Asymmetric Synthesis of Functionalized Pyrrolizidines by Organocatalytic and Pot-economy Strategy.

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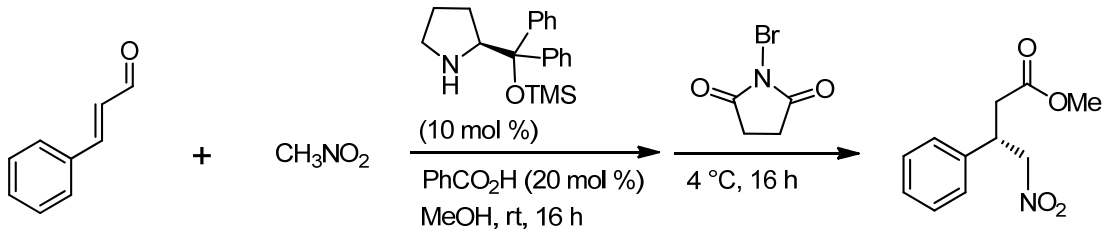
SUPPORTING INFORMATION:

Contents:

General Procedure. All solvents were reagent grade. Chemicals were purchased from Aldrich, TCI or Acros Chemical Co. Reactions were normally carried out under nitrogen atmosphere in glassware. Merck silica gel 60 (particle size 0.04-0.063 mm) was employed for flash chromatography. Melting points are uncorrected. ^{1}H NMR spectra were obtained in $CDCl_3$ unless otherwise noted at 400 MHz (Bruker DPX-400**)** or 500 MHz (Varian-Unity INOVA-500). ^{13}C NMR spectra were obtained at 100 MHz or 125 MHz. *E.e.* values were measured by HPLC on a chiral column (0.46 cm ID x 25 cm, particle size 5 μ) by elution with THF-hexane. Unless otherwise noted, the flow rate of the indicated elution solvent is maintained at 1 mL/min, and the retention time of a compound is recorded accordingly. HPLC was equipped with the ultraviolet and refractive index detectors. The melting point was recorded on a melting point apparatus (MPA100 – Automated melting point system, Stanford Research Systems, Inc.) and is uncorrected. The optical rotation values were recorded with a Jasco-P-2000 digital polarimeter.

Preparation of (*S*)-methyl 4-nitro-3-phenylbutanoate (2a)



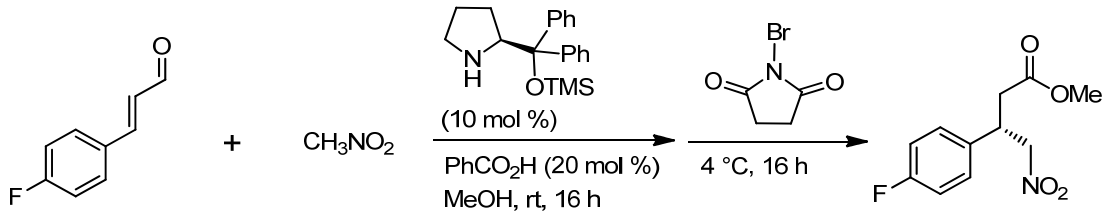
1a **2a**

To a solution of cinnamaldehyde (158.6 mg, 1.2 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (39.1 mg, 0.12 mmol, 0.1 equiv) and benzoic acid (29.3 mg, 0.24 mmol, 0.2 equiv) in methanol (2.4 mL) was added nitromethane (219.7 mg, 3.6 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (320.4 mg, 1.8 mmol, 1.5 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 15:10:75 EtOAc–CH_2Cl_2–hexane (R_f = 0.31) to afford product **2a** (155.4 mg, 58% yield) as a pale yellow oil. Selected spectroscopic data of **2a**: $[\alpha]_D^{23}$ –19.6 (*c* 0.5, CH_2Cl_2);[1] IR (neat): 3022, 2953, 1736, 1554, 1440, 1371, 1162, 1081, 998, 891, 768, 704 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.19 (m, 5H), 4.72 (dd, J = 12.6, 6.9 Hz, 1H), 4.62 (dd, J = 12.6, 7.9 Hz, 1H), 4.00 – 3.93 (m, 1H), 3.61 (s, 3H), 2.81 – 2.73 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1 (C), 138.3 (C), 129.01 (2 CH), 128.0 (CH), 127.3 (2 CH), 79.4 (CH_2), 51.9 (CH_3), 40.1 (CH), 37.5 (CH_2);[2] MS (*m/z*, relative intensity): 223 (M^+, 0.4), 192 (18), 177 (32), 176 (90), 145 (43), 135 (54), 118 (86), 117 (100), 104 (57), 91 (50), 77 (33); exact mass calculated for $C_{11}H_{13}O_4N$ (M^+): 223.0845; found: 223.0844.

[1] Lit. $[\alpha]_D^{20}$ –18.8 (*c* 0.5, CH_2Cl_2), Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

[2] (a) Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519. (b) Baschieri, A.; Bernardi, L.; Ricci, A.; Suresh, S.; Adamo, M. F. A. *Angew. Chem. Int. Ed.* **2009**, *48*, 9342 – 9345.

Prearation of (*S*)-methyl 3-(4-fluorophenyl)-4-nitrobutanoate (2b)



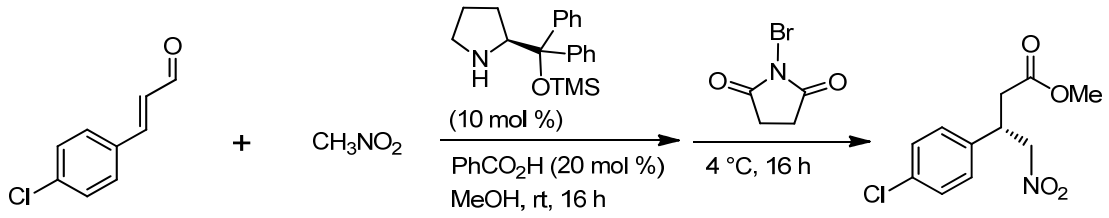
1b **2b**

To a solution of (*E*)-3-(4-fluorophenyl)acrylaldehyde **1b** (150.1 mg, 1.0 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (32.6 mg, 0.1 mmol, 0.1 equiv) and benzoic acid (24.4 mg, 0.2 mmol, 0.2 equiv) in methanol (2 mL) was added nitromethane (183.1 mg, 3.0 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (267 mg, 1.5 mmol, 1.5 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 5:10:85 EtOAc–CH_2Cl_2–hexane (R_f = 0.14) to afford product **2b** (149.6 mg, 62% yield) as a yellow oil. Selected spectroscopic data of **2b**: $[\alpha]_D^{26}$ –19.2 (*c* 0.4, CH_2Cl_2);[3] IR (neat): 2956, 2924, 1735, 1605, 1554, 1511, 1436, 1377, 1225, 1161, 1103, 836 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.21 – 7.16 (m, 2 H), 7.04 – 6.98 (m, 2 H), 4.70 (dd, *J* = 12.6, 6.8 Hz, 1 H), 4.59 (dd, *J* = 12.6, 8.1 Hz, 1 H), 3.99 – 3.92 (m, 1 H), 3.62 (s, 3 H), 2.79 – 2.67 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9 (C), 162.3 (d, *J* = 245 Hz, C), 134.0 (d, *J* = 4 Hz, C), 129.0 (d, *J* = 9 Hz, 2 CH), 116.1 (d, *J* = 22 Hz, 2 CH), 79.3 (CH_2), 52.0 (CH_3), 39.5 (CH), 37.5 (CH_2);[4] MS (*m/z*, relative intensity): 241 (M^+, 0.3), 195 (14), 194 (89), 163 (18), 153 (40), 136 (90), 135 (100), 122 (70), 109 (34); exact mass calculated for $C_{11}H_{12}O_4NF$ (M^+): 241.0750; found: 241.0750.

[3] Lit. $[\alpha]_D^{20}$ –19.4 (*c* 0.5, CH_2Cl_2), Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

[4] Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

Preparation of (*S*)-methyl 3-(4-chlorophenyl)-4-nitrobutanoate (2c)



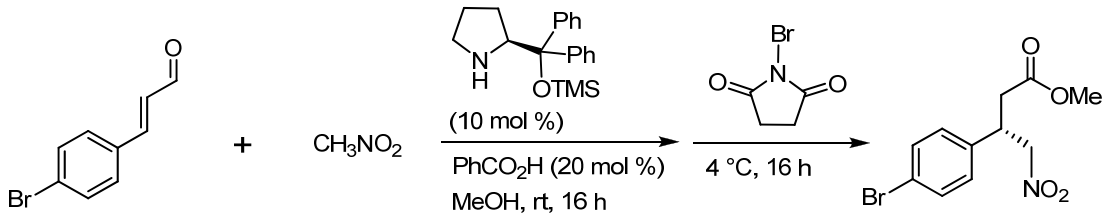
1c **2c**

To a solution of (*E*)-3-(4-chlorophenyl)acrylaldehyde **1c** (166.6 mg, 1.0 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (32.6 mg, 0.1 mmol, 0.1 equiv) and benzoic acid (24.4 mg, 0.2 mmol, 0.2 equiv) in methanol (2 mL) was added nitromethane (183.1 mg, 3.0 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (534 mg, 3.0 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 5:10:85 EtOAc–CH_2Cl_2–hexane (R_f = 0.17) to afford product **2c** (170.1 mg, 66% yield) as a yellow oil. Selected spectroscopic data of **2c**: $[\alpha]_D^{27}$ –13.2 (*c* 0.4, CH_2Cl_2);[5] IR (neat): 2954, 2922, 2849, 1736, 1552, 1493, 1436, 1378, 1262, 1166, 1095, 1014, 830, 794, 732 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.30 (d, J = 8.5 Hz, 2 H), 7.15 (d, J = 8.5 Hz, 2 H), 4.70 (dd, J = 12.8, 6.9 Hz, 1 H), 4.59 (dd, J = 12.8, 8.2 Hz, 1 H), 3.99 – 3.91 (m, 1 H), 3.62 (s, 3 H), 2.79 – 2.67 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8 (C), 136.7 (C), 134.0 (C), 129.3 (2 CH), 128.7 (2 CH), 79.1 (CH_2), 52.1 (CH_3), 39.5 (CH), 37.4 (CH_2);[6] MS (*m/z*, relative intensity): 259 (M^++2, 0.3), 257 (M^+, 0.8), 226 (8), 212 (30), 211 (15), 210 (100), 179 (18), 169 (43), 152 (96), 151 (64), 138 (54), 115 (45); exact mass calculated for $C_{11}H_{12}O_4NCl$ (M^+): 257.0455; found: 257.0457.

[5] Lit. $[\alpha]_D^{20}$ –13.2 (*c* 0.5, CH_2Cl_2), Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

[6] (a) Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519. (b) Vesely, J.; Zhao, G.-L.; Bartoszewicz, A.; Cordova, A. *Tetrahedron Lett.* **2008**, 49, 4209 – 4212. (c) Palomo, C.; Landa, A.; Mielgo, A.; Oiarbide, M.; Puente, A.; Vera, S. Angew. Chem. Int. Ed. **2007**, 46, 8431 – 8435.

Preparation of (*S*)-methyl 3-(4-bromophenyl)-4-nitrobutanoate (2d)



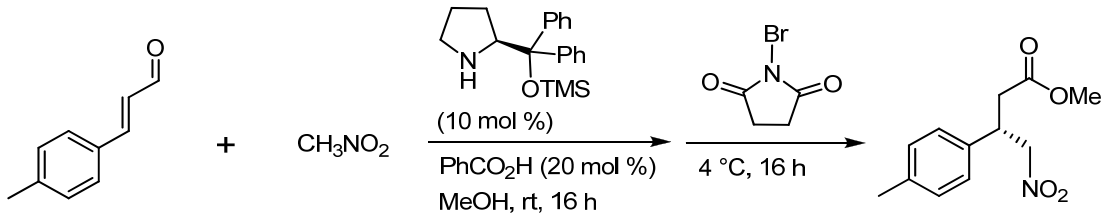
1d **2d**

To a solution of (*E*)-3-(4-bromophenyl)acrylaldehyde **1d** (253.3 mg, 1.2 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (39.1 mg, 0.12 mmol, 0.1 equiv) and benzoic acid (29.3 mg, 0.24 mmol, 0.2 equiv) in methanol (2.4 mL) was added nitromethane (219.7 mg, 3.6 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (640.7 mg, 3.6 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 7:14:79 EtOAc–CH_2Cl_2–hexane (R_f = 0.26) to afford the product **2d** (217.5 mg, 60% yield) as a pale yellow oil. Selected spectroscopic data of **2d**: $[\alpha]_D^{26}$ –12.0 (*c* 0.4, CH_2Cl_2);[7] IR (neat): 2953, 2922, 1733, 1552, 1490, 1436, 1375, 1265, 1200, 1168, 1109, 1075, 1010, 824, 727 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, J = 8.5 Hz, 2H), 7.09 (d, J = 8.5 Hz, 2H), 4.70 (dd, J = 12.7, 6.8 Hz, 1H), 4.59 (dd, J = 12.7, 8.0 Hz, 1H), 3.98 – 3.90 (m, 1H), 3.62 (s, 3H), 2.78 – 2.67 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8 (C), 137.2 (C), 132.3 (2 CH), 129.0 (2 CH), 122.1 (C), 79.0 (CH_2), 52.1 (CH_3), 39.6 (CH), 37.3 (CH_2);[8] MS (*m/z*, relative intensity): 303 (M^++3, 1), 301 (M^++1, 1), 272 (5), 270 (5), 256 (94), 254 (100), 225 (16), 223 (13), 215 (27), 213 (29), 198 (63), 196 (70), 184 (28), 182 (29), 116 (79), 115 (36); exact mass calculated for $C_{11}H_{12}O_4NBr$ (M^+): 300.9950; found: 300.9951.

[7] Lit. $[\alpha]_D^{20}$ –12.2 (*c* 0.5, CH_2Cl_2), Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

[8] Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

Preparation of (*S*)-methyl 4-nitro-3-p-tolylbutanoate (2e)



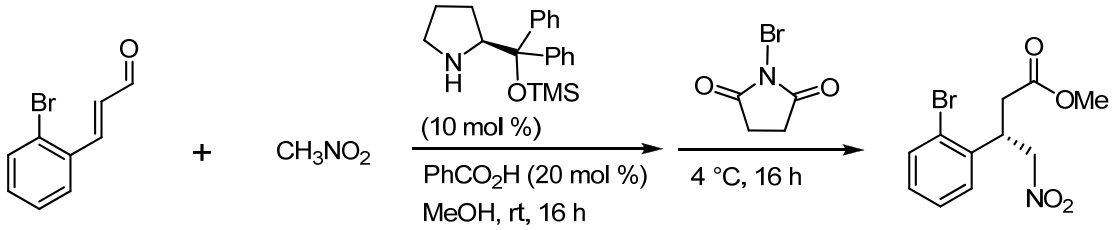
1e **2e**

To a solution of (*E*)-3-*p*-tolylacrylaldehyde (**1e**, 146.2 mg, 1.0 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (32.6 mg, 0.1 mmol, 0.1 equiv) and benzoic acid (24.4 mg, 0.2 mmol, 0.2 equiv) in methanol (2 mL) was added nitromethane (183.1 mg, 3.0 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (534.0 mg, 3.0 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 5:10:85 EtOAc–CH_2Cl_2–hexane (R_f = 0.20) to afford the product **2e** (125.7 mg, 53% yield) as a yellow oil. Selected spectroscopic data of **2e**: $[\alpha]_D^{26}$ –17.0 (c 0.4, CH_2Cl_2);[9] IR (neat): 2953, 2923, 1735, 1554, 1515, 1436, 1377, 1198, 1168, 1115, 996, 879, 817, 719 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.17 – 7.00 (m, 4 H), 4.69 (dd, J = 12.5, 7.5 Hz, 1 H), 4.59 (dd, J = 12.5, 7.5 Hz, 1 H), 3.96 – 3.89 (m, 1 H), 3.61 (s, 3 H), 2.79 – 2.67 (m, 2 H), 2.30 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1 (C), 137.8 (C), 135.2 (C), 129.8 (2 CH), 127.1 (2 CH), 79.5 (CH_2), 51.9 (CH_3), 39.8 (CH), 37.6 (CH_2), 21.0 (CH_3);[10] MS (*m/z*, relative intensity): 237 (M^+, 1), 206 (7), 191 (13), 190 (100), 159 (15), 149 (24), 132 (82), 131 (75), 118 (32), 117 (29), 115 (19), 91 (16); exact mass calculated for $C_{12}H_{15}O_4N$ (M^+): 237.1001; found: 237.1001.

[9] Lit. $[\alpha]_D^{20}$ –17.3 (*c* 0.5, CH_2Cl_2), Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

[10] Jensen, K. L.; Poulsen, P. H.; Donslund, B. S.; Morana, F.; Jørgensen, K. A. *Org. Lett.* **2012**, *14*, 1516 – 1519.

Preparation of (*S*)-methyl 3-(2-bromophenyl)-4-nitrobutanoate (2f)

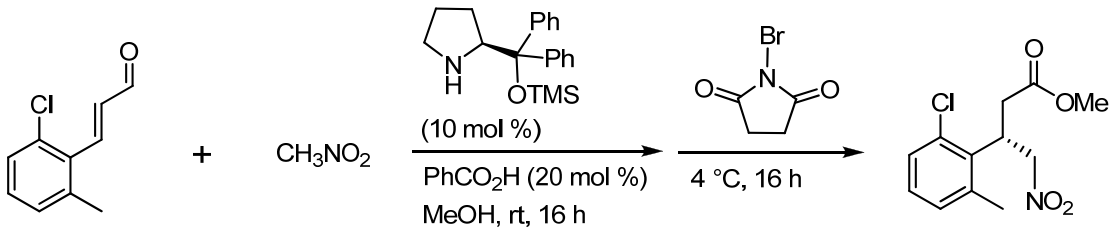


1f **2f**

To a solution of (*E*)-3-(2-bromophenyl)acrylaldehyde (**1f**, 211.1 mg, 1.0 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (32.6 mg, 0.1 mmol, 0.1 equiv) and benzoic acid (24.4 mg, 0.2 mmol, 0.2 equiv) in methanol (2 mL) was added nitromethane (183.1 mg, 3.0 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (534.0 mg, 3.0 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 7:14:79 EtOAc–CH_2Cl_2–hexane (R_f = 0.26) to afford the product **2f** (175.2 mg, 58% yield) as a pale yellow oil.[11] Selected spectroscopic data of **2f**: $[\alpha]_D^{26}$ –14.3 (*c* 0.4, CH_2Cl_2); IR (neat): 2953, 2915, 1736, 1554, 1473, 1436, 1377, 1200, 1173, 1022, 758, 731 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.59 (dd, J = 8.0, 1.3 Hz, 1 H), 7.30 – 7.26 (m, 1 H), 7.20 – 7.11 (m, 2 H), 4.80 – 4.71 (m, 2 H), 4.50 – 4.43 (m, 1 H), 3.63 (s, 3 H), 2.91 – 2.80 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9 (C), 137.1 (C), 133.8 (CH), 129.4 (CH), 128.0 (CH), 127.8 (CH), 124.5 (C), 77.5 (CH_2), 52.0 (CH_3), 38.9 (CH), 36.0 (CH_2); MS (*m/z*, relative intensity): 303 (M^++3, 0.1), 301 (M^++1, 0.1), 272 (2), 270 (2), 223 (20), 222 (100), 215 (11), 213 (12), 184 (12), 182 (12), 175 (53), 161 (91), 116 (90), 115 (68); exact mass calculated for $C_{11}H_{12}O_4NBr$ (M^+): 300.9950; found: 300.9947.

[11] The racemic **2f** was prepared from 3-bromobenzaldehyde by the Wittig reaction followed by the Michael addition of nitromethane and applied in the synthesis of novel piperidine inhibitors of farnesyltransferase. However, no details of physical characteristics and spectra data were reported, see: (a) Nara, S.; Tanaka, R.; Eishima, J.; Hara, M.; Takahashi, Y.; Otaki, S.; Foglesong, R. J.; Hughes, P. F.; Turkington, S.; Kanda, Y. *J. Med. Chem.* **2003**, *46*, 2467 – 2473. (b) Tanaka, R.; Rubio, A.; Harn, N. K.; Gernert, D.; Grese, T. A.; Eishima, J.; Hara, M.; Yoda, Nobuyuki; Ohashi, R.; Kuwabara, T.; Soga, S.; Akinaga, S.; Nara, S.; Kanda, Y. *Bioorg. Med. Chem.* **2007**, *15*, 1363 – 1382.

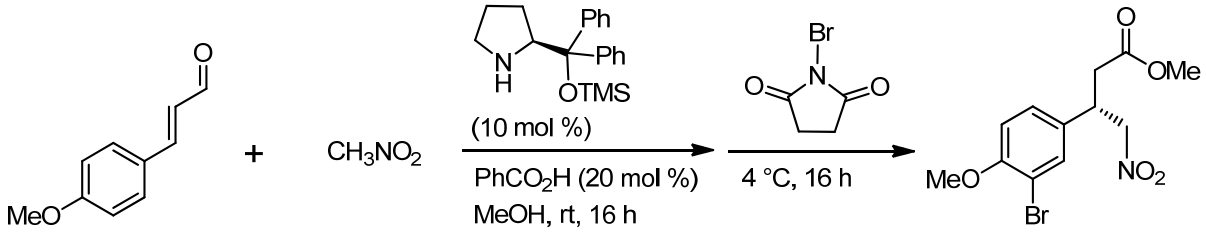
Preparation of (S)-methyl 3-(2-chloro-6-methylphenyl)-4-nitrobutanoate (2g)



1g **2g**

To a solution of (*E*)-3-(2-chloro-6-methylphenyl)acrylaldehyde (**1g**, 100.0 mg, 0.55 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (19.5 mg, 0.06 mmol, 0.1 equiv) and benzoic acid (14.7 mg, 0.12 mmol, 0.2 equiv) in methanol (2 mL) was added nitromethane (109.9 mg, 1.8 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (320.4 mg, 1.8 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 5:10:85 EtOAc–CH_2Cl_2–hexane (R_f = 0.37) to afford the product **2g** (89.9 mg, 60% yield) as a pale yellow oil.f Selected spectroscopic data of **2g**: $[\alpha]_D^{26}$ –15.8 (*c* 0.4, CH_2Cl_2); IR (neat): 2954, 2917, 1736, 1554, 1435, 1377, 1217, 1194, 1173, 1124, 1090, 1048, 1001, 845, 776 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.21 – 7.14 (m, 1 H), 7.13 – 7.05 (m, 2 H), 5.02 (dd, J = 13.1, 8.0 Hz, 1 H), 4.89 (dd, J = 13.1, 6.7 Hz, 1 H), 4.62 – 4.54 (m, 1 H), 3.62 (s, 3 H), 3.10 – 2.96 (m, 2 H), 2.50 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.6 (C), 140.3 (C), 134.0 (C), 133.4 (C), 129.9 (CH), 129.3 (CH), 128.8 (CH), 76.9 (CH_2), 52.0 (CH_3), 35.2 (CH), 35.1 (CH_2), 21.2 (CH_3); MS (*m/z*, relative intensity): 273 (M^++2, 0.26), 271 (M^+, 0.61), 240 (14), 236 (61), 209 (23), 189 (35), 175 (46), 167 (100), 165 (82), 129 (57), 115 (100); exact mass calculated for $C_{12}H_{14}O_4NCl$ (M^+): 271.0611; found: 271.0612.

Preparation of (*S*)-methyl 3-(4-methoxyphenyl)-4-nitrobutanoate (2h)



1h **2h**

To a solution of (*E*)-3-(4-methoxyphenyl)acrylaldehyde (**1h**, 97.3 mg, 0.6 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (19.5 mg, 0.06 mmol, 0.1 equiv) and benzoic acid (14.7 mg, 0.12 mmol, 0.2 equiv) in methanol (1.2 mL) was added nitromethane (109.9 mg, 1.8 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (320.4 mg, 1.8 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 7:14:79 EtOAc–CH_2Cl_2–hexane (R_f = 0.26) to afford the product **2h** (113.6 mg, 57% yield) as a pale yellow oil.[12] Selected spectroscopic data of **2h**: m.p. 100–101 °C; $[\alpha]_D^{26}$ –13.3 (c 0.4, CH_2Cl_2); IR (neat): 2951, 2841, 1732, 1552, 1498, 1436, 1375, 1286, 1259, 1168, 1054, 1017, 881, 814, 718 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.38 (d, J = 2.3 Hz, 1 H), 7.13 (dd, J = 8.5, 2.3 Hz, 1 H), 6.83 (d, J = 8.5 Hz, 1 H), 4.68 (dd, J = 12.6, 6.8 Hz, 1 H), 4.57 (dd, J = 12.6, 8.0 Hz, 1 H), 3.93 – 3.85 (m, 1 H), 3.85 (s, 3 H), 3.63 (s, 3 H), 2.75 – 2.70 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9 (C), 155.6 (C), 132.0 (CH), 131.7 (C), 127.6 (CH), 112.18 (CH), 112.17 (C), 79.3 (CH_2), 56.2 (CH_3), 52.0 (CH_3), 39.1 (CH), 37.5 (CH_2);[13] MS (*m/z*, relative intensity): 333 (M^++2, 9), 331 (M^+, 9), 286 (94), 284 (95), 228 (51), 226 (52), 214 (33), 212 (34), 147 (28), 146 (100); exact mass calculated for $C_{12}H_{14}BrNO_5$ (M^+): 331.0055; found:.331.0050.

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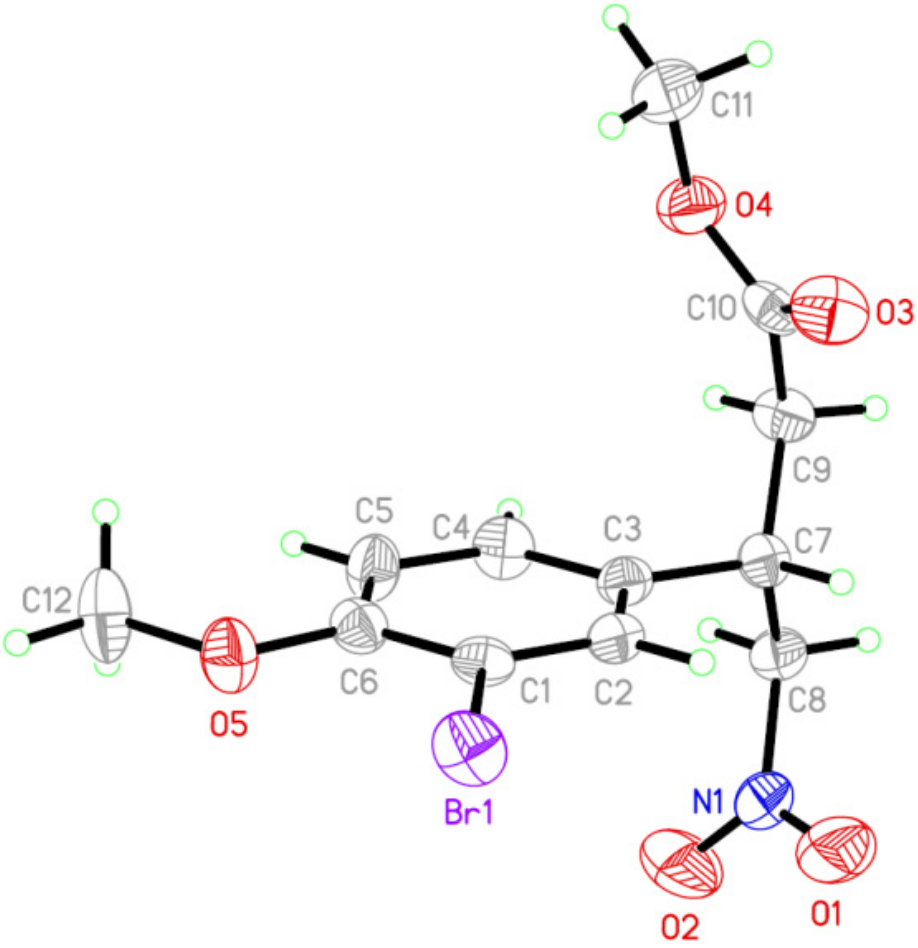
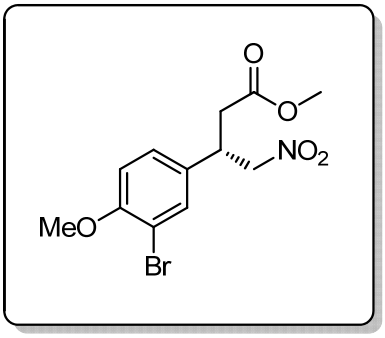
[12] Racemic **2h** was prepared for the synthesis of the antidepressant rolipram, see: Scmidt, B.; Elizarov, N.; Berger, R.; Petersen, M. H. *Synthesis* **2013**, *45*, 1174–1180.

[13] The chemical shift of ^{13}C NMR spectra is consistent with the theoretical calculation by SPARTAN'14, equilibrium geometry, density function model, MMFF, EDF2/6-31G: δ 172.7, 156.0, 135.8, 131.3, 124.3, 110.8, 110.6, 78.8, 54.4, 52.2, 38.5, 35.4. An error (δ 144.2) was reported in the aforementioned literature: δ 171.0, 155.8, 144.2, 132.2, 131.9, 127.8, 112.4, 79.5, 56.4, 52.2, 39.3, 37.7





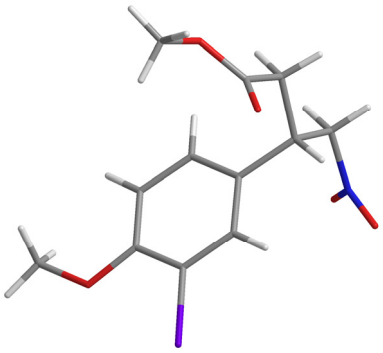
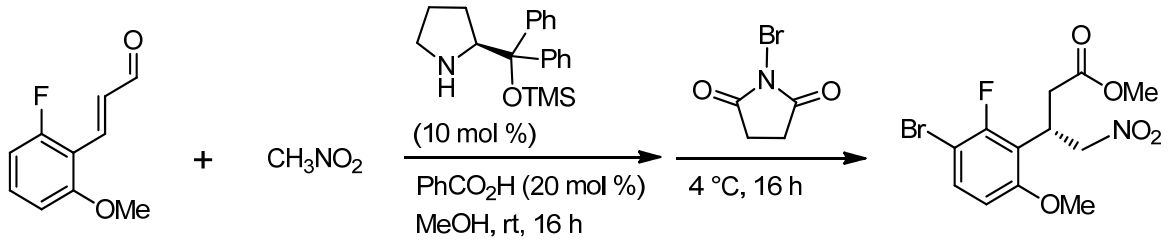


Figure S1. ORTEP and Stereo plots for X-ray crystal structures of (–)-**2h**.

CCDC-1420364 contains the supplementary crystallographic data for (–)-**2h**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

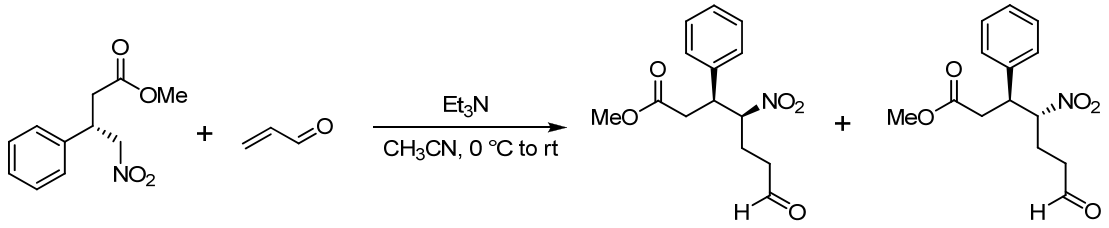
Preparation of (*S*)-methyl 3-(3-bromo-6-fluoro-2-methoxyphenyl)-4-nitrobutanoate (2i)



1i **2i**

To a solution of (*E*)-3-(2-fluoro-6-methoxyphenyl)acrylaldehyde (**1i**, 180.2 mg, 1.0 mmol), (*S*)-2-(diphenyl(trimethylsilyloxy)methyl)pyrrolidine (32.6 mg, 0.1 mmol, 0.1 equiv) and benzoic acid (24.4 mg, 0.2 mmol, 0.2 equiv) in methanol (2 mL) was added nitromethane (183.1 mg, 3.0 mmol, 3.0 equiv) at ambient temperature. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction temperature was then reduced to 4 °C, followed by the addition of *N*-bromosuccinimide (534 mg, 3.0 mmol, 3.0 equiv) to the reaction mixture. The resulting solution was vigorously stirred at 4 °C for 16 h until the completion of the reaction, as monitored by TLC. The reaction mixture was flushed through a small silica gel column with 20% EtOAc–hexane and concentrated *in vacuo* to give the orange oil residue. The crude product was purified by flash column chromatography with 5:10:85 EtOAc–CH_2Cl_2–hexane (R_f = 0.14) to afford the product **2i** (203.1 mg, 58% yield) as a pale yellow oil. Selected spectroscopic data of **2i**: $[\alpha]_D^{26}$ –11.2 (*c* 0.3, CH_2Cl_2); IR (neat): 2951, 2844, 1738, 1603, 1554, 1478, 1439, 1377, 1282, 1219, 1175, 1088, 998, 881, 803 cm^{-1}; ^1H NMR (400 MHz, acetone-d_6) δ 7.53 (dd, *J* = 8.5, 8.5 Hz, 1 H), 6.88 (d, *J* = 8.5 Hz, 1 H), 5.05 – 4.90 (m, 2 H), 4.57 – 4.47 (m, 1 H), 3.94 (s, 3 H), 3.58 (s, 3 H), 2.91 (d, *J* = 7.5 Hz, 2 H); ^{13}C NMR (100 MHz, acetone-d_6): *δ* 171.8 (C), 159.4 (d, *J* = 4 Hz, C), 158.2 (d, *J* = 241 Hz, C), 133.3 (d, *J* = 2 Hz, CH), 117.0 (d, *J* = 17 Hz, C), 109.8 (d, *J* = 4 Hz, CH), 100.4 (d, *J* = 23 Hz, C), 77.5 (d, *J* = 2 Hz, CH_2), 56.8 (CH_3), 51.9 (CH_3), 35.4 (d, *J* = 1 Hz, CH_2), 32.0 (d, *J* = 3 Hz, CH); MS (*m/z*, relative intensity): 351 (M^++2, 20), 349 (M^+, 20), 304 (17), 302 (17), 273 (32), 271 (32), 245 (36), 243 (38), 230 (34), 229 (35), 164 (76), 150 (54), 149 (66), 136 (100); exact mass calculated for $C_{12}H_{13}O_5NBrF$ (M^+): 348.9961; found: 348.9958.

Preparation of (3*S*,4*S*)-methyl 4-nitro-7-oxo-3-phenylheptanoate (*syn*-3a) and (3*S*,4*R*)-methyl 4-nitro-7-oxo-3-phenylheptanoate (*anti*-3a)



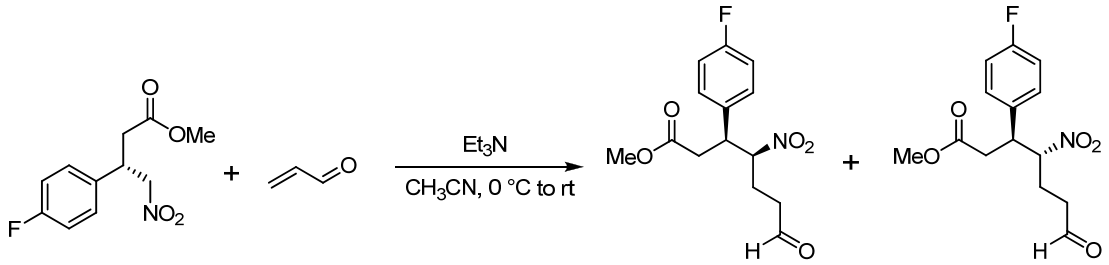
2a *syn*-**3a** *anti*-**3a**

To a solution of **2a** (22.3 mg, 0.1 mmol) and Et_3N (10.1 mg, 0.1 mmol, 1 equiv) in CH_3CN (0.9 mL) was added a solution of acrylaldehyde (11.2 mg, 0.2 mmol, 2.0 equiv) in CH_3CN (0.1 mL) at 0 °C. The resulting solution was stirred at room temperature for 18 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.33 for *syn*-**3a**, R_f = 0.17 for *anti*-**3a**, in 25% EtOAc–hexane) to afford the product *syn*-**3a** (16.5 mg, 59% yield) and *anti*-**3a** (6.4 mg, 23% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3a**: $[\alpha]_D^{28}$ –4.6 (*c* 0.5. CH_2Cl_2); IR (neat): 2953, 2849, 1736, 1548, 1495, 1437, 1367, 1258, 1166, 1089, 851, 770, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.62 (s, 1 H), 7.37 – 7.25 (m, 3 H), 7.22 – 7.17 (m, 2 H), 4.82 – 4.75 (m, 1 H), 3.69 – 3.63 (m, 1 H), 3.50 (m, 3 H), 2.75 (dd, J = 15.8, 10.1 Hz, 1 H), 2.62 (dd, J = 15.8, 4.4 Hz, 1 H), 2.49 – 2.33 (m, 2 H), 2.03 – 1.93 (m, 1 H), 1.89 – 1.81 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2 (CH), 170.7 (C), 137.2 (C), 129.2 (2 CH), 128.2 (CH), 128.1 (2 CH), 91.1 (CH), 51.8 (CH_3), 45.7 (CH), 39.6 (CH_2), 37.7 (CH_2), 24.3 (CH_2); MS (*m/z*, relative intensity): 279 (M^+, 1), 248 (5), 233 (7), 215 (10), 204 (17), 201 (50), 173 (18), 159 (26), 155 (23), 129 (100), 121 (54); exact mass calculated for $C_{14}H_{17}O_5N$ (M^+): 279.1107; found: 279.1107.

Selected spectroscopic data of *anti*-**3a**: $[\alpha]_D^{28}$ 1.6 (*c* 0.5, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.73 (s, 1 H), 7.31 – 7.24 (m, 3 H), 7.16 – 7.12 (m, 2 H), 4.87 – 4.81 (m, 1 H), 3.71 – 3.66 (m, 1 H), 3.58 (s, 3 H), 2.91 (dd, J = 16.3, 6.1 Hz, 1 H), 2.76 (dd, J = 16.3, 8.5 Hz, 1 H), 2.62 – 2.45 (m, 2 H), 2.28 – 2.08 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.5 (CH), 171.3 (C), 137.4 (C), 128.8 (2 CH), 128.2 (CH), 127.9 (2 CH), 90.7 (CH), 52.0 (CH_3), 45.2 (CH), 39.8 (CH_2), 36.5 (CH_2), 23.5 (CH_2).

Preparation of (3*S*,4*S*)-methyl 3-(4-fluorophenyl)-4-nitro-7-oxoheptanoate (*syn*-3b) and (3*S*,4*R*)-methyl 3-(4-fluorophenyl)-4-nitro-7-oxoheptanoate (*anti*-3b)



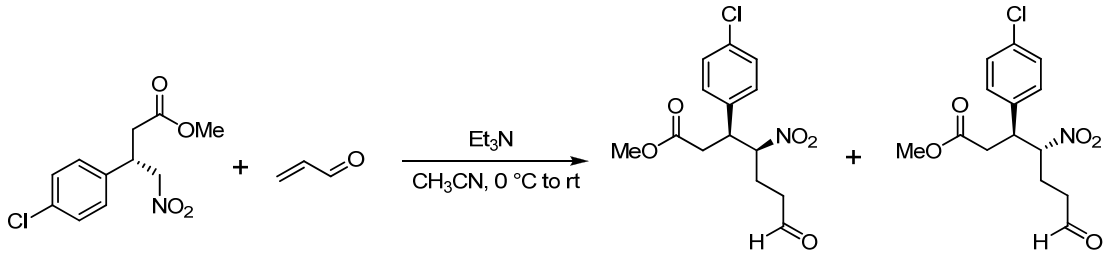
2b *syn*-**3b** *anti*-**3b**

To a solution of **2b** (48.2 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 24 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.29 for *syn*-**3b**, R_f = 0.11 for *anti*-**3b**, in 25% EtOAc–hexane) to afford the product *syn*-**3b** (36.3 mg, 61% yield) and *anti*-**3b** (14.9 mg, 25% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3b**: $[\alpha]_D^{28}$ –3.0 (c 0.5, CH_2Cl_2); IR (neat): 2954, 2848, 1736, 1605, 1549, 1511, 1437, 1364, 1226, 1163, 1103, 1015, 840, 773 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.64 (s, 1 H), 7.21 – 7.16 (m, 2 H), 7.03 (dd, J = 8.5, 8.5 Hz, 2 H), 4.79 – 4.73 (m, 1 H), 3.69 – 3.62 (m, 1 H), 3.51 (s, 3 H), 2.71 (dd, J = 15.9, 10.1Hz, 1 H), 2.61 (dd, J = 15.9, 4.6 Hz, 1 H), 2.51 – 2.36 (m, 2 H), 2.00 – 1.91 (m, 1 H), 1.89 – 1.80 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2 (CH), 170.6 (C), 162.4 (d, J = 245 Hz, C), 133.0 (d, J = 3 Hz, C), 129.8 (d, J = 9 Hz, 2 CH), 116.2 (d, J = 22 Hz, 2 CH), 90.9 (CH), 51.86 (CH_3), 45.0 (CH), 39.5 (CH_2), 37.7 (CH_2), 24.2 (CH_2); MS (*m/z*, relative intensity): 297 (M^+, 1), 266 (3), 251 (5), 233 (8), 222 (12), 219 (31), 194 (16), 191 (16), 177 (26), 173 (18), 149 (41), 147 (100), 139 (85), 122 (52), 109 (79); exact mass calculated for $C_{14}H_{16}O_5NF$ (M^+): 297.1013; found: 297.1015.

Selected spectroscopic data of *anti*-**3b**: $[\alpha]_D^{28}$ 2.9 (*c* 0.2, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.74 (s, 1 H), 7.12 (dd, J = 8.5, 5.2 Hz, 2 H), 6.97 (dd, J = 8.5, 8.5 Hz, 2 H), 4.83 – 4.78 (m, 1 H), 3.70 – 3.63 (m, 1 H), 3.58 (s, 3 H), 2.89 (dd, J = 16.3, 5.8 Hz, 1 H), 2.72 (dd, J = 16.2, 8.8 Hz, 1 H), 2.62 – 2.46 (m, 2 H), 2.28 – 2.20 (m, 1 H), 2.16 – 2.06 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4 (CH), 171.1 (C), 162.4 (d, J = 245 Hz, C), 133.1 (d, J = 3 Hz, C), 129.6 (d, J = 8 Hz, 2 CH), 115.8 (d, J = 21 Hz, 2 CH), 90.7 (CH), 52.0 (CH_3), 44.5 (CH), 39.7 (CH_2), 36.6 (CH_2), 23.6 (CH_2).

Preparation of (3*S*,4*S*)-methyl 3-(4-chlorophenyl)-4-nitro-7-oxoheptanoate (*syn*-3c) and (3*S*,4*R*)-methyl 3-(4-chlorophenyl)-4-nitro-7-oxoheptanoate (*anti*-3c)



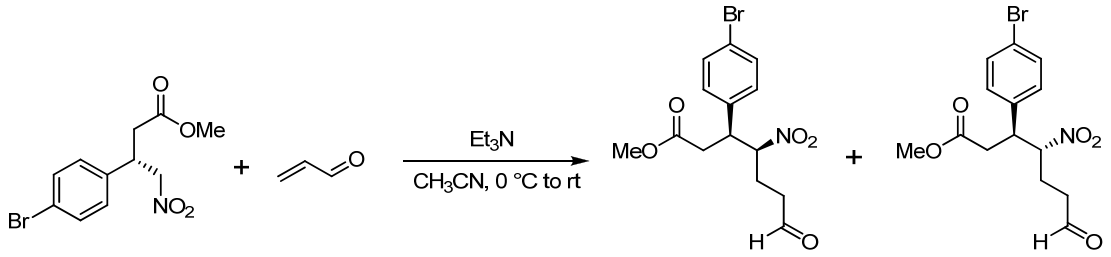
2c *syn*-**3c** *anti*-**3c**

To a solution of **2c** (51.5 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 22 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.35 for *syn*-**3c**, R_f = 0.16 for *anti*-**3c**, in 25% EtOAc–hexane) to afford the product *syn*-**3c** (33.9 mg, 54% yield) and *anti*-**3c** (18.8 mg, 30% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3c**: $[\alpha]_D^{28}$ –8.5 (*c* 0.5, CH_2Cl_2); IR (neat): 2953, 2844, 1735, 1549, 1493, 1437, 1415, 1362, 1235, 1167, 1093, 1014, 833, 728 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.63 (s, 1 H), 7.31 (d, J = 8.5 Hz, 2 H), 7.15 (J = 8.5 Hz, 2 H), 4.79 – 4.72 (m, 1 H), 3.67 – 3.61 (m, 1 H), 3.50 (s, 3 H), 2.71 (dd, J = 16.0, 10.2 Hz, 1 H), 2.61 (dd, J = 16.0, 4.5 Hz, 1 H), 2.50 – 2.35 (m, 2 H), 1.99 – 1.90 (m, 1 H), 1.87 – 1.79 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2 (CH), 170.5 (C), 135.6 (C), 134.1 (C), 129.5 (2 CH), 129.4 (2 CH), 90.7 (CH), 51.9 (CH_3), 45.0 (CH), 39.4 (CH_2), 37.4 (CH_2), 24.2 (CH_2); MS (*m/z*, relative intensity): 315 (M^++2, 0.1), 313 (M^+, 0.2), 282 (6), 267 (16), 235 (55), 210 (25), 207 (24), 193 (43), 165 (70), 163 (94), 155 (100); exact mass calculated for $C_{14}H_{16}O_5NCl$ (M^+): 313.0717; found: 313.0719.

Selected spectroscopic data of *anti*-**3c**: $[\alpha]_D^{28}$ 13.4 (*c* 0.25, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.74 (s, 1 H), 7.25 (d, J = 8.5 Hz, 2 H), 7.08 (J = 8.5 Hz, 2 H), 4.83 – 4.78 (m, 1 H), 3.66 – 3.58 (m, 1 H), 3.58 (s, 3 H), 2.89 (dd, J = 16.2, 5.8 Hz, 1 H), 2.72 (dd, J = 16.2, 8.8 Hz, 1 H), 2.63 – 2.45 (m, 2 H), 2.28 – 2.19 (m, 1 H), 2.16 – 2.07 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.3 (CH), 170.9 (C), 135.8 (C), 134.0 (C), 129.2 (2 CH), 129.0 (2 CH), 90.5 (CH), 52.0 (CH_3), 44.5 (CH), 39.6 (CH_2), 36.4 (CH_2), 23.5 (CH_2).

Preparation of (3*S*,4*S*)-methyl 3-(4-bromophenyl)-4-nitro-7-oxoheptanoate (*syn*-3d) and (3*S*,4*R*)-methyl 3-(4-bromophenyl)-4-nitro-7-oxoheptanoate (*anti*-3d)



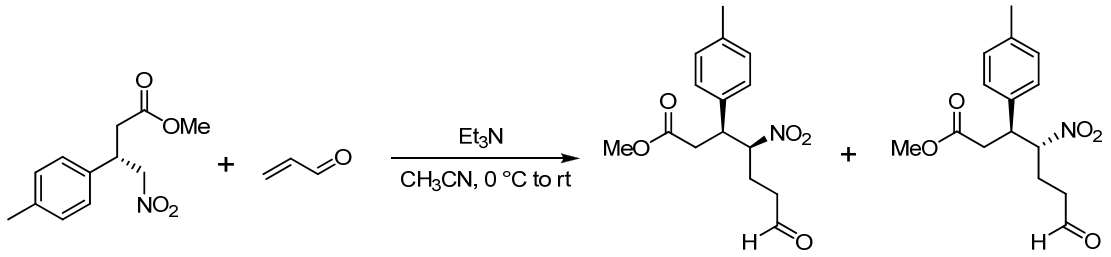
2d *syn*-**3d** *anti*-**3d**

To a solution of **2d** (60.4 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 22 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.31 for *syn*-**3d**, R_f = 0.16 for *anti*-**3d**, in 25% EtOAc–hexane) to afford the product *syn*-**3d** (40.8 mg, 57% yield) and *anti*-**3d** (17.9 mg, 25% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3d**: $[\alpha]_D^{28}$ –2.6 (*c* 0.5, CH_2Cl_2); IR (neat): 2924, 2851, 2730, 1731,1548, 1488, 1436, 1412, 1361, 1235, 1166, 1109, 1072, 1011, 827, 722 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.65 (s, 1 H), 7.47 (d, J = 8.5 Hz, 2 H), 7.09 (d, J = 8.5 Hz, 2 H), 4.79 – 4.73 (m, 1 H), 3.67 – 3.61 (m, 1 H), 3.52 (s, 3 H), 2.71 (dd, J = 16.0, 10.2 Hz, 1 H), 2.61 (dd, J = 16.0, 4.4 Hz, 1 H), 2.49 – 2.35 (m, 2 H), 2.03 – 1.90 (m, 1 H), 1.88 – 1.80 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.2 (CH), 170.5 (C), 136.3 (C), 132.4 (2 CH), 129.8 (2 CH), 122.3 (C), 90.7 (CH), 51.9 (CH_3), 45.2 (CH), 39.5 (CH_2), 37.5 (CH_2), 24.3 (CH_2); MS (*m/z*, relative intensity): 359 (M^++2, 0.5), 357 (M^+, 0.5), 328 (4), 326 (5), 313 (16), 311 (17), 281 (58), 279 (59), 237 (48), 209 (67), 201 (76), 199 (79), 184 (52), 182 (54), 128 (100), 116 (91), 115 (89); exact mass calculated for $C_{14}H_{16}O_5NBr$ (M^+): 357.0212; found: 357.0212.

Selected spectroscopic data of *anti*-**3d**: $[\alpha]_D^{28}$ 14.0 (*c* 0.4, CH_2Cl_2) ^1H NMR (400 MHz,$CDCl_3$): δ 9.73 (s, 1 H), 7.41 (d, J = 8.4 Hz, 2 H), 7.02 (d, J = 8.4 Hz, 2 H), 4.86 – 4.78 (m, 1 H), 3.65 – 3.61 (m, 1 H), 3.58 (s, 3 H), 2.88 (dd, J = 16.3, 5.7 Hz, 1 H), 2.71 (dd, J = 16.3, 8.8 Hz, 1 H), 2.63 – 2.44 (m, 2 H), 2.27 – 2.19 (m, 1 H), 2.15 – 2.06 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.3 (CH), 171.0 (C), 136.5 (C), 132.0 (2 CH), 129.6 (2 CH), 122.3 (C), 90.5 (CH), 52.1 (CH_3), 44.7 (CH), 39.7 (CH_2), 36.4 (CH_2), 23.6 (CH_2).

Preparation of (3*S*,4*S*)-methyl 4-nitro-7-oxo-3-*p*-tolylheptanoate (*syn*-3e) and (3*S*,4*R*)-methyl 4-nitro-7-oxo-3-*p*-tolylheptanoate (*anti*-3e)



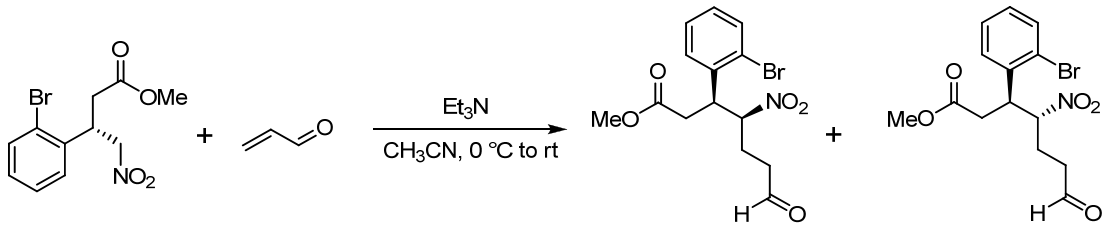
2e *syn*-**3e** *anti*-**3e**

To a solution of **2e** (47.5 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 17 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.31 for *syn*-**3e**, R_f = 0.16 for *anti*-**3e**, in 25% EtOAc–hexane) to afford the product *syn*-**3e** (41.7 mg, 71% yield) and *anti*-**3e** (10.6 mg, 18% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3e**: $[\alpha]_D^{28}$ –3.5 (*c* 0.5, CH_2Cl_2); IR (neat): 2924, 2849, 2730, 1735, 1548, 1514, 1436, 1362, 1255, 1163, 1116, 1020, 959, 820, 722 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.63 (s, 1 H), 7.13 (d, J = 8.0 Hz, 2 H), 7.07 (d, J = 8.0 Hz, 2 H), 4.78 – 4.72 (m, 1 H), 3.65 – 3.58 (m, 1 H), 3.50 (s, 3 H), 2.73 (dd, J = 15.7, 10.2 Hz, 1 H), 2.60 (dd, J = 15.7, 4.4 Hz, 1 H), 2.48 – 2.33 (m, 2 H), 2.30 (s, 3 H), 2.02 – 1.92 (m, 1 H), 1.90 – 1.81 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.3 (CH), 170.8 (C), 137.9 (C), 134.1 (C), 129.9 (2 CH), 127.9 (2 CH), 91.2 (CH), 51.8 (CH_3), 45.3 (CH), 39.6 (CH_2), 37.7 (CH_2), 24.3 (CH_2), 21.1 (CH_3); MS (*m/z*, relative intensity): 293 (M^+, 0.4), 262 (7), 246 (19), 215 (44), 190 (33), 173 (31), 169 (28), 143 (99), 135 (100), 129 (62), 105 (49); exact mass calculated for $C_{15}H_{19}O_5N$ (M^+): 293.1263; found: 293.1262.

Selected spectroscopic data of *anti*-**3e**: $[\alpha]_D^{28}$ 9.4 (*c* 0.5, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.73 (s, 1 H), 7.09 (d, J = 8.0 Hz, 2 H), 7.02 (d, J =8.0 Hz, 2 H), 4.84 – 4.78 (m, 1 H), 3.65 – 3.57 (m, 1 H), 3.58 (s, 3 H), 2.89 (dd, J = 16.2, 6.0 Hz, 1 H), 2.74 (dd, J = 16.2, 8.6 Hz, 1 H), 2.61 – 2.45 (m, 2 H), 2.28 (s, 3 H), 2.26 – 2.07 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.5 (CH), 171.4 (C), 137.9 (C), 134.3 (C), 129.5 (2 CH), 127.7 (2 CH), 90.8 (CH), 52.0 (CH_3), 44.8 (CH), 39.8 (CH_2), 36.5 (CH_2), 23.4 (CH_2), 21.1 (CH_3).

Preparation of (3*S*,4*S*)-methyl 3-(2-bromophenyl)-4-nitro-7-oxoheptanoate (*syn*-3f) and (3*S*,4*R*)-methyl 3-(2-bromophenyl)-4-nitro-7-oxoheptanoate (*anti*-3f)



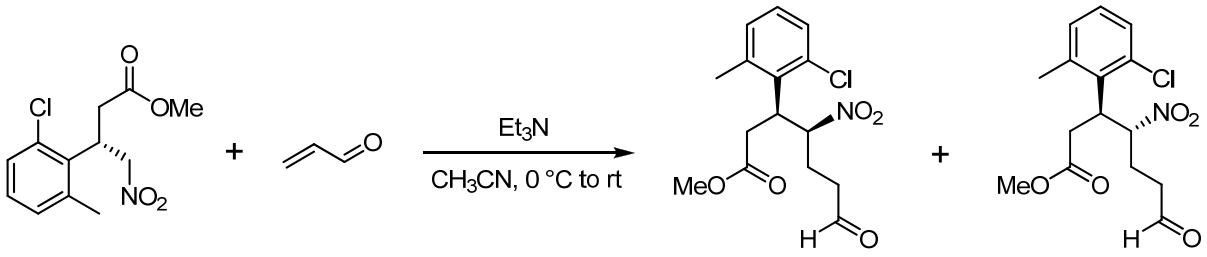
2f *syn*-**3f** *anti*-**3f**

To a solution of **2f** (60.4 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 20 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.29 for *syn*-**3f**, R_f = 0.20 for *anti*-**3f**, in 25% EtOAc–hexane) to afford the product *syn*-**3f** (40.8 mg, 57% yield) and *anti*-**3f** (16.5 mg, 23% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3f**: $[\alpha]_D^{28}$ –29.2 (*c* 0.5, CH_2Cl_2); IR (neat): 2953, 2849, 2726, 1736, 1549, 1473, 1436, 1362, 1252, 1167, 1022, 851, 756 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6): δ 9.64 (s, 1 H), 7.68 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.49 (dd, *J* = 8.0, 2.0 Hz, 1 H), 7.41 (ddd, *J* = 8.0, 7.5, 1.0 Hz, 1 H), 7.25 (ddd, *J* = 8.0, 7.5, 2.0 Hz, 1 H), 5.01 – 4.99 (m, 1 H), 4.38 – 4.36 (m, 1 H), 3.49 (s, 3 H), 2.96 (dd, *J* = 16.0, 10.0 Hz, 1 H), 2.75 (dd, *J* = 16.0, 4.5 Hz, 1 H), 2.53 – 2.50 (m, 2 H), 2.21 – 2.13 (m, 1 H), 1.94 – 1.87 (m, 1 H); ^{13}C NMR (125 MHz, acetone-d_6): δ 200.6 (CH), 171.1 (C), 138.6 (C), 134.3 (CH), 130.4 (CH), 129.8 (CH), 129.3 (CH), 126.4 (C), 92.1 (CH), 51.9 (CH_3), 44.4 (CH), 40.2 (CH_2), 37.1 (CH_2), 24.8 (CH_2); MS (*m/z*, relative intensity): 359 (M^++2, 0.7), 357 (M^+, 0.7), 279 (12), 278 (65), 209 (22), 201 (30), 199 (32), 184 (23), 182 (25), 171 (22), 161 (100); Exact mass calculated for $C_{14}H_{16}O_5NBr$ (M^+): 357.0212; found: 357.0210.

Selected spectroscopic data of *anti*-**3f**: $[\alpha]_D^{28}$ –7.5 (*c* 0.5, CH_2Cl_2); ^1H NMR (500 MHz, acetone-d_6): δ 9.73 (s, 1 H), 7.62 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.45 (dd, *J* = 8.0, 2.0 Hz, 1 H), 7.37 (ddd, J = 8.0, 8.0, 1.0 Hz, 1 H), 7.21 (ddd, J = 8.0, 8.0, 2.0 Hz, 1 H), 5.20 – 5.12 (m, 1 H), 4.42 – 4.35 (m, 1 H), 3.55 (s, 3 H), 3.03 – 2.91 (m, 2 H), 2.66 – 2.54 (m, 2 H), 2.41 – 2.34 (m, 1 H), 2.26 – 2.18 (m, 1 H); ^{13}C NMR (125 MHz, acetone-d_6): δ 200.8 (CH), 171.5 (C), 138.9 (C), 134.3 (CH), 130.4 (CH), 129.7 (CH), 128.9 (CH), 125.9 (C), 91.0 (CH), 52.1 (CH_3), 44.4 (CH), 40.4 (CH_2), 36.6 (CH_2), 24.1 (CH_2). Two aryl carbons are broadened and disappeared due to the slow rotation and coalescence phenomenon.

Preparation of (3*S*,4*S*)-methyl 3-(2-chloro-6-methylphenyl)-4-nitro-7-oxoheptanoate (*syn*-3g) and (3*S*,4*R*)-methyl 3-(2-chloro-6-methylphenyl)-4-nitro-7-oxoheptanoate (*anti*-3g)



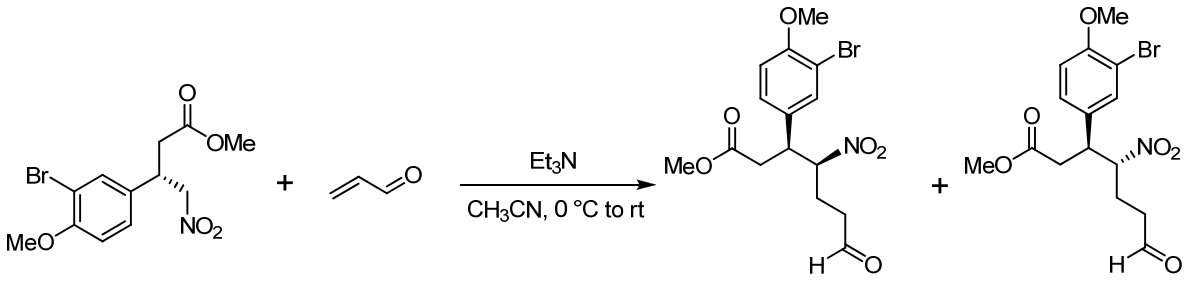
2g *syn*-**3g** *anti*-**3g**

To a solution of **2f** (54.3 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 19 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 15% EtOAc–hexane (R_f = 0.43 for *syn*-**3g**, R_f = 0.31 for *anti*-**3g**, in 25% EtOAc–hexane) to afford the product *syn*-**3g** (18.4 mg, 28% yield) and *anti*-**3g** (36.1 mg, 55% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3g**: $[\alpha]_D^{28}$ –21.9 (*c* 0.5, CH_2Cl_2); IR (neat): 2953, 2847, 2730, 1738, 1551, 1437, 1369, 1232, 1200, 1168, 1129, 1071, 969, 848, 780 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.62 (s, 1 H), 7.20 (dd, *J* = 6.6, 2.8 Hz, 1 H), 7.14 – 7.07 (m, 2 H), 5.45 – 5.39 (m, 1 H), 4.35 – 4.29 (m, 1 H), 3.50 (s, 3 H), 3.37 (dd, *J* = 16.5, 9.9 Hz, 1 H), 2.60 (dd, *J* = 16.5, 4.4 Hz, 1 H), 2.56 (s, 3 H), 2.43 – 2.37 (m, 2 H), 2.02 – 1.92 (m, 1 H), 1.76 – 1.67 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.9 (CH), 171.3 (C), 141.1 (C), 133.4 (C), 133.3 (C), 130.0 (CH), 129.5 (CH), 129.0 (CH), 89.7 (CH), 51.8 (CH_3), 40.5 (CH), 39.9 (CH_2), 35.2 (CH_2), 24.3 (CH_2), 21.7 (CH_3); MS (*m/z*, relative intensity): 326 (M^+-1, 4), 292 (28), 249 (13), 236 (35), 223 (38), 177 (73), 169 (96), 129 (71), 115 (100); exact mass calculated for $C_{15}H_{18}O_5NCl$ (M^+): 327.0874; found: 327.0872.

Selected spectroscopic data of *anti*-**3g**: $[\alpha]_D^{28}$ 17.1 (*c* 0.5, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.77 (s, 1 H), 7.15 (dd, *J* = 7.0, 2.4 Hz, 1 H), 7.05 – 6.99 (m, 2 H), 5.56 – 5.50 (m, 1 H), 4.33 – 4.27 (m, 1 H), 3.59 (s, 3 H), 3.17 (dd, *J* = 16.7, 8.0 Hz, 1 H), 2.90 (dd, *J* = 16.7, 5.2 Hz, 1 H), 2.60 – 2.56 (m, 2 H), 2.44 (s, 3 H), 2.40 – 2.31 (m, 1 H), 2.23 – 2.14 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.3 (CH), 171.9 (C), 140.7 (C), 133.8 (C), 133.4 (C), 129.9 (CH), 129.3 (CH), 128.9 (CH), 88.6 (CH), 52.1 (CH_3), 40.0 (CH), 39.6 (CH_2), 34.9 (CH_2), 24.5 (CH_2), 20.9 (CH_3).

Preparation of (3*S*,4*S*)-methyl 3-(3-bromo-4-methoxyphenyl)-4-nitro-7-oxoheptanoate (*syn*-3h) and (3*S*,4*R*)-methyl 3-(3-bromo-4-methoxyphenyl)-4-nitro-7-oxoheptanoate (*anti*-3h)



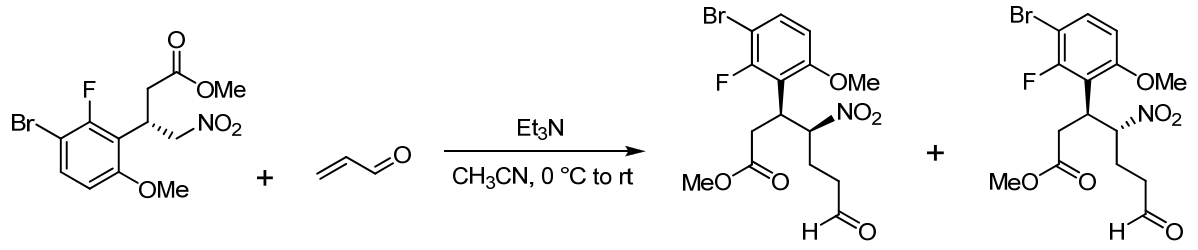
2h *syn*-**3h** *anti*-**3h**

To a solution of **2h** (66.4 mg, 0.2 mmol) and Et_3N (20.2 mg, 0.2 mmol, 1 equiv) in CH_3CN (1.8 mL) was added a solution of acrylaldehyde (22.4 mg, 0.4 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 13 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 20% EtOAc–hexane (R_f = 0.27 for *syn*-**3h**, R_f = 0.17 for *anti*-**3h**, in 30% EtOAc–hexane) to afford the product *syn*-**3h** (46.6 mg, 60% yield) and *anti*-**3h** (16.3 mg, 21% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3h**: $[\alpha]_D^{28}$ 1.1 (*c* 1.6, CH_2Cl_2); IR (neat): 2926, 2842, 2730, 1733, 1603, 1548, 1497, 1437, 1361, 1284, 1259, 1166, 1054, 1018, 816 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.65 (s, 1 H), 7.38 (d, J = 2.2 Hz, 1 H), 7.13 (dd, J = 8.4, 2.2 Hz, 1 H), 6.86 (d, J = 8.4 Hz, 1 H), 4.75 – 4.69 (m, 1 H), 3.87 (s, 3 H), 3.62 – 3.55 (m, 1 H), 3.53 (s, 3 H), 2.69 (dd, J = 16.0, 10.1 Hz, 1 H), 2.60 (dd, J = 16.0, 4.6 Hz, 1 H), 2.51 – 2.36 (m, 2 H), 2.02 – 1.92 (m, 1 H), 1.92 – 1.82 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2 (CH), 170.6 (C), 155.7 (C), 132.7 (CH), 130.7 (C), 128.4 (CH), 112.30 (CH), 112.26 (C), 90.9 (CH), 56.2 (CH_3), 51.9 (CH_3), 44.6 (CH), 39.5 (CH_2), 37.6 (CH_2), 24.3 (CH_2); MS (*m/z*, relative intensity): 389 (M^++2, 13), 387 (M^+, 13), 342 (23), 340 (23), 311 (31), 309 (31), 286 (44), 284 (46), 239 (44), 231 (97), 229 (100), 212 (43), 158 (55), 146 (67); exact mass calculated for $C_{15}H_{18}O_6NBr$ (M^+): 387.0317; found: 387.0319.

Selected spectroscopic data of *anti*-**3h**: $[\alpha]_D^{26}$ 9.1 (*c* 0.6, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.73 (s, 1H), 7.33 (d, J = 2.2 Hz, 1H), 7.05 (dd, J = 8.5, 2.2 Hz, 1H), 6.80 (d, J = 8.5 Hz, 1H), 4.82 – 4.76 (m, 1H), 3.84 (s, 3H), 3.60 (s, 3H), 3.55 – 3.50 (m, 1H), 2.86 (dd, J = 16.2, 5.8 Hz, 1H), 2.70 (dd, J = 16.2, 8.7 Hz, 1H), 2.62 – 2.46 (m, 2H), 2.27 – 2.18 (m, 1H), 2.16 – 2.06 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.3 (CH), 171.1 (C), 155.7 (C), 132.7 (CH), 130.9 (C), 128.1 (CH), 112.0 (2CH), 111.9 (C), 90.7 (CH), 56.2 (CH_3), 52.1 (CH_3), 44.1 (CH), 39.7 (CH_2), 36.6 (CH_2), 23.6 (CH_2).

Prerpation of (3*S*,4*S*)-methyl 3-(3-bromo-6-fluoro-2-methoxy-4-methylphenyl)-4-nitro-7-oxoheptanoate (*syn*-3i) and (3*S*,4*R*)-methyl 3-(3-bromo-6-fluoro-2-methoxyphenyl)-4-nitro-7-oxoheptanoate (*anti*-3i)

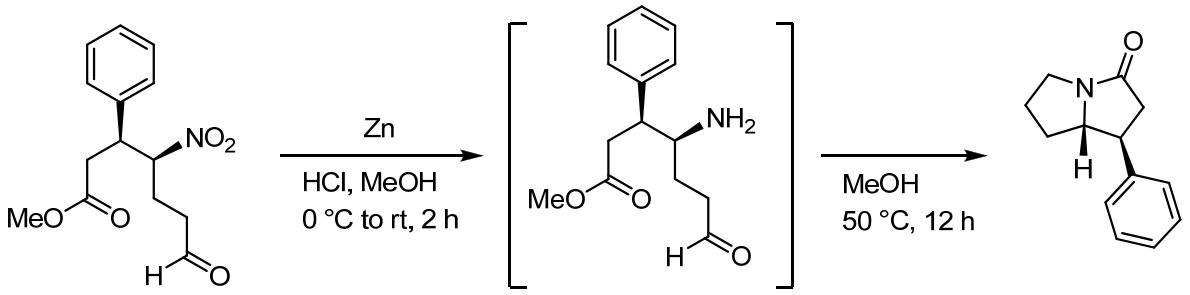


To a solution of **2i** (50.0 mg, 0.14 mmol) and Et_3N (14 mg, 0.14 mmol, 1 equiv) in CH_3CN (0.8 mL) was added a solution of acrylaldehyde (16 mg, 0.29 mmol, 2.0 equiv) in CH_3CN (0.2 mL) at 0 °C. The resulting solution was stirred at room temperature for 16 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. The crude product was purified by flash column chromatography with 20% EtOAc–hexane (R_f = 0.27 for *syn*-**3i**, R_f = 0.17 for *anti*-**3i**, in 30% EtOAc–hexane) to afford the product *syn*-**3i** (32.8 mg, 57% yield) and *anti*-**3i** (11.4 mg, 20% yield) as colorless oils.

Selected spectroscopic data of *syn*-**3i**: $[\alpha]_D^{28}$ –10.7 (*c* 0.5, CH_2Cl_2); IR (neat): 2948, 2844, 2730, 1738, 1551, 1478, 1439, 1362, 1284, 1222, 1170, 1088, 804 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.64 (s, 1 H), 7.42 (dd, J = 9.0, 8.0 Hz, 1 H), 6.62 (dd, *J* = 9.0, 1.6 Hz, 1 H), 5.10 – 5.04 (m, 1 H), 4.32 – 4.26 (m, 1 H), 3.88 (s, 3 H), 3.51 (s, 3 H), 3.01 (dd, *J* = 15.2, 10.4 Hz, 1 H), 2.52 (dd, *J* = 16.0, 4.4 Hz, 1 H), 2.44 – 2.41 (m, 2 H), 2.01 – 1.91 (m, 1 H), 1.85 – 1.76 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2 (CH), 170.8 (C), 157.7 (d, *J* = 245 Hz, C), 157.9 (d, *J* = 7 Hz, C), 133.0 (d, *J* = 2 Hz, CH), 114.8 (d, *J* = 16 Hz, C) 108.3 (d, *J* = 4 Hz, CH), 100.7 (d, *J* = 24 Hz, C), 88.8 (CH), 56.3 (CH_3), 51.8 (CH_3), 39.3 (CH_2), 35.9 (CH), 35.0 (CH_2), 24.4 (CH_2); MS (*m/z*, relative intensity): 407 (M^++2, 79), 405 (M^+, 80), 329 (60), 327 (62), 301 (54), 299 (58), 259 (42), 257 (51), 249 (75), 247 (77), 219 (65), 217 (76), 202 (56), 150 (40), 136 (100); exact mass calculated for $C_{15}H_{17}BrFNO_6$ (M^+): 405.0223; found: 405.0220.

Selected spectroscopic data of *anti*-**3i**: $[\alpha]_D^{28}$ 14.3 (*c* 0.5, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$): δ 9.75 (s, 1 H), 7.37 (dd, *J* = 8.8, 8.0 Hz, 1 H), 6.55 (dd, *J* = 8.8, 1.2 Hz, 1 H), 5.08 – 5.03 (m, 1 H), 4.25 (q, *J* = 7.9 Hz, 1 H), 3.80 (s, 3 H), 3.60 (s, 3 H), 2.95 – 2.84 (m, 2 H), 2.62 – 2.48 (m, 2 H), 2.36 – 2.25 (m, 1 H), 2.19 – 2.10 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4 (CH), 171.5 (C), 157.6 (d, *J* = 245 Hz, C),158.0 (d, *J* = 6 Hz, C) 132.8 (d, *J* = 2 Hz, CH), 115.2 (d, *J* = 17 Hz, C), 108.2 (d, *J* = 3 Hz, CH), 100.6 (d, *J* = 24 Hz, C), 88.5 (CH), 56.2 (CH_3), 52.0 (CH_3), 39.7 (CH_2), 35.9 (CH), 34.6 (CH_2), 24.0 (CH_2).

Preparation of (1*S*,7a*S*)-1-phenyltetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4a)



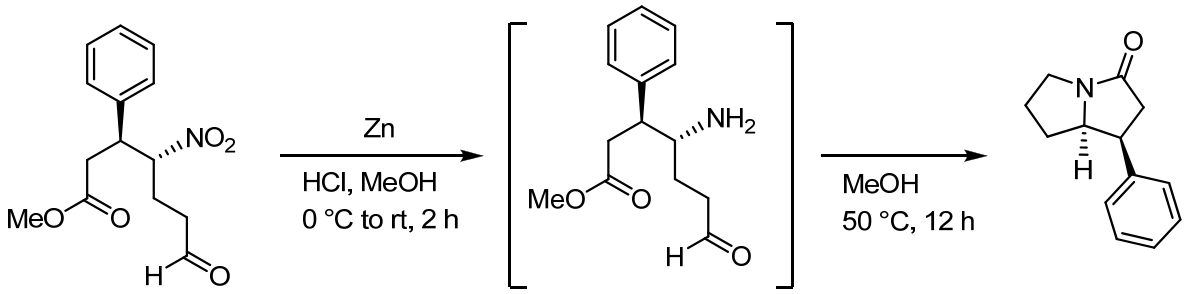
syn-**3a** *syn*-**4a**

To a solution of *syn*-**3a** (10.0 mg, 0.04 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL) was slowly added zinc powder (46.8 mg, 0.72 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *syn*-**4a** in EtOAc) to afford the product *syn*-**4a** (5.6 mg, 78% yield) as a colorless oil.

Selected spectroscopic data of *syn*-**4a**: $[\alpha]_D^{26}$ –90 (*c* 0.22, CH_2Cl_2);[14] IR (neat): 2964, 2876, 1689, 1497, 1410, 1260, 1098, 1014, 799, 752, 701, 663 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.34 (dd, *J* = 7.5, 7.5 Hz, 2 H), 7.27 – 7.24 (m, 3 H), 3.96 – 3.91 (m, 1 H), 3.64 – 3.59 (m, 1 H), 3.30 – 3.25 (m, 1 H), 3.13 – 3.09 (m, 1 H), 2.97 (dd, *J* = 15.5, 11.0 Hz, 1 H), 2.81 (dd, *J* = 15.5, 8.5 Hz, 1 H), 2.17 – 1.98 (m, 3 H), 1.58 – 1.51 (m, 1 H);[15] ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.2 (C), 140.6 (C), 128.8 (2 CH), 127.1 (CH), 127.0 (2 CH), 68.7 (CH), 48.6 (CH), 43.2 (CH_2), 41.4 (CH_2), 31.4 (CH_2), 27.0 (CH_2); MS (*m/z*, relative intensity): 201 (M^+, 40), 181 (4), 169 (7), 131 (7), 104 (100), 70 (62), 69 (21); exact mass calculated for $C_{13}H_{15}ON$ (M^+) 201.1154; found: 201.1158.

[14] Lit. $[\alpha]_D^{22}$ –84.0 (*c* 0.5, CH_2Cl_2); Zoute, L.; Kociok-Koehn, G.; Frost, C. G. *Org. Lett.* **2009**, *11*, 2491 – 2494.
[15] Zoute, L.; Kociok-Koehn, G.; Frost, C. G. *Org. Lett.* **2009**, *11*, 2491 – 2494.

Preparation of (1*S*,7a*R*)-1-phenyltetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4a)

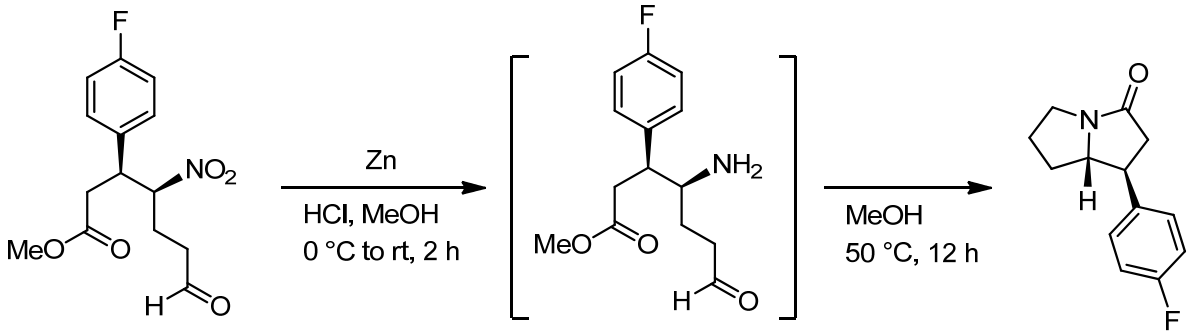


anti-**3a** *anti*-**4a**

To a solution of *anti*-**3a** (10.0 mg, 0.04 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL) was slowly added zinc powder (46.8 mg, 0.72 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.47 for *anti*-**4a** in EtOAc) to afford the product *anti*-**4a** (5.5 mg, 76% yield) as a colorless oil.
Selected spectroscopic data of *anti*-**4a**: $[\alpha]_D^{29}$ 146 (*c* 0.16, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30 (dd, J = 7.5, 7.5 Hz, 2 H), 7.23 (dd, J = 7.5, 7.5 Hz, 1 H), 7.08 (d, J = 7.5 Hz, 2 H), 4.27 – 4.23 (m, 1 H), 3.67 (t, J = 8.0 Hz, 1 H), 3.52 – 3.47 (m, 1 H), 3.17 (dd, J = 16.5, 8.5 Hz, 1 H), 3.08 – 3.04 (m, 1 H), 2.65 (dd, J = 16.5, 2.0 Hz, 1 H), 1.92 – 1.86 (m, 1 H), 1.85 – 1.78 (m, 1 H), 1.5 – 1.44 (m, 1 H), 0.95 – 0.86 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.3 (C), 140.6 (C), 128.6 (2 CH), 127.7 (2 CH), 127.1 (CH), 66.0 (CH), 41.8 (CH), 41.6 (CH_2), 41.4 (CH_2), 26.8 (CH_2), 26.4 (CH_2).[16]

[16] Zoute, L.; Kociok-Koehn, G.; Frost, C. G. *Org. Lett.* **2009**, *11*, 2491 – 2494.

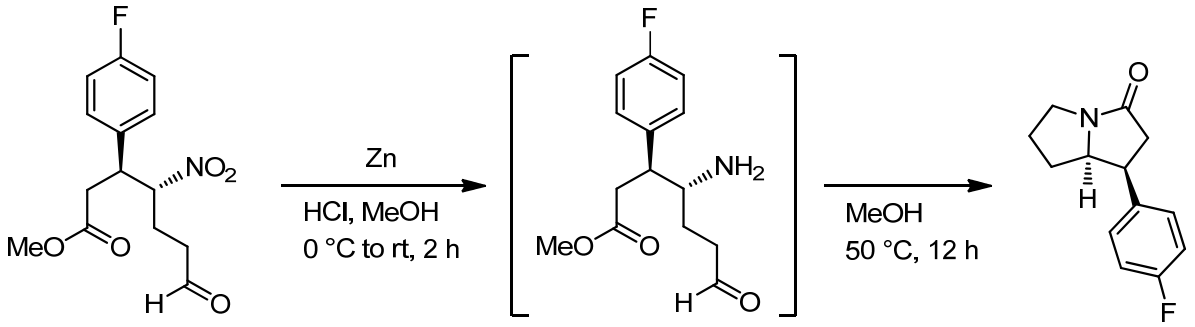
Preparation of (1*S*,7a*S*)-1-(4-fluorophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4b)



syn-**3b** *syn*-**4b**

To a solution of *syn*-**3b** (19.0 mg, 0.06 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.3 mL) was slowly added zinc powder (83.7 mg, 1.28 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (15 mL x 3) and washed with brine (15 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.45 for *syn*-**4b** in EtOAc) to afford the product *syn*-**4b** (7.2 mg, 51% yield) as a light yellow oil. Selected spectroscopic data of *syn*-**4b**: $[\alpha]_D^{27}$ –42.7 (*c* 0.5, CH_2Cl_2); IR (neat): 2968, 2878, 1692, 1603, 1511, 1413, 1259, 1224, 1161, 1096, 1015, 838, 794 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.20 (dd, J = 8.5, 5.5 Hz, 2 H), 7.02 (dd, J = 8.5, 8.5 Hz, 2 H), 3.90 – 3.86 (m, 1 H), 3.63 – 3.58 (m, 1 H), 3.25 – 3.22 (m, 1 H), 3.13 – 3.08 (m, 1 H), 2.91 (dd, J = 16.5, 12.0 Hz, 1 H), 2.79 (dd, J = 16.5, 8.5 Hz, 1 H), 2.17 – 1.99 (m, 3 H), 1.56 – 1.49 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.9 (C), 161.8 (d, J = 244.5 Hz, C), 136.2 (d, J = 3 Hz, C), 128.5 (d, J = 8.3 Hz, 2 CH), 115.6 (d, J = 21.5 Hz, 2 CH), 68.7 (CH), 47.9 (CH), 43.2 (CH_2), 41.4 (CH_2), 31.3 (CH_2), 27.0 (CH_2); MS (*m/z*, relative intensity): 219 (M^+, 67), 191 (1), 176 (1), 162 (1), 148 (7), 133 (5), 122 (100), 101 (11), 70 (100); exact mass calculated for $C_{13}H_{14}ONF$ (M^+) 219.1059; found: 219.1058.

Preparation of (1*S*,7a*R*)-1-(4-fluorophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4b)

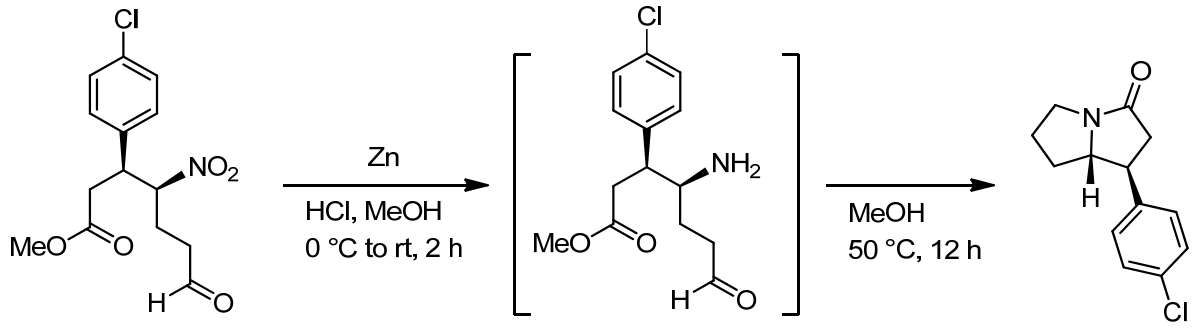


anti-**3b** *anti*-**4b**

To a solution of *anti*-**3b** (10.0 mg, 0.03 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL) was slowly added zinc powder (44.2 mg, 0.68 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.44 for *anti*-**4b** in EtOAc) to afford the product *anti*-**4a** (3.8 mg, 52% yield) as a light yellow oil. Selected spectroscopic data of *anti*-**4a**: $[\alpha]_D^{27}$ 277.1 (*c* 0.2, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.06 – 7.02 (m, 2 H), 7.00 – 6.96 (m, 2 H), 4.26 – 4.21 (m, 1 H), 3.67 – 3.64 (m, 1 H), 3.51 – 3.46 (m, 1 H), 3.17 (dd, J = 16.5, 8.5 Hz, 1 H), 3.08 – 3.03 (m, 1 H), 2.59 (dd, J = 16.5, 2.0 Hz, 1 H), 1.95 – 1.86 (m, 1 H), 1.84 – 1.76 (m, 1 H), 1.51 – 1.45 (m, 1 H), 0.93 – 0.84 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.0 (C), 161.8 (d, J = 244.5 Hz, C) 136.3 (d, J = 3.3 Hz, C), 129.1 (d, J = 7.8 Hz, 2 CH), 115.5 (d, J = 21 Hz, 2 CH), 65.9 (d, J = 1 Hz, CH), 41.8 (CH_2), 41.4 (CH_2), 41.2 (CH), 26.8 (CH_2), 26.3 (CH_2).[17]

[17] Zoute, L.; Kociok-Koehn, G.; Frost, C. G. *Org. Lett.* **2009**, *11*, 2491 – 2494.

Preparation of (1*S*,7a*S*)-1-(4-chlorophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4c)

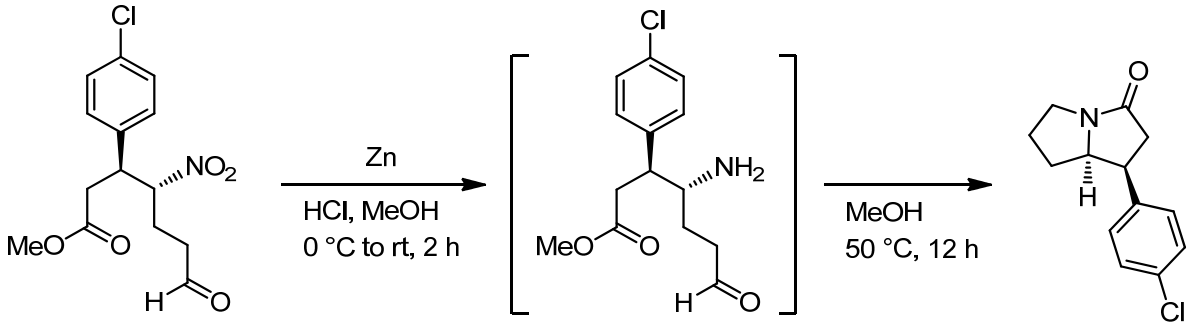


syn-**3c** *syn*-**4c**

To a solution of *syn*-**3c** (21.0 mg, 0.07 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.35 mL) was slowly added zinc powder (87.6 mg, 1.34 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.45 for *syn*-**4c** in EtOAc) to afford the product *syn*-**4c** (9.6 mg, 61% yield) as a colorless oil.

Selected spectroscopic data of *syn*-**4c**: $[\alpha]_D^{27}$ –54.1 (*c* 0.5, CH_2Cl_2); IR (neat): 2970, 2878, 1692, 1494, 1413, 1333, 1286, 1259, 1204, 1170, 1090, 1014, 831, 790 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.30 (d, J = 8.0 Hz,, 2 H), 7.17 (d, J = 8.0 Hz, 2 H), 3.90 – 3.85 (m, 1 H), 3.63 – 3.58 (m, 1 H), 3.27 – 3.21 (m, 1 H), 3.13 – 3.08 (m, 1 H), 2.91 (dd, J = 16.0, 11.5 Hz, 1 H), 2.79 (dd, J = 16.0, 8.5 Hz, 1 H), 2.17 – 1.99 (m, 3 H), 1.56 – 1.47 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.8 (C), 139.0 (C), 132.9 (C), 128.9 (2 CH), 128.4 (2 CH), 68.5 (CH), 47.9 (CH), 43.1 (CH_2), 41.4 (CH_2), 31.3 (CH_2), 27.0 (CH_2); MS (*m/z*, relative intensity): 237 (M^++2, 8), 235 (M^+, 26), 172 (4), 140 (28), 139 (8), 138 (100), 103 (12), 102 (5), 70 (96); exact mass calculated for $C_{13}H_{14}ONCl$ (M^+) 235.0764; found: 235.0764.

Preparation of (1*S*,7a*R*)-1-(4-chlorophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4c)

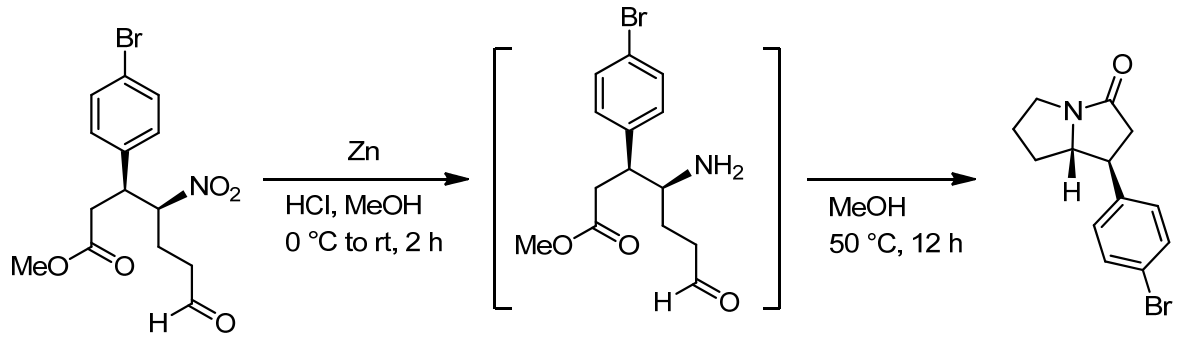


anti-**3c** *anti*-**4c**

To a solution of *anti*-**3c** (15.0 mg, 0.05 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.25 mL) was slowly added zinc powder (62.8 mg, 0.96 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.44 for *anti*-**4c** in EtOAc) to afford the product *anti*-**4c** (6.2 mg, 55% yield) as a colorless oil.

Selected spectroscopic data of *anti*-**4c**: $[\alpha]_D^{27}$ 154.2 (*c* 0.5, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.27 (d, J = 8.0 Hz, 2 H), 7.02 (d, J = 8.0 Hz,, 2 H), 4.26 – 4.22 (m, 1 H), 3.66 – 3.63 (m, 1 H), 3.51 – 3.45 (m, 1 H), 3.17 (dd, J = 17.0, 8.5 Hz, 1 H), 3.08 – 3.03 (m, 1 H), 2.58 (dd, J = 17.0, 2.0 Hz, 1 H), 1.95 – 1.86 (m, 1 H), 1.84 – 1.77 (m, 1 H), 1.51 – 1.46 (m, 1 H), 0.92 – 0.84 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.8 (C), 139.1(C), 132.9 (C), 129.0 (2 CH), 128.8 (2 CH), 65.8 (CH), 41.6 (CH_2), 41.4 (CH_2), 41.3 (CH), 26.8 (CH_2), 26.3 (CH_2).

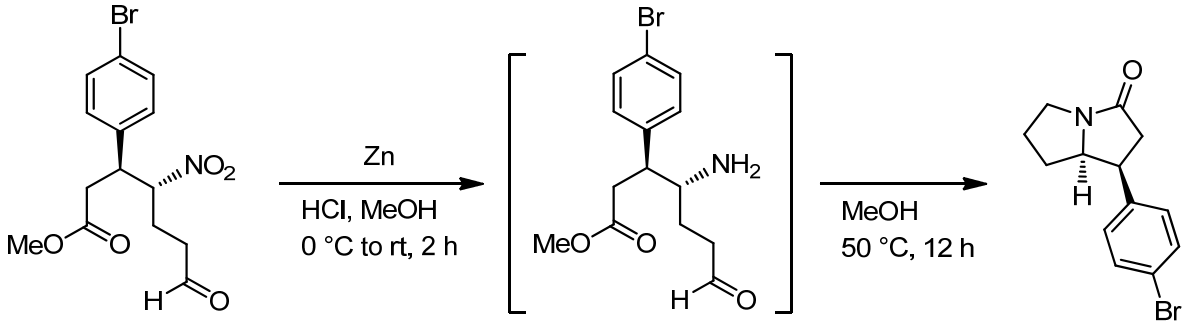
Preparation of (1*S*,7a*S*)-1-(4-bromophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4d)



syn-**3d** *syn*-**4d**

To a solution of *syn*-**3d** (30.0 mg, 0.08 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.4 mL) was slowly added zinc powder (109.8 mg, 1.68 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (15 mL x 3) and washed with brine (15 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.43 for *syn*-**4d** in EtOAc) to afford the product *syn*-**4d** (13.0 mg, 55% yield) as a light yellow oil. Selected spectroscopic data of *syn*-**4d**: $[\alpha]_D^{27}$ –53 (*c* 1.09, CH_2Cl_2); IR (neat): 2970, 2881, 1690, 1490, 1418, 1333, 1283, 1202, 1170, 1075, 1008, 908, 826 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.45 (d, J = 8.5 Hz, 2 H), 7.12 (d, J = 8.5 Hz, 2 H), 3.90 – 3.85 (m, 1 H), 3.63 – 3.58 (m, 1 H), 3.25 – 3.20 (m, 1 H), 3.13 – 3.08 (m, 1 H), 2.91 (dd, J = 16.5, 11.5 Hz, 1 H), 2.79 (dd, J = 16.0, 8.5 Hz, 1 H), 2.17 – 1.99 (m, 3 H), 1.58 – 1.50 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.1 (C), 139.8 (C), 132.2 (2 CH), 129.0 (2 CH), 121.2 (C), 68.8 (CH), 48.3 (CH), 43.3 (CH_2), 41.7 (CH_2), 31.6 (CH_2), 27.2 (CH_2); MS (*m/z*, relative intensity): 281 (M^++2, 41), 279 (M^+, 41), 184 (96), 182 (100), 172 (8), 130 (6), 115 (8), 103 (24), 102 (16), 77 (15), 69 (64); Exact mass calculated for $C_{13}H_{14}ONBr$ (M^+) 279.0259; found: 279.0260.

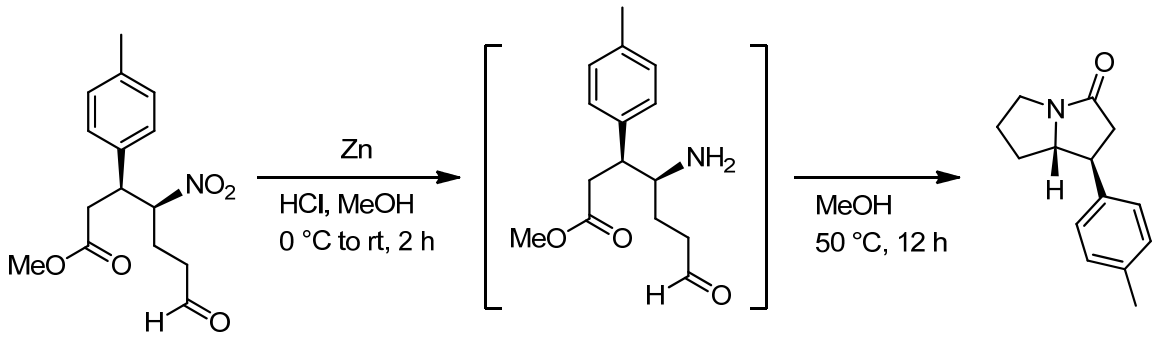
Preparation of (1*S*,7a*R*)-1-(4-bromophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4d)



anti-**3d** *anti*-**4d**

To a solution of *anti*-**3d** (10.0 mg, 0.03 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.15 mL) was slowly added zinc powder (36.4 mg, 0.56 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (15 mL x 3) and washed with brine (15 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.42 for *anti*-**4d** in EtOAc) to afford the product *anti*-**4d** (4.6 mg, 59% yield) as a light yellow oil. Selected spectroscopic data of *anti*-**4d**: $[\alpha]_D^{27}$ 157.8 (*c* 0.14, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 (d, J = 8.5 Hz, 2H), 6.96 (d, J = 8.5 Hz, 2H), 4.26 – 4.22 (m, 1H), 3.65 – 3.60 (m, 1H), 3.51 – 3.45 (m, 1H), 3.17 (dd, J = 17.0, 8.5 Hz, 1H), 3.08 – 3.03 (m, 1H), 2.58 (dd, J = 17.0, 2.5 Hz, 1H), 1.95 – 1.86 (m, 1H), 1.85 – 1.78 (m, 1H), 1.52 – 1.46 (m, 1H), 0.93 – 0.85 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.8 (C), 139.6 (C), 131.8 (2 CH), 129.4 (2 CH), 121.0 (C), 65.7 (CH), 41.6 (CH_2), 41.40 (CH_2), 41.39 (CH), 26.8 (CH_2), 26.3 (CH_2).

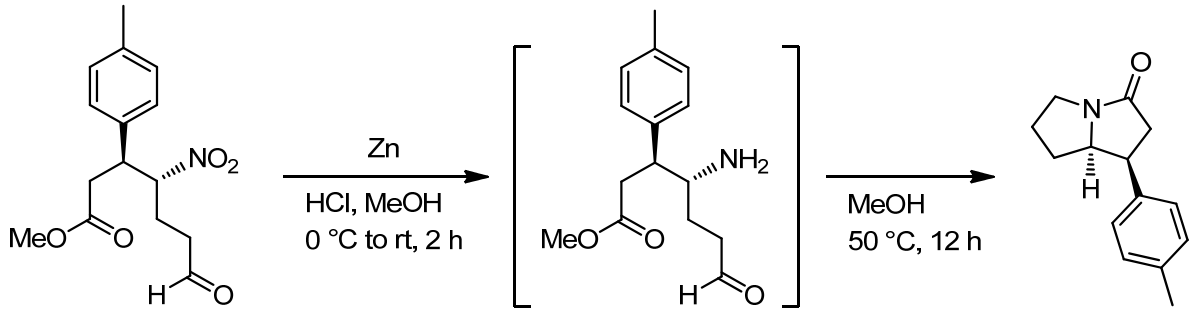
Preparation of (1*S*,7a*S*)-1-(*p*-tolyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4e)



syn-**3e** *syn*-**4e**

To a solution of *syn*-**3e** (30.0 mg, 0.1 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.5 mL) was slowly added zinc powder (133.4 mg, 2.0 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (15 mL x 3) and washed with brine (15 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.51 for *syn*-**4e** in EtOAc) to afford the product *syn*-**4e** (13.8 mg, 63% yield) as a light yellow oil. Selected spectroscopic data of *syn*-**4e**: $[\alpha]_D^{25}$ –49.5 (*c* 1.39, CH_2Cl_2); IR (neat): 2967, 2876, 1690, 1515, 1412, 1259, 1090, 1020, 819, 666, 578, 528 cm^{-1}; ^1H NMR (500 MHz,$CDCl_3$): δ 7.15 –7.12 (m, 4 H), 3.93 –3.88 (m, 1 H), 3.63 – 3.57 (m, 1 H), 3.26 – 3.20 (m, 1 H), 3.12 – 3.07 (m, 1 H), 2.94 (dd, *J* = 16.0, 11.5 Hz, 1 H), 2.77 (dd, *J* = 16.0, 8.5 Hz, 1 H), 2.32 (s, 3 H), 2.15 – 1.98 (m, 3 H), 1.56 – 1.49 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.2 (C), 137.5 (C), 136.8 (C), 129.4 (2CH), 126.9 (2CH), 68.7 (CH), 48.3 (CH), 43.2 (CH_2), 41.3 (CH_2), 31.3 (CH_2), 27.0 (CH_2), 21.0 (CH_3); MS (*m/z*, relative intensity): 215 (M^+, 51), 144 (3), 119 (9), 118 (100), 117 (24), 91 (8), 70 (17); exact mass calculated for $C_{14}H_{17}ON$ (M^+) 215.1310; found: 215.1313.

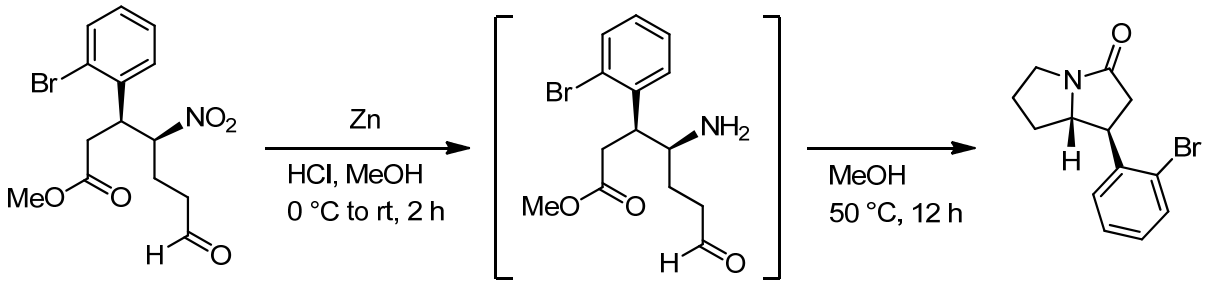
Preparation of (1*S*,7a*R*)-1-(*p*-tolyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4e)



anti-**3e** *anti*-**4e**

To a solution of *anti*-**3e** (13.0 mg, 0.04 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL) was slowly added zinc powder (57.5 mg, 0.88 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.50 for *anti*-**4e** in EtOAc) to afford the product *anti*-**4e** (5.2 mg, 55% yield) as a light yellow oil. Selected spectroscopic data of *anti*-**4e**: $[\alpha]_D^{25}$ 101.6 (*c* 0.16, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.10 (d, *J* = 8.0 Hz, 2 H), 6.96 (d, *J* = 8.0 Hz, 2 H), 4.25 – 4.20 (m, 1 H), 3.65 – 3.61 (m, 1 H), 3.51 – 3.46 (m, 1 H), 3.15 (dd, *J* = 17.0, 8.5 Hz, 1 H), 3.08 – 3.03 (m, 1 H), 2.62 (dd, *J* = 17.0, 2.5 Hz, 1 H), 2.31 (s, 3 H), 1.92 – 1.86 (m, 1 H), 1.84 – 1.77 (m, 1 H) 1.50 – 1.44 (m, 1 H), 0.96 – 0.90 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4 (C), 137.5 (C), 136.7 (C), 129.3 (2 CH), 127.6 (2 CH), 66.1 (CH), 41.8 (CH), 41.5 (2 CH_2), 26.8 (CH_2), 26.4 (CH_2), 21.0 (CH_3).

Preparation of (1*S*,7a*S*)-1-(2-bromophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4f)

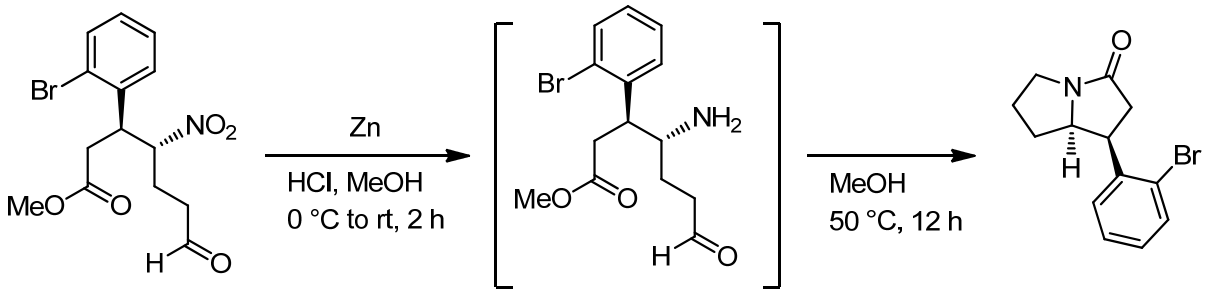


syn-**3f** *syn*-**4f**

To a solution of *syn*-**3f** (17.0 mg, 0.05 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.25 mL) was slowly added zinc powder (61.5 mg, 0.94 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *syn*-**4f** in EtOAc) to afford the product *syn*-**4f** (9.0 mg, 68% yield) as a colorless oil.

Selected spectroscopic data of *syn*-**4f**: $[\alpha]_D^{28}$ –18.5 (*c* 0.77, CH_2Cl_2); IR (neat): 2926, 2876, 1685, 1473, 1439, 1419, 1333, 1286, 1200, 1022, 904, 765, 666 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.56 (d, J = 8.0 Hz, 1H), 7.40 (d, J = 8.0 Hz, 1H), 7.33 – 7.30 (m, 1H), 7.11 – 7.08 (m, 1H), 3.96 – 3.92 (m, 1H), 3.87 – 3.81 (m, 1H), 3.65 – 3.59 (m, 1H), 3.13 – 3.08 (m, 1H), 2.87 (d, J = 9.5 Hz, 2H), 2.18 – 2.10 (m, 1H), 2.07 – 1.95 (m, 2H), 1.61 – 1.53 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.2 (C), 140.0 (C), 133.3 (CH), 128.5 (CH), 128.0 (CH), 127.7 (CH), 124.7 (C), 68.4 (CH), 46.2 (CH), 42.8 (CH_2), 41.4 (CH_2), 31.2 (CH_2), 26.8 (CH_2); MS (*m/z*, relative intensity): 281 (M^++2, 12), 279 (M^+, 12), 265 (2), 204 (4), 202 (4), 184 (22), 182 (22), 103 (18), 70 (100); exact mass calculated for $C_{13}H_{14}ONBr$ (M^+) 279.0259; found: 279.0262.

Preparation of (1*S*,7a*R*)-1-(2-bromophenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4f)

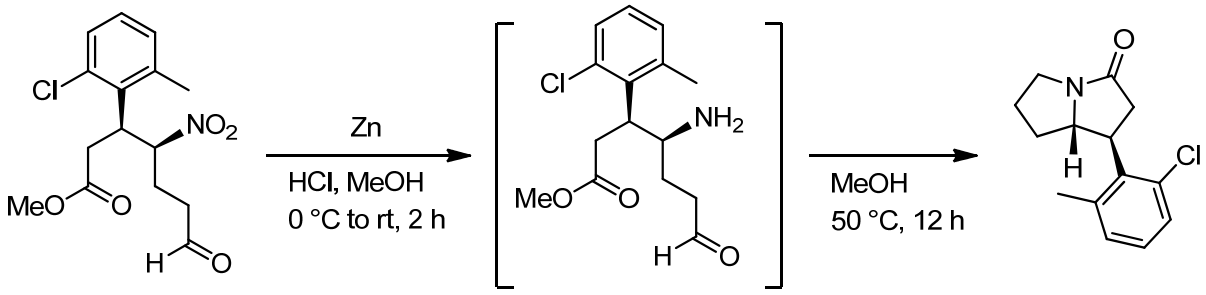


anti-**3f** *anti*-**4f**

To a solution of *anti*-**3f** (12.0 mg, 0.03 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.15 mL) was slowly added zinc powder (44.2 mg, 0.68 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *anti*-**4f** in EtOAc) to afford the product *anti*-**4f** (5.0 mg, 53% yield) as a colorless oil.
Selected spectroscopic data of *anti*-**4f**: $[\alpha]_D^{26}$ 209.2 (*c* 0.38, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57 (dd, J = 8.0, 1.5 Hz, 1H), 7.30 – 7.27 (m, 1H), 7.14 – 7.09 (m, 2H), 4.37 – 4.33 (m, 1H), 4.18 – 4.15 (m, 1H), 3.43 – 3.38 (m, 1H), 3.22 – 3.17 (m, 1H), 3.09 – 3.04 (m, 1H), 2.65 (dd, J = 17.0, 2.0 Hz, 1H), 1.97 – 1.87 (m, 1H), 1.83 – 1.76 (m, 1H), 1.71 – 1.65 (m, 1H), 0.86 – 0.73 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.5 (C), 139.7 (C), 132.9 (CH), 128.6 (CH), 128.0 (2 CH), 125.4 (C), 65.3 (CH), 41.2 (2 CH_2), 40.8 (CH), 26.9 (CH_2), 26.3 (CH_2).[18]

[18] Zoute, L.; Kociok-Koehn, G.; Frost, C. G. *Org. Lett.* **2009**, *11*, 2491 – 2494.

Preparation of
(1*S*,7a*S*)-1-(2-chloro-6-methylphenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4g)



syn-**3g** *syn*-**4g**

To a solution of *syn*-**3g** (15.0 mg, 0.05 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.25 mL) was slowly added zinc powder (60.2 mg, 0.92 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *syn*-**4g** in EtOAc) to afford the product *syn*-**4g** (7.5 mg, 66% yield) as a white solid.
Selected data of *syn*-**4g**: mp: 118–120 °C; $[\alpha]_D^{25}$ –28.7 (c 0.43, CH_2Cl_2); IR (neat): 2964, 2881, 1688, 1454, 1412, 1284, 1211, 1122, 850, 777, 734, 680 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.24 – 7.21 (m, 1 H), 7.08 – 7.05 (m, 2 H), 4.32 – 4.27 (m, 1 H), 3.89 – 3.83 (m, 1 H), 3.66 – 3.60 (m, 1 H), 3.44 (dd, J = 17.0, 10.5 Hz, 1 H), 3.16 – 3.11 (m, 1 H), 2.70 (dd, J = 16.5, 10.0 Hz, 1 H), 2.38 (s, 3 H), 2.19 – 2.12 (m, 1 H), 2.06 – 1.97 (m, 2 H), 1.45 – 1.36 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.9 (C), 138.7 (C), 135.6 (C), 134.3 (C), 129.7 (CH), 129.1 (CH), 127.9 (CH), 65.8 (CH), 41.5 (CH), 41.3 (CH_2), 40.1(CH_2), 32.7(CH_2), 26.7 (CH_2), 21.2 (CH_3); MS (*m/z*, relative intensity): 251 (M^++2, 0.7), 249 (M^+, 2), 186 (1), 152 (18), 117 (15), 115 (11), 70 (100), 58 (29); exact mass calculated for $C_{14}H_{16}ONCl$ (M^+) 249.0920; found: 249.0921.

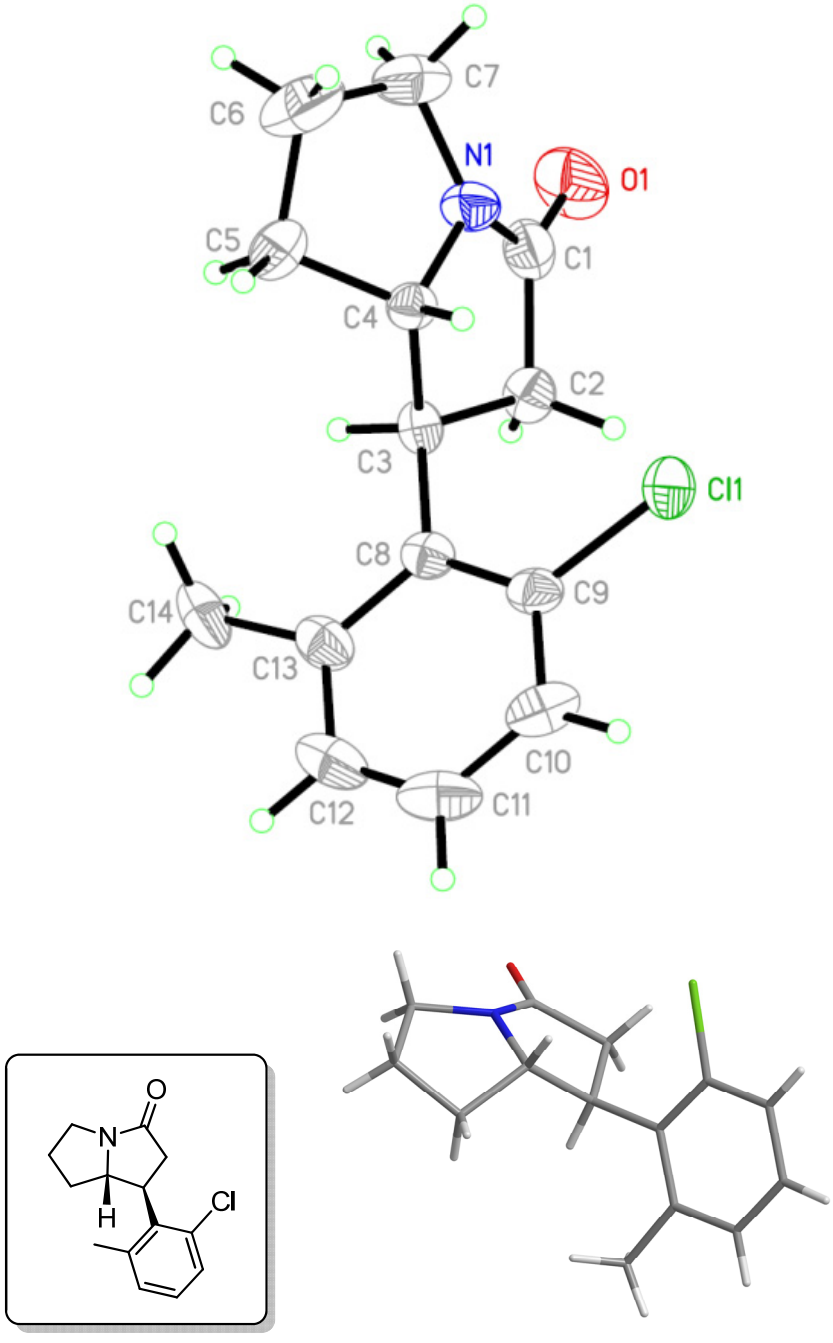
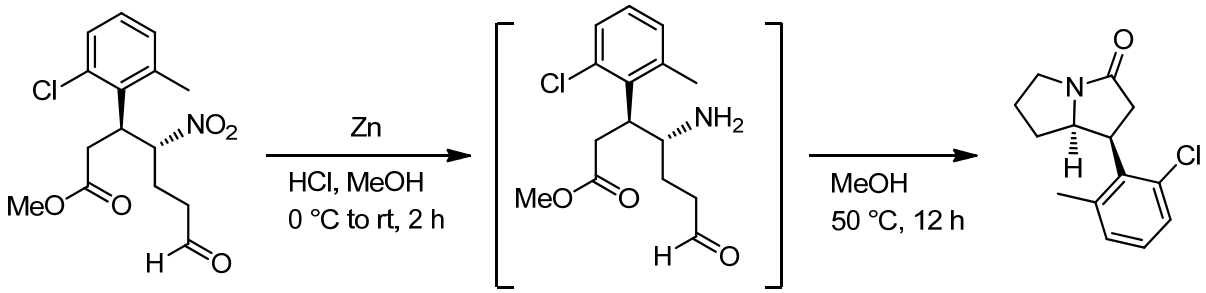


Figure S1. ORTEP and Stereo plots for X-ray crystal structures of (–)-*syn*-**4g**.

CCDC-1420368 contains the supplementary crystallographic data for (–)-*syn*-**4g**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

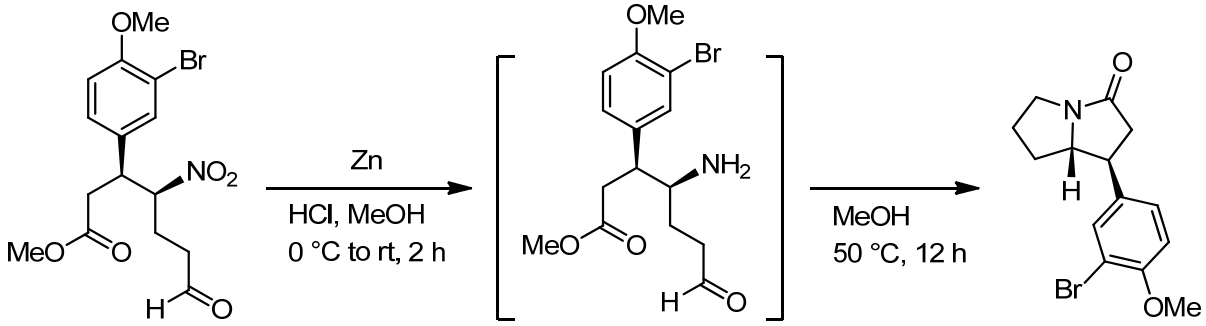
Preparation of
(1*S*,7a*R*)-1-(2-chloro-6-methylphenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4g)



anti-**3g** *anti*-**4g**

To a solution of *anti*-**3g** (26.0 mg, 0.08 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.4 mL) was slowly added zinc powder (104.0 mg, 1.6 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.28 for *anti*-**4g** in EtOAc) to afford the product *anti*-**4g** (13.1 mg, 66% yield) as a white solid.
Selected data of *anti*-**4g**: mp: 93–94 °C; $[\alpha]_D^{25}$ 139.4 (*c* 0.56, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.22 – 7.19 (m, 1 H), 7.06 – 7.02 (m, 2 H), 4.34 – 4.29 (m, 2 H), 3.58 – 3.52 (m, 1 H), 3.19 – 3.12 (m, 2 H), 2.97 (d, J = 17.5 Hz, 1 H), 2.33 (s, 3 H), 2.16 – 2.10 (m, 1 H), 2.07 – 1.96 (m, 1 H), 1.44 – 1.39 (m, 1 H), 1.27 – 1.20 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.2 (C), 138.8 (C), 136.9 (C), 134.6 (C), 129.7 (CH), 128.9 (CH), 127.7 (CH), 64.4 (CH), 41.4 (CH_2), 40.3 (CH_2), 35.6 (CH), 26.8 (CH_2), 26.5 (CH_2), 22.0 (CH_3).

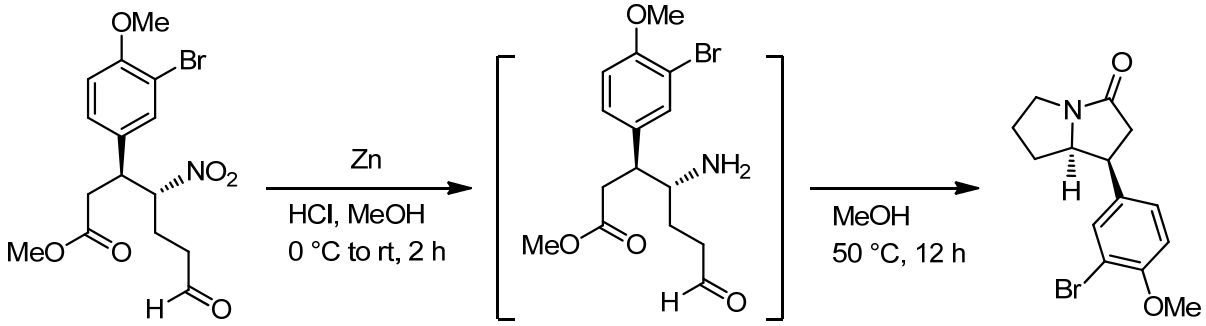
Preparation of
(1*S*,7a*S*)-1-(3-bromo-4-methoxyphenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4h)



syn-**3h** *syn*-**4h**

To a solution of *syn*-**3h** (16.0 mg, 0.04 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL) was slowly added zinc powder (53.6 mg, 0.82 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *syn*-**4h** in EtOAc) to afford the product *syn*-**4h** (7.3 mg, 57% yield) as a yellow solid.
Selected data of *syn*-**4h**: mp: 107 – 108 °C; $[\alpha]_D^{27}$ –30.6 (*c* 0.36, CH_2Cl_2); IR (neat): 2965, 2924, 2852, 1691, 1500, 1408, 1287, 1259, 1181, 1092, 1055, 1018, 881, 814 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.42 (d, J = 2.5 Hz, 1 H), 7.14 (dd, J = 8.5, 2.5 Hz, 1 H), 6.85 (d, J = 8.5 Hz, 1 H), 3.87 (s, 3 H), 3.90 – 3.85 (m, 1 H), 3.63 – 3.57 (m, 1 H), 3.21 – 3.16 (m, 1 H), 3.12 – 3.07 (m, 1 H), 2.89 (dd, J = 16.5, 12.0 Hz, 1 H), 2.77 (dd, J = 16.5, 8.5 Hz, 1 H), 2.15 – 1.99 (m, 3 H), 1.55 – 1.48 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.9 (C), 155.0 (C), 134.1 (C), 131.9 (CH), 127.0 (CH), 112.1 (CH), 112.0 (C), 68.7 (CH), 56.3 (CH_3), 47.5 (CH), 43.2 (CH_2), 41.4 (CH_2), 31.3 (CH_2), 27.0 (CH_2); MS (*m/z*, relative intensity): 309 (M^+, 2), 213 (4), 198 (7), 187 (13), 142 (8), 91 (52), 80 (63), 57 (100); exact mass calculated for $C_{14}H_{16}O_2NBr$ (M^+) 309.0364; found: 309.0361.

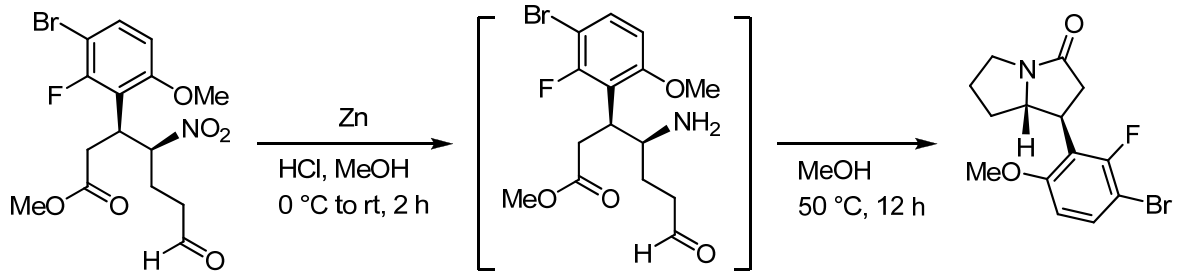
Preparation of
(1*S*,7a*R*)-1-(3-bromo-4-methoxyphenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*anti*-4h)



anti-**3h** *anti*-**4h**

To a solution of *anti*-**3h** (10.0 mg, 0.03 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.15 mL) was slowly added zinc powder (34.0 mg, 0.52 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.42 for *anti*-**4h** in EtOAc) to afford the product *anti*-**4h** (5.1 mg, 64% yield) as a yellow solid.
Selected data of *anti*-**4h**: mp: 170 ^{o}C (decomposed); $[\alpha]_D^{27}$ 127.5 (*c* 0.17, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.27 (s, 1 H), 6.99 (d, J = 8.5 Hz, 1 H), 6.82 (d, J = 8.5 Hz, 1 H), 4.23 – 4.18 (m, 1 H), 3.86 (s, 3 H), 3.61 – 3.58 (m, 1 H), 3.53 – 3.47 (m, 1 H), 3.15 (dd, J = 17.0, 8.5 Hz, 1 H), 3.08 – 3.03 (m, 1 H), 2.57 (d, J = 17.0 Hz, 1 H), 1.94 – 1.88 (m, 1 H), 1.86 – 1.82 (m, 1 H), 1.52 – 1.49 (m, 1 H), 1.00 – 0.99 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.9 (C), 154.9 (C), 134.2 (C), 132.8 (CH), 127.2 (CH), 112.0 (CH), 111.8 (C), 65.9 (CH), 56.3 (CH_3), 41.6 (CH_2), 41.4 (CH_2), 40.7 (CH), 26.8 (CH_2), 26.4 (CH_2).

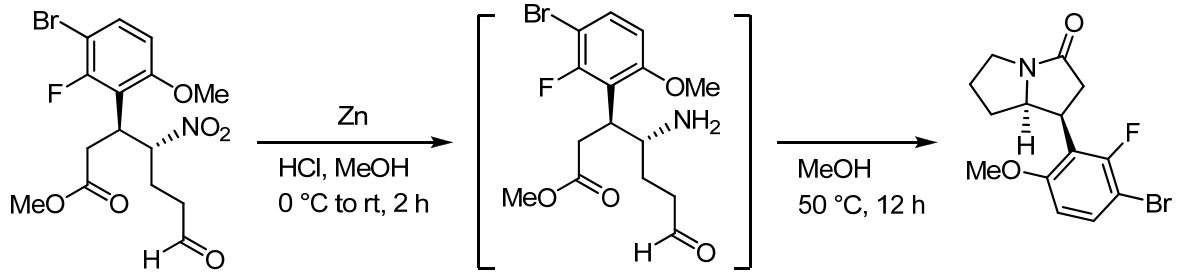
Preparation of
(1*S*,7a*S*)-1-(3-bromo-6-fluoro-2-methoxyphenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4i)



syn-**3i** *syn*-**4i**

To a solution of *syn*-**3i** (22.0 mg, 0.05 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.25 mL) was slowly added zinc powder (70.6 mg, 1.08 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *syn*-**4i** in EtOAc) to afford the product *syn*-**4i** (10.7 mg, 60% yield) as a yellow oil.
Selected data of *syn*-**4i**: $[\alpha]_D^{27}$ –33.2 (*c* 0.96, CH_2Cl_2); IR (neat): 2947, 2841, 1690, 1602, 1573, 1478, 1440, 1412, 1365, 1282, 1224, 1167, 1075, 802 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.37 (dd, J = 9.0, 8.0 Hz, 1 H), 6.59 (dd, J = 9.0, 1.0 Hz, 1 H), 4.12 – 4.09 (m, 1 H), 3.82 (s, 3 H), 3.76 – 3.70 (m, 1 H), 3.63 – 3.58 (m, 1 H), 3.28 (dd, J = 16.5, 11.5 Hz, 1 H), 3.11 – 3.07 (m, 1 H), 2.58 (dd, J = 16.5, 9.0 Hz, 1 H), 2.11 – 2.09 (m, 1 H), 2.02 – 1.93 (m, 2 H), 1.45 – 1.41 (m, 1 H); ^{13}C NMR (125 MHz,$CDCl_3$): δ 173.8 (C), 157.5 (d, J = 243.1 Hz, C), 158.0 (d, J = 6.9 Hz, C) 131.5 (d, J = 2.3 Hz, CH), 117.3 (d, J = 16.5 Hz, C), 107.9 (d, J = 3.1 Hz, CH), 100.6 (d, J = 22.9 Hz, C), 65.5 (d, J = 2.8 Hz, CH), 56.0 (CH_3), 41.5 (CH_2), 40.2 (d, J = 2.3 Hz, CH_2), 37.5 (d, J = 1.4 Hz, CH), 31.6 (CH_2), 26.8 (CH_2); MS (*m/z*, relative intensity): 329 (M^++2, 35), 327 (M^+, 36), 232 (46), 230 (49), 217 (15), 215 (15), 136 (75), 108 (16), 107 (18), 70 (100); exact mass calculated for $C_{14}H_{15}O_2NBrF$ (M^+) 327.0270; found: 327.0267.

Preparation of
(1*S*,7a*R*)-1-(3-bromo-6-fluoro-2-methoxyphenyl)tetrahydro-1*H*-pyrrolizin-3(2*H*)-one
(*anti*-4i)

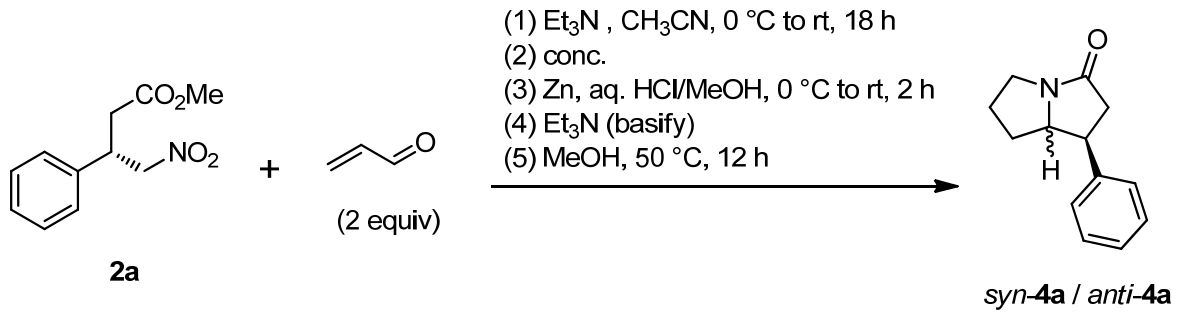


anti-**3i** *anti*-**4i**

To a solution of *anti*-**3i** (8.0 mg, 0.02 mmol) in conc. aqueous HCl–MeOH (1:1 v/v, 0.1 mL) was slowly added zinc powder (26.2 mg, 0.4 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The reaction mixture was filtered, and the filtrate was neutralized to *ca.* pH = 8 by the addition of $NaHCO_3$. The solution was extracted with EtOAc (10 mL x 3) and washed with brine (10 mL). The organic extract was dried over $MgSO_4$ and concentrated *in vacuo* to give a yellow oil residue. A solution of the residue in MeOH (1.0 mL) was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane (R_f = 0.48 for *anti*-**4i** in EtOAc) to afford the product *anti*-**4i** (3.8 mg, 59% yield) as a yellow oil.

Selected data of *anti*-**4i**: $[\alpha]_D^{27}$ 39.4 (*c* 0.3, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (d, J = 9.0 Hz, 1 H), 6.57 (d, J = 9.0 Hz, 1 H), 4.28 – 4.21 (m, 2 H), 3.77 (s, 3 H), 3.53 – 3.47 (m, 1 H), 3.15 – 3.08 (m, 2 H), 2.57 (d, J = 16.5 Hz, 1 H), 2.06 – 1.93 (m, 2 H), 1.54 – 1.48 (m, 1 H), 1.08 – 1.00 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.71 (C), 158.2 (C), 157.1 (d, J = 243 Hz, C), 131.6 (d, J = 2.4 Hz, CH), 118.6 (C), 107.9 (d, J = 3.1 Hz, CH), 100.6 (d, J = 23.9 Hz, C), 64.3 (CH), 56.0 (CH_3), 41.5 (CH_2), 40.8 (d, J = 2.4 Hz, CH_2), 31.6 (d, J = 3.3 Hz, CH), 27.0 (CH_2), 26.8 (CH_2).

One-pot Synthesis of tetrahydro-1*H*-pyrrolizin-3(2*H*)-one (*syn*-4a and *anti*-4a)

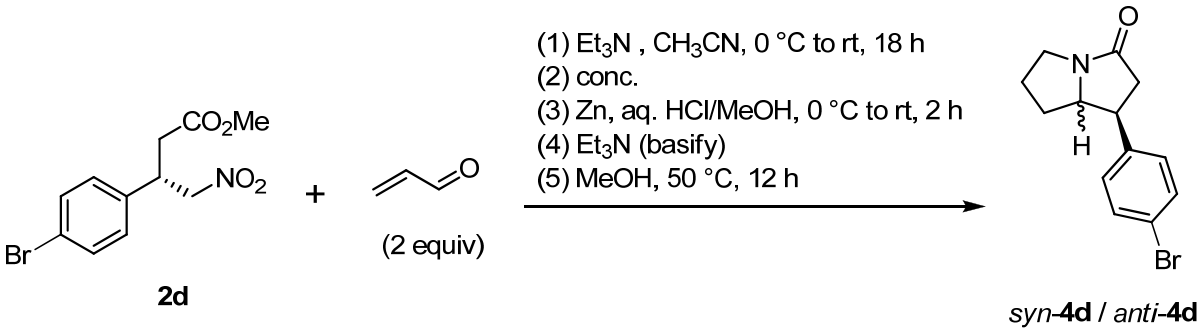


2a

syn-**4a** / *anti*-**4a**

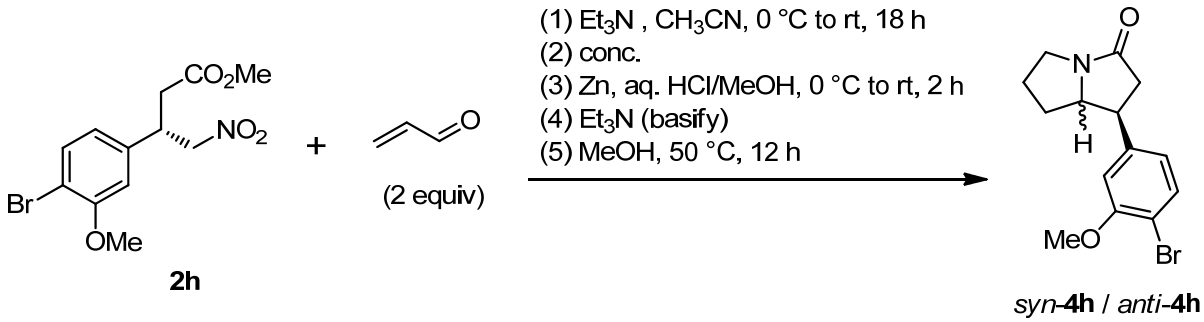
To a solution of **2a** (22.3 mg, 0.1 mmol) and Et_3N (10.1 mg, 0.1 mmol, 1 equiv) in CH_3CN (0.9 mL) was added a solution of acrylaldehyde (11.2 mg, 0.2 mmol, 2.0 equiv) in CH_3CN (0.1 mL) at 0 °C. The resulting solution was stirred at room temperature for 18 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. To the crude product was added a solution of conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL). To the solution was slowly added zinc powder (130.6 mg, 2.0 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The solution was cooled to 0 °C and was basified by the addition of Et_3N. To the solution was added MeOH (1.0 mL), and the mixture was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane to afford the product *syn*-**4a** and *anti*-**4a** (11.5 mg, 57% yield) as a colorless oil. Further separation by HPLC [ZORBAX SIL column 4.6 mm x 250 mm, elute: *n*-hexane/*i*-PrOH/MeOH = 50:40:10, detector: 254 nm, flow rate: 0.5 mL/min), t1 = 10.77 min (*syn*-**4a**), t2 = 13.17 min (*anti*-**4a**) or ZORBAX SIL column 9.4 mm x 250 mm, elute: *n*-hexane/*i*-PrOH/MeOH = 50:40:10, detector: 254 nm, flow rate: 4.0 mL/min), t1 = 6.09 min (*syn*-**4a**), t2 = 7.95 min (*anti*-**4a**)] gave 7.8 mg of *syn*-**4a** (39% yield) and 2.4 mg of *anti*-**4a** (12% yield).

One-pot Synthesis of tetrahydro-1*H*-pyrrolizin-3(2*H*)-one syn-4d and anti-4d



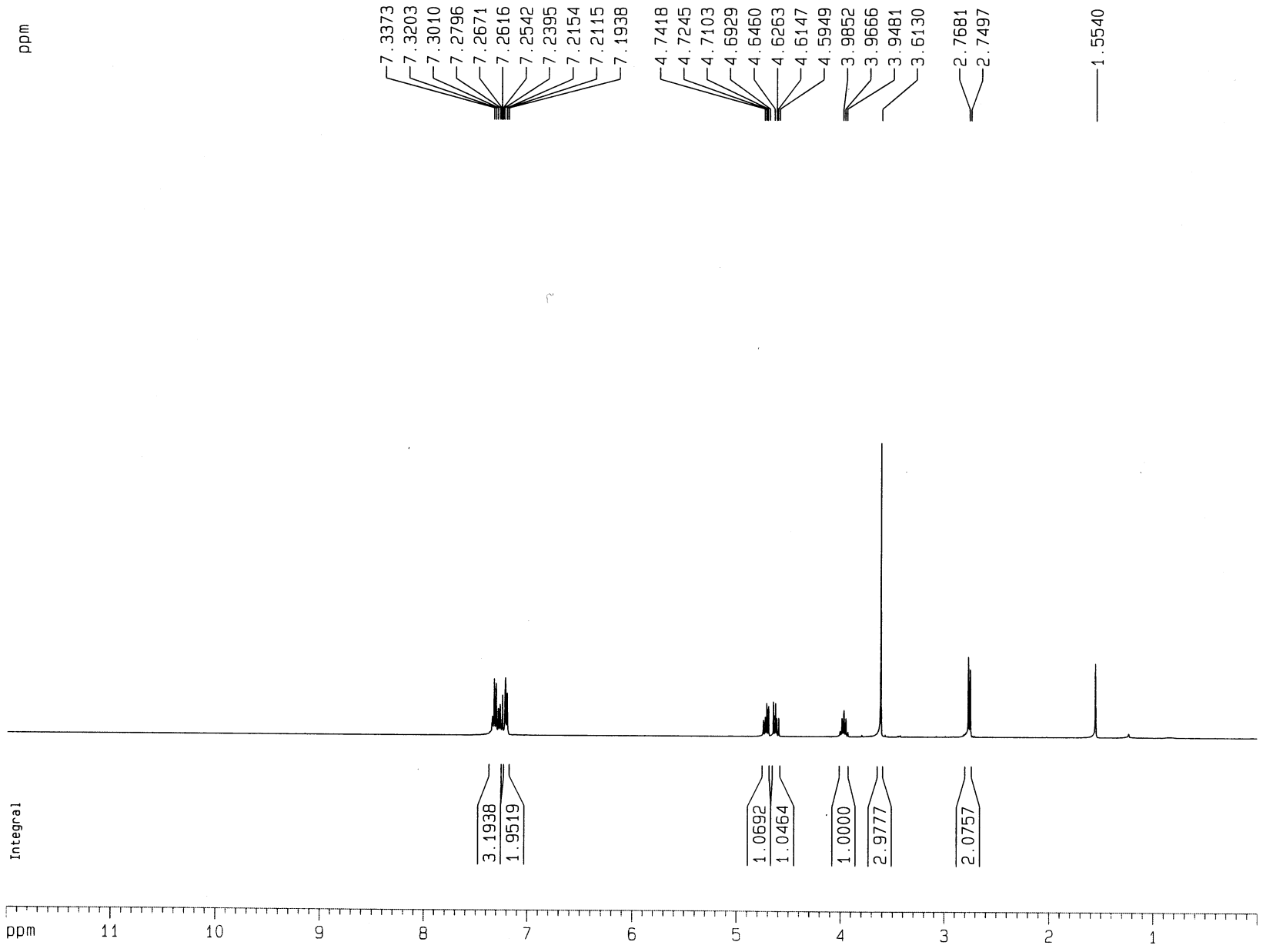
To a solution of **2d** (30.2 mg, 0.1 mmol) and Et_3N (10.1 mg, 0.1 mmol, 1 equiv) in CH_3CN (0.9 mL) was added a solution of acrylaldehyde (11.2 mg, 0.2 mmol, 2.0 equiv) in CH_3CN (0.1 mL) at 0 °C. The resulting solution was stirred at room temperature for 18 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. To the crude product was added a solution of conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL). To the solution was slowly added zinc powder (130.6 mg, 2.0 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The solution was cooled to 0 °C and was basified by the addition of Et_3N. To the solution was added MeOH (1.0 mL), and the mixture was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane to afford the product *syn*-**4d** and *anti*-**4d** (15.8 mg, 56% yield) as a colorless oil.

One-pot Synthesis of tetrahydro-1*H*-pyrrolizin-3(2*H*)-one syn-4h and anti-4h



To a solution of **2h** (33.2 mg, 0.1 mmol) and Et_3N (10.1 mg, 0.1 mmol, 1 equiv) in CH_3CN (0.9 mL) was added a solution of acrylaldehyde (11.2 mg, 0.2 mmol, 2.0 equiv) in CH_3CN (0.1 mL) at 0 °C. The resulting solution was stirred at room temperature for 18 h until the completion of the reaction, as monitored by TLC. The reaction solution was concentrated *in vacuo* to give the yellow oil residue. To the crude product was added a solution of conc. aqueous HCl–MeOH (1:1 v/v, 0.2 mL). To the solution was slowly added zinc powder (130.6 mg, 2.0 mmol, 20 equiv) over three portions at 0 °C. The resulting mixture was stirred and gradually warmed up to ambient temperature over 2 h until the completion of the reaction, as monitored by TLC. The solution was cooled to 0 °C and was basified by the addition of Et_3N. To the solution was added MeOH (1.0 mL), and the mixture was heated to 50 °C for 12 h until the completion of the reaction, as monitored by TLC. The solution was concentrated *in vacuo*, and the residue was purified by flash column chromatography with 80% EtOAc–hexane to afford the product *syn*-**4h** and *anti*-**4h** (16.4 mg, 53% yield) as a colorless oil.

Fig S43. 1H NMR (CDCl3, 400 MHz) of compound 2a

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Current Data Parameters
NAME                  LCH-2-269
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141208
Time                      17.18
INSTRUM                   spect
PROBHD          5 mm QNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   5995.204 Hz
FIDRES                0.365918 Hz
AQ                    1.3664756 sec
RG                        4096
DW                      83.400 usec
DE                       6.50 usec
TE                      300.0 K
D1                    1.50000000 sec

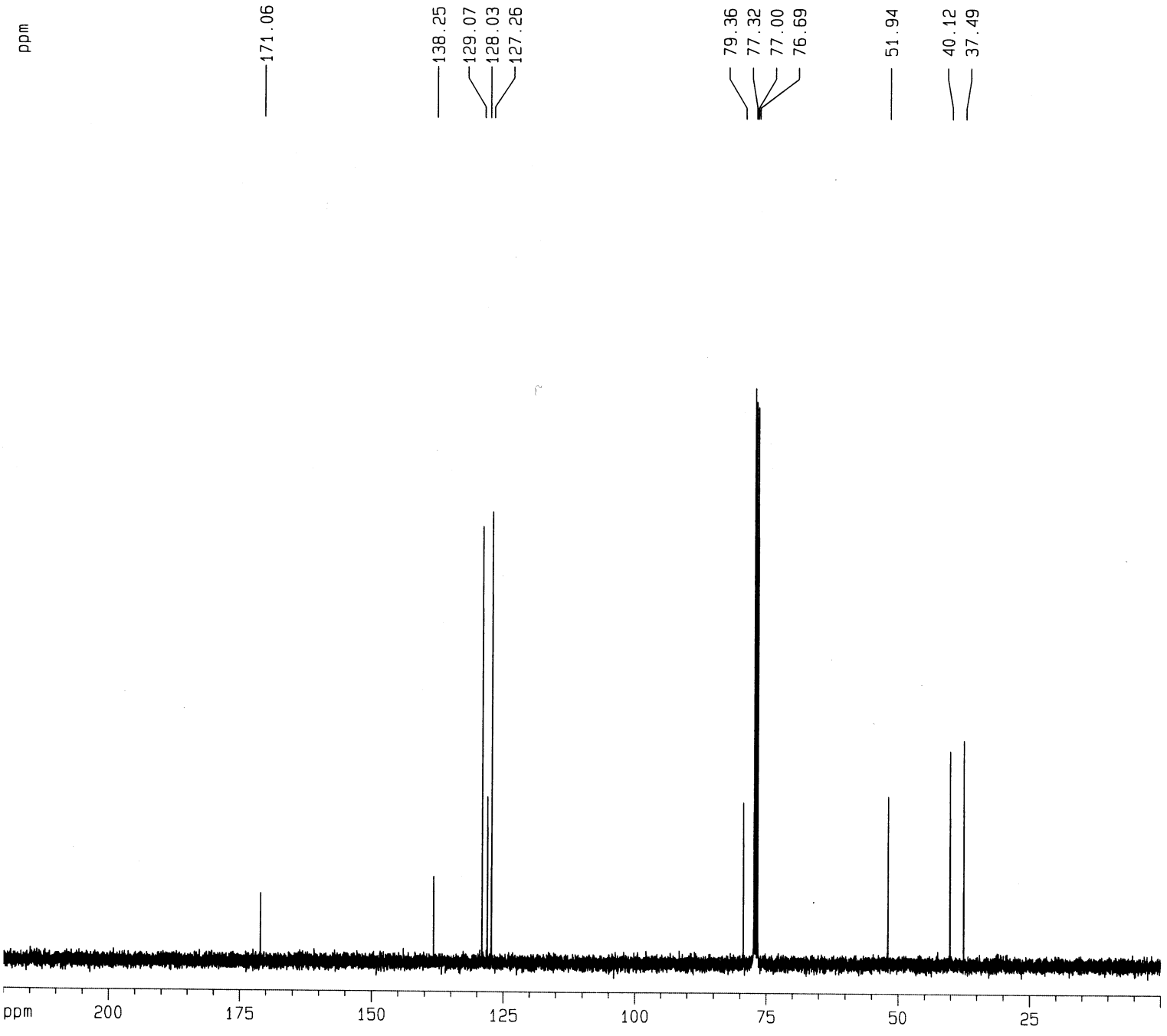
============ CHANNEL f1 =============
NUC1                         1H
P1                       11.90 usec
PL1                      -3.00 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                  400.1300179 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                     12.000 ppm
F1                     4801.56 Hz
F2P                     -0.000 ppm
F2                       -0.00 Hz
PPMCM                 0.55814 ppm/cm
HZCM                223.32837 Hz/cm
```

C13 spectrum of

Fig S44. 13C NMR (CDCl3, 100 MHz) of compound 2a




```
Current Data Parameters
NAME                  LCH-2-269
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141207
Time                      20.33
INSTRUM                   spect
PROBHD           5 mm QNP  1H
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          819
DS                            4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                          256
DW                     19.900 usec
DE                      6.50 usec
TE                      300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

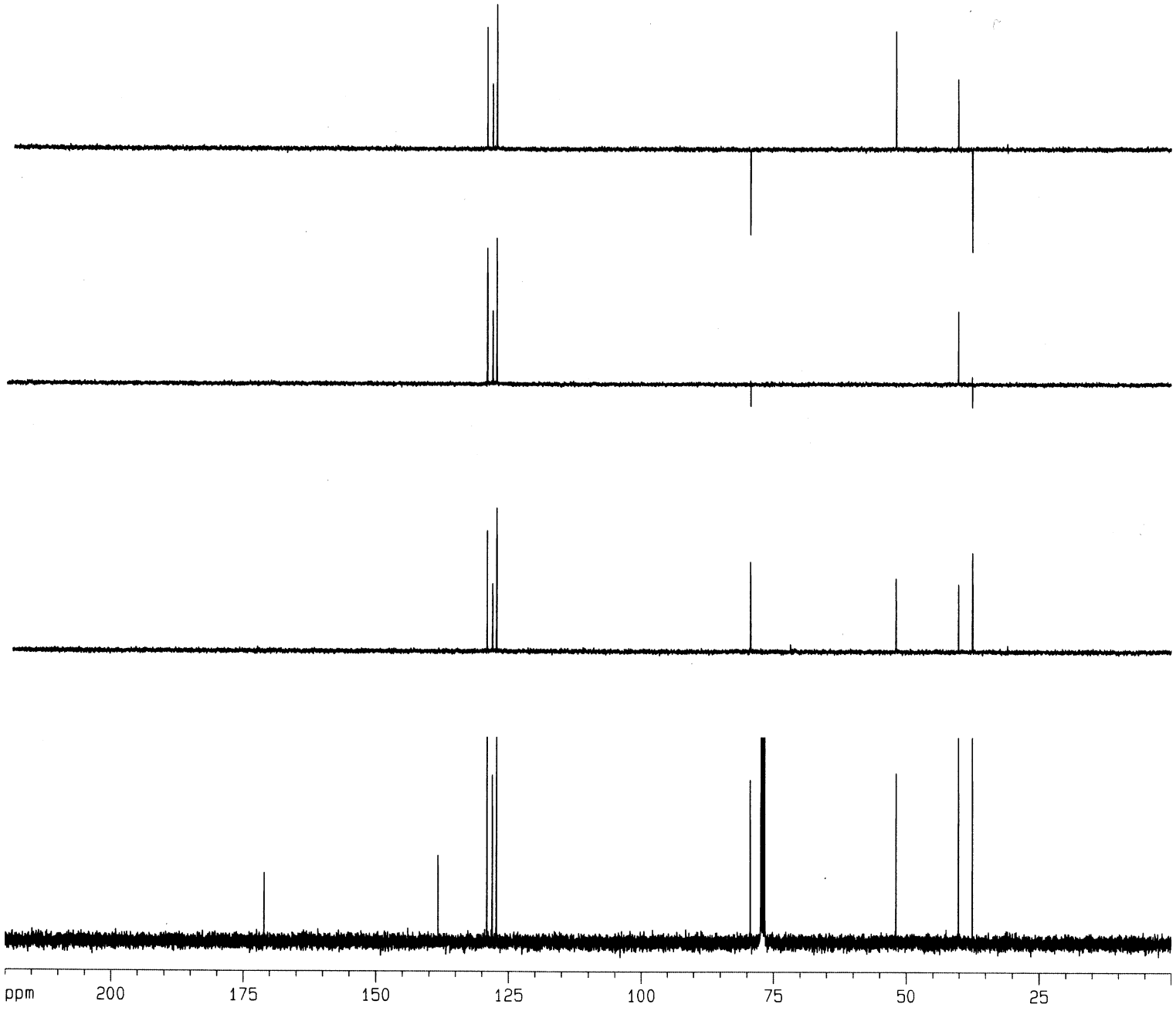
============ CHANNEL f1 =============
NUC1                        13C
P1                       10.20 usec
PL1                       0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     14.50 dB
PL13                     17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127731 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     220.000 ppm
F1                    22134.81 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                 11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S45. DEPT of compound 2a




```
Current Data Parameters
NAME          LCH-2-269
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20141207
Time              20.33
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  819
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

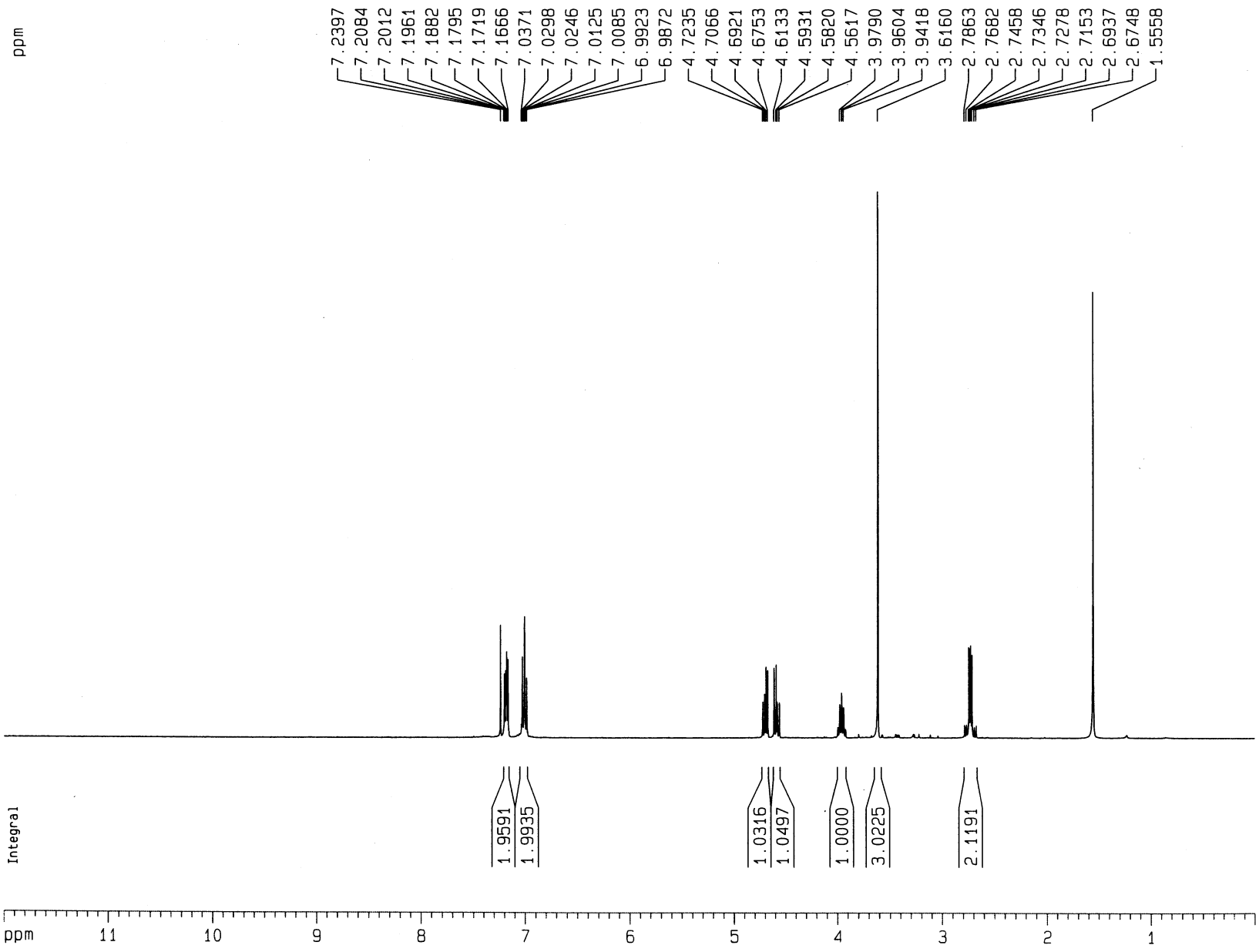
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                32768
SF           100.6127731 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```

Fig S46. 1H NMR (CDCl3, 400 MHz) of compound 2b




```
Current Data Parameters
NAME                  LCH-2-292
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141001
Time                      22.09
INSTRUM                   spect
PROBHD           5 mm GNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   5995.204 Hz
FIDRES                0.365918 Hz
AQ                    1.3664756 sec
RG                       5792.6
DW                       83.400 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.50000000 sec

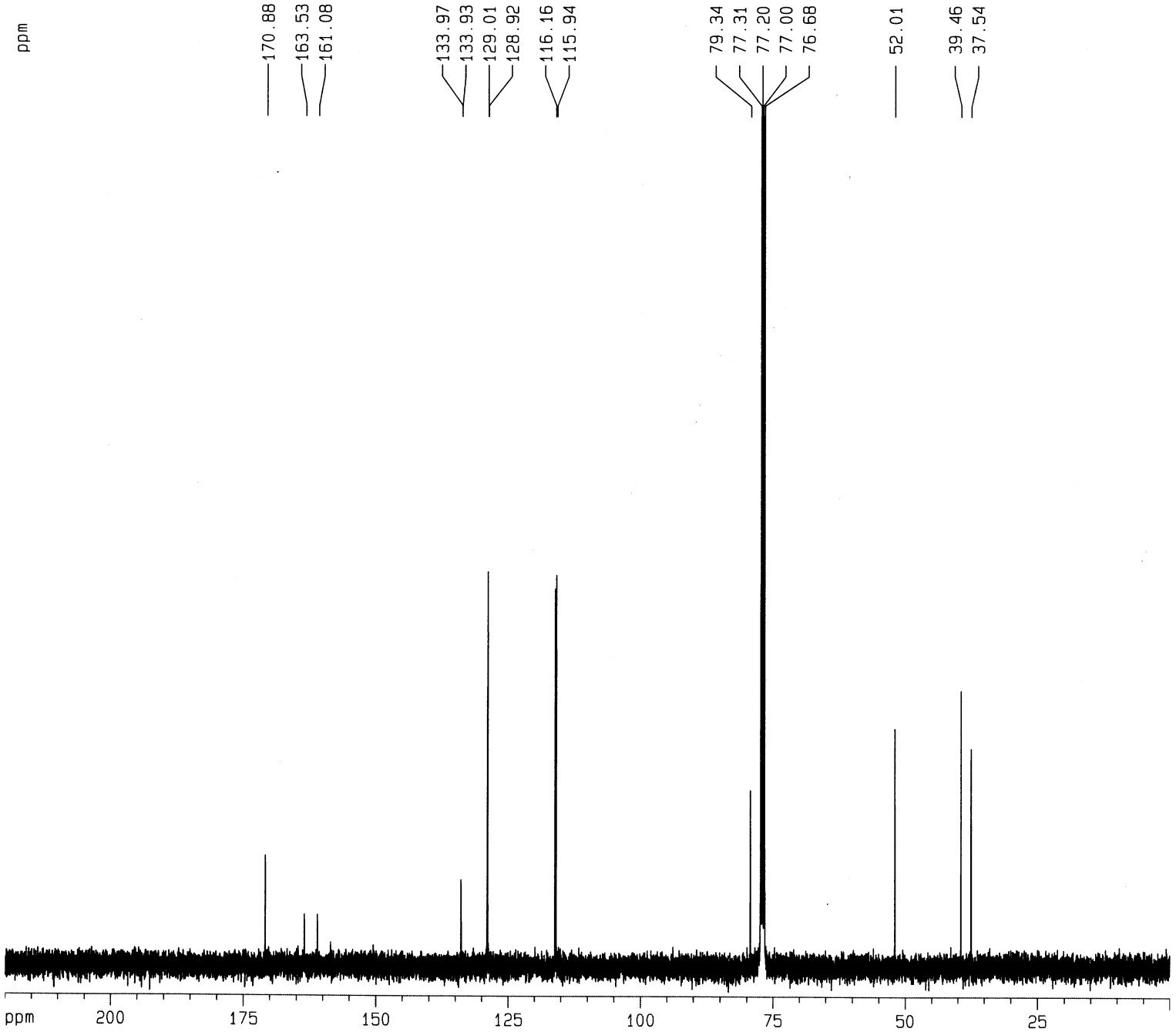
============ CHANNEL f1 =============
NUC1                         1H
P1                       11.90 usec
PL1                      -3.00 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                  400.1300179 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                      12.000 ppm
F1                      4801.56 Hz
F2P                      -0.000 ppm
F2                       -0.00 Hz
PPMCM                  0.55814 ppm/cm
HZCM                 223.32838 Hz/cm
```

C13 spectrum of

Fig S47. 13C NMR (CDCl3, 100 MHz) of compound 2b




```
Current Data Parameters
NAME                  LCH-2-292
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141003
Time                       2.43
INSTRUM                   spect
PROBHD           5 mm QNP  1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        3072
DS                           4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                         256
DW                      19.900 usec
DE                       6.50 usec
TE                      300.0 K
D1                   2.00000000 sec
d11                  0.03000000 sec
d12                  0.00002000 sec

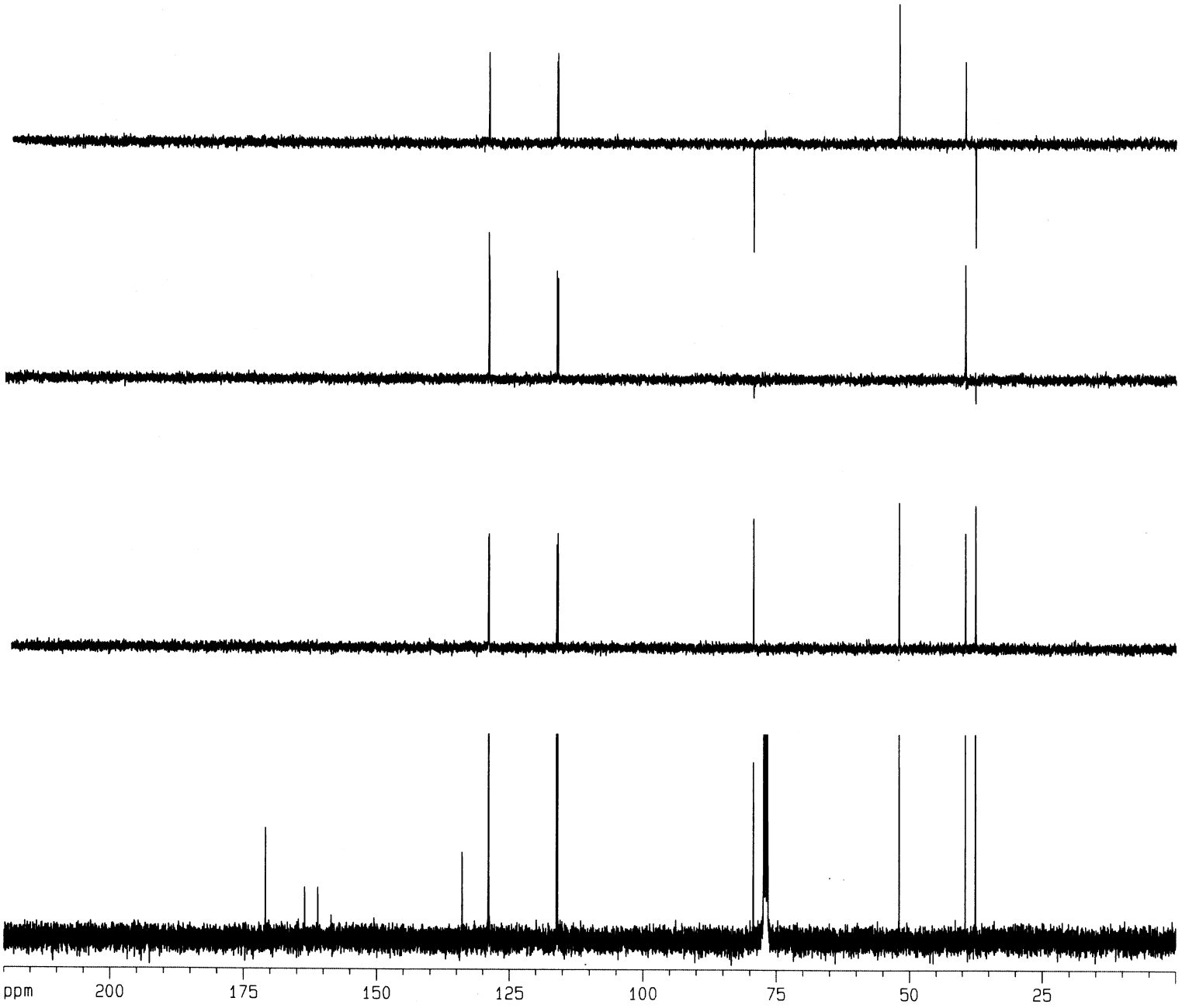
============ CHANNEL f1 =============
NUC1                      13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127715 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      220.000 ppm
F1                     22134.81 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                 11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S48. DEPT of compound 2b

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Current Data Parameters
NAME            LCH-2-292
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20141003
Time                2.43
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

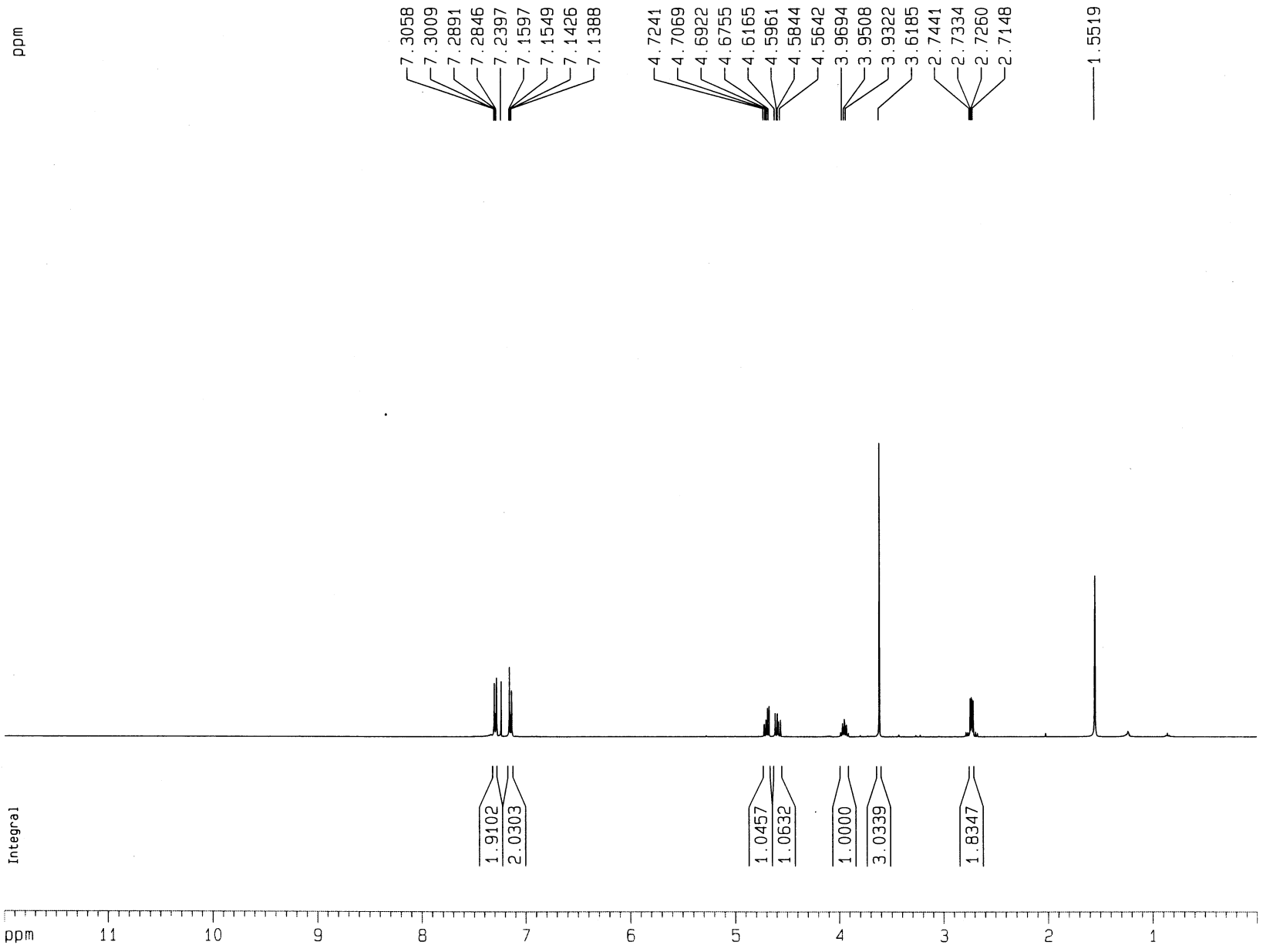
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127715 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S49. 1H NMR (CDCl3, 400 MHz) of compound 2c

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Current Data Parameters
NAME          LCH-2-288
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20141010
Time               0.12
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG           zg30
TD               16384
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                6502
DW              83.400 usec
DE               6.50 usec
TE              300.0 K
D1           1.50000000 sec

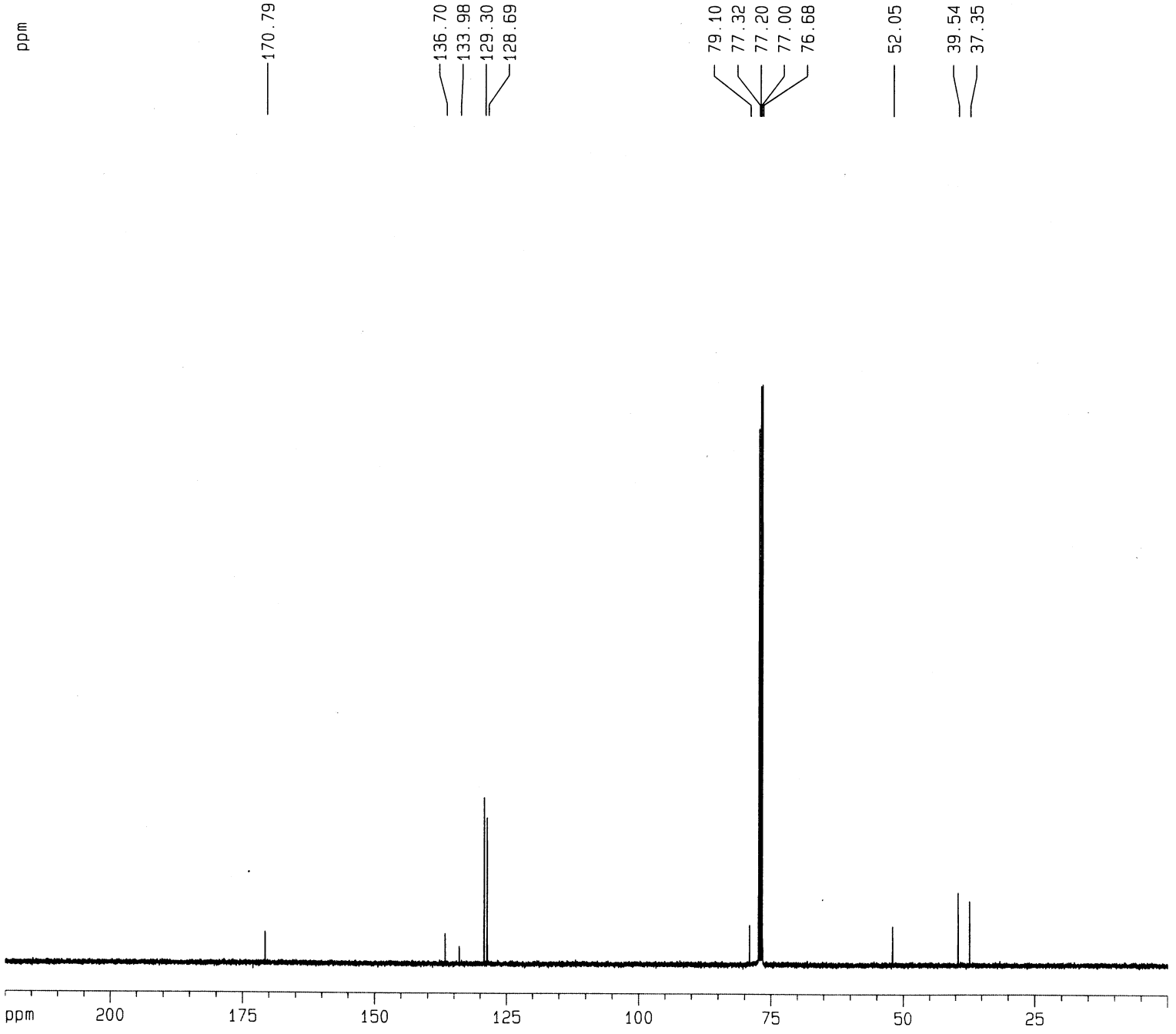
============ CHANNEL f1 =============
NUC1                1H
P1              11.90 usec
PL1             -3.00 dB
SFO1       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                 EM
SSB                  0
LB              0.10 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX              21.50 cm
F1P            12.000 ppm
F1             4801.56 Hz
F2P            -0.000 ppm
F2              -0.00 Hz
PPMCM         0.55814 ppm/cm
HZCM        223.32837 Hz/cm
```

C13 spectrum of

Fig S50. 13C NMR (CDCl3, 100 MHz) of compound 2c




```
Current Data Parameters
NAME            LCH-2-288
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20141010
Time                3.02
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

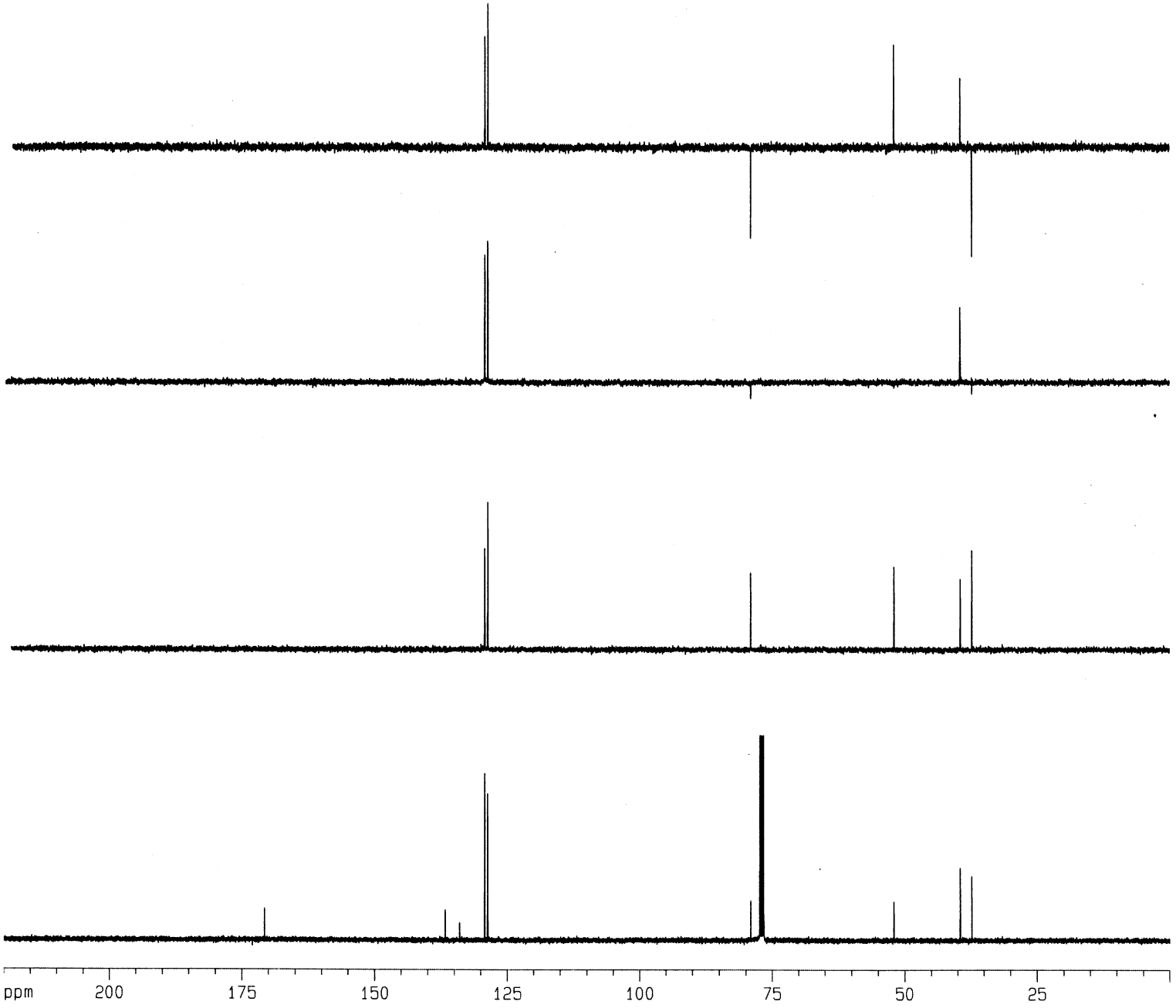
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127715 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```

Fig S51. DEPT of compound 2c

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Current Data Parameters
NAME          LCH-2-288
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
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Time               3.02
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

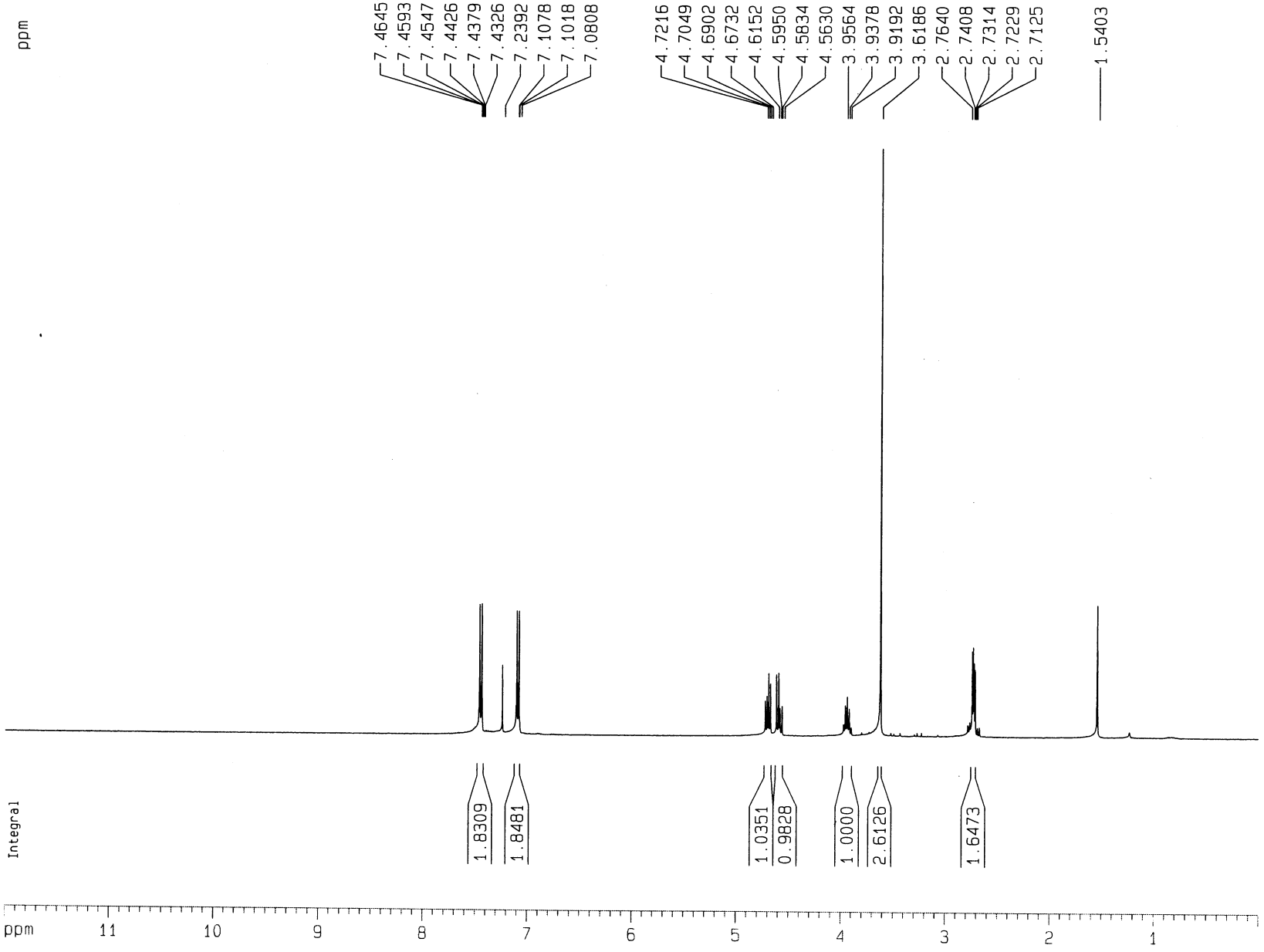
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127715 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S52. 1H NMR (CDCl3, 400 MHz) of compound 2d

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Current Data Parameters
NAME                  LCH-2-342
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141215
Time                      18.07
INSTRUM                   spect
PROBHD          5 mm QNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                5995.204 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                        6502
DW                      83.400 usec
DE                       6.50 usec
TE                      300.0 K
D1                 1.50000000 sec

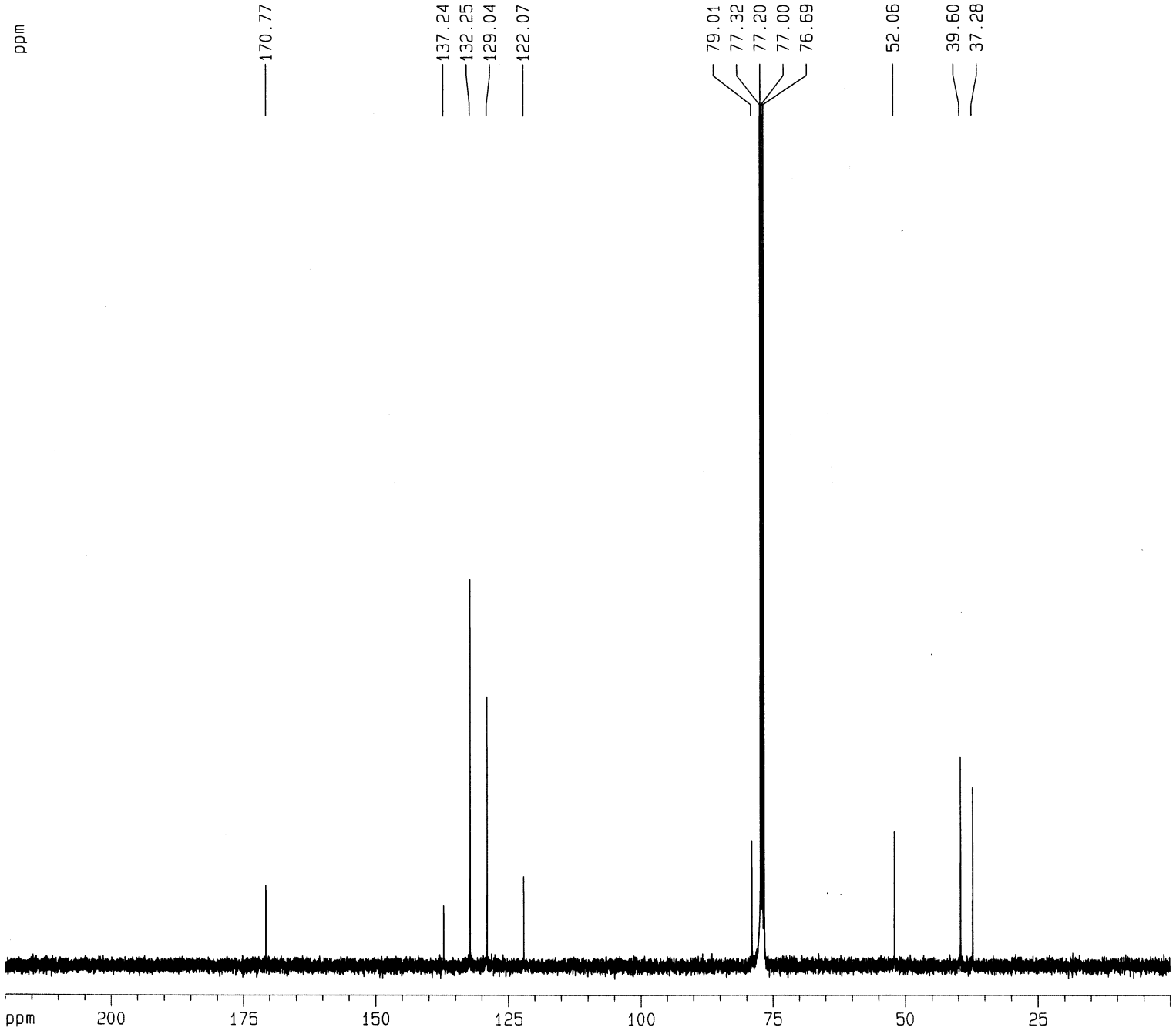
============ CHANNEL f1 =============
NUC1                         1H
P1                       11.90 usec
PL1                      -3.00 dB
SFO1               400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                 400.1300179 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                     12.000 ppm
F1                     4801.56 Hz
F2P                    -0.000 ppm
F2                      -0.00 Hz
PPMCM                  0.55814 ppm/cm
HZCM                 223.32837 Hz/cm
```

C13 spectrum of

Fig S53. 13C NMR (CDCl3, 100 MHz) of compound 2d




```
Current Data Parameters
NAME            LCH-2-342
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20141216
Time                2.29
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

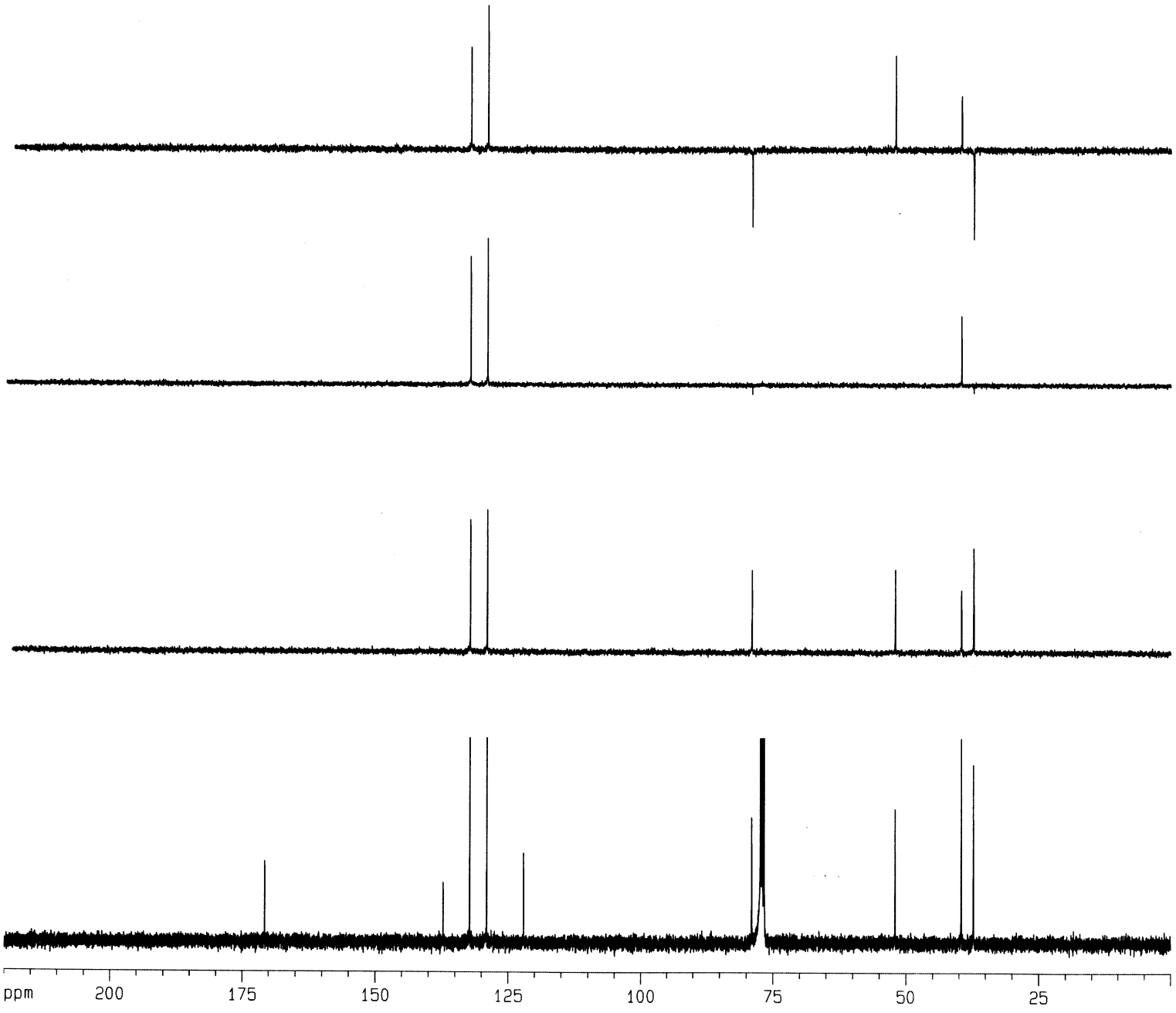
============ CHANNEL f1 =============
NUC1                 13C
P1                 10.20 usec
PL1                 0.00 dB
SF01         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -3.00 dB
PL12               14.50 dB
PL13               17.50 dB
SF02         400.1326008 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127715 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
F1P                220.000 ppm
F1               22134.81 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM           11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```

Fig S54. DEPT of compound 2d




```
Current Data Parameters
NAME          LCH-2-342
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20141216
Time               2.29
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3276
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

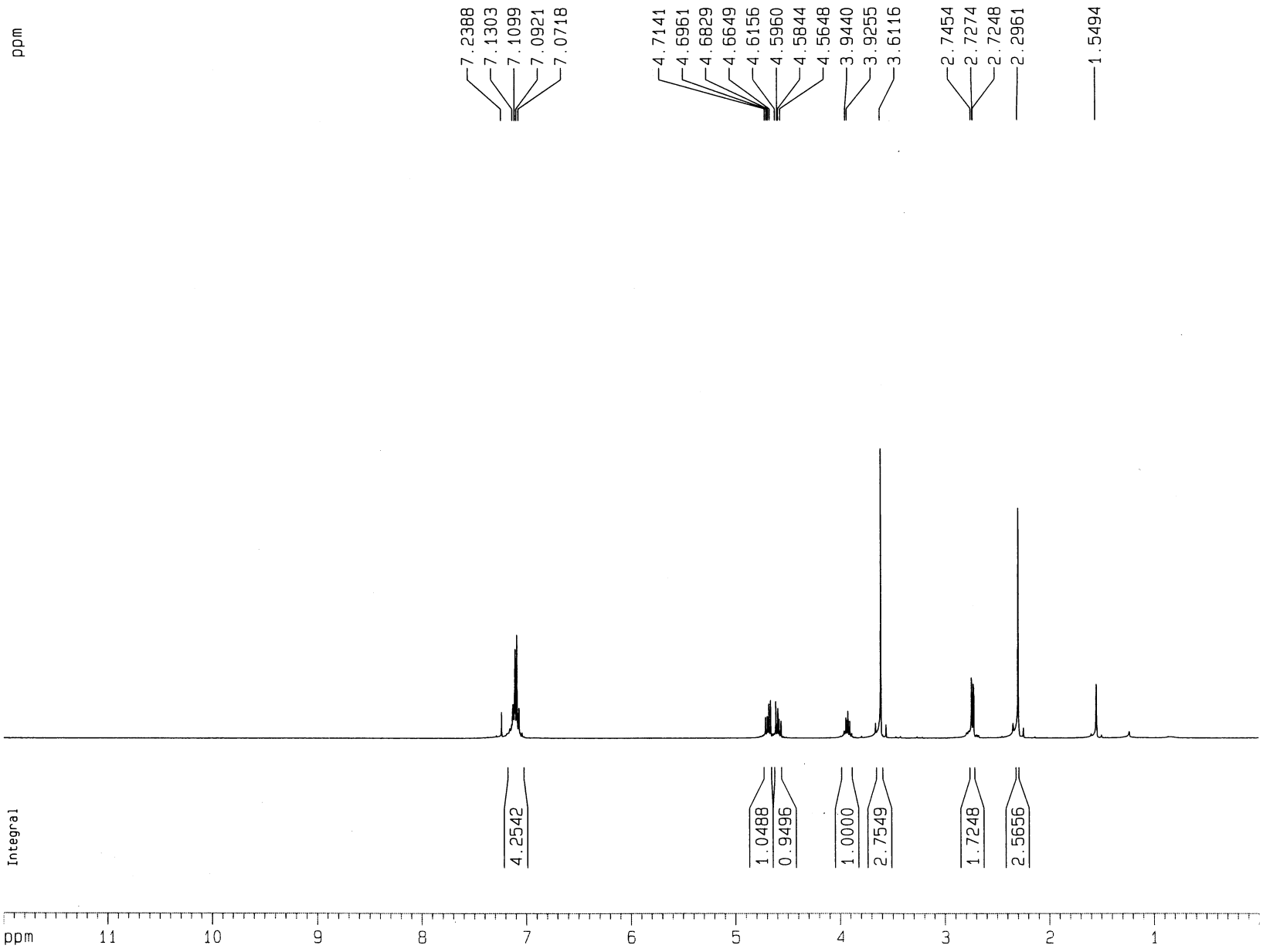
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                32768
SF           100.6127715 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S55. 1H NMR (CDCl3, 400 MHz) of compound 2e




```
Current Data Parameters
NAME                  LCH-2-350
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141227
Time                      23.51
INSTRUM                   spect
PROBHD          5 mm QNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   5995.204 Hz
FIDRES                0.365918 Hz
AQ                    1.3664756 sec
RG                       3649.1
DW                       83.400 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.50000000 sec

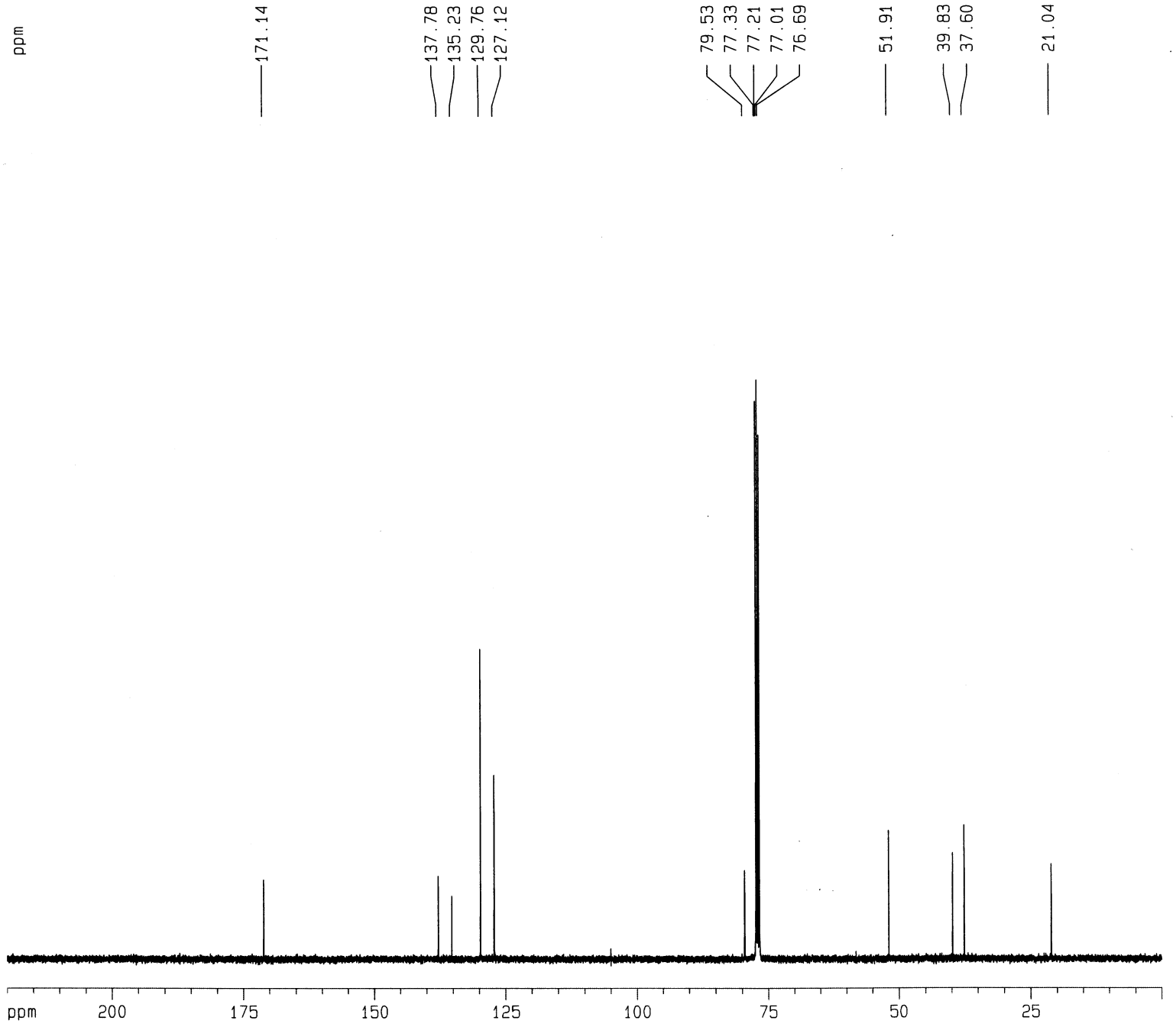
============ CHANNEL f1 =============
NUC1                         1H
P1                       11.90 usec
PL1                      -3.00 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                  400.1300179 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                      12.000 ppm
F1                      4801.56 Hz
F2P                      -0.000 ppm
F2                       -0.00 Hz
PPMCM                  0.55814 ppm/cm
HZCM                 223.32837 Hz/cm
```

C13 spectrum of

Fig S56. 13C NMR (CDCl3, 100 MHz) of compound 2e




```
Current Data Parameters
NAME                  LCH-2-350
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141228
Time                       2.54
INSTRUM                   spect
PROBHD           5 mm QNP  1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        3276
DS                           4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                         256
DW                      19.900 usec
DE                       6.50 usec
TE                      300.0 K
D1                   2.00000000 sec
d11                  0.03000000 sec
d12                  0.00002000 sec

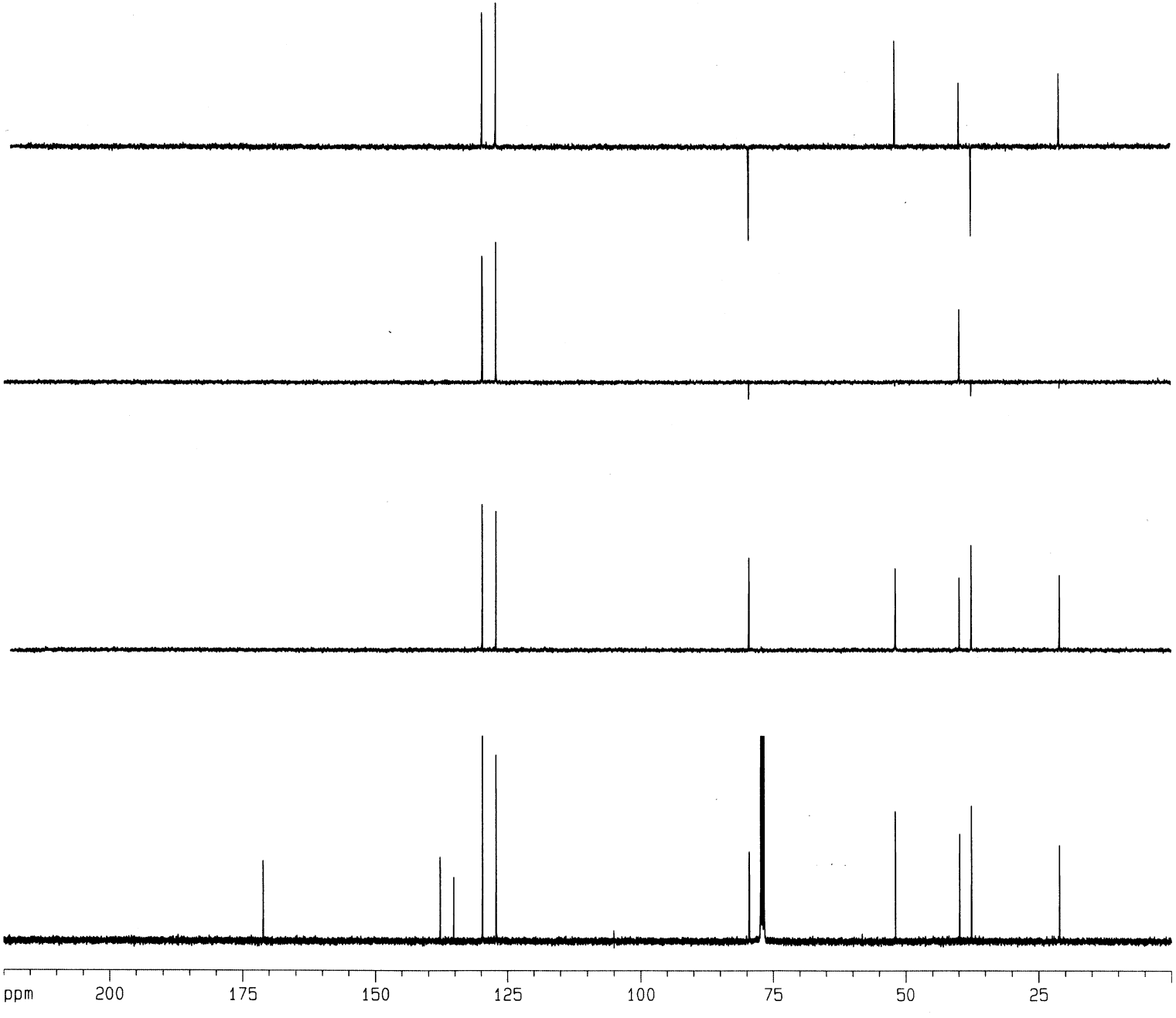
============ CHANNEL f1 =============
NUC1                       13C
P1                       10.20 usec
PL1                       0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     14.50 dB
PL13                     17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127692 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     220.000 ppm
F1                    22134.81 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S57. DEPT of compound 2e




```
Current Data Parameters
NAME          LCH-2-350
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20141228
Time               2.54
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3276
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

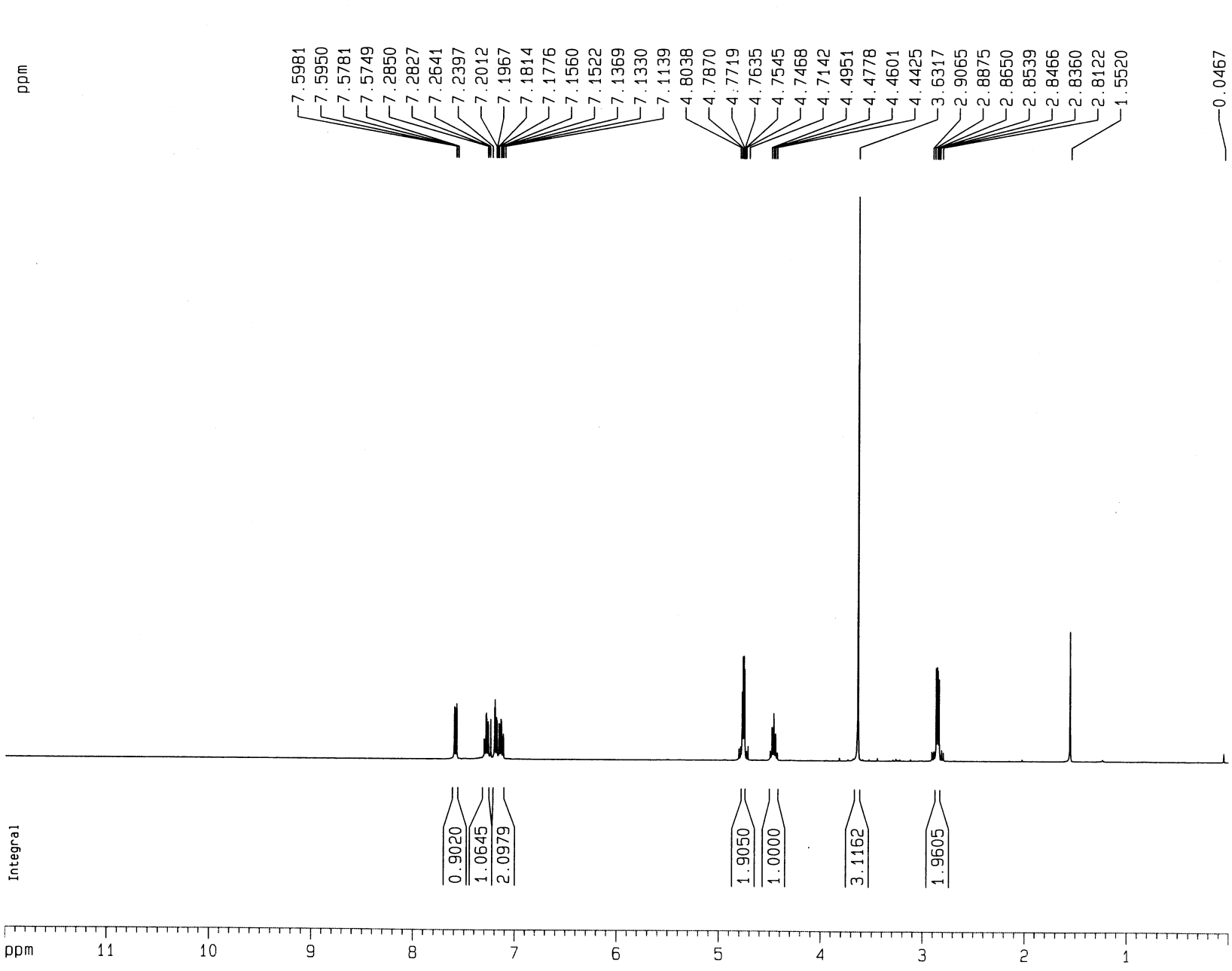
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127692 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S58. 1H NMR (CDCl3, 400 MHz) of compound 2f




```
Current Data Parameters
NAME                  LCH-2-386
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150311
Time                      20.35
INSTRUM                   spect
PROBHD      5 mm QNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                5995.204 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    3649.1
DW                   83.400 usec
DE                    6.50 usec
TE                   300.0 K
D1                 1.50000000 sec

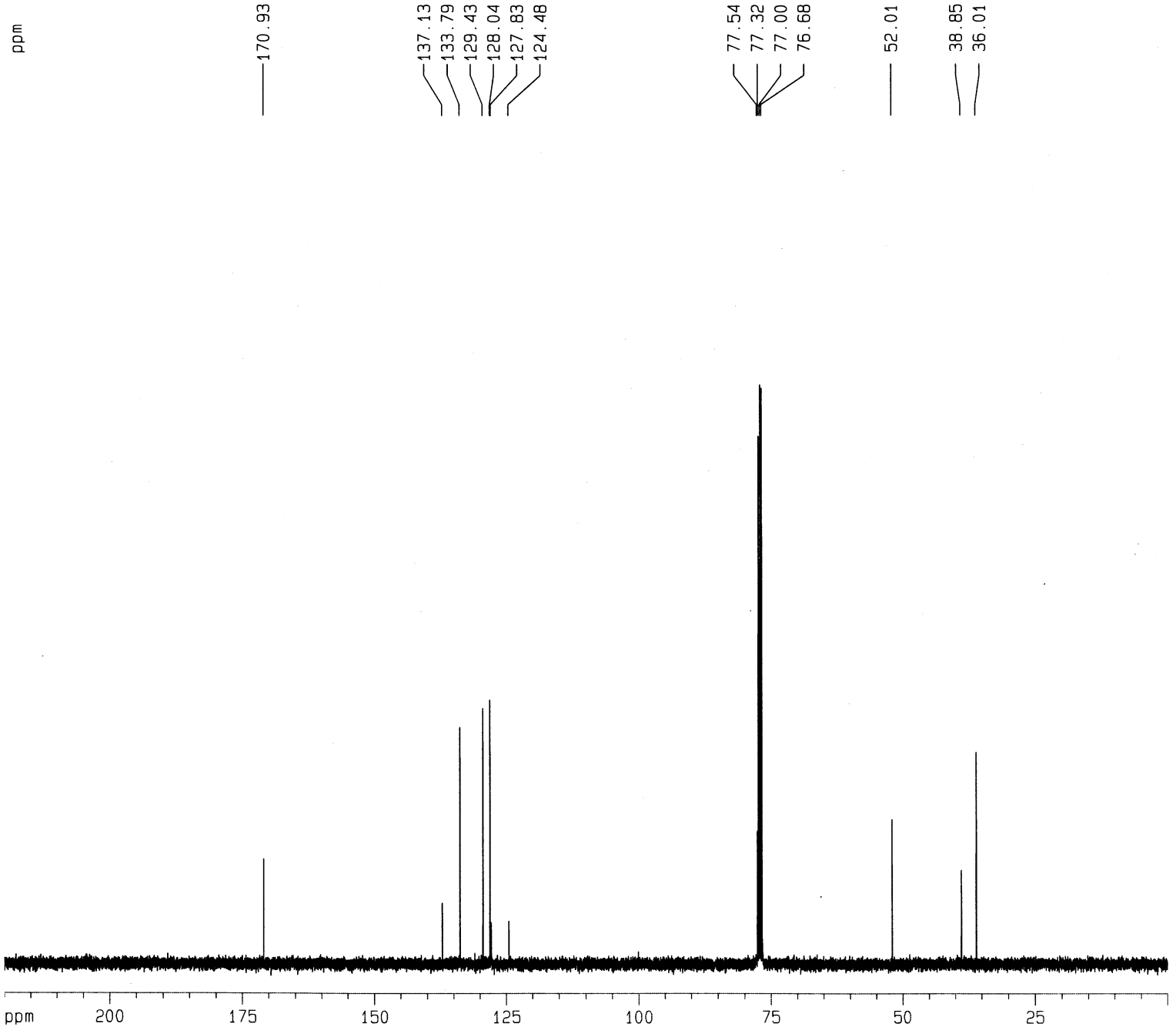
============ CHANNEL f1 ============
NUC1                         1H
P1                       11.90 usec
PL1                      -3.00 dB
SFO1               400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                 400.1300179 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                     12.000 ppm
F1                     4801.56 Hz
F2P                     -0.000 ppm
F2                      -0.00 Hz
PPMCM                 0.55814 ppm/cm
HZCM                223.32838 Hz/cm
```

C13 spectrum of

Fig S59. 13 NMR (CDCl3, 100 MHz) of compound 2f




```
Current Data Parameters
NAME                  LCH-2-386
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150311
Time                      21.22
INSTRUM                   spect
PROBHD      5 mm QNP  1H
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          819
DS                            4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                          256
DW                     19.900 usec
DE                      6.50 usec
TE                      300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

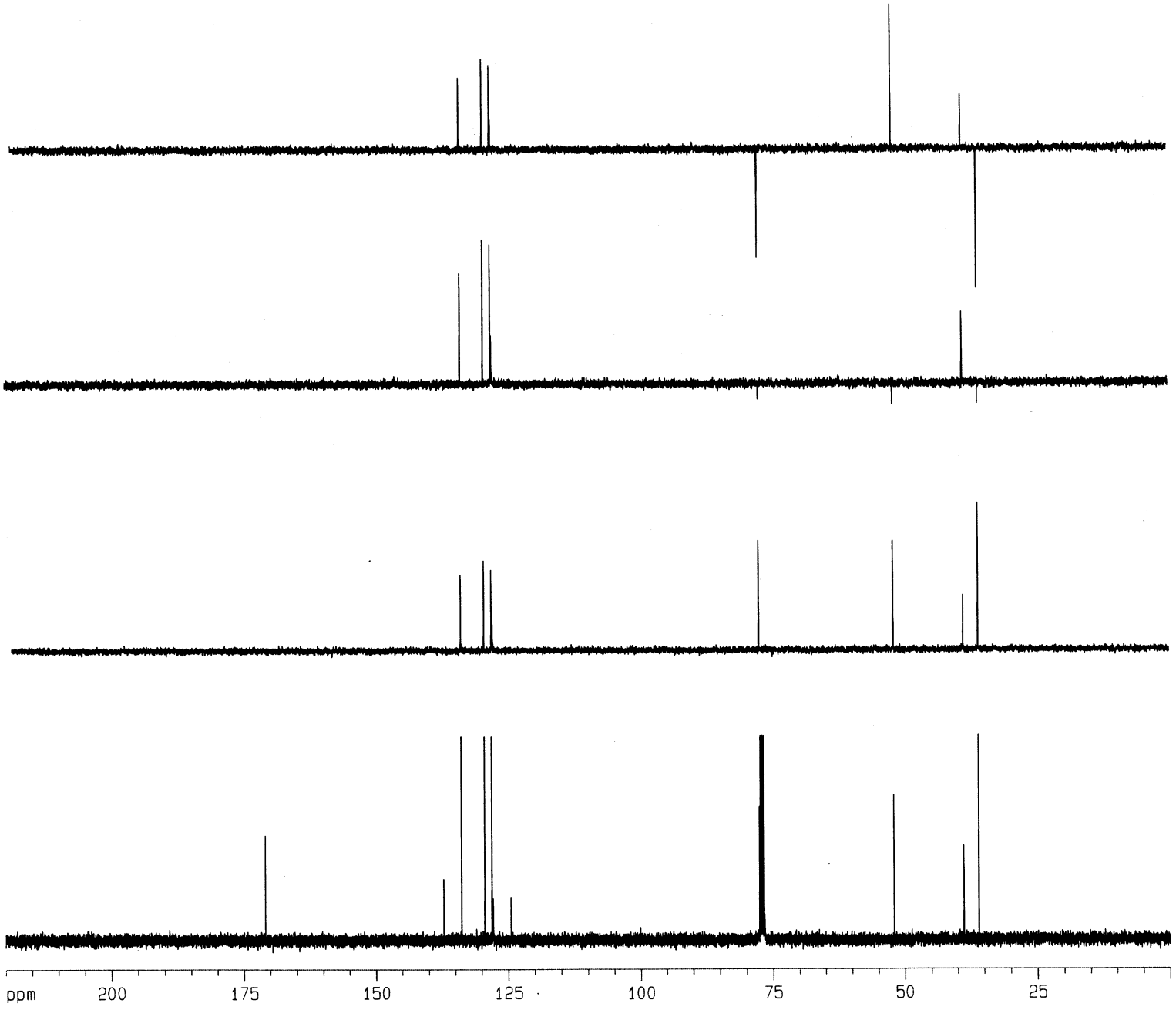
============ CHANNEL f1 =============
NUC1                        13C
P1                       10.20 usec
PL1                       0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     14.50 dB
PL13                     17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127708 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     220.000 ppm
F1                    22134.81 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                 11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S60. DEPT of compound 2f




```
Current Data Parameters
NAME                  LCH-2-386
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150311
Time                      21.22
INSTRUM                   spect
PROBHD          5 mm QNP  1H
PULPROG                 zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                           819
DS                             4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                           256
DW                        19.900 usec
DE                         6.50 usec
TE                        300.0 K
D1                   2.00000000 sec
d11                  0.03000000 sec
d12                  0.00002000 sec

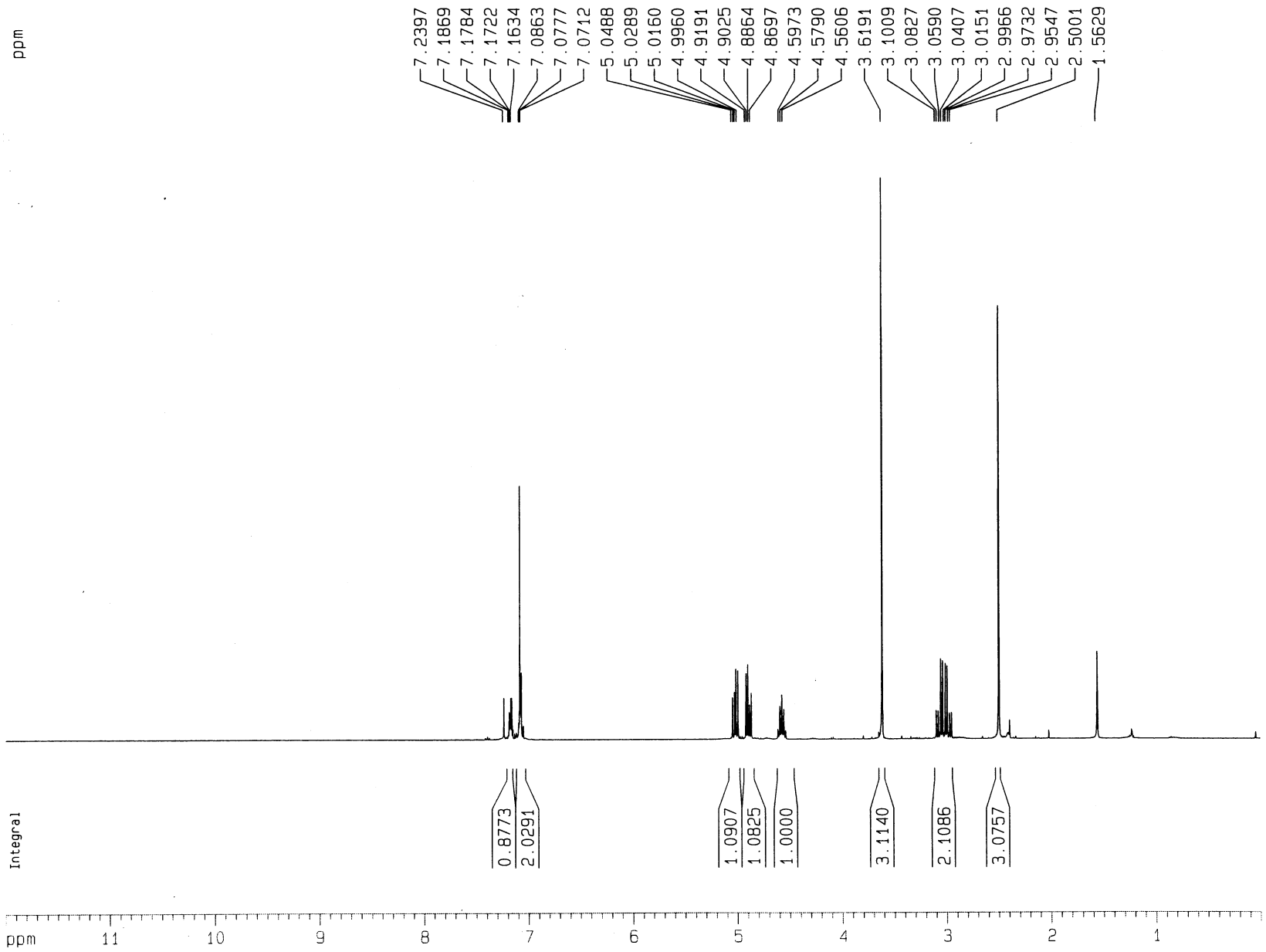
============ CHANNEL f1 =============
NUC1                         13C
P1                        10.20 usec
PL1                        0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     90.00 usec
PL2                       -3.00 dB
PL12                      14.50 dB
PL13                      17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                           32768
SF                  100.6127708 MHz
WDW                             EM
SSB                              0
LB                            0.30 Hz
GB                               0
PC                            1.40

1D NMR plot parameters
CX                           20.00 cm
F1P                         220.000 ppm
F1                        22134.81 Hz
F2P                         0.000 ppm
F2                            0.00 Hz
PPMCM                    11.00000 ppm/cm
HZCM                  1106.74048 Hz/cm
```

Fig S61. 1H NMR (CDCl3, 400 MHz) of compound 2g




```
Current Data Parameters
NAME                  LCH-2-369
EXPNO                         6
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150412
Time                      19.05
INSTRUM                   spect
PROBHD      5 mm QNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   5995.204 Hz
FIDRES                0.365918 Hz
AQ                    1.3664756 sec
RG                        3251
DW                      83.400 usec
DE                       6.50 usec
TE                      300.0 K
D1                    1.50000000 sec

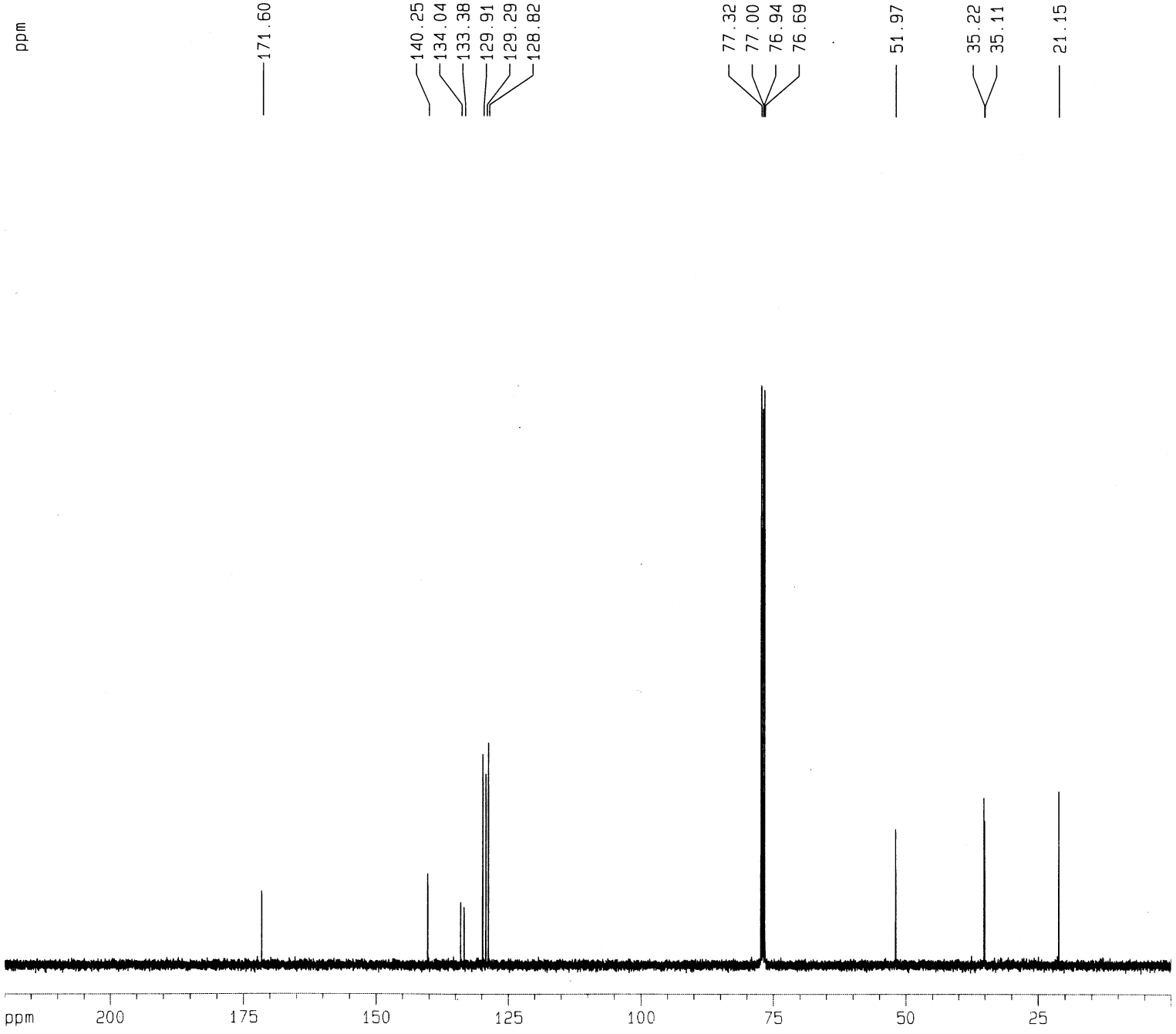
============ CHANNEL f1 =============
NUC1                         1H
P1                       11.90 usec
PL1                      -3.00 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                  400.1300179 MHz
WDW                         EM
SSB                          0
LB                       0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                     12.000 ppm
F1                     4801.56 Hz
F2P                     -0.000 ppm
F2                      -0.00 Hz
PPMCM                 0.55814 ppm/cm
HZCM                223.32837 Hz/cm
```

C13 spectrum of

Fig S62. 13C NMR (CDCl3, 100 MHz) of compound 2g




```
Current Data Parameters
NAME            LCH-2-369
EXPNO                   7
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150412
Time                20.03
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             25125.629 Hz
FIDRES          0.383387 Hz
AQ              1.3042164 sec
RG                    256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

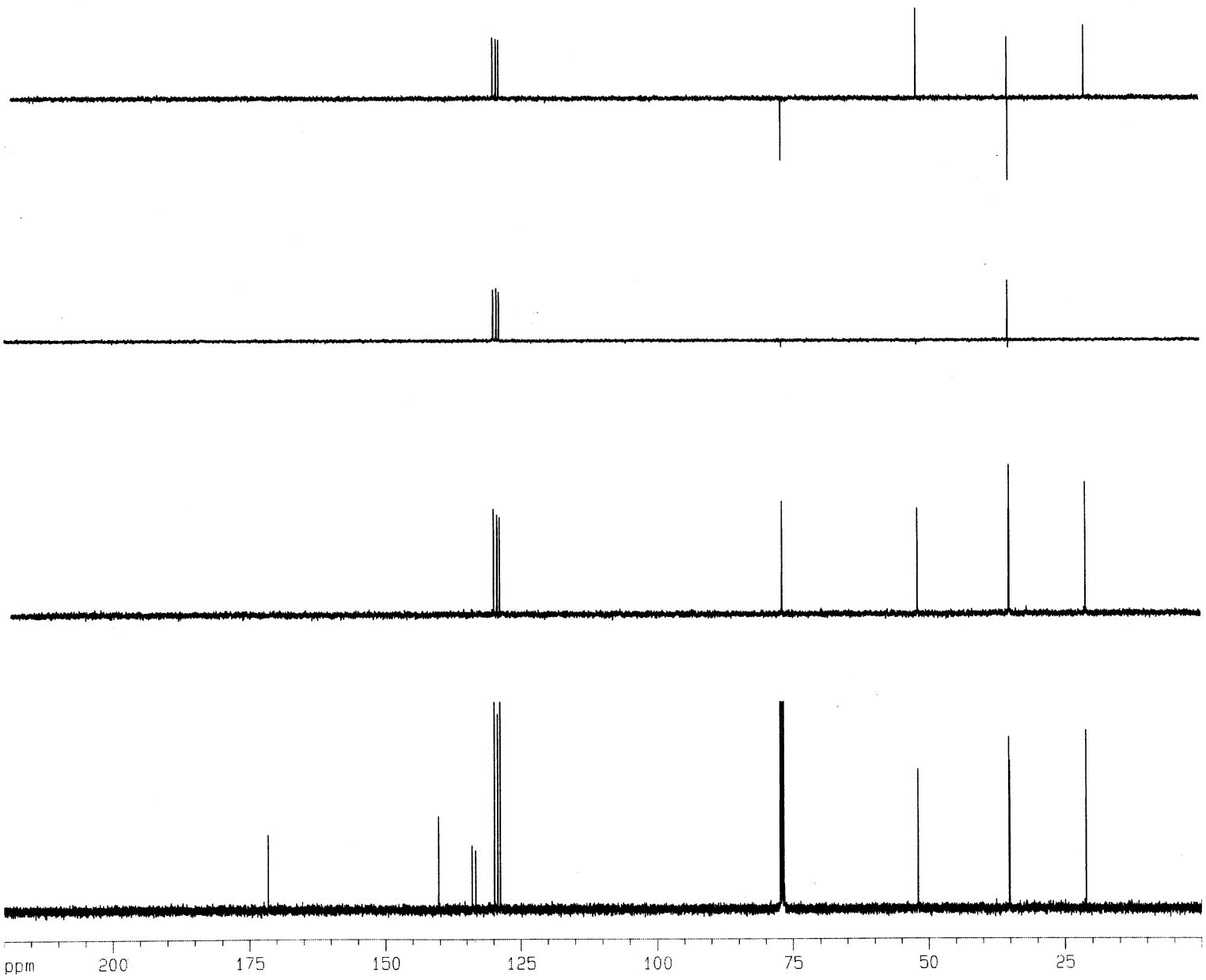
============ CHANNEL f1 =============
NUC1                 13C
P1                 10.20 usec
PL1                 0.00 dB
SFO1          100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -3.00 dB
PL12               14.50 dB
PL13               17.50 dB
SFO2          400.1326008 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127715 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```

Fig S63. DEPT of compound 2g




```
Current Data Parameters
NAME          LCH-2-369
EXPNO                 7
PROCNO                1

F2 - Acquisition Parameters
Date_          20150412
Time              20.03
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

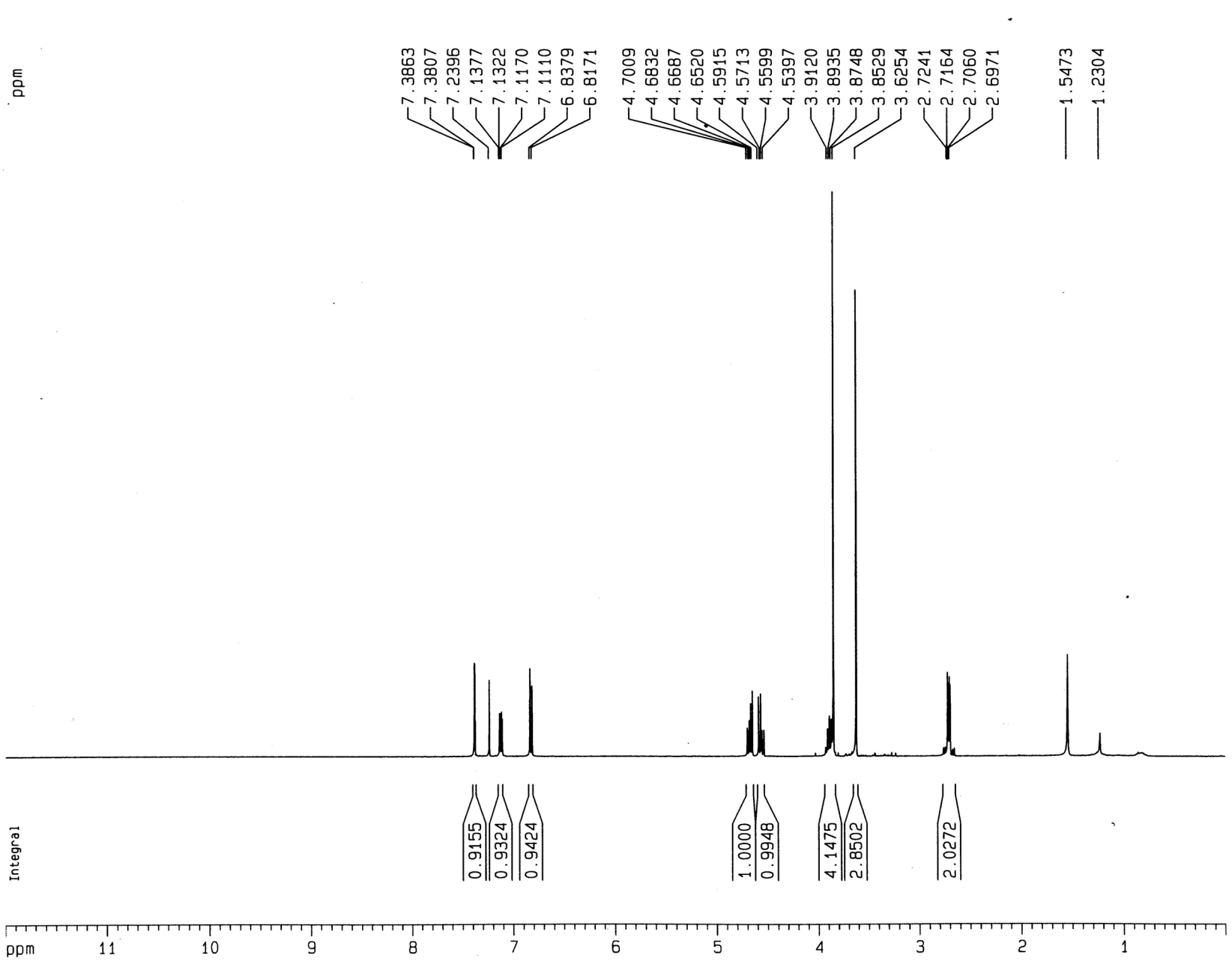
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                32768
SF          100.6127715 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
```

Fig S64. 1H NMR (CDCl3, 400 MHz) of compound 2h



Current Data Parameters

NAME	LCH-2-287
EXPNO	9
PROCNO	1

F2 - Acquisition Parameters

Date_	20150415
Time	0.39
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	5792.6
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

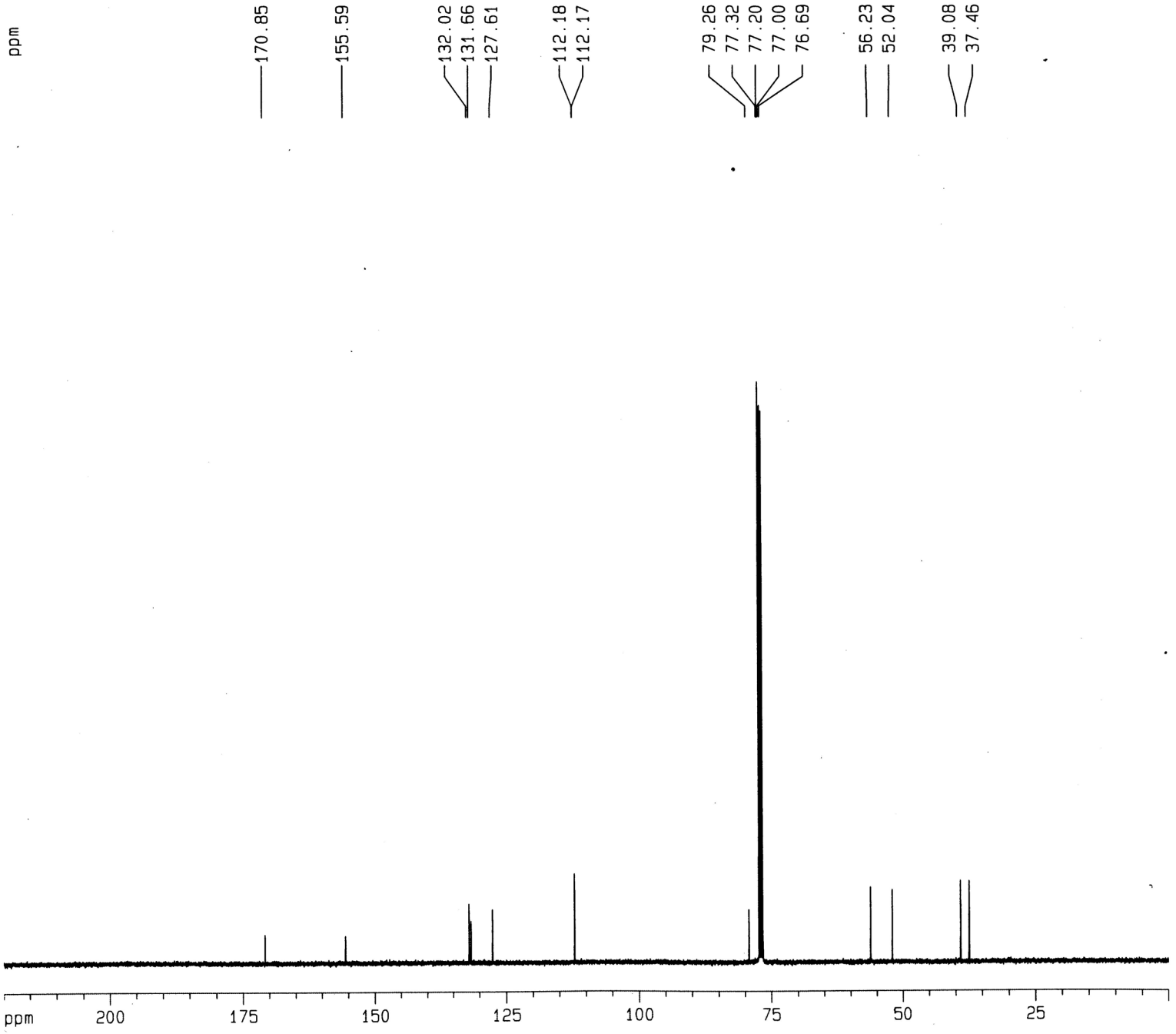
SI	8192
SF	400.1300179 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	0.55814 ppm/cm
HZCM	223.32838 Hz/cm

C13 spectrum of

Fig S65. 13C NMR (CDCl3, 100 MHz) of compound 2h




```
Current Data Parameters
NAME                  LCH-2-287
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150415
Time                       4.03
INSTRUM                   spect
PROBHD           5 mm QNP  1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        3686
DS                           4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                         256
DW                      19.900 usec
DE                       6.50 usec
TE                      300.0 K
D1                   2.00000000 sec
d11                  0.03000000 sec
d12                  0.00002000 sec

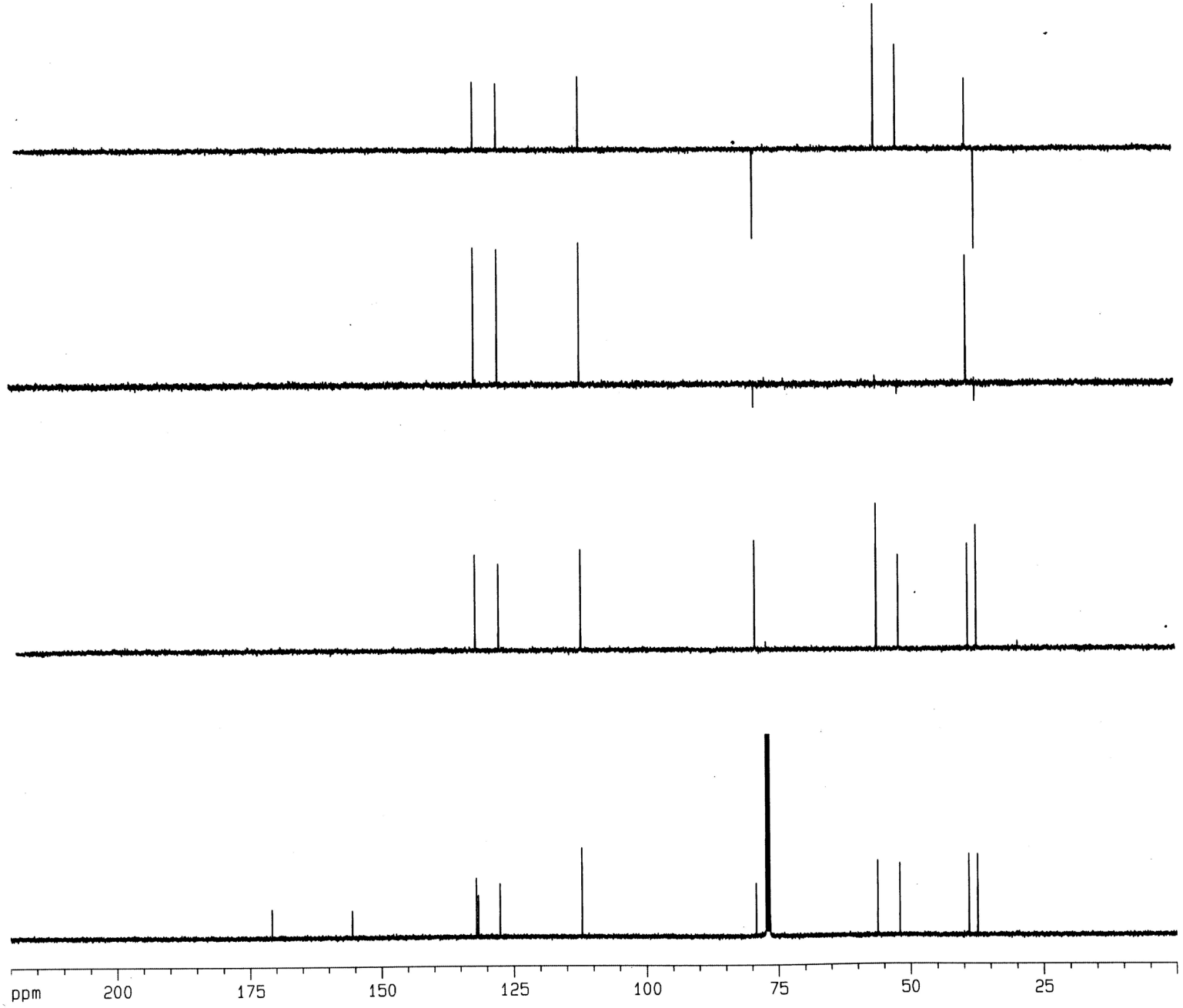
============ CHANNEL f1 =============
NUC1                       13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127715 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      220.000 ppm
F1                     22134.81 Hz
F2P                        0.000 ppm
F2                         0.00 Hz
PPMCM                  11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S66. DEPT of compound 2h




```
Current Data Parameters
NAME                  LCH-2-287
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150415
Time                       4.03
INSTRUM                   spect
PROBHD      5 mm QNP  1H
PULPROG                 zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                          3686
DS                             4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                    1.3042164 sec
RG                           256
DW                        19.900 usec
DE                         6.50 usec
TE                        300.0 K
D1                    2.00000000 sec
d11                   0.03000000 sec
d12                   0.00002000 sec

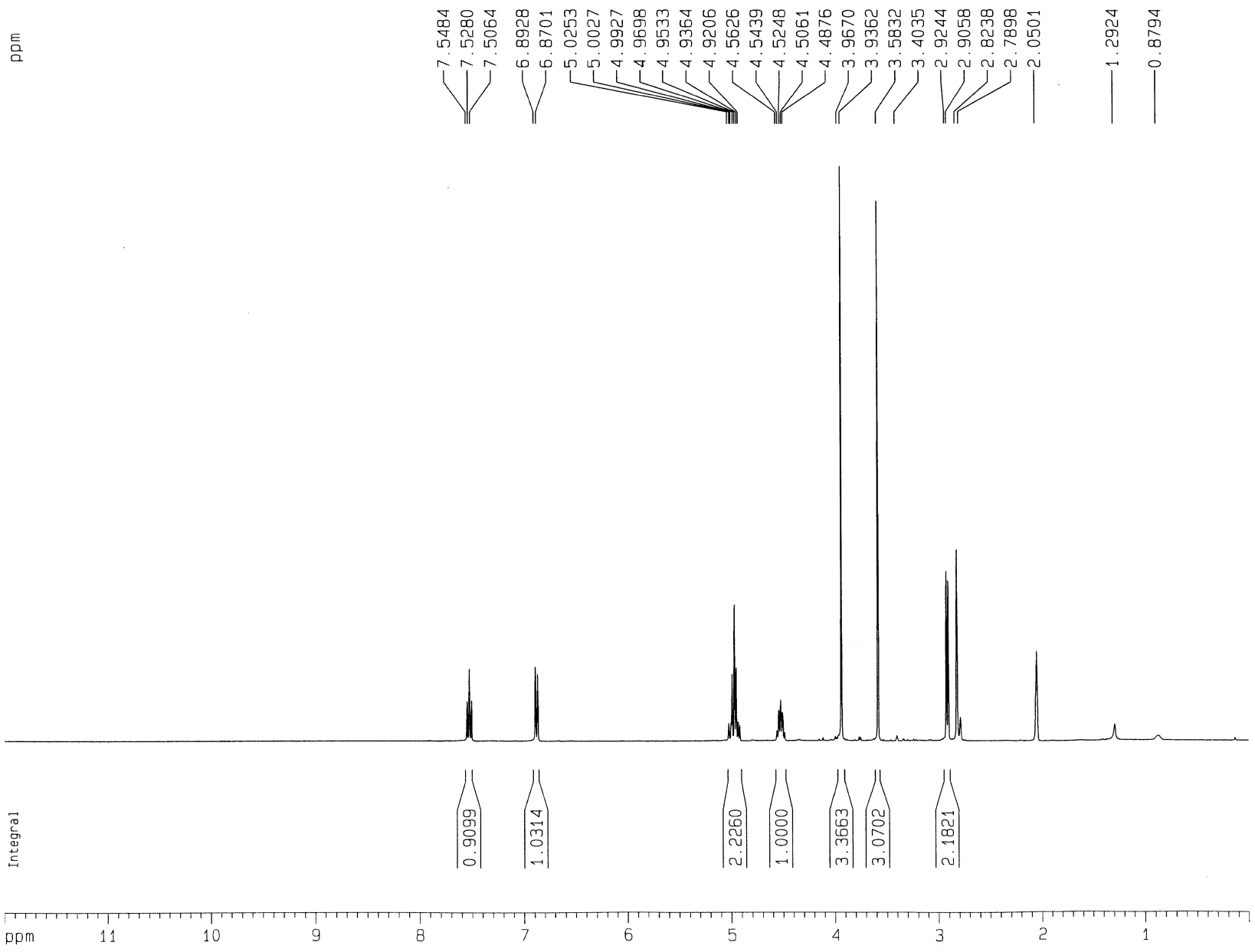
============ CHANNEL f1 =============
NUC1                         13C
P1                        10.20 usec
PL1                        0.00 dB
SF01                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                         1H
PCPD2                     90.00 usec
PL2                       -3.00 dB
PL12                      14.50 dB
PL13                      17.50 dB
SF02                400.1326008 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127715 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      220.000 ppm
F1                     22134.81 Hz
F2P                       0.000 ppm
F2                         0.00 Hz
PPMCM                  11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S67. 1H NMR (acetone-d6, 400 MHz NMR) of compound 2i



Current Data Parameters

NAME	LCH-2-383
EXPNO	17
PROCNO	1

F2 - Acquisition Parameters

Date_	20150601
Time	17.40
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	Aceton
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	2896.3
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

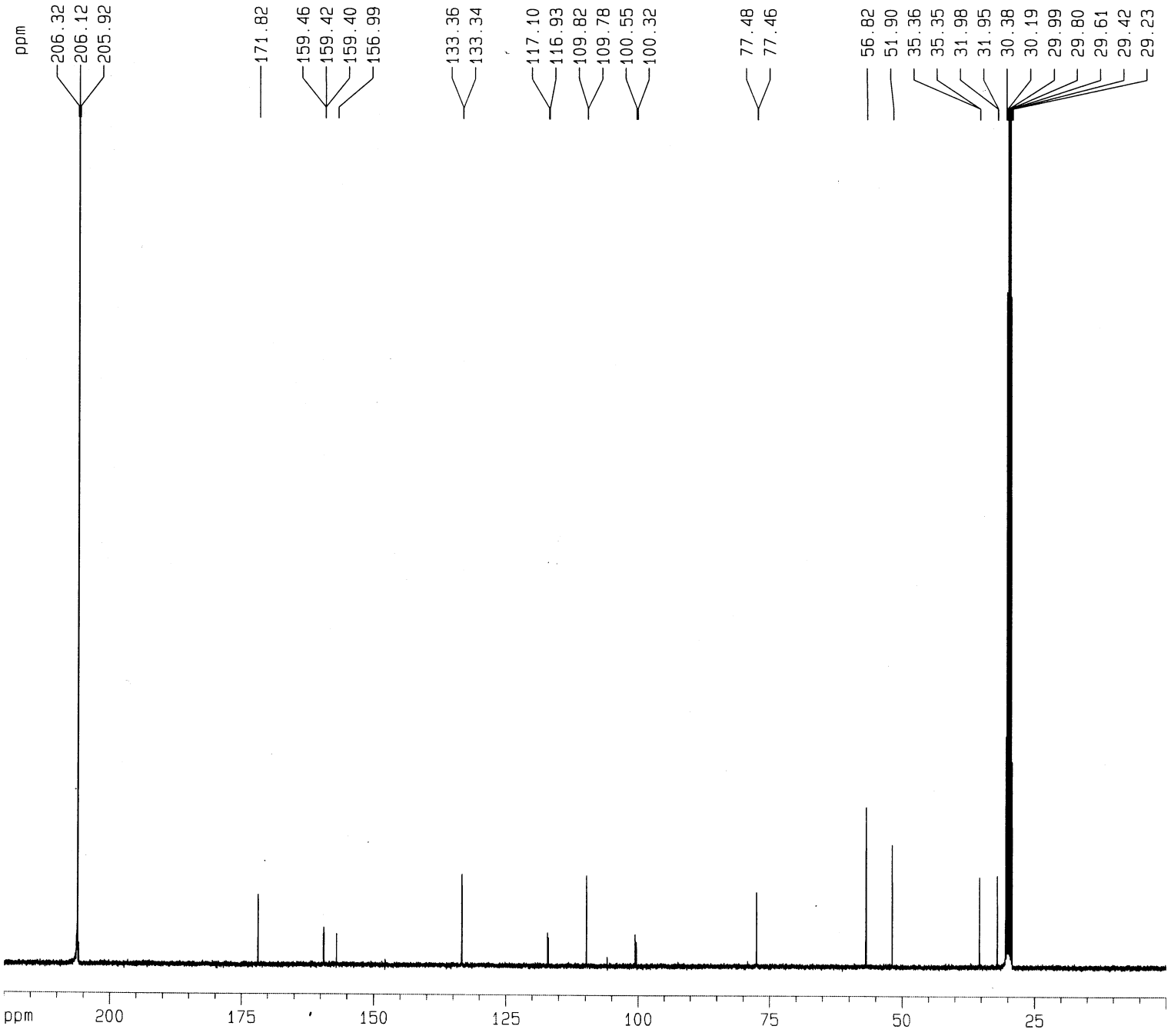
SI	8192
SF	400.1300066 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	0.55814 ppm/cm
HZCM	223.32837 Hz/cm

C13 spectrum of

Fig S68. 13C NMR (acetone-d6, 100 MHz) of compound 2i




```
Current Data Parameters
NAME          LCH-2-383
EXPNO                13
PROCNO                1

F2 - Acquisition Parameters
Date_          20150423
Time               3.47
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3686
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

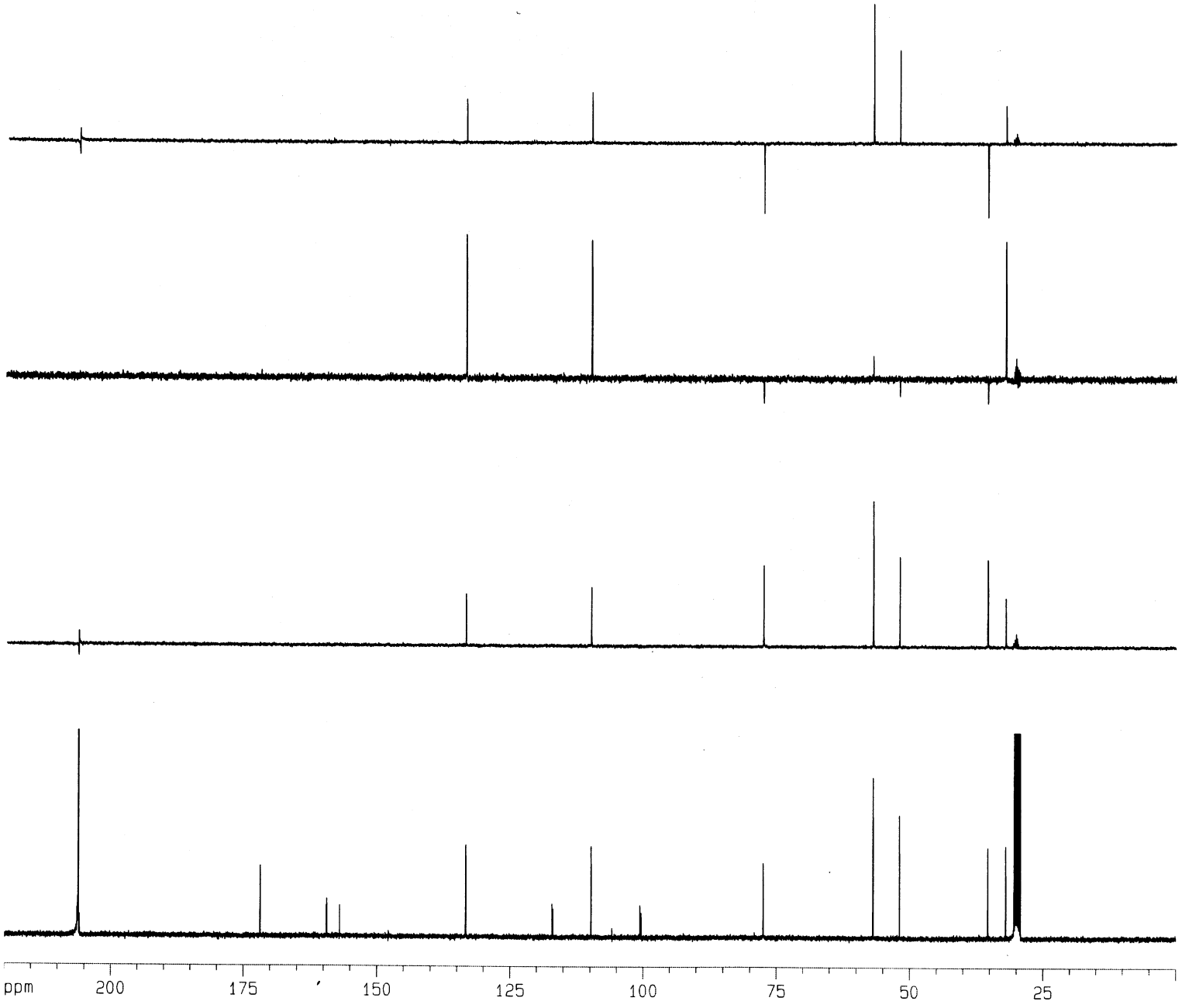
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6126837 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.79 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73950 Hz/cm
```

Fig S69. DEPT of compound 2i




```
Current Data Parameters
NAME                 LCH-2-383
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20150423
Time                      3.47
INSTRUM                  spect
PROBHD      5 mm QNP  1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        3686
DS                           4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                    1.3042164 sec
RG                         256
DW                      19.900 usec
DE                       6.50 usec
TE                      300.0 K
D1                    2.00000000 sec
d11                   0.03000000 sec
d12                   0.00002000 sec

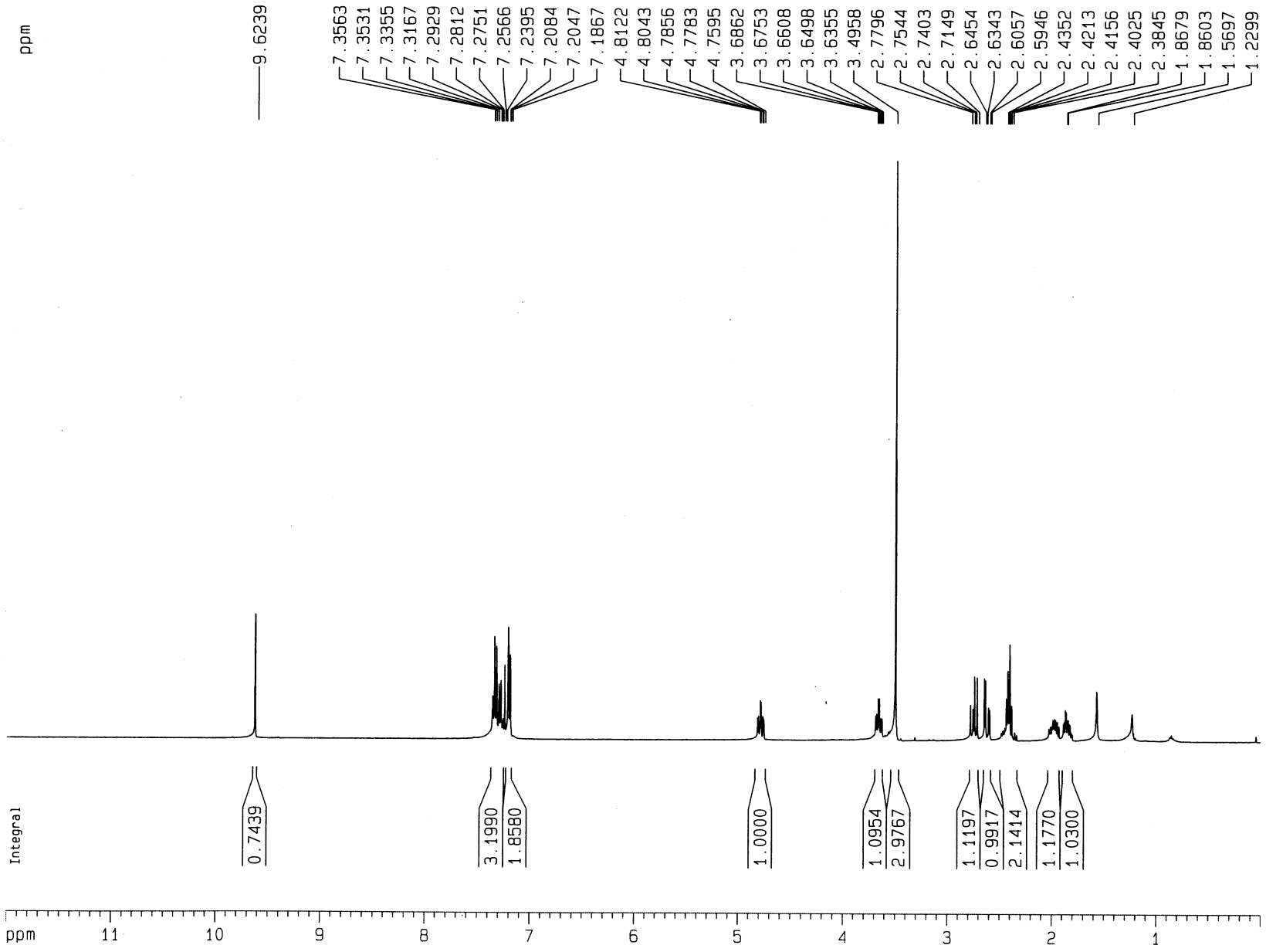
============ CHANNEL f1 =============
NUC1                        13C
P1                       10.20 usec
PL1                       0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     14.50 dB
PL13                     17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6126837 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     220.000 ppm
F1                    22134.79 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                 11.00000 ppm/cm
HZCM                1106.73950 Hz/cm
```

Fig S70. 1H NMR (CDCl3, 400 MHz) of compound syn-3a



Current Data Parameters

NAME	LCH-2-308-f1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20141127
Time	22.25
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	2896.3
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

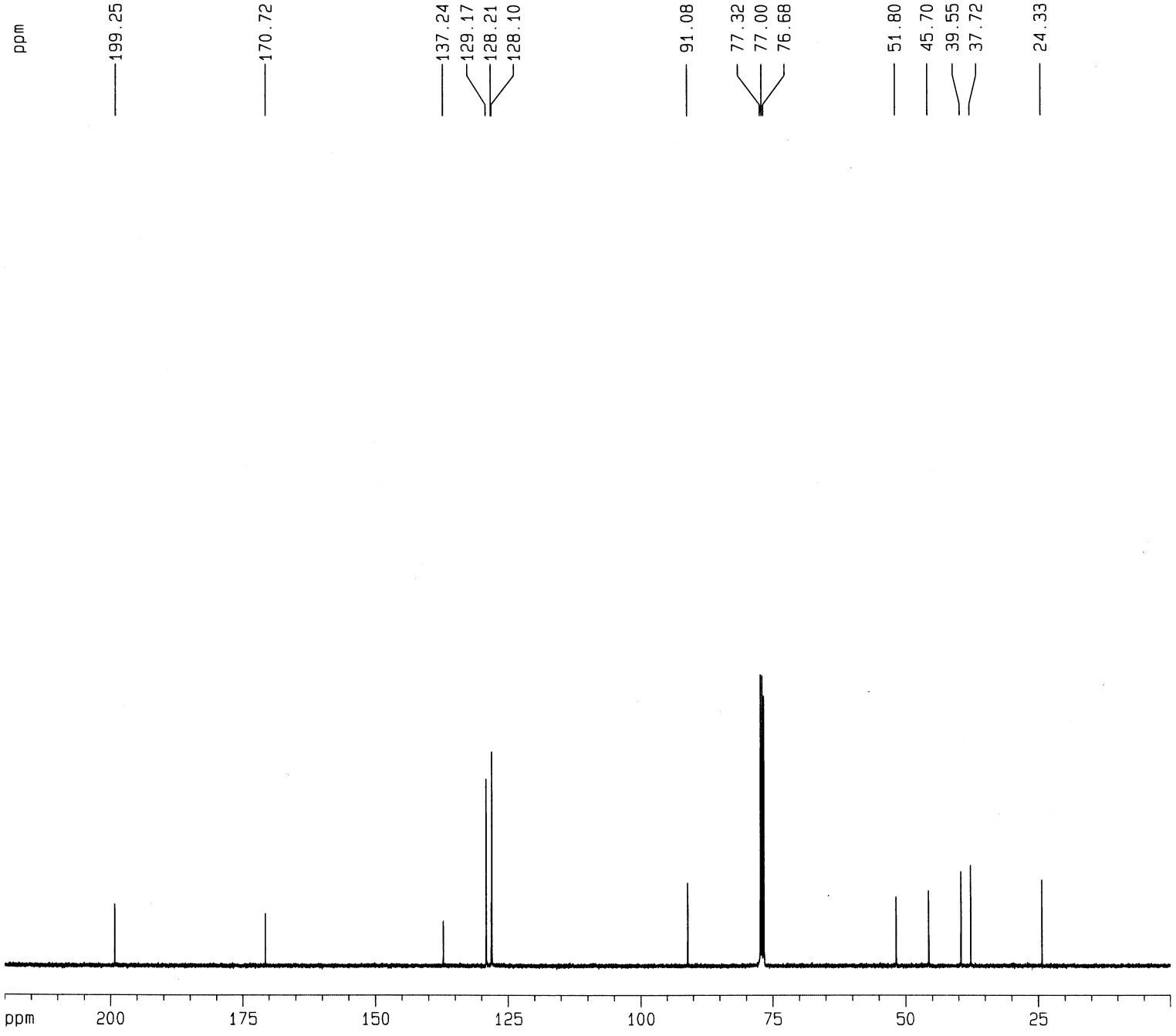
SI	8192
SF	400.1300179 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	0.55814 ppm/cm
HZCM	223.32838 Hz/cm

C13 spectrum of

Fig S71. 13C NMR (CDCl3, 100 MHz) of compound syn-3a




```
Current Data Parameters
NAME        LCH-2-308-f1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141128
Time                1.52
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

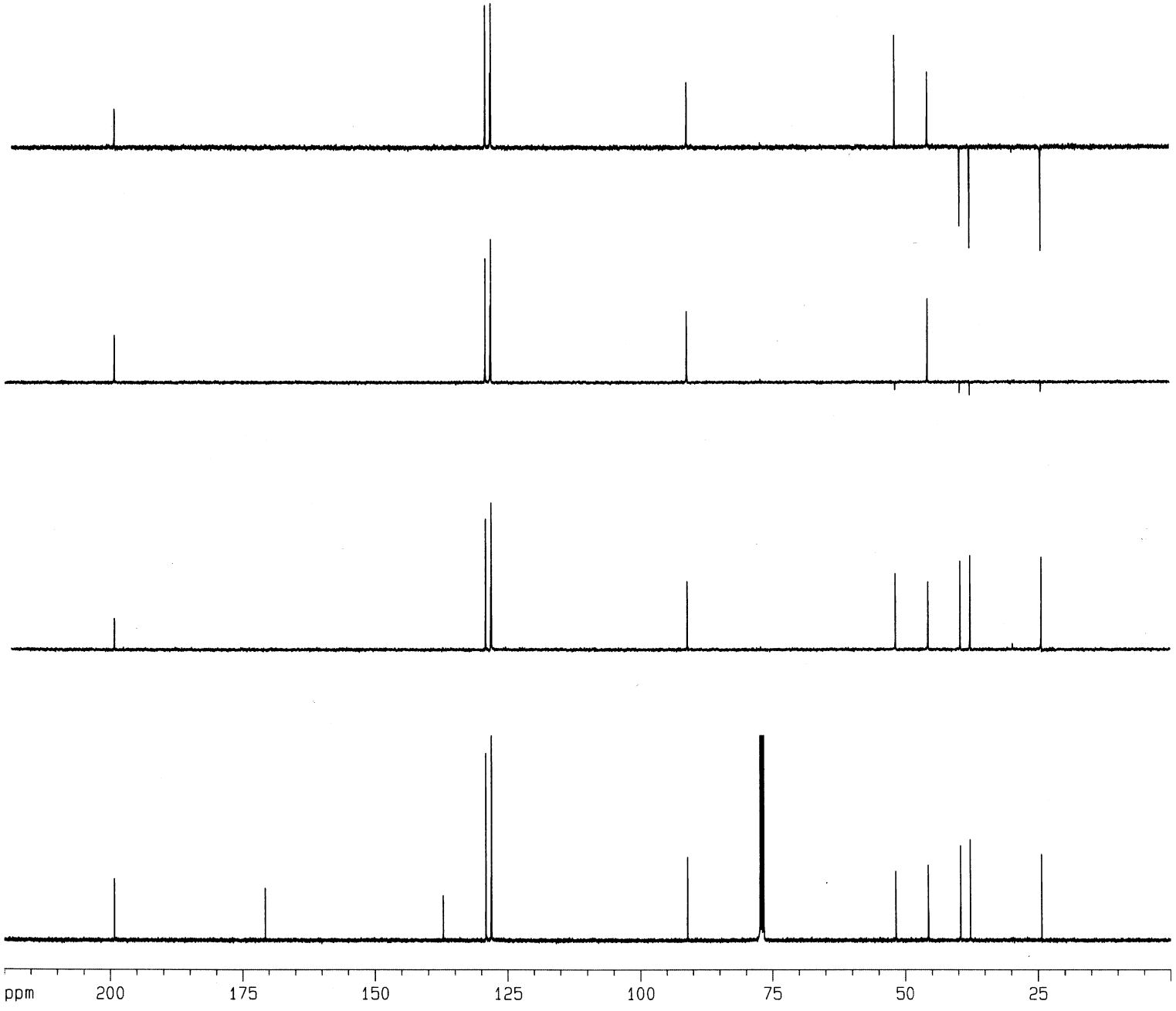
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127715 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S72. DEPT of compound syn-3a




```
Current Data Parameters
NAME        LCH-2-308-f1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141128
Time                1.52
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3686
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

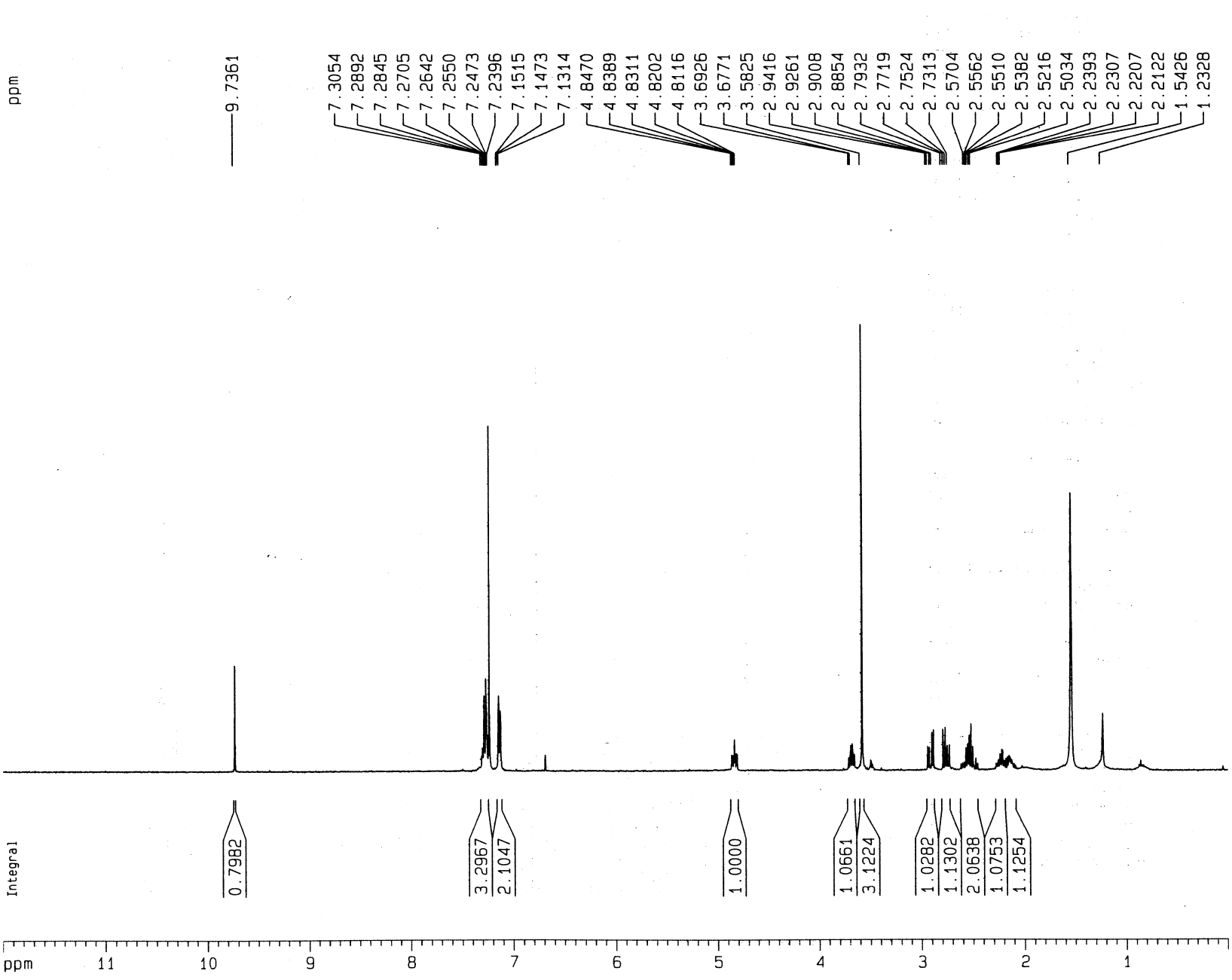
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SF01         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SF02         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127715 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S73. 1H NMR (CDCl3, 400 MHz) of compound anti-3a




```
Current Data Parameters
NAME        LCH-2-308-f2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141205
Time               14.59
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG            10321.3
DW             83.400 usec
DE              6.50 usec
TE             300.0 K
D1          1.50000000 sec

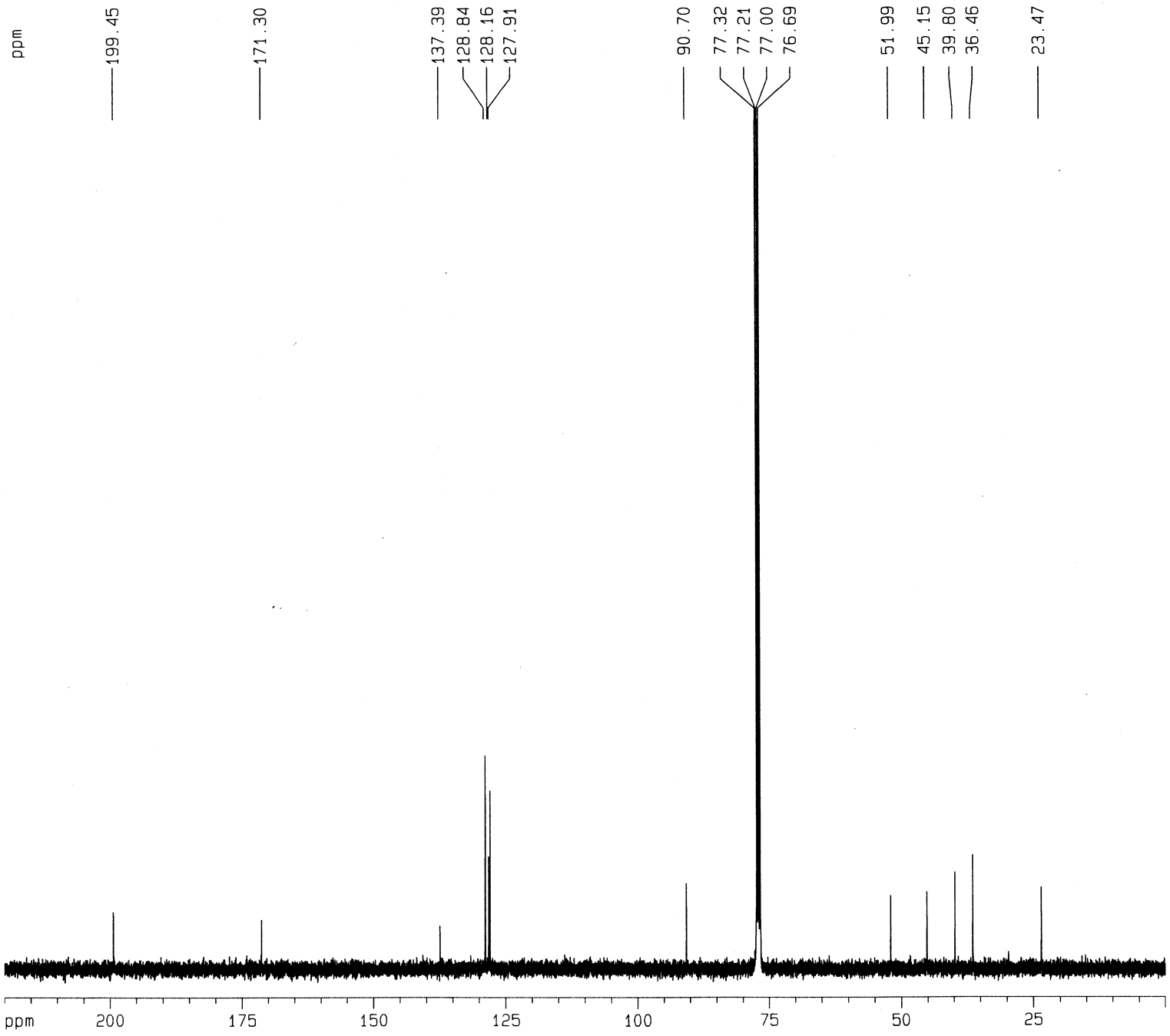
============ CHANNEL f1 =============
NUC1                1H
P1             11.90 usec
PL1             -3.00 dB
SFO1       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                 EM
SSB                 0
LB              0.10 Hz
GB                  0
PC              1.00

1D NMR plot parameters
CX             21.50 cm
F1P           12.000 ppm
F1            4801.56 Hz
F2P           -0.000 ppm
F2             -0.00 Hz
PPMCM         0.55814 ppm/cm
HZCM        223.32837 Hz/cm
```

C13 spectrum of

Fig S74. 13C NMR (CDCl3, 100 MHz) of compound anti-3a




```
Current Data Parameters
NAME        LCH-2-308-f2
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141208
Time                3.03
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  4096
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW               19.900 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

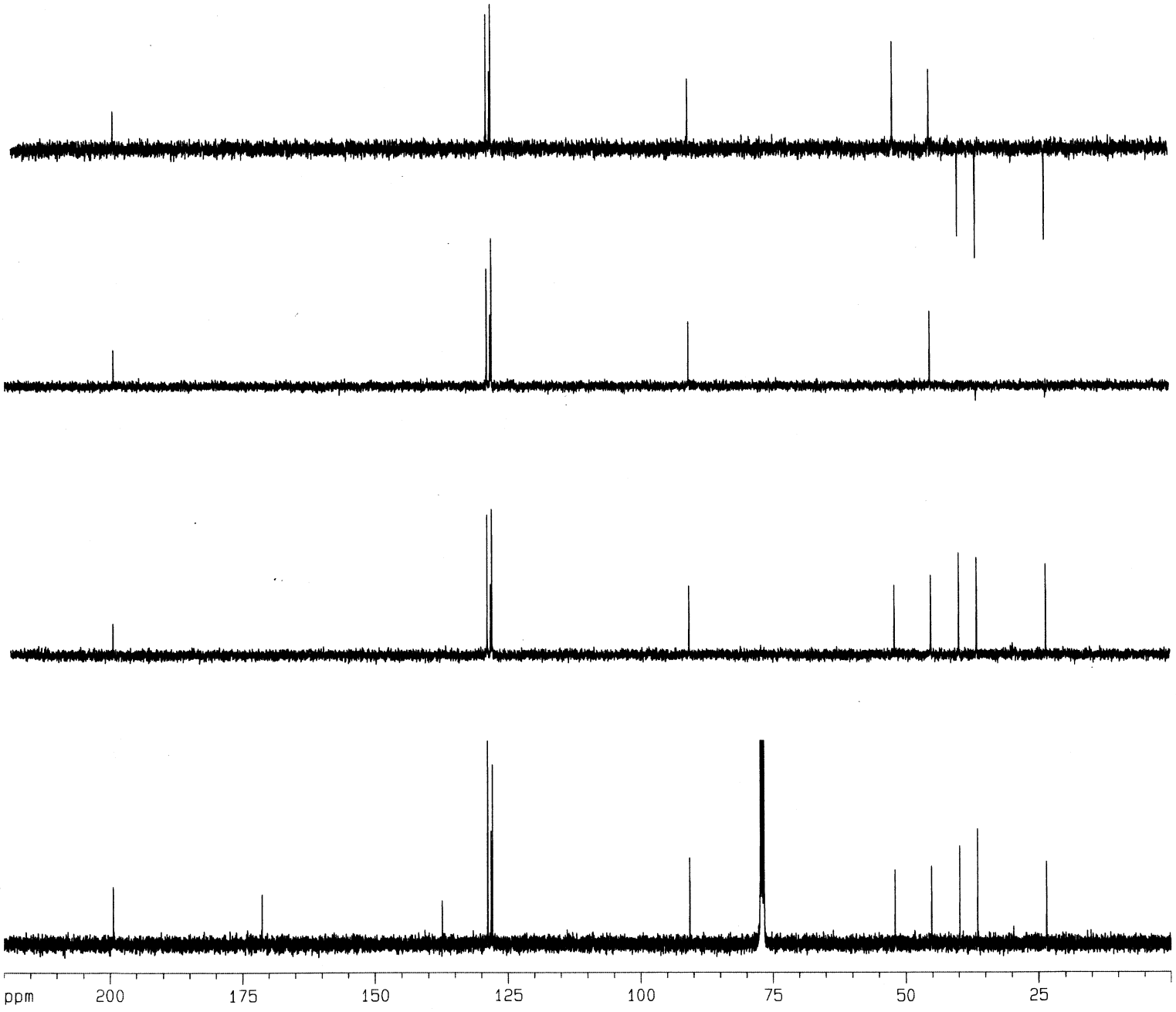
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2           90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
```

Fig S75. DEPT of compound anti-3a




```
Current Data Parameters
NAME        LCH-2-308-f2
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141208
Time                3.03
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

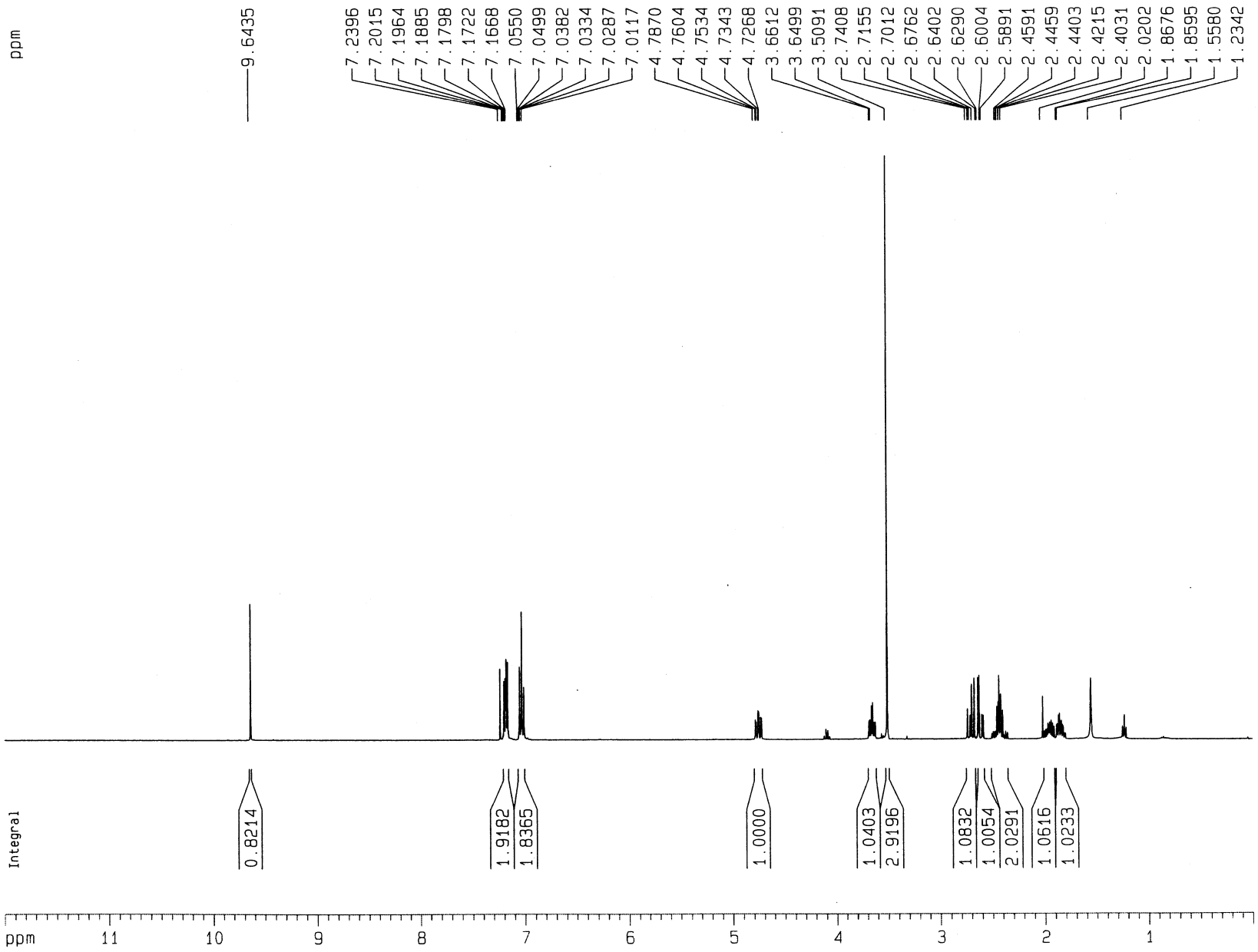
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
```

Fig S76. 1H NMR (CDCl3, 400 MHz) of compound syn-3b




```
Current Data Parameters
NAME        LCH-2-375-p1
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150306
Time                1.01
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG                  4096
DW              83.400 usec
DE               6.50 usec
TE               300.0 K
D1          1.50000000 sec

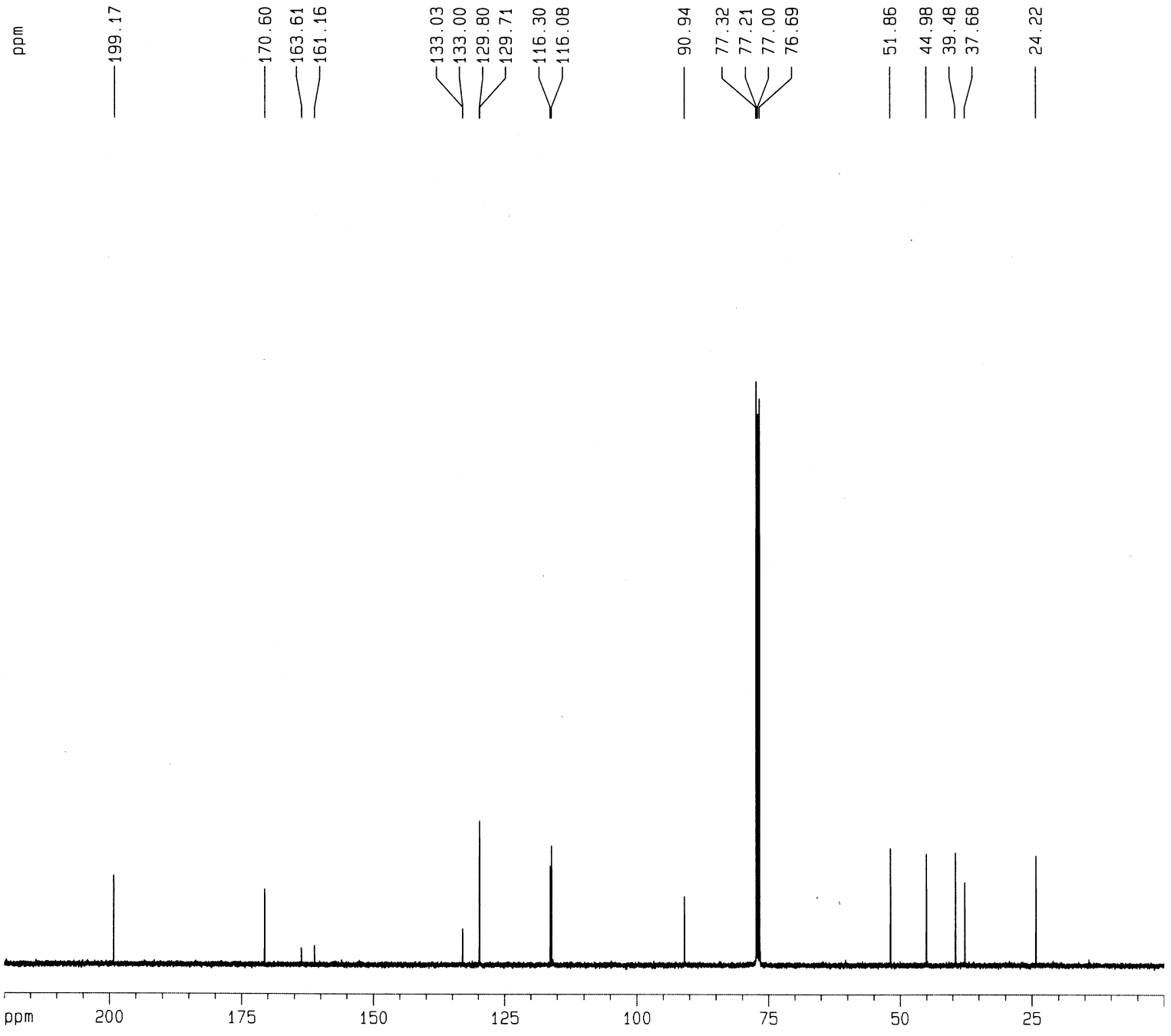
============ CHANNEL f1 =============
NUC1                1H
P1              11.90 usec
PL1             -3.00 dB
SFO1       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                 EM
SSB                    0
LB              0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              21.50 cm
F1P            12.000 ppm
F1            4801.56 Hz
F2P            -0.000 ppm
F2              -0.00 Hz
PPMCM         0.55814 ppm/cm
HZCM        223.32838 Hz/cm
```

C13 spectrum of

Fig S77. 13C NMR (CDCl3, 100 MHz) of compound syn-3b




```
Current Data Parameters
NAME        LCH-2-375-p1
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150306
Time                4.26
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

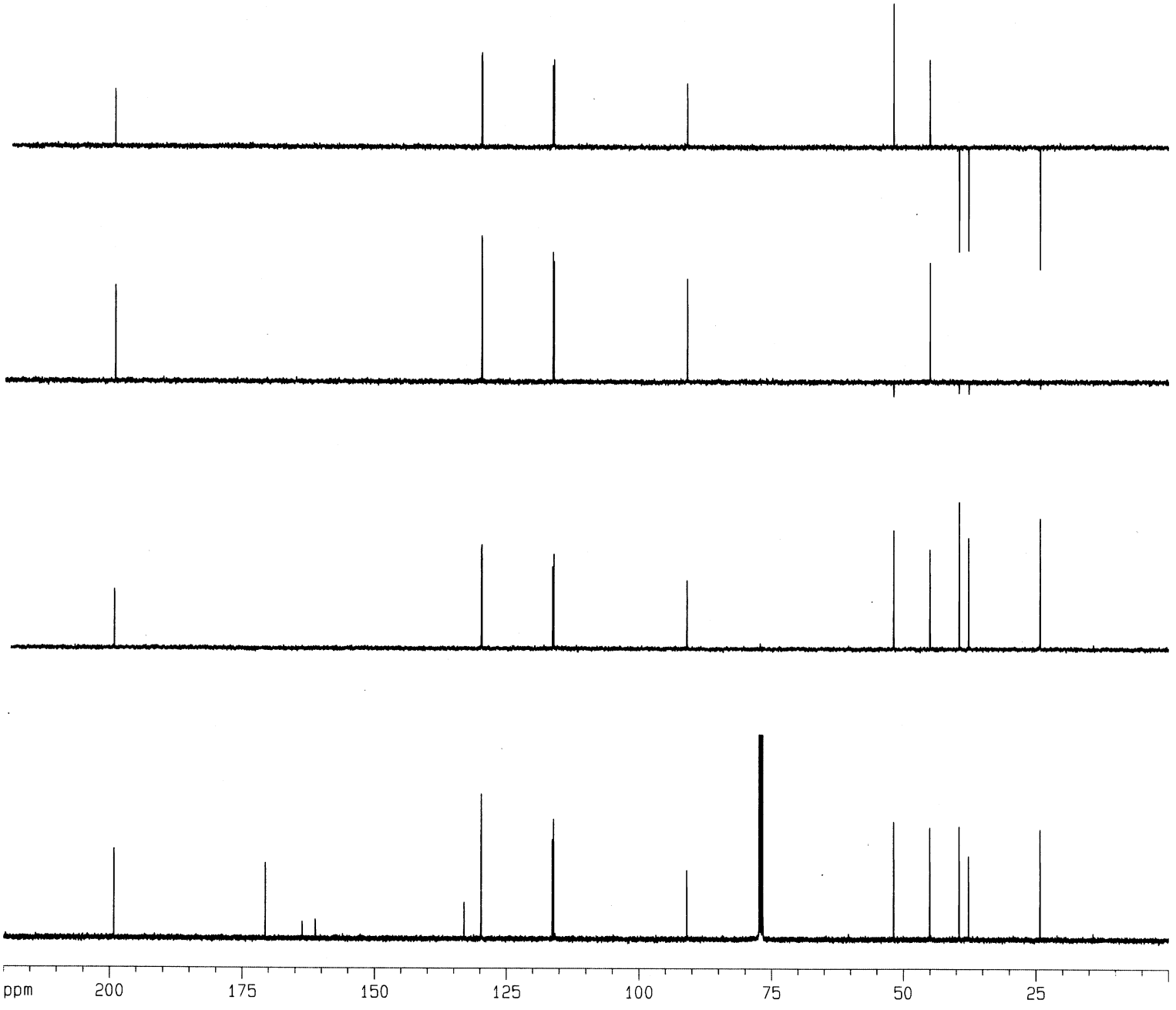
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S78. DEPT of compound syn-3b




```
Current Data Parameters
NAME        LCH-2-375-p1
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150306
Time                4.26
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3686
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

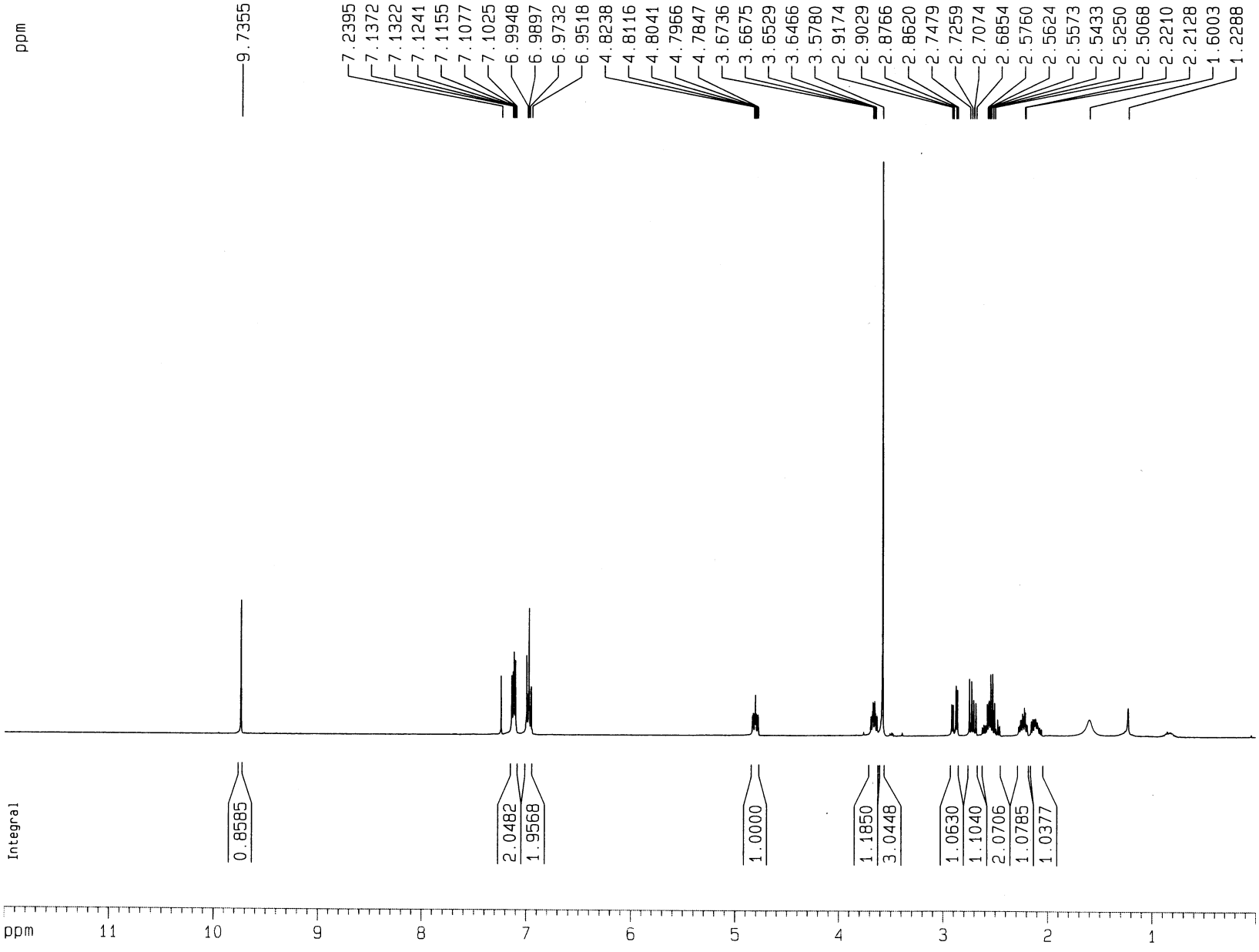
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127700 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S79. 1H NMR (CDCl3, 400 MHz) of compound anti-3b



Current Data Parameters

NAME	LCH-2-375-p2
EXPNO	8
PROCNO	1

F2 - Acquisition Parameters

Date_	20150318
Time	21.05
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	2896.3
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

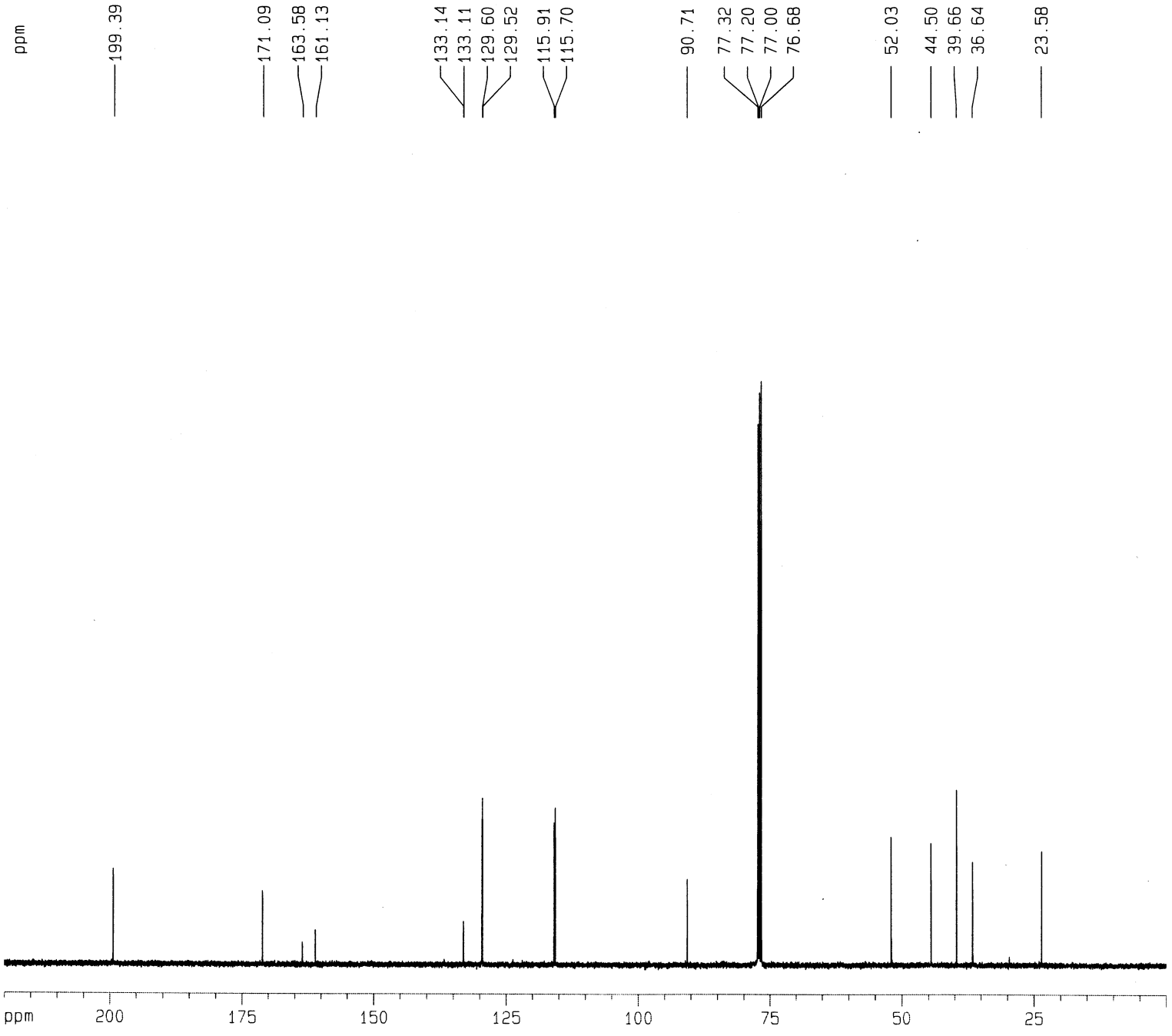
SI	8192
SF	400.1300179 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	0.55814 ppm/cm
HZCM	223.32838 Hz/cm

C13 spectrum of

Fig S80. 13C NMR (CDCl3, 100 MHz) of compound anti-3b




```
Current Data Parameters
NAME        LCH-2-375-p2
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150319
Time                3.12
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

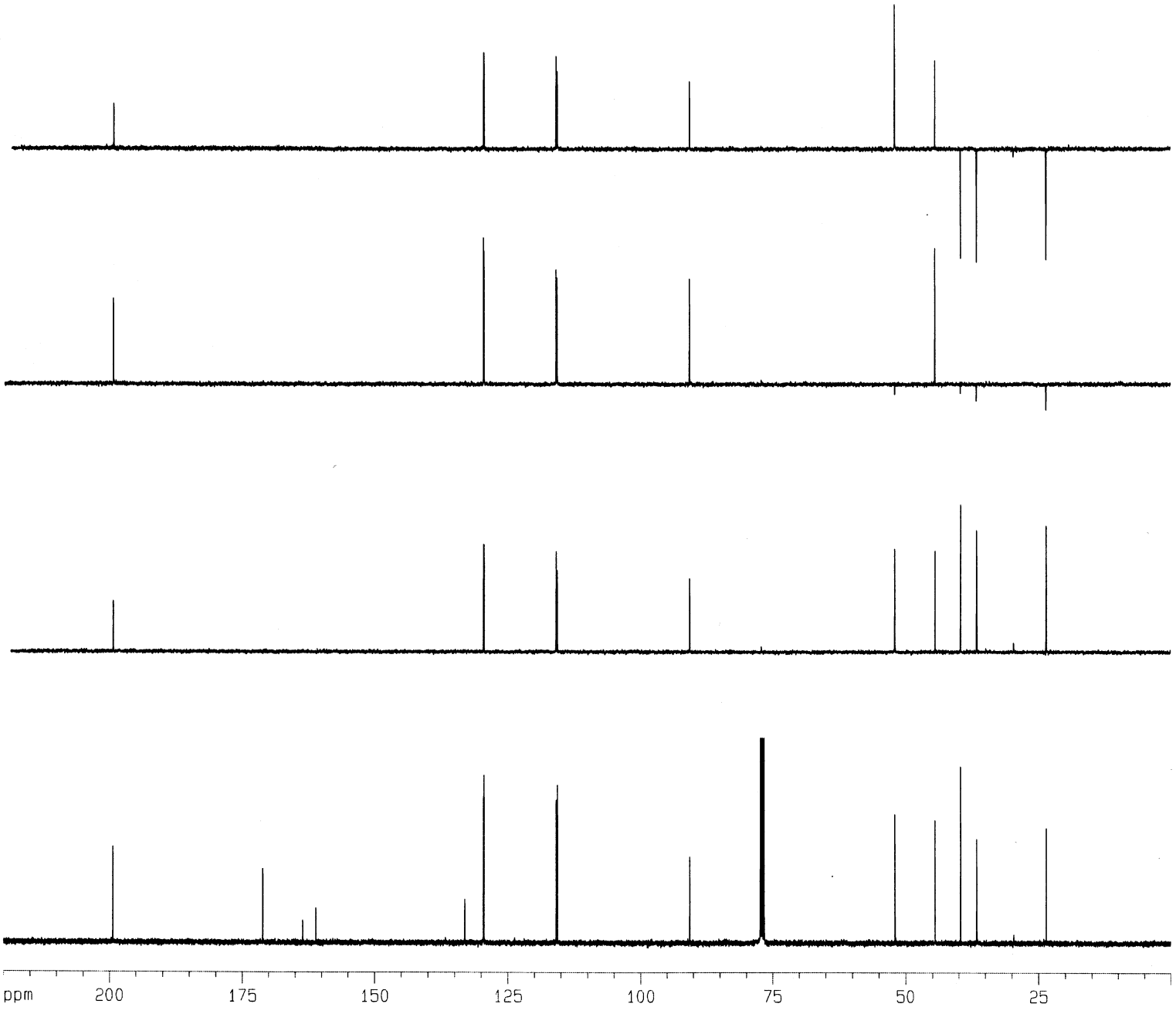
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127723 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```

Fig S81. DEPT of compound anti-3b




```
Current Data Parameters
NAME        LCH-2-375-p2
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150319
Time                3.12
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3276
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

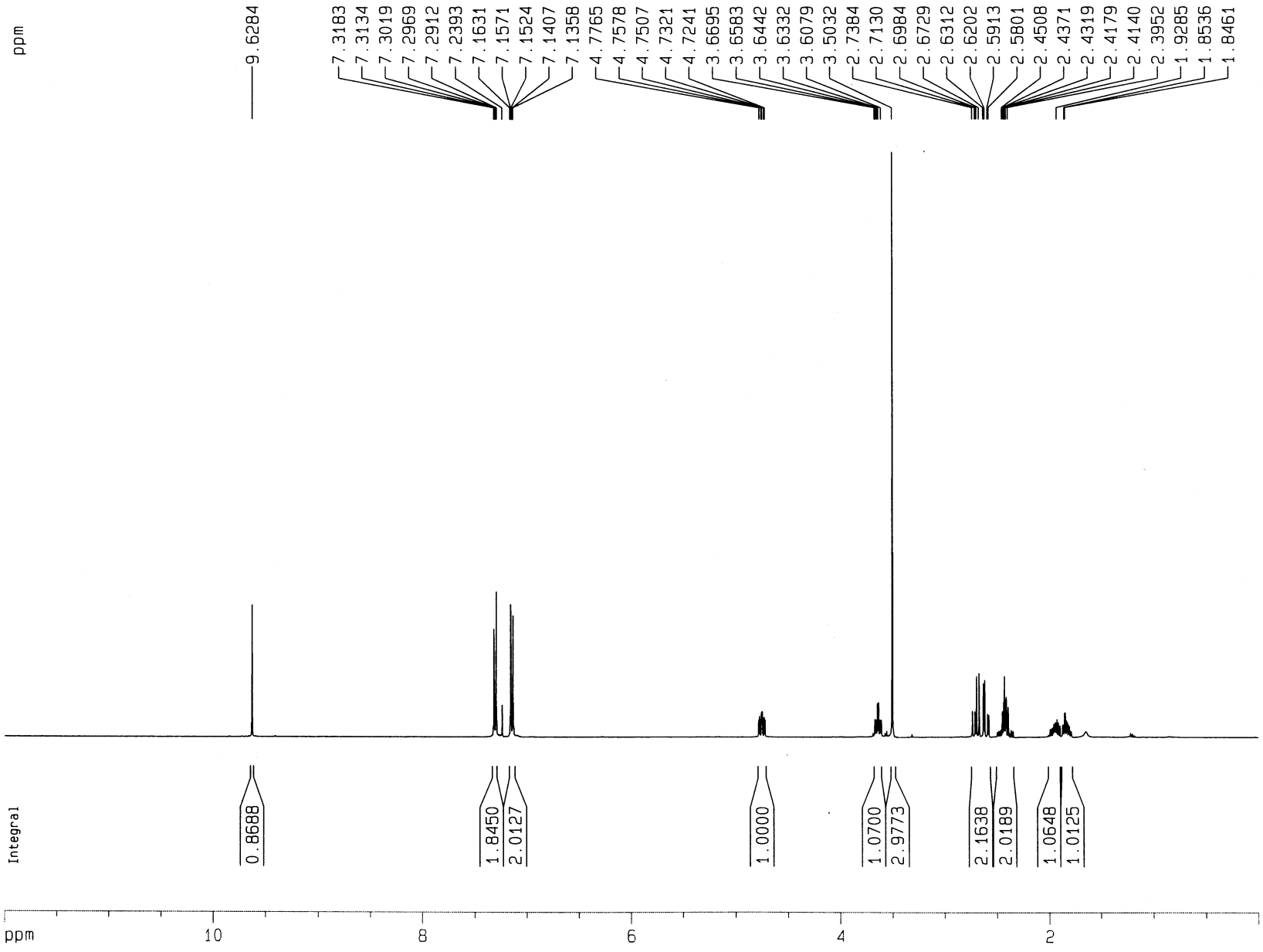
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127723 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S82. 1H NMR (CDCl3, 400 MHz) of compound syn-3c



Current Data Parameters

NAME	LCH-2-381-p1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20150320
Time	0.23
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	1290.2
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

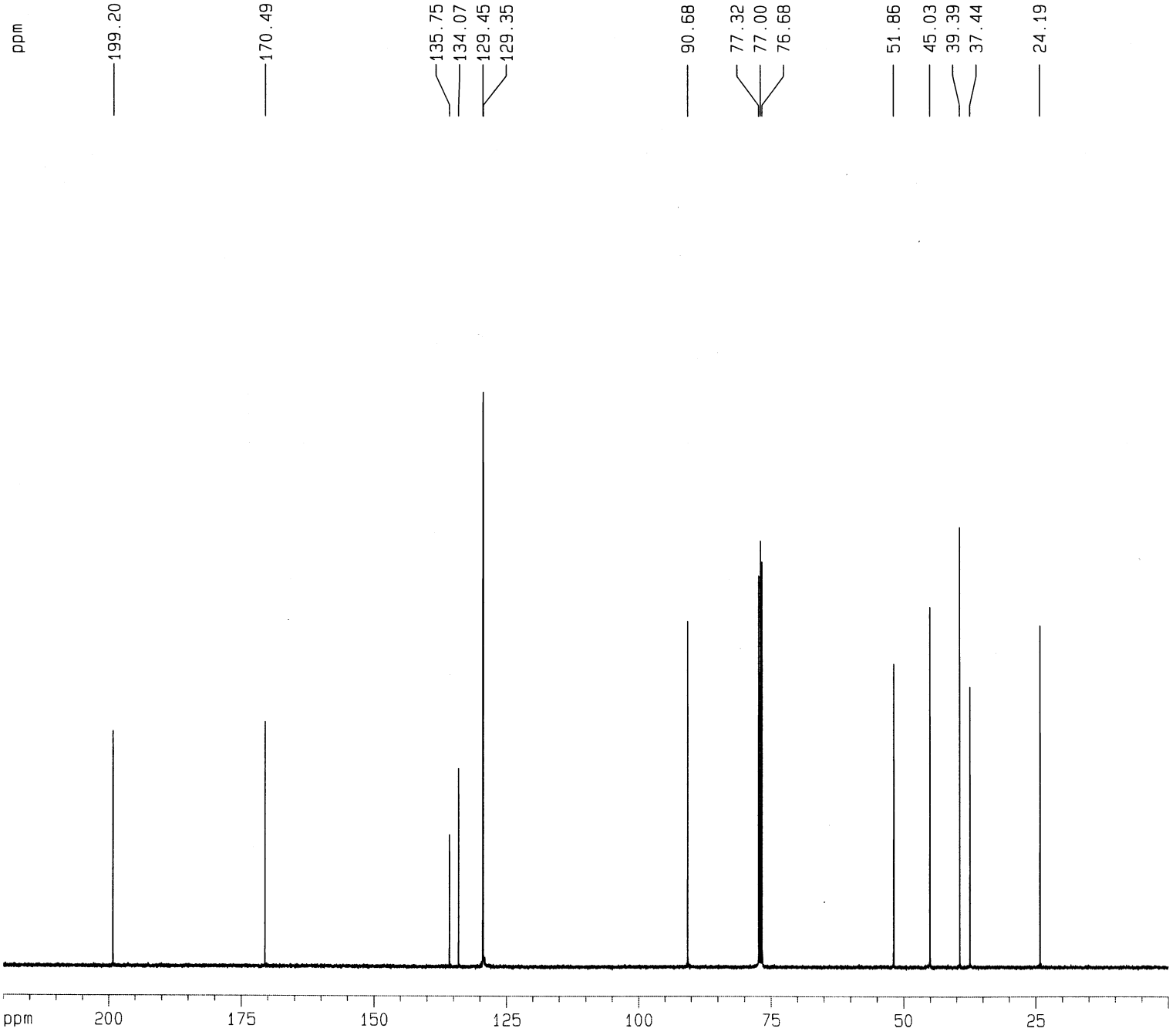
SI	8192
SF	400.1300179 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	0.55814 ppm/cm
HZCM	223.32838 Hz/cm

C13 spectrum of

Fig S83. 13C NMR (CDCl3, 100 MHz) of compound syn-3c




```
Current Data Parameters
NAME        LCH-2-381-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150320
Time                3.36
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3481
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

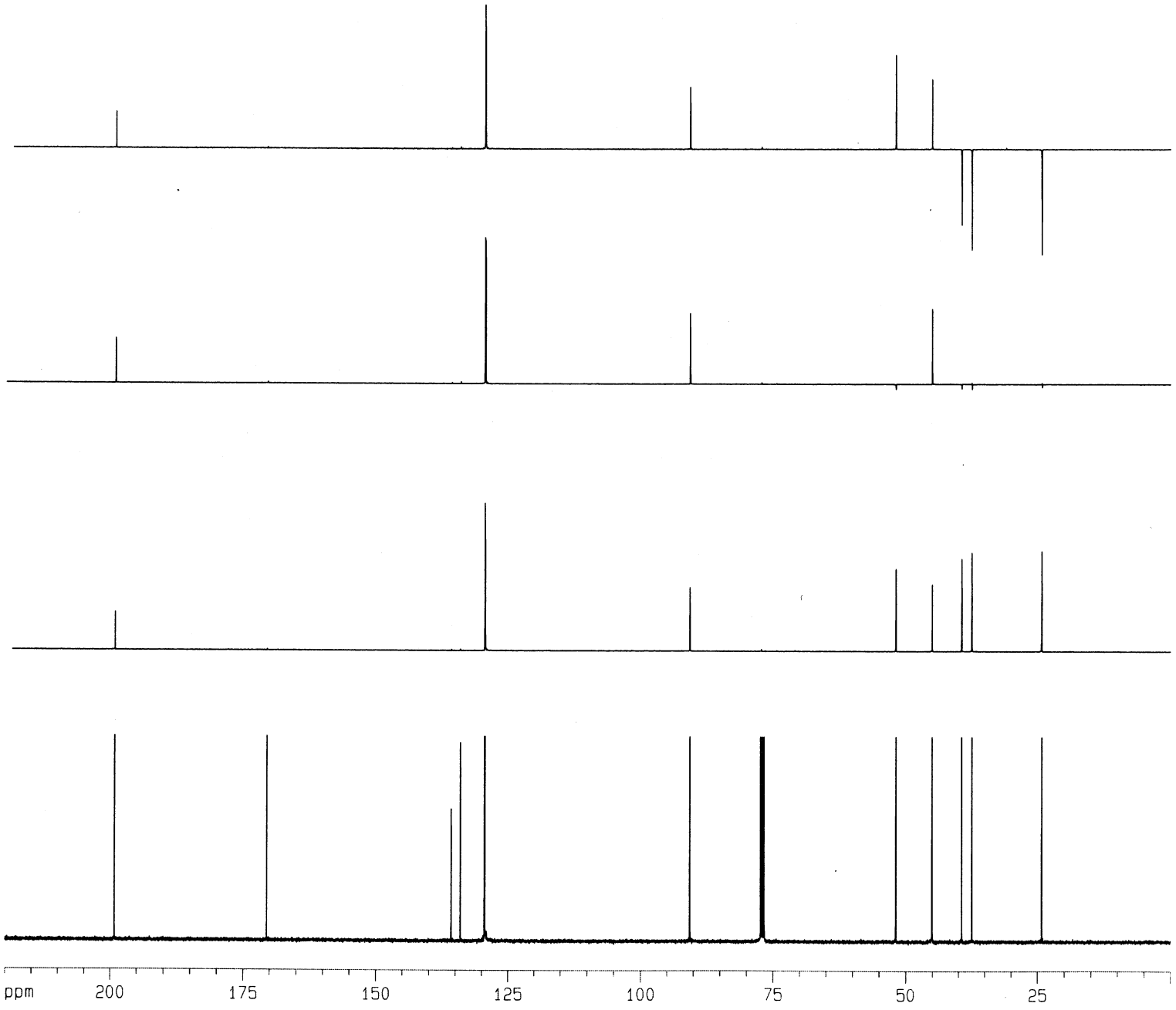
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127754 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM          1106.74060 Hz/cm
```

Fig S84. DEPT of compound syn-3c




```
Current Data Parameters
NAME        LCH-2-381-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150320
Time                3.36
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3481
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

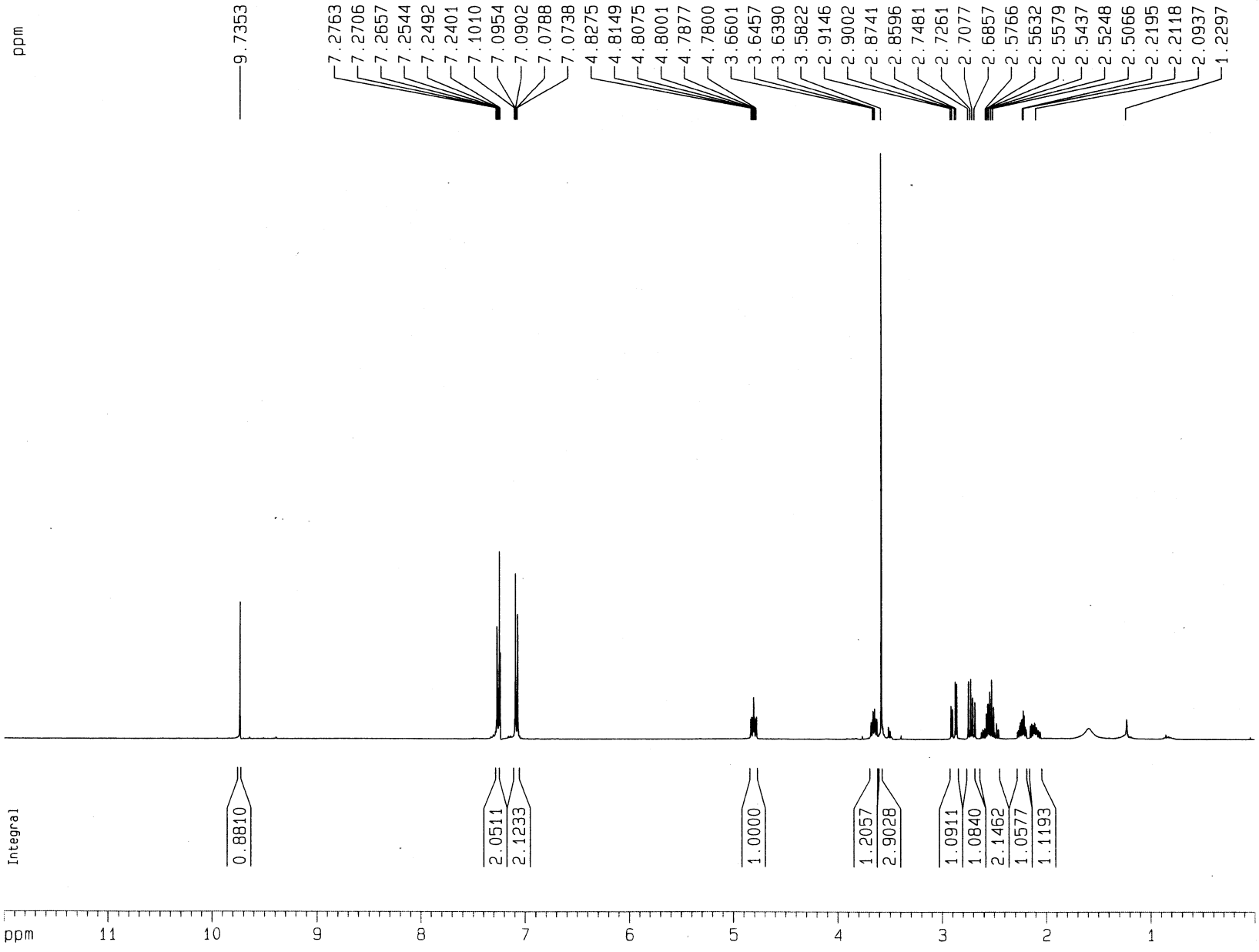
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127754 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S85. 1H NMR (CDCl3, 400 MHz) of compound anti-3c




```
Current Data Parameters
NAME        LCH-2-381-p2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150327
Time                1.03
INSTRUM            spect
PROBHD      5 mm GNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG                  4096
DW              83.400 usec
DE               6.50 usec
TE              300.0 K
D1           1.50000000 sec

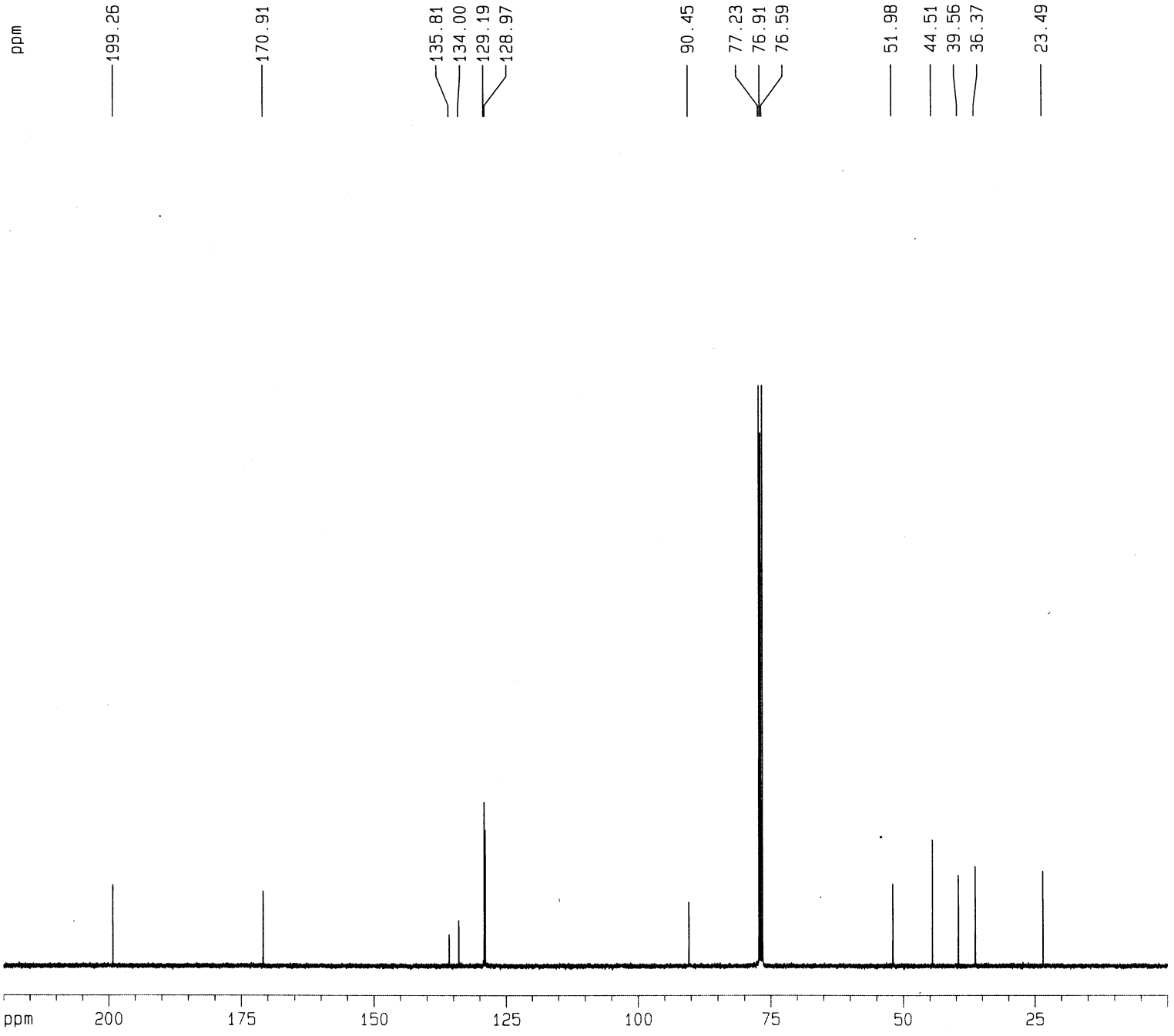
============ CHANNEL f1 =============
NUC1                  1H
P1              11.90 usec
PL1             -3.00 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF          400.1300179 MHz
WDW                   EM
SSB                    0
LB              0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                21.50 cm
F1P               12.000 ppm
F1               4801.56 Hz
F2P              -0.000 ppm
F2                -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM          223.32838 Hz/cm
```

C13 spectrum of

Fig S86. 13C NMR (CDCl3, 100 MHz) of compound anti-3c




```
Current Data Parameters
NAME        LCH-2-381-p2
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150327
Time                4.05
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW               19.900 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

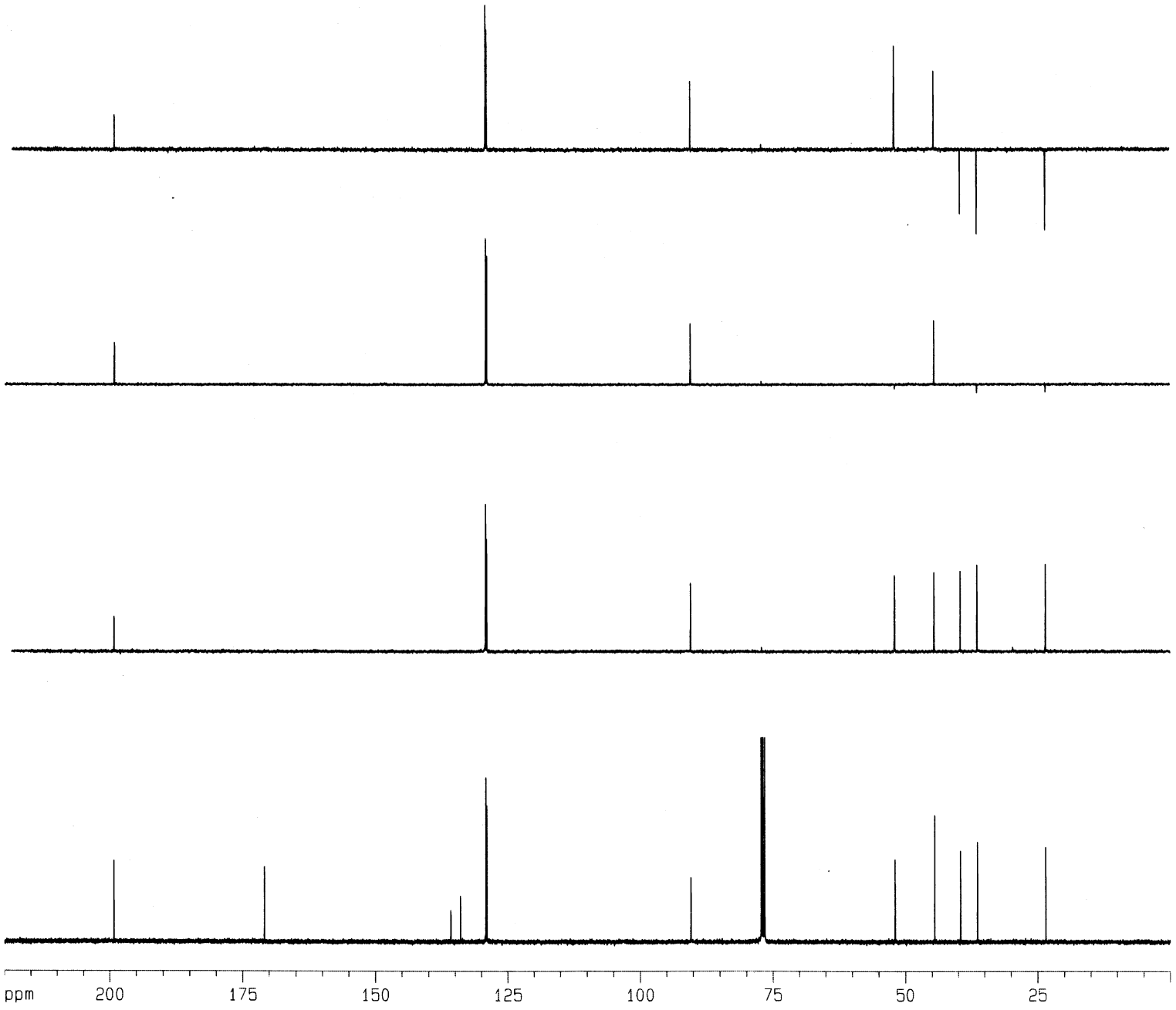
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127807 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1              22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74060 Hz/cm
```

Fig S87. DEPT of compound anti-3c




```
Current Data Parameters
NAME        LCH-2-381-p2
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150327
Time                4.05
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

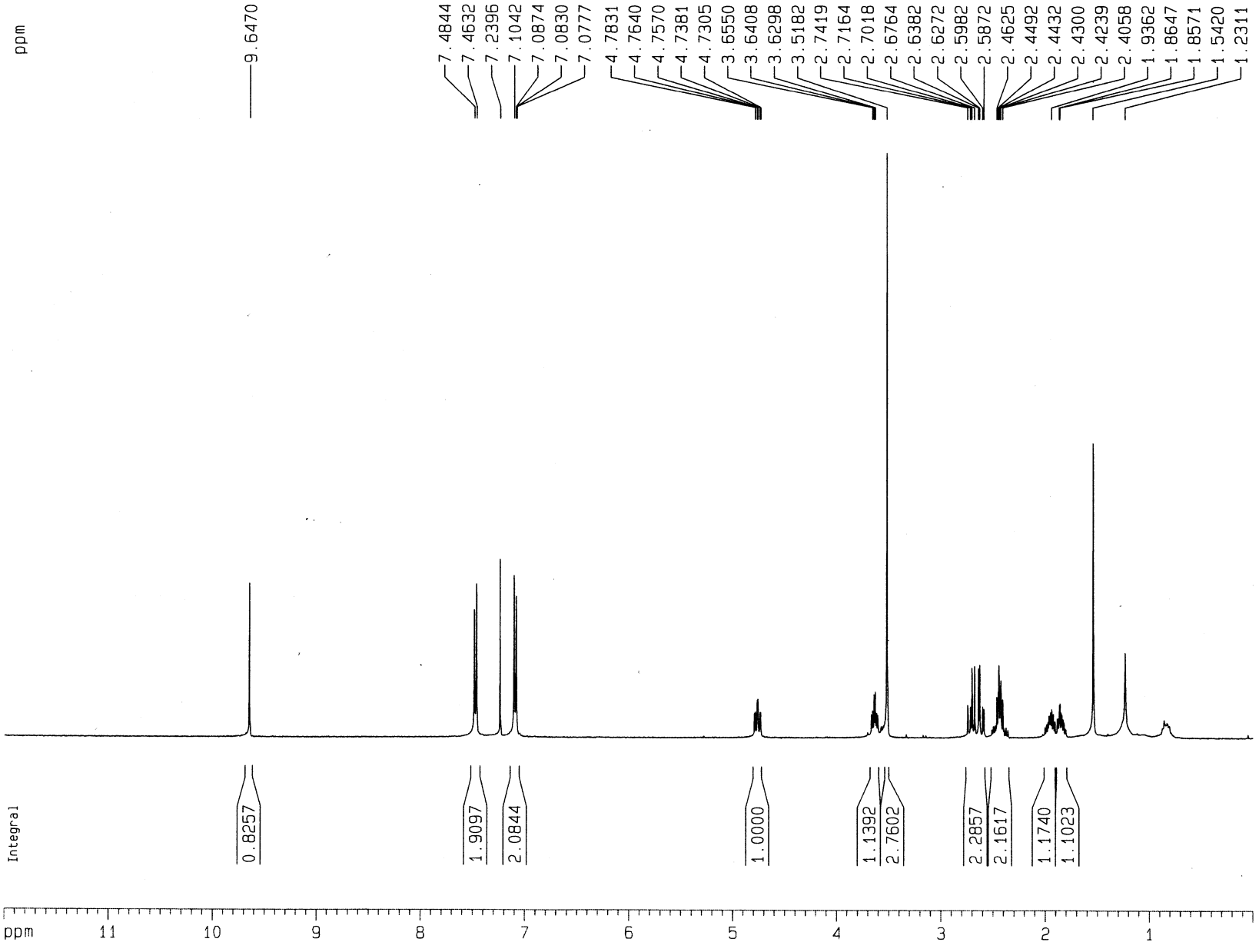
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127807 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S88. 1H NMR (CDCl3, 400 MHz) of compound syn-3d




```
Current Data Parameters
NAME        LCH-2-349-p1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141226
Time                0.23
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG                  6502
DW               83.400 usec
DE                6.50 usec
TE               300.0 K
D1           1.50000000 sec

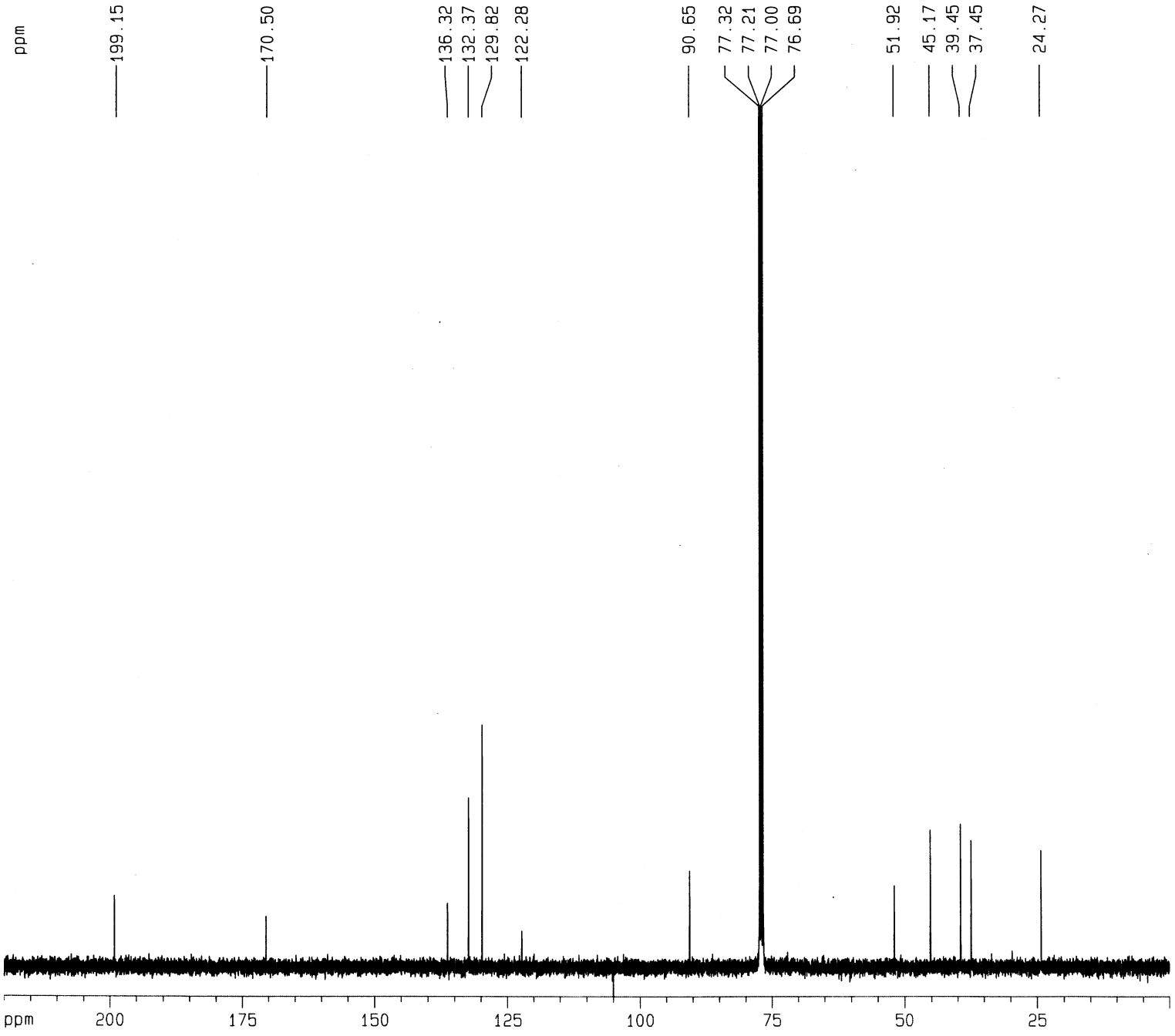
============ CHANNEL f1 =============
NUC1                  1H
P1                11.90 usec
PL1               -3.00 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF          400.1300179 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00

1D NMR plot parameters
CX                21.50 cm
F1P              12.000 ppm
F1              4801.56 Hz
F2P              -0.000 ppm
F2                -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM          223.32838 Hz/cm
```

C13 spectrum of

Fig S89. 13C NMR (CDCl3, 100 MHz) of compound syn-3d




```
Current Data Parameters
NAME        LCH-2-349-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141226
Time                3.25
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3276
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

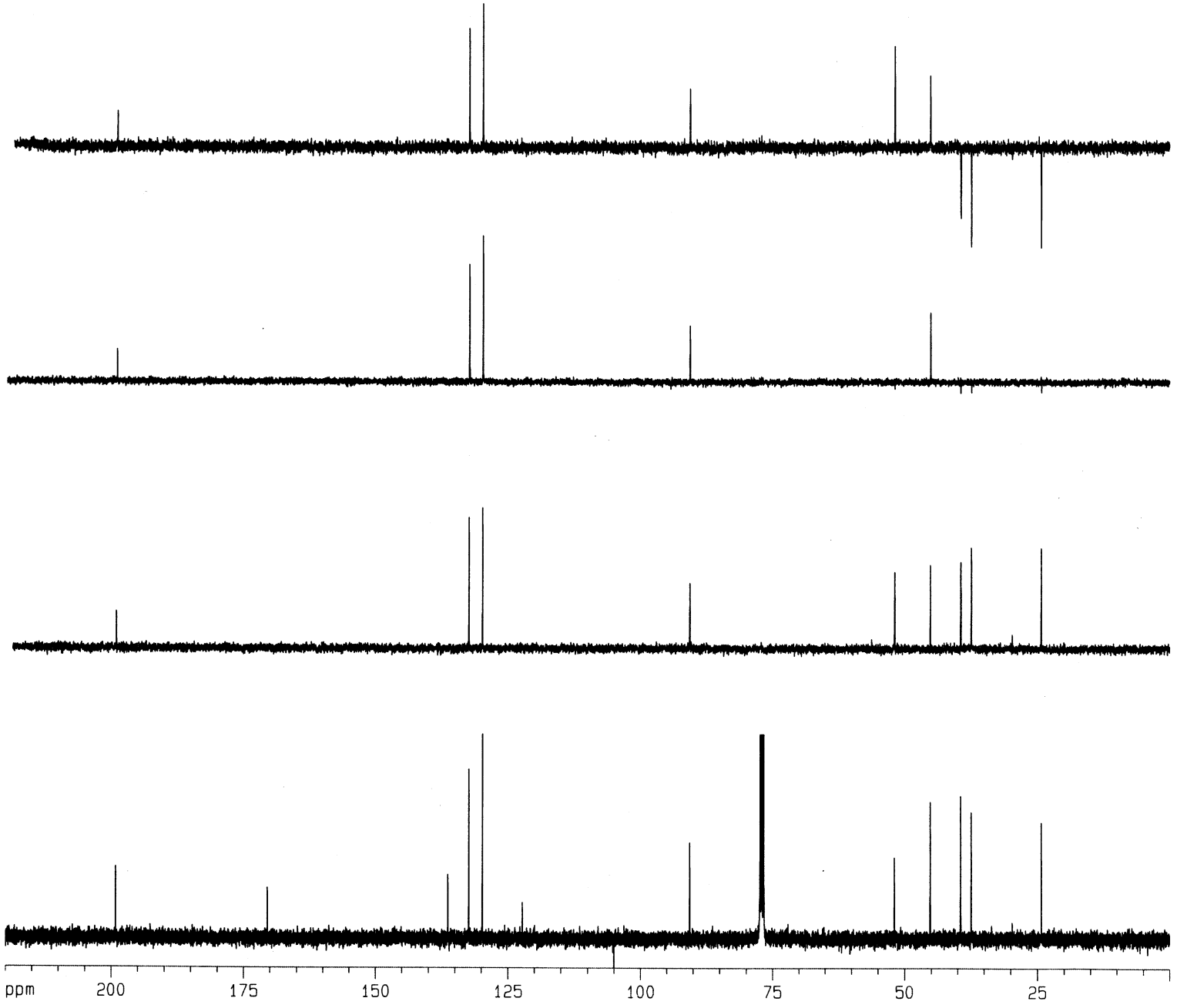
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
F1P             220.000 ppm
F1            22134.81 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM          1106.74048 Hz/cm
```

Fig S90. DEPT of compound syn-3d




```
Current Data Parameters
NAME        LCH-2-349-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141226
Time                3.25
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3276
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

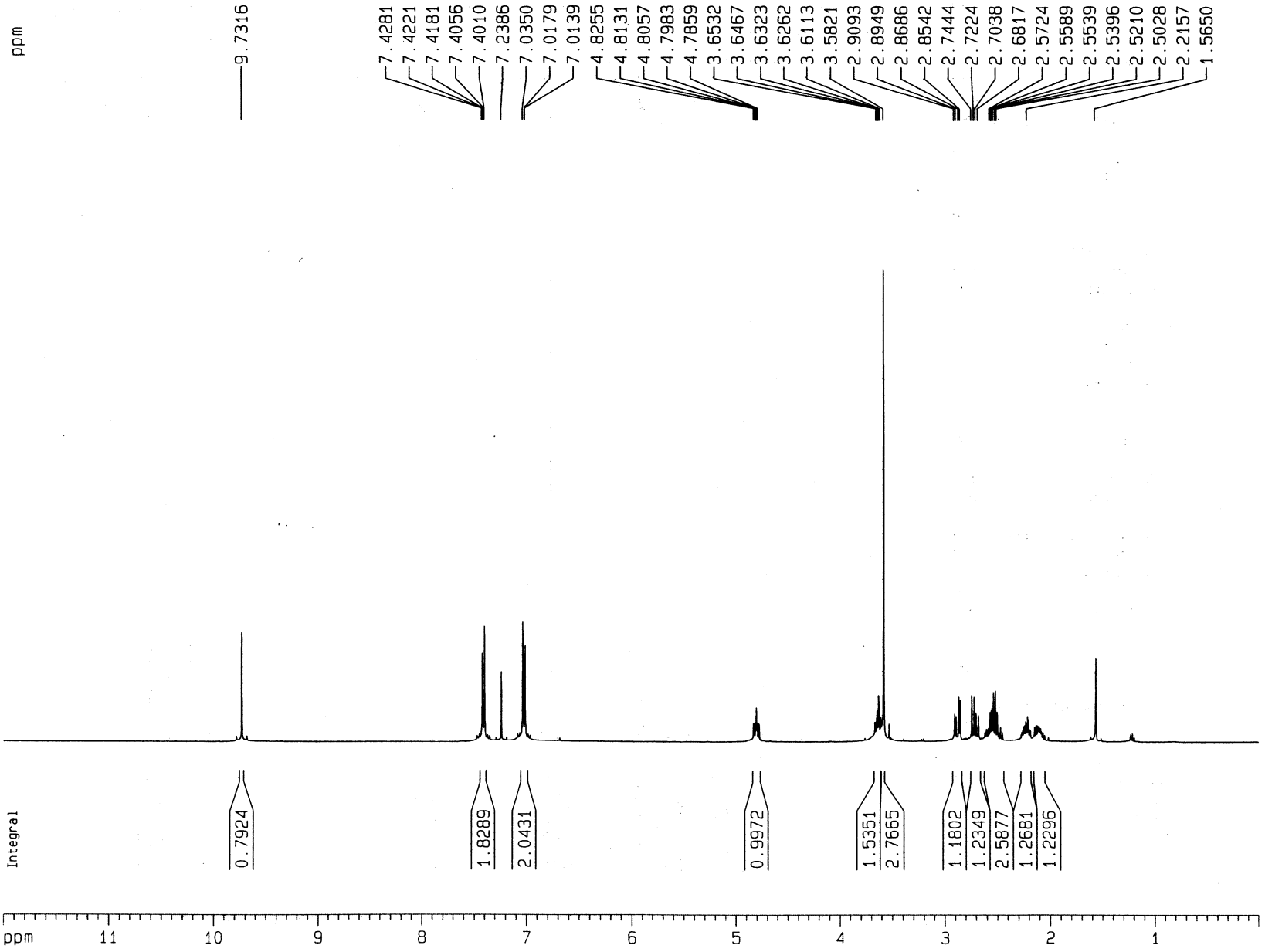
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S91. 1H NMR (CDCl3, 400 MHz) of compound anti-3d




```
Current Data Parameters
NAME        LCH-2-349-p2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141227
Time                0.00
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             5995.204 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                  3251
DW                83.400 usec
DE                 6.50 usec
TE                300.0 K
D1             1.50000000 sec

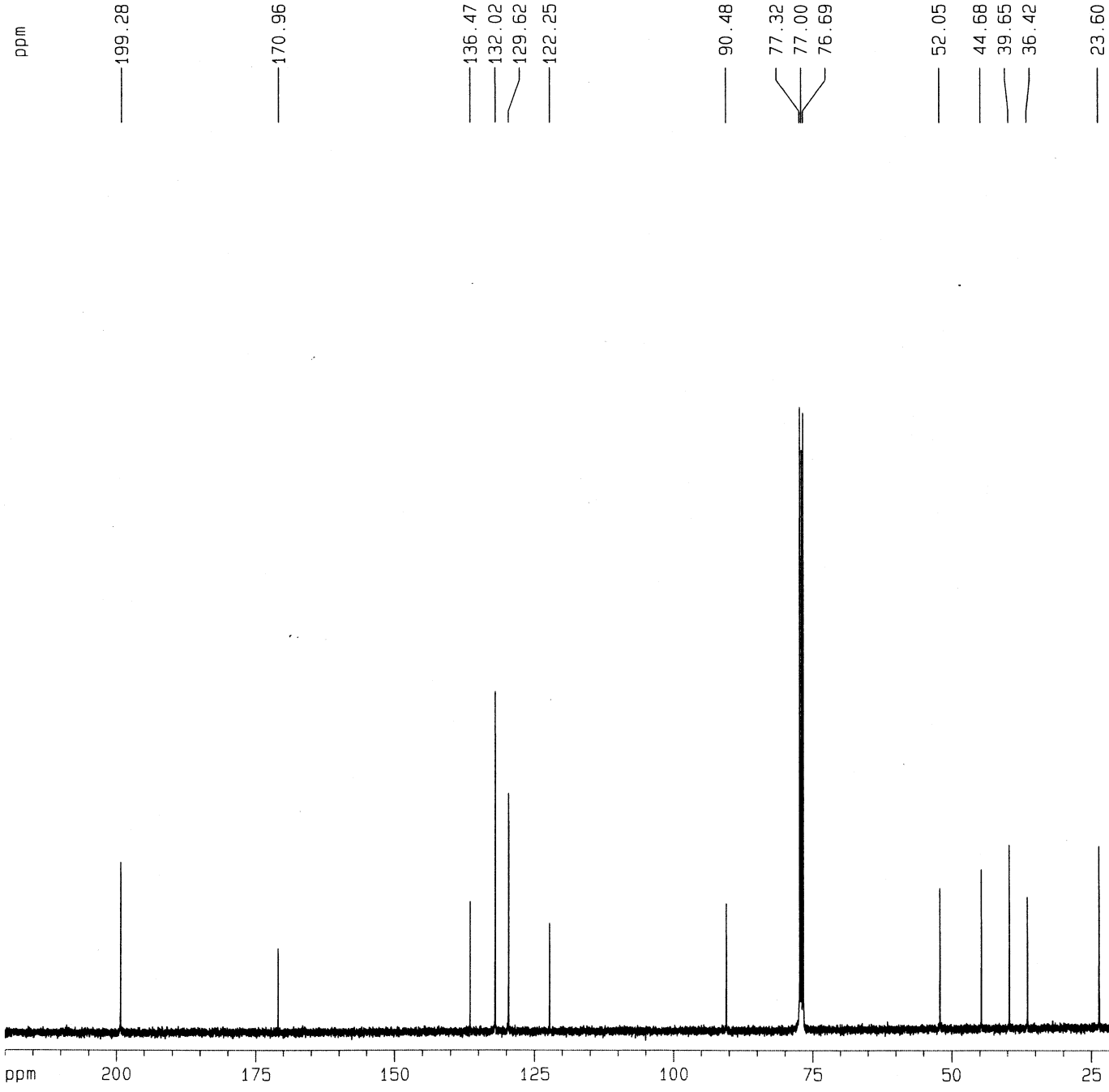
============ CHANNEL f1 =============
NUC1                  1H
P1                 11.90 usec
PL1                -3.00 dB
SFO1          400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF            400.1300179 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 21.50 cm
F1P                12.000 ppm
F1                4801.56 Hz
F2P               -0.000 ppm
F2                 -0.00 Hz
PPMCM           0.55814 ppm/cm
HZCM          223.32837 Hz/cm
```

C13 spectrum of

Fig S92. 13C NMR (CDCl3, 100 MHz) of compound anti-3d




```
Current Data Parameters
NAME        LCH-2-349-p2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141227
Time                3.02
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

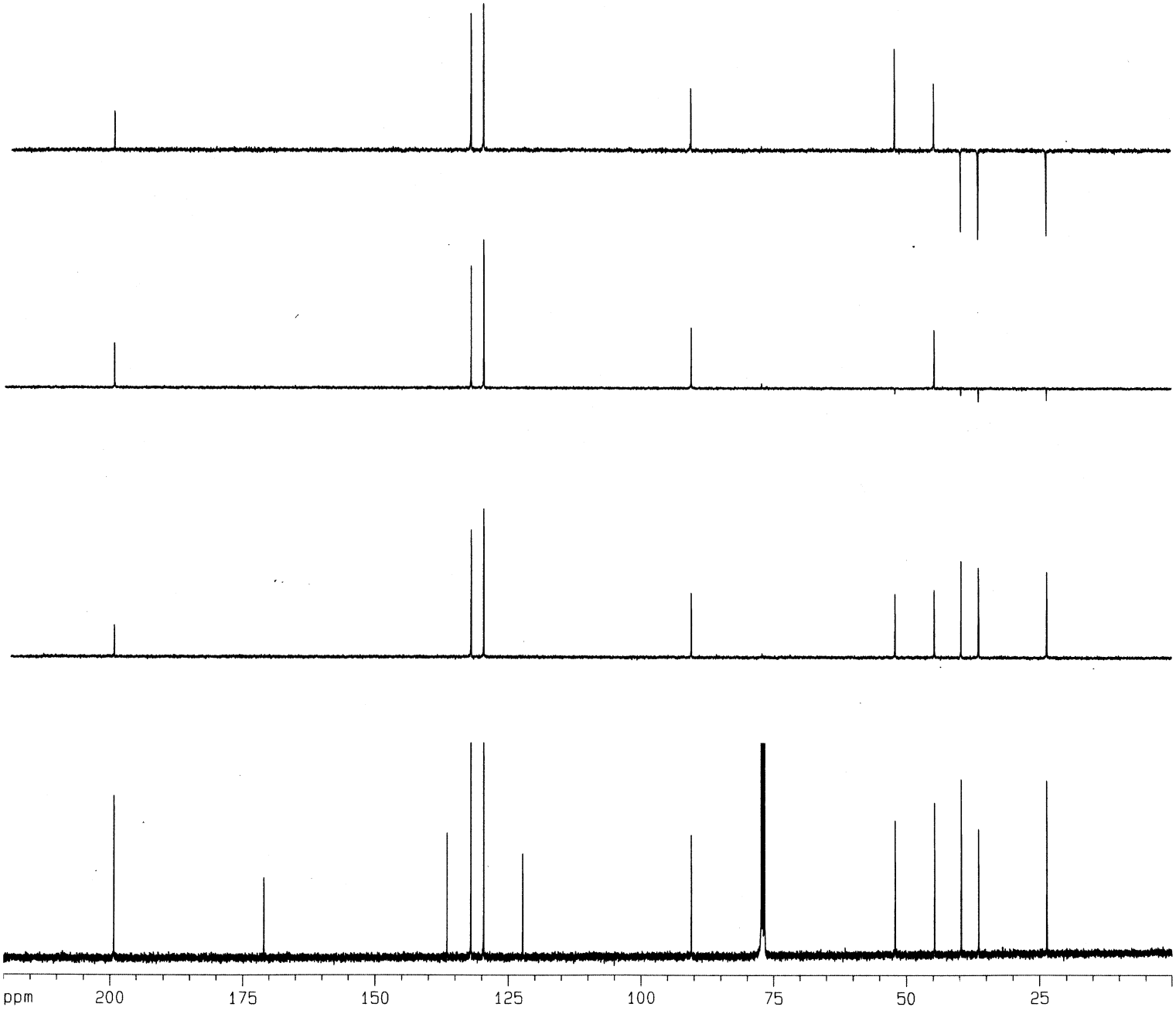
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SF01         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SF02         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127700 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S93. DEPT of compound anti-3d




```
Current Data Parameters
NAME        LCH-2-349-p2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141227
Time                3.02
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

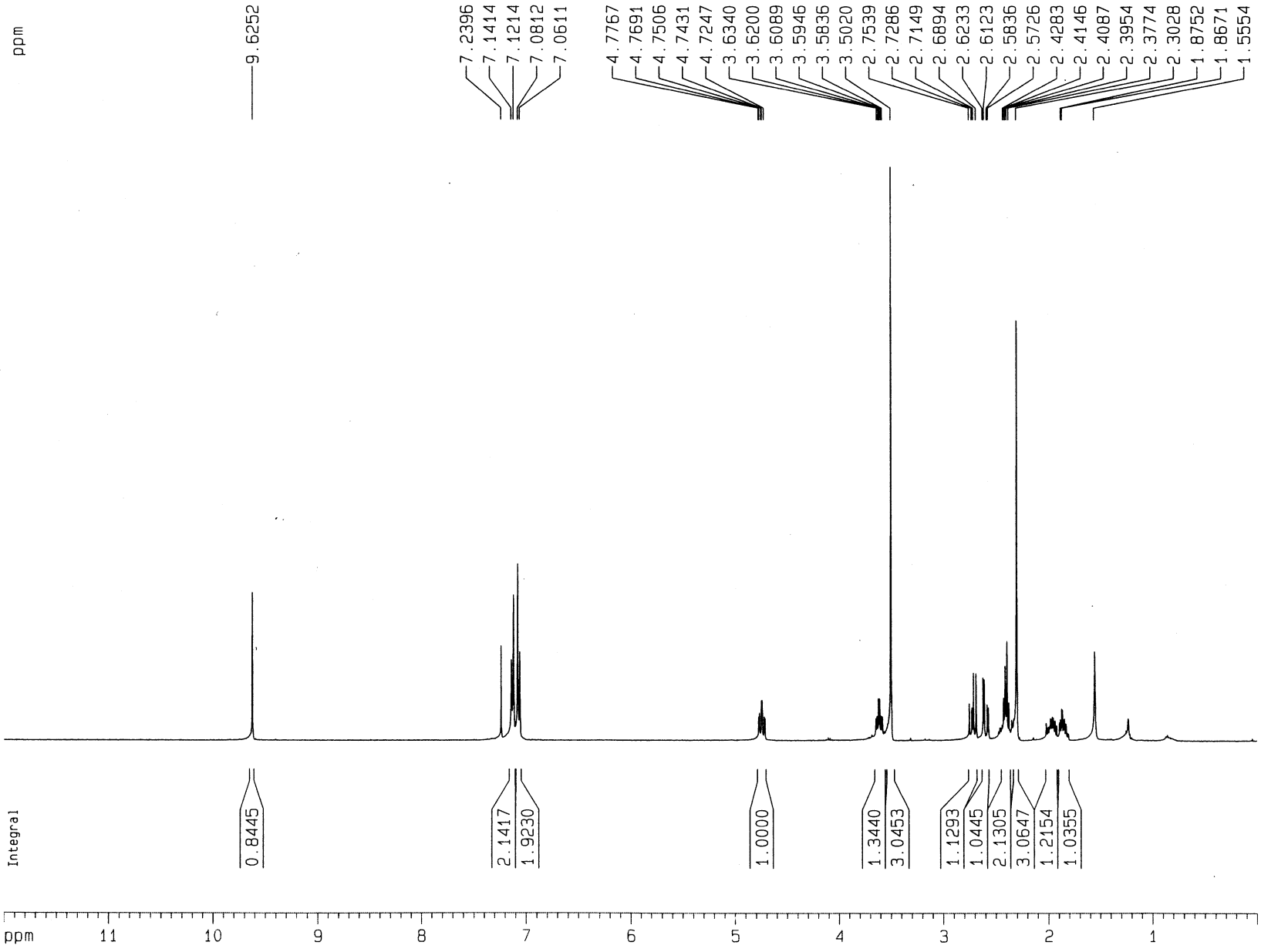
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127700 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S94. 1H NMR (CDCl3, 400 MHz) of compound syn-3e




```
Current Data Parameters
NAME                  LCH-2-353
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150110
Time                      20.21
INSTRUM                   spect
PROBHD          5 mm QNP  1H
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                5995.204 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                        4096
DW                  83.400 usec
DE                   6.50 usec
TE                   300.0 K
D1                1.50000000 sec

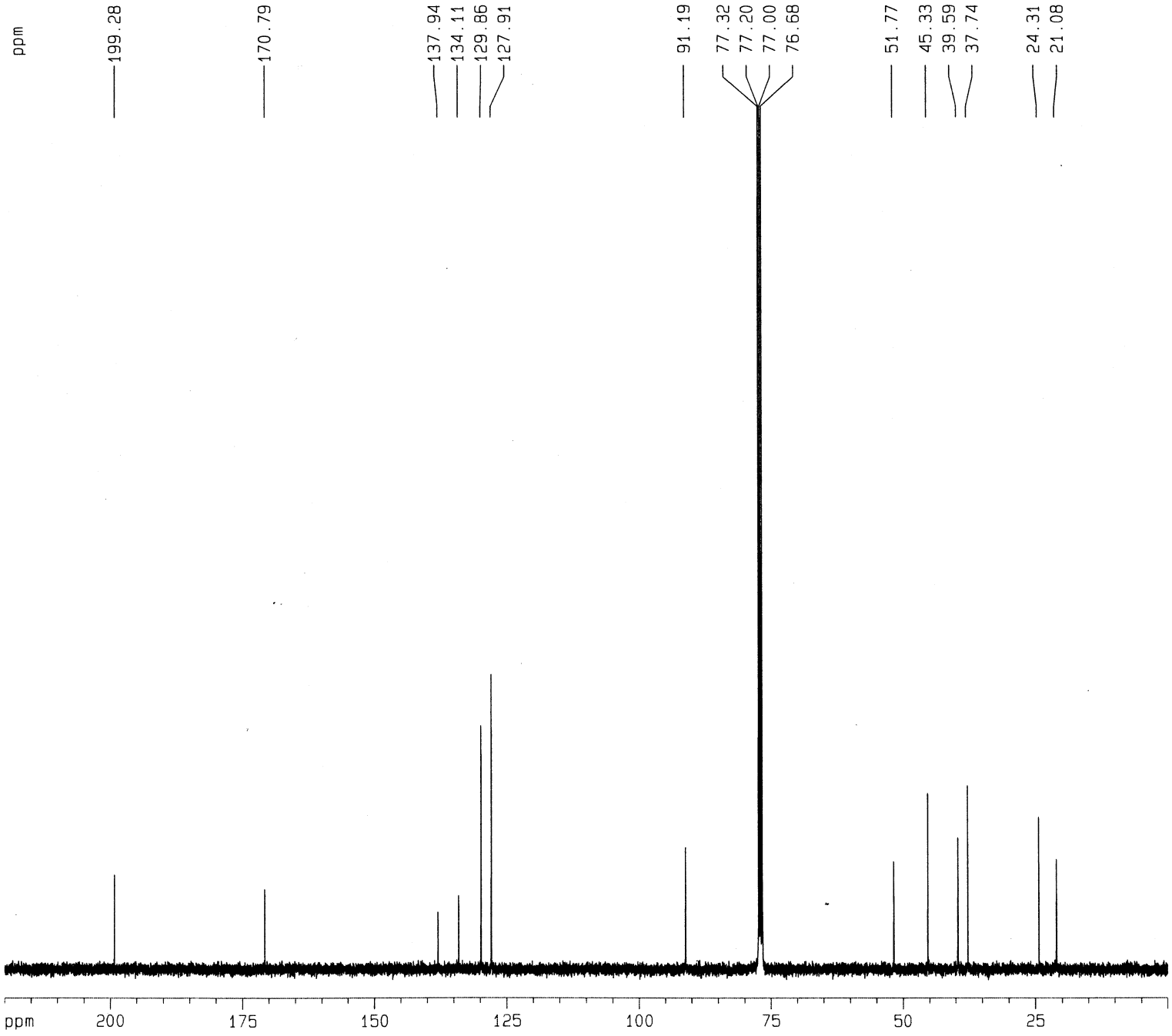
============ CHANNEL f1 =============
NUC1                         1H
P1                    11.90 usec
PL1                   -3.00 dB
SFO1           400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                400.1300179 MHz
WDW                         EM
SSB                          0
LB                    0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                    21.50 cm
F1P                  12.000 ppm
F1                  4801.56 Hz
F2P                 -0.000 ppm
F2                   -0.00 Hz
PPMCM              0.55814 ppm/cm
HZCM             223.32838 Hz/cm
```

C13 spectrum of

Fig S95. 13C NMR (CDCl3, 100 MHz) of compound syn-3e




```
Current Data Parameters
NAME          LCH-2-353
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150110
Time              23.46
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3686
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

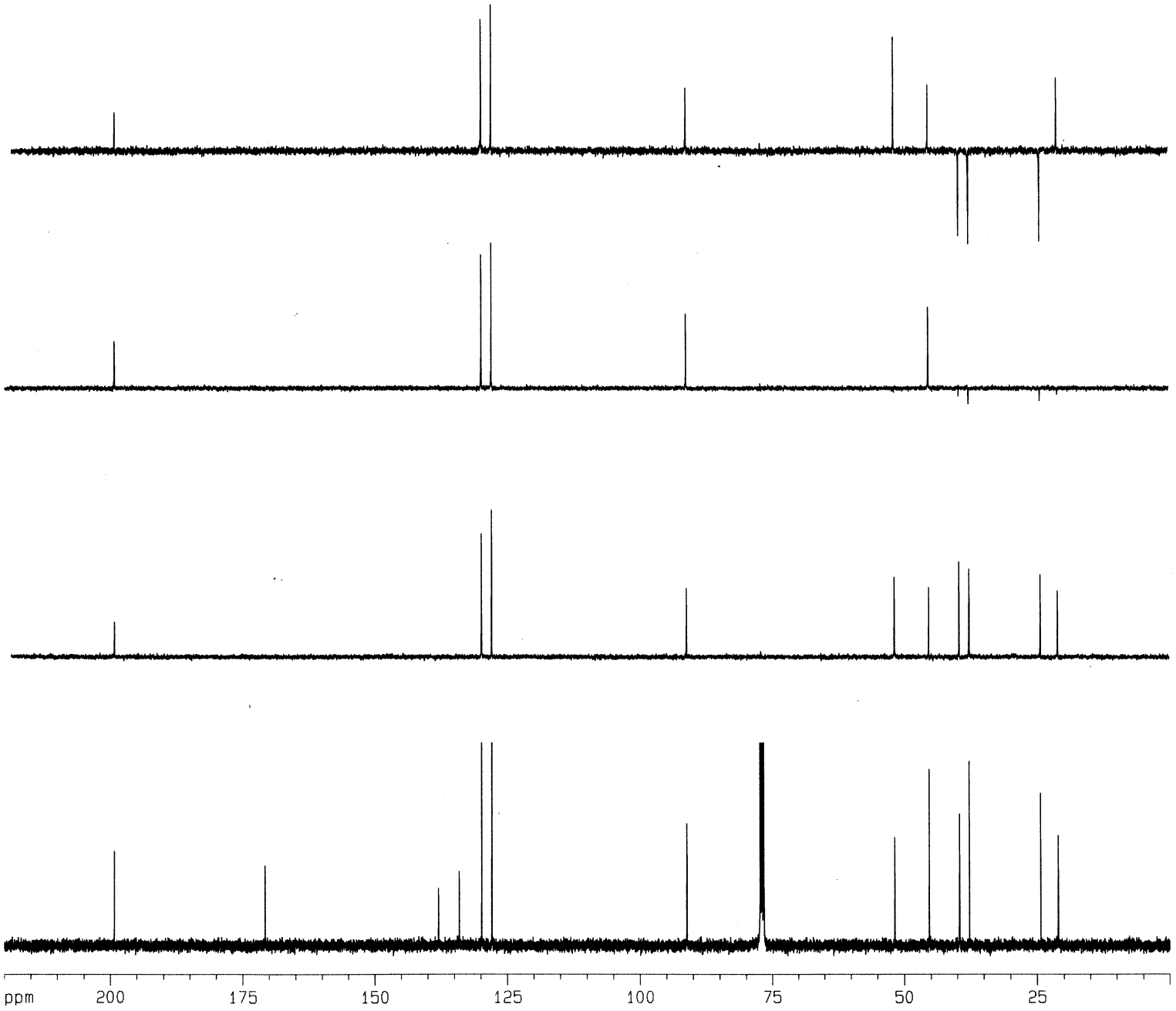
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127715 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
```

Fig S96. DEPT of compound syn-3e




```
Current Data Parameters
NAME                  LCH-2-353
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150110
Time                      23.46
INSTRUM                   spect
PROBHD     5 mm QNP  1H
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         3686
DS                            4
SWH                  25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                          256
DW                     19.900 usec
DE                      6.50 usec
TE                      300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

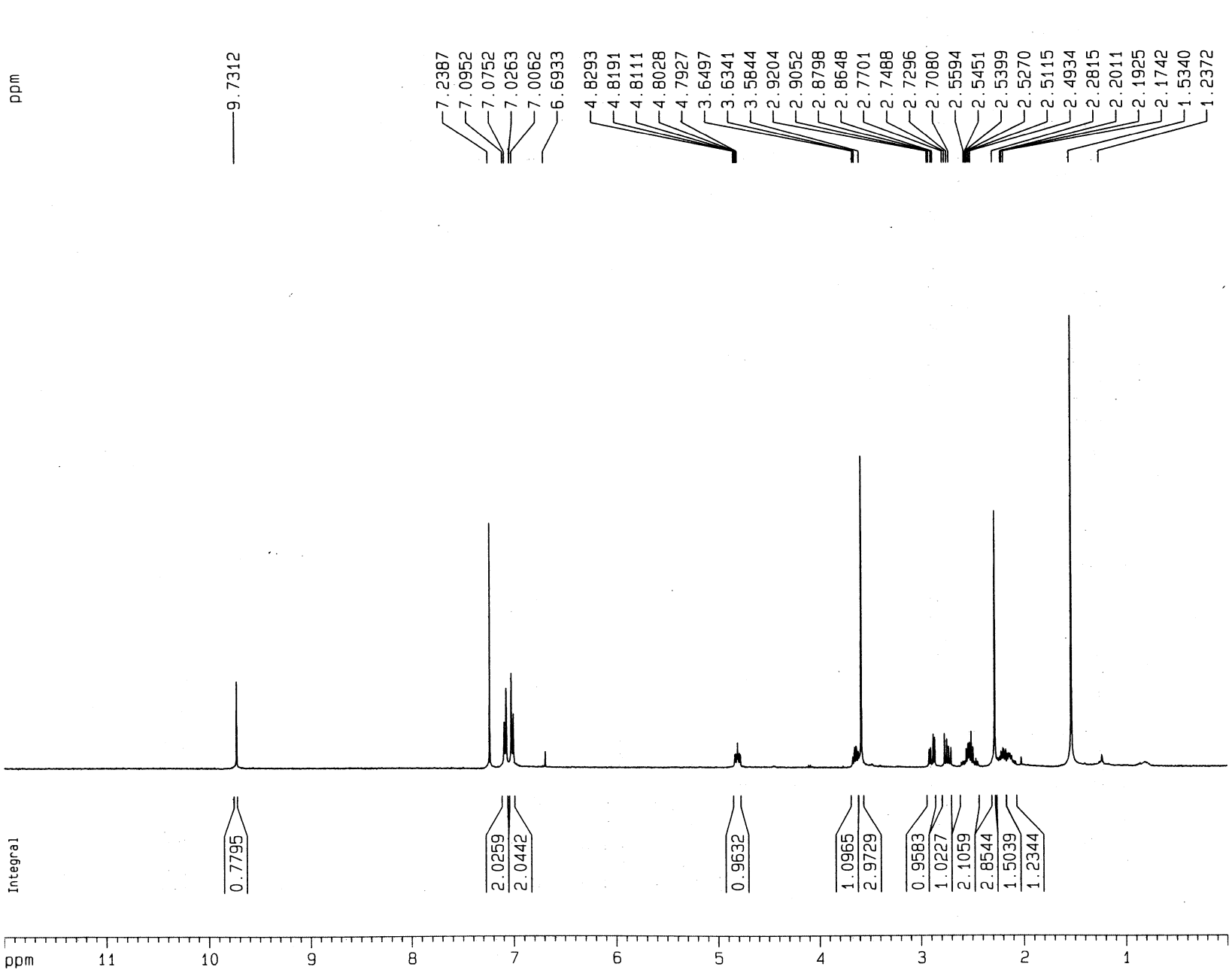
============ CHANNEL f1 =============
NUC1                        13C
P1                       10.20 usec
PL1                       0.00 dB
SFO1                100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     14.50 dB
PL13                     17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127715 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     220.000 ppm
F1                    22134.81 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 11.00000 ppm/cm
HZCM                1106.74048 Hz/cm
```

Fig S97. 1H NMR (CDCl3, 400 MHz) of compound anti-3e




```
Current Data Parameters
NAME        LCH-2-353-p2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150111
Time               17.32
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG                 13004
DW              83.400 usec
DE               6.50 usec
TE              300.0 K
D1           1.50000000 sec

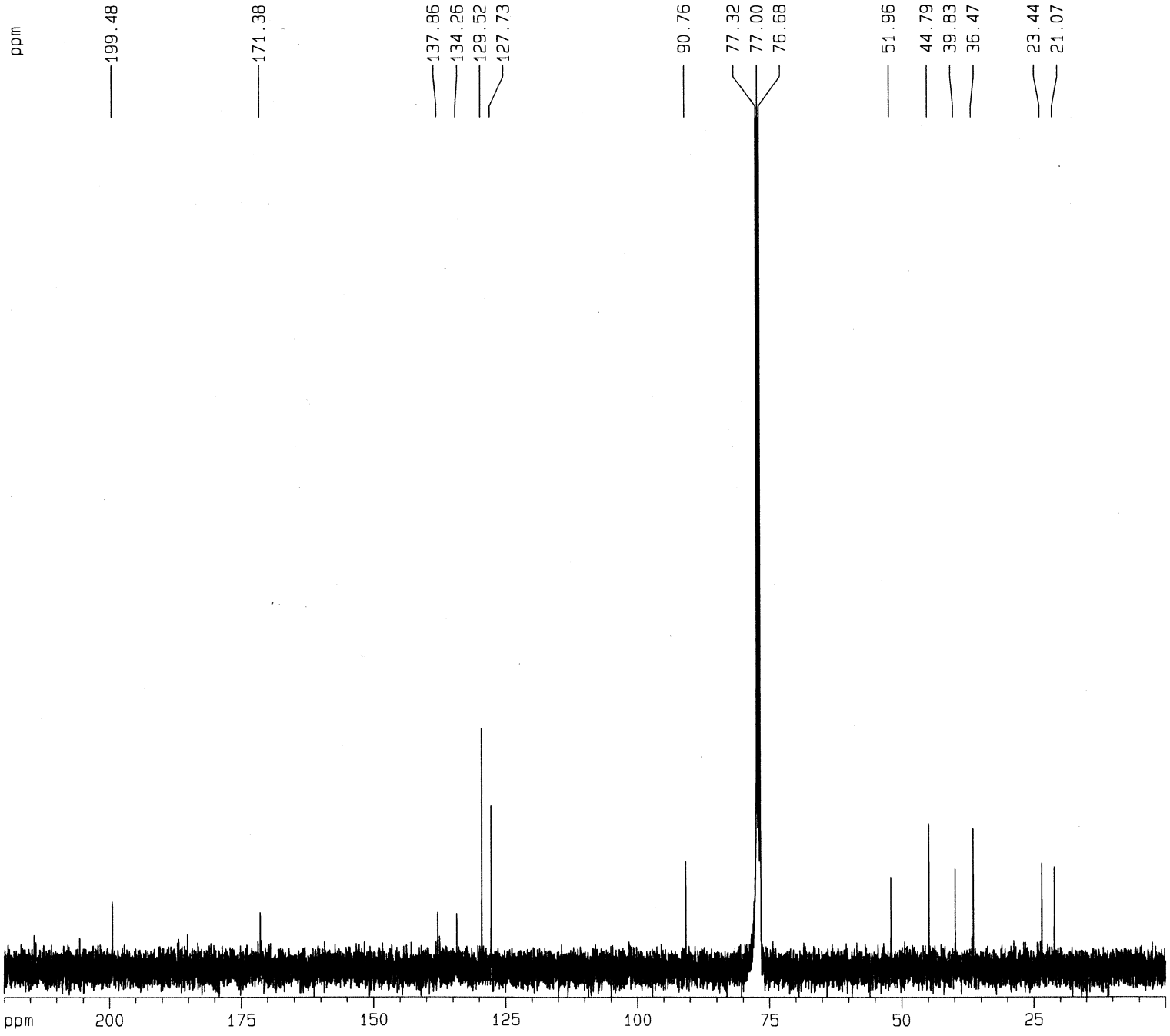
============ CHANNEL f1 =============
NUC1                  1H
P1                11.90 usec
PL1               -3.00 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF           400.1300179 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                21.50 cm
F1P               12.000 ppm
F1               4801.56 Hz
F2P               -0.000 ppm
F2                 -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM          223.32837 Hz/cm
```

C13 spectrum of

Fig S98. 13C NMR (CDCl3, 100 MHz) of compound anti-3e




```
Current Data Parameters
NAME        LCH-2-353-p2
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150115
Time                2.48
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW              19.900 usec
DE               6.50 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

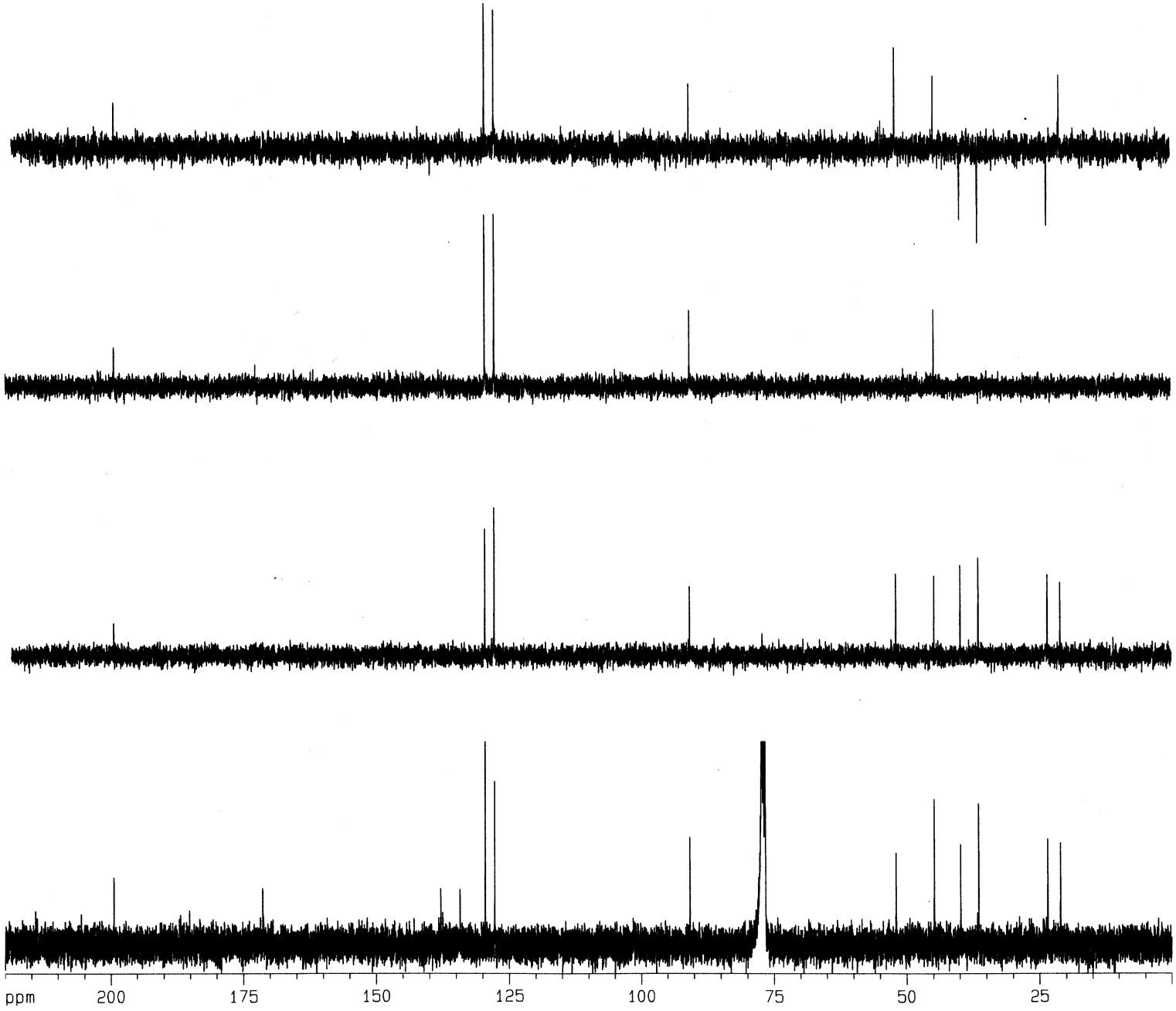
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SF01        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2           90.00 usec
PL2             -3.00 dB
PL12            14.50 dB
PL13            17.50 dB
SF02        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127708 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S99. DEPT of compound anti-3e




```
Current Data Parameters
NAME        LCH-2-353-p2
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150115
Time                2.48
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

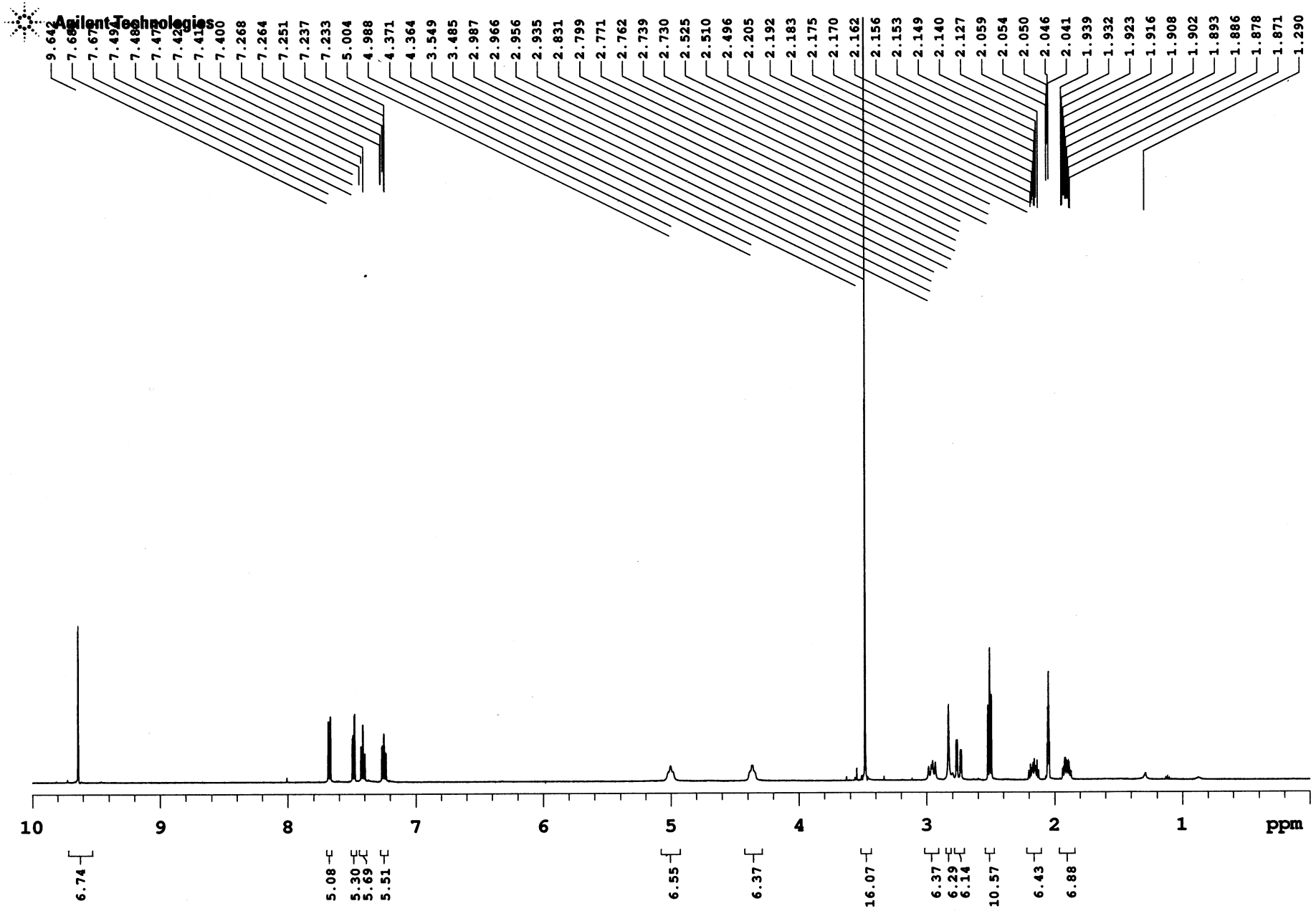
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127708 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

LCH-02-393-f1

Sample Name	**LCH-02-393-f1**	Pulse sequence	**s2pul**	Temperature	**50**	Study owner	**vnmr2**
Date collected	**2015-04-16**	Solvent	**Acetone**	Spectrometer	**---**	Operator	**vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-393/p1/dacetone/H1.fid

Fig S100. 1H NMR (acetone-d6, 500 MHz) of compound syn-3f

Plot date 2015-06-17

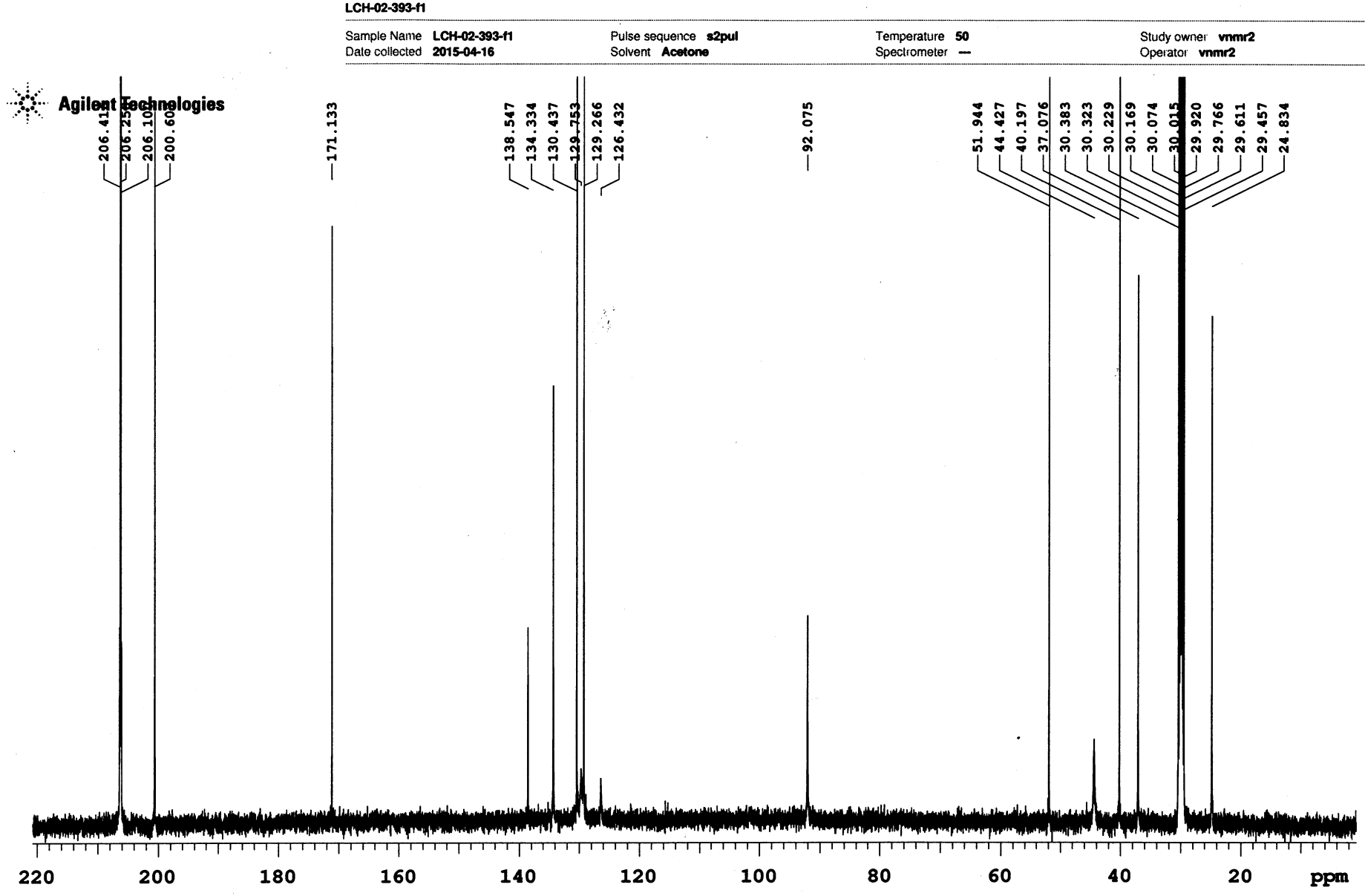


Fig S101. 13C NMR (acetone-d6 125 MHz) of compound syn-3f

LCH-02-393-f1

Sample Name **LCH-02-393-f1**	Pulse sequence **DEPT**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-17**	Solvent **Acetone**	Spectrometer **—**	Operator **vnmr2**

Agilent Technologies

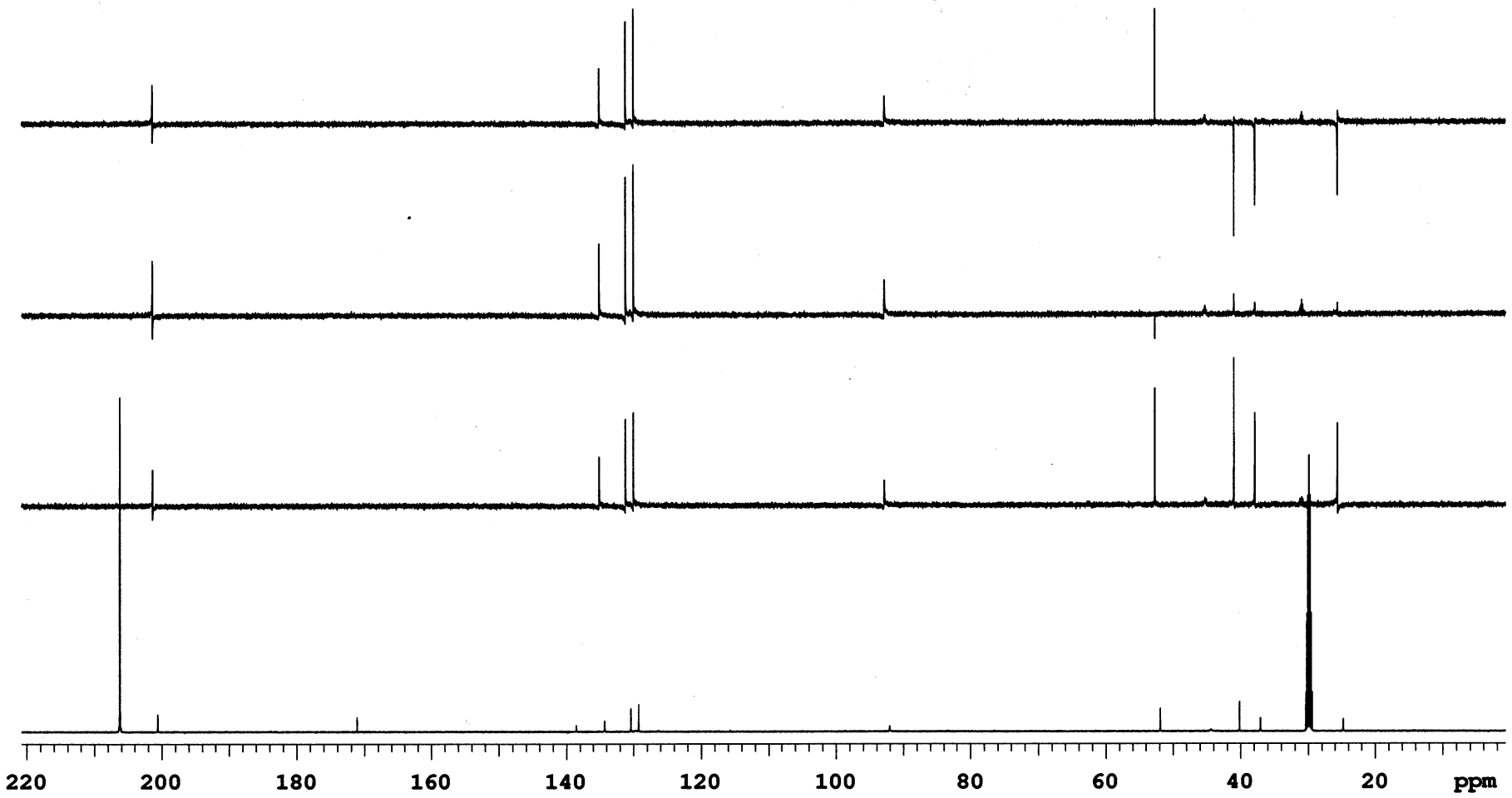
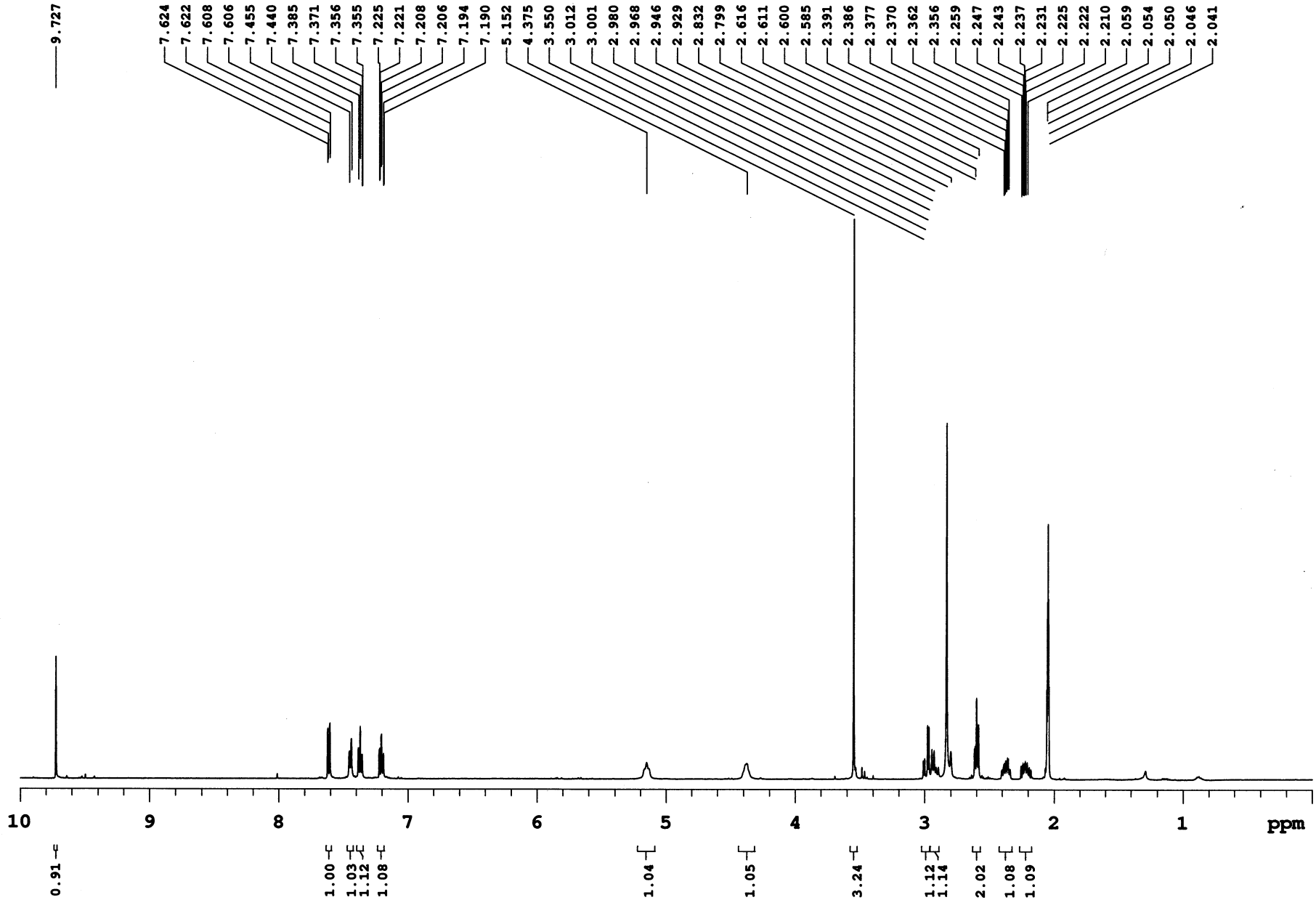


Fig S102. DEPT of compound syn-3f

LCH-02-393-p2

Sample Name **LCH-02-393-p2**	Pulse sequence **s2pul**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-20**	Solvent **Acetone**	Spectrometer **—**	Operator **vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-393/p2/dacetone/H1.fid

Fig S103. 1H NMR (acetone-d6, 500 MHz) of compound anti-3f

Plot date 2015-11-07

LCH-02-393-f2

Sample Name **LCH-02-393-f2**
Date collected **2015-04-20**
Pulse sequence **s2pul**
Solvent **Acetone**
Temperature **50**
Spectrometer **—**
Study owner **vnmr2**
Operator **vnmr2**

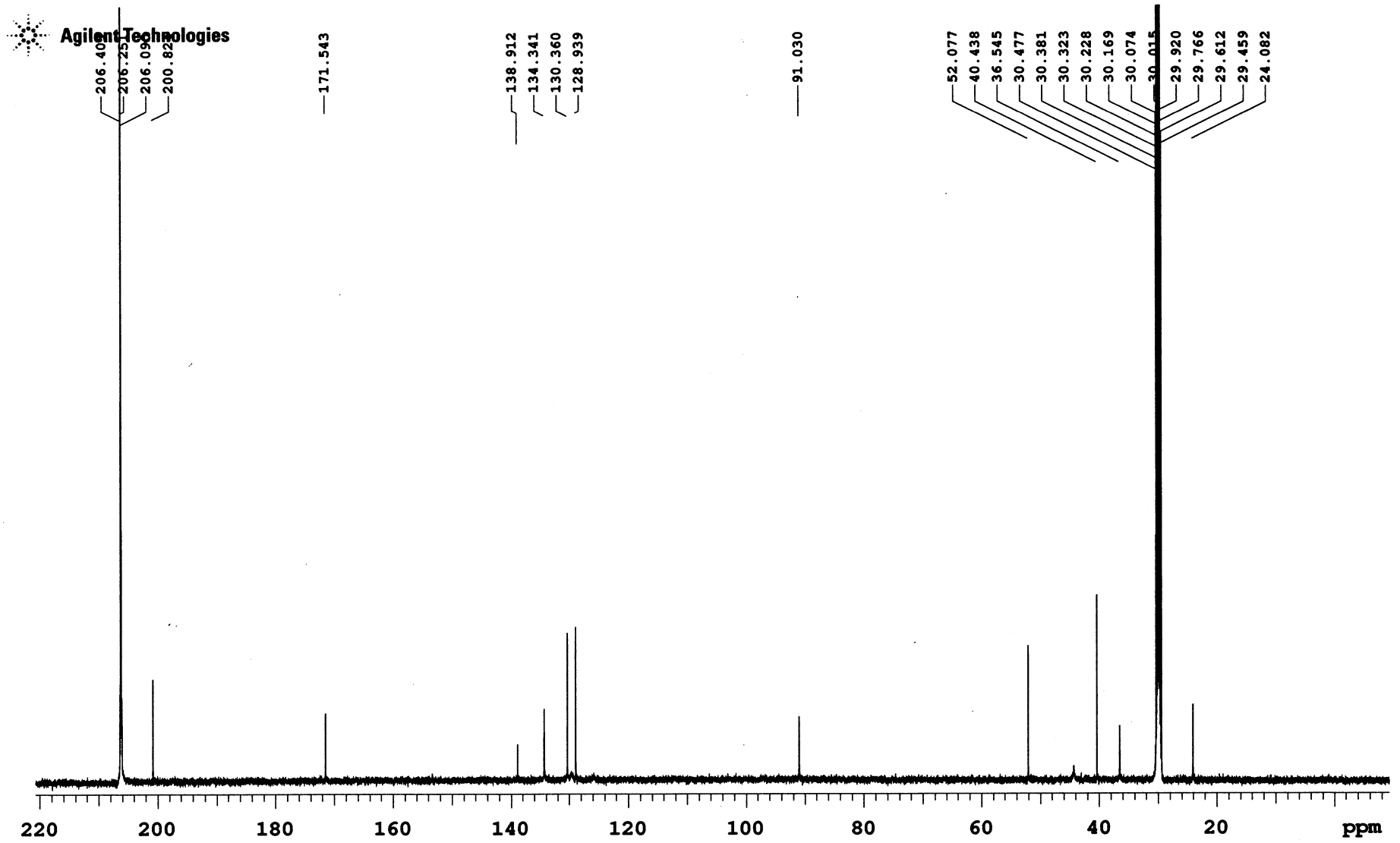


Fig S104. 13C NMR (acetone-d6, 125 MHz) of compound anti-3f

LCH-02-393-f2

Sample Name **LCH-02-393-f2**	Pulse sequence **DEPT**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-20**	Solvent **Acetone**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

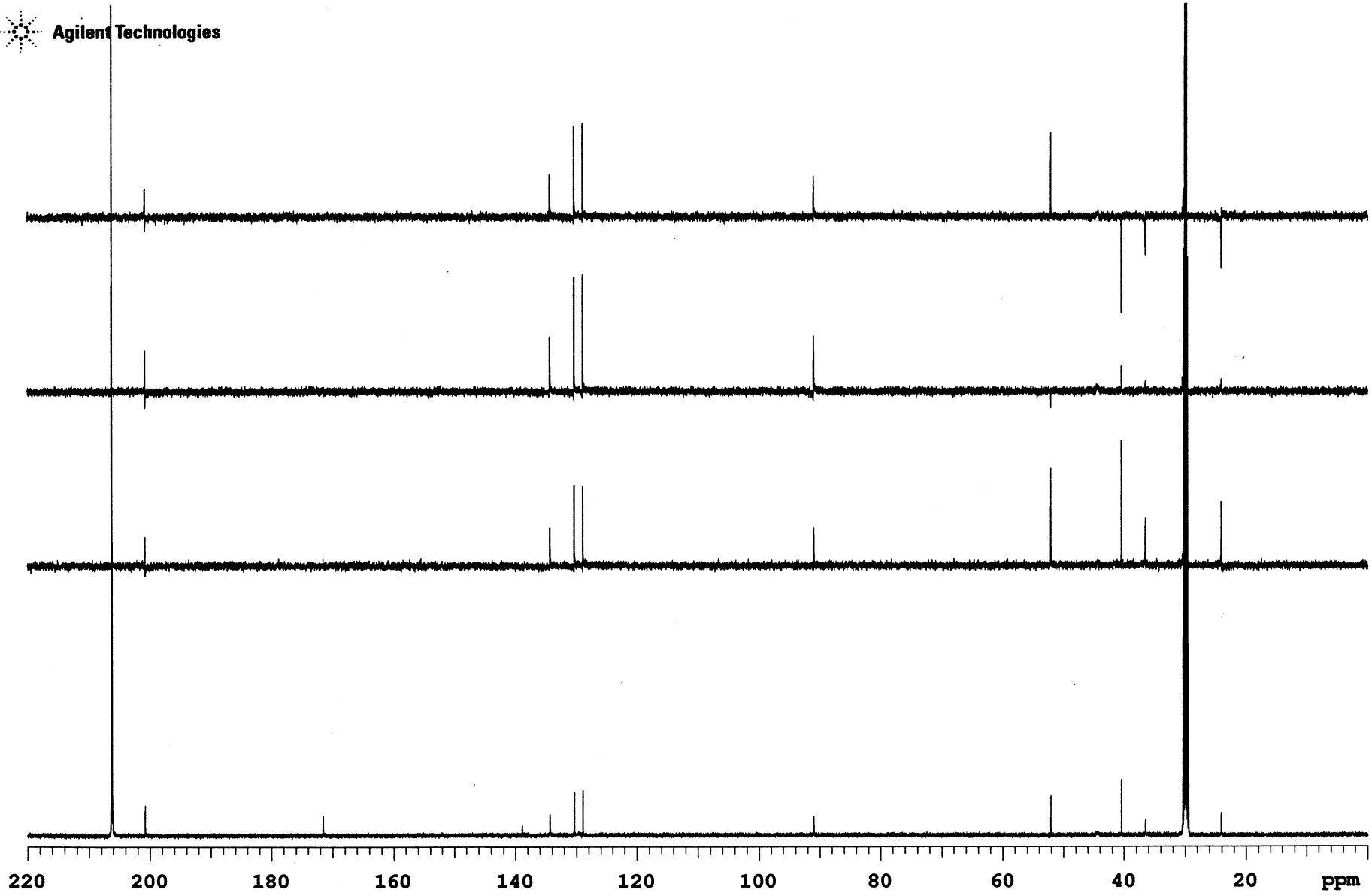


Fig S105. DEPT of compound anti-3f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-393/p2/dacetone/DEPT.fid

Plot date 2015-06-17

LCH-02-393-p2

Sample Name **LCH-02-393-p2**
Date collected **2015-11-02**
Pulse sequence **gHSQC**
Solvent **acetone**
Temperature **25**
Spectrometer **Agilent-NMR-inova500**
Study owner **vnmr2**
Operator **vnmr2**

Agilent Technologies

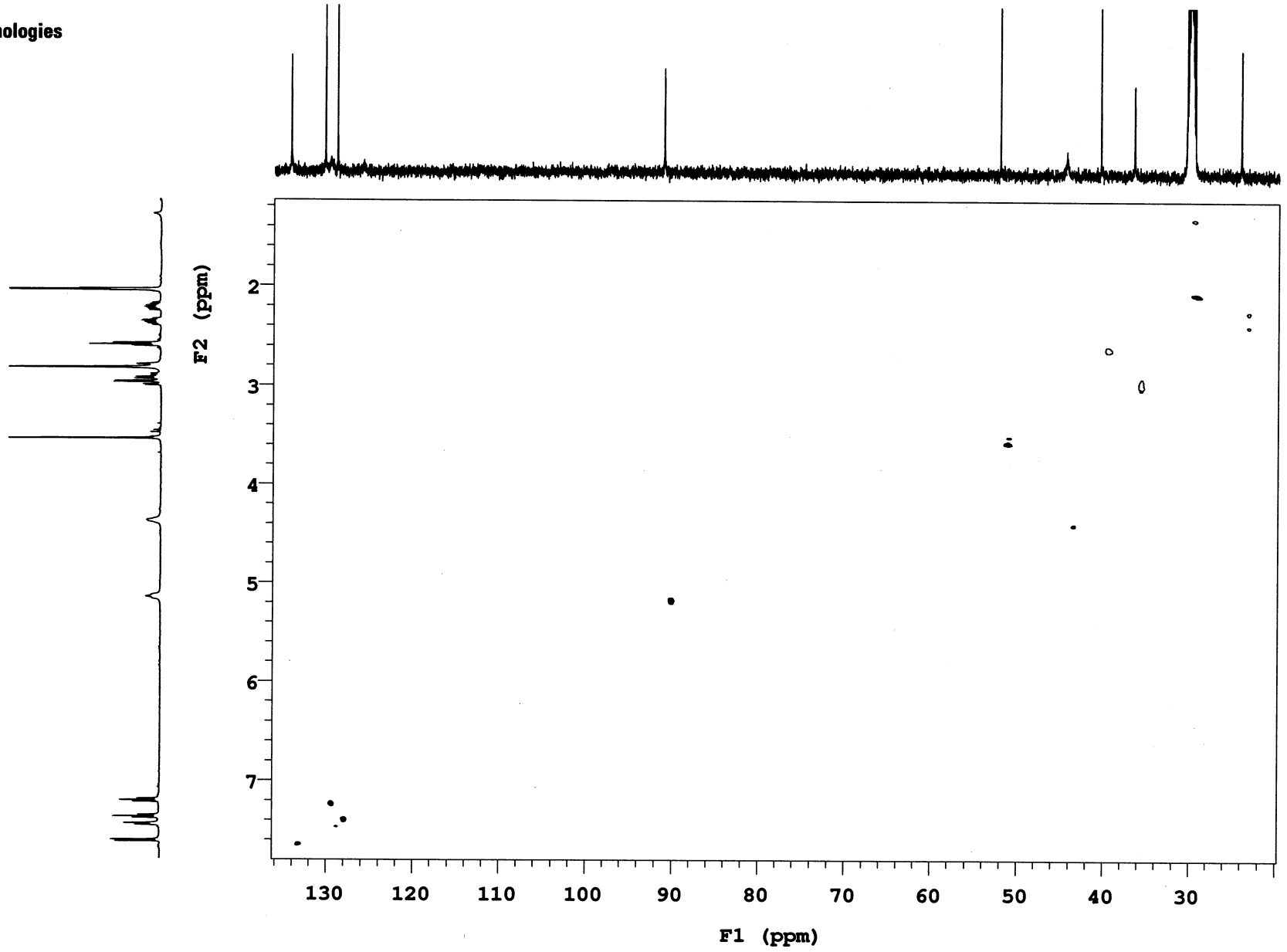
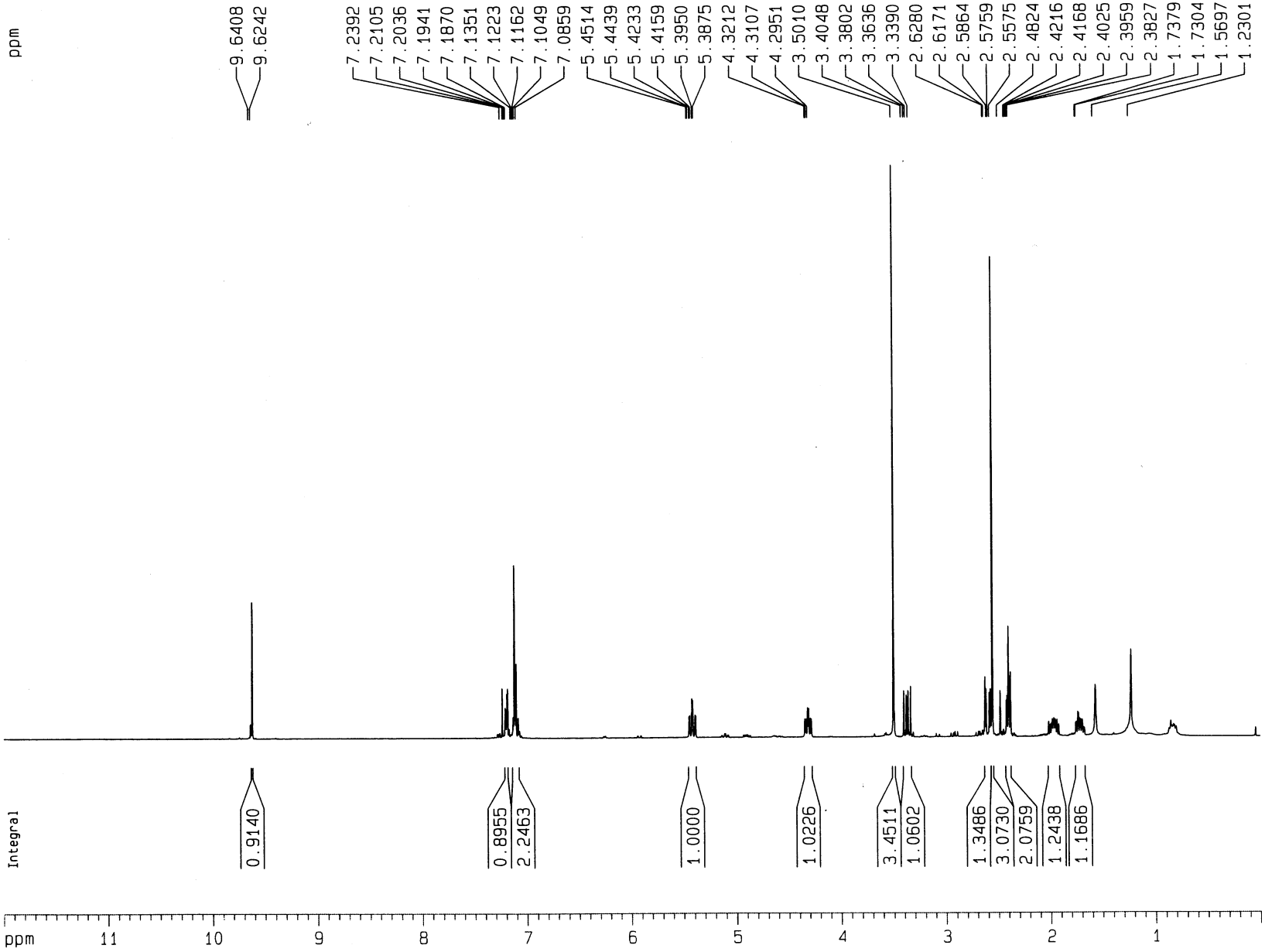


Fig S106. HSQC of compound anti-3f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-393-p2/gHSQC_02

Plot date 2015-11-02

Fig S107. 1H NMR (CDCl3, 400 MHz) of compound syn-3g



Current Data Parameters

NAME	LCH-2-373-p1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20150203
Time	0.30
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	2298.8
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

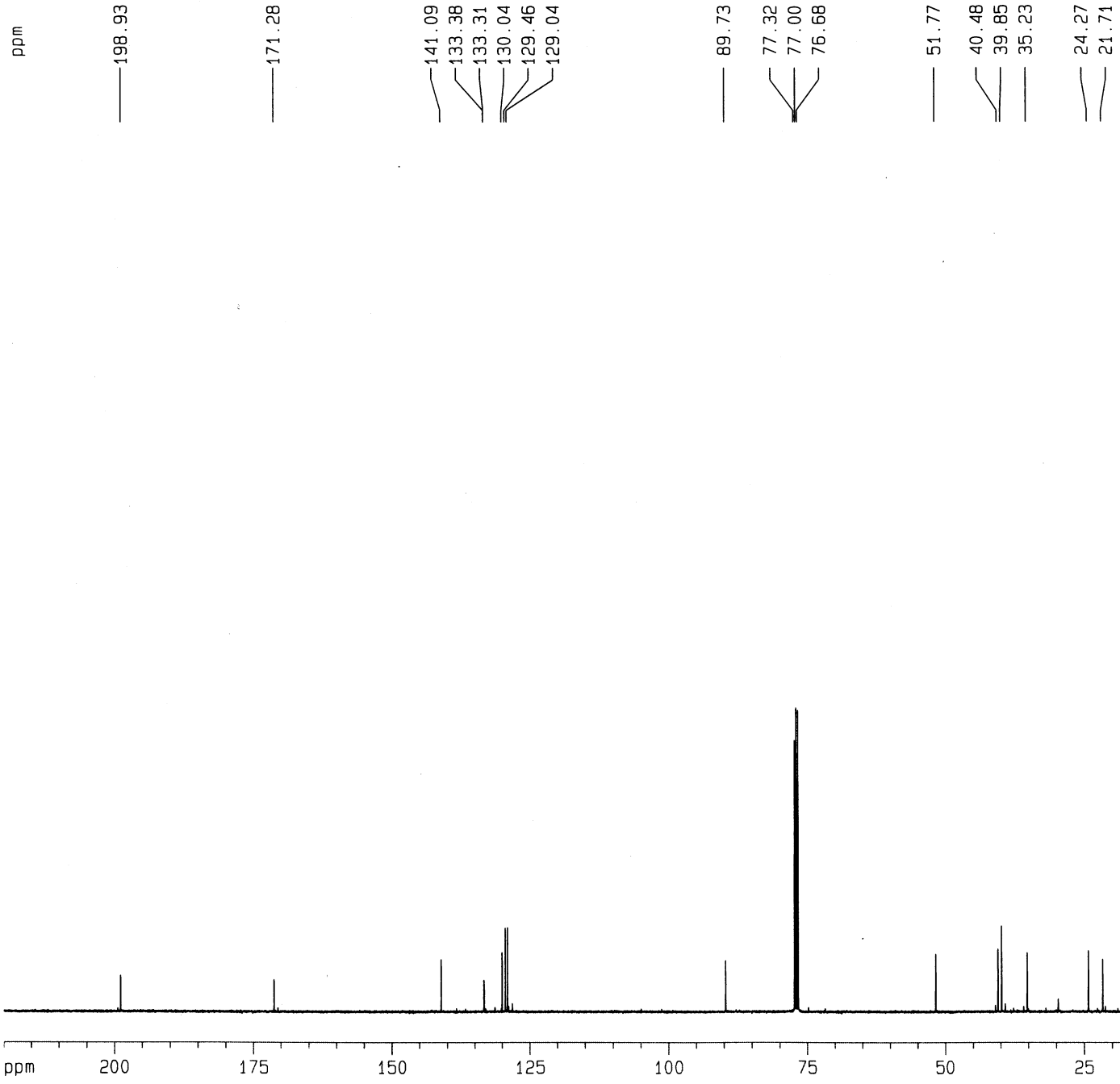
SI	8192
SF	400.1300179 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	0.55814 ppm/cm
HZCM	223.32838 Hz/cm

C13 spectrum of

Fig S108. 13C NMR (CDCl3, 100 MHz) of compound syn-3g




```
Current Data Parameters
NAME        LCH-2-373-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150203
Time                3.55
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

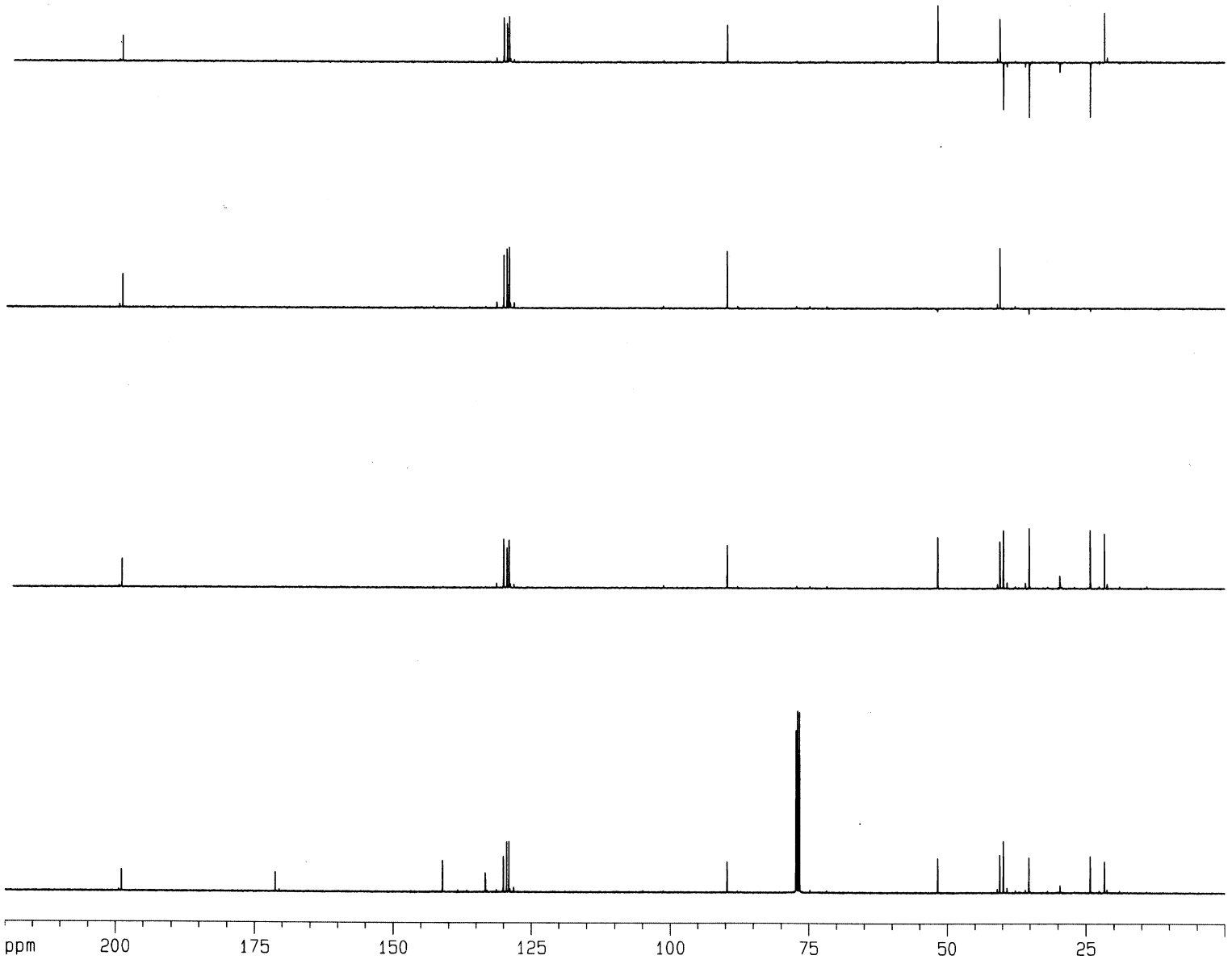
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127723 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S109. DEPT of compound syn-3g




```
Current Data Parameters
NAME        LCH-2-373-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150203
Time                3.55
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

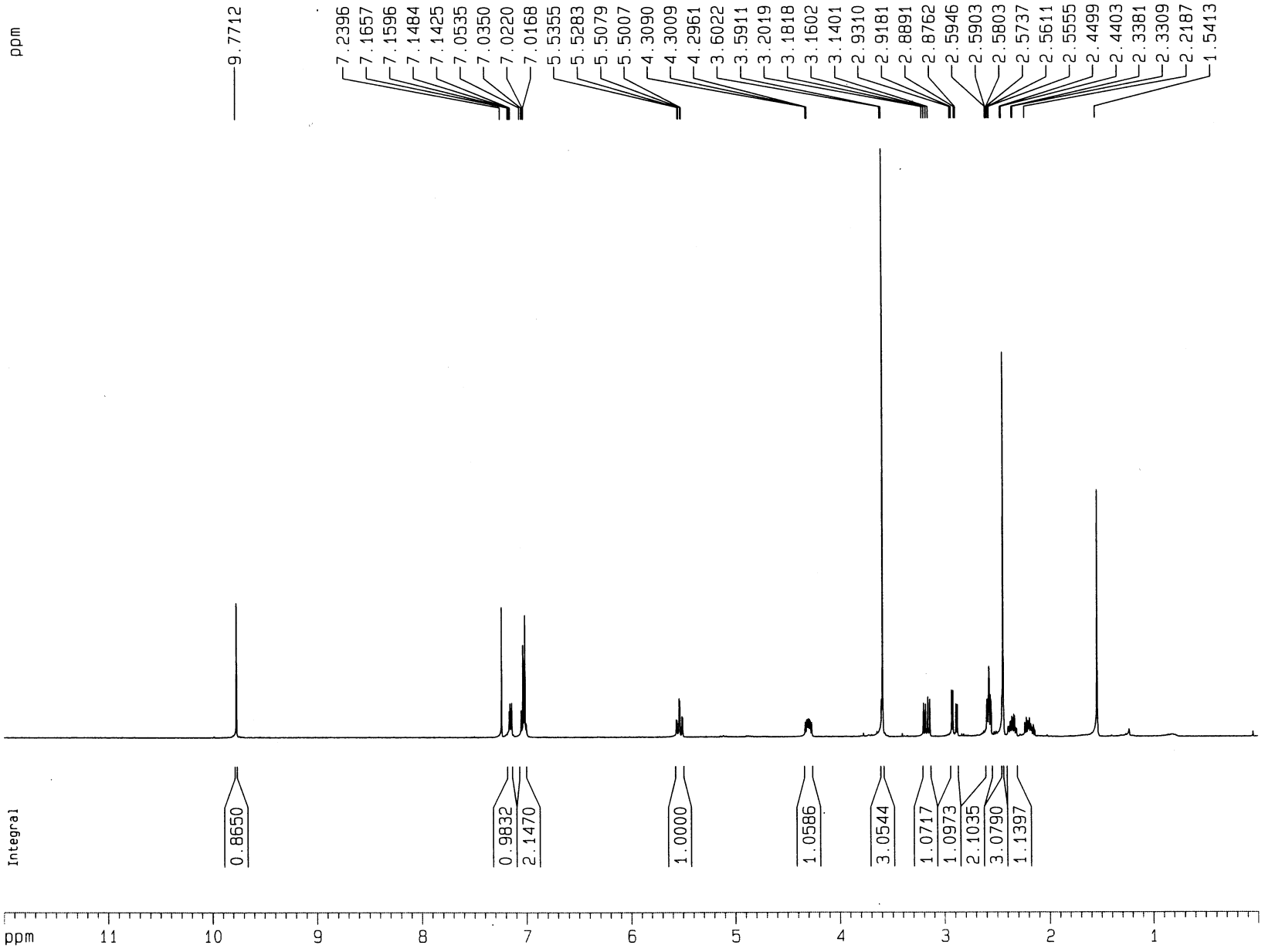
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127723 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S110. 1H NMR (CDCl3, 400 MHz) of compound anti-3g




```
Current Data Parameters
NAME        LCH-2-373-p2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150206
Time               23.43
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG                  6502
DW              83.400 usec
DE               6.50 usec
TE              300.0 K
D1           1.50000000 sec

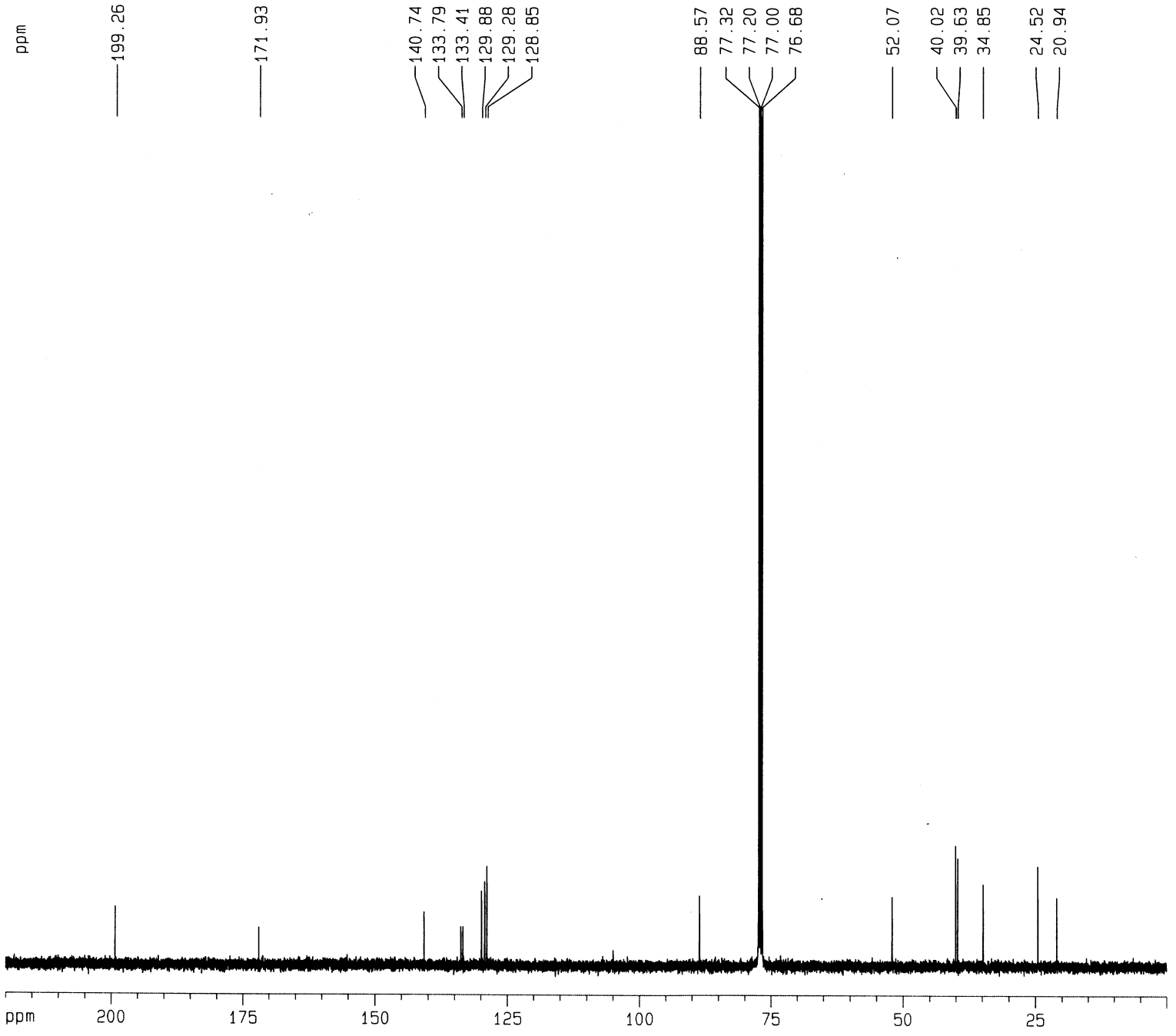
============ CHANNEL f1 =============
NUC1                1H
P1              11.90 usec
PL1             -3.00 dB
SFO1       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                 EM
SSB                    0
LB              0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              21.50 cm
F1P            12.000 ppm
F1             4801.56 Hz
F2P            -0.000 ppm
F2              -0.00 Hz
PPMCM         0.55814 ppm/cm
HZCM        223.32838 Hz/cm
```

C13 spectrum of

Fig S111. 13C NMR (CDCl3, 100 MHz) of compound anti-3g




```
Current Data Parameters
NAME        LCH-2-373-p2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150207
Time                3.07
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

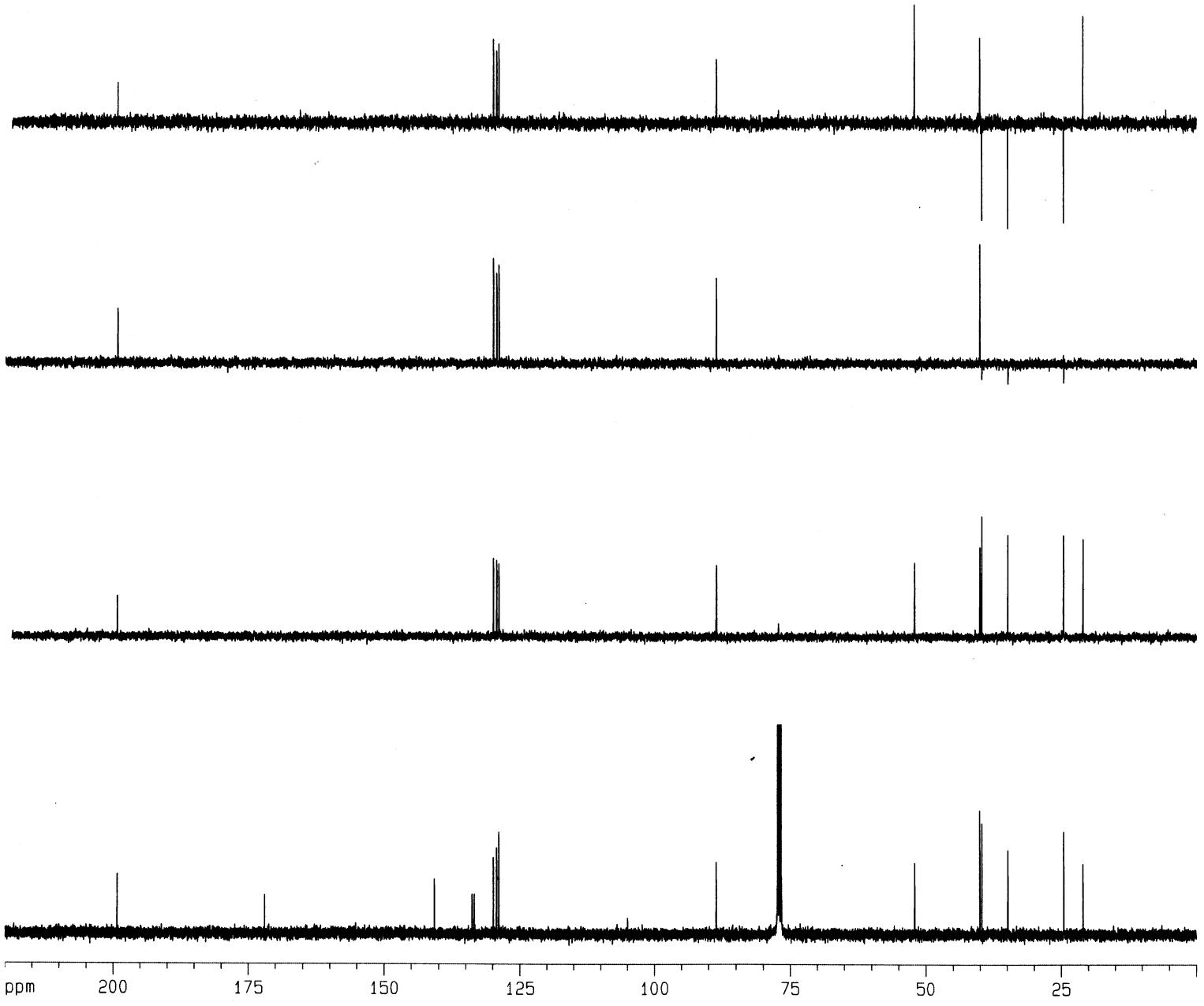
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127708 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S112. DEPT of compound anti-3g




```
Current Data Parameters
NAME       LCH-2-373-p2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150207
Time               3.07
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3686
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

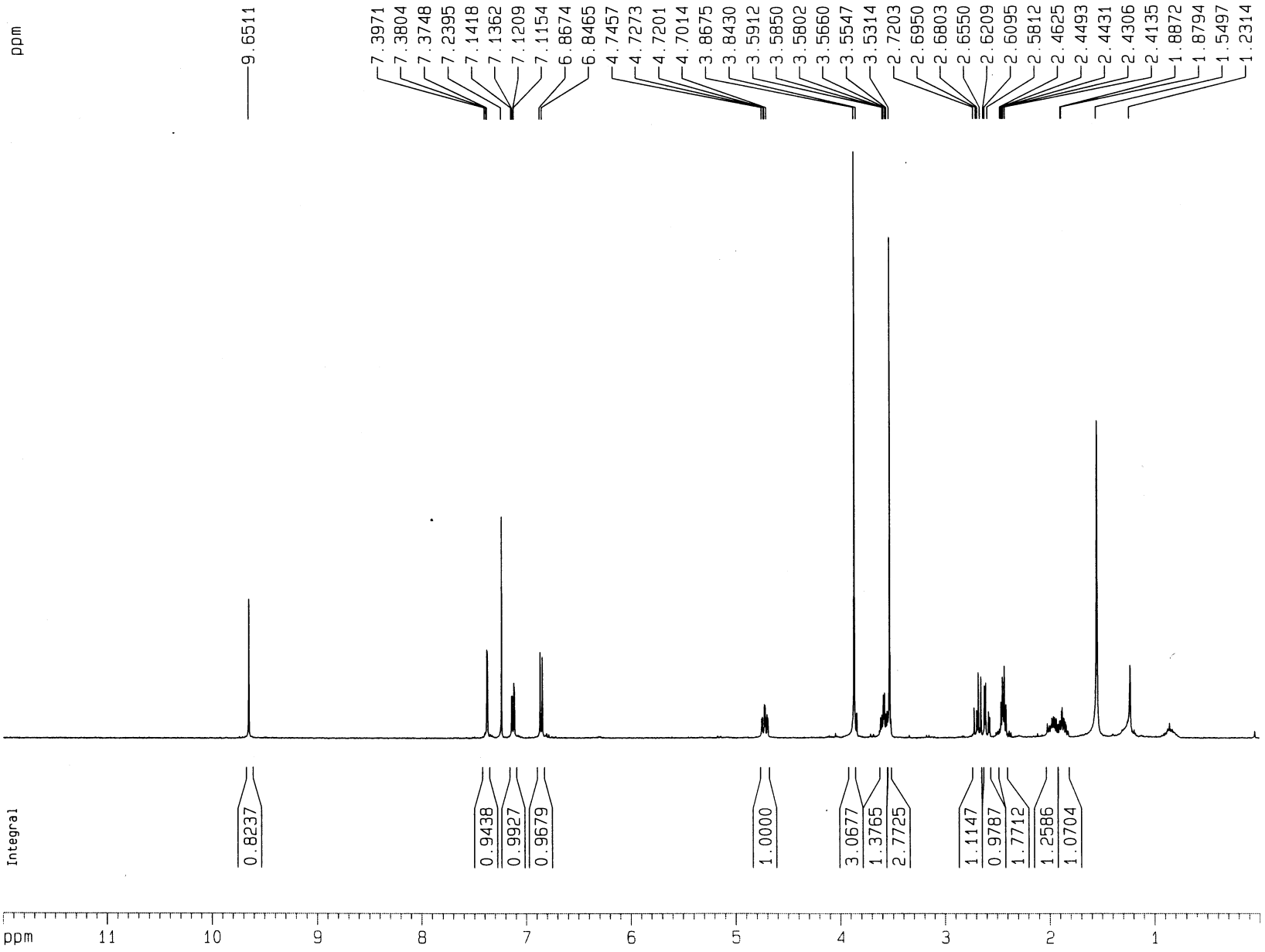
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127708 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S113. 1H NMR (CDCl3, 400 MHz) of compound syn-3h




```
Current Data Parameters
NAME        LCH-2-390-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150410
Time                0.51
INSTRUM            spect
PROBHD      5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG                  6502
DW              83.400 usec
DE               6.50 usec
TE               300.0 K
D1          1.50000000 sec

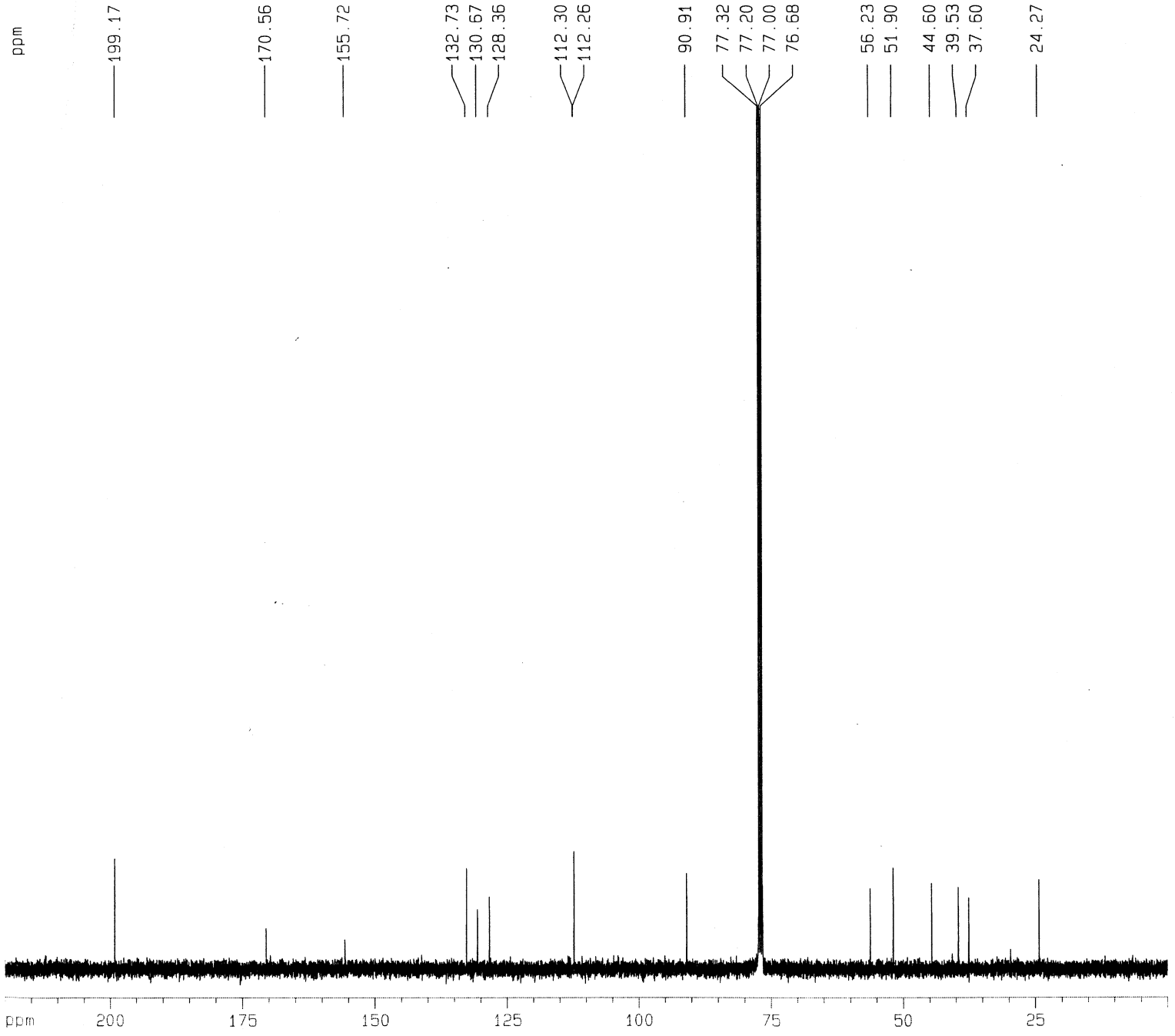
============ CHANNEL f1 =============
NUC1                1H
P1              11.90 usec
PL1             -3.00 dB
SFO1       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                   EM
SSB                    0
LB              0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                21.50 cm
F1P               12.000 ppm
F1               4801.56 Hz
F2P              -0.000 ppm
F2                -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM         223.32838 Hz/cm
```

C13 spectrum of

Fig S114. 13C NMR (CDCl3, 100 MHz) of compound syn-3h




```
Current Data Parameters
NAME        LCH-2-390-p1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150410
Time                3.53
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

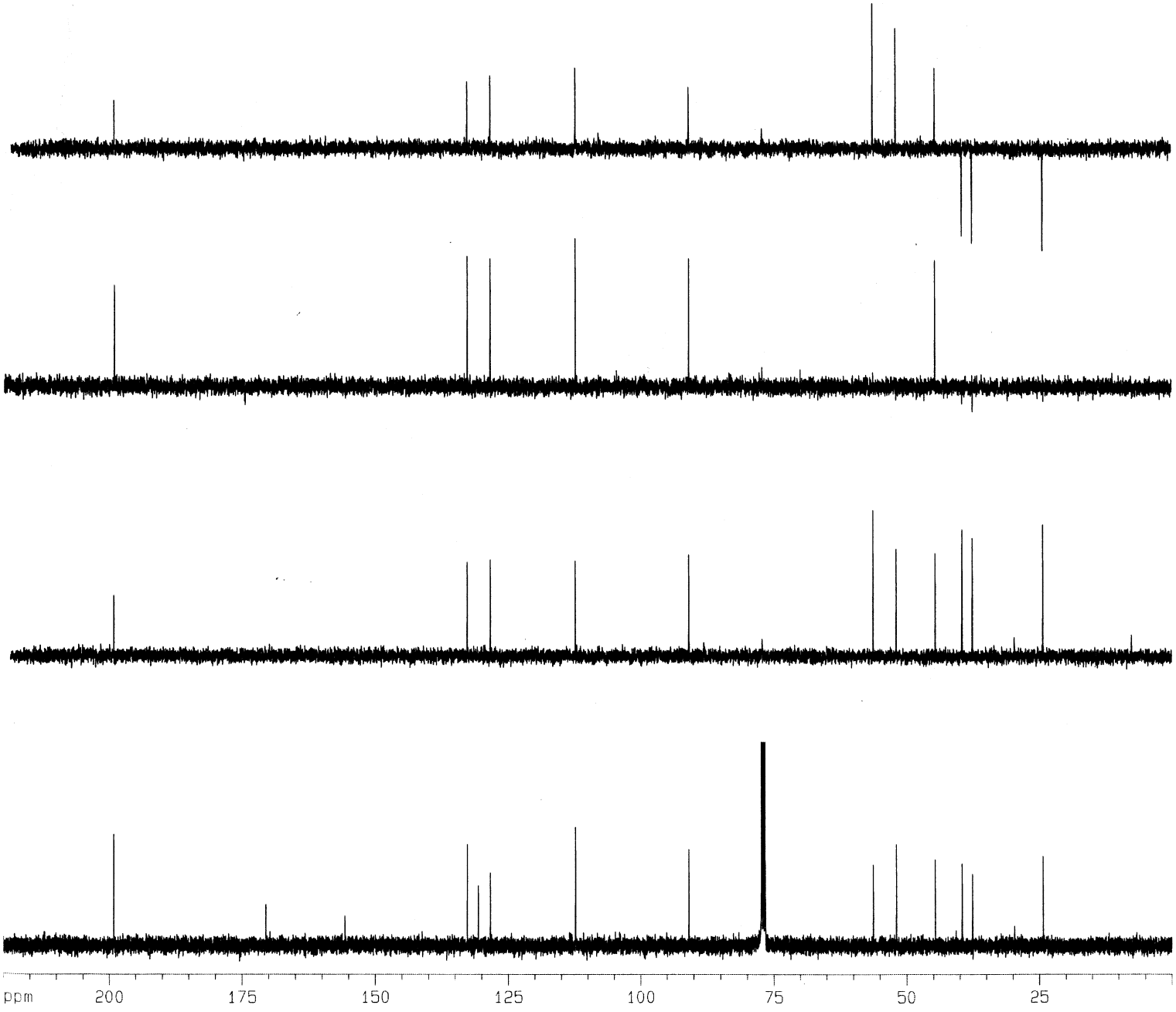
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127700 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S115. DEPT of compound syn-3h




```
Current Data Parameters
NAME        LCH-2-390-p1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150410
Time                3.53
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3276
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

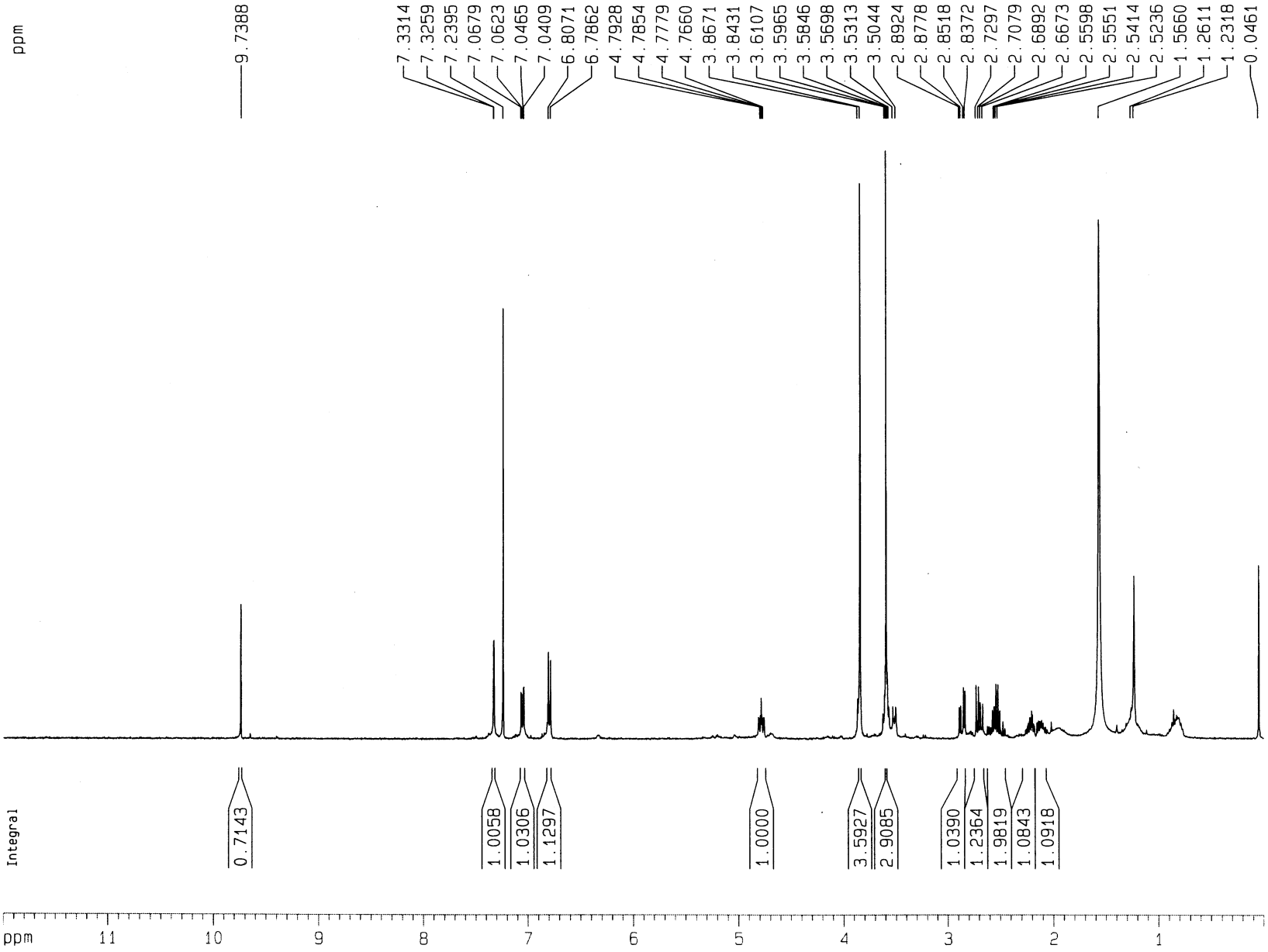
============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S116. 1H NMR (CDCl3, 400 MHz) of compound anti-3h




```
Current Data Parameters
NAME        LCH-2-390-p2
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150411
Time                1.38
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             5995.204 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                  8192
DW                83.400 usec
DE                 6.50 usec
TE                300.0 K
D1           1.50000000 sec

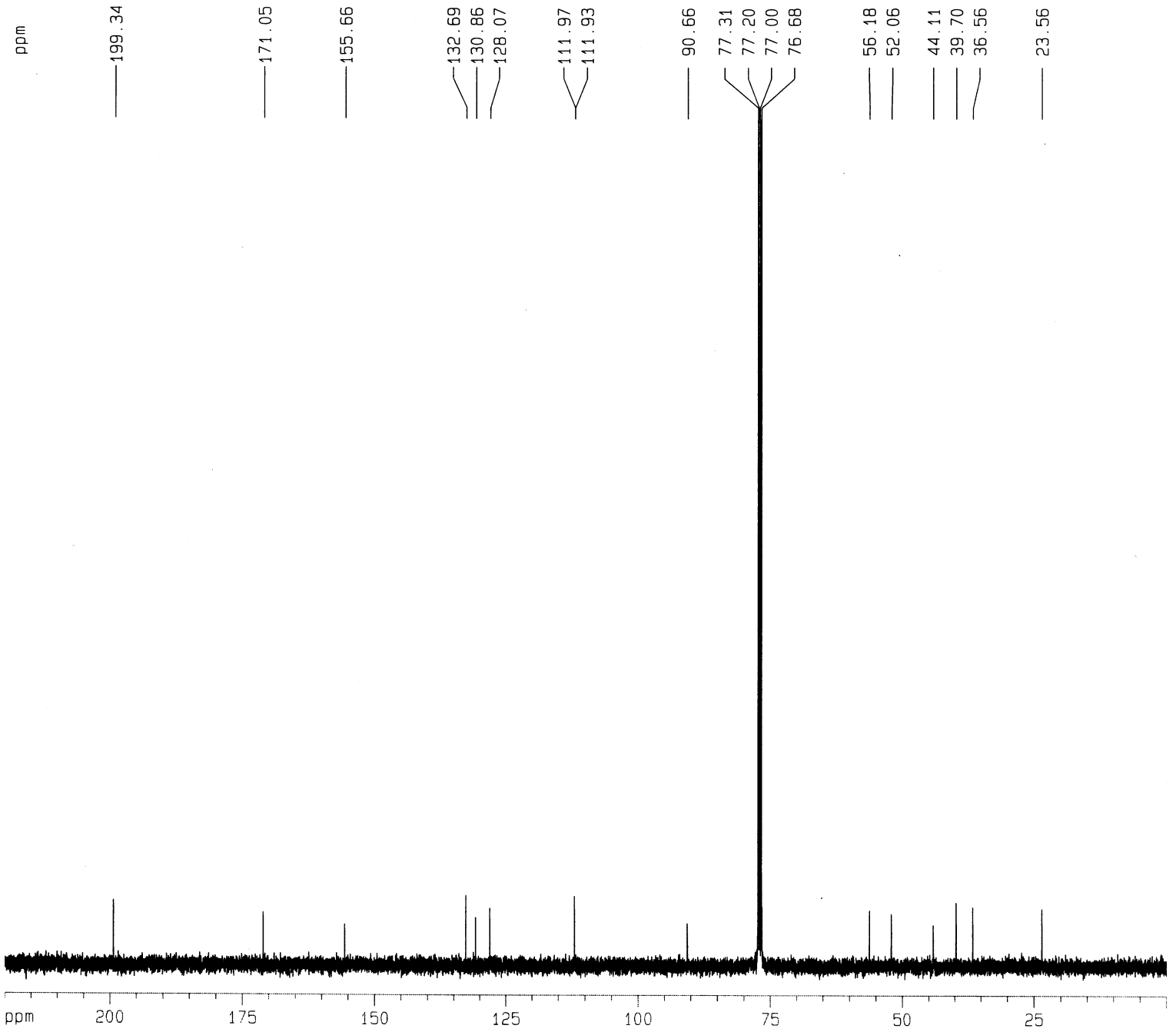
============ CHANNEL f1 ============
NUC1                1H
P1                11.90 usec
PL1               -3.00 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF           400.1300179 MHz
WDW                 EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                21.50 cm
F1P              12.000 ppm
F1              4801.56 Hz
F2P             -0.000 ppm
F2               -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM         223.32838 Hz/cm
```

C13 spectrum of

Fig S117. 13C NMR (CDCl3, 100 MHz) of compound anti-3h




```
Current Data Parameters
NAME        LCH-2-390-p2
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150411
Time                4.40
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

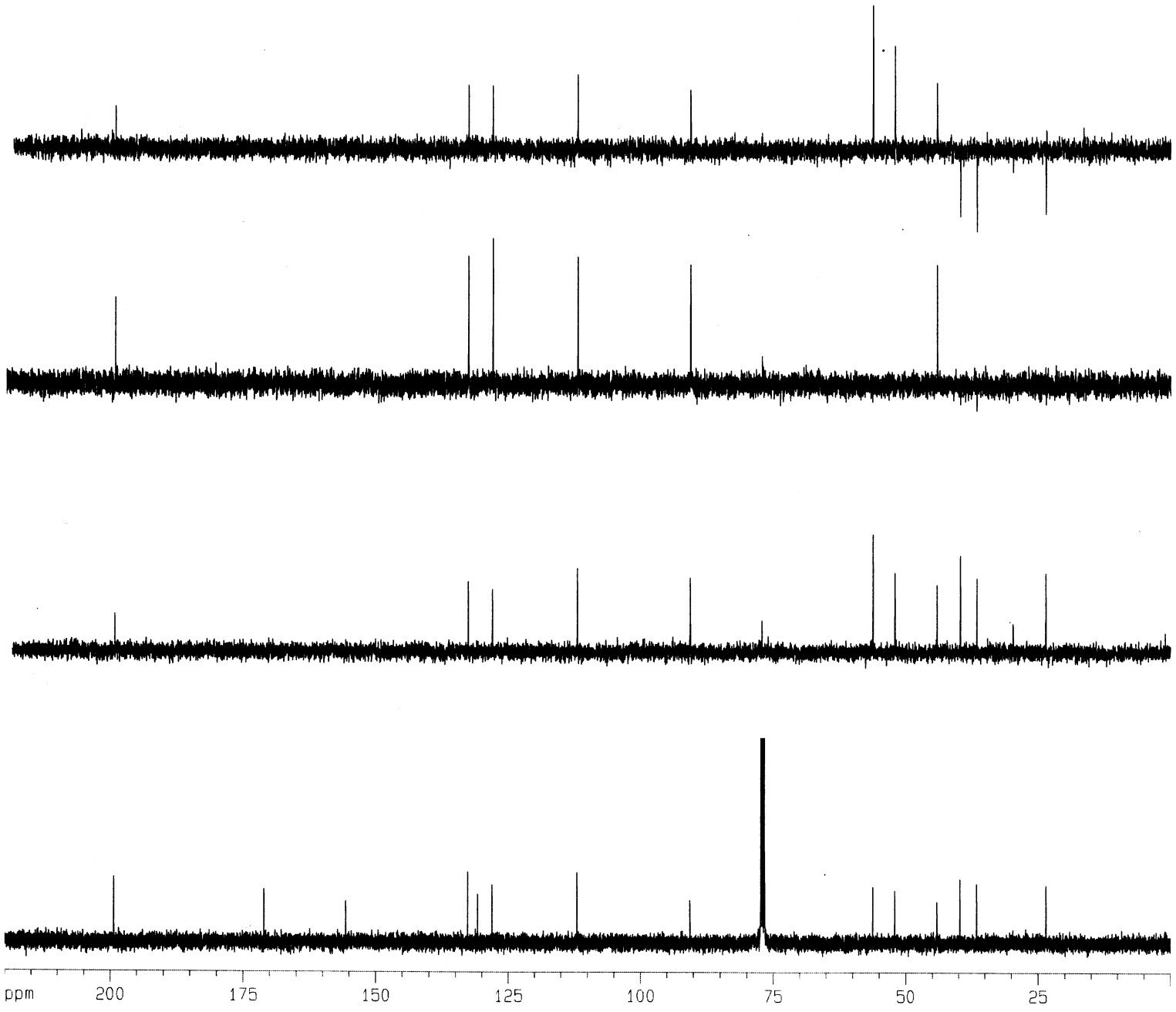
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S118. DEPT of compound anti-3h




```
Current Data Parameters
NAME        LCH-2-390-p2
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150411
Time                4.40
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3276
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

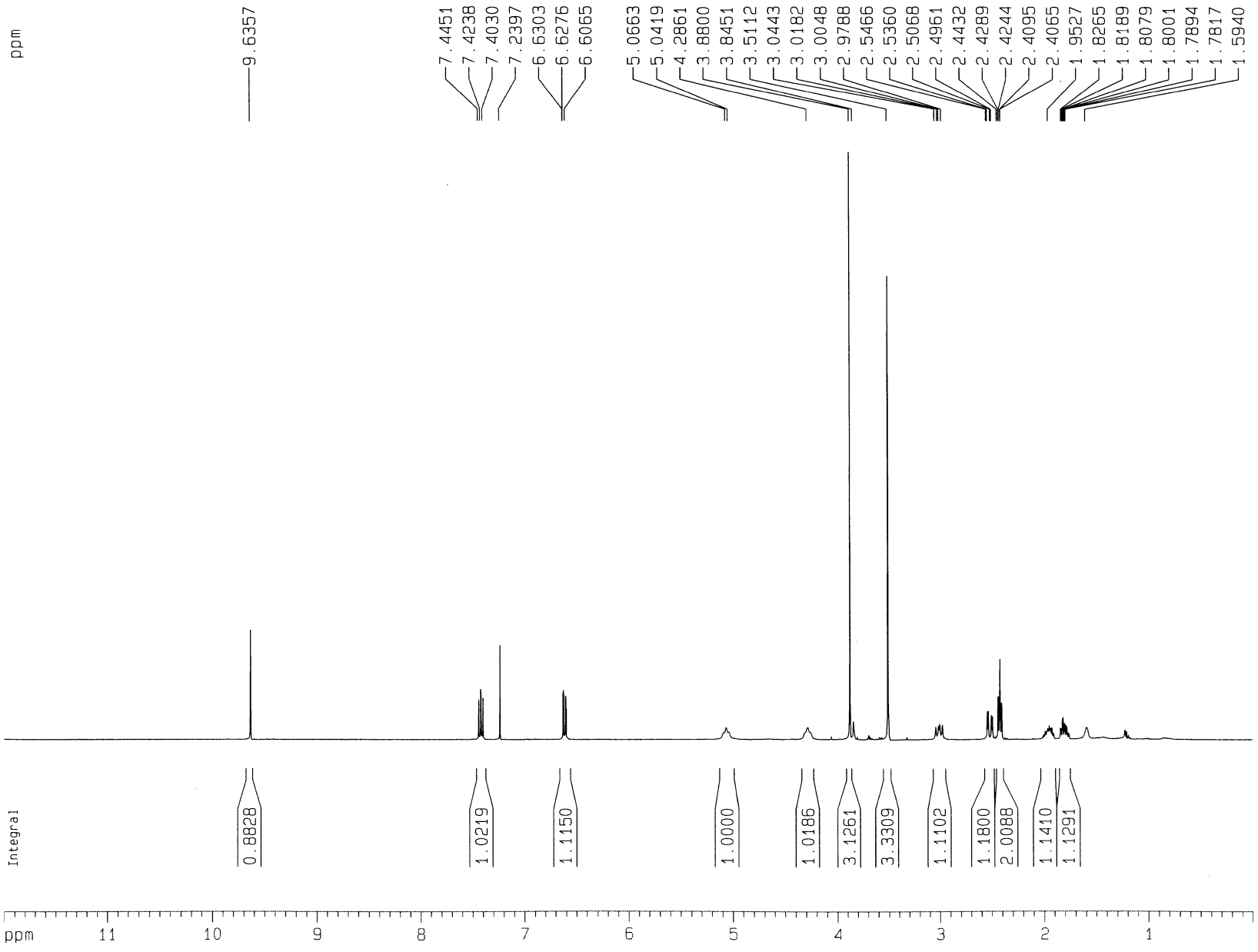
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127700 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S119. 1H NMR (CDCl3, 400 MHz) of compound syn-3i




```
Current Data Parameters
NAME        LCH-2-394-p1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150405
Time               21.58
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG              2298.8
DW              83.400 usec
DE               6.50 usec
TE              300.0 K
D1           1.50000000 sec

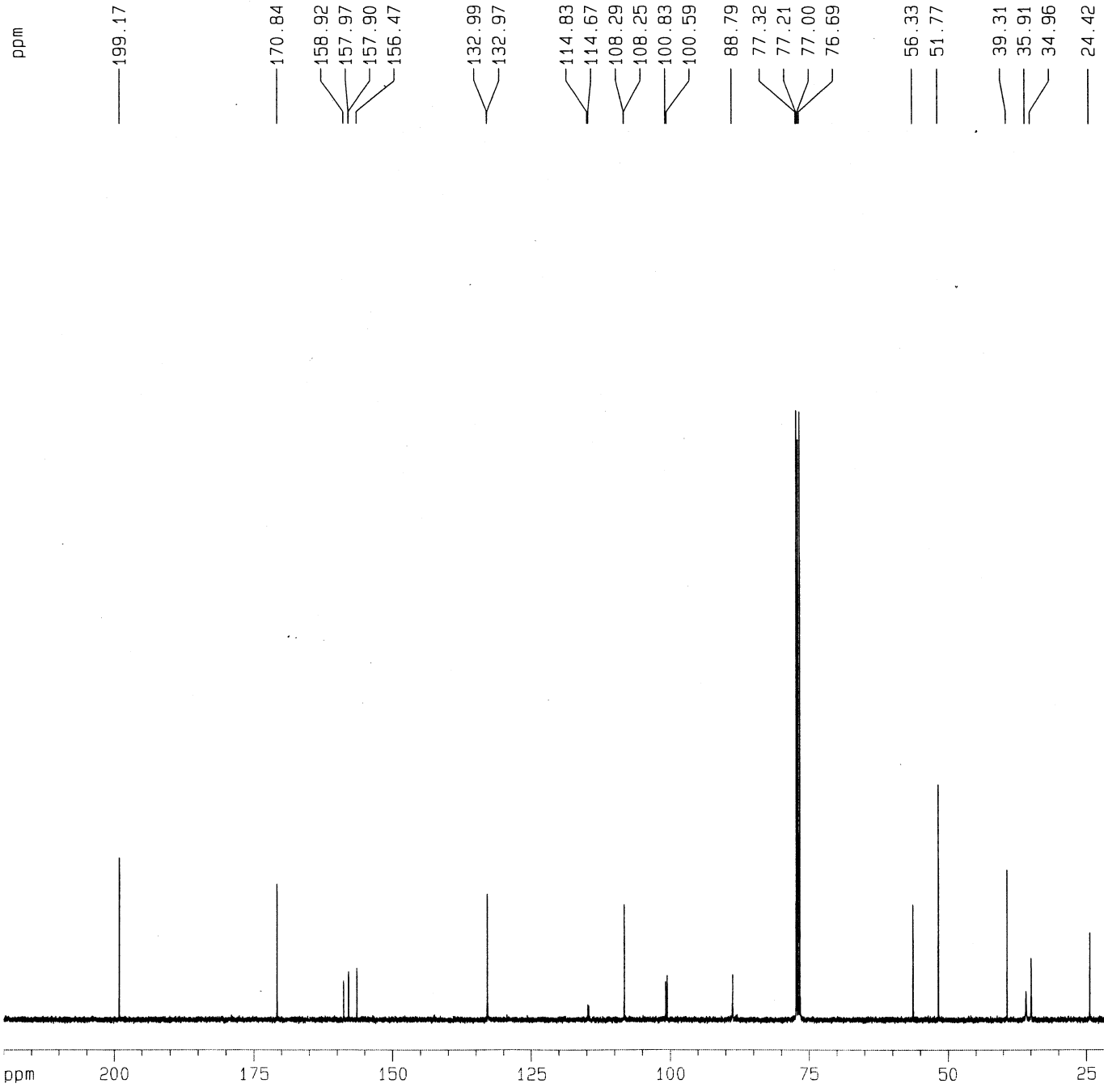
============ CHANNEL f1 =============
NUC1                1H
P1              11.90 usec
PL1             -3.00 dB
SF01       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                 EM
SSB                 0
LB              0.10 Hz
GB                  0
PC                1.00

1D NMR plot parameters
CX                21.50 cm
F1P               12.000 ppm
F1              4801.56 Hz
F2P              -0.000 ppm
F2               -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM          223.32837 Hz/cm
```

C13 spectrum of

Fig S120. 13C NMR (CDCl3, 100 MHz) of compound syn-3i




```
Current Data Parameters
NAME        LCH-2-394-p1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150406
Time                1.46
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  4096
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

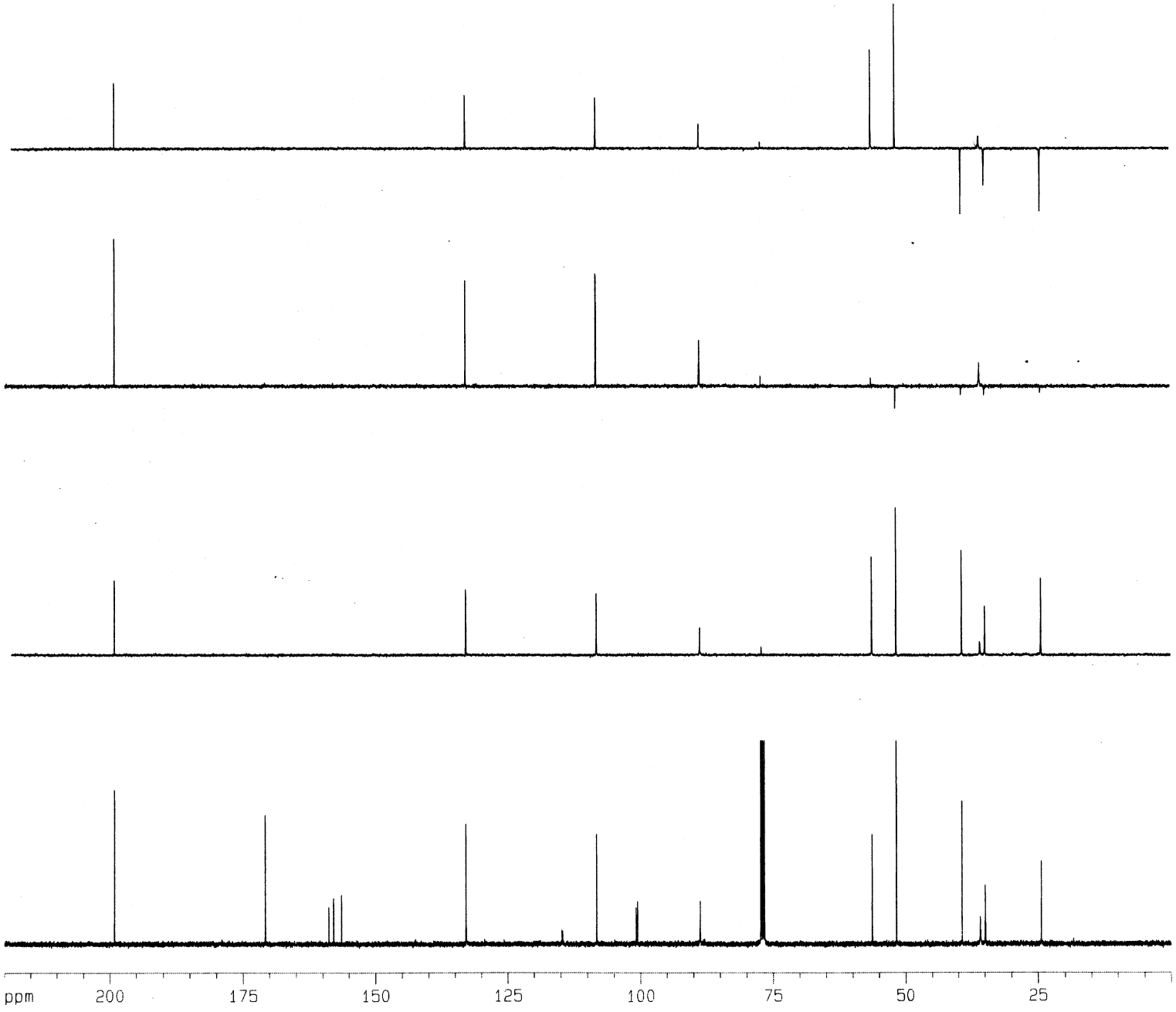
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127723 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S121. DEPT of compound syn-3i




```
Current Data Parameters
NAME        LCH-2-394-p1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150406
Time                1.46
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  4096
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

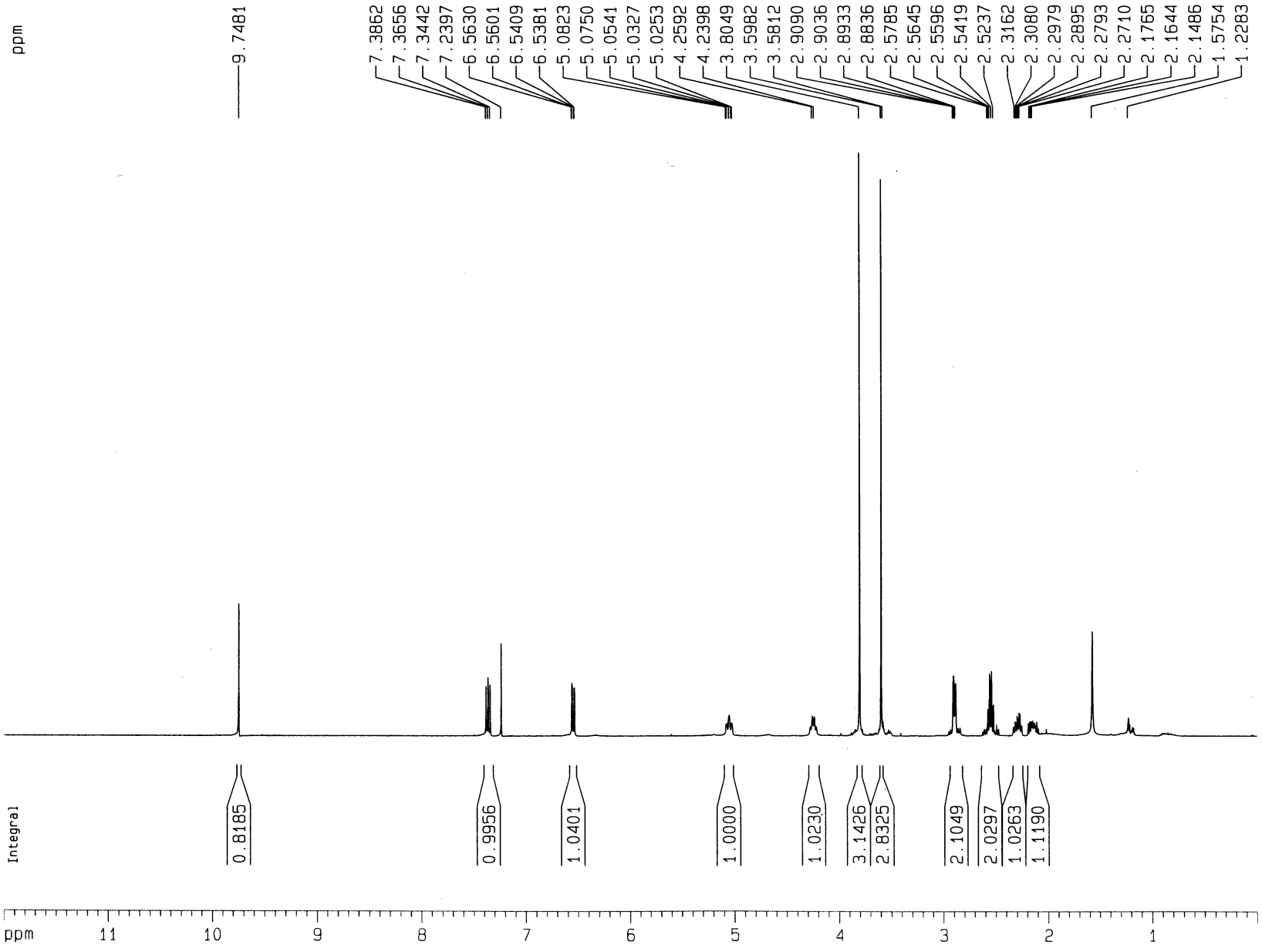
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127723 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S122. 1H NMR (CDCl3, 400 MHz) of compound anti-3i




```
Current Data Parameters
NAME        LCH-2-394-p2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150406
Time               23.49
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 16384
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           5995.204 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG              3649.1
DW              83.400 usec
DE               6.50 usec
TE              300.0 K
D1           1.50000000 sec

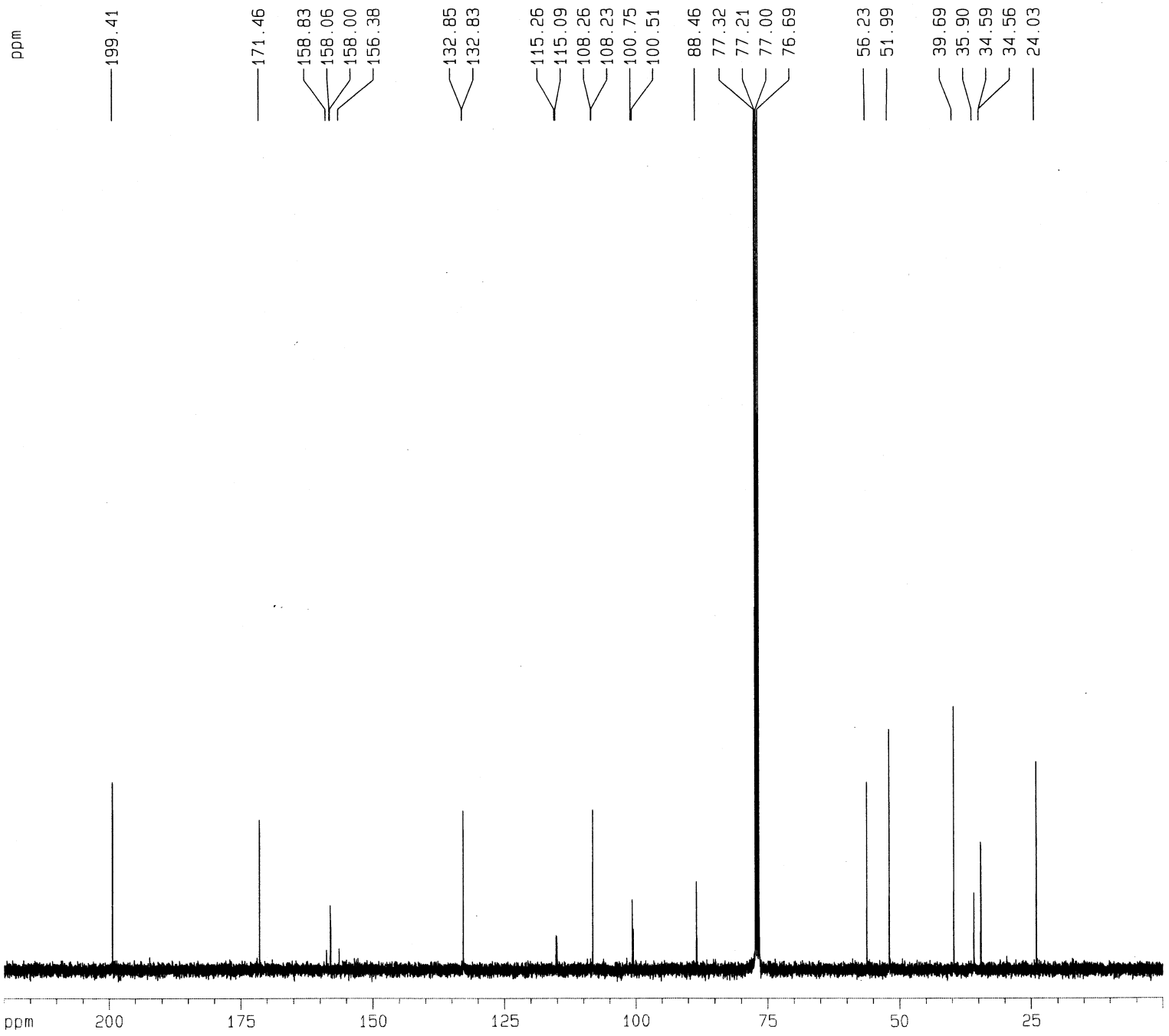
============ CHANNEL f1 =============
NUC1                1H
P1              11.90 usec
PL1             -3.00 dB
SFO1       400.1326008 MHz

F2 - Processing parameters
SI                  8192
SF         400.1300179 MHz
WDW                 EM
SSB                  0
LB              0.10 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX                21.50 cm
F1P               12.000 ppm
F1              4801.56 Hz
F2P              -0.000 ppm
F2               -0.00 Hz
PPMCM          0.55814 ppm/cm
HZCM         223.32838 Hz/cm
```

C13 spectrum of

Fig S123. 13C NMR (CDCl3, 100 MHz) of compound anti-3i




```
Current Data Parameters
NAME        LCH-2-394-p2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150407
Time                3.15
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3686
DS                     4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                   256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

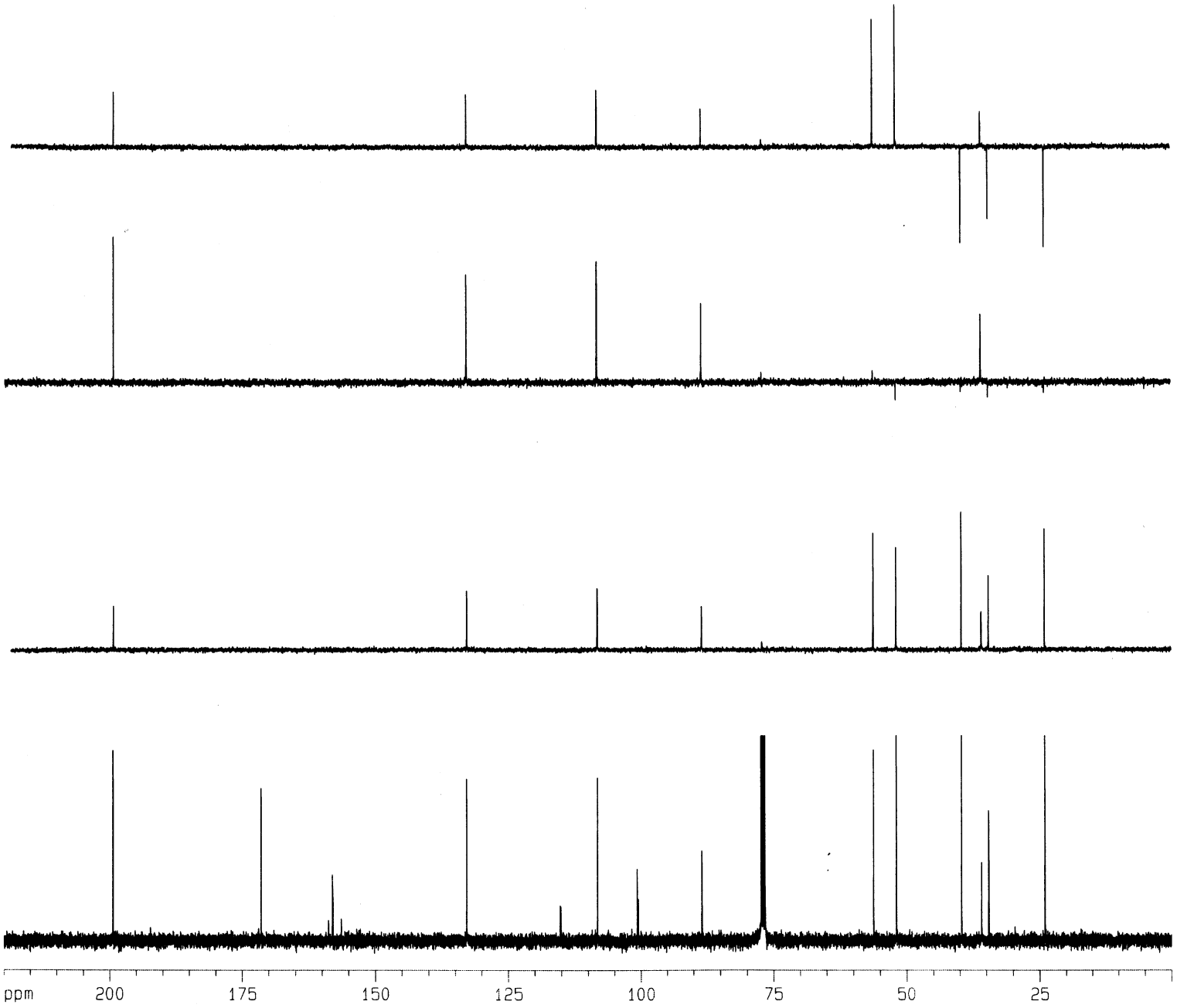
============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SF01         100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SF02         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127715 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.74048 Hz/cm
```

Fig S124. DEPT of compound anti-3i




```
Current Data Parameters
NAME        LCH-2-394-p2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150407
Time                3.15
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3686
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

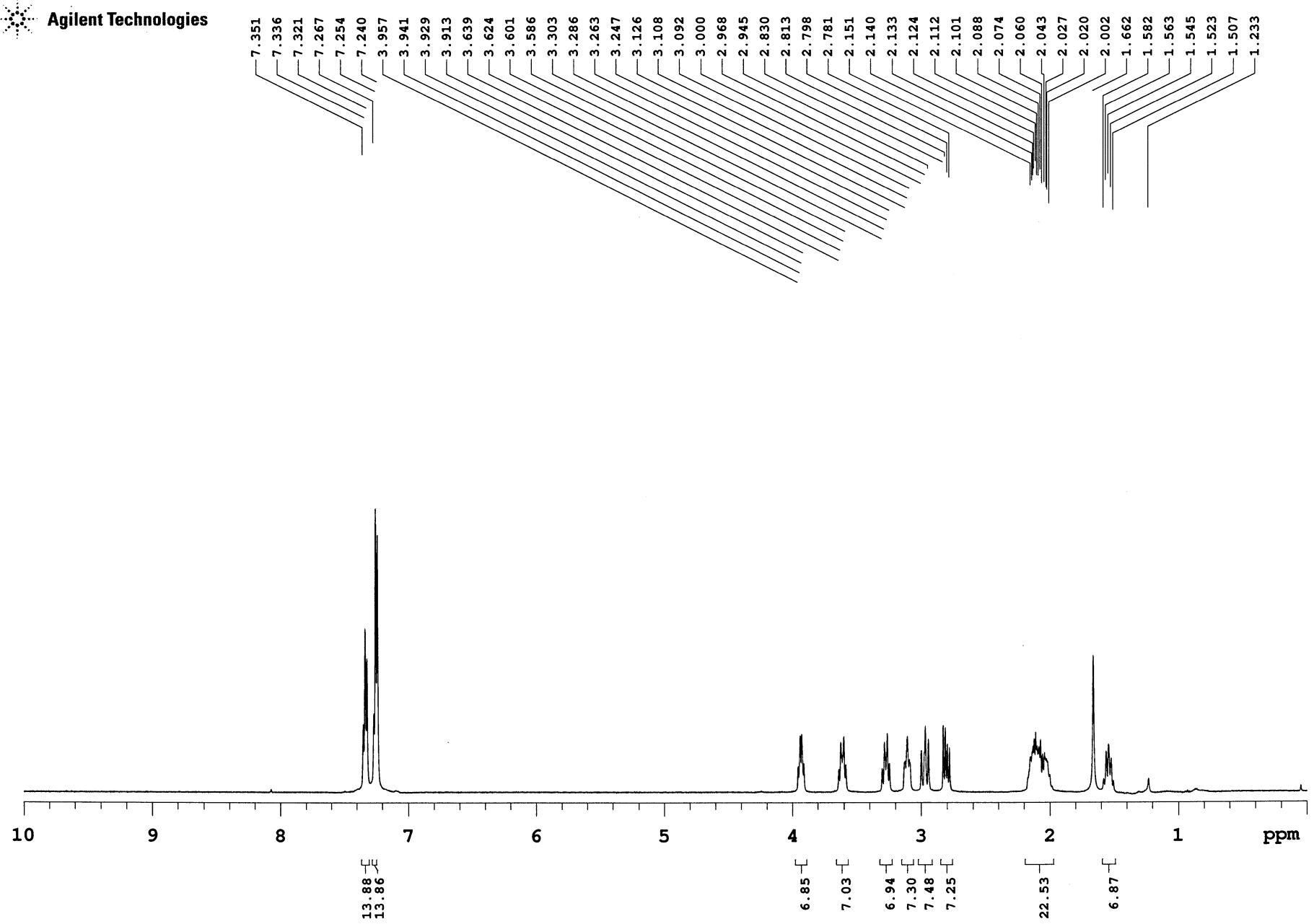
F2 - Processing parameters
SI                 32768
SF          100.6127715 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.74048 Hz/cm
```

LCH-2-332

Sample Name	**LCH-2-332**	Pulse sequence	**s2pul**	Temperature	**60**	Study owner	**vnmr2**
Date collected	**2014-12-04**	Solvent	**cdcl3**	Spectrometer	**--**	Operator	**vnmr2**

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-332/H1.fid

Plot date 2015-10-23

Fig S125. 1H NMR (CDCl3, 500 MHz) of compound syn-4a

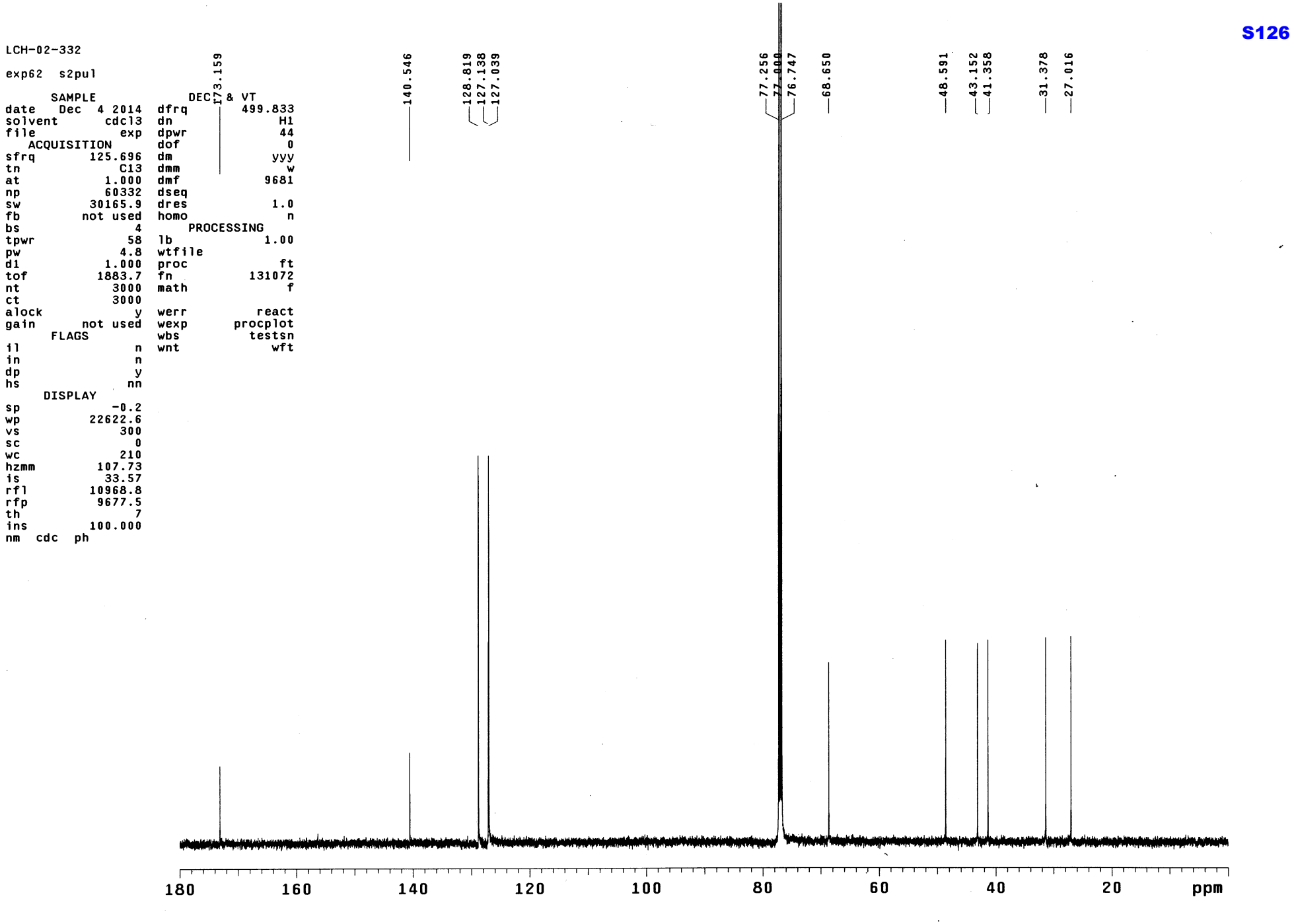


Fig S126. 13C NMR (CDCl3, 125 MHz) of compound syn-4a

Fig S127. DEPT of compound syn-4a

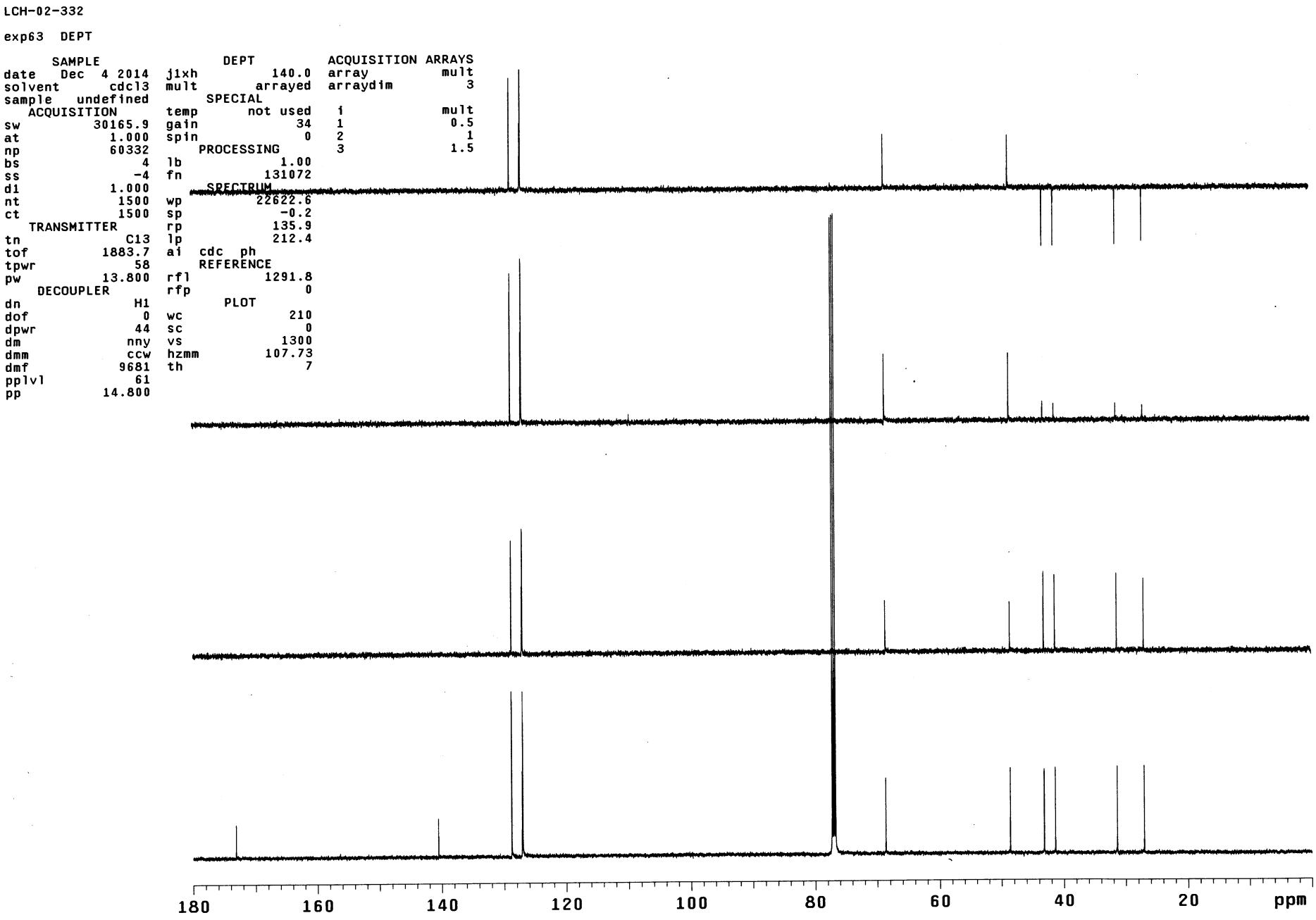


Fig S128. HSQC of compound syn-4a

LCH-02-332

exp66 gHSQC

SAMPLE	
date	Dec 4 2014
solvent	cdcl3
sample	undefined
ACQUISITION	
sw	4001.6
at	0.128
np	1024
fb	not used
ss	32
d1	1.000
nt	8
2D ACQUISITION	
sw1	21367.5
ni	128
phase	arrayed
TRANSMITTER	
tn	H1
sfrq	499.832
tof	-499.9
tpwr	61
pw	13.900
DECOUPLER	
dn	C13
dof	-2515.1
dm	nny
dmm	ccp
dmf	32258
dpwr	42
pwxlvl	60
pwx	10.700
HSQC	
j1xh	140.0
nullflg	y
mult	2

FLAGS	
hs	n
sspul	y
PFGflg	y
hsglvl	1009
SPECIAL	
temp	not used
gain	54
spin	0
GRADIENTS	
gzlvl1	1009
gt1	0.002000
gzlvl3	507
gt3	0.001000
gstab	0.000500
F2 PROCESSING	
gf	0.059
gfs	not used
fn	1024
F1 PROCESSING	
gf1	0.006
gfs1	not used
proc1	lp
fn1	2048
DISPLAY	
sp	688.7
wp	3079.4
sp1	3072.1
wp1	13459.0
rfl	1653.5
rfp	1630.9
rfl1	7417.0
rfp1	6107.0
PLOT	
wc	150.0
sc	6.2
wc2	116.2
sc2	0
vs	133
th	6
ai cdc ph	

ACQUISITION ARRAYS	
array	phase
arraydim	256
i	phase
1	1
2	2

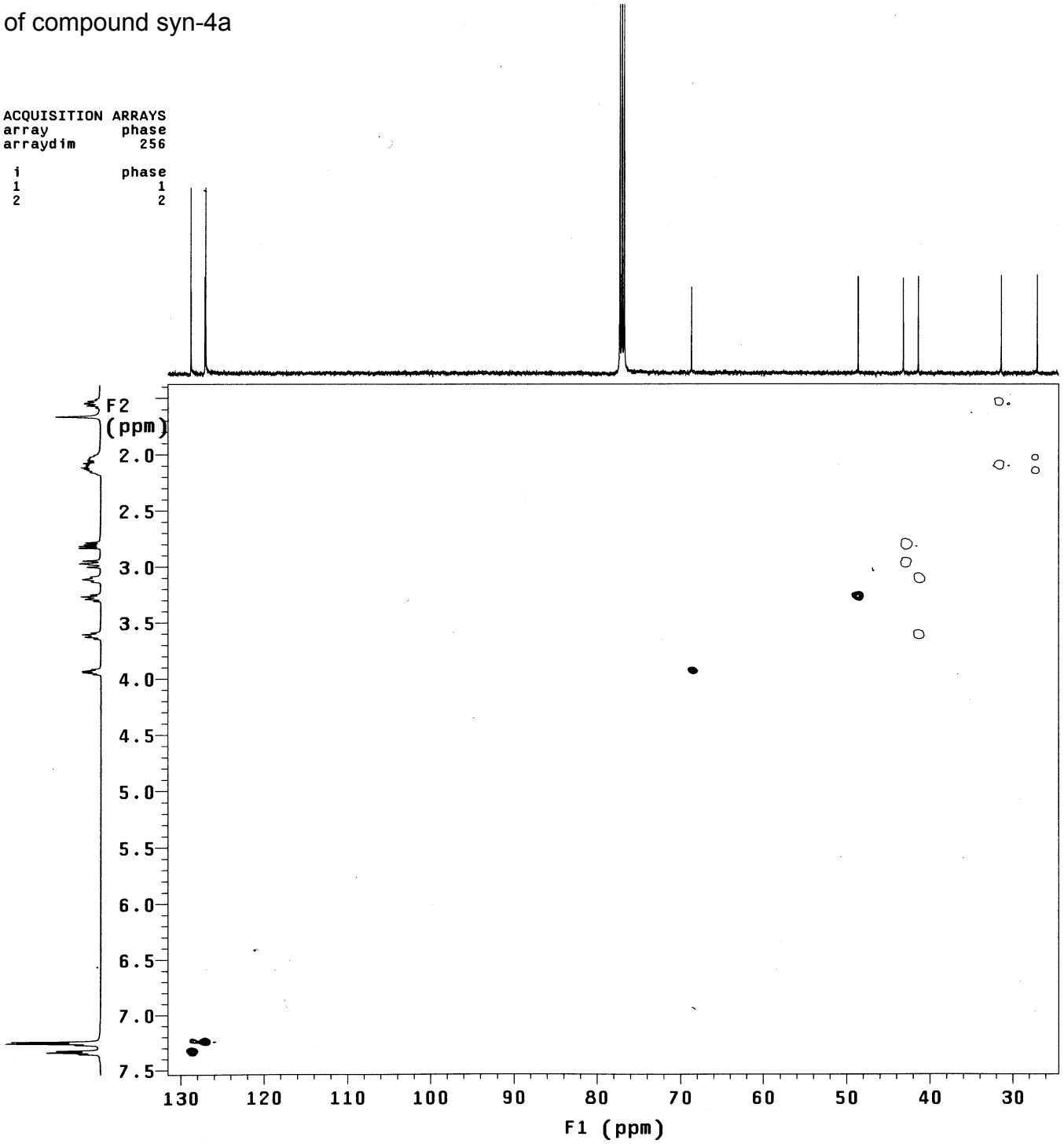


Fig S129. COSY of compound syn-4a

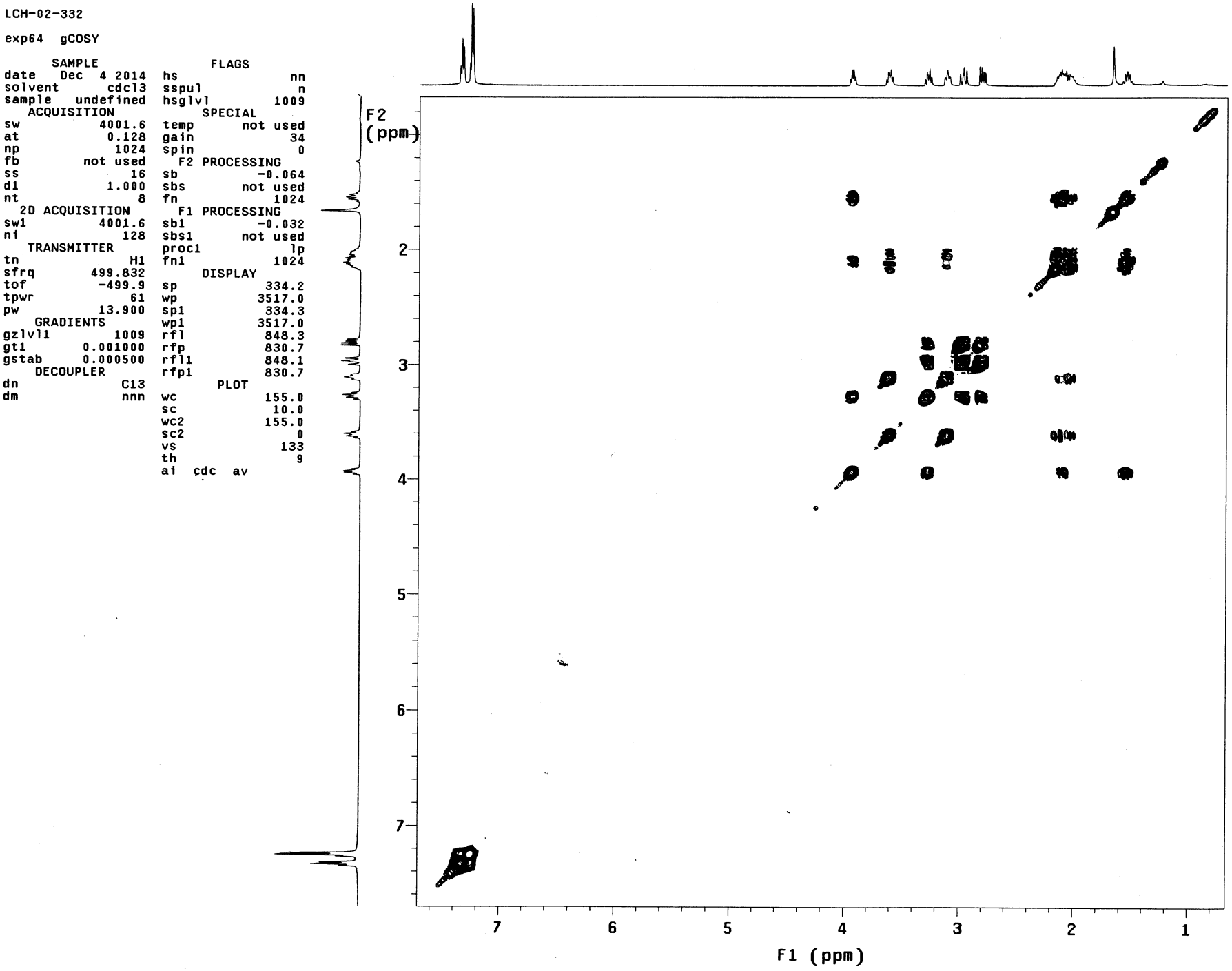
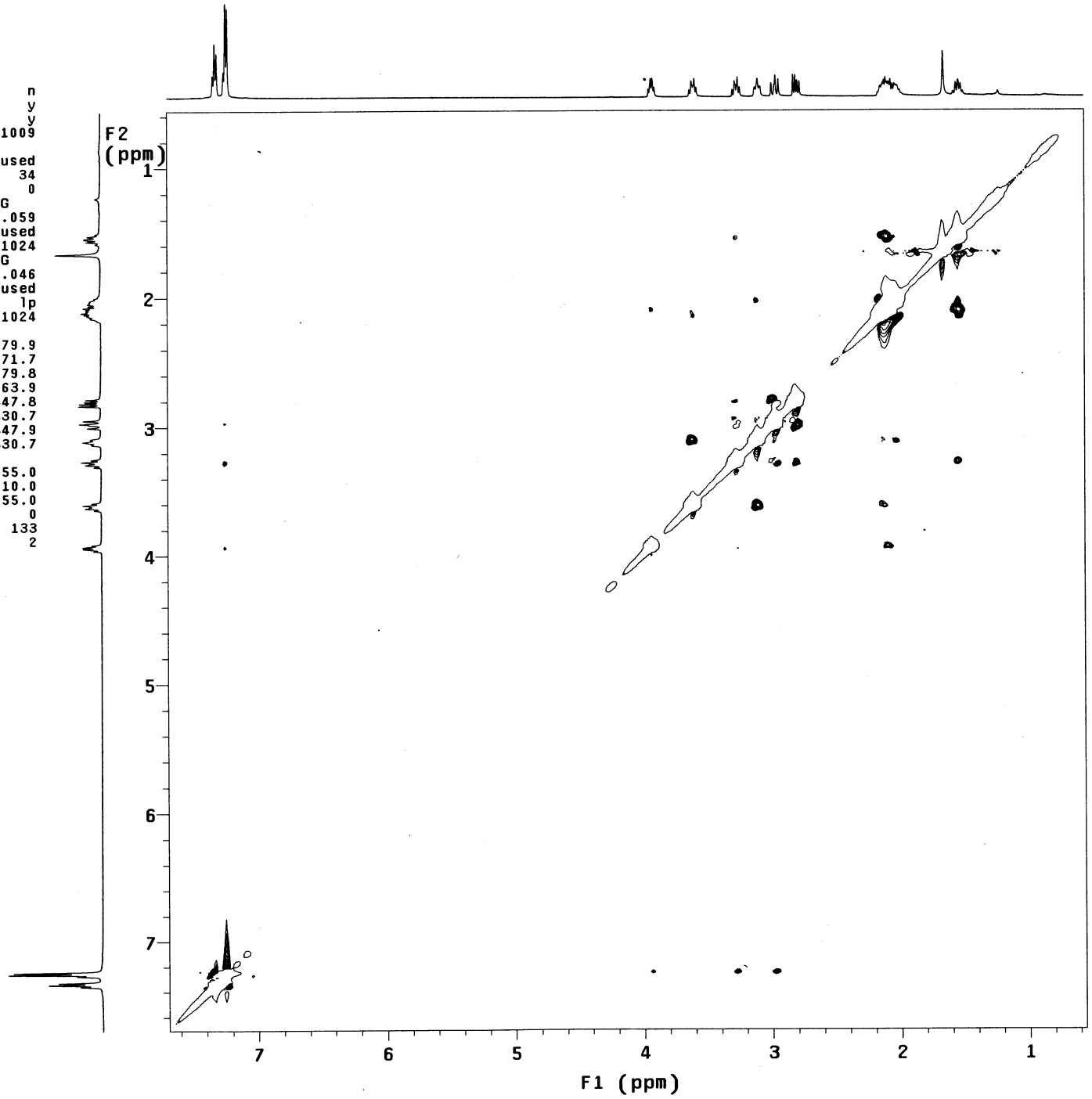


Fig S130. NOESY of compound syn-4a

LCH-02-332

exp65 NOESY

SAMPLE		FLAGS	
date	Dec 4 2014	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1009
sw	4001.6	SPECIAL	
at	0.128	temp	not used
np	1024	gain	34
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.059
nt	16	gfs	not used
2D ACQUISITION		fn	1024
sw1	4001.6	F1 PROCESSING	
ni	200	gf1	0.046
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.832	fn1	1024
tof	-499.9	DISPLAY	
tpwr	61	sp	279.9
pw	13.900	wp	3571.7
NOESY		sp1	279.8
mix	0.600	wp1	3563.9
PRESATURATION		rf1	847.8
satmode	nnnn	rfp	830.7
satpwr	0	rf11	847.9
satdly	0	rfp1	830.7
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	133
		th	2
		ai ph	



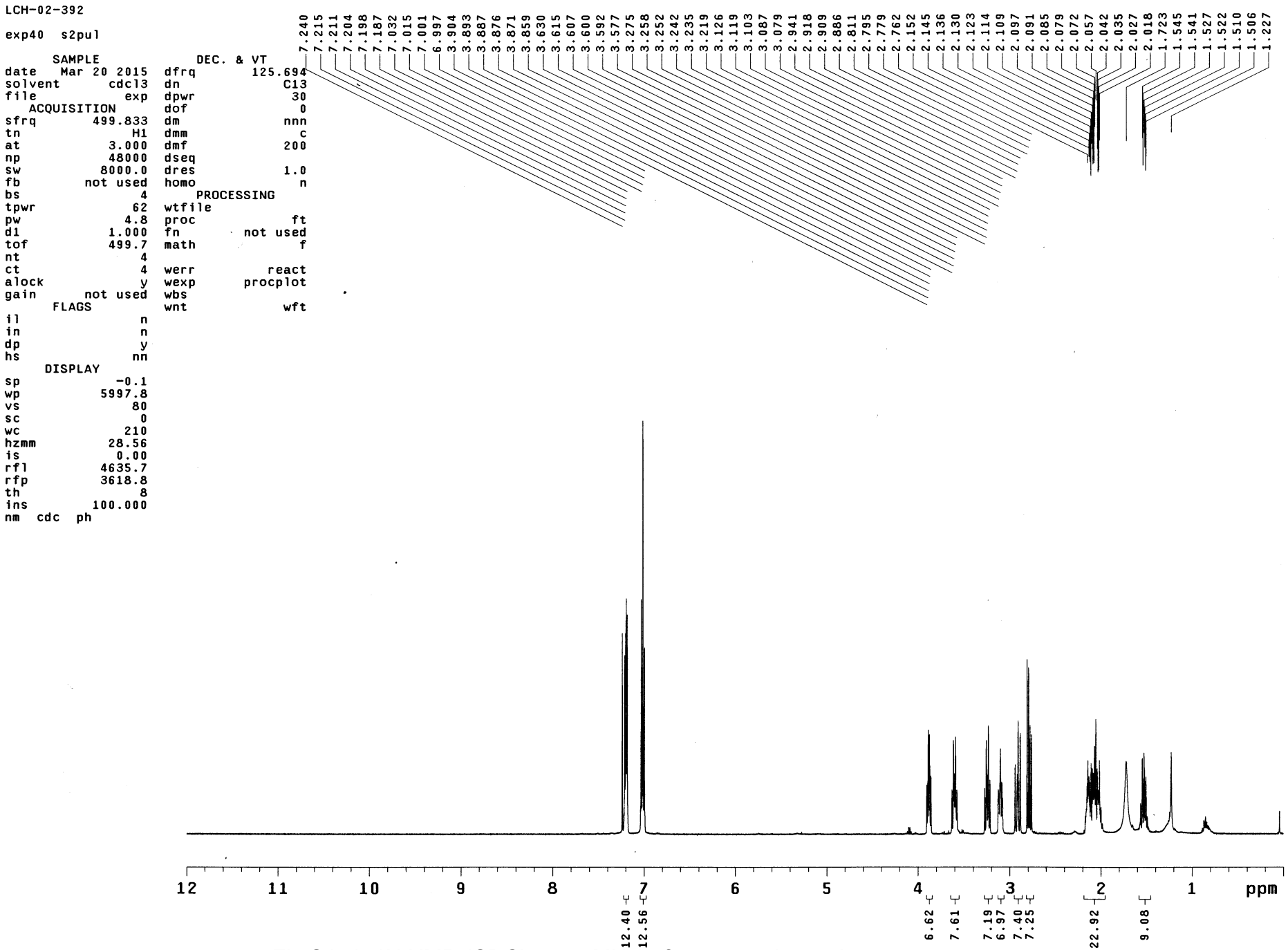


Fig S131. 1H NMR (CDCl3, 500 MHz) of compound syn-4b

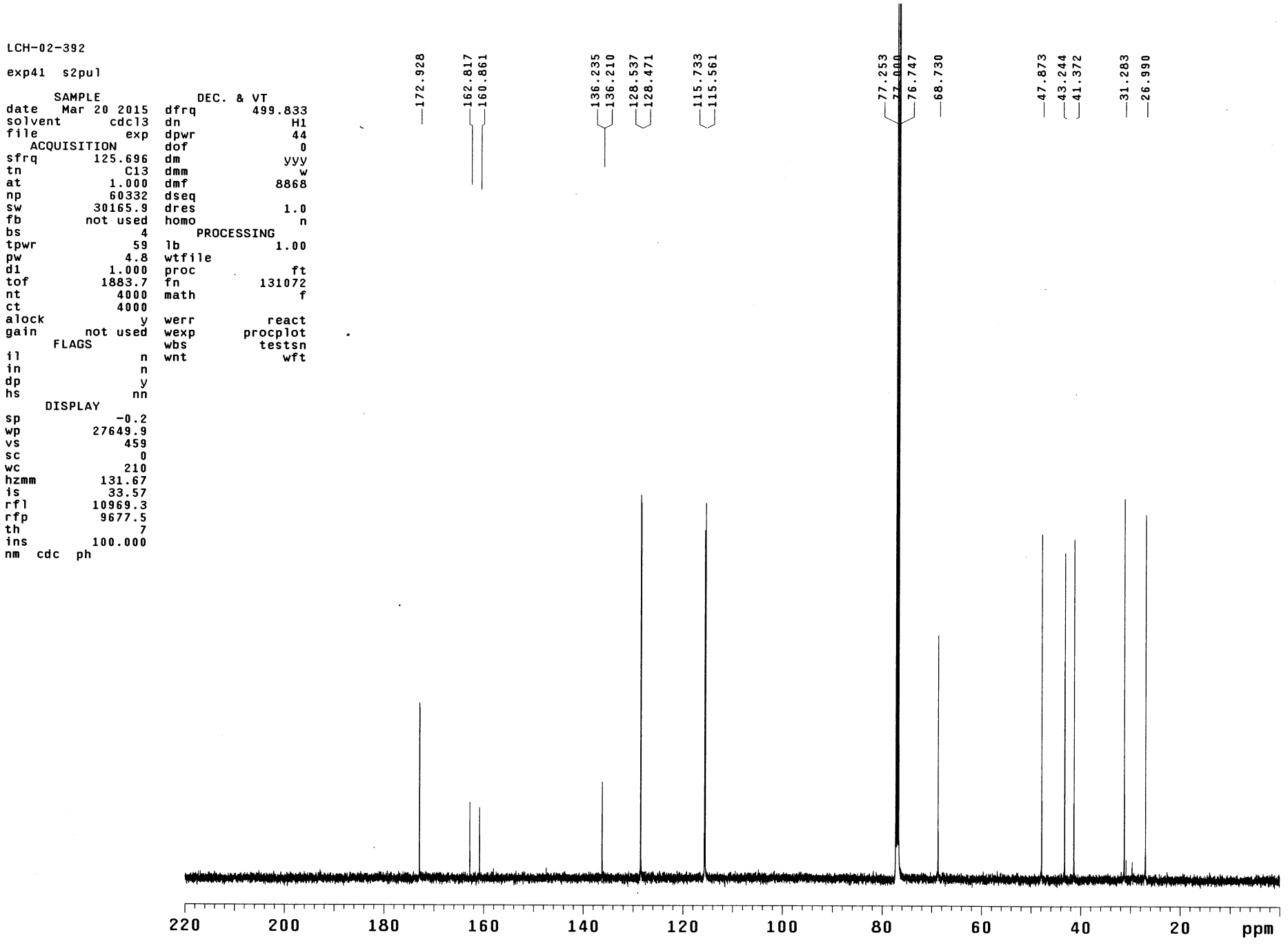


Fig S132. 13C NMR (CDCl3, 125 MHz) of compound syn-4b

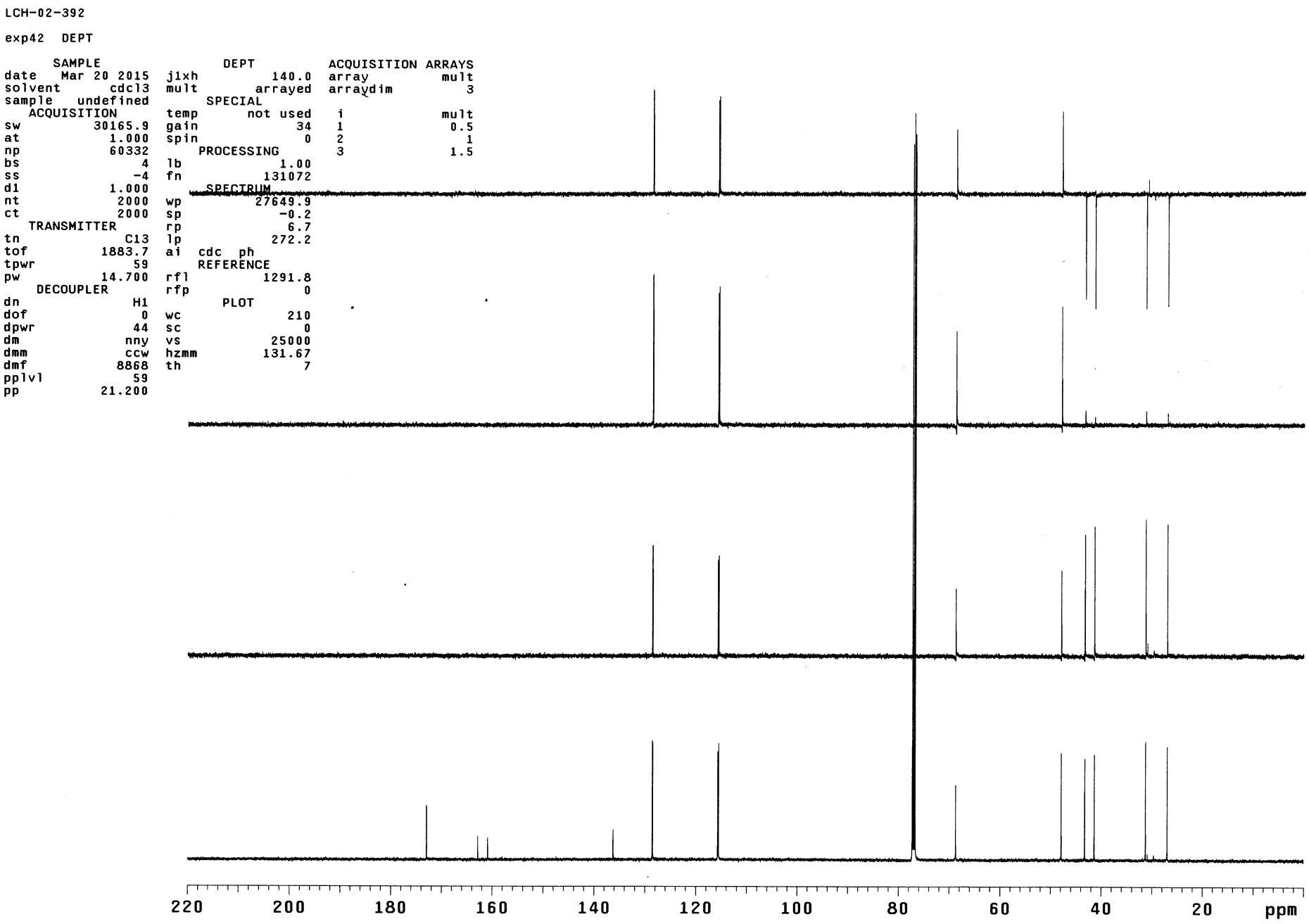


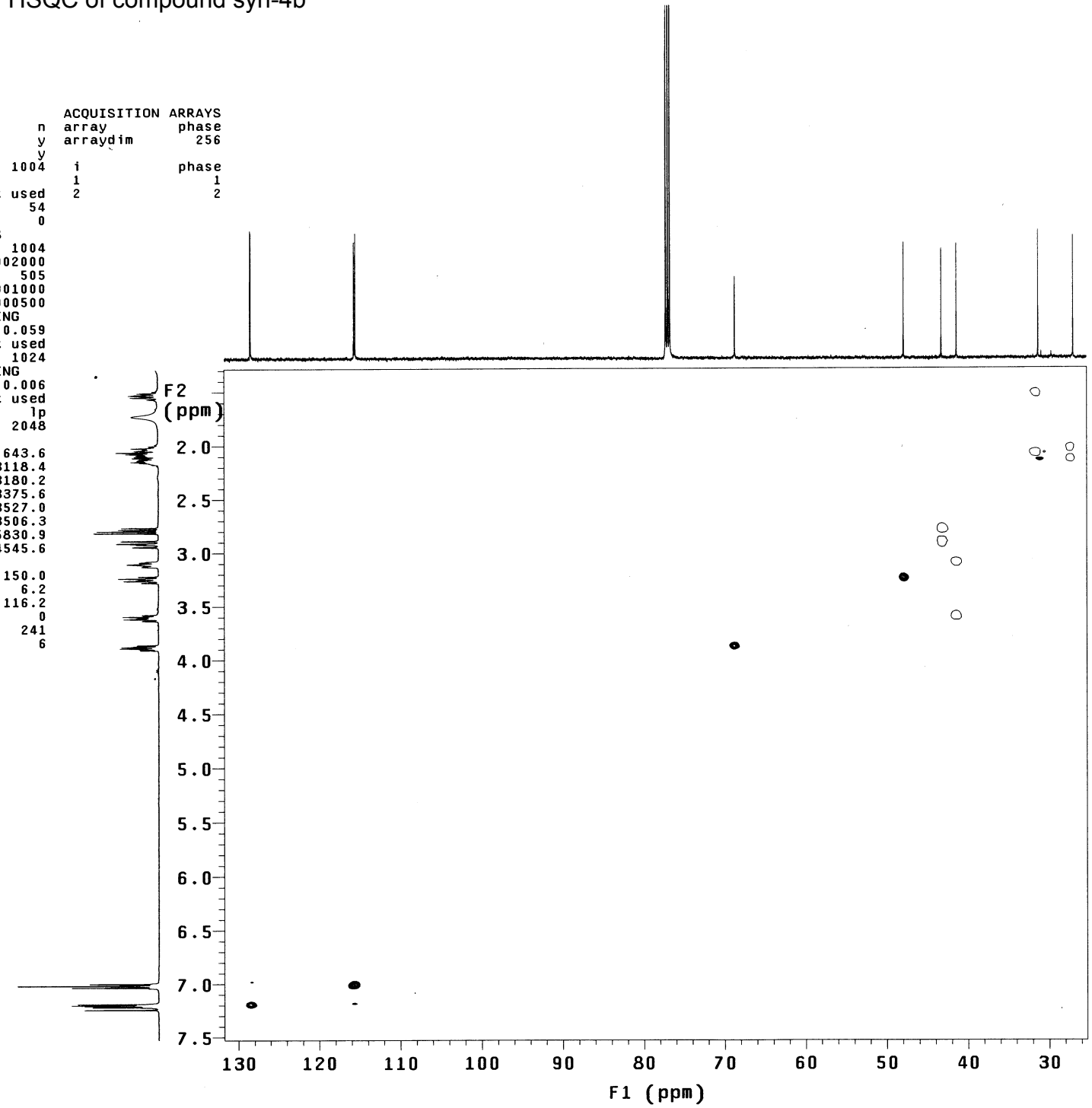
Fig S133. DEPT of compound syn-4b

Fig S134. HSQC of compound syn-4b

LCH-02-392

exp45 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Mar 20 2015	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1004	i	phase
sw	4001.6	SPECIAL		1	1
at	0.128	temp	not used	2	2
np	1024	gain	54		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1004		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.832	fn	1024		
tof	-499.9	F1 PROCESSING			
tpwr	62	gf1	0.006		
pw	13.800	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	643.6		
dmm	ccp	wp	3118.4		
dmf	32258	sp1	3180.2		
dpwr	43	wp1	13375.6		
pwxlvl	61	rfl	3527.0		
pwx	11.000	rfp	3506.3		
HSQC		rfl1	15830.9		
j1xh	140.0	rfp1	14545.6		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	241		
		th	6		
		ai cdc ph			



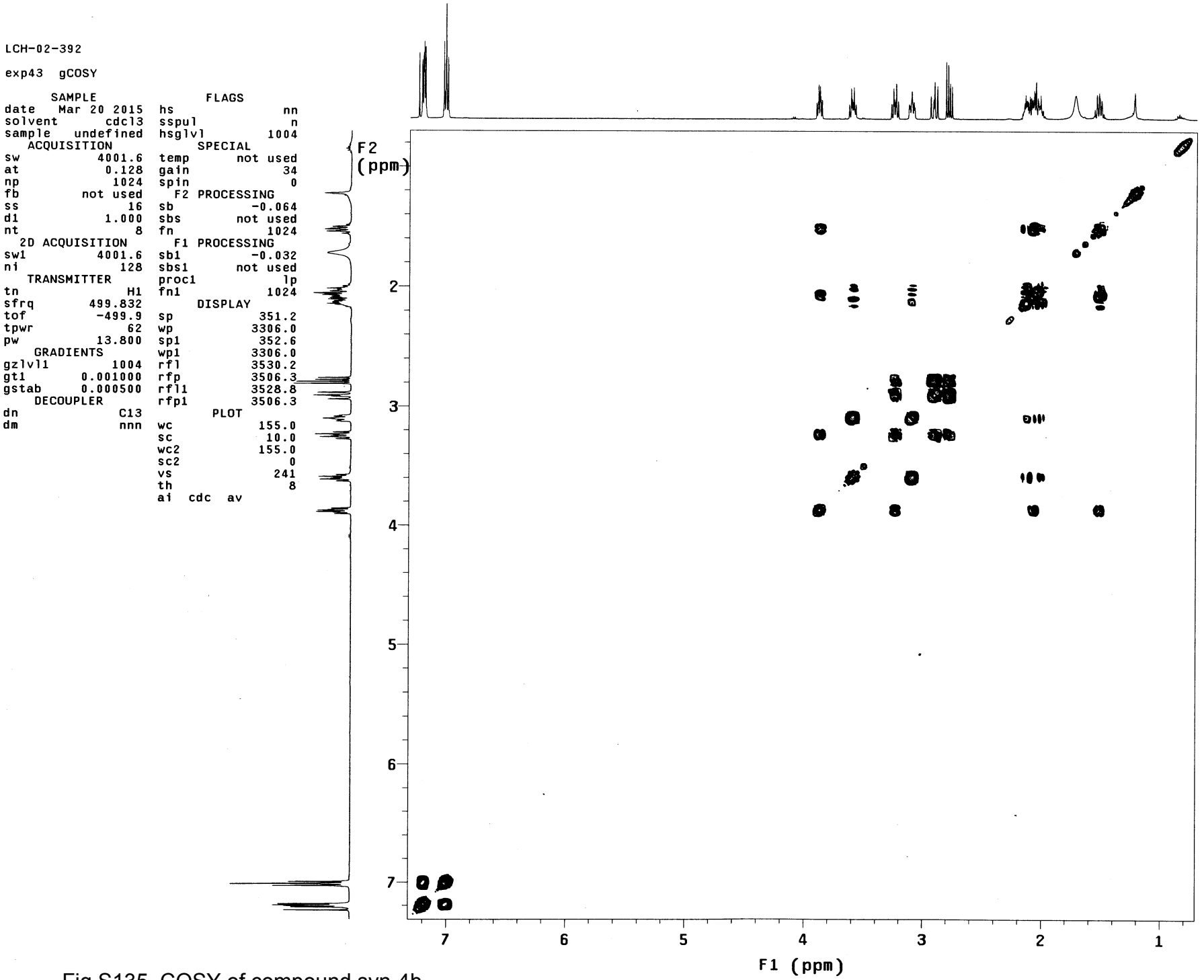


Fig S135. COSY of compound syn-4b

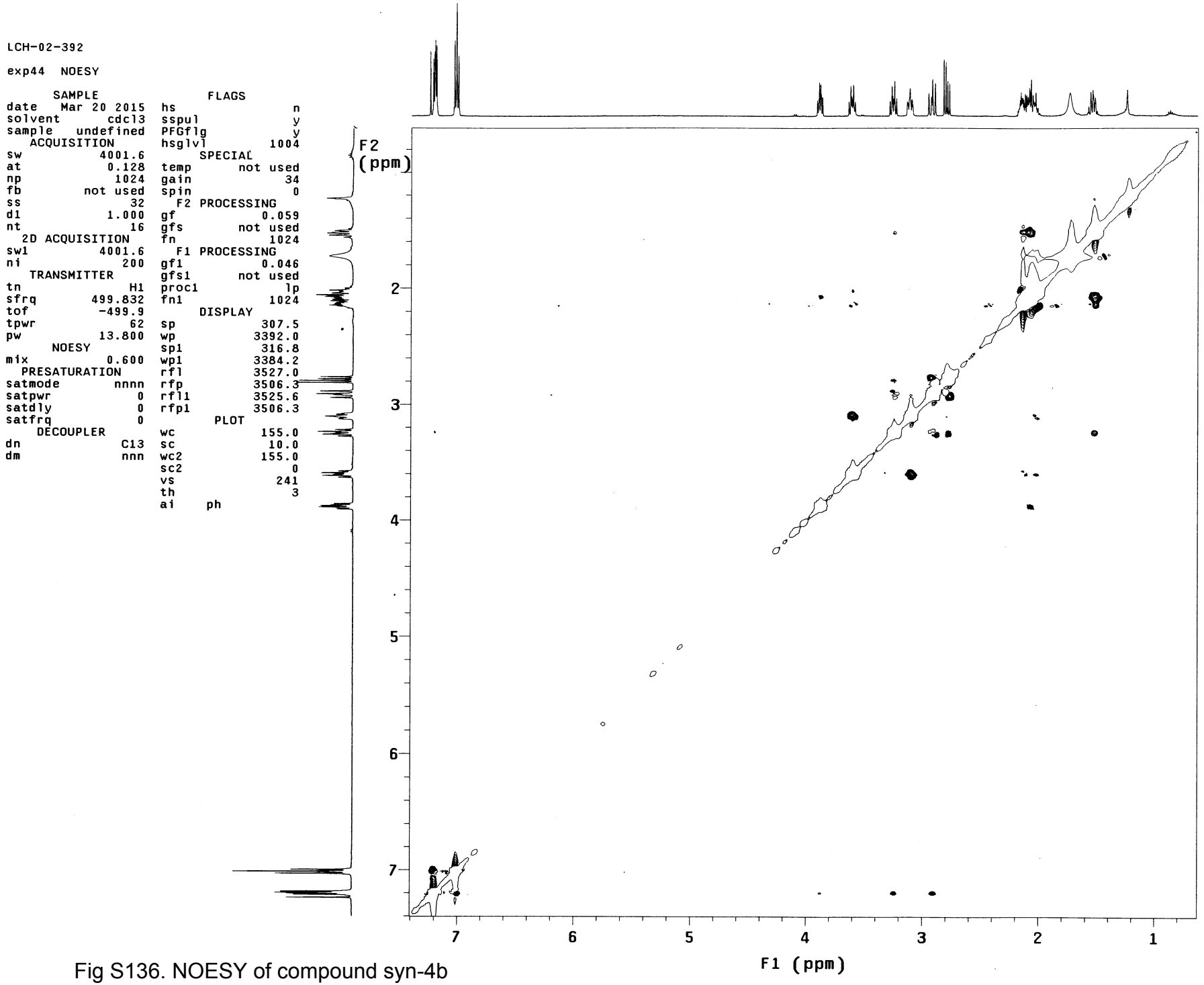
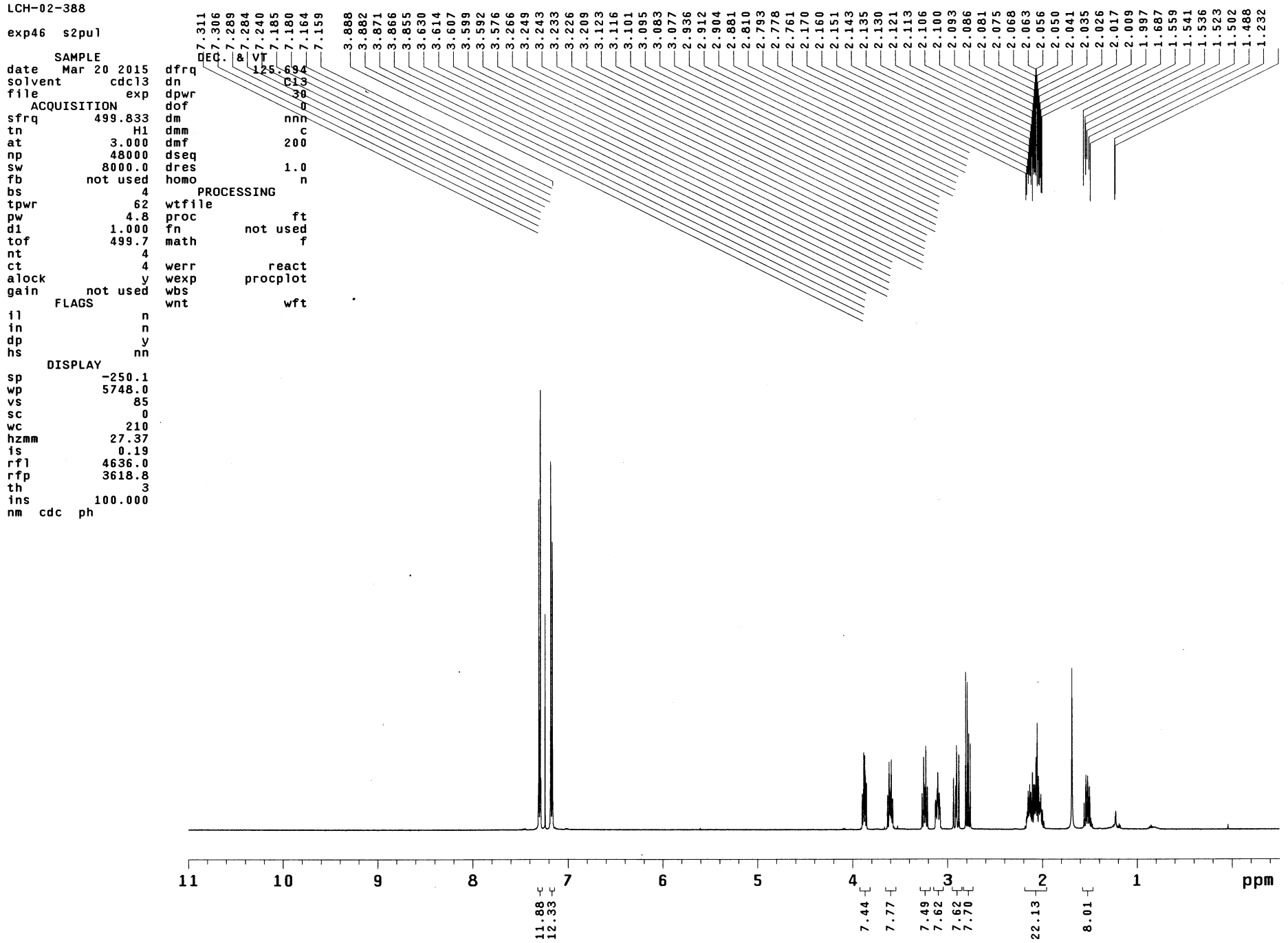


Fig S136. NOESY of compound syn-4b

Fig S137. 1H NMR (CDCl3, 500 MHz NMR) of compound syn-4c



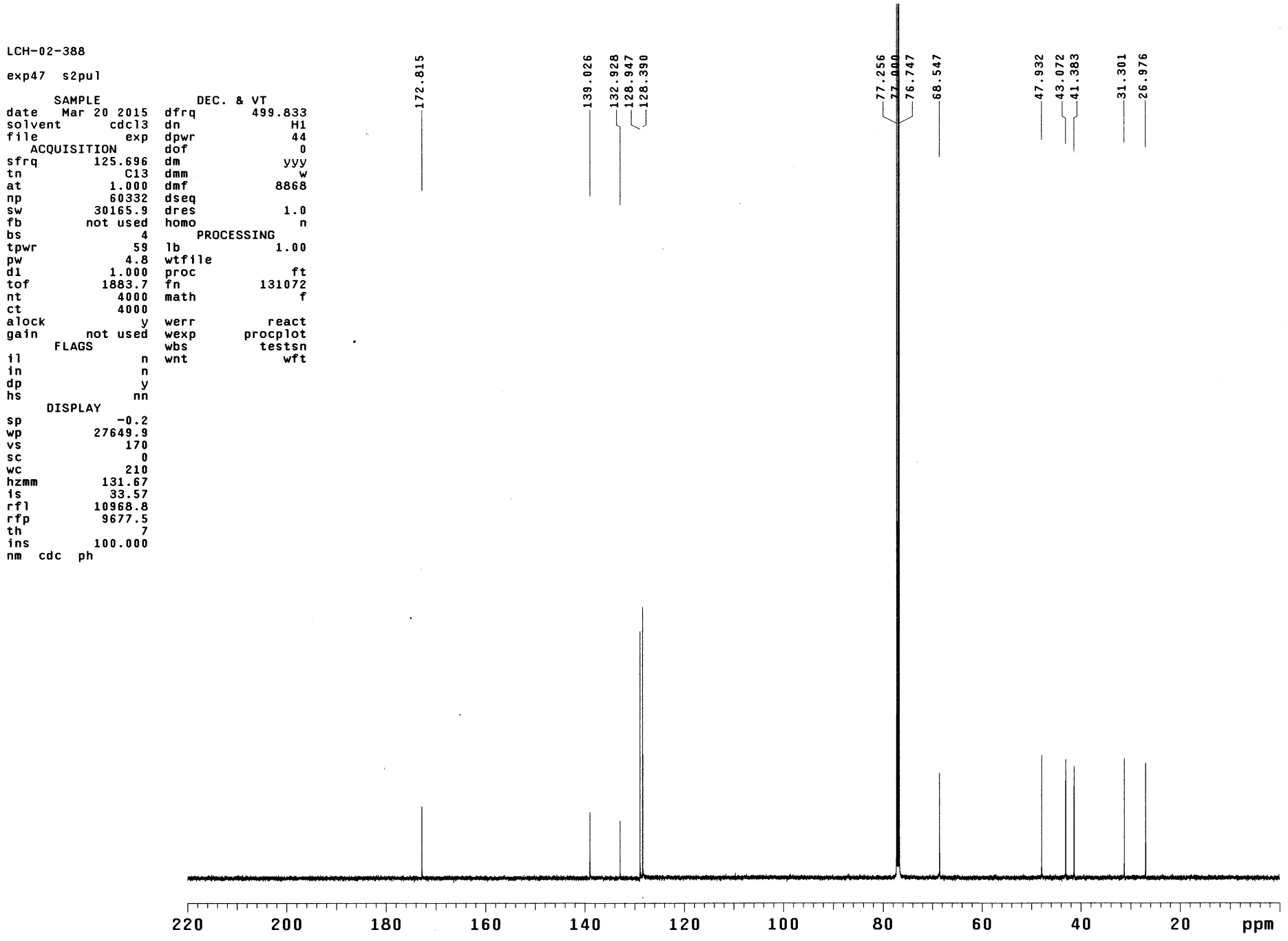
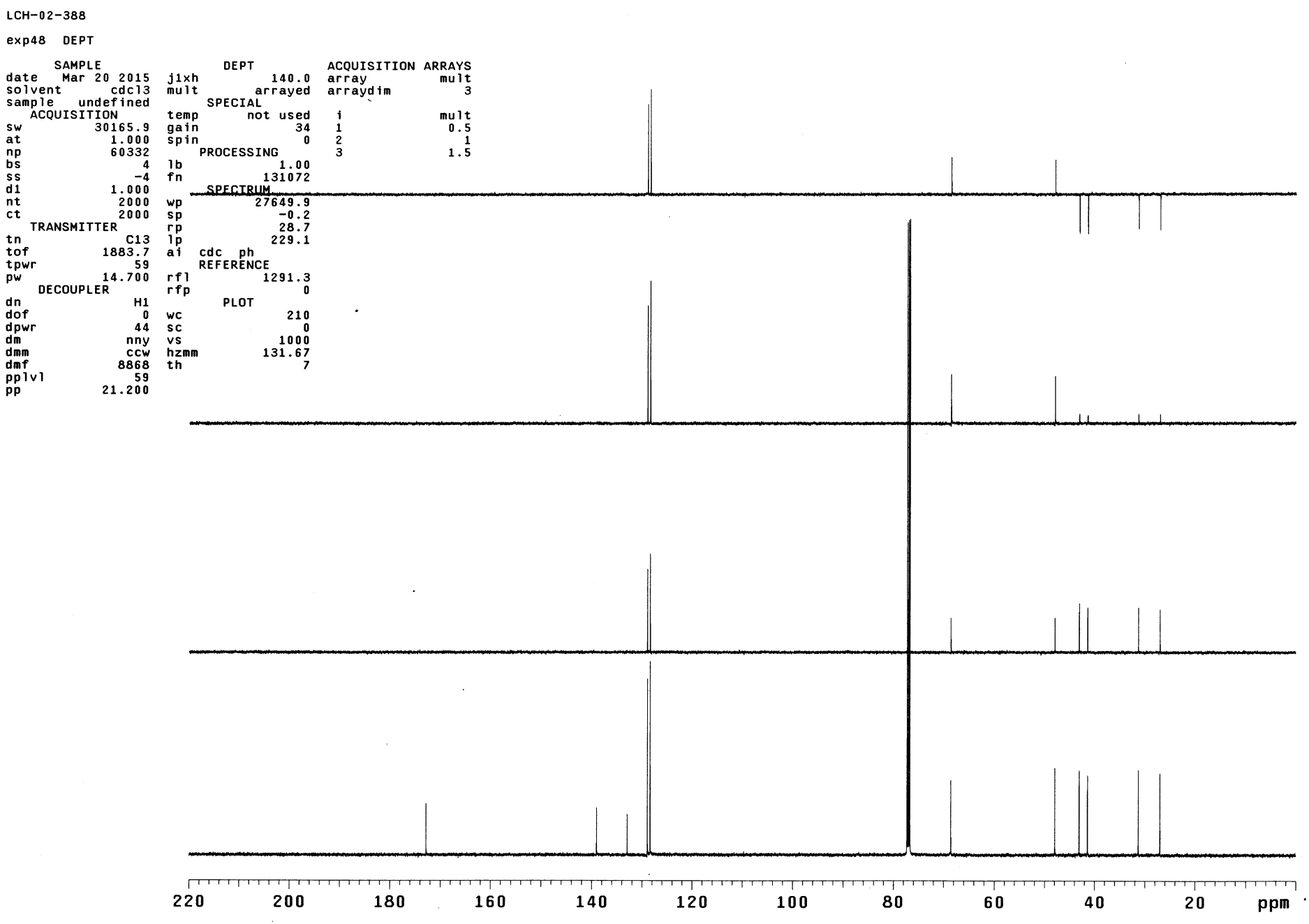


Fig S138. 13C NMR (CDCl3, 125 MHz NMR) of compound syn-4c

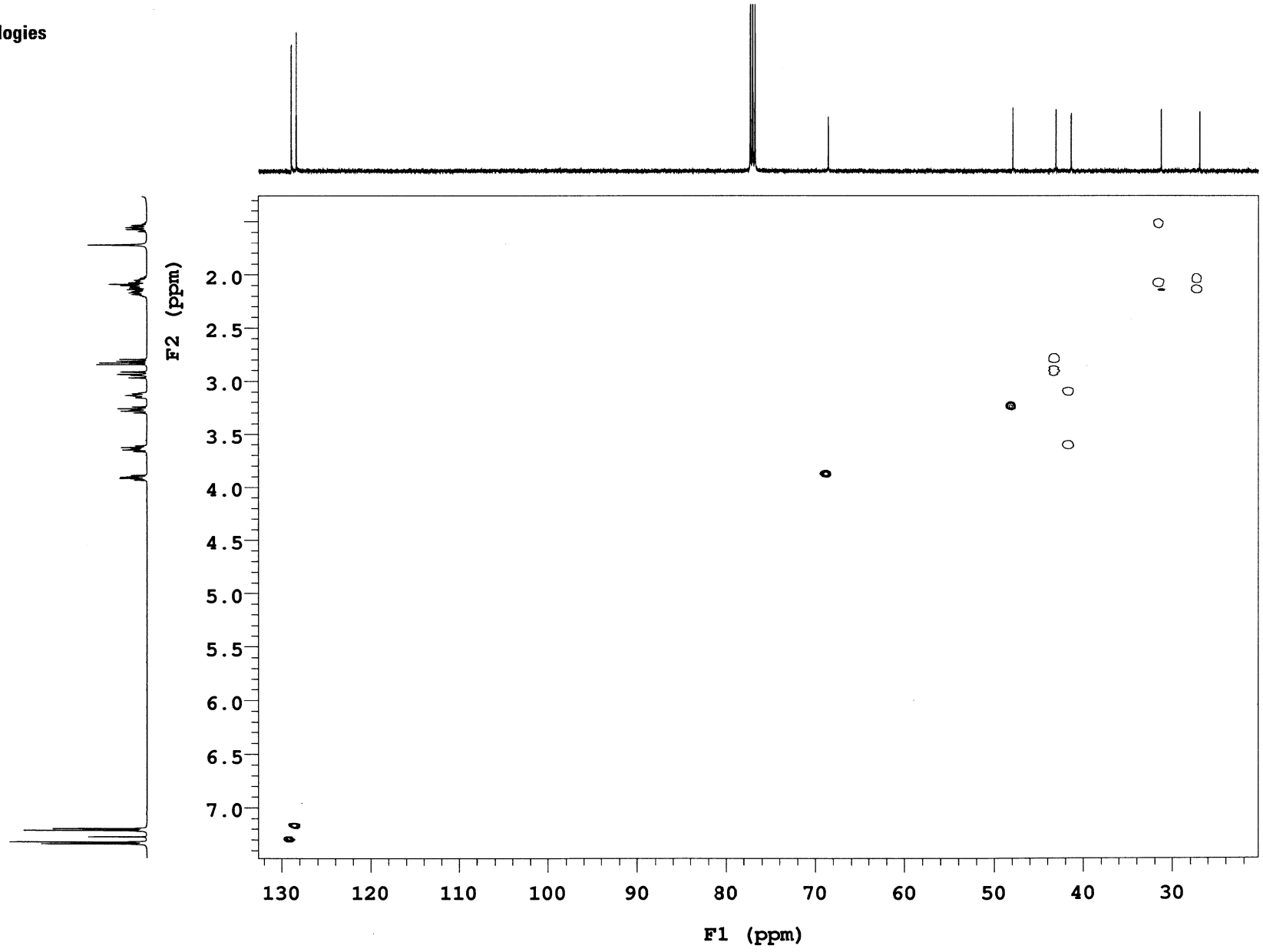
Fig S139. DEPT of compound syn-4c



LCH-02-388

Sample Name	LCH-02-388	Pulse sequence	gHSQC	Temperature	50	Study owner	vnmr2
Date collected	2015-04-23	Solvent	cdcl3	Spectrometer	---	Operator	vnmr2

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-388/HSQC.fid

Fig S140. HSQC of compound syn-4c

Plot date 2015-10-29

Fig S141. COSY of compound syn-4c

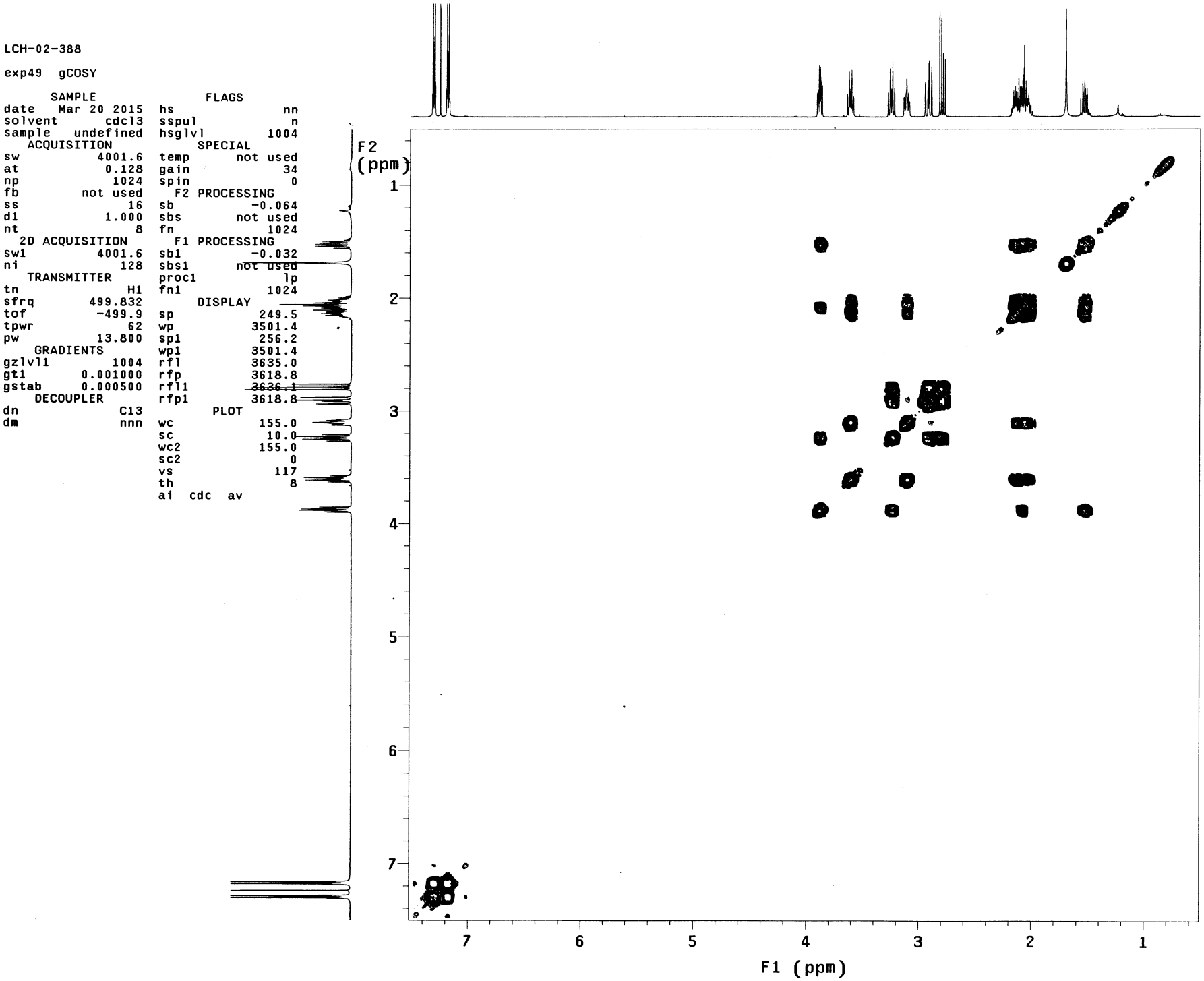
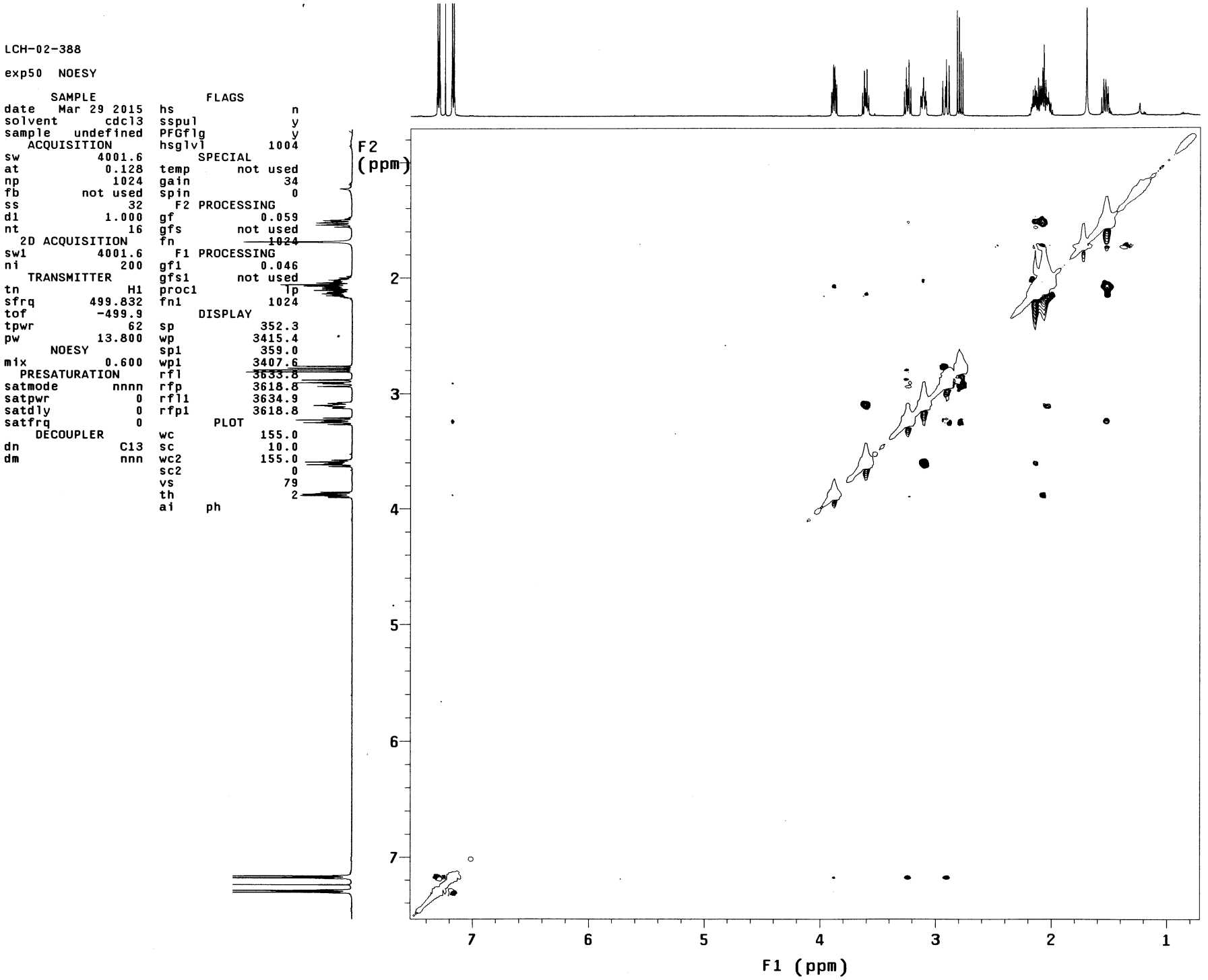


Fig S142. NOESY of compound syn-4c



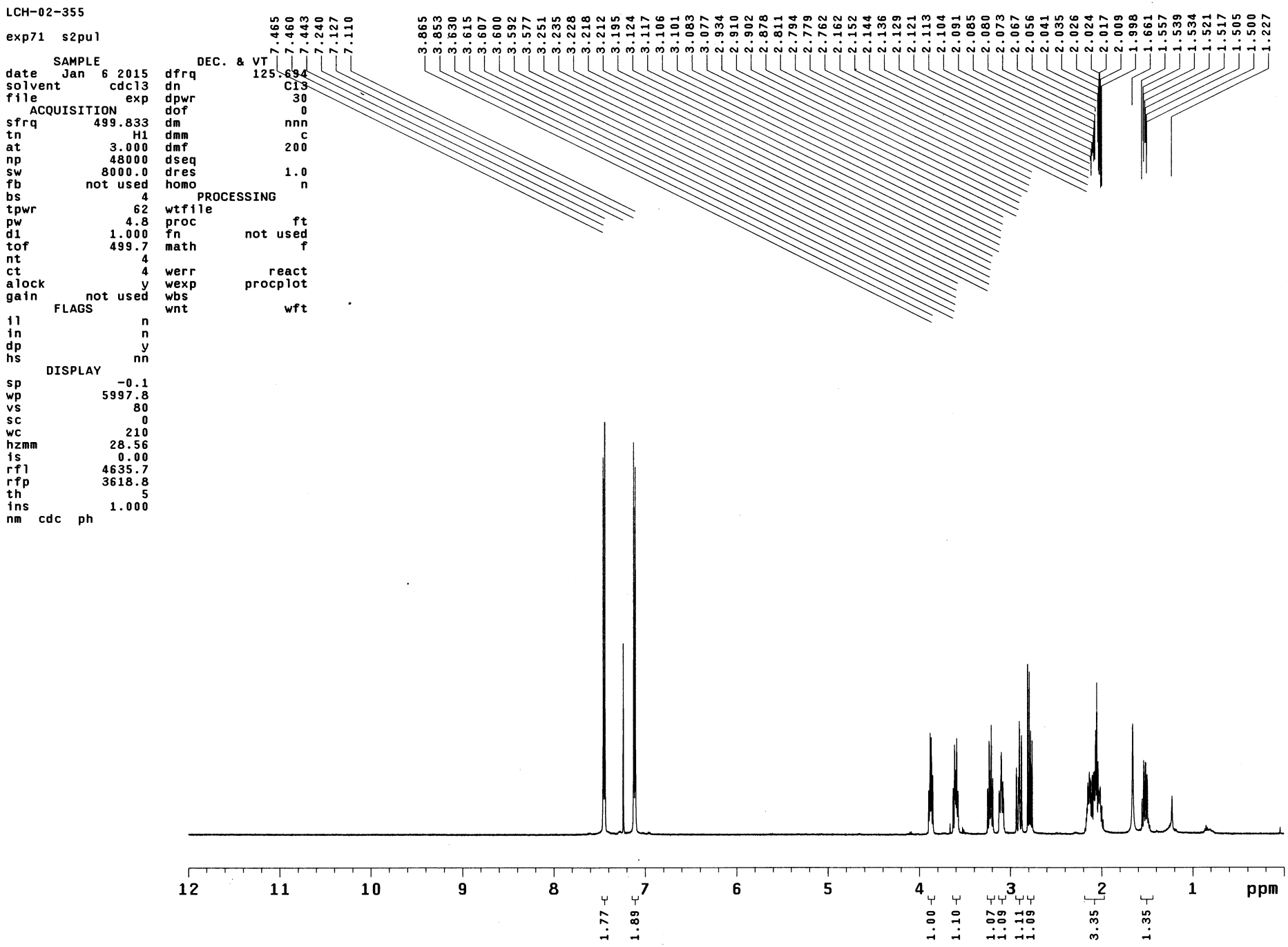


Fig S143. 1H NMR (CDCl3, 500 MHz) of compound syn-4d

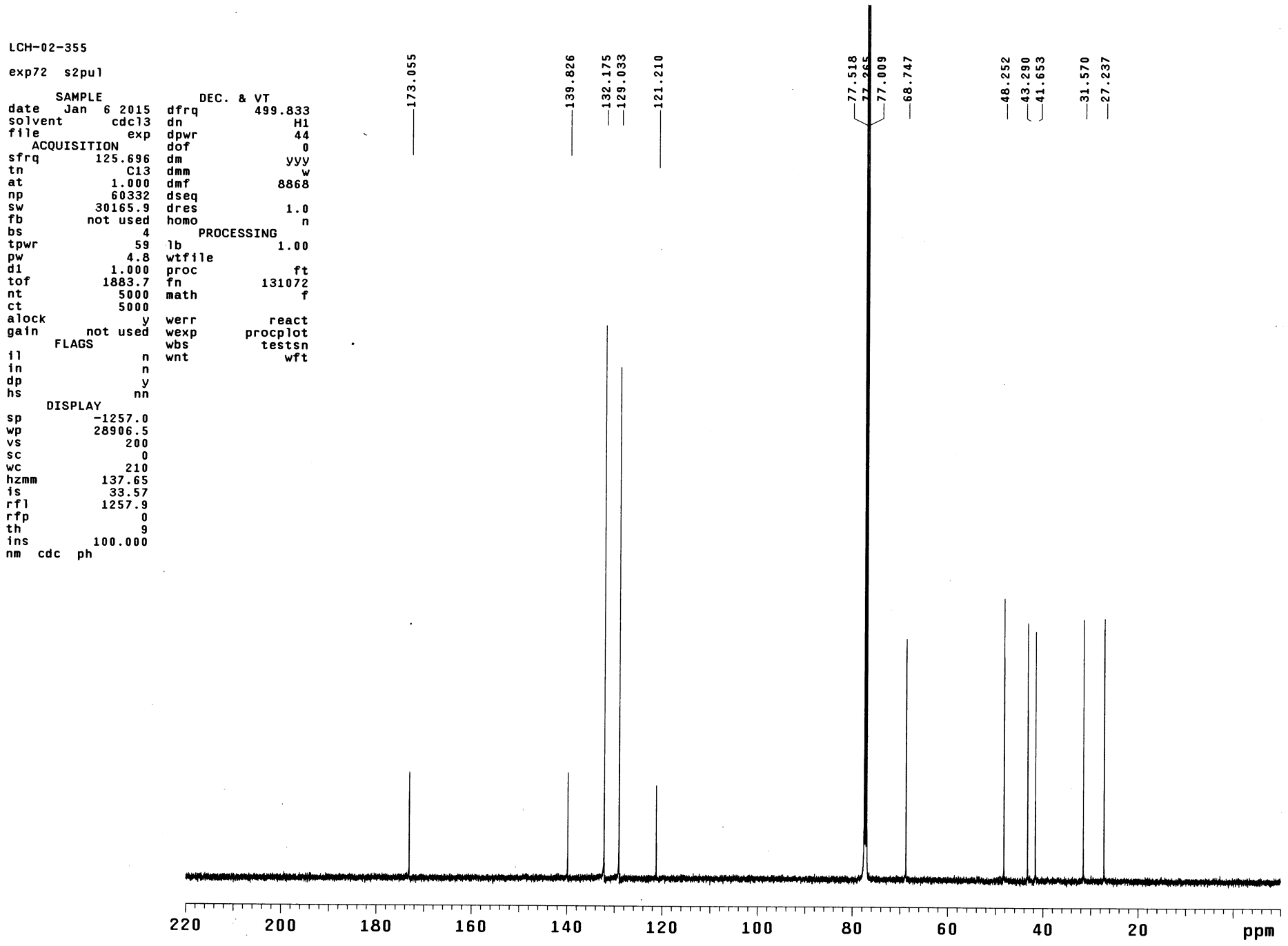


Fig S144. 13C NMR (CDCl3, 125 MHz) of compound syn-4d

Fig S145. DEPT of compound syn-4d

LCH-02-355

exp73 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Jan 6 2015	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	30165.9	gain	34	1	0.5
at	1.000	spin	0	2	1
np	60332	PROCESSING		3	1.5
bs	4	lb	1.00		
ss	-4	fn	131072		
d1	1.000	SPECTRUM			
nt	2500	wp	28906.5		
ct	2500	sp	-1257.2		
TRANSMITTER		rp	-160.4		
tn	C13	lp	220.7		
tof	1883.7	ai	cdc ph		
tpwr	59	REFERENCE			
pw	14.700	rfl	1290.8		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	44	sc	0		
dm	nny	vs	600		
dmm	ccw	hzmm	137.65		
dmf	8868	th	7		
pplvl	59				
pp	21.200				

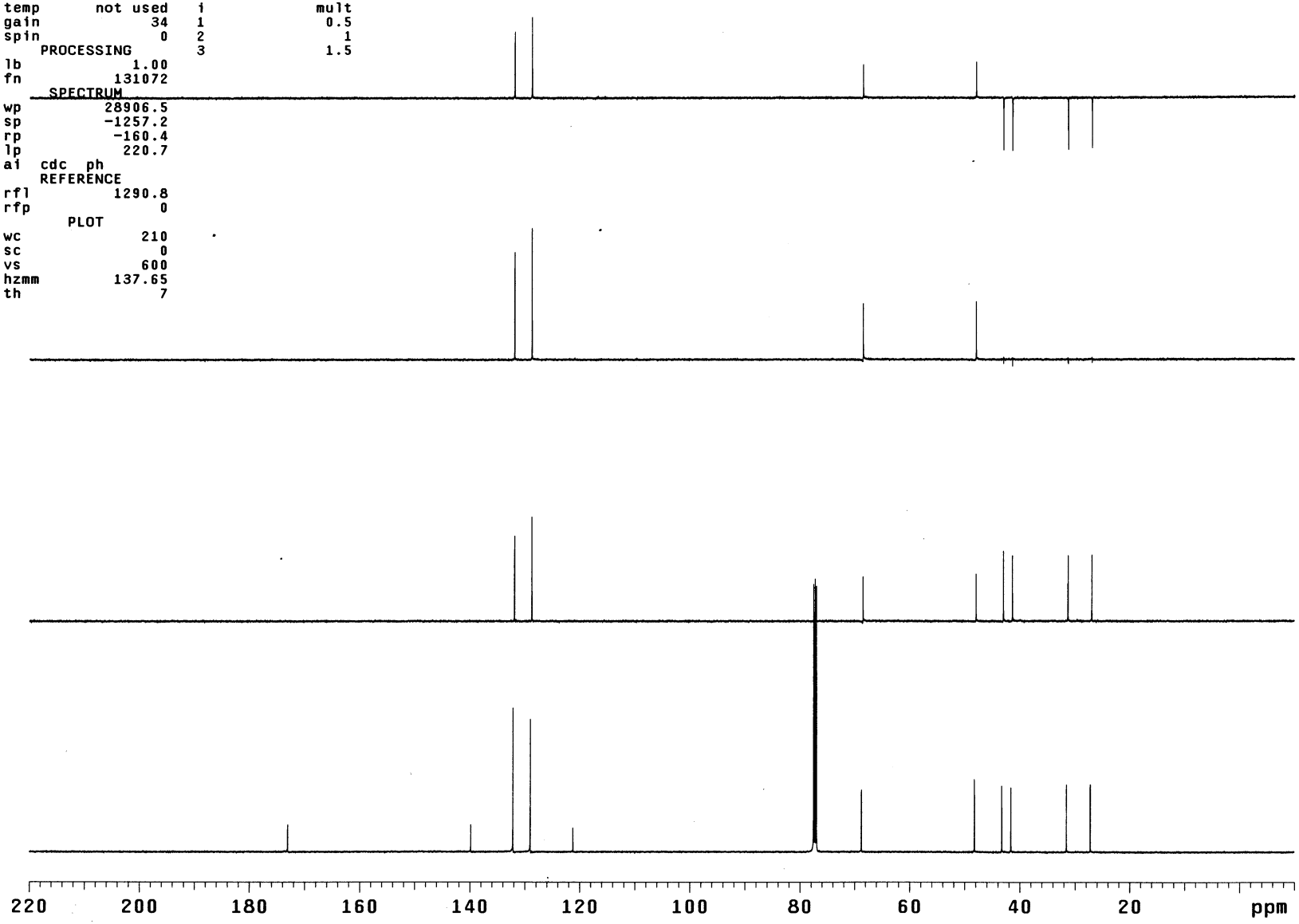


Fig S146. HSQC of compound syn-4d

LCH-02-355

exp76 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Jan 6 2015	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1004	i	phase
sw	4001.6	SPECIAL		1	1
at	0.128	temp	not used	2	2
np	1024	gain	54		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1004		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.832	fn	1024		
tof	-499.9	F1 PROCESSING			
tpwr	62	gf1	0.006		
pw	13.800	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	715.9		
dmm	ccp	wp	3118.4		
dmf	32258	sp1	3226.7		
dpwr	43	wp1	13730.3		
pwxlvl	61	rf1	3739.7		
pwx	11.000	rfp	3728.7		
HSQC		rfl1	17871.8		
j1xh	140.0	rfp1	16612.1		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	232		
		th	6		
		ai cdc ph			

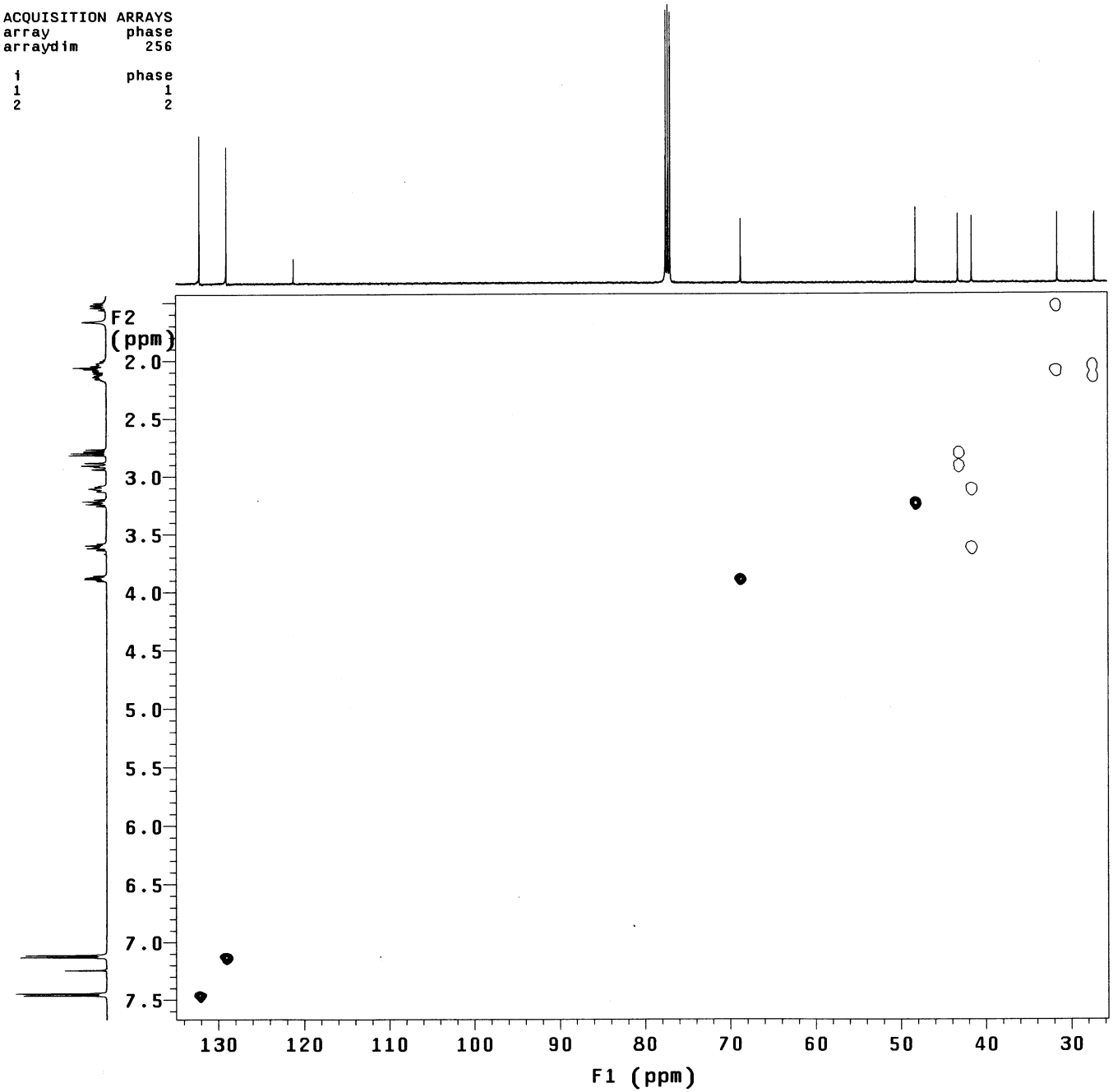


Fig S147. COSY of compound syn-4d

LCH-02-355

exp74 gCOSY

SAMPLE		FLAGS	
date	Jan 6 2015	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1004
ACQUISITION		SPECIAL	
sw	4001.6	temp	not used
at	0.128	gain	34
np	1024	spin	0
fb	not used	F2 PROCESSING	
ss	16	sb	-0.064
d1	1.000	sbs	not used
nt	8	fn	1024
2D ACQUISITION		F1 PROCESSING	
sw1	4001.6	sb1	-0.032
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	1024
sfrq	499.832	DISPLAY	
tof	-499.9	sp	348.4
tpwr	62	wp	3540.5
pw	13.800	sp1	340.1
GRADIENTS		wp1	3548.3
gzlvl1	1004	rfl	3637.7
gt1	0.001000	rfp	3618.8
gstab	0.000500	rfl1	3638.2
DECOUPLER		rfp1	3618.8
dn	C13	PLOT	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	232
		th	9
		ai cdc av	

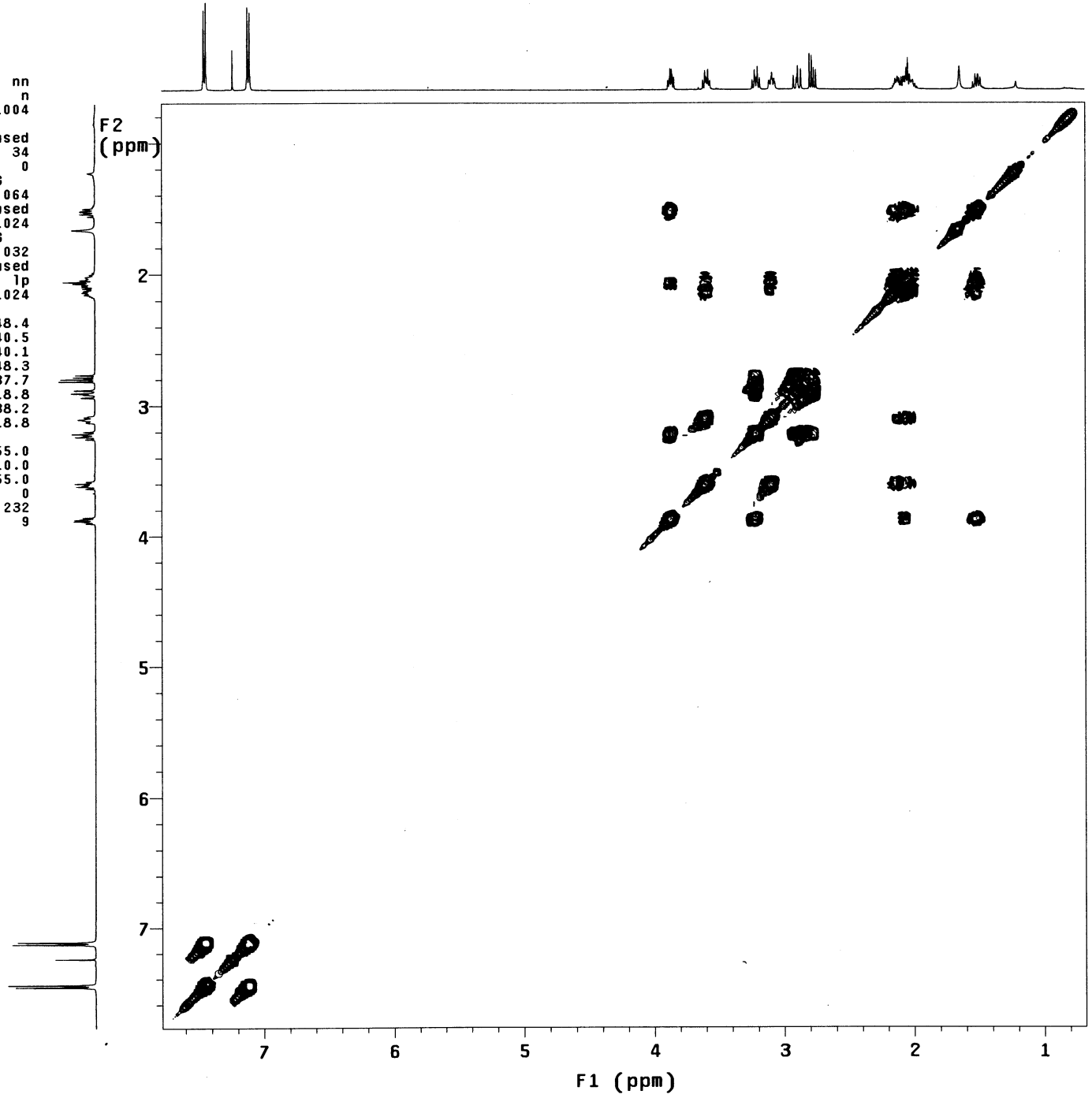


Fig S148. NOESY of compound syn-4d

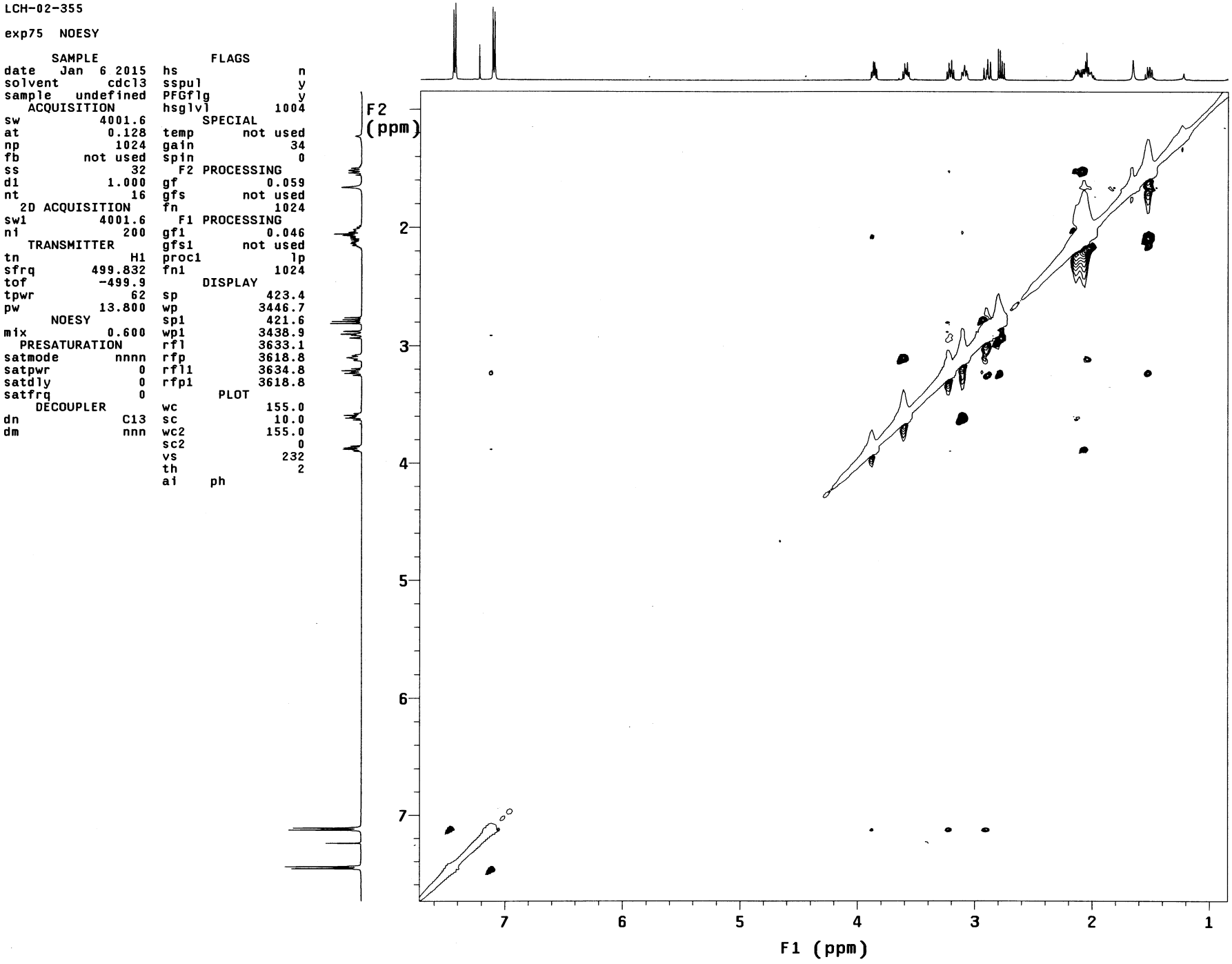
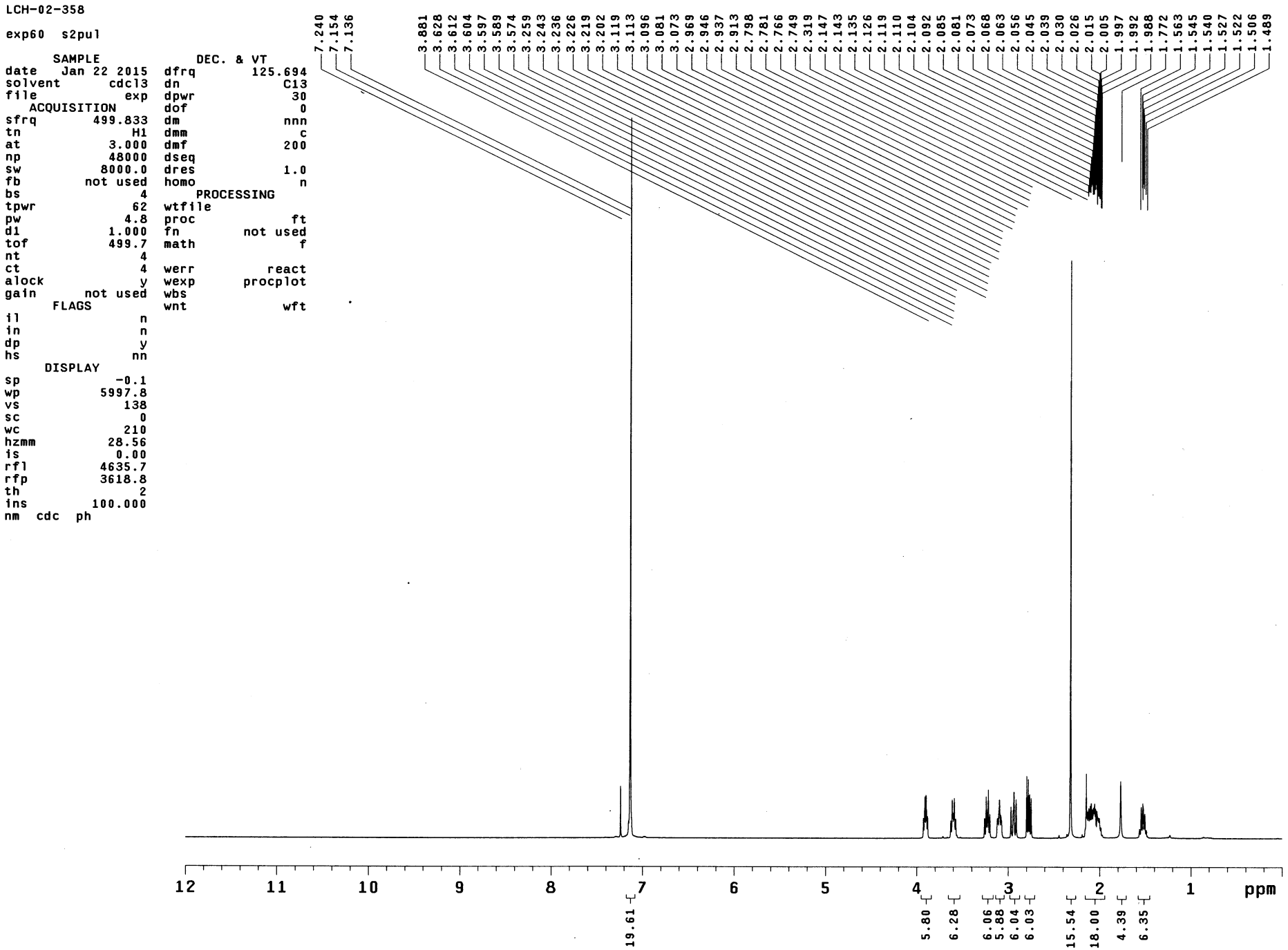


Fig S149. 1H NMR (CDCl3, 500 MHz) of compound syn-4e



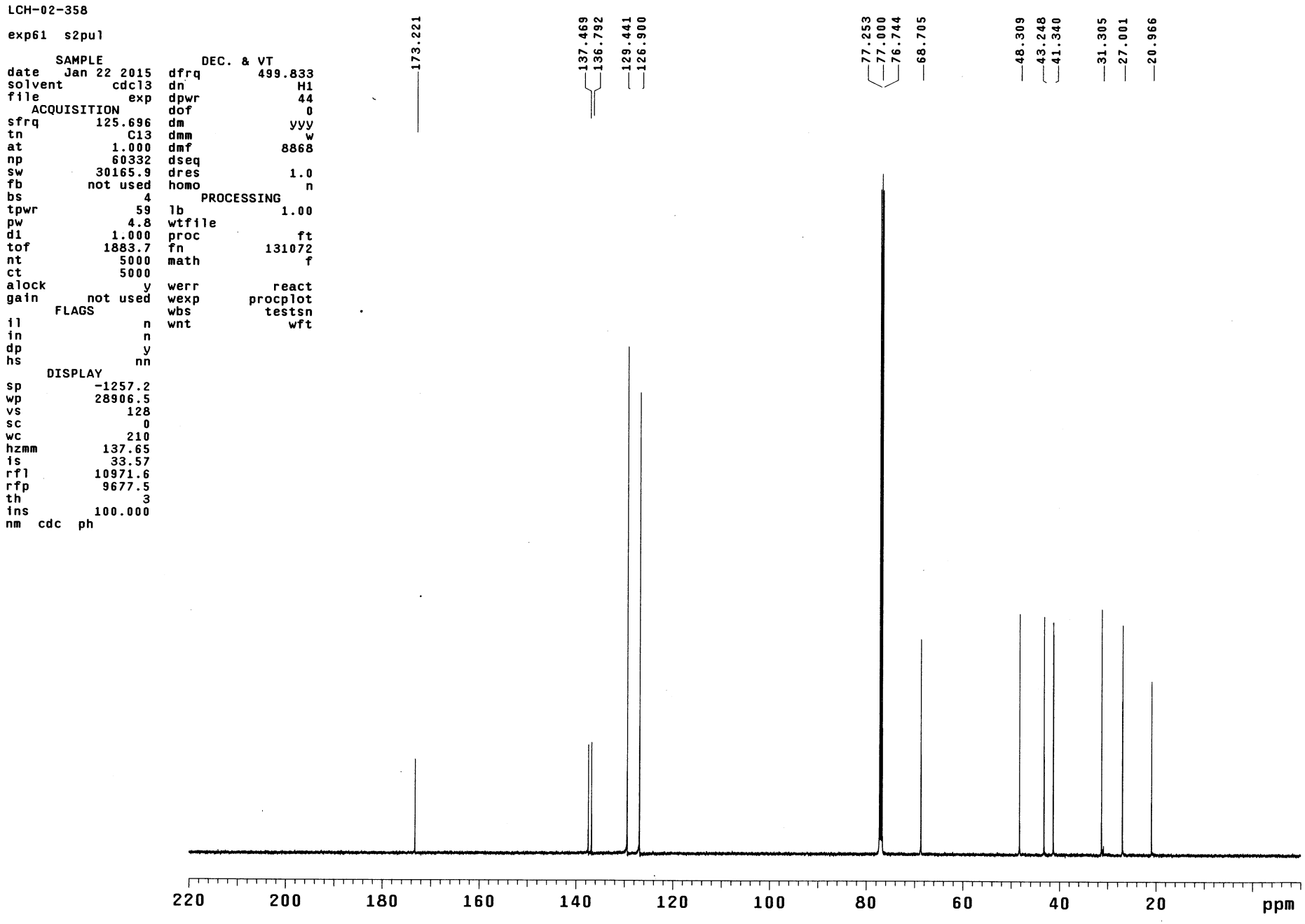


Fig S150. 13C NMR (CDCl3, 125 MHz) of compound syn-4e

Fig S151. DEPT of compound syn-4e

LCH-02-358

exp62 DEPT

SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Jan 22 2015	j1xh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	3
sample	undefined	SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	30165.9	gain	34	1	0.5
at	1.000	spin	0	2	1
np	60332	PROCESSING		3	1.5
bs	4	lb	1.00		
ss	-4	fn	131072		
d1	1.000	SPECTRUM			
nt	2500	wp	28906.5		
ct	2500	sp	-1257.2		
TRANSMITTER		rp	-167.3		
tn	C13	lp	248.4		
tof	1883.7	ai	cdc ph		
tpwr	59	REFERENCE			
pw	14.700	rfl	1294.1		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	210		
dpwr	44	sc	0		
dm	nny	vs	300		
dmm	ccw	hzmm	137.65		
dmf	8868	th	7		
pplvl	59				
pp	21.200				

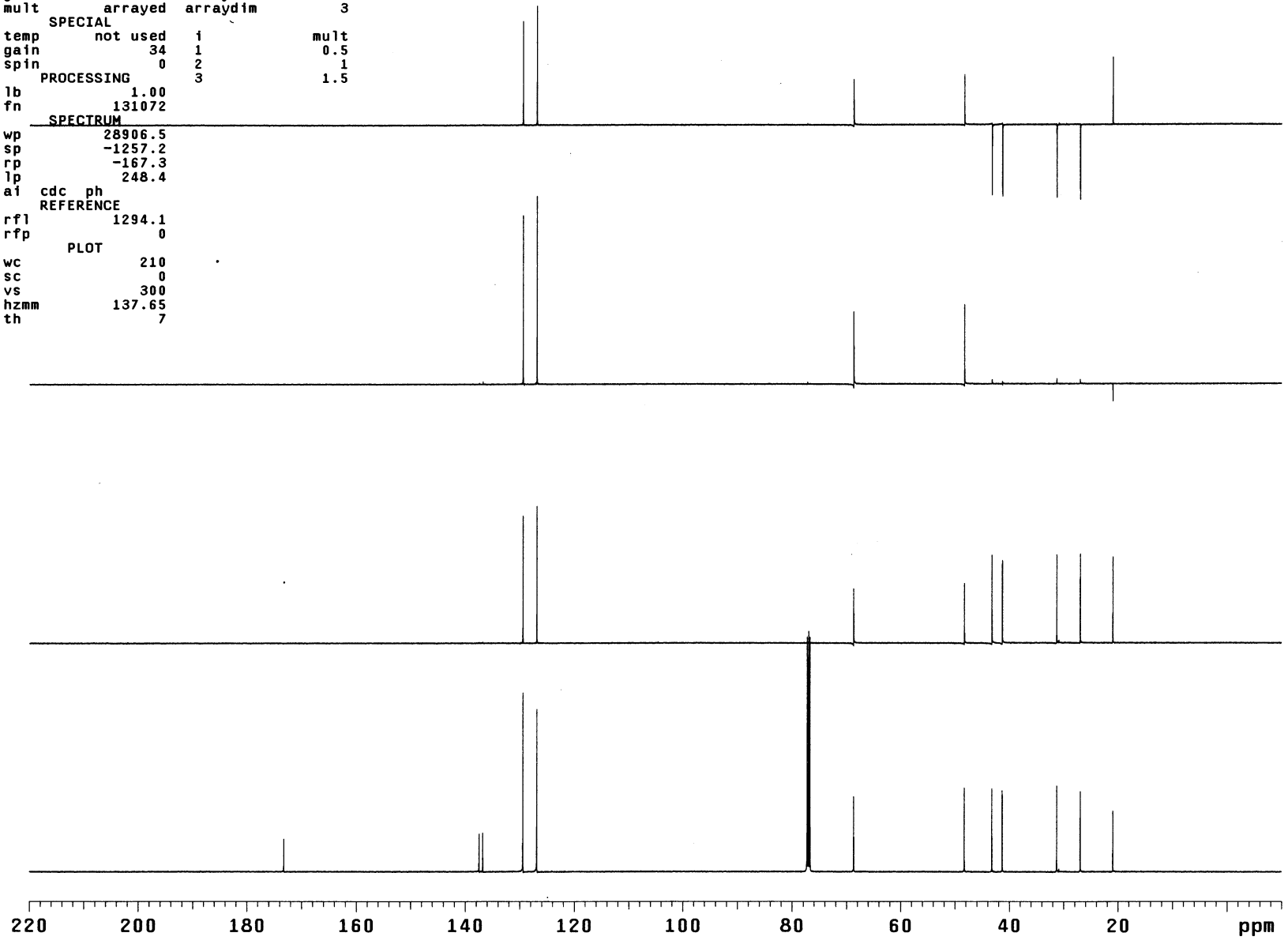
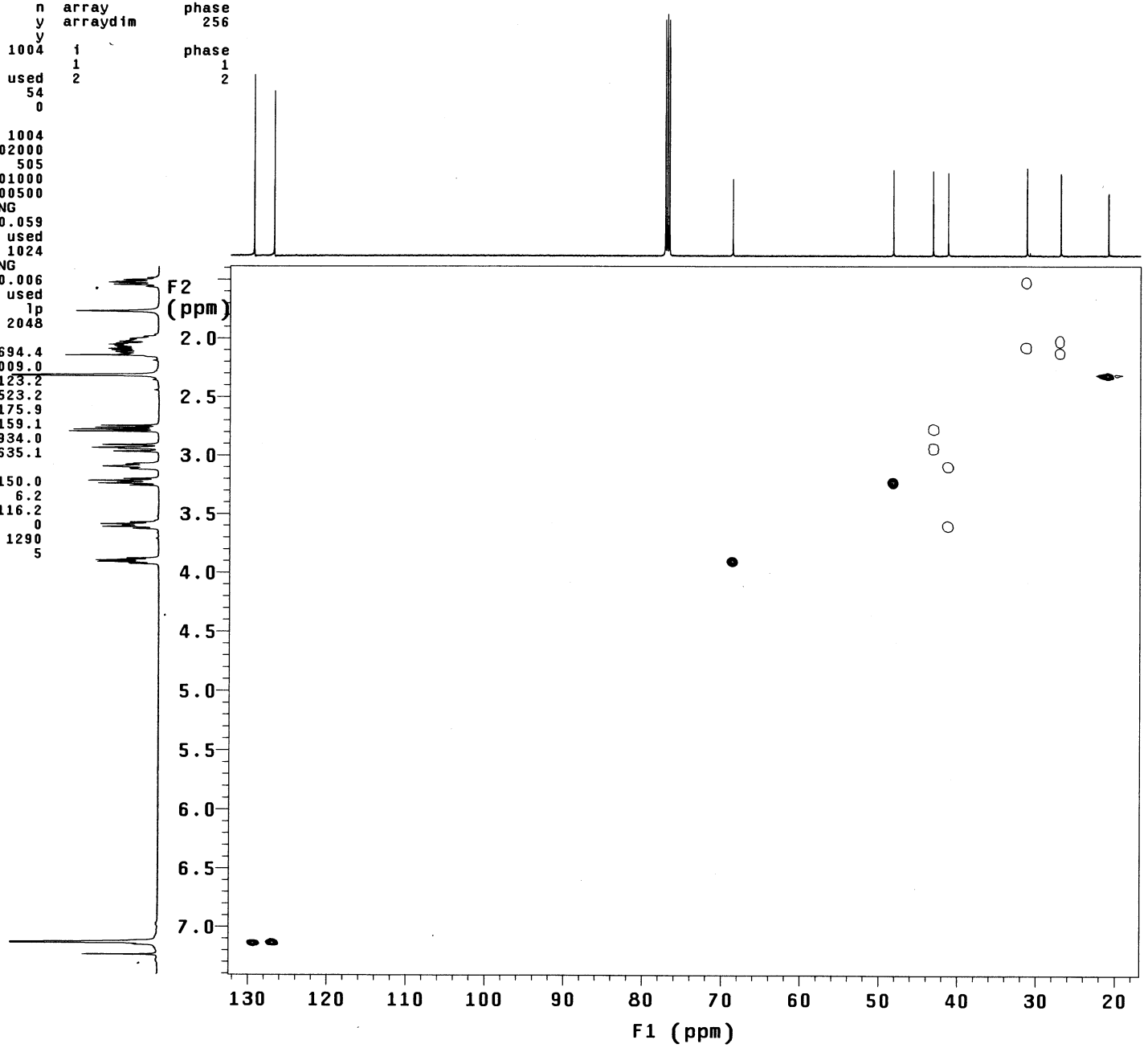


Fig S152. HSQC of compound syn-4e

LCH-02-358

exp65 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Jan 22 2015	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1004	1	phase
sw	4001.6	SPECIAL		1	1
at	0.128	temp	not used	2	2
np	1024	gain	54		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1004		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.832	fn	1024		
tof	-499.9	F1 PROCESSING			
tpwr	62	gf1	0.006		
pw	13.800	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	694.4		
dmm	ccp	wp	3009.0		
dmf	32258	sp1	2123.2		
dpwr	43	wp1	14523.2		
pwxlvl	61	rfl	1175.9		
pwx	11.000	rfp	1159.1		
HSQC		rfl1	3934.0		
j1xh	140.0	rfp1	2635.1		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	1290		
		th	5		
		ai cdc ph			



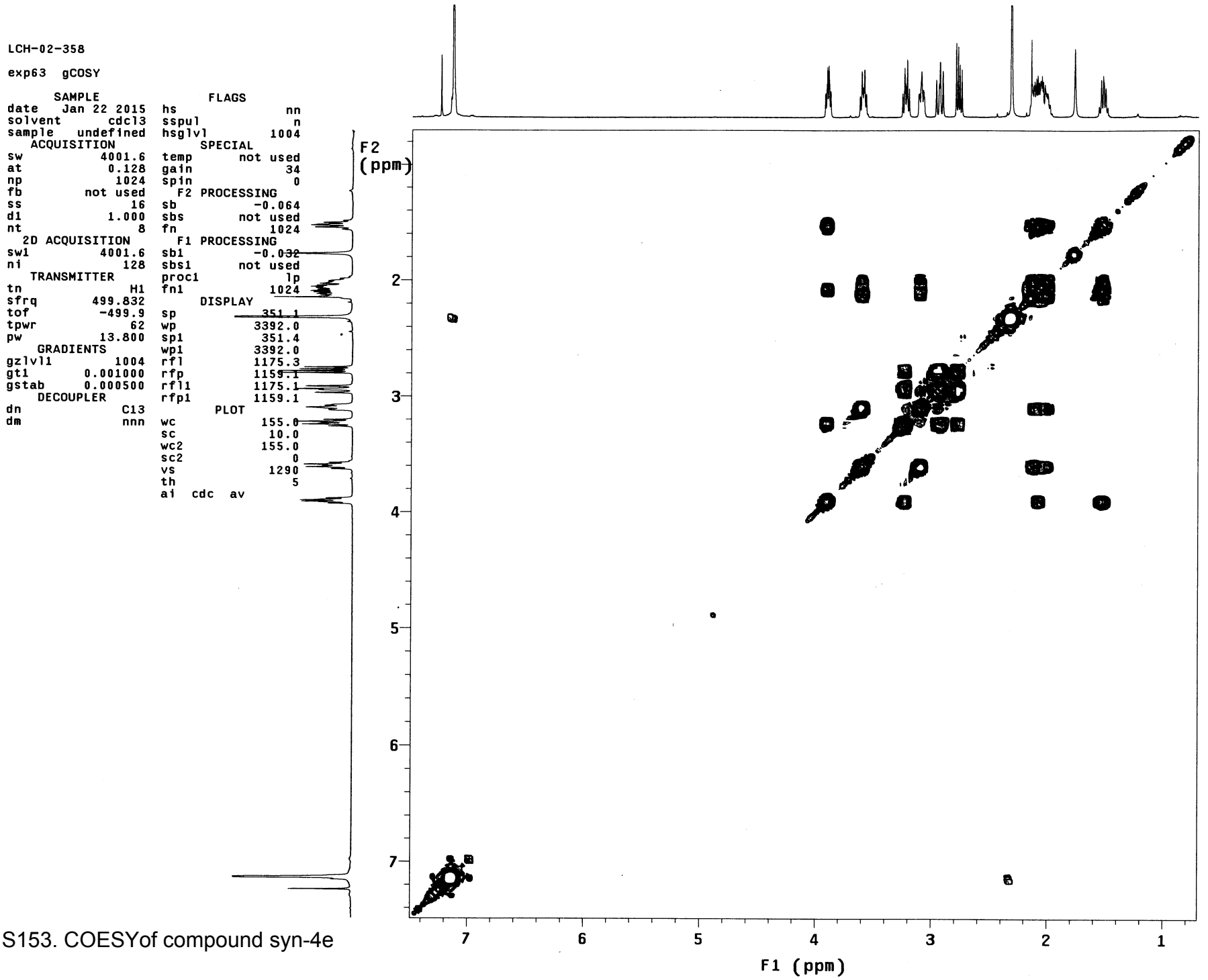


Fig S153. COESYof compound syn-4e

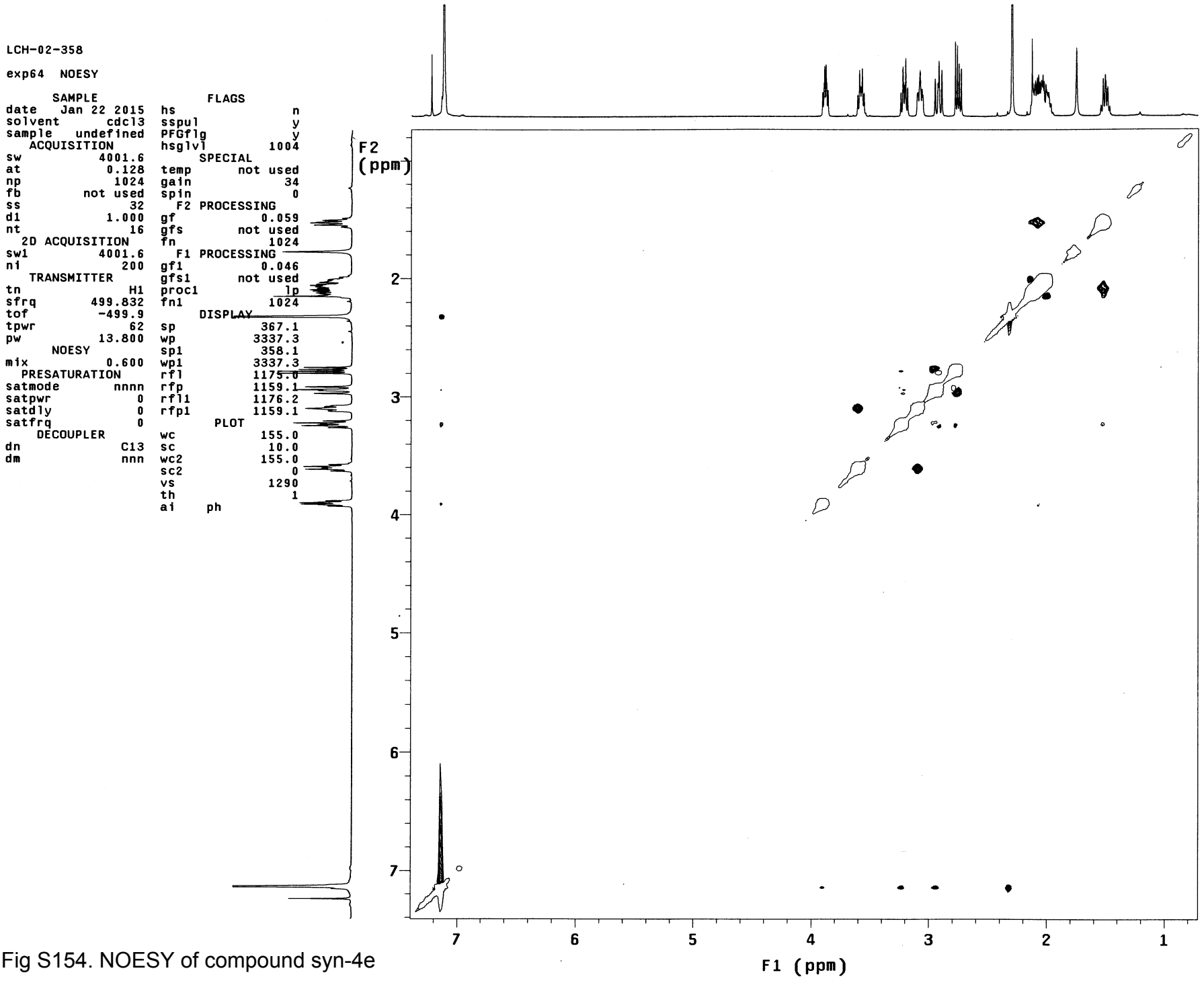
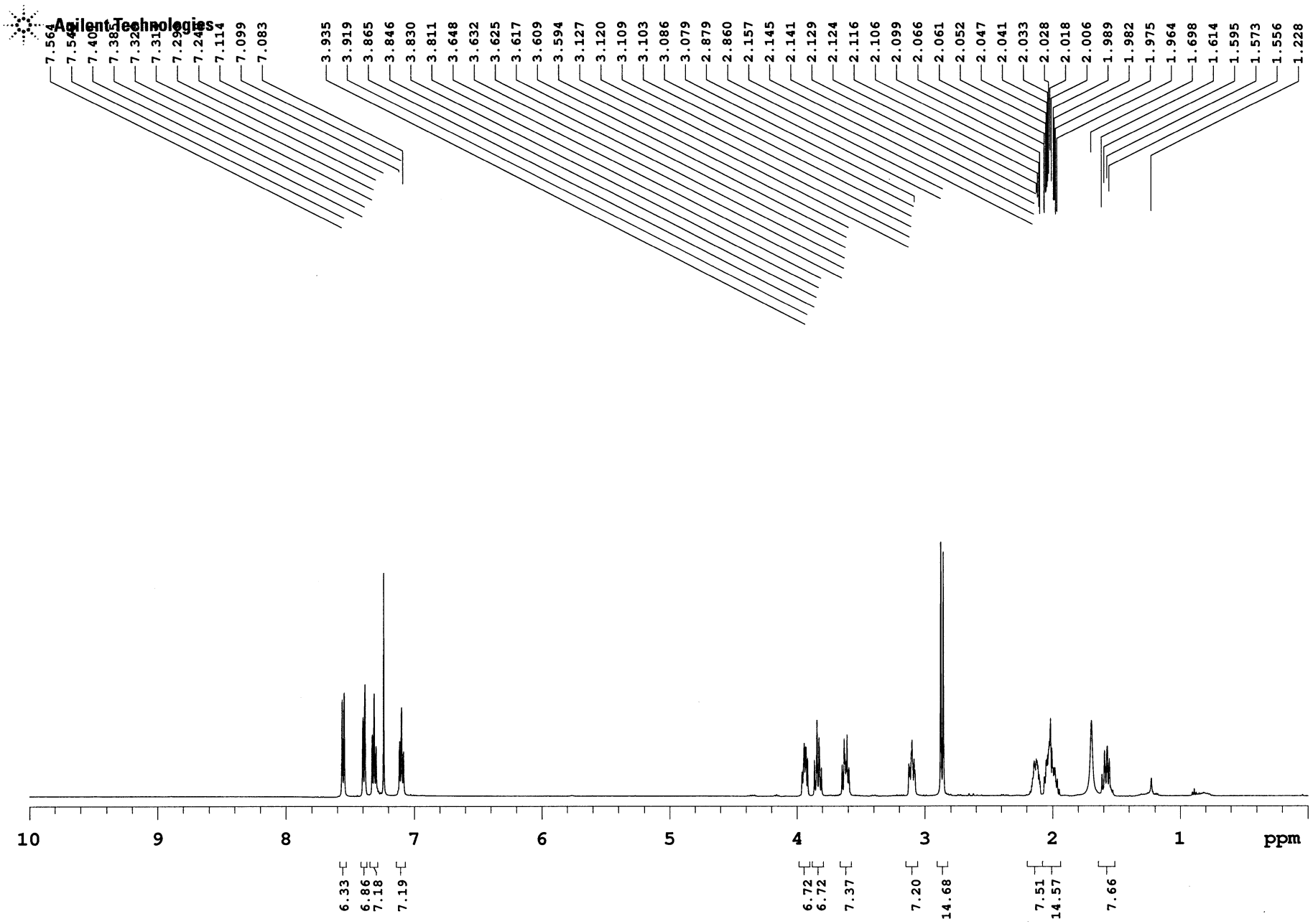


Fig S154. NOESY of compound syn-4e

LCH-02-405

Sample Name	**LCH-02-405**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2015-05-13**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-405/PROTON_05.fid

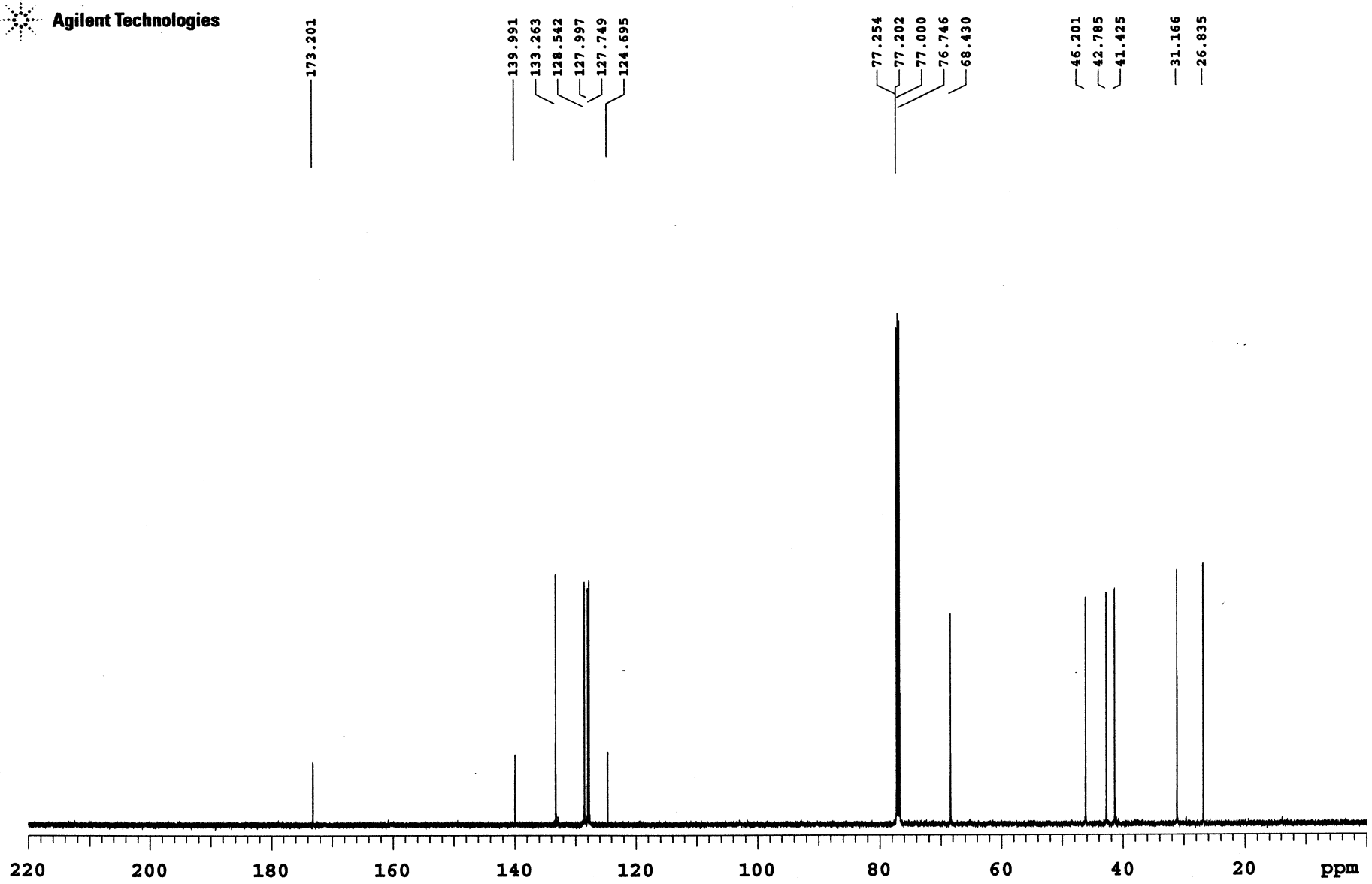
Fig S155. 1H NMR (CDCl3, 500 MHz) of compound syn-4f

Plot date 2015-10-23

LCH-02-405

Sample Name	**LCH-02-405**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2015-05-13**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

Agilent Technologies



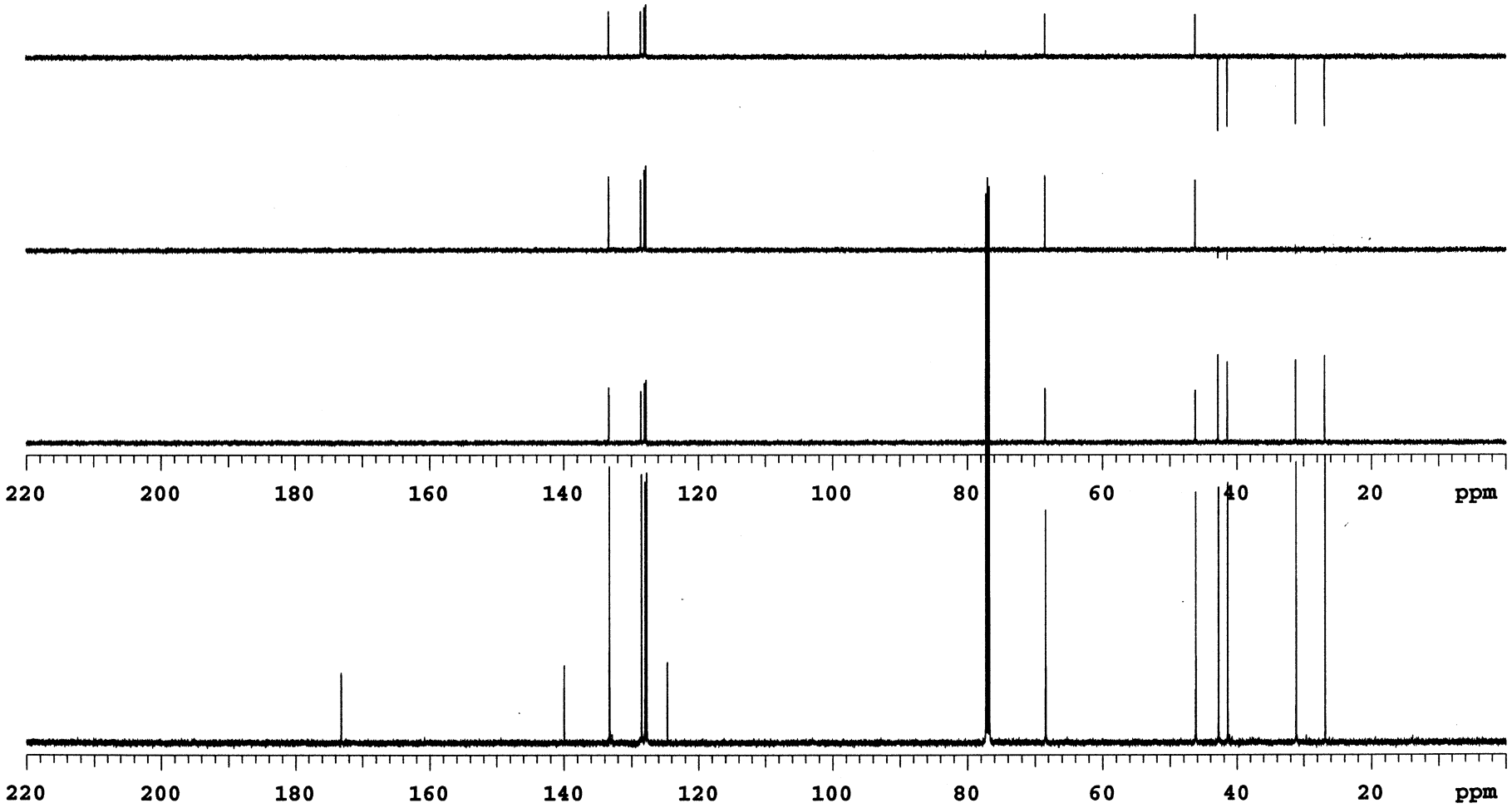
Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-405/CARBON_01

Fig S156. 13C NMR (CDCl3, 125 MHz) of compound syn-4f

Plot date 2015-05-19

Sample Name **LCH-02-405**	Pulse sequence **DEPT**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-05-13**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-405/DEPT_01.fid

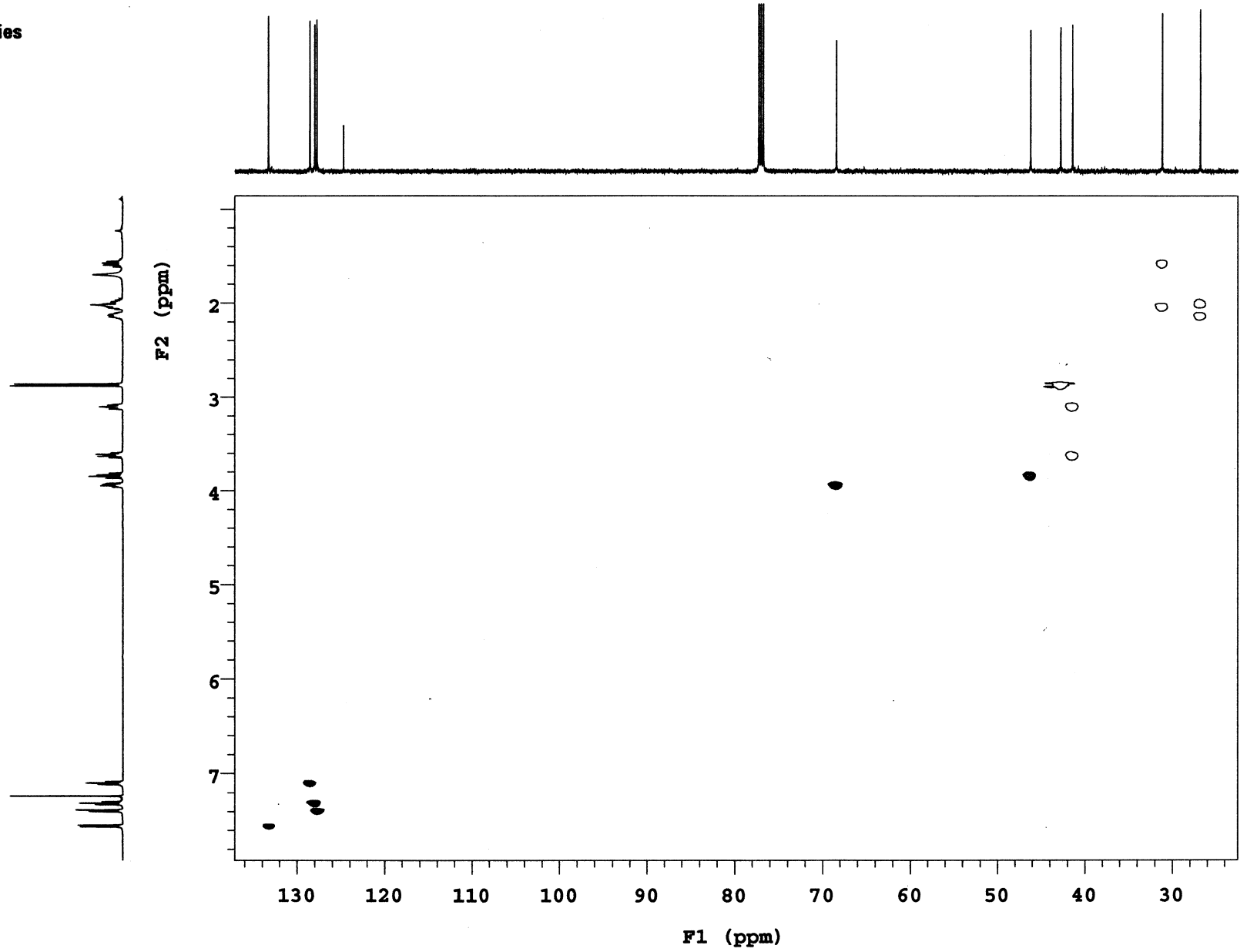
Fig S157. DEPT of compound syn-4f

Plot date 2015-05-19

Agilent Technologies

LCH-02-405

Sample Name **LCH-02-405**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-05-13**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-405/gHSQC_01

Fig S158. HSQC of compound syn-4f

Plot date 2015-05-19

Agilent Technologies

LCH-02-405

Sample Name	LCH-02-405	Pulse sequence	gCOSY	Temperature	25	Study owner	vnmr2
Date collected	2015-05-13	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

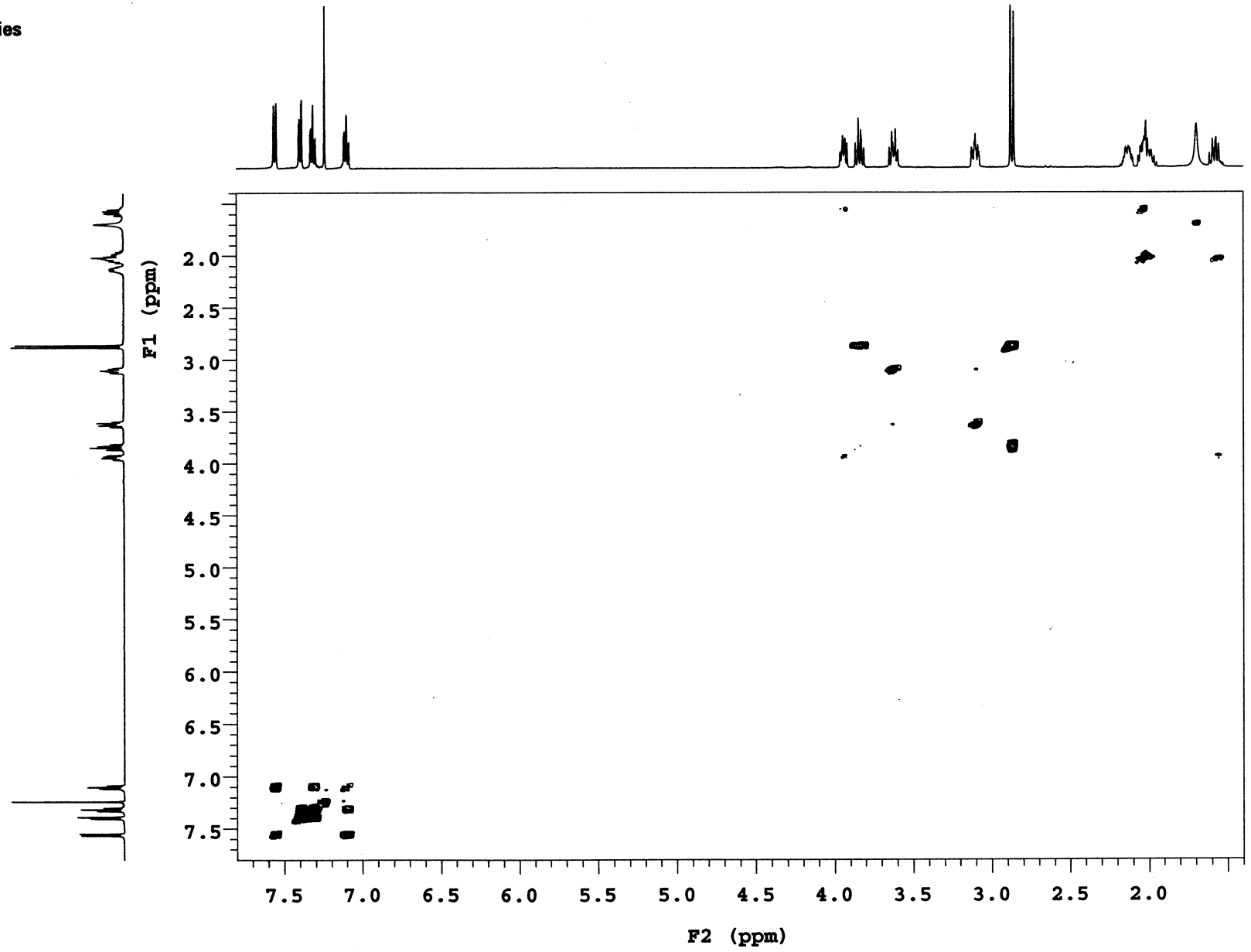


Fig S159. COSY of compound syn-4f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-405/gCOSY_01.fid

Plot date 2015-05-19

LCH-02-405

Sample Name	LCH-02-405	Pulse sequence	NOESY	Temperature	25	Study owner	vnmr2
Date collected	2015-05-13	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

Agilent Technologies

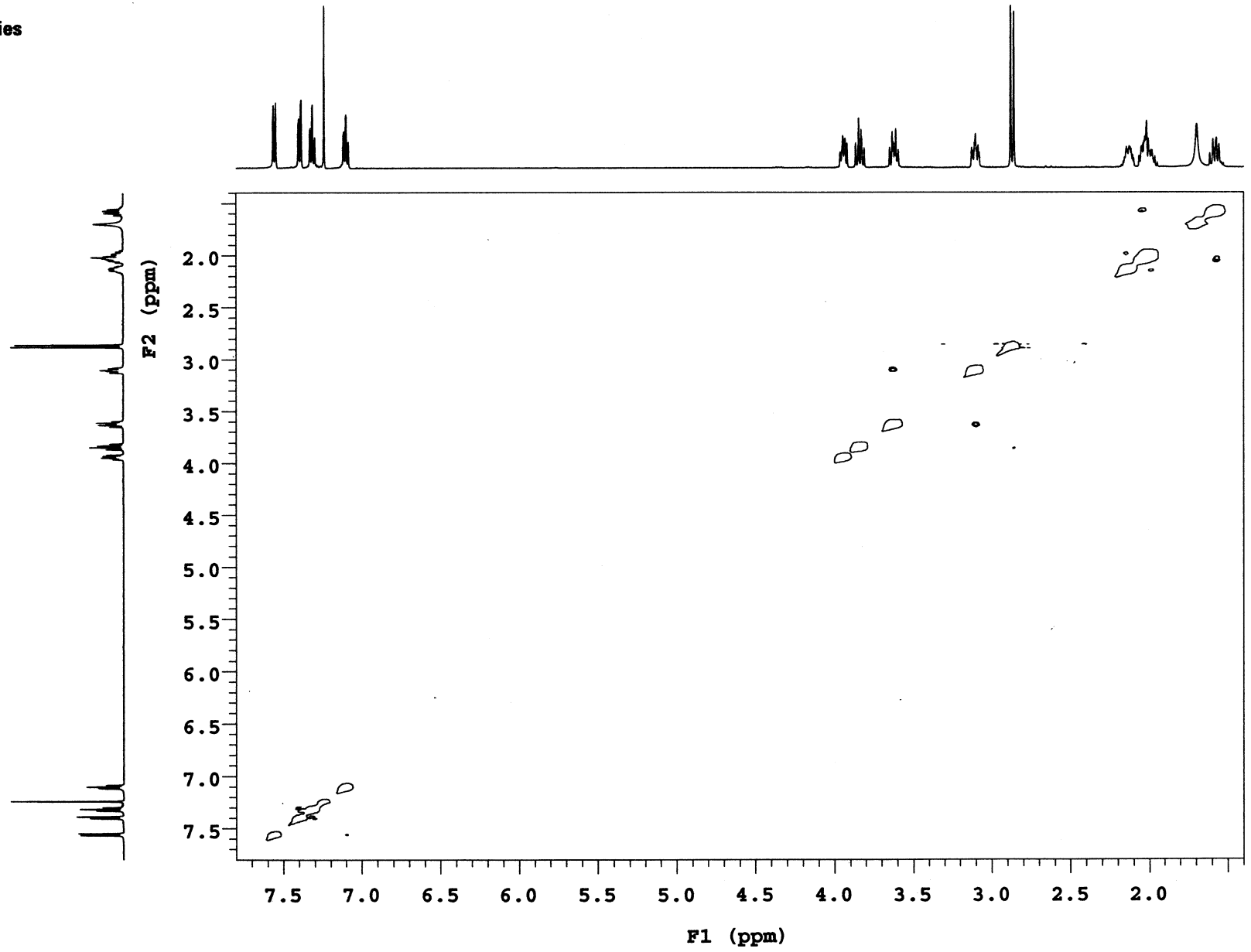


Fig S160. NOESY of compound syn-4f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-405/NOESY_01

Plot date 2015-05-19

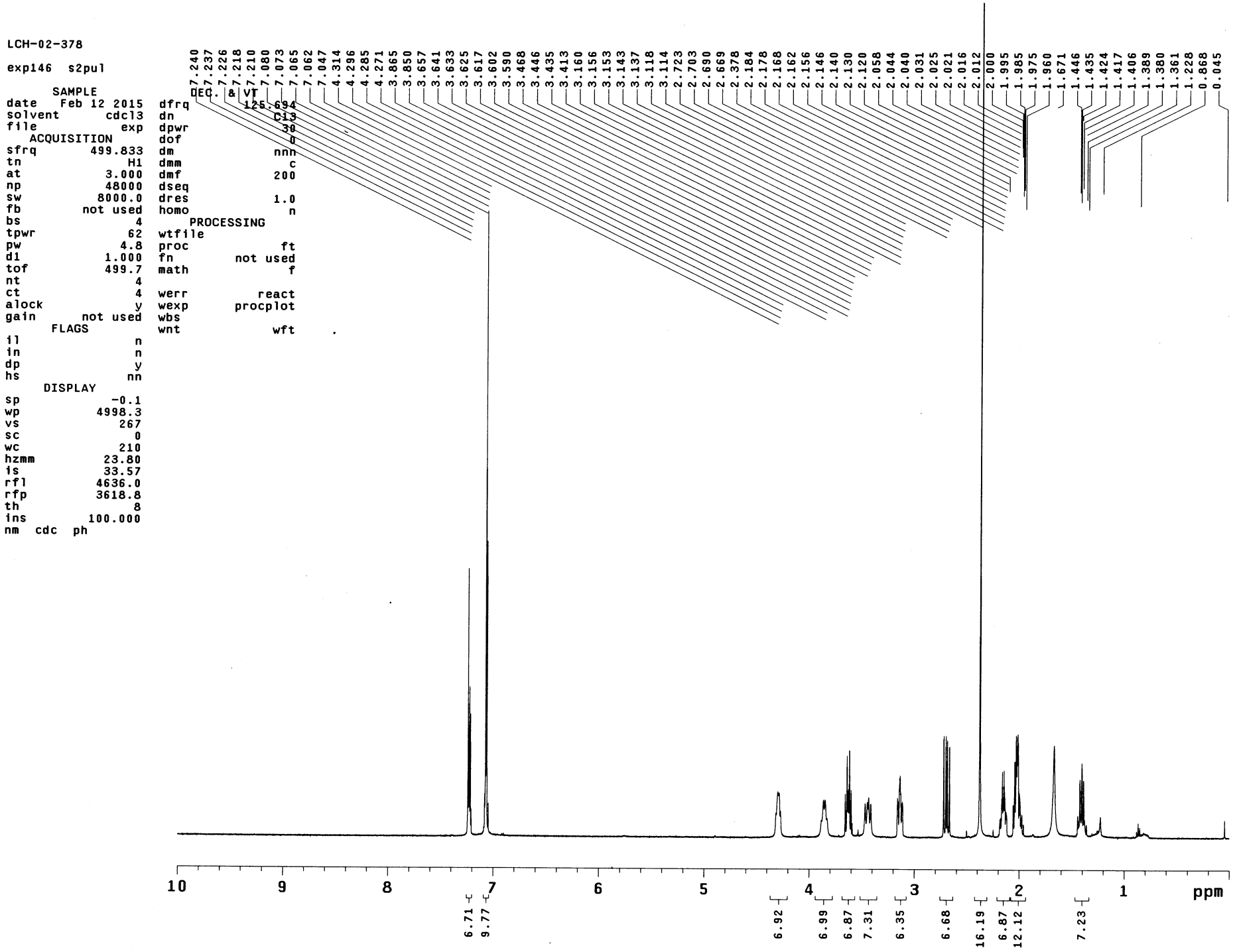


Fig S161. 1H NMR (CDCl3, 500 MHz) of compound syn-4g

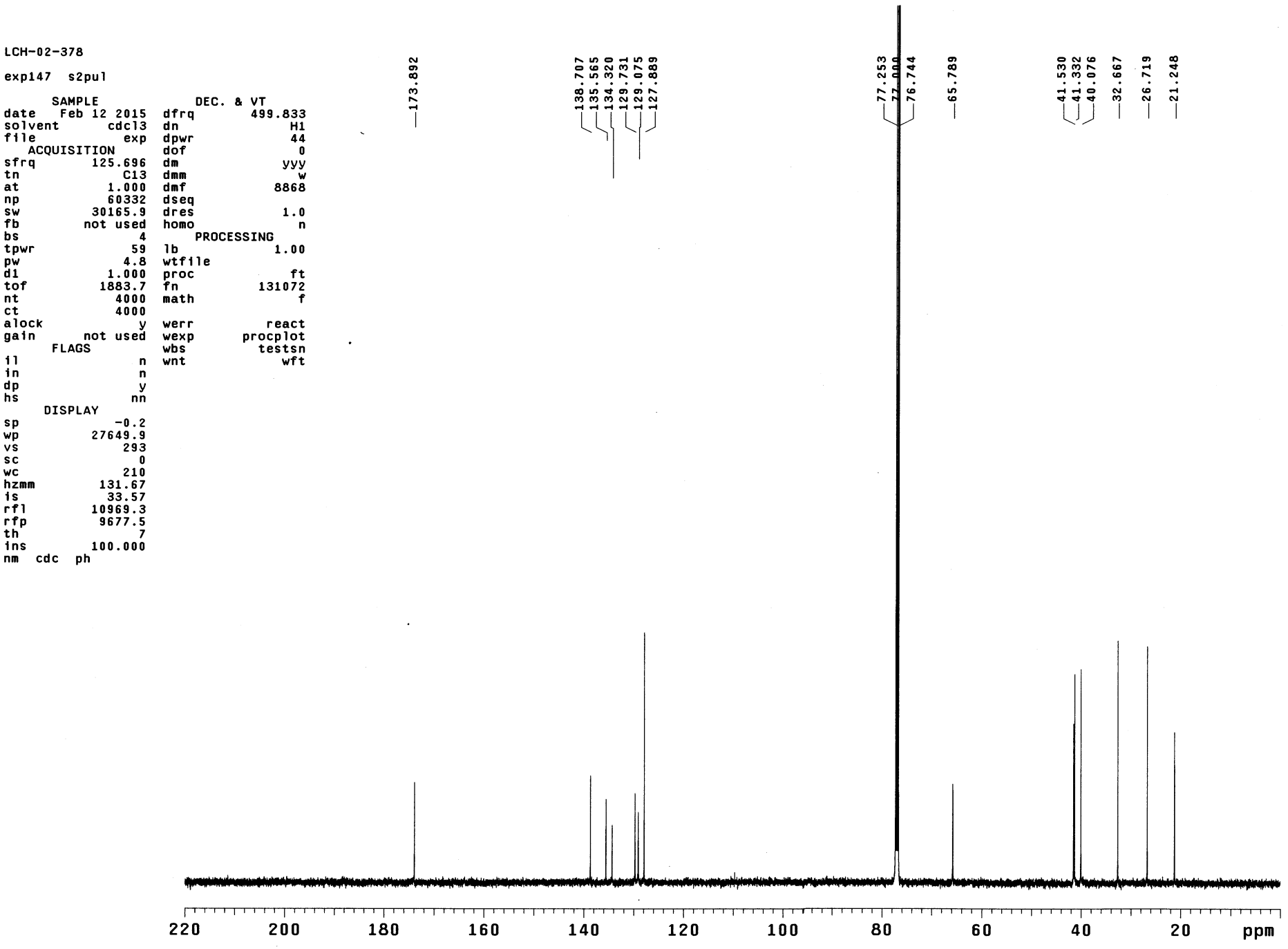


Fig S162. 13C NMR (CDCl3, 125 MHz) of compound syn-4g

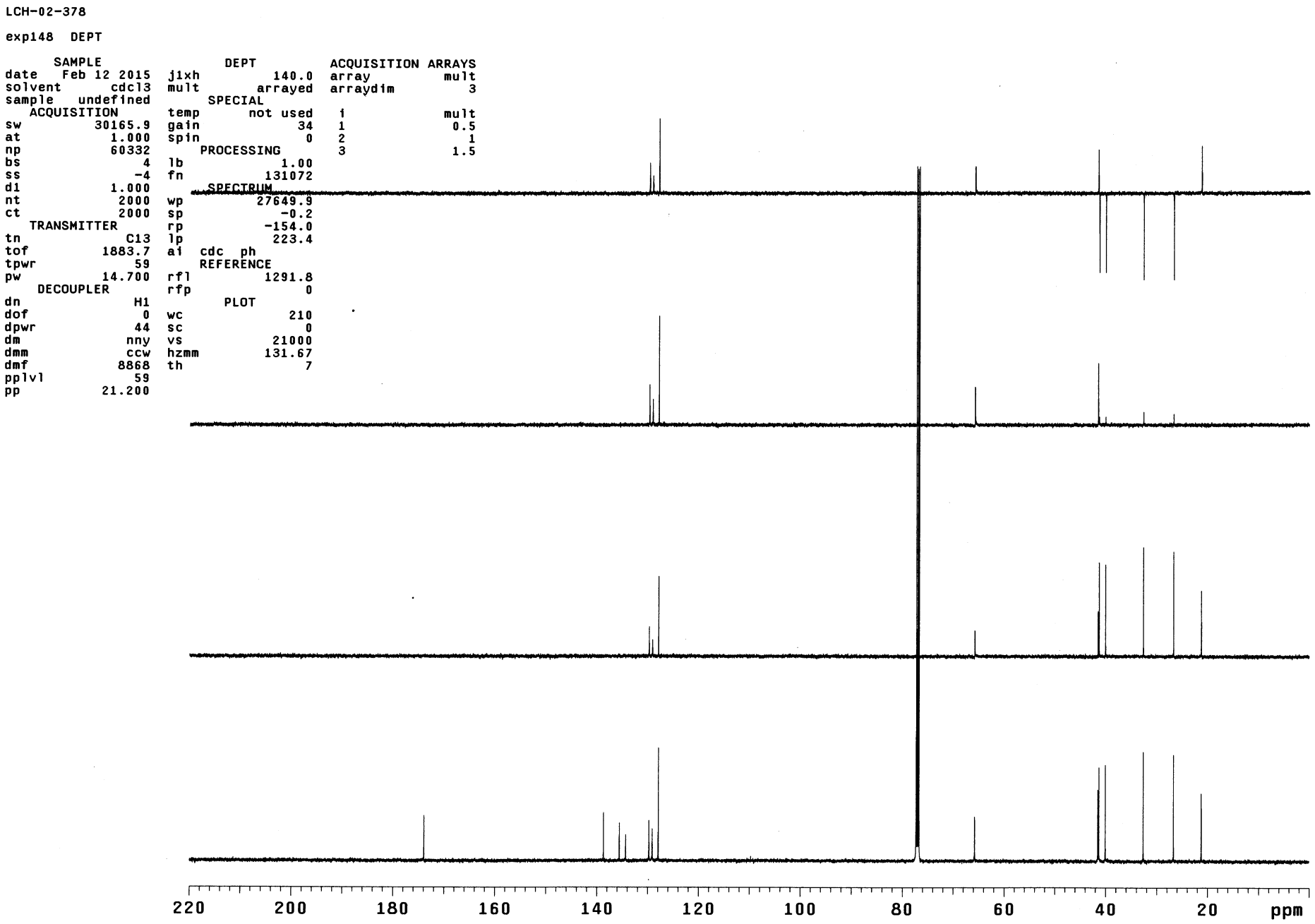
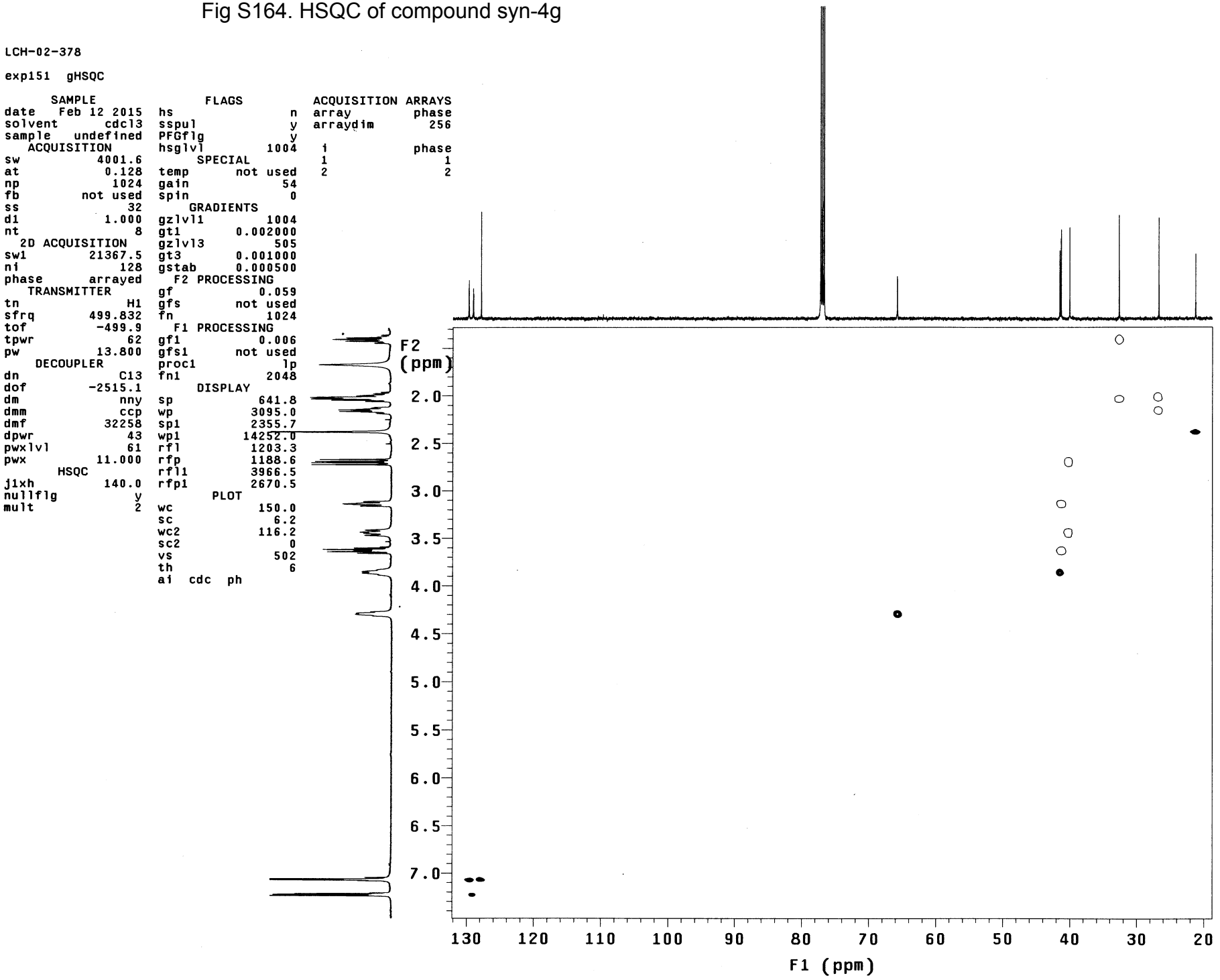


Fig S163. DEPT of compound syn-4g

Fig S164. HSQC of compound syn-4g



LCH-02-378

exp149 gCOSY

SAMPLE		FLAGS	
date	Feb 12 2015	hs	nn
solvent	cdcl3	sspul	n
sample	undefined	hsglvl	1004
ACQUISITION		**SPECIAL**	
sw	4001.6	temp	not used
at	0.128	gain	34
np	1024	spin	0
fb	not used	**F2 PROCESSING**	
ss	16	sb	-0.064
d1	1.000	sbs	not used
nt	8	fn	1024
2D ACQUISITION		**F1 PROCESSING**	
sw1	4001.6	sb1	-0.032
ni	128	sbs1	not used
TRANSMITTER		proc1	lp
tn	H1	fn1	1024
sfrq	499.832	**DISPLAY**	
tof	-499.9	sp	322.3
tpwr	62	wp	3431.1
pw	13.800	sp1	328.2
GRADIENTS		wp1	3423.2
gzlvl1	1004	rfl	1202.4
gt1	0.001000	rfp	1188.6
gstab	0.000500	rfl1	1204.3
DECOUPLER		rfp1	1188.6
dn	C13	**PLOT**	
dm	nnn	wc	155.0
		sc	10.0
		wc2	155.0
		sc2	0
		vs	502
		th	6
		ai cdc av	

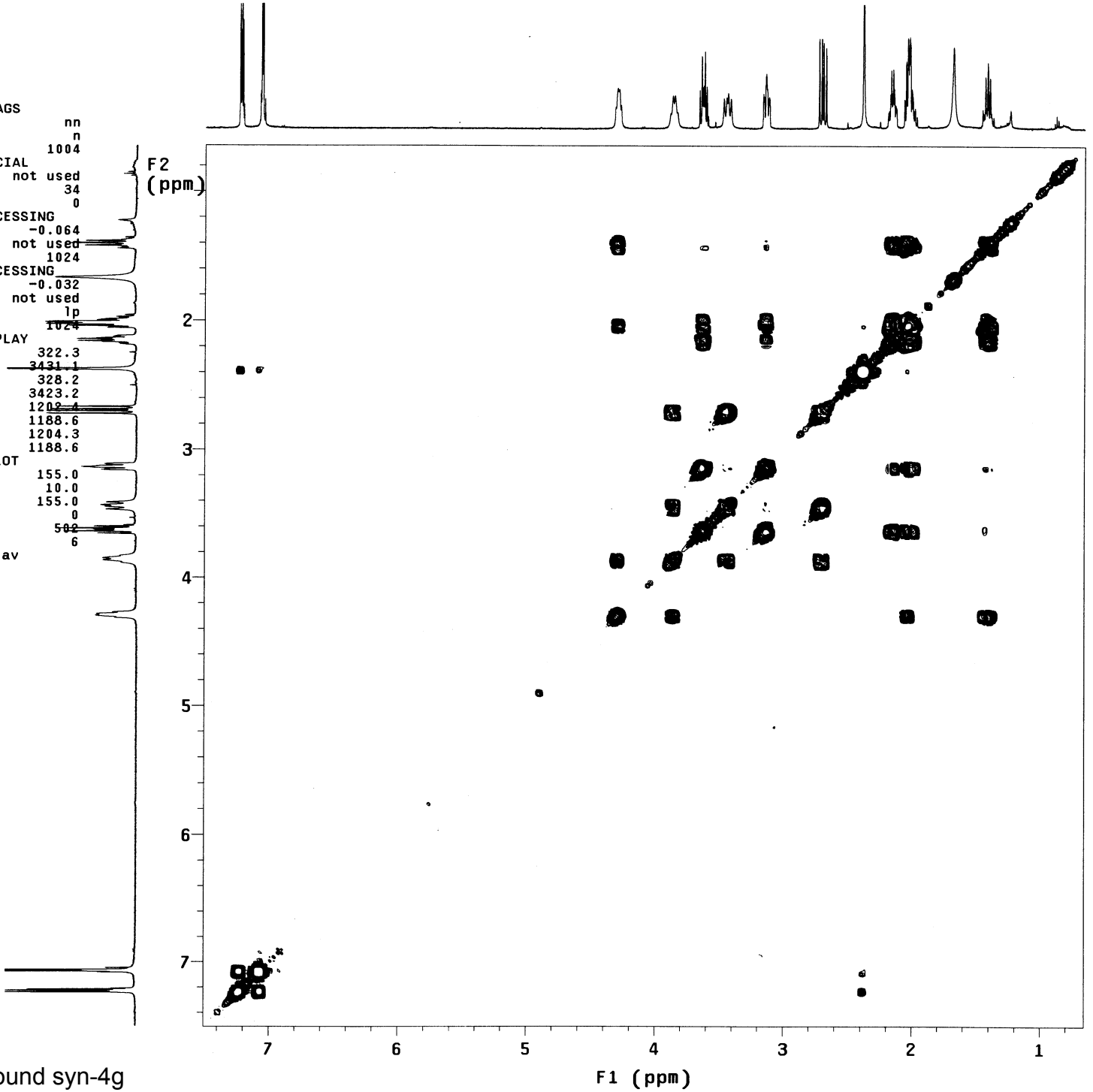


Fig S165. COSY of compound syn-4g

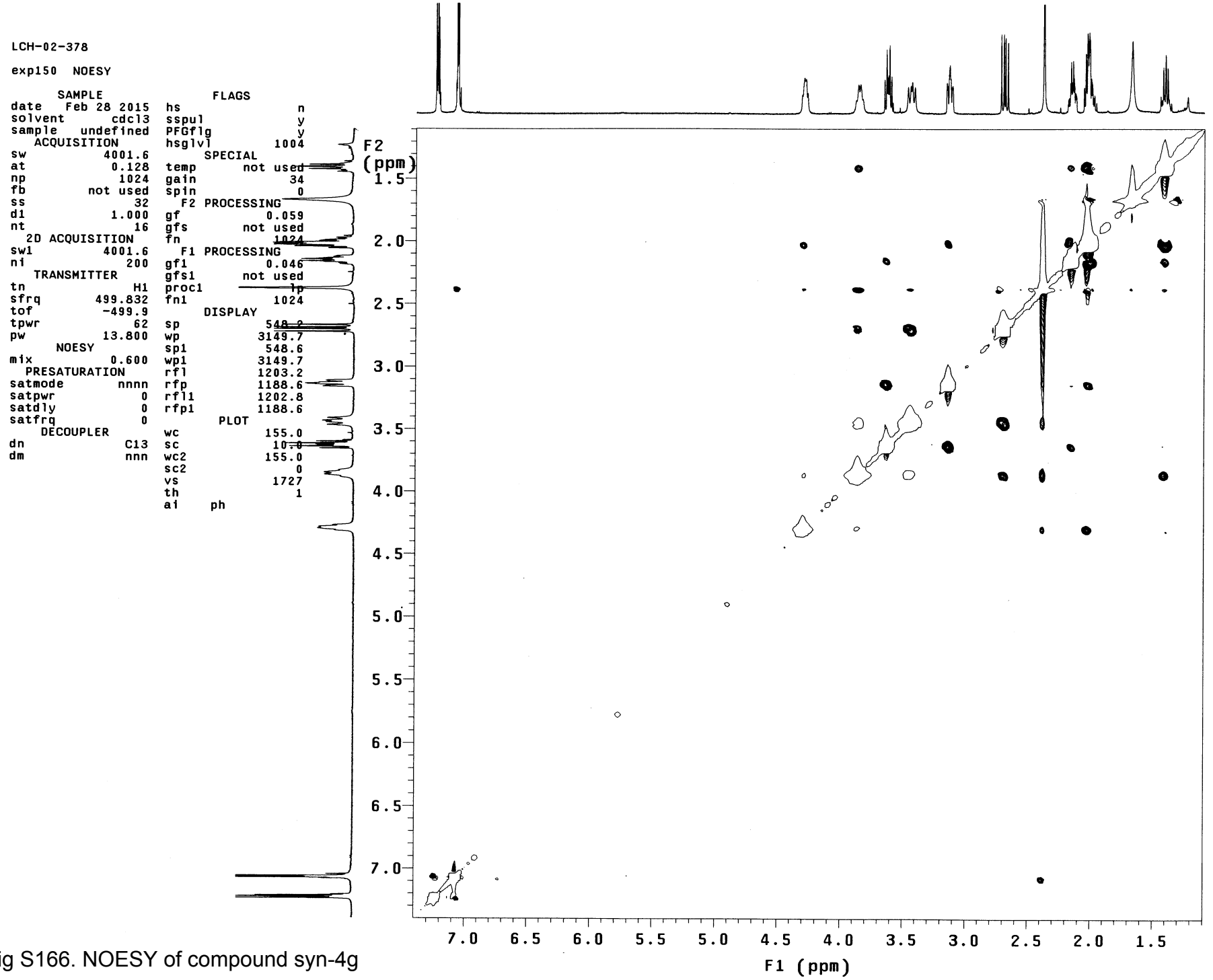
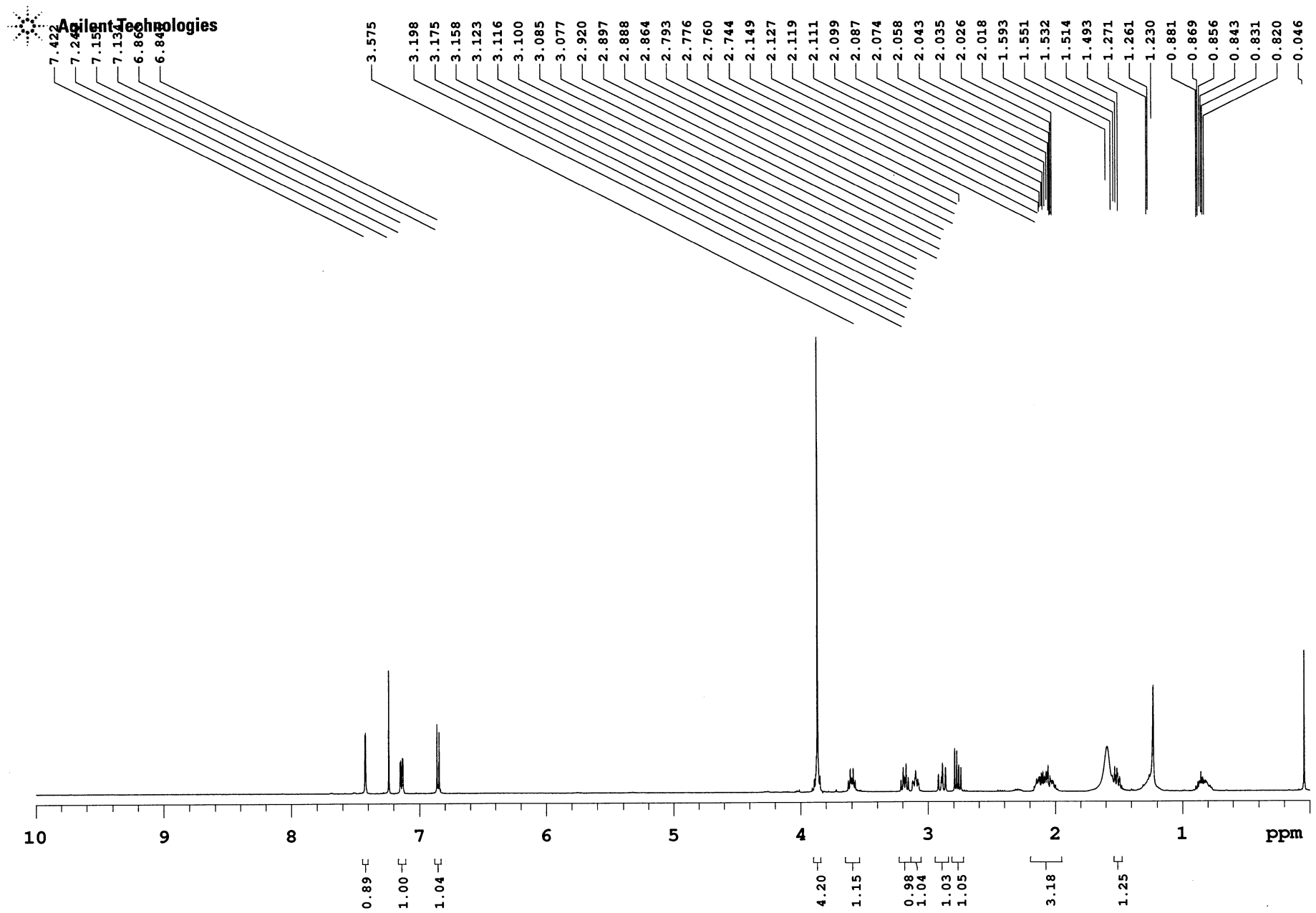


Fig S166. NOESY of compound syn-4g

LCH-02-396

Sample Name	LCH-02-396	Pulse sequence	PROTON	Temperature	25	Study owner	vnmr2
Date collected	2015-06-09	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-396/PROTON_02.fid

Fig S167. 1H NMR (CDCl3, 500 MHz) of compound syn-4h

Plot date 2015-10-24

LCH-02-396

Sample Name **LCH-02-396** | Pulse sequence **s2pul** | Temperature **50** | Study owner **vnmr2**
Date collected **2015-04-12** | Solvent **cdcl3** | Spectrometer **---** | Operator **vnmr2**

Agilent Technologies

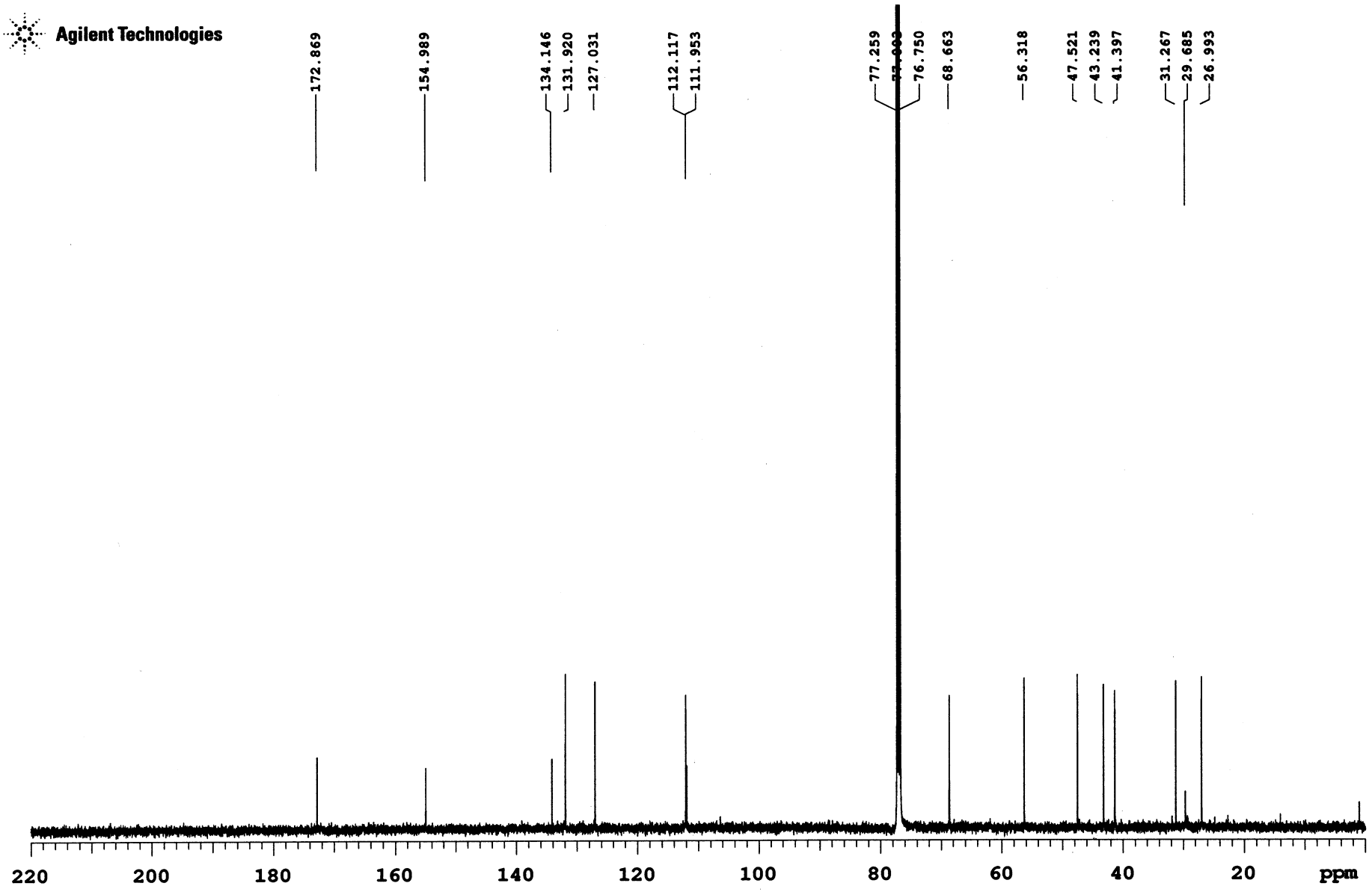


Fig S168. 13C NMR (CDCl3, 125 MHz) of compound syn-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-396/C13.fid

Plot date 2015-05-28

LCH-02-396

Sample Name **LCH-02-396**	Pulse sequence **DEPT**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-12**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

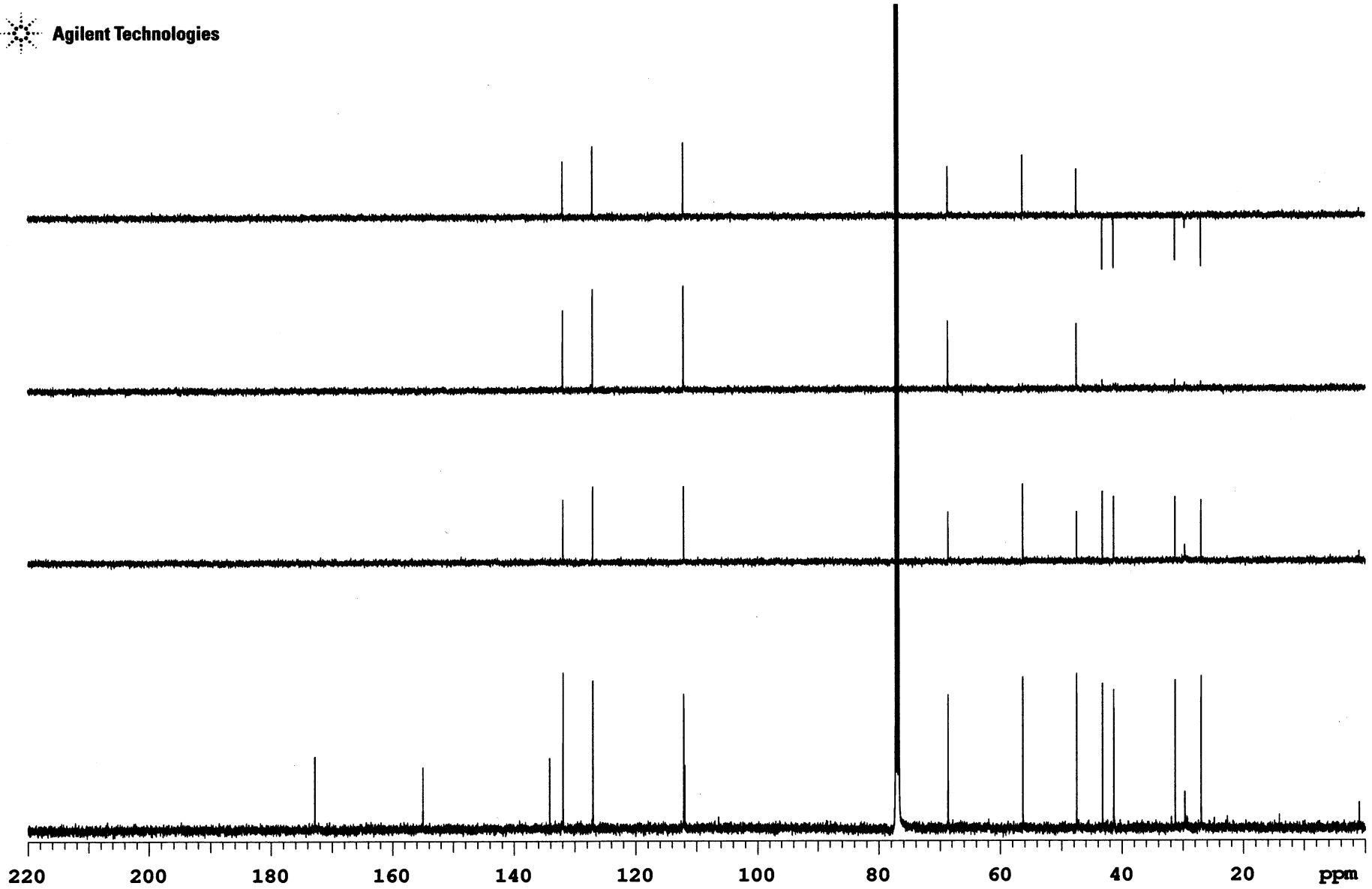


Fig S169. DEPT of compound syn-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-396/DEPT.fid

Plot date 2015-05-28

LCH-02-396

Sample Name	LCH-02-396	Pulse sequence	gHSQC	Temperature	25	Study owner	vnmr2
Date collected	2015-06-09	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

Agilent Technologies

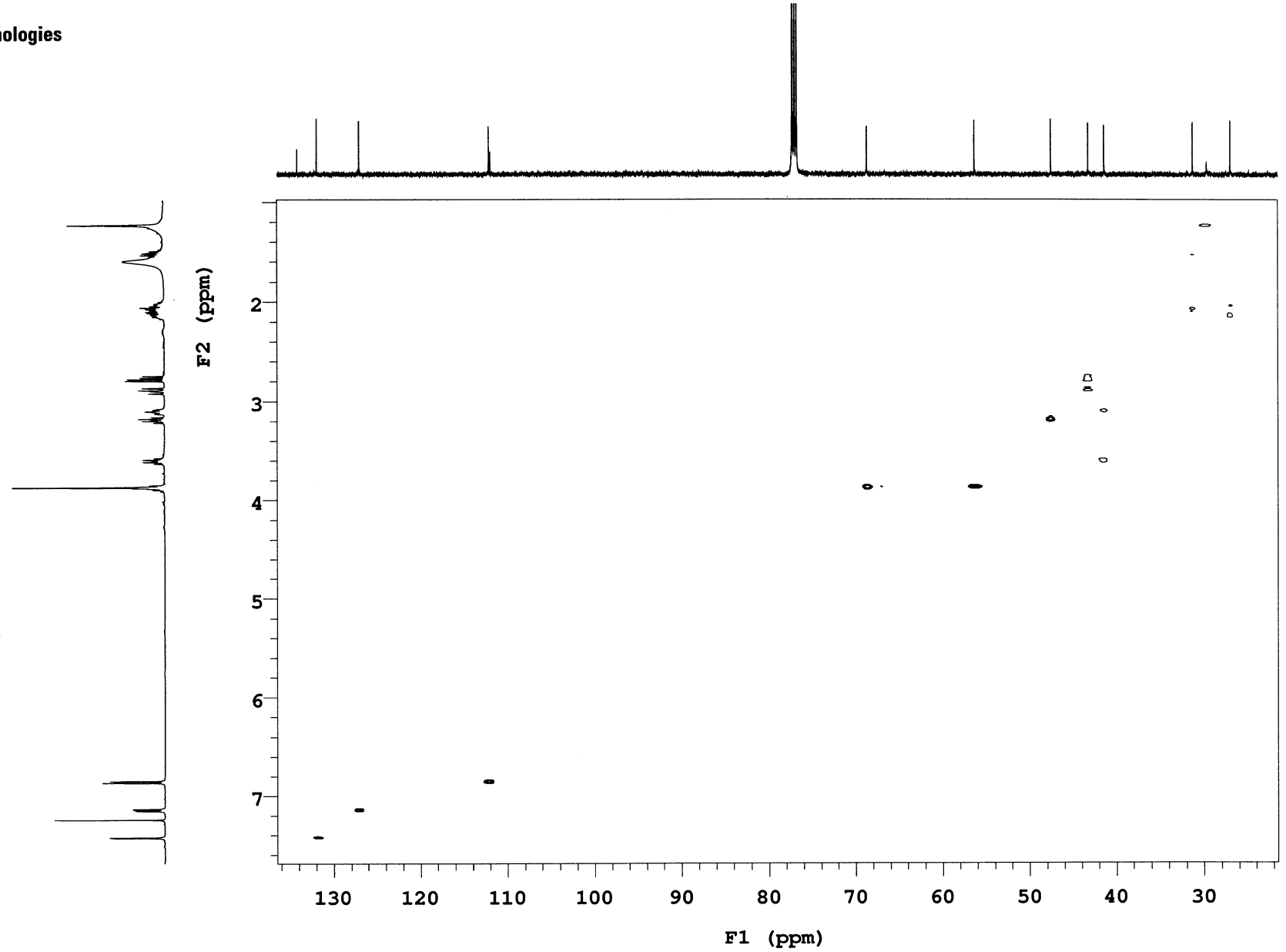


Fig S170. HSQC of compound syn-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-396/gHSQC_01.fid

Plot date 2015-10-29

LCH-02-396

Sample Name	**LCH-02-396**	Pulse sequence	**gCOSY**	Temperature	**50**	Study owner	**vnmr2**
Date collected	**2015-04-13**	Solvent	**cdcl3**	Spectrometer	**---**	Operator	**vnmr2**

Agilent Technologies

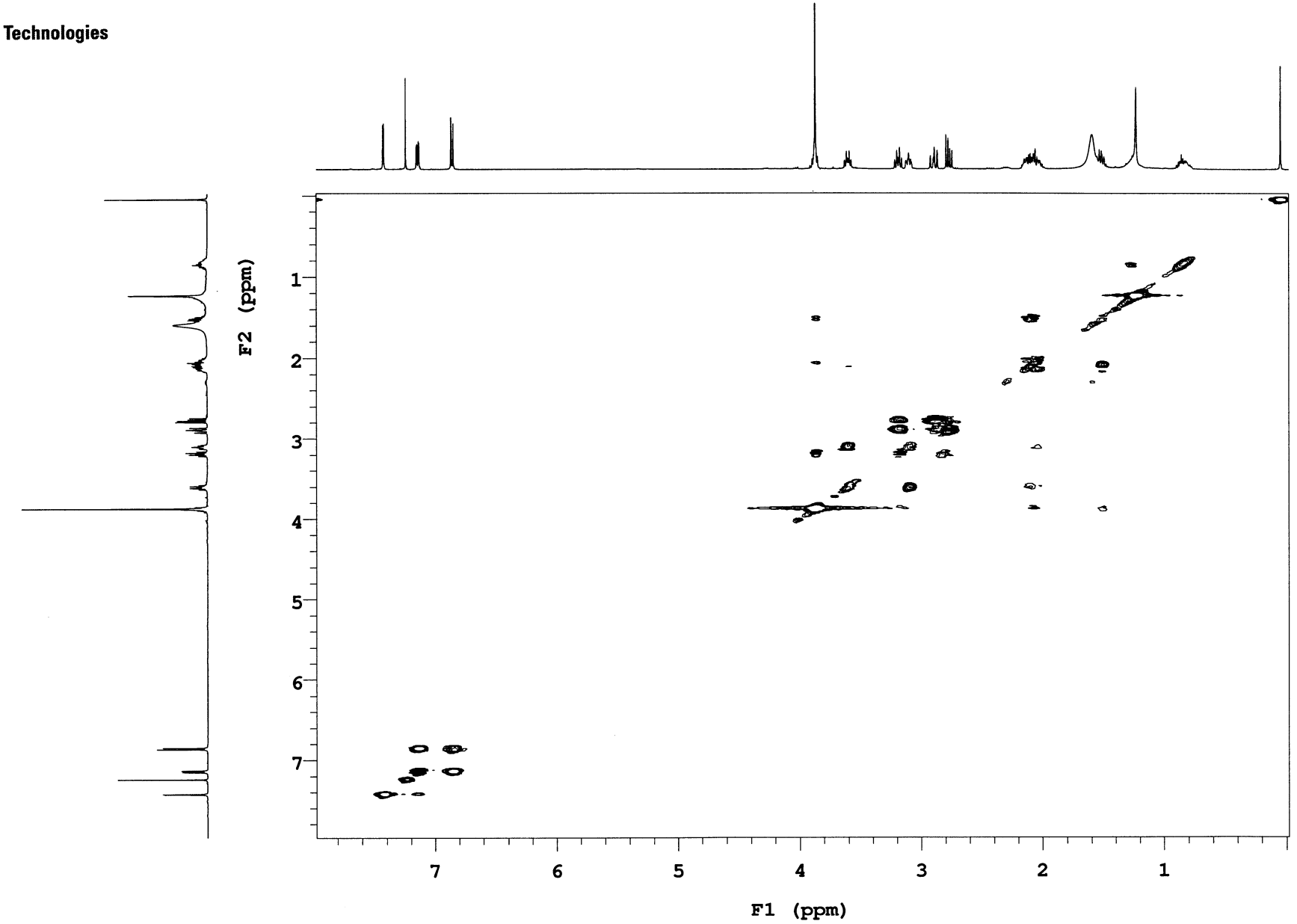


Fig S171. COSY of compound syn-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-396/COSY.fid

Plot date 2015-10-24

LCH-02-396

Sample Name **LCH-02-396**	Pulse sequence **NOESY**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-13**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

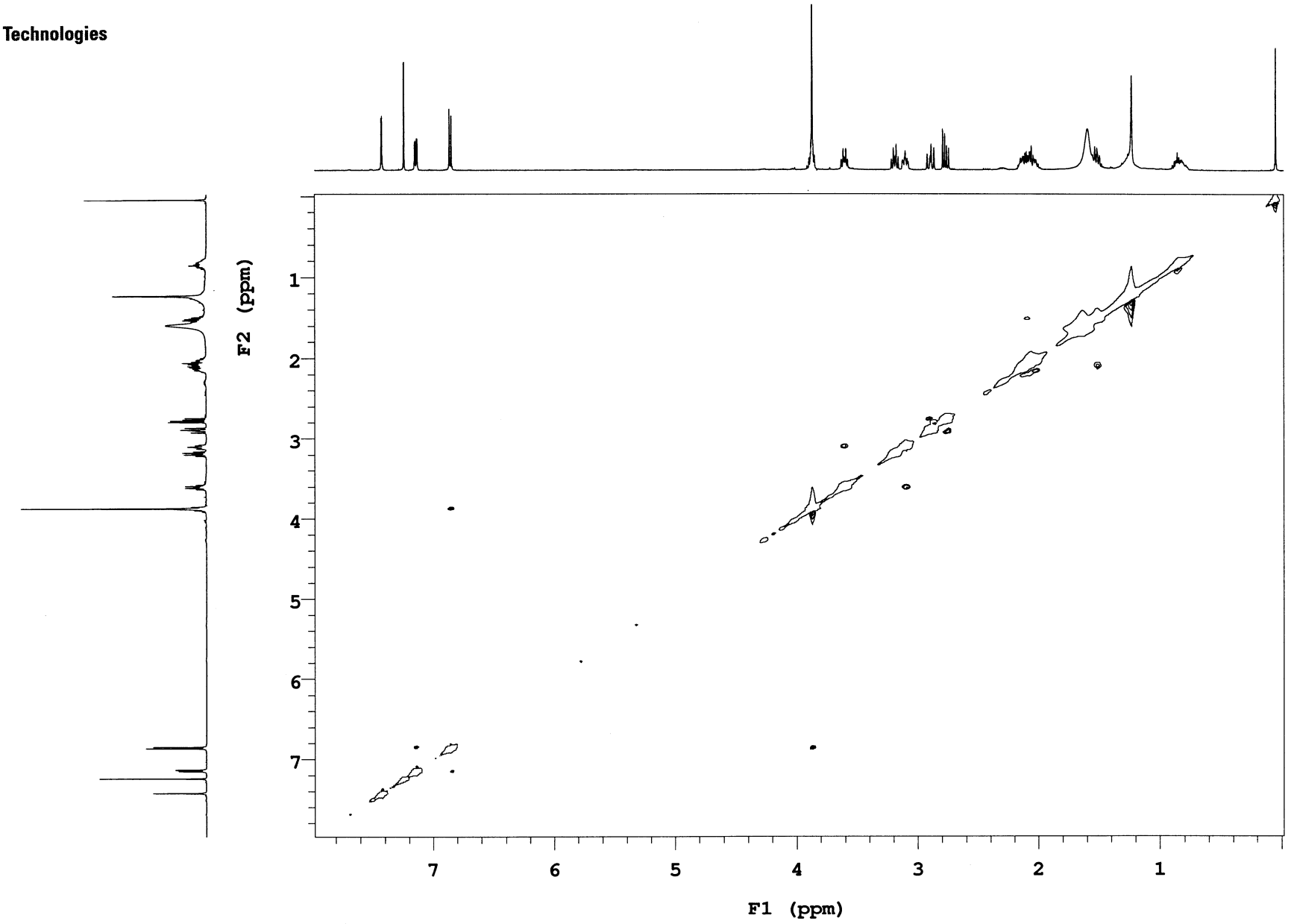
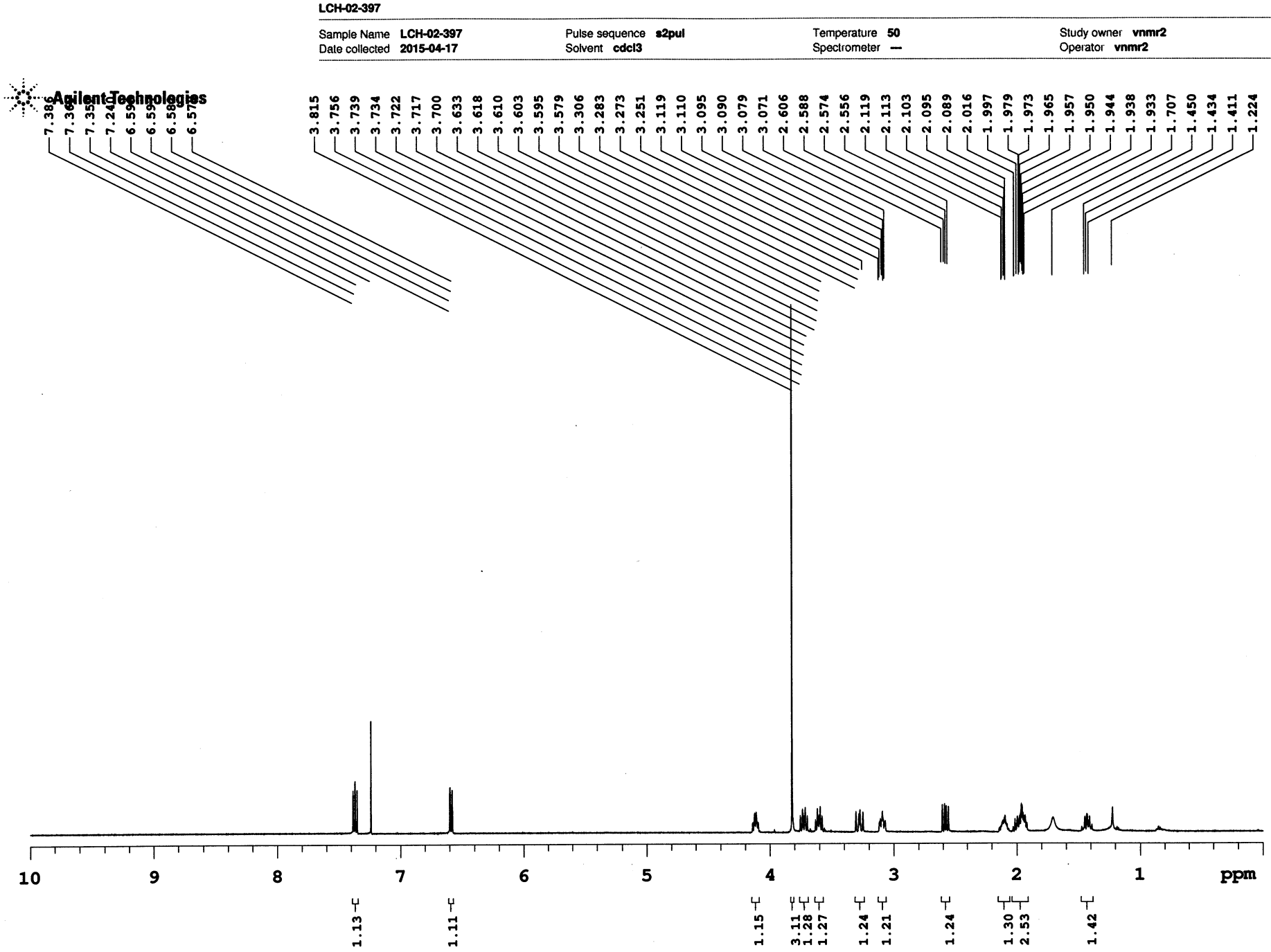


Fig S172. NOESY of compound syn-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-396/NOESY.fid

Plot date 2015-10-29



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-397/H1.fid

Fig S173. 1H NMR (CDCl3, 500 MHz) of compound syn-4i

Plot date 2015-10-20

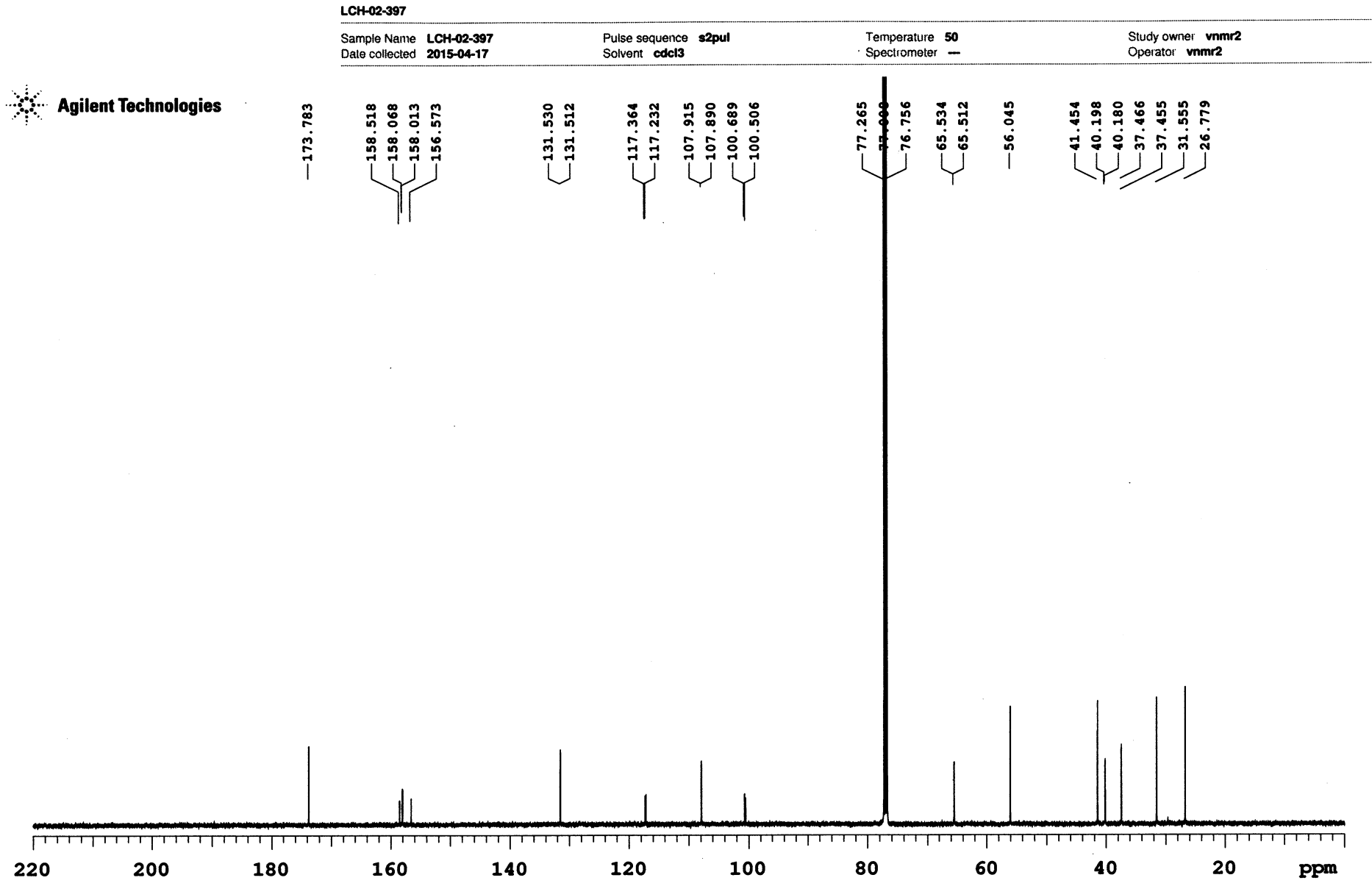


Fig S174. 13C NMR (CDCl3, 125 MHz) of compound syn-4i

LCH-02-397

Sample Name **LCH-02-397**	Pulse sequence **DEPT**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-17**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

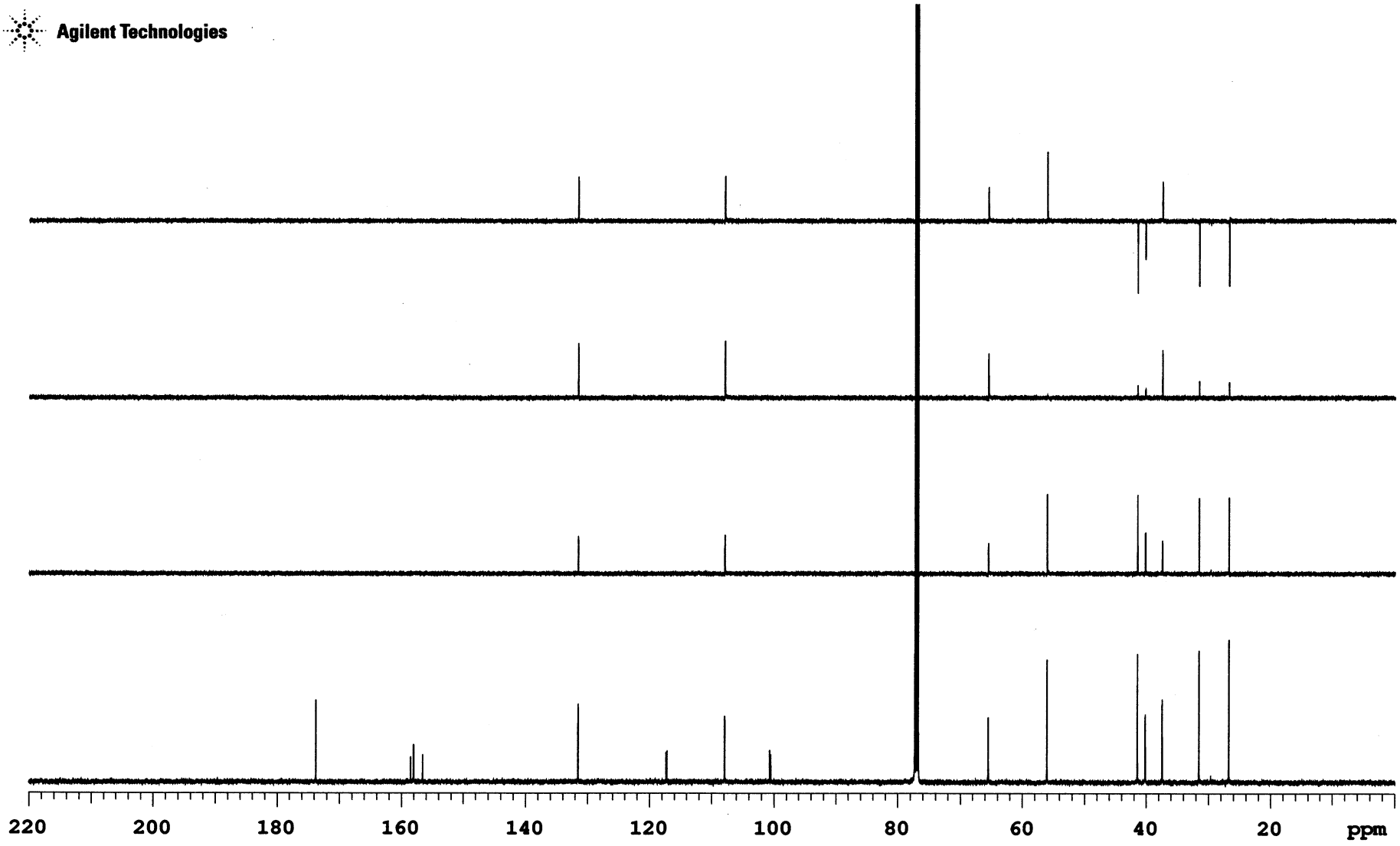


Fig S175. DEPT of compound syn-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-397/DEPT.fid

Plot date 2015-06-04

LCH-02-397

Sample Name **LCH-02-397**	Pulse sequence **gHSQC**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-17**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

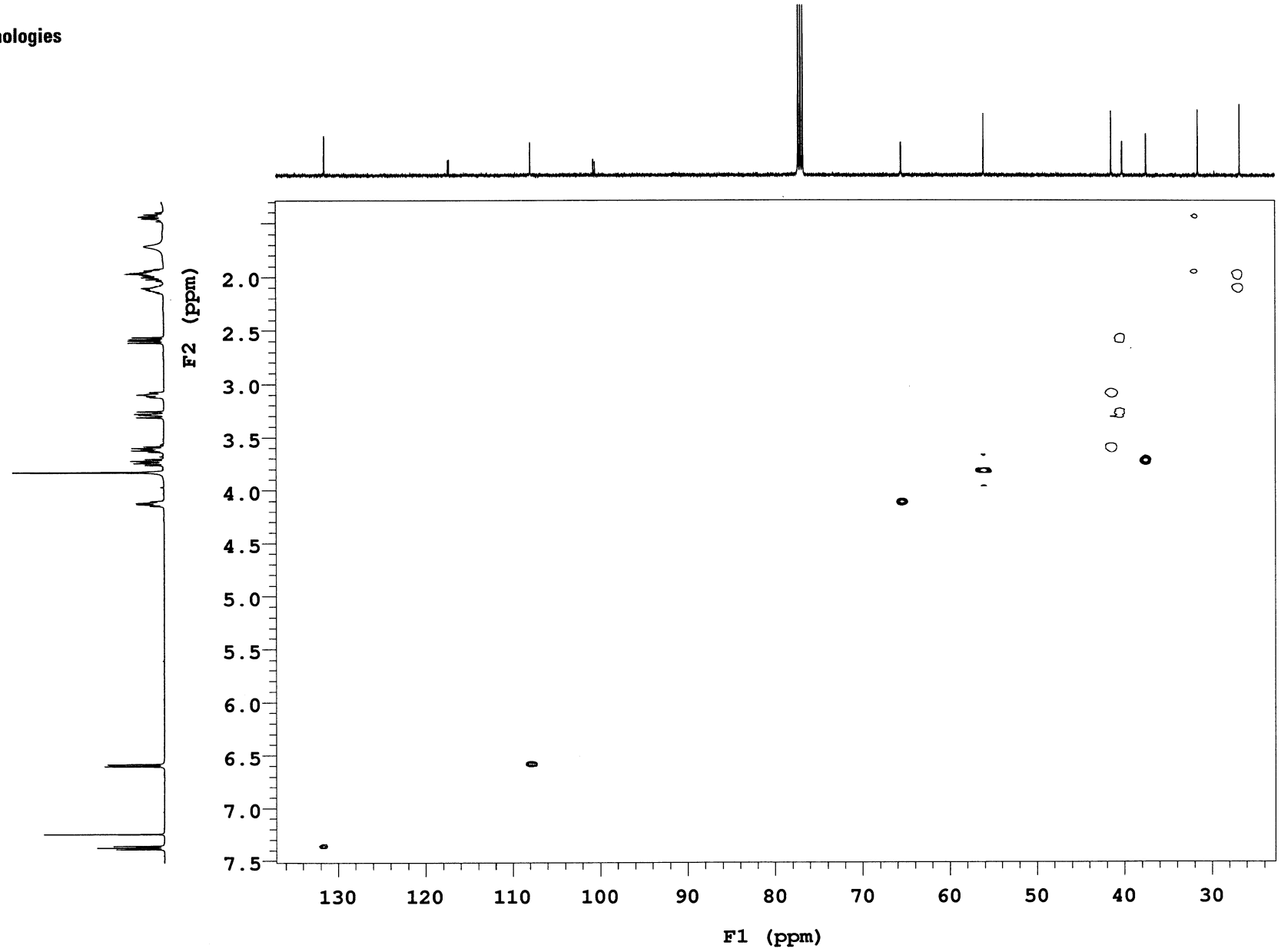


Fig S176. HSQC of compound syn-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-397/HSQC.fid

Plot date 2015-10-29

LCH-02-397

Sample Name **LCH-02-397**	Pulse sequence **gCOSY**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-17**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

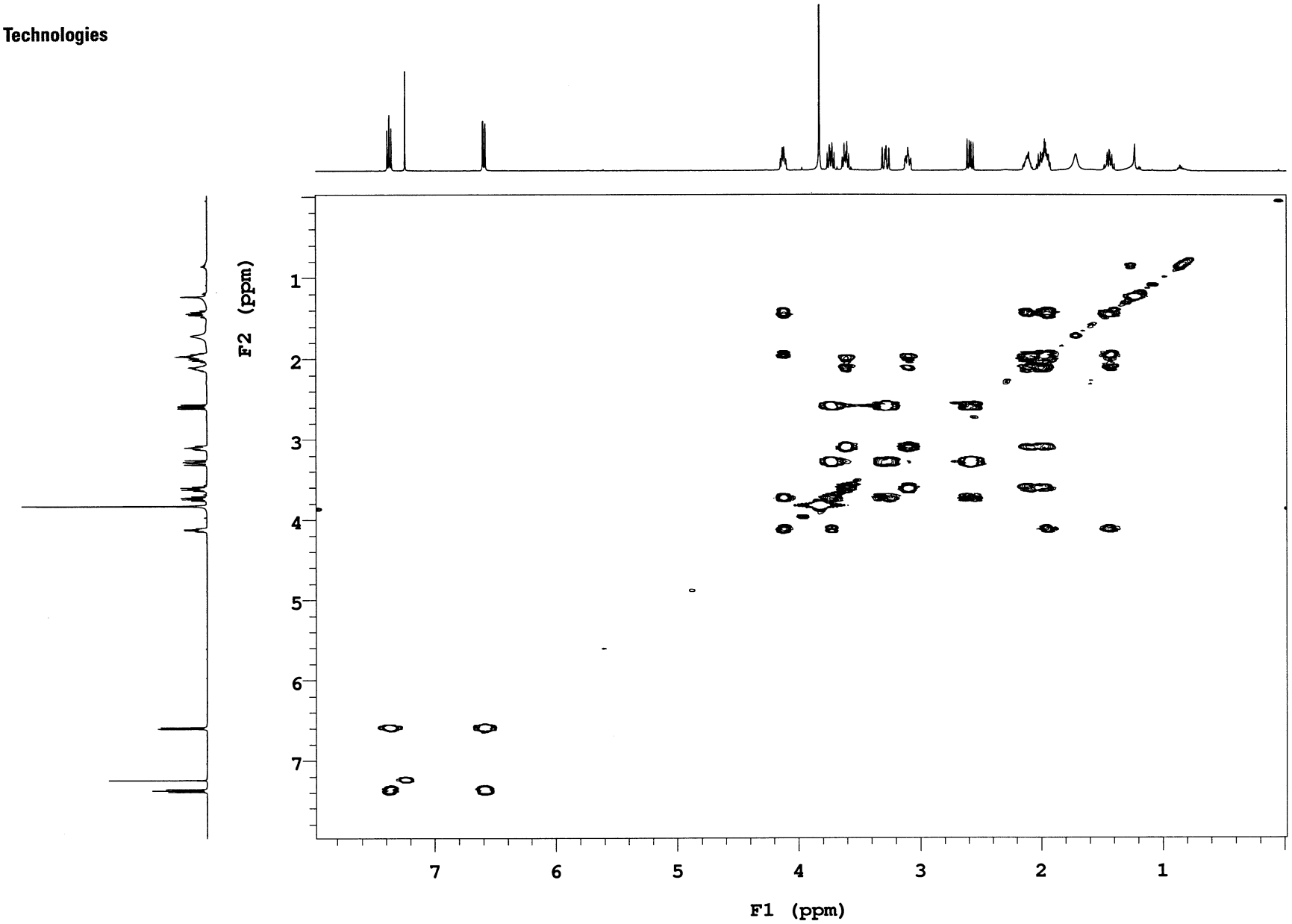


Fig S177. COSY of compound syn-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-397/COSY.fid

Plot date 2015-10-24

LCH-02-397

Sample Name	**LCH-02-397**	Pulse sequence	**NOESY**	Temperature	**50**	Study owner	**vnmr2**
Date collected	**2015-04-17**	Solvent	**cdcl3**	Spectrometer	**---**	Operator	**vnmr2**

Agilent Technologies

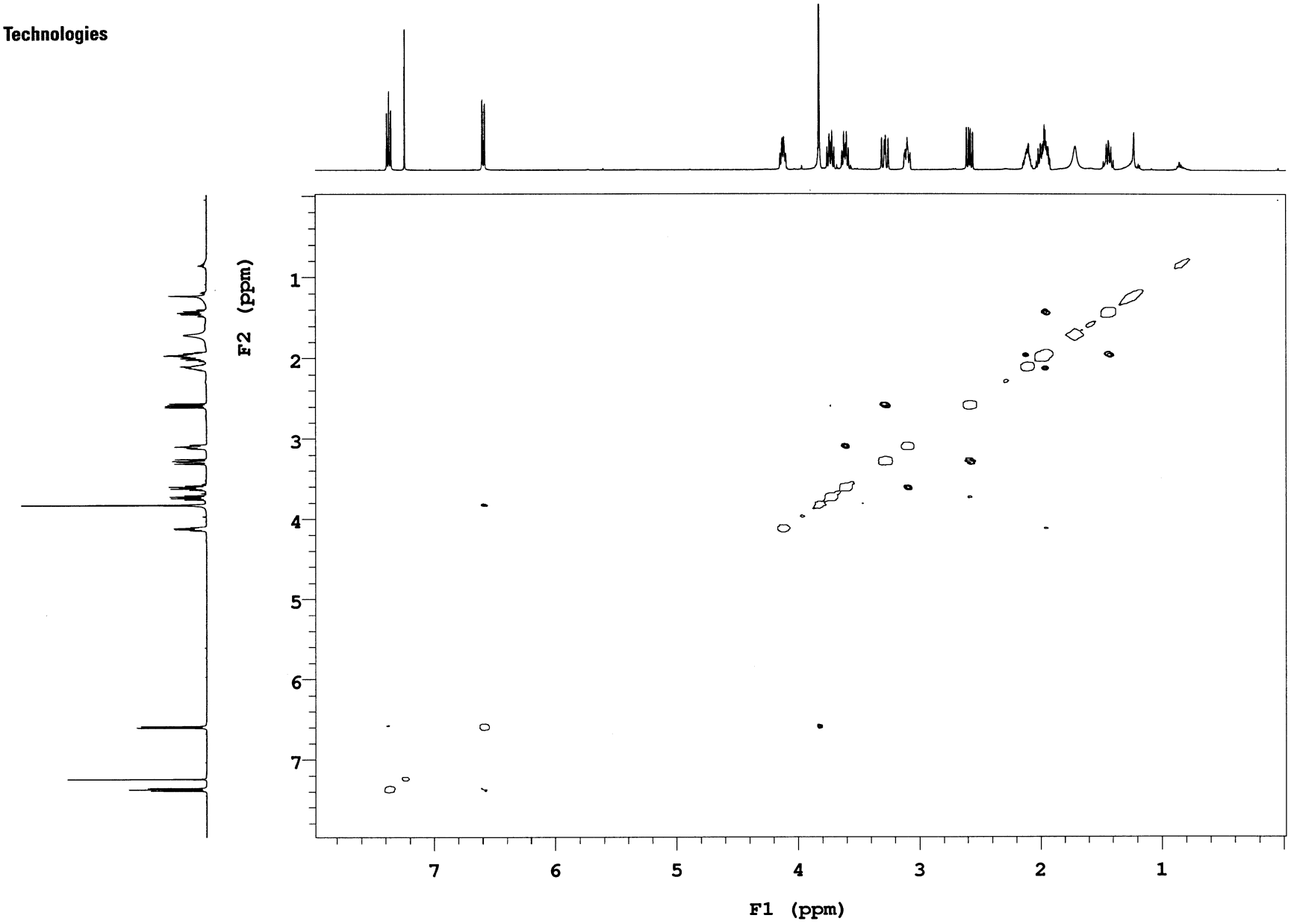


Fig S178. NOESY of compound syn-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-397/NOESY.fid

Plot date 2015-10-29

Fig S179. 1H NMR (CDCl3, 500 MHz) of compound anti-4a

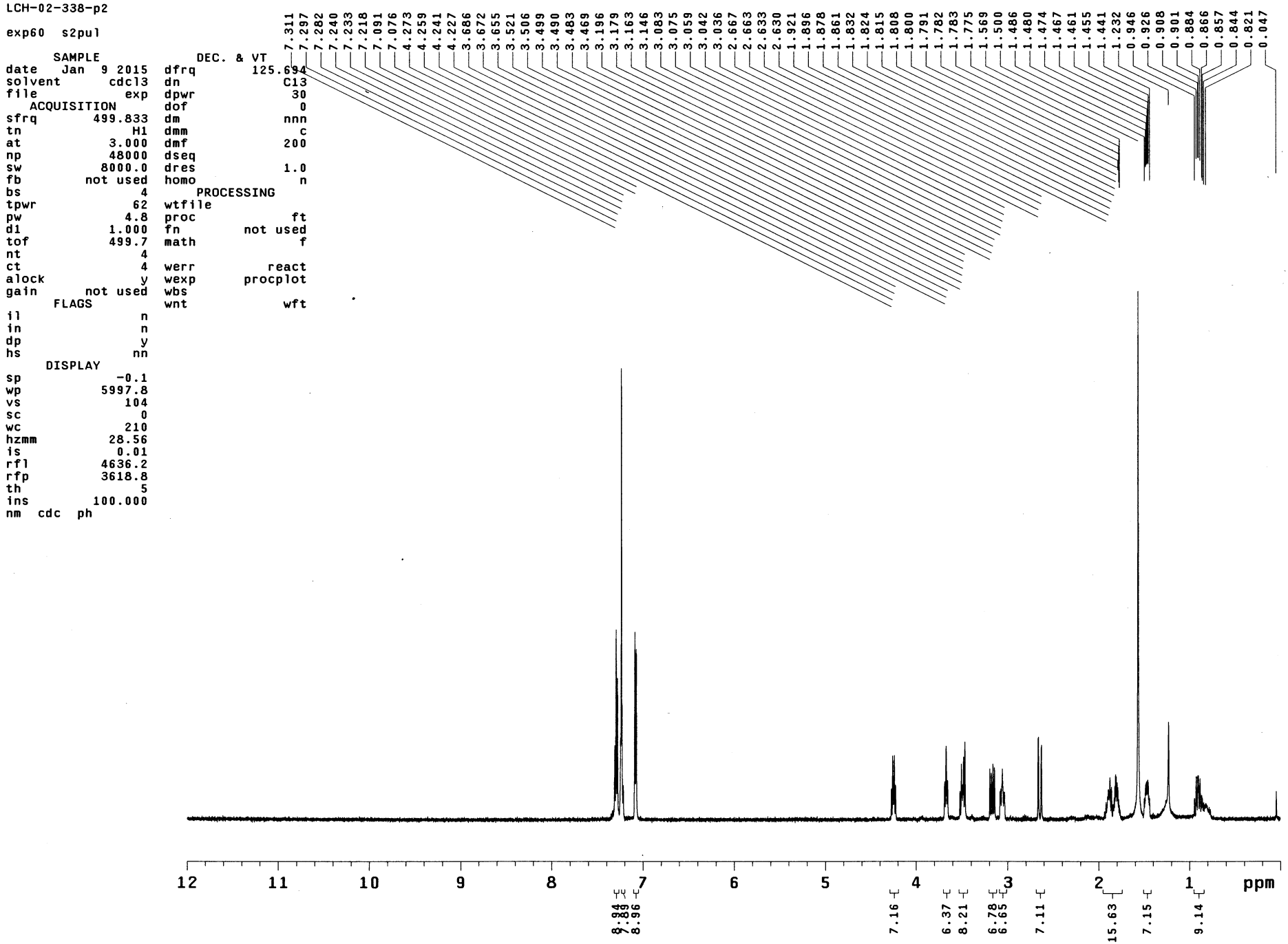
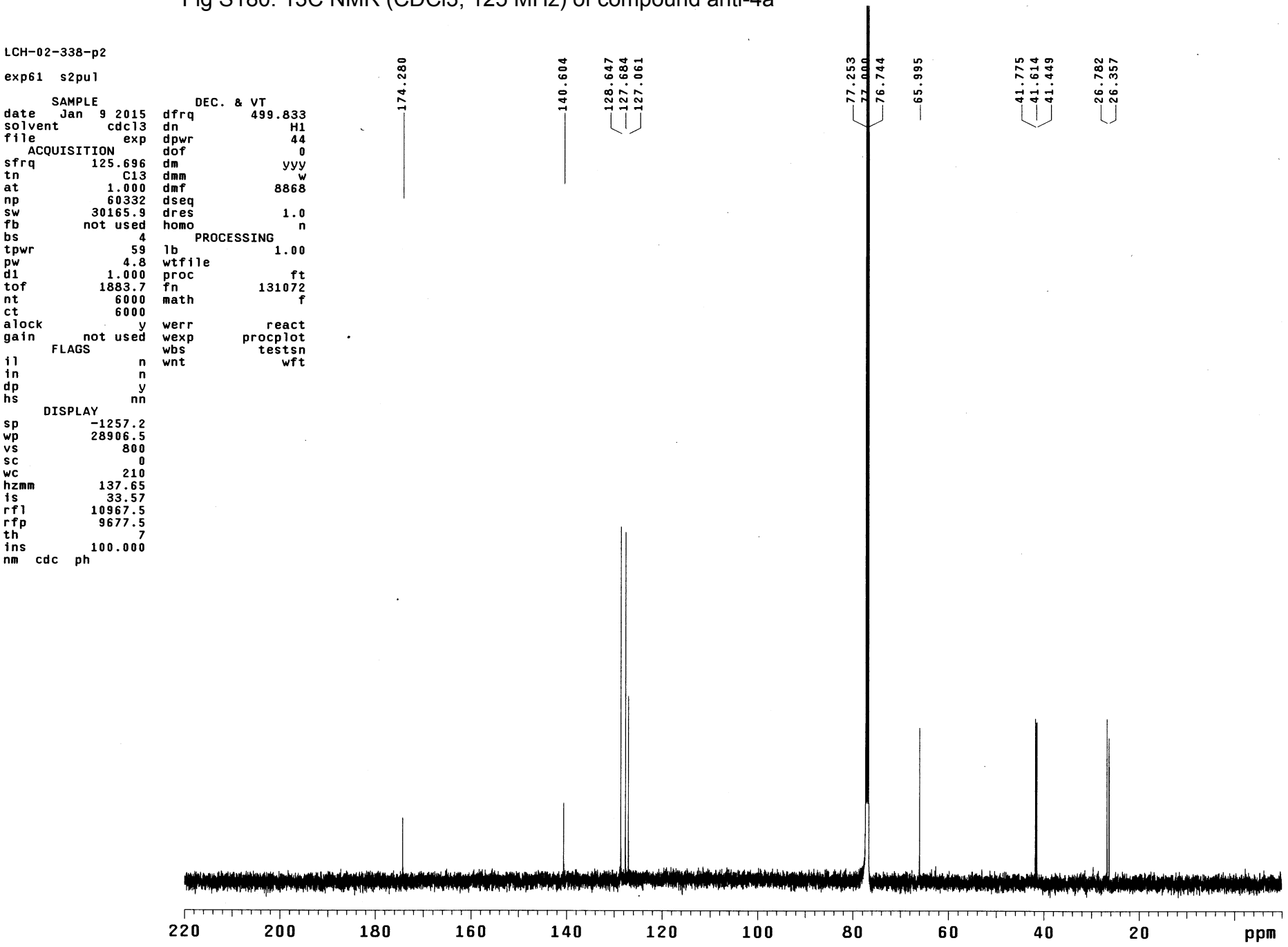


Fig S180. 13C NMR (CDCl3, 125 MHz) of compound anti-4a



LCH-02-338-p2

Sample Name **LCH-02-338-p2**	Pulse sequence **DEPT**	Temperature **100**	Study owner **vnmr2**
Date collected **2015-01-09**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

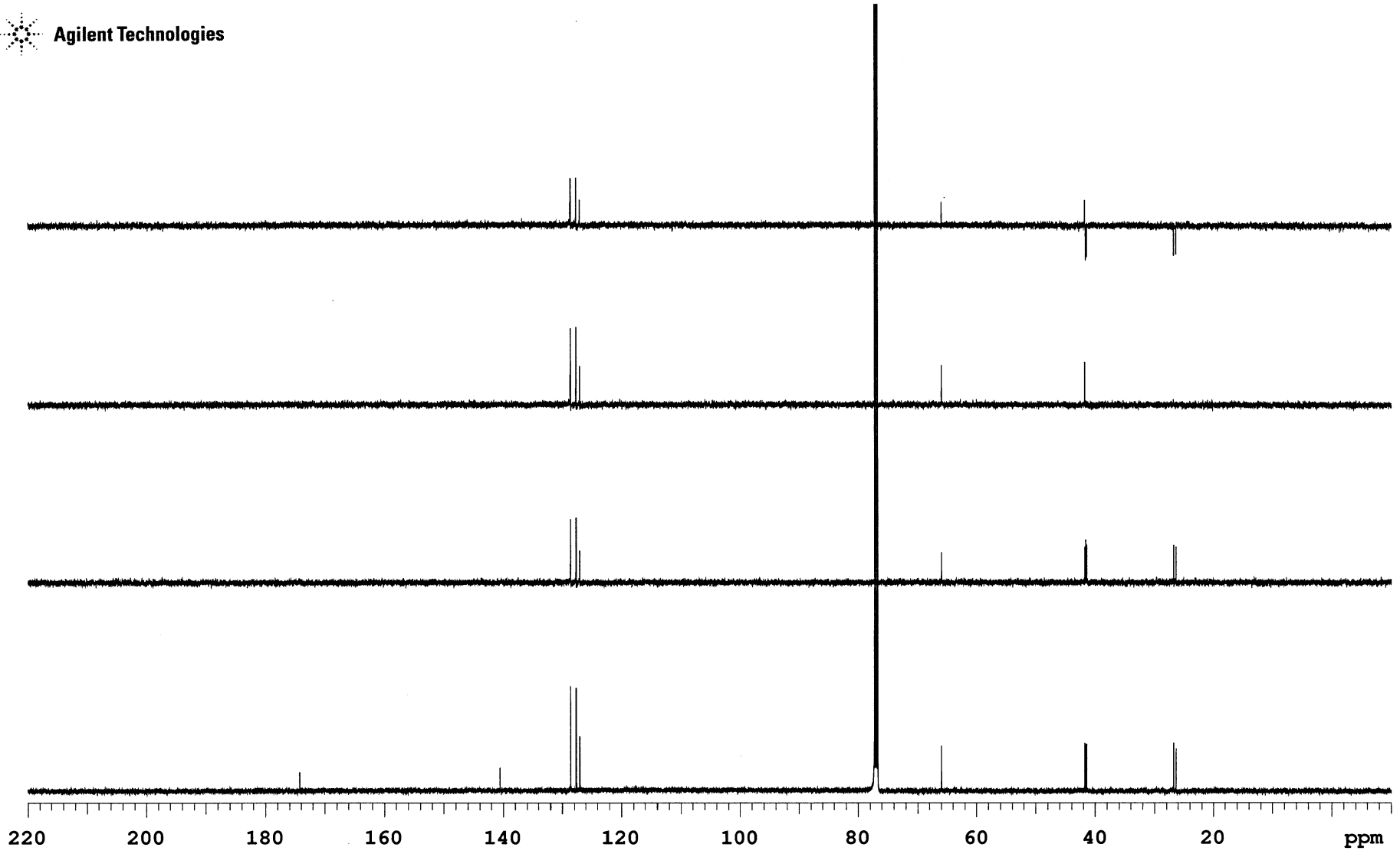


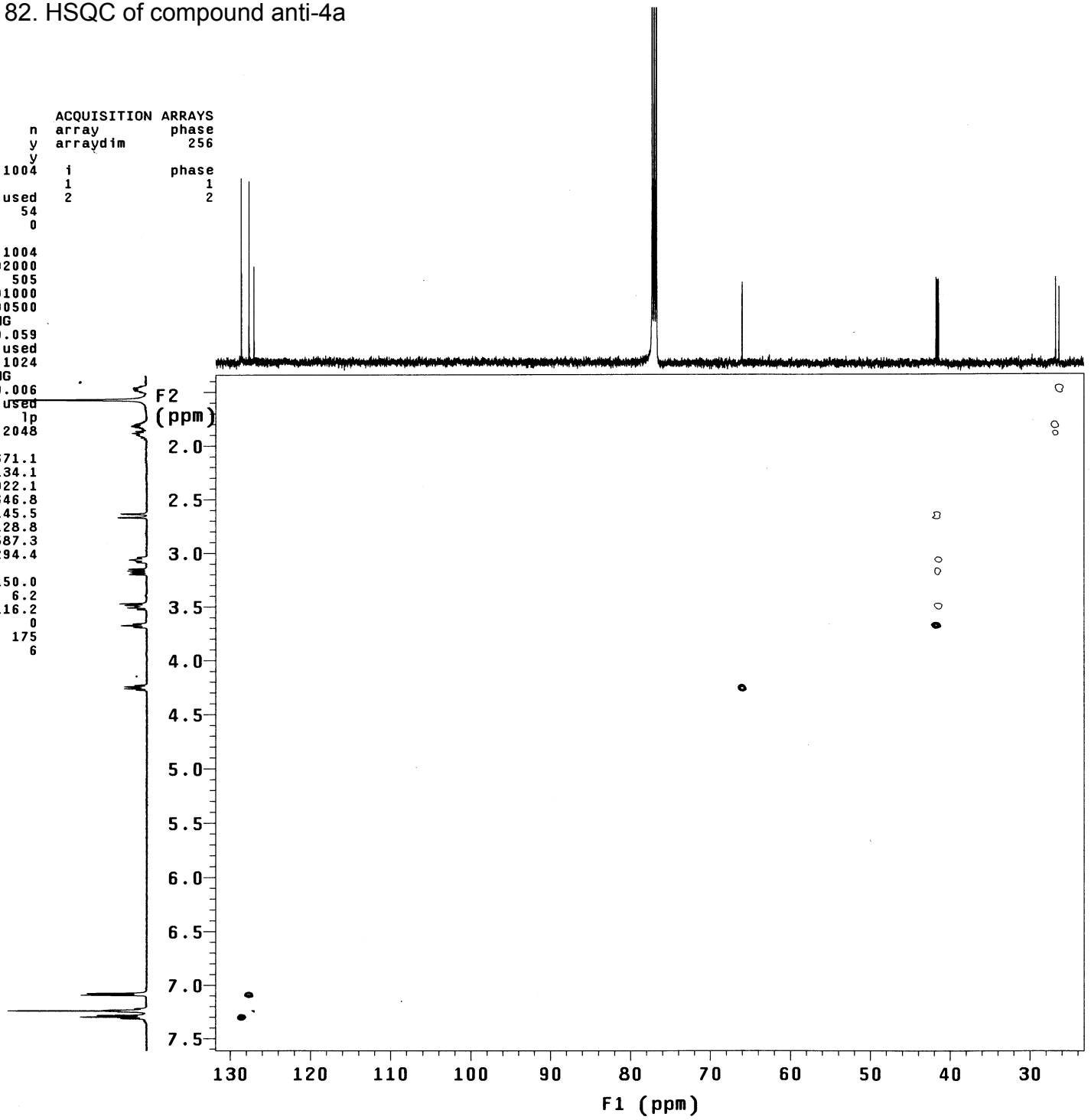
Fig S181. DEPT of compound anti-4a

Fig S182. HSQC of compound anti-4a

LCH-02-338-p2

exp65 gHSQC

SAMPLE		FLAGS		ACQUISITION ARRAYS	
date	Jan 9 2015	hs	n	array	phase
solvent	cdcl3	sspul	y	arraydim	256
sample	undefined	PFGflg	y		
ACQUISITION		hsglvl	1004	i	phase
sw	4001.6	SPECIAL		1	1
at	0.128	temp	not used	2	2
np	1024	gain	54		
fb	not used	spin	0		
ss	32	GRADIENTS			
d1	1.000	gzlvl1	1004		
nt	8	gt1	0.002000		
2D ACQUISITION		gzlvl3	505		
sw1	21367.5	gt3	0.001000		
ni	128	gstab	0.000500		
phase	arrayed	F2 PROCESSING			
TRANSMITTER		gf	0.059		
tn	H1	gfs	not used		
sfrq	499.832	fn	1024		
tof	-499.9	F1 PROCESSING			
tpwr	62	gf1	0.006		
pw	13.800	gfs1	not used		
DECOUPLER		proc1	lp		
dn	C13	fn1	2048		
dof	-2515.1	DISPLAY			
dm	nny	sp	671.1		
dmm	ccp	wp	3134.1		
dmf	32258	sp1	2922.1		
dpwr	43	wp1	13646.8		
pwxlvl	61	rfl	2145.5		
pwx	11.000	rfp	2128.8		
HSQC		rfl1	9587.3		
j1xh	140.0	rfp1	8294.4		
nullflg	y	PLOT			
mult	2	wc	150.0		
		sc	6.2		
		wc2	116.2		
		sc2	0		
		vs	175		
		th	6		
		ai cdc ph			



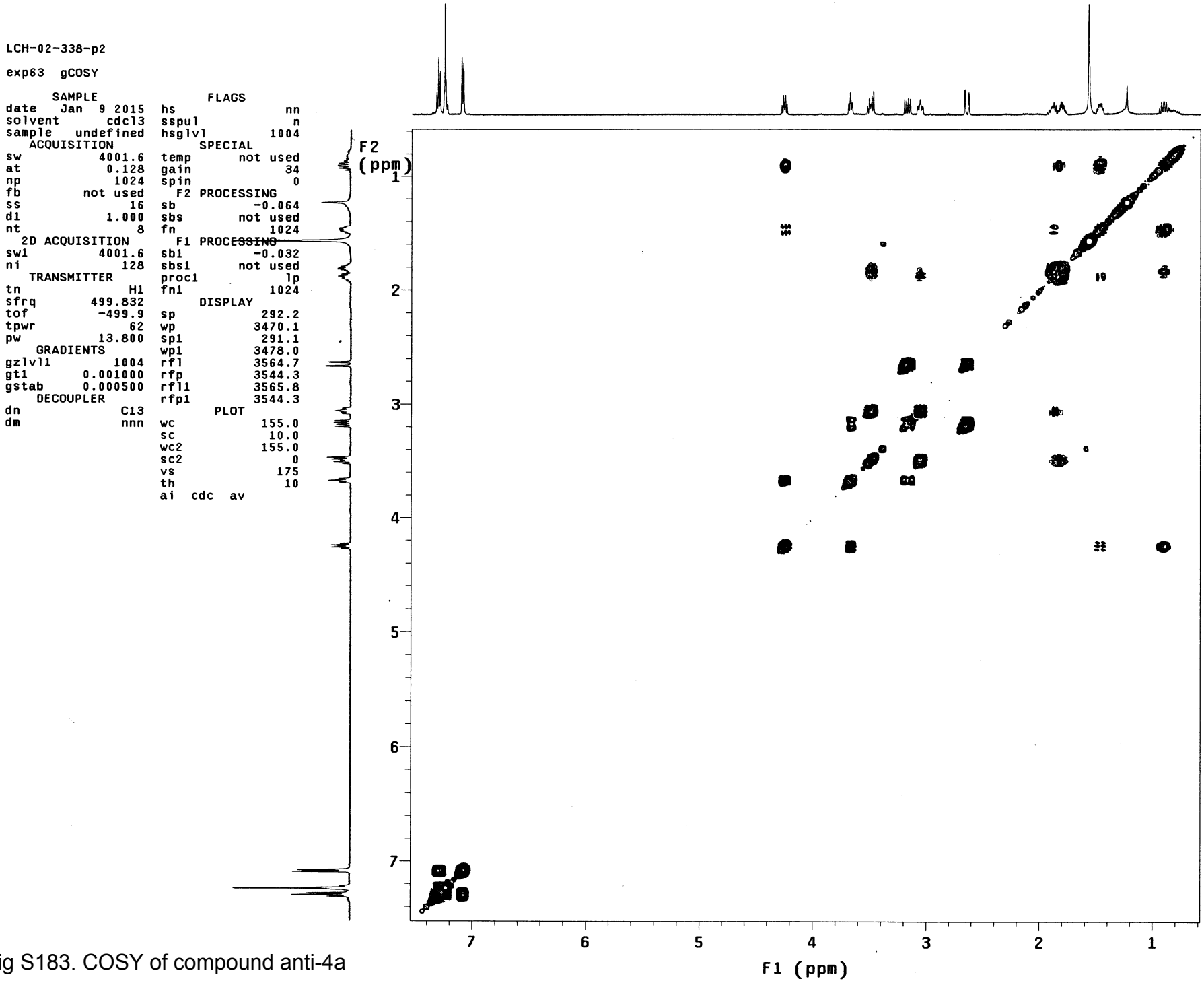


Fig S183. COSY of compound anti-4a

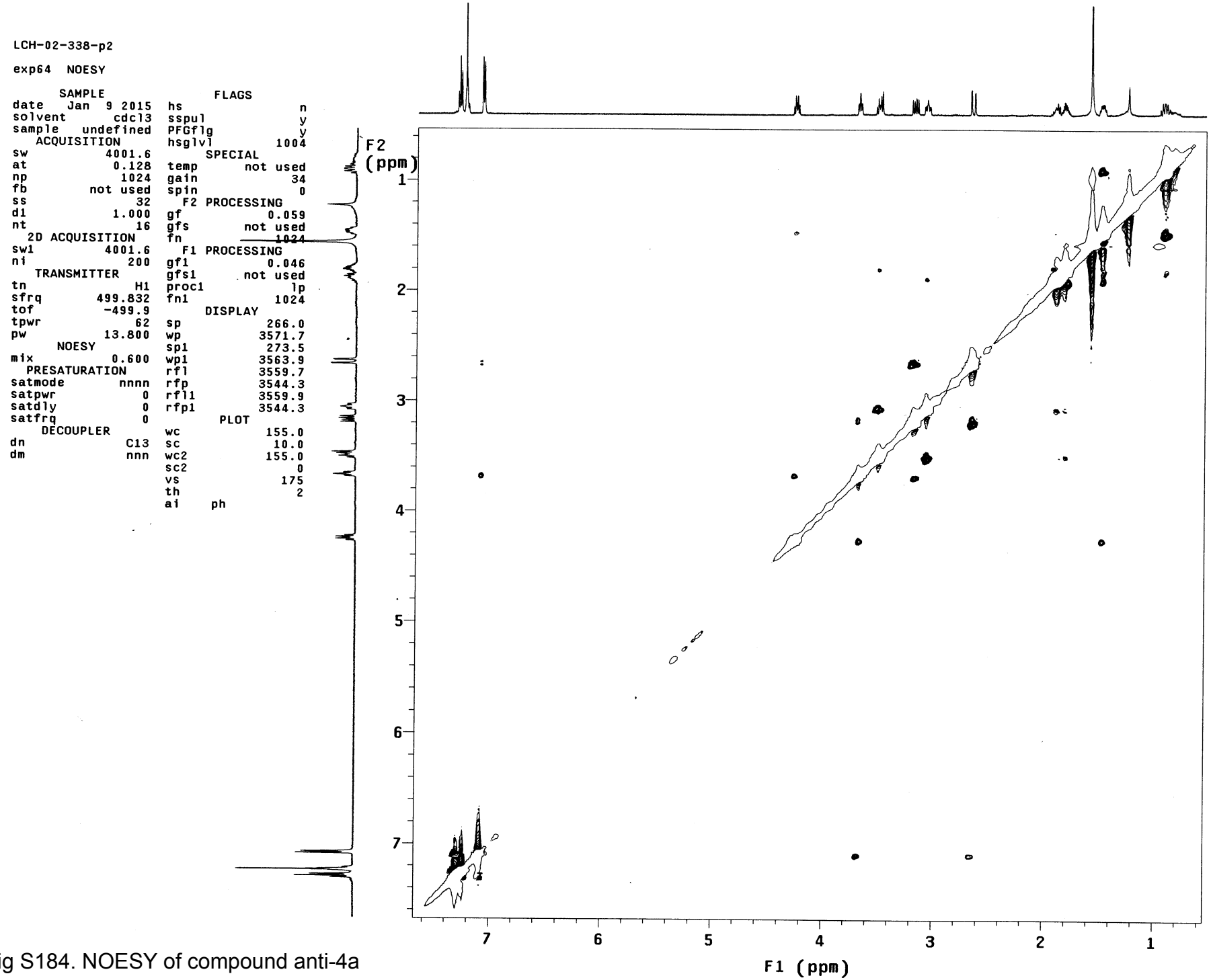
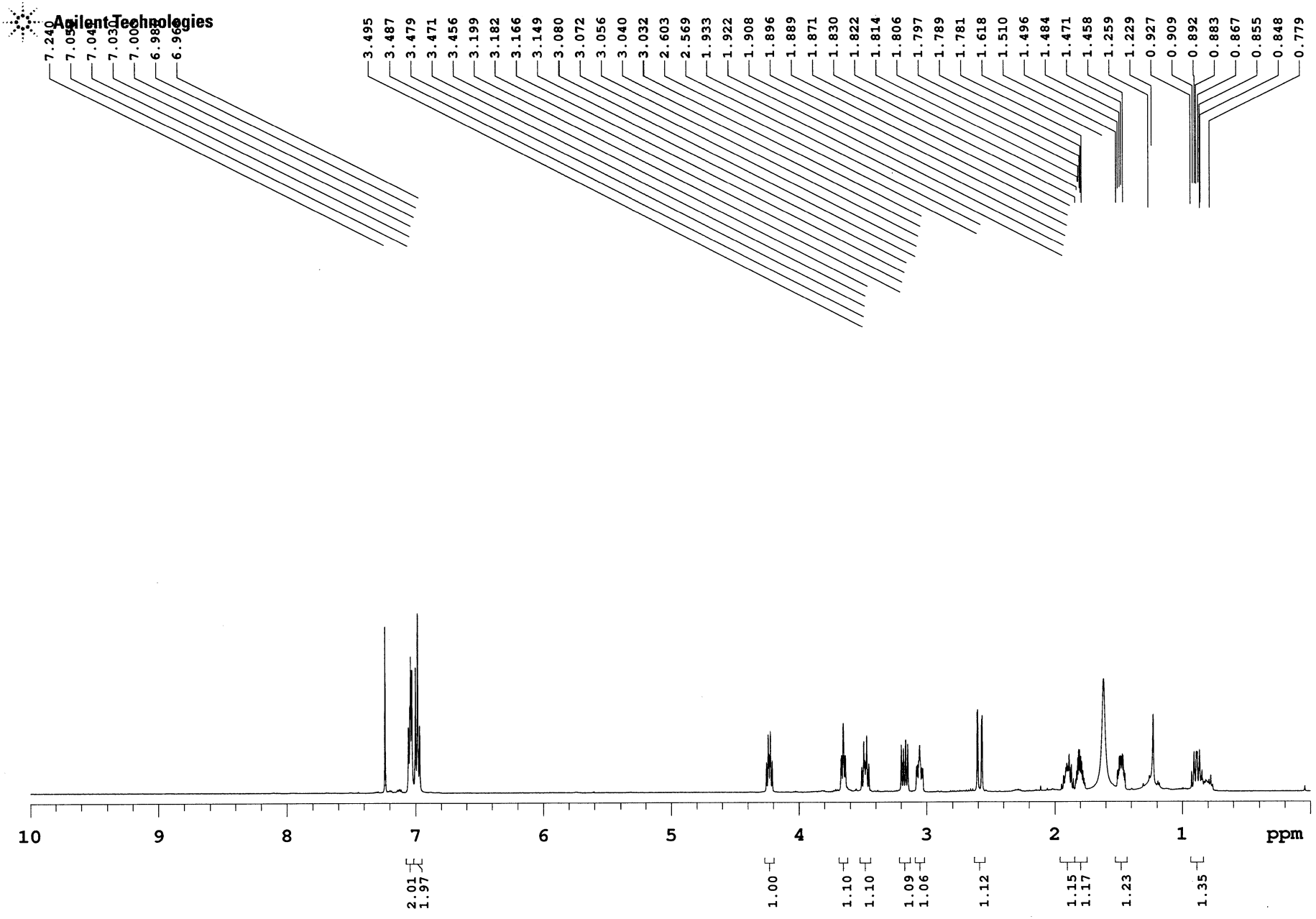


Fig S184. NOESY of compound anti-4a

LCH-02-395

Sample Name	**LCH-02-395**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2015-06-04**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-395/PROTON_01.fid

Fig S185. 1H NMR (CDCl3, 500 MHz) of compound anti-4b

Plot date 2015-10-24

LCH-02-395

Sample Name **LCH-02-395** Pulse sequence **s2pul** Temperature **50** Study owner **vnmr2**
Date collected **2015-04-13** Solvent **cdcl3** Spectrometer **---** Operator **vnmr2**

Agilent Technologies

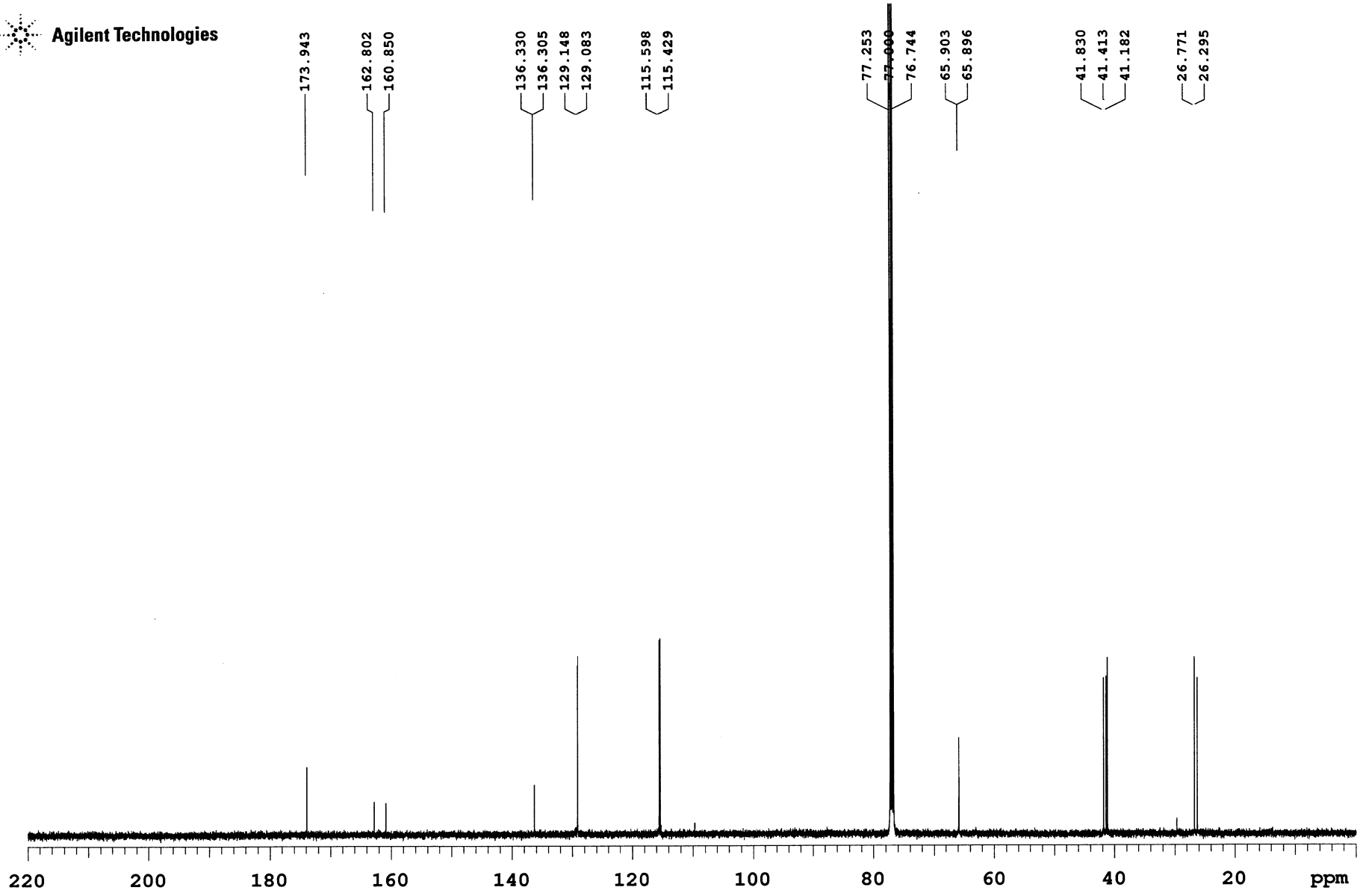


Fig S186. 13C NMR (CDCl3, 125 MHz) of compound anti-4b

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-395/C13.fid

Plot date 2015-10-27

LCH-02-395

Sample Name **LCH-02-395**	Pulse sequence **DEPT**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-14**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

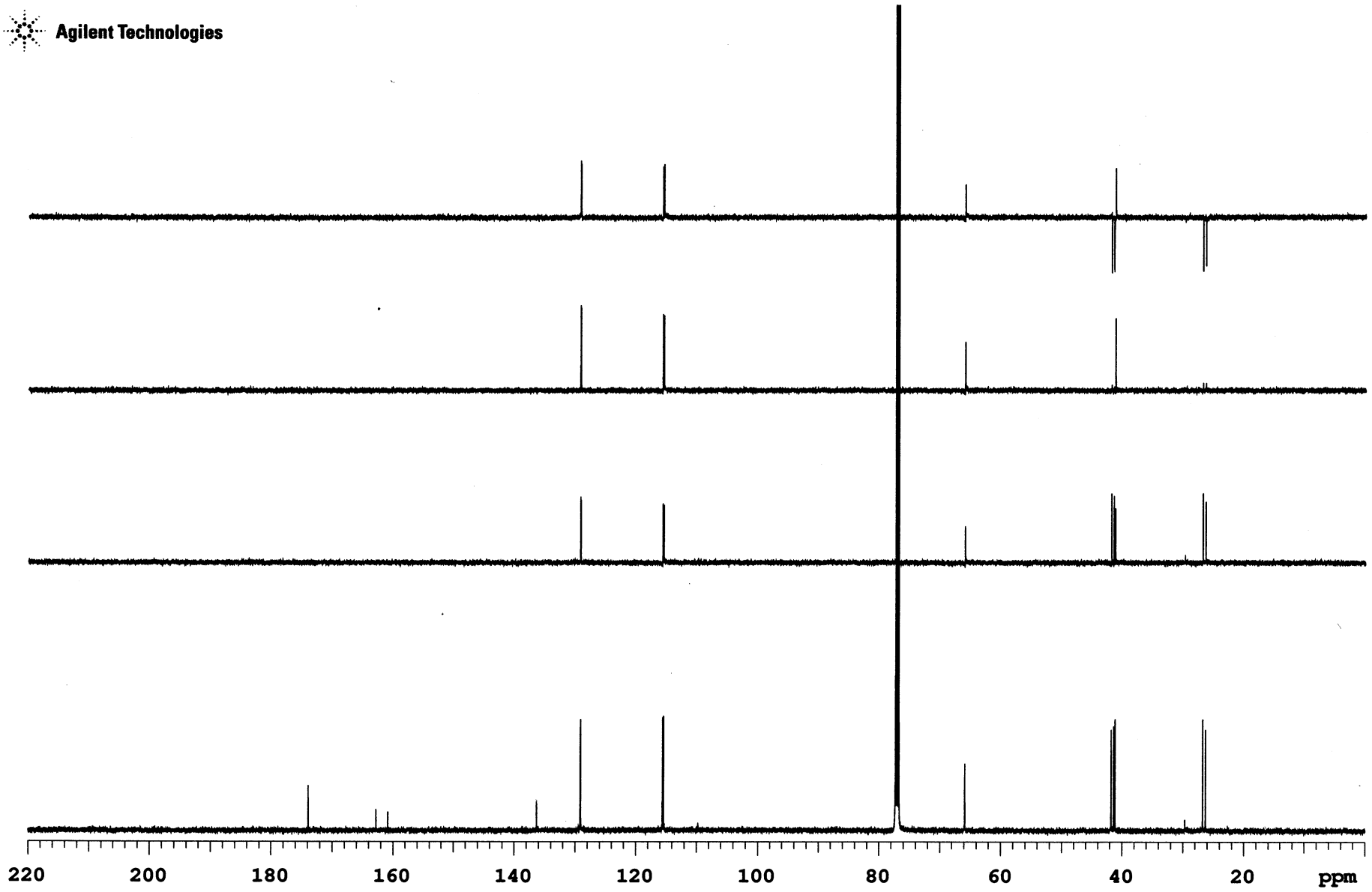


Fig S187. DEPT of compound anti-4b

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-395/DEPT.fid

Plot date 2015-07-15

LCH-02-395

Sample Name **LCH-02-395**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-06-04**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

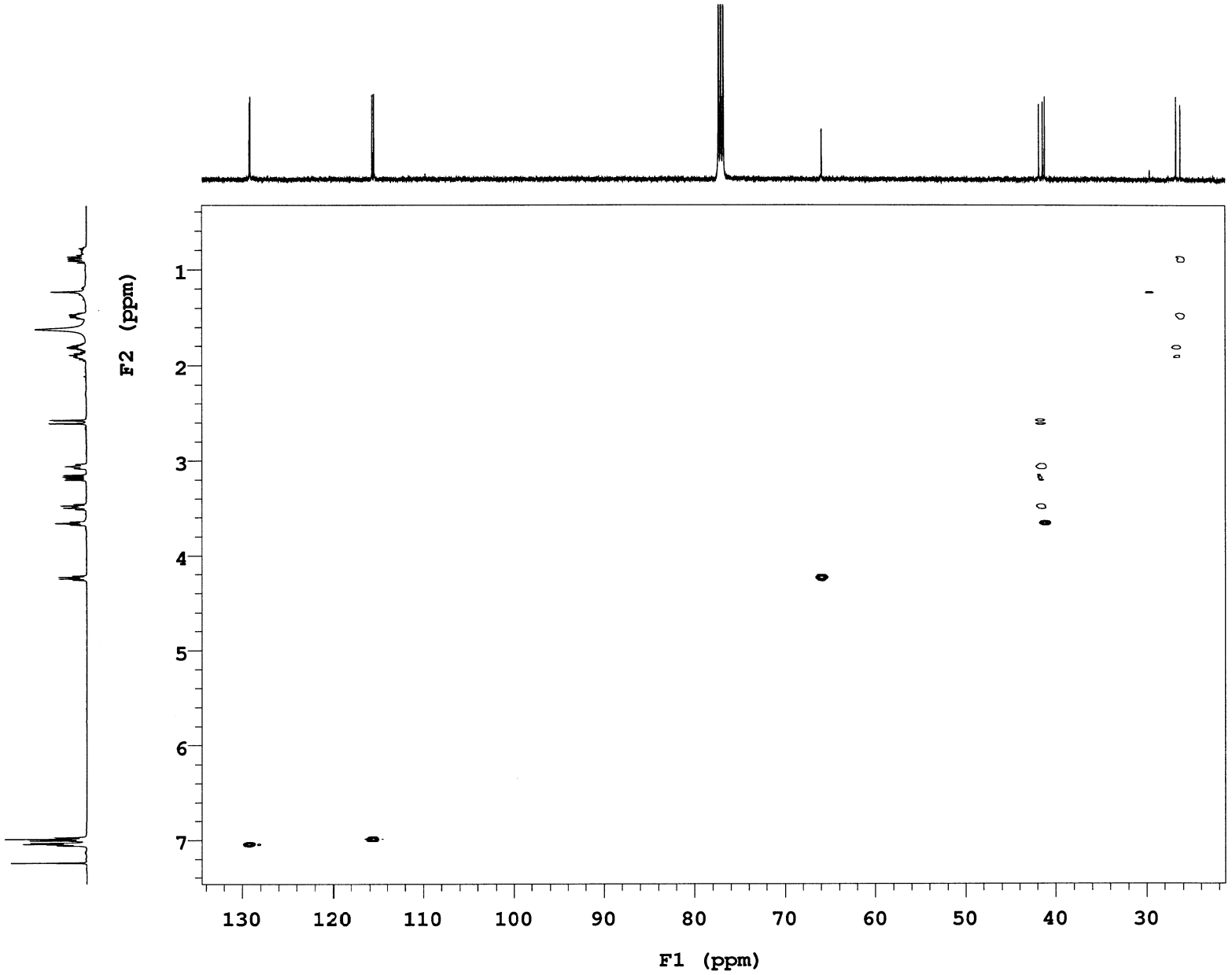


Fig S188. HSQC of compound anti-4b

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-395/gHSQC_01.fid

Plot date 2015-10-24

LCH-02-395

Sample Name **LCH-02-395**	Pulse sequence **gCOSY**	Temperature **50**	Study owner **vnmr2**
Date collected **2015-04-14**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

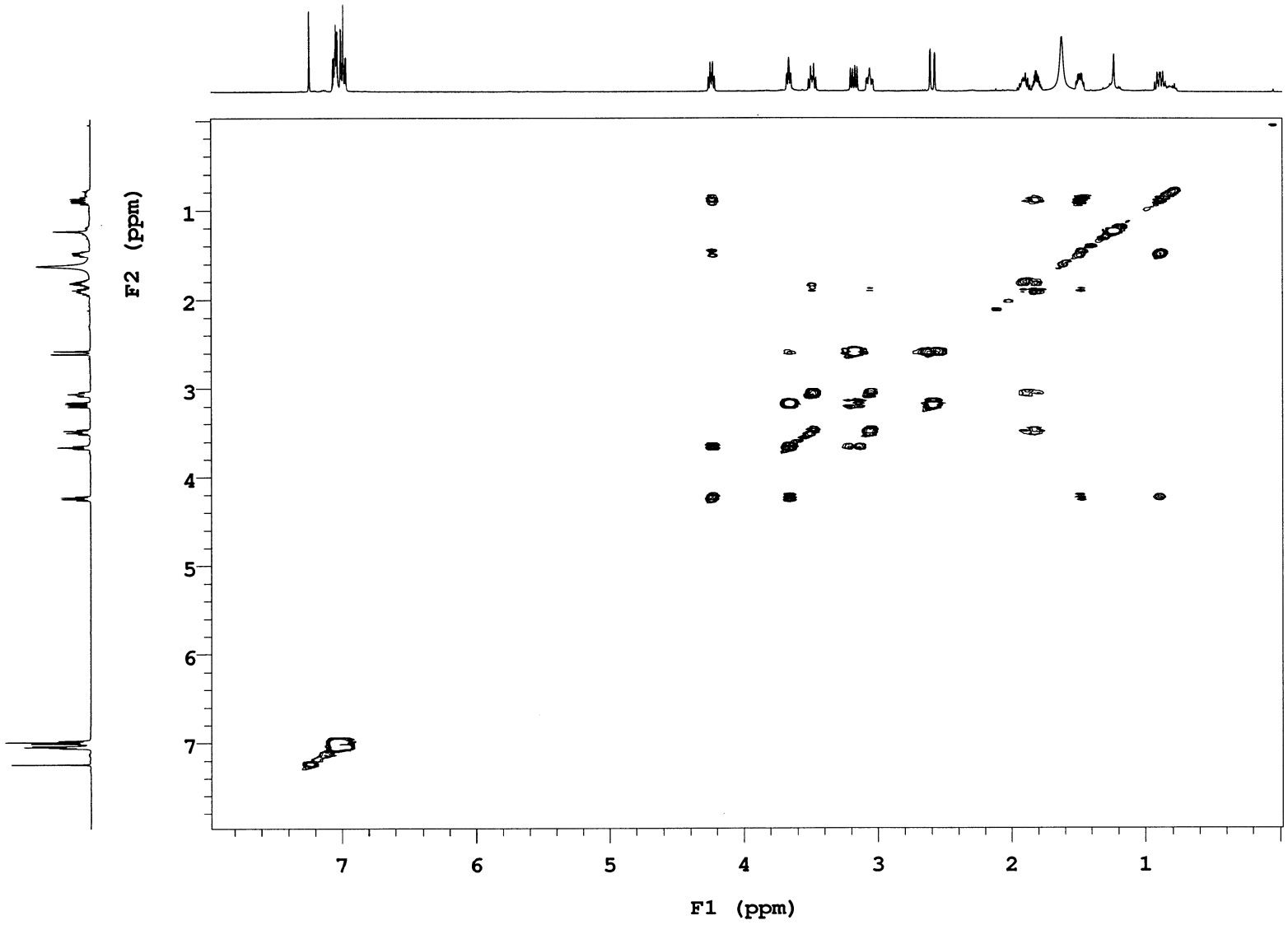


Fig S189. COSY of compound anti-4b

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-395/COSY.fid

Plot date 2015-10-24

LCH-02-395

Sample Name	**LCH-02-395**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2015-06-04**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

Agilent Technologies

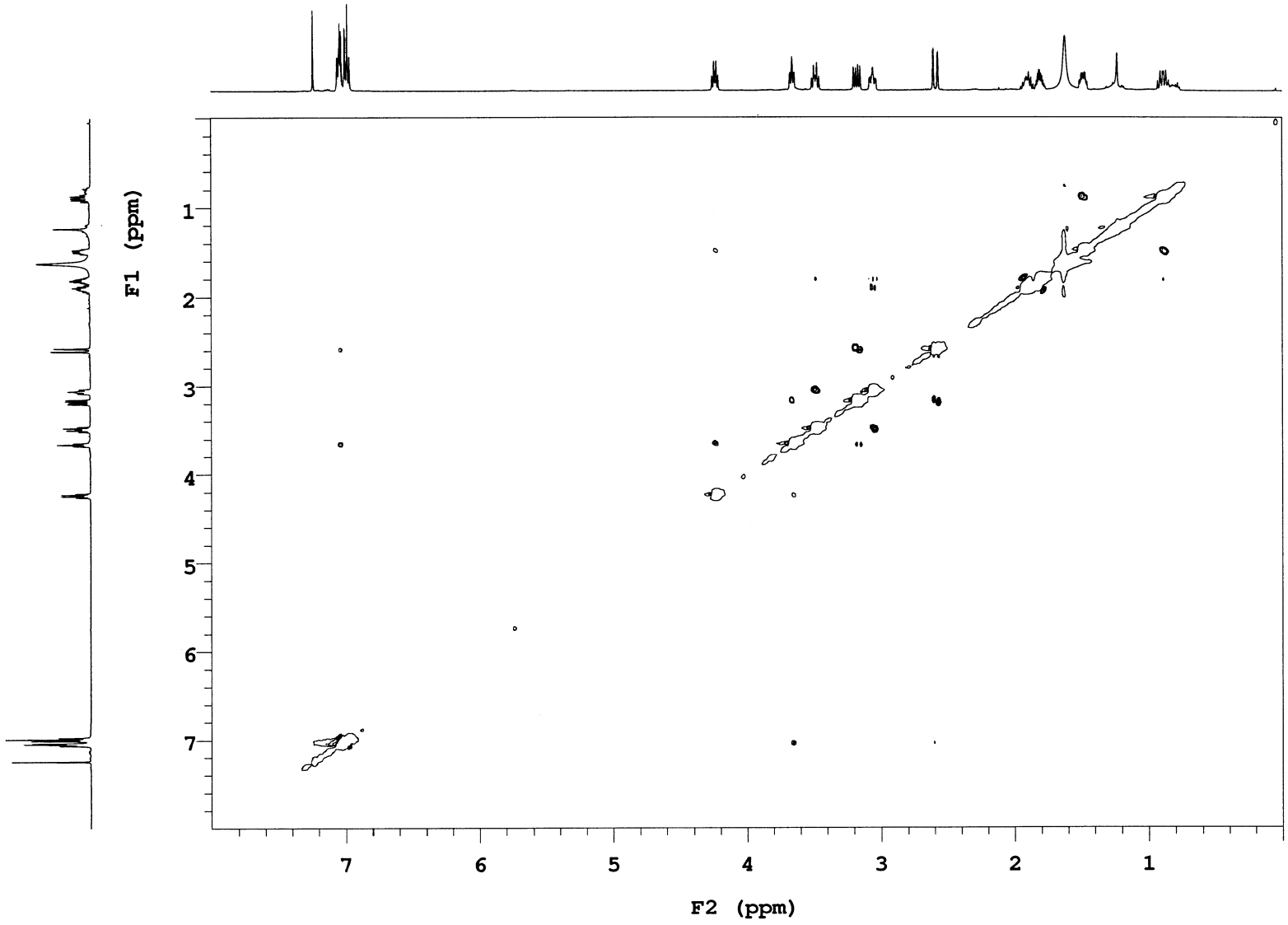


Fig S190. NOESY of compound anti-4b

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-395/NOESY_01.fid

Plot date 2015-10-24

Fig S191. 1H NMR (CDCl3, 500 MHz) of compound anti-4c

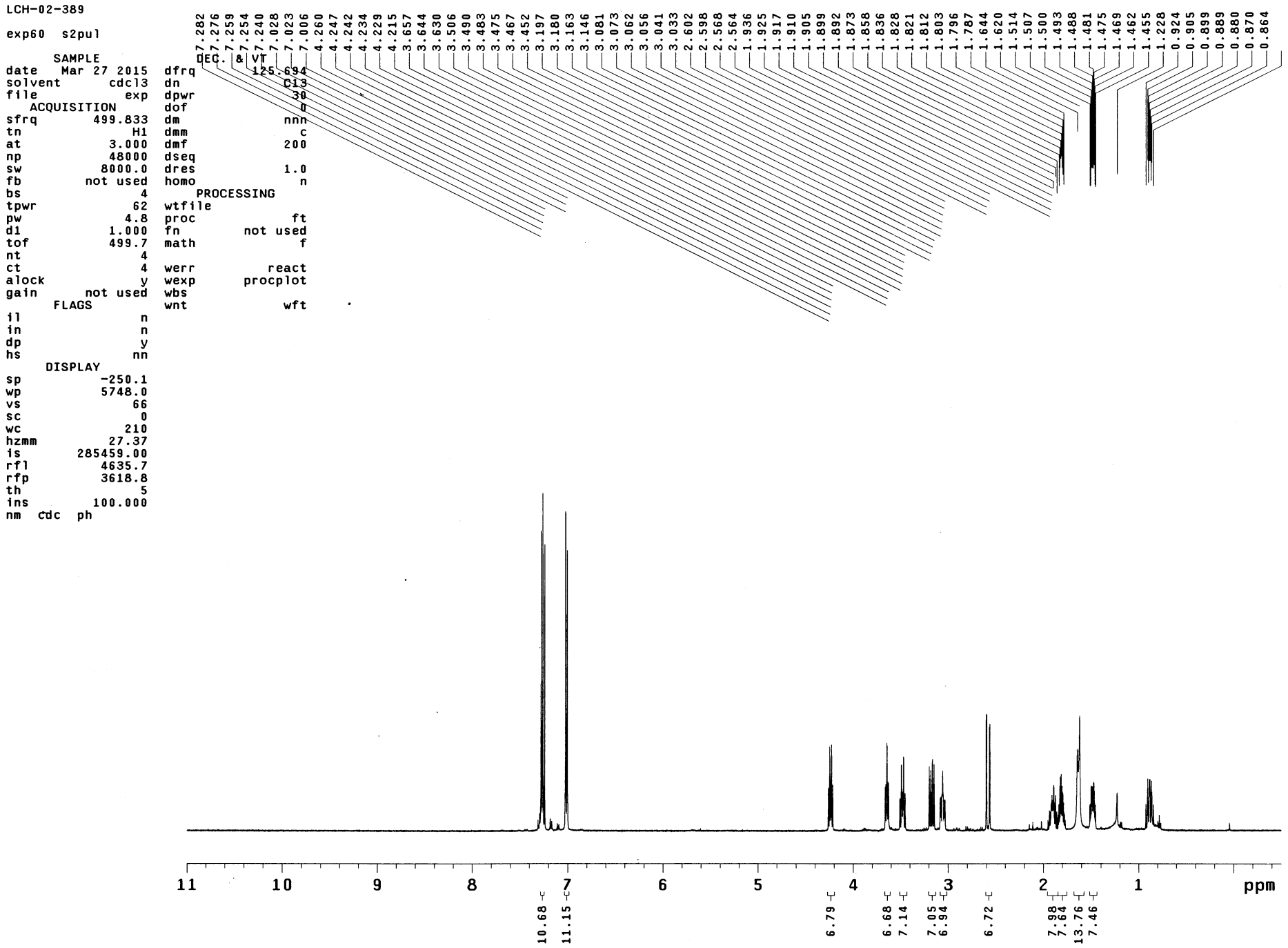


Fig S192. 13C NMR (CDCl3, 125 MHz) of compound anti-4c

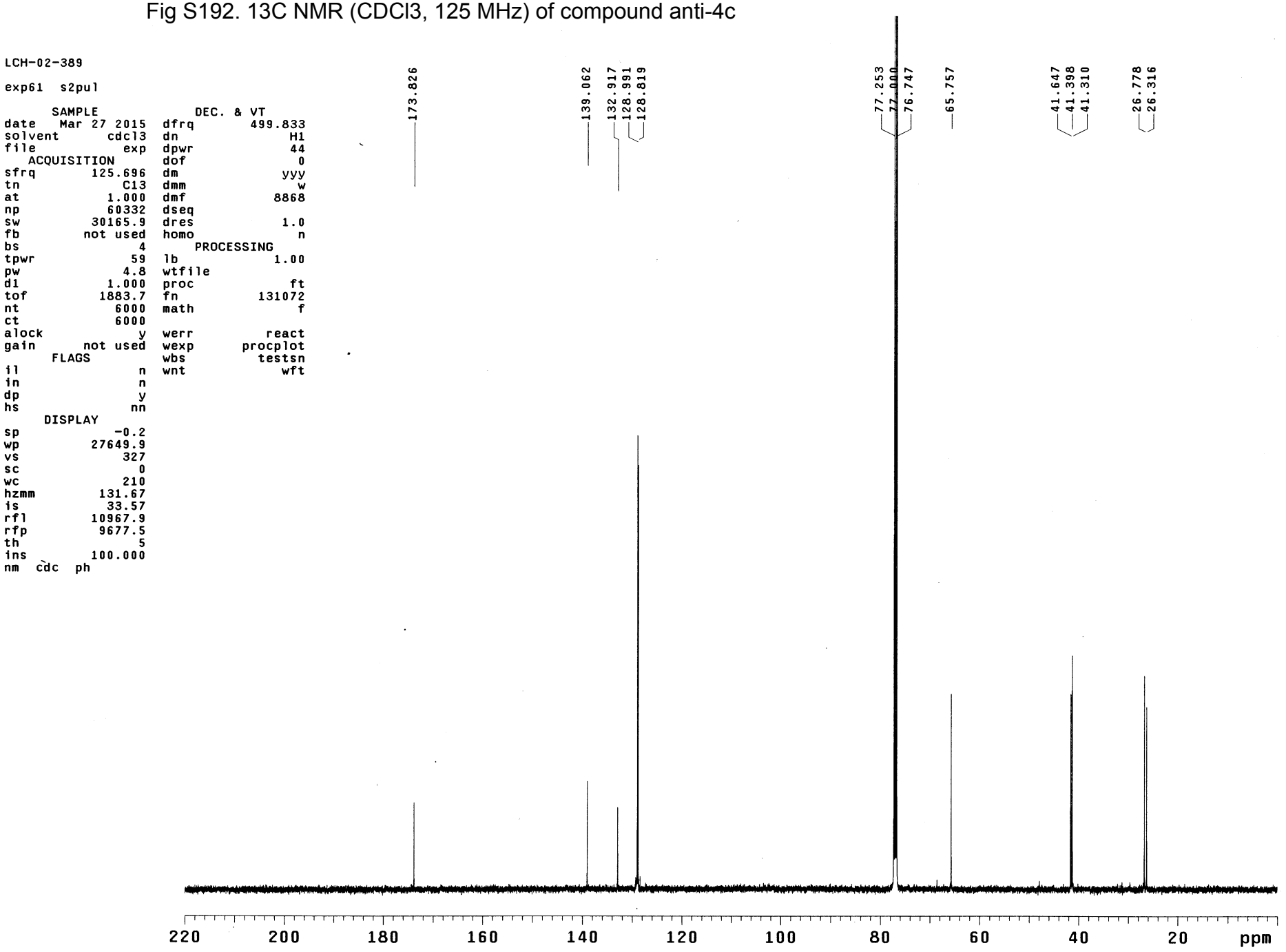


Fig S193. DEPT of compound anti-4c

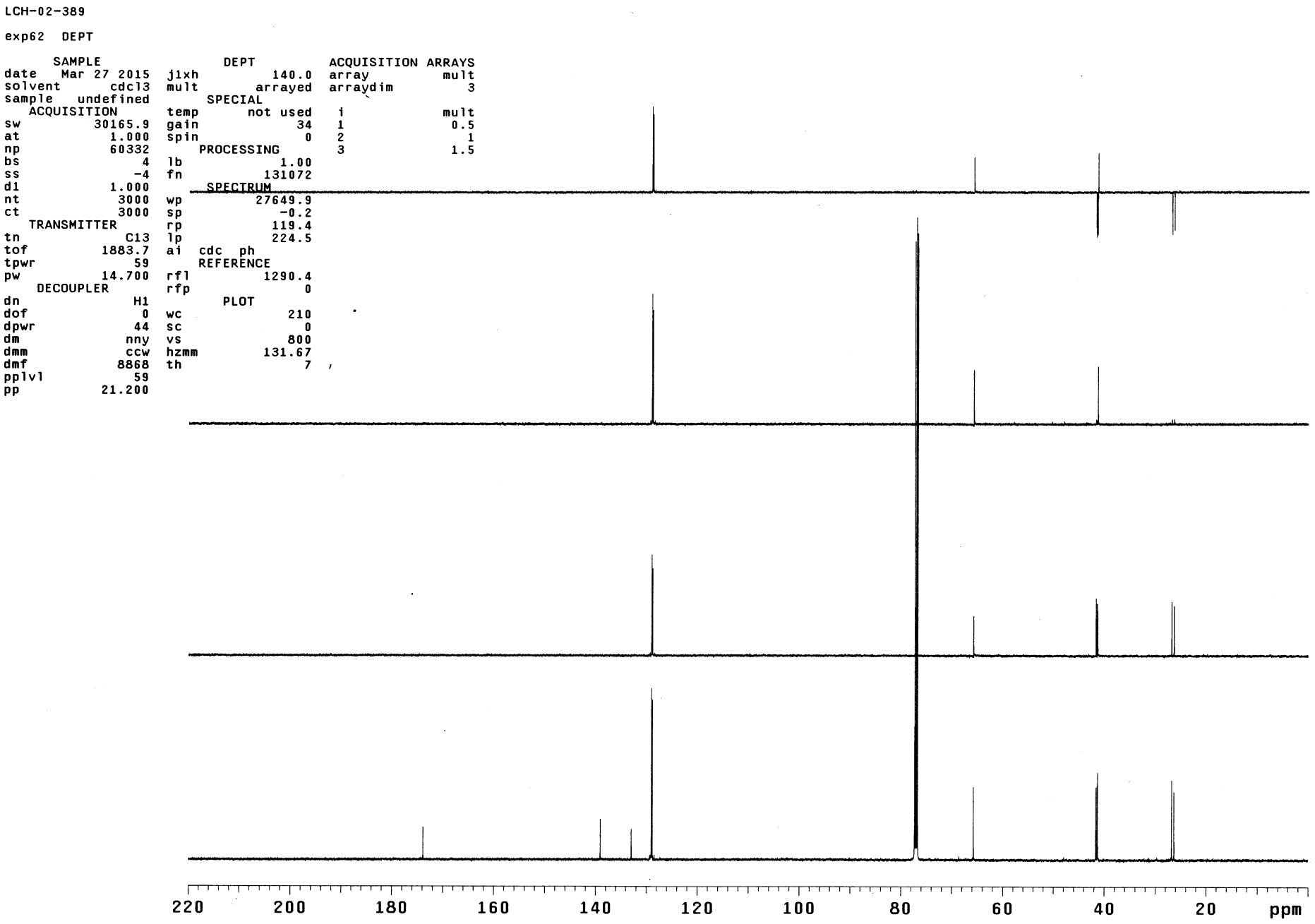
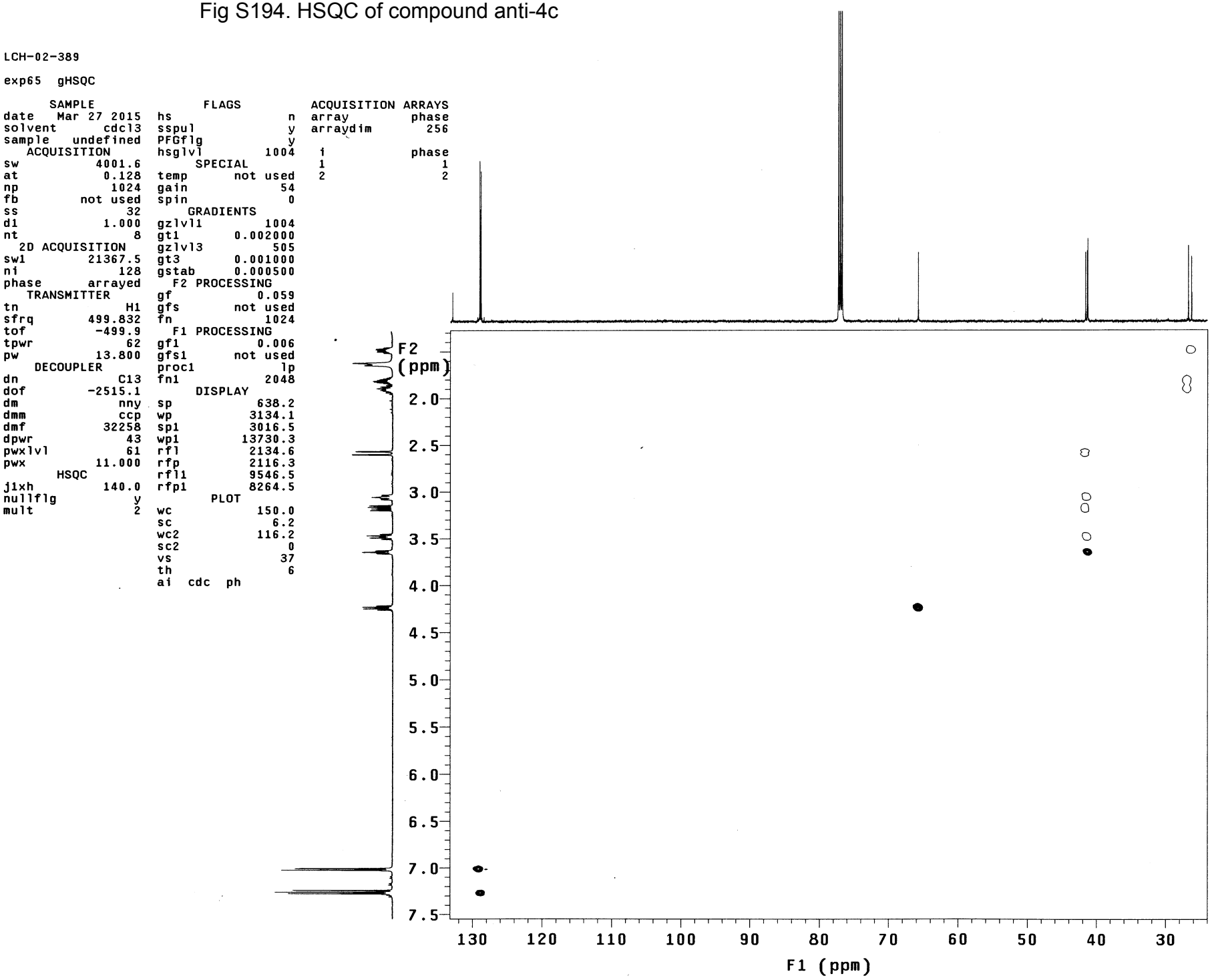


Fig S194. HSQC of compound anti-4c



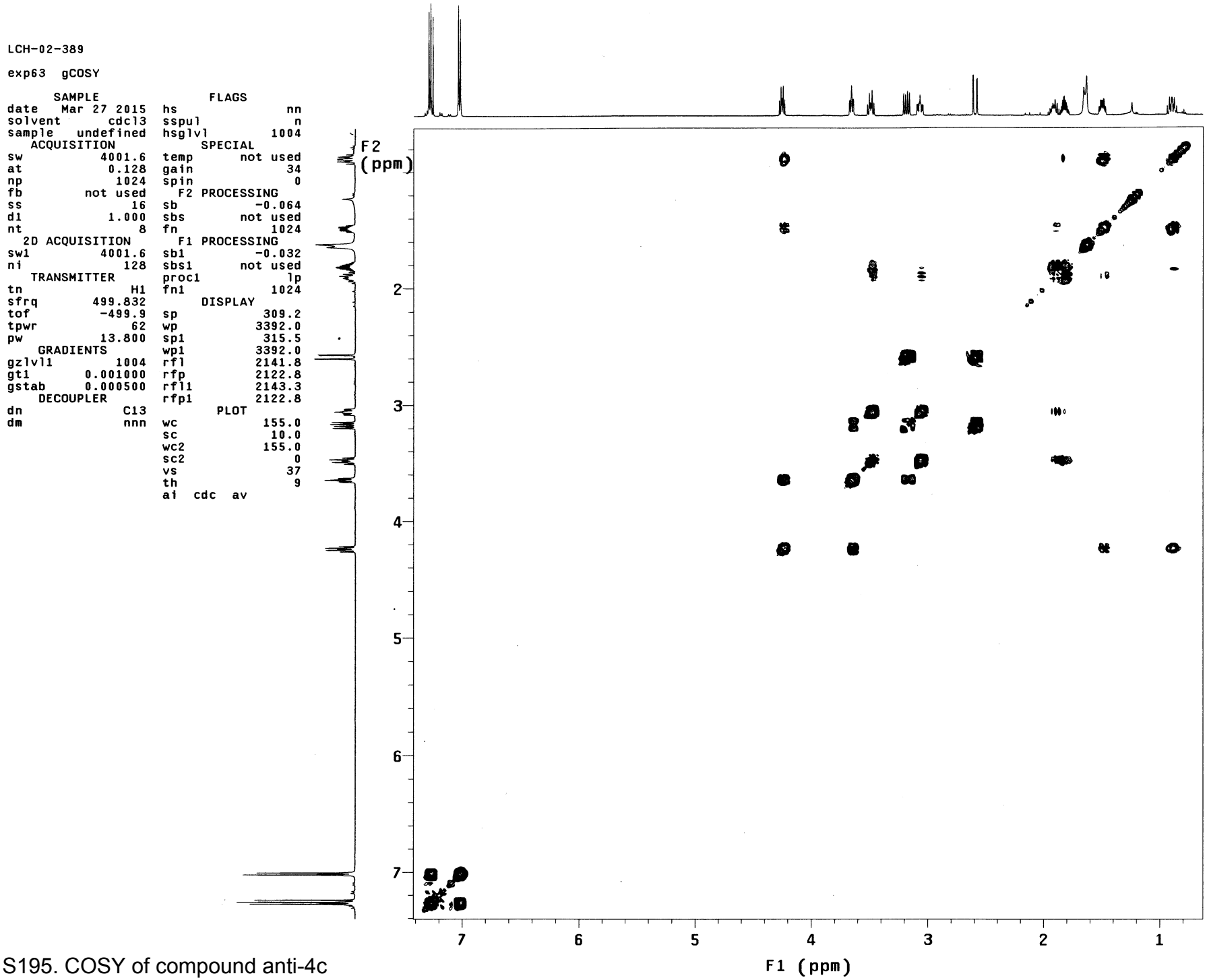


Fig S195. COSY of compound anti-4c

LCH-02-389

exp64 NOESY

SAMPLE		FLAGS	
date	Mar 27 2015	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1004
sw	4001.6	SPECIAL	
at	0.128	temp	not used
np	1024	gain	34
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.059
nt	16	gfs	not used
2D ACQUISITION		fn	1024
sw1	4001.6	F1 PROCESSING	
ni	200	gf1	0.046
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.832	fn1	1024
tof	-499.9	DISPLAY	
tpwr	62	sp	322.1
pw	13.800	wp	3446.7
NOESY		sp1	328.6
mix	0.600	wp1	3438.9
PRESATURATION		rfl	2134.3
satmode	nnnn	rfp	2120.3
satpwr	0	rfl1	2135.6
satdly	0	rfp1	2120.3
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	37
		th	2
		ai ph	

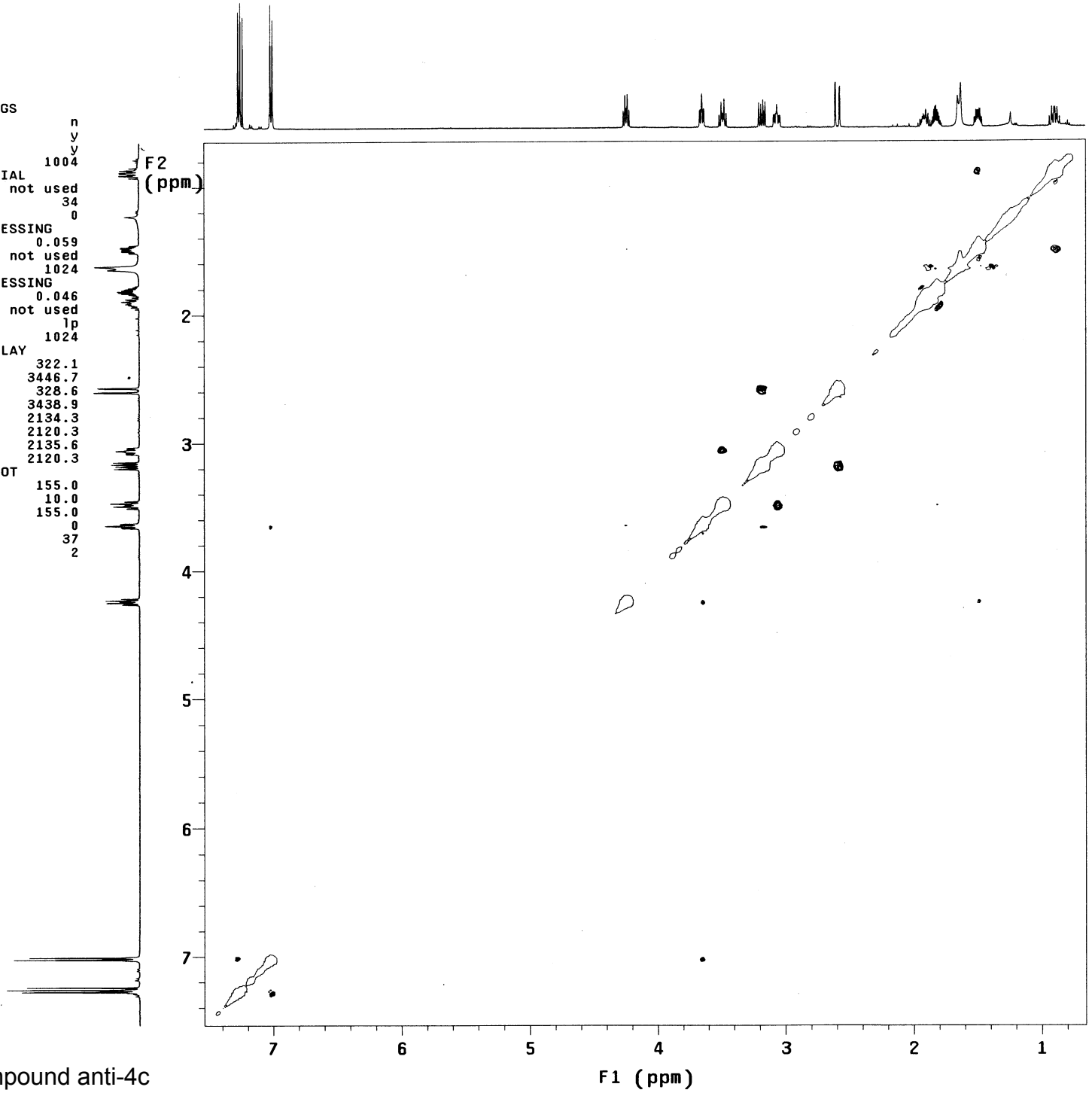


Fig S196. NOESY of compound anti-4c

Fig S197. 1H NMR (CDCl3, 500 MHz) of compound anti-4d

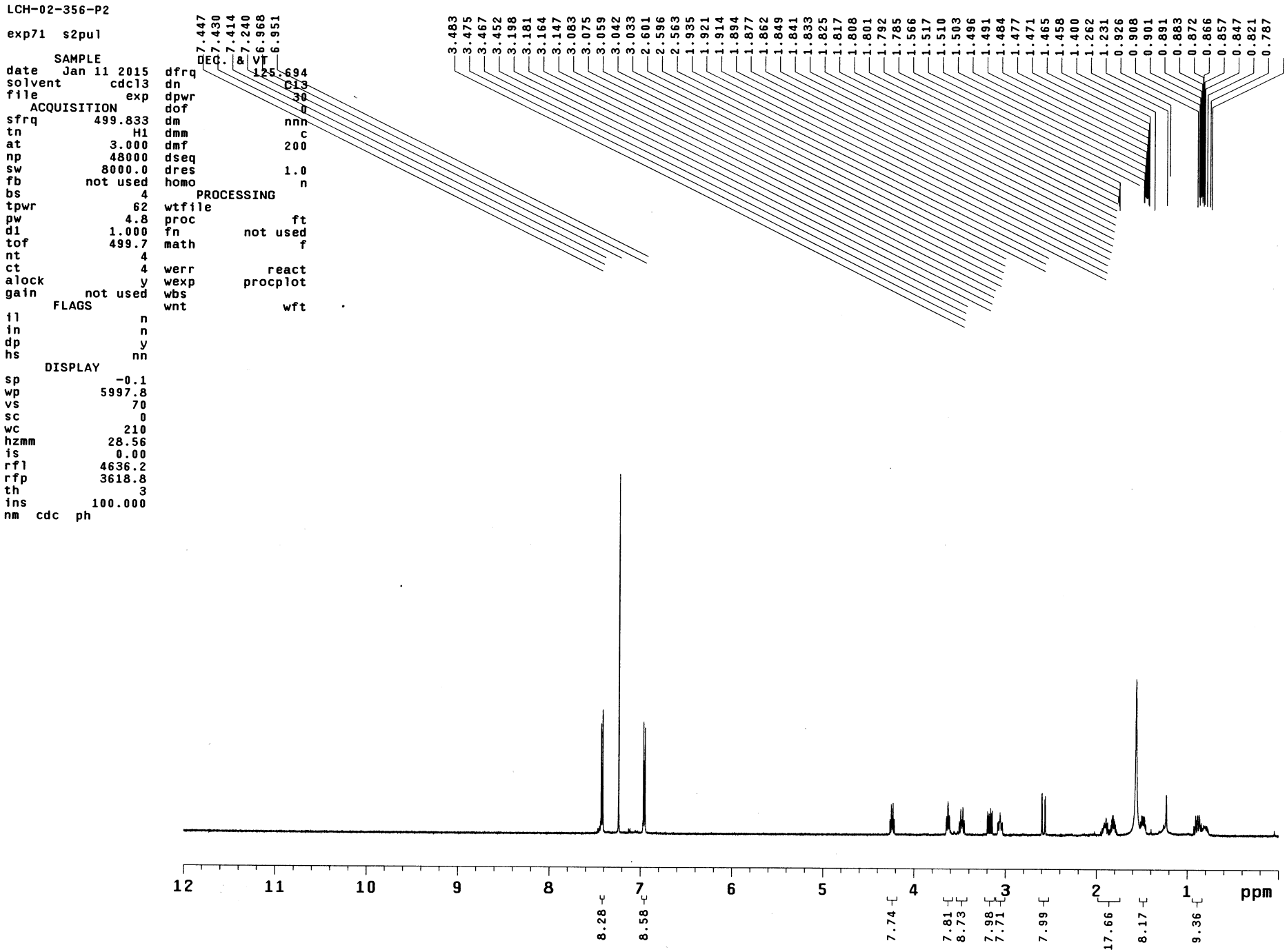


Fig S198. 13C NMR (CDCl3, 125 MHz) of compound anti-4d

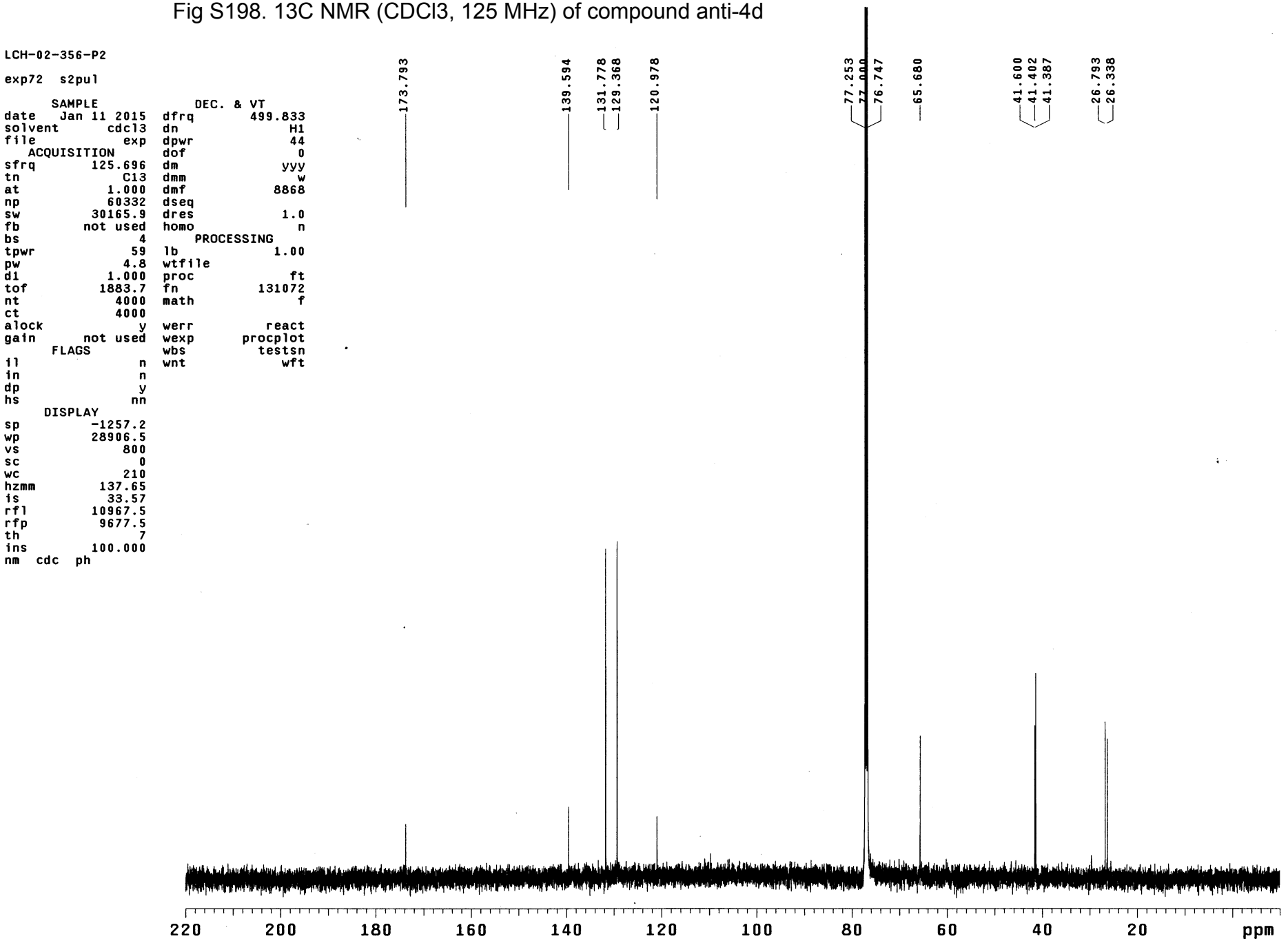


Fig S199. DEPT of compound anti-4d

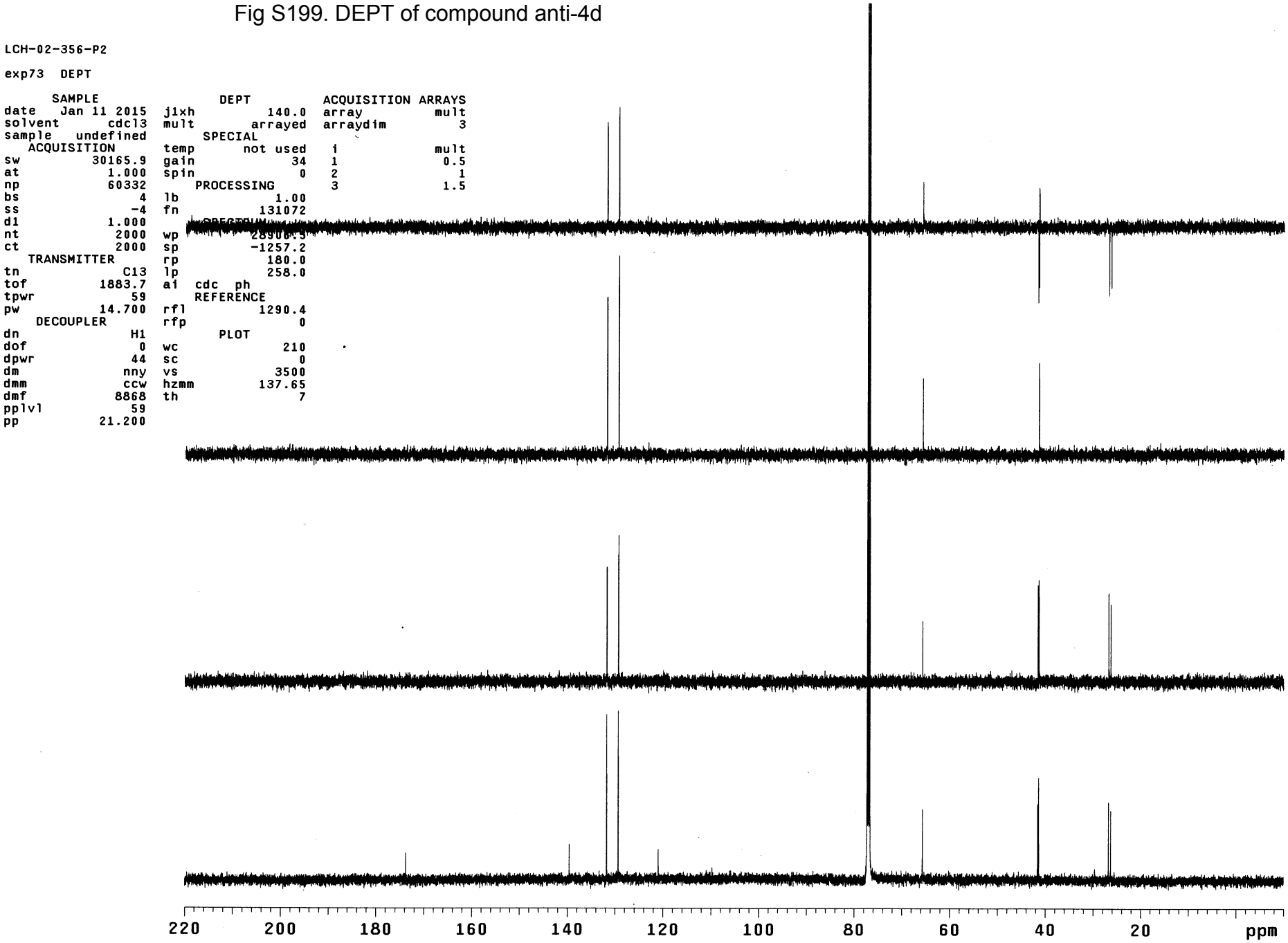
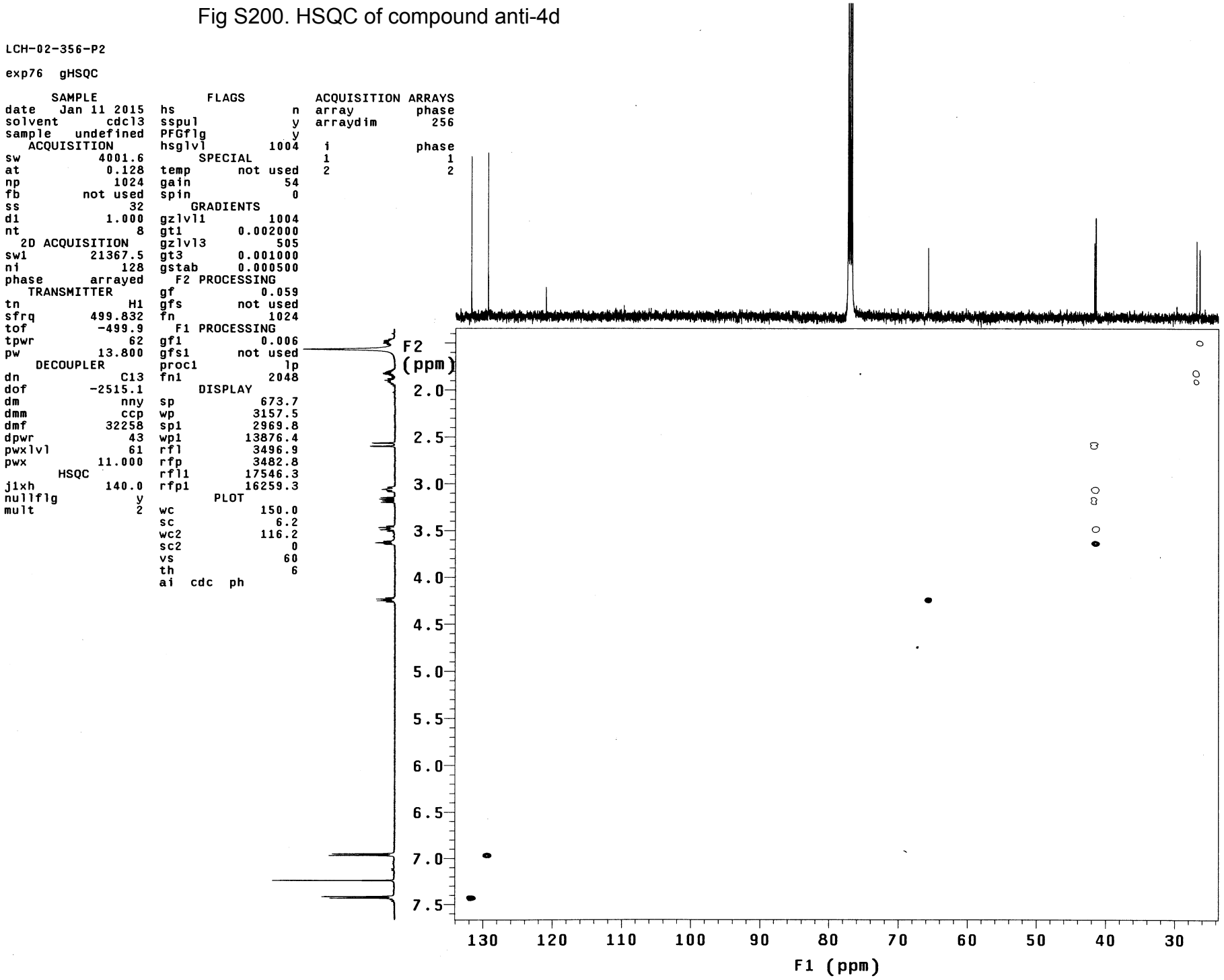


Fig S200. HSQC of compound anti-4d



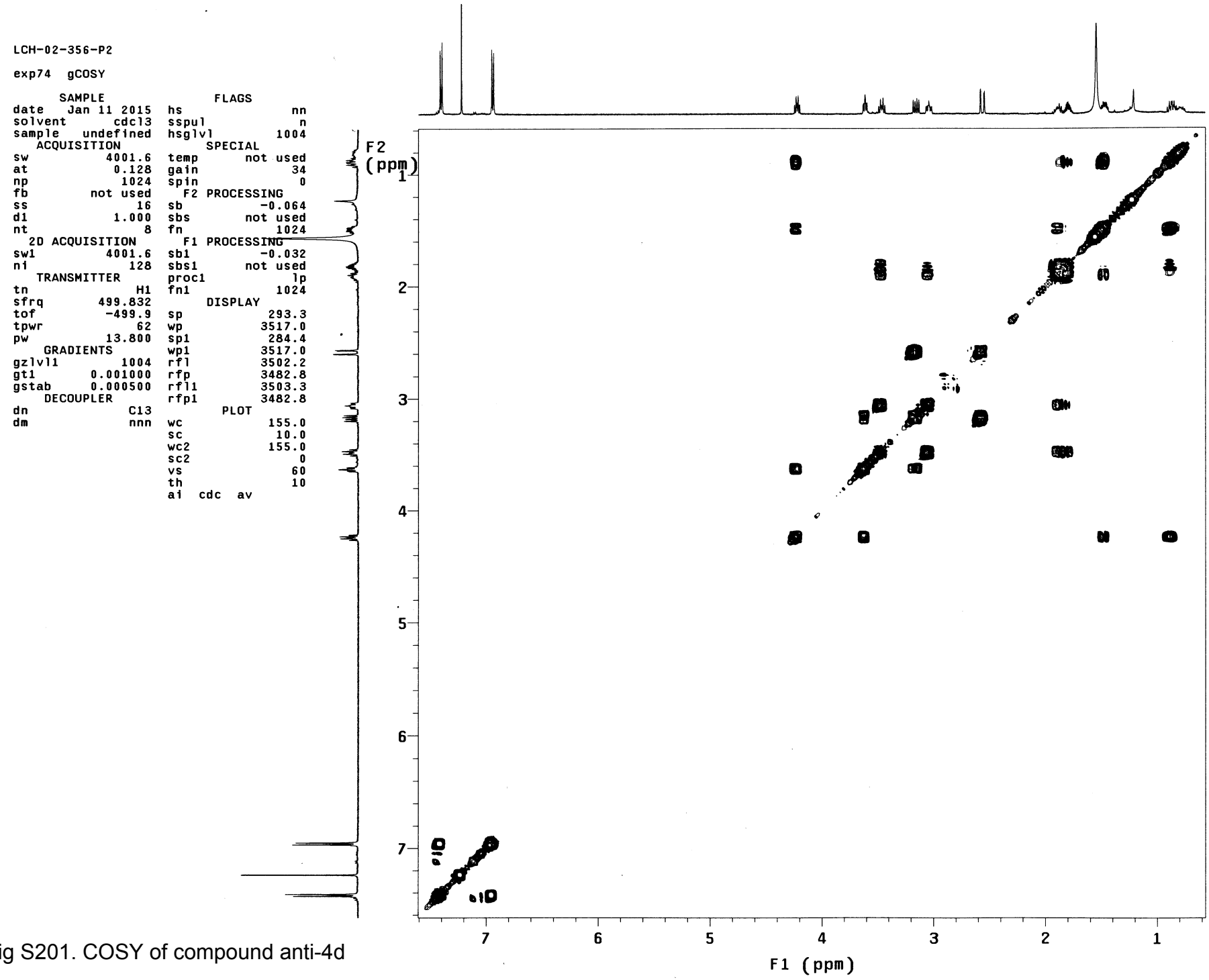


Fig S201. COSY of compound anti-4d

LCH-02-356-P2

exp75 NOESY

SAMPLE		FLAGS	
date	Jan 11 2015	hs	n
solvent	cdcl3	sspul	y
sample	undefined	PFGflg	y
ACQUISITION		hsglvl	1004
sw	4001.6	SPECIAL	
at	0.128	temp	not used
np	1024	gain	34
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.059
nt	16	gfs	not used
2D ACQUISITION		fn	1024
sw1	4001.6	F1 PROCESSING	
ni	200	gf1	0.046
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.832	fn1	1024
tof	-499.9	DISPLAY	
tpwr	62	sp	297.8
pw	13.800	wp	3556.1
NOESY		sp1	286.9
mix	0.600	wp1	3556.1
PRESATURATION		rfl	3497.6
satmode	nnnn	rfp	3482.8
satpwr	0	rfl1	3636.7
satdly	0	rfp1	3618.8
satfrq	0	PLOT	
DECOUPLER		wc	155.0
dn	C13	sc	10.0
dm	nnn	wc2	155.0
		sc2	0
		vs	60
		th	2
		ai ph	

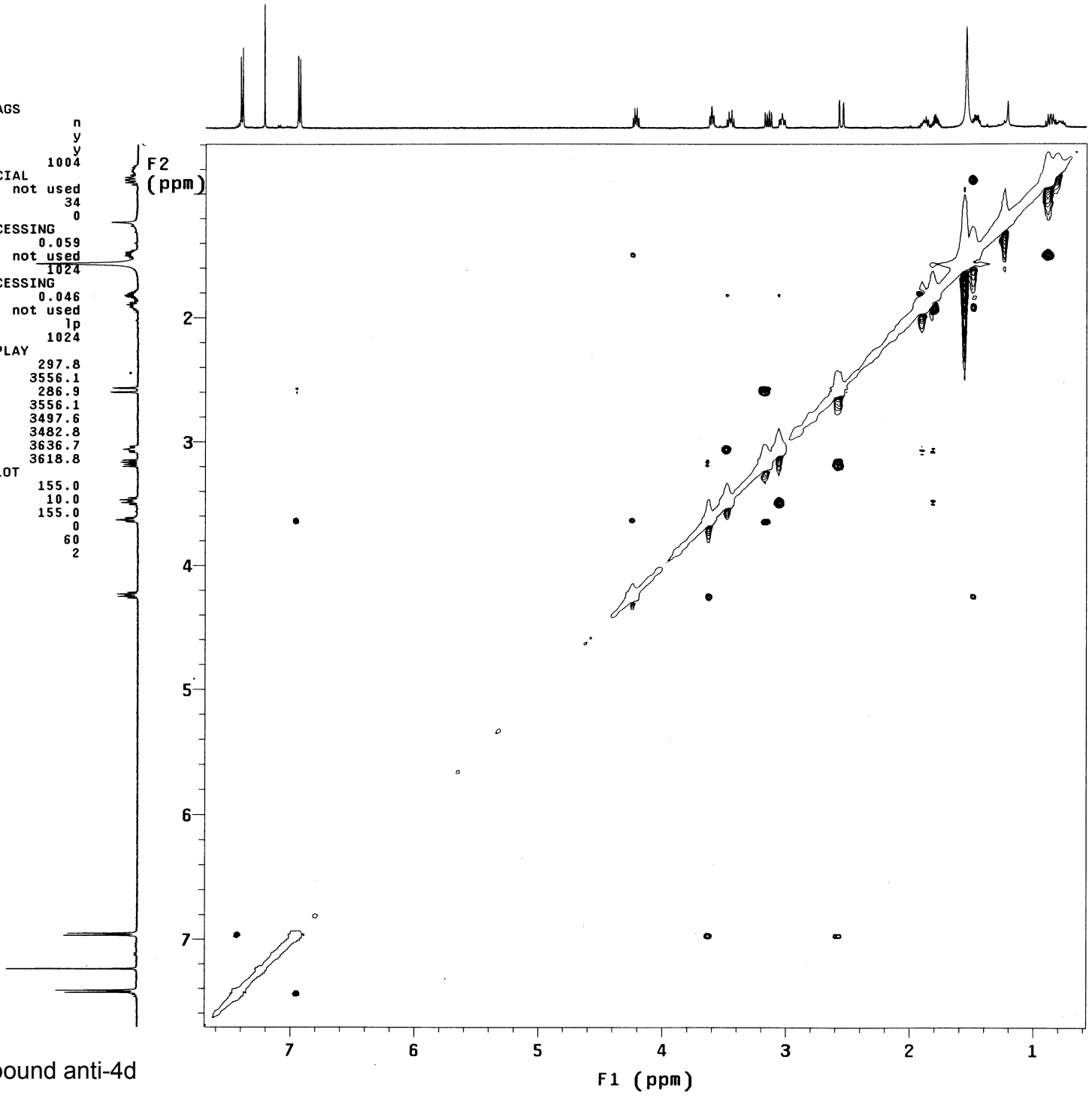
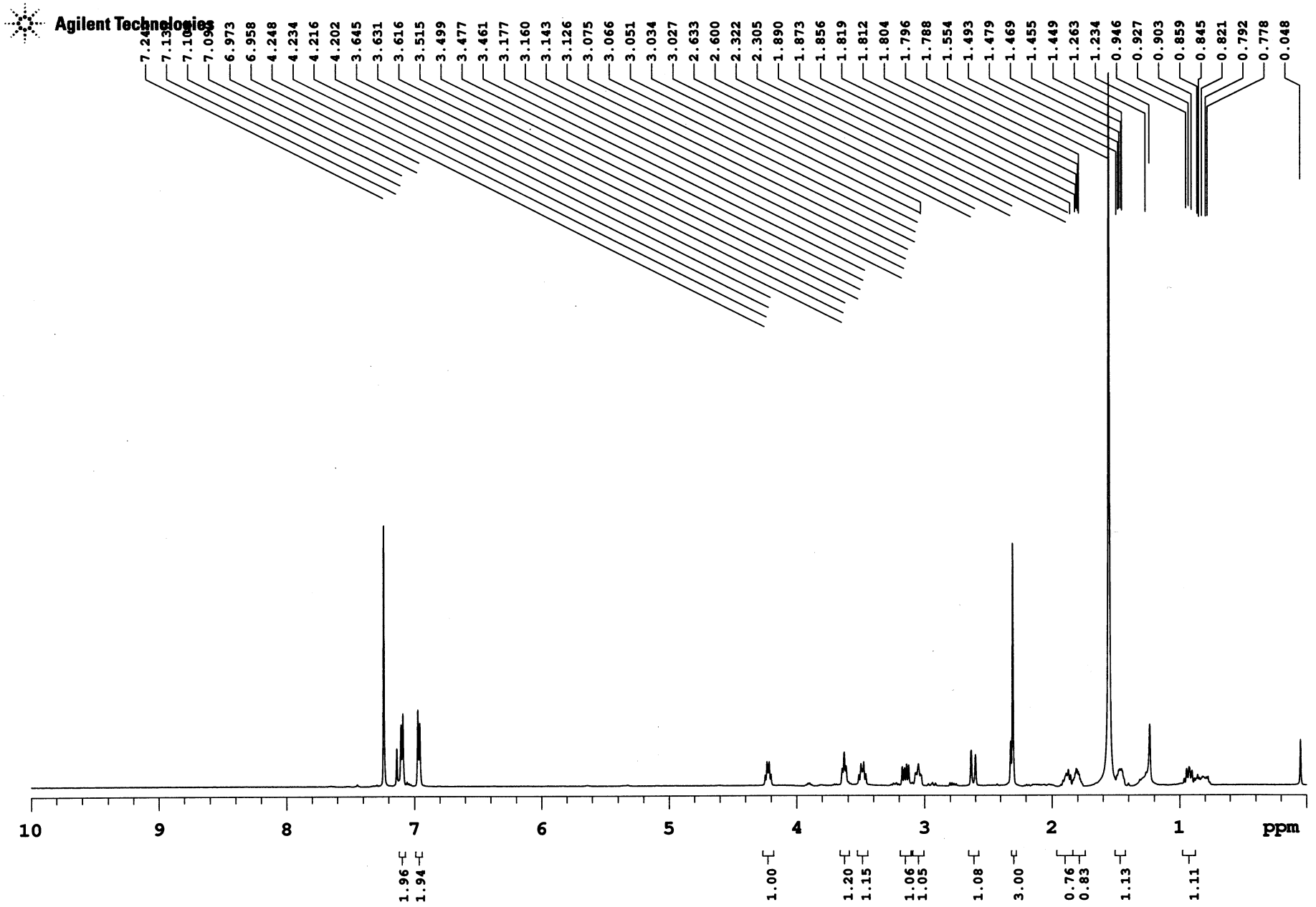


Fig S202. NOESY of compound anti-4d

LCH-02-391

Sample Name	LCH-02-391	Pulse sequence	PROTON	Temperature	25	Study owner	vnmr2
Date collected	2015-10-30	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-391/PROTON_14.fid

Plot date 2015-11-02

Fig S203. 1H NMR (CDCl3, 500 MHz) of compound anti-4e

LCH-02-391

Sample Name **LCH-02-391** Pulse sequence **s2pul** Temperature **130** Study owner **vnmr2**
Date collected **2015-04-03** Solvent **cdcl3** Spectrometer **---** Operator **vnmr2**

Agilent Technologies

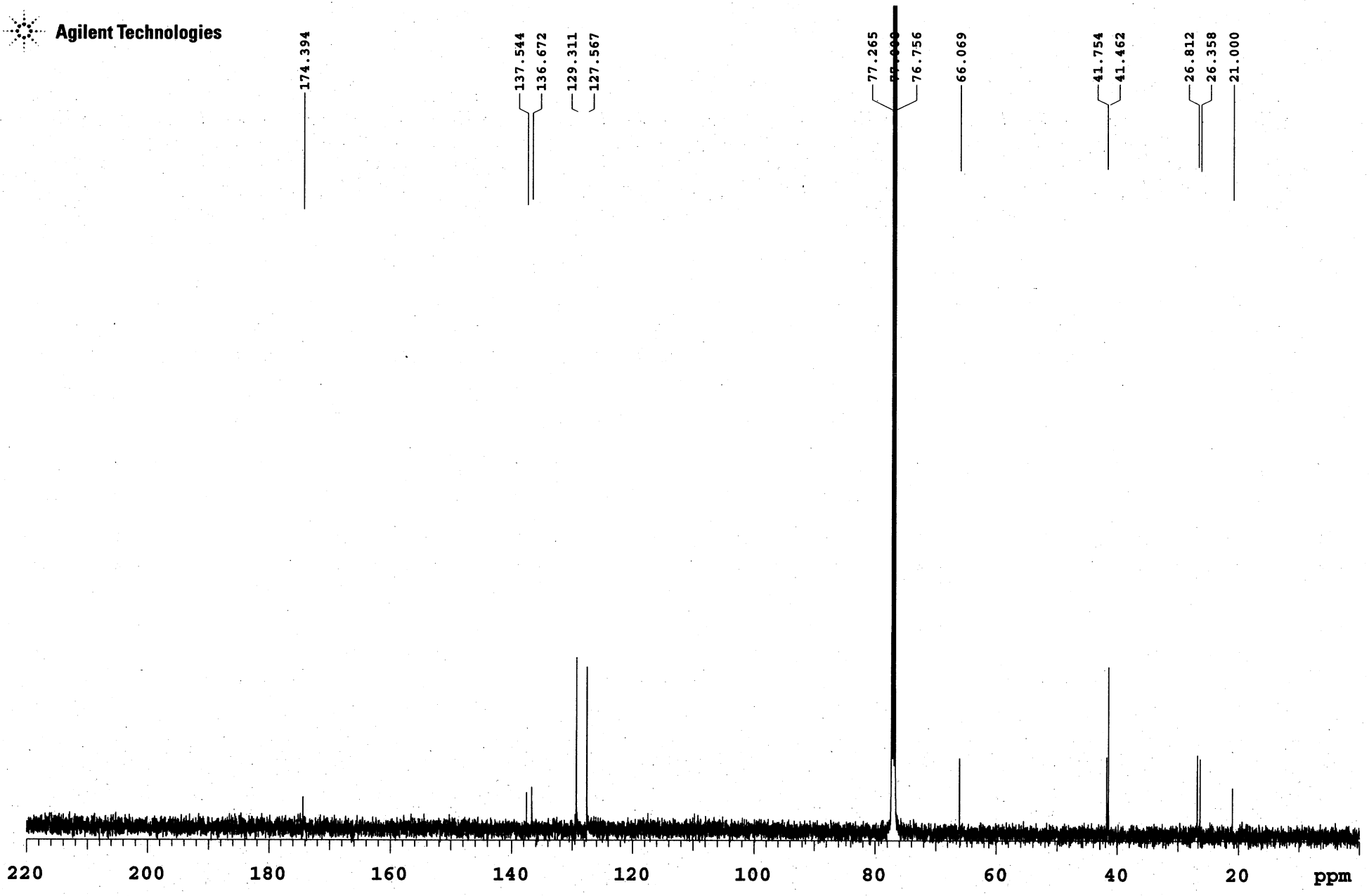


Fig S204. 13C NMR (CDCl3, 125 MHz) of compound anti-4e

LCH-02-391

Sample Name **LCH-02-391**	Pulse sequence **DEPT**	Temperature **130**	Study owner **vnmr2**
Date collected **2015-04-03**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

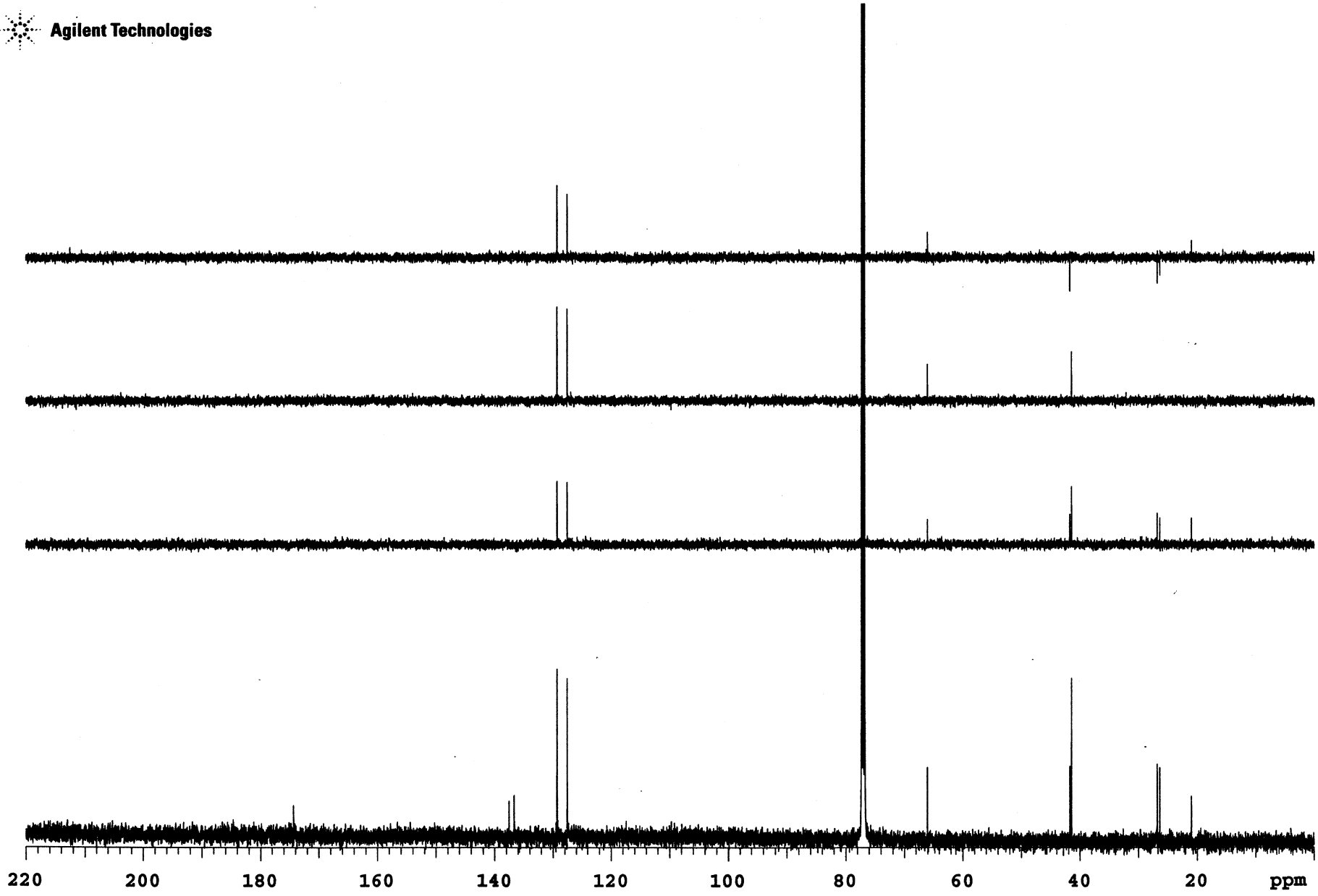


Fig S205. DEPT of compound anti-4e

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-391/DEPT.fid

Plot date 2015-06-18

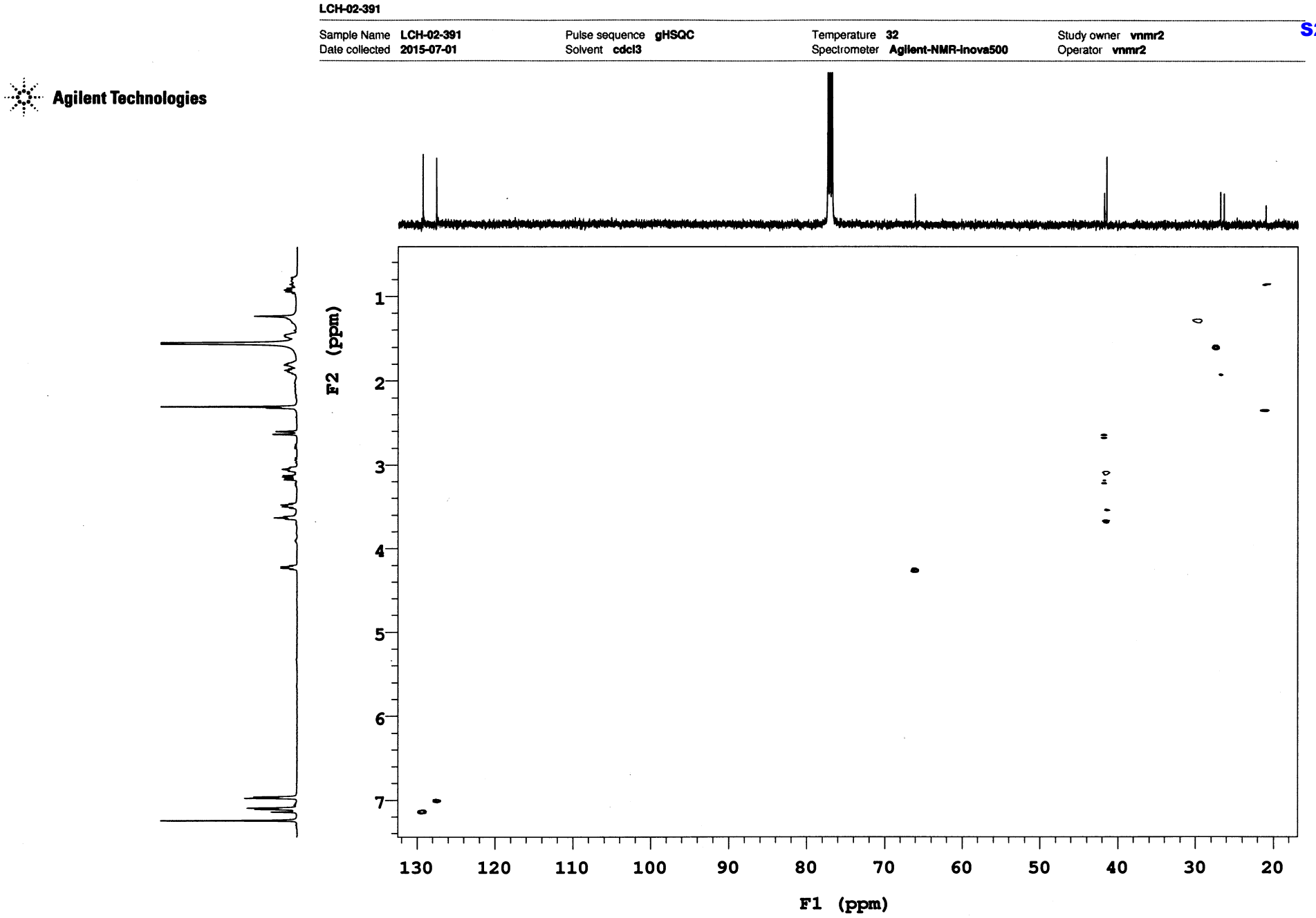


Fig S206. HSQC of compound anti-4e

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-391/gHSQC_03.fid

Plot date 2015-11-02

LCH-02-391

Sample Name **LCH-02-391**	Pulse sequence **gCOSY**	Temperature **130**	Study owner **vnmr2**
Date collected **2015-04-03**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

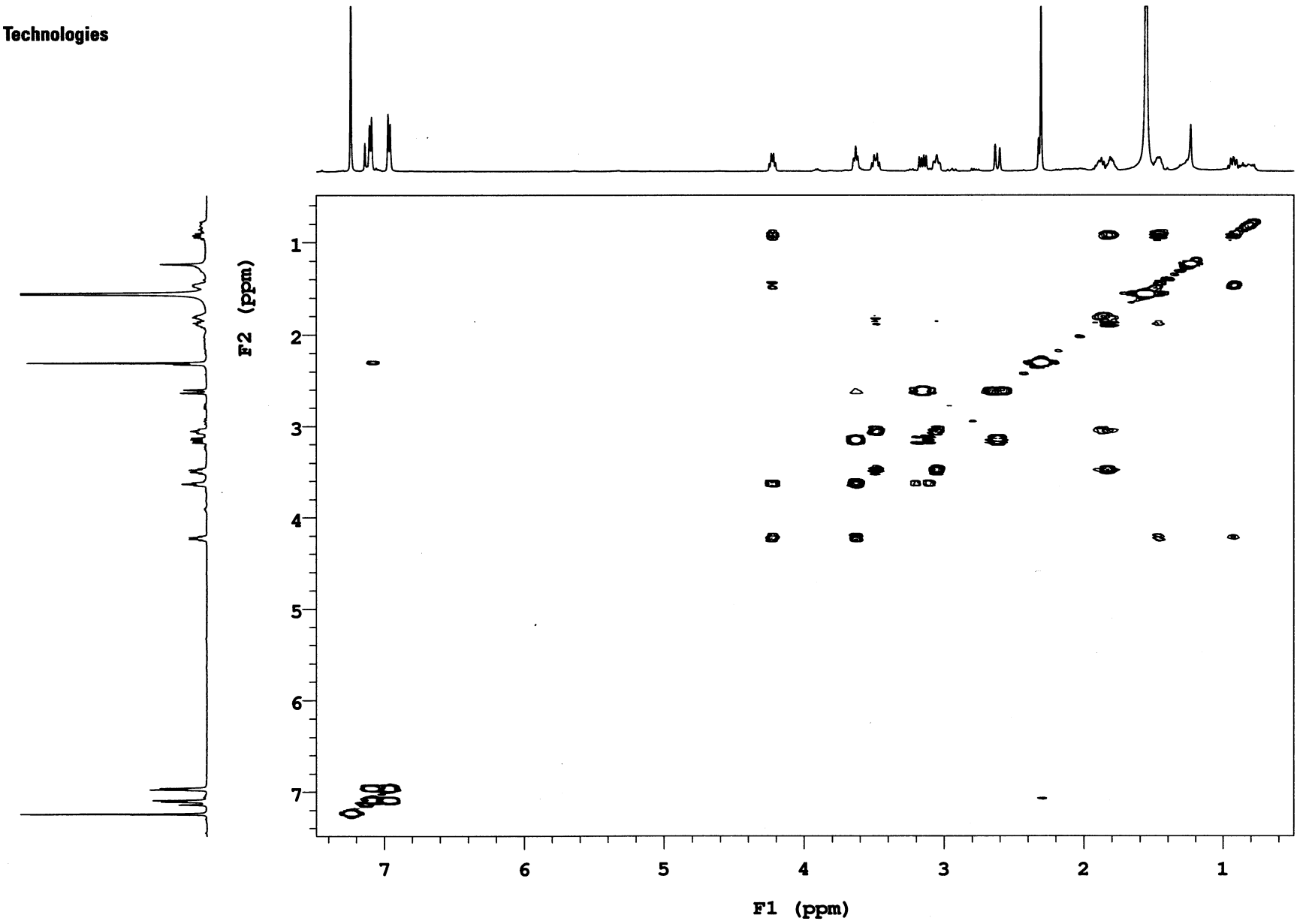


Fig S207. COSY of compound anti-4e

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-391/COSY.fid

Plot date 2015-11-02

LCH-02-391

Sample Name **LCH-02-391**	Pulse sequence **NOESY**	Temperature **130**	Study owner **vnmr2**
Date collected **2015-04-03**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

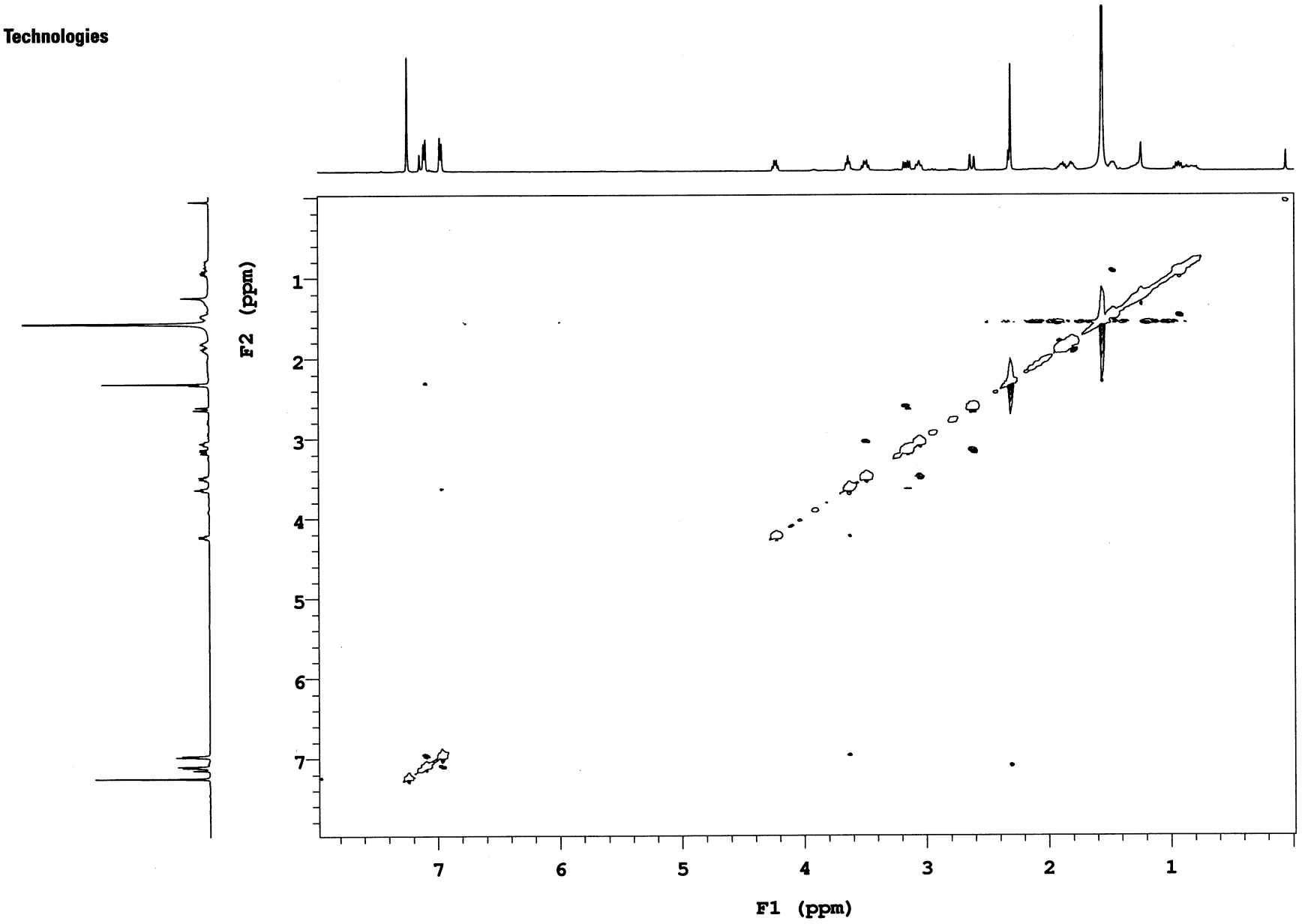


Fig S208. NOESY of compound anti-4e

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-391/NOESY.fid

Plot date 2015-11-02

LCH-02-406

Sample Name	**LCH-02-406**	Pulse sequence	**PROTON**	Temperature	**26**	Study owner	**vnmr2**
Date collected	**2015-06-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

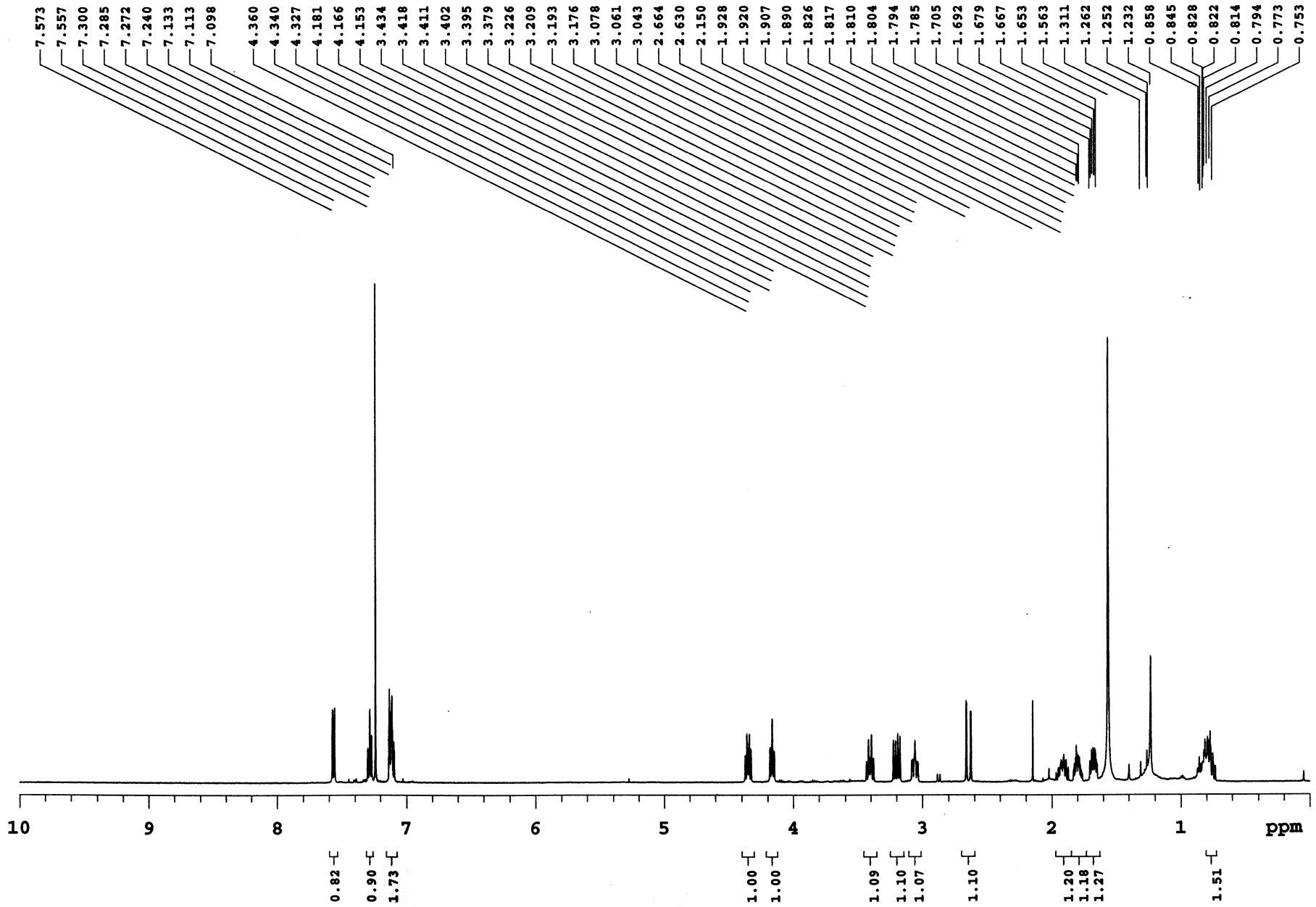


Fig S209. 1H NMR (CDCl3, 500 MHz) of compound anti-4f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-406/PROTON_06.fid

Plot date 2015-09-02

LCH-02-406

Sample Name **LCH-02-406** | Pulse sequence **CARBON** | Temperature **26** | Study owner **vnmr2**
Date collected **2015-06-23** | Solvent **cdcl3** | Spectrometer **Agilent-NMR-inova500** | Operator **vnmr2**

Agilent Technologies

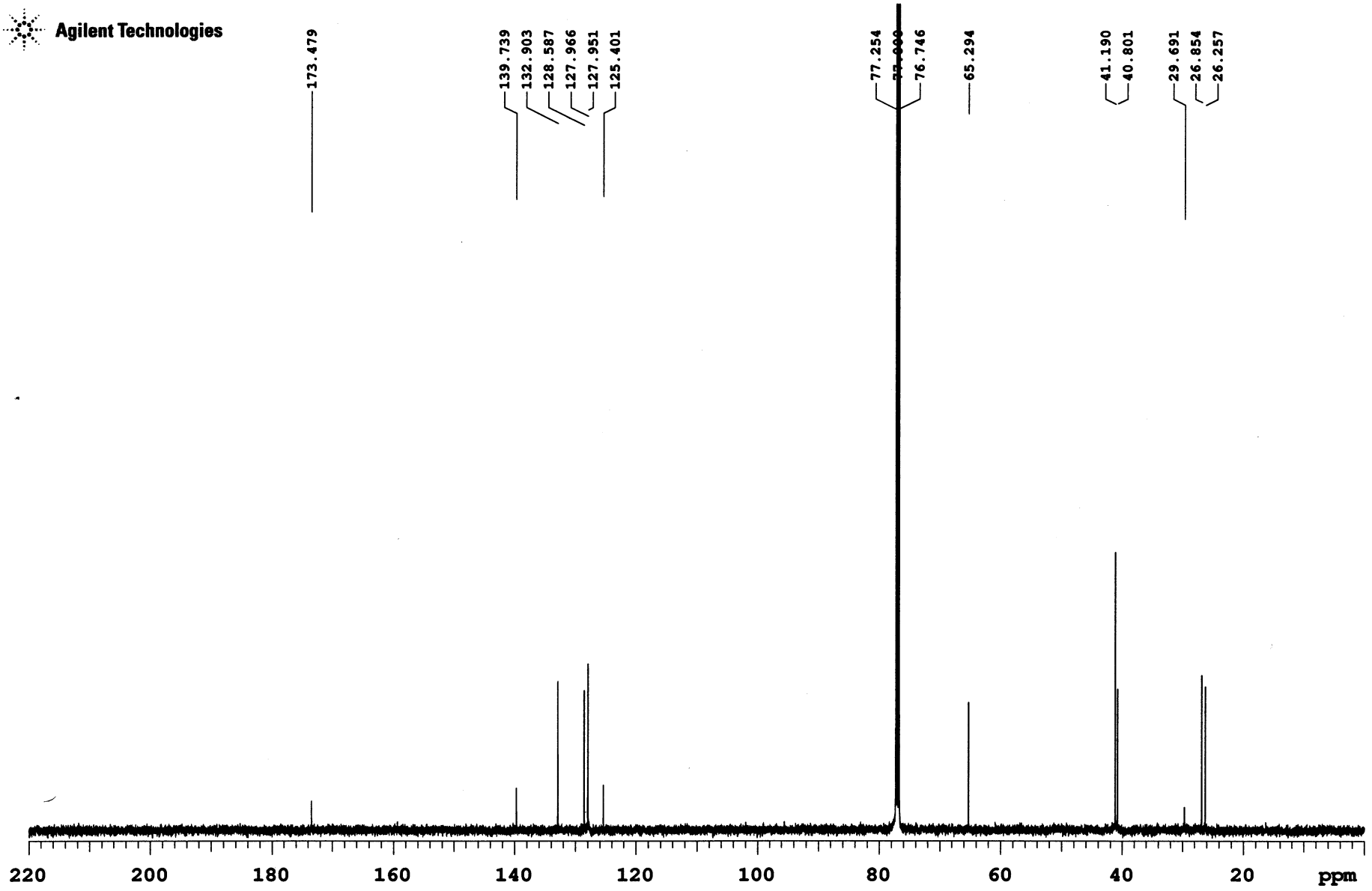


Fig S210. 13C NMR (CDCl3, 125 MHz) of compound anti-4f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-406/CARBON_02

Plot date 2015-06-24

LCH-02-406

Sample Name **LCH-02-406**	Pulse sequence **DEPT**	Temperature **26**	Study owner **vnmr2**
Date collected **2015-06-23**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

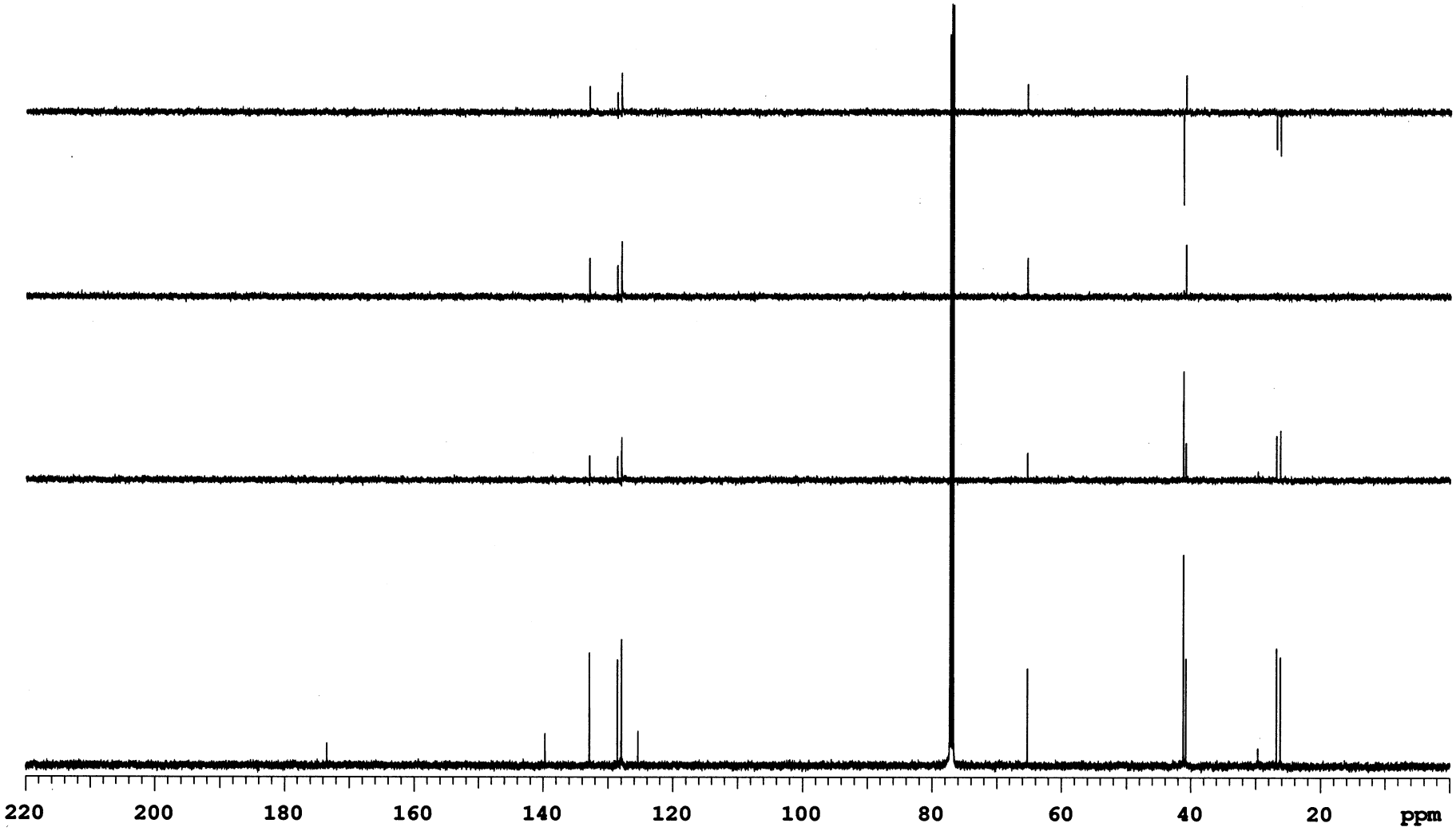


Fig S211. DEPT of compound anti-4f

LCH-02-406

Sample Name	LCH-02-406	Pulse sequence	gHSQC	Temperature	25	Study owner	vnmr2
Date collected	2015-05-19	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

Agilent Technologies

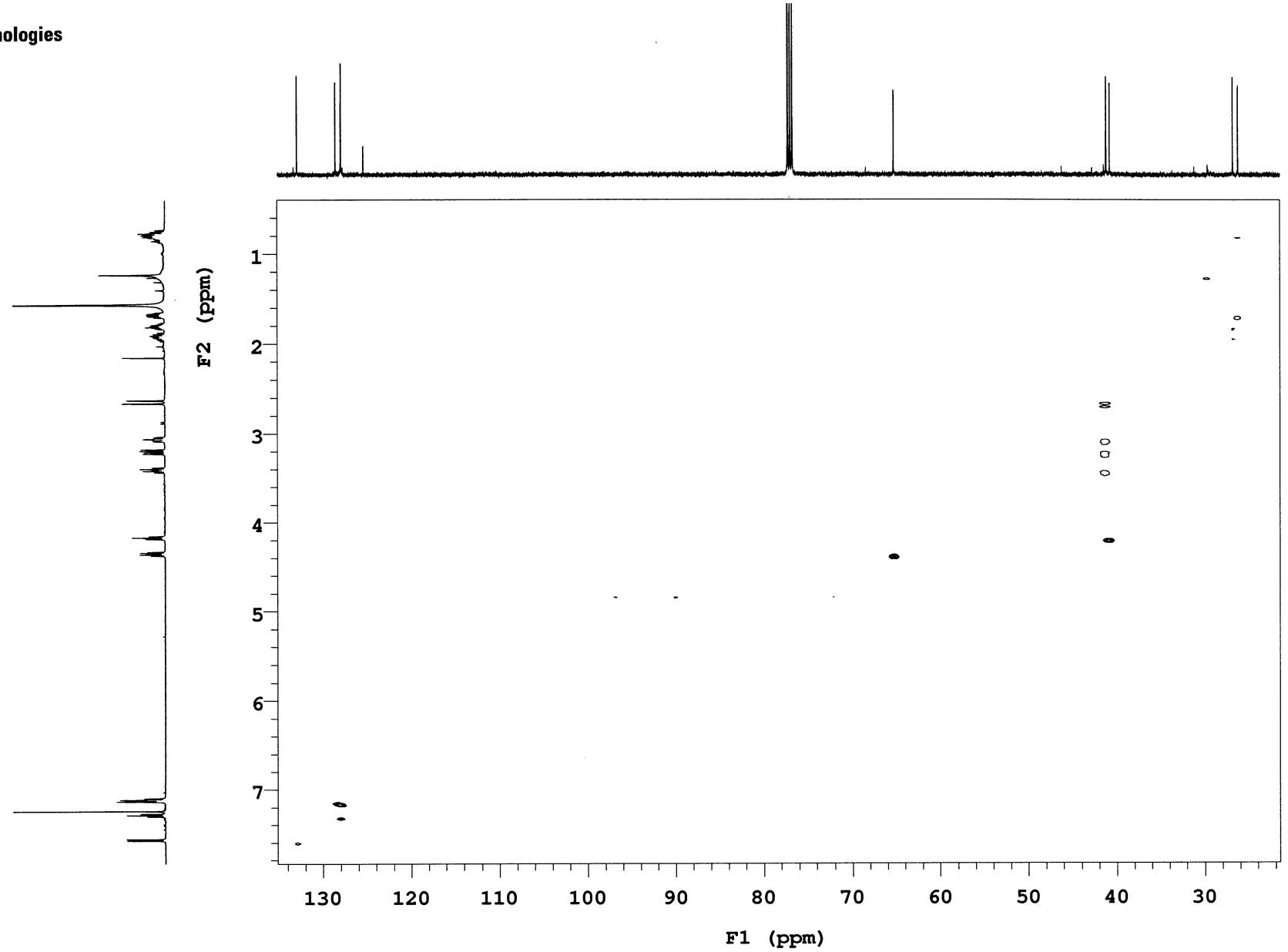


Fig S212. HSQC of compound anti-4f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-406/gHSQC_01

Plot date 2015-10-24

LCH-02-406

Sample Name	LCH-02-406	Pulse sequence	gCOSY	Temperature	26	Study owner	vnmr2
Date collected	2015-06-24	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

Agilent Technologies

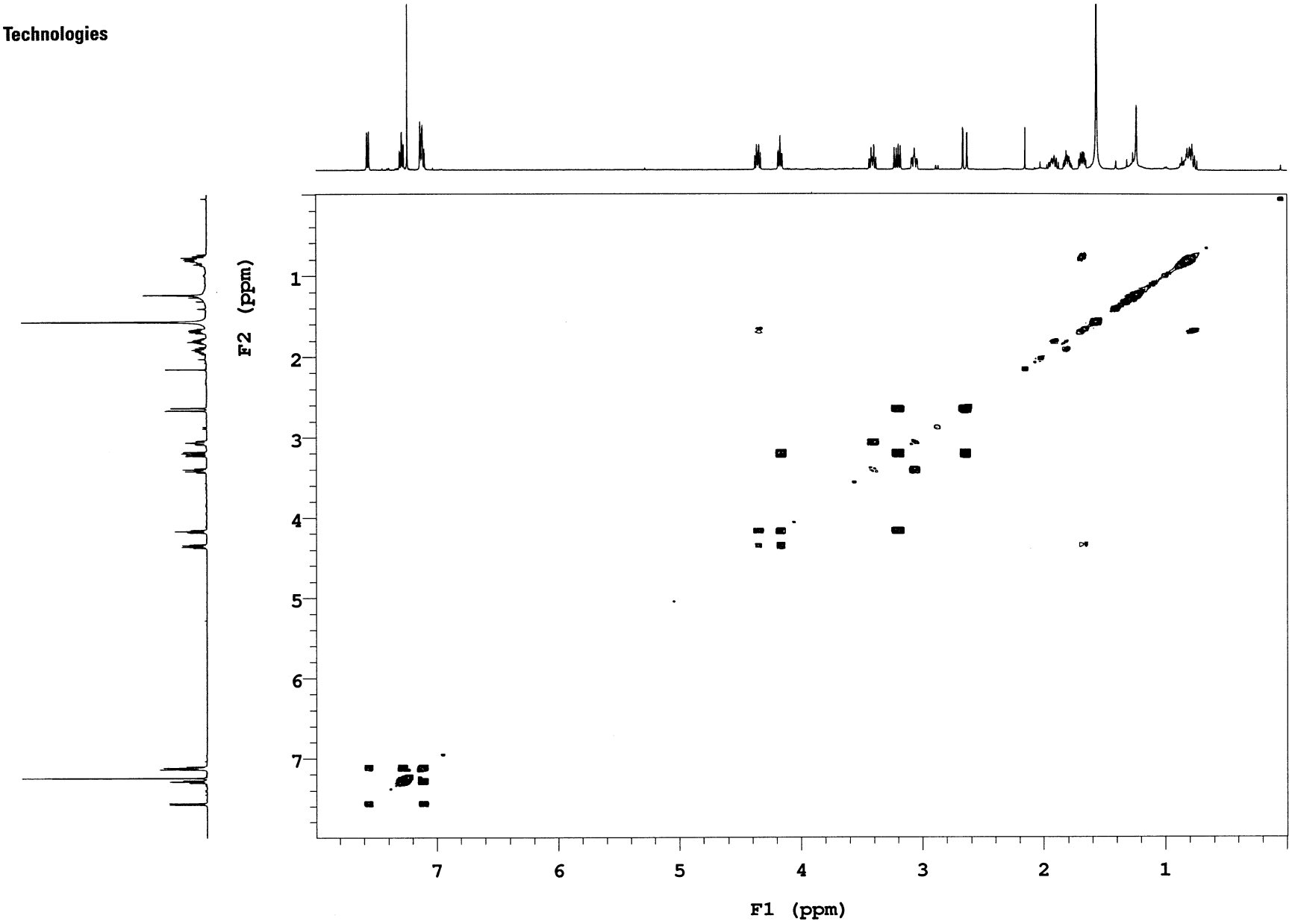


Fig S213. COSY of compound anti-4f

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-406/gCOSY_02

Plot date 2015-10-24

LCH-02-406

Sample Name	**LCH-02-406**	Pulse sequence	**NOESY**	Temperature	**26**	Study owner	**vnmr2**
Date collected	**2015-06-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

Agilent Technologies

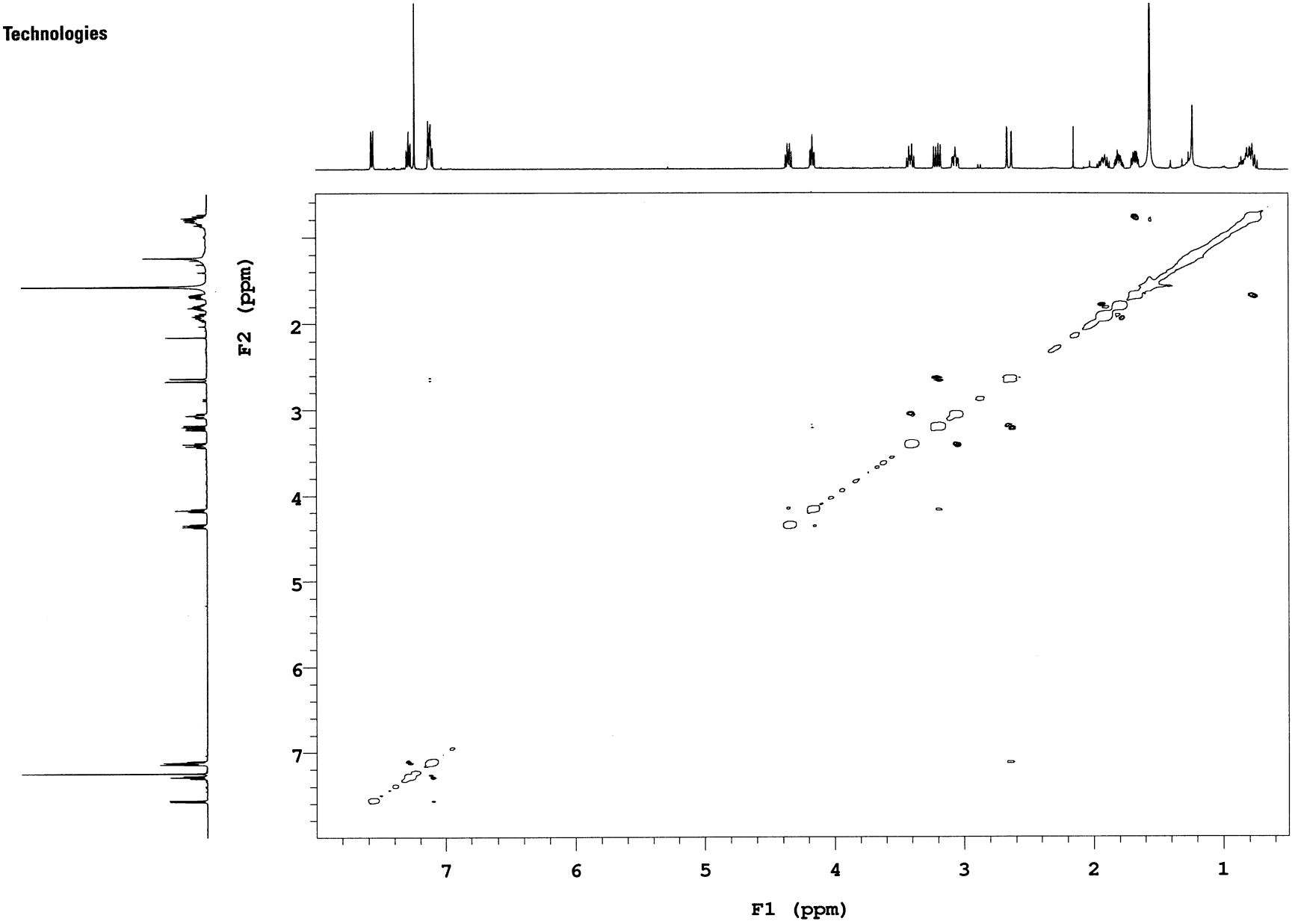


Fig S214. NOESY of compound anti-4f

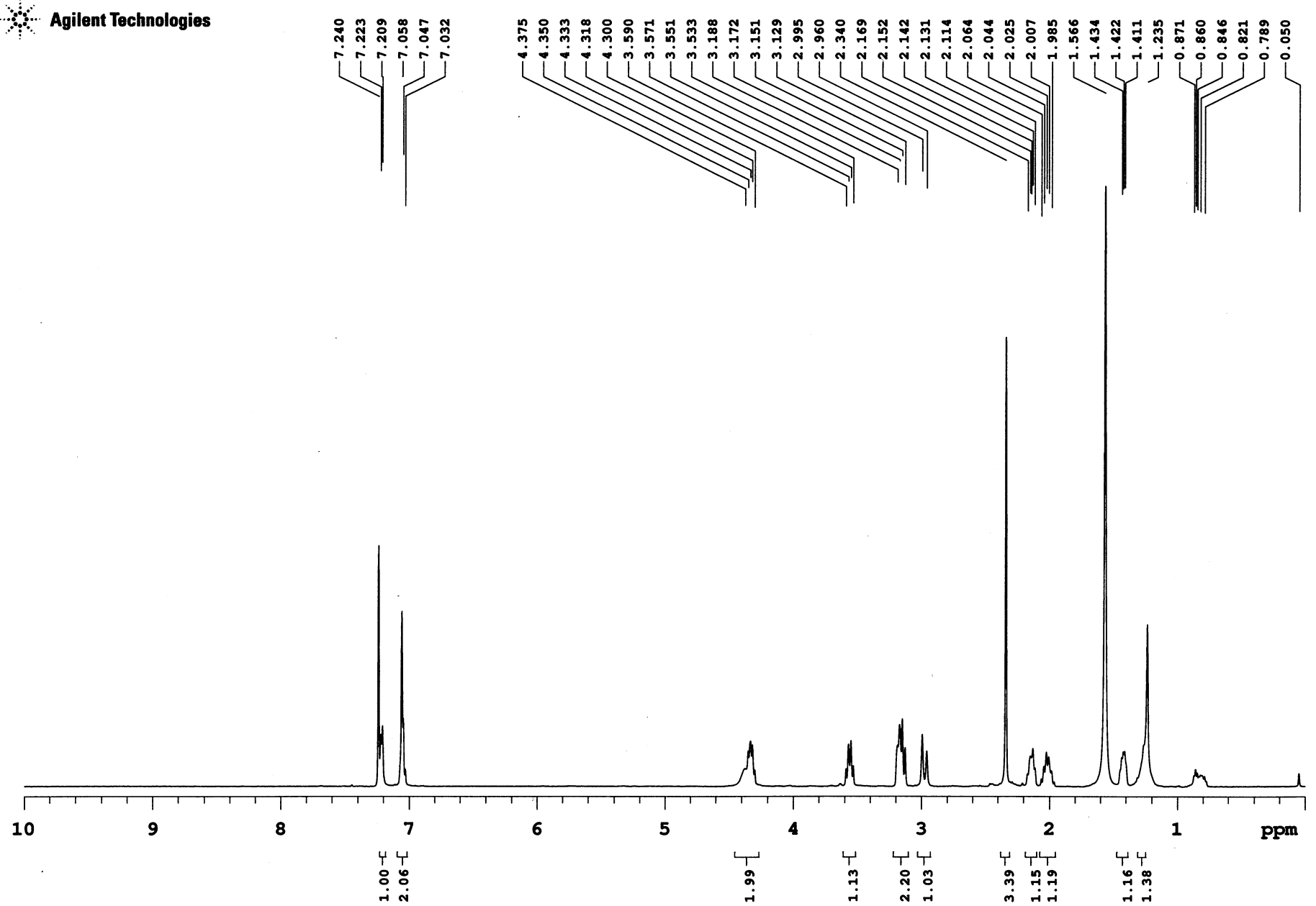
Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-406/NOESY_02

Plot date 2015-10-24

LCH-02-379

Sample Name **LCH-02-379**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-10-30**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-379/PROTON_10.fid

Fig S215. 1H NMR (CDCl3, 500 MHz) of compound anti-4g

Plot date 2015-11-02

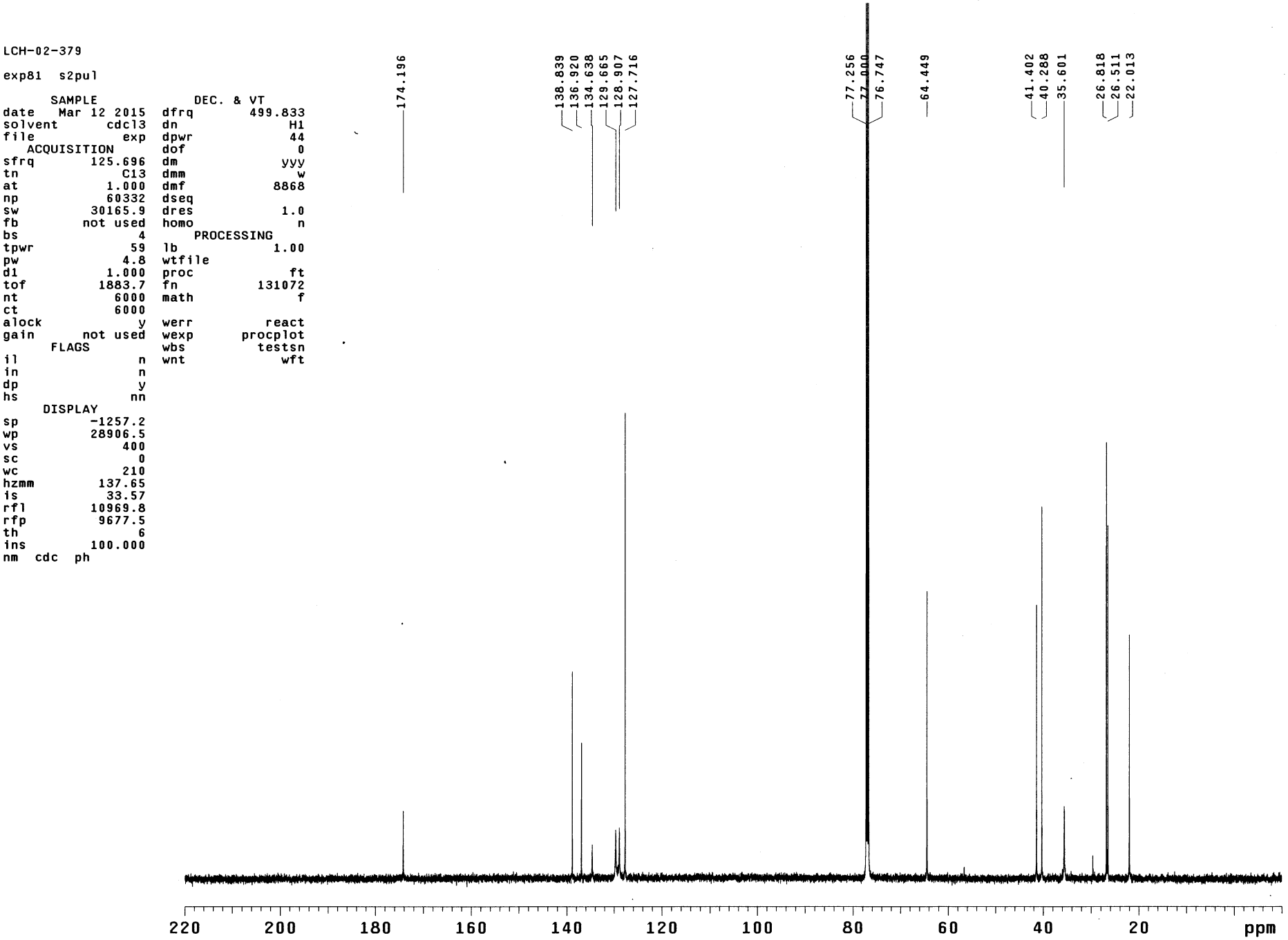
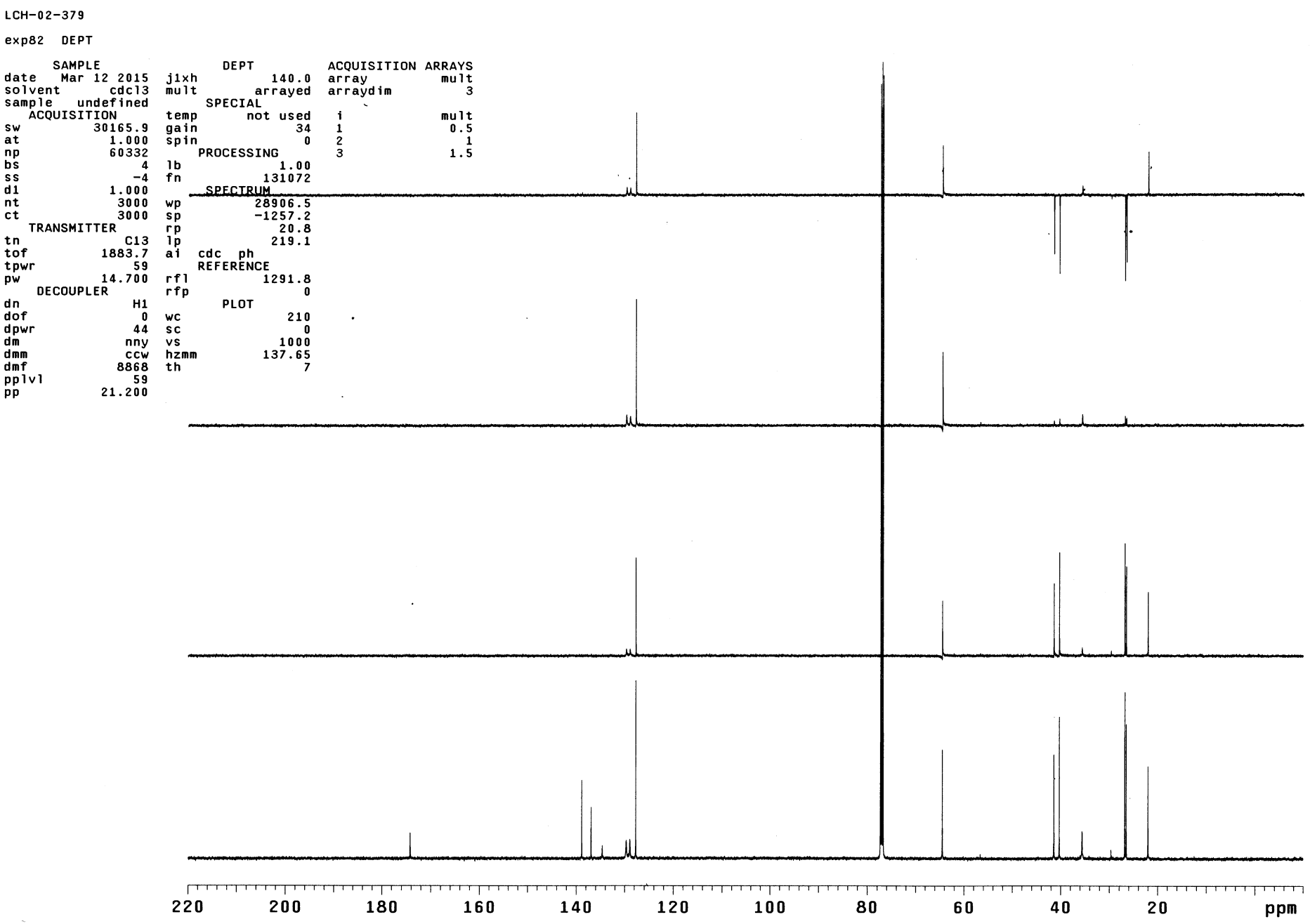


Fig S216. 13C NMR (CDCl3, 125 MHz) of compound anti-4g

Fig S217. DEPT of compound anti-4g



LCH-02-379

Sample Name **LCH-02-379**	Pulse sequence **gHSQC**	Temperature **130**	Study owner **vnmr2**
Date collected **2015-03-12**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

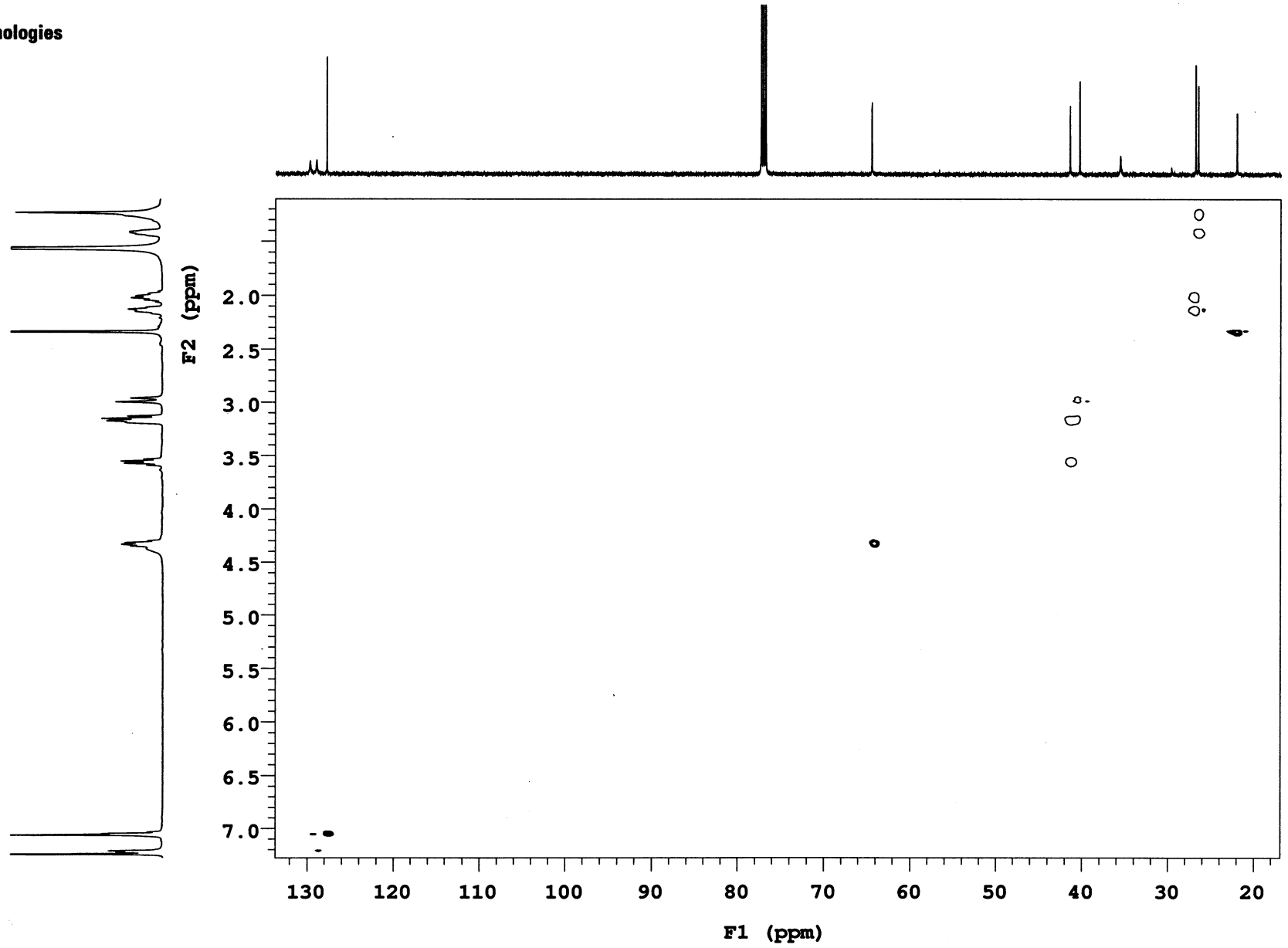


Fig S218. HSQC of compound anti-4g

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-379/new/HSQC.fid

Plot date 2015-11-02

LCH-02-379

Sample Name **LCH-02-379**	Pulse sequence **gCOSY**	Temperature **130**	Study owner **vnmr2**
Date collected **2015-03-12**	Solvent **cdcl3**	Spectrometer **---**	Operator **vnmr2**

Agilent Technologies

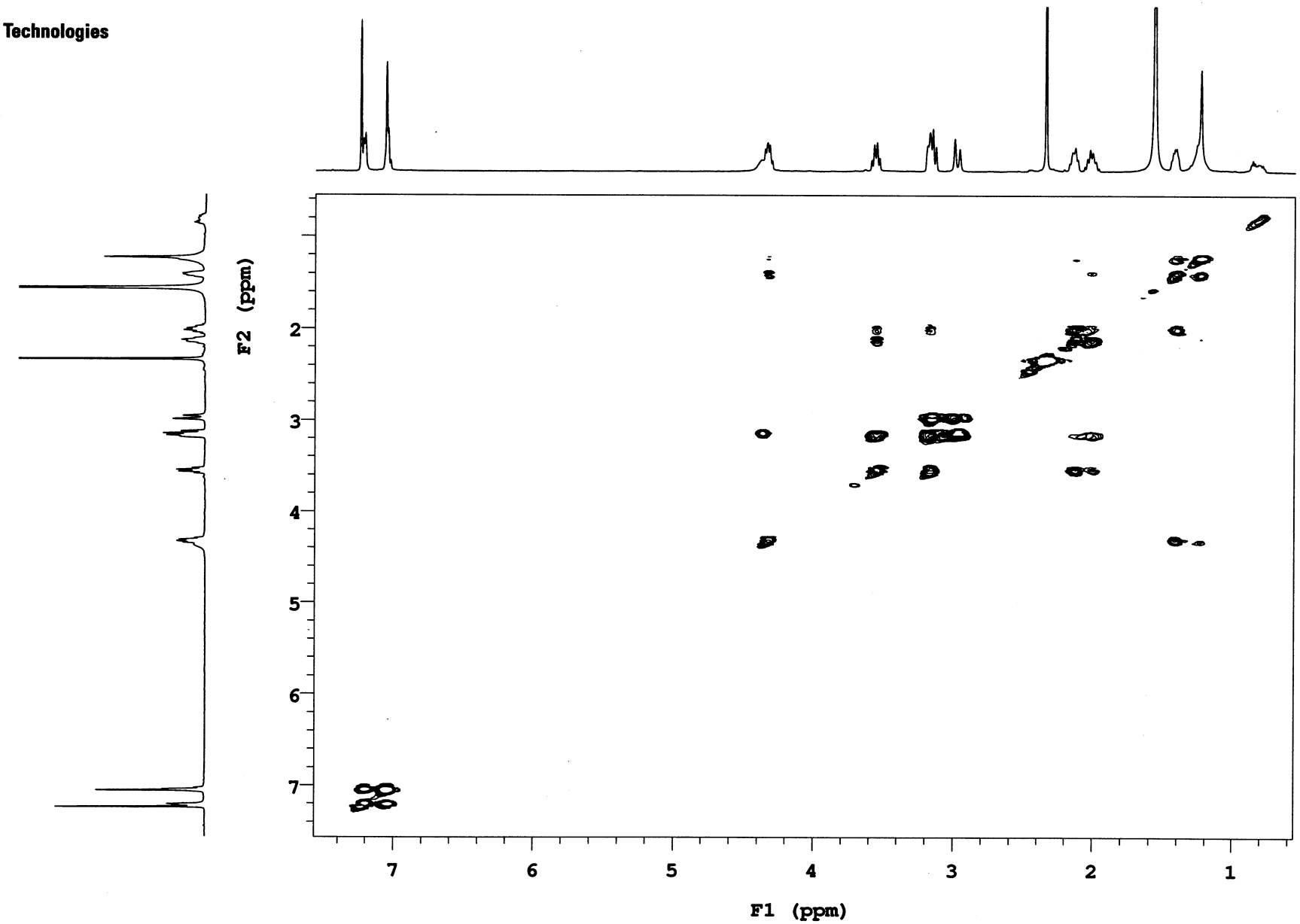


Fig S219. COSY of compound anti-4g

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-379/new/COSY.fid

Plot date 2015-11-02

LCH-02-379

Sample Name **LCH-02-379**
Date collected **2015-03-12**

Pulse sequence **NOESY**
Solvent **cdcl3**

Temperature **130**
Spectrometer **---**

Study owner **vnmr2**
Operator **vnmr2**

Agilent Technologies

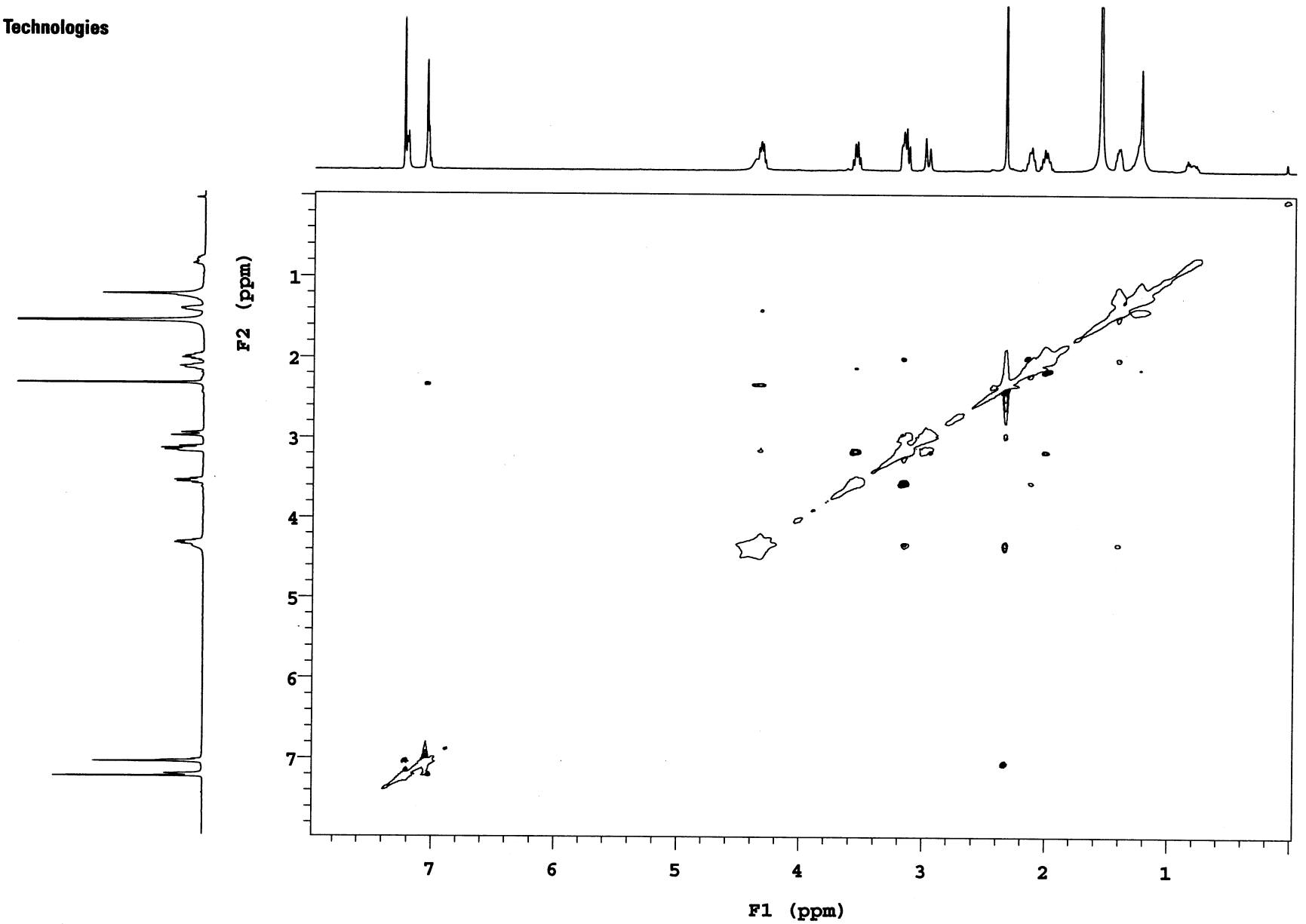


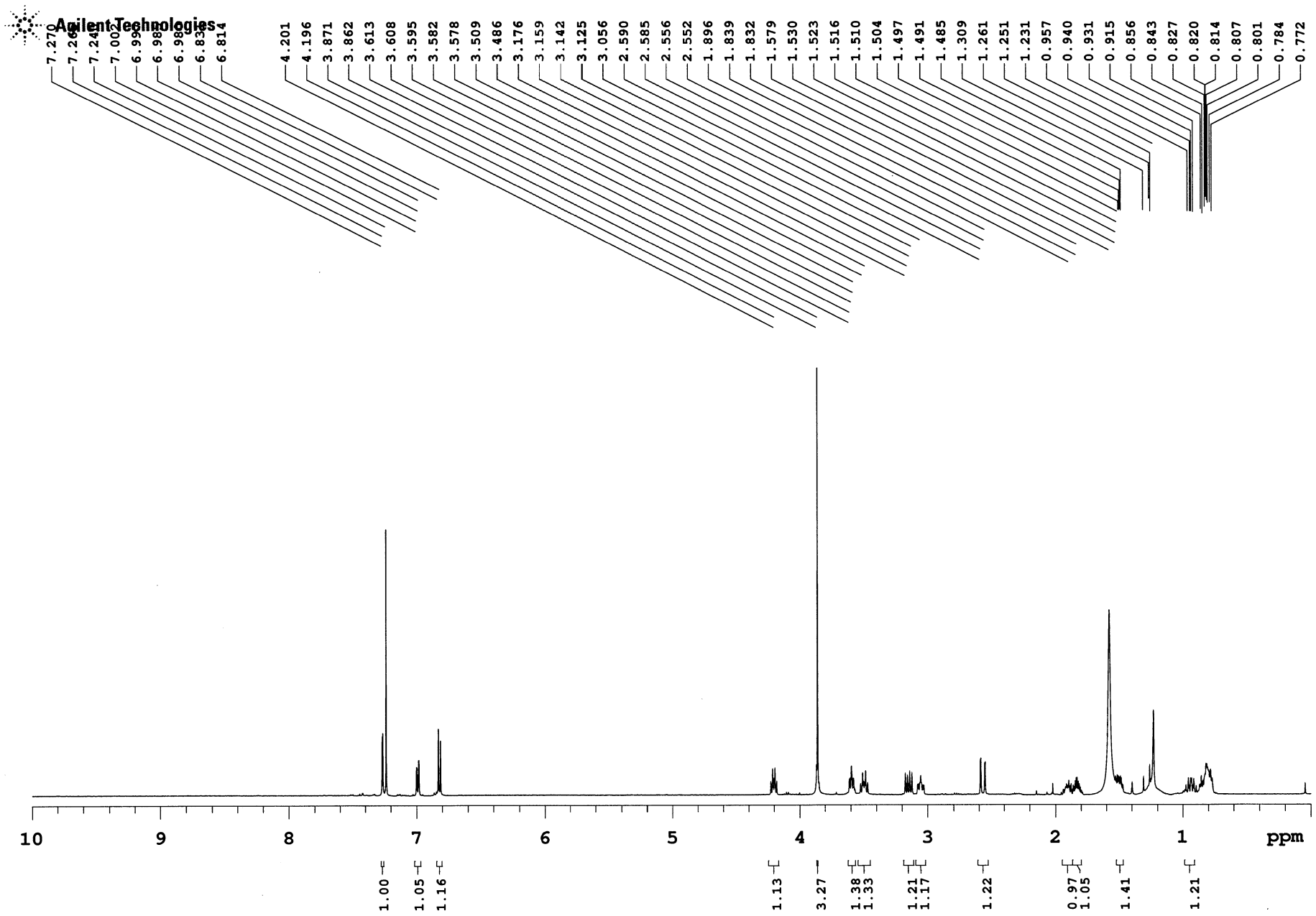
Fig S220. NOESY of compound anti-4g

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-379/new/NOESY.fid

Plot date 2015-11-02

LCH-02-399

Sample Name	LCH-02-399	Pulse sequence	PROTON	Temperature	25	Study owner	vnmr2
Date collected	2015-06-13	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-399/PROTON_09.fid

Fig S221. 1H NMR (CDCl3, 500 MHz) of compound anti-4h

Plot date 2015-10-24

LCH-02-399

Sample Name **LCH-02-399**
Date collected **2015-06-13**
Pulse sequence **CARBON**
Solvent **cdcl3**
Temperature **25**
Spectrometer **Agilent-NMR-inova500**
Study owner **vnmr2**
Operator **vnmr2**

Agilent Technologies

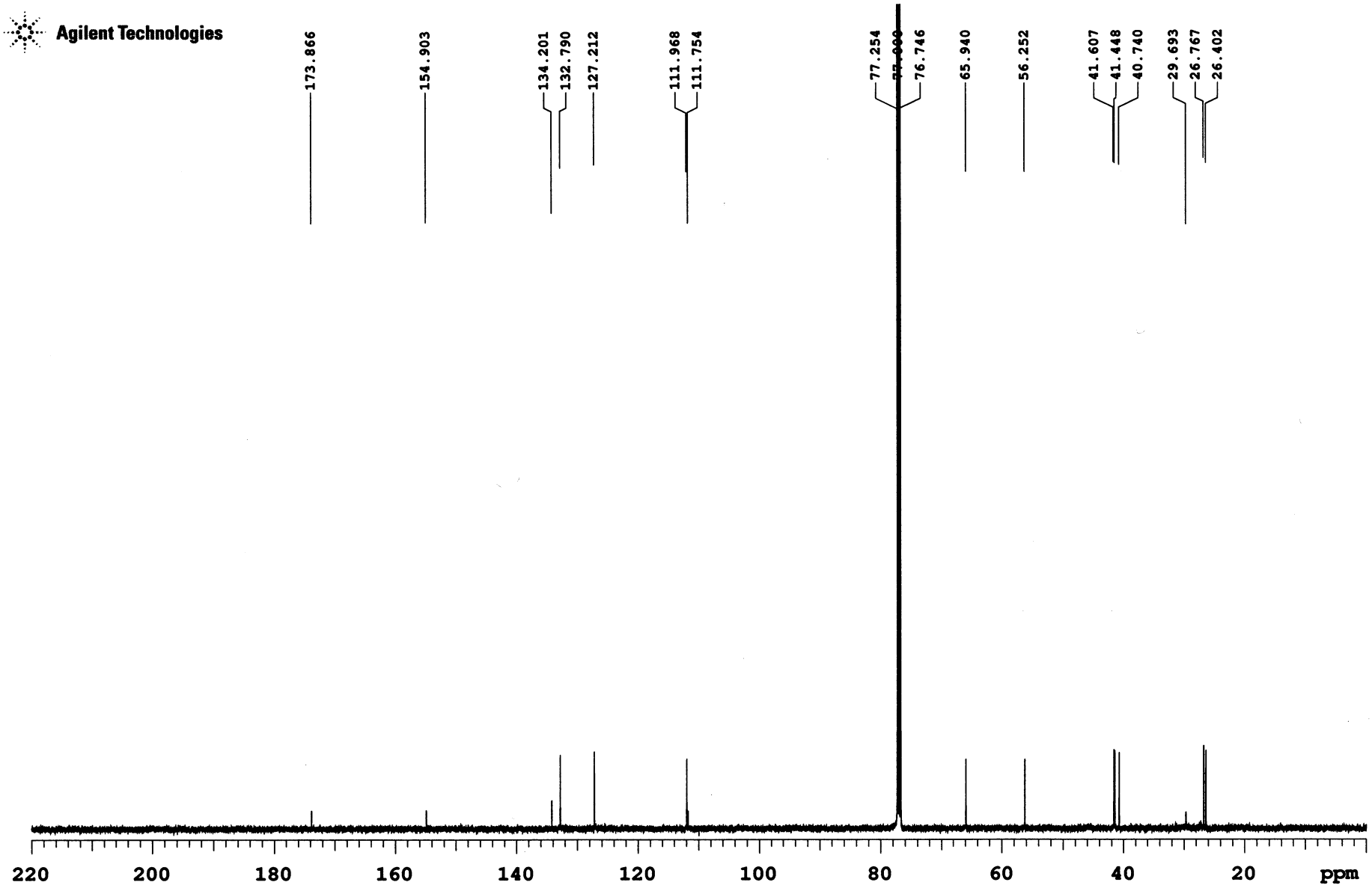


Fig S222. 13C NMR (CDCl3, 125 MHz) of compound anti-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-399/CARBON_02

Plot date 2015-06-15

Sample Name **LCH-02-399**	Pulse sequence **DEPT**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-06-13**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

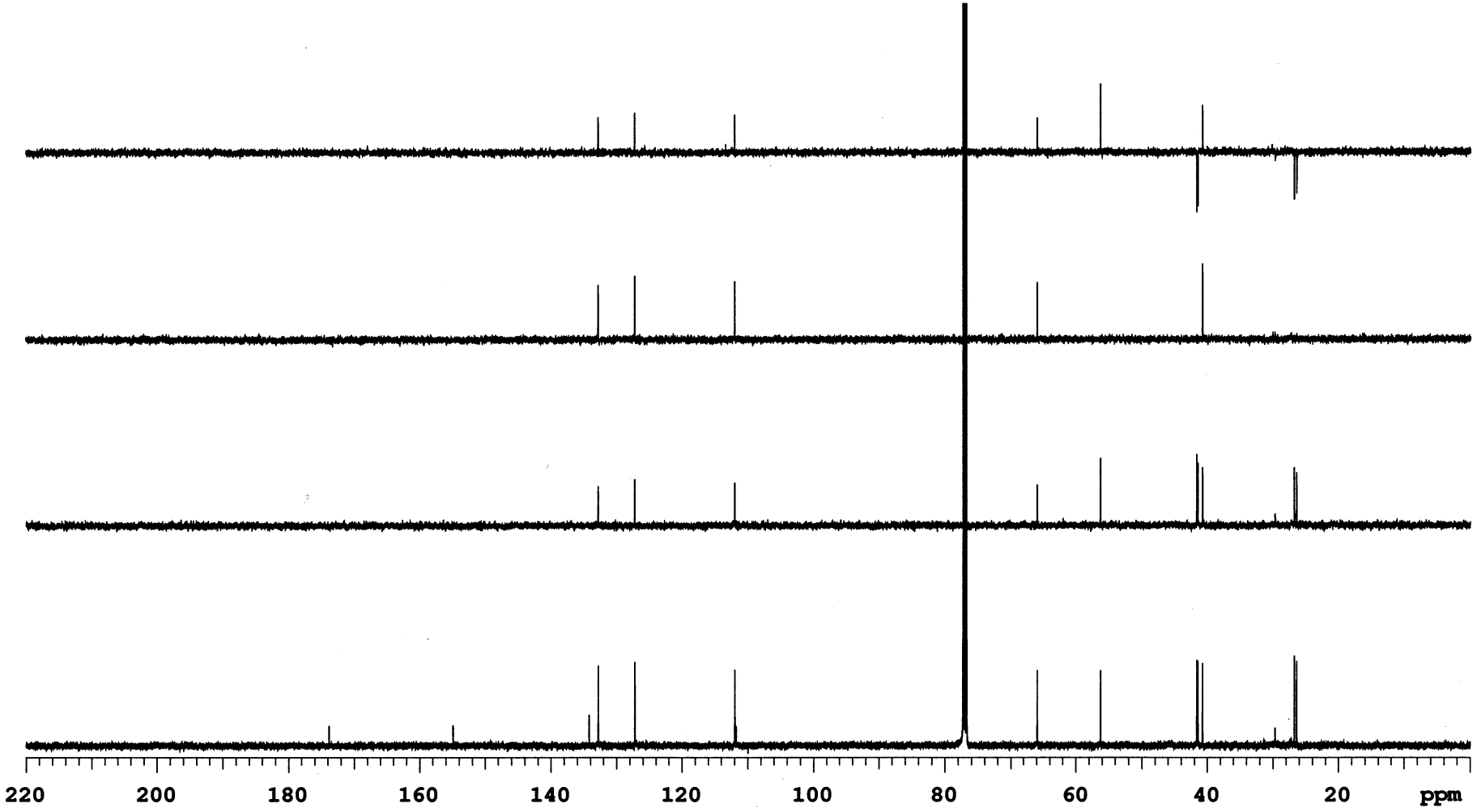


Fig S223. DEPT of compound anti-4h

LCH-02-399

Sample Name **LCH-02-399**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-06-03**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

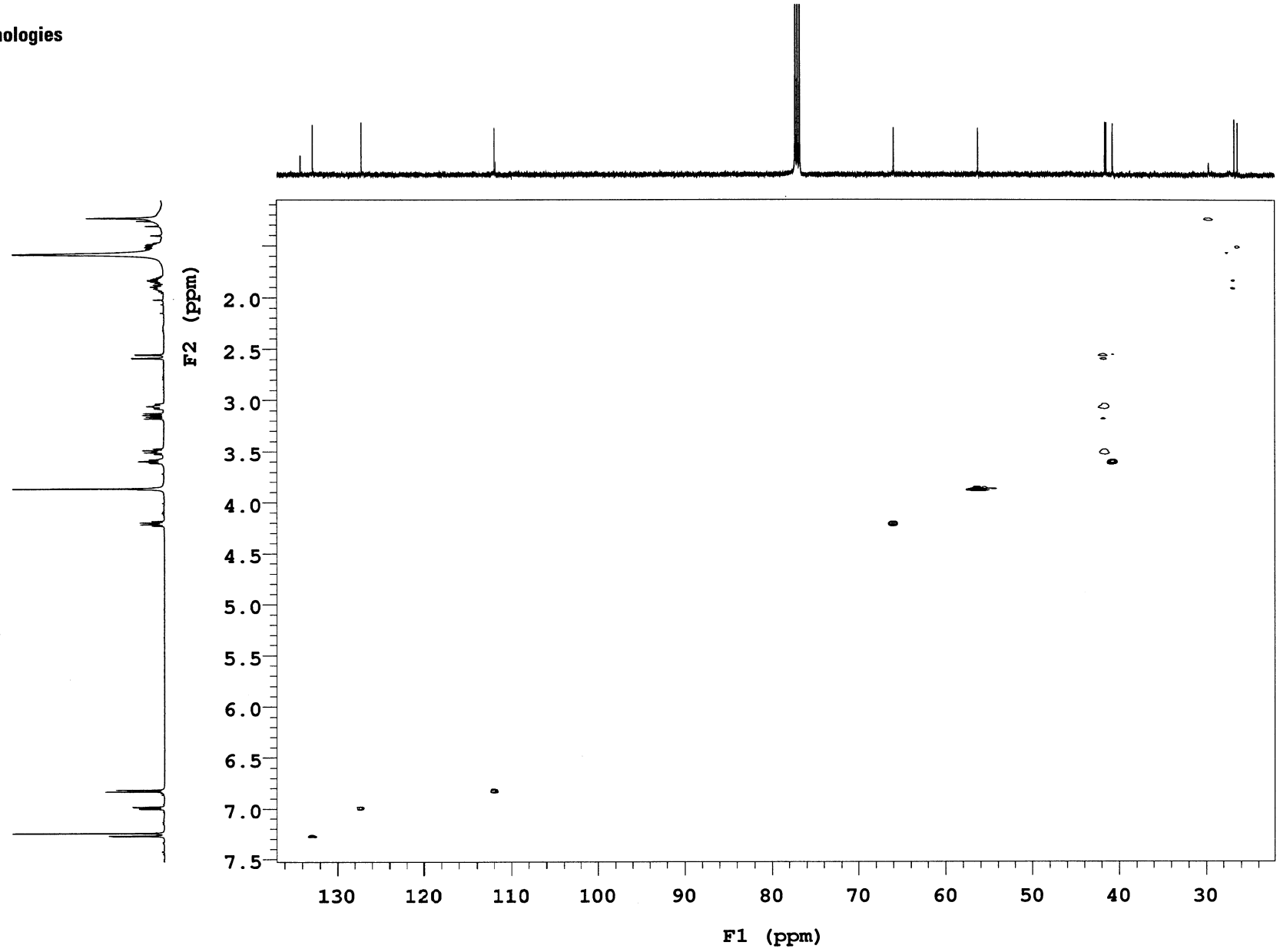


Fig S224. HSQC of compound anti-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-399/gHSQC_01.fid

Plot date 2015-10-29

LCH-02-399

Sample Name **LCH-02-399**	Pulse sequence **gCOSY**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-06-13**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

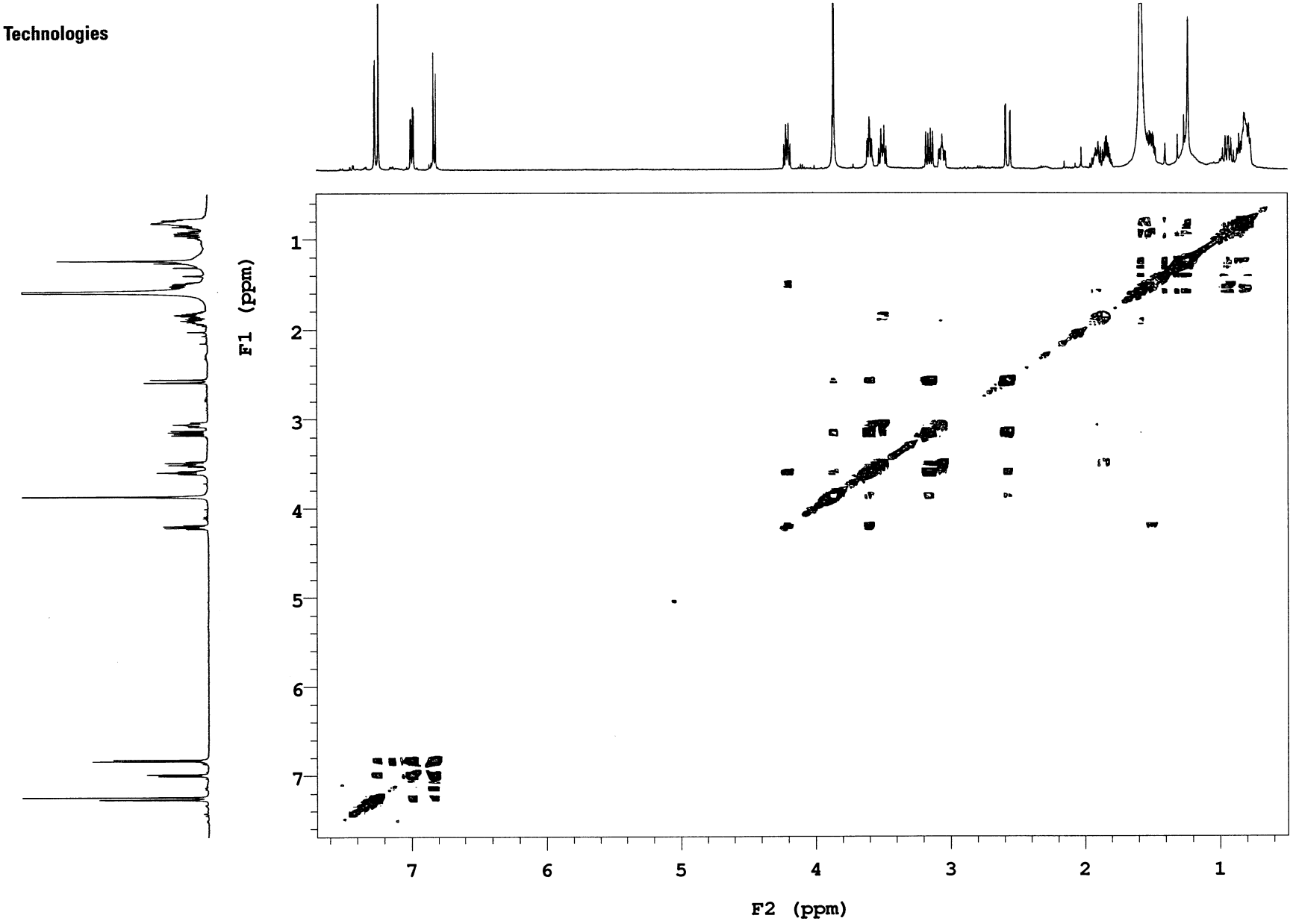


Fig S225. COSY of compound anti-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-399/gCOSY_02.fid

Plot date 2015-10-24

LCH-02-399

Sample Name **LCH-02-399**	Pulse sequence **NOESY**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-06-13**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

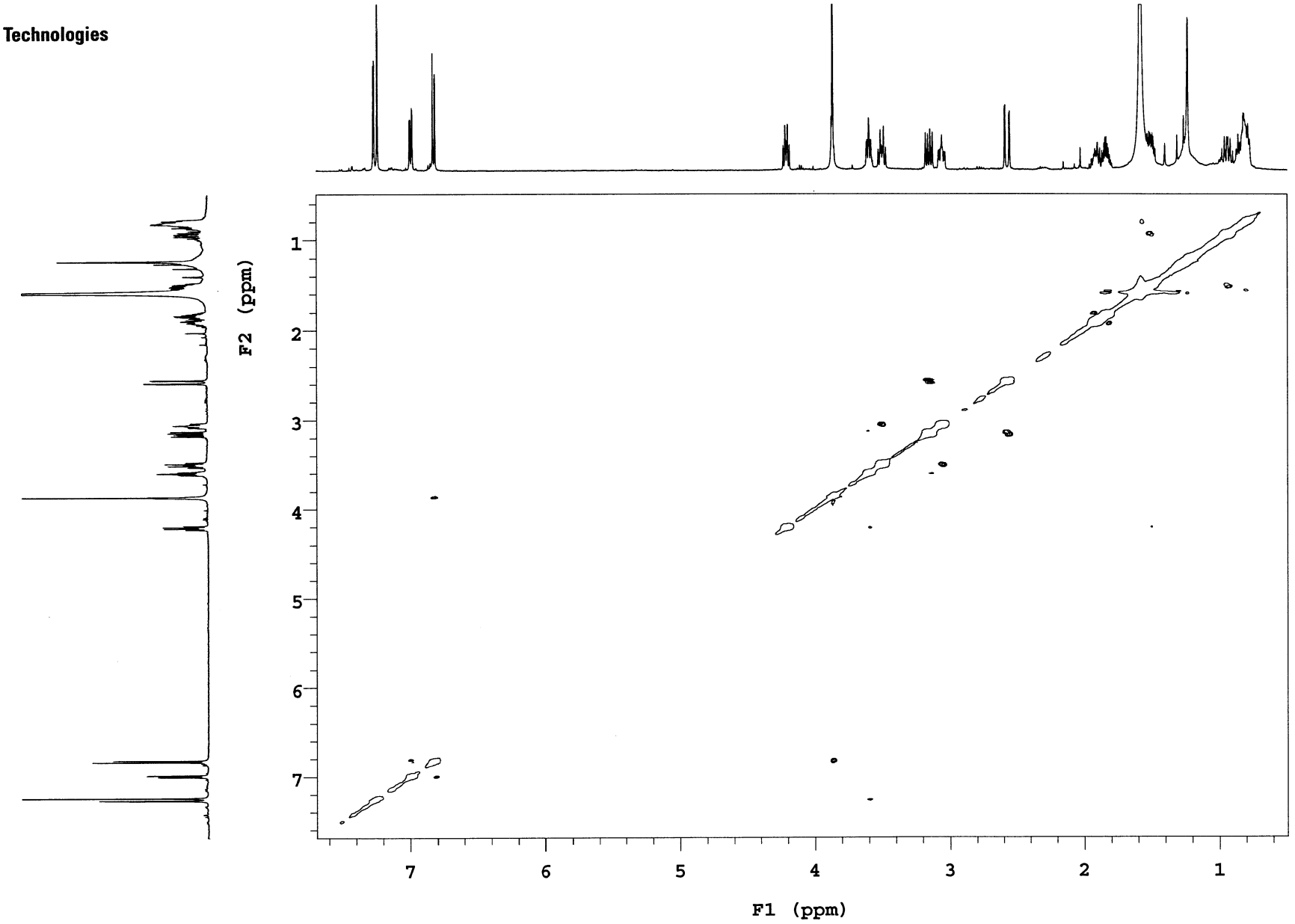


Fig S226. NOESY of compound anti-4h

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-399/NOESY_02.fid

Plot date 2015-10-24

LCH-02-407

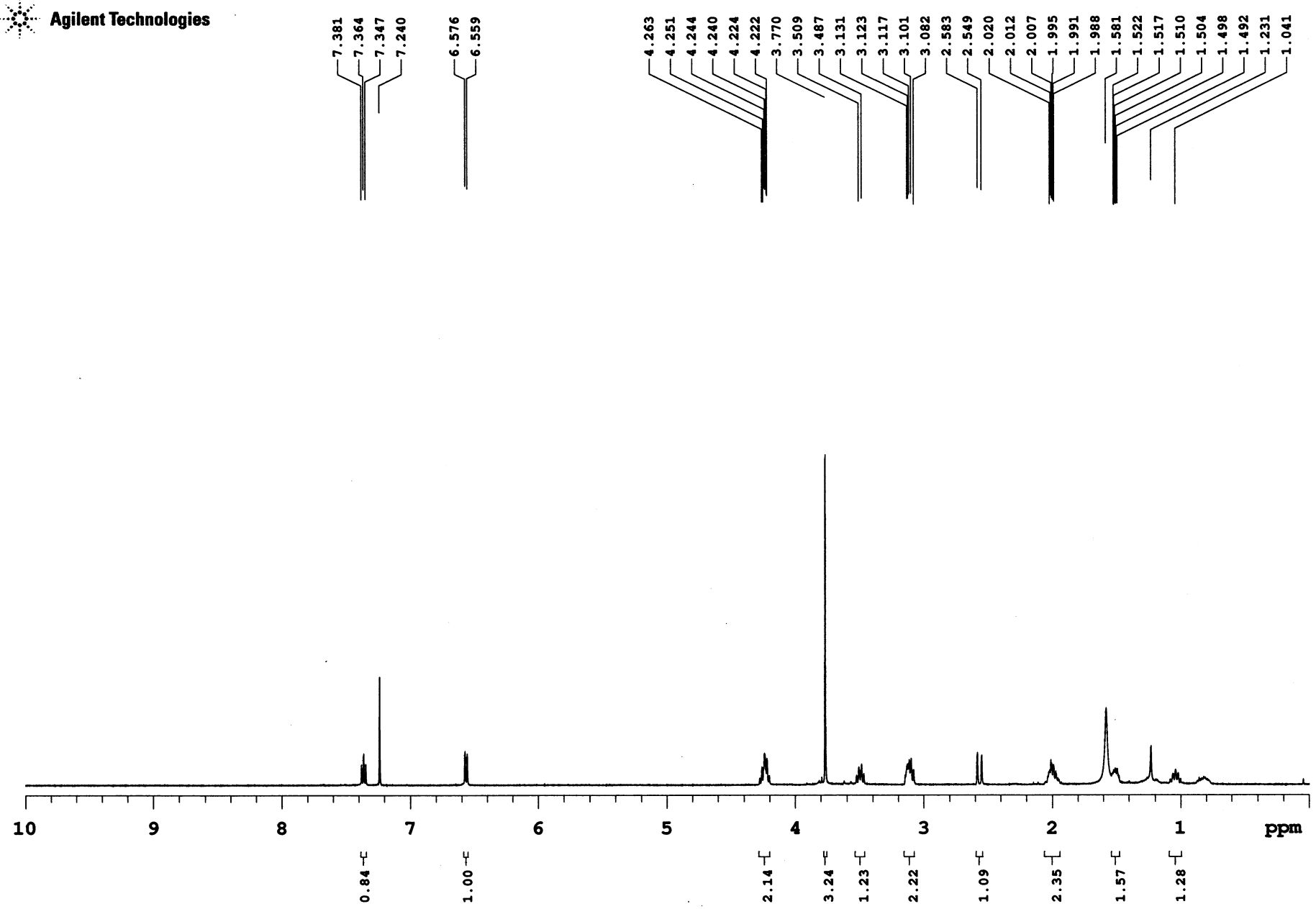
Sample Name **LCH-02-407**
Date collected **2015-05-06**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Spectrometer **Agilent-NMR-inova500**

Study owner **vnmr2**
Operator **vnmr2**

Agilent Technologies



Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-407/PROTON_01.fid

Fig S227. 1H NMR (CDCl3, 500 MHz) of compound anti-4i

Plot date 2015-06-09

LCH-02-407

Sample Name **LCH-02-407**
Date collected **2015-05-06**
Pulse sequence **CARBON**
Solvent **cdcl3**
Temperature **25**
Spectrometer **Agilent-NMR-inova500**
Study owner **vnmr2**
Operator **vnmr2**

Agilent Technologies

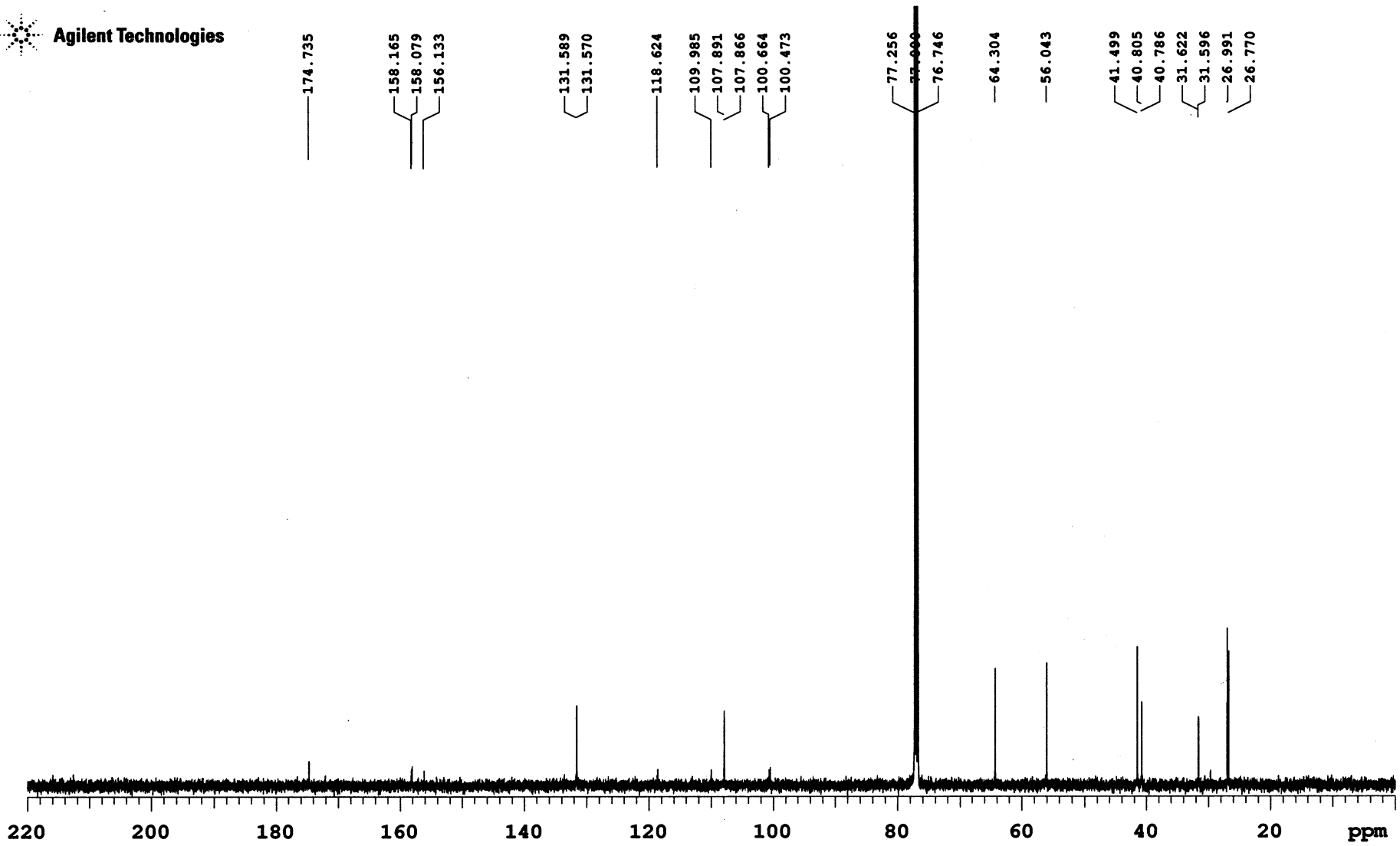


Fig S228. 13C NMR (CDCl3, 125 MHz) of compound anti-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-407/CARBON_01.fid

Plot date 2015-06-09

LCH-02-407

Sample Name **LCH-02-407**	Pulse sequence **DEPT**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-05-07**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

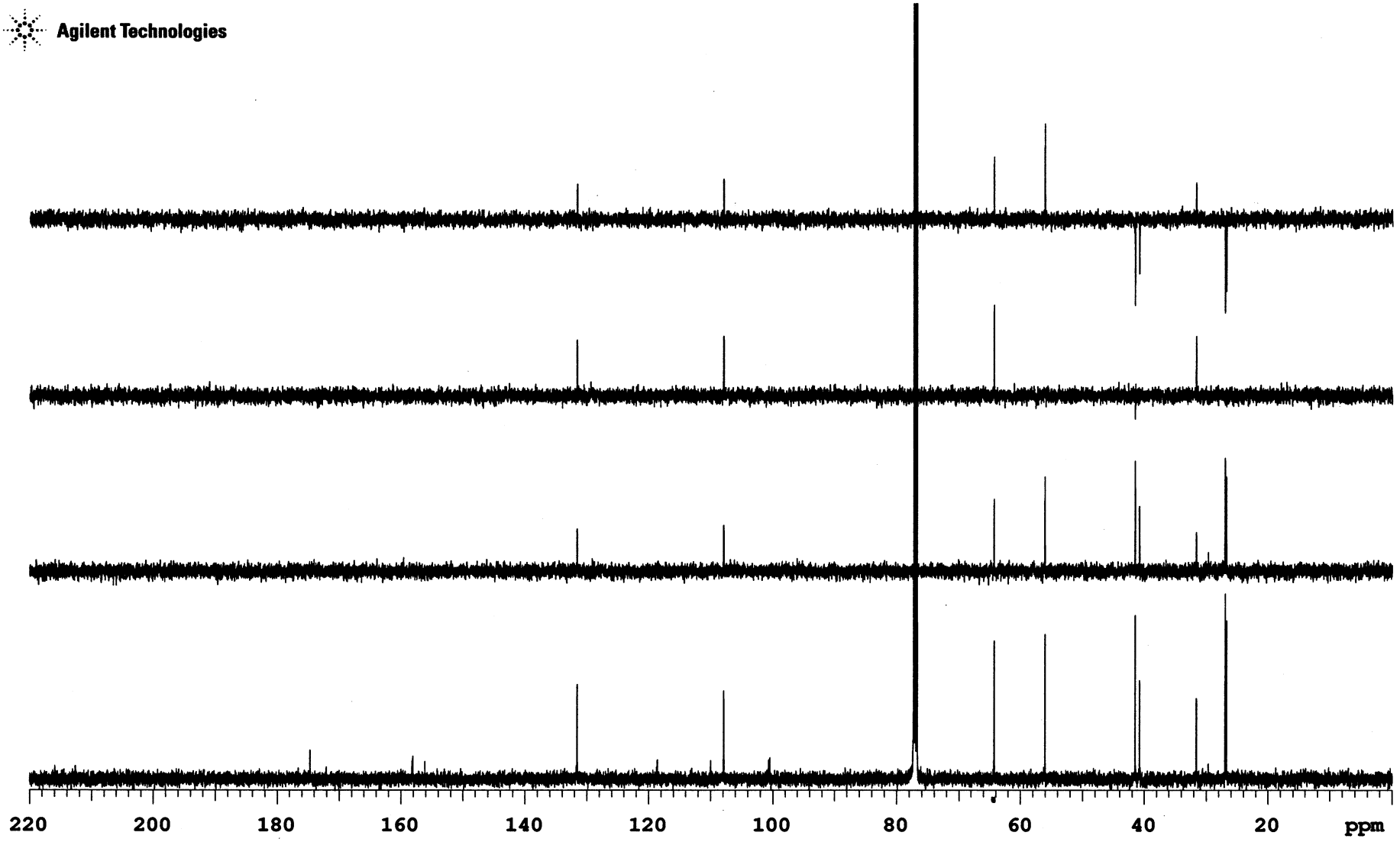


Fig S229. DEPT of compound anti-4i

LCH-02-407

Sample Name **LCH-02-407**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-05-07**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

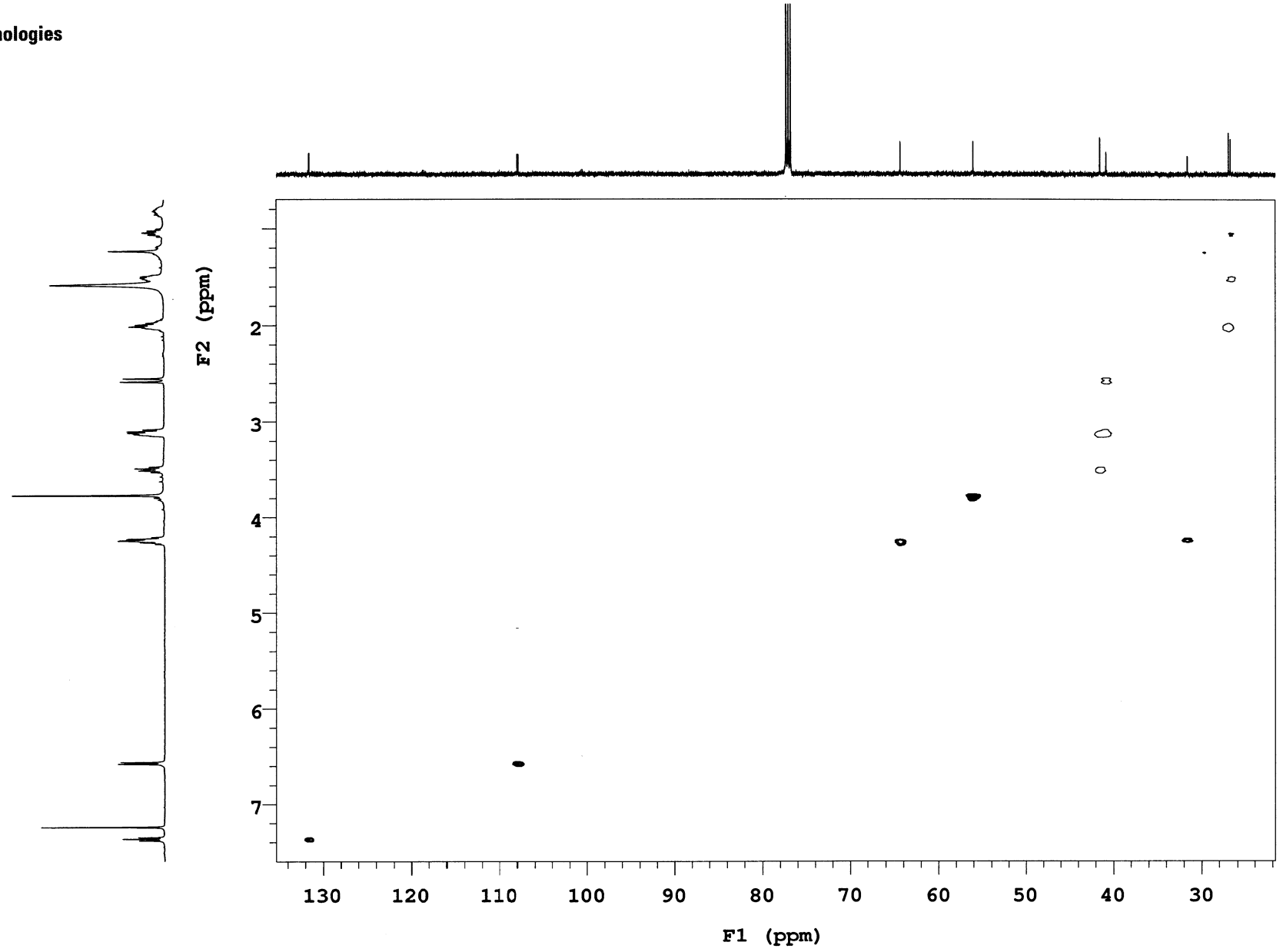


Fig S230. HSQC of compound anti-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-407/gHSQC_01.fid

Plot date 2015-10-29

LCH-02-407

Sample Name	LCH-02-407	Pulse sequence	gCOSY	Temperature	25	Study owner	vnmr2
Date collected	2015-05-07	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

Agilent Technologies

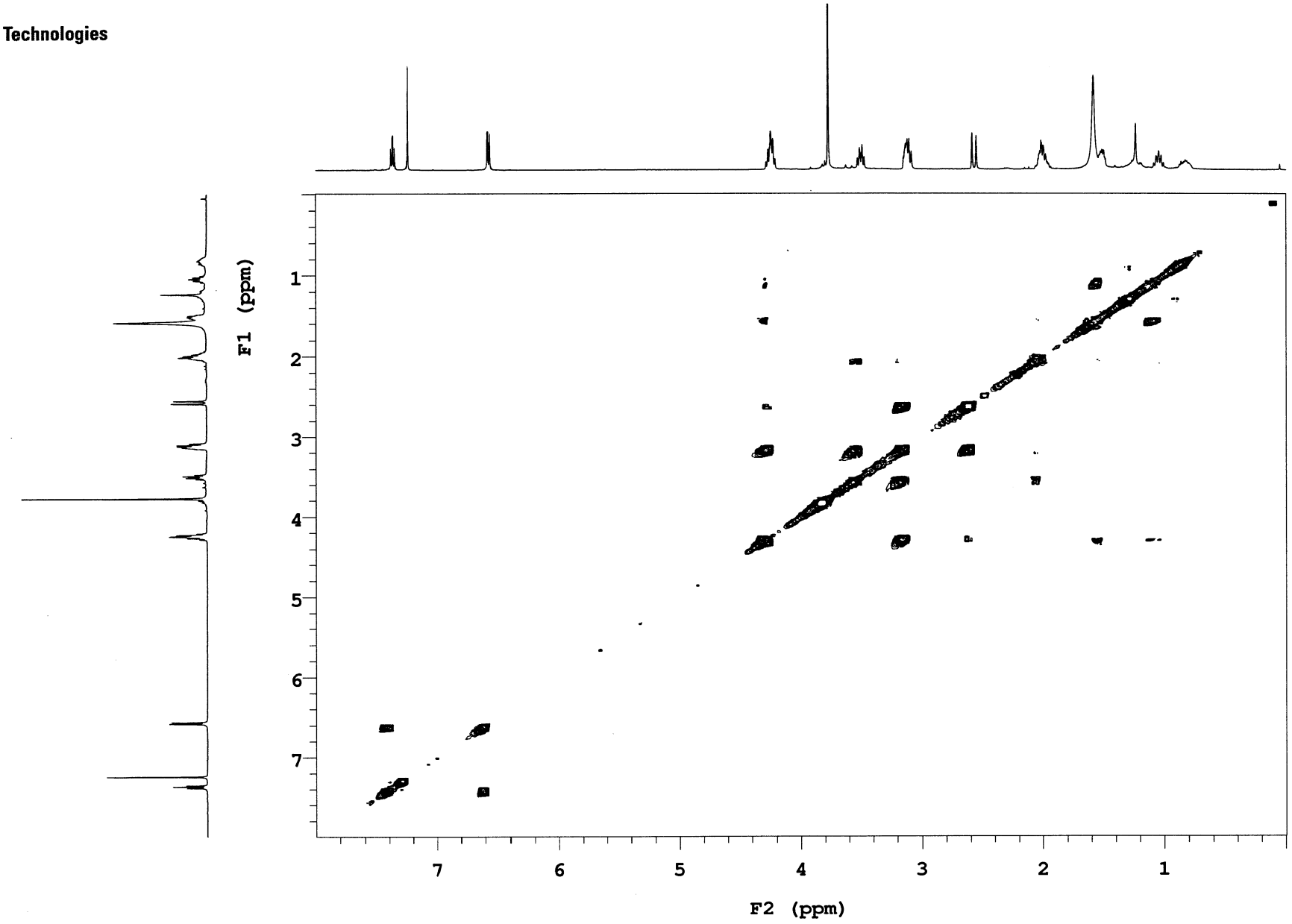


Fig S231. COSY of compound anti-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-407/gCOSY_01.fid

Plot date 2015-10-24

LCH-02-407

Sample Name **LCH-02-407**	Pulse sequence **NOESY**	Temperature **25**	Study owner **vnmr2**
Date collected **2015-05-07**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

Agilent Technologies

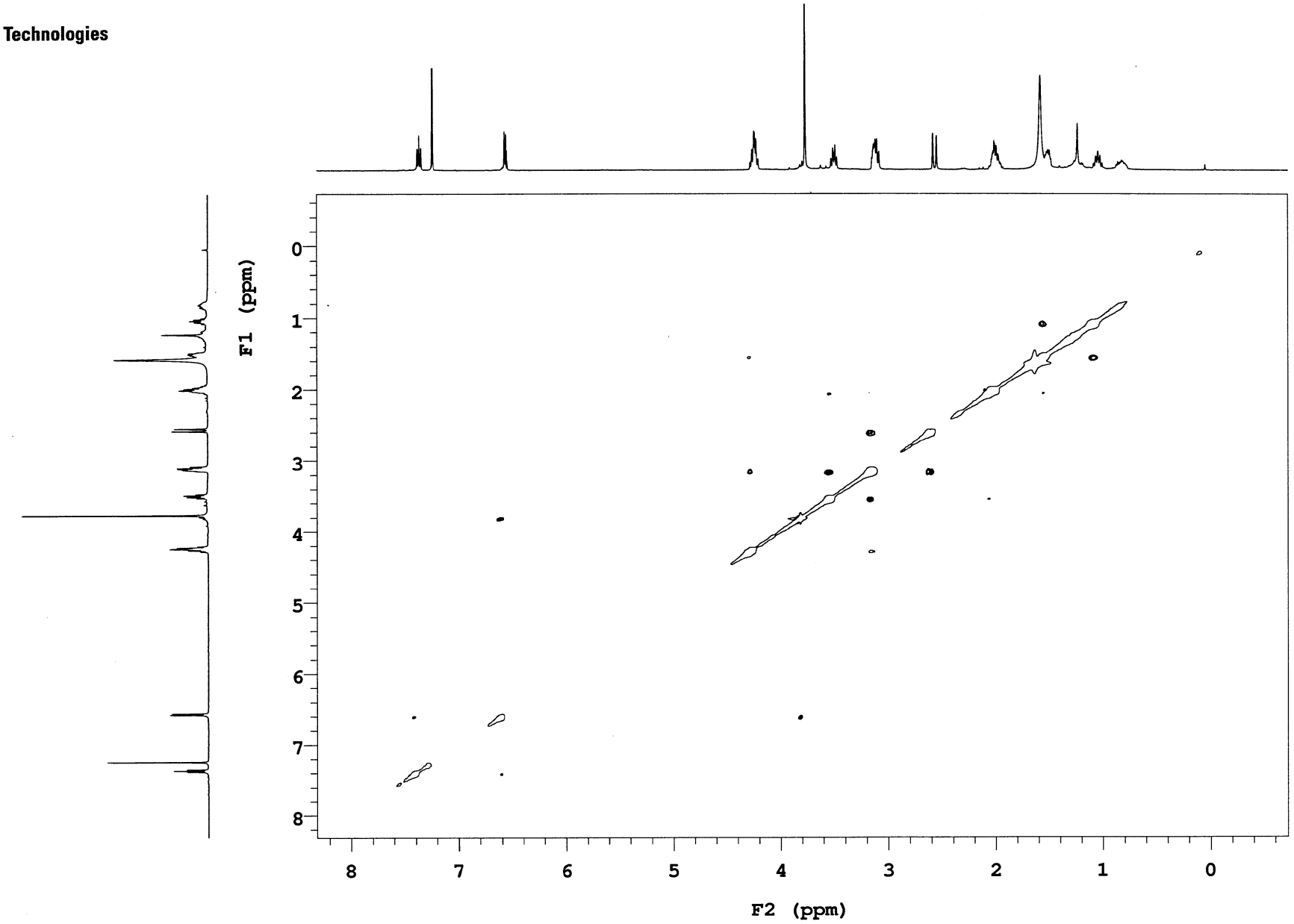


Fig S232. NOESY of compound anti-4i

Data file /home/vnmr2/vnmrsys/data/511/LCH/LCH-02-407/NOESY_01.fid

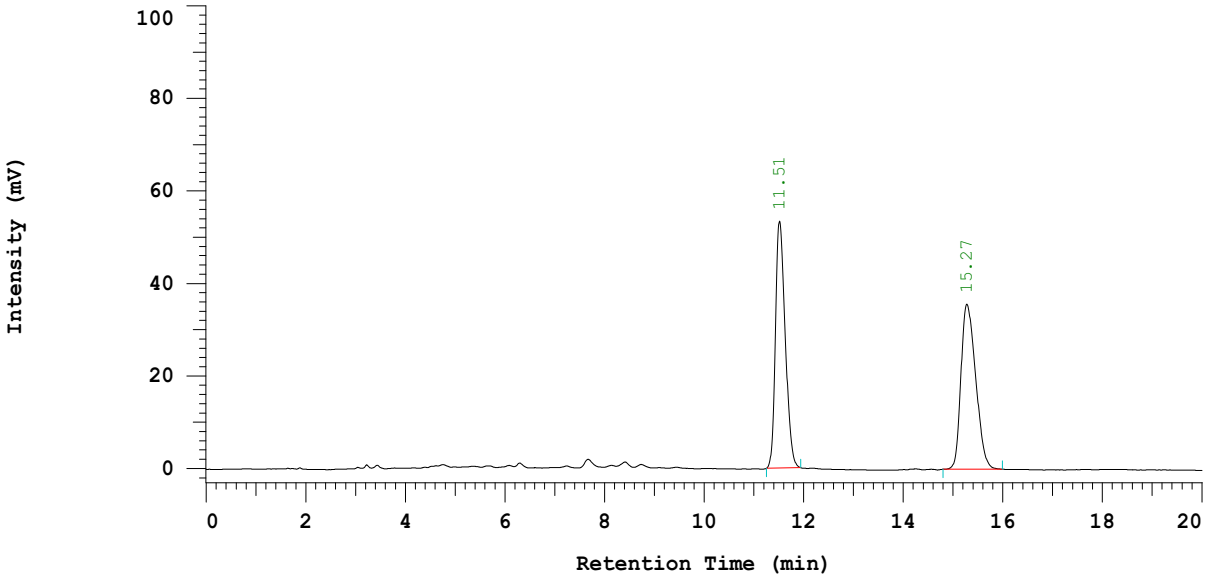
Plot date 2015-10-24

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/13 02:50 下午
Reported Date and Time: 2015/03/13 03:36 下午
Processed Date and Time: 2015/03/13 03:35 下午
Data Path: D:\LCH\DATA\0036\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0036
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-269-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	11.51	723788	53343	49.650
2	15.27	733995	35642	50.350
		1457783	88985	100.000

Peak rejection level: 20000

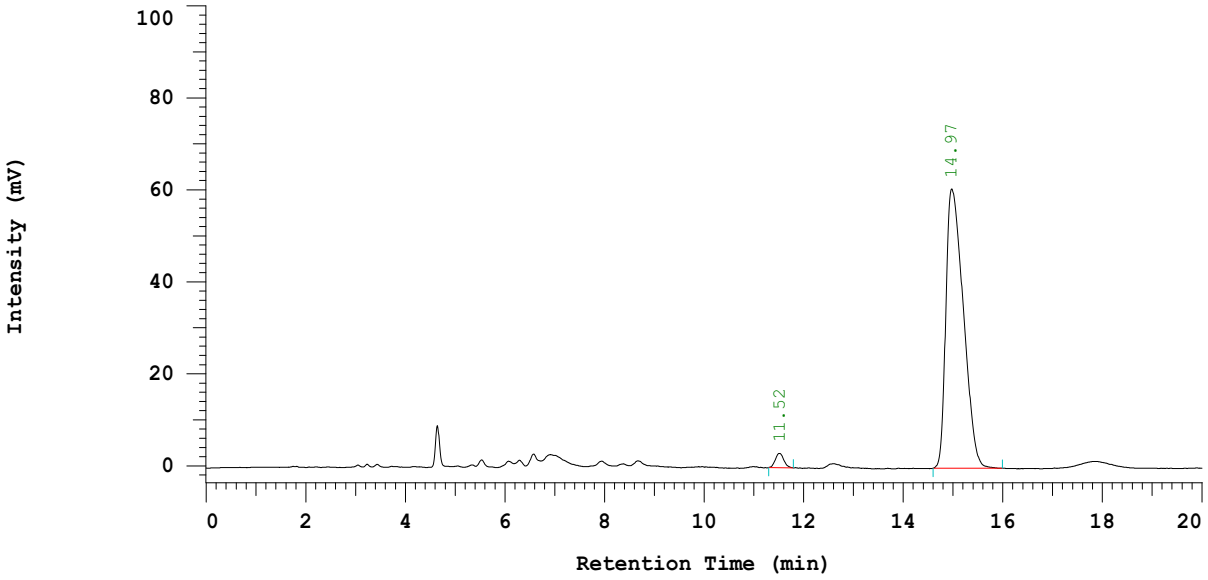
Fig S233. HPLC analysis of the racemic compound 2a, as a standard for comparison (Table 1, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/13 03:11 下午
Reported Date and Time: 2015/05/14 09:22 下午
Processed Date and Time: 2015/05/14 09:22 下午
Data Path: D:\LCH\DATA\0037\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0037
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-269-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	11.52	36342	3076	2.401
2	14.97	1477088	60717	97.599
		1513430	63793	100.000

Peak rejection level: 20000

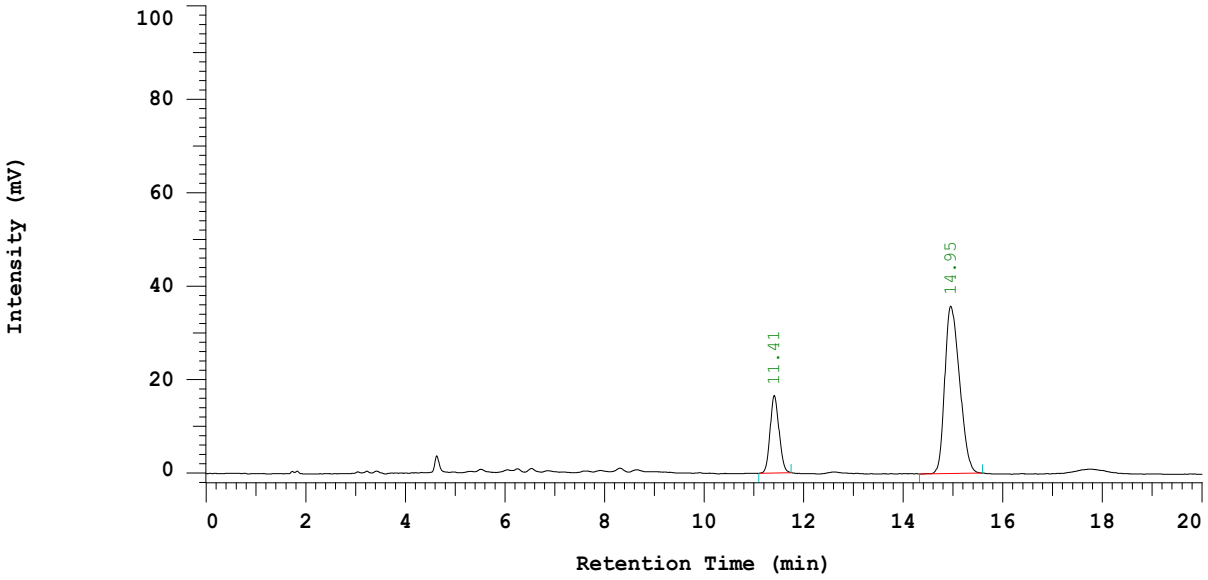
Fig S234. HPLC analysis of the chiral compound 2a obtained, (Table 1, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/13 03:32 下午
Reported Date and Time: 2015/03/13 04:36 下午
Processed Date and Time: 2015/03/13 04:35 下午
Data Path: D:\LCH\DATA\0038\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0038
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-269-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	11.41	207472	16580	21.873
2	14.95	741073	35792	78.127
		948545	52372	100.000

Peak rejection level: 20000

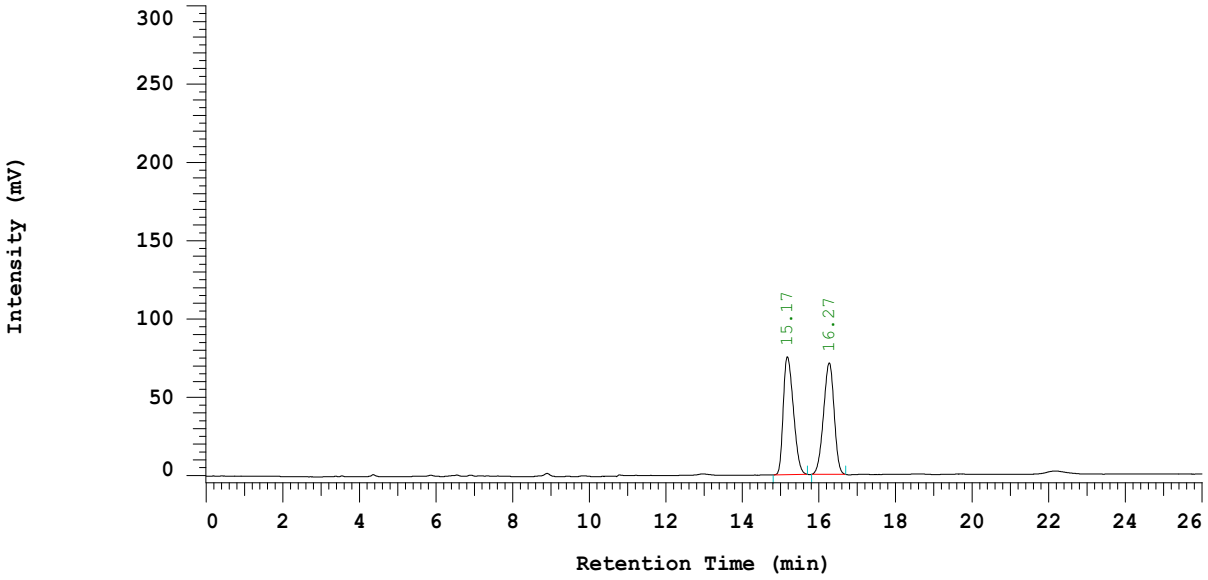
Fig S235. HPLC analysis of the mixture of chiral compound 2a obtained and the racemic compound 2a, for comparison (Table 1, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/20 07:30 下午
Reported Date and Time: 2015/05/20 08:18 下午
Processed Date and Time: 2015/05/20 08:18 下午
Data Path: D:\LCH\DATA\0217\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0217
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-371-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 5%IPA/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IC
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	15.17	1369149	75358	50.167
2	16.27	1360038	71311	49.833
		2729187	146669	100.000

Peak rejection level: 10000

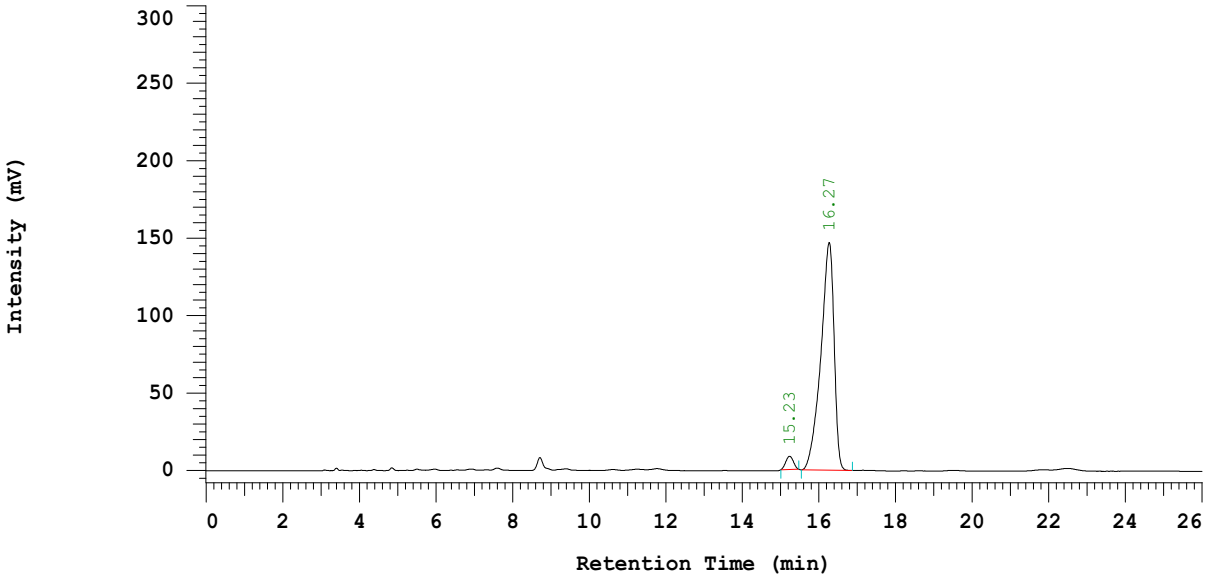
Fig S236. HPLC analysis of the racemic compound 2b, as a standard for comparison (Table 1, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/20 09:09 下午
Reported Date and Time: 2015/05/20 09:38 下午
Processed Date and Time: 2015/05/20 09:38 下午
Data Path: D:\LCH\DATA\0220\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0220
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-371-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 5%IPA/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IC
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	15.23	119698	8672	3.292
2	16.27	3515907	146995	96.708
		3635605	155667	100.000

Peak rejection level: 10000

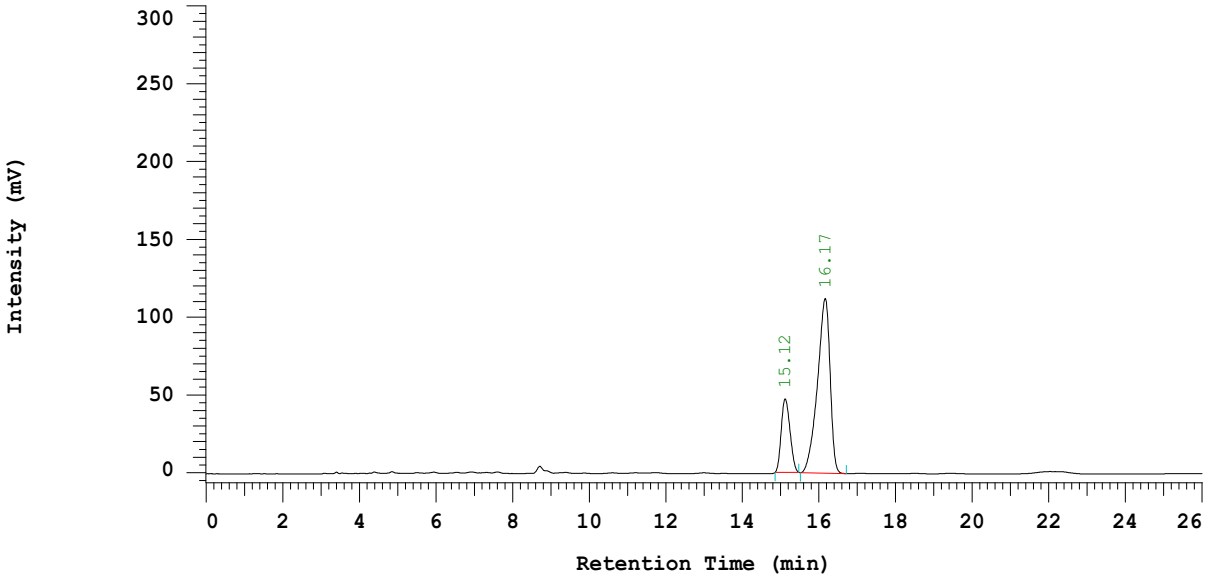
Fig S237. HPLC analysis of the chiral compound 2b obtained, (Table 1, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/20 08:38 下午
Reported Date and Time: 2015/05/20 09:07 下午
Processed Date and Time: 2015/05/20 09:07 下午
Data Path: D:\LCH\DATA\0219\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0219
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-371-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 5%IPA/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IC
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	15.12	746147	47242	22.567
2	16.17	2560145	112207	77.433
		3306292	159449	100.000

Peak rejection level: 10000

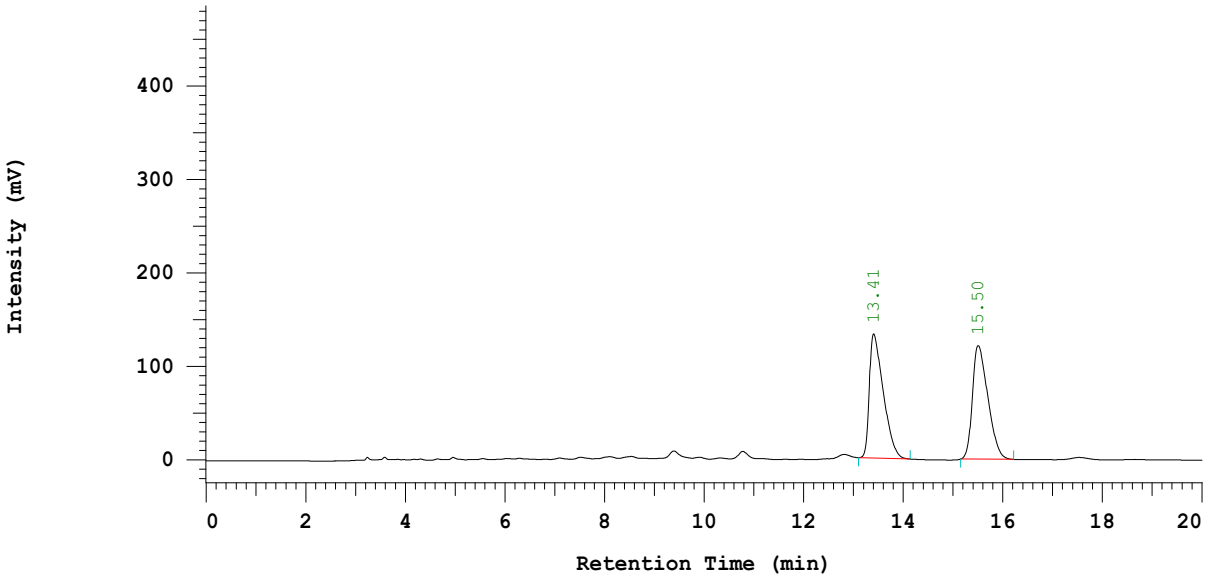
Fig S238. HPLC analysis of the mixture of chiral compound 2b obtained and the racemic compound 2b, for comparison (Table 1, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/08 07:28 下午
Reported Date and Time: 2015/03/08 07:59 下午
Processed Date and Time: 2015/03/08 07:58 下午
Data Path: D:\LCH\DATA\0033\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0033
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-288-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.41	2594974	132986	49.657
2	15.50	2630874	121354	50.343
		5225848	254340	100.000

Peak rejection level: 20000

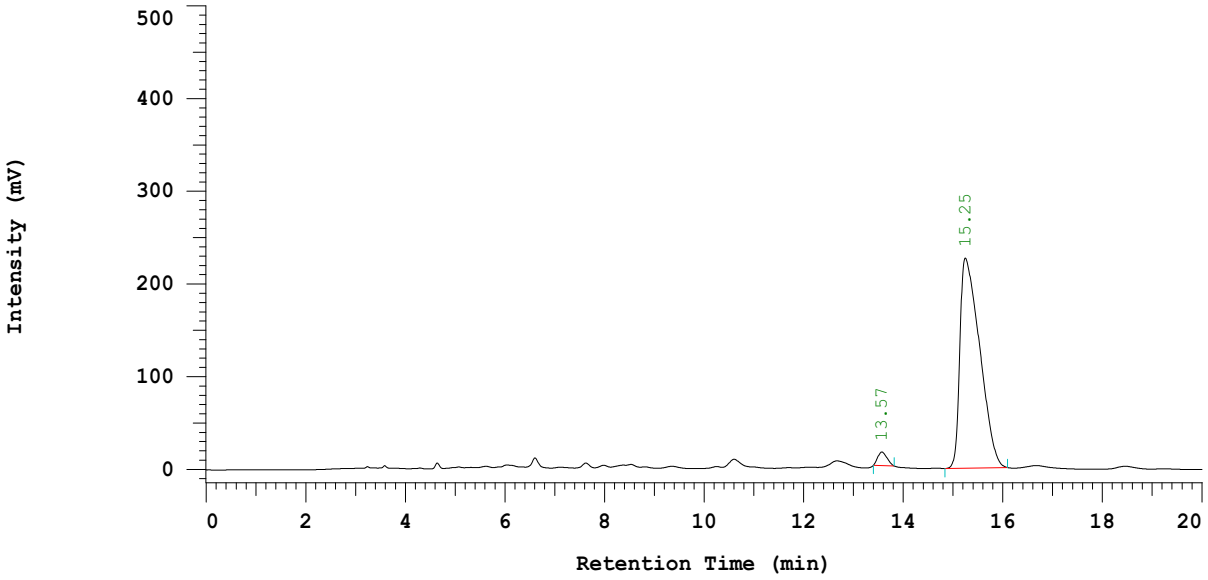
Fig S239. HPLC analysis of the racemic compound 2c, as a standard for comparison (Table 1, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/08 07:49 下午
Reported Date and Time: 2015/03/13 03:30 下午
Processed Date and Time: 2015/03/13 03:29 下午
Data Path: D:\LCH\DATA\0034\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0034
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-288-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.57	189705	14656	2.940
2	15.25	6261858	226509	97.060
		6451563	241165	100.000

Peak rejection level: 20000

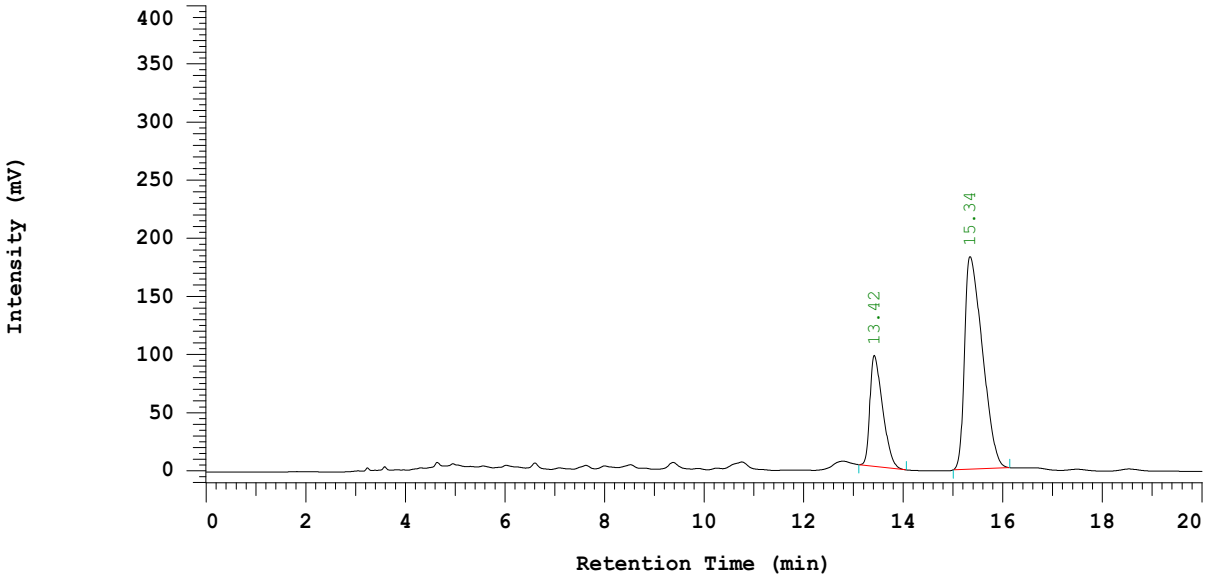
Fig S240. HPLC analysis of the chiral compound 2c obtained, (Table 1, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/08 08:11 下午
Reported Date and Time: 2015/03/08 08:36 下午
Processed Date and Time: 2015/03/08 08:36 下午
Data Path: D:\LCH\DATA\0035\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0035
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-288-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.42	1698138	95452	27.129
2	15.34	4561361	182701	72.871
		6259499	278153	100.000

Peak rejection level: 20000

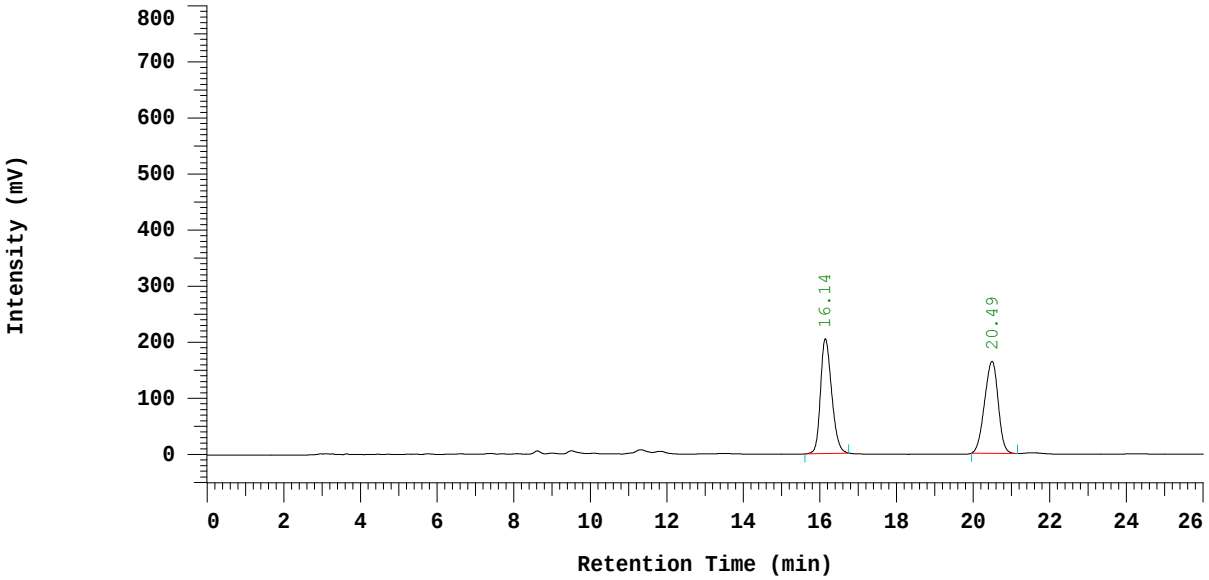
Fig S241. HPLC analysis of the mixture of chiral compound 2c obtained and the racemic compound 2c, for comparison (Table 1, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/01/18 06:13 下午
Reported Date and Time: 2015/10/29 01:26 下午
Processed Date and Time: 2015/10/29 01:26 下午
Data Path: D:\LCH\DATA\0008\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0008
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-342-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 238 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 238 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	16.14	4095295	204628	50.415
2	20.49	4027838	163845	49.585
		8123133	368473	100.000

Peak rejection level: 10000

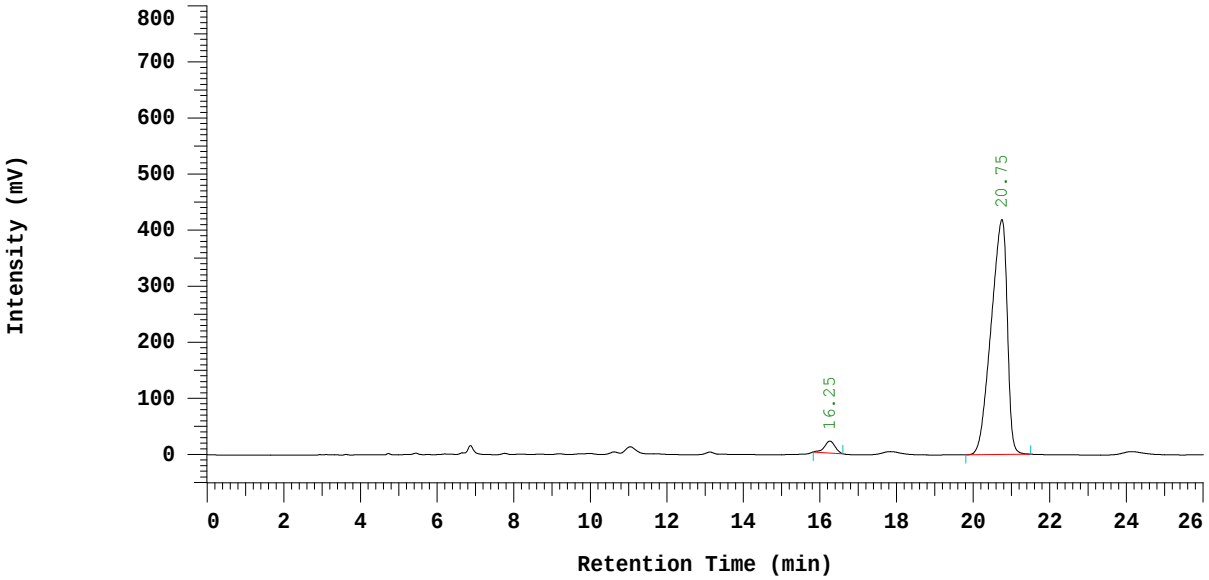
Fig S242. HPLC analysis of the racemic compound 2d, as a standard for comparison (Table 1, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/01/18 06:45 下午
Reported Date and Time: 2015/10/29 01:36 下午
Processed Date and Time: 2015/10/29 01:35 下午
Data Path: D:\LCH\DATA\0009\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0009
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-342-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 238 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 238 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	16.25	397741	21125	3.170
2	20.75	12147809	418596	96.830
		12545550	439721	100.000

Peak rejection level: 10000

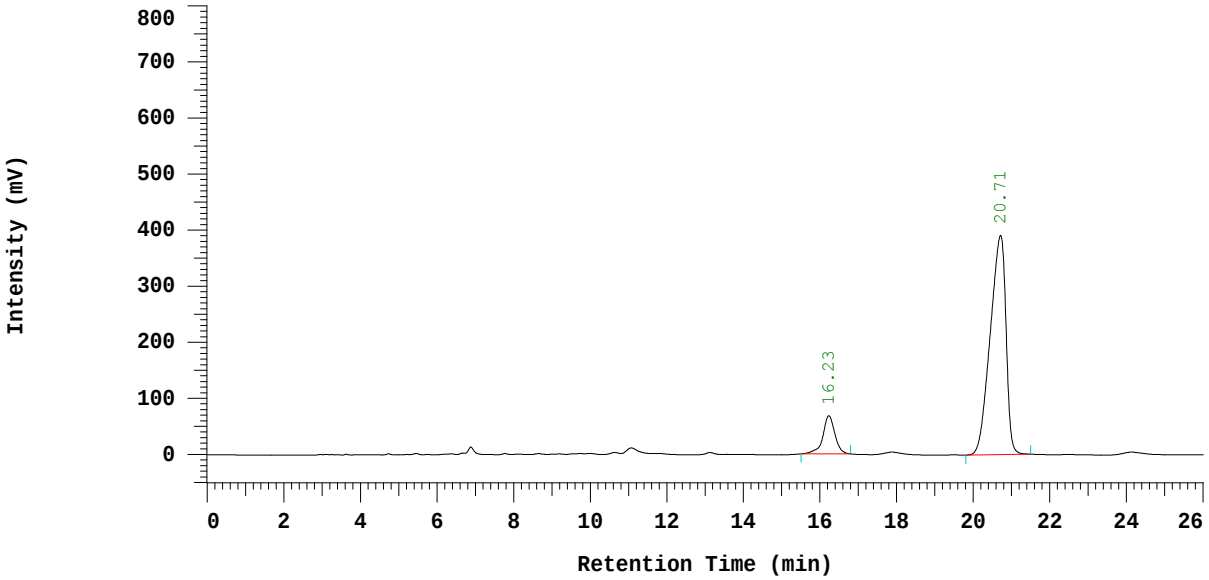
Fig S243. HPLC analysis of the chiral compound 2d obtained, (Table 1, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/01/18 07:16 下午
Reported Date and Time: 2015/10/29 01:39 下午
Processed Date and Time: 2015/10/29 01:39 下午
Data Path: D:\LCH\DATA\0010\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0010
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-342-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 238 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 238 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	16.23	1464419	67944	11.661
2	20.71	11093313	390807	88.339
		12557732	458751	100.000

Peak rejection level: 10000

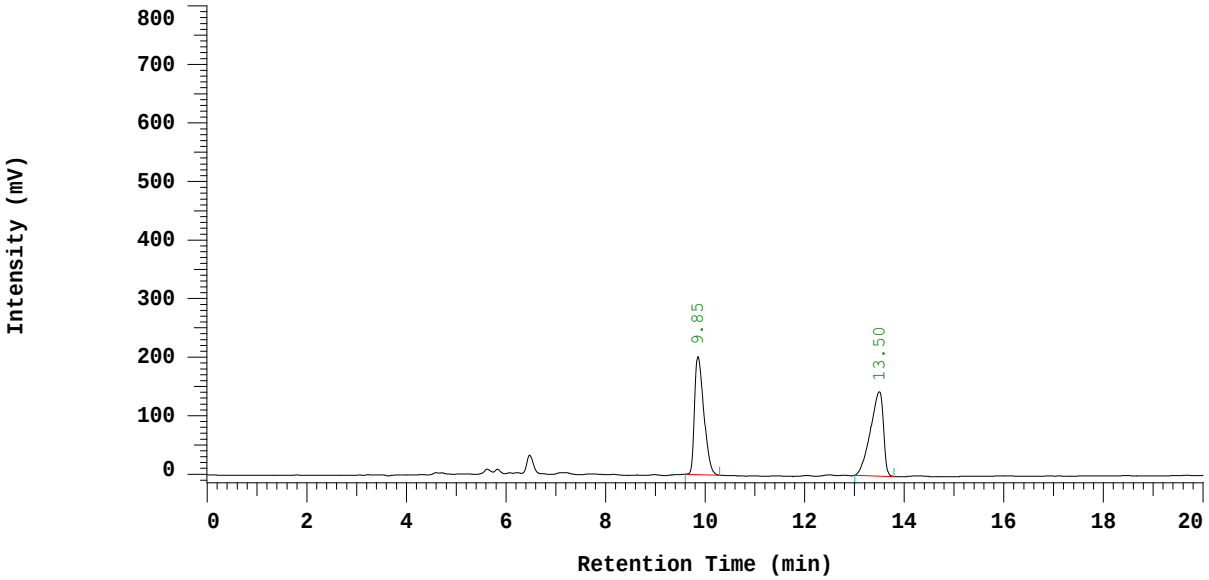
Fig S244. HPLC analysis of the mixture of chiral compound 2d obtained and the racemic compound 2d, for comparison (Table 1, entry 4)

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/02/12 01:56 下午
Reported Date and Time: 2015/10/29 01:45 下午
Processed Date and Time: 2015/10/29 01:45 下午
Data Path: D:\LCH\DATA\0021\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0021
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-350-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.85	2622204	201404	50.085
2	13.50	2613273	143943	49.915
		5235477	345347	100.000

Peak rejection level: 10000

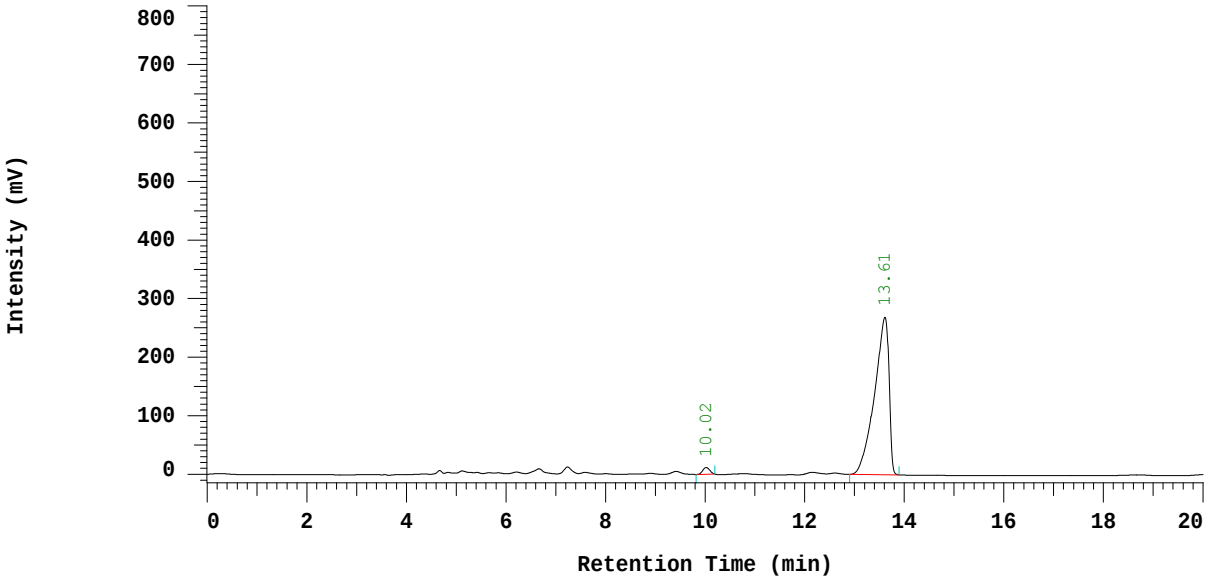
Fig S245. HPLC analysis of the racemic compound 2e, as a standard for comparison (Table 1, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/02/12 02:17 下午
Reported Date and Time: 2015/10/29 01:48 下午
Processed Date and Time: 2015/10/29 01:47 下午
Data Path: D:\LCH\DATA\0022\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0022
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-350-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.02	114431	11553	2.062
2	13.61	5435543	269019	97.938
		5549974	280572	100.000

Peak rejection level: 10000

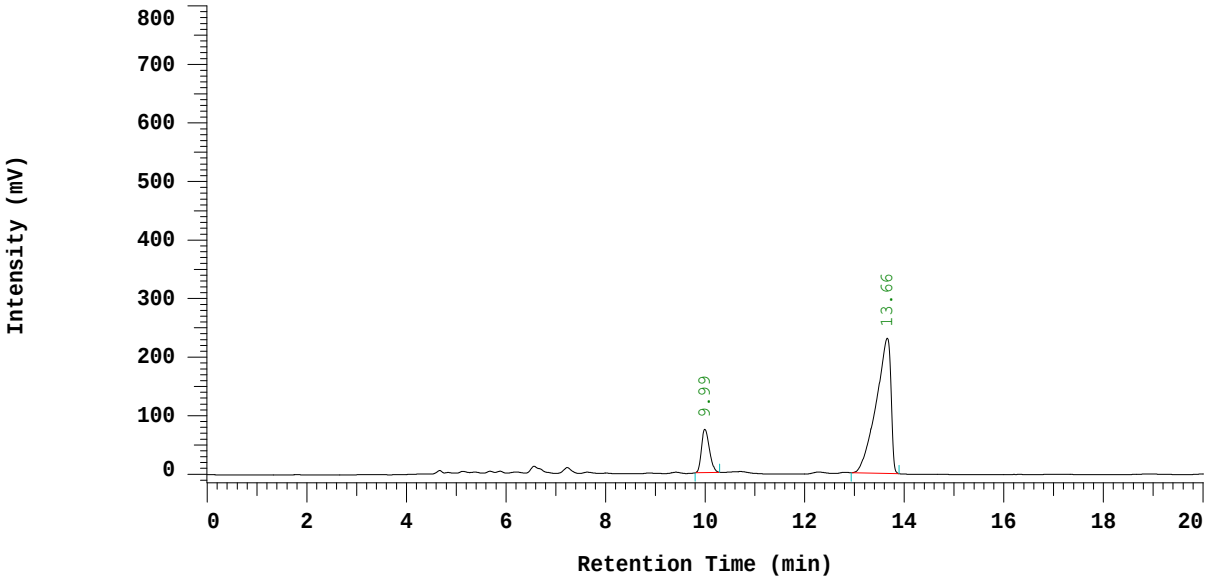
Fig S246. HPLC analysis of the chiral compound 2e obtained, (Table 1, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/02/12 03:02 下午
Reported Date and Time: 2015/10/29 01:54 下午
Processed Date and Time: 2015/10/29 01:53 下午
Data Path: D:\LCH\DATA\0024\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0024
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-350-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.99	828335	74045	14.778
2	13.66	4776816	230969	85.222
		5605151	305014	100.000

Peak rejection level: 10000

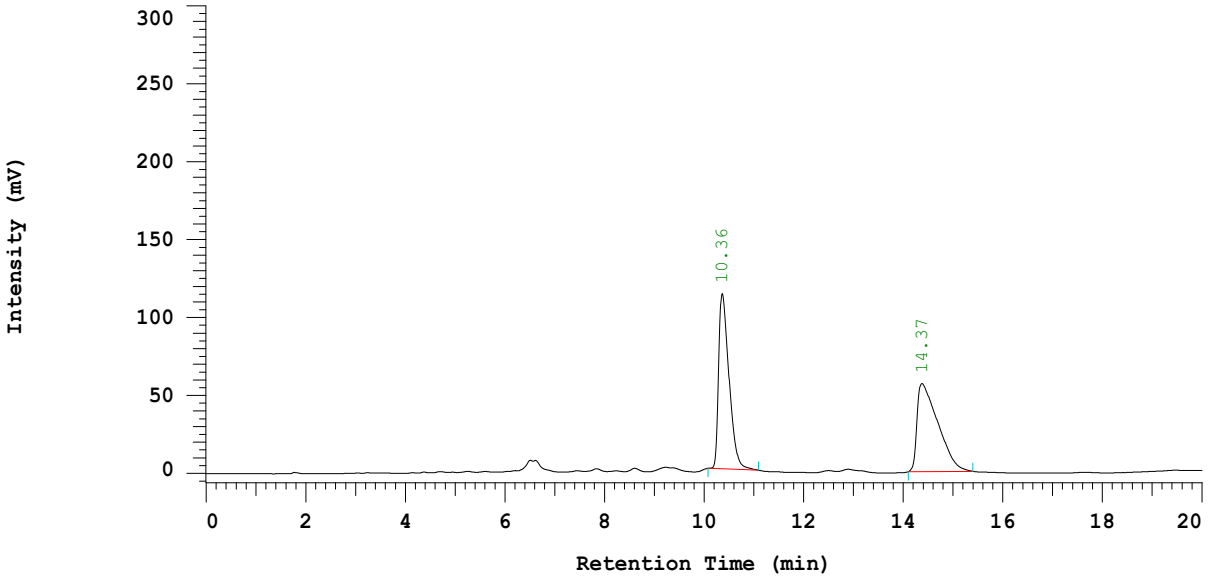
Fig S247. HPLC analysis of the mixture of chiral compound 2e obtained and the racemic compound 2e, for comparison (Table 1, entry 5)

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/13 03:59 下午
Reported Date and Time: 2015/03/13 04:32 下午
Processed Date and Time: 2015/03/13 04:31 下午
Data Path: D:\LCH\DATA\0039\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0039
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-386-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.36	1634777	112482	49.731
2	14.37	1652464	56534	50.269
		3287241	169016	100.000

Peak rejection level: 20000

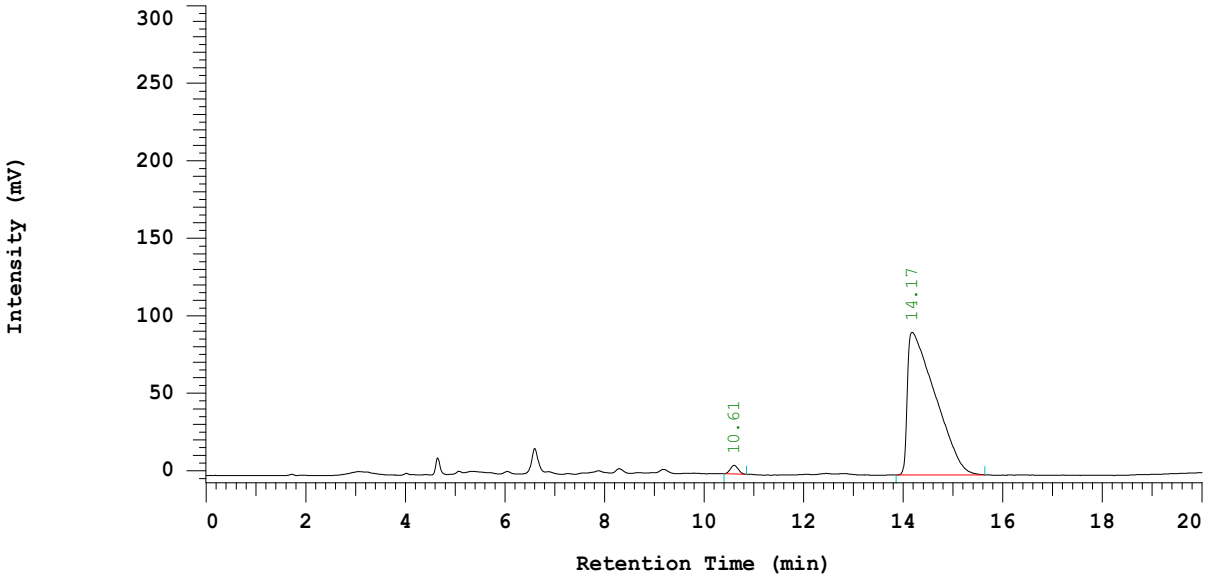
Fig S248. HPLC analysis of the racemic compound 2f, as a standard for comparison (Table 1, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/13 04:24 下午
Reported Date and Time: 2015/05/14 09:40 下午
Processed Date and Time: 2015/05/14 09:40 下午
Data Path: D:\LCH\DATA\0040\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0040
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-386-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.61	61835	5629	1.704
2	14.17	3567935	92012	98.296
		3629770	97641	100.000

Peak rejection level: 10000

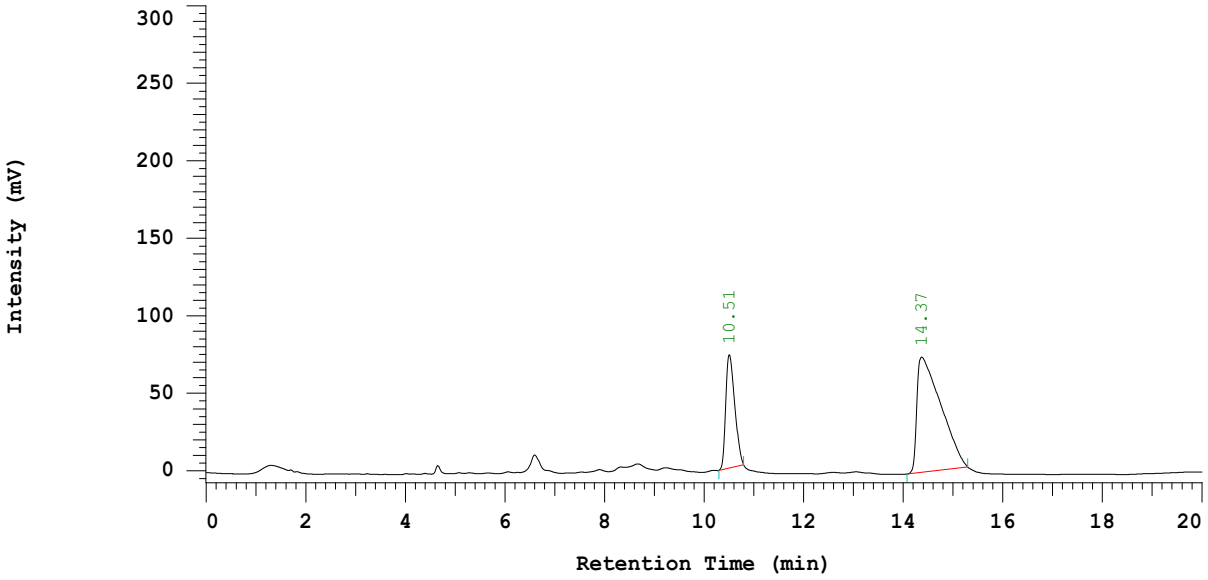
Fig S249. HPLC analysis of the chiral compound 2f obtained, (Table 1, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/13 04:45 下午
Reported Date and Time: 2015/03/13 05:07 下午
Processed Date and Time: 2015/03/13 05:06 下午
Data Path: D:\LCH\DATA\0041\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0041
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-386-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.51	909069	72872	26.899
2	14.37	2470476	74395	73.101
		3379545	147267	100.000

Peak rejection level: 20000

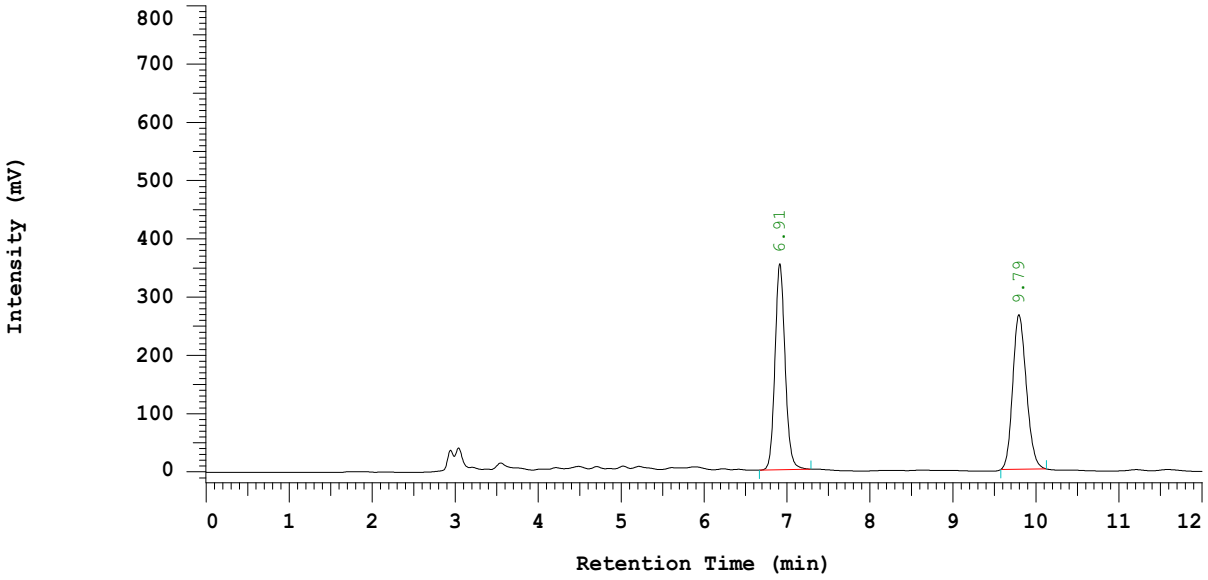
Fig S250. HPLC analysis of the mixture of chiral compound 2f obtained and the racemic compound 2f, for comparison (Table 1, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/15 11:25 下午
Reported Date and Time: 2015/03/16 12:07 上午
Processed Date and Time: 2015/03/16 12:07 上午
Data Path: D:\LCH\DATA\0056\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0056
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-369-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 225 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 225 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.91	2982874	353394	49.690
2	9.79	3020036	265506	50.310
		6002910	618900	100.000

Peak rejection level: 20000

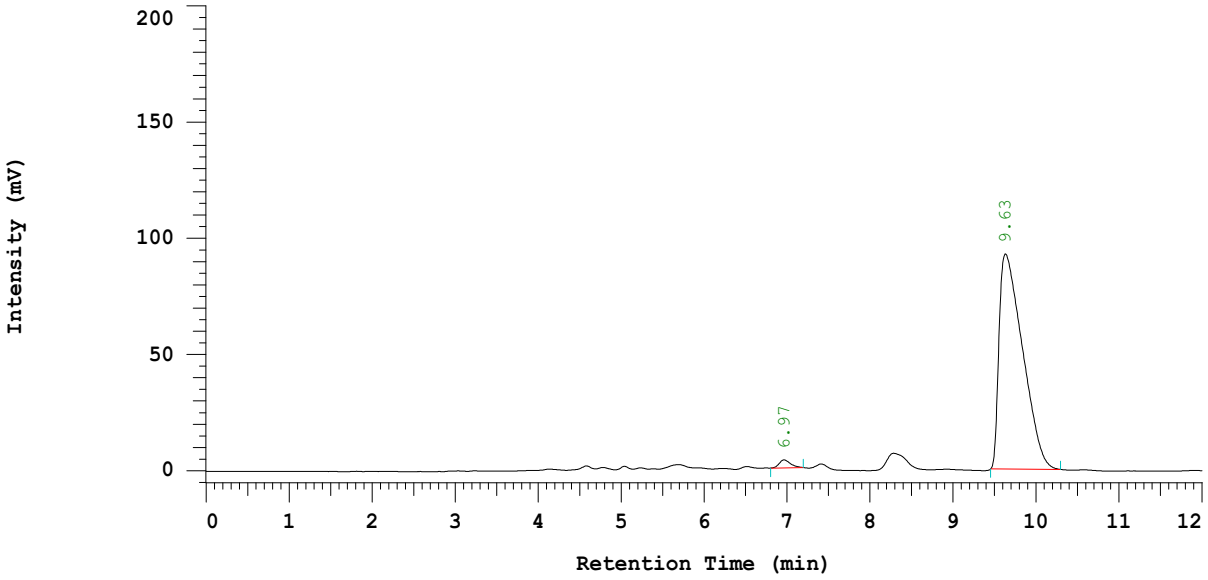
Fig S251. HPLC analysis of the racemic compound 2g, as a standard for comparison (Table 1, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/16 12:04 上午
Reported Date and Time: 2015/04/02 10:46 下午
Processed Date and Time: 2015/04/02 10:45 下午
Data Path: D:\LCH\DATA\0058\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0058
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-369-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.97	32430	3450	1.756
2	9.63	1814235	92466	98.244
		1846665	95916	100.000

Peak rejection level: 20000

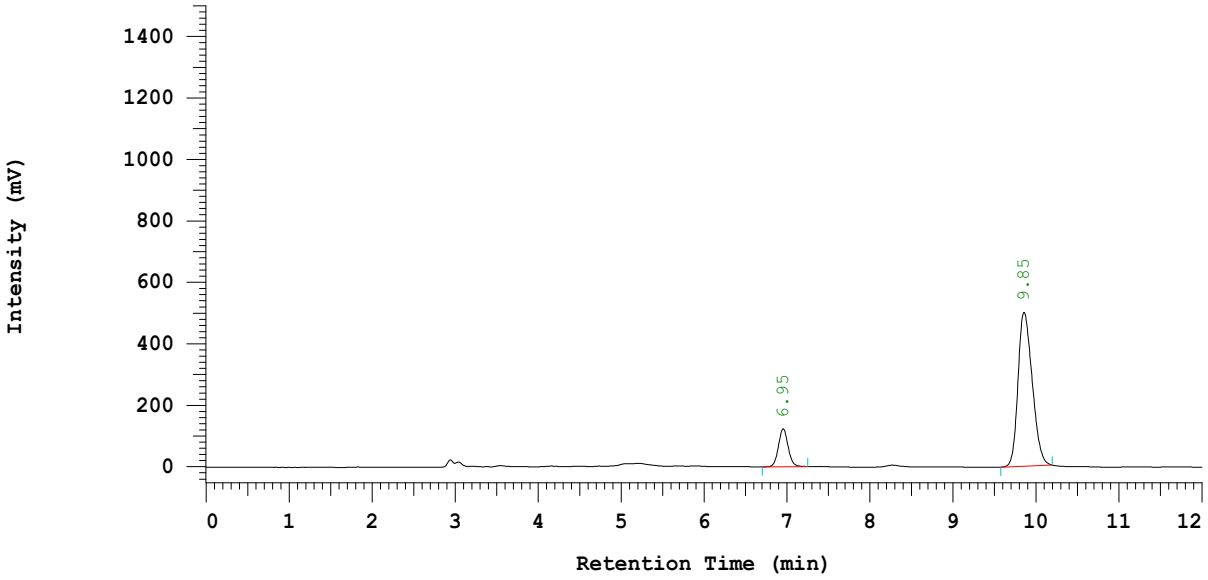
Fig S252. HPLC analysis of the chiral compound 2g obtained, (Table 1, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/16 12:37 上午
Reported Date and Time: 2015/03/16 12:51 上午
Processed Date and Time: 2015/03/16 12:51 上午
Data Path: D:\LCH\DATA\0060\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0060
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-369-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 225 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 225 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.95	1000607	123762	14.399
2	9.85	5948759	500921	85.601
		6949366	624683	100.000

Peak rejection level: 20000

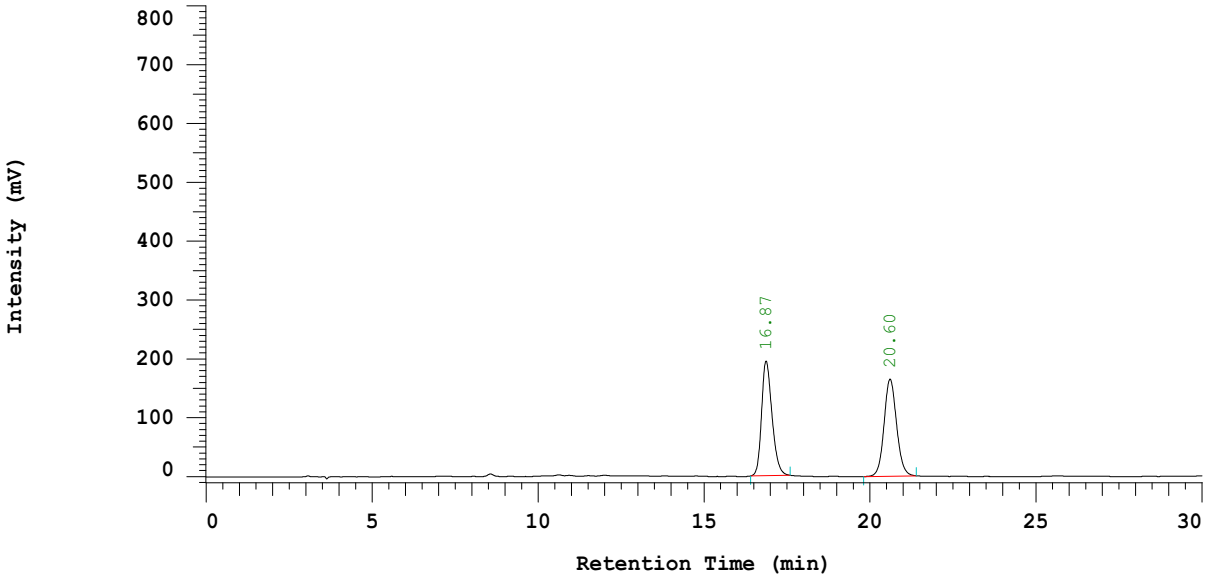
Fig S253. HPLC analysis of the mixture of chiral compound 2g obtained and the racemic compound 2g, for comparison (Table 1, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/07 12:50 上午
Reported Date and Time: 2015/04/30 12:32 上午
Processed Date and Time: 2015/04/30 12:32 上午
Data Path: D:\LCH\DATA\0030\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0030
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-287-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 238 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 238 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	16.87	4235650	195107	49.727
2	20.60	4282184	164949	50.273
		8517834	360056	100.000

Peak rejection level: 20000

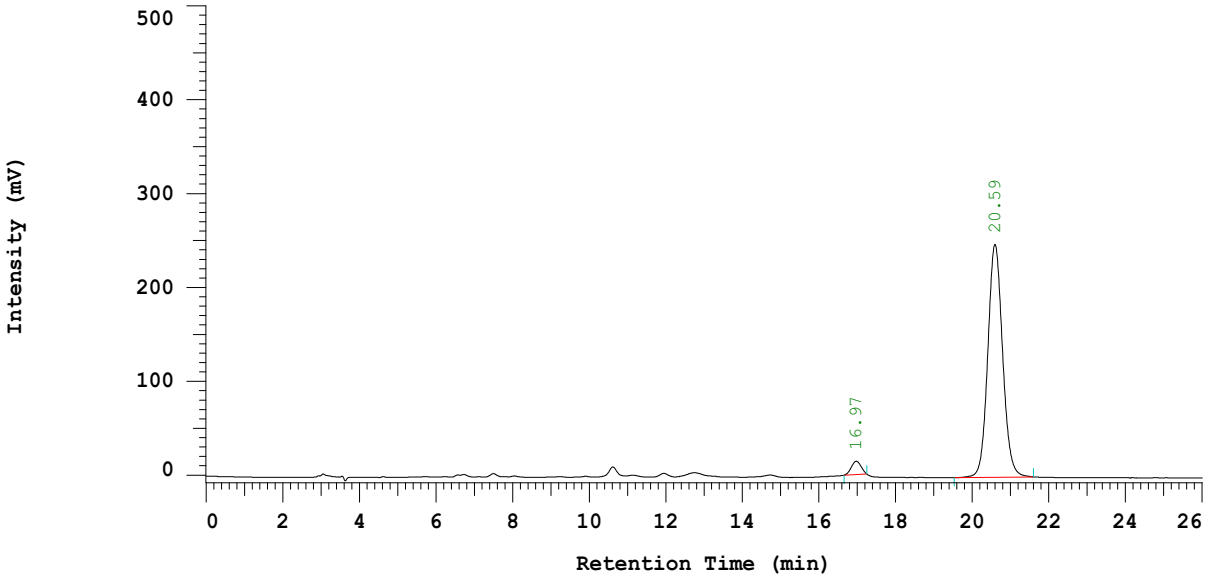
Fig S254. HPLC analysis of the racemic compound 2h, as a standard for comparison (Table 1, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/07 01:22 上午
Reported Date and Time: 2015/05/27 07:43 下午
Processed Date and Time: 2015/05/27 07:42 下午
Data Path: D:\LCH\DATA\0031\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0031
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-287-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 238 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 238 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	16.97	248557	14233	3.622
2	20.59	6613528	247824	96.378
		6862085	262057	100.000

Peak rejection level: 10000

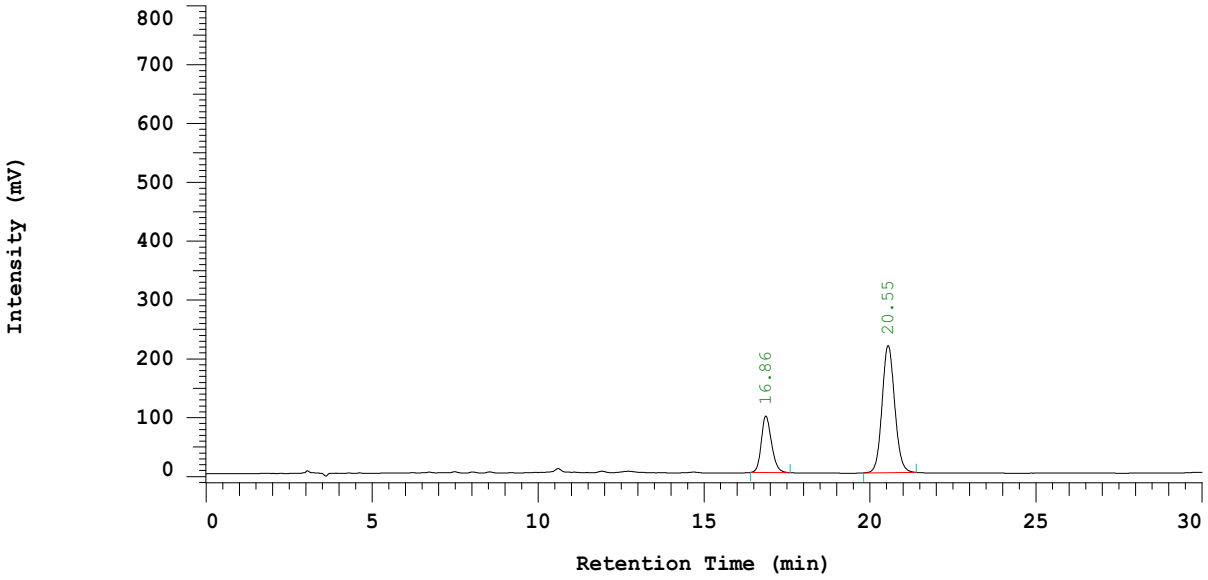
Fig S255. HPLC analysis of the chiral compound 2h obtained, (Table 1, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/07 01:55 上午
Reported Date and Time: 2015/04/30 12:33 上午
Processed Date and Time: 2015/04/30 12:33 上午
Data Path: D:\LCH\DATA\0032\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0032
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-287-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 238 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 238 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	16.86	2045501	95989	26.507
2	20.55	5671227	216163	73.493
		7716728	312152	100.000

Peak rejection level: 20000

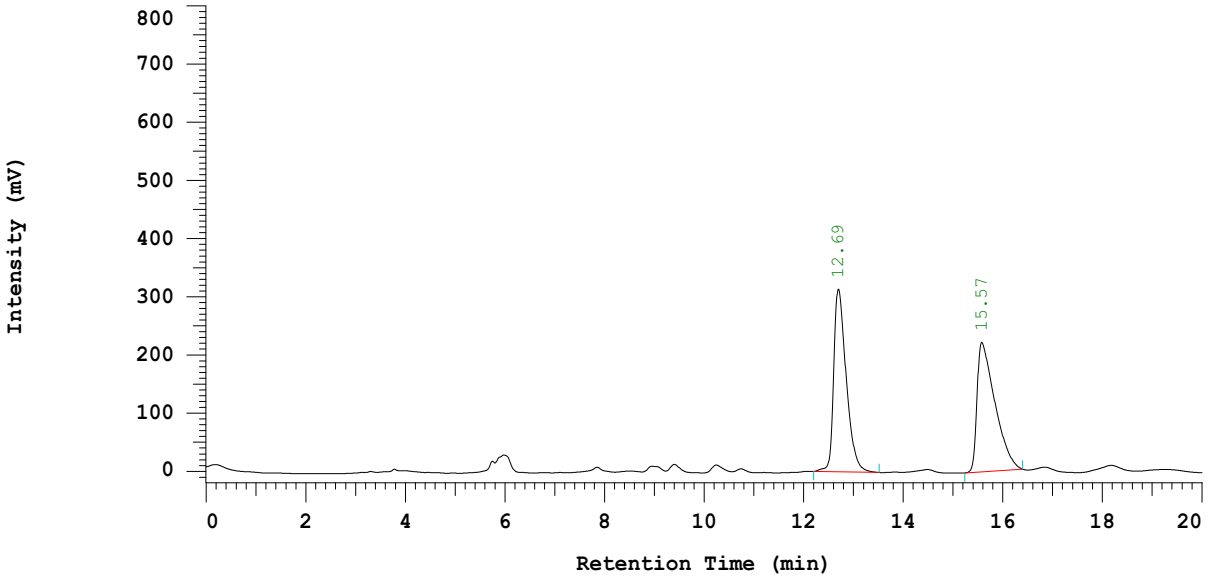
Fig S256. HPLC analysis of the mixture of chiral compound 2h obtained and the racemic compound 2h, for comparison (Table 1, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/15 09:14 下午
Reported Date and Time: 2015/03/15 09:39 下午
Processed Date and Time: 2015/03/15 09:39 下午
Data Path: D:\LCH\DATA\0052\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0052
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-383-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 5%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	12.69	5494214	314179	49.643
2	15.57	5573252	222546	50.357
		11067466	536725	100.000

Peak rejection level: 20000

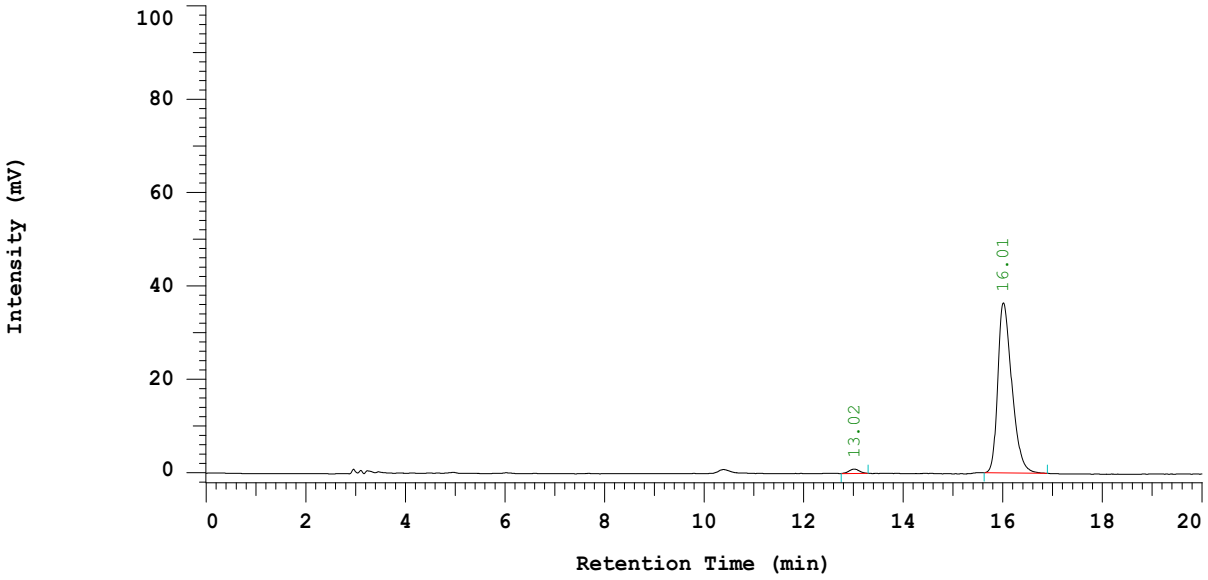
Fig S257. HPLC analysis of the racemic compound 2i, as a standard for comparison (Table 1, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/15 10:43 下午
Reported Date and Time: 2015/05/14 09:29 下午
Processed Date and Time: 2015/05/14 09:29 下午
Data Path: D:\LCH\DATA\0055\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0055
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-383-chiral
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 5%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.02	13479	927	1.857
2	16.01	712503	36384	98.143
		725982	37311	100.000

Peak rejection level: 10000

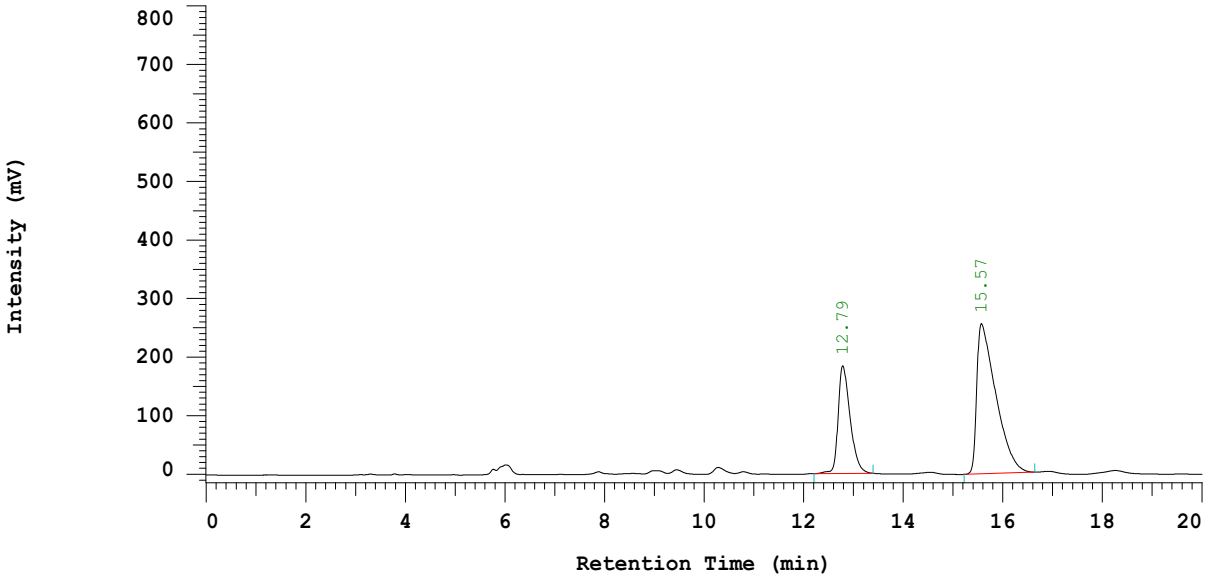
Fig S258. HPLC analysis of the chiral compound 2i obtained, (Table 1, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/03/15 09:58 下午
Reported Date and Time: 2015/03/15 10:27 下午
Processed Date and Time: 2015/03/15 10:27 下午
Data Path: D:\LCH\DATA\0054\
Processing Method: test-IPA/Hx

System (acquisition): Sys 1
Series: 0054
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-383-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 5%IPA/Hx-1ml/min-col IB

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-IPA/Hx
Column Type: IB
Method Developer: WL
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	12.79	3085049	184151	31.077
2	15.57	6842001	256654	68.923
		9927050	440805	100.000

Peak rejection level: 20000

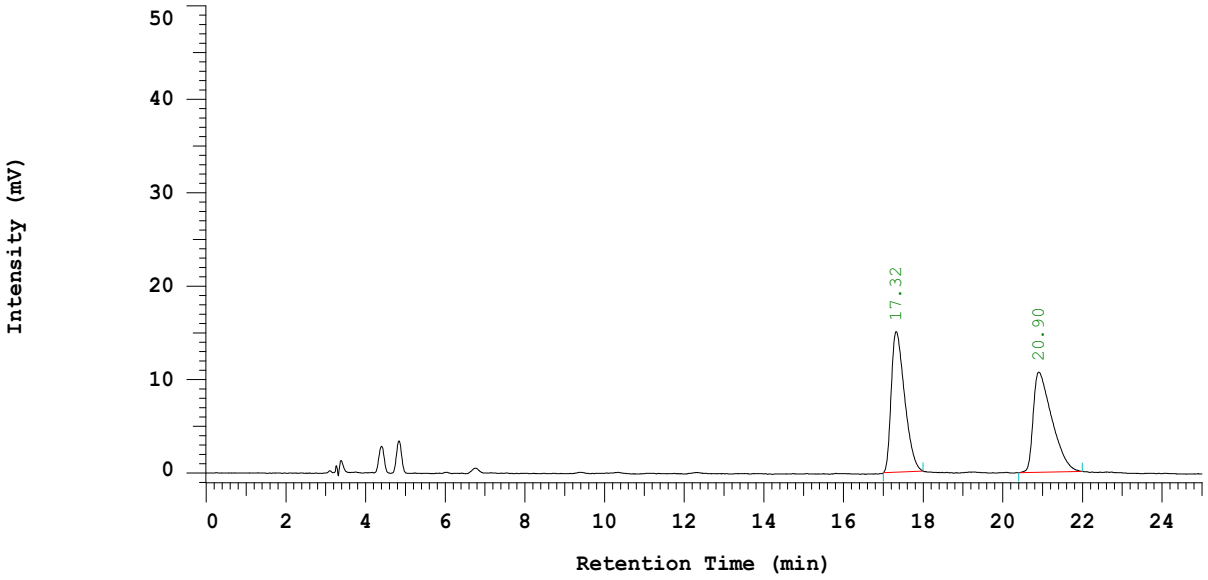
Fig S259. HPLC analysis of the mixture of chiral compound 2i obtained and the racemic compound 2i, for comparison (Table 1, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/23 09:31 下午
Reported Date and Time: 2015/04/23 10:09 下午
Processed Date and Time: 2015/04/23 10:09 下午
Data Path: D:\LCH\DATA\0139\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0139
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-308-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	17.32	349089	15055	49.995
2	20.90	349154	10714	50.005
		698243	25769	100.000

Peak rejection level: 10000

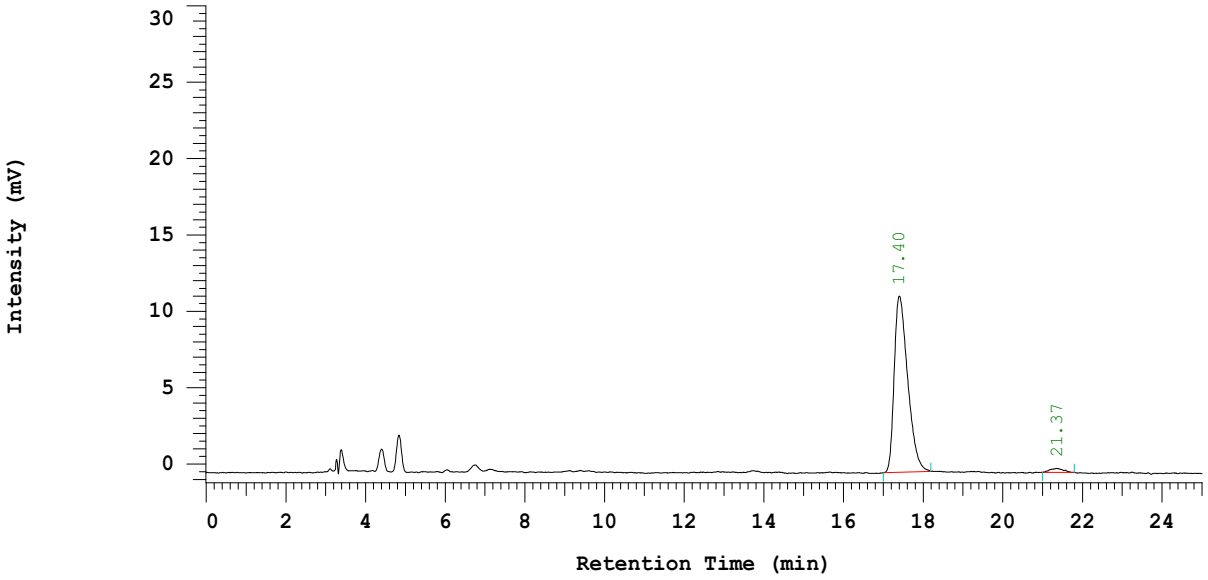
Fig S260. HPLC analysis of the racemic compound syn-3a, as a standard for comparison (Table 3, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/23 09:57 下午
Reported Date and Time: 2015/04/23 11:43 下午
Processed Date and Time: 2015/04/23 11:43 下午
Data Path: D:\LCH\DATA\0140\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0140
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-308-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	17.40	269907	11531	97.826
2	21.37	5997	263	2.174
		275904	11794	100.000

Peak rejection level: 1000

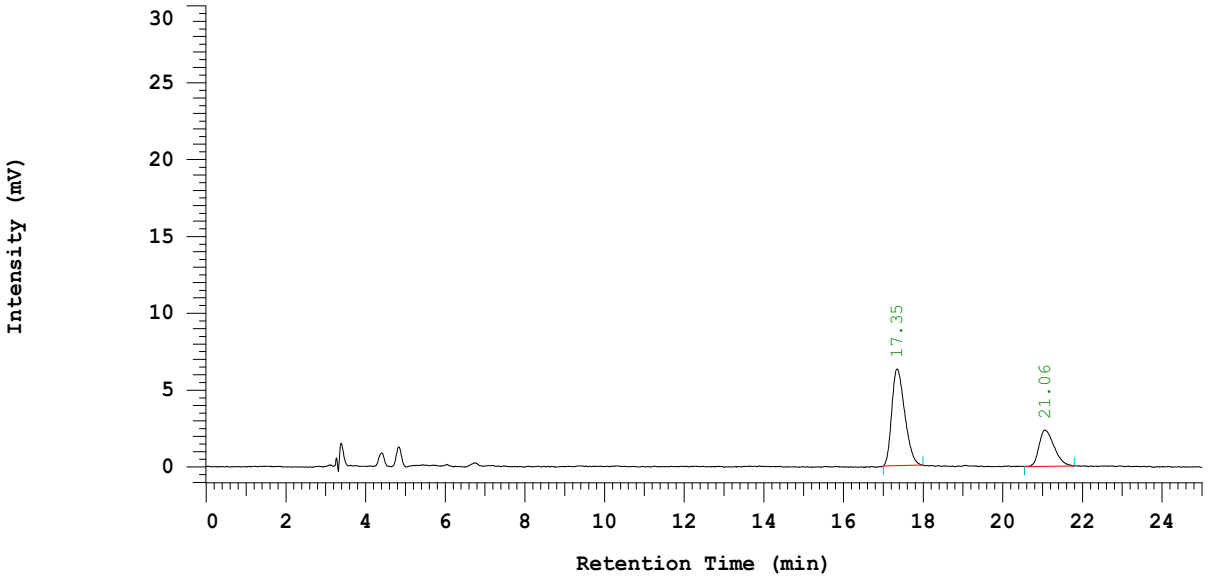
Fig S261 HPLC analysis of the chiral compound syn-3a obtained, (Table 3, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/23 10:24 下午
Reported Date and Time: 2015/04/23 10:52 下午
Processed Date and Time: 2015/04/23 10:52 下午
Data Path: D:\LCH\DATA\0141\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0141
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-308-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	17.35	140953	6284	69.560
2	21.06	61683	2357	30.440
		202636	8641	100.000

Peak rejection level: 1000

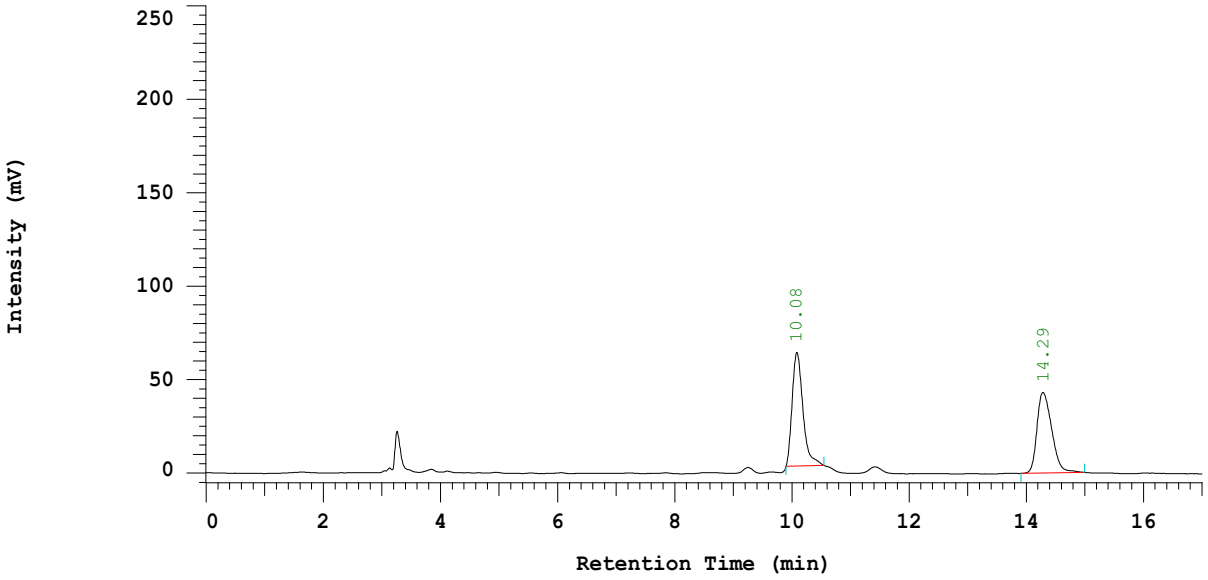
Fig S262. HPLC analysis of the mixture of chiral compound syn-3a obtained and the racemic compound syn-3a, for comparison (Table 3, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/23 11:47 下午
Reported Date and Time: 2015/04/24 12:14 上午
Processed Date and Time: 2015/04/24 12:14 上午
Data Path: D:\LCH\DATA\0144\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0144
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-308-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.08	776979	60682	50.597
2	14.29	758640	43194	49.403
		1535619	103876	100.000

Peak rejection level: 1000

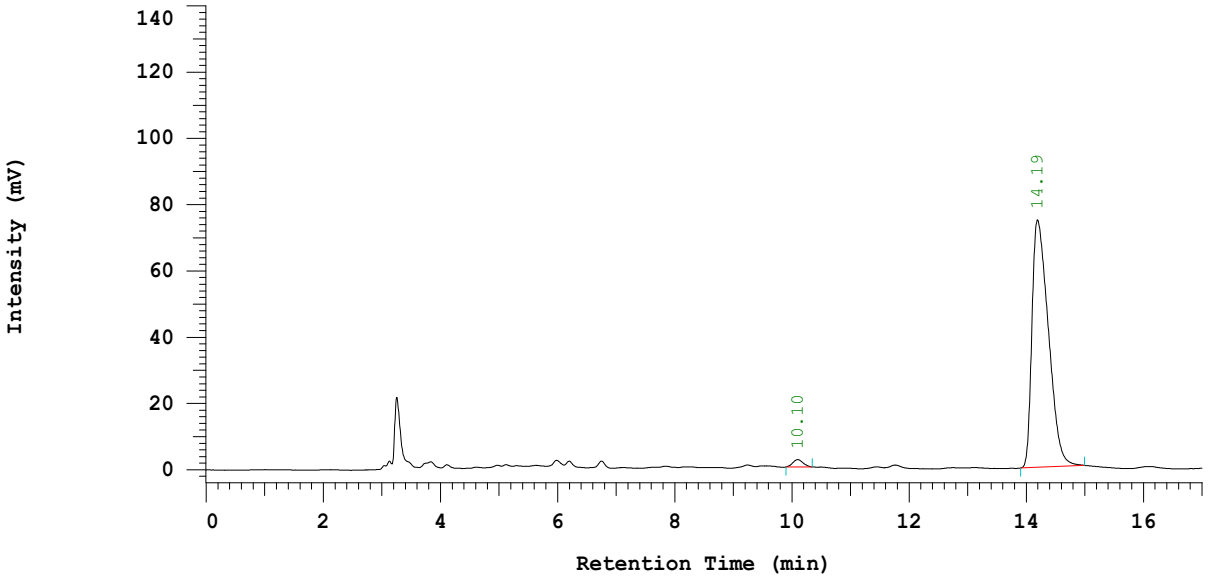
Fig S263. HPLC analysis of the racemic compound anti-3a, as a standard for comparison (Table 3, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/23 11:08 下午
Reported Date and Time: 2015/05/14 09:58 下午
Processed Date and Time: 2015/05/14 09:57 下午
Data Path: D:\LCH\DATA\0142\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0142
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-308-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.10	27073	2231	1.820
2	14.19	1460646	74626	98.180
		1487719	76857	100.000

Peak rejection level: 1000

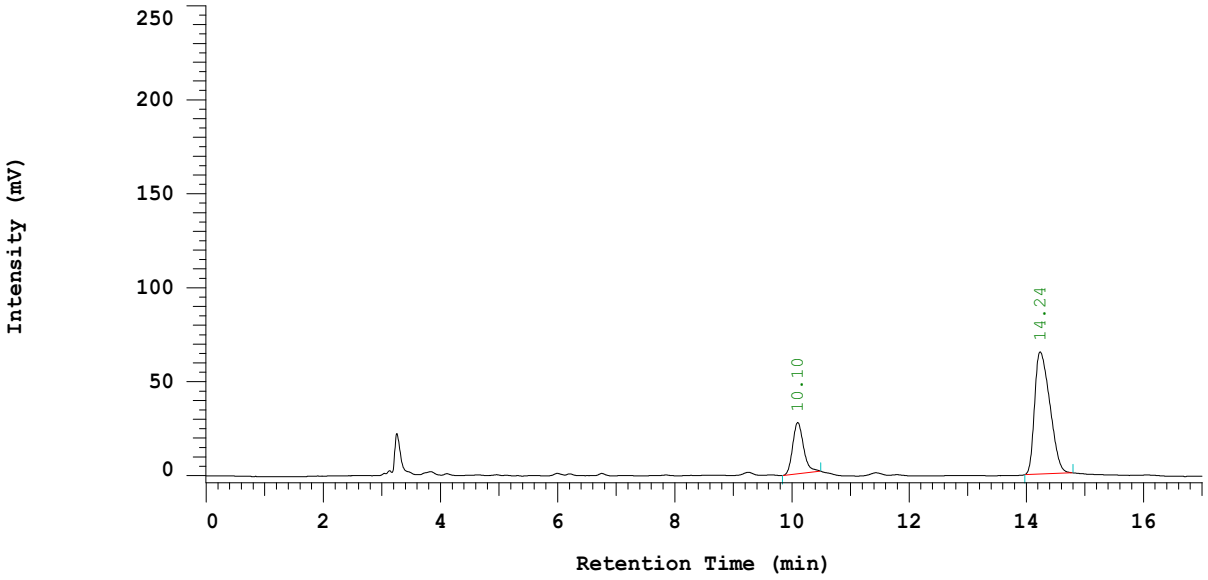
Fig S264. HPLC analysis of the chiral compound anti-3a obtained, (Table 3, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/23 11:28 下午
Reported Date and Time: 2015/04/23 11:53 下午
Processed Date and Time: 2015/04/23 11:53 下午
Data Path: D:\LCH\DATA\0143\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0143
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-308-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	10.10	351832	27301	22.987
2	14.24	1178714	64963	77.013
		1530546	92264	100.000

Peak rejection level: 1000

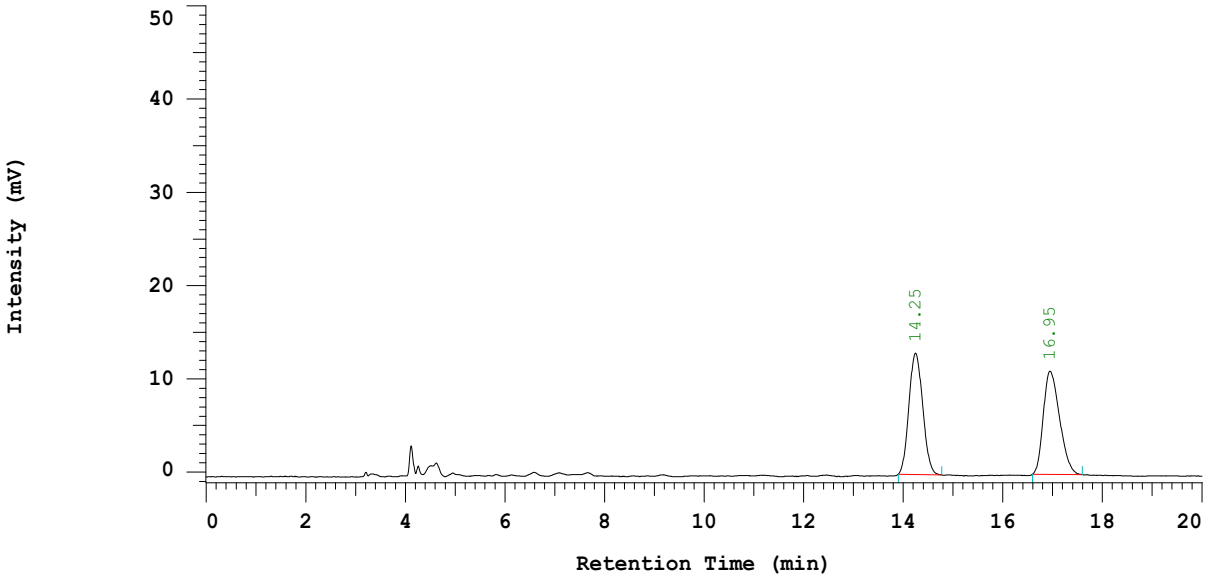
Fig S265. HPLC analysis of the mixture of chiral compound anti-3a obtained and the racemic compound anti-3a, for comparison (Table 3, entry 1).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 03:20 下午
Reported Date and Time: 2015/05/01 03:44 下午
Processed Date and Time: 2015/05/01 03:43 下午
Data Path: D:\LCH\DATA\0186\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0186
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-375-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	14.25	251538	13003	50.088
2	16.95	250657	11062	49.912
		502195	24065	100.000

Peak rejection level: 1000

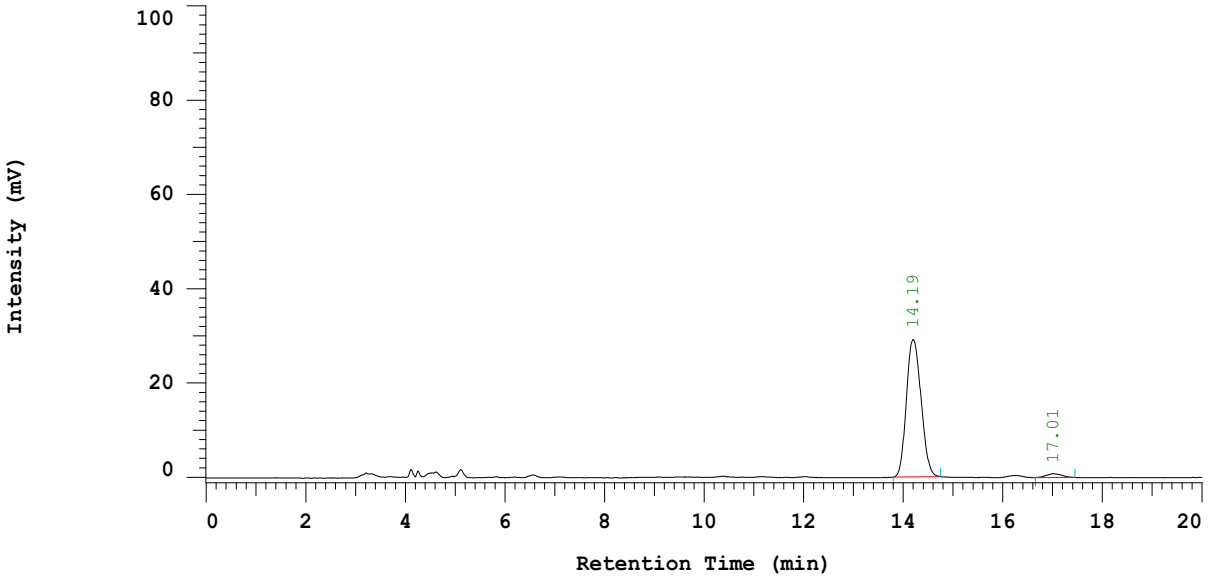
Fig S266. HPLC analysis of the racemic compound syn-3b, as a standard for comparison (Table 3, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 03:41 下午
Reported Date and Time: 2015/05/01 04:07 下午
Processed Date and Time: 2015/05/01 04:07 下午
Data Path: D:\LCH\DATA\0187\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0187
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-375-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	14.19	595134	29120	97.351
2	17.01	16191	775	2.649
		611325	29895	100.000

Peak rejection level: 1000

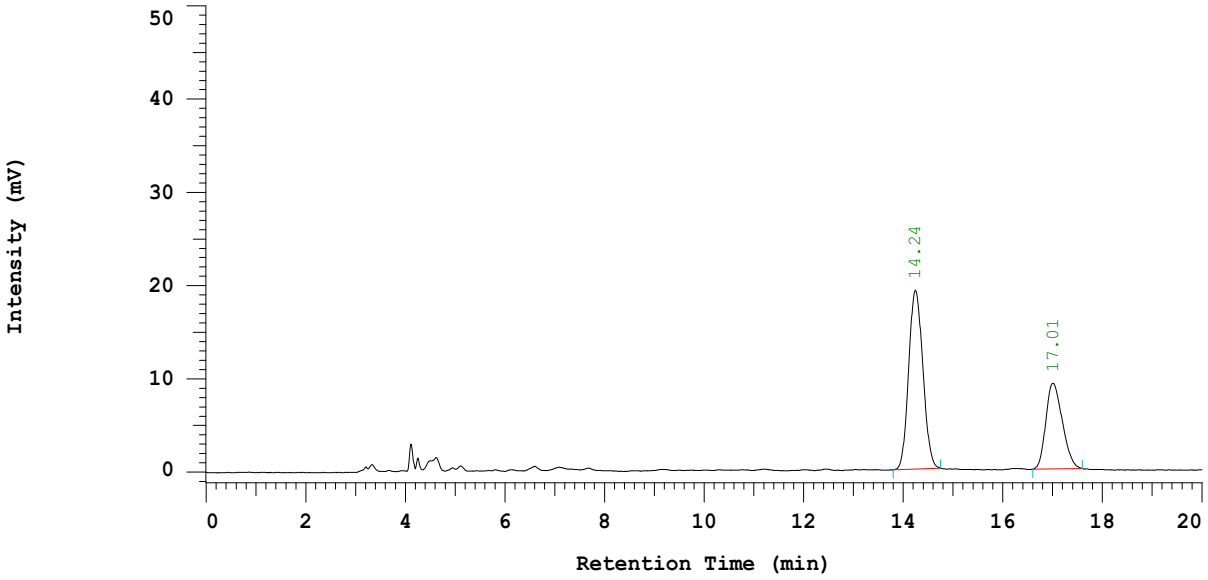
Fig S267. HPLC analysis of the chiral compound syn-3b obtained, (Table 3, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 04:02 下午
Reported Date and Time: 2015/05/01 04:27 下午
Processed Date and Time: 2015/05/01 04:27 下午
Data Path: D:\LCH\DATA\0188\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0188
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-375-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	14.24	380977	19164	64.287
2	17.01	211643	9184	35.713
		592620	28348	100.000

Peak rejection level: 1000

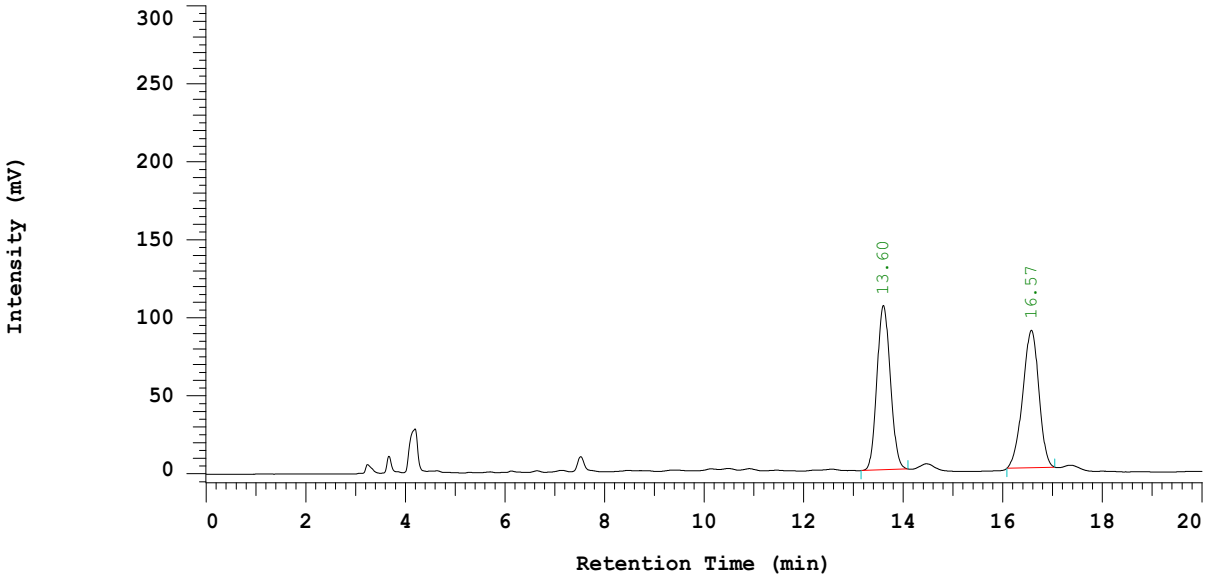
Fig S268. HPLC analysis of the mixture of chiral compound syn-3b obtained and the racemic compound syn-3b for comparison (Table 3, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 04:54 下午
Reported Date and Time: 2015/05/01 05:20 下午
Processed Date and Time: 2015/05/01 05:19 下午
Data Path: D:\LCH\DATA\0189\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0189
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-375-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.60	1969948	105280	49.658
2	16.57	1997048	88086	50.342
		3966996	193366	100.000

Peak rejection level: 1000

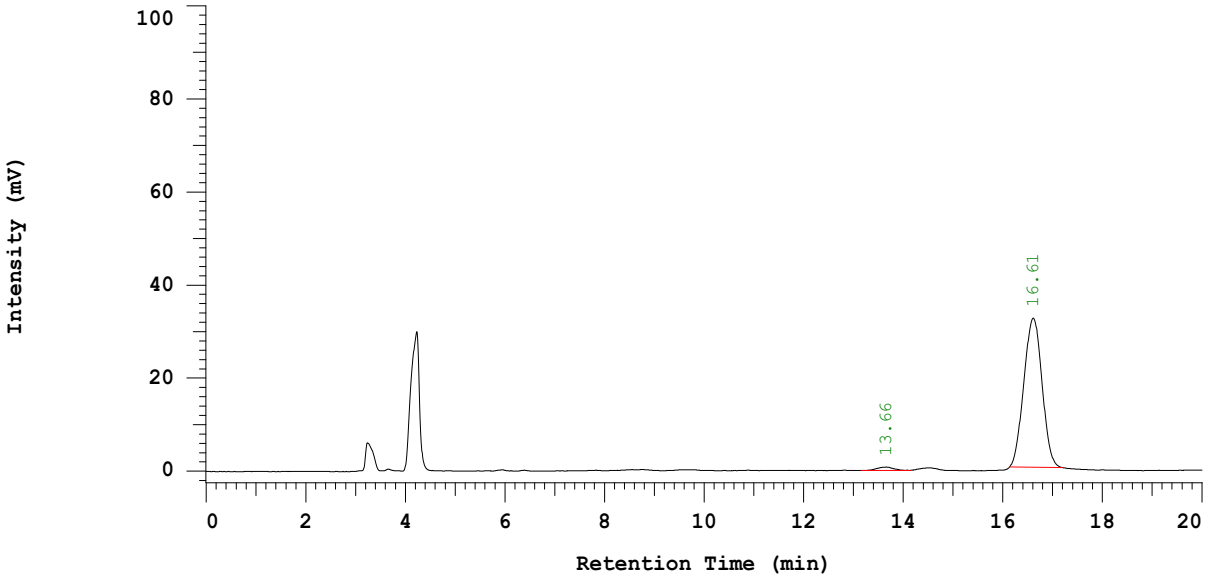
Fig S269. HPLC analysis of the racemic compound anti-3b, as a standard for comparison (Table 3, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 07:04 下午
Reported Date and Time: 2015/05/01 07:33 下午
Processed Date and Time: 2015/05/01 07:32 下午
Data Path: D:\LCH\DATA\0193\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0193
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-375-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.66	16837	730	2.013
2	16.61	819775	32016	97.987
		836612	32746	100.000

Peak rejection level: 1000

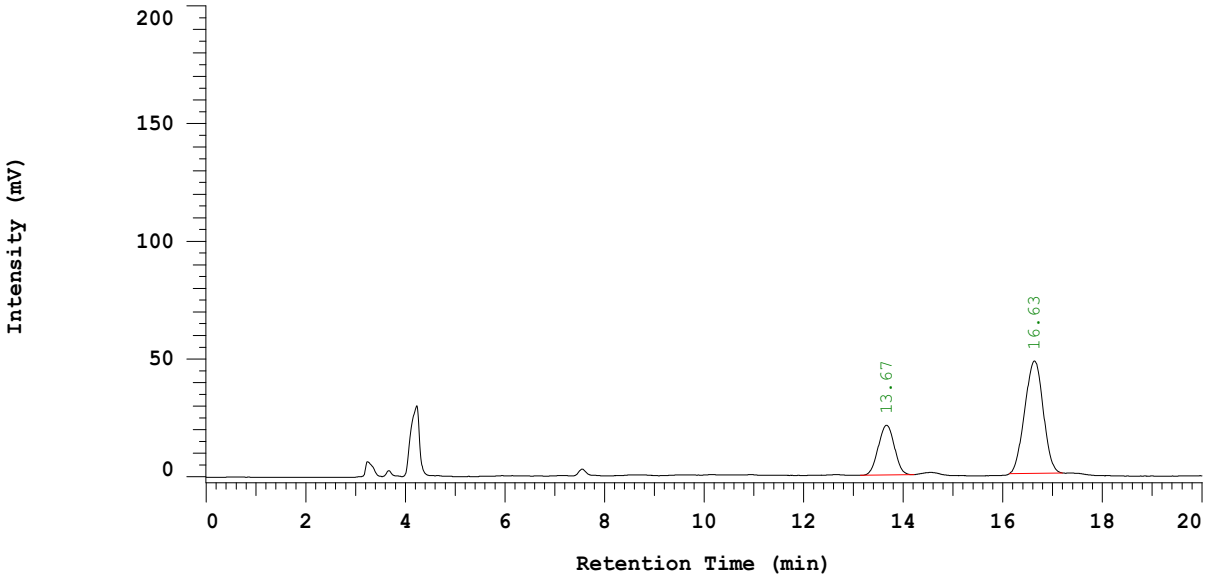
Fig S270. HPLC analysis of the chiral compound anti-3b obtained, (Table 3, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 07:26 下午
Reported Date and Time: 2015/05/01 07:49 下午
Processed Date and Time: 2015/05/01 07:49 下午
Data Path: D:\LCH\DATA\0194\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0194
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-375-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.67	474193	21059	27.698
2	16.63	1237803	47739	72.302
		1711996	68798	100.000

Peak rejection level: 1000

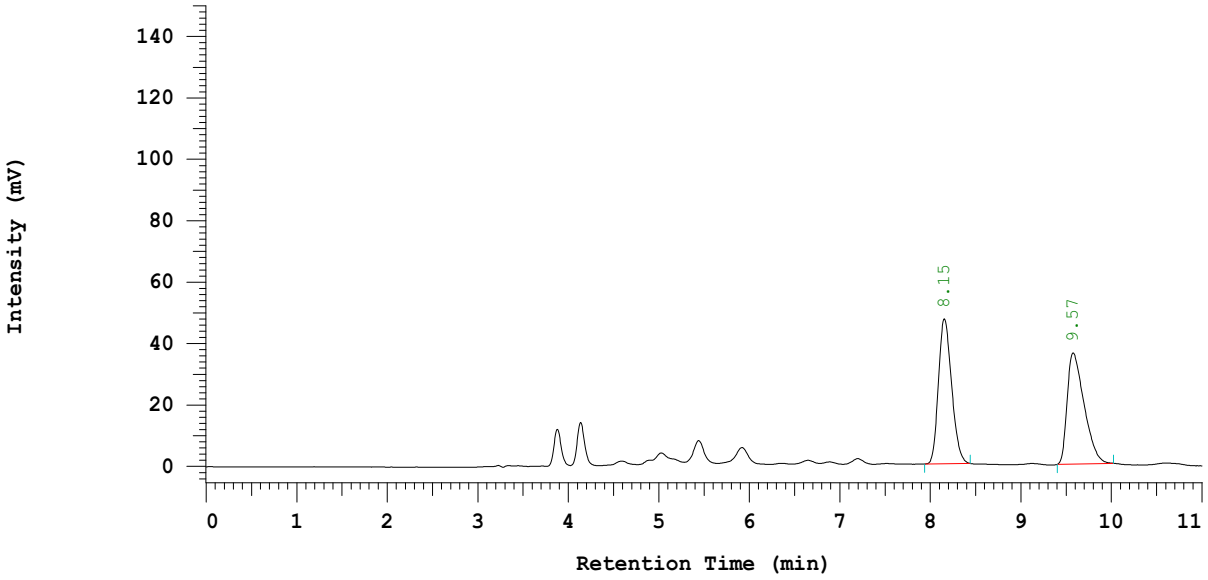
Fig S271. HPLC analysis of the mixture of chiral compound anti-3b obtained and the racemic compound anti-3b, for comparison (Table 3, entry 2).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/24 12:38 下午
Reported Date and Time: 2015/04/24 12:55 下午
Processed Date and Time: 2015/04/24 12:55 下午
Data Path: D:\LCH\DATA\0145\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0145
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-381-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.15	475814	47191	50.375
2	9.57	468739	36175	49.625
		944553	83366	100.000

Peak rejection level: 1000

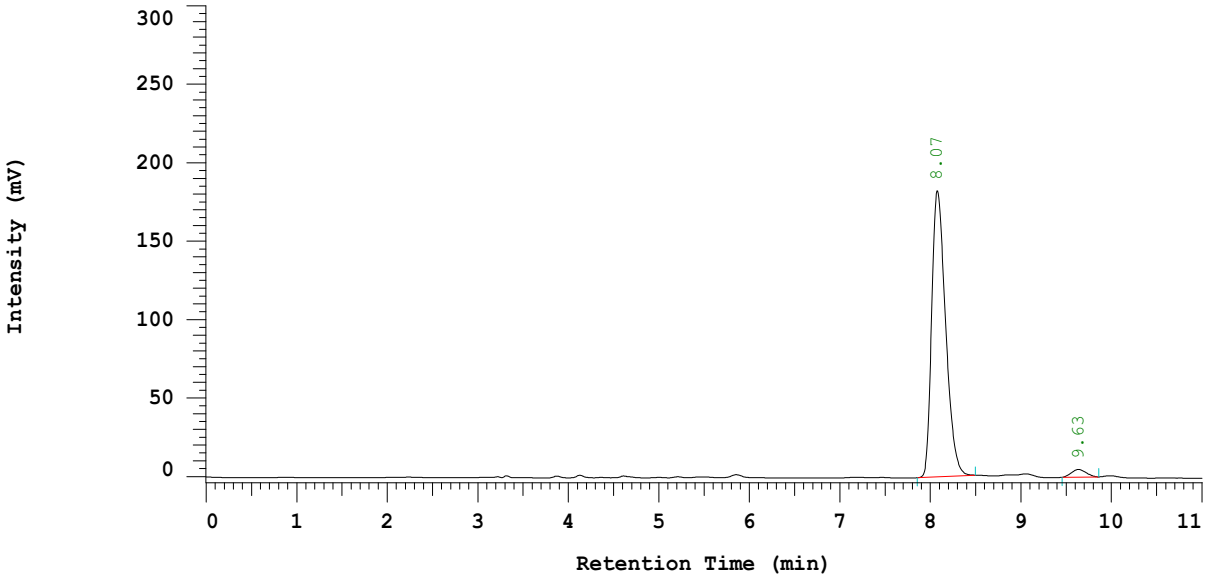
Fig S272. HPLC analysis of the racemic compound syn-3c, as a standard for comparison (Table 3, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/24 12:51 下午
Reported Date and Time: 2015/05/14 10:02 下午
Processed Date and Time: 2015/05/14 10:00 下午
Data Path: D:\LCH\DATA\0146\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0146
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-381-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.07	1943130	182237	97.360
2	9.63	52688	4777	2.640
		1995818	187014	100.000

Peak rejection level: 1000

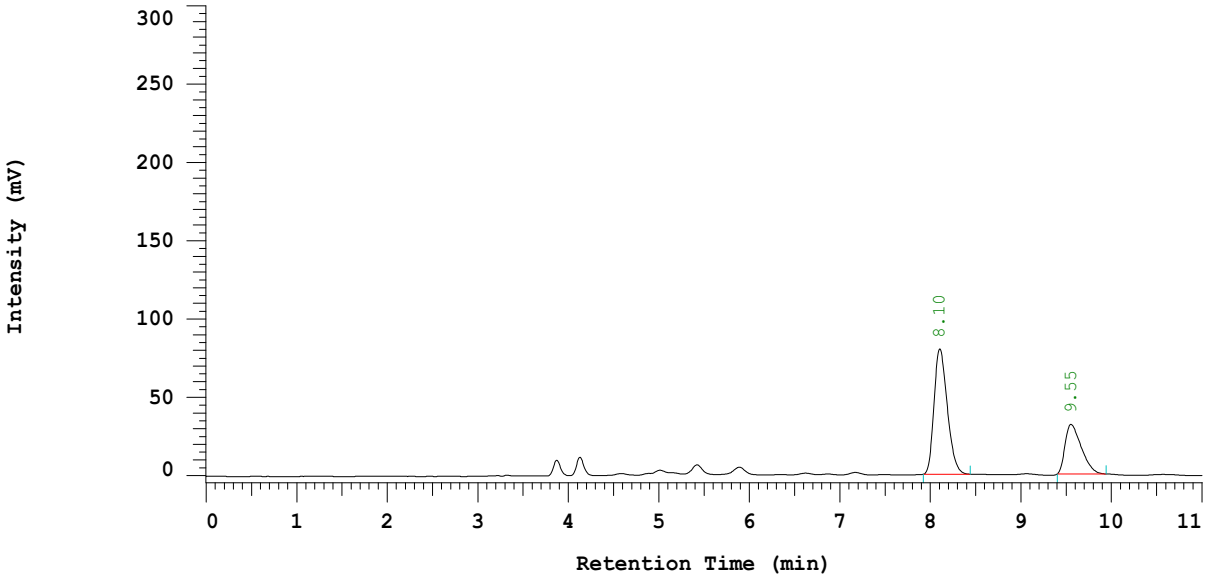
Fig S273. HPLC analysis of the chiral compound syn-3c obtained, (Table 3, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/24 01:06 下午
Reported Date and Time: 2015/04/24 01:21 下午
Processed Date and Time: 2015/04/24 01:20 下午
Data Path: D:\LCH\DATA\0147\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0147
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-381-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.10	814219	80026	67.363
2	9.55	394492	31670	32.637
		1208711	111696	100.000

Peak rejection level: 1000

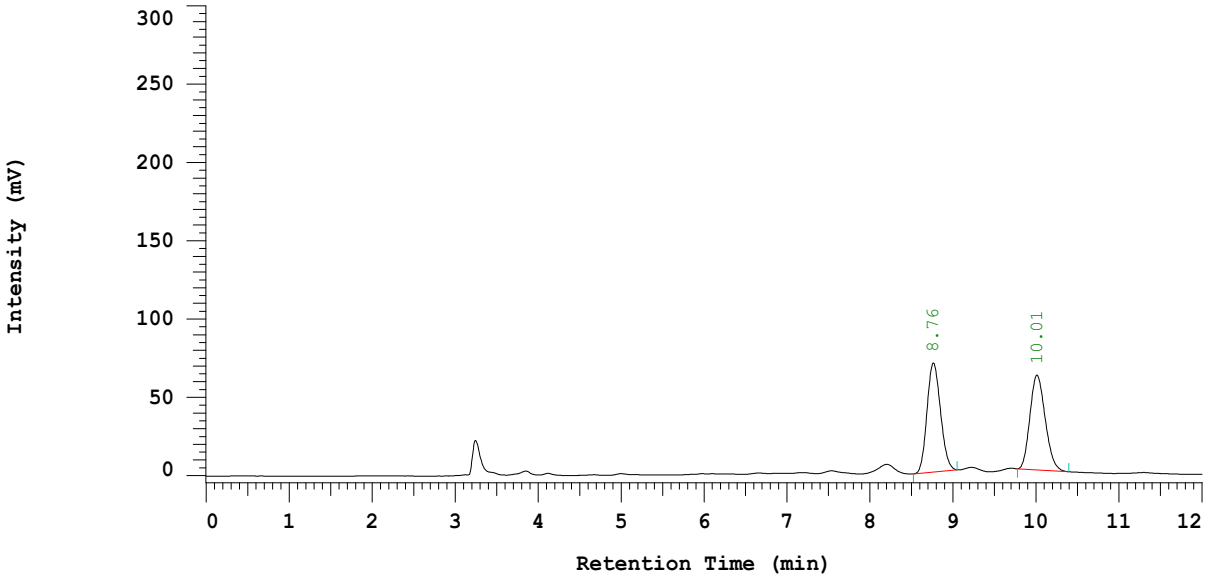
Fig S274. HPLC analysis of the mixture of chiral compound syn-3c obtained and the racemic compound syn-3c, for comparison (Table 3, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/24 02:45 下午
Reported Date and Time: 2015/04/24 03:01 下午
Processed Date and Time: 2015/04/24 03:01 下午
Data Path: D:\LCH\DATA\0149\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0149
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-381-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.76	789721	69654	50.269
2	10.01	781268	60538	49.731
		1570989	130192	100.000

Peak rejection level: 1000

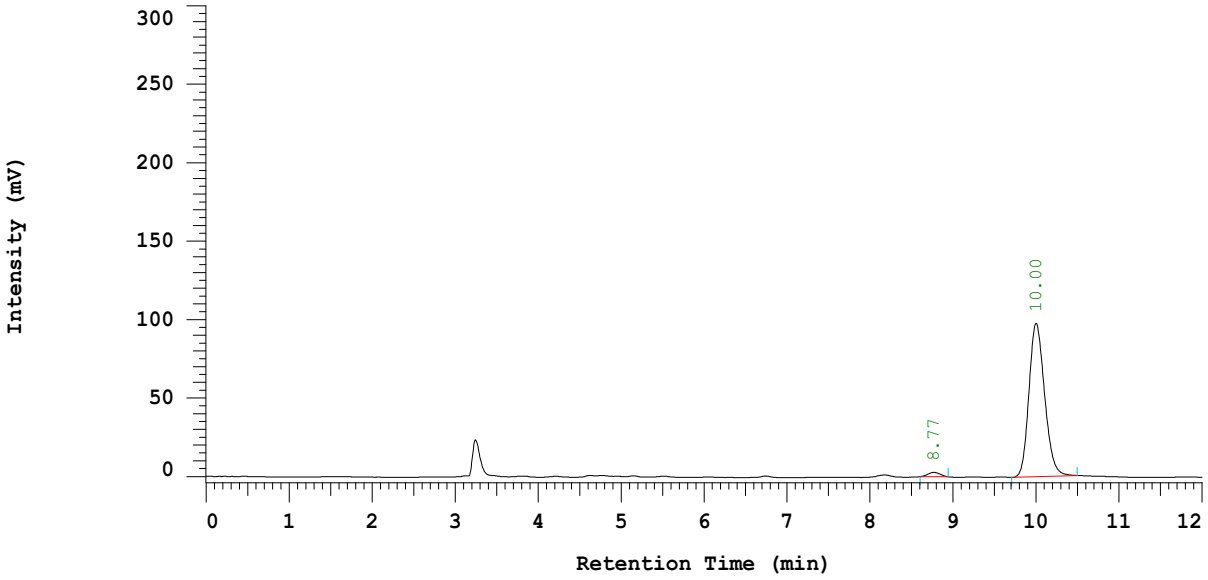
Fig S275. HPLC analysis of the racemic compound anti-3c, as a standard for comparison (Table 3, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/24 02:59 下午
Reported Date and Time: 2015/05/14 10:17 下午
Processed Date and Time: 2015/05/14 10:16 下午
Data Path: D:\LCH\DATA\0150\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0150
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-381-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.77	26996	2671	2.055
2	10.00	1286640	97810	97.945
		1313636	100481	100.000

Peak rejection level: 1000

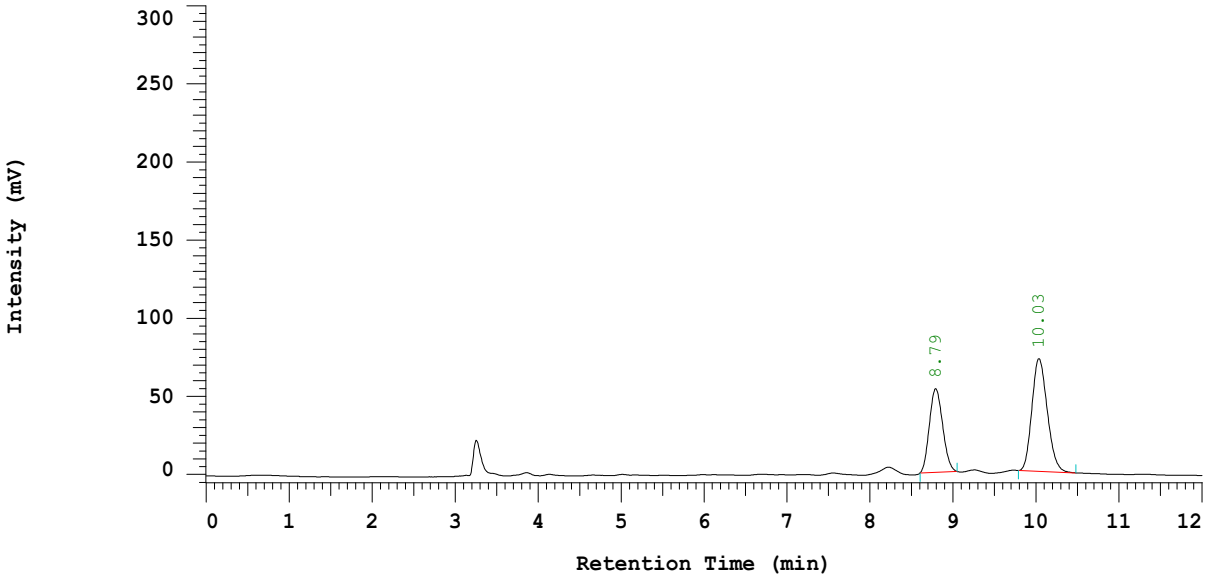
Fig S276. HPLC analysis of the chiral compound anti-3c obtained, (Table 3, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/24 03:15 下午
Reported Date and Time: 2015/04/24 03:57 下午
Processed Date and Time: 2015/04/24 03:56 下午
Data Path: D:\LCH\DATA\0151\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0151
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-381-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.79	596010	53615	38.921
2	10.03	935315	72186	61.079
		1531325	125801	100.000

Peak rejection level: 1000

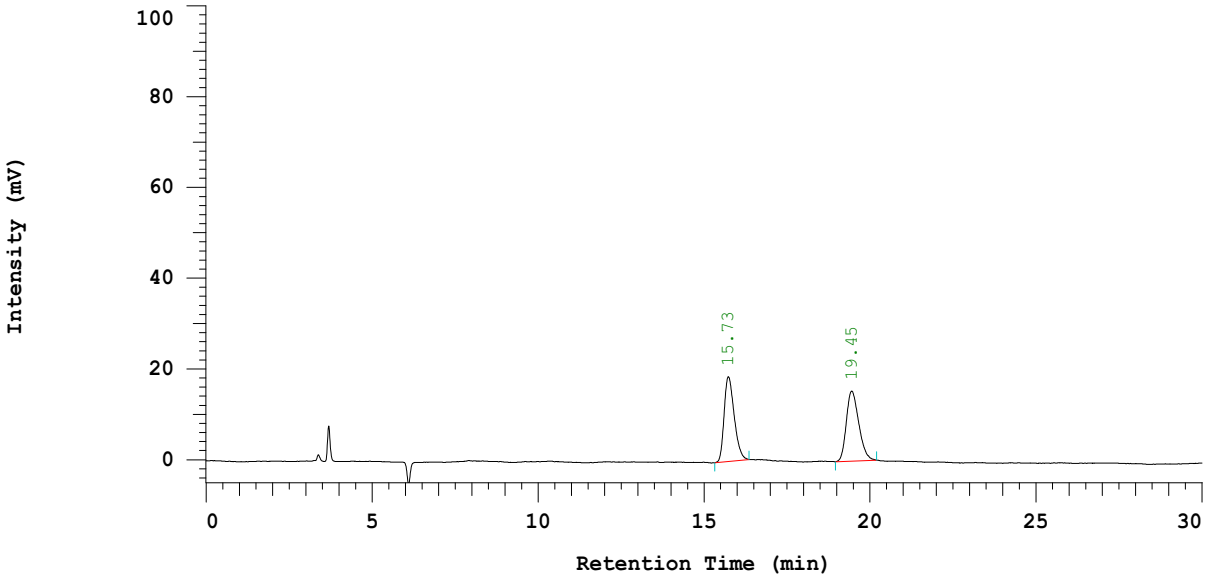
Fig S277. HPLC analysis of the mixture of chiral compound anti-3c obtained and the racemic compound anti-3c, for comparison (Table 3, entry 3).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/01/20 11:17 下午
Reported Date and Time: 2015/01/21 12:36 上午
Processed Date and Time: 2015/01/21 12:35 上午
Data Path: D:\LCH\DATA\0017\
Processing Method: test-Ea/Hx

System (acquisition): Sys 1
Series: 0017
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-364-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	15.73	390216	18660	49.534
2	19.45	397564	15420	50.466
		787780	34080	100.000

Peak rejection level: 1000

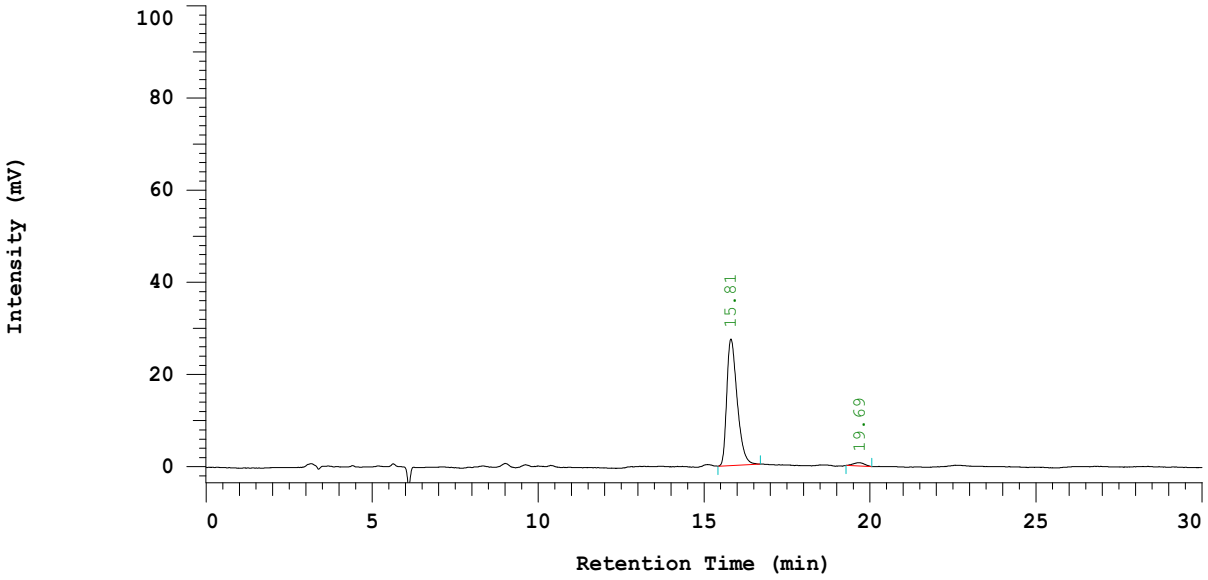
Fig S278. HPLC analysis of the racemic compound syn-3d, as a standard for comparison (Table 3, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/01/20 11:48 下午
Reported Date and Time: 2015/04/15 09:30 下午
Processed Date and Time: 2015/04/15 09:29 下午
Data Path: D:\LCH\DATA\0018\
Processing Method: test-Ea/Hx

System (acquisition): Sys 1
Series: 0018
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-364-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	15.81	593048	27455	97.136
2	19.69	17486	734	2.864
		610534	28189	100.000

Peak rejection level: 10000

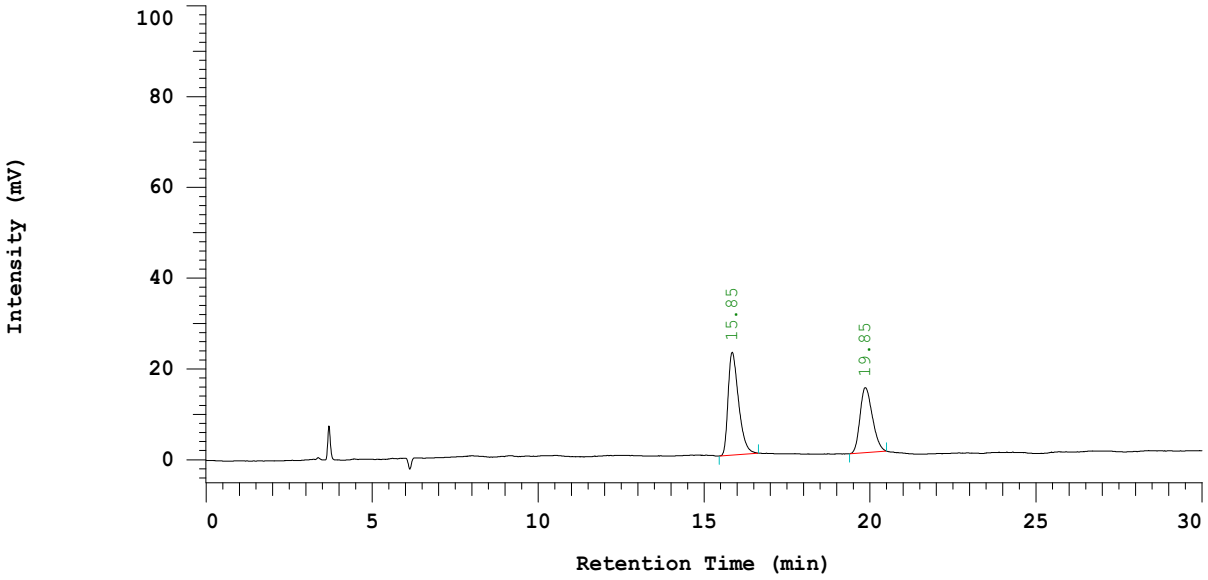
Fig S279. HPLC analysis of the chiral compound syn-3d obtained, (Table 3, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/01/21 01:43 下午
Reported Date and Time: 2015/01/21 02:23 下午
Processed Date and Time: 2015/01/21 02:23 下午
Data Path: D:\LCH\DATA\0020\
Processing Method: test-Ea/Hx

System (acquisition): Sys 1
Series: 0020
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-364-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	15.85	491706	22616	57.011
2	19.85	370764	14324	42.989
		862470	36940	100.000

Peak rejection level: 1000

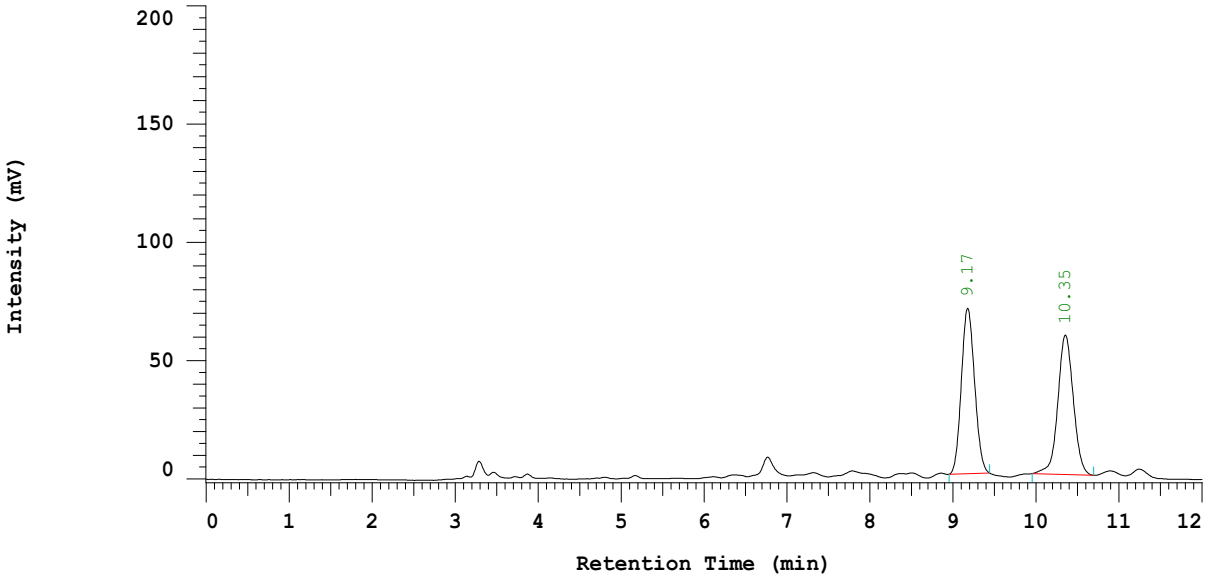
Fig S280. HPLC analysis of the mixture of chiral compound syn-3d obtained and the racemic compound syn-3d, for comparison (Table 3, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/16 03:50 下午
Reported Date and Time: 2015/04/16 04:09 下午
Processed Date and Time: 2015/04/16 04:09 下午
Data Path: D:\LCH\DATA\0125\
Processing Method: test-Ea/Hx

System (acquisition): Sys 1
Series: 0125
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-364-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.17	772922	70025	50.353
2	10.35	762097	58991	49.647
		1535019	129016	100.000

Peak rejection level: 20000

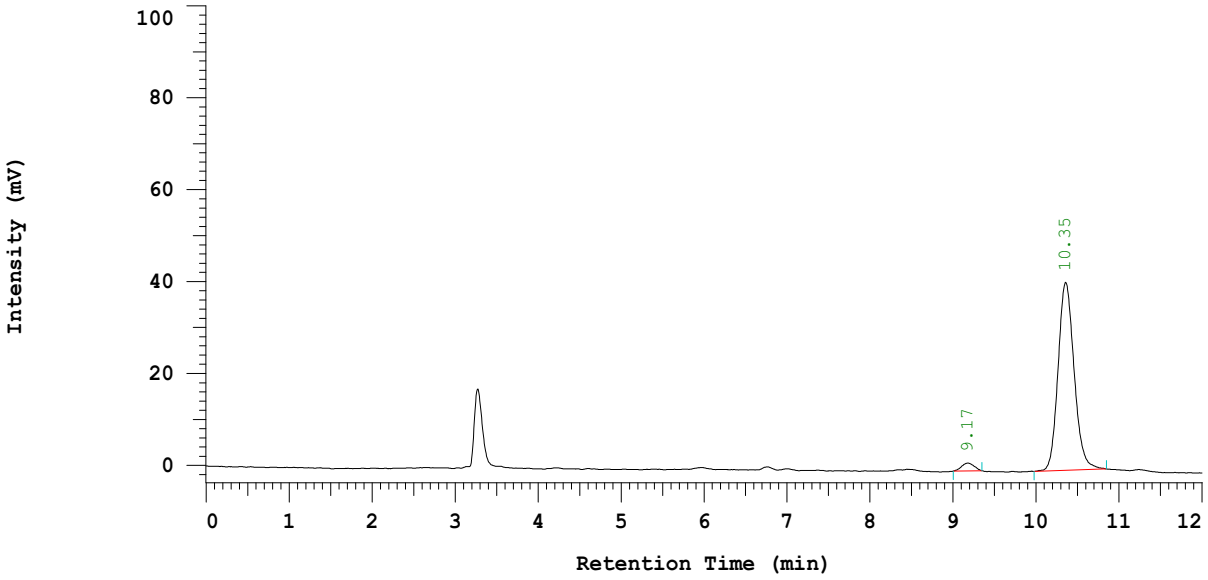
Fig S281. HPLC analysis of the racemic compound anti-3d, as a standard for comparison (Table 3, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/16 04:22 下午
Reported Date and Time: 2015/04/16 04:44 下午
Processed Date and Time: 2015/04/16 04:43 下午
Data Path: D:\LCH\DATA\0127\
Processing Method: test-Ea/Hx

System (acquisition): Sys 1
Series: 0127
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-364-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.17	18055	1738	3.189
2	10.35	548034	40901	96.811
		566089	42639	100.000

Peak rejection level: 10000

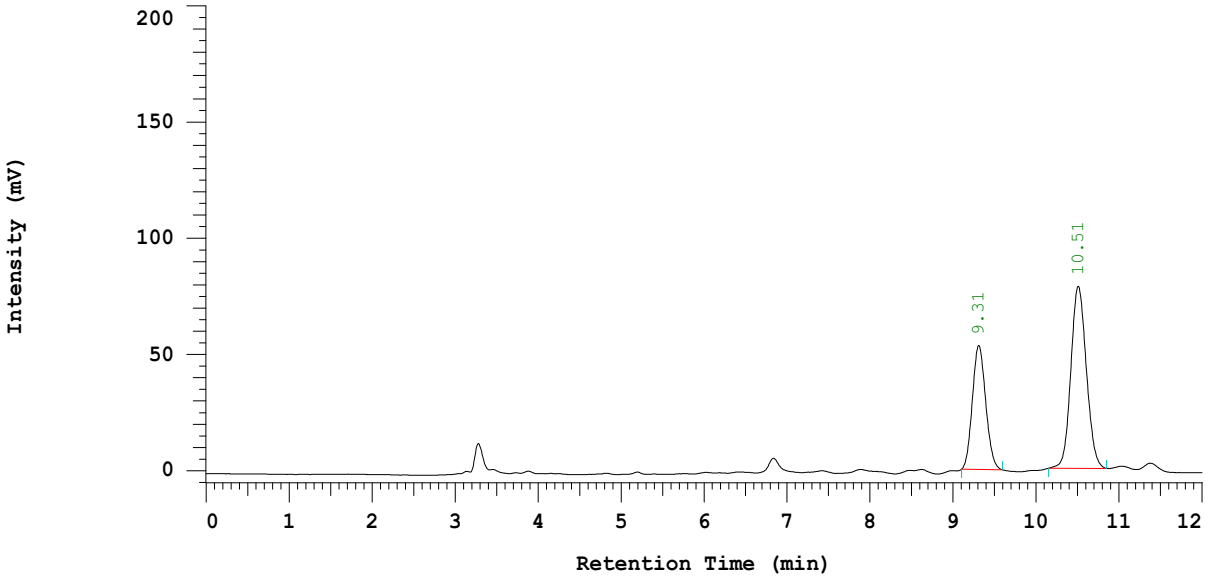
Fig S282. HPLC analysis of the chiral compound anti-3d obtained, (Table 3, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/16 04:38 下午
Reported Date and Time: 2015/04/16 04:54 下午
Processed Date and Time: 2015/04/16 04:54 下午
Data Path: D:\LCH\DATA\0128\
Processing Method: test-Ea/Hx

System (acquisition): Sys 1
Series: 0128
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-364-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: test-Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.31	601249	53299	37.178
2	10.51	1015947	78309	62.822
		1617196	131608	100.000

Peak rejection level: 10000

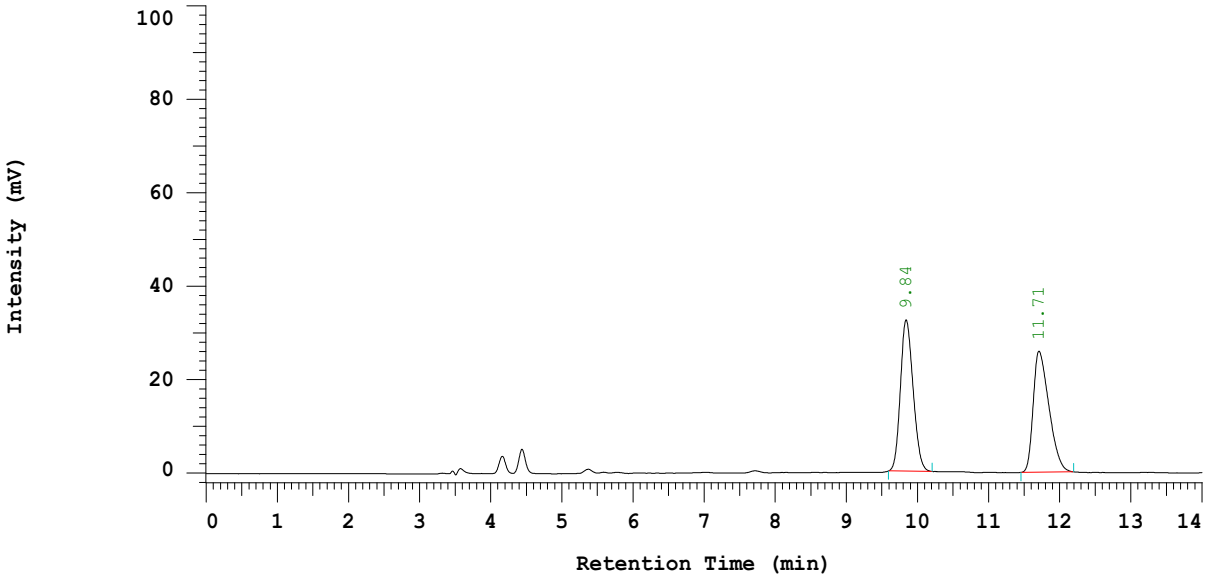
Fig S283. HPLC analysis of the mixture of chiral compound anti-3d obtained and the racemic compound anti-3d, for comparison (Table 3, entry 4).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/27 07:56 下午
Reported Date and Time: 2015/04/27 08:15 下午
Processed Date and Time: 2015/04/27 08:15 下午
Data Path: D:\LCH\DATA\0155\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0155
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-382-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.84	400794	32380	50.000
2	11.71	400799	25885	50.000
		801593	58265	100.000

Peak rejection level: 1000

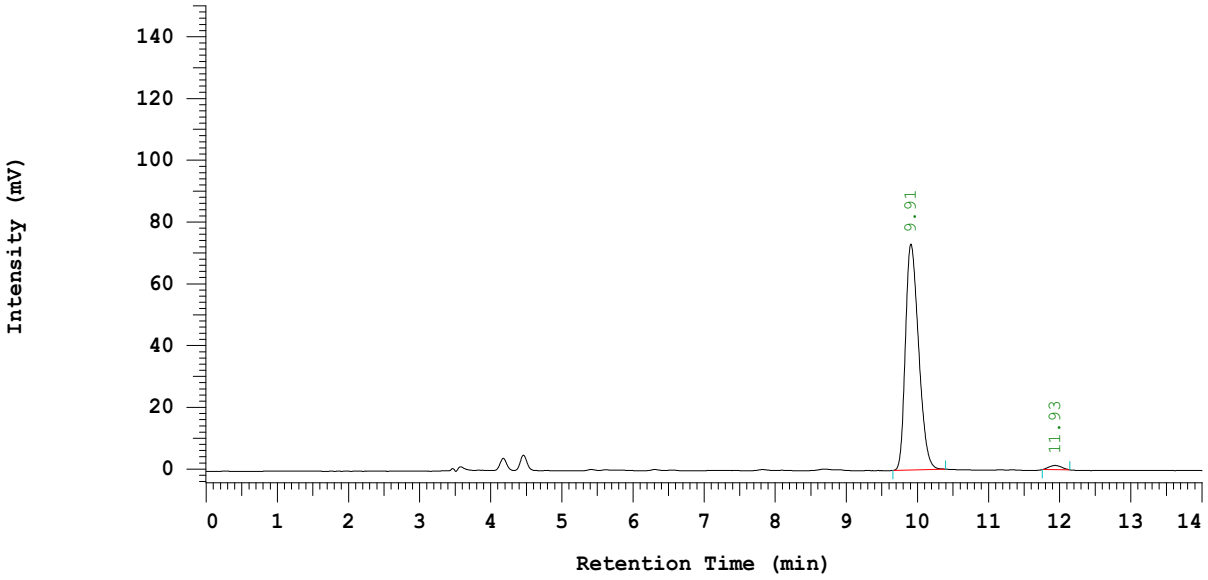
Fig S284. HPLC analysis of the racemic compound syn-3e, as a standard for comparison (Table 3, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/27 08:12 下午
Reported Date and Time: 2015/04/27 08:34 下午
Processed Date and Time: 2015/04/27 08:33 下午
Data Path: D:\LCH\DATA\0156\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0156
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-382-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.91	939737	73189	98.168
2	11.93	17541	1400	1.832
		957278	74589	100.000

Peak rejection level: 1000

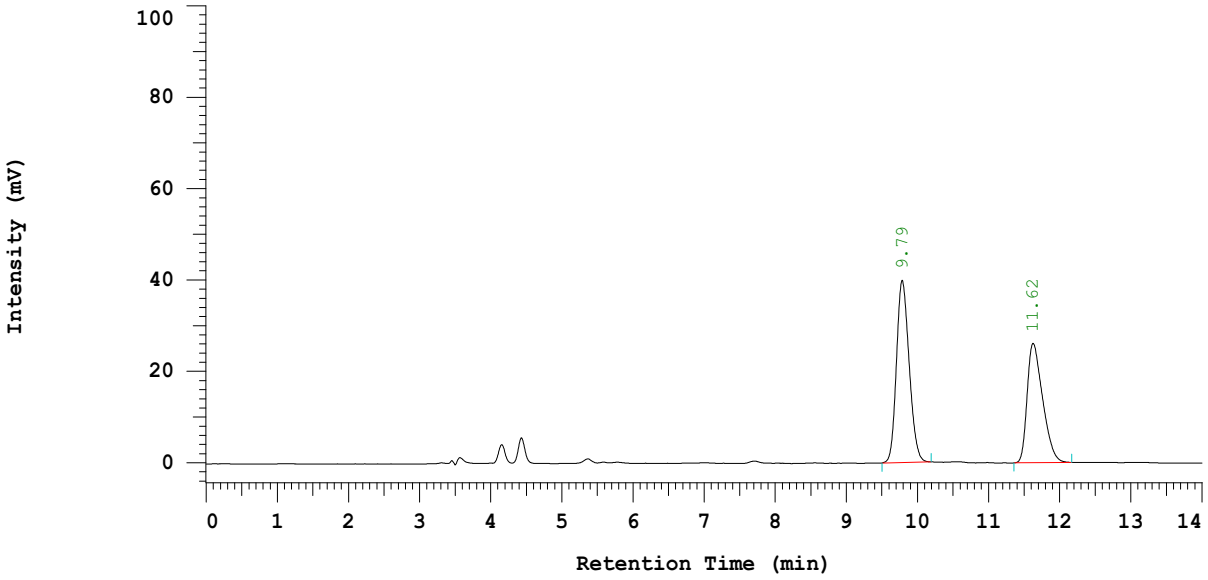
Fig S285. HPLC analysis of the chiral compound syn-3e obtained, (Table 3, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/27 08:29 下午
Reported Date and Time: 2015/04/27 08:48 下午
Processed Date and Time: 2015/04/27 08:48 下午
Data Path: D:\LCH\DATA\0157\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0157
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-382-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.79	493419	39842	55.418
2	11.62	396942	26126	44.582
		890361	65968	100.000

Peak rejection level: 1000

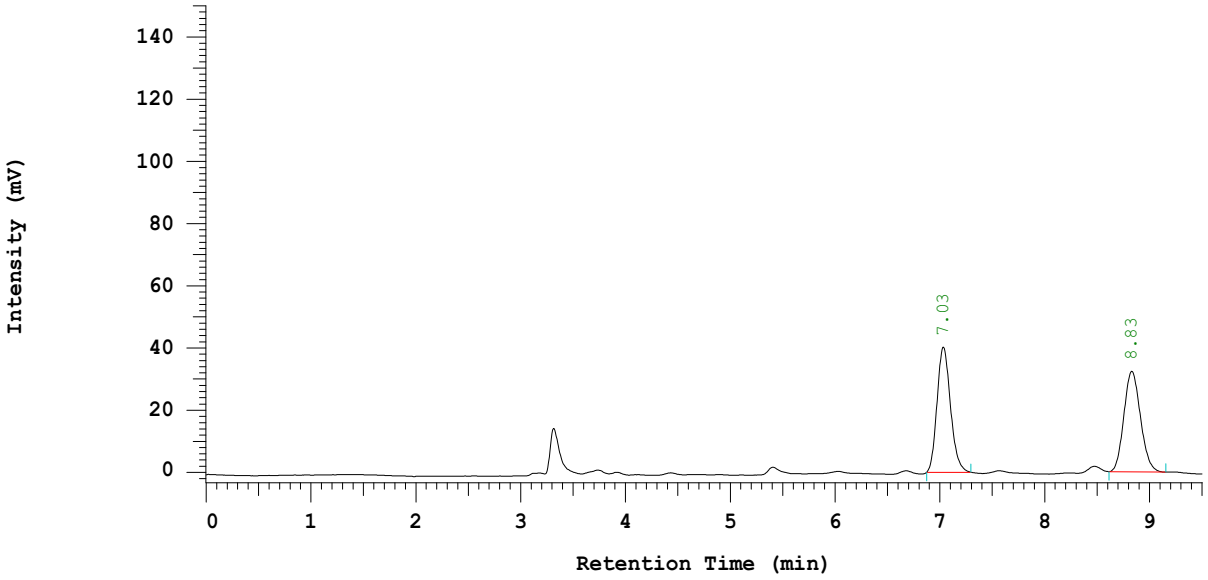
Fig S286. HPLC analysis of the mixture of chiral compound syn-3e obtained and the racemic compound syn-3e, for comparison (Table 3, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/27 10:53 下午
Reported Date and Time: 2015/04/27 11:07 下午
Processed Date and Time: 2015/04/27 11:07 下午
Data Path: D:\LCH\DATA\0164\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0164
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-382-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 20%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	7.03	357929	40262	50.187
2	8.83	355262	32294	49.813
		713191	72556	100.000

Peak rejection level: 1000

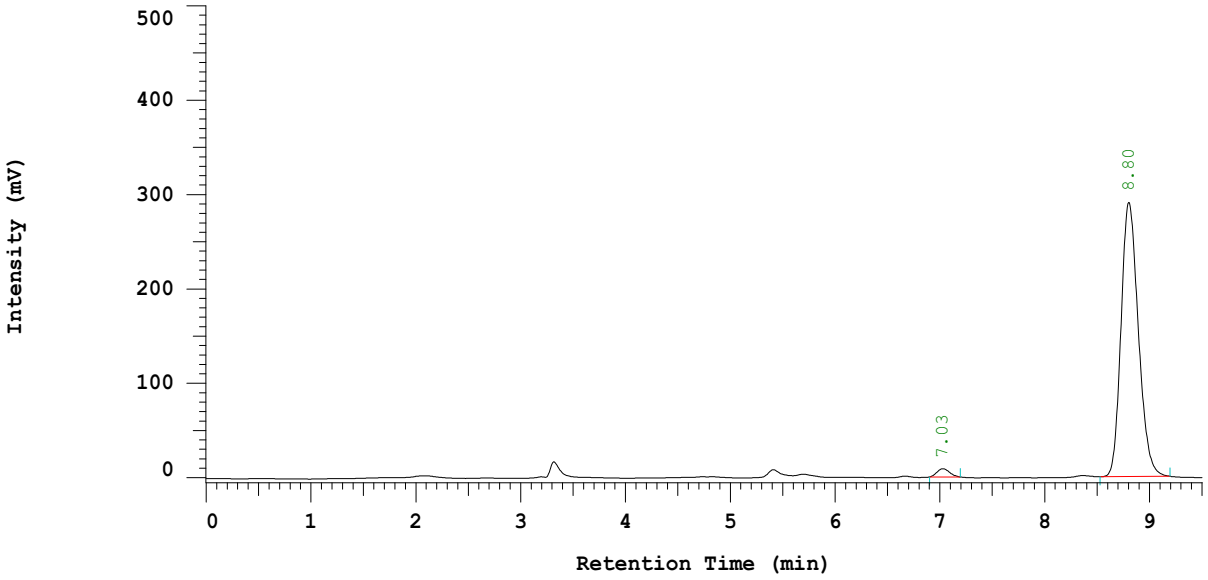
Fig S287. HPLC analysis of the racemic compound anti-3e, as a standard for comparison (Table 3, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/27 10:20 下午
Reported Date and Time: 2015/04/27 10:37 下午
Processed Date and Time: 2015/04/27 10:36 下午
Data Path: D:\LCH\DATA\0162\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0162
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-382-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 20%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	7.03	70715	8640	2.084
2	8.80	3322014	290282	97.916
		3392729	298922	100.000

Peak rejection level: 1000

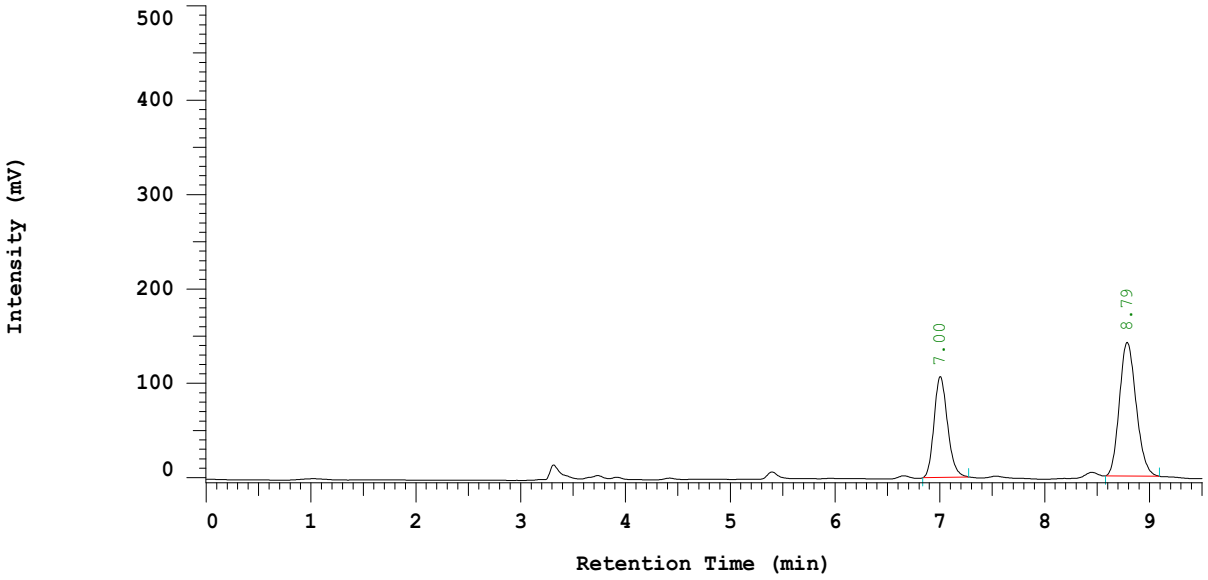
Fig S288. HPLC analysis of the chiral compound anti-3e obtained, (Table 3, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/27 10:33 下午
Reported Date and Time: 2015/04/27 10:48 下午
Processed Date and Time: 2015/04/27 10:48 下午
Data Path: D:\LCH\DATA\0163\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0163
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-382-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 20%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 254 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 254 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	7.00	949789	106573	37.741
2	8.79	1566822	141665	62.259
		2516611	248238	100.000

Peak rejection level: 1000

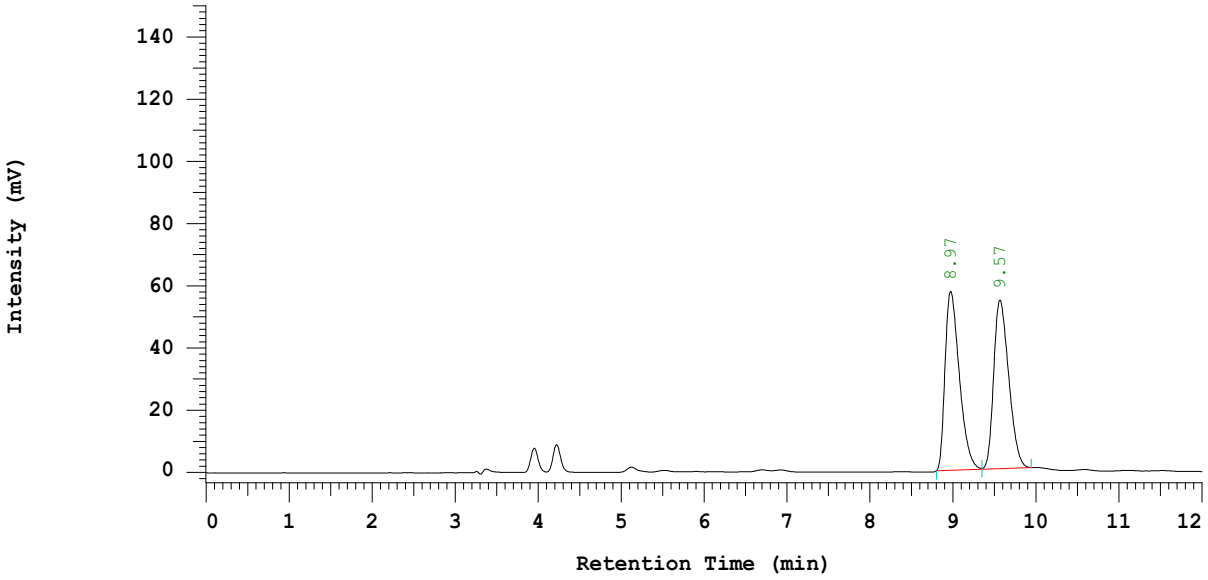
Fig S289. HPLC analysis of the mixture of chiral compound anti-3e obtained and the racemic compound anti-3e, for comparison (Table 3, entry 5).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 01:53 下午
Reported Date and Time: 2015/04/29 02:12 下午
Processed Date and Time: 2015/04/29 02:12 下午
Data Path: D:\LCH\DATA\0165\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0165
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-393-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.97	686258	57534	50.320
2	9.57	677535	54122	49.680
		1363793	111656	100.000

Peak rejection level: 1000

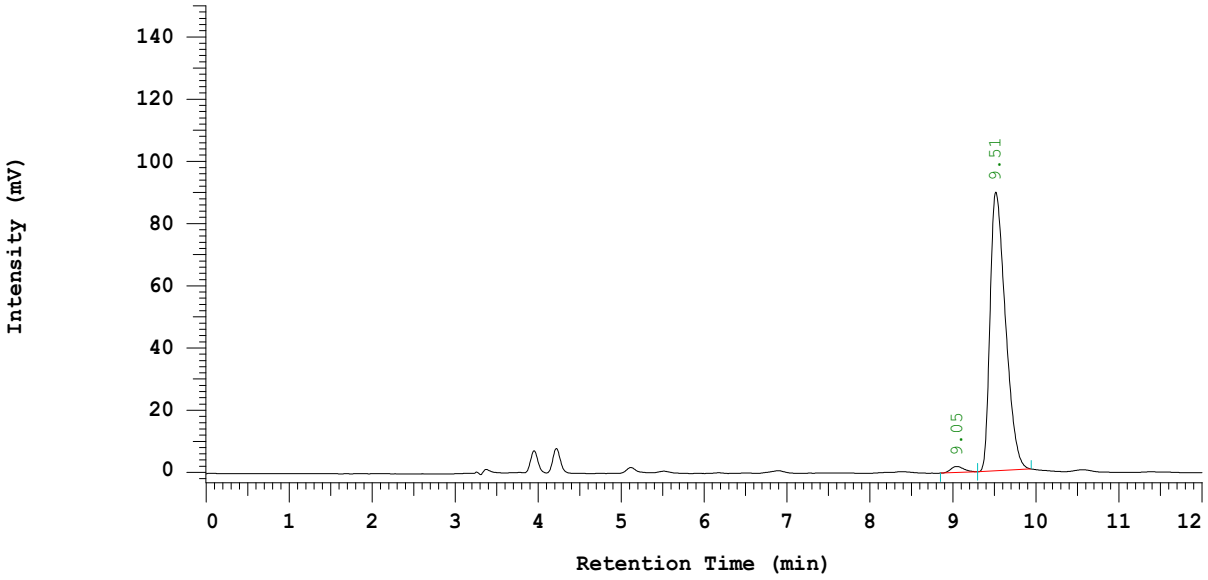
Fig S290. HPLC analysis of the racemic compound syn-3f, as a standard for comparison (Table 3, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 02:14 下午
Reported Date and Time: 2015/04/29 02:42 下午
Processed Date and Time: 2015/04/29 02:41 下午
Data Path: D:\LCH\DATA\0166\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0166
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-393-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.05	21871	1969	1.866
2	9.51	1150175	89553	98.134
		1172046	91522	100.000

Peak rejection level: 1000

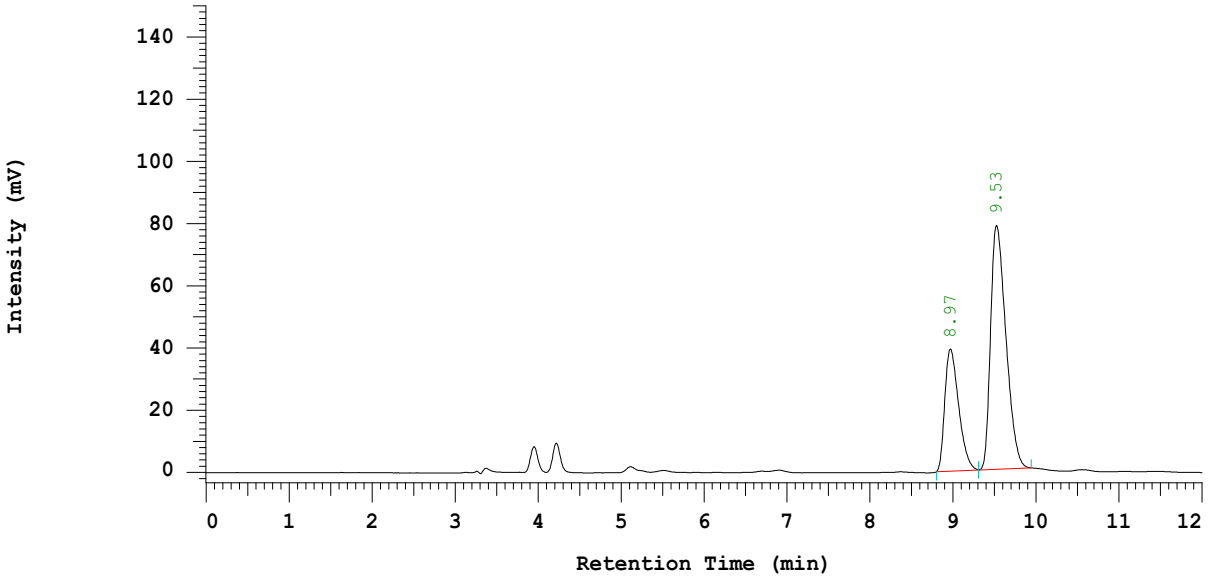
Fig S291. HPLC analysis of the chiral compound syn-3f obtained, (Table 3, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 02:30 下午
Reported Date and Time: 2015/04/29 02:44 下午
Processed Date and Time: 2015/04/29 02:44 下午
Data Path: D:\LCH\DATA\0167\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0167
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-393-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	8.97	454229	39255	31.175
2	9.53	1002788	78340	68.825
		1457017	117595	100.000

Peak rejection level: 1000

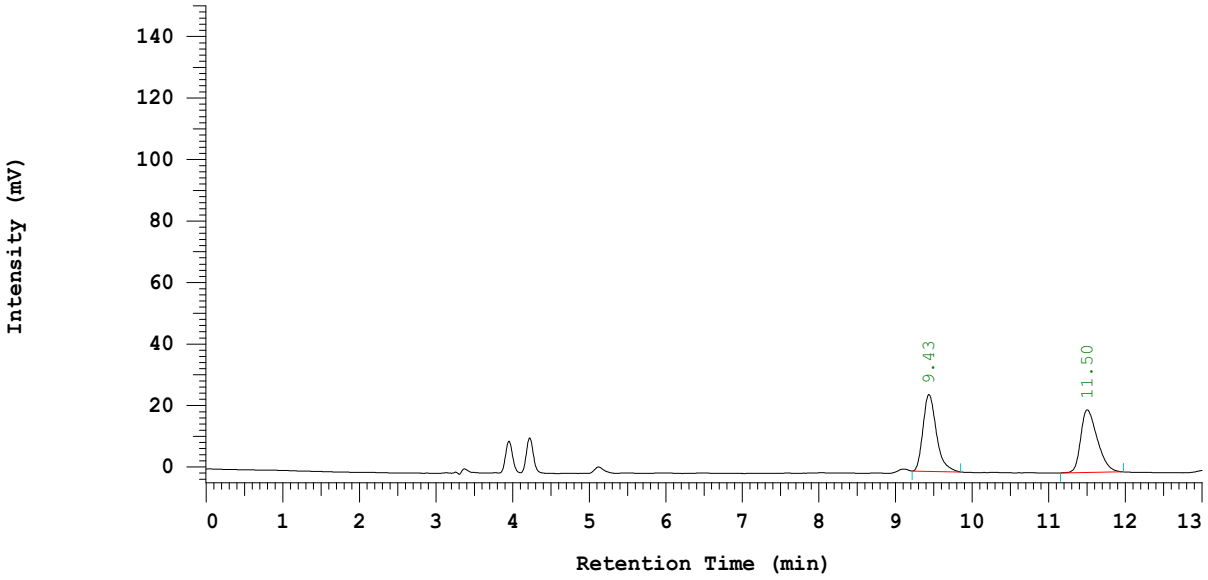
Fig S292. HPLC analysis of the mixture of chiral compound syn-3f obtained and the racemic compound syn-3f, for comparison (Table 3, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 03:34 下午
Reported Date and Time: 2015/04/29 04:08 下午
Processed Date and Time: 2015/04/29 04:08 下午
Data Path: D:\LCH\DATA\0169\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0169
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-393-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.43	306604	24970	49.909
2	11.50	307724	20392	50.091
		614328	45362	100.000

Peak rejection level: 1000

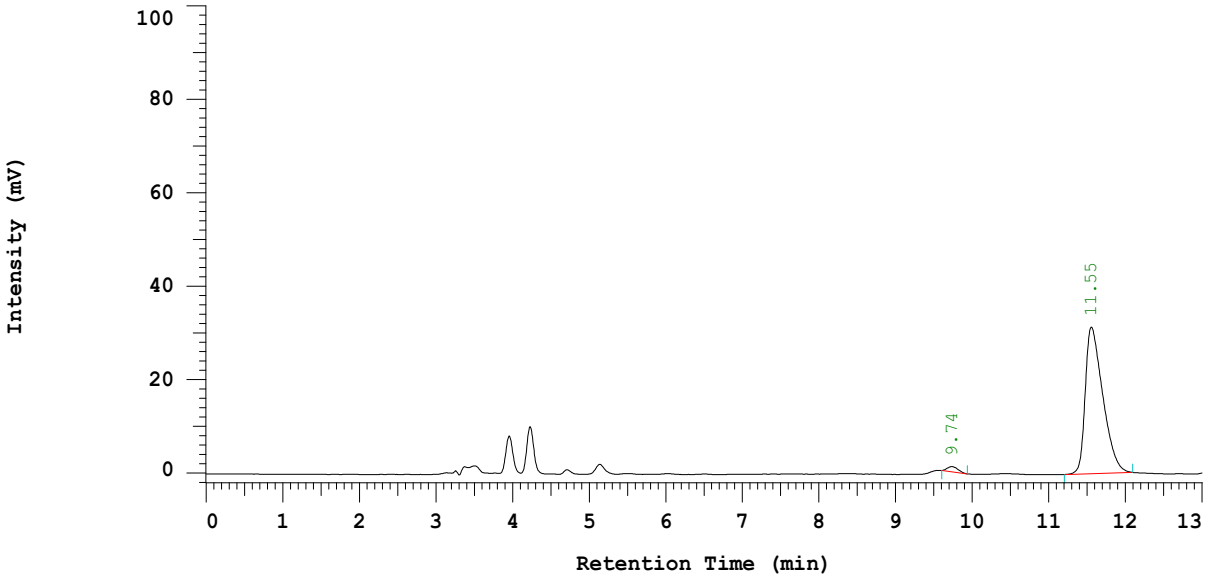
Fig S293. HPLC analysis of the racemic compound anti-3f, as a standard for comparison (Table 3, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 04:32 下午
Reported Date and Time: 2015/04/29 04:50 下午
Processed Date and Time: 2015/04/29 04:50 下午
Data Path: D:\LCH\DATA\0172\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0172
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-393-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.74	11237	1135	2.187
2	11.55	502688	31402	97.813
		513925	32537	100.000

Peak rejection level: 1000

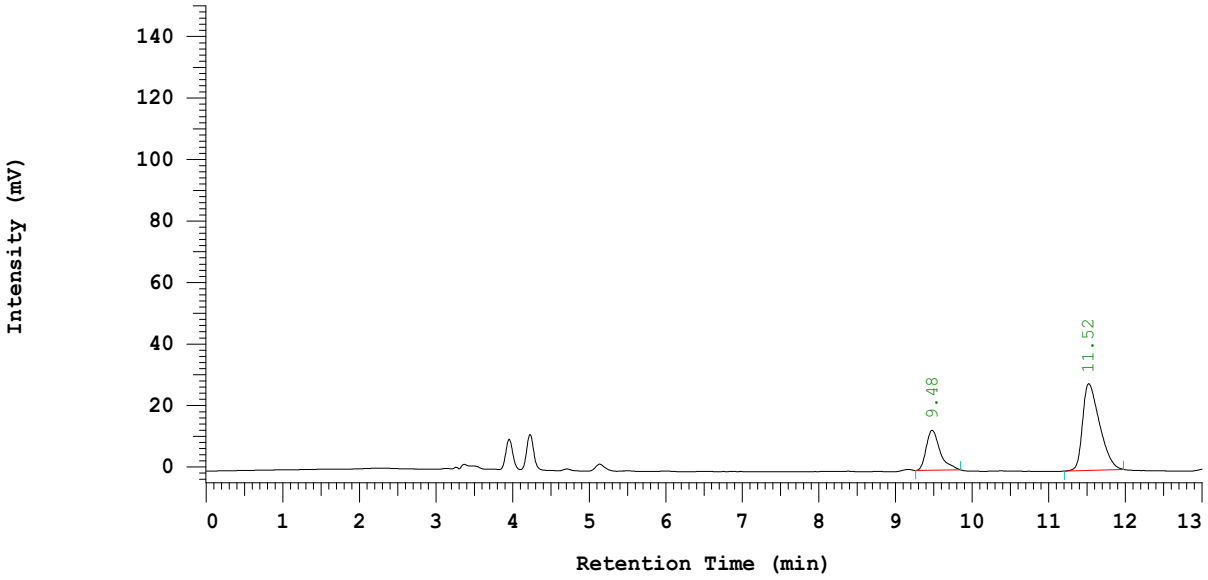
Fig S294. HPLC analysis of the chiral compound anti-3f obtained, (Table 3, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 04:07 下午
Reported Date and Time: 2015/04/29 04:21 下午
Processed Date and Time: 2015/04/29 04:21 下午
Data Path: D:\LCH\DATA\0171\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0171
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-393-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	9.48	163712	13011	27.269
2	11.52	436646	28238	72.731
		600358	41249	100.000

Peak rejection level: 1000

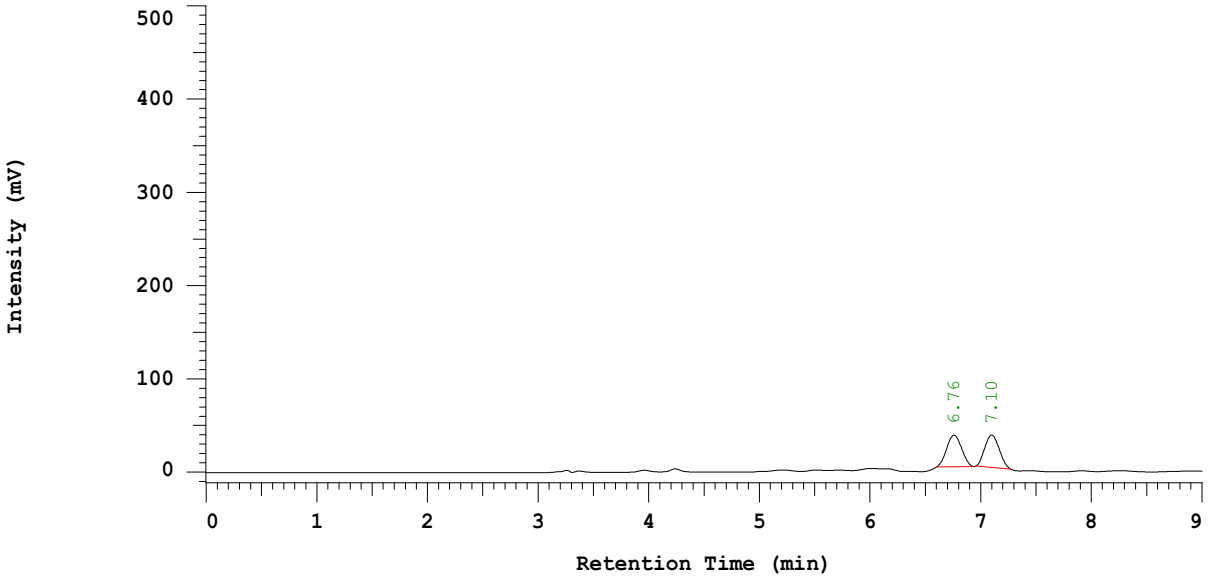
Fig S295. HPLC analysis of the mixture of chiral compound anti-3f obtained and the racemic compound anti-3f, for comparison (Table 3, entry 6).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 04:01 下午
Reported Date and Time: 2015/05/04 04:46 下午
Processed Date and Time: 2015/05/04 04:46 下午
Data Path: D:\LCH\DATA\0205\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0205
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-373-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.76	317541	33910	49.810
2	7.10	319961	34930	50.190
		637502	68840	100.000

Peak rejection level: 1000

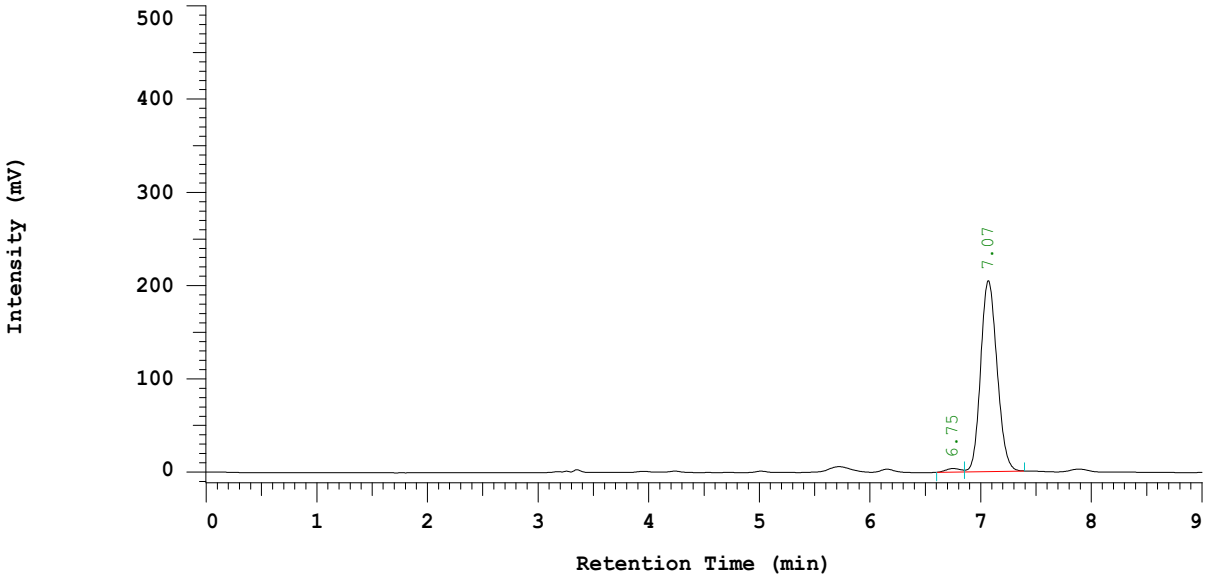
Fig S296. HPLC analysis of the racemic compound syn-3g, as a standard for comparison (Table 3, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 04:12 下午
Reported Date and Time: 2015/05/04 05:38 下午
Processed Date and Time: 2015/05/04 05:37 下午
Data Path: D:\LCH\DATA\0206\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0206
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-373-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.75	34523	3738	1.610
2	7.07	2109226	204574	98.390
		2143749	208312	100.000

Peak rejection level: 1000

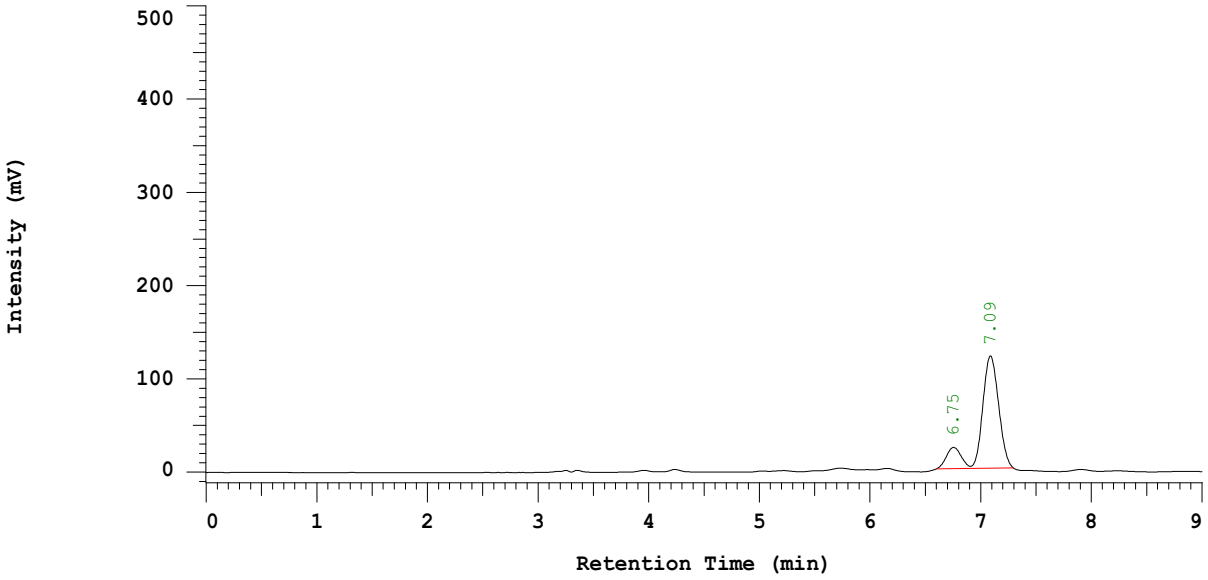
Fig S297. HPLC analysis of the chiral compound syn-3g obtained, (Table 3, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 04:32 下午
Reported Date and Time: 2015/05/04 04:50 下午
Processed Date and Time: 2015/05/04 04:50 下午
Data Path: D:\LCH\DATA\0207\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0207
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-373-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.75	211715	22681	15.140
2	7.09	1186680	120487	84.860
		1398395	143168	100.000

Peak rejection level: 1000

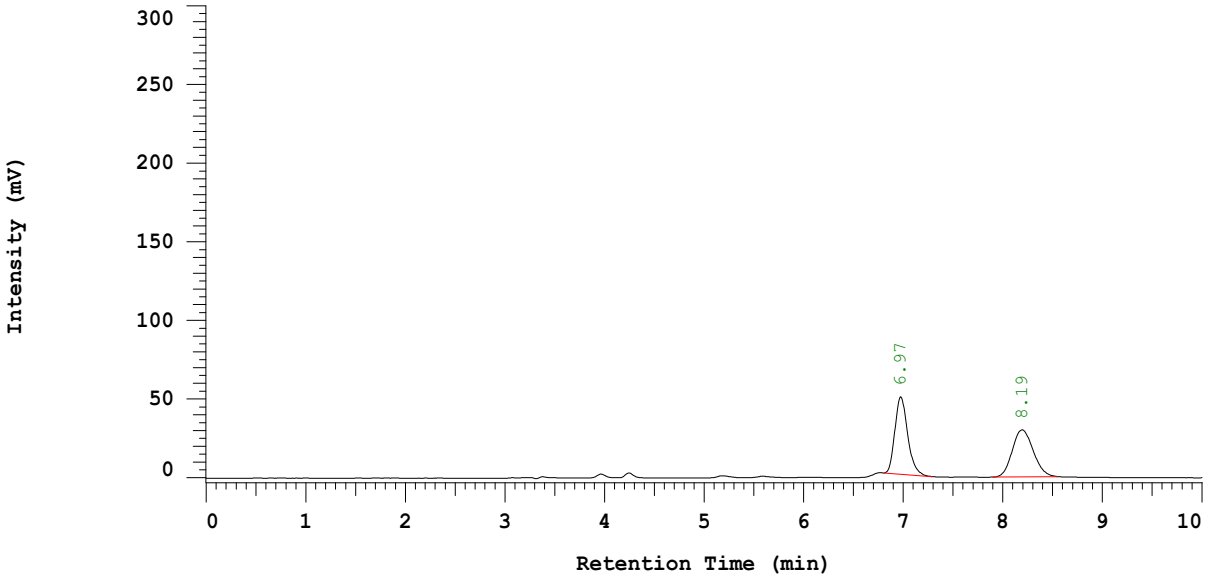
Fig S298. HPLC analysis of the mixture of chiral compound syn-3g obtained and the racemic compound syn-3g, for comparison (Table 3, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 04:55 下午
Reported Date and Time: 2015/05/04 05:16 下午
Processed Date and Time: 2015/05/04 05:16 下午
Data Path: D:\LCH\DATA\0208\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0208
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-373-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.97	440388	49217	50.258
2	8.19	435862	29917	49.742
		876250	79134	100.000

Peak rejection level: 1000

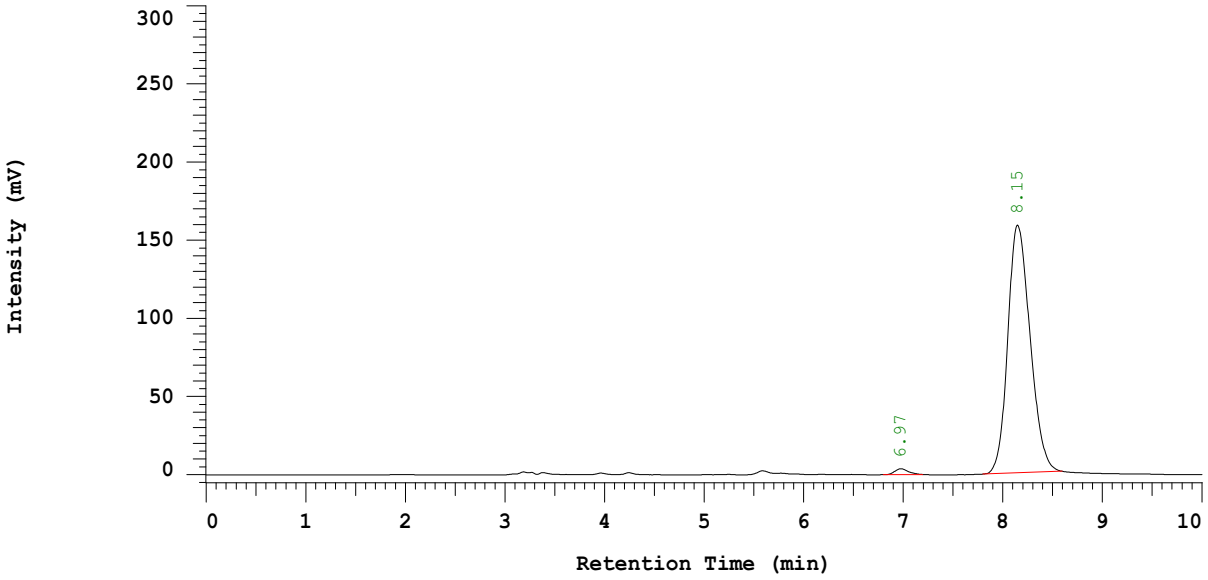
Fig S299. HPLC analysis of the racemic compound anti-3g, as a standard for comparison (Table 3, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 05:06 下午
Reported Date and Time: 2015/05/04 05:34 下午
Processed Date and Time: 2015/05/04 05:33 下午
Data Path: D:\LCH\DATA\0209\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0209
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-373-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.97	35648	3771	1.424
2	8.15	2467842	158545	98.576
		2503490	162316	100.000

Peak rejection level: 1000

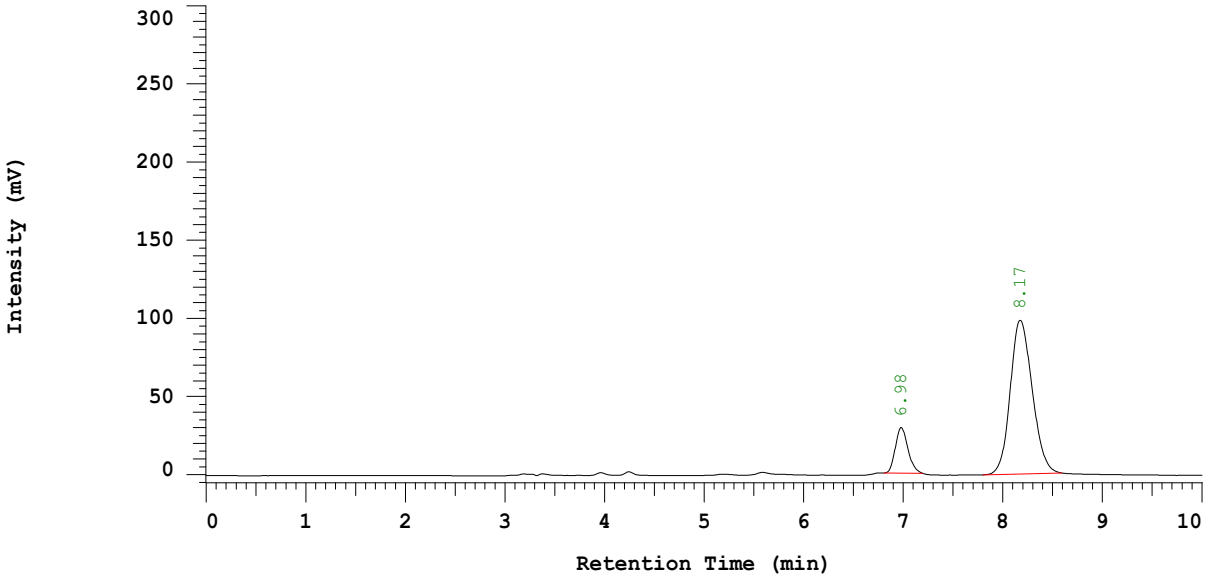
Fig S300. HPLC analysis of the chiral compound anti-3g obtained, (Table 3, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 05:20 下午
Reported Date and Time: 2015/05/04 05:33 下午
Processed Date and Time: 2015/05/04 05:32 下午
Data Path: D:\LCH\DATA\0210\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0210
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-373-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 15%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.98	256545	29146	14.522
2	8.17	1510111	98393	85.478
		1766656	127539	100.000

Peak rejection level: 1000

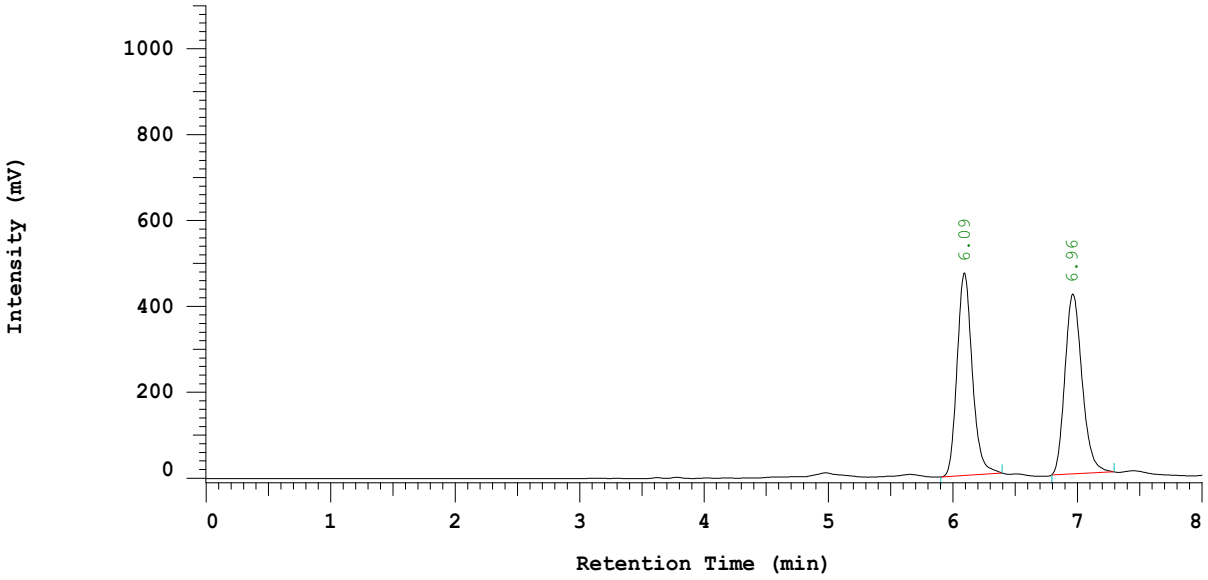
Fig S301. HPLC analysis of the mixture of chiral compound anti-3g obtained and the racemic compound anti-3g, for comparison (Table 3, entry 7).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 10:26 下午
Reported Date and Time: 2015/04/29 10:38 下午
Processed Date and Time: 2015/04/29 10:37 下午
Data Path: D:\LCH\DATA\0178\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0178
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-390-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 22%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 290 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 290 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.09	3977158	471063	49.840
2	6.96	4002636	418795	50.160
		7979794	889858	100.000

Peak rejection level: 1000

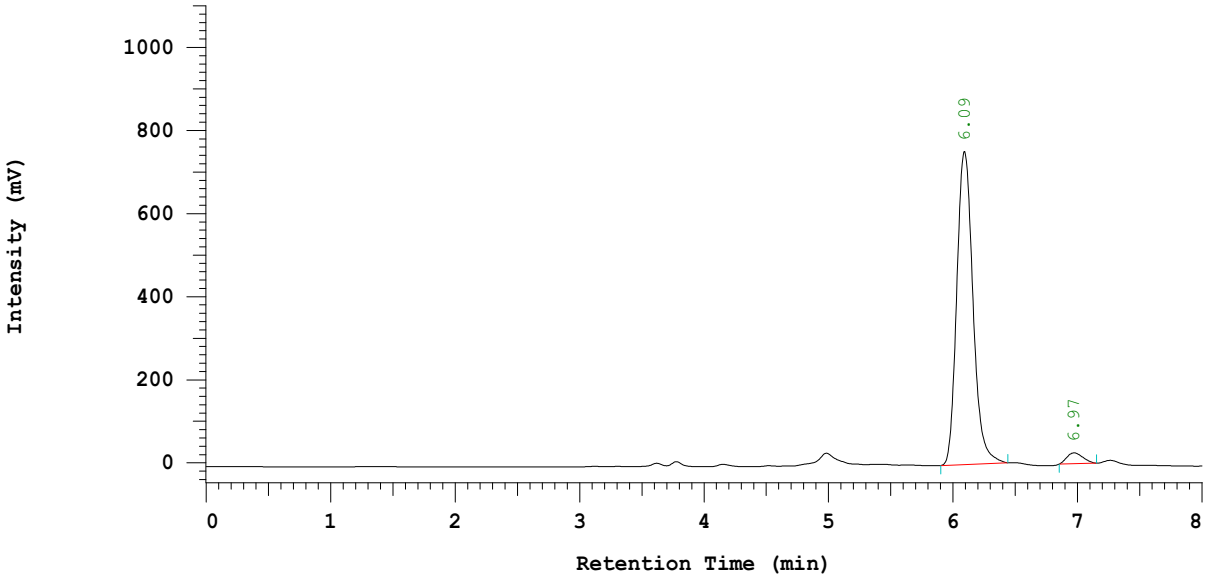
Fig S302. HPLC analysis of the racemic compound syn-3h, as a standard for comparison (Table 3, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 10:39 下午
Reported Date and Time: 2015/04/30 01:03 上午
Processed Date and Time: 2015/04/30 01:03 上午
Data Path: D:\LCH\DATA\0179\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0179
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-390-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 22%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 290 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 290 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.09	6795782	752923	96.677
2	6.97	233598	26366	3.323
		7029380	779289	100.000

Peak rejection level: 1000

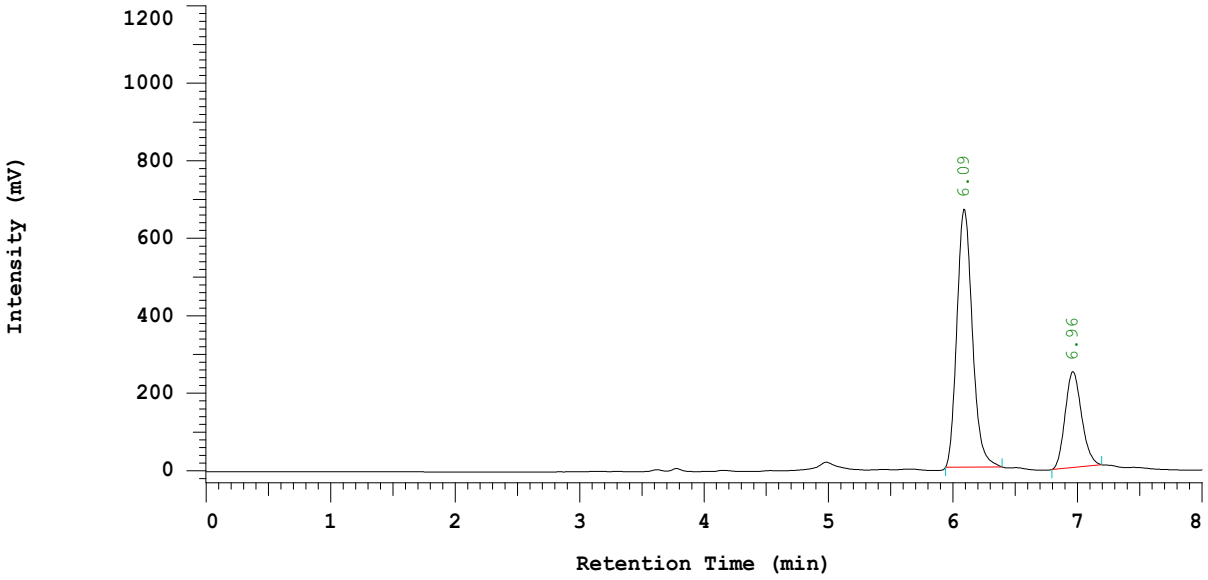
Fig S303. HPLC analysis of the chiral compound syn-3h obtained, (Table 3, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 10:54 下午
Reported Date and Time: 2015/04/29 11:03 下午
Processed Date and Time: 2015/04/29 11:03 下午
Data Path: D:\LCH\DATA\0180\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0180
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-390-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 22%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 290 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 290 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.09	5724797	665222	71.651
2	6.96	2265054	247311	28.349
		7989851	912533	100.000

Peak rejection level: 1000

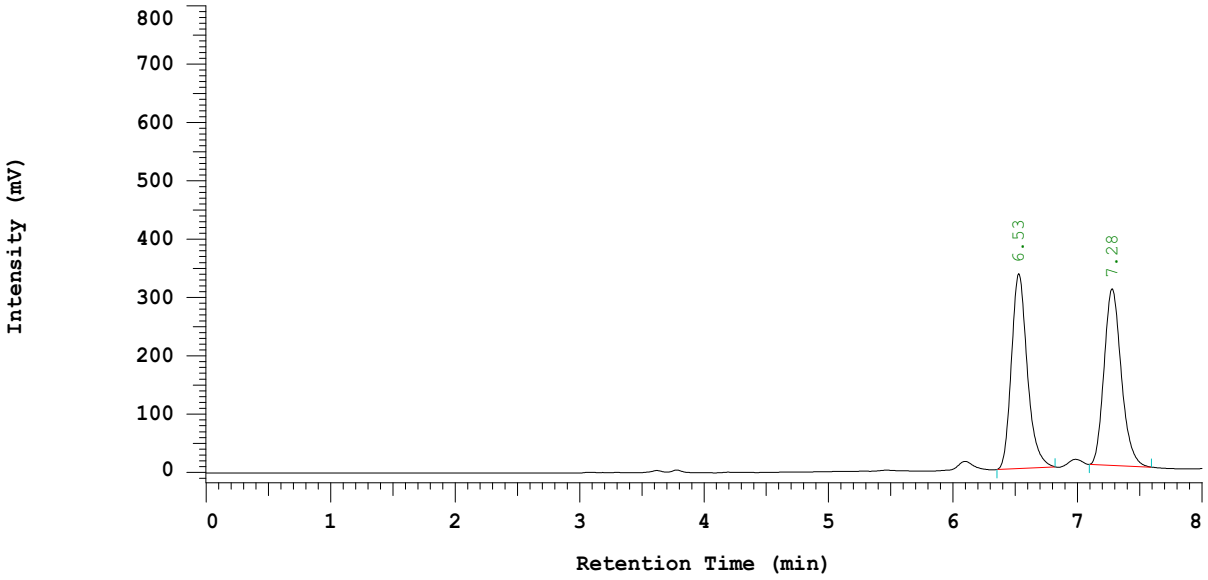
Fig S304. HPLC analysis of the mixture of chiral compound syn-3h obtained and the racemic compound syn-3h, for comparison (Table 3, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/29 11:35 下午
Reported Date and Time: 2015/04/30 12:41 上午
Processed Date and Time: 2015/04/30 12:40 上午
Data Path: D:\LCH\DATA\0182\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0182
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-390-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 22%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.53	2954061	334115	50.438
2	7.28	2902795	302695	49.562
		5856856	636810	100.000

Peak rejection level: 1000

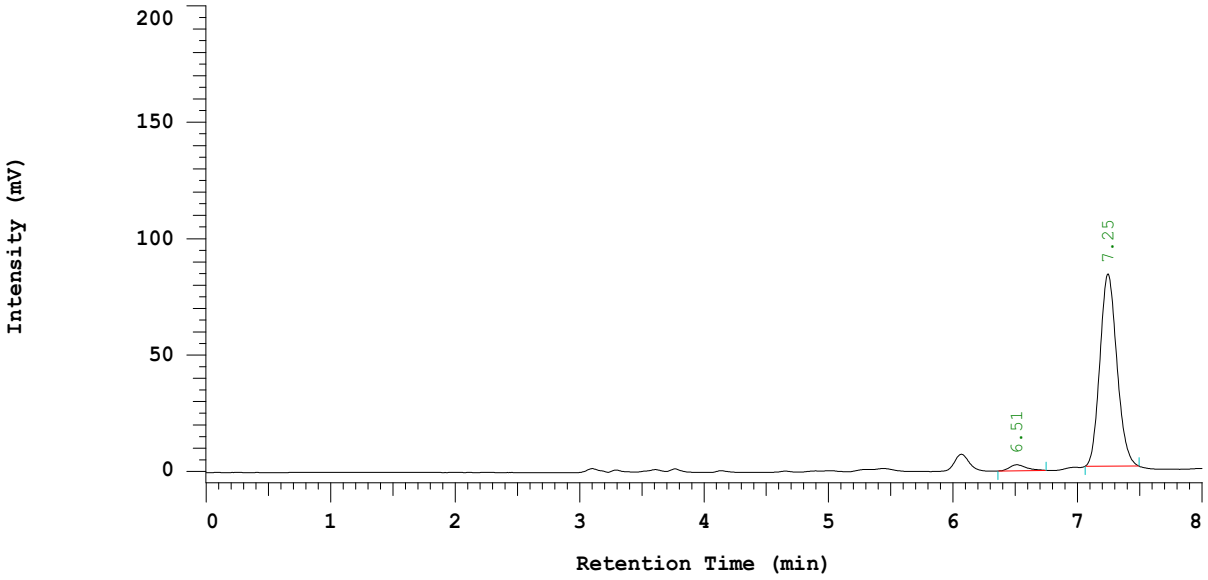
Fig S305. HPLC analysis of the racemic compound anti-3h, as a standard for comparison (Table 3, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/30 12:06 上午
Reported Date and Time: 2015/04/30 01:18 上午
Processed Date and Time: 2015/04/30 01:17 上午
Data Path: D:\LCH\DATA\0184\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0184
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-390-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 22%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.51	23850	2608	2.960
2	7.25	781903	82489	97.040
		805753	85097	100.000

Peak rejection level: 1000

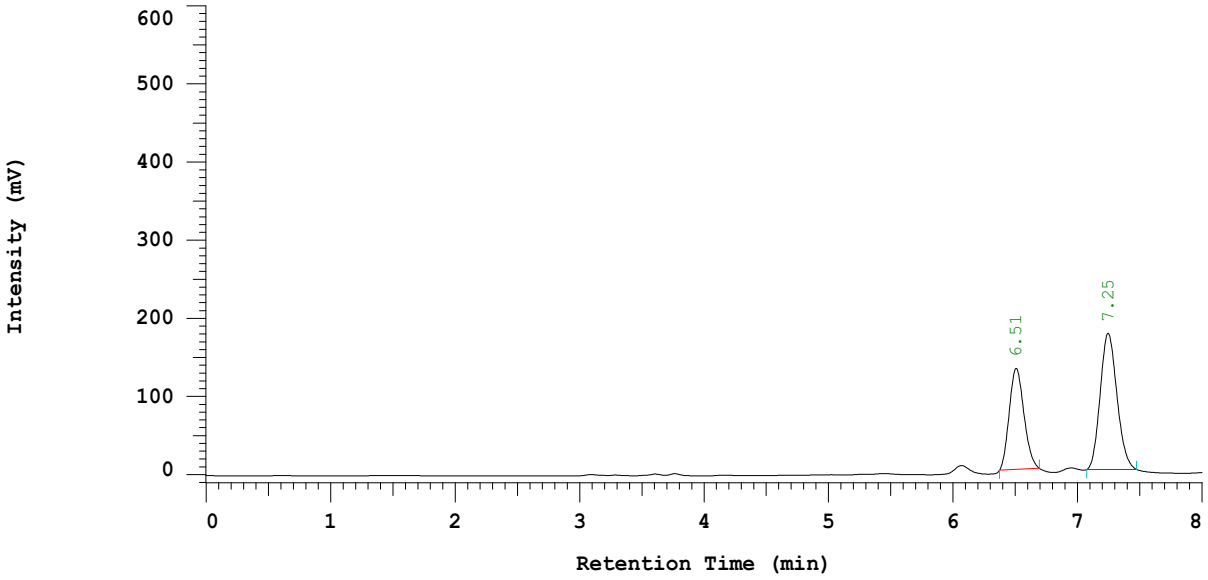
Fig S306. HPLC analysis of the chiral compound anti-3h obtained, (Table 3, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/04/30 12:16 上午
Reported Date and Time: 2015/04/30 12:35 上午
Processed Date and Time: 2015/04/30 12:35 上午
Data Path: D:\LCH\DATA\0185\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0185
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-390-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 22%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.51	1061408	129392	39.487
2	7.25	1626552	174423	60.513
		2687960	303815	100.000

Peak rejection level: 1000

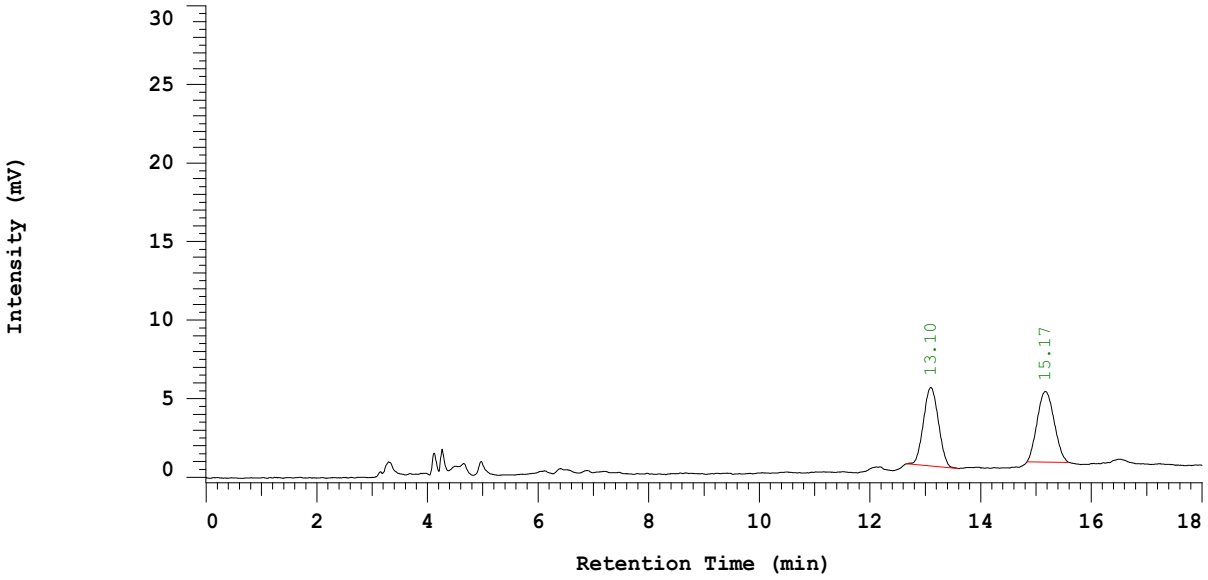
Fig S307. HPLC analysis of the mixture of chiral compound anti-3h obtained and the racemic compound anti-3h, for comparison (Table 3, entry 8).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 08:20 下午
Reported Date and Time: 2015/05/01 08:54 下午
Processed Date and Time: 2015/05/01 08:54 下午
Data Path: D:\LCH\DATA\0195\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0195
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-394-p1-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.10	94586	5011	49.593
2	15.17	96140	4495	50.407
		190726	9506	100.000

Peak rejection level: 1000

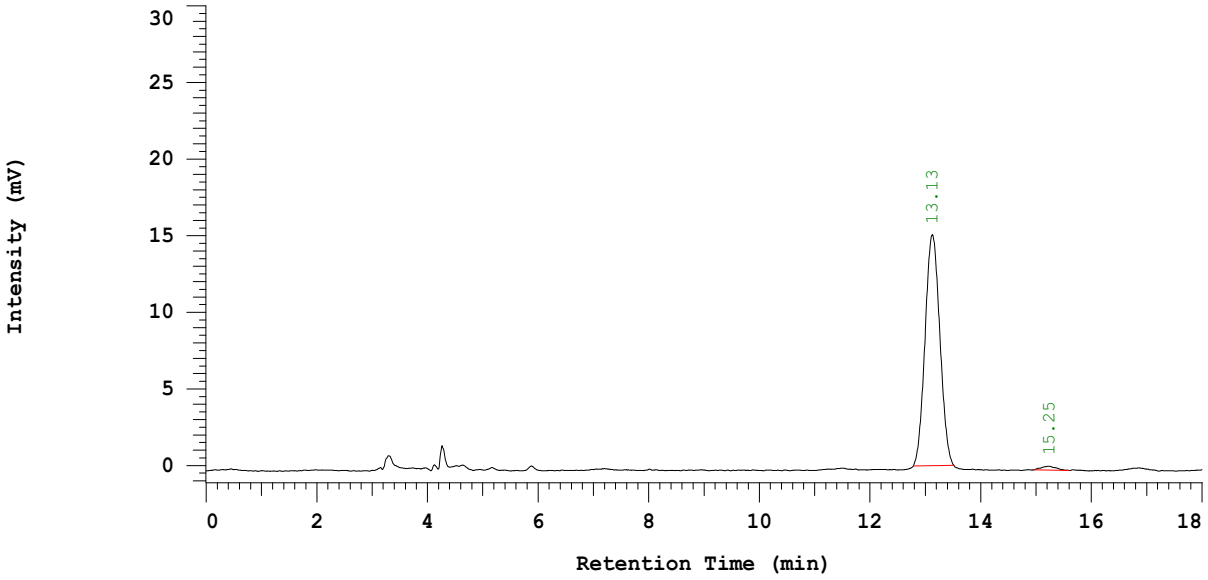
Fig S308. HPLC analysis of the racemic compound syn-3i, as a standard for comparison (Table 3, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 08:45 下午
Reported Date and Time: 2015/05/01 09:13 下午
Processed Date and Time: 2015/05/01 09:13 下午
Data Path: D:\LCH\DATA\0196\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0196
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-394-p1-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.13	280782	15070	98.356
2	15.25	4692	255	1.644
		285474	15325	100.000

Peak rejection level: 1000

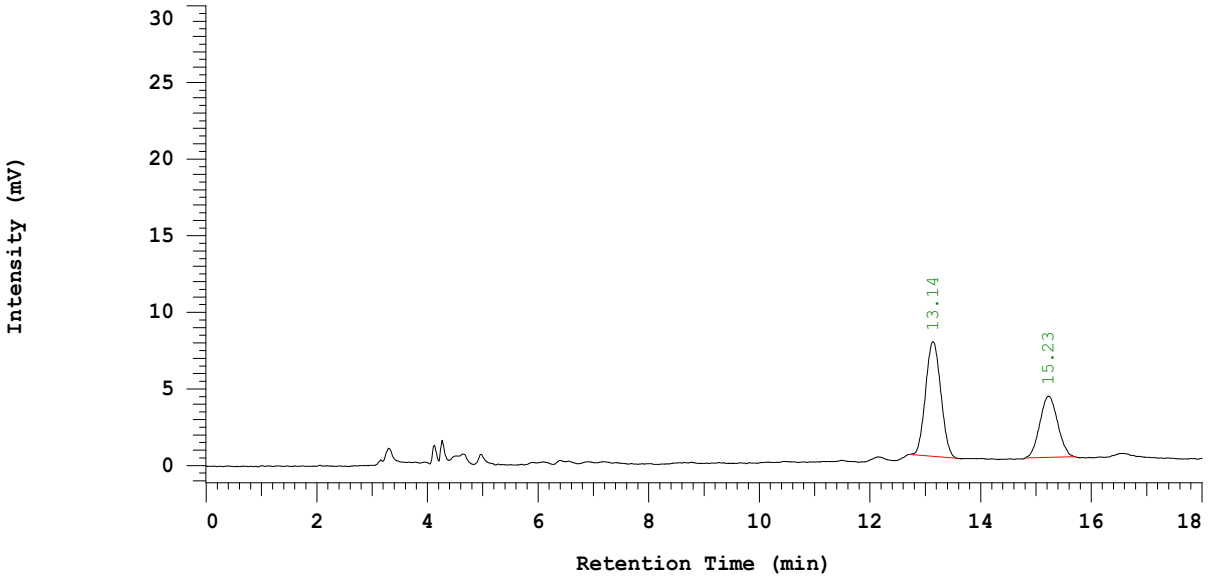
Fig S309. HPLC analysis of the chiral compound syn-3i obtained, (Table 3, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/01 09:05 下午
Reported Date and Time: 2015/05/01 09:26 下午
Processed Date and Time: 2015/05/01 09:25 下午
Data Path: D:\LCH\DATA\0197\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0197
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-394-p1-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 10%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 300 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 300 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	13.14	143572	7477	61.616
2	15.23	89437	3990	38.384
		233009	11467	100.000

Peak rejection level: 1000

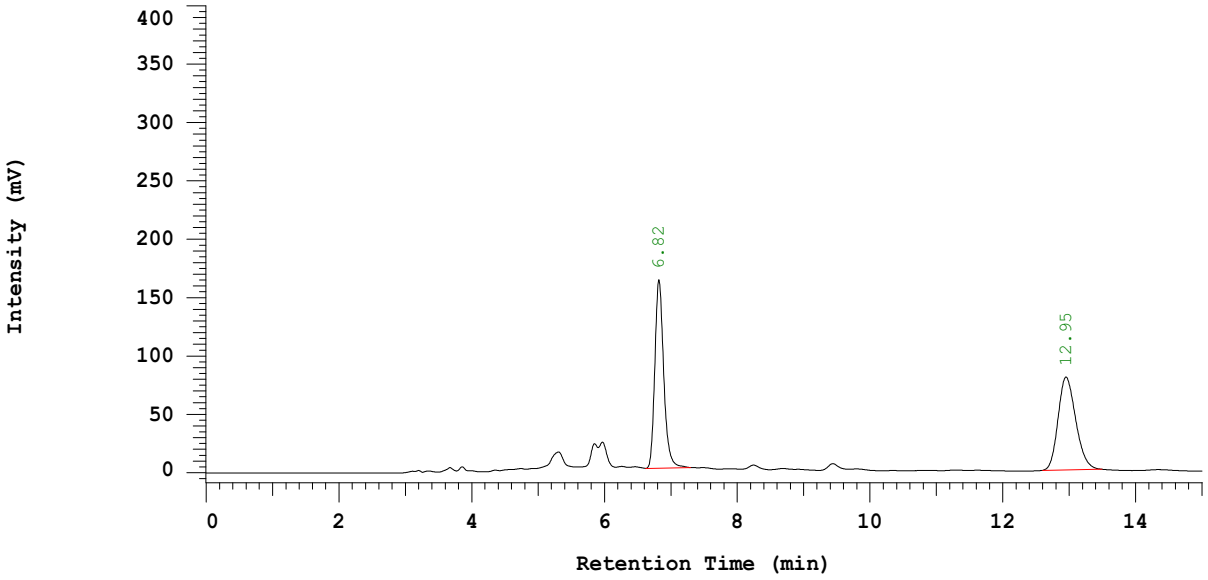
Fig S310. HPLC analysis of the mixture of chiral compound syn-3i obtained and the racemic compound syn-3i, for comparison (Table 3, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 01:36 下午
Reported Date and Time: 2015/05/04 01:57 下午
Processed Date and Time: 2015/05/04 01:56 下午
Data Path: D:\LCH\DATA\0201\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0201
Application(data): LCH
Vial Number: 1
Sample Name: LCH-2-394-p2-rac
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 20%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.82	1495523	161453	50.471
2	12.95	1467638	79505	49.529
		2963161	240958	100.000

Peak rejection level: 1000

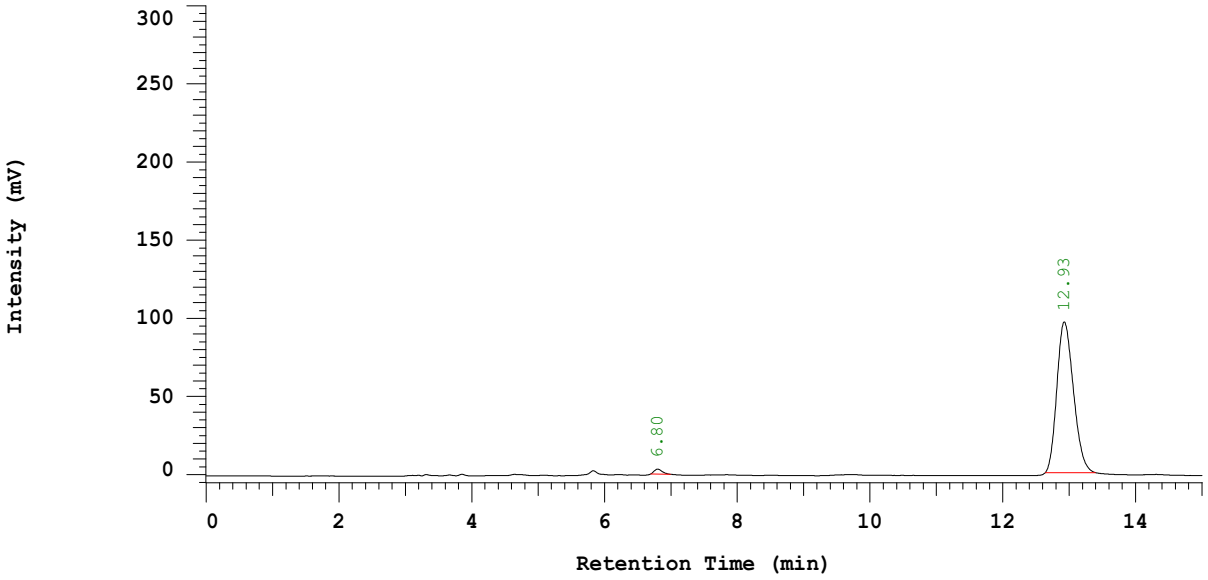
Fig S311. HPLC analysis of the racemic compound anti-3i, as a standard for comparison (Table 3, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 01:52 下午
Reported Date and Time: 2015/05/04 02:22 下午
Processed Date and Time: 2015/05/04 02:22 下午
Data Path: D:\LCH\DATA\0202\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0202
Application(data): LCH
Vial Number: 2
Sample Name: LCH-2-394-p2-chi
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 20%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.80	25929	3153	1.532
2	12.93	1666300	96362	98.468
		1692229	99515	100.000

Peak rejection level: 1000

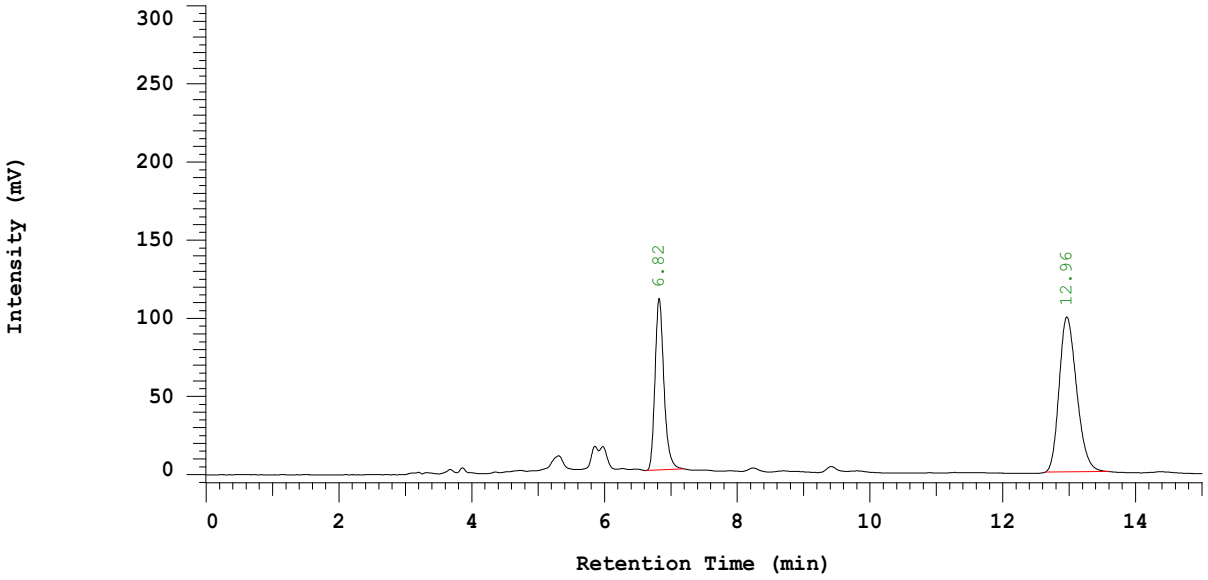
Fig S312. HPLC analysis of the chiral compound anti-3i obtained, (Table 3, entry 9).

D-2000 Elite HPLC System Manager Report

Analyzed Date and Time: 2015/05/04 02:08 下午
Reported Date and Time: 2015/05/04 02:26 下午
Processed Date and Time: 2015/05/04 02:26 下午
Data Path: D:\LCH\DATA\0203\
Processing Method: Ea/Hx

System (acquisition): Sys 1
Series: 0203
Application(data): LCH
Vial Number: 3
Sample Name: LCH-2-394-p2-co
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 20.0 ul
Sample Description: 20%Ea/Hx-1ml/min-col IC

Chrom Type: Fixed WL Chromatogram, 280 nm



Processing Method: Ea/Hx
Column Type: IC
Method Developer: YVW
Method Description:

Chrom Type: Fixed WL Chromatogram, 280 nm
Peak Quantitation: AREA
Calculation Method: EXT-STD
Scale Factor 1: 1.000

No.	RT	Area	Height	Area %
1	6.82	1018340	109718	36.013
2	12.96	1809398	99118	63.987
		2827738	208836	100.000

Peak rejection level: 1000

Fig S313. HPLC analysis of the mixture of chiral compound anti-3i obtained and the racemic compound anti-3i, for comparison (Table 3, entry 9).