(Supporting Information)

Asymmetric synthesis of (α*R*)-polyfluoroalkylated prolinols based on the perfluoroalkyl-induced highly stereoselective reduction of perfluoroalkyl *N*-Boc-pyrrolidyl ketones

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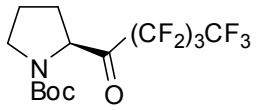
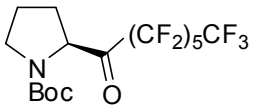
Experimental section

(*S*)-*tert*-butyl 2-(2,2,3,3,4,4,5,5,5-nonafluoropentanoyl)pyrrolidine-1-carboxylate (1a).



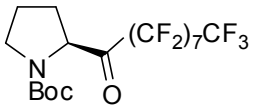
To a solution of methyl (*S*)-*N*-(*tert*-butoxycarbonyl)pyrrolidine-2-carboxylate (0.461 g, 2 mmol) in dry Et_2O (15 ml) was added perfluorobutyl iodide (2.076 g, 6 mmol) at room temperature under argon. After the mixture was stirred at room temperature for 20 min, methyllithium-lithium bromide complex (1.5 M Et_2O solution, 4.0 ml, 6 mmol) was added dropwise and allowed to react for 3 h at -78 °C. The reaction mixture was quenched with NH_4Cl-10 % HCl (v/v = 1:1) aq solution (30 ml), and then subjected to extraction with Et_2O (30 ml×3). The organic layer was washed with brine (70 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-Et_2O=2:1) gave **1a** (78%, 0.647 g). *Rf* 0.45 (hexane-Et_2O=2:1); $[\alpha]_D^{26}$ -9.49° (c = 1.00, $CHCl_3$); IR (KBr) 1763 (C=O), 1701 (C=O) cm^{-1}; HRMS (CI) Found *m/z* 418.1069. Calcd for $C_{14}H_{17}O_3NF_9$: M+H, 418.1065; **Major isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.40 (9H, s), 1.80-2.03 (3H, m), 2.35-2.42 (1H, m), 3.51-3.61 (2H, m), 4.84 (1H, t, *J* = 9.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.1 (s), 28.0 (s), 29.7 (s), 46.5 (s), 61.2 (s), 81.0 (s), 105.2-118.9 (4C, m), 153.1 (s), 193.1 (t, *J* = 26.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -47.95--47.76 (2F, m), -44.83 (2F, m), -40.54--40.44 (2F, m), -3.28--3.20 (3F, m); **Minor isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46 (9H, s), 1.80-2.03 (3H, m), 2.24-2.33 (1H, m), 3.42-3.48 (2H, m), 4.85 (1H, t, *J* = 8.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.3 (s), 28.2 (s), 28.7 (s), 46.6 (s), 60.9 (s), 80.3 (s), 105.2-118.9 (4C, m), 153.9 (s), 193.5 (t, *J* = 26.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -47.95--47.76 (2F, m), -44.97--44.88 (2F, m), -41.20--41.08 (2F, m), -3.28--3.20 (3F, m).

(*S*)-*tert*-butyl 2-(2,2,3,3,4,4,5,5,6,6,7,7,7-tridecafluoroheptanoyl)pyrrolidine-1-carboxylate (1b).



Rf 0.62 (hexane-Et_2O=2:1); mp 45.1-46.2 °C; $[\alpha]_D^{27}$ -9.71° (c = 1.00, $CHCl_3$); IR (KBr) 1761 (C=O), 1699 (C=O) cm^{-1}; HRMS (FAB) Found *m/z* 517.0927. Calcd for $C_{16}H_{16}O_3NF_{17}$: M, 517.0923; **Major isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.40 (9H, s), 1.84-2.05 (3H, m), 2.35-2.42 (1H, m), 3.51-3.61 (2H, m), 4.84 (1H, t, *J* = 8.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.1 (s), 28.0 (s), 29.7 (s), 46.5 (s), 61.2 (s), 81.0 (s), 105.5-118.9 (6C, m), 153.1 (s), 193.1 (t, *J* = 25.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -50.71--50.66 (2F, m), -47.37 (2F, m), -46.07--46.05 (4F, m), -42.6--42.56 (2F, m), -5.43--5.37 (3F, m); **Minor isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46 (9H, s), 1.84-2.05 (3H, m), 2.26-2.33 (1H, m), 3.42-3.48 (2H, m), 4.85 (1H, t, *J* = 9.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.3 (s), 28.2 (s), 28.7 (s), 46.6 (s), 60.9 (s), 80.4 (s), 105.2-118.9 (6C, m), 153.9 (s), 193.6 (t, *J* = 25.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -50.71--50.66 (2F, m), -47.37 (2F, m), -46.23--46.18 (4F, m), -43.21 (2F, t, *J* = 12.6 Hz), -5.43--5.37 (3F, m).

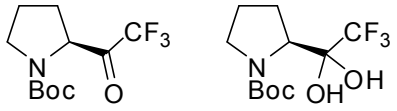
(*S*)-*tert*-butyl 2-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-heptadecafluorononanoyl)pyrrolidine-1-carboxylate (1c).



Rf 0.60 (hexane-Et_2O=2:1); mp 51.3-52.6 °C; $[\alpha]_D^{27}$ -8.20° (c = 1.00, $CHCl_3$); IR (KBr) 1760 (C=O), 1699 (C=O) cm^{-1}; HRMS (FAB) Found *m/z* 617.0853. Calcd for $C_{18}H_{16}O_3NF_{17}$: M, 617.0859; **Major isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.40 (9H, s), 1.82-2.05 (3H, m), 2.35-2.42 (1H, m), 3.51-3.61 (2H, m), 4.85 (1H, t, *J* = 9.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.1 (s), 28.0 (s), 29.7 (s), 46.5 (s), 61.2 (s), 81.0 (s), 106.8-118.9 (8C, m), 153.1 (s), 193.4 (t, *J* = 25.4 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -50.73--50.64 (2F, m), -47.26 (2F, m), -46.43--46.38 (8F, m), -42.64--42.52 (2F, m), -5.37 (3F, t, J = 9.9Hz); **Minor isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46 (9H, s), 1.82-2.05 (3H, m), 2.26-2.32 (1H,

m), 3.42-3.48 (2H, m), 4.84 (1H, t, J = 10.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.3 (s), 28.2 (s), 28.8 (s), 46.6 (s), 60.9 (s), 80.4 (s), 106.8-118.9 (8C, m), 153.96 (s), 193.6 (t, J = 26.2 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -50.73--50.64 (2F, m), -47.26 (2F, m), -46.13--45.76 (8F, m), -43.16 (2F, t, J = 12.6 Hz), -5.37 (3F, t, J = 9.9 Hz).

(*S*)-*tert*-butyl 2-(2,2,2-trifluoroacetyl)pyrrolidine-1-carboxylate (1d), (*S*)-*tert*-butyl 2-(2,2,2-trifluoro-1,1-dihydroxyethyl)pyrrolidine-1-carboxylate (1d-hydrate).



To a solution of methyl (*S*)-*N*-(*tert*-butoxycarbonyl)pyrrolidine-2-carboxylate (0.698 g, 3 mmol) in pentane (5 ml) was added trifluoromethyltrimethylsilane (CF_3SiMe_4) (0.660 g, 4.6 mmol) at room temperature under argon. To the reaction mixture was added dropwise tetrabutylammonium fluoride (TBAF) (1.0 M THF solution, 0.15 ml, 0.15 mmol) at -78 °C, and the reaction mixture was then allowed to warm slowly to room temperature, and stirred for 18 h. The reaction mixture was quenched with sat. $NaHCO_3$ aq solution (70 ml) at 0 °C and then subjected to extraction with Et_2O (30 ml×3). The organic layer was washed with brine (80 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-Et_2O=5:1) gave a mixture of **1d** and **1d**-hydrate (0.570 g, **1d** : **1d**-hydrate = 40 : 60, 28% for **1d**, 40% for **1d**-hydrate).

The mixture of 1d and 1d-hydrate (1d : 1d-hydrate = 56 : 44). *Rf* 0.30 (hexane-Et_2O=5:1); $[\alpha]_D^{30}$ -0.24° (c = 1.00, $CHCl_3$); IR (NaCl) 3365 (OH), 1686 (C=O), 1645 (C=O) cm^{-1}; HRMS (CI) Found *m/z* 268.1147. Calcd for $C_{11}H_{17}O_3NF_3$: M+H, 268.1161.

(*S*)-*tert*-butyl 2-(2,2,2-trifluoroacetyl)pyrrolidine-1-carboxylate (1d). Major isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.30 (9H, s), 1.79-1.98 (4H, m), 3.33-3.52 (2H, m), 4.65 (1H, dd, J = 8.9, 4.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.7 (s), 28.8 (s), 30.6 (s), 47.1 (s), 61.1 (s), 81.6 (s), 116.3 (q, J = 293.3 Hz), 153.8 (s), 191.4 (q, J = 33.3 Hz); ^{19}F NMR (372 MHz, $CDCl_3$) δ -1.24 (3F, s); **Minor isomer:** ^{1}H

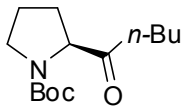
NMR (400 MHz, $CDCl_3$) δ 1.37 (9H, s), 1.79-1.98 (4H, m), 3.33-3.52 (2H, m), 4.71 (1H, dd *J* = 8.7, 3.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.0 (s), 28.9 (s), 30.3 (s), 47.2 (s), 61.0 (s), 81.2 (s), 116.3 (q, *J* = 293.3 Hz), 154.8 (s), 191.4 (q, *J* = 33.3 Hz); ^{19}F NMR (372 MHz, $CDCl_3$) δ -1.18 (3F, s).

(*S*)-*tert*-butyl 2-(2,2,2-trifluoro-1,1-dihydroxyethyl)pyrrolidine-1-carboxylate (1d-hydrate). ^1H NMR (400 MHz, $CDCl_3$) δ 1.40 (9H, s), 1.60-1.67 (1H, m), 1.79-1.98 (1H, m), 2.10-2.34 (1H, m), 3.22 (dt, *J* = 10.6, 6.6 Hz), 3.33-3.52 (1H, m), 4.08 (1H, dd, *J* = 7.6, 5.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.1 (s), 27.7 (s), 28.7 (s), 48.5 (s), 62.1 (s), 82.6 (s), 95.8 (q, *J* = 29.5 Hz), 124.0 (q, *J* = 290.0 Hz), 159.8 (s); ^{19}F NMR (372 MHz, $CDCl_3$) δ -6.67 (3F, s).

(*S*)-*tert*-butyl 2-pentanoylpyrrolidine-1-carboxylate (1e), (*S*)-*tert*-butyl 2-(5-hydroxynonan-5-yl)pyrrolidine-1-carboxylate (2).

To a solution of methyl (*S*)-*N*-(*tert*-butoxycarbonyl)pyrrolidine-2-carboxylate (0.459 g, 2 mmol) in dry Et_2O (15 ml) was added dropwise *n*-BuLi (1.6 M *n*-hexane solution, 3.75 ml, 6 mmol) for 2 h at -78 °C. The reaction was quenched with NH_4Cl-10 % HCl (v/v = 1:1) aq solution (30 ml) and then subjected to extraction with Et_2O (30 ml×3). The organic layer was washed with brine (30 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-AcOEt=5:1) gave **1e** (47%, 0.239 g) and **2** (41%, 0.257 g).

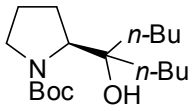
(*S*)-*tert*-butyl 2-pentanoylpyrrolidine-1-carboxylate (1e).



Rf 0.13 (hexane-AcOEt=10:1); $[\alpha]_D^{22}$ -16.5° (c = 1.00, $CHCl_3$); IR (KBr) 1724.6 (C=O), 1697.6 (C=O) cm^{-1}; HRMS (EI) found: *m/z* 255.1837. Calcd for $C_{14}H_{25}NO_3$: M,255.1834; **Major isomer:** ^1H NMR (400 MHz, $CDCl_3$) δ 0.67 (3H, t, *J* = 7.6 Hz), 1.02-1.65 (7H, m), 1.16 (9H, s), 1.85-1.99 (1H, m), 2.11-

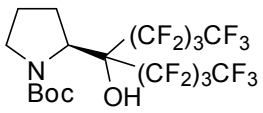
2.29 (2H, m), 3.16-3.29 (2H, m), 4.00-4.03 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4 (s), 21.9 (s), 23.1 (s), 24.9 (s), 27.8 (s), 29.4 (s), 37.7 (s), 46.2 (s), 64.6 (s), 79.3 (s), 153.3 (s), 209.3 (s); **Minor isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.65 (3H, t, J = 7.6 Hz), 1.02-1.65 (7H, m), 1.22 (9H, s), 1.85-1.99 (1H, m), 2.11-2.29 (2H, m), 3.16-3.29 (2H, m), 4.07-4.10 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4 (s), 21.8 (s), 23.9 (s), 24.8 (s), 27.9 (s), 28.3 (s), 38.2 (s), 46.4 (s), 64.2 (s), 79.0 (s), 154.0 (s), 209.4 (s).

(*S*)-*tert*-butyl 2-(5-hydroxynonan-5-yl)pyrrolidine-1-carboxylate (2).



Rf 0.26 (hexane-AcOEt = 10:1); $[\alpha]_D^{22}$ -52.2° (c = 1.00, $CHCl_3$); IR (KBr) 3360.4 (OH), 1693.7 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 313.2627. Calcd for $C_{18}H_{35}NO_3$: M, 313.2617; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.81 (6H, t, m), 1.09-1.65 (14H, m), 1.37 (9H, s), 1.72 (1H, m), 1.93 (1H, m), 3.02-3.12 (1H, m), 3.57 (1H, br s), 3.91 (1H, t, J = 6.1 Hz), 5.44 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0 (s), 23.2 (s), 23.4 (s), 23.6 (s), 24.1 (s), 25.1 (s), 25.9 (s), 28.2 (s), 35.2 (s), 38.8 (s), 48.2 (s), 65.1 (s), 76.5 (s), 80.1 (s), 157.6 (s).

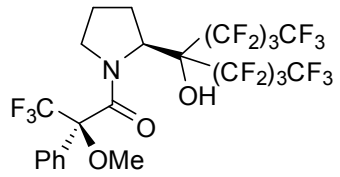
(*S*)-*tert*-butyl 2-(1,1,1,2,2,3,3,4,4,6,6,7,7,8,8,9,9,9-octadecafluoro-5-hydroxynonan-5-yl)-pyrrolidine-1-carboxylate (3).



To a solution of (*S*)-*tert*-butyl 2-(2,2,3,3,4,4,5,5,5-nonafluoropentanoyl)pyrrolidine-1-carboxylate **1a** (1.257 g, 3 mmol) in dry Et_2O (15 ml) was added perfluorobutyl iodide (5.287 g, 15 mmol) at room temperature under argon. After the reaction mixture was stirred at room temperature for 20 min,

methyllithium-lithium bromide complex (1.5 M Et_2O solution, 11 ml, 17 mmol) was added dropwise for 2 h at -78 °C. The reaction mixture was quenched with NH_4Cl-10 % HCl (v/v = 1:1) aq solution (80 ml) and then subjected to extraction with Et_2O (30 ml×3). The organic layer was washed with brine (70 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-Et_2O=10:1) gave **3** (55%, 1.052 g). *Rf* 0.63 (hexane-Et_2O=10:1); $[\alpha]_D^{22}$ -39.2° (c = 1.00, $CHCl_3$); IR (KBr) 3474 (OH), 1647 (C=O) cm^{-1}; HRMS (FAB) Found *m/z* 638.0992. Calcd for $C_{18}H_{18}O_3NF_{18}$: M+H, 638.0999; **Major isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47 (9H, s), 1.72-1.83 (1H, m), 1.94-1.99 (1H, m), 2.31-2.33 (2H, m), 3.18-3.25 (1H, m), 3.76-3.81 (1H, m), 4.66 (1H, t, *J* = 8.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.8 (s), 27.2 (s), 28.0 (s), 47.7 (s), 62.6 (s), 83.1 (s), 85.0 (quint), 108.0-122.2 (8C, m), 160.0 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ -49.64- -45.72 (4F, m), -44.02- -39.22 (4F, m), -34.61- -28.84 (4F, m), -3.11 (6F, t, *J* = 10.3 Hz); **Minor isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47 (9H, s), 1.72-1.83 (1H, m), 1.94-1.99 (1H, m), 2.31-2.33 (2H, m), 3.18-3.25 (1H, m), 3.76-3.81 (1H, m), 4.66 (1H, t, *J* = 8.1 Hz), 8.86 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.8 (s), 27.2 (s), 28.0 (s), 47.7 (s), 62.6 (s), 83.1 (s), 85.0 (quint), 108.0-122.2 (8C, m), 160.0 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ -49.64- -45.72 (4F, m), -44.02--39.22 (4F, m), -34.61- -28.84 (4F, m), -2.91 (6F, t, *J* = 10.3 Hz).

(*R*)-3,3,3-trifluoro-2-methoxy-1-((*S*)-2-(1,1,1,2,2,3,3,4,4,6,6,7,7,8,8,9,9,9-octadecafluoro-5-hydroxynonan-5-yl)pyrrolidin-1-yl)-2-phenylpropan-1-one (4).

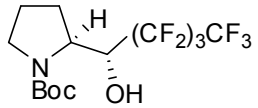


(*S*)-*tert*-Butyl 2-(1,1,1,2,2,3,3,4,4,6,6,7,7,8,8,9,9,9-octadecafluoro-5-hydroxynonan-5-yl)-pyrrolidine-1-carboxylate (0.269 g, 0.5 mmol) was dissolved in CH_2Cl_2 (3 ml) and TFA (3 ml) at 0 °C. After the

mixture was stirred at room temperature for 1.5 h, the reaction was quenched with Na_2CO_3 aq solution (50 ml) and then subjected to extraction with CH_2Cl_2 (30 ml×3). The organic layer was washed with brine (70 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. To a solution of the residue in THF was added aqueous NaOH (1.0 M, 0.5 ml, 0.5 mmol) and (+)-MTPA acid chloride (0.200 g, 1 mmol). After the mixture was stirred at room temperature for 2 h, the reaction mixture was quenched with $NaHCO_3$ aq solution (60 ml), and then subjected to extraction with Et_2O (30 ml×3). The organic layer was washed with brine (70 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-AcOEt=10:1) gave **4** (79%, 0.298 g). *Rf* 0.30 (hexane-AcOEt=10:1); mp 116.5-117.0 °C; $[\alpha]_D^{25}$ 11.3 (c = 1.00, $CHCl_3$); IR (NaCl) 3022.9 (OH), 1619.9 (C=O) cm^{-1}; HRMS (FAB) Found m/z 754.0871. Calcd for $C_{23}H_{17}F_{21}NO_3$: M+H, 754.0871; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.61-1.74 (1H, m), 1.88-1.89 (1H, m), 2.21-2.36 (3H, m), 3.65 (3H, s), 4.02 (1H, dt, *J* = 8.7, 2.3 Hz), 5.17 (1H, t, *J* = 8.3 Hz), 7.33-7.50 (5H, m), 8.59 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.7 (s), 25.7 (s), 47.4 (d, *J* = 8.2 Hz), 56.1 (s), 63.1 (s), 85.3 (q, *J* = 26.2 Hz), 86.0 (quint), 123.4 (q, *J* = 289.8 Hz), 126.9 (s), 128.3 (s), 129.7 (s), 132.8 (s), 171.2 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ -51.66--49.31 (4F, m), -45.04--44.24 (2F, m), -42.77--41.83 (2F, m), -36.64--35.84 (2F, m), -34.44--33.65 (1F, m), -32.36--31.56 (1F, m), -5.48 (3F, t, *J* = 10.7 Hz), -5.41 (3F, t, *J* = 10.7 Hz), 4.07 (3F, s).

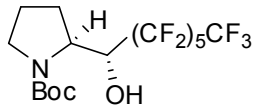
Typical procedure for the reduction of ketones. To a solution of $NaBH_4$ (0.076 g, 2 mmol) in EtOH (5 ml) was added an EtOH solution (3 ml) of (*S*)-*tert*-butyl 2-(2,2,3,3,4,4,5,5,5-nonafluoropentanoyl)pyrrolidine-1-carboxylate **1a** (0.417 g, 1 mmol) at 0 °C under argon. After the mixture was stirred at room temperature for 7 h, the reaction was quenched with 10 % HCl aq solution (60 ml), and then subjected to extraction with Et_2O (30 ml×3). The organic layer was washed with brine (70 ml), and dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-CH_2Cl_2=1:2) gave (α*R*)-**5a** (78%, 0.325 g).

(*S*)-*tert*-butyl 2-((*R*)-2,2,3,3,4,4,5,5,5-nonafluoro-1-hydroxypentyl)pyrrolidine-1-carboxylate ((α*R*)-5a).



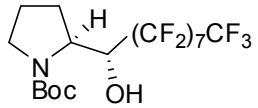
Rf 0.33 (hexane-CH_2Cl_2=1:2); mp 74.6-75.3 °C; $[\alpha]_D^{25}$ -19.9° (c = 1.00, $CHCl_3$); IR (KBr) 3250 (OH), 1682 (C=O) cm^{-1}; HRMS (FAB) Found *m/z* 420.1212. Calcd for $C_{14}H_{19}F_9NO_3$: M+H, 420.1221; Anal. Calcd for C, 40.10; H, 4.33; N, 3.34. Found: C, 39.80; H, 4.18; N, 3.35; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.39 (9H, s), 1.78-1.86 (3H, m), 1.93-2.01 (1H, m), 3.25-3.40 (2H, m), 3.85 (1H, dt, *J* = 19.9, 8.5 Hz), 4.26 (1H, t, *J* = 8.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.7 (s), 28.0 (s), 28.7 (s), 46.8 (s), 56.8 (s), 73.5-73.9 (m), 81.4 (s), 105.9-122.1 (4C, m), 158.8 (s); ^{19}F NMR (372 MHz, $CDCl_3$) δ -52.48--51.66 (2F, m), -49.96--47.71 (2F, m), -46.36--41.03 (2F, m), -5.73 (3F, s).

(*S*)-*tert*-butyl 2-((*R*)-2,2,3,3,4,4,5,5,6,6,7,7,7-tridecafluoro-1-hydroxyheptyl)pyrrolidine-1-carboxylate ((α*R*)-5b).



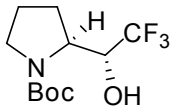
Rf 0.61 (hexane-CH_2Cl_2=1:2); mp 55.1-57.0 °C; $[\alpha]_D^{29}$ -33.39° (c = 1.00, $CHCl_3$); IR (KBr) 3244 (OH), 1652 (C=O) cm^{-1}; HRMS (FAB) Found *m/z* 520.1157. Calcd for $C_{16}H_{19}F_{13}NO_3$: M+H, 520.1157; Anal. Calcd for C, 37.01; H, 3.49; N, 2.70. Found: C, 37.18; H, 3.45; N, 2.57; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.44 (9H, s), 1.84-1.88 (3H, m), 2.00-2.06 (1H, m), 3.30-3.44 (2H, m), 3.89 (1H, dt, *J* = 20.5, 8.4 Hz), 4.31 (1H, t, *J* = 8.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.6 (s), 28.1 (s), 28.7 (s), 46.8 (s), 56.8 (s), 73.8 (t, *J* = 22.5 Hz), 81.5 (s), 105.6-121.6 (6C, m), 158.86 (s); ^{19}F NMR (372 MHz, $CDCl_3$) δ -51.40 (1F, d, *J* = 280.0 Hz), -51.40 (1F, d, *J* = 296.0 Hz), -50.15 (1F, d, *J* = 296.0 Hz), -48.44--44.93 (6F, m), -41.32 (1F, d, *J* = 280.0 Hz), -5.42 (3F, t, *J* = 9.9Hz).

(*S*)-*tert*-butyl 2-((*R*)-2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-heptadecafluoro-1-hydroxynonyl)pyrrolidine-1-carboxylate ((α*R*)-5c).



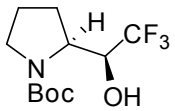
Rf 0.52 (hexane-CH_2Cl_2=1:2); mp 68.6-69.6 °C; $[\alpha]_D^{29}$ -30.83° (c = 1.00, $CHCl_3$); IR (KBr) 3284 (OH), 1652 (C=O) cm^{-1}; HRMS (FAB) Found *m/z* 620.1071. Calcd for $C_{18}H_{19}F_{17}NO_3$: M+H, 620.1093; Anal. Calcd for C, 34.91; H, 2.93; N, 2.26. Found: C, 34.75; H, 2.93; N, 2.39; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.43 (9H, s), 1.83-1.88 (3H, m), 2.00-2.05 (1H, m), 3.29-3.41 (2H, m), 3.89 (1H, dt, *J* = 19.8, 8.0 Hz), 4.30 (1H, t, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.4 (s), 28.8 (s), 29.4 (s), 47.5 (s), 57.5 (s), 74.4 (t, *J* = 23.8 Hz), 82.2 (s), 108.2-119.6 (8C, m), 159.5 (s); ^{19}F NMR (372 MHz, $CDCl_3$) δ -51.56 (1F, d, *J* = 280.0 Hz), -51.07 (1F, d, *J* = 293.0 Hz), -50.51 (1F, d, *J* = 293.0 Hz), -48.28--44.93 (10F, m), -41.38 (1F, d, *J* = 280.0 Hz), -5.48 (3F, t, *J* = 9.9 Hz).

(*S*)-*tert*-butyl 2-((*R*)-2,2,2-trifluoro-1-hydroxyethyl)pyrrolidine-1-carboxylate ((α*R*)- 5d).



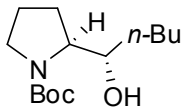
Rf 0.38 (CH_2Cl_2); $[\alpha]_D^{23}$ -5.0° (c = 1.00, $CHCl_3$); IR (KBr) 3325.7 (OH), 1655.1 (C=O) cm^{-1}; HRMS (FAB) found: *m/z* 270.1324. Calcd for $C_{11}H_{19}F_3NO_3$: M+H, 270.1317; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.40 (9H, s), 1.77-2.06 (4H, m), 3.24-3.30 (1H, m), 3.36-3.42 (1H, m), 3.64-3.73 (1H, m), 4.06-4.11 (1H, m), 5.79 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.8 (s), 28.0 (s), 46.9 (s), 57.4 (s), 74.1 (q, *J* = 28.1 Hz), 81.3 (s), 124.6 (q, *J* = 283.4 Hz), 158.6 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ 2.93 (3F, s).

(*S*)-*tert*-butyl 2-((*S*)-2,2,2-trifluoro-1-hydroxyethyl)pyrrolidine-1-carboxylate ((α*S*)-5d).



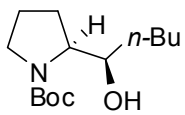
Rf 0.10 (CH_2Cl_2); mp 124.2-125.0 °C; $[\alpha]_D^{23}$ -56.8° (c = 1.00, $CHCl_3$); IR (KBr) 3325.7 (OH), 1655.1 (C=O) cm^{-1}; HRMS (FAB) found: *m/z* 270.1324. Calcd for $C_{11}H_{19}F_3NO_3$: M+H, 270.1317; Anal. Calcd for C, 49.07; H, 6.74; N, 5.20. Found: C, 48.98; H, 6.50; N, 5.19. **Major isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47 (9H, s), 1.78-2.23 (4H, m), 3.27-3.78 (2H, m), 4.02-4.19 (1H, m), 4.25-4.36 (1H, m), 5.48 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.2 (s), 26.4 (s), 28.2 (s), 47.3 (s), 58.1 (s), 70.5 (quint, *J* = 28.7 Hz), 80.4 (s), 124.8 (q, *J* = 284.3 Hz), 156.1 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ 0.87 (3F, s), **Minor isomer:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47 (9H, s), 1.78-2.23 (4H, m), 3.27-3.78 (2H, m), 4.02-4.19 (1H, m), 4.25-4.36 (1H, m), 5.48 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.2 (s), 25.3 (s), 28.2 (s), 46.3 (s), 56.7 (s), 70.5 (quint, *J* = 28.7 Hz), 80.4 (s), 124.6 (q, *J* = 283.5 Hz), 156.1 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ 0.87 (3F, s).

(*S*)-*tert*-butyl 2-((*S*)-1-hydroxypentyl)pyrrolidine-1-carboxylate ((α*S*)-5e).



Rf 0.09 (hexane-AcOEt=10:1); $[\alpha]_D^{25}$ -31.9° (c = 1.00, $CHCl_3$); IR (NaCl) 3395.1 (OH), 1666.2 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 257.1998. Calcd for $C_{14}H_{27}NO_3$: M, 257.1991; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.84 (3H, t, *J* = 7.2 Hz), 1.17-1.56 (6H, m), 1.40 (9H, s), 1.66-1.93 (4H, m), 3.22 (1H, ddd, *J* = 10.9, 7.0, 5.6 Hz), 3.40 (2H, m), 3.74 (1H, ddd, *J* = 8.2, 8.2, 4.2 Hz), 4.78 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0 (s), 22.7 (s), 24.0 (s), 27.1 (s), 28.3 (s), 28.5 (s), 34.5 (s), 47.1 (s), 62.7 (s), 75.5 (s), 80.2 (s), 157.9 (s).

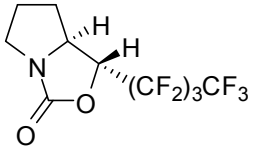
(*S*)-*tert*-butyl 2-((*R*)-1-hydroxypentyl)pyrrolidine-1-carboxylate ((α*R*)-5e).



Rf 0.03 (hexane-AcOEt=10:1); $[\alpha]_D^{25}$ -15.5° (c = 1.00, $CHCl_3$); IR (NaCl) 3398.0 (OH), 1675.8 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 257.1995. Calcd for $C_{14}H_{27}NO_3$: M, 257.1991; ^1H NMR (400 MHz, $CDCl_3$) δ 0.84 (3H, t, *J* = 7.2 Hz), 1.20-1.41 (6H, m), 1.41 (9H, s), 1.63-2.04 (4H, m), 3.18 (1H, ddd, *J* = 10.1, 7.0, 6.8 Hz), 3.47 (1H, br s), 3.69 (1H, m), 3.87 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0 (s), 22.7 (s), 24.2 (s), 27.4 (s), 28.4 (s), 31.7 (s), 48.0 (s), 63.1 (s), 73.3 (s), 79.7 (s), 156.2 (s).

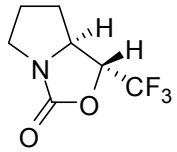
A typical procedure for the synthesis of oxazolidonone. (*S*)-*tert*-Butyl 2-((*R*)-2,2,3,3,4,4,5,5,5-nonafluoro-1-hydroxypentyl)pyrrolidine-1-carboxylate ((α*R*)-**5a**) (0.419g, 1 mmol) was dissolved in CH_2Cl_2 (3 ml) and TFA (3 ml) at 0 °C. After the mixture was stirred at room temperature for 1.5 h, the reaction was quenched with Na_2CO_3 aq solution (60 ml) and then subjected to extraction with CH_2Cl_2 (30 ml×3). The organic layer was washed with brine (70 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. To a solution of the residue in CH_2Cl_2 (5 ml) was added TEA (0.321 g, 3 mmol) and ethyl chloroformate (0.190 g, 1.8 mmol) at 0 °C. After the mixture was stirred at room temperature for 20 h, the reaction was quenched with 10% HCl aq solution (70 ml) and then subjected to extraction with CH_2Cl_2 (30 ml×3). The organic layer was washed with $NaHCO_3$ (70 ml), dried over Na_2SO_4, and concentrated by distillation under reduced pressure. To a suspension of NaH (0.026 g, 1.1 mmol) in DMF (2 ml) was added the residue in DMF solution (3 ml) at 0 °C. After the mixture was stirred at room temperature for 20 h, the reaction was quenched with $NaHCO_3$ aq solution (50 ml) and then subjected to extraction with AcOEt (30 ml×3). The organic layer was washed with H_2O (50 ml), and dried over Na_2SO_4, and concentrated by distillation under reduced pressure. Purification of the residue by silica gel column chromatography (hexane-Et_2O=2:1) gave **6a** (28%, 0.097 g).

(1*R*,7a*S*)-1-(perfluorobutyl)-tetrahydropyrrolo[1,2-c]oxazol-3(1*H*)-one (6a).



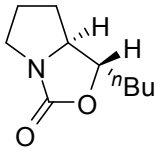
Rf 0.10 (hexane-Et_2O=2:1); mp 73.7-75.2 °C; $[\alpha]_D^{25}$ -12.9 (c = 1.00, $CHCl_3$); IR (KBr) 1761.6 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 345.0419. Calcd for $C_{10}H_8F_9NO_2$: M, 345.0411; Anal. Calcd for C, 34.80; H, 2.34; N, 4.06. Found: C, 35.14; H, 2.60; N, 4.39; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.52-1.68 (1H, m), 1.91-2.03 (1H, m), 2.07-2.18 (2H, m), 3.18-3.24 (1H, m), 3.58 (1H, dt, *J* = 14.2, 5.7 Hz), 3.99-4.04 (1H, m), 4.70 (1H, dt, *J* = 18.7, 4.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.4 (s), 30.6 (s), 45.7 (s), 58.7 (s), 74.8 (dd, *J* = 32.4, 22.5 Hz), 158.5 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ -52.75--51.87 (1F, m), -51.84--49.68 (2F, m), -49.03--48.25 (1F, m), -47.62--47.47 (2F, m), -5.48 (3F, tt, *J* = 9.5, 2.8 Hz).

(1*R*,7a*S*)-1-(trifluoromethyl)-tetrahydropyrrolo[1,2-c]oxazol-3(1*H*)-one (6d).



Rf 0.15 (hexane-Et_2O=1:1); $[\alpha]_D^{25}$ -36.5 (c = 0.31, $CHCl_3$); IR (NaCl) 1776.1 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 195.0502. Calcd for $C_7H_8F_3NO_2$: M, 195.0507; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.50-1.60 (1H, m), 1.90-2.04 (1H, m), 2,04-2.23 (2H, m), 3.21 (1H, ddd, *J* = 12.3, 8.3, 3.1 Hz), 3.59 (1H, dt, *J* = 14.2, 5.6 Hz), 3.88 (1H, ddd, *J* = 9.5, 6.0, 3.3 Hz), 4.54 (1H, dq, *J* = 12.7, 3.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3 (s), 30.4 (s), 45.7 (s), 59.3 (s), 75.3 (q, *J* = 34.7 Hz), 122.8 (q, *J* = 279.9 Hz), 158.9 (s); ^{19}F NMR (376 MHz, $CDCl_3$) δ -4.10 (3F, d, *J* = 6.1 Hz).

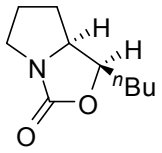
(1*S*,7a*S*)-1-butyl-tetrahydropyrrolo[1,2-c]oxazol-3(1*H*)-one (6c).



Rf 0.25 (hexane-AcOEt=2:1); $[\alpha]_D^{26}$ -25.9 (c = 1.00, $CHCl_3$); IR (NaCl) 1753.0 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 183.1253. Calcd for $C_{10}H_{17}NO_2$: M, 183.1259; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.64 (3H, t, *J* = 7.0 Hz), 1.06-1.26 (5H, m), 1.38-1.54 (2H, m), 1.59-1.83 (3H m), 2.85 (1H, ddd, *J* = 11.0, 8.9, 4.5

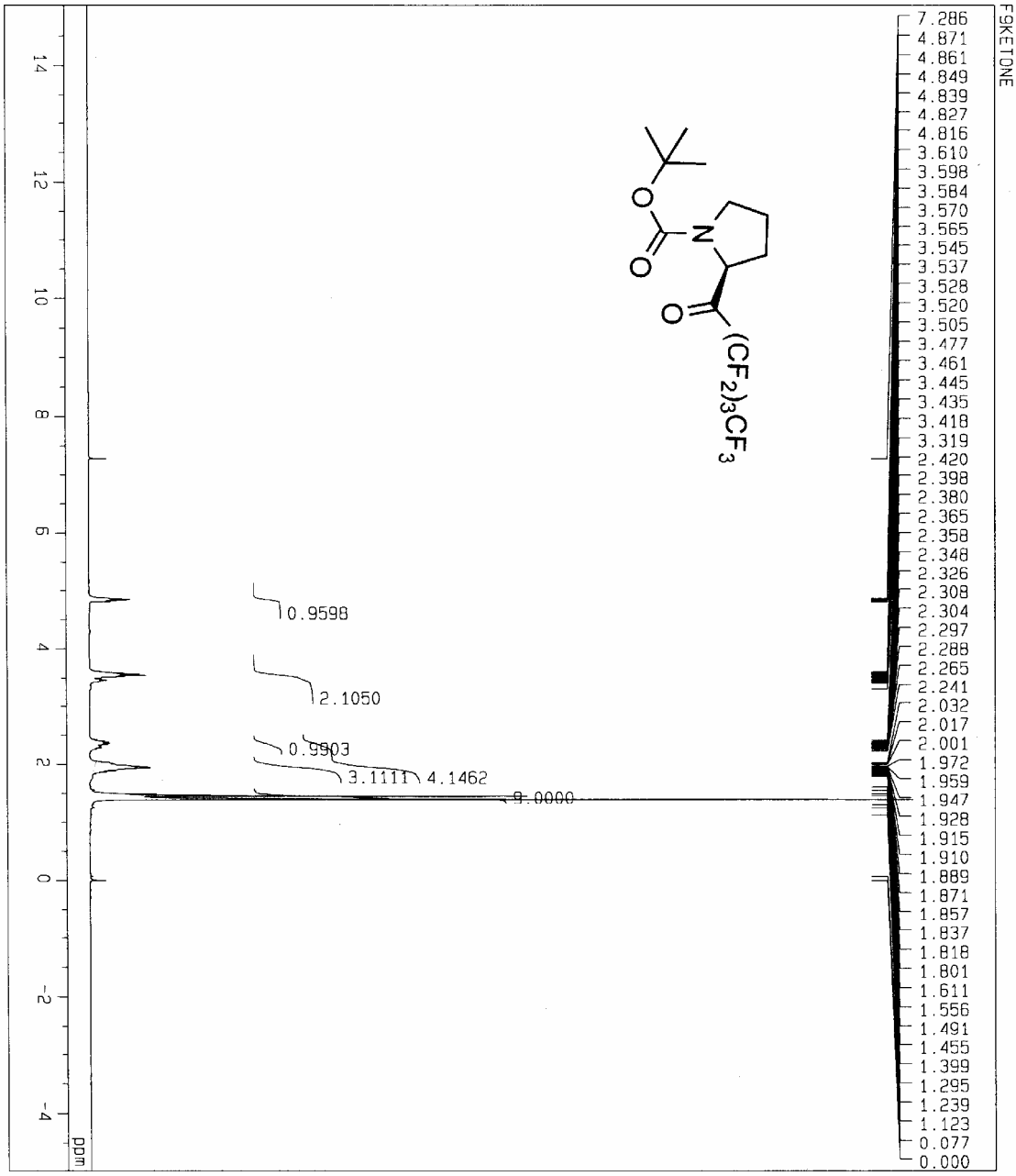
Hz), 3.29 (2H, ddd, *J* = 13.0, 7.2, 4.2 Hz), 4.00 (1H, ddd, *J* = 7.4, 5.8, 4.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.2 (s), 21.6 (s), 25.1 (s), 26.0 (s), 30.1 (s), 34.2 (s), 44.8 (s), 63.9 (s), 80.1 (s), 160.4 (s).

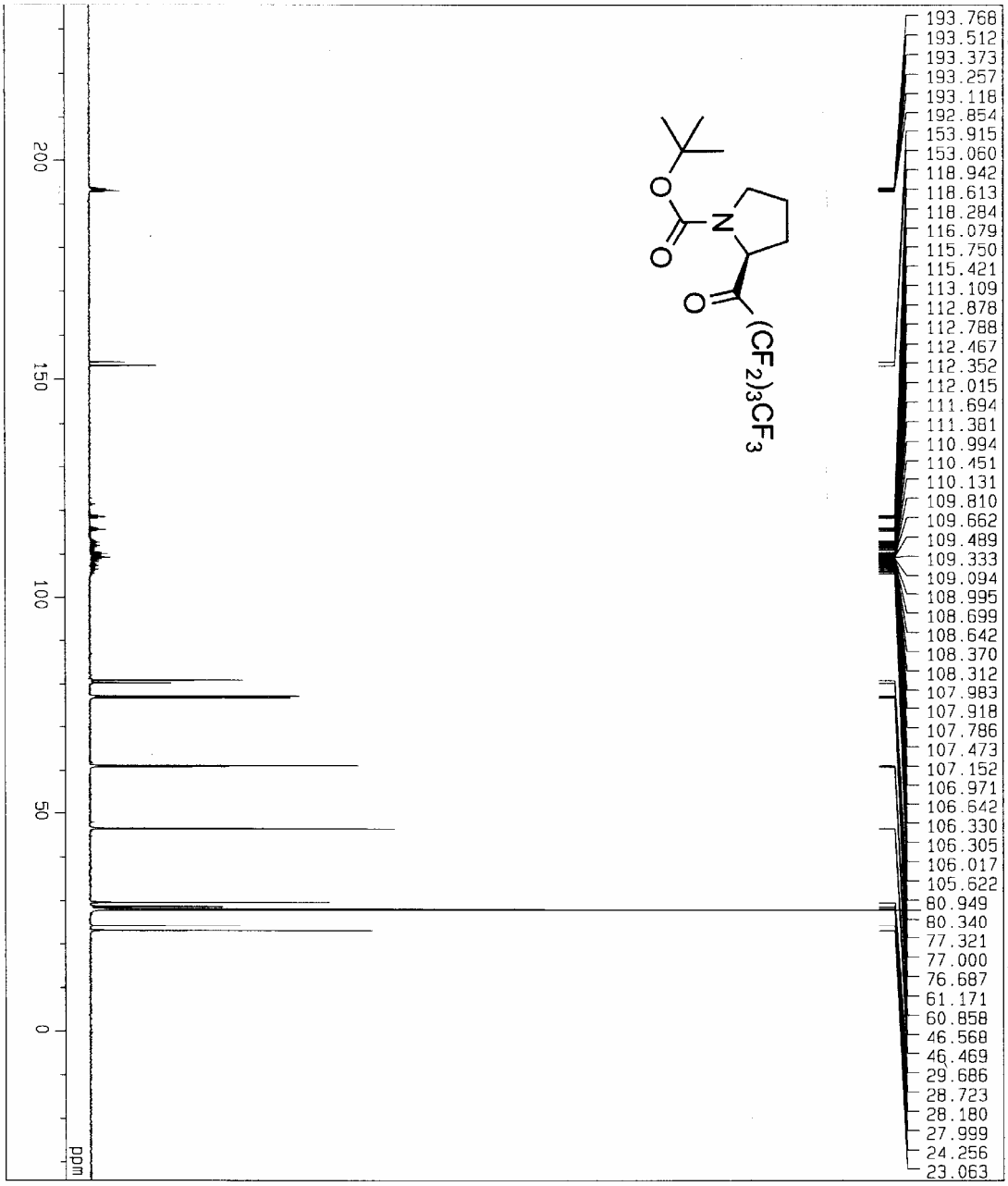
(1*R*,7a*S*)-1-butyl-tetrahydropyrrolo[1,2-c]oxazol-3(1*H*)-one (6c').[1]



Rf 0.30 (hexane-AcOEt=2:1); $[\alpha]_D^{26}$ -10.9 (c = 0.99, $CHCl_3$); IR (NaCl) 1753.0 (C=O) cm^{-1}; HRMS (EI) Found *m/z* 183.1263. Calcd for $C_{10}H_{17}NO_2$: M, 183.1259; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.75 (3H, t, *J* = 7.0 Hz), 1.18-1.37 (5H, m), 1.40-1.48 (1H, m), 1.54-1.65 (2H, m), 1.67-1.77 (1H, m), 1.86-1.95 (1H, m), 3.00 (1H, ddd, *J* = 12.1, 8.9, 2.5 Hz), 3.44 (1H, dt, *J* = 14.7, 5.7 Hz), 3.65 (1H, ddd, *J* = 11.7, 6.3, 4.5 Hz), 4.46 (1H, ddd, *J* = 8.3, 7.4, 5.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5 (s), 22.0 (s), 24.4 (s), 24.7 (s), 27.6 (s), 29.8 (s), 45.3 (s), 62.9 (s), 76.0 (s), 161.4 (s).

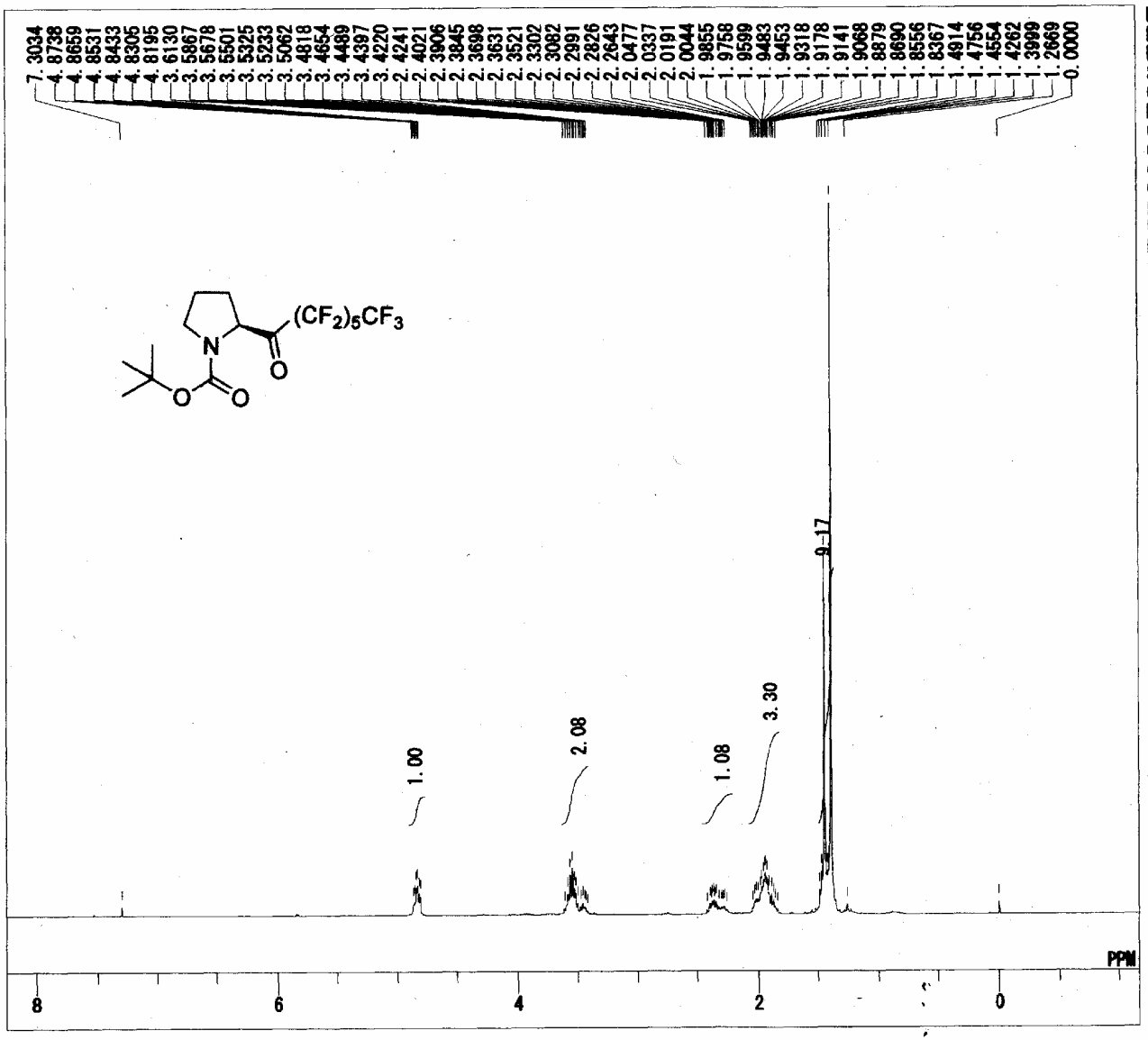
[1] Bejjani, J.; Chemla, F.; Audouin, M. *J. Org. Chem.* **2003**, *68*, 9747.


F9KETONE
7.286
4.871
4.861
4.849
4.839
4.827
4.816
3.610
3.598
3.584
3.570
3.565
3.545
3.537
3.528
3.520
3.505
3.477
3.461
3.445
3.435
3.418
3.319
2.420
2.398
2.380
2.365
2.358
2.348
2.326
2.308
2.304
2.297
2.288
2.265
2.241
2.032
2.017
2.001
1.972
1.959
1.947
1.928
1.915
1.910
1.889
1.871
1.857
1.837
1.818
1.801
1.611
1.556
1.491
1.455
1.399
1.295
1.239
1.123
0.077
0.000
(CF2)3CF3
0.9598
2.1050
0.9903
3.1111
4.1462
9.0000
ppm
14
12
10
8
6
4
2
0
-2
-4
31-JAN-2003 12:16:54.18
EXMOD : SINGL
IRMOD : NON
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
PW1 : 4.50 usec
POINT : 16384
SAMPO : 16384
TIMES : 8
SCANS : 8
DUMMY : 4
FREQU : 7993.61 Hz
FILTR : 4000 Hz
ACQTM : 2.0496 sec
DEADT : 72.75 usec
DELAY : 50.00 usec
PD : 1.9040 sec
ADBIT : 16
RGAIN : 13
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRPW : 50.0 usec
IRATN : 511
IRRNS : 0
IRBP1 : 30
IRBP2 : 5
BF : 0.10 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 7993.61 Hz
XS : 0.00 Hz
REFVL : 0.00 ppm
OFILE : ALPHA
MENUF : A1H
SHMFL : TH5AT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 23
LKPHS : 66.09 deg
LKSIG : 948
CTEMP : 19.4 c
CSPED : 11 Hz
FIELD : -676

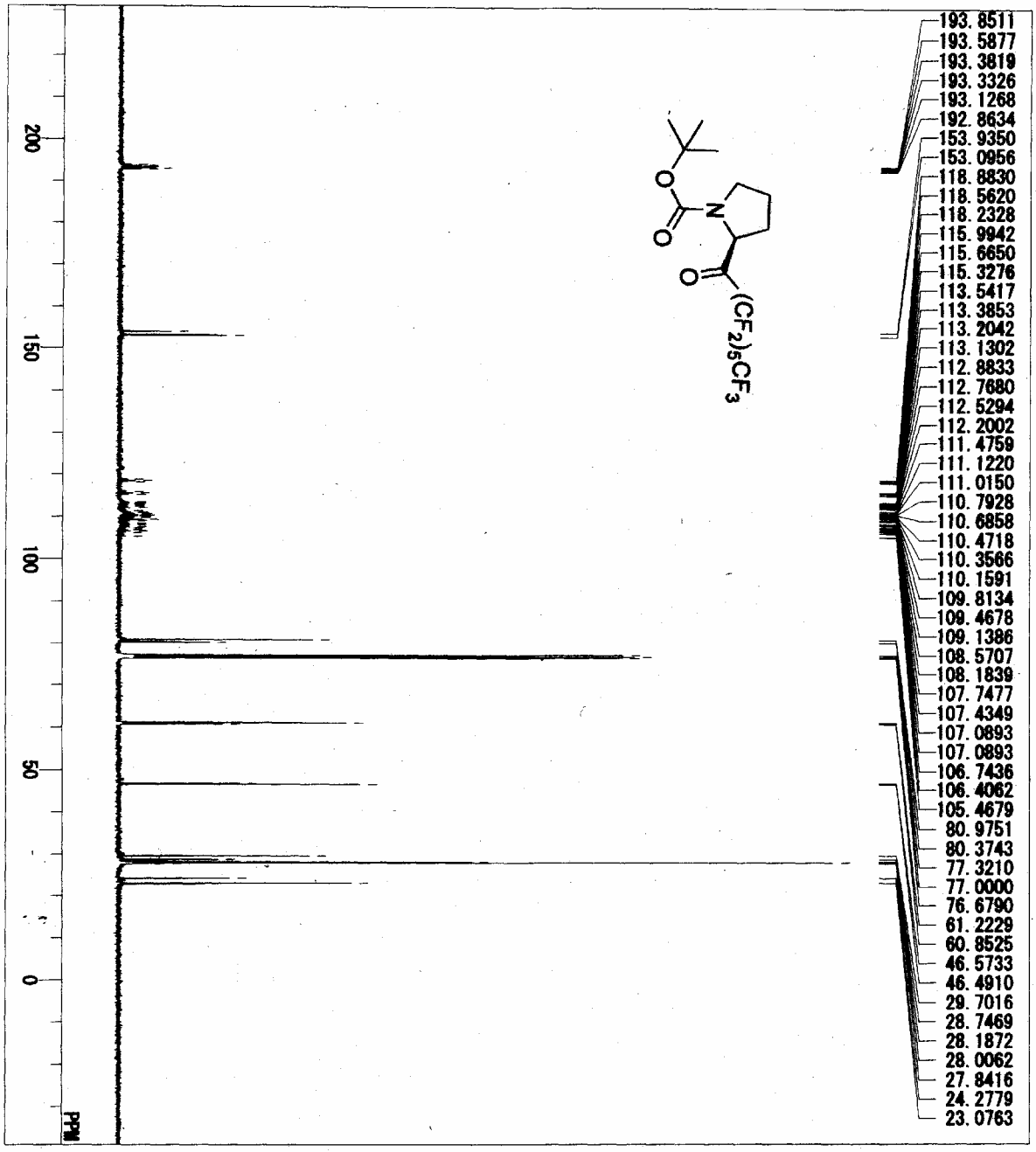


17-FEB-2003 15:46:04.53
EXMOD : SINGL
IRMOD : BCM
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
PW1 : 4.90 usec
POINT : 32768
SAMPO : 32768
TIMES : 2048
SCANS : 1505
DUMMY : 2
FREQU : 27100.27 Hz
FILTR : 13550 Hz
ACQTM : 1.2091 sec
DEADT : 19.69 usec
DELAY : 14.76 usec
PD : 2.5000 sec
ADBIT : 16
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRRNS : 0
IRBP1 : 30
IRBP2 : 5
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 27100.27 Hz
XS : 0.00 Hz
REFVL : 77.00 ppm
DFILE : ALPHA
MENUF : A13C
SHMFL : TH5AT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 23
LKPHS : 66.09 deg
LKSIG : 1219
CTEMP : 20.8 c
CSPED : 12 Hz
FIELD : -631

D:¥岩田¥Rxn14 1H(CDCl3)-2.als

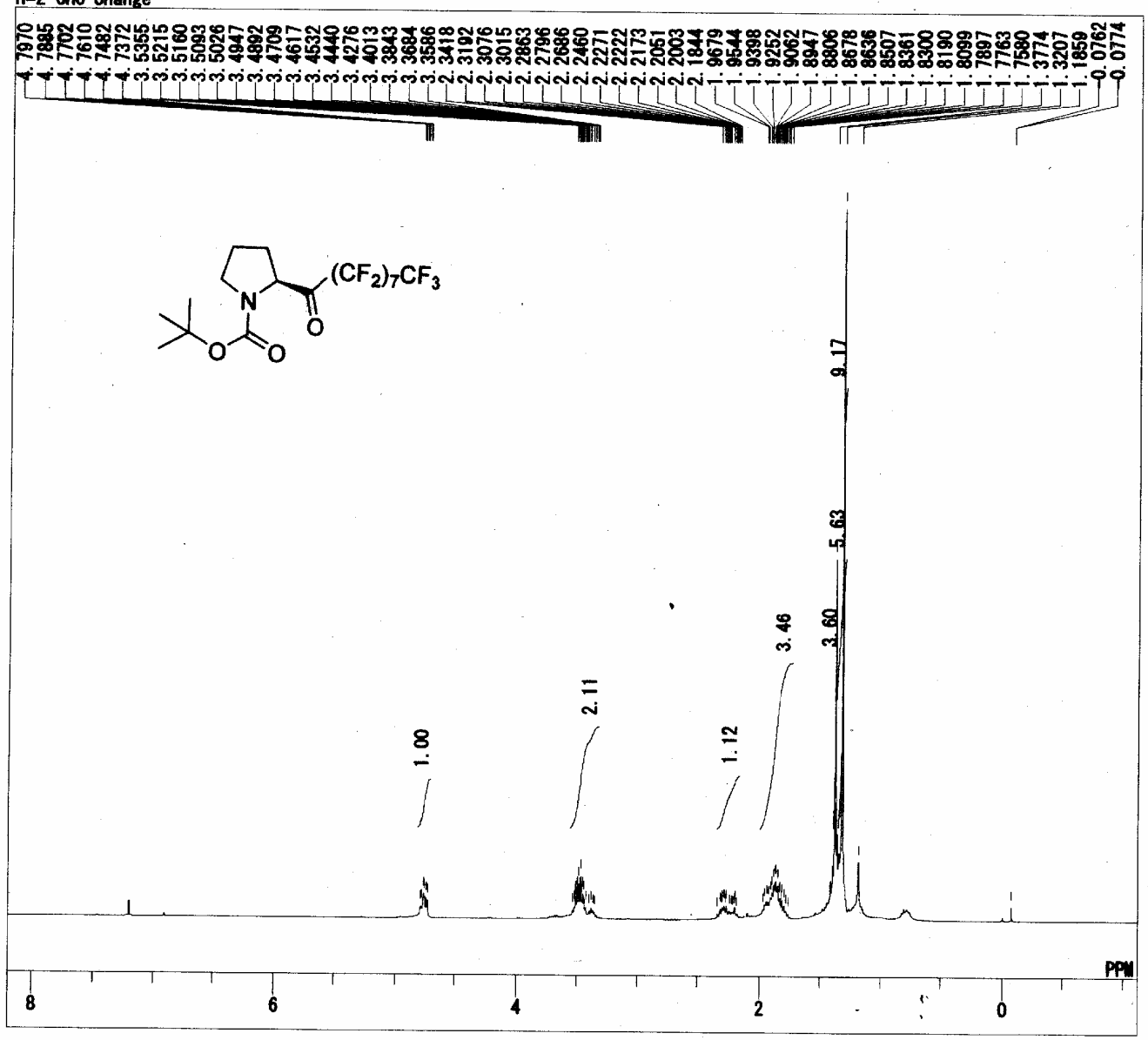


DATIM	Thu Jun 14 15:45:32 2007
HELEV	72.00 %
N2LEV	89.60 %
SLVNT	CDCL3
CTEMP	20.0 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
PW2	10.00 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	8
SCANS	8
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	8
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥岩田¥Rxn14 1H(CDCl3)-2.als
SF	TH5ATFQ2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	713
CSPED	10 Hz
FILDC	
FILDF	



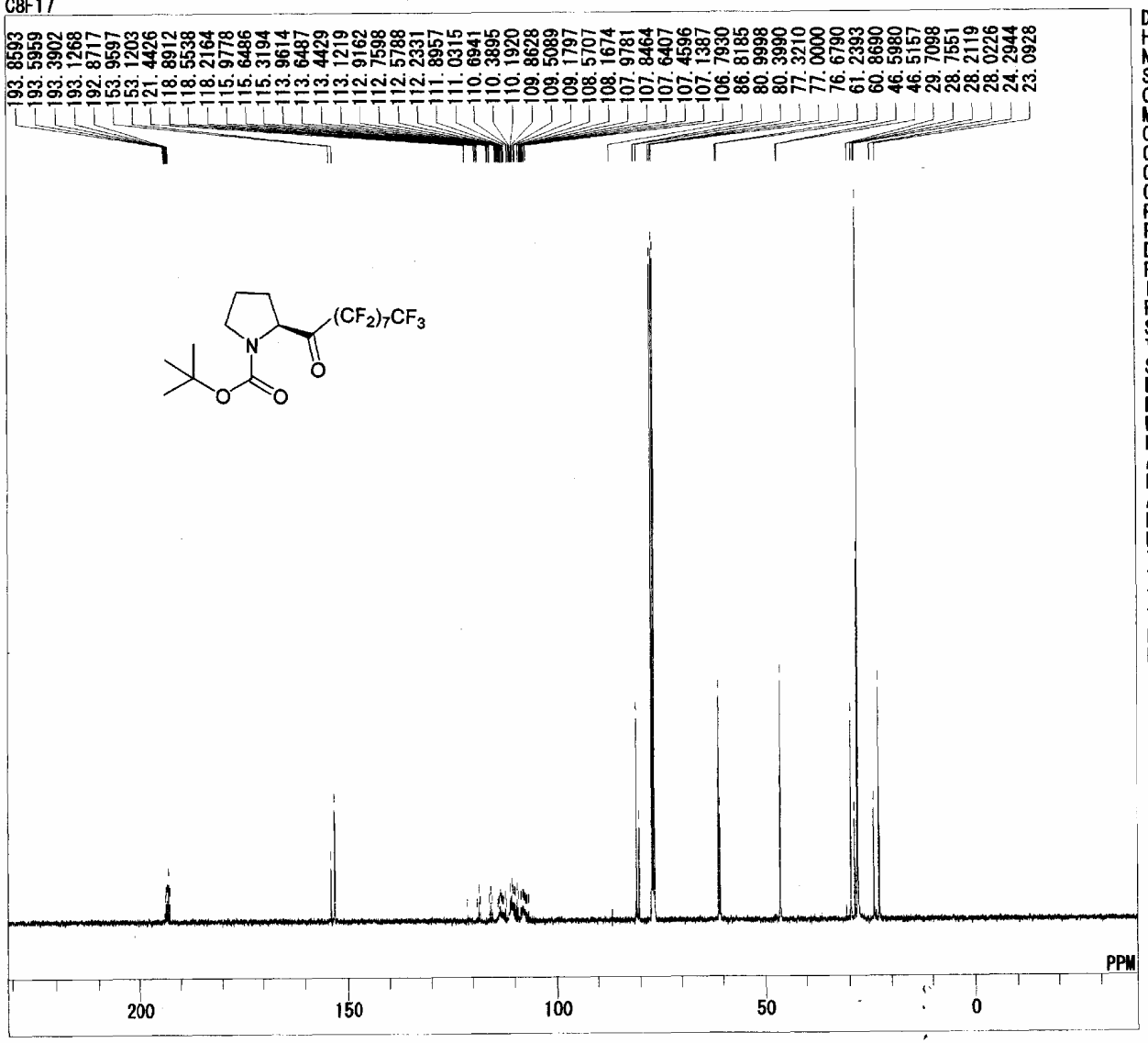
DATIM Thu Jun 14 17:03:58 2007
HELEV 72.40 %
N2LEV 89.20 %
SLVNT CDCL3
CTEMP 20.2 c
MENUF 13C BCM
OBNUC 13C
OFR 99.45 MHz
OBSET 94.00 KHz
OBFIN 10309.00 Hz
PW1 4.80 usec
PW2 10.00 usec
DEADT 20.00 usec
PREDL 0.20000 msec
INT 1.0000 msec
POINT 32768
SPO 32768
TIMES 1216
SCANS 1216
DUMMY 1
FREQU 26845.64 Hz
FLT
DELAY 14.90 usec
ACQTM 1.2206 sec
PD 1.7790 sec
ADBIT 16
RGAIN 25
BF 2.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel.complete.decoupling:Set_IRRPW
IRNUC 1H
IFR 395.75 MHz
IRSET 124.00 KHz
IRFIN 10277.00 Hz
IRRPW 50 usec
IRATN 511
DFILE D:¥嶋田¥Rxn14 13C(CDCL3).als
SF TMSATF02
LKSET 12.90 KHz
LKFIN 104.0 Hz
LKLEV 180
LGAIN 28
LKPHS 326
LKSIG 787
CSPED 10 Hz
FILDC
FILDF

D:¥岩田¥Rxn15 1H(CDCL3)-2.als
n=2 CHO change

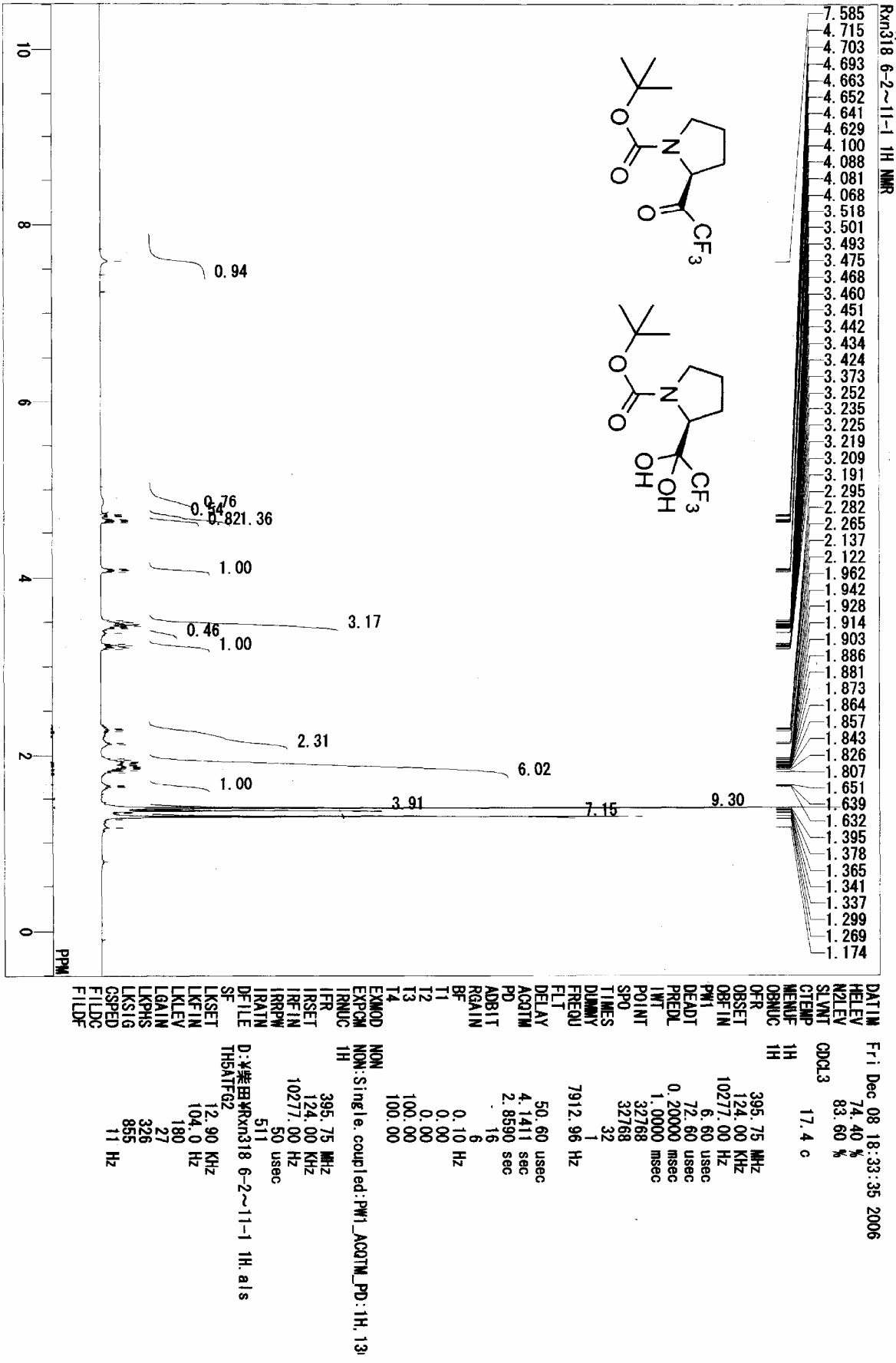


DATIM	Tue Jun 26 16:41:38 2007
HELEV	94.00 %
N2LEV	89.20 %
SLVNT	CDCL3
CTEMP	20.2 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
PW2	10.00 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	8
SCANS	8
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	10
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥岩田¥Rxn15 1H(CDCL3)-2.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	703
CSPED	12 Hz
FILDC	
FILDF	

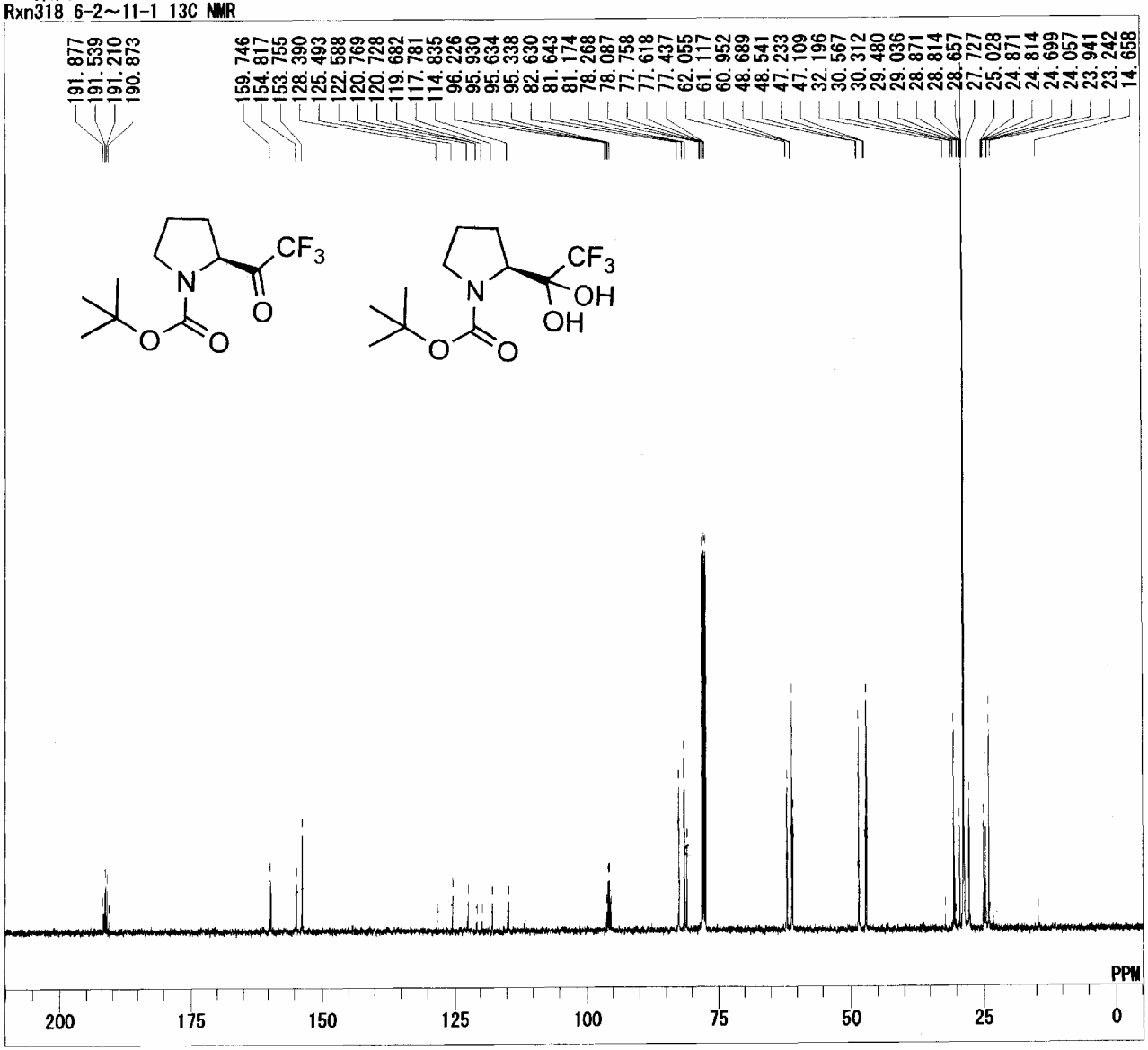
D:¥岩田¥Rxn18 13C(CDCl3).als
C8F17



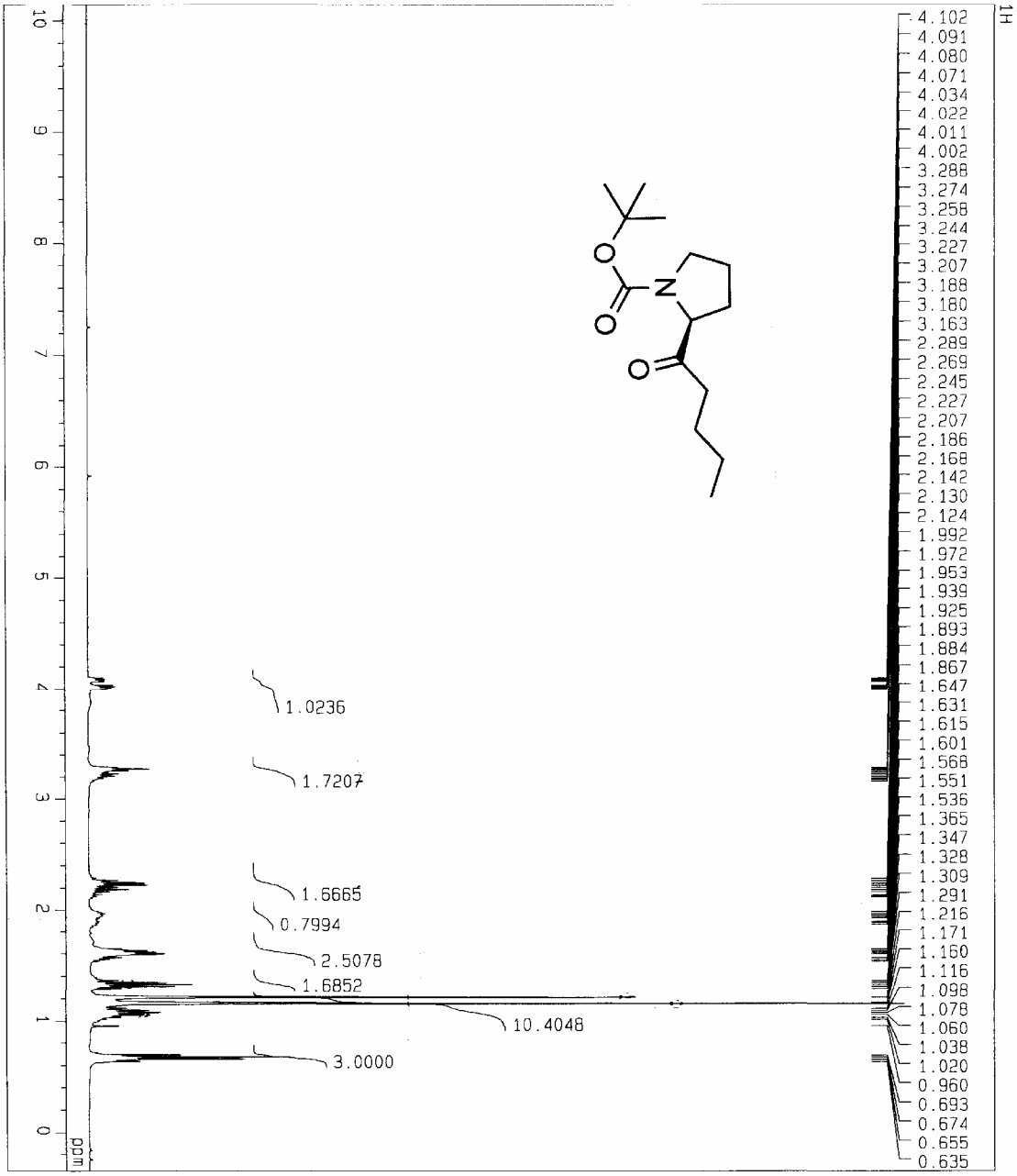
DATIM	Thu Jul 26 00:47:59 2007
HELEV	86.00 %
N2LEV	94.80 %
SLVNT	CDCL3
CTEMP	21.8 c
MENUF	13C BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.80 usec
PW2	10.00 usec
DEADT	20.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1856
SCANS	1856
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	26
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥岩田¥Rxn18 13C(CDCl3).als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	663
CSPED	13 Hz
FILDC	
FILDF	

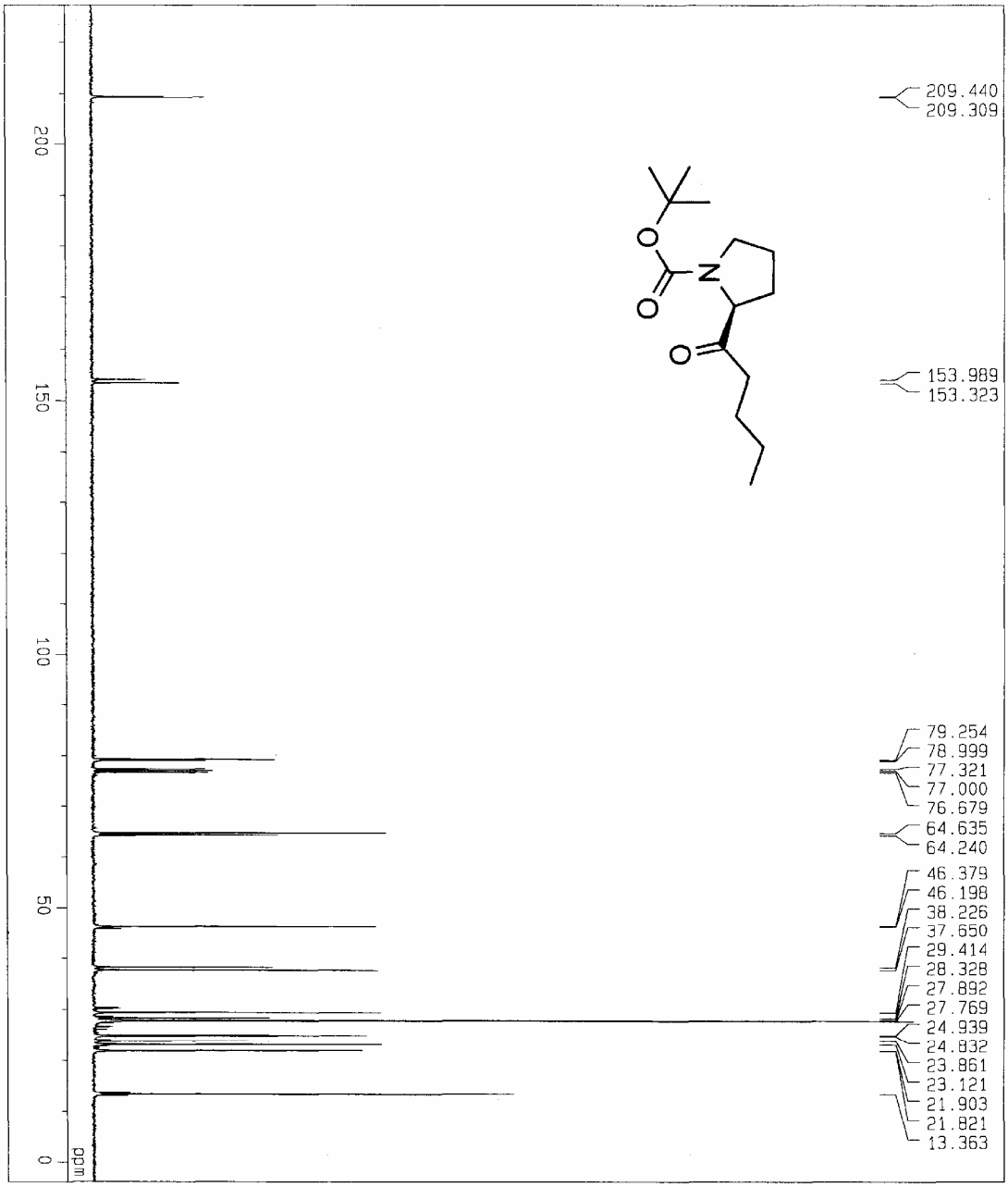

D:¥柴田¥Rxn318 6-2~11-1 1H.als
Rxn318 6-2~11-1 1H NMR
7.585
4.715
4.703
4.693
4.663
4.652
4.641
4.629
4.100
4.088
4.081
4.068
3.518
3.501
3.493
3.475
3.468
3.460
3.451
3.442
3.434
3.424
3.373
3.252
3.235
3.225
3.219
3.209
3.191
2.295
2.282
2.265
2.137
2.122
1.962
1.942
1.928
1.914
1.903
1.886
1.881
1.873
1.864
1.857
1.843
1.826
1.807
1.651
1.639
1.632
1.395
1.378
1.365
1.341
1.337
1.299
1.269
1.174
0.94
0.76
0.54
0.82
1.36
1.00
3.17
0.46
1.00
2.31
6.02
1.00
3.91
7.15
9.30
PPM
DATIM Fri Dec 08 18:33:35 2006
HELEV 74.40 %
N2LEV 83.60 %
SLVNT CDCL3
CTEMP 17.4 c
MENUF 1H
OBNUC 1H
OFR 395.75 MHz
OBSET 124.00 KHz
OBFIN 10277.00 Hz
PW1 6.60 usec
DEADT 72.60 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 32
DUMMY 1
FREQU 7912.96 Hz
FLT
DELAY 50.60 usec
ACQTM 4.1411 sec
PD 2.8590 sec
ADBIT 16
RGAIN 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD NON
EXPCM NON:Single couple:PW1_ACQTM_PD:1H,13
IRNUC 1H
IFR 395.75 MHz
IRSET 124.00 KHz
IRFIN 10277.00 Hz
IRRPW 50 usec
IRATN 511
DFILE D:¥柴田¥Rxn318 6-2~11-1 1H.als
SF THSATF62
LKSET 12.90 KHz
LKFIN 104.0 Hz
LKLEV 180
LGAIN 27
LKPHS 326
LKSIG 855
GSPED 11 Hz
FILDC
FILDF

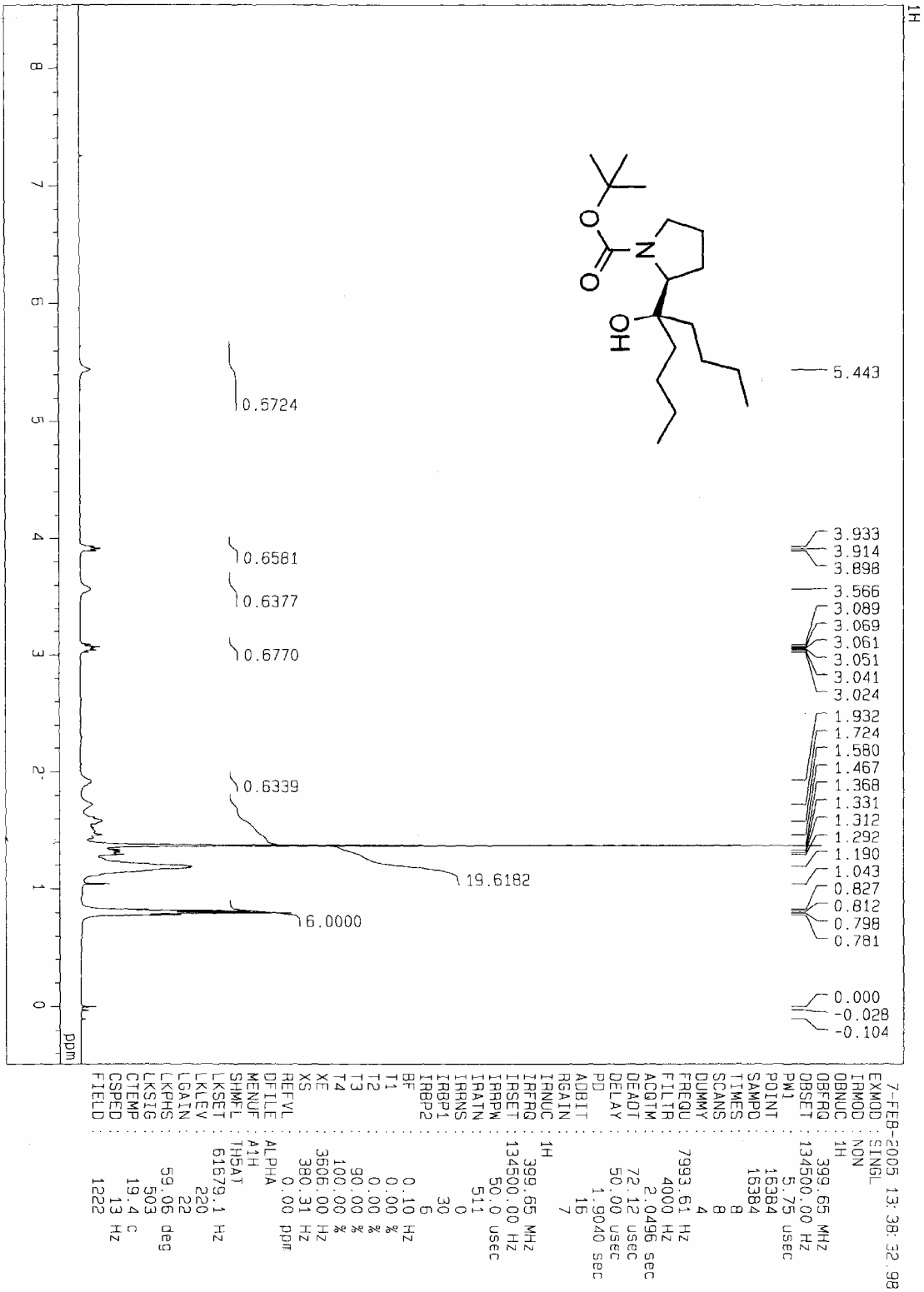
D:¥柴田¥Rxn318 6-2～11-1 13C.als
Rxn318 6-2～11-1 13C NMR

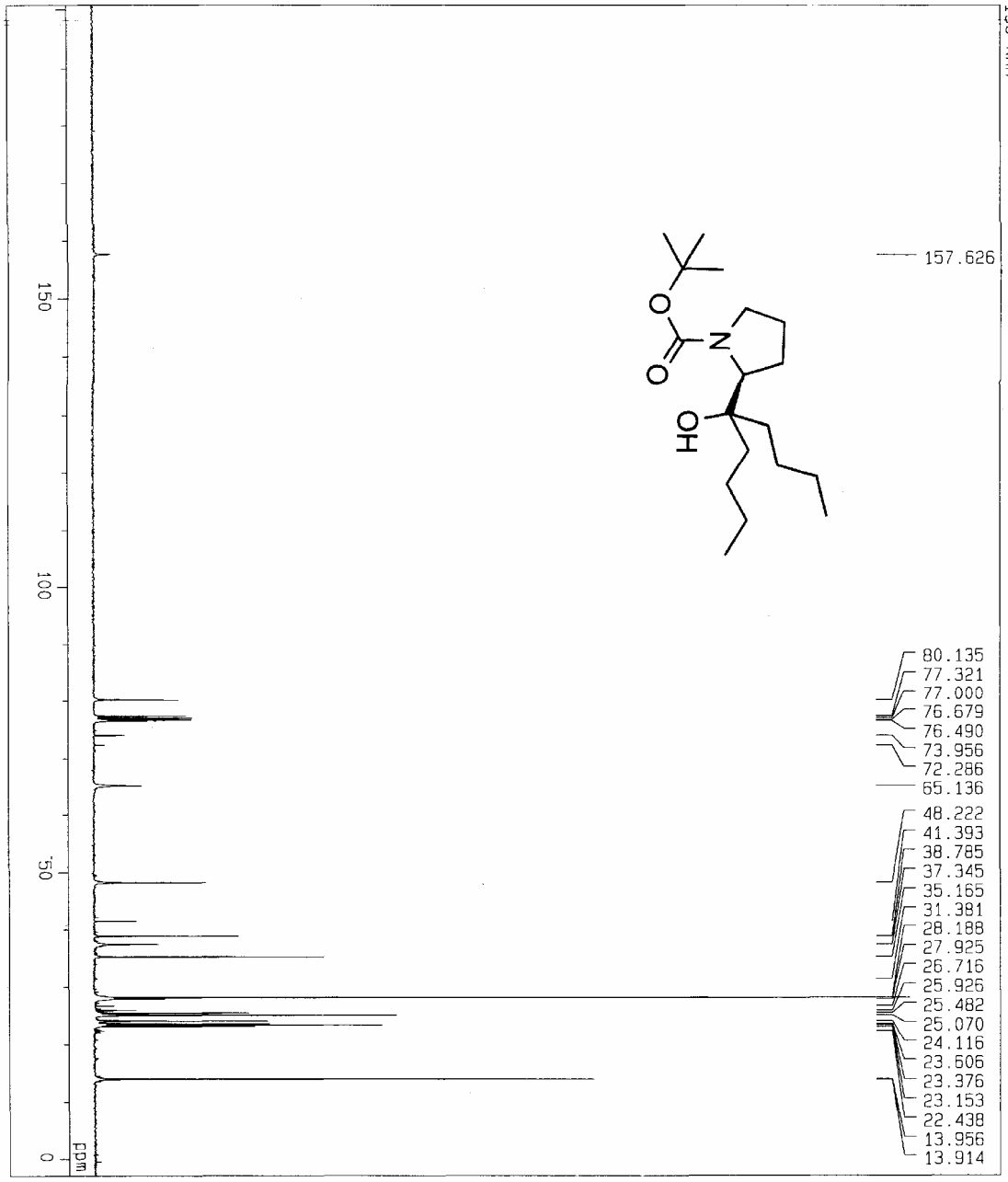


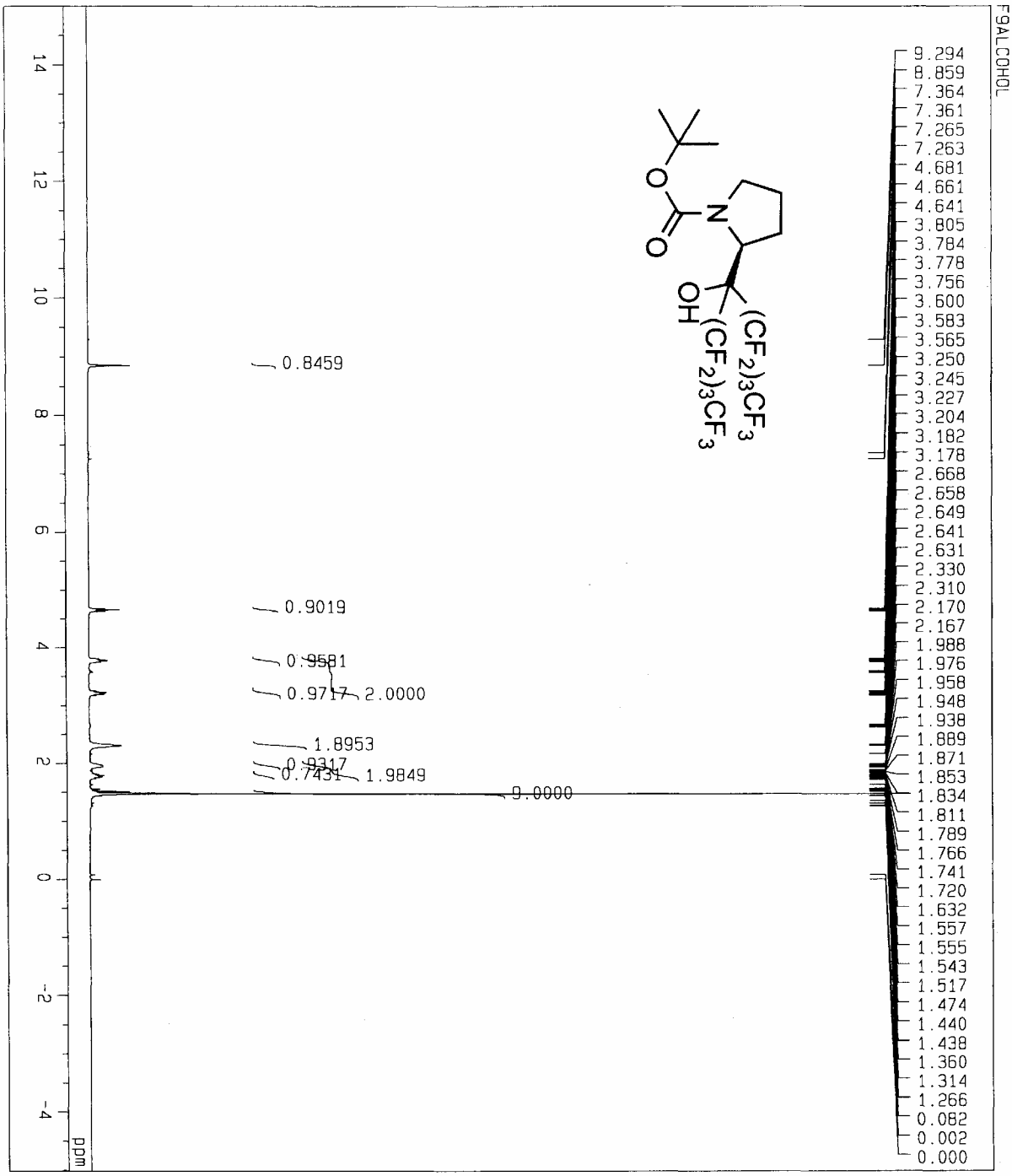
DATIM	Fri Dec 08 18:50:41 2006
HELEV	74.40 %
N2LEV	96.40 %
SLVNT	CDCL3
CTEMP	18.6 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1000
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn318 6-2～11-1 13C.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	878
CSPED	12 Hz
FILDC	
FILDF	


1H
1.0236
1.7207
1.6665
0.7994
2.5078
1.6852
10.4048
3.0000
ppm
7-FEB-2005 16:10:36.23
EXMOD : SINGL
IRMOD : NON
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
PW1 : 5.75 usec
POINT : 16384
SAMPO : 16384
TIMES : 8
SCANS : 8
DUMMY : 4
FREQU : 7993.61 Hz
FILTR : 4000 Hz
ACQTM : 2.0496 sec
DEADT : 72.12 usec
DELAY : 50.00 usec
PD : 1.9040 sec
ADBIT : 16
RGAIN : 7
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRRNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 0.10 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 4192.93 Hz
XS : 32.69 Hz
REFVL : 7.24 ppm
DFILE : ALPHA
MENUF : A1H
SHMFL : THSAT
LKSET : 51679.1 Hz
LKLEV : 220
LGAIN : 23
LKPHS : 59.06 deg
LKSIG : 759
CTEMP : 18.9 c
CSPED : 13 Hz
FIELD : 11B4


13C NMR
209.440
209.309
153.989
153.323
79.254
78.999
77.321
77.000
76.679
64.635
64.240
46.379
46.198
38.226
37.650
29.414
28.328
27.892
27.769
24.939
24.832
23.861
23.121
21.903
21.821
13.363
200
150
100
50
0
ppm
7-FEB-2005 16:25:51.24
EXMOD : SINGL
IRMOD : BCM
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
PW1 : 4.90 usec
POINT : 32768
SAMPO : 32768
TIMES : 1000
SCANS : 153
DUMMY : 2
FREQU : 27100.27 Hz
FILTR : 13550 Hz
ACQTM : 1.2091 sec
DEADT : 19.69 usec
DELAY : 14.76 usec
PD : 2.5000 sec
ADBIT : 16
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRRNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 23198.32 Hz
XS : -1141.31 Hz
REFVL : 77.00 ppm
DFILE : ALPHA
MENUF : A13C
SHMFL : THSAT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 23
LKPHS : 59.06 deg
LKSIG : 759
CTEMP : 19.9 c
CSPED : 14 Hz
FIELD : 1184

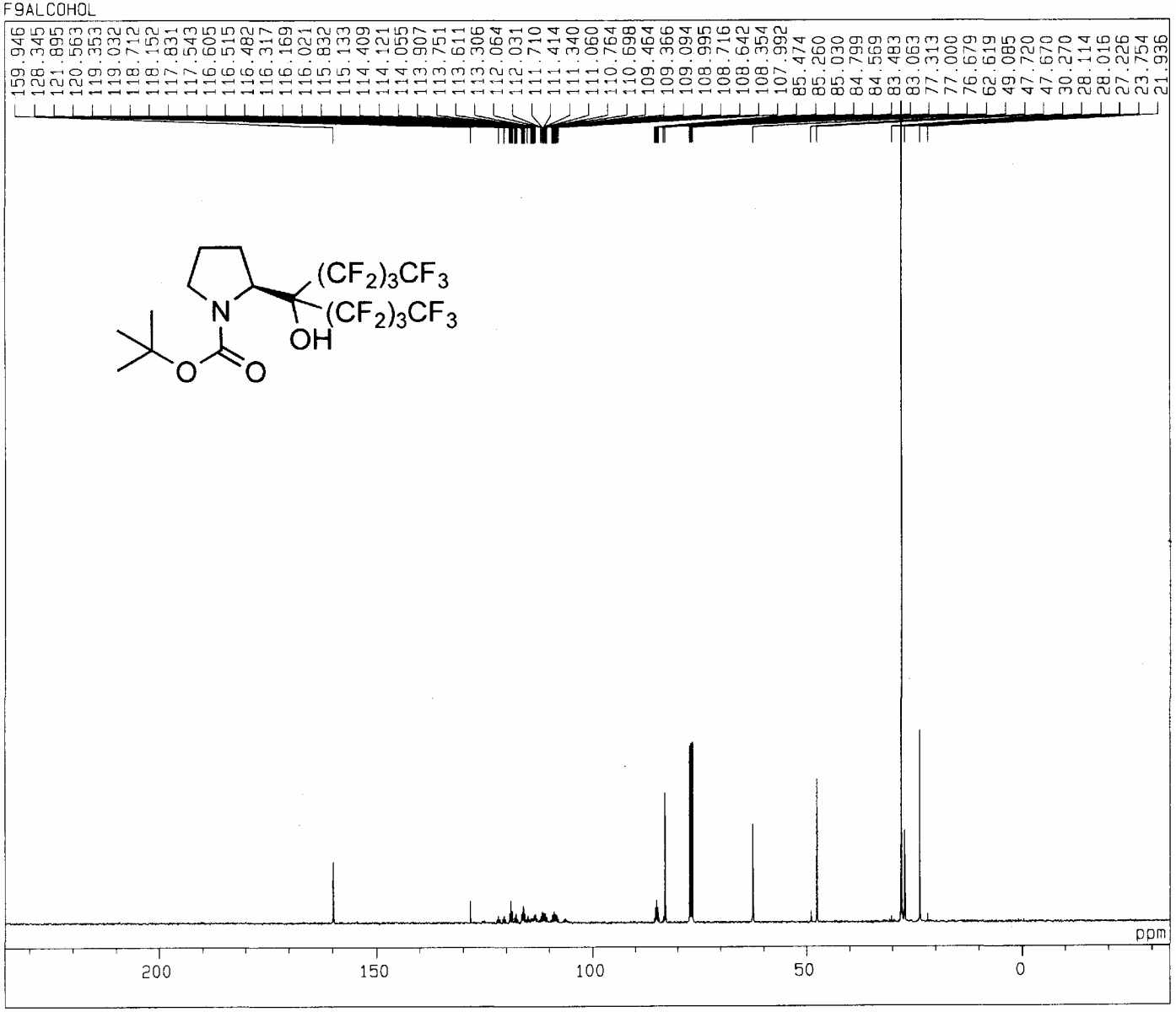

1H
5.443
0.5724
3.933
3.914
3.898
0.6581
0.6377
3.566
3.089
3.069
3.061
3.051
3.041
3.024
0.6770
1.932
1.724
1.580
1.467
1.368
1.331
1.312
1.292
1.190
0.6339
19.6182
1.043
0.827
0.812
0.798
0.781
6.0000
0.000
-0.028
-0.104
ppm
7-FEB-2005 13:38:32.98
EXMOD : SINGL
IRMOD : NON
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
PW1 : 5.75 usec
POINT : 16384
SAMPD : 16384
TIMES : 8
SCANS : 8
DUMMY : 4
FREQU : 7993.61 Hz
FILTR : 4000 Hz
ACQTM : 2.0496 sec
DEADT : 72.12 usec
DELAY : 50.00 usec
PD : 1.9040 sec
ADBIT : 16
RGAIN : 7
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRPPW : 50.0 usec
IRATN : 511
IRNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 0.10 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 3606.00 Hz
XS : 380.31 Hz
REFVL : 0.00 ppm
DFILE : ALPHA
MENUF : A1H
SHMFL : THSAT
LKSET : 61579.1 Hz
LKLEV : 220
LGAIN : 22
LKPHS : 59.06 deg
LKSIG : 503
CTEMP : 19.4 C
CSPED : 13 Hz
FIELD : 1222


13C NMR
157.626
80.135
77.321
77.000
76.679
76.490
73.956
72.286
65.136
48.222
41.393
38.785
37.345
35.165
31.381
28.188
27.925
26.716
25.926
25.482
25.070
24.116
23.606
23.376
23.153
22.438
13.956
13.914
150
100
50
0
ppm
OH
N
O
O
7-FEB-2005 13:55:27.05
EXMOD : SINGL
IRMOD : BCM
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
PW1 : 4.90 usec
POINT : 32768
SAMPO : 32768
TIMES : 1000
SCANS : 202
DUMMY : 2
FREQU : 27100.27 Hz
FILTR : 13550 Hz
ACQTM : 1.2091 sec
DEADT : 19.69 usec
DELAY : 14.76 usec
PD : 2.5000 sec
ADBIT : 16
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRRNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 20473.24 Hz
XS : 147.63 Hz
REFVL : 77.00 ppm
DFILE : ALPHA
MENUF : A13C
SHMFL : TH5AT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 22
LKPHS : 59.06 deg
LKSIG : 503
CTEMP : 19.6 C
CSPED : 14 Hz
FIELD : 1222

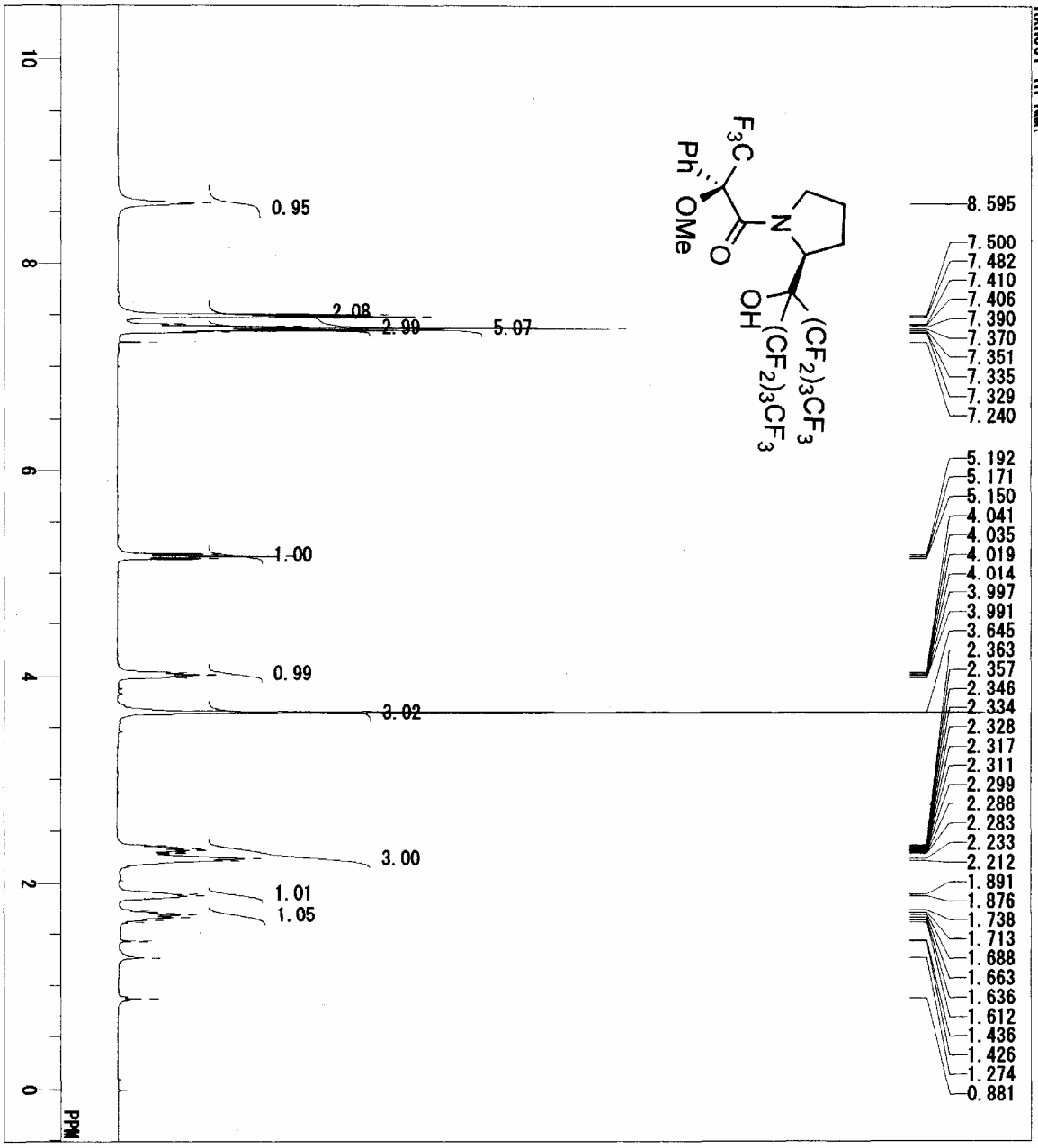


3-FEB-2003 17:09:21.21
EXMOD : SINGL
IRMOD : NON
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
PW1 : 5.75 usec
POINT : 16384
SAMPO : 16384
TIMES : 8
SCANS : 8
DUMMY : 4
FREQU : 7993.61 Hz
FILTR : 4000 Hz
ACQTM : 2.0496 sec
DEADT : 72.12 usec
DELAY : 50.00 usec
PD : 1.9040 sec
ADBIT : 16
RGAIN : 13
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRNNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 0.10 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 7993.61 Hz
XS : 0.00 Hz
REFVL : 0.00 ppm
DFILE : ALPHA
MENUF : A1H
SHMFL : TH5AT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 22
LKPHS : 66.09 deg
LKSIG : 937
CTEMP : 19.5 c
CSPED : 13 Hz
FIELD : -674

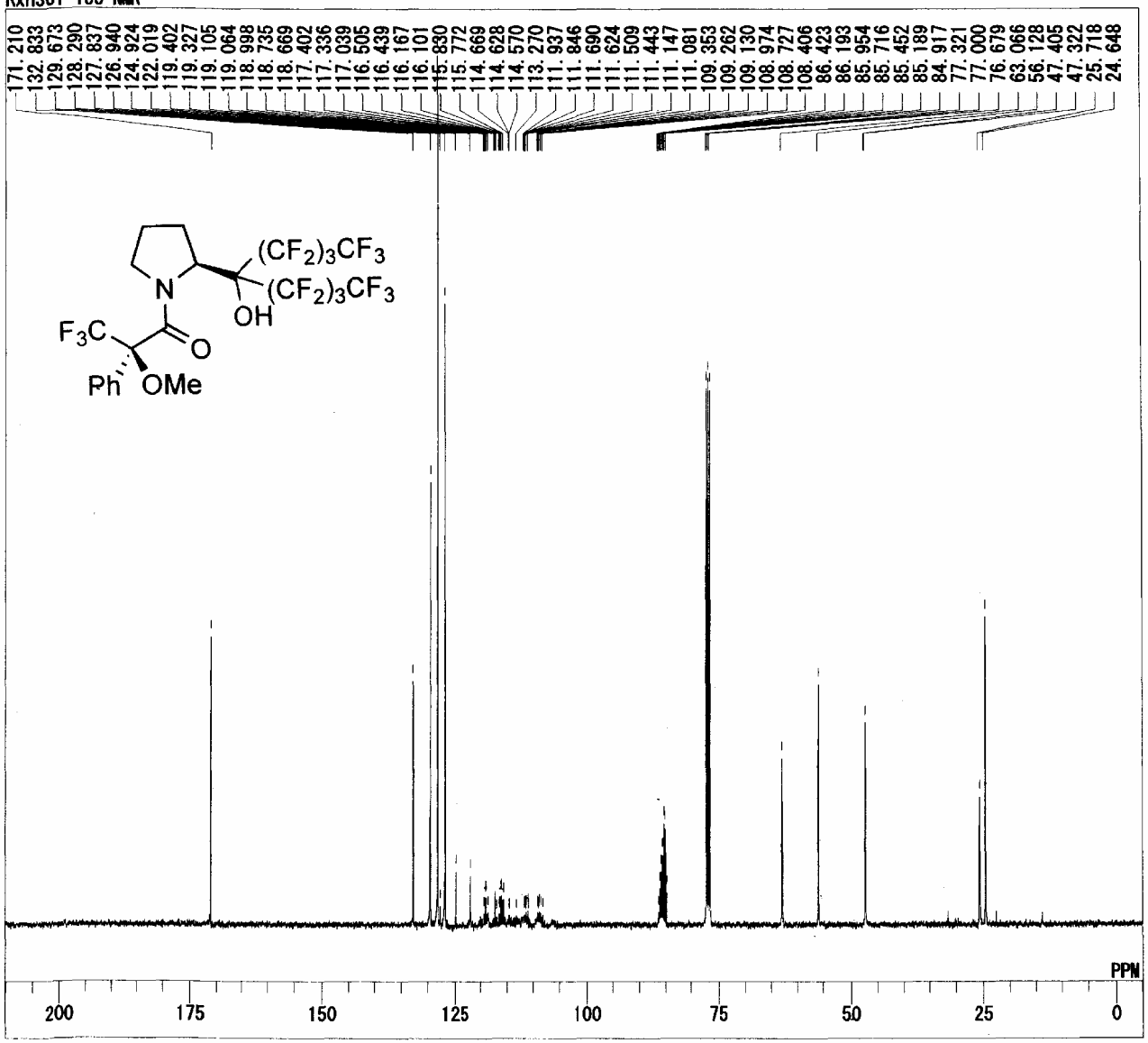
F9ALCOHOL




```
 4-FEB-2003 00: 40: 39.17
EXMOD : SINGL
IRMOD : BCM
OBNUC : 13C
OBFRQ :   100.40 MHz
OBSET : 135500.00 Hz
PW1   :     4.90 usec
POINT :    32768
SAMPO :    32768
TIMES :     2048
SCANS :     1438
DUMMY :        2
FREQU : 27100.27 Hz
FILTR :    13550 Hz
ACQTM :      1.2091 sec
DEADT :    19.69 usec
DELAY :    14.76 usec
PD    :      2.5000 sec
ADBIT :       16
RGAIN :       23
IRNUC : 1H
IRFRQ :  399.65 MHz
IRSET : 134500.00 Hz
IRRPW :     50.0 usec
IRATN :      511
IRRNS :        0
IRBP1 :       30
IRBP2 :        6
BF    :     2.00 Hz
T1    :     0.00 %
T2    :     0.00 %
T3    :    90.00 %
T4    :   100.00 %
XE    : 27100.27 Hz
XS    :     0.00 Hz
REFVL :    77.00 ppm
DFILE : ALPHA
MENUF : A13C
SHMFL : TH5AT
LKSET :  61679.1 Hz
LKLEV :      220
LGAIN :       23
LKPHS :    66.09 deg
LKSIG :     1071
CTEMP :     20.1 c
CSPED :       13 Hz
FIELD :     -673
```

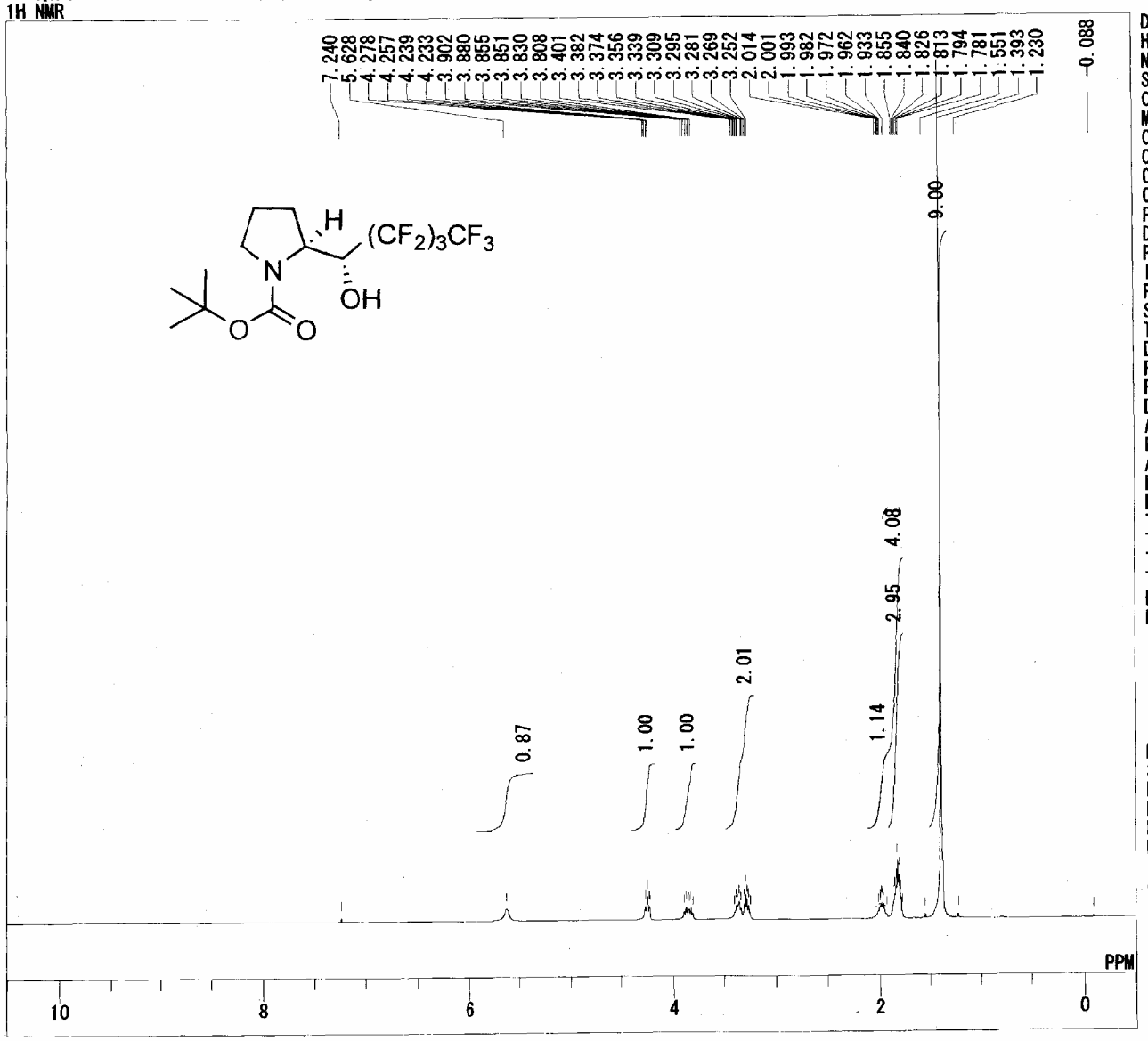

D:¥集田¥Rxn361 1H.als
Rxn361 1H NMR
8.595
7.500
7.482
7.410
7.406
7.390
7.370
7.351
7.335
7.329
7.240
5.192
5.171
5.150
4.041
4.035
4.019
4.014
3.997
3.991
3.645
2.363
2.357
2.346
2.334
2.328
2.317
2.311
2.299
2.288
2.283
2.233
2.212
1.891
1.876
1.738
1.713
1.688
1.663
1.636
1.612
1.436
1.426
1.274
0.881
0.95
2.08
2.99
5.07
1.00
0.99
3.02
3.00
1.01
1.05
Ph
OMe
OH
$(CF_2)_3CF_3$
$(CF_2)_3CF_3$
PPM
DATIM Tue Feb 27 10:38:13 2007
HELEV 76.80 %
N2LEV 92.80 %
SLVNT CDCL3
CTEMP 20.7 c
MENUF 1H
OBNUC 1H
OFR 395.75 MHz
OBSET 124.00 KHz
OBFIN 10277.00 Hz
PW1 6.60 usec
PW2 10.00 usec
DEADT 72.60 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 32
SCANS 32
DUMMY 1
FREQU 7912.96 Hz
FLT
DELAY 50.60 usec
ACQTM 4.1411 sec
PD 2.8590 sec
ADBIT 16
RGAIN 10
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM_PD:1H, 13
IRNUC 1H
IFR 395.75 MHz
IRSET 124.00 KHz
IRFIN 10277.00 Hz
IRRPW 50 usec
IRATN 511
DFILE D:¥集田¥Rxn361 1H.als
SF TH5ATF02
LKSET 12.90 KHz
LKFIN 104.0 Hz
LKLEV 180
LGAIN 26
LKPHS 326
LKSIG 651
CSPED 14 Hz
FILDC
FILDF

D:¥柴田¥Rxn361 13C.als
Rxn361 13C NMR



DATIM	Tue Feb 27 11:39:28 2007
HELEV	76.80 %
N2LEV	92.40 %
SLVNT	CDCL3
CTEMP	20.6 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
PW2	10.00 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1500
SCANS	1211
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn361 13C.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	674
CSPED	14 Hz
FILDC	
FILDF	

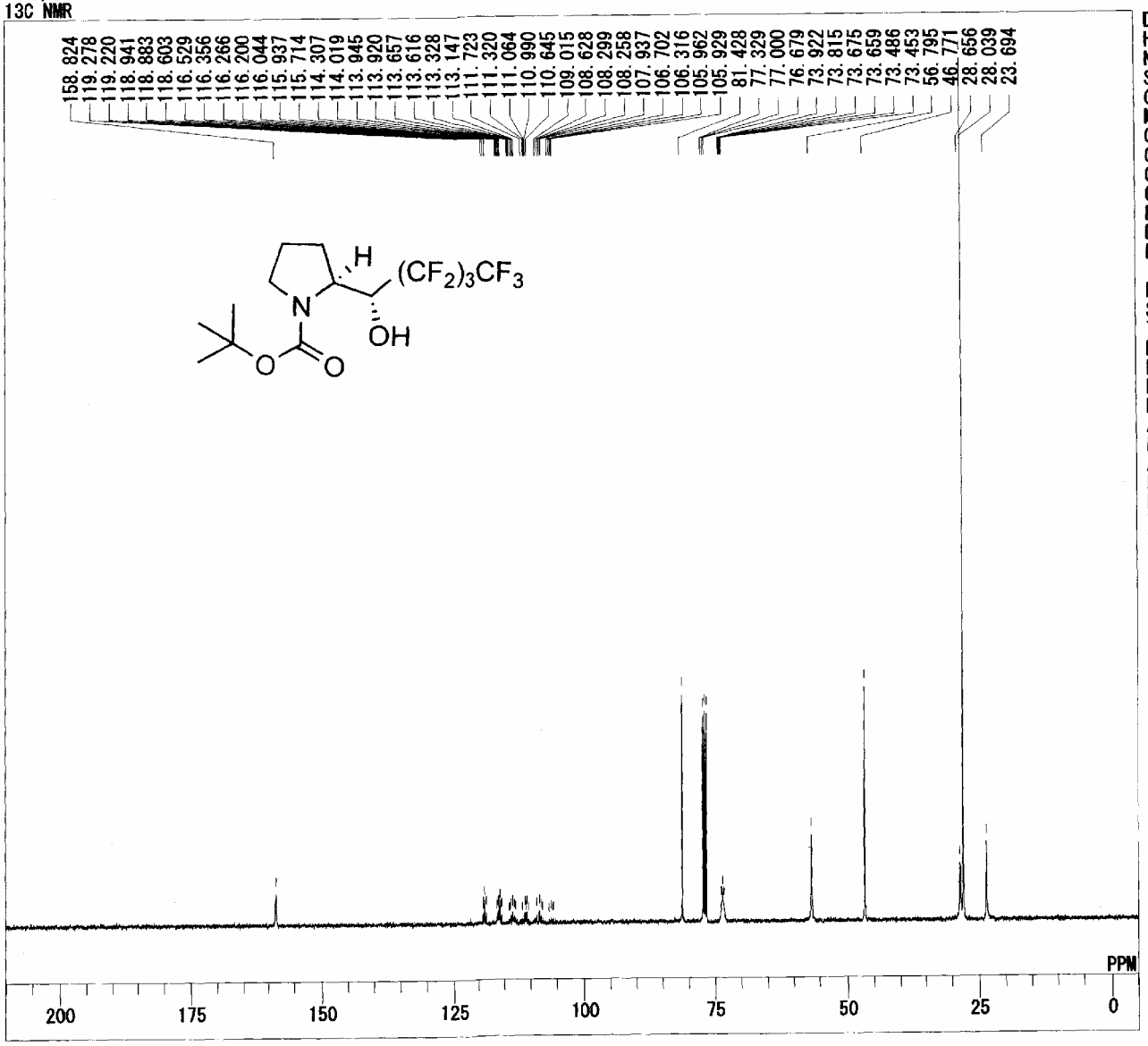
D:¥柴田¥mono-C4F9 alcohol (aR, 2S) (major isomer) 1H.
1H NMR



DATIM	Tue Nov 21 14:58:07 2006
HELEV	80.00 %
N2LEV	91.20 %
SLVNT	CDCL3
CTEMP	15.3 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	32
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	6
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥mono-C4F9 alcohol (aR, 2S) (m
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	628
CSPED	12 Hz
FILDC	
FILDF	

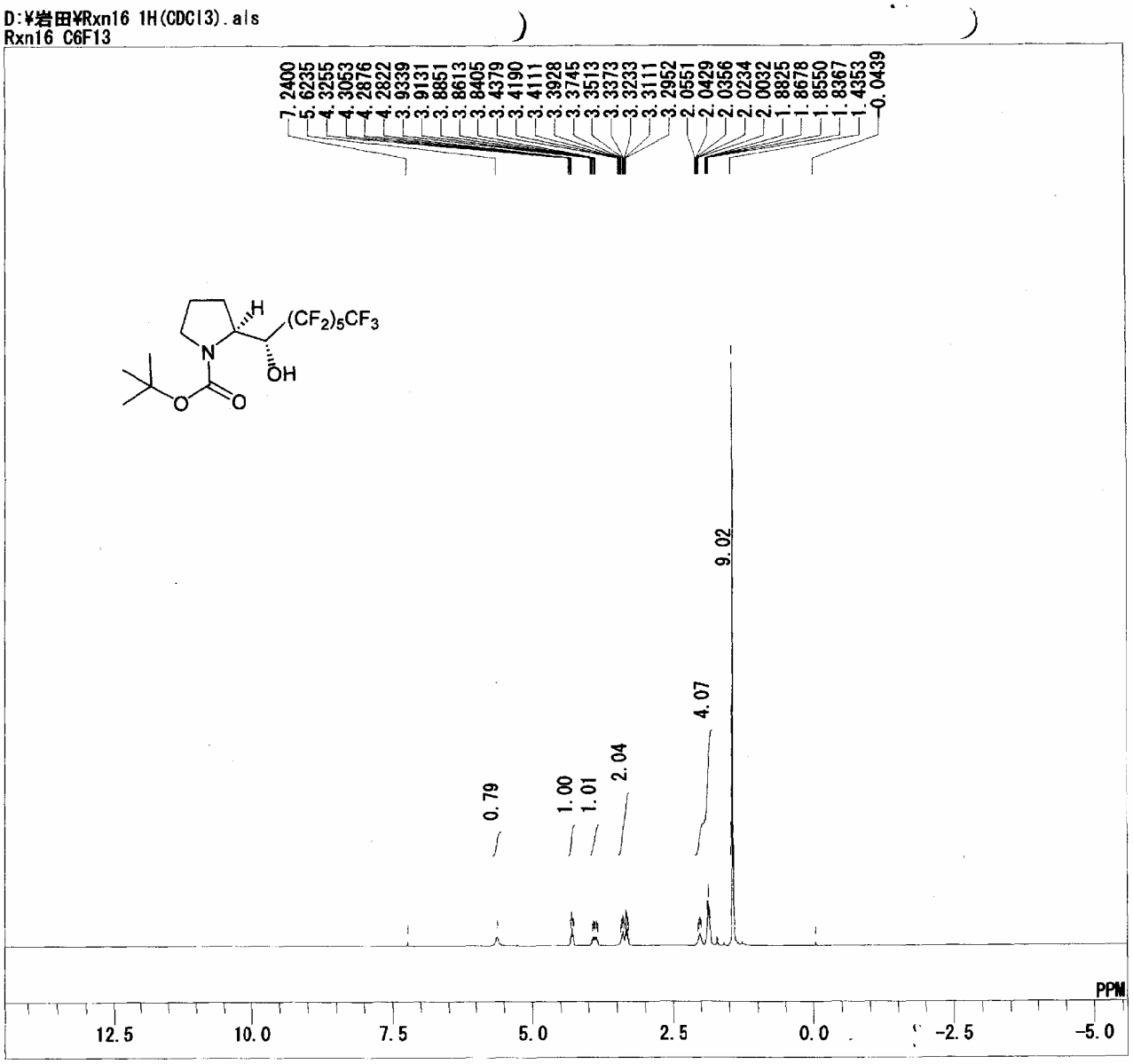
D:¥柴田¥mono-C4F9 alcohol (aR, 2S) (major isomer) 13C D.als

13C NMR



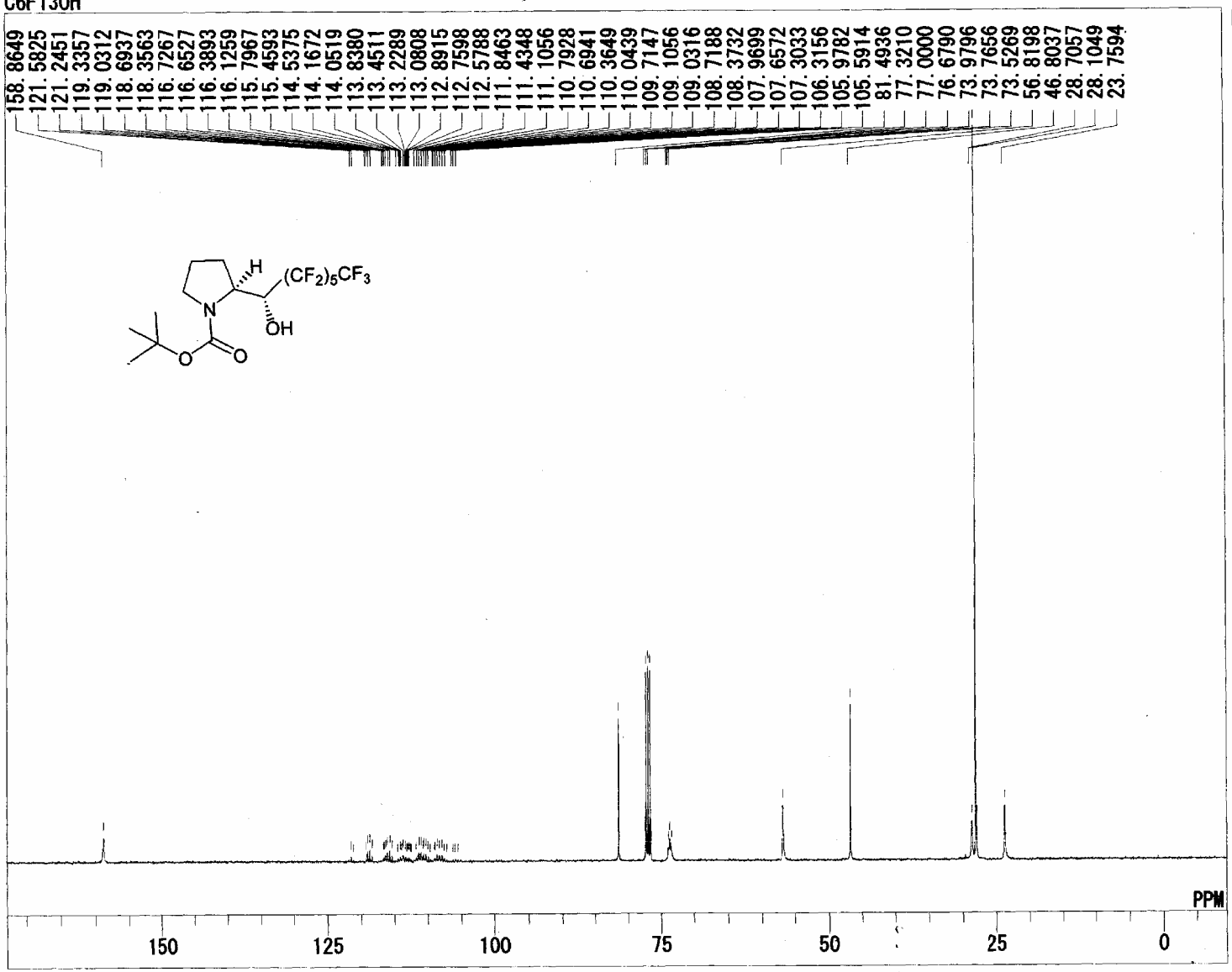
DATIM	Tue Nov 21 15:18:10 2006
HELEV	80.00 %
N2LEV	90.80 %
SLVNT	CDCL3
CTEMP	18.0 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1000
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥mono-C4F9 alcohol (aR, 2S) (m
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	645
CSPED	12 Hz
FILDC	
FILDF	

D:¥岩田¥Rxn16 1H(CDCl3).als
Rxn16 C6F13

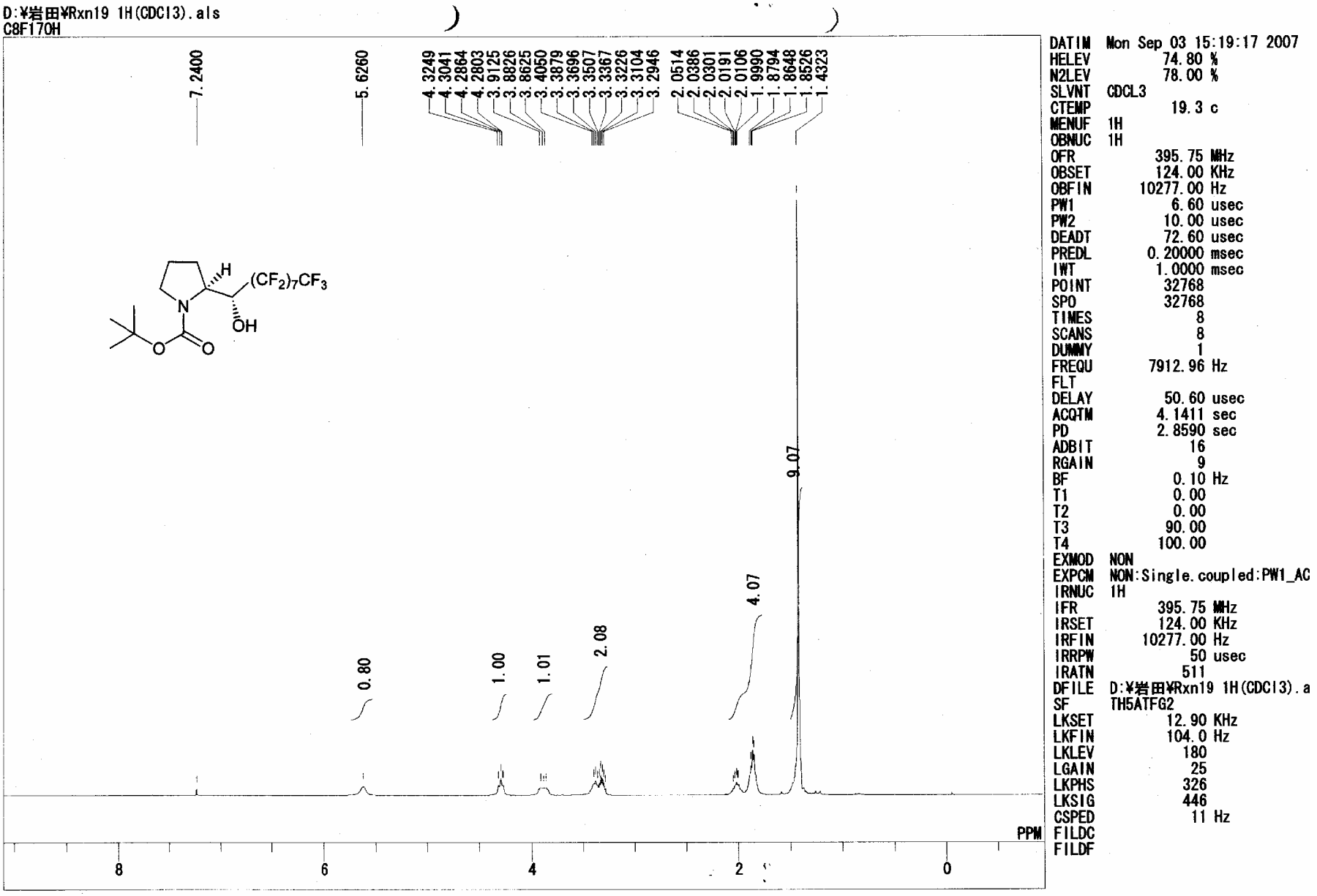


DATIM	Mon Aug 20 16:09:29 2007
HELEV	80.40 %
N2LEV	78.00 %
SLVNT	CDCL3
CTEMP	22.5 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
PW2	10.00 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	8
SCANS	8
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	9
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥岩田¥Rxn16 1H(CDCl3).als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	25
LKPHS	326
LKSIG	465
CSPED	14 Hz
FILDC	
FILDF	

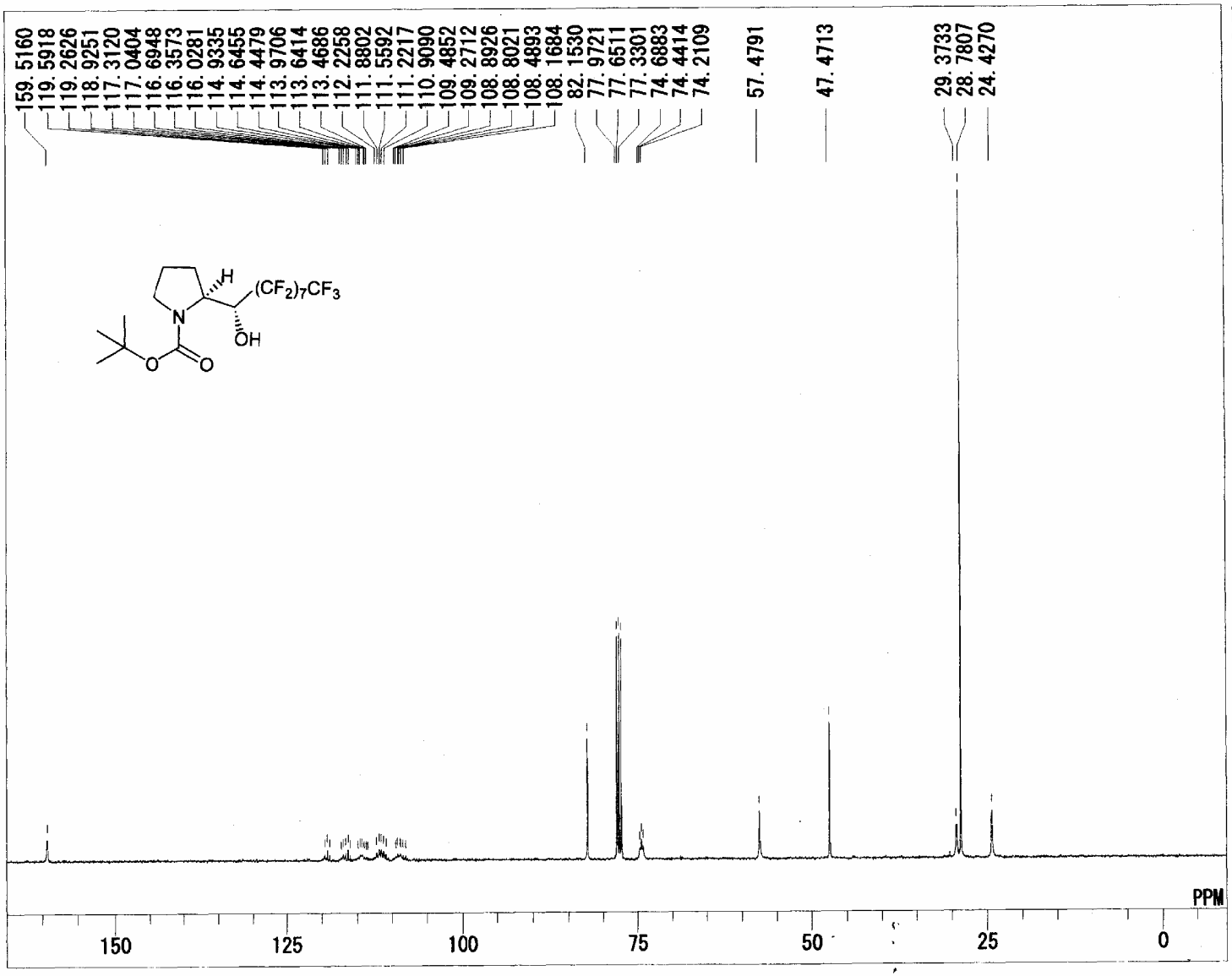
D:¥岩田¥Rxn16 13C(CDCl3).als
C6F13OH



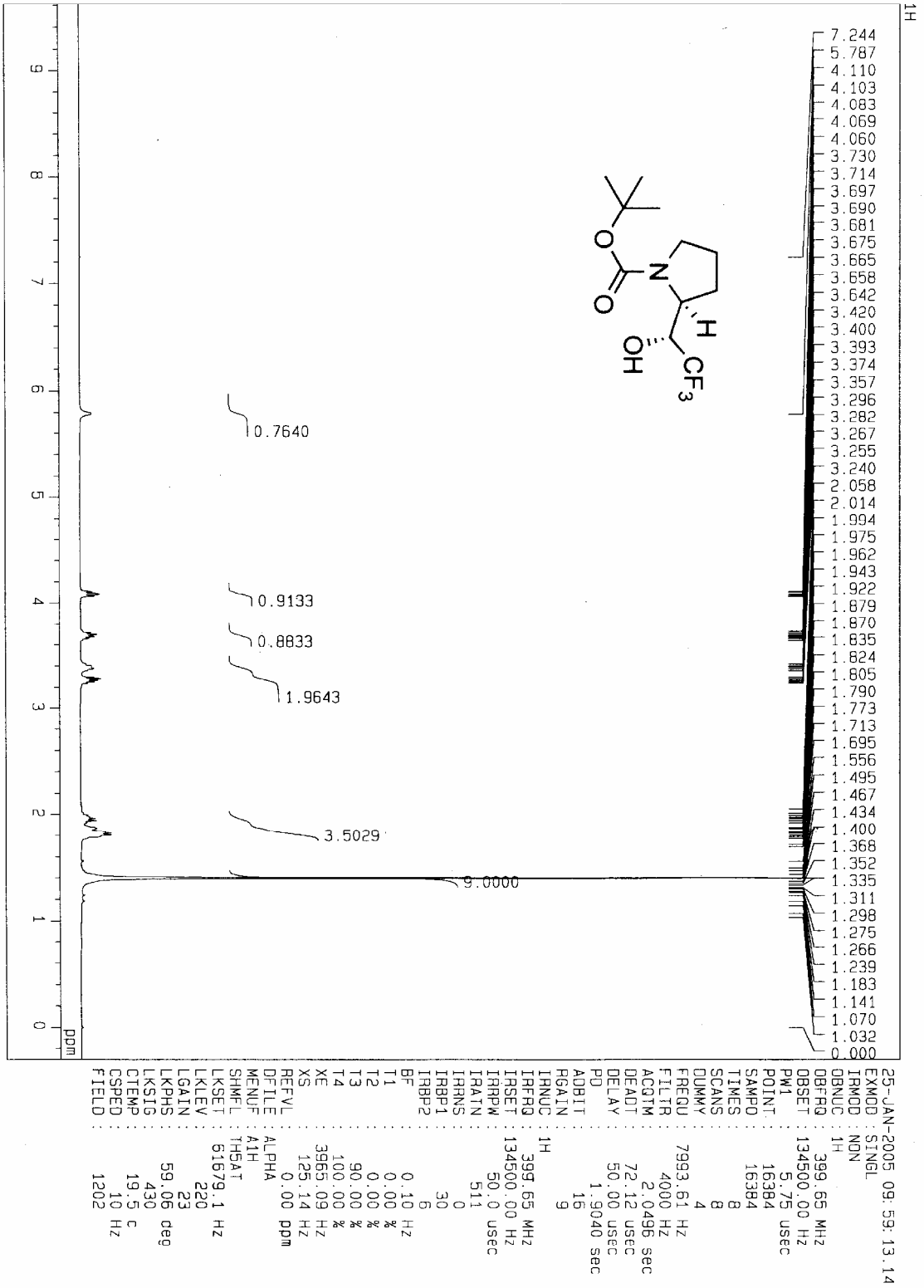
MENUF	13C BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.80 usec
DEADT	20.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1440
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	26
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.d
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥岩田¥Rxn16 13C(
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	473
CSPED	10 Hz
FILDC	
FILDF	

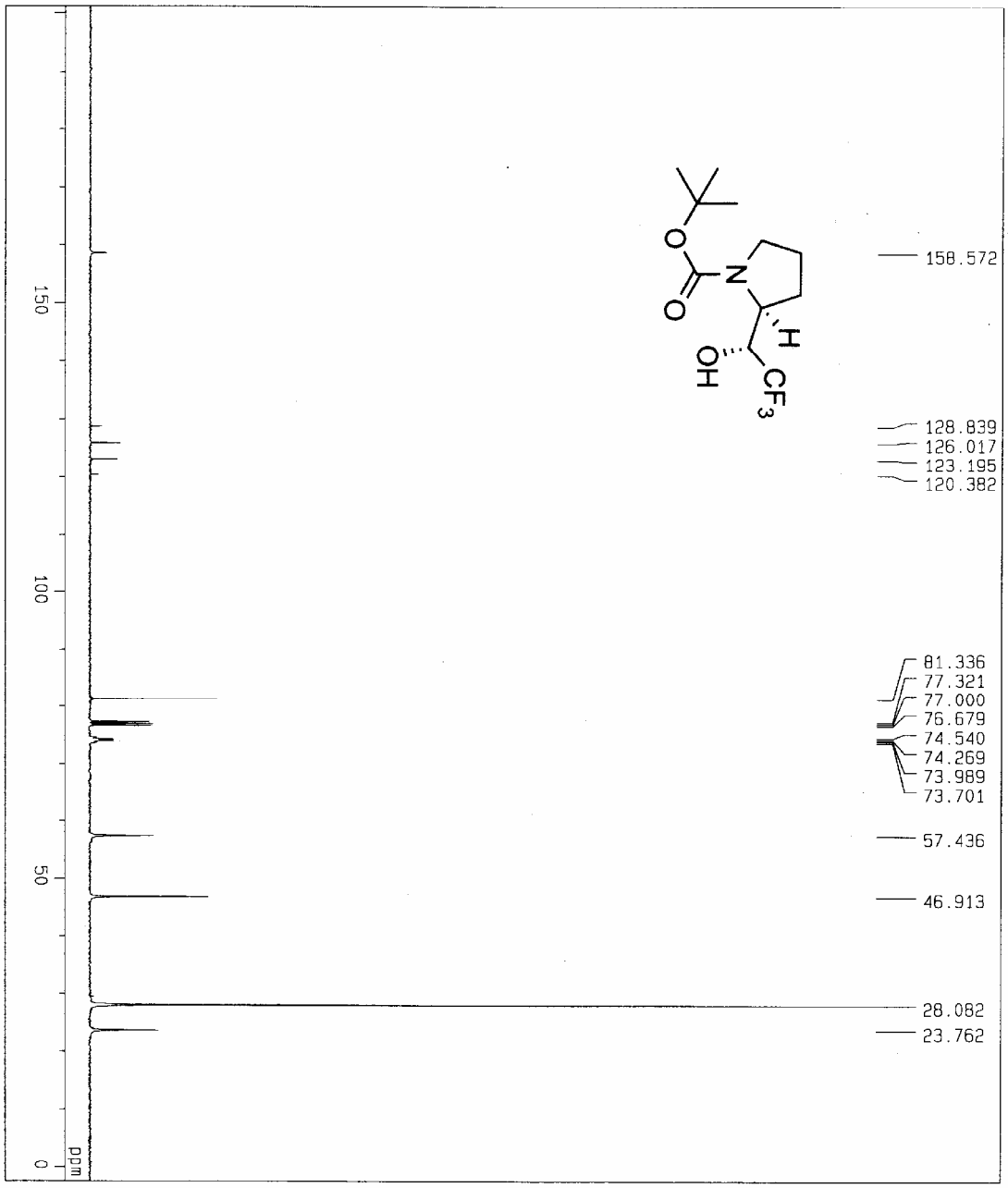

D:¥岩田¥Rxn19 1H(CDCl3).als
C8F17OH
7.2400
5.6260
4.3249
4.3041
4.2864
4.2803
3.9125
3.8826
3.8625
3.4050
3.3879
3.3696
3.3507
3.3367
3.3226
3.3104
3.2946
2.0514
2.0386
2.0301
2.0191
2.0106
1.9990
1.8794
1.8648
1.8526
1.4323
0.80
1.00
1.01
2.08
4.07
9.07
PPM
8
6
4
2
0
DATIM Mon Sep 03 15:19:17 2007
HELEV 74.80 %
N2LEV 78.00 %
SLVNT CDCL3
CTEMP 19.3 c
MENUF 1H
OBNUC 1H
OFR 395.75 MHz
OBSET 124.00 KHz
OBFIN 10277.00 Hz
PW1 6.60 usec
PW2 10.00 usec
DEADT 72.60 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 8
SCANS 8
DUMMY 1
FREQU 7912.96 Hz
FLT
DELAY 50.60 usec
ACQTM 4.1411 sec
PD 2.8590 sec
ADBIT 16
RGAIN 9
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_AC
IRNUC 1H
IFR 395.75 MHz
IRSET 124.00 KHz
IRFIN 10277.00 Hz
IRRPW 50 usec
IRATN 511
DFILE D:¥岩田¥Rxn19 1H(CDCl3).a
SF TH5ATFG2
LKSET 12.90 KHz
LKFIN 104.0 Hz
LKLEV 180
LGAIN 25
LKPHS 326
LKSIG 446
CSPED 11 Hz
FILDC
FILDF

C:¥WINNMR98¥COMMON¥_DEFAULT.ALS

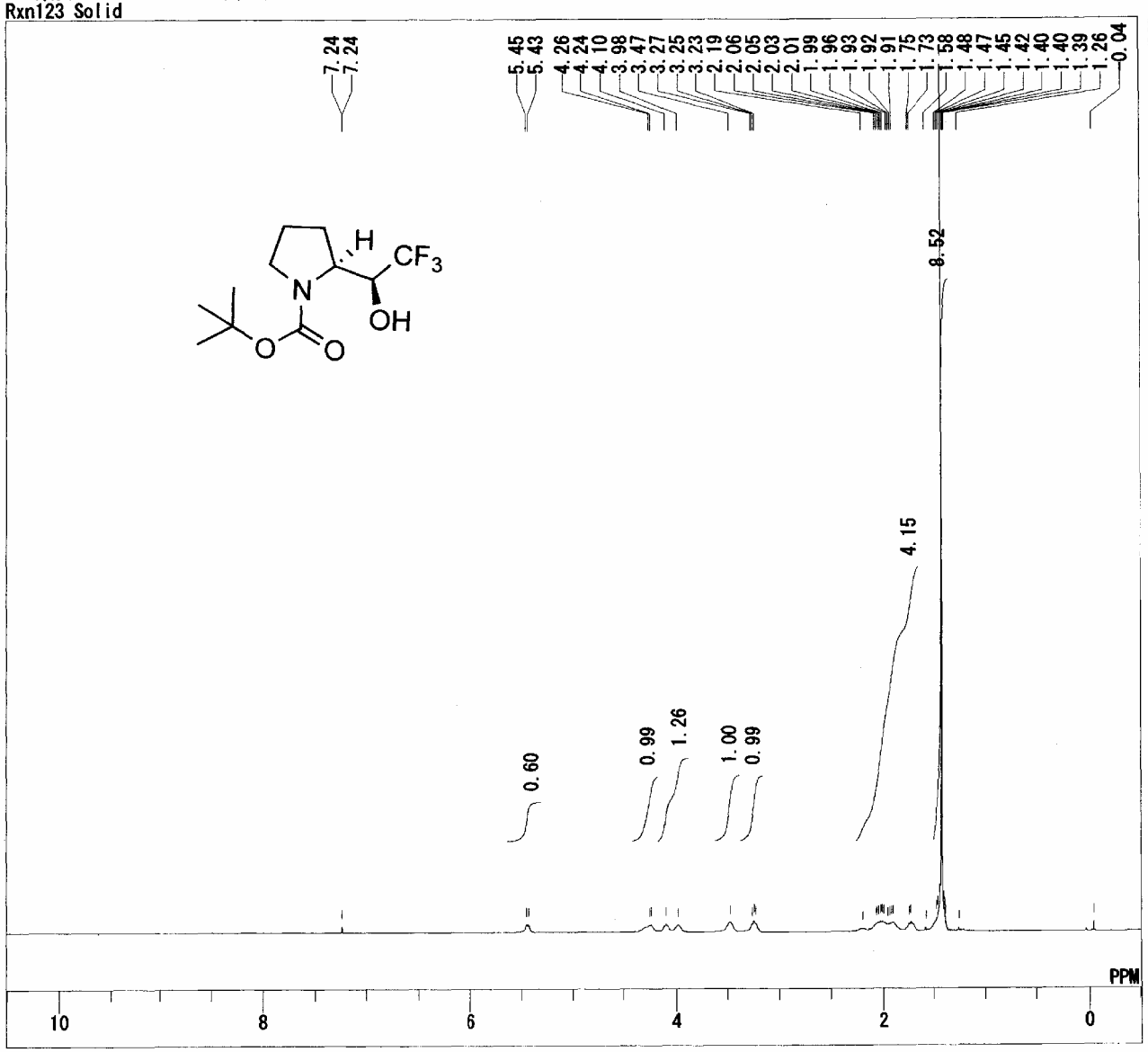


MENUF	13C BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.80 usec
DEADT	20.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1520
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	26
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.d
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	C:¥WINNMR98¥COMMON
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	447
CSPED	11 Hz
FILDC	
FILDF	


1H
7.244
5.787
4.110
4.103
4.083
4.069
4.060
3.730
3.714
3.697
3.690
3.681
3.675
3.665
3.658
3.642
3.420
3.400
3.393
3.374
3.357
3.296
3.282
3.267
3.255
3.240
2.058
2.014
1.994
1.975
1.962
1.943
1.922
1.879
1.870
1.835
1.824
1.805
1.790
1.773
1.713
1.695
1.556
1.495
1.467
1.434
1.400
1.368
1.352
1.335
1.311
1.298
1.275
1.266
1.239
1.183
1.141
1.070
1.032
0.000
0.7640
0.9133
0.8833
1.9643
3.5029
9.0000
OH
CF3
ppm
25-JAN-2005 09:59:13.14
EXMOD : SINGL
IRMOD : NON
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
PW1 : 5.75 usec
POINT : 16384
SAMPO : 16384
TIMES : 8
SCANS : 8
DUMMY : 4
FREQU : 7993.61 Hz
FILTR : 4000 Hz
ACQTM : 2.0496 sec
DEADT : 72.12 usec
DELAY : 50.00 usec
PD : 1.9040 sec
ADBIT : 16
RGAIN : 9
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRRNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 0.10 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 3965.09 Hz
XS : 125.14 Hz
REFVL : 0.00 ppm
DFILE : ALPHA
MENUF : A1H
SHMFL : THSAT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 23
LKPHS : 59.06 deg
LKSIG : 430
CTEMP : 19.5 c
CSPED : 10 Hz
FIELD : 1202

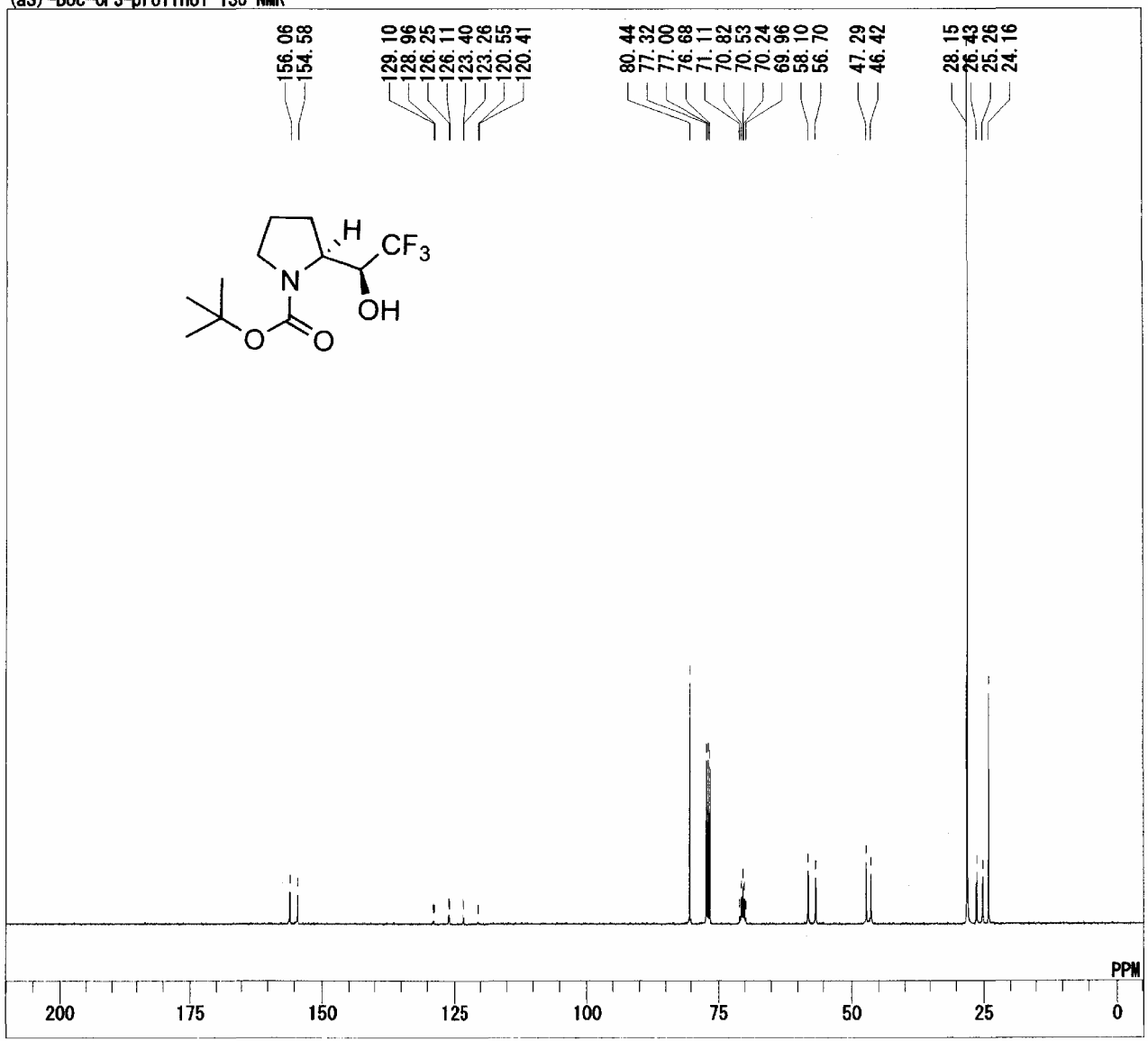

13C NMR
158.572
128.839
126.017
123.195
120.382
81.336
77.321
77.000
76.679
74.540
74.269
73.989
73.701
57.436
46.913
28.082
23.762
150
100
50
0
ppm
25-JAN-2005 10:14:36.35
EXMOD : SINGL
IRMOD : BCM
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
PW1 : 4.90 usec
POINT : 32768
SAMPO : 32768
TIMES : 1000
SCANS : 160
DUMMY : 2
FREQU : 27100.27 Hz
FILTR : 13550 Hz
ACQTM : 1.2091 sec
DEADT : 19.69 usec
DELAY : 14.76 usec
PD : 2.5000 sec
ADBIT : 16
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRRPW : 50.0 usec
IRATN : 511
IRNS : 0
IRBP1 : 30
IRBP2 : 6
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
XE : 20473.24 Hz
XS : 111.24 Hz
REFVL : 77.00 ppm
DFILE : ALPHA
MENUF : A13C
SHMFL : TH5AT
LKSET : 61679.1 Hz
LKLEV : 220
LGAIN : 23
LKPHS : 59.05 deg
LKSIG : 430
CTEMP : 20.8 c
CSPED : 13 Hz
FIELD : 1202

D:¥柴田¥Boc-CF3-Solid-(S, S) 1H.als
Rxn123 Solid

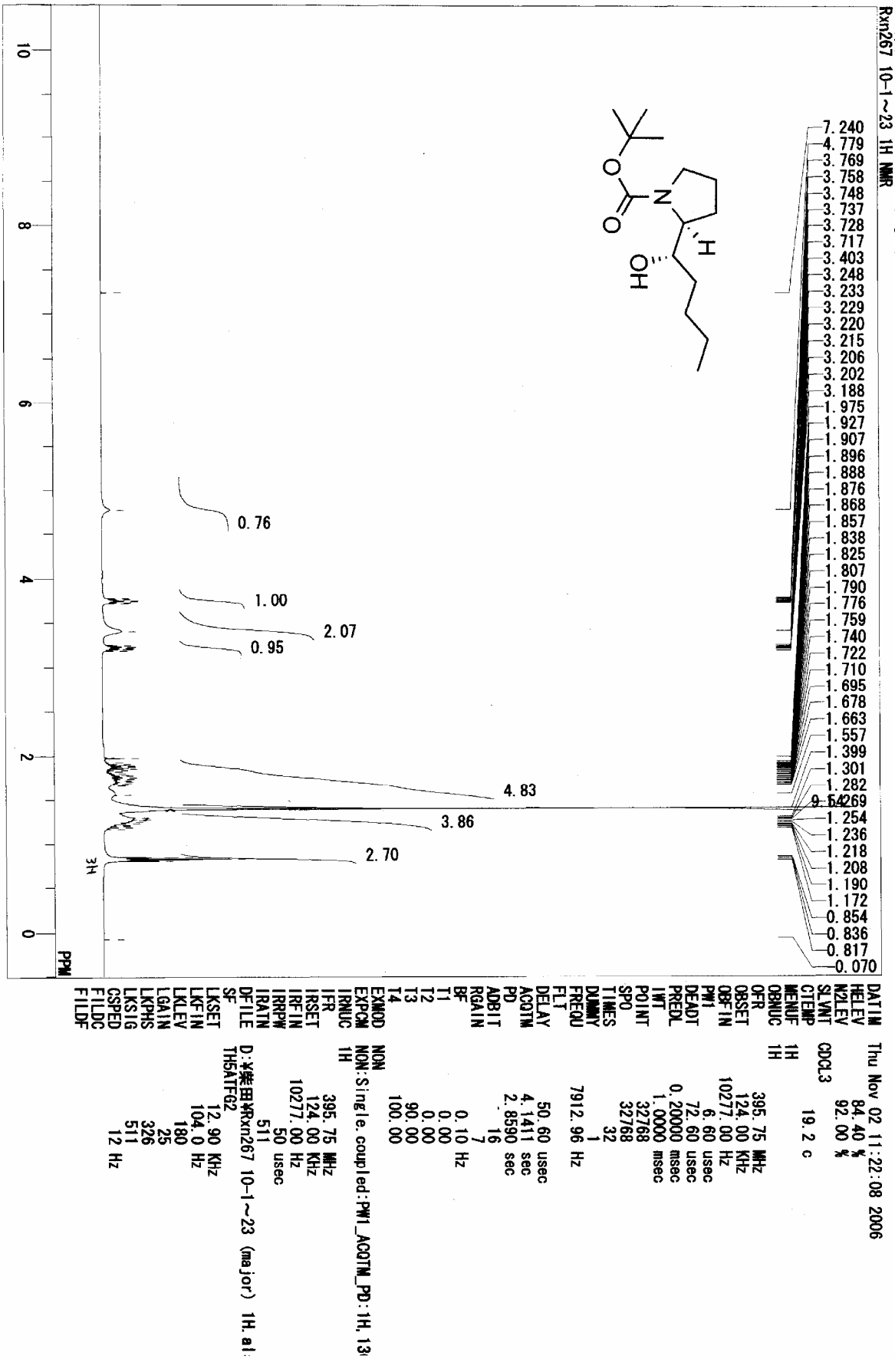


DATIM	Mon Oct 03 17:08:14 2005
HELEV	93.60 %
N2LEV	68.80 %
SLVNT	CDCL3
CTEMP	22.2 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.50 usec
PW2	10.00 usec
DEADT	72.70 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	8
SCANS	8
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	4.9290 sec
ADBIT	16
RGAIN	9
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Boc-CF3-Solid-(S, S) 1H.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	65
LKSIG	576
CSPED	13 Hz
FILDC	
FILDF	

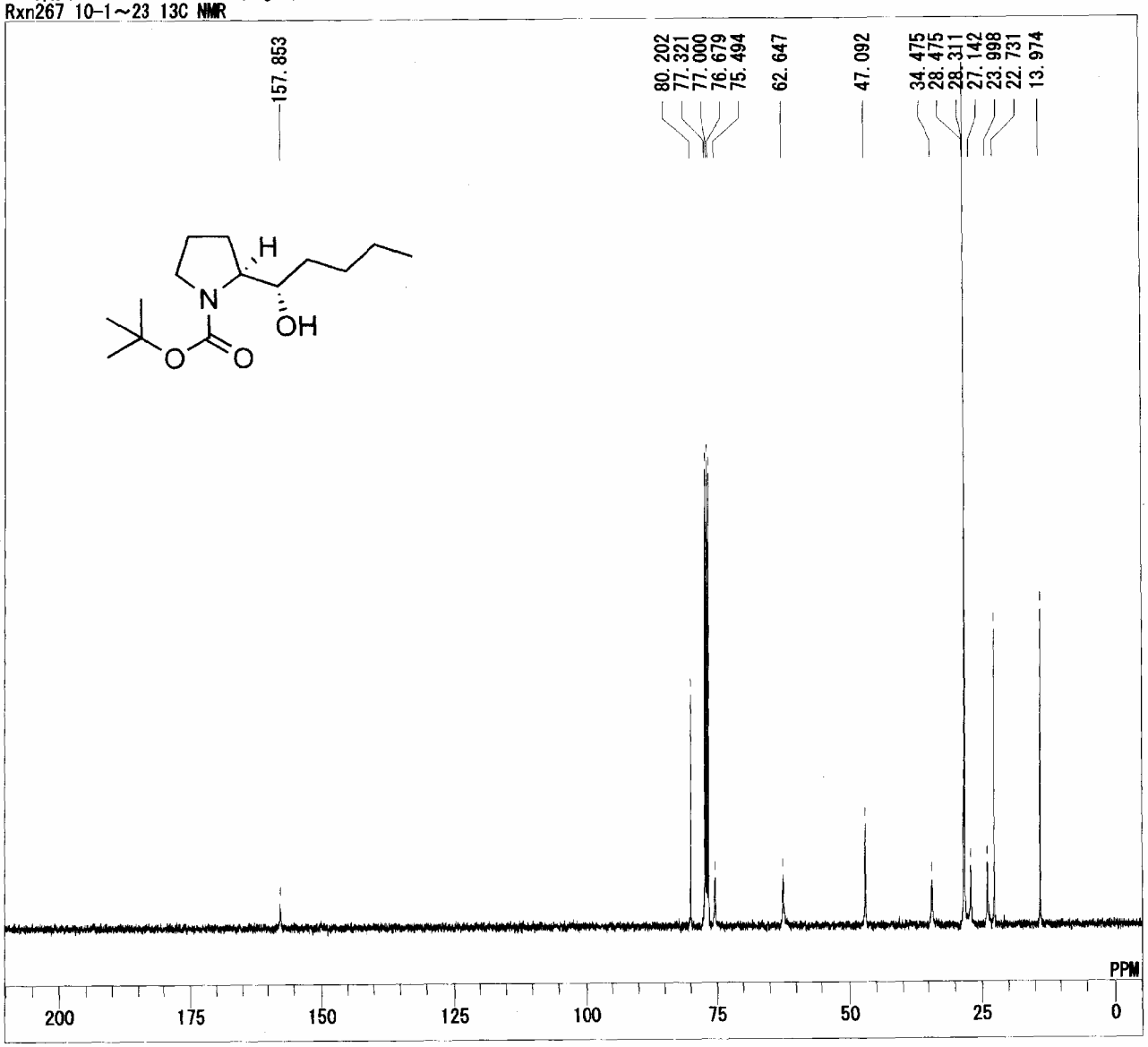
D:¥柴田¥(aS)-Boc-CF3-prolinol 13C-FID.als
(aS)-Boc-CF3-prolinol 13C NMR



DATIM	Wed Feb 21 16:39:51 2007
HELEV	79.60 %
N2LEV	86.00 %
SLVNT	CDCL3
CTEMP	20.6 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
PW2	10.00 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1500
SCANS	1247
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1:2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥(aS)-Boc-CF3-prolinol 13C-FID.
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	25
LKPHS	335
LKSIG	400
CSPED	11 Hz
FILDC	
FILDF	

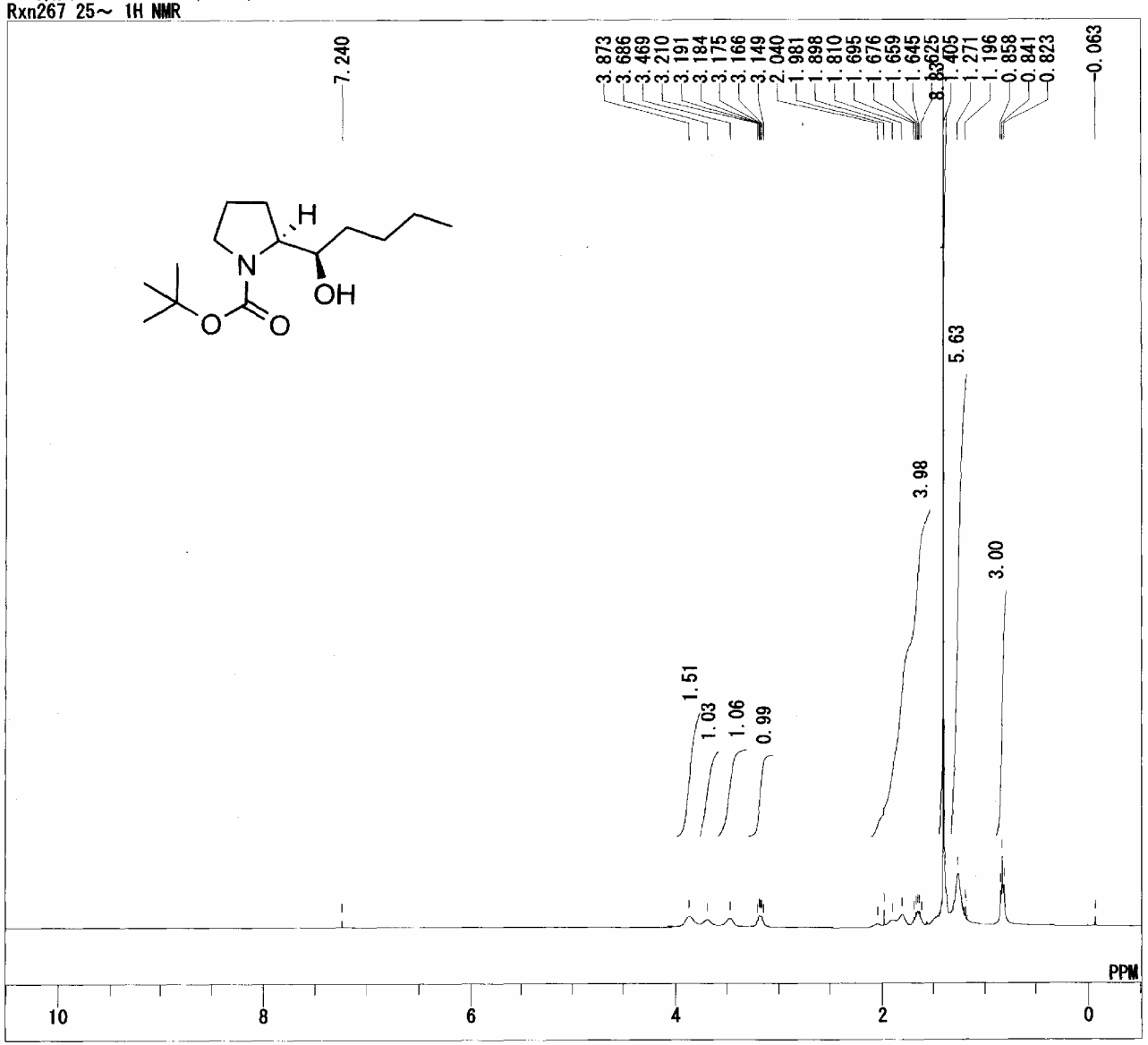

D:¥柴田¥Rxn267 10-1~23 (major) 1H.als
Rxn267 10-1~23 1H NMR
7.240
4.779
3.769
3.758
3.748
3.737
3.728
3.717
3.403
3.248
3.233
3.229
3.220
3.215
3.206
3.202
3.188
1.975
1.927
1.907
1.896
1.888
1.876
1.868
1.857
1.838
1.825
1.807
1.790
1.776
1.759
1.740
1.722
1.710
1.695
1.678
1.663
1.557
1.399
1.301
1.282
1.269
1.254
1.236
1.218
1.208
1.190
1.172
0.854
0.836
0.817
0.070
0.76
1.00
2.07
0.95
4.83
3.86
2.70
3H
PPM
DATIM Thu Nov 02 11:22:08 2006
HELEV 84.40 %
N2LEV 92.00 %
SLVNT CDCL3
CTEMP 19.2 c
MENUF 1H
OBNUC 1H
OFR 395.75 MHz
OBSET 124.00 KHz
OBFIN 10277.00 Hz
PW1 6.60 usec
DEADT 72.60 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 32
DUMMY 1
FREQU 7912.96 Hz
FLT
DELAY 50.60 usec
ACQTM 4.1411 sec
PD 2.8590 sec
ADBIT 16
RGAIN 7
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC 1H
IFR 395.75 MHz
IRSET 124.00 KHz
IRFIN 10277.00 Hz
IRRPW 50 usec
IRATN 511
DFILE D:¥柴田¥Rxn267 10-1~23 (major) 1H.al
SF TH5ATF62
LKSET 12.90 KHz
LKFIN 104.0 Hz
LKLEV 180
LGAIN 25
LKPHS 326
LKSIG 511
GSPED 12 Hz
FILDC
FILDF

D:¥柴田¥Rxn267 10-1～23 (major) 13C-FID.als
Rxn267 10-1～23 13C NMR



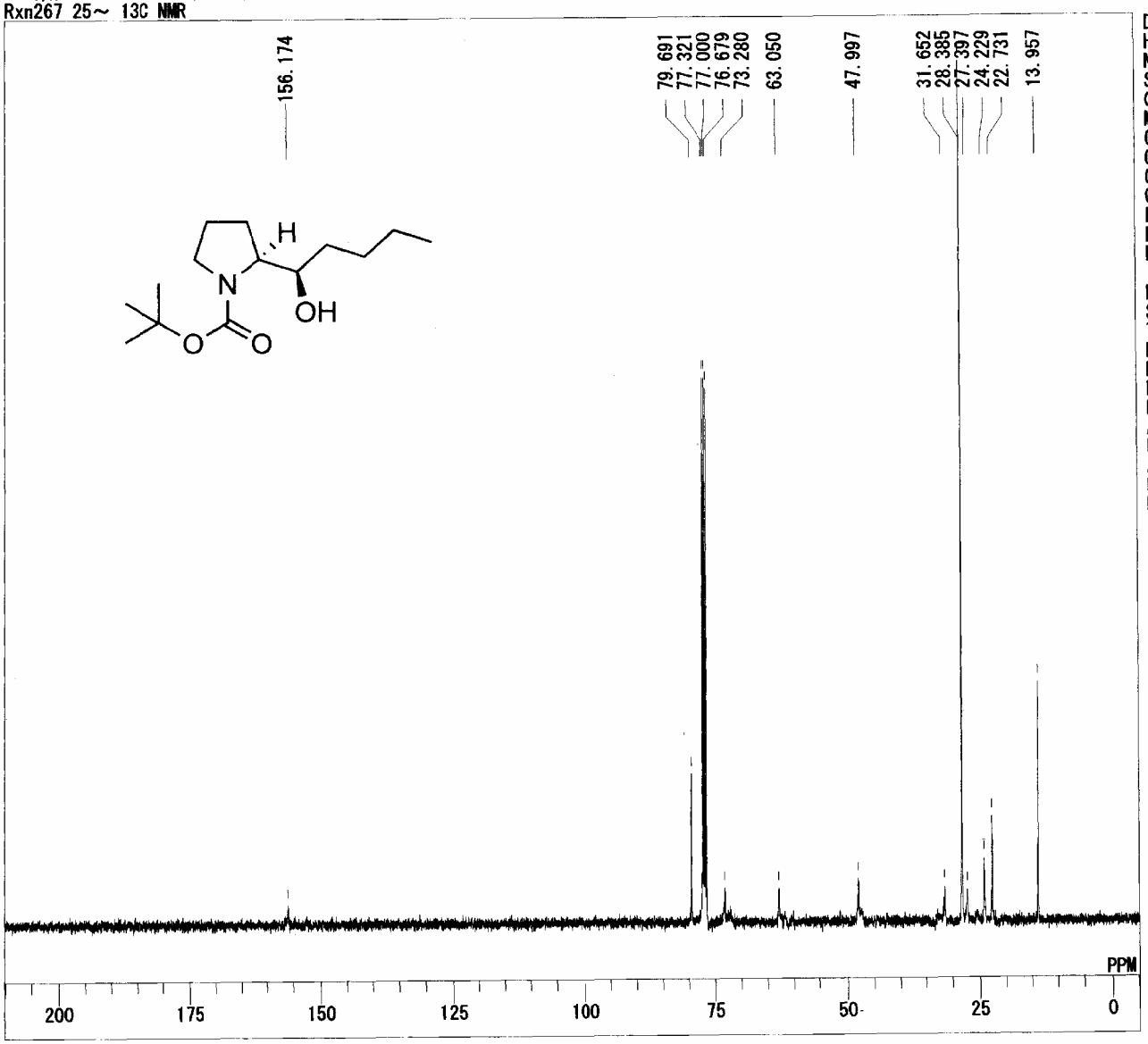
DATIM	Thu Nov 02 11:44:45 2006
HELEV	84.40 %
N2LEV	92.40 %
SLVNT	CDCL3
CTEMP	20.2 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1000
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	24
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn267 10-1～23 (major) 13C-F
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	25
LKPHS	326
LKSIG	508
CSPED	12 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn267 25～(minor) 1H-FID.als
Rxn267 25～ 1H NMR



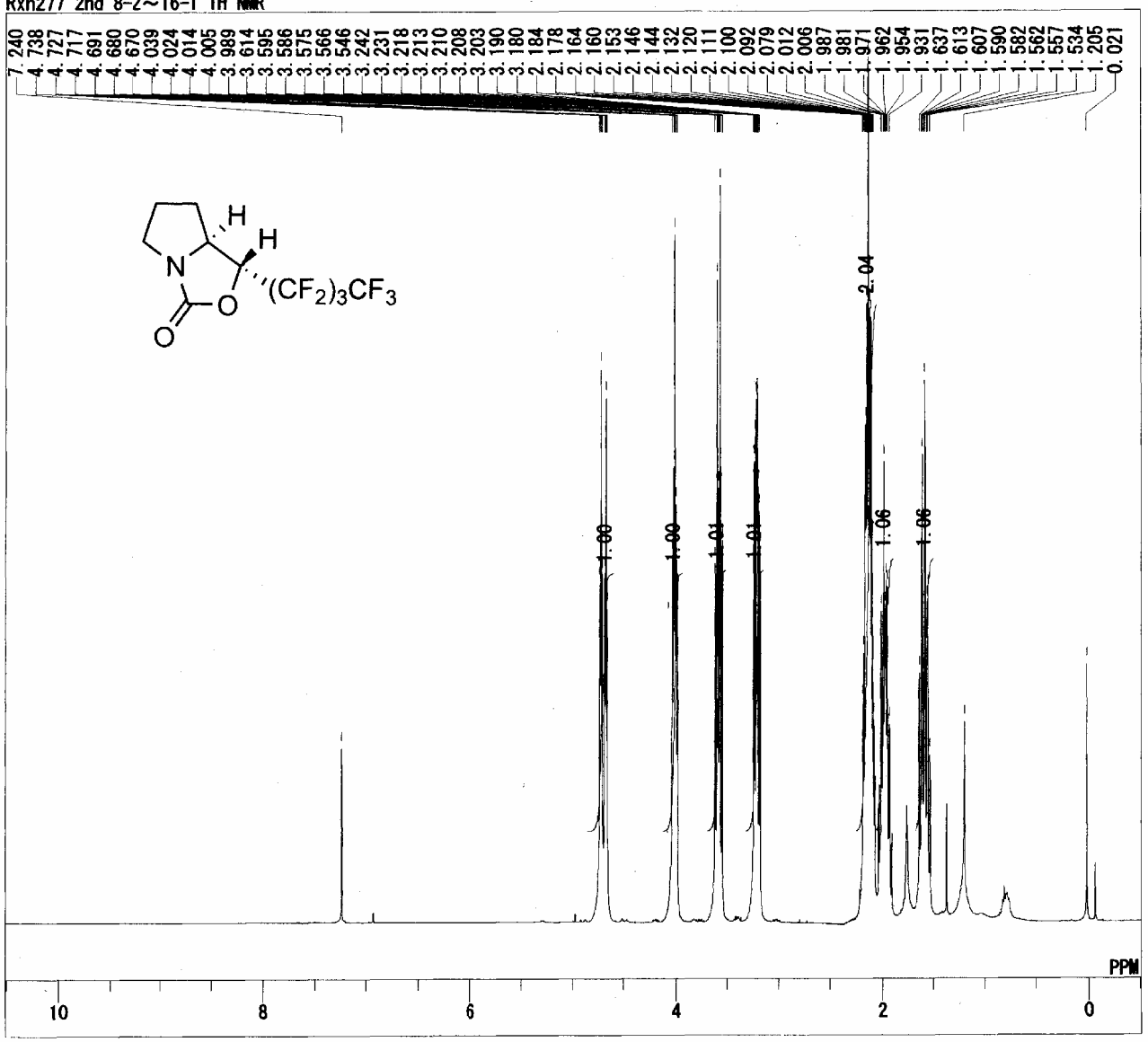
DATIM	Thu Nov 02 11:53:31 2006
HELEV	84.40 %
N2LEV	92.00 %
SLVNT	CDCL3
CTEMP	18.4 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	32
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	8
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn267 25～(minor) 1H-FID.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	1000
CSPED	12 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn267 25～(minor) 13C.als
Rxn267 25～ 13C NMR



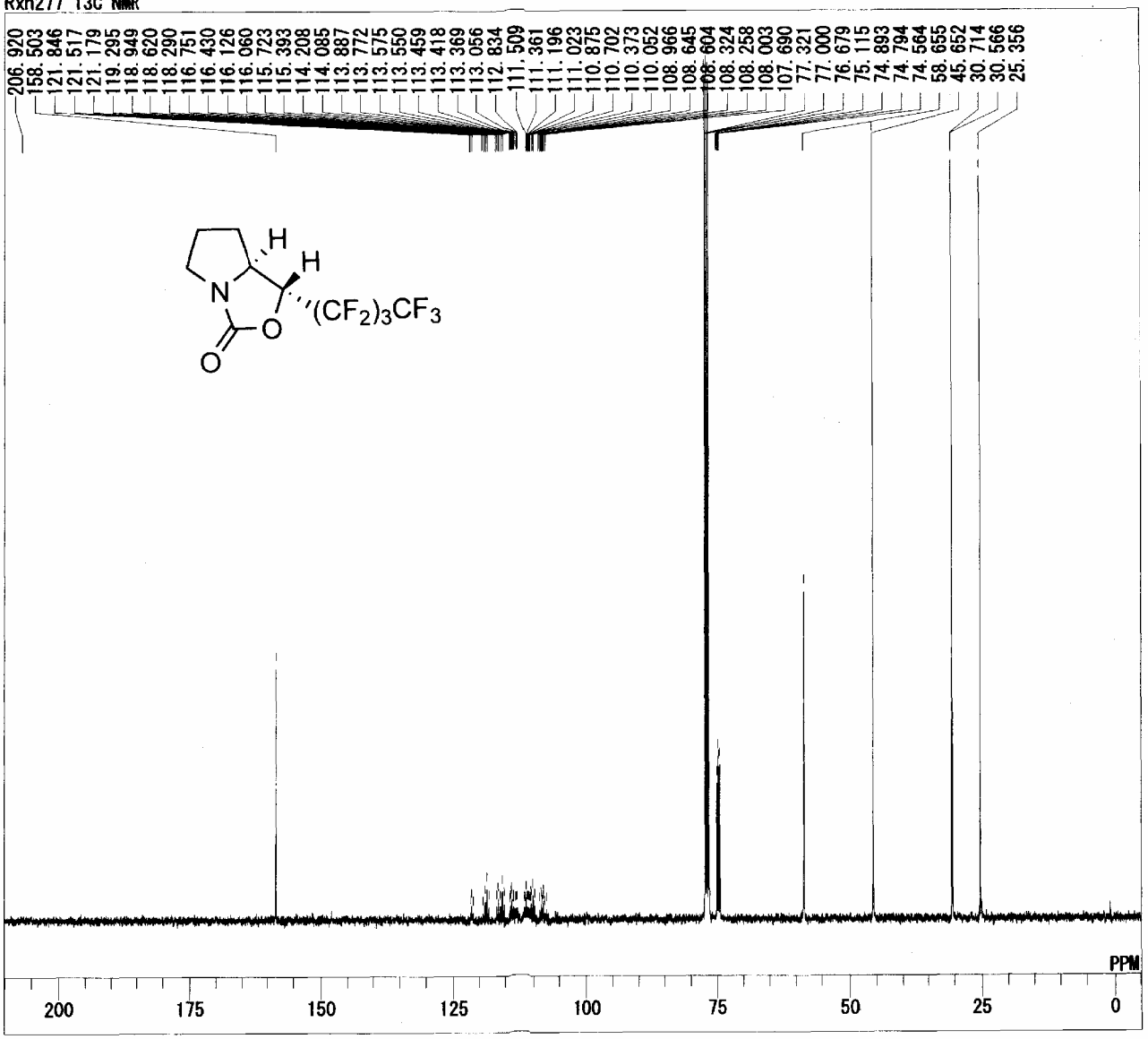
DATIM	Thu Nov 02 12:08:48 2006
HELEV	84.40 %
N2LEV	92.00 %
SLVNT	CDCL3
CTEMP	19.8 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1000
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn267 25～(minor) 13C.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	984
CSPED	12 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn277 2nd 8-2～16-1 1H.als
Rxn277 2nd 8-2～16-1 1H NMR



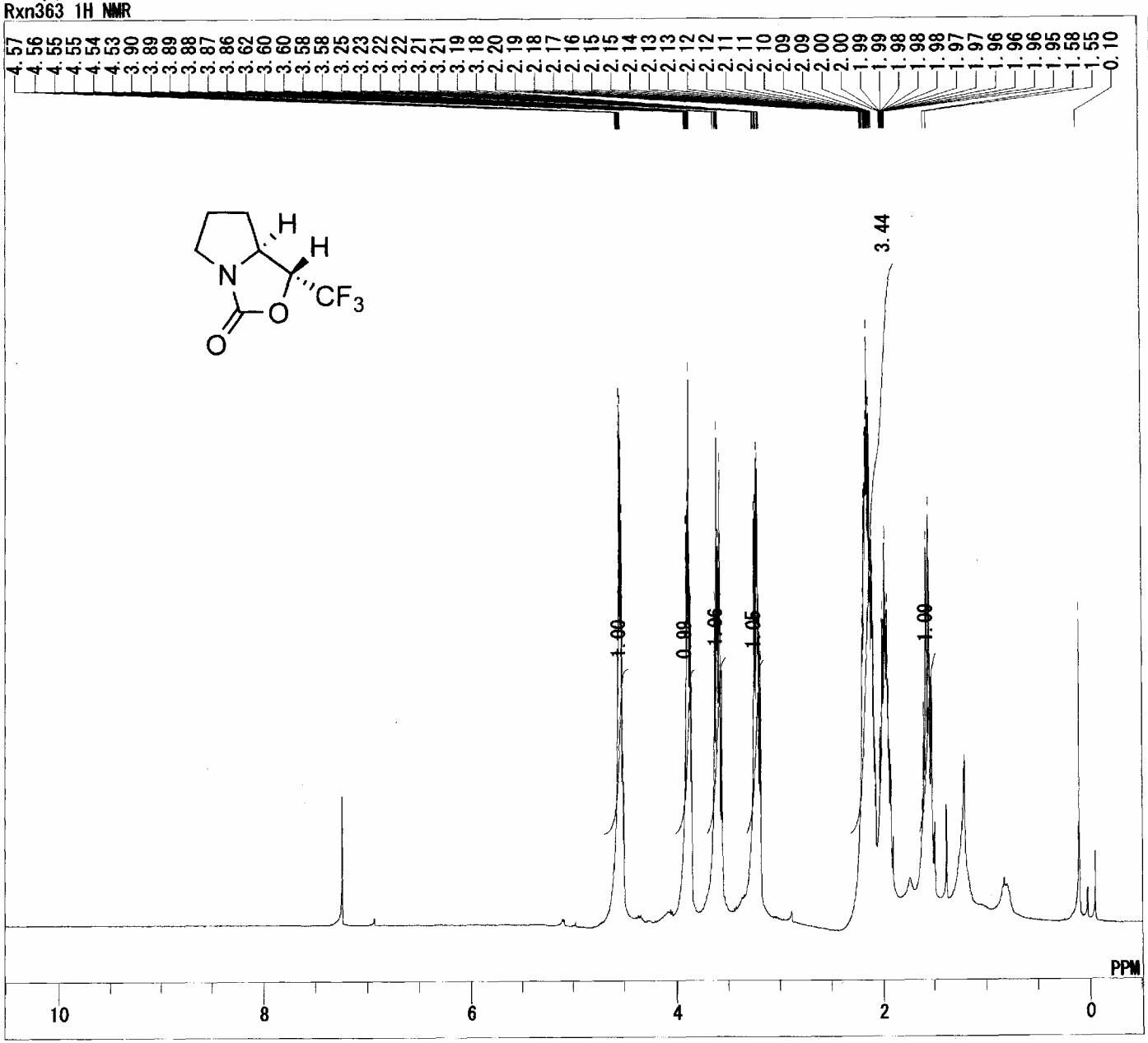
DATIM	Mon Oct 16 21:18:53 2006
HELEV	88.80 %
N2LEV	95.60 %
SLVNT	CDCL3
CTEMP	21.5 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	32
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	10
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn277 2nd 8-2～16-1 1H.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	24
LKPHS	326
LKSIG	493
CSPED	12 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn277 13C-FID.als
Rxn277 13C NMR



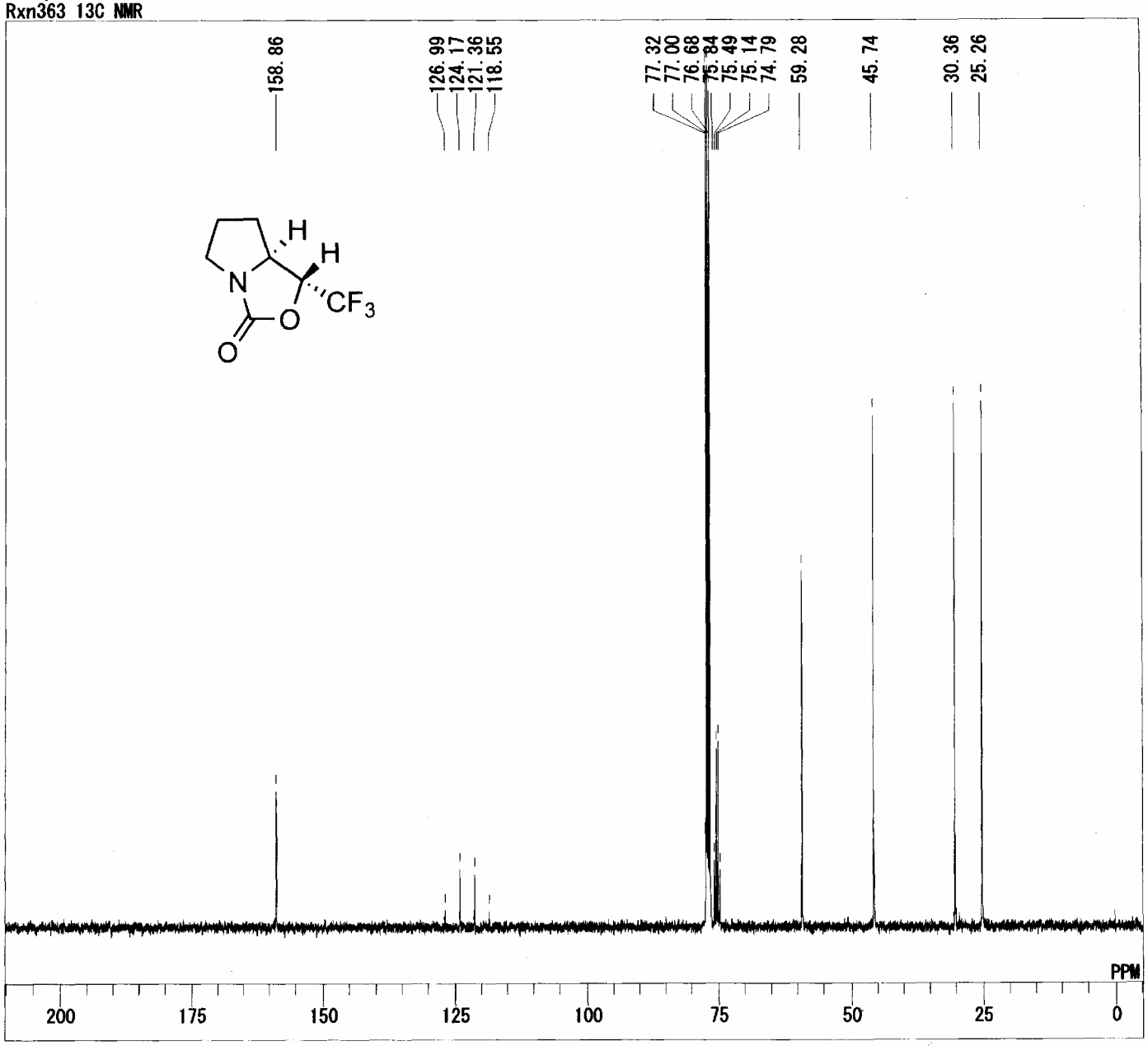
DATIM	Tue Nov 14 15:45:05 2006
HELEV	81.60 %
N2LEV	90.80 %
SLVNT	CDCL3
CTEMP	17.2 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	2000
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn277 13C-FID.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	1028
CSPED	14 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn363 1H-FID.als
Rxn363 1H NMR



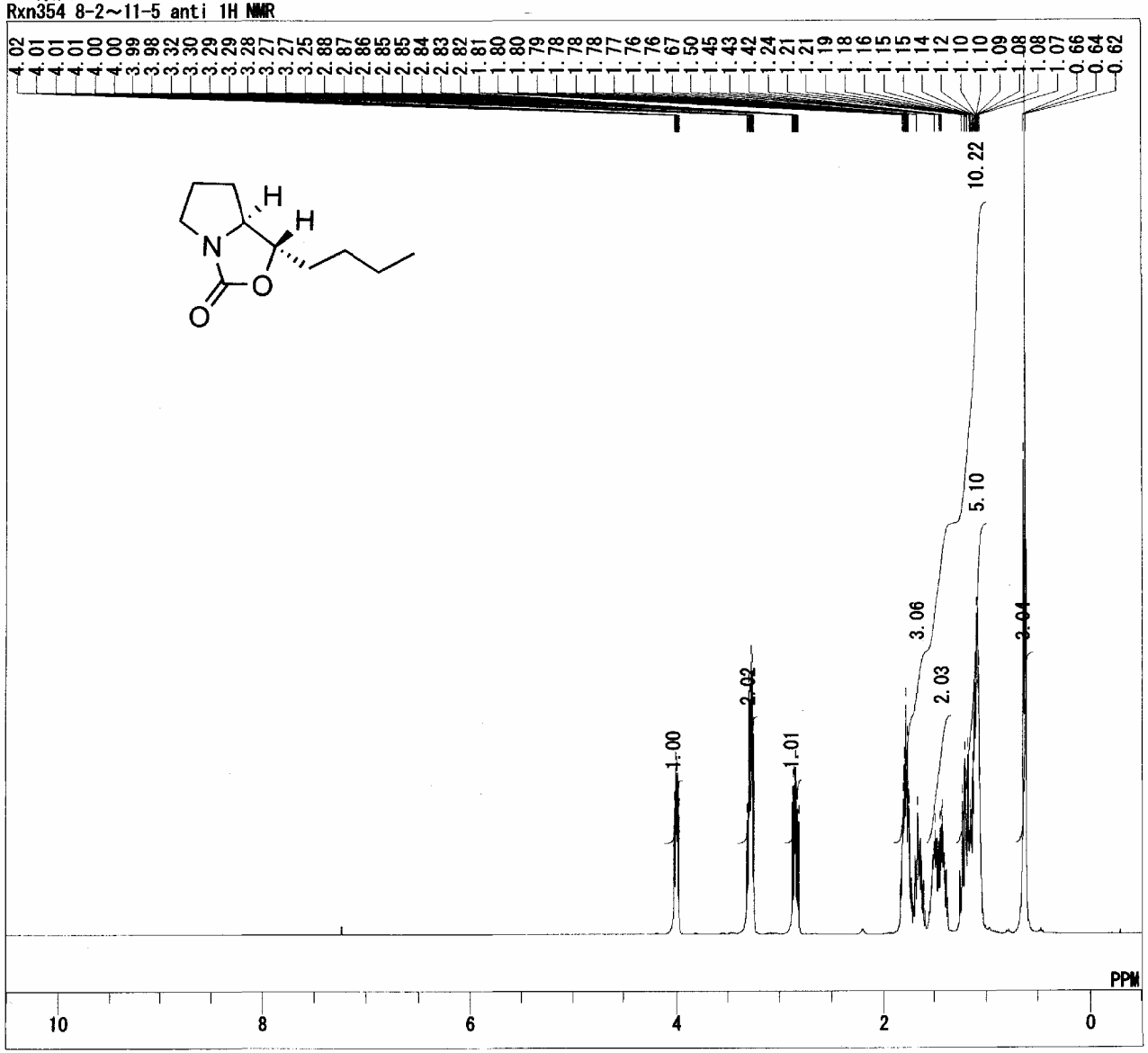
DATIM	Fri Mar 02 14:25:50 2007
HELEV	76.00 %
N2LEV	86.00 %
SLVNT	CDCL3
CTEMP	19.7 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
PW2	10.00 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	32
SCANS	32
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	12
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn363 1H-FID.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	335
CSPED	11 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn363 13C.als
Rxn363 13C NMR



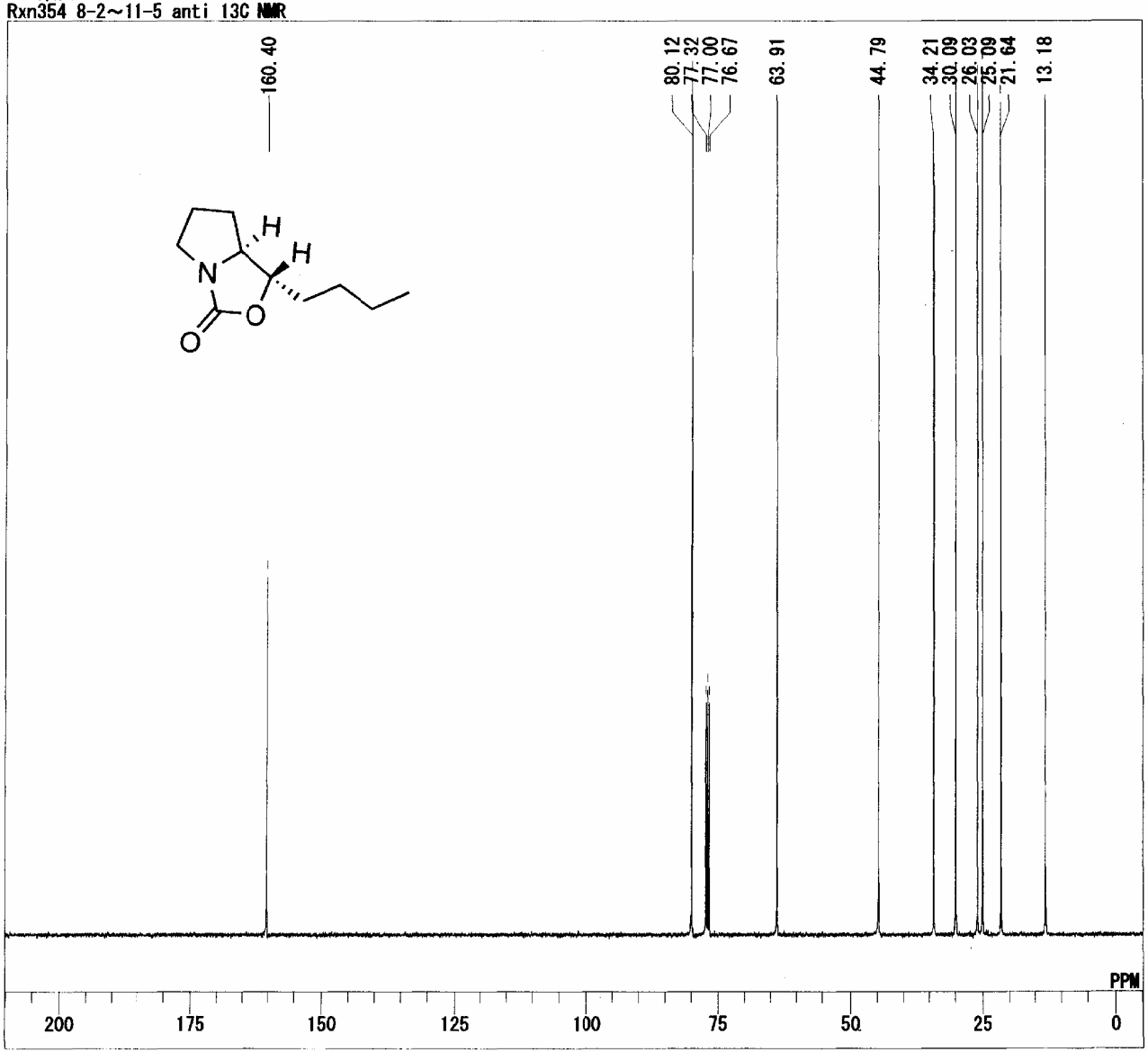
DATIM	Fri Mar 02 15:43:46 2007
HELEV	76.00 %
N2LEV	86.00 %
SLVNT	CDCL3
CTEMP	20.6 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
PW2	10.00 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	2000
SCANS	1423
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Se
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn363 13C.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	26
LKPHS	326
LKSIG	386
CSPED	10 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn354 8-2～11-5 anti 1H.als
Rxn354 8-2～11-5 anti 1H NMR



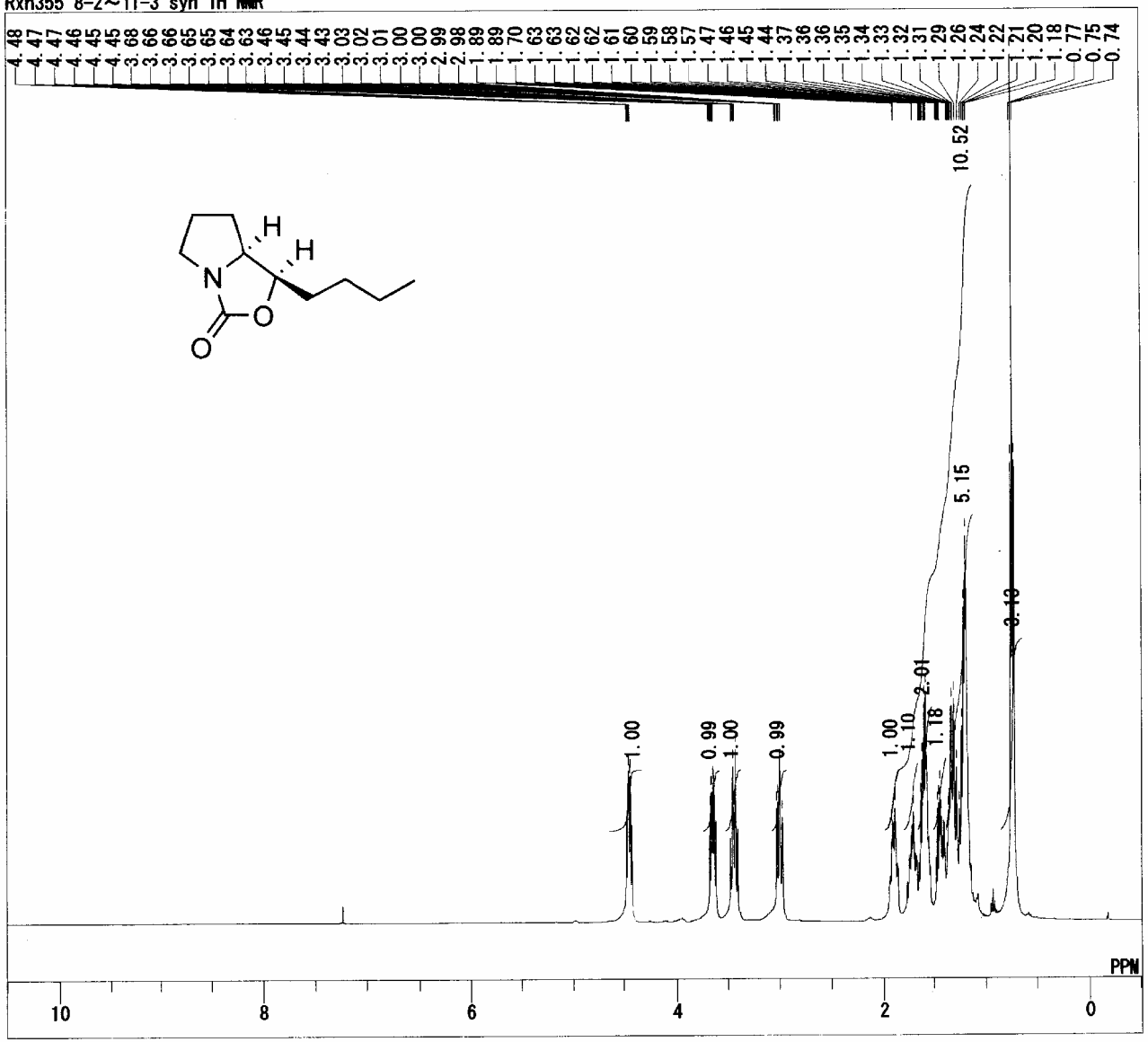
DATIM	Sun Feb 11 18:21:31 2007
HELEV	82.00 %
N2LEV	85.60 %
SLVNT	CDCL3
CTEMP	19.1 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
PW2	10.00 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	32
SCANS	32
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	3
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn354 8-2～11-5 anti 1H.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	450
CSPED	13 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn354 8-2～11-5 anti 13C.als
Rxn354 8-2～11-5 anti 13C NMR



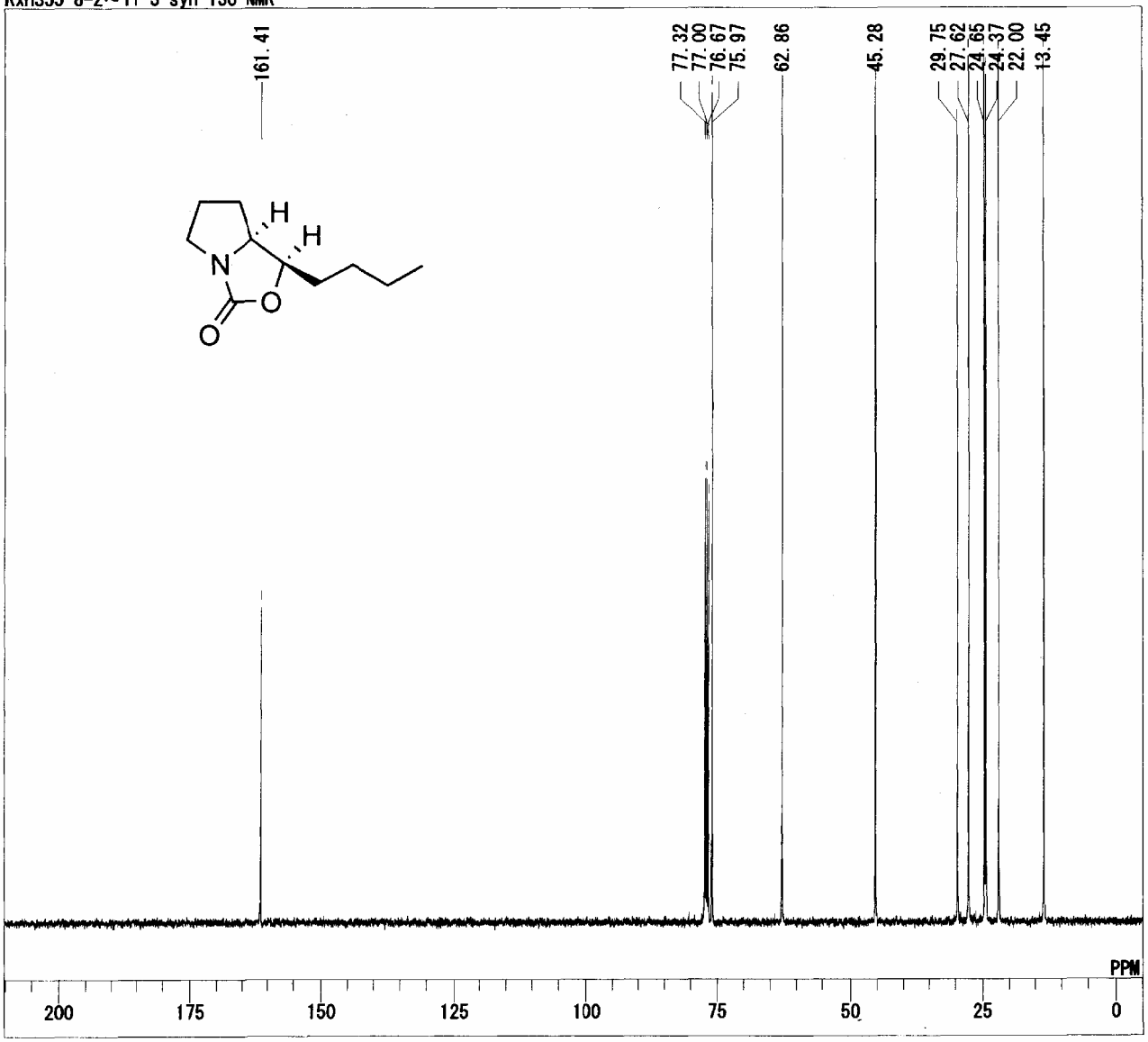
DATIM	Sun Feb 11 18:39:13 2007
HELEV	82.00 %
N2LEV	85.60 %
SLVNT	CDCL3
CTEMP	20.5 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
PW2	10.00 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1500
SCANS	343
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	24
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn354 8-2～11-5 anti 13C.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	27
LKPHS	326
LKSIG	478
CSPED	13 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn355 8-2～11-3 syn 1H.als
Rxn355 8-2～11-3 syn 1H NMR



DATIM	Sun Feb 11 18:49:04 2007
HELEV	82.00 %
N2LEV	85.60 %
SLVNT	CDCL3
CTEMP	19.3 c
MENUF	1H
OBNUC	1H
OFR	395.75 MHz
OBSET	124.00 KHz
OBFIN	10277.00 Hz
PW1	6.60 usec
PW2	10.00 usec
DEADT	72.60 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	32
SCANS	32
DUMMY	1
FREQU	7912.96 Hz
FLT	
DELAY	50.60 usec
ACQTM	4.1411 sec
PD	2.8590 sec
ADBIT	16
RGAIN	5
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:1H,13
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn355 8-2～11-3 syn 1H.als
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	25
LKPHS	326
LKSIG	379
CSPED	12 Hz
FILDC	
FILDF	

D:¥柴田¥Rxn355 8-2～11-3 syn 13C-FID.als
Rxn355 8-2～11-3 syn 13C NMR



DATIM	Sun Feb 11 19:08:37 2007
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N2LEV	85.20 %
SLVNT	CDCL3
CTEMP	20.0 c
MENUF	BCM
OBNUC	13C
OFR	99.45 MHz
OBSET	94.00 KHz
OBFIN	10309.00 Hz
PW1	4.60 usec
PW2	10.00 usec
DEADT	20.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1500
SCANS	381
DUMMY	1
FREQU	26845.64 Hz
FLT	
DELAY	14.90 usec
ACQTM	1.2206 sec
PD	1.7790 sec
ADBIT	16
RGAIN	25
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRPW
IRNUC	1H
IFR	395.75 MHz
IRSET	124.00 KHz
IRFIN	10277.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	D:¥柴田¥Rxn355 8-2～11-3 syn 13C-FID.a
SF	TH5ATFG2
LKSET	12.90 KHz
LKFIN	104.0 Hz
LKLEV	180
LGAIN	25
LKPHS	326
LKSIG	396
CSPED	11 Hz
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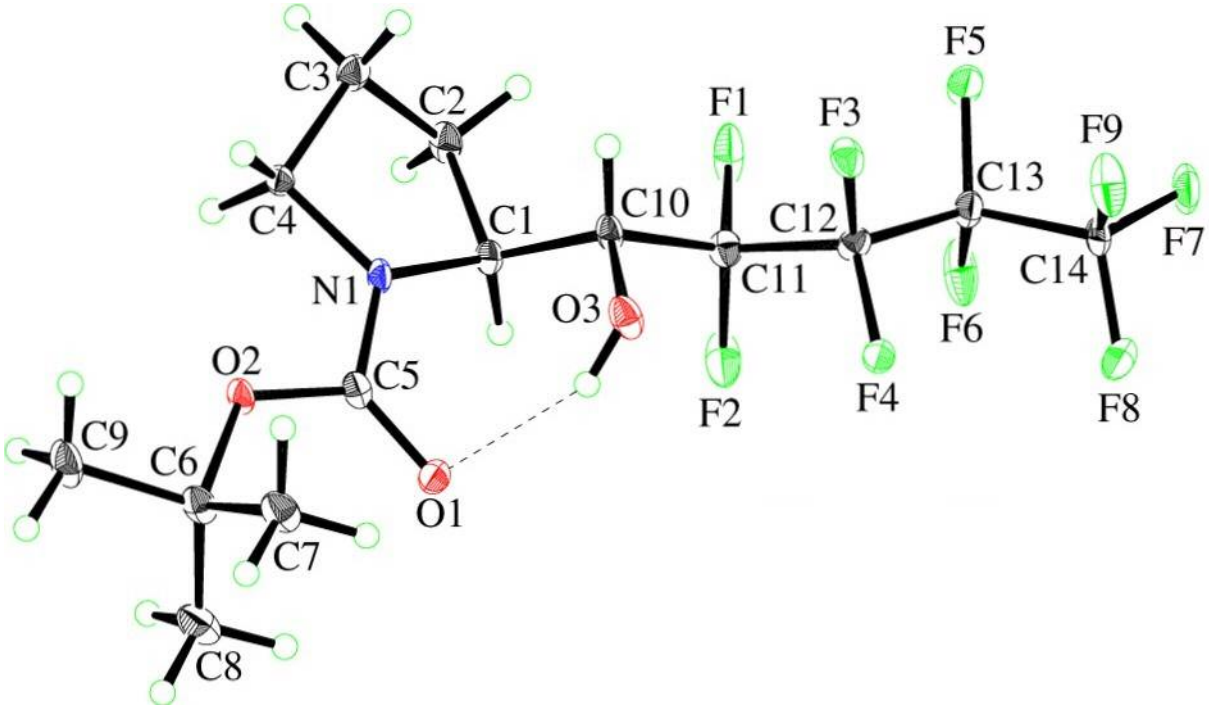
FILDF	



The crystal data of (α*R*)-**5a**. Empirical formula: $C_{14}H_{18}F_9NO_3$ Formula weight: 419.28; Crystal description: Platelet, Crystal color: Colorless; Crystal size: 0.10 x 0.10 x 0.05 mm; Crystal system: orthorhombic; Space group: P 21 21 2; Unit cell dimensions: a = 8.811(5) Å, b = 12.403(7) Å, c = 16.004(9) Å, α= 90°, β= 90°, γ = 90°, Residuals: R = 0.0965, Rw = 0.1549.

Table 1. Crystal data and structure refinement for funabiki5a.

Identification code	funabiki5a	
Empirical formula	C14 H18 F9 N O3	
Formula weight	419.29	
Temperature	123(2) K	
Wavelength	0.71070 Å	
Crystal system	orthorhombic	
Space group	P 21 21 2	
Unit cell dimensions	a = 8.811(5) Å	α= 90°.
	b = 12.403(7) Å	β= 90°.
	c = 16.004(9) Å	γ = 90°.
Volume	1749.0(17) $Å^3$	
Z	4	
Density (calculated)	1.592 Mg/m^3	
Absorption coefficient	0.172 mm^{-1}	
F(000)	856	
Crystal size	0.10 x 0.10 x 0.05 mm^3	
Theta range for data collection	3.03 to 27.47°.	
Index ranges	-11<=h<=9, -15<=k<=16, -20<=l<=15	
Reflections collected	14312	
Independent reflections	2287 [R(int) = 0.0808]	
Completeness to theta = 27.47°	99.8 %	
Max. and min. transmission	0.9914 and 0.9830	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2287 / 0 / 248	
Goodness-of-fit on F^2	1.377	
Final R indices [I>2sigma(I)]	R1 = 0.0965, wR2 = 0.1549	
R indices (all data)	R1 = 0.1052, wR2 = 0.1575	
Largest diff. peak and hole	0.291 and -0.327 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for funabiki5a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	5414(6)	6909(4)	5096(3)	18(1)
C(1)	5921(7)	7868(5)	4611(4)	18(1)
C(2)	6055(8)	7396(5)	3718(4)	25(2)
C(3)	4942(9)	6453(5)	3714(4)	25(2)
C(4)	5174(8)	5943(5)	4565(4)	19(1)
C(5)	5690(8)	6856(5)	5925(3)	19(1)
O(1)	6069(6)	7626(3)	6357(3)	23(1)
O(2)	5487(6)	5844(3)	6206(2)	23(1)
C(6)	5489(8)	5590(5)	7119(4)	22(1)
C(7)	4196(9)	6199(6)	7534(4)	31(2)
C(8)	7008(9)	5817(7)	7502(4)	35(2)
C(9)	5148(10)	4391(6)	7100(4)	34(2)
C(10)	4793(8)	8795(5)	4684(4)	19(1)
O(3)	4265(6)	8977(4)	5507(3)	27(1)
C(11)	5475(8)	9853(5)	4381(4)	21(1)
F(1)	5743(6)	9809(3)	3541(2)	41(1)
F(2)	6826(5)	10042(4)	4753(3)	49(1)
C(12)	4517(8)	10881(5)	4523(4)	20(1)
F(3)	3028(4)	10622(3)	4406(2)	26(1)
F(4)	4644(5)	11208(3)	5325(2)	29(1)
C(13)	4876(8)	11853(5)	3964(4)	22(1)
F(5)	4321(6)	11658(3)	3198(2)	38(1)
F(6)	6392(5)	11963(3)	3910(3)	43(1)
C(14)	4229(10)	12945(5)	4239(4)	28(2)
F(7)	4480(6)	13667(3)	3647(3)	38(1)
F(8)	4869(7)	13291(3)	4934(3)	52(2)
F(9)	2751(6)	12865(4)	4361(3)	47(1)

Table 3. Bond lengths [Å] and angles [°] for funabiki5a.

N(1)-C(5)	1.350(7)
N(1)-C(4)	1.485(7)
N(1)-C(1)	1.489(7)
C(1)-C(10)	1.524(9)
C(1)-C(2)	1.549(8)
C(1)-H(1)	1.0000
C(2)-C(3)	1.526(9)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.515(8)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(1)	1.226(7)
C(5)-O(2)	1.345(7)
O(2)-C(6)	1.494(7)
C(6)-C(8)	1.498(10)
C(6)-C(9)	1.518(9)
C(6)-C(7)	1.520(9)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-O(3)	1.415(7)

C(10)-C(11)	1.524(9)
C(10)-H(10)	1.0000
O(3)-H(3)	0.8400
C(11)-F(2)	1.350(8)
C(11)-F(1)	1.366(7)
C(11)-C(12)	1.546(9)
C(12)-F(4)	1.351(7)
C(12)-F(3)	1.364(8)
C(12)-C(13)	1.534(8)
C(13)-F(5)	1.342(7)
C(13)-F(6)	1.346(8)
C(13)-C(14)	1.534(9)
C(14)-F(8)	1.319(8)
C(14)-F(9)	1.320(9)
C(14)-F(7)	1.323(7)

C(5)-N(1)-C(4)	123.3(5)
C(5)-N(1)-C(1)	119.8(5)
C(4)-N(1)-C(1)	112.9(5)
N(1)-C(1)-C(10)	111.5(5)
N(1)-C(1)-C(2)	101.7(5)
C(10)-C(1)-C(2)	113.9(5)
N(1)-C(1)-H(1)	109.8
C(10)-C(1)-H(1)	109.8
C(2)-C(1)-H(1)	109.8
C(3)-C(2)-C(1)	104.1(5)
C(3)-C(2)-H(2A)	110.9
C(1)-C(2)-H(2A)	110.9
C(3)-C(2)-H(2B)	110.9
C(1)-C(2)-H(2B)	110.9
H(2A)-C(2)-H(2B)	109.0
C(4)-C(3)-C(2)	103.3(6)

C(4)-C(3)-H(3A)	111.1
C(2)-C(3)-H(3A)	111.1
C(4)-C(3)-H(3B)	111.1
C(2)-C(3)-H(3B)	111.1
H(3A)-C(3)-H(3B)	109.1
N(1)-C(4)-C(3)	101.3(5)
N(1)-C(4)-H(4A)	111.5
C(3)-C(4)-H(4A)	111.5
N(1)-C(4)-H(4B)	111.5
C(3)-C(4)-H(4B)	111.5
H(4A)-C(4)-H(4B)	109.3
O(1)-C(5)-O(2)	125.1(5)
O(1)-C(5)-N(1)	124.4(6)
O(2)-C(5)-N(1)	110.5(5)
C(5)-O(2)-C(6)	121.5(5)
O(2)-C(6)-C(8)	111.2(5)
O(2)-C(6)-C(9)	100.7(5)
C(8)-C(6)-C(9)	111.7(6)
O(2)-C(6)-C(7)	108.7(5)
C(8)-C(6)-C(7)	113.4(6)
C(9)-C(6)-C(7)	110.3(6)
C(6)-C(7)-H(7A)	109.5
C(6)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(6)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(6)-C(8)-H(8A)	109.5
C(6)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(6)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5

H(8B)-C(8)-H(8C)	109.5
C(6)-C(9)-H(9A)	109.5
C(6)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(6)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
O(3)-C(10)-C(11)	106.8(5)
O(3)-C(10)-C(1)	114.0(5)
C(11)-C(10)-C(1)	111.6(5)
O(3)-C(10)-H(10)	108.1
C(11)-C(10)-H(10)	108.1
C(1)-C(10)-H(10)	108.1
C(10)-O(3)-H(3)	109.5
F(2)-C(11)-F(1)	106.7(6)
F(2)-C(11)-C(10)	110.9(5)
F(1)-C(11)-C(10)	110.3(5)
F(2)-C(11)-C(12)	105.9(5)
F(1)-C(11)-C(12)	105.8(5)
C(10)-C(11)-C(12)	116.6(5)
F(4)-C(12)-F(3)	106.3(5)
F(4)-C(12)-C(13)	107.5(5)
F(3)-C(12)-C(13)	107.7(5)
F(4)-C(12)-C(11)	110.0(5)
F(3)-C(12)-C(11)	108.1(5)
C(13)-C(12)-C(11)	116.7(5)
F(5)-C(13)-F(6)	108.7(5)
F(5)-C(13)-C(14)	106.6(5)
F(6)-C(13)-C(14)	107.3(6)
F(5)-C(13)-C(12)	108.5(5)
F(6)-C(13)-C(12)	108.8(5)
C(14)-C(13)-C(12)	116.7(5)

F(8)-C(14)-F(9)	108.8(7)
F(8)-C(14)-F(7)	108.1(5)
F(9)-C(14)-F(7)	108.8(6)
F(8)-C(14)-C(13)	111.8(6)
F(9)-C(14)-C(13)	110.0(6)
F(7)-C(14)-C(13)	109.3(6)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for funabiki5a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	26(3)	9(2)	20(2)	3(2)	1(2)	0(2)
C(1)	19(3)	14(3)	21(3)	2(2)	3(3)	0(3)
C(2)	35(4)	20(3)	20(3)	4(3)	7(3)	0(3)
C(3)	43(4)	16(3)	17(3)	-4(2)	5(3)	3(3)
C(4)	29(4)	16(3)	13(3)	-1(2)	-3(3)	2(3)
C(5)	20(3)	20(3)	15(3)	3(2)	1(3)	8(3)
O(1)	37(3)	17(2)	16(2)	-2(2)	-2(2)	0(2)
O(2)	42(3)	15(2)	11(2)	4(2)	6(2)	6(2)
C(6)	30(4)	24(3)	11(3)	6(2)	8(3)	4(3)
C(7)	39(4)	37(4)	15(3)	2(3)	6(3)	15(4)
C(8)	39(5)	44(5)	21(3)	10(3)	-2(3)	10(4)
C(9)	53(5)	26(4)	23(3)	11(3)	7(3)	10(4)
C(10)	24(3)	15(3)	16(3)	4(2)	0(3)	2(3)
O(3)	33(3)	26(2)	21(2)	6(2)	9(2)	9(2)
C(11)	21(4)	19(3)	24(3)	1(3)	-1(3)	-1(3)
F(1)	69(3)	17(2)	36(2)	7(2)	27(2)	12(2)
F(2)	19(2)	22(2)	105(4)	11(3)	-18(2)	-3(2)
C(12)	25(4)	17(3)	19(3)	-1(2)	2(3)	-2(3)
F(3)	19(2)	22(2)	38(2)	6(2)	-4(2)	3(2)
F(4)	55(3)	17(2)	16(2)	0(1)	-7(2)	1(2)
C(13)	28(4)	16(3)	21(3)	1(3)	2(3)	1(3)
F(5)	75(3)	22(2)	16(2)	1(2)	-2(2)	-1(2)
F(6)	26(2)	24(2)	80(3)	22(2)	15(2)	-1(2)
C(14)	42(5)	8(3)	34(4)	6(3)	-8(3)	-4(3)
F(7)	62(3)	13(2)	38(2)	6(2)	1(2)	4(2)
F(8)	96(4)	19(2)	39(2)	-3(2)	-23(3)	-4(3)
F(9)	46(3)	28(3)	68(3)	9(2)	14(3)	11(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for funabiki5a.

	x	y	z	U(eq)
H(1)	6943	8107	4811	22
H(2A)	5771	7940	3293	30
H(2B)	7102	7145	3605	30
H(3A)	3884	6709	3646	30
H(3B)	5182	5938	3261	30
H(4A)	4268	5531	4744	23
H(4B)	6072	5463	4570	23
H(7A)	4405	6975	7516	46
H(7B)	4101	5966	8117	46
H(7C)	3246	6049	7237	46
H(8A)	7797	5438	7186	52
H(8B)	7013	5567	8083	52
H(8C)	7205	6595	7487	52
H(9A)	4153	4271	6842	51
H(9B)	5142	4107	7671	51
H(9C)	5930	4019	6773	51
H(10)	3898	8624	4324	22
H(3)	4923	8769	5851	40

Table 6. Torsion angles [°] for funabiki5a.

C(5)-N(1)-C(1)-C(10)	-82.7(7)
C(4)-N(1)-C(1)-C(10)	119.2(6)
C(5)-N(1)-C(1)-C(2)	155.6(6)
C(4)-N(1)-C(1)-C(2)	-2.6(7)
N(1)-C(1)-C(2)-C(3)	26.2(6)
C(10)-C(1)-C(2)-C(3)	-93.9(6)
C(1)-C(2)-C(3)-C(4)	-40.6(7)
C(5)-N(1)-C(4)-C(3)	-179.2(6)
C(1)-N(1)-C(4)-C(3)	-21.9(7)
C(2)-C(3)-C(4)-N(1)	37.5(7)
C(4)-N(1)-C(5)-O(1)	170.9(6)
C(1)-N(1)-C(5)-O(1)	15.1(10)
C(4)-N(1)-C(5)-O(2)	-9.3(9)
C(1)-N(1)-C(5)-O(2)	-165.1(5)
O(1)-C(5)-O(2)-C(6)	9.1(10)
N(1)-C(5)-O(2)-C(6)	-170.7(5)
C(5)-O(2)-C(6)-C(8)	-64.1(8)
C(5)-O(2)-C(6)-C(9)	177.4(6)
C(5)-O(2)-C(6)-C(7)	61.4(8)
N(1)-C(1)-C(10)-O(3)	44.6(7)
C(2)-C(1)-C(10)-O(3)	158.9(5)
N(1)-C(1)-C(10)-C(11)	165.6(5)
C(2)-C(1)-C(10)-C(11)	-80.0(7)
O(3)-C(10)-C(11)-F(2)	73.8(7)
C(1)-C(10)-C(11)-F(2)	-51.4(7)
O(3)-C(10)-C(11)-F(1)	-168.2(5)
C(1)-C(10)-C(11)-F(1)	66.6(7)
O(3)-C(10)-C(11)-C(12)	-47.5(7)
C(1)-C(10)-C(11)-C(12)	-172.7(5)
F(2)-C(11)-C(12)-F(4)	-45.2(7)

F(1)-C(11)-C(12)-F(4)	-158.2(5)
C(10)-C(11)-C(12)-F(4)	78.7(7)
F(2)-C(11)-C(12)-F(3)	-160.9(5)
F(1)-C(11)-C(12)-F(3)	86.0(6)
C(10)-C(11)-C(12)-F(3)	-37.0(7)
F(2)-C(11)-C(12)-C(13)	77.6(7)
F(1)-C(11)-C(12)-C(13)	-35.4(7)
C(10)-C(11)-C(12)-C(13)	-158.5(6)
F(4)-C(12)-C(13)-F(5)	-161.4(5)
F(3)-C(12)-C(13)-F(5)	-47.2(7)
C(11)-C(12)-C(13)-F(5)	74.5(7)
F(4)-C(12)-C(13)-F(6)	80.5(7)
F(3)-C(12)-C(13)-F(6)	-165.3(5)
C(11)-C(12)-C(13)-F(6)	-43.5(7)
F(4)-C(12)-C(13)-C(14)	-41.0(8)
F(3)-C(12)-C(13)-C(14)	73.2(7)
C(11)-C(12)-C(13)-C(14)	-165.1(6)
F(5)-C(13)-C(14)-F(8)	-170.9(6)
F(6)-C(13)-C(14)-F(8)	-54.6(7)
C(12)-C(13)-C(14)-F(8)	67.8(8)
F(5)-C(13)-C(14)-F(9)	68.1(7)
F(6)-C(13)-C(14)-F(9)	-175.6(5)
C(12)-C(13)-C(14)-F(9)	-53.2(8)
F(5)-C(13)-C(14)-F(7)	-51.3(7)
F(6)-C(13)-C(14)-F(7)	65.1(7)
C(12)-C(13)-C(14)-F(7)	-172.6(6)

Symmetry transformations used to generate equivalent atoms: