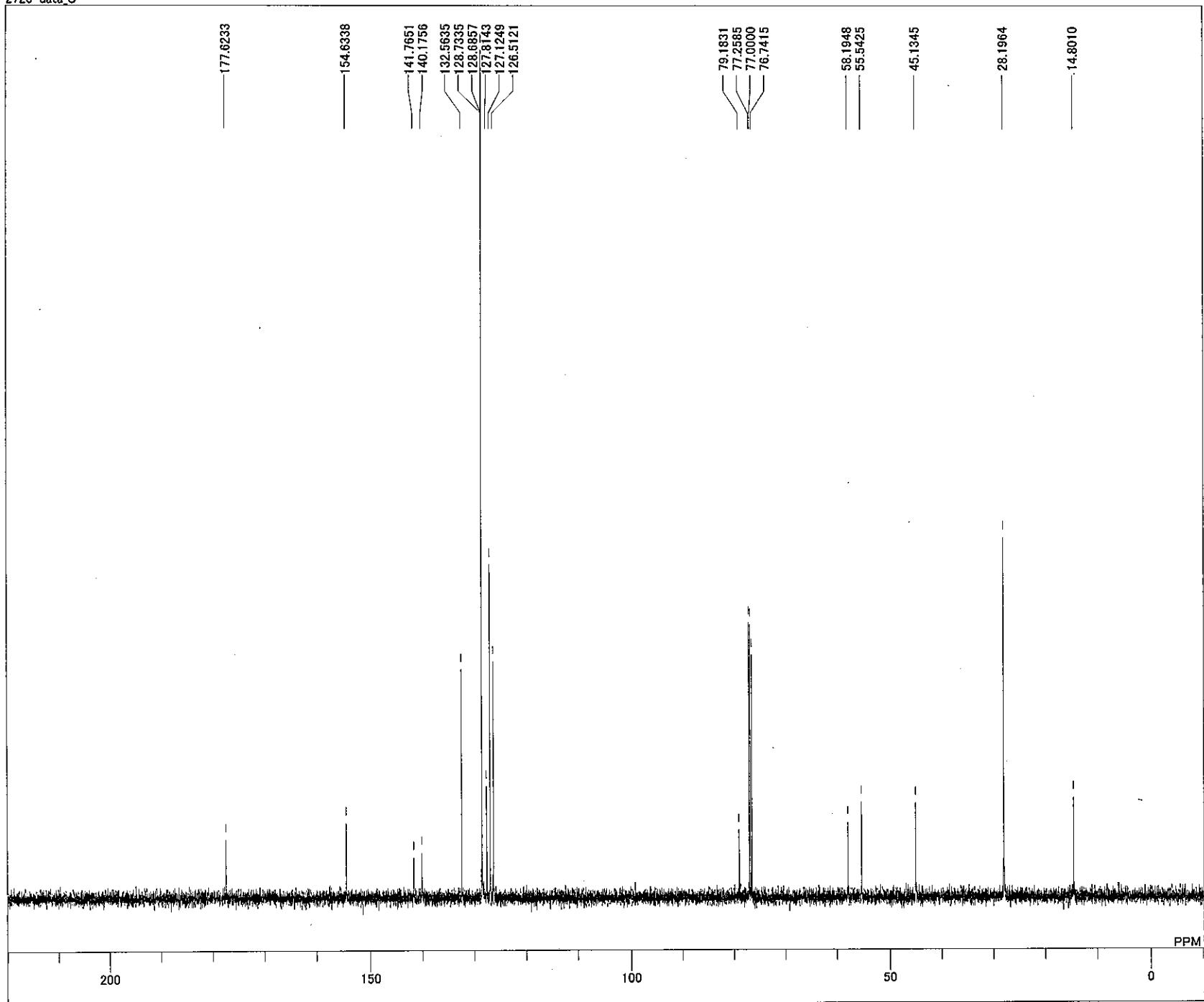


7x (minor)

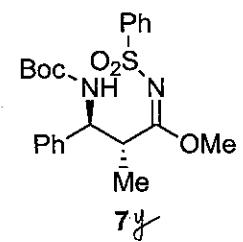


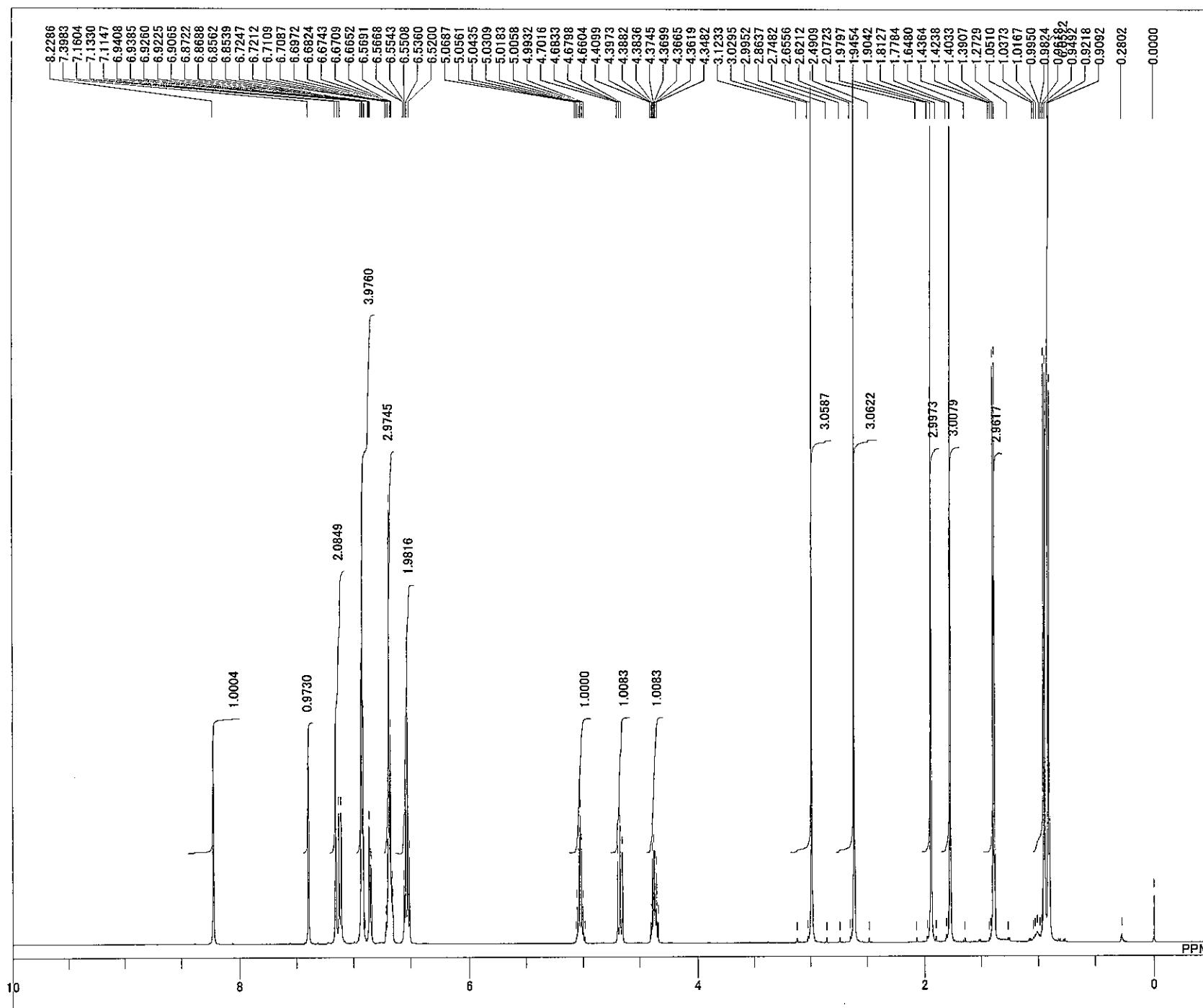
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2720-data
DATIM 11-08-2007 13:51:24
OBNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 29.7 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28



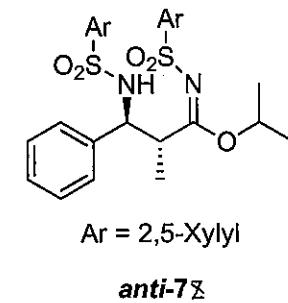


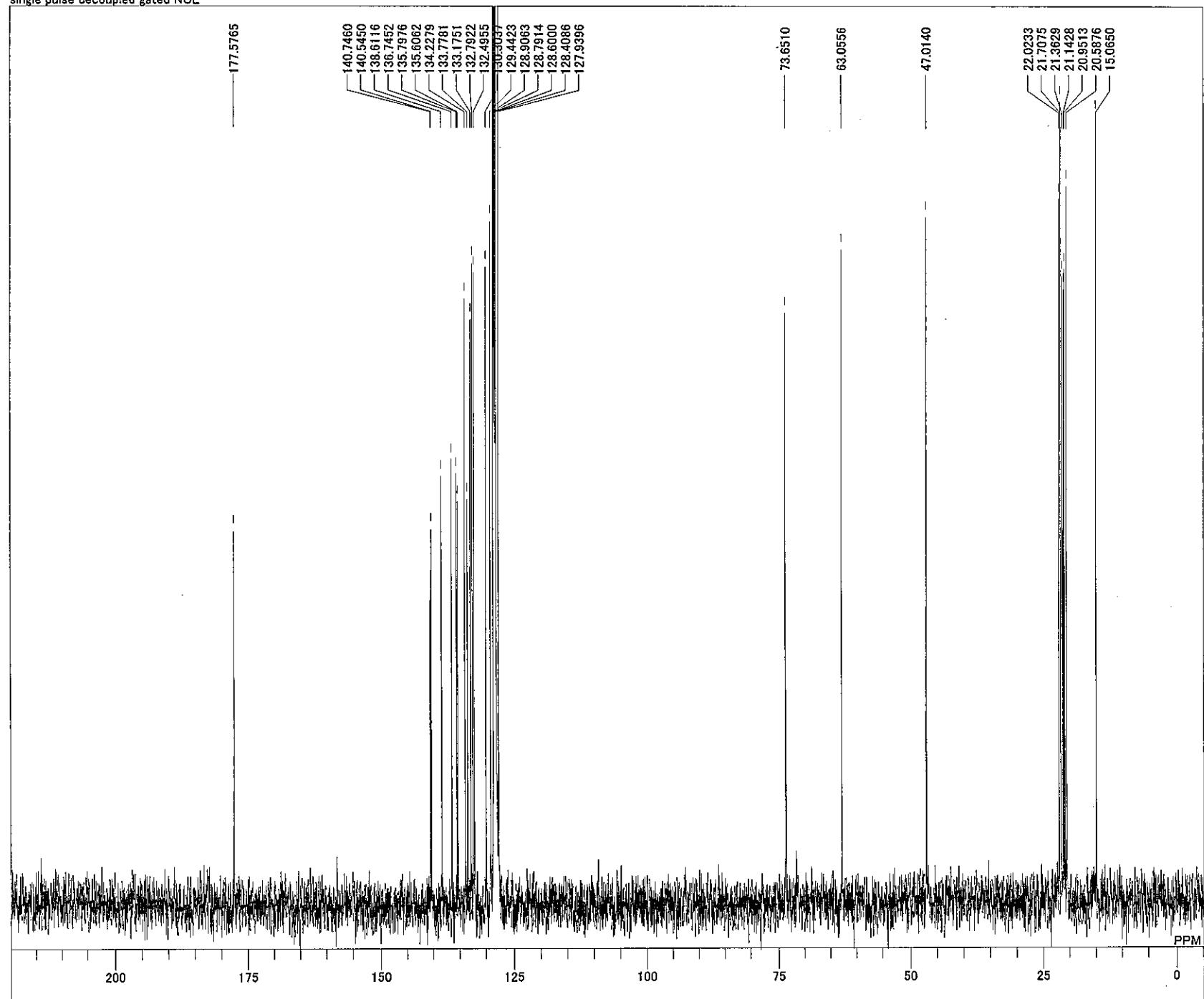
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2720-data_C
DATIM 11-08-2007 13:55:18
13C
OBNUC 13C NMR.ex2
EXMOD 124.51 MHz
OBFRQ 3.45 KHz
OBSET 6.00 Hz
OBFIN POINT 26214
FREQU 31249.52 Hz
SCANS 68
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 29.9 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



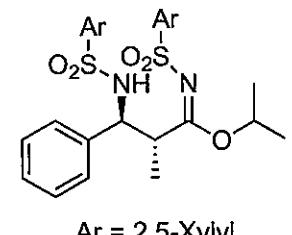


¥¥Eca¥data¥florian¥fb436prana-1.jdf
 DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRQ
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN
 08-08-2007 20:20:50
 1H
 1H NMR.ex2
 495.13 MHz
 4.38 KHz
 9.64 Hz
 16400
 9286.78 Hz
 8
 1.7642 sec
 5.0000 sec
 6.50 usec
 1H
 27.8 c
 C6D6
 0.00 ppm
 0.12 Hz
 30

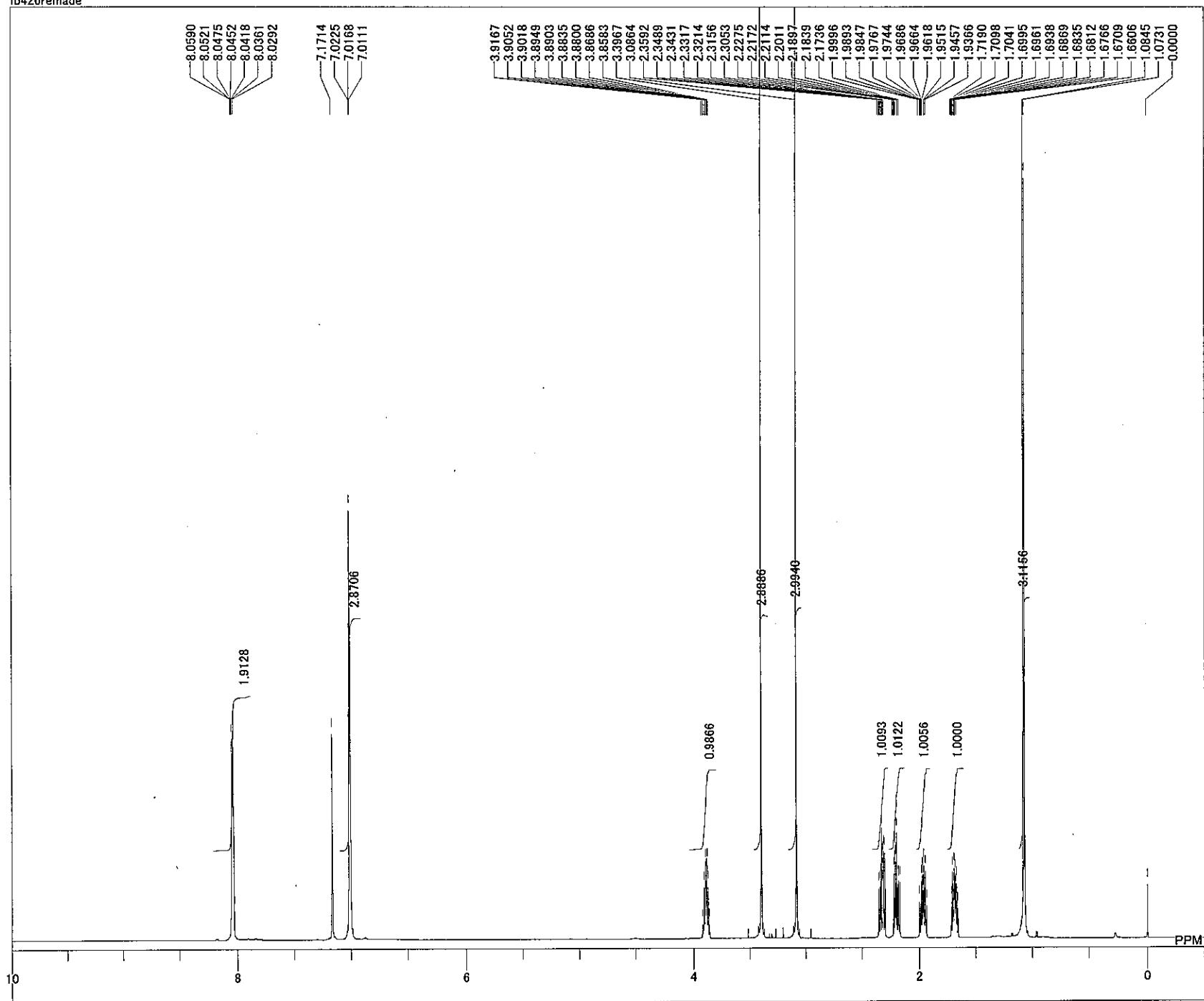




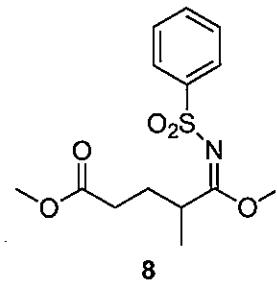
DFILE C:\Documents and Settings\All Users\Docu
COMNT single pulse decoupled gated NOE
DATIM 08-08-2007 20:25:10
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26224
FREQU 31249.52 Hz
SCANS 67
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 28.1 c
SLVNT C6D6
EXREF 128.60 ppm
BF 0.12 Hz
RGAIN 50

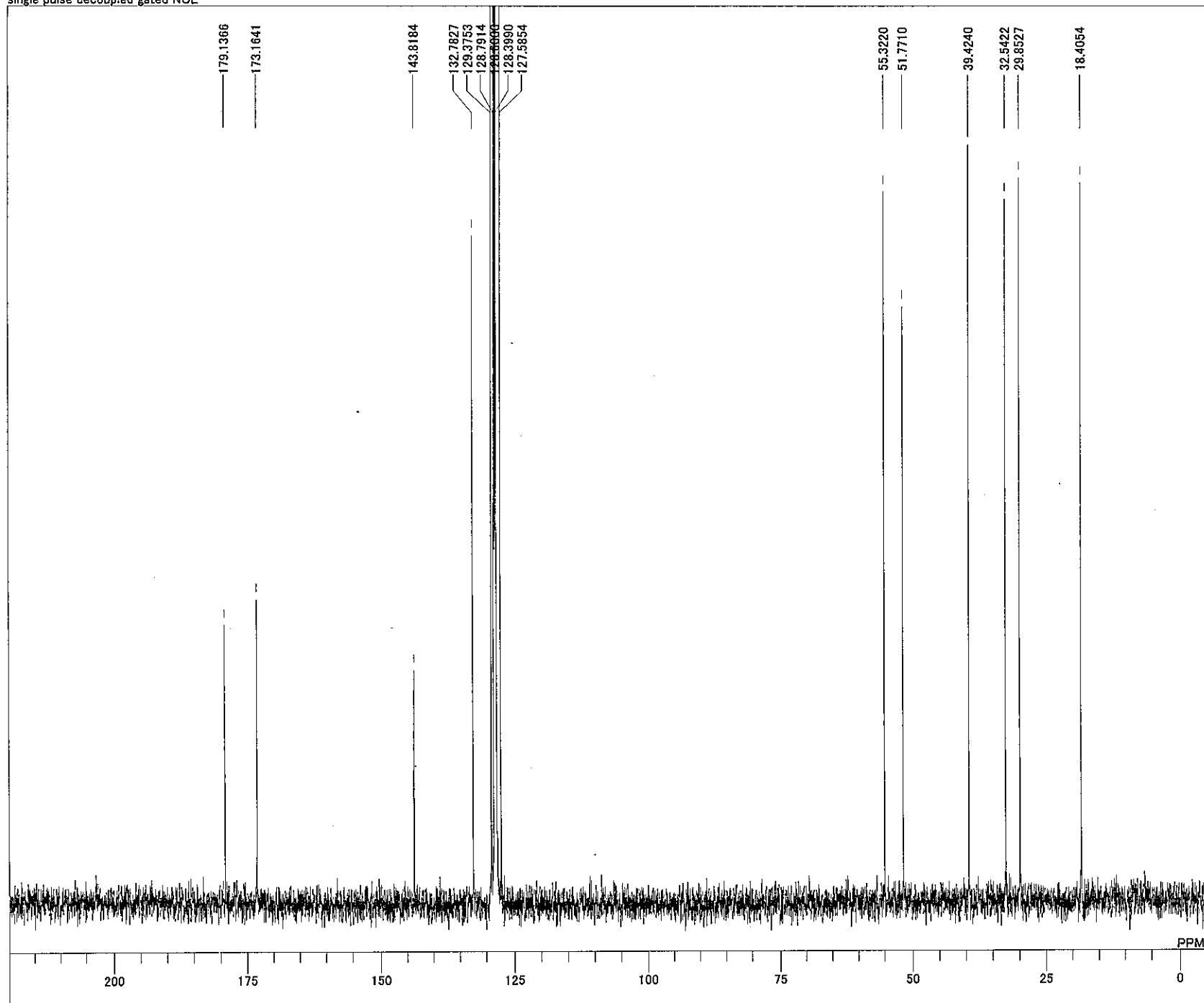


anti-7 α

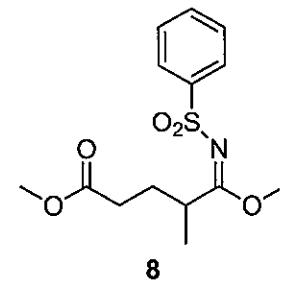


C:\Documents and Settings\ALICE2\デスク
fb426remade
28-08-2007 21:59:22
1H
single_pulse.ex2
600.17 MHz
5.30 kHz
5.47 Hz
20480
14076.79 Hz
16
1.4549 sec
4.0000 sec
9.10 usec
1H
29.5 c
C6D6
0.00 ppm
0.12 Hz
36

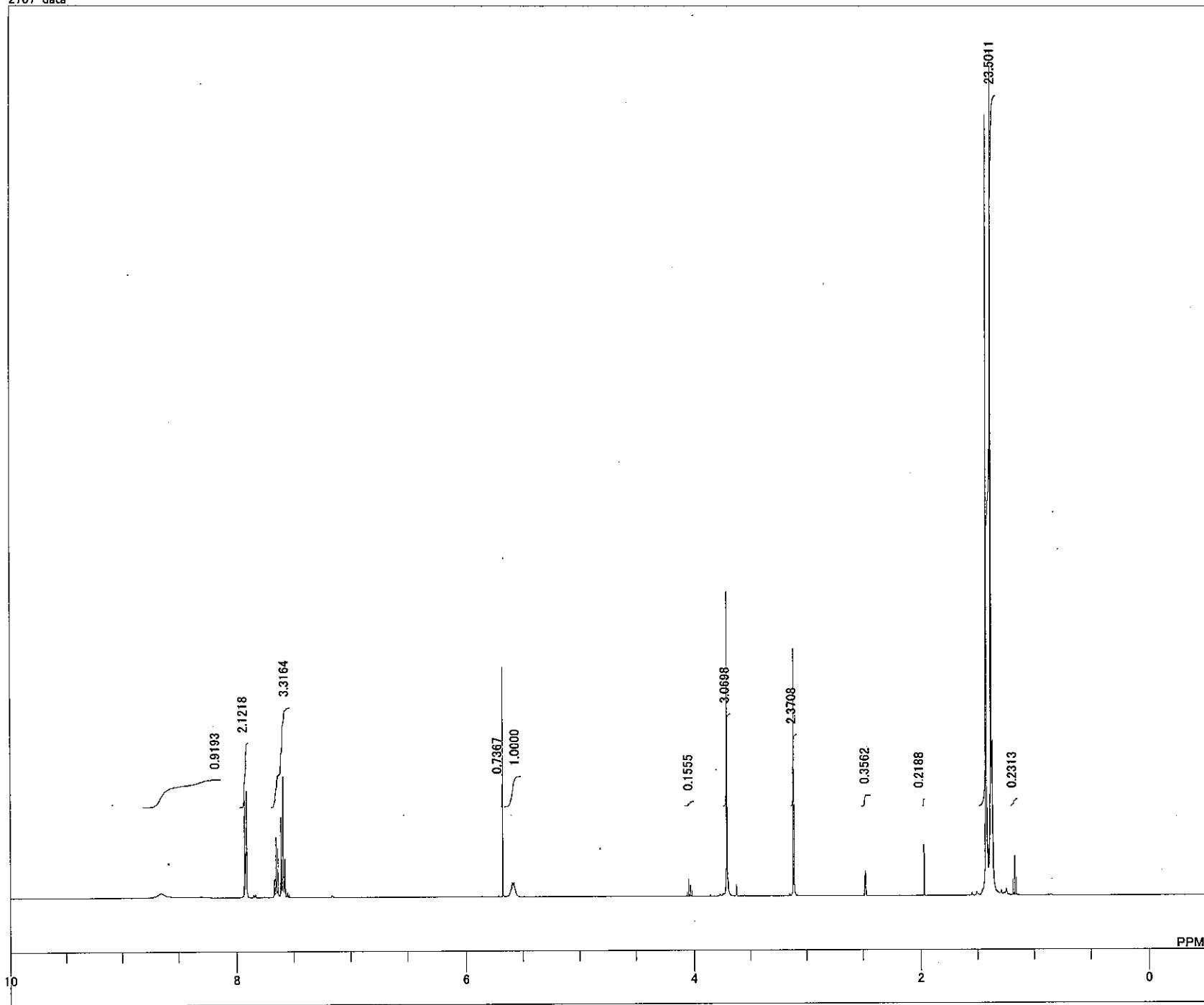




DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\Documents and Settings\All Users\Docu
single pulse decoupled gated NOE
01-07-2007 16:06:25
13C
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26224
31249.52 Hz
77
0.8389 sec
2.0000 sec
3.67 usec
1H
25.4 c
C6D6
128.60 ppm
1.20 Hz
50

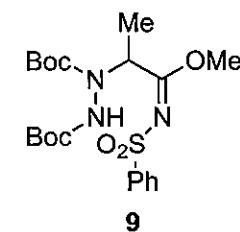


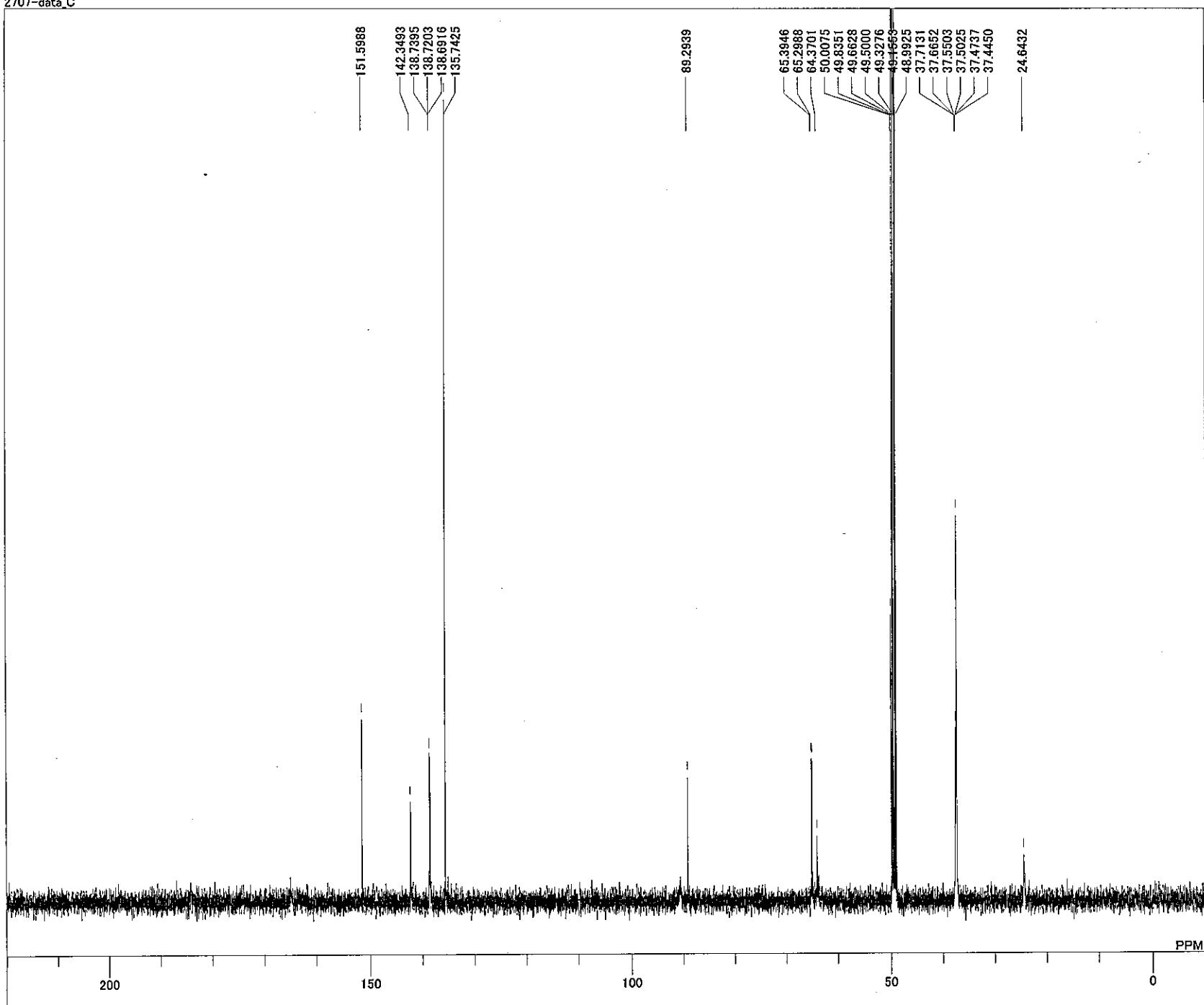
140



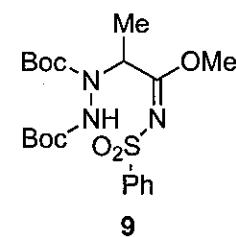
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

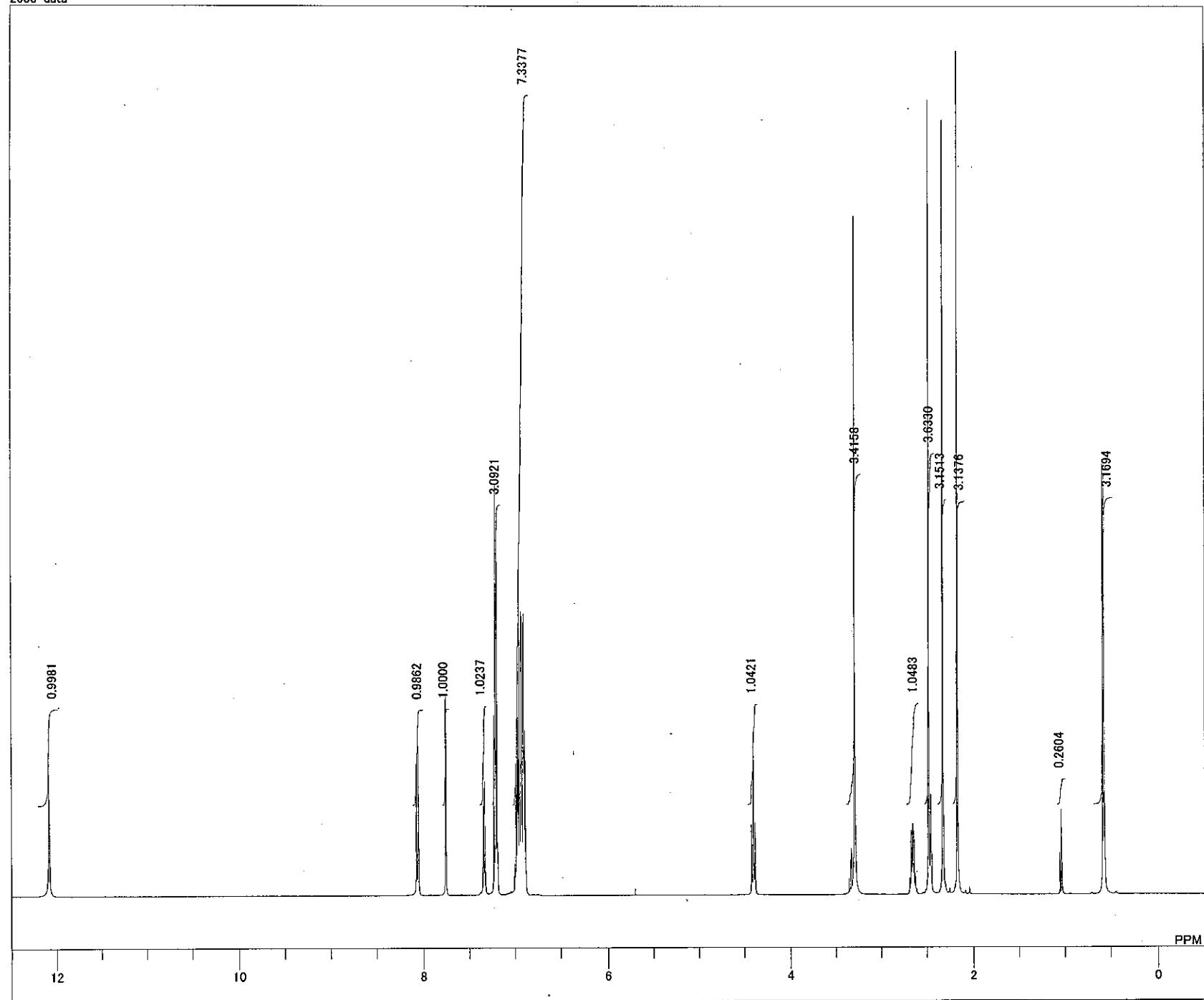
2707-data
11-08-2007 18:51:58
1H
1H NMR.ex2
495.13 MHz
4.38 kHz
9.64 Hz
13107
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec
70.0 c
DMSO
2.49 ppm
0.12 Hz
36



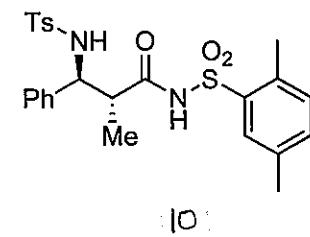


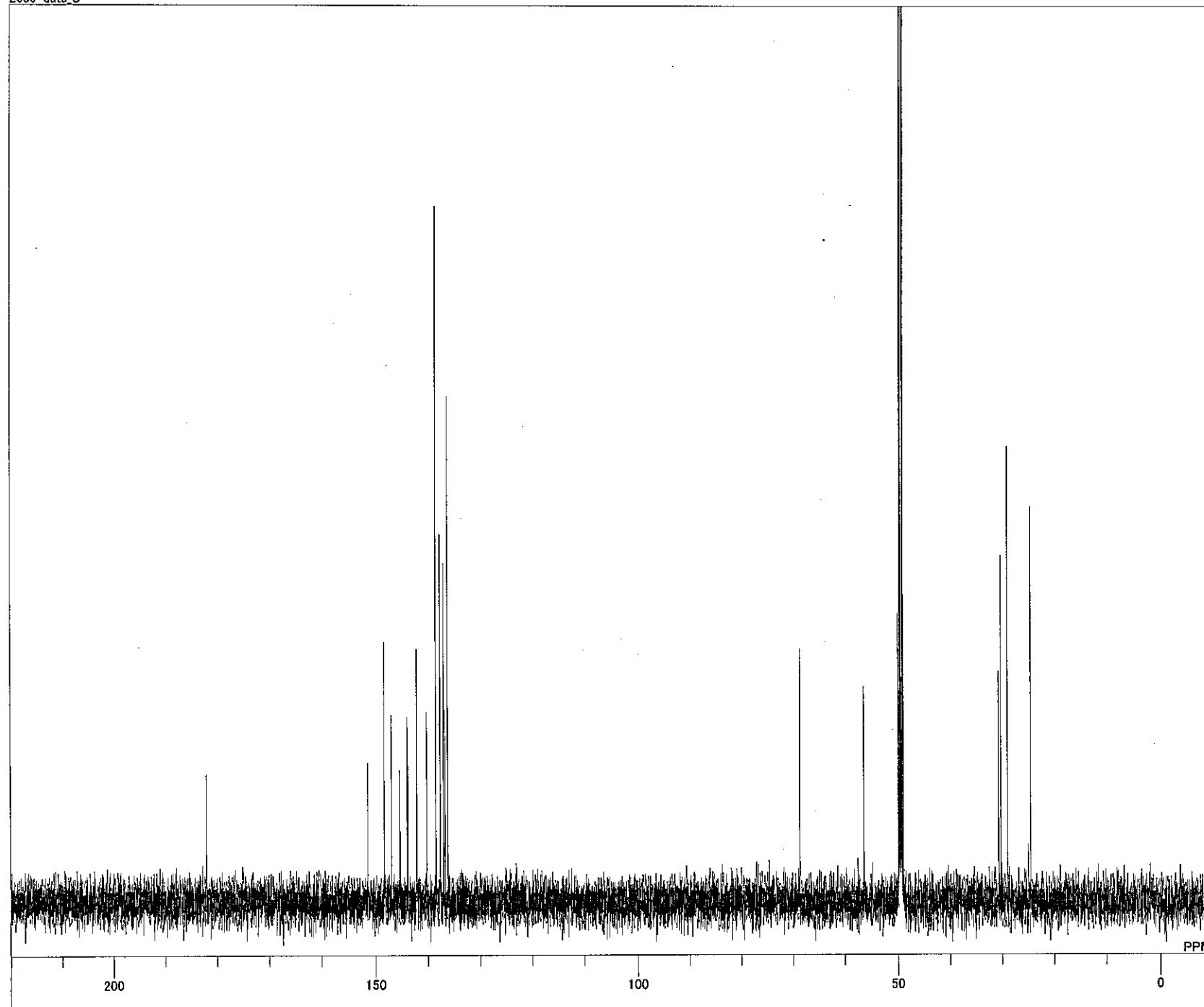
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2707-data_C
DATIM 11-08-2007 19:39:58
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 kHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 1000
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 70.0 c
SLVNT DMSO
EXREF 49.50 ppm
BF 0.12 Hz
RGAIN 60



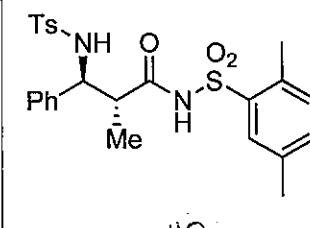


DFILE C:\Documents and Settings\All Users\Docu
COMNT 2685-data
DATIM 11-08-2007 18:34:30
1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
1H 29.3 c
IRNUC DMSO
CTEMP 3.30 ppm
SLVNT 0.12 Hz
EXREF 34
BF RGAIN





C:\Documents and Settings\All Users\Docu
2685-data_C
11-08-2007 18:44:60
13C
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26214
31249.52 Hz
124
0.8389 sec
2.0000 sec
3.67 usec
1H
29.7 c
DMSO
49.50 ppm
0.12 Hz
RGAIN 56



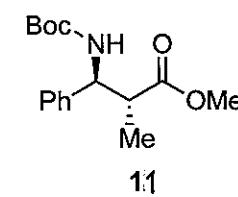
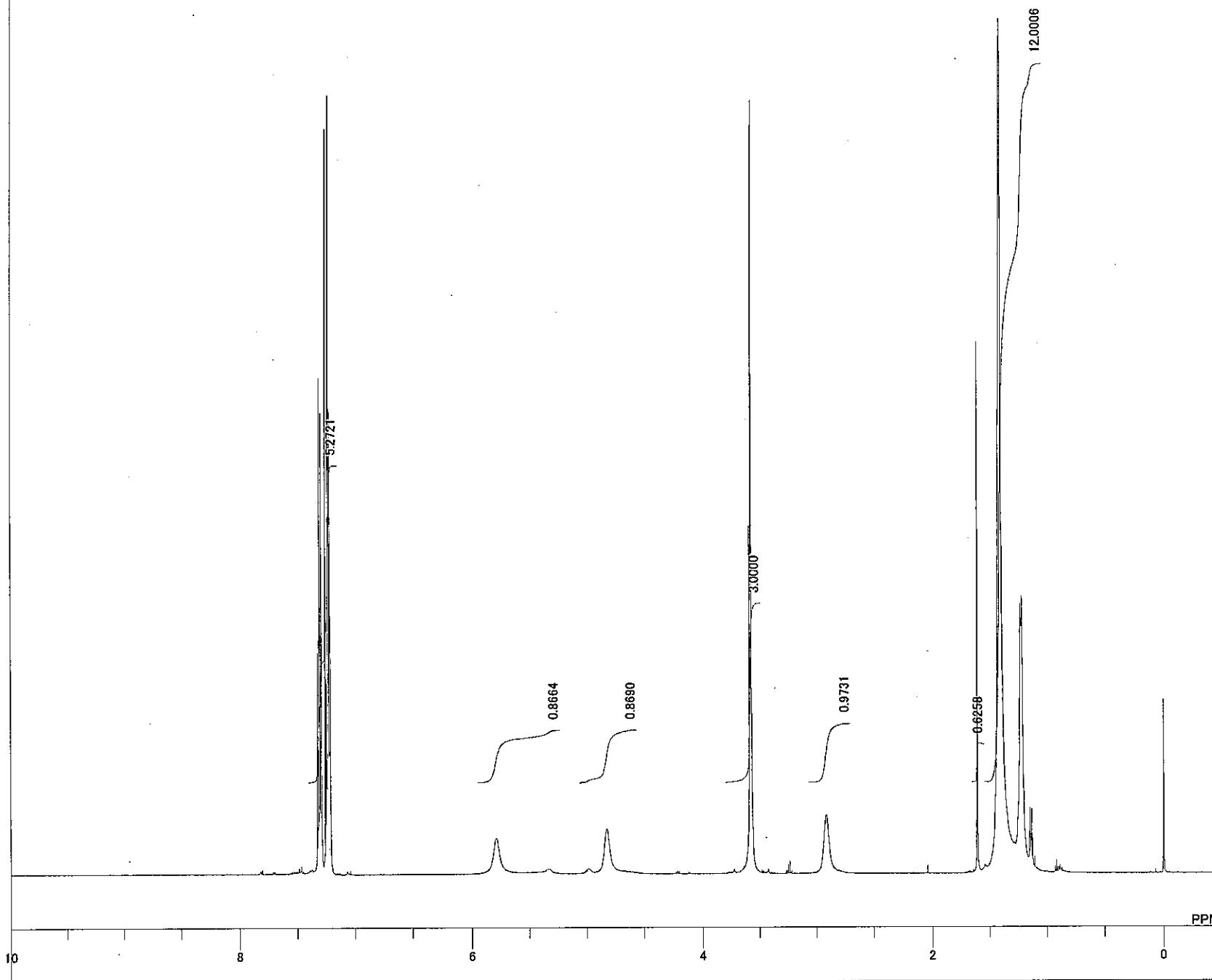
¹³O

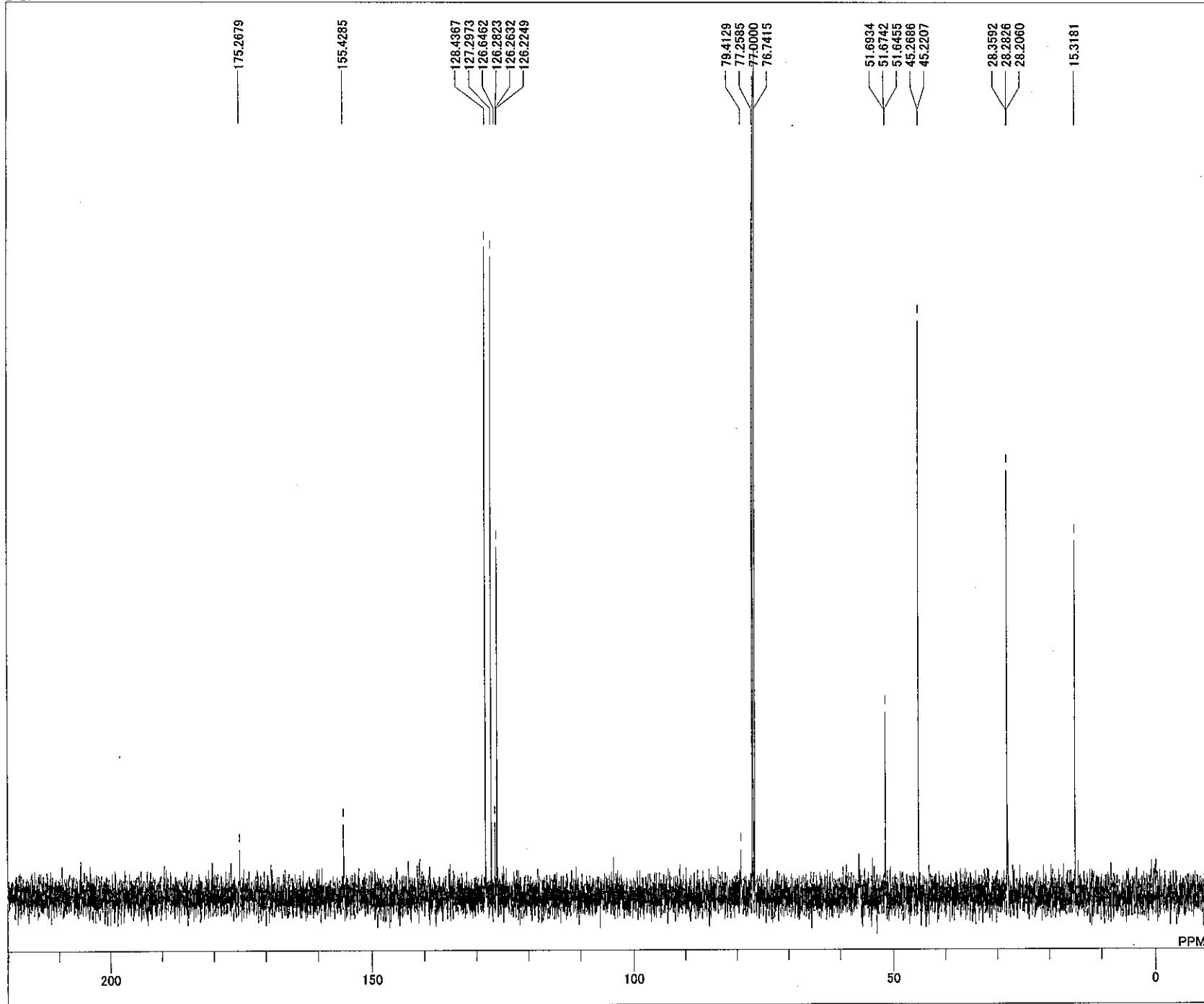
C:\Documents and Settings\All Users\Docu
2723\lowconcentration
06-08-2007 21:19:23

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

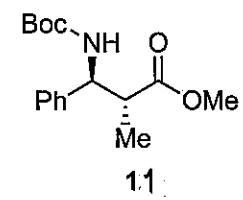
1H NMR.ex2
495.13 MHz
4.38 KHz
9.64 Hz
13107
7429.31 Hz
8
1.7642 sec
5.0000 sec
6.50 usec

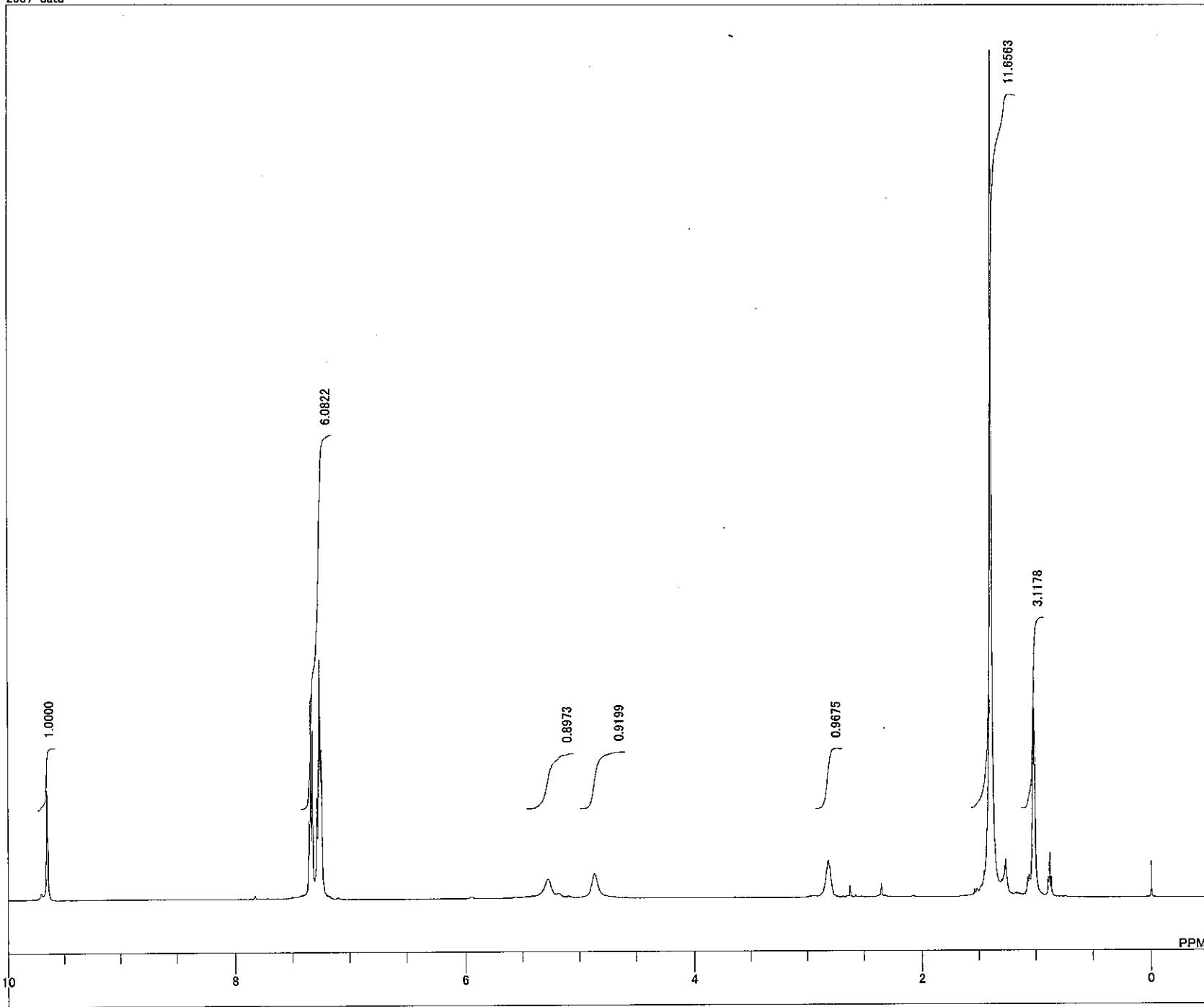
1H
28.1 c
CDCL₃
0.00 ppm
0.12 Hz
40



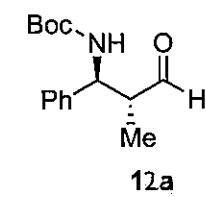


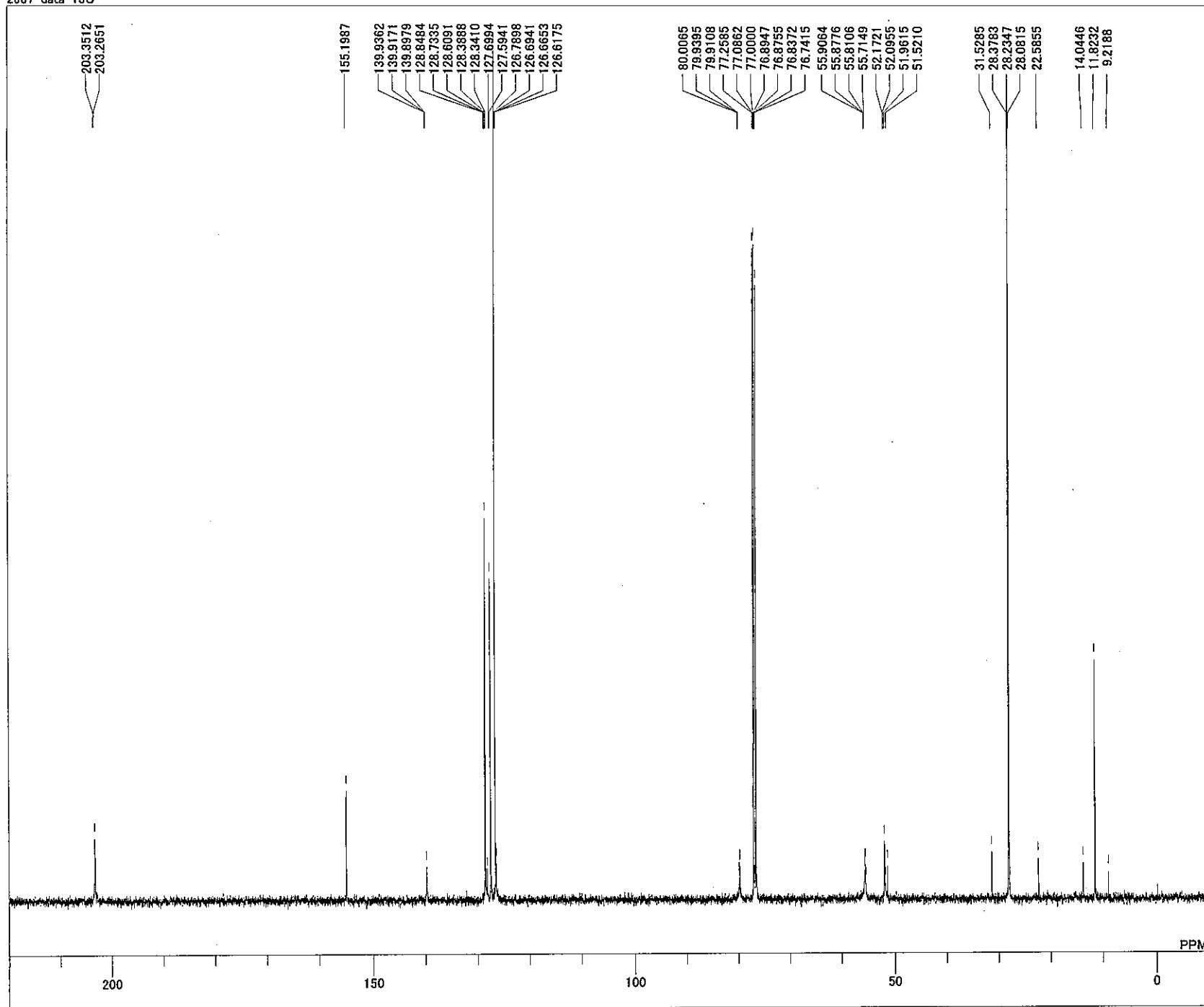
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
C:\Documents and Settings\All Users\Docu
2723
06-08-2007 13:24:51
13C
13C NMR.ex2
124.51 MHz
3.45 kHz
6.00 Hz
26214
31249.52 Hz
88
0.8389 sec
2.0000 sec
3.67 usec
1H
28.8 c
CDCL₃
77.00 ppm
0.12 Hz
52



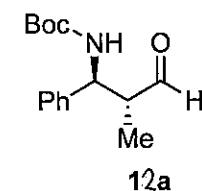


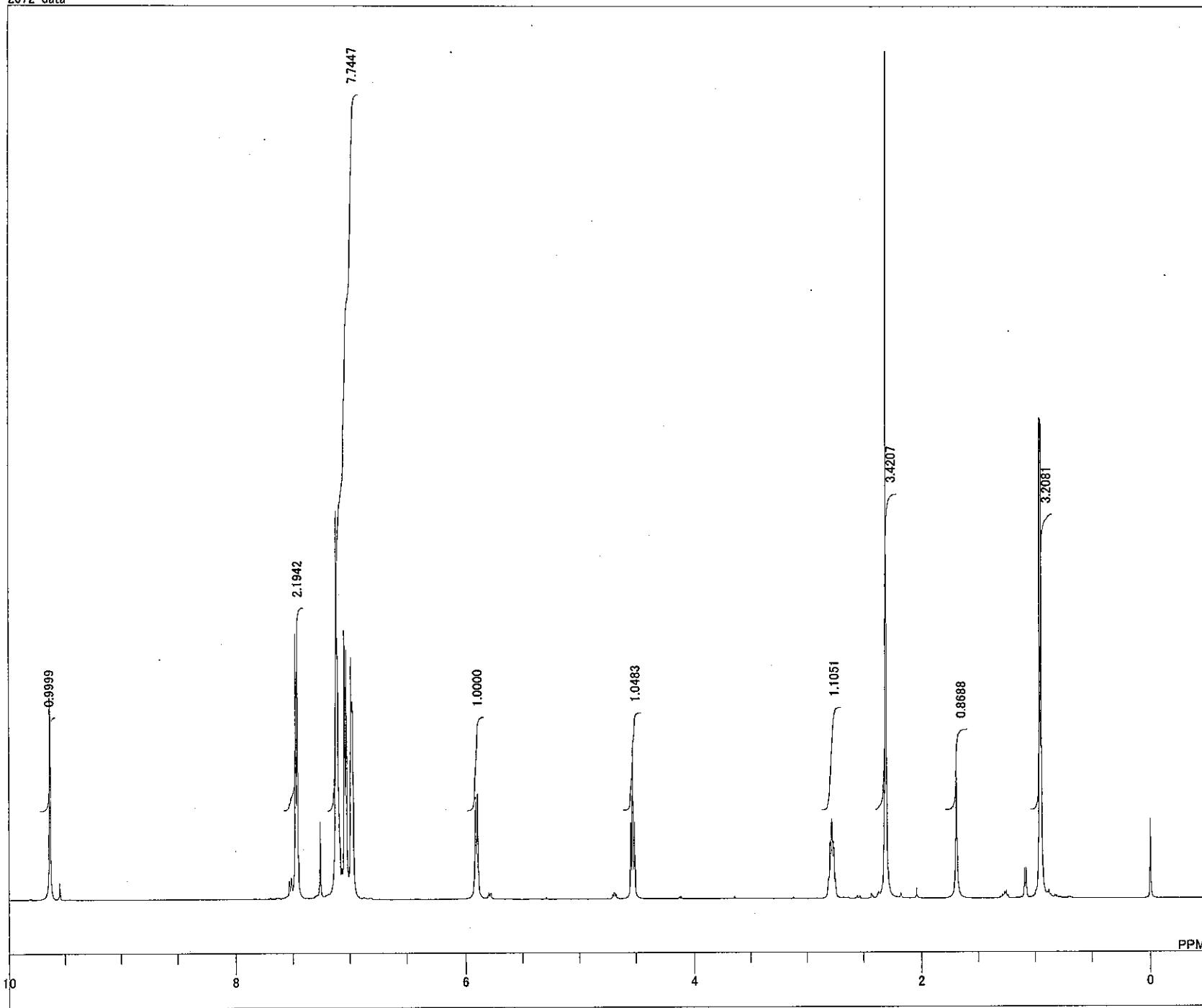
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2687-data
DATIM 21-08-2007 14:36:42
DNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 29.0 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 32



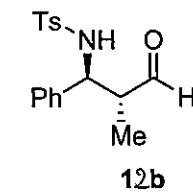


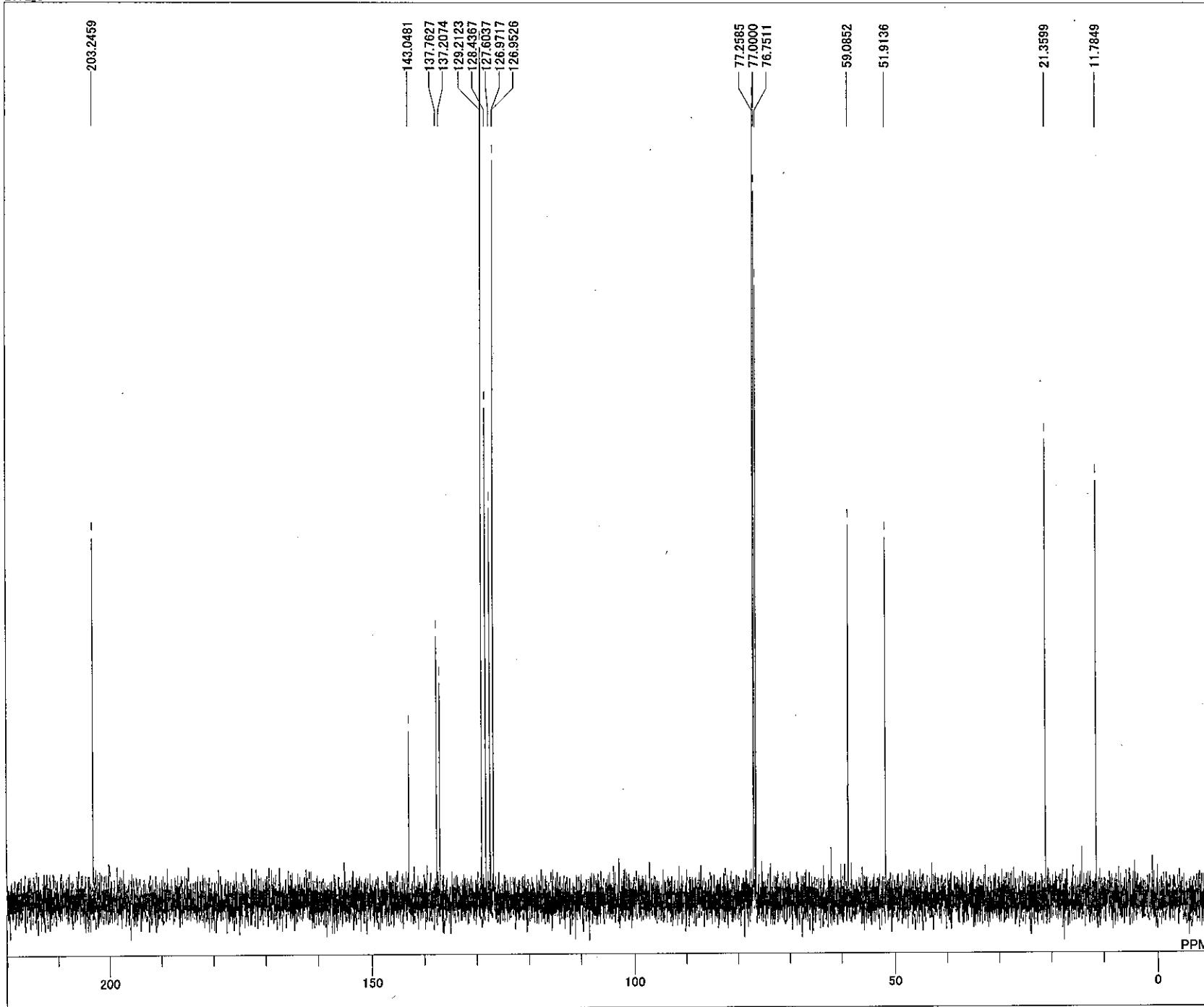
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2687-data-13C
DATIM 21-08-2007 15:16:32
13C
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU 31249.52 Hz
SCANS 817
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
1H
IRNUC 29.4 c
CTEMP CDCL₃
SLVNT 77.00 ppm
EXREF 1.20 Hz
BF 52
RGAIN



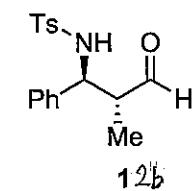


DFILE C:\Documents and Settings\All Users\Docu
COMNT 2672-data
DATIM 21-08-2007 14:30:26
1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
1H
IRNUC 28.9 c
CTEMP CDCL₃
SLVNT 0.00 ppm
EXREF BF 1.20 Hz
RGAIN 40

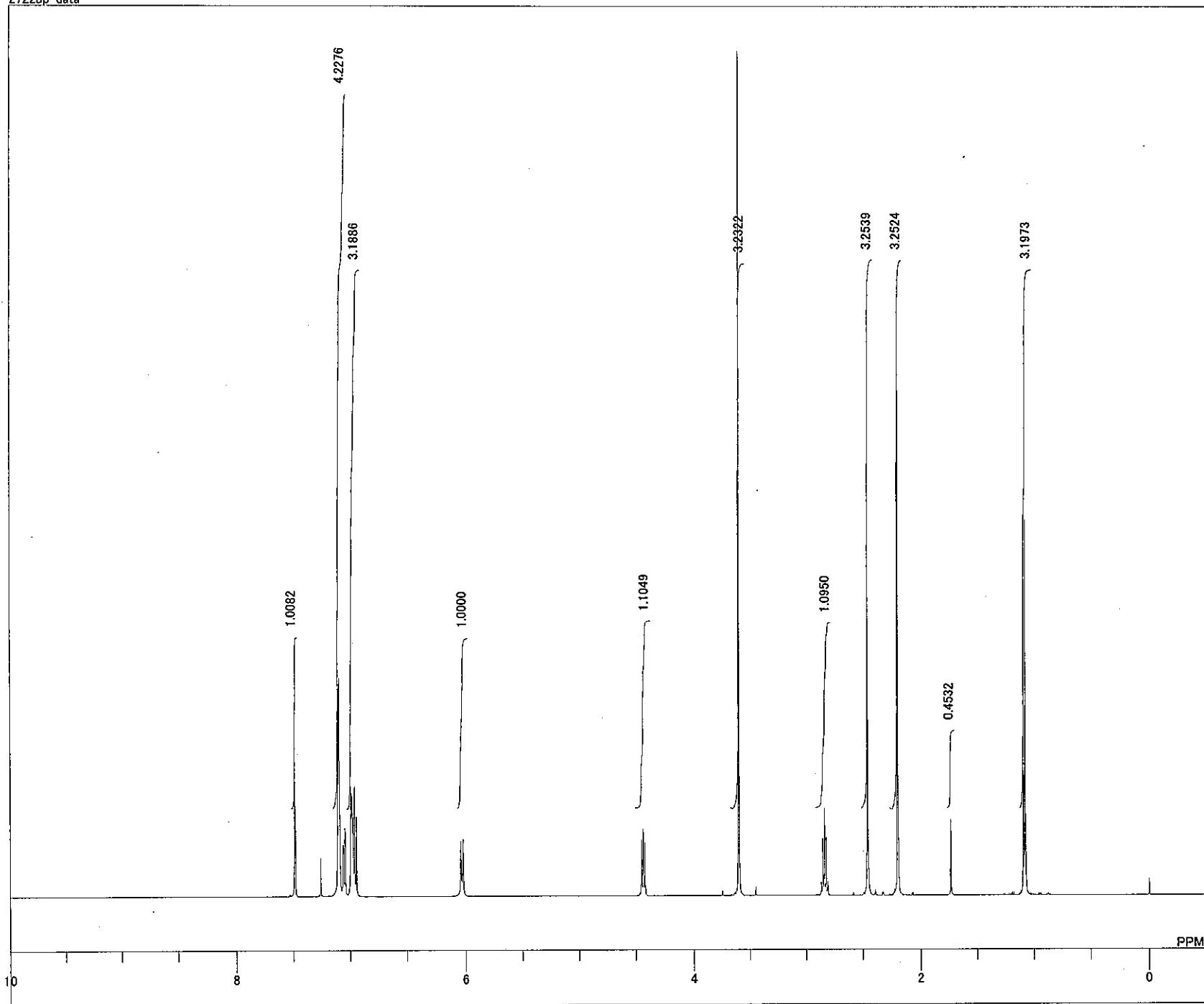




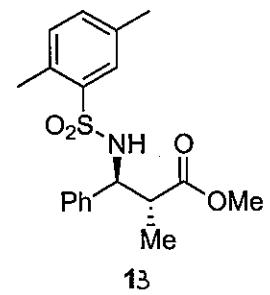
DFILE C:\Documents and Settings\All Users\Docu
COMNT 2672_C
DATIM 11-07-2007 10:33:42
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 64
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

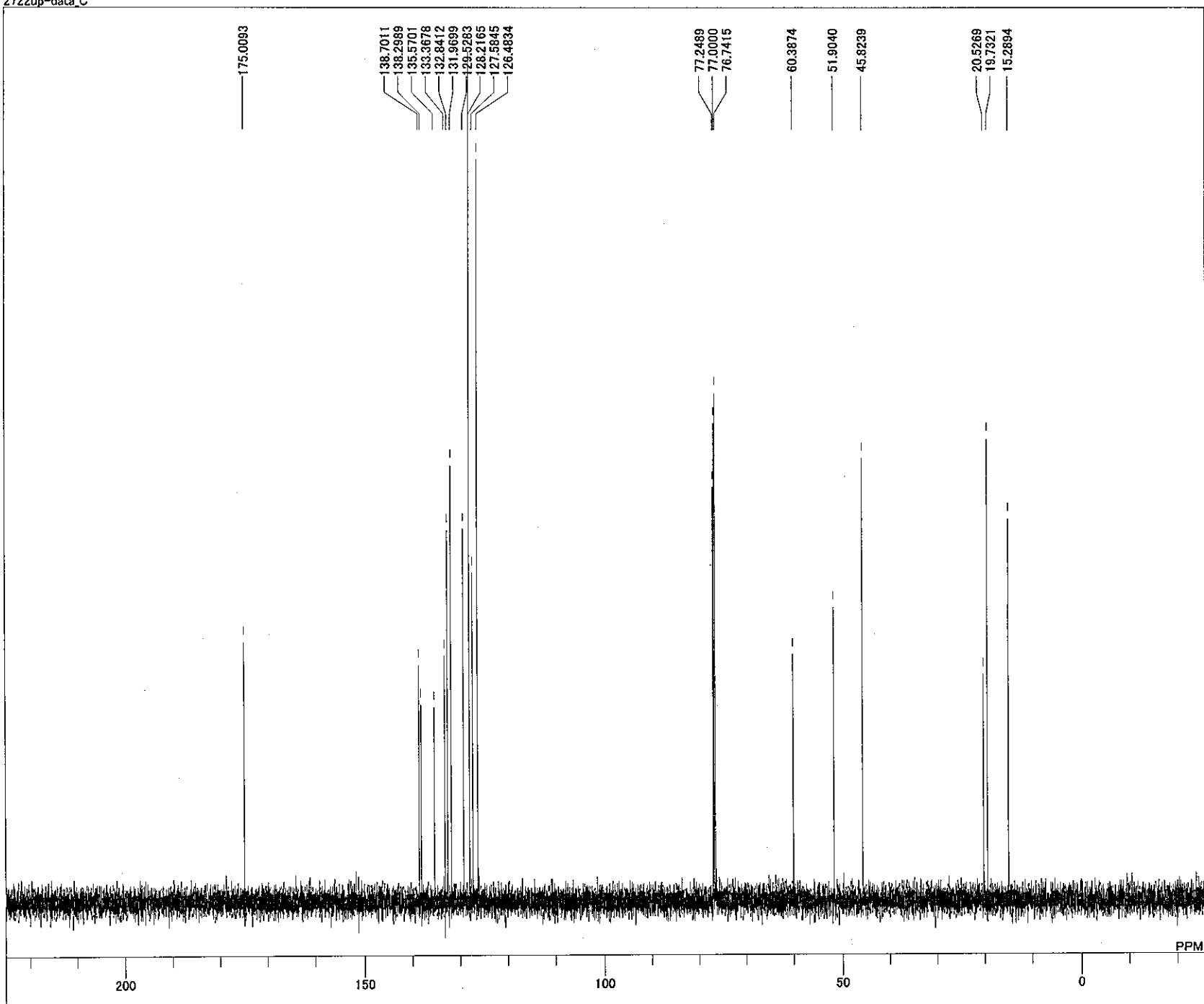


150



DFILE C:\Documents and Settings\All Users\Docu
COMNT 2722up-data
DATIM 11-08-2007 13:29:50
OBNUC 1H
EXMOD 1H NMR.ex2
OBFRQ 495.13 MHz
OBSET 4.38 kHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 29.6 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34





DFILE C:\Documents and Settings\All Users\Docu
COMNT 2722up-data_C
DATIM 11-08-2007 13:33:34
OBNUC 13C
EXMOD 13C NMR.ex2
OBFRQ 124.51 MHz
OBSET 3.45 kHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 63
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 29.8 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 52

