Supporting Information

Efficient synthesis of mibefradil analogues: an insight into in vitro stability

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Table of Contents

I. Experimental Procedures for Scheme 4

General Methods

All reactions were carried out under dry nitrogen unless otherwise indicated. Commercially available reagents were used without further purification. Solvents and gases were dried according to standard procedures. Organic solvents were evaporated with reduced pressure using a rotary evaporator. Analytical thin layer chromatography (TLC) was performed using glass plates precoated with silica gel (0.25 mm). TLC plates were visualized by exposure to UV light (UV), and then were visualized with a *p*-anisaldehyde stain followed by brief heating on hot plate. Flash column chromatography was performed using silica gel 60 (230-400 mesh, Merck) with the indicated solvents. ^{1}H and ^{13}C spectra were recorded on Bruker 300, Bruker 400 or Varian 300 NMR spectrometers. ^{1}H NMR spectra are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), integration, and coupling constant (*J*) in Hertz (Hz). ^{1}H NMR chemical shifts are reported relative to $CDCl_3$ (7.26 ppm). ^{13}C NMR was recorded relative to the central line of $CDCl_3$ (77.0 ppm). HPLC data were acquired from a Waters Alliance System with UV detector set to at 254 and 280 nm. Samples were injected (10 μL) onto a Waters Sunfire 4.6 x 150 mm, 5.0 μM, C18 column maintained at 25.8 °C. A linear gradient from 30% to 100% **B** (MeCN) in 20 min was followed by pumping 100% **B** for another 10 minutes with **A** being H_2O + 0.1 M NH_4OAc (or NH_4HCO_2). The flow rate was 1.0 mL/min.

Benzyl 4-(2-aminophenylamino)-4-oxobutyl(methyl)carbamate (17) : To a solution of 4-(methylamino)butyric acid hydrochloride (1.00 g, 6.51 mmol) in 4 N NaOH (5 mL) was added benzyl chloroformate (1.02 mL, 7.16 mmol) at 0 °C. The reaction mixture was allowed to stir for 1 h at room temperature. The resulting solution was extracted with diethyl ether (3 x 20 mL). The organic layers were washed with brine, dried over anhydrous $MgSO_4$ and

concentrated *in vacuo*. Concentration afforded the Cbz-protected amine (1.28 g, 78%), which was used for the next step without purification. The spectroscopic data were identical with those reported in the literature[1]; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.26-7.36 (m, 5H), 5.12 (s, 2H), 3.33-3.35 (m, 2H), 2.93 (s, 3H), 2.34-2.37 (m, 2H), 1.85-1.87 (m, 2H).

Benzyl 4-(2-aminophenylamino)-4-oxobutyl(methyl)carbamate (19a): To a solution of the Cbz protected amine **17** (1.28 g, 5.09 mmol) in distilled THF (4 mL) was added TEA (1.07 mL) and a solution of isobutyl chloroformate (0.67 mL, 5.09 mmol) in distilled THF (1 mL) at −15 °C. The reaction mixture was allowed to stir for 3 h at the same temperature. Then, a solution of 1,2-phenylene diamine **18a** (0.61 g, 5.60 mmol) in distilled THF (5 mL) was added to the reaction mixture at the same temperature. The reaction mixture was allowed to stir for 2 h at room temperature. The solvent was partially removed under reduced pressure. The resulting mixture was diluted aq $NaHCO_3$/ethyl acetate and extracted with ethyl acetate (3 x 30 mL). The organic layers were washed with brine, dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel (EtOAc:*n*-hexane = 4:1) to afford amide **19a** (1.40 g, 86%); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.34-7.40 (m, 6H), 7.00-7.04 (m, 1H), 6.74-6.79 (m, 2H), 5.13 (s, 2H), 3.41-3.45 (m, 2H), 2.94 (s, 3H), 2.31-2.35 (m, 2H), 1.90-1.97 (m, 2H).

Benzyl (4-((2-amino-4-methylphenyl)amino)-4-oxobutyl)carbamate (19b): The following the same procedure as that used for the synthesis of **19a**, the reaction of amine **17** (0.817 mg, 3.45 mmol), TEA (721 μl, 5.17 mmol), iso-butyl chloroformate (451 μl, 3.45 mmol), and 3,4-diaminotoluene **18b** (463 mg, 3.79 mmol) in dry THF gave amide **19b** (0.91 mg, 78%) as a

[1] M. Tegoni, L. Ferretti, F. Sansone, M. Remelli, V. Bertolasi, F. Dallavalle, *Chem. Eur. J.* **2007**, *13*, 1300.

white solid after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.89 (s, 1H) 7.37-7.31 (m, 5H), 7.06 (d, J = 8.5 Hz, 1H), 6.54 (s, 1H), 5.21 (s, 1H), 5.06 (s, 2H), 3.96 (s, 1H), 3.27-3.20 (m, 2H), 2.35 (t, J = 6.1Hz, 2H), 2.22 (s, 3H) 1.88-1.81 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 171.5, 157.2, 140.7, 137.0, 136.4, 128.6, 128.1, 125.4, 121.5, 119.9, 118.2, 66.9, 40.1, 33.7, 26.6, 21.0. LRMS-EI (*m/z*): $[M]^+$calcd for $C_{19}H_{23}N_3O_3$ 341.17, found 341.

Benzyl (4-((2-amino-4-fluorophenyl)amino)-4-oxobutyl)carbamate (19c): The following the same procedure as that used for the synthesis of **19a**, the reaction of amine **17** (799 mg, 3.36 mmol), TEA (702 ml, 5.04 mmol), iso-butyl chloroformate (439 ml, 3.36 mmol), and 4-fluoro-1,2-phenylenediamine **18c** (466 mg, 3.69 mmol) in dry THF gave the carbamate **19c** (899 mg, 78%) as a brown solid after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.35 (s, 5H), 7.17 (t, J = 6.8 Hz, 1H), 6.49-6.43 (m, 2H), 5.10 (s, 2H), 5.06 (s, 1H), 3.32 (m, J = 6.1 Hz, 2H), 2.40 (t, J = 6.5 Hz, 2H), 1.90 (m, J = 6.2 Hz, 2H).^{13}C-NMR (100 MHz, MeOD) δ 173.4, 162.1 (d, ^{1}J = 239.6 Hz), 157.7 144.7, 137.0, 128.1, 127.8 (d, ^{3}J = 10.5 Hz), 127.5 (d, ^{3}J = 13.9 Hz), 118.9, 103.3 (d, ^{2}J = 22.9 Hz), 102.2 (d, ^{2}J = 25.8 Hz), 66.0, 39.8, 32.7, 25.7. LRMS-EI (*m/z*): $[M]^+$calcd for $C_{18}H_{20}FN_3O_3$ 345.15, found 345.

Benzyl (4-((2-amino-4-chlorophenyl)amino)-4-oxobutyl)carbamate (19d): The following the same procedure as that used for the synthesis of **19a**, the reaction of amine **17** (1.51 g, 6.38 mmol), TEA (1.33 ml, 9.57 mmol), isobutyl chloroformate (910 μL, 6.38 mmol), and 4-chloro-1,2-phenylenediamine (1.00 g, 7.02 mmol) in dry THF gave amide **19d** (1.67 g, 72%) as a yellow solid after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.34 (s, 5H), 7.20 (d, J = 8.2 Hz, 1H),

6.72-6.65 (m, 2H), 5.09 (s, 2H), 4.08 (s, 2H), 3.28 (t, J = 5.7 Hz, 2H), 2.36 (t, J = 6.3 Hz, 2H), 1.88 (m, J = 6.5 Hz, 2H).^{13}C-NMR (100 MHz, $CDCl_3$) δ 172.5, 157.3, 142.5, 136.5, 132.2, 128.6, 128.2, 128.0, 127.2, 122.0, 118.3, 116.6, 66.8, 40.1, 33.5, 26.3.

Benzyl (4-((2-amino-4-bromophenyl)amino)-4-oxobutyl)carbamate (19e): The following the same procedure as that used for the synthesis of **19a**, the reaction of amine **17** (1.43 g, 6.02 mmol), TEA (1.26 ml, 9.02 mmol), isobutyl chloroformate (786 µl, 6.02 mmol), and 4-bromo-1,2-phenylenediamine **18e** (1.24 g, 6.62 mmol) in dry THF gave amide **19e** (1.64 g, 67 %) as a white solid after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (400 MHz, MeOD) δ 7.33-7.25 (m, 5H), 6.99 (d, J = 8.4 Hz, 1H), 6.95 (d, J = 2.2 Hz, 1H), 6.73 (dd, J = 2.2, 8.4 Hz, 1H), 5.05 (s, 2H), 3.19 (m, J = 3.7 Hz, 2H), 2.41 (t, J = 7.4 Hz, 2H), 1.86 (m, J = 6.9 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 173.2, 157.6, 144.0, 136.9, 129.4, 128.2, 127.7, 127.5, 122.2, 120.0, 118.8, 118.0, 66.2, 39.9, 33.0, 25.8.

Benzyl (4-((2-amino-4,5-dichlorophenyl)amino)-4-oxobutyl)carbamate (19f): The following the same procedure as that used for the synthesis of **19a**, the reaction of amine **17** (1.62 g, 6.83 mmol), TEA (1.43 ml, 10.6 mmol), isobutyl chloroformate (893 µl, 6.83 mmol), and 4,5-dichloro-1,2-phenylenediamine **18f** (1.33 g, 7.52 mmol) in dry THF gave amide **19f** (2.51 g, 93%) as a pale orange solid after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (400 MHz, MeOD) δ 7.33-7.25 (m, 6H), 6.91 (s, 1H), 5.01 (s, 2H), 3.23-3.17 (m, 2H), 2.43 (t, J = 7.2 Hz, 2H), 1.87 (m, J = 6.96 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 173.1, 157.7, 142.3, 137.0, 129.4, 128.1, 127.6, 127.4, 126.9, 122.8, 118.6, 116.7, 66.1, 39.7, 32.8, 25.6

Benzyl (4-((2-amino-4-nitrophenyl)amino)-4-oxobutyl)carbamate (19g): The following the same procedure as that used for the synthesis of **19a**, the reaction of amine **17** (695 mg, 2.93 mmol), TEA (613 ml, 4.40 mmol), isobutyl chloroformate (383 ml, 2.93 mmol), and 4-nitro-1,2-phenylenediamine (494 mg, 3.22 mmol) in dry THF gave amide **19g** (0.26 g, 24%) as a orange solid after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (300 MHz, MeOD) δ 8.28 (d, J = 2.5 Hz, 1H), 8.06 (dd, J = 2.6, 9.1 Hz, 1H), 7.50-7.42 (m, 5H), 6.93 (d, J = 9.1 Hz, 1H), 5.22 (s, 2H), 3.31 (t, J = 7.2 Hz, 2H), 2.63 (t, J = 7.2 Hz, 2H), 2.04 (m, J = 6.9 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 173.6, 157.7, 149.9, 137.0, 136.8, 128.1, 127.6, 127.4, 123.4, 123.1, 120.8, 113.7, 66.1, 39.7, 32.7, 25.5

3-(1*H*-Benzo[*d*]imidazol-2-yl)-*N*-methylpropan-1-amine (19a'): To a solution of the amide **19a** (1.40 g, 4.10 mmol) in toluene (30 mL) was added *p*-toluenesulfonic acid monohydrate (1.17 g, 6.15 mmol) at room temperature. The reaction mixture was treated under reflux condition with a Dean-Stark apparatus. The solvent was removed under reduced pressure. The resulting mixture was extracted with CH_2Cl_2 (3 x 30 mL). The organic layers were dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel (EtOAc only) to afford benzimidazole **19a'** (0.60 g, 45%). The spectroscopic data were identical with those reported in the literature[13]; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.57-7.58 (m, 2H), 7.33-7.38 (m, 5H), 7.19-7.22 (m, 2H), 5.23 (s, 2H), 3.40-3.44 (m, 2H), 2.96 (s, 3H), 2.86-2.90 (m, 2H), 1.99 (m, 2H).

Benzyl (3-(1*H*-benzo[*d*]imidazol-2-yl)propyl)carbamate (19b'): The following the same procedure as that used for the synthesis of **19a'**, the reaction of amide **19b** (798 g, 2.44 mmol) and *p*-toluenesulfonic acid monohydrate (695 mg, 3.65 mmol) in dry benzene (24 ml) gave benzimidazole **19b'** (0.32 g, 42%) as a yellow solid after purification by column

chromatography on silica gel (only EtOAc); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.46 (d, J = 8.1 Hz, 1H), 7.34 (s, 5H), 7.04 (d, J = 8.1 Hz, 1H), 5.36 (t, J = 6.4 Hz, 1H), 5.13 (s, 2H), 3.28 (q, J = 6.1 Hz, 2H), 2.92 (t, J = 6.5 Hz, 2H), 2.45 (s, 3H), 1.97-1.91 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 157.6, 154.1, 138.3, 137.0, 136.4, 131.9, 129.1, 128.6, 128.2, 128.0, 123.6, 114.6, 114.2, 66.9, 39.7, 29.0, 25.7, 21.6. LRMS-EI (*m/z*): $[M]^+$ calcd for $C_{19}H_{21}N_3O_2$ 323.16, found 323.

Benzyl (3-(5-fluoro-1*H*-benzo[*d*]imidazol-2-yl)propyl)carbamate (19c'): The following the same procedure as that used for the synthesis of **19a'**, the reaction of amide **19c** (975 mg, 2.82 mmol) and *p*-toluenesulfonic acid monohydrate (591 mg, 3.11 mmol) in dry toluene (25 ml) gave benzimidazole **19c'** (0.69 g, 75%) as a white solid after purification by column chromatography on silica gel (only EtOAc); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.45-7.41 (m, 1H), 7.35 (s, 5H), 7.21 (dd, J = 2.2, 9.0 Hz, 1H), 6.94 (td, J = 2.3, 9.2 Hz, 1H), 5.60 (t, J = 6.2 Hz, 1H), 5.04 (s, 2H), 3.25 (q, J = 6.2 Hz, 2H), 2.91 (t, J = 6.7 Hz, 2H), 1.97-1.89 (m, 2H). LRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{18}FN_3O_2$ 327.14, found 327.

Benzyl (3-(5-chloro-1*H*-benzo[*d*]imidazol-2-yl)propyl)carbamate (19d'): The following the same procedure as that used for the synthesis of **19a'**, the reaction of amide **19d** (1.67 g, 4.62 mmol) and *p*-toluenesulfonic acid monohydrate (958 mg, 5.08 mmol) in dry toluene (28 ml) gave benzimidazole **19d'** (573 mg, 36%) as a brown oil after purification by column chromatography on silica gel (only EtOAc); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.48 (d, J = 7.7 Hz, 1H), 7.37 (s, 5H), 7.21 (dd, J = 1.8, 8.5 Hz, 1H) 5.50 (t, J = 6.2 Hz, 1H), 5.17 (s, 2H), 3.31 (q, J = 6.2 Hz, 2H), 2.95 (t, J = 6.6 Hz, 2H), 1.97 (m, J = 6.2 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 157.9, 155.5, 139.2, 136.2, 131.0, 128.6, 128.3, 128.0, 127.8, 122.7, 115.5, 114.7, 67.1, 39.5, 29.0, 25.5. LRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{18}ClN_3O_2$ 343.11,

found 343.

Benzyl (3-(5-bromo-1*H*-benzo[*d*]imidazol-2-yl)propyl)carbamate (19e'): The following the same procedure as that used for the synthesis of **19a'**, the reaction of amide **19e** (1.64 g, 4.04 mmol) and *p*-toluenesulfonic acid monohydrate (923 mg, 4.85 mmol) in dry toluene (40 ml) gave benzimidazole **19e'** (651 mg, 41%) as a brown oil after purification by column chromatography on silica gel (only EtOAc); ^{1}H-NMR (400 MHz, MeOD) δ 7.58 (d, J = 0.8 Hz, 1H), 7.27 (d, J = 8.5 Hz, 1H), 7.19-7.13 (m, 6H), 3.16 (t, J = 6.4 Hz, 2H), 2.82 (t, J = 7.5 Hz, 2H), 1.96 (m, J = 7.1 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 157.5, 156.0, 139.6, 136.8, 136.5, 128.1, 127.6, 127.4, 124.9, 117.2, 115.3, 114.8, 66.2, 39.9, 28.0, 25.8

Benzyl (3-(5,6-dichloro-1*H*-benzo[*d*]imidazol-2-yl)propyl)carbamate (19f'): The following the same procedure as that used for the synthesis of **19a'**, the reaction of amide **19f** (2.51 g, 6.33 mmol) and *p*-toluenesulfonic acid monohydrate (1.32 g, 6.96 mmol) in dry toluene (30 ml) gave benzimidazole **19f'** (624 mg, 26%) as a yellow solid after purification by column chromatography on silica gel (only EtOAc); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 11.43 (s, 1H), 7.74 (s, 1H), 7.57 (s, 1H), 7.36 (s, 5H), 5.20 (t, J = 6.3 Hz, 1H), 5.16 (s, 2H), 3.29 (q, J = 6.2 Hz, 2H), 2.92 (t, J = 6.3 Hz, 2H), 1.92 (m, J = 4.0, 2H)

Benzyl (3-(5-nitro-1*H*-benzo[*d*]imidazol-2-yl)propyl)carbamate (19g'): The following the same procedure as that used for the synthesis of **19a'**, the reaction of amide **19a** (358 mg, 962 μmol) and *p*-toluenesulfonic acid monohydrate (220 mg, 1.15 mmol) in dry benzene (10 ml) gave benzimidazole **19g'** (296 mg, 87%) as a yellow solid after purification by column chromatography on silica gel (only EtOAc); ^{1}H-NMR (400 MHz, MeOD) δ 8.24 (d, J = 1.7 Hz, 1H), 7.98 (dd, J = 2.2, 8.8 Hz, 1H), 7.46 (d, J = 8.9 Hz, 1H), 7.26-7.20 (m, 5H), 5.00 (s,

3H), 3.21 (t, *J* = 6.7 Hz, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.02 (m, *J* = 7.2 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 159.7, 157.6, 143.1, 141.9, 138.2, 136.9, 128.0, 127.6, 127.3, 117.5, 113.4, 111.0, 66.1, 39.8, 27.7, 25.9. LRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{18}N_4O_4$ 354.13, found 354.

3-(1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4a): To a solution of the Cbz-protected amino benzimidazole **19a'** (600 mg, 1.86 mmol) in MeOH (8 mL) was added Pd/C (60 mg, 10 %(w/w)) at room temperature. The reaction mixture was stirred for 3 h under a hydrogen atmosphere (with the aid of a hydrogen balloon). It was filtered through a pad of Celite® and the solvent was removed under reduce pressure. Concentration afforded amine **5** (351 mg, quantitative) as a brown solid, which was used for the next step without further purification; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.51-7.54 (m, 2H), 7.17-7.24 (m, 2H), 3.07 (t, *J* = 6.5 Hz, 2H), 2.78 (t, *J* = 5.9 Hz, 2H), 2.49 (s, 3H), 1.98 (m, 2H).

3-(5-Methyl-1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4b): The following the same procedure as that used for the synthesis of **4a**, the reaction of carbamate **19b'** (515 g, 1.59 mmol) and Pd/C (51 mg, 5 %(w/w) in dry MeOH (15 ml) gave amine **4b** (131 mg, 43%) as a brown oil after purification by column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4Cl = 80:4:1:1); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.1 Hz, 1H), 7.28 (s, 1H), 6.97 (d, *J* = 8.1, 1H), 5.77 (s, 2H), 2.94 (t, *J* = 6.8 Hz, 2H), 2.73 (t, *J* = 6.0 Hz, 2H), 2.39 (s, 3H), 1.91 (t, *J* = 6.6 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 155.0, 138.7, 137.2, 131.6, 123.3, 114.4, 114.1, 41.3, 30.9, 27.1, 21.6. LRMS-EI (*m/z*): $[M]^+$ calcd for $C_{11}H_{15}N_3$ 189.13, found 189.

3-(5-Fluoro-1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4c): The following the same

procedure as that used for the synthesis of **4a**, the reaction of carbamate **19c'** (699 mg, 2.01 mmol) and Pd/C (69 mg, 10 %(w/w) in dry MeOH (21 ml) gave amine **4c** (374 mg, ≥ 99%) as a pale pink solid ;^{1}H-NMR (300 MHz, MeOD) δ 7.41 (dd, J = 4.7, 8.7 Hz, 1H), 7.16 (dd, J = 2.3, 9.1 Hz, 1H), 6.92 (td, J = 3.4, 6.8 Hz, 1H), 2.91 (t, J = 7.5 Hz, 2H), 2.77 (t, J = 7.2 Hz, 2H), 2.91 (q, J = 7.5 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 159.3 (d, ^{1}J = 235.0 Hz), 156.0, 138.5 (d, ^{3}J = 13.0 Hz), 134.0, 114.6 (d, ^{3}J = 10 Hz), 109.7 (d, ^{2}J = 25 Hz), 100.1 (d, ^{2}J = 26 Hz), 40.3, 30.1, 25.8. LRMS-EI (*m/z*): $[M]^{+}$ calcd for $C_{10}H_{12}FN_3$ 193.10, found 193.

3-(5-Chloro-1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4d): The following the same procedure as that used for the synthesis of **4a**, the reaction of carbamate **19d'** (573 mg, 1.67 mmol) and Pd/C (60 mg, 10%(w/w)) in dry MeOH (15 ml) gave amine **4c** (334 mg, 96%) as a yellow oil; ^{1}H-NMR (400 MHz, MeOD) δ 7.51 (s, 1H), 7.43 (d, J = 6.4 Hz, 1H), 7.15 (dd, J = 1.2, 6.6 Hz, 1H), 3.08 (t, J = 6.5 Hz, 2H), 2.94 (t, J = 5.8 Hz, 2H), 1.96 (m, J = 6.2 Hz, 2H) .^{13}C-NMR (100 MHz, MeOD) δ 156.1, 139.2, 136.6, 127.4, 122.2, 114.8, 114.0, 40.0, 29.0, 25.7. LRMS-EI (*m/z*): $[M]^{+}$ calcd for $C_{10}H_{12}ClN_3$ 209.07, found 209.

3-(5-Bromo-1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4e): The solution of the *N*-Cbz protected amine **19e'** (221 mg, 569 μmol) in 6N HCl (5 mL) was allowed to warm to reflux for 1 h. Concentration afforded isopropylchroman-3-ol **4e** (175 mg, ≥ 99%) as a brown solid; ^{1}H-NMR (400 MHz, MeOD) δ 7.94 (d, J = 1.2 Hz, 1H), 7.70-7.65 (m, 2H), 3.39 (t, J = 8.0 Hz, 2H), 3.17 (t, J = 7.4 Hz, 2H), 2.37 (m, J = 7.9 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 153.7, 132.1, 130.1, 129.3, 118.8, 116.5, 115.1, 38.4, 24.4, 23.6.

3-(5,6-Dichloro-1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4f): The following the same procedure as that used for the synthesis of **4a**, the reaction of carbamate **19f'** (601 mg, 1.59

mmol) and Pd/C (60 mg, 10 %(w/w) in dry MeOH (15 ml) gave amine **4f** (354 mg, 91%) as a pale red brown solid; ^{1}H-NMR (400 MHz, MeOD) δ 7.45 (s, 2H), 2.87 (t, J = 7.5 Hz, 2H), 2.75 (t, J = 7.2 Hz, 2H), 1.95 (m, J = 7.3 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 157.5, 137.6, 125.3, 115.3, 40.1, 29.2, 25.8

3-(5-Nitro-1*H*-benzo[*d*]imidazol-2-yl)propan-1-amine (4g): The following the same procedure as that used for the synthesis of **4e**, the reaction of carbamate **19g'** (140 mg, 396 μmol) in 6 N HCl (4 ml) gave amine **4g** (111 mg, ≥ 99%) as a pale yellow solid; ^{1}H-NMR (300 MHz, MeOD) δ 8.69 (d, J = 1.9 Hz, 1H), 8.47 (dd, J = 1.8, 9.0 Hz, 1H), 7.98 (d, J = 9.0 Hz, 1H), 3.40 (t, J = 7.0 Hz, 2H), 3.36 (t, J = 7.5 Hz, 2H), 2.34 (m, J = 7.7 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 157.2, 145.9, 135.0, 131.0, 121.2, 113.5, 110.3, 38.4, 24.3, 24.1.

3-(1-Tosyl-1*H*-benzo[*d*]imidazol-2-yl)propan-1-ol (20a'): To a solution of alcohol **20a** (35.7 mg, 188 μmol) in dry CH_2Cl_2, THF (3 ml/3 ml) was added *p*-TsCl (119 mg, 624 μmol), DMAP (3.50 mg, 28.4 μmol) and TEA(57.4 mg, 568 μmol) at 0 °C. The reaction mixture was allowed to stir for 4 h at room temperature. The resulting mixture was extracted with CH_2Cl_2. The organic layers were washed with brine, dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel (EtOAc:*n*-hexane = 4:1) to afford the protected amine **20a'** (106 mg, 57%); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.04-8.01 (m, 1H), 7.80-7.78 (m, 2H), 7.64-7.61 (m, 1H), 7.36-7.24 (m, 4H), 3.78 (t, J = 5.8 Hz, 2H), 3.55 (s, 1H), 3.32 (t, J = 7.1 Hz, 2H), 2.35 (s, 3H), 2.21-2.15 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 155.0, 146.1, 141.5, 135.4, 133.1, 130.3, 126.8, 124.9, 124.7, 119.7, 113.6, 61.9, 30.1, 27.1, 21.7.

3-(1-Tosyl-1*H*-benzo[*d*]imidazol-2-yl)propanal (21a): Dess-Martin periodinane (72.9 mg,

172 μmol) was added to a solution of alcohol **20a'** (51.6 mg, 156 μmol) in CH_2Cl_2 (2 ml). After stirring for 2 h, the reaction mixture was quenched with $Na_2S_2O_3$ and sat. $NaHCO_3$. The resulting mixture was extracted with Et_2O. The organic layers were washed with brine, dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel (EtOAc:*n*-hexane = 1:1) to afford the aldehyde **21a** (38.4 mg, 75%) as a colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 8.01-7.99 (m, 1H), 7.85 (dd, *J* = 1.7, 6.7 Hz, 2H), 7.62-7.60 (m, 1H), 7.33-7.31 (m, 4H), 3.50 (t, *J* = 6.8 Hz, 2H), 3.15 (t, *J* = 6.8 Hz, 2H), 2.38 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 200.2, 153.2, 146.1, 141.8, 135.3, 133.3, 130.3, 126.9, 124.9, 124.6, 119.9, 113.5, 40.3, 22.7, 21.7.

3-(5-Methyl-1-tosyl-1*H*-benzo[*d*]imidazol-2-yl)propan-1-ol (20b'): The following the same procedure as that used for the synthesis of **20a'**, the reaction of benzimidazole **20b** (35.7 mg, 188 μmol), *p*-TsCl (39.4 mg, 206 μmol), DMAP (1.15 mg, 9.38 μmol) and TEA (19.0 mg, 188 μmol) in dry CH_2Cl_2, THF (1:1) gave the N-protected benzimidazole **20b'** (34.2 mg, 53%) after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 4:1); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.89-7.83 (m, 1H), 7.78-7.56 (m, 2H), 7.50-7.41(m, 1H), 7.27-7.24 (m, 2H), 7.16-7.11 (m, 1H), 3.76 (t, *J* = 5.8 Hz, 2H), 3.28 (q, *J* = 6.6 Hz, 2H), 2.49 (s, 1H), 2.41 (s, 2H), 2.36 (d, *J* = 4.2 Hz, 3H), 2.15 (m, 2H).

3-(1-Tosyl-1*H*-benzo[*d*]imidazol-2-yl)propanal (21b): The following the same procedure as that used for the synthesis of **21a**, the reaction of alcohol **20b'** (34.2 mg, 100 μmol) and Dess-Martin periodinane (46.3 mg, 110 μmol) in dry CH_2Cl_2 (1 ml) gave aldehyde **21b (**25.4 mg, 75%) as a brown oil after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 1:1); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.90 (s, 1H), 7.87-7.81 (m, 3H), 7.45-7.40 (m, 1H), 7.30 (t, *J* = 7.2 Hz, 2H), 7.14 (t, *J* = 9.0 Hz, 1H), 3.49-3.44 (m, 2H), 3.14 (t, *J* = 6.8 Hz,

2H), 2.49 (s, 1H), 2.42 (s, 2H), 2.39 (d, J = 4.4 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 200.3, 153.2, 152.6, 145.9, 142.0, 139.8, 135.4, 135.3, 135.1, 134.6, 133.5, 131.3, 130.3, 130.3, 126.8, 126.2, 126.0, 119.8, 119.3, 113.5, 113.0, 40.4, 22.7, 22.0, 21.7, 21.3

2-(1*H*-Benzo[*d*]imidazol-2-yl)ethanol (23a): *o*-Phenylenediamine **22** (3.31 mL, 48.5 mmol) and 3-hydroxypropionitrile (4.20 g, 38.8 mmol) dissolved in conc. HCl (18 mL) were refluxed for 14h. The reaction mixture was neutralized with ammonia solution until pH 8 was reached at ice-bath. Neutralization gave brown powder which was filtered with water to give benzimidazole **23a**[2] (2.56 g, 41%); ^{1}H-NMR (400 MHz, MeOD) δ 7.49-7.50 (m, 2H), 7.17-7.20 (m, 2H), 3.99 (t, J = 6.6 Hz, 2H), 3.09 (t, J = 6.6 Hz, 2H).^{13}C-NMR (100 MHz, MeOD) δ 153.0, 137.1, 122.3, 113.9, 59.5, 31.7.

2-(5-Methyl-1*H*-benzo[*d*]imidazol-2-yl)ethanol (23b): The following the same procedure as that used for the synthesis of **23a**, the reaction of 3,4-diaminotoluene (2.00 g, 16.37 mmol) and 3-hydroxypropionitrile (1.45 g, 20.46 mmol) dissolved in conc. HCl (10 mL) gave benzimidazole **23b** [3] (0.36 g, 13%) as a white solid after purification by column chromatography on silica gel (EtOAc:MeOH = 10:1); ^{1}H-NMR (400 MHz, MeOD) δ 7.38 (d, J = 8.2 Hz, 1H), 7.29 (s, 1H), 7.01 (dd, J = 1.0, 8.2 Hz, 1H), 3.99 (t, J = 6.6 Hz, 2H), 3.07 (t, J = 6.6 Hz, 2H), 2.42 (s, 3H). ^{13}C-NMR (100 MHz, MeOD) δ 152.7, 138.0, 136.6, 131.6, 123.2, 113.8, 113.6, 59.7, 32.0, 20.3.

2-(5-Fluoro-1*H*-benzo[*d*]imidazol-2-yl)ethanol (23c): The following the same procedure as that used for the synthesis of **23a**, the reaction of 4-fluoro-1,2-phenylenediamine (2.00 g,

[2] G. B. Bachman, L. V. Heisy, *J. Am. Chem. Soc.* **1949**, *71*, 1985-8.
[3] S. Akihama, M. Okude, K. Sato, S. Iwabauchi, Yakugaku Zasshi 1968, 88, 684-689.

15.86 mmol) and 3-hydroxypropionitrile (1.24 g, 17.44 mmol) dissolved in conc. HCl (10 mL) gave benzimidazole **23c** (1.70 g, 59%) as a brown powder; ^{1}H-NMR (400 MHz, MeOD) δ 7.49-7.52 (m, 1H), 7.25 (dd, J = 1.7, 6.8 Hz, 1H), 7.01 (td, J = 1.8, 6.9 Hz, 1H), 4.02 (t, J = 4.8 Hz, 2H), 3.14 (t, J = 4.8 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 159.4 (d, J = 236.0 Hz), 154.5, 137.6 (d, J = 13.0 Hz), 133.7, 114.6 (d, J = 11.0 Hz), 110.2 (d, J = 25.0 Hz), 100.1 (d, J = 27.0 Hz), 59.4, 31.7.

2-(5-Chloro-1*H*-benzo[*d*]imidazol-2-yl)ethanol (23d): The following the same procedure as that used for the synthesis of **23a**, the reaction of 4-chloro-1,2-phenylenediamine (5.00 g, 35.08 mmol) and 3-hydroxypropionitrile (3.12 g, 4.85 mmol) dissolved in conc. HCl (16 mL) gave benzimidazole **23d**[3] (1.53 g, 22%) as a pale-yellow solid after purification by column chromatography on silica gel (EtOAc:MeOH = 10:1); ^{1}H-NMR (400 MHz, MeOD) δ 7.48 (d, J = 1.7 Hz, 1H), 7.42 (d, J = 8.6 Hz, 1H), 7.15 (dd, J = 2.0, 8.6 Hz, 1H), 4.00 (t, J = 6.5 Hz, 2H), 3.09 (t, J = 6.5 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 154.6, 139.1, 136.6, 127.3, 122.1, 114.8, 114.0, 59.5, 32.0.

2-(2-(1*H*-Benzo[*d*]imidazol-2-yl)ethoxy)isoindoline-1,3-dione (23a'): To a solution of *N*-hydroxyphthalimide (700 mg, 4.29 mmol), benzimidazole **23a** (580 mg, 3.58 mmol), and PPh_3 (1.31 g, 5.01 mmol) in distilled THF (17 mL) was added DIAD (0.97 mL, 5.01 mmol) at 0 °C. The reaction mixture was stirred for 1.5h at room temperature. The solvent of reaction mixture was removed under reduced pressure. The resulting mixture was extracted with EtOAc and washed with brine. The organic layer was dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica-gel (EtOAc:*n*-hexane = 2:1) to give *N*-alkoxyphtalimide **23a'** (0.21 g, 19%); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.77-7.80 (m, 2H), 7.72-7.73 (m, 2H), 7.56-7.59 (m, 2H), 7.18-

7.21 (m, 2H), 4.59 (t, *J* = 5.3 Hz, 2H), 3.34 (t, *J* = 5.4Hz, 2H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 164.4 151.5 135.0 128.6 123.9 122.3 76.3 28.6.

2-(2-(5-Methyl-1*H*-benzo[*d*]imidazol-2-yl)ethoxy)isoindoline-1,3-dione (23b'): The following the same procedure as that used for the synthesis of **23a'**, the reaction of *N*-hydroxyphthalimide (330 mg, 2.02 mmol), methylbenzimidazole **23b** (356 mg, 2.02 mmol), PPh_3 (583 mg, 2.22 mmol) and DIAD (0.43 mL, 2.22 mmol) in distilled THF (12 mL) gave the *N*-alkoxyphthalimide **23b'** (350 mg, 54%) as a white solid after purification by column chromatography on silica gel (EtOAc:$CHCl_3$=1:1);^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.80-7.81 (m, 2H), 7.72-7.75 (m, 2H), 7.44 (d, *J* = 8.0 Hz, 1H) 7.34 (s, 1H), 7.01 (dd, *J* = 1.1, 8.2 Hz, 1H) 4.56 (t, *J* = 5.4 Hz, 2H), 3.29 (t, *J* = 5.4 Hz, 2H).

2-(2-(5-Fluoro-1*H*-benzo[*d*]imidazol-2-yl)ethoxy)isoindoline-1,3-dione (23c'): The following the same procedure as that used for the synthesis of **23a'**, the reaction of *N*-hydroxyphthalimide (1.53 g, 9.38 mmol), fluorobenzimidazole **23c** (1.69 g, 9.38 mmol), PPh_3 (2.71 g, 10.32 mmol) and DIAD (2.00 mL, 10.32 mmol) in distilled THF (60 mL) gave the *N*-alkoxyphtalimide **23c'** (0.17 g, 6%) as a white solid after purification by column chromatography on silica gel (EtOAc:$CHCl_3$ = 1:1); ^{1}H-NMR (400 MHz, DMSO) δ 7.84 (s, 4H), 7.41-7.44 (m, 1H), 7.24 (dd, *J* = 2.2, 9.5 Hz, 1H), 6.96 (t, *J* = 8.5 Hz, 1H), 4.64 (t, *J* = 6.8 Hz, 2H), 3.31 (t, *J* = 6.8 Hz, 2H). ^{13}C-NMR (100 MHz, DMSO) δ 163.7, [158.8 (d, *J* = 234.0 Hz) and 158.5 (d, *J* = 232.0 Hz)], [153.6 and 152.6], [144.0 (d, *J* = 12.0 Hz) and 134.7 (d, *J* = 14.0 Hz)], [140.2 and 131.3], 135.2, 129.1, 123.6, [119.3 (d, *J* = 10.0 Hz) and 111.9 (d, *J* = 1.0 Hz)], [109.9 (d, *J* = 25.0 Hz) and 109.3 (d, *J* = 24.0 Hz)], [104.1 (d, *J* = 23.0 Hz) and 97.8 (d, *J* = 27.0 Hz)], 75.3, 28.8.

2-(2-(5-Chloro-1*H*-benzo[*d*]imidazol-2-yl)ethoxy)isoindoline-1,3-dione (23d'): The following the same procedure as that used for the synthesis of **23a'**, the reaction of *N*-hydroxyphthalimide (292 mg, 1.79 mmol), chlorobenzimidazole **23d** (352 mg, 1.79 mmol), PPh_3 (516 mg, 1.97 mmol) and DIAD (0.38 mL, 1.97 mmol) in distilled THF (10 mL) gave the *N*-alkoxy phthalimide **23d'** (44 mg, 7%) as a white solid after purification by column chromatography on silica gel (EtOAc:$CHCl_3$ = 1:1); ^{1}H-NMR (400 MHz, DMSO) δ 7.84 (s, 4H), 7.44-7.50 (m, 2H), 7.11-7.16 (m, 1H), 4.64 (t, J = 6.6 Hz, 2H), 3.31 (t, J = 6.6 Hz, 2H). ^{13}C-NMR (100 MHz, DMSO) δ 163.7, [153.6 and 153.1], [144.6 and 142.4], 135.2, [135.4 and 133.5], 129.0, [126.5 and 125.8], 123.6, [122.2 and 121.7], [119.8 and 118.0], [112.6 and 111.2], 75.2, 28.8.

***O*-(2-(1*H*-Benzo[*d*]imidazol-2-yl)ethyl)hydroxylamine hydrochloride (5a):** To a solution of *N*-alkoxyphthalimide **23a'** (120 mg, 0.39 mmol) in EtOH (3 mL) was added hydrazine monohydrate at room temperature. The reaction mixture was stirred for 3h at 80 °C. The resulting solution was filtered through a pad of silica gel and washed with EtOAc. The filtrate was solidified by treatment of 1M solution of HCl in Et_2O (0.39 mL) to give *N*-alkoxyamine **5a** (45 mg, 54%); ^{1}H-NMR (300 MHz, MeOD) δ 7.81-7.85 (m, 2H), 7.61-7.65 (m, 2H), 4.62 (t, J = 5.8 Hz, 2H), 3.69 (t, J = 5.9 Hz, 2H).

***O*-(2-(5-Methyl-1*H*-benzo[*d*]imidazol-2-yl)ethyl)hydroxylamine hydrochloride (5b):** The following the same procedure as that used for the synthesis of **5a**, the reaction of *N*-alkoxyphthalimide **23b'** (350 mg, 1.09 mmol), hydrazine monohydrate in EtOH (4 mL) gave the *N*-alkoxyamine **5b** (120 mg, 48 %); ^{1}H-NMR (400 MHz, MeOD) δ 7.70 (d, J = 8.4 Hz, 1H), 7.62 (d, J = 0.6 Hz, 1H), 7.45 (dd, J = 0.7, 8.5 Hz, 1H), 4.66 (t, J = 5.8 Hz, 2H), 3.70 (t, J = 5.8 Hz, 2H), 2.56 (s, 3H). ^{13}C-NMR (100 MHz, MeOD) δ 149.4, 137.1, 131.2, 128.9,

127.8, 113.0, 113.0, 70.7, 25.6, 20.3.

***O*-(2-(5-Fluoro-1*H*-benzo[*d*]imidazol-2-yl)ethyl)hydroxylamine hydrochloride (5c):** The following the same procedure as that used for the synthesis of **5a**, the reaction of *N*-alkoxyphthalimide **23c'** (167 mg, 0.51 mmol), hydrazine monohydrate in EtOH (2 mL) gave the *N*-alkoxyamine **5c** (101 mg, 85 %); ^{1}H-NMR (400 MHz, MeOD) δ 7.87 (dd, J = 4.3, 9.1 Hz, 1H), 7.63 (d, J = 2.2, 8.2 Hz, 1H), 7.44 (td, J = 2.4, 9.3 Hz, 1H), 4.67 (t, J = 5.8 Hz, 2H), 3.72 (t, J = 5.8 Hz, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 161.1 (d, J = 243.0 Hz), 151.3, 131.5 (d, J = 13.0 Hz), 127.5, 115.2 (d, J = 10.0 Hz), 114.8 (d, J = 26.0 Hz), 100.2 (d, J = 28.0 Hz), 70.6, 25.7.

***O*-(2-(5-Chloro-1*H*-benzo[*d*]imidazol-2-yl)ethyl)hydroxylamine hydrochloride (5d):** The following the same procedure as that used for the synthesis of **5a**, the reaction of *N*-alkoxyphthalimide **23d'** (140 mg, 0.41 mmol), hydrazine monohydrate in EtOH (3 mL) gave the *N*-alkoxyamine **5d** (52 mg, 51%); ^{1}H-NMR (400 MHz, MeOD) δ 7.91 (s, 1H) 7.83-7.85 (m, 1H) 7.63-7.67 (m, 1H) 4.62-4.66 (m, 2H), 3.69-3.72 (m, 2H). ^{13}C-NMR (100 MHz, MeOD) δ 151.3, 132.0, 131.7, 129.7, 126.9, 114.9, 113.6, 70.5, 25.7.

II. Experimental Procedures for 2ab-g, 2cb-g, 2db, and 2fb-d in Scheme 5

(±)-(3*S*,4*S*)-7-Fluoro-4-isopropyl-3-(2-((3-(6-methyl-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)chroman-3-ol (2ab)

The following the same procedure as that used for the synthesis of **2aa**, the reaction of amine **4b (**19.7 mg, 104 μmol), sodium cyanoborohydride (6.53 mg, 104 μmol), aldehyde **3** (26.2 mg, 104 μmol) and acetic acid in MeOH (2 mL) gave amine **2ab** (13.5 mg, 31%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4OH = 80:20:1:1) as a brown oil; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.38 (d, J = 8.2 Hz, 1H), 7.26 (d, J = 6.9 Hz, 1H), 7.02 (d, J = 8.1 Hz, 1H), 6.94 (dd, J = 6.7, 8.3 Hz, 1H), 6.57 (td, J = 2.6, 8.3 Hz, 1H), 6.49 (dd, J = 2.5, 10.2 Hz, 1H) 4.11 (d, J = 11.0 Hz, 1H), 3.95 (dd, J = 1.6, 10.9 Hz, 1H), 3.00-2.92 (m, 4H), 2.76-2.65 (m, 2H) 2.57 (s, 1H), 2.44 (s, 3H), 2.02 (m, J = 6.1 Hz, 2H), 1.70 (m, J = 6.1 Hz, 2H), 1.18 (d, J = 6.9 Hz, 3H), 0.65 (d, J = 6.9 Hz, 3H).^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.3 (d, ^1J = 242 Hz), 153.8, 153.6 (d, ^3J = 12 Hz), 138.0, 136.7, 132.2, 131.7 (d, ^3J = 10 Hz), 123.8, 118.8 (d, ^4J = 3 Hz), 114.6, 114.1, 107.1 (d, ^2J = 22 Hz), 103.1 (d, ^2J = 25 Hz), 71.0, 67.8, 51.6, 47.7, 44.8, 34.6, 27.8, 27.0, 26.6, 25.5, 21.6, 21.0. HPLC: 96.4%, RT 17.52 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{33}FN_3O_2$ 426.2551, found 426.2555.

(±)-(3*S*,4*S*)-7-Fluoro-3-(2-((3-(6-fluoro-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)-4-isopropylchroman-3-ol (2ac)

The following the same procedure as that used for the synthesis of **2aa**, the reaction of amine **4c** (53.7 mg, 278 μmol), sodium cyanoborohydride (8.46 mg, 139 μmol), aldehyde **3** (35.9 mg, 139 μmol) and acetic acid in MeOH (3 mL) gave amine **2ac** ((15.6 mg, 26%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4OH =

80:20:1:1) as a brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.36 (dd, J = 4.6, 8.7 Hz, 1H), 7.16 (dd, J = 2.1, 8.9 Hz, 1H), 6.95-6.89 (m, 2H), 6.57 (td, J = 2.5, 8.3 Hz, 1H), 6.49 (dd, J = 2.5, 10.1 Hz, 1H), 4.10 (d, J = 11.0 Hz, 1H), 3.96 (dd, J = 1.5, 11.0 Hz, 1H), 3.10-3.01 (m, 4H), 2.97 (t, J = 6.4 Hz, 2H), 2.87-2.75 (m, 2H), 2.57 (s, 1H), 2.40-2.33 (m, 1H), 2.04 (t, J = 5.9 Hz, 2H), 1.82 (m, 2H), 1.16 (d, J = 6.9 Hz, 3H), 0.64 (d, J = 5.9 Hz, 3H).^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.3 (d, ^{1}J = 243 Hz), 159.3 (d, ^{1}J = 237 Hz), 155.3 (d, ^{4}J = 2 Hz), 153.5 (d, ^{3}J = 11 Hz), 138.3 (d, ^{3}J = 12 Hz), 134.5, 131.7 (d, ^{3}J = 10 Hz), 118.7 (d, ^{4}J = 3 Hz), 115.1 (d, ^{3}J = 10 Hz), 110.6 (d, ^{2}J = 25 Hz), 107.2 (d, ^{2}J = 22 Hz), 103.2 (d, ^{2}J = 24 Hz), 100.9 (d, ^{2}J = 26 Hz), 70.9, 67.8, 51.4, 47.9, 44.6, 34.8, 27.9, 26.8, 26.2, 25.4, 21.0. HPLC: 96.2%, RT 13.38 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{30}F_2N_3O_2$ 430.2301, found 430.2303.

(±)-(3*S*,4*S*)-3-(2-((3-(6-Chloro-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2ad)

The following the same procedure as that used for the synthesis of **2aa**, the reaction of amine **4d** (31.7 mg, 151 μmol), sodium triacetoxyborohydride (48.0 mg, 227 μmol), aldehyde **3** (38.1 mg, 151 μmol) and acetic acid in MeOH (3 mL) gave the secondary amine **2ad** (17.4 mg, 26%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4OH = 80:20:1:1) as a white oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 1.8 Hz, 1H), 7.40 (d, J = 8.5 Hz, 1H), 7.16 (dd, J = 1.9, 8.5 Hz, 1H), 6.94 (dd, J = 6.6, 8.5 Hz, 1H), 6.56 (td, J = 2.6, 8.3 Hz, 1H), 6.49 (dd, J = 2.6, 10.2 Hz, 1H) 4.10 (d, J = 11.0 Hz, 1H), 3.94 (dd, J = 2.0, 11.0 Hz, 1H), 2.97-2.94 (m, 4H), 2.73 (m, J = 6.1 Hz, 1H), 2.65 (m, J = 6.1 Hz, 1H), 2.56 (s, 1H), 2.42-2.34 (m, 1H), 2.03-1.99 (m, 2H), 1.74-1.56 (m, 2H), 1.17 (d, J = 7.0 Hz, 3H), 0.66 (d, J = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^{1}J = 242 Hz), 155.2, 153.6 (d, ^{3}J = 11 Hz), 139.2, 137.0, 131.7 (d, ^{3}J = 9 Hz), 127.9, 122.8, 118.8 (d, ^{4}J = 3 Hz), 115.3, 114.7, 107.1 (d, ^{2}J = 21 Hz), 103.2 (d, ^{2}J = 24 Hz), 71.3, 67.9,

51.7, 47.5, 45.1, 34.6, 27.9, 27.1, 26.5, 25.5, 21.0. HPLC: 93.0%, RT 16.65 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{30}ClFN_3O_2$ 446.2005, found 446.2008.

(±)-(3*S*,4*S*)-3-(2-((3-(6-Bromo-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2ae)

The following the same procedure as that used for the synthesis of **2aa**, the reaction of amine **4e** (41.5 mg, 143 μmol), sodium cyanoborohydride (5.99 mg, 95.2 μmol) and aldehyde **3** (24.6 mg, 95.2 μmol) in MeOH (2 mL) gave amine **2ae** (6.70 mg, 14%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4OH = 80:20:1:1) as a brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 1.5 Hz, 1H), 7.31-7.24 (m, 2H), 6.95 (dd, J = 6.5, 8.5 Hz, 1H), 6.58 (td, J = 2.6, 8.3 Hz, 1H), 6.50 (dd, J = 2.5, 10.1 Hz, 1H), 4.12 (d, J = 11.1 Hz, 1H), 3.99 (dd, J = 1.8, 11.1 Hz, 1H), 3.15-3.01 (m, 2H), 2.97 (t, J = 6.4 Hz, 2H), 2.92-2.91 (m, 2H), 2.59 (s, 1H), 2.41-2.32 (m, 1H), 2.04 (t, J = 6.0 Hz, 2H), 1.89 (t, J = 5.8 Hz, 2H), 1.18 (d, J = 7.0 Hz, 3H), 0.65 (d, J = 6.9 Hz, 3H).^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 243 Hz), 154.3, 153.4 (d, ^3J = 12 Hz), 138.3, 136.0, 131.7 (d, ^3J = 10 Hz), 130.9, 126.0, 118.2 (d, ^4J = 3 Hz), 117.6, 115.9, 107.6 (d, ^2J = 21 Hz), 103.3(d, ^2J = 24 Hz), 70.2, 67.2, 50.9, 48.1, 43.9, 34.5, 28.1, 26.7, 25.4, 23.4, 21.0. HPLC: 95.2%, RT 13.72 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{30}BrFN_3O_2$ 490.1500, found 490.1501.

(±)-(3*S*,4*S*)-3-(2-((3-(5,6-Dichloro-1H-benzo[d]imidazol-2-yl)propyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2af)

The following the same procedure as that used for the synthesis of **2aa**, the reaction of amine **4f** (60.4 mg, 2.47 mmol), sodium cyanoborohydride (20.7 mg, 333 μmol), aldehyde **3** (42.6 mg, 165 μmol) and acetic acid in MeOH (3 mL) gave amine **2af** (36.6 mg, 46%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4OH =

80:20:1:1) as a brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 6.94 (dd, J = 6.6, 8.5 Hz, 1H), 6.56 (td, J = 2.6, 8.3 Hz, 1H), 6.48 (dd, J = 2.6, 10.2 Hz, 1H) 4.08 (d, J = 10.9 Hz, 1H), 3.91 (dd, J = 2.0, 11.0 Hz, 1H), 2.97-2.93 (m, 4H), 2.71 (m, J = 6.1 Hz, 1H), 2.63 (m, J = 6.1 Hz, 1H), 2.54 (s, 1H), 2.38-2.29 (m, 1H), 2.00 (m, J = 6.5 Hz, 2H), 1.68-1.51 (m, 2H), 1.16 (d, J = 7.0 Hz, 3H), 0.64 (d, J = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.3 (d, ^{1}J = 242 Hz), 156.5, 153.5 (d, ^{3}J = 12 Hz), 137.8, 131.7 (d, ^{3}J = 10 Hz), 126.1, 118.7 (d, ^{4}J = 3 Hz), 115.9, 107.2 (d, ^{2}J = 22 Hz), 103.2 (d, ^{2}J = 24 Hz), 71.6, 67.9, 51.7, 47.6, 45.2, 34.6, 27.8, 27.4, 26.5, 25.5, 21.0. HPLC: 91.5%, RT 4.31 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{29}Cl_2FN_3O_2$ 480.1615, found 480.1617.

(±)-(3*S*,4*S*)-7-Fluoro-4-isopropyl-3-(2-((3-(6-nitro-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)chroman-3-ol (2ag)

Acetic acid was added to a solution of amine **4g** (44.3 mg, 171 μmol) in anhydrous MeOH (3 mL) until pH 6 was reached. To this solution was added sodium cyanoborohydride (10.8 mg, 171 μmol), aldehyde **3** (43.5 mg, 171 μmol). After stirring for 3.5 h at room temperature, the reaction mixture was stirred for an additional 5 h at 45 °C. The reaction was monitored by TLC, quenched with saturated aqueous $NaHCO_3$ (15 mL) after completion of the reaction and extracted with CH_2Cl_2 (3 x 20 mL). The organic layers were washed with brine, dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel (CH_2Cl_2:MeOH:H_2O:NH_4OH = 80:20:1:1) to afford amine **2ag** (17.6 mg, 24%) as a yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.47 (d, J = 2.1 Hz, 1H), 8.13 (dd, J = 2.2, 8.9 Hz, 1H), 7.55 (d, J = 8.9 Hz, 1H), 6.96 (dd, J = 6.5, 8.5 Hz, 1H), 6.57 (td, J = 2.6, 8.3 Hz, 1H), 6.50 (dd, J = 2.6, 10.1 Hz, 1H) 4.13 (d, J = 11.0 Hz, 1H), 3.96 (dd, J = 2.0, 11.0 Hz, 1H), 3.06 (t, J = 6.7 Hz, 2H), 3.03-2.95 (m, 2H) 2.79-2.73 (m, 1H), 2.70-2.64 (m, 1H), 2.59 (d, J = 2.1 Hz, 1H), 2.45-2.37 (m, 1H), 2.09 (m, J

= 6.3 Hz, 2H), 1.70-1.63 (m, 2H), 1.21 (d, *J* = 7.0 Hz, 3H), 0.69 (d, *J* = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 243 Hz), 158.57, 153.5 (d, ^3J = 12 Hz), 143.2, 142.4, 138.0, 131.7 (d, ^3J = 10 Hz), 118.5 (d, ^4J = 3 Hz), 118.2, 114.1, 111,7, 107.4 (d, ^2J = 21 Hz), 103.3 (d, ^2J = 24 Hz), 71.4, 67.6, 51.5, 47.7, 44.9, 34.5, 28.0, 26.8, 26.1, 25.5, 21.0. HPLC: 98.2%, RT 12.66 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{30}FN_4O_4$ 457.2246, found 457.2246.

(±)-(3*S*,4*S*)-7-Fluoro-4-isopropyl-3-(2-(isopropyl(3-(6-methyl-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)chroman-3-ol (2cb)

The following the same procedure as that used for the synthesis of **2ca**, the reaction of amine **2ab (**33.3 mg, 78.3 μmol), acetic acid (8.96 μL, 156 μmol), sodium cyanoborohydride (9.84 mg, 156 μmol) and acetone (23.0 μL, 313 μmol) in MeOH (1 mL) gave amine **2cb** (36.6 mg, 46%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) as a pale brown oil; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 10.9 Hz, 1H), 7.30 (s, 1H), 7.03 (d, *J* = 10.9 Hz, 1H), 6.92 (dd, *J* = 8.9, 11.0 Hz, 1H), 6.55 (td, *J* = 3.4, 11.1 Hz, 1H), 6.48 (dd, *J* = 3.3, 13.6 Hz, 1H), 4.12 (d, *J* = 14.5 Hz, 1H), 3.93 (dd, *J* = 2.2, 14.6 Hz, 1H), 3.20 (m, 1H), 2.96-2.89 (m, 2H), 2.75 (t, *J* = 7.6 Hz, 2H), 2.56-2.45 (m, 2H), 2.45-2.34 (m, 4H), 2.15-2.01 (m, 2H), 1.66-1.60 (m, 2H), 1.16 (d, *J* = 9.3 Hz, 3H), 1.07 (d, *J* = 8.8 Hz, 6H), 0.64 (d, *J* = 9.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 242 Hz), 154.1, 153.6 (d, ^3J = 12 Hz), 138.1, 136.8, 132.1, 131.6 (d, ^3J = 10 Hz), 123.7, 118.8 (d, ^4J = 3 Hz), 114.5, 114.1, 107.0 (d, ^2J = 21 Hz), 103.1 (d, ^2J = 24 Hz), 71.1, 68.4, 51.4, 49.8, 48.7, 45.5, 32.2, 27.7, 26.5, 25.6, 25.5, 21.6, 20.9, 17.3, 16.6. HPLC: 95.9%, RT 16.91 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{28}H_{39}FN_3O_2$ 468.2021, found 468.3024.

(±)-(3*S*,4*S*)-7-Fluoro-3-(2-((3-(6-fluoro-1*H*-benzo[*d*]imidazol-2-yl)propyl)(isopropyl)amino)ethyl)-4-isopropylchroman-3-ol (2cc)

The following the same procedure as that used for the synthesis of **2ca**, the reaction of amine **2ac** (46.7 mg, 109 μmol), acetic acid (12.5 μL, 217 μmol), sodium cyanoborohydride (13.7 mg, 217 μmol) and acetone (31.9 μL, 435 μmol) in MeOH (1 mL) gave amine **2cc** (12.7 mg, 25%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) as a pale brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.22 (d, J = 8.12 Hz, 2H), 7.00-6.93 (m, 2H), 6.57 (td, J = 2.6, 8.3 Hz, 1H), 6.50 (dd, J = 2.5, 10.2 Hz, 1H), 4.15 (d, J = 10.9 Hz, 1H), 3.95 (dd, J = 1.9, 10.8 Hz, 1H), 3.20 (m, 1H), 2.99-2.85 (m, 2H), 2.72 (t, J = 5.6 Hz, 2H), 2.60 (s, 1H), 2.57-2.44 (m, 3H), 2.13-2.00 (m, 2H), 1.66-1.55 (m, 2H), 1.18 (d, J = 7.0 Hz, 3H), 1.07 (dd, J = 4.3, 6.5 Hz, 6H), 0.67 (d, J = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 242 Hz), 159.3 (d, ^1J = 236 Hz), 155.7, 153.6 (d, ^3J = 12 Hz), 138.5, 135.2, 131.5 (d, ^3J = 9 Hz), 118.7 (d, ^4J = 3 Hz), 114.9, 110.3 (d, ^2J = 25 Hz), 107.1 (d, ^2J = 22 Hz), 103.1 (d, ^2J = 25 Hz), 100.8, 71.6, 68.5, 51.5, 49.2, 48.2, 45.6, 32.1, 27.7, 26.3, 26.1, 25.5, 20.8, 17.4, 16.6. HPLC: 95.5%, RT 15.93 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{36}F_2N_3O_2$ 472.2770, found 472.2771.

(±)-(3*S*,4*S*)-3-(2-((3-(6-Chloro-1*H*-benzo[*d*]imidazol-2-yl)propyl)(isopropyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2cd)

The following the same procedure as that used for the synthesis of **2ca**, the reaction of amine **2ad** (29.7 mg, 66.6 μmol), acetic acid (7.63 μL, 133 μmol), sodium cyanoborohydride (8.37 mg, 133 μmol) and acetone (19.6 μL, 266 μmol) in MeOH (1 mL) gave amine **2cd** (4.50 mg, 14%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) as a pale brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.53 (s, 1H), 7.45 (dd, J = 7.2 Hz, 1H), 7.19 (dd, J = 1.8, 8.5 Hz, 1H), 6.95 (d, J = 6.6, 8.3 Hz, 1H), 6.57 (td, J = 2.6, 8.3 Hz, 1H), 6.50

(dd, *J* = 2.6, 10.2 Hz, 1H), 4.15 (d, *J* = 10.8 Hz, 1H), 3.95 (dd, *J* = 1.8, 10.9 Hz, 1H), 3.21 (m, 1H), 2.99-2.86 (m, 2H), 2.73 (t, *J* = 5.5 Hz, 2H), 2.60 (s, 1H), 2.57-2.40 (m, 3H), 2.15-2.00 (m, 2H), 1.65-1.55(m, 2H), 1.18 (d, *J* = 7.0 Hz, 3H), 1.07 (dd, *J* = 4.3, 6.5 Hz, 6H), 0.68 (d, *J* = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 242 Hz), 155.5, 153.6 (d, ^3J = 12 Hz), 139.4, 137.0, 131.5 (d, ^3J = 9 Hz), 127.8, 122.7, 118.6 (d, ^4J = 2 Hz), 115.3, 114.7, 107.1 (d, ^2J = 21 Hz), 103.1 (d, ^2J = 24 Hz), 71.4, 68.4, 51.4, 49.7, 48.4, 45.6, 32.1, 27.7, 26.3, 25.7, 25.5, 20.8, 17.4, 16.6. HPLC: 94.8%, RT 16.74 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{36}ClFN_3O_2$ 488.2475, found 488.2477.

(±)-(3*S*,4*S*)-3-(2-((3-(6-Bromo-1*H*-benzo[*d*]imidazol-2-yl)propyl)(isopropyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2ce)

The following the same procedure as that used for the synthesis of **2ca**, the reaction of amine **2ae** (63.4 mg, 129 μmol), acetic acid (14.4 μL, 259 μmol), sodium cyanoborohydride (16.2 mg, 259 μmol) and acetone (38.0 μL, 517 μmol) in MeOH (1.3 mL) gave amine **2ce** (20.2 mg, 29%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) as a brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.67 (s, 1H), 7.39 (d, *J* = 8.6 Hz, 1H), 6.94 (dd, J = 6.6, 8.5 Hz, 1H), 6.56 (td, *J* = 2.6, 8.3 Hz, 1H), 6.50 (dd, *J* = 2.6, 10.2 Hz, 1H), 4.13 (d, *J* = 10.8 Hz, 1H), 3.93 (dd, *J* = 2.0, 10.9 Hz, 1H), 3.22-3.14 (m, 1H), 2.99-2.86 (m, 2H), 2.72 (t, *J* = 5.6 Hz, 2H), 2.58-2.48 (m, 3H), 2.45-2.38 (m, 1H), 2.13-1.99 (m, 2H), 1.62-1.58 (m, 2H), 1.16 (d, *J* = 7.0 Hz, 3H), 1.06 (dd, *J* = 3.9, 6.6 Hz, 6H), 0.66 (d, *J* = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 243 Hz), 155.6, 153.6 (d, ^3J = 12 Hz), 140.4, 137.7, 131.5 (d, ^3J = 10 Hz), 125.3, 118.7 (d, ^4J = 3 Hz), 117.8, 115.7, 115.1, 107.1 (d, ^2J = 21 Hz), 103.1 (d, ^2J = 24 Hz), 71.7, 68.5, 51.5, 49.0, 48.1, 45.6, 32.1, 27.6, 26.2, 26.1 25.5, 20.8, 17.5, 16.6. HPLC: 95.5%, RT 16.91 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{36}BrFN_3O_2$ 532.1969, found 532.1971.

(±)-(3*S*,4*S*)-3-(2-((3-(5,6-Dichloro-1*H*-benzo[*d*]imidazol-2-yl)propyl)(isopropyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2cf)

The following the same procedure as that used for the synthesis of **2ca**, the reaction of amine **2af** (7.20 mg, 15.0 μmol), acetic acid (1.72 μL, 30.0 μmol), sodium cyanoborohydride (1.88 mg, 30.0 μmol) and acetone (4.40 μL, 60.0 μmol) in MeOH (150 μL) gave amine **2cf** (3.70 mg, 47%) after purification by column chromatography on silica gel (CH_2Cl_2 : MeOH = 10 : 1) as a colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.60 (s, 1H), 6.94 (dd, J = 6.5, 8.4 Hz, 1H), 6.58 (td, J = 2.6, 8.3 Hz, 1H), 6.50 (dd, J = 2.5, 10.1 Hz, 1H), 4.12 (d, J = 10.9 Hz, 1H), 3.91 (dd, J = 1.9, 10.9 Hz, 1H), 3.30 (m, J = 6.64 Hz, 1H), 3.04-2.92 (m, 2H), 2.84 (t, J = 5.7 Hz, 2H), 2.66 (m, 2H), 2.47 (s, 1H), 2.43-2.35 (m, 1H), 2.22-2.07 (m, 2H), 1.70-1.67 (m, 1H), 1.18-1.12 (m, 9H), 0.66 (d, J = 6.9 Hz, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$) δ 162.4 (d, J = 243 Hz), 156.8, 153.6 (d, J = 12 Hz), 138.0, 131.5 (d, J = 9 Hz), 126.0, 118.6 (d, J = 3 Hz), 115.9, 107.1 (d, J = 22 Hz), 103.1 (d, J = 25 Hz), 71.6, 68.4, 51.4, 49.2, 48.2, 45.6, 32.1, 27.7, 26.2, 25.9, 25.4, 20.8, 17.5, 16.5. HPLC: 88.9%, RT 4.97 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{35}Cl_2FN_3O_2$ 522.2085, found 522.2086.

(±)-(3*S*,4*S*)-7-Fluoro-4-isopropyl-3-(2-(isopropyl(3-(6-nitro-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)chroman-3-ol (2cg)

The following the same procedure as that used for the synthesis of **2ca**, the reaction of amine **2ag** (40.2 mg, 88.0 μmol), acetic acid (10.1 μL, 176 μmol), sodium cyanoborohydride (11.7 mg, 176 μmol) and acetone (25.9 μL, 352 μmol) in MeOH (1 mL) gave amine **2cg** (17.1 mg, 39%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) as a yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H), 8.17 (dd, J = 2.1, 8.8 Hz, 1H), 7.56 (d, J = 8.76 Hz, 1H), 6.96 (d, J = 6.7, 8.3 Hz, 1H), 6.57 (td, J = 2.5, 8.3 Hz, 1H), 6.51 (dd, J =

2.5, 10.1 Hz, 1H), 4.17 (d, *J* = 10.8 Hz, 1H), 3.95 (dd, *J* = 1.5, 10.8 Hz, 1H), 3.22 (m, 1H), 3.06-2.92 (m, 2H), 2.75 (t, *J* = 5.3 Hz, 2H), 2.62-2.49 (m, 3H), 2.47-2.40 (m, 1H), 2.19-2.02 (m, 2H), 1.63 (t, *J* = 4.9 Hz, 2H), 1.18 (d, *J* = 7.0 Hz, 3H), 1.08 (dd, *J* = 6.7, 9.1 Hz, 6H), 0.69 (d, *J* = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^1J = 243 Hz), 159.3, 153.6 (d, ^3J = 11 Hz), 143.4, 142.9, 138.4, 131.5 (d, ^3J = 9 Hz), 118.6 (d, ^4J = 3 Hz), 118.2, 114.0, 111.9, 107.2 (d, ^2J = 21 Hz), 103.2 (d, ^2J = 24 Hz), 71.9, 68.6, 51.4, 49.0, 48.1, 45.7, 32.0, 27.7, 26.5, 26.2, 25.5, 20.8, 17.7, 16.4. HPLC: 96.7%, RT 14.98 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{36}FN_4O_4$ 499.2715, found 499.2716.

(±)-(3*S*,4*S*)-7-Fluoro-4-isopropyl-3-(2-((2,2,2-trifluoroethyl)amino)ethyl)chroman-3-ol (24b)

To a solution of the aldehyde **3** (31.8 mg, 126 μmol) in dry MeOH (1.5 mL) was sequentially added trifluoroethylamine (28.5 mg, 288 μmol) and sodium cyanoborohydride (13.6 mg, 216 μmol) at room temperature. The reaction was allowed to stir for 18 h at the same temperature. The reaction mixture was quenched with $NaHCO_3$ (10 mL), extracted with CH_2Cl_2 (3 x 15 mL) and washed with brine. The organic layers were dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) to afford amine **24b** (38.8 mg, 80%) as a colorless oil.; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 6.92 (dd, *J* = 6.6, 8.5 Hz, 1H), 6.54 (td, *J* = 2.6, 8.4 Hz, 1H), 6.47 (dd, *J* = 2.6, 10.2 Hz, 1H), 4.04 (d, *J* = 11.0 Hz, 1H), 3.87 (dd, *J* = 2.2, 11.1 Hz, 2H), 3.25-3.12 (m, 2H), 3.05-2.95 (m, 2H), 2.52-2.50 (m, 1H), 2.36-2.27 (m, 1H), 1.68-1.58 (m, 2H), 1.13 (d, *J* = 6.9 Hz, 3H), 0.61 (d, *J* = 7.0 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.3 (d, ^1J = 242 Hz), 153.6 (d, ^3J = 12 Hz), 131.7 (d, ^3J = 10 Hz), 125.0 (q, ^1J = 277 Hz), 119.1 (d, ^4J = 3 Hz), 107.0 (d, ^2J = 21 Hz), 103.1 (d, ^2J = 24 Hz), 70.6, 68.0, 51.5, 50.2 (q, ^2J = 31 Hz), 45.4, 35.9, 27.9, 25.4, 21.3.

(±)-(3*S*,4*S*)-3-(2-(Cyclopropyl(3-(5-methyl-1*H*-benzo[*d*]imidazol-2-yl)propyl)amino)ethyl)-7-fluoro-4-isopropylchroman-3-ol (2db)

The following the same procedure as that used for the synthesis of **2da**, the reaction of amine **24a** (22.9 mg, 78.1 μmol), acetic acid (8.49 μL, 148 μmol), sodium cyanoborohydride (9.32 mg, 148 μmol) and aldehyde **21b** (25.4 mg, 74.2 μmol) in MeOH (1 mL) gave tertiary amine intermediate; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.80-7.77 (m, 2H), 7.51 (d, J = 8.2 Hz, 1H), 7.43 (t, J = 0.8 Hz, 1H), 7.29-7.25 (m, 2H), 7.17-7.13 (m, 1H), 6.92 (dd, J = 6.6, 8.4 Hz, 1H), 6.54 (td, J = 2.6, 8.3 Hz, 1H), 6.48 (dd, J = 2.6, 10.3 Hz, 1H), 4.00 (d, J = 11.0 Hz, 1H), 3.90 (dd, J = 1.8, 11.0 Hz, 1H), 3.12(m, J = 7.0 Hz, 1H), 3.00-2.94 (m, 1H), 2.91-2.77 (m, 3H), 2.51 (s, 2H), 2.50 (s, 2H), 2.38 (d, J = 4.4 Hz, 3H), 2.35-2.26 (m, 1H), 2.22-2.12 (m, 2H), 1.83-1.78 (m, 1H), 1.64 (t, J = 4.9 Hz, 2H), 1.11 (d, J = 7.0 Hz, 3H), 0.59 (d, J = 7.0 Hz, 7H).

The reaction of the above tertiary amine (37.6 mg, 61.0 μmol) and tetrabutylammonium fluoride (607 μmol, 1.0 M solution in THF) in THF (1 mL) gave amine **2db** (16.0 mg, 57%) after purification by column chromatography on silica gel (CH_2Cl_2:MeOH = 10:1) as a brown oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.2 Hz, 1H), 7.33 (s, 1H), 7.02 (dd, J = 1.0, 8.2 Hz, 1H), 6.94 (dd, J = 6.6, 8.5 Hz, 1H), 6.57 (td, J = 3.6, 6.9 Hz, 1H), 6.49 (dd, J = 2.6, 10.2 Hz, 1H), 4.11 (d, J = 10.8 Hz, 1H), 3.93 (dd, J = 2.1, 10.8 Hz, 1H), 2.93-2.83 (m, 4H), 2.65-2.58 (m, 3H), 2.47 (s, 3H), 2.43-2.34 (m, 1H), 2.24-2.11 (m, 2H), 1.68 (m, 1H), 1.57 (t, J = 5.7 Hz, 2H), 1.19 (d, J = 7.0 Hz, 3H), 0.66-0.55 (m, 7H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, ^{1}J = 242 Hz), 154.1, 153.5 (d, ^{3}J = 14 Hz), 138.2, 137.0, 132.0, 131.6 (d, ^{3}J = 10 Hz), 123.7, 118.8 (d, ^{4}J = 3 Hz), 114.8, 114.3, 107.0 (d, ^{2}J = 22 Hz), 103.1 (d, ^{2}J = 25 Hz), 71.6, 68.3, 54.2, 53.1 51.4, 37.9, 32.2, 27.8, 26.6, 25.5, 25.3, 21.6, 21.0, 6.7, 6.3.

HPLC: 96.1%, RT 19.07 min. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{28}H_{37}FN_3O_2$ 466.2864, found 466.2868.

(±)-(3*S*,4*S*)-2-(7-Fluoro-3-hydroxy-4-isopropylchroman-3-yl)acetaldehyde *O*-(2-(6-methyl-1*H*-benzo[*d*]imidazol-2-yl)ethyl) oxime (2fb)

The following the same procedure as that used for the synthesis of **2fa**, the reaction of aldehyde **3** (41 mg, 0.16 mmol), K_2CO_3 (34 mg, 0.24 mmol) and *N*-alkoxyamine **5b** (41 mg, 0.18 mmol) in MeOH (1 mL) gave an inseparable mixture of oximes **2fb** (17 mg, 25%, *E/Z* = 1:1) after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 1:1); ***isomer A***: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.48 (t, *J* = 6.0 Hz, 1H), 7.41-7.45 (m, 1H), 7.30-7.32 (m, 1H), 7.02-7.08 (m, 1H), 6.93-6.98 (m, 1H), 6.49-6.62 (m, 2H), 4.38-4.42 (m, 2H), 4.06 (d, *J* = 11.0 Hz, 1H), 3.87 (dd, *J* = 2.0, 11.0 Hz, 1H), 3.18-3.30 (m, 2H), 2.58-2.63 (m, 1H), 2.36-2.44 (m, 6H), 1.14 (d, *J* = 7.0 Hz, 3H), 0.66 (d, *J* = 6.6 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 243.0 Hz), 153.6, 151.9 149.0, 138.0 (broad), 136.7 (broad), 132.3, 131.7, 123.9, 118.5, 114.6 (broad), 114.2 (broad), 107.5 (d, *J* = 21.0 Hz), 103.4 (d, *J* = 24.0 Hz), 71.1, 70.4, 68.2, 50.8, 38.5, 29.3, 28.3, 25.1, 21.0. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{29}FN_3O_3$ 426.2188, found 426.2186; ***isomer B***: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.41-7.45 (m, 1H), 7.30-7.32 (m, 1H), 6.93-7.08 (m, 3H), 6.49-6.62 (m, 2H), 4.38-4.42 (m, 2H), 4.13 (d, *J* = 11.2 Hz, 1H), 3.92 (dd, *J* = 2.0, 11.0 Hz, 1H), 3.18-3.30 (m, 2H), 2.58-2.63 (m, 2H), 2.36-2.44 (m, 5H), 1.17 (d, *J* = 7.0 Hz, 3H), 0.68 (d, *J* = 6.4 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 243.0 Hz), 153.5, 152.1, 148.8 138.0 (broad), 136.7 (broad), 132.3, 131.6, 123.9, 118.5, 114.6 (broad), 114.2 (broad), 107.5 (d, *J* = 21.0 Hz), 103.3 (d, *J* = 24.0 Hz), 71.4, 70.6, 68.7, 51.0, 35.4, 29.3, 28.1, 25.2, 21.0. HPLC (mixture): 88.2%, RT 5.83 min.

(±)-(3*S*,4*S*)-2-(7-Fluoro-3-hydroxy-4-isopropylchroman-3-yl)acetaldehyde *O*-(2-(6-fluoro-1*H*-benzo[*d*]imidazol-2-yl)ethyl) oxime (2fc)

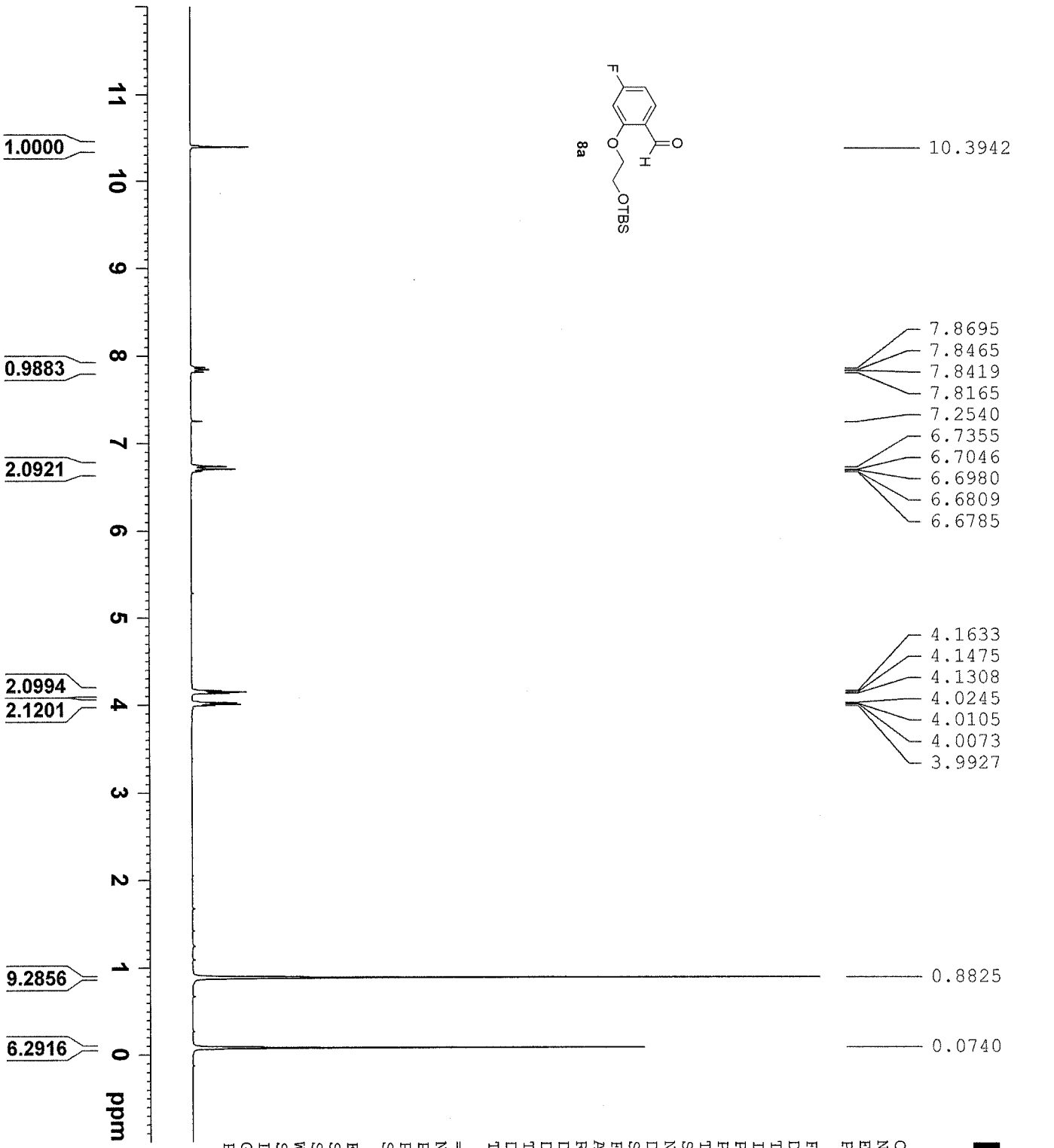
The following the same procedure as that used for the synthesis of **2fa**, the reaction of aldehyde **3** (48 mg, 0.19 mmol), K_2CO_3 (39 mg, 0.29 mmol) and *N*-alkoxyamine **5c** (52 mg, 0.21 mmol) in MeOH (1 mL) gave an inseparable mixture of oximes **2fc** (17 mg, 21%, *E/Z* = 2:1) after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 1:1); ***major isomer***: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.46-7.53 (m, 2H), 7.23-7.25 (m, 1H), 6.93-7.02 (m, 2H), 6.51-6.62 (m, 2H), 4.42-4.46 (m, 2H), 4.05 (d, *J* = 11.0 Hz, 1H), 3.88 (dd, *J* = 1.9, 11.0 Hz, 1H), 3.25-3.34 (m, 2H), 2.59-2.63 (m, 1H), 2.31-2.42 (m, 3H), 1.13 (d, *J* = 7.0 Hz, 3H), 0.66 (d, *J* = 7.0 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 163.4 (d, *J* = 243.0 Hz), 159.6 (d, *J* = 237.0 Hz), 153.4 (broad), 149.2, 137.5 (broad), 133.8 (broad), 131.7, 131.6, 118.4, 115.2 (broad), 111.3 (d, *J* = 25.0 Hz), 107.6 (d, *J* = 21.0 Hz), 103.4 (d, *J* = 24.0 Hz), 101.0 (d, *J* = 26.0 Hz), 70.8, 70.5, 68.2, 50.8, 38.5, 29.1, 28.3, 25.1, 21.0. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{23}H_{26}F_2N_3O_3$ 430.1937, found 430.1933; ***minor isomer***: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.46-7.53 (m, 1H), 7.23-7.25 (m, 1H), 6.93-7.02 (m, 3H), 6.51-6.62 (m, 2H), 4.42-4.46 (m, 2H), 4.12 (d, *J* = 9.8 Hz, 1H), 3.90-3.93 (m, 1H), 3.25-3.34 (m, 2H), 2.59-2.63 (m, 3H), 2.31-2.42 (m, 1H), 1.16 (d, *J* = 7.0 Hz, 3H), 0.67 (d, *J* = 7.0 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 163.5 (d, *J* = 243.0 Hz), 159.6 (d, *J* = 237.0 Hz), 153.4 (broad), 148.7, 137.5 (broad), 133.8 (broad), 131.7, 131.6, 118.4, 115.2 (broad), 11123 (d, *J* = 26.0 Hz), 107.6 (d, *J* = 19.0 Hz), 103.4 (d, *J* = 24.0 Hz), 101.0 (d, *J* = 26.0 Hz), 71.1, 70.9, 68.5, 51.0, 35.4, 29.4, 28.2, 25.2, 21.0. HPLC (mixture): 86.9%, RT 3.52 min.

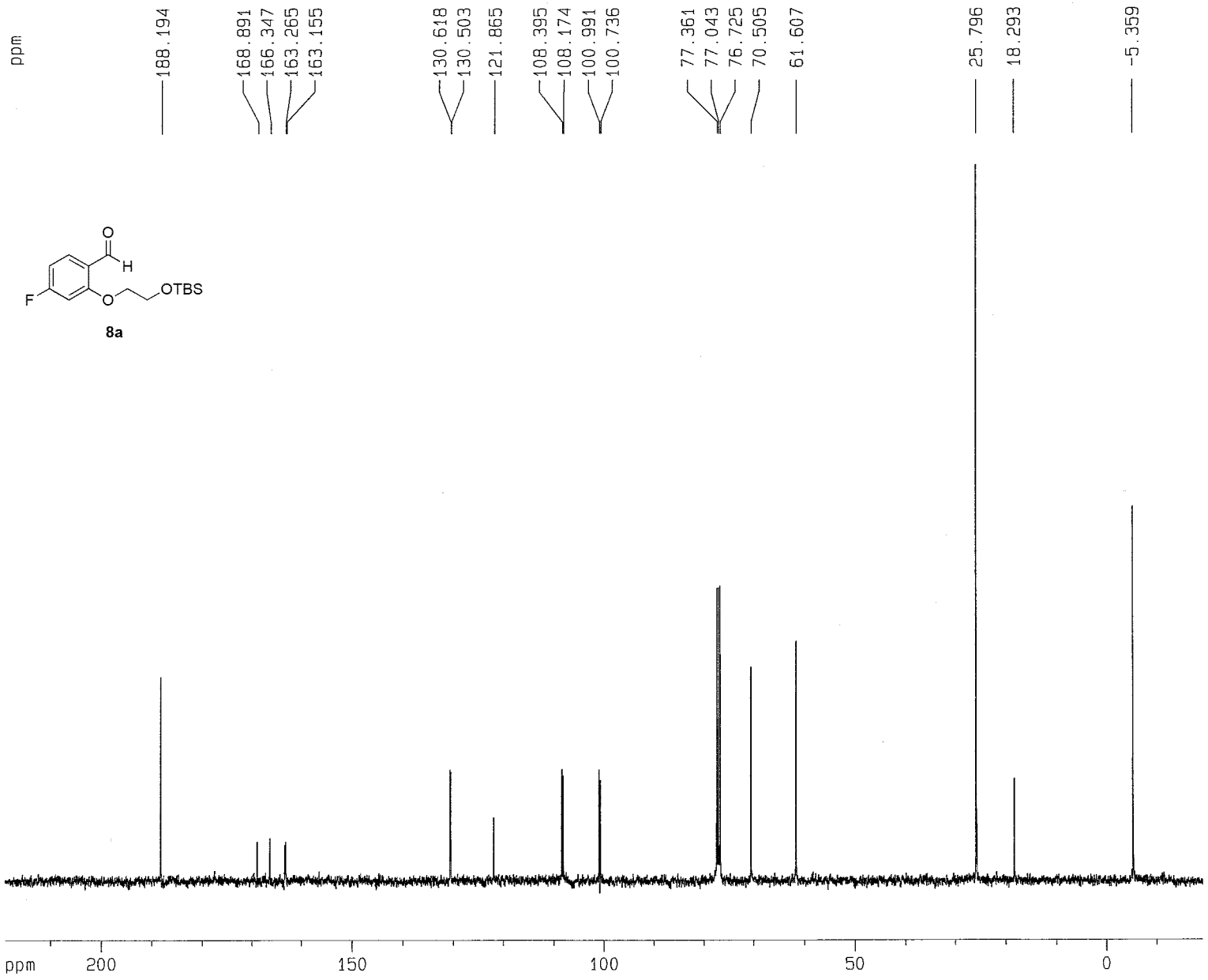
(±)-(3*S*,4*S*)-2-(7-Fluoro-3-hydroxy-4-isopropylchroman-3-yl)acetaldehyde *O*-(2-(6-chloro-1*H*-benzo[*d*]imidazol-2-yl)ethyl) oxime (2fd)

The following the same procedure as that used for the synthesis of **2fa**, the reaction of

aldehyde **3** (48 mg, 0.19 mmol), K_2CO_3 and *N*-alkoxyamine **5d** (52 mg, 0.21 mmol) in MeOH (1 mL) gave the inseparable mixture of oximes **2fd** (18 mg, 21%, *E/Z* = 2:1) after purification by column chromatography on silica gel (EtOAc:*n*-hexane = 1:1); ***major isomer***: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.4-7.53 (m, 2H), 7.41-7.43 (m, 1H), 7.16-7.19 (m, 1H), 6.93-6.97 (m, 1H), 6.52-6.63 (m, 2H), 4.40-4.44 (m, 2H), 4.07 (d, *J* = 11.4 Hz, 1H), 3.86 (dd, *J* = 1.9, 10.9 Hz, 1H), 3.22-3.32 (m, 2H), 2.58 (s, 1H), 2.04-2.41 (m, 3H), 1.11 (d, *J* = 5.0 Hz, 3H), 0.66 (d, *J* = 6.5 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 243.0 Hz), 153.5 (broad), 149.0, 139.0 (broad), 137.0 (broad), 131.7 (d, *J* = 100.0 Hz), 128.1, 122.0, 118.4, 115.6 (broad), 114.7 (broad),107.6 (d, *J* = 21.0 Hz), 103.4 (d, *J* = 24.0 Hz), 70.9, 70.6, 68.2, 50.8, 38.5, 29.4, 28.3, 25.1, 21.0. HRMS-ESI (*m/z*): $[M+H]^+$ calcd for $C_{23}H_{26}ClFN_3O_3$ 446.1641, found 446.1638; ***minor isomer***: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.4-7.53 (m, 2H), 7.41-7.43 (m, 1H), 7.16-7.19 (m, 1H), 6.93-6.97 (m, 1H), 6.52-6.63 (m, 2H), 4.40-4.44 (m, 2H), 4.12 (d, *J* = 9.9 Hz, 1H), 3.91 (dd, *J* = 1.9, 10.9 Hz, 1H), 3.22-3.32 (m, 2H), 2.60-2.62 (m, 3H), 2.04-2.41 (m, 1H), 1.15 (d, *J* = 5.9 Hz, 3H), 0.68 (d, *J* = 6.8 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 163.4 (d, *J* = 242.0 Hz), 153.5 (broad), 148.6, 139.0 (broad), 137.0 (broad), 131.7(d, *J* = 100.0 Hz), 128.0, 122.0, 118.4, 115.6 (broad), 114.7 (broad), 107.7 (d, *J* = 21.0 Hz), 103.4 (d, *J* = 24.0 Hz), 71.2, 70.9, 68.5, 51.0, 35.4, 29.3, 28.2, 25.2, 20.9. HPLC (mixture): 86.3%, RT 6.40 min.

III. Spectral Data



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1D NMR plot parameters
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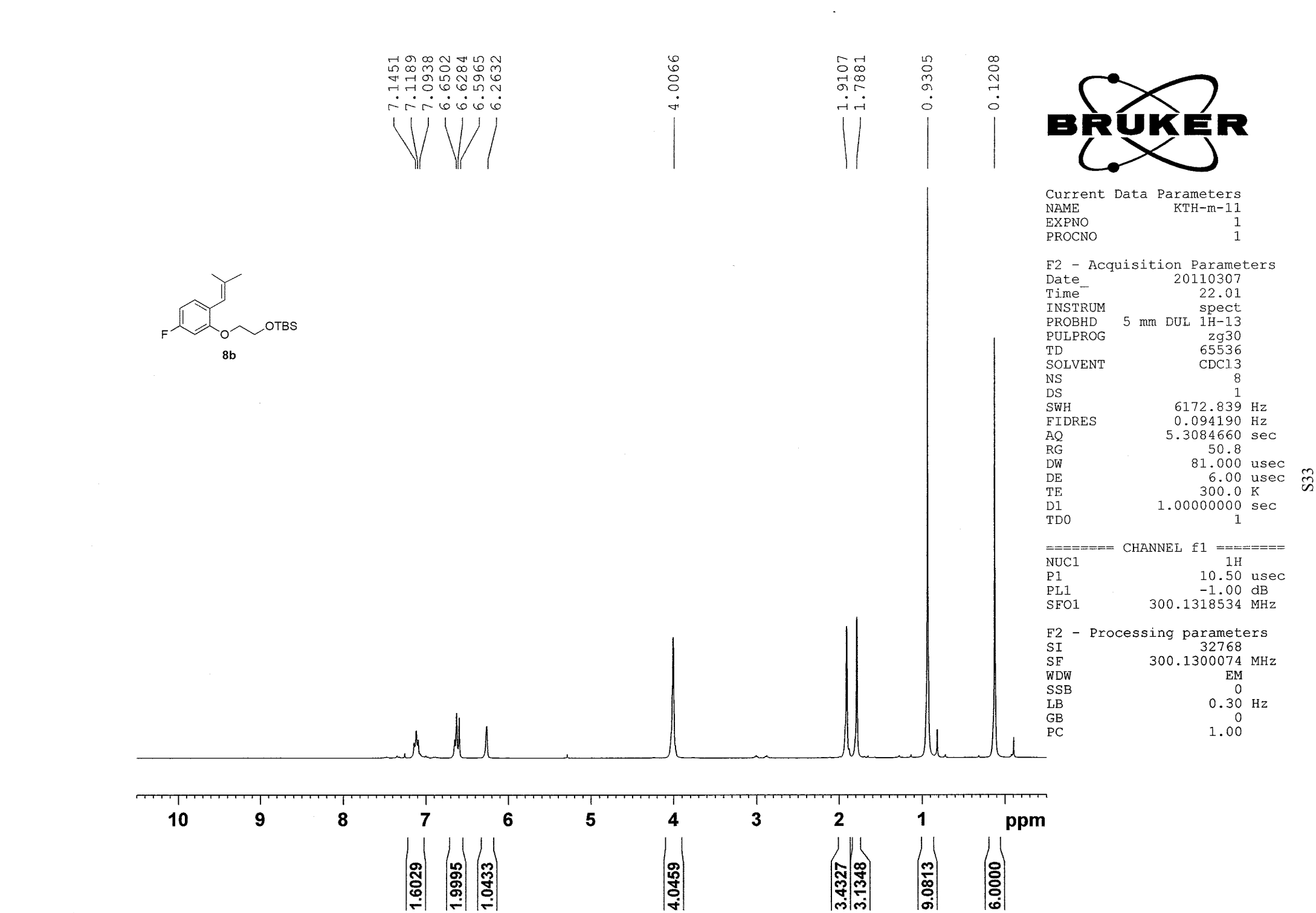

BRUKER

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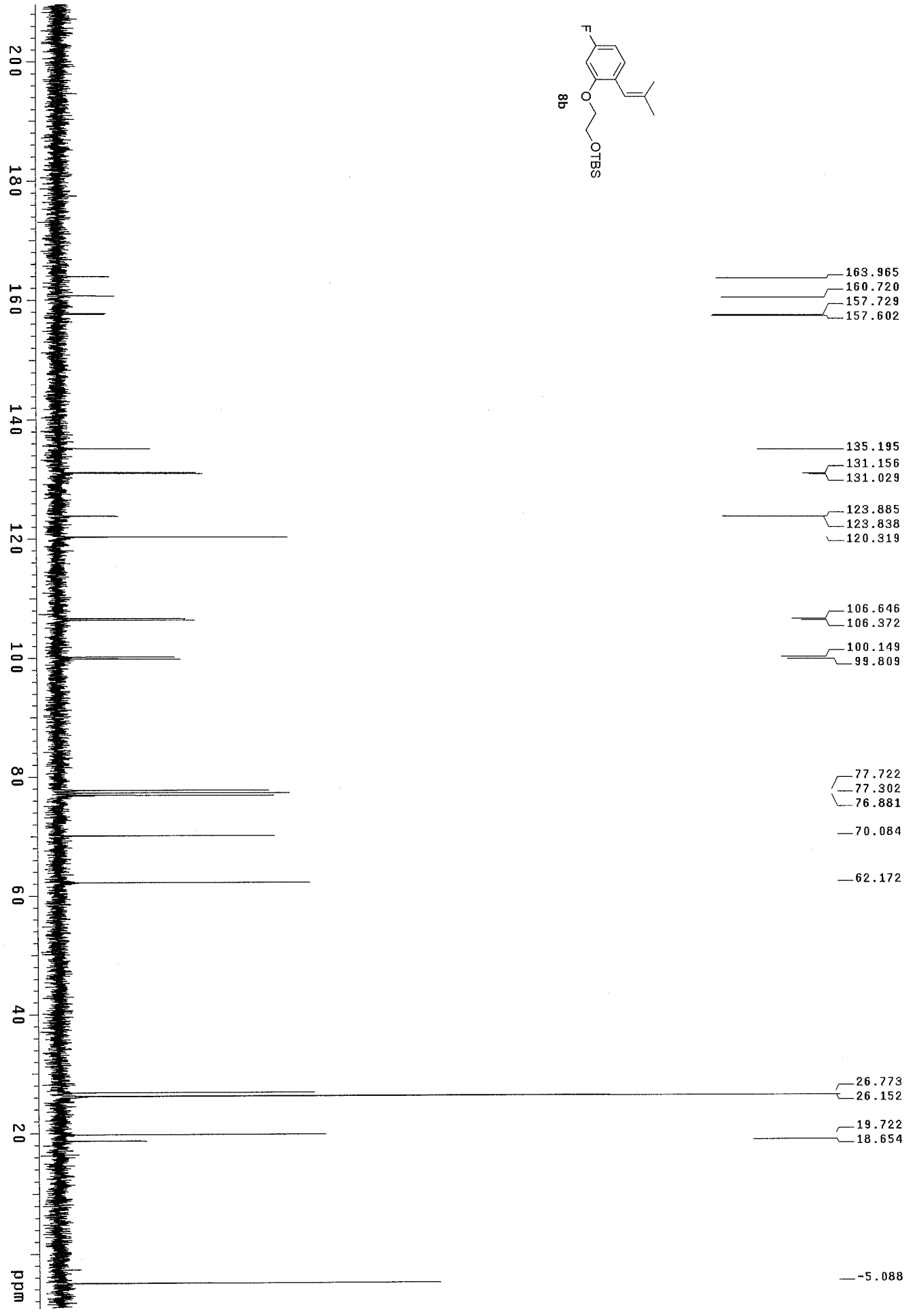
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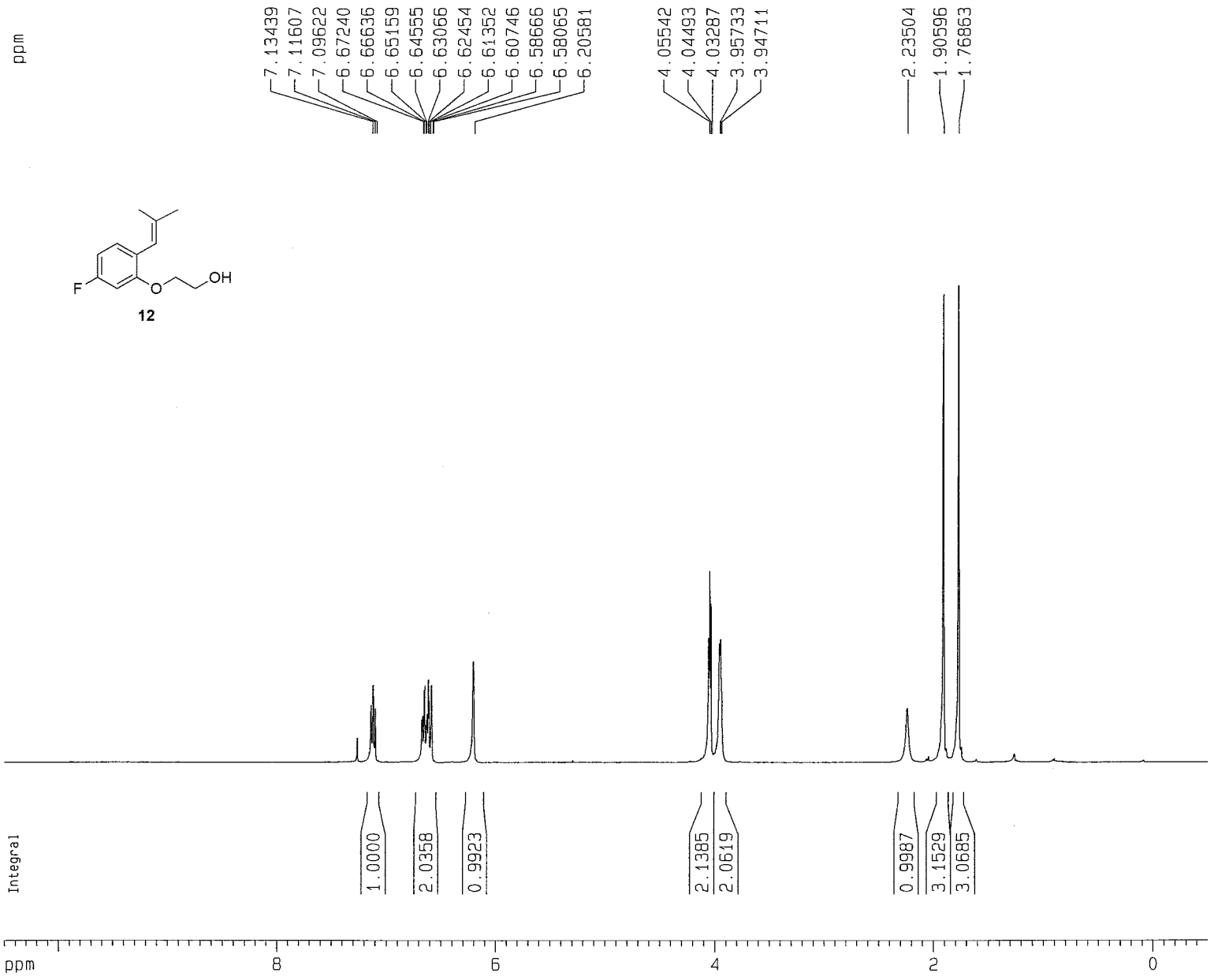

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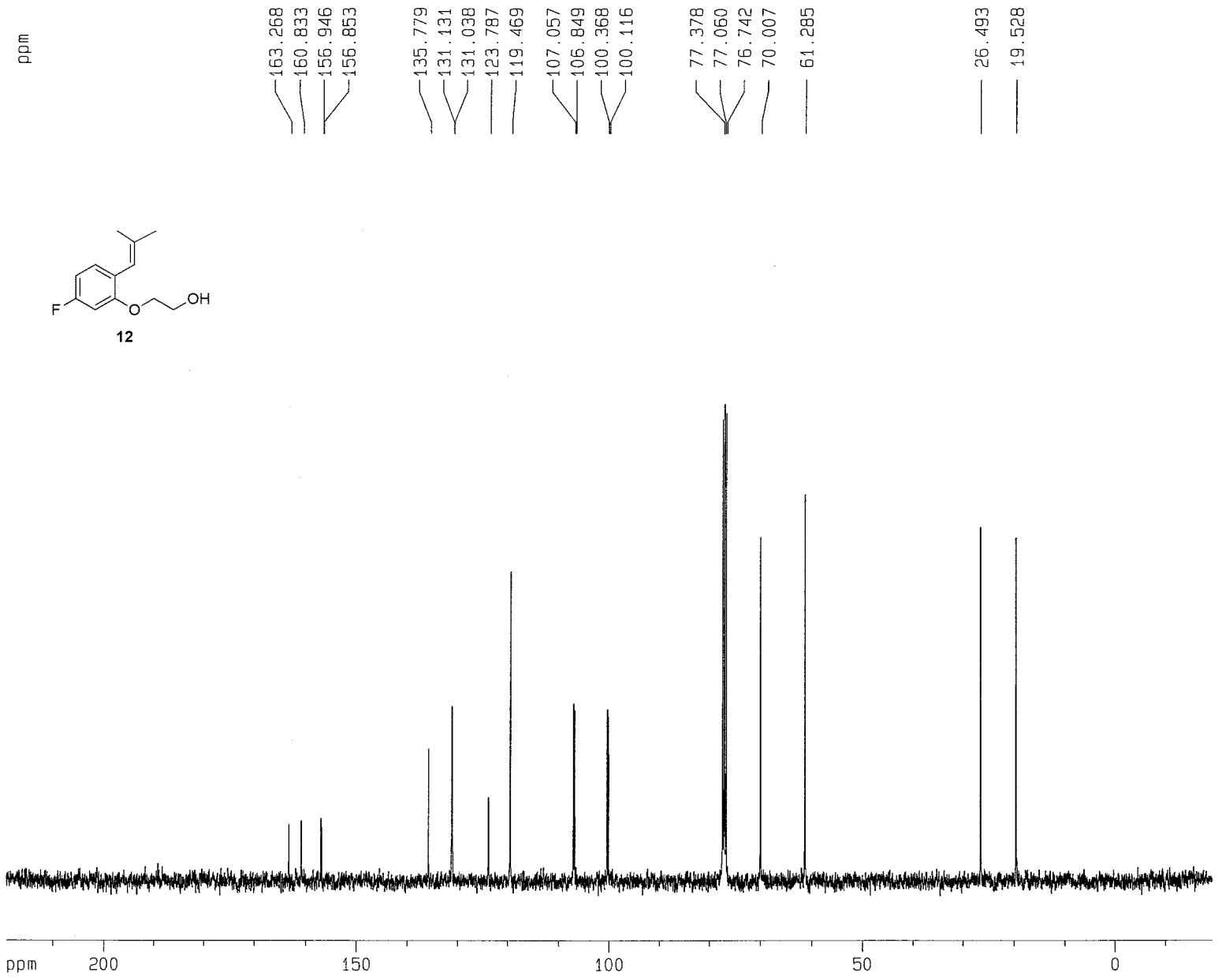


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AQ                 1.3664756 sec
RG                        128
DW                    20.850 usec
DE                     6.00 usec
TE                     297.3 K
D1                 2.00000000 sec
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DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
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PL12                  18.90 dB
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SFO2              400.1316005 MHz

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PC                     1.40

1D NMR plot parameters
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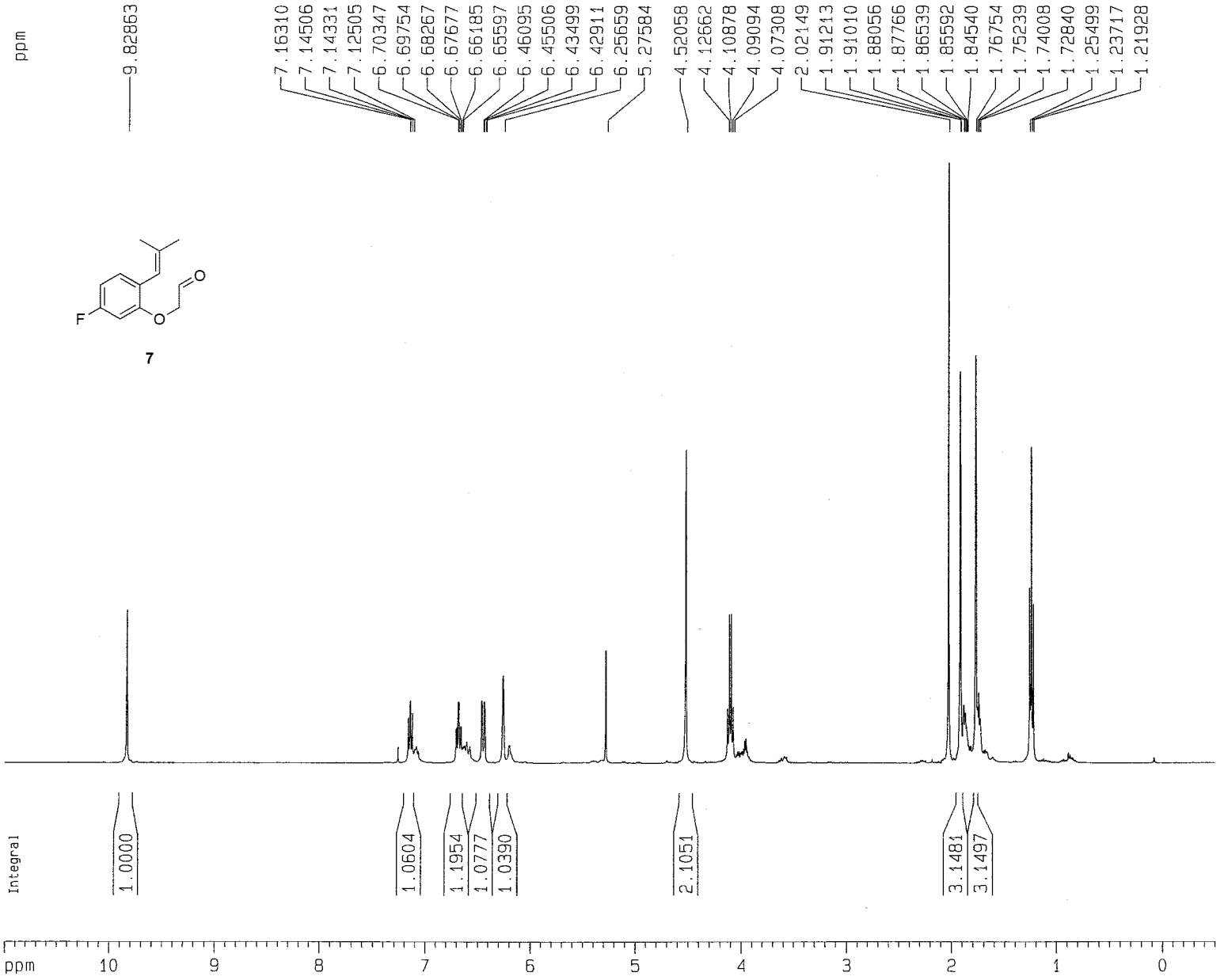

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======== CHANNEL f1 ========

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F2 - Processing parameters

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GB	0
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1D NMR plot parameters

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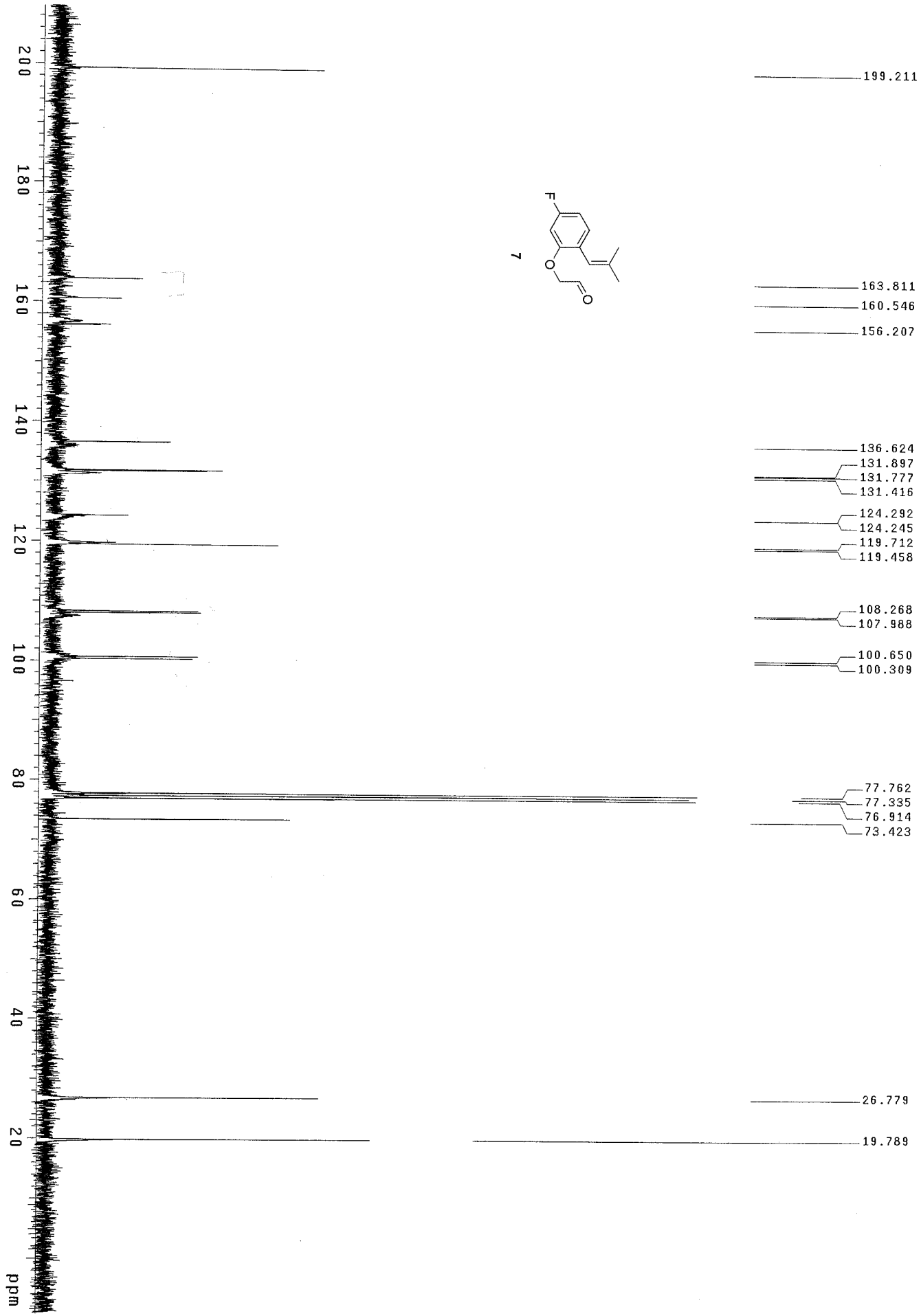
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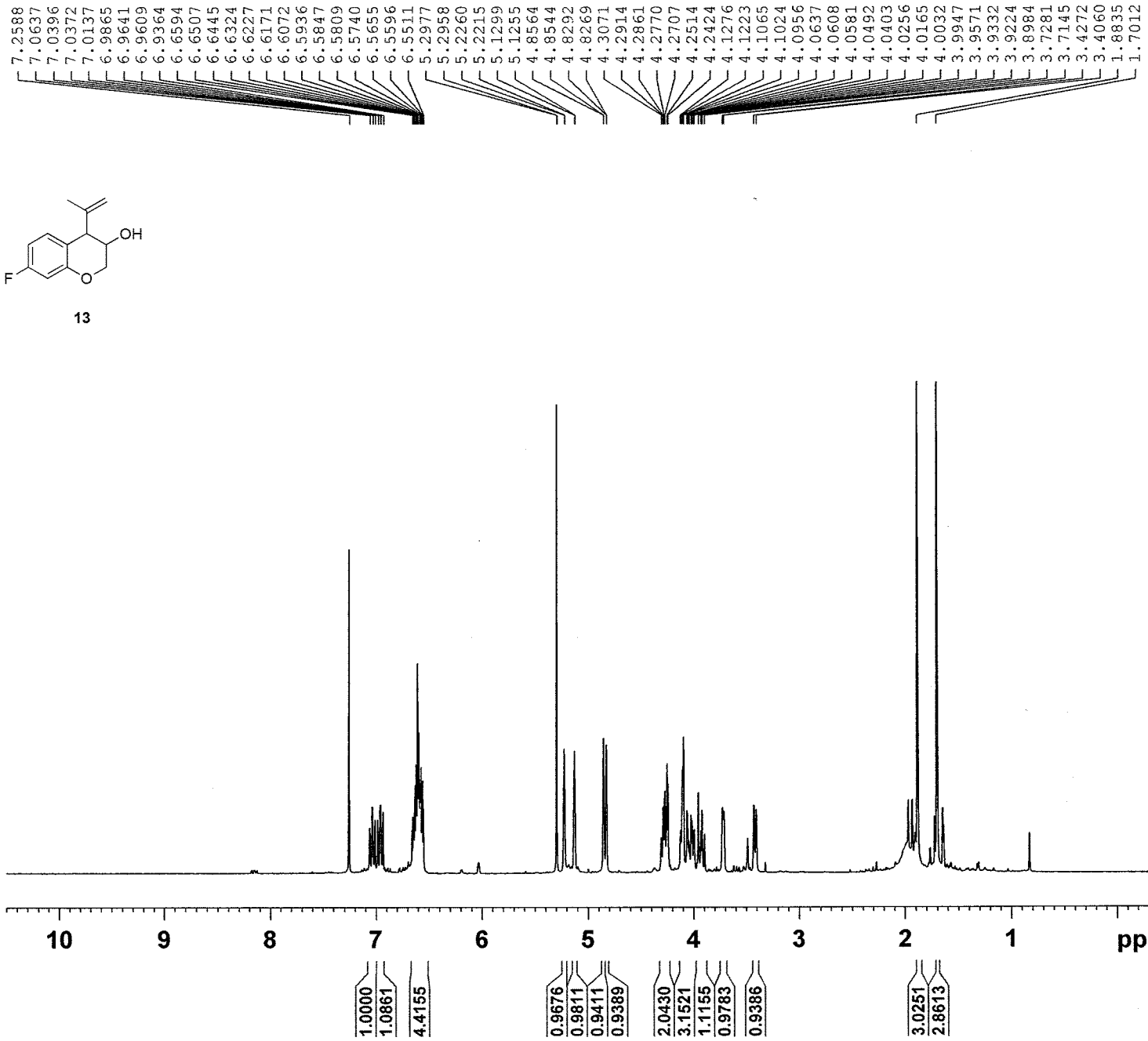
20
ppm

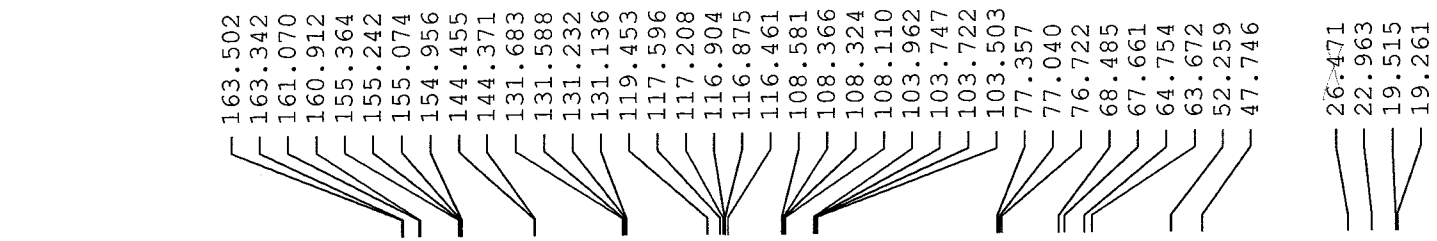


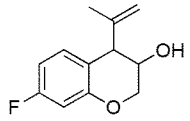
Current Data Parameters
NAME KTH-m-36
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110402
Time 19.29
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 724.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

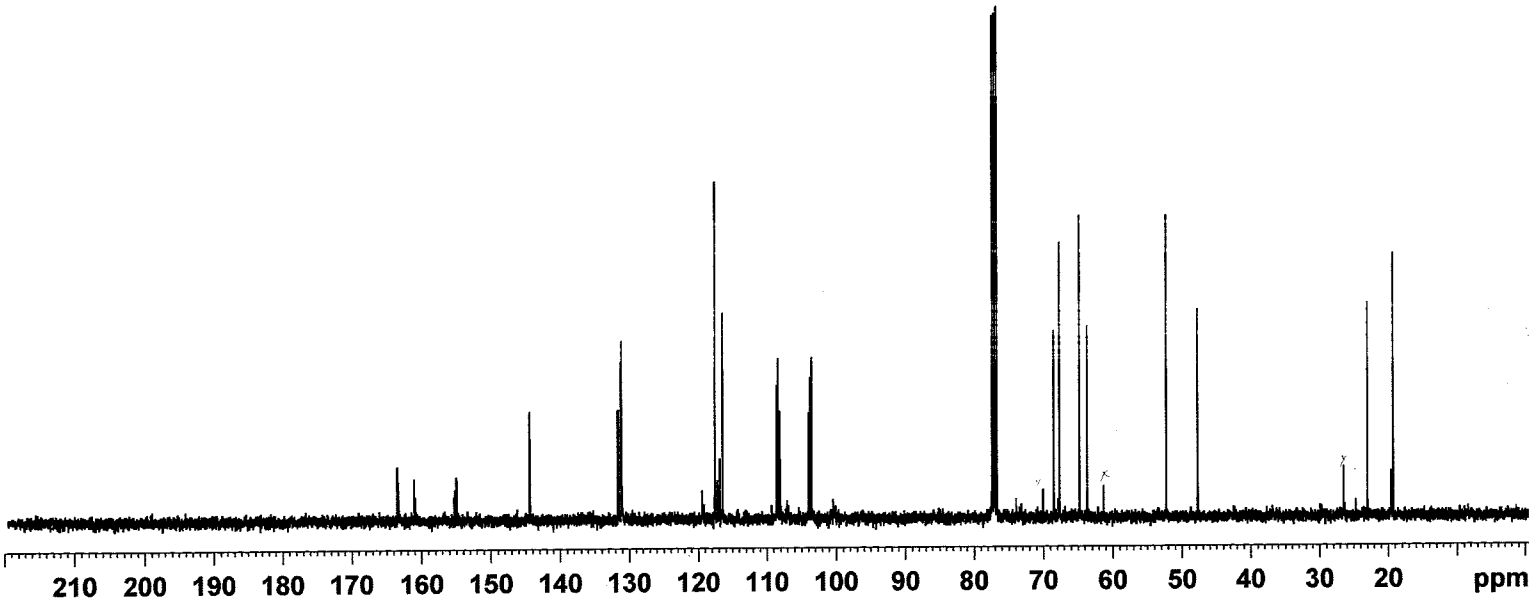
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





13



Current Data Parameters

NAME	2011-03-17
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20110317
Time	12.44
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	420
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3632196 sec
RG	32768
DW	20.800 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

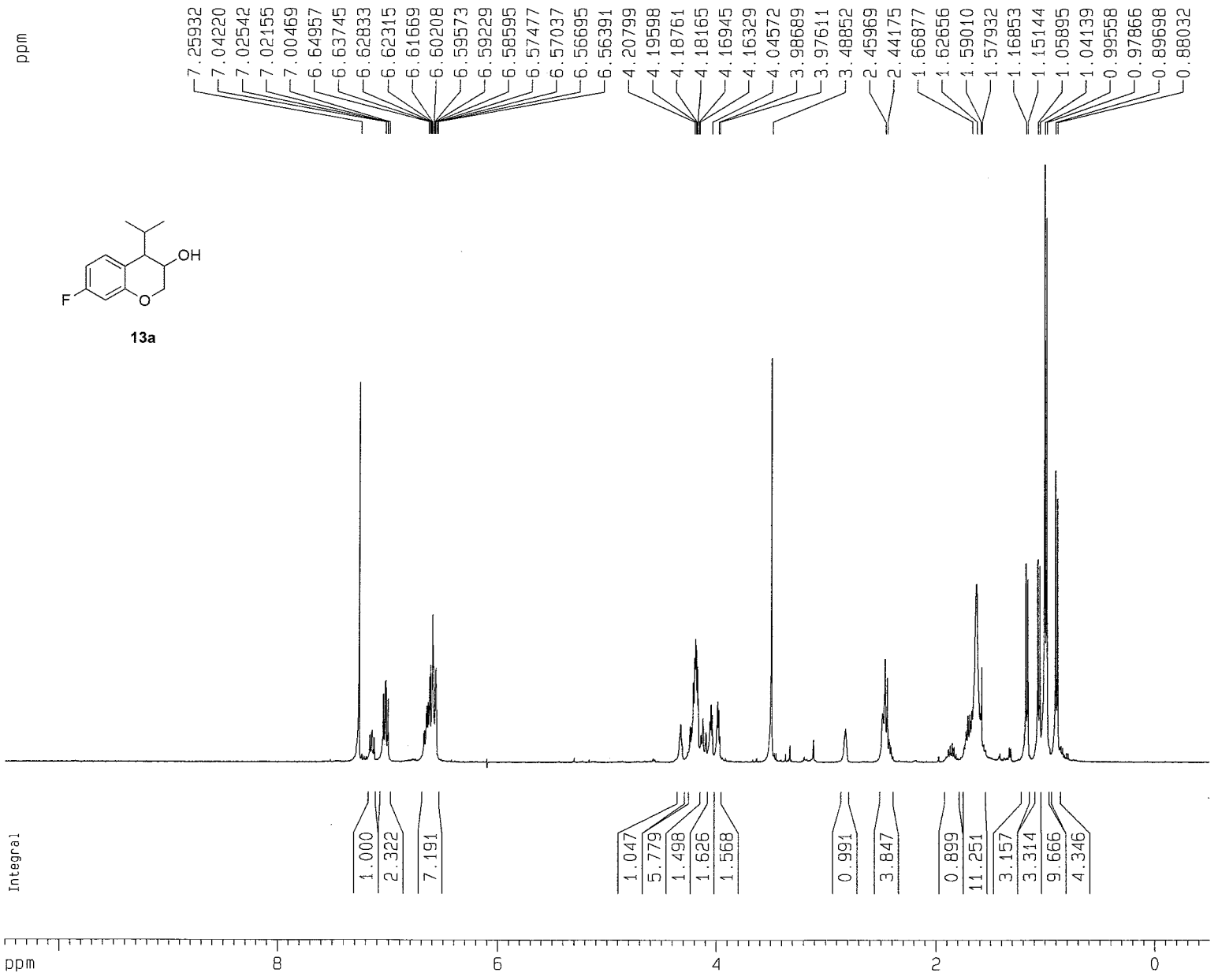
NUC1	13C
P1	7.50 usec
PL1	1.00 dB
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-2.00 dB
PL12	17.41 dB
PL13	21.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



Current Data Parameters

NAME	KTH-m-62
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20110604
Time	16.07
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	0
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.00 usec
TE	296.7 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

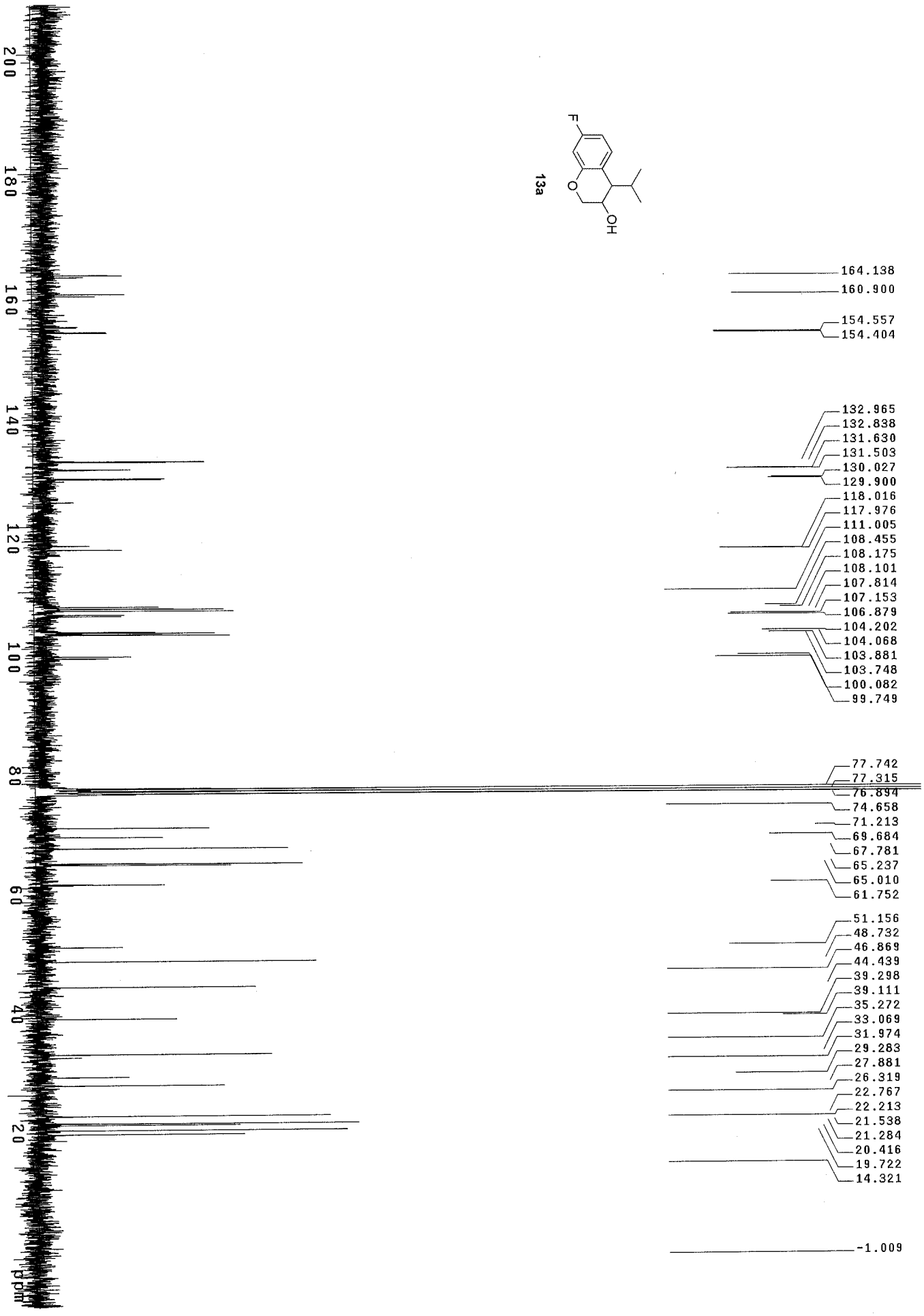
NUC1	1H
P1	11.50 usec
PL1	1.40 dB
SFO1	400.1324710 MHz

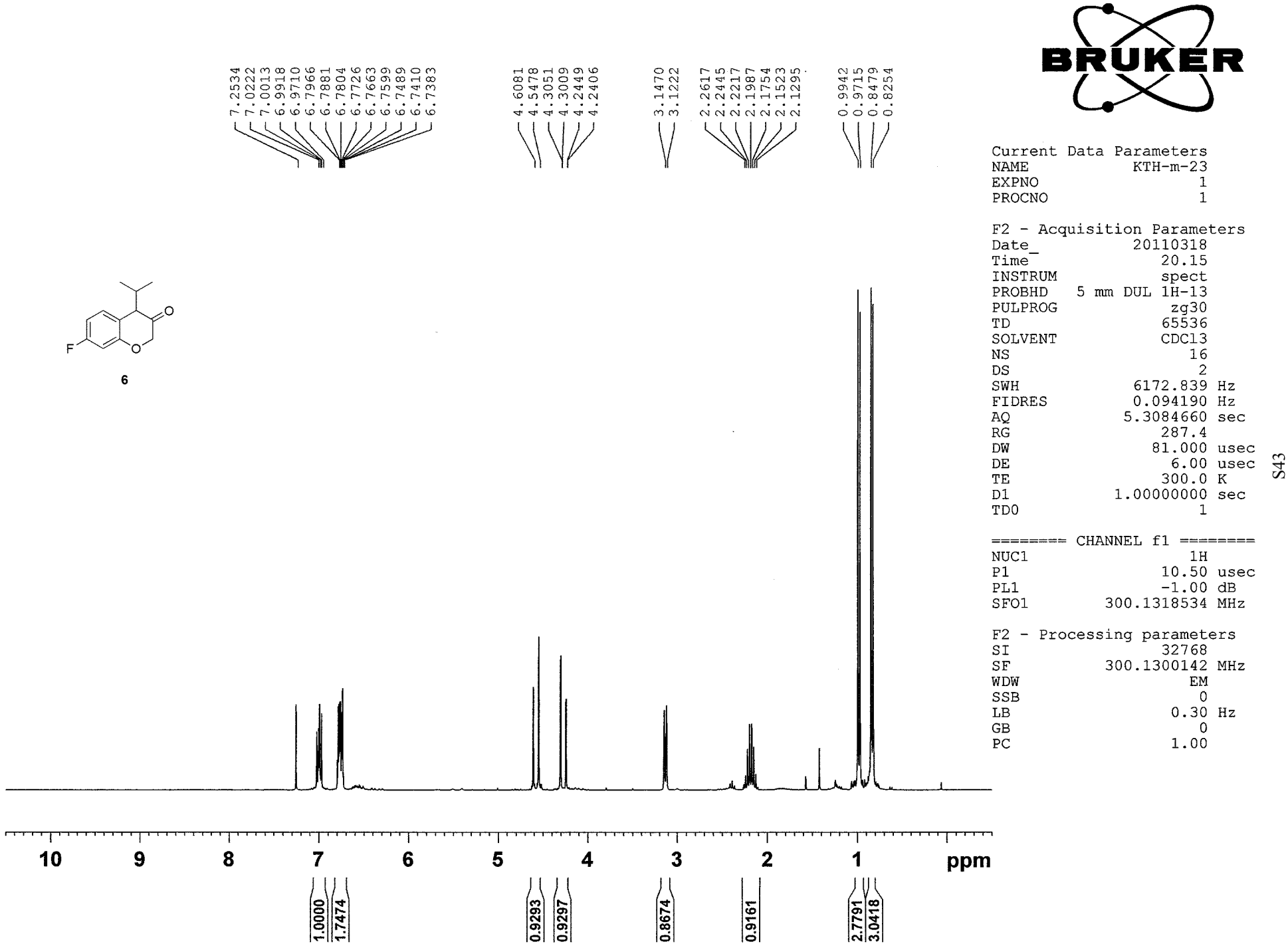
F2 - Processing parameters

SI	32768
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	4201.37 Hz
F2P	-0.500 ppm
F2	-200.06 Hz
PPMCM	0.55000 ppm/cm
HZCM	220.07150 Hz/cm


13a
F
O
OH
164.138
160.900
154.557
154.404
132.965
132.838
131.630
131.503
130.027
129.900
118.016
117.976
111.005
108.455
108.175
108.101
107.814
107.153
106.879
104.202
104.068
103.881
103.748
100.082
99.749
77.742
77.315
76.894
74.658
71.213
69.684
67.781
65.237
65.010
61.752
51.156
48.732
46.869
44.439
39.298
39.111
35.272
33.069
31.974
29.283
27.881
26.319
22.767
22.213
21.538
21.284
20.416
19.722
14.321
-1.009
200
180
160
140
120
100
80
60
40
20
ppm



Current Data Parameters

NAME	KTH-m-23
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

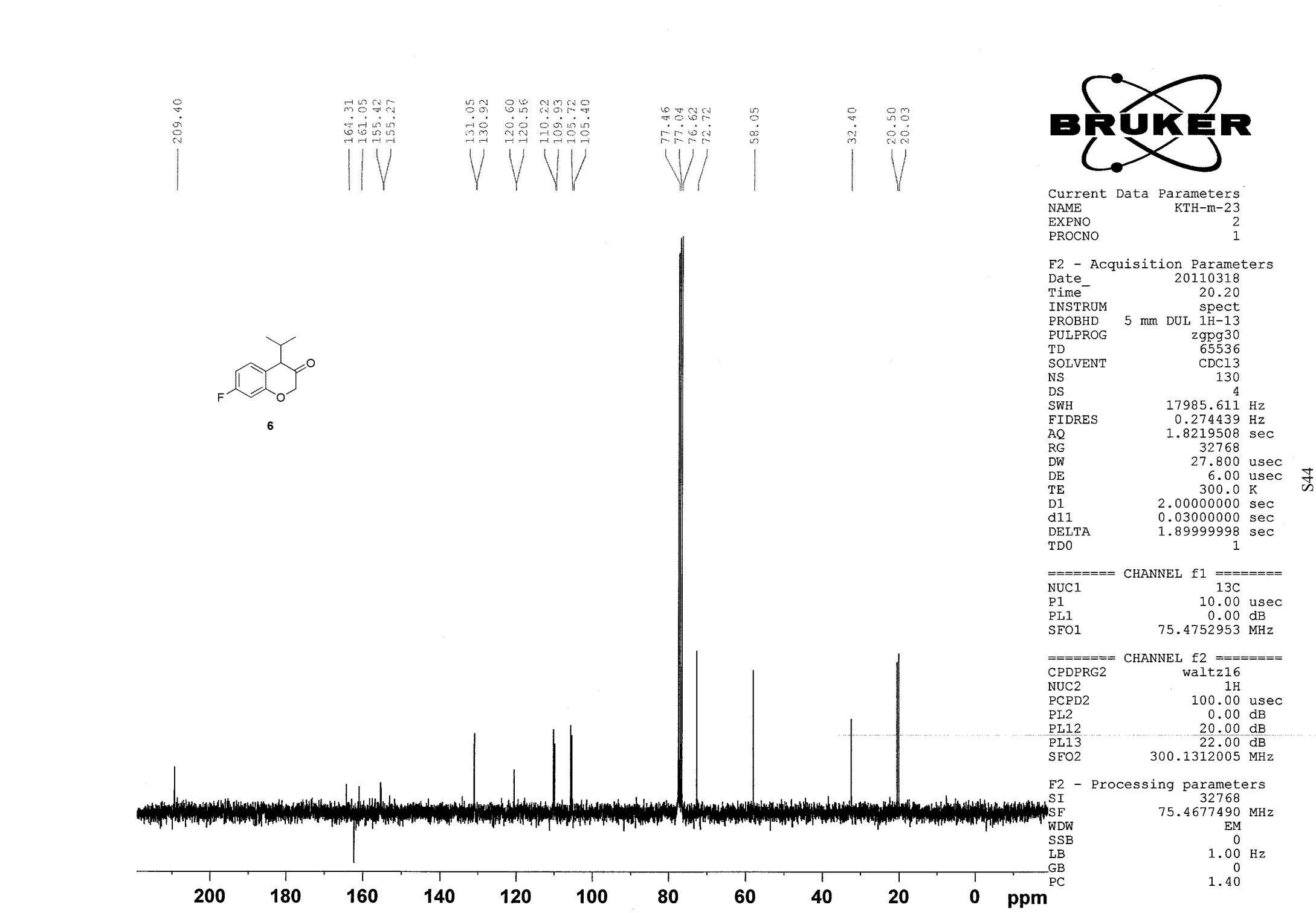
Date_	20110318
Time	20.15
INSTRUM	spect
PROBHD	5 mm DUL 1H-13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	287.4
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	10.50 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300142 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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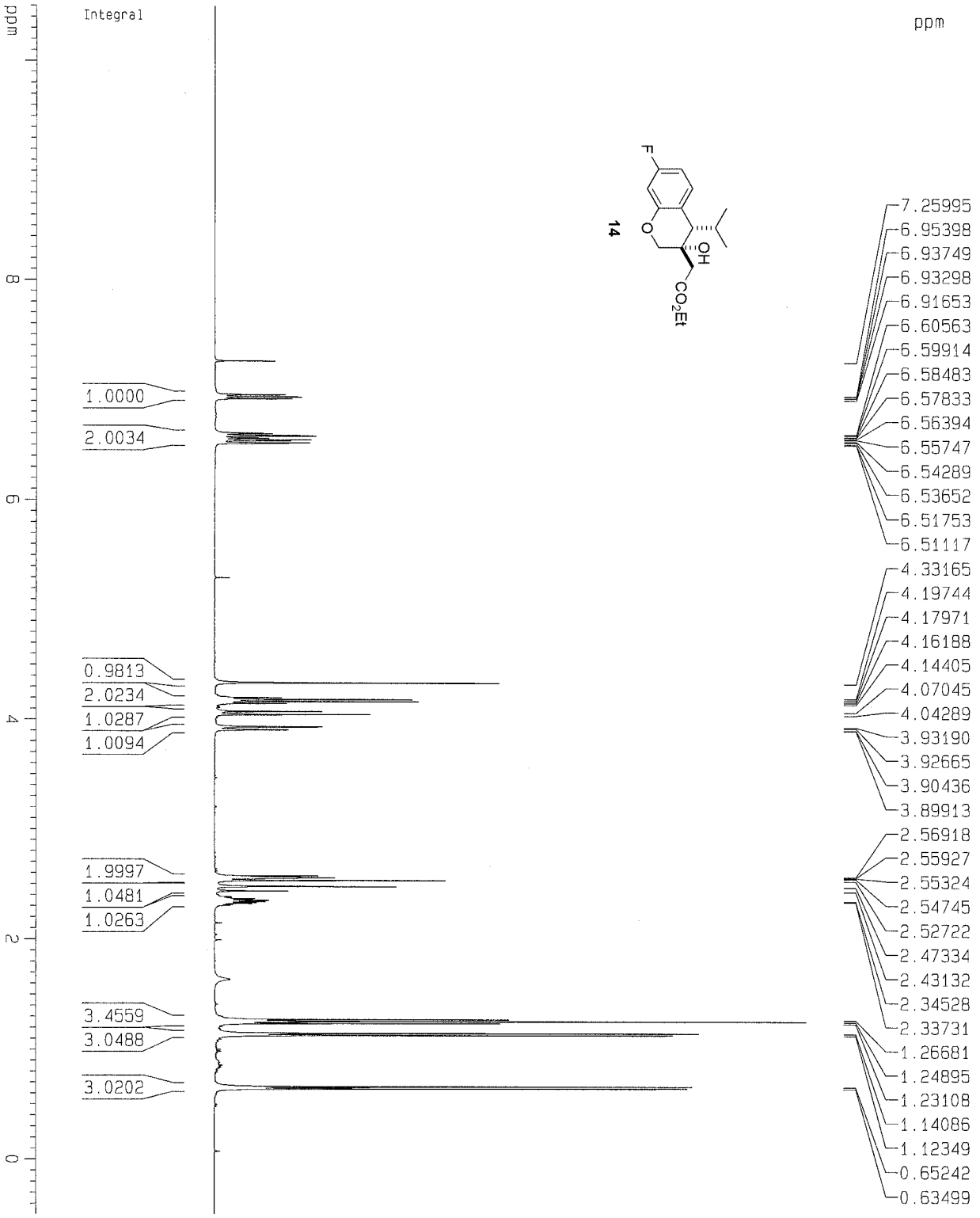
Current Data Parameters
NAME KTH-m-23
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110318
Time 20.20
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

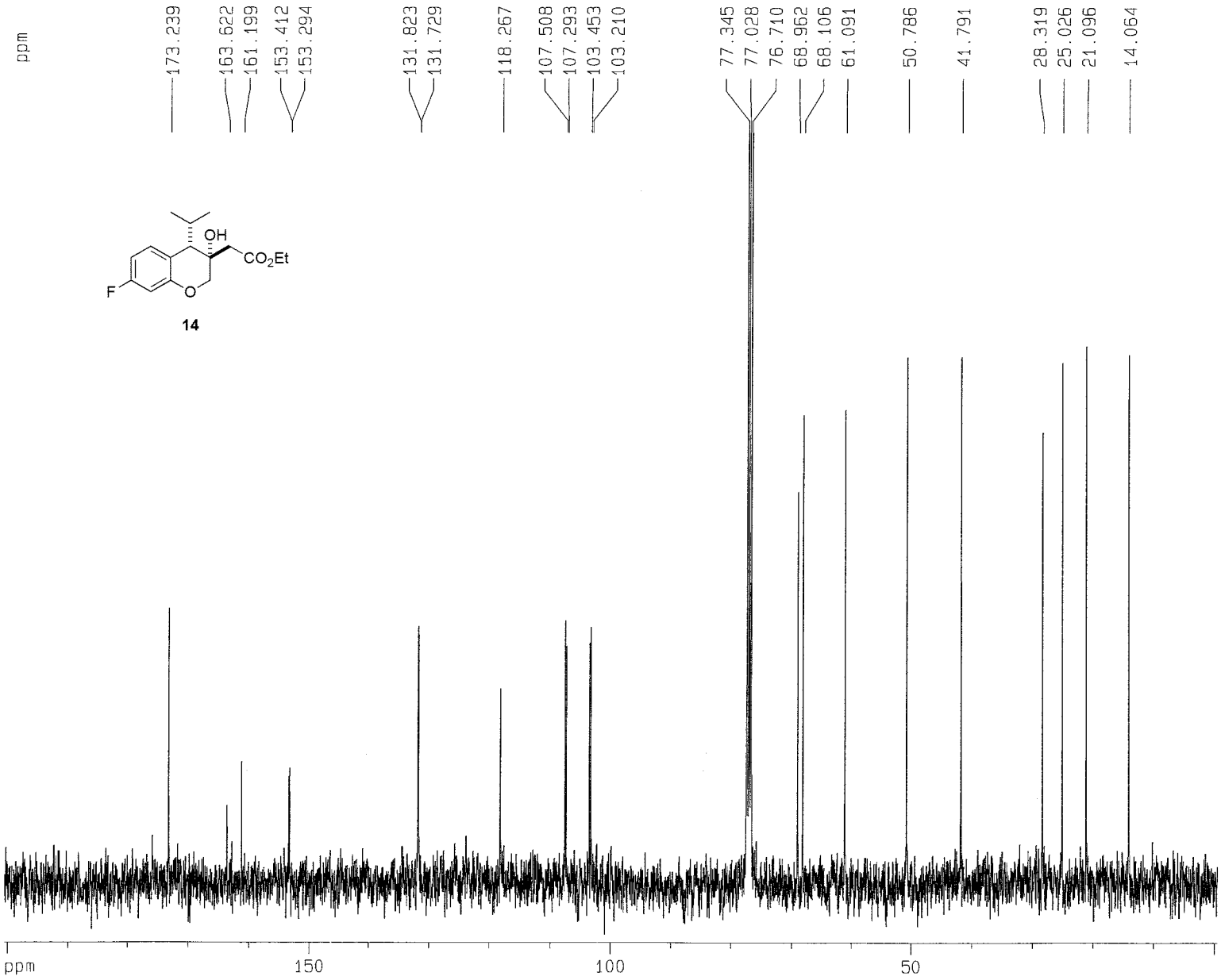
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 22.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME                  KTH-m-105
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20110903
Time_                 15.26
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT               MeOD
NS                       16
DS                        0
SWH                8278.146 Hz
FIDRES                0.126314 Hz
AQ                 3.9584243 sec
RG                       71.8
DW                    60.400 usec
DE                     6.00 usec
TE                      297.3 K
D1                 1.00000000 sec
d11                0.03000000 sec
DELTA              0.69999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.50 usec
PL1                     1.40 dB
SFO1             400.1324710 MHz

======== CHANNEL f2 ========
CPDPRG2                 off
NUC2                    off
PCPD2                80.00 usec
PL2                  120.00 dB
PL12                 120.00 dB
PL13                 120.00 dB
SFO2             400.1324710 MHz

F2 - Processing parameters
SI                      32768
SF               400.1300094 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      10.00 cm
F1P                   10.500 ppm
F1                  4201.37 Hz
F2P                   -0.500 ppm
F2                  -200.06 Hz
PPMCM                 0.55000 ppm/cm
HZCM                220.07150 Hz/cm
```



Current Data Parameters

NAME	KTH-m-105
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20110903
Time	15.28
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	84
DS	0
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	128
DW	20.850 usec
DE	6.00 usec
TE	297.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	8.60 usec
PL1	4.00 dB
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

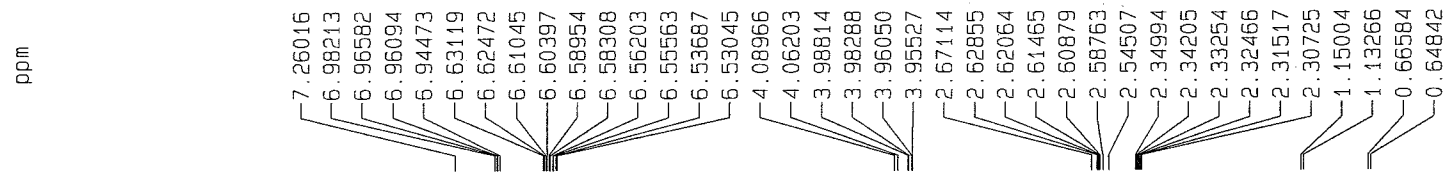
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.40 dB
PL12	18.90 dB
PL13	22.63 dB
SFO2	400.1316005 MHz

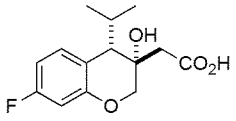
F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

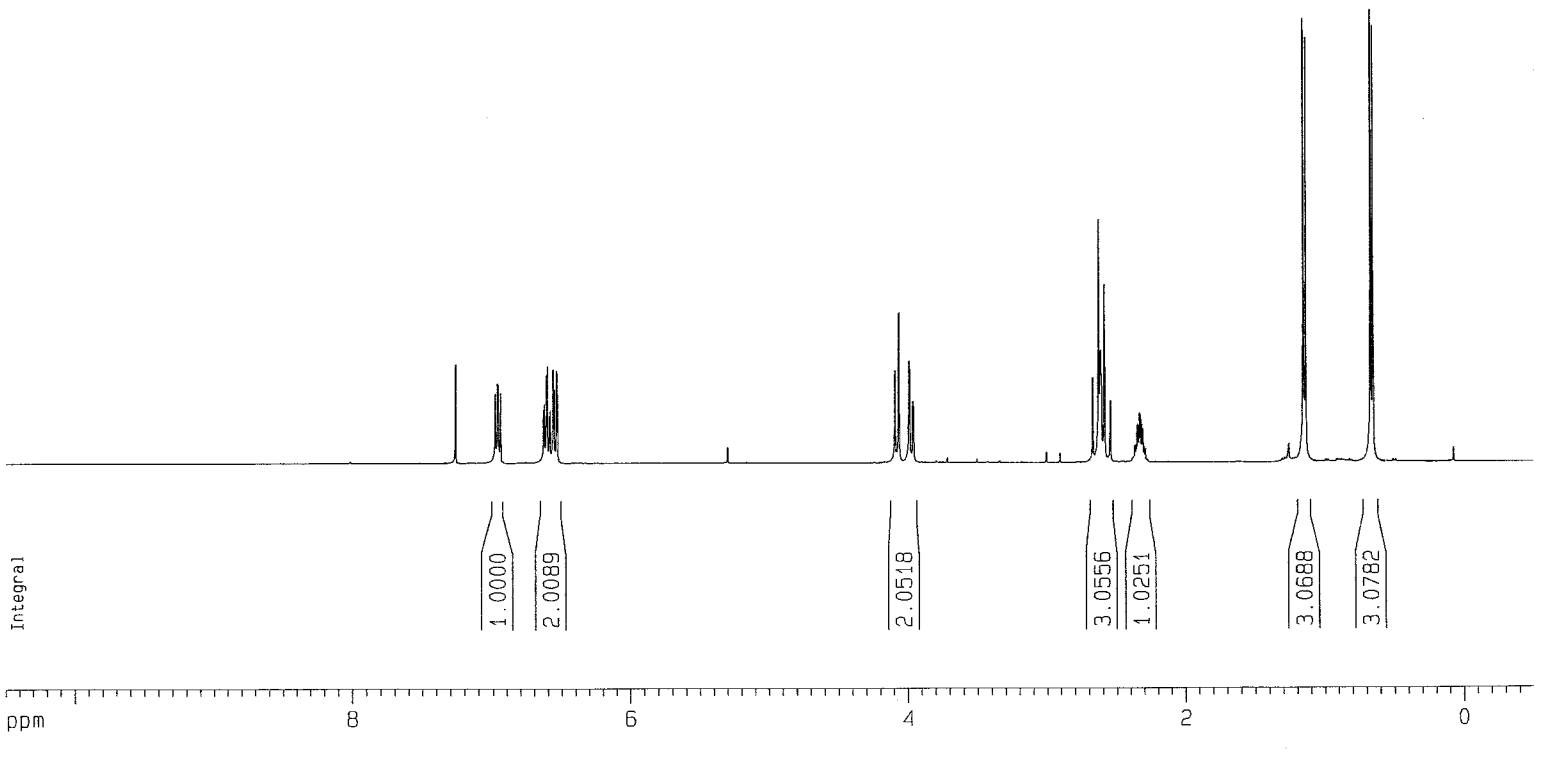
1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	200.500 ppm
F1	20172.86 Hz
F2P	-0.500 ppm
F2	-50.31 Hz
PPMCM	10.05000 ppm/cm
HZCM	1011.15833 Hz/cm

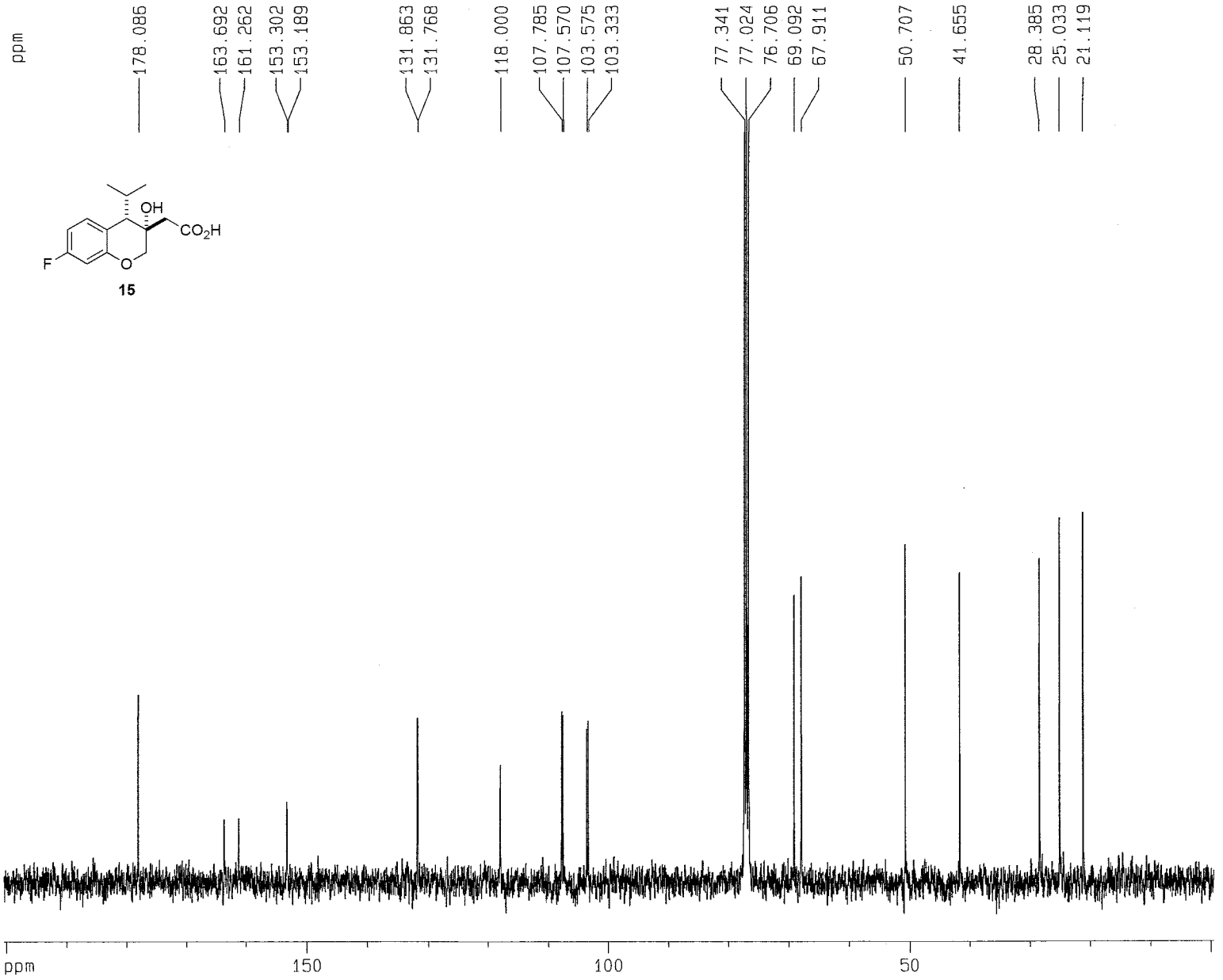




15




```
Current Data Parameters
NAME                  KTH-m-57
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110519
Time                     17.11
INSTRUM                  spect
PROBHD    5 mm Dual 13C/
PULPROG                  zg30
TD                       65536
SOLVENT                  MeOD
NS                          16
DS                           0
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                       161.3
DW                    60.400 usec
DE                     6.00 usec
TE                     297.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                     11.50 usec
PL1                     1.40 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF                400.1300094 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                     20.00 cm
CY                      6.00 cm
F1P                   10.500 ppm
F1                  4201.37 Hz
F2P                   -0.500 ppm
F2                  -200.06 Hz
PPMCM                0.55000 ppm/cm
HZCM               220.07150 Hz/cm
```



Current Data Parameters

NAME	KTH-m-57
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20110519
Time	17.13
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	149
DS	0
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	128
DW	20.850 usec
DE	6.00 usec
TE	297.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	8.60 usec
PL1	4.00 dB
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

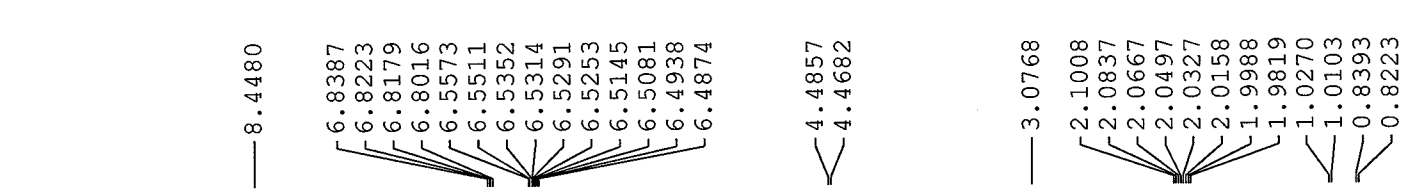
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.40 dB
PL12	18.90 dB
PL13	22.63 dB
SFO2	400.1316005 MHz

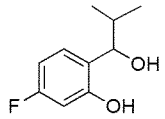
F2 - Processing parameters

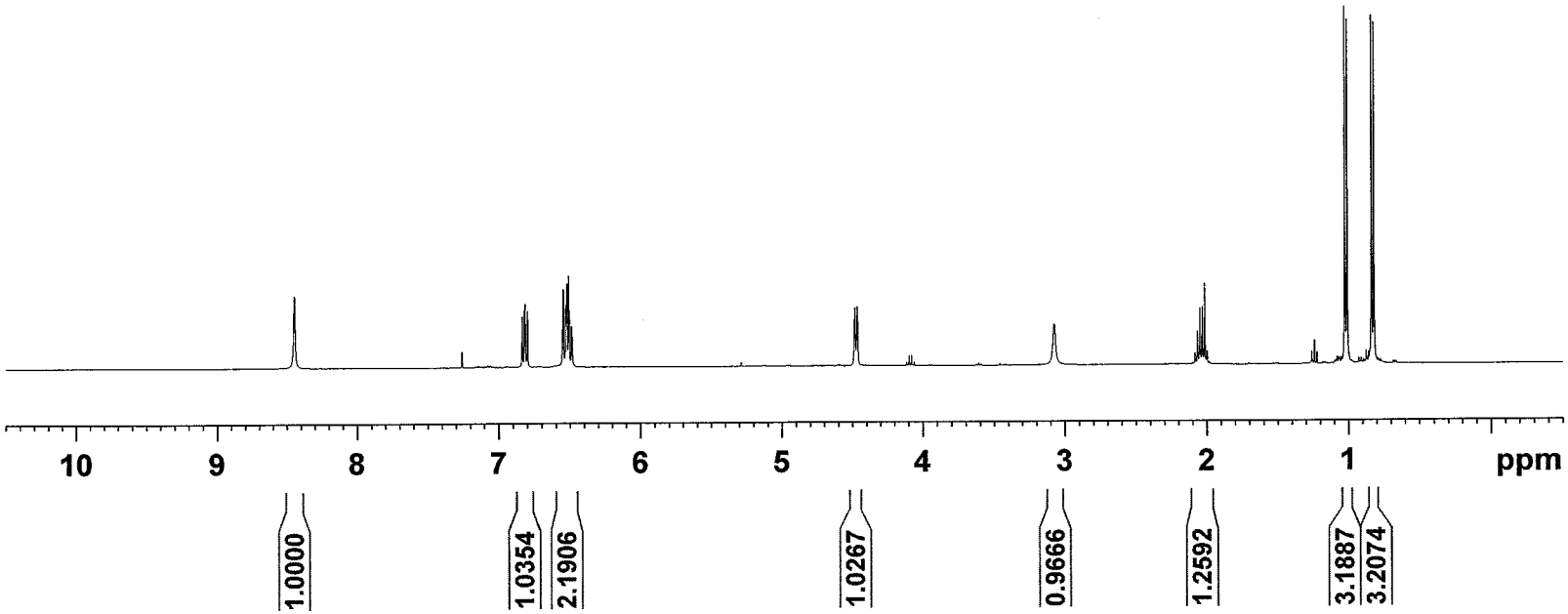
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	20.00 cm
F1P	200.500 ppm
F1	20172.86 Hz
F2P	-0.500 ppm
F2	-50.31 Hz
PPMCM	10.05000 ppm/cm
HZCM	1011.15833 Hz/cm





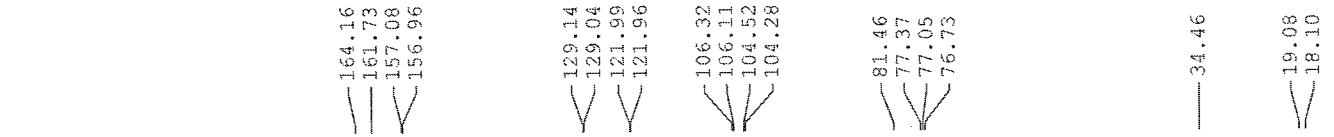


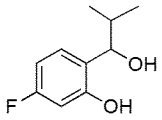
Current Data Parameters
NAME LJE-201
EXPNO 1
PROCNO 1

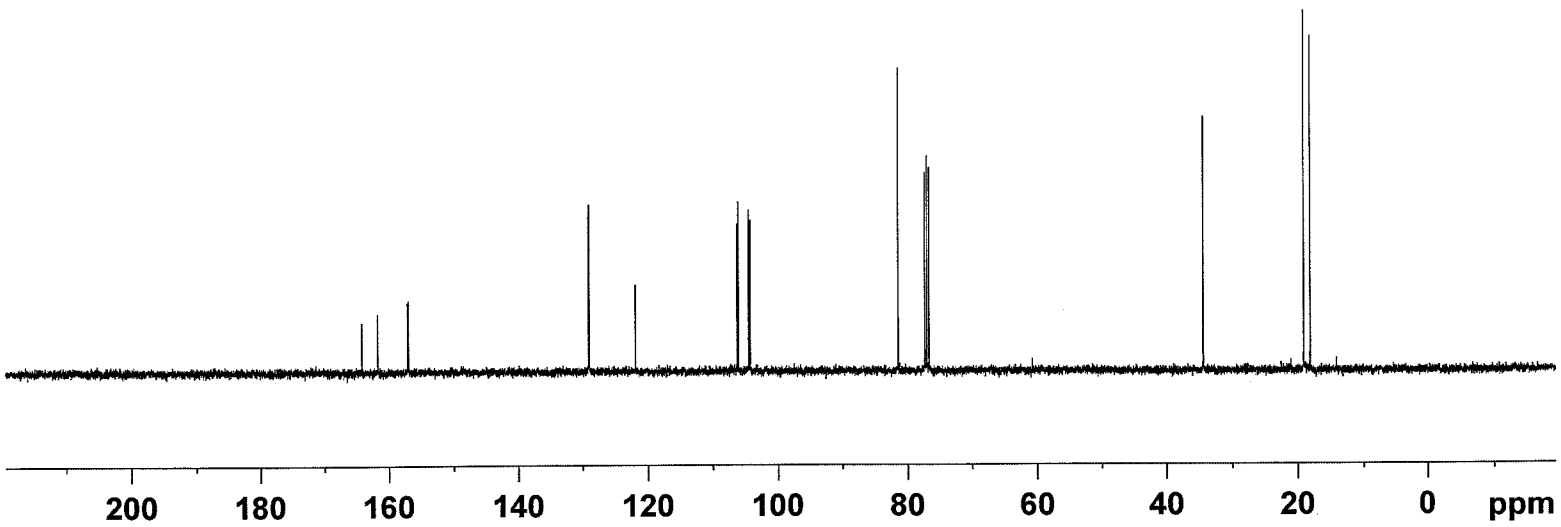
F2 - Acquisition Parameters
Date_ 20120713
Time 19.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.2
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







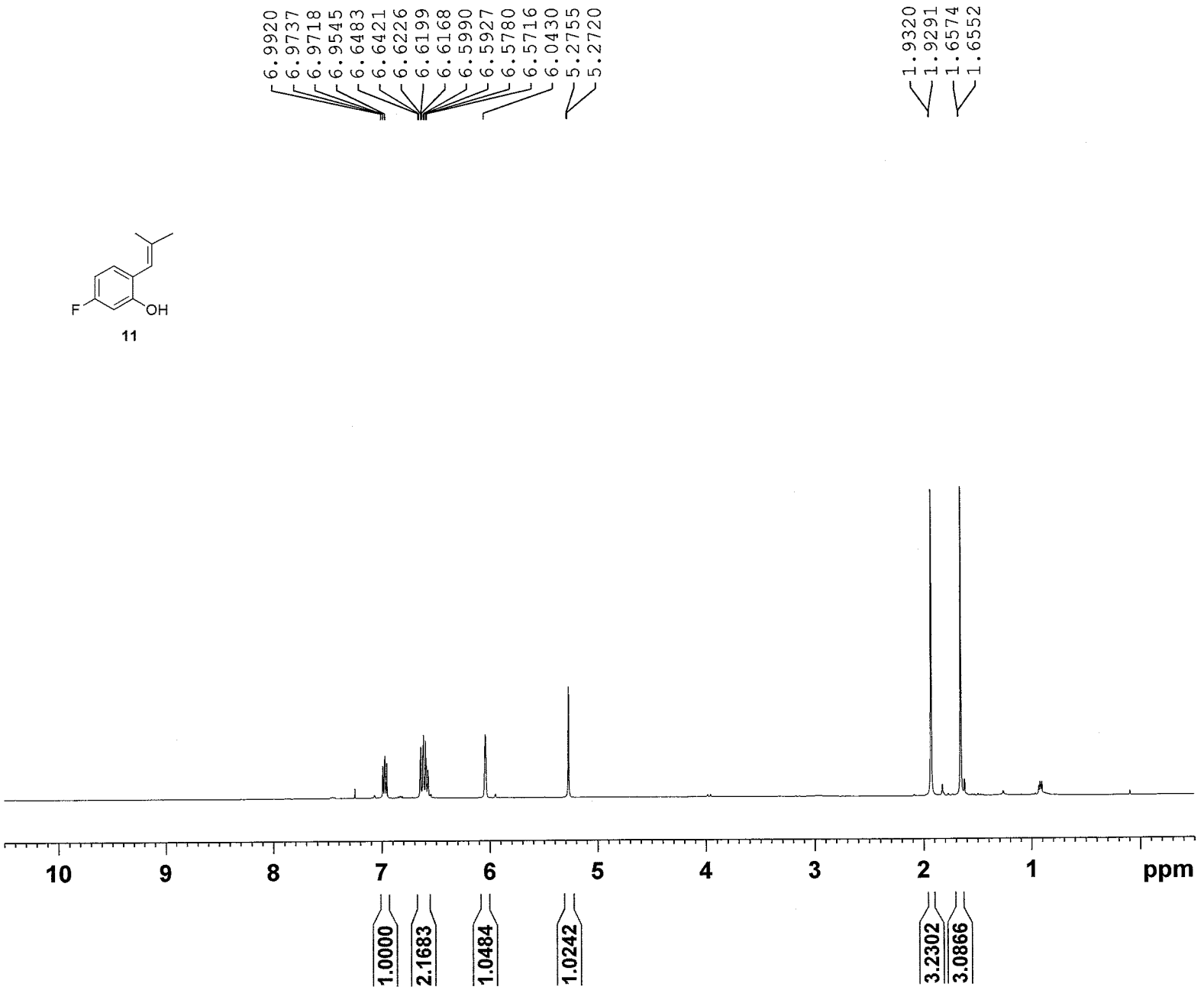
Current Data Parameters
NAME LJE-201
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120713
Time 19.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 35
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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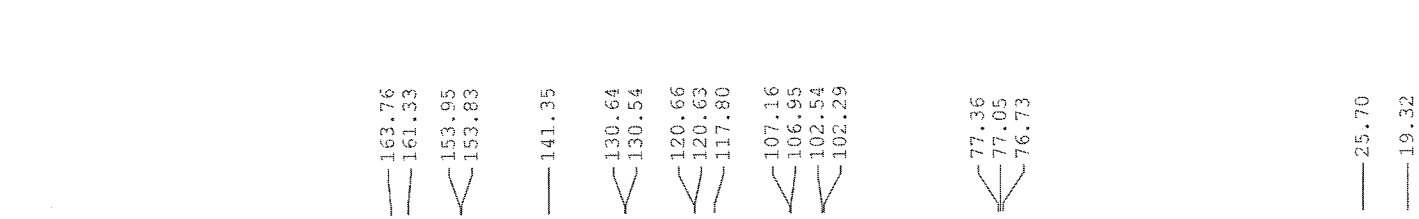
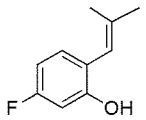
Current Data Parameters
NAME LJE-200
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120719
Time 20.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.2
DW 60.800 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

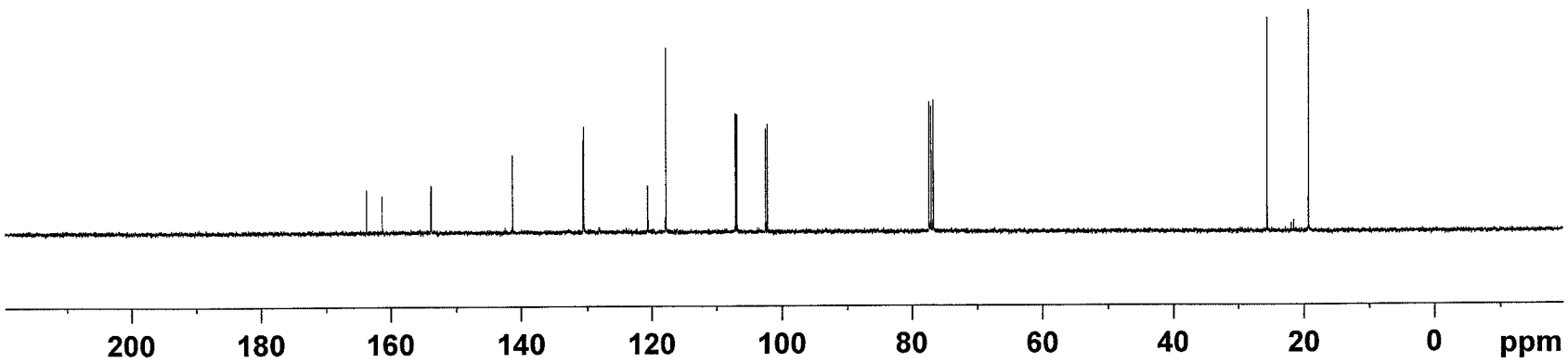
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100165 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

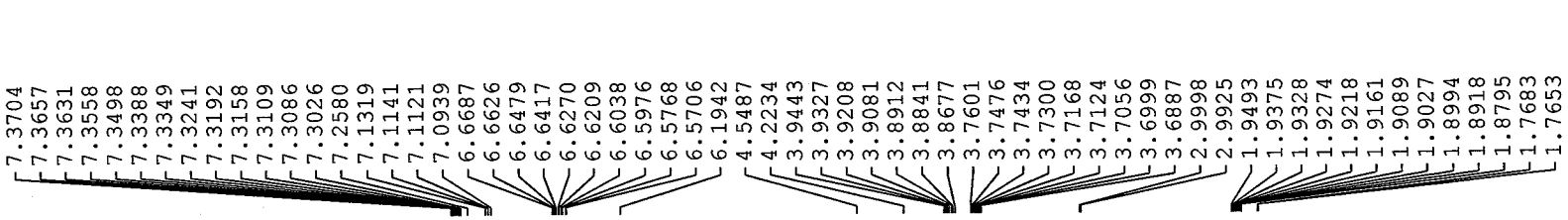


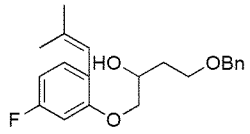


11

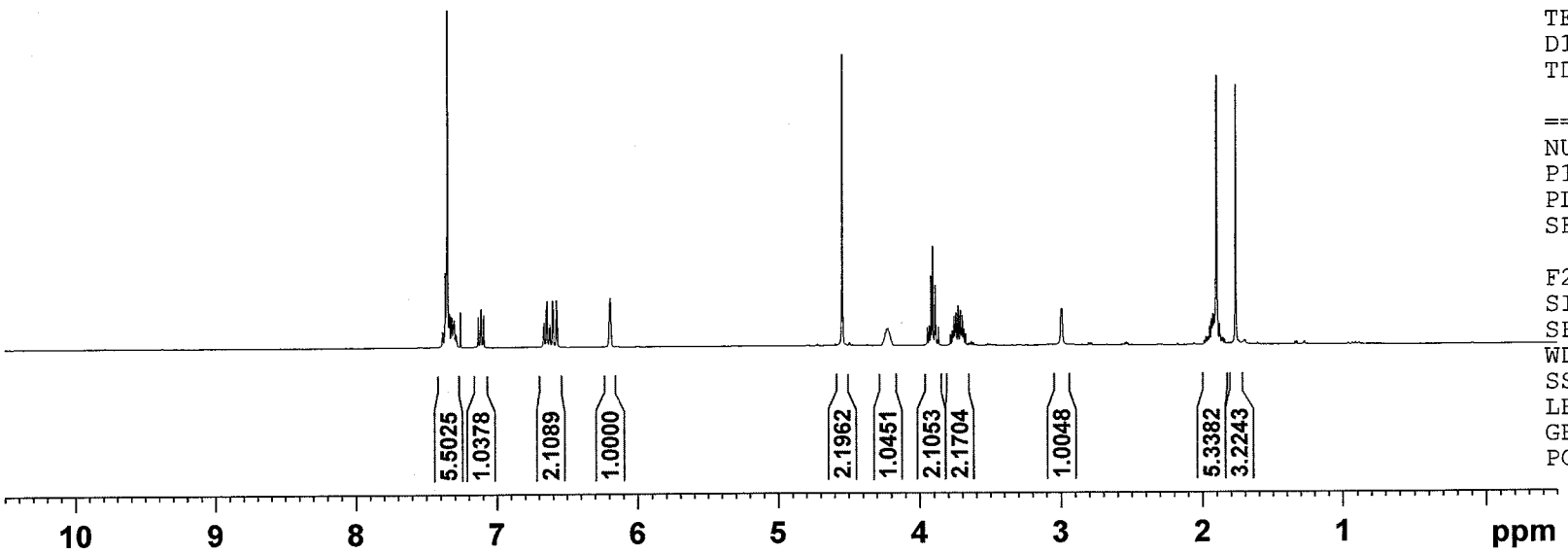



```
Current Data Parameters
NAME                  LJE-200
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20120719
Time                  20.05
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      96
DS                       4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                    190.62
DW                    20.800 usec
DE                     6.50 usec
TE                    298.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PLW1              52.00000000 W
SFO1             100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 90.00 usec
PLW2              12.00000000 W
PLW12              0.33333001 W
PLW13              0.27000001 W
SFO2             400.2116008 MHz

F2 - Processing parameters
SI                       32768
SF               100.6328860 MHz
WDW                         EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```





11a



Current Data Parameters

NAME	LJE-260
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

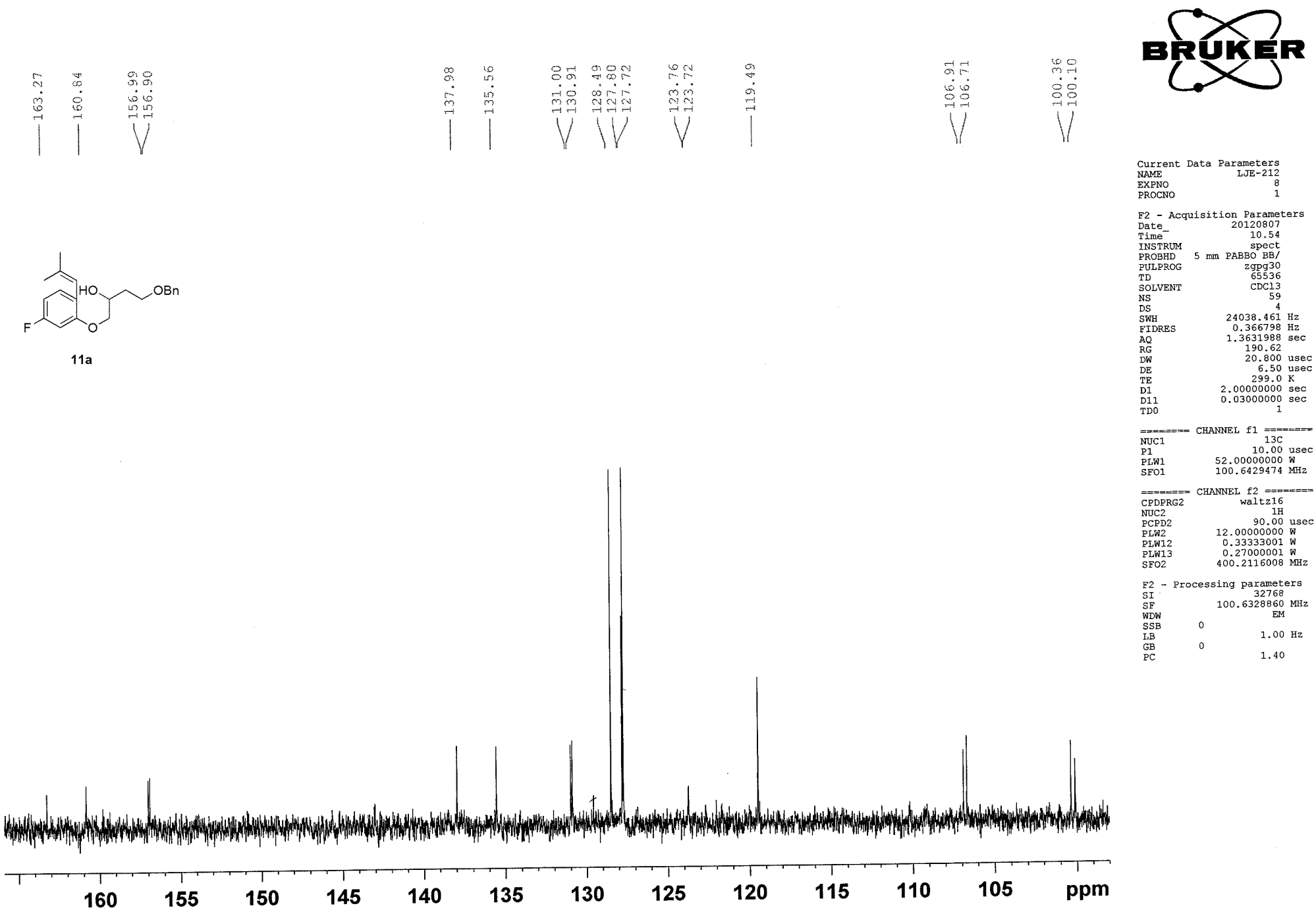
Date_	20121114
Time	14.37
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	56.34
DW	60.800 usec
DE	6.50 usec
TE	297.2 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	15.00 usec
PLW1	12.00000000 W
SFO1	400.2124715 MHz

F2 - Processing parameters

SI	65536
SF	400.2100136 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

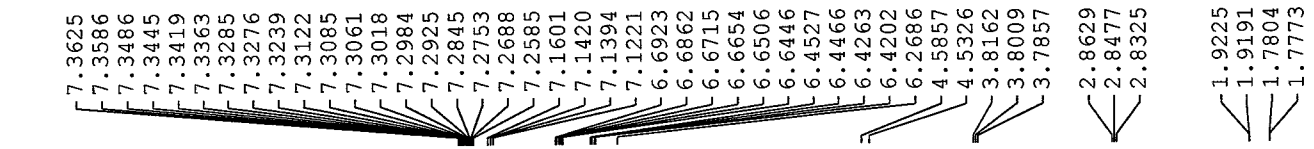
Current Data Parameters
NAME LJE-212
EXPNO 8
PROCNO 1

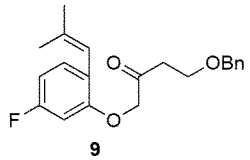
F2 - Acquisition Parameters
Date_ 20120807
Time 10.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 59
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

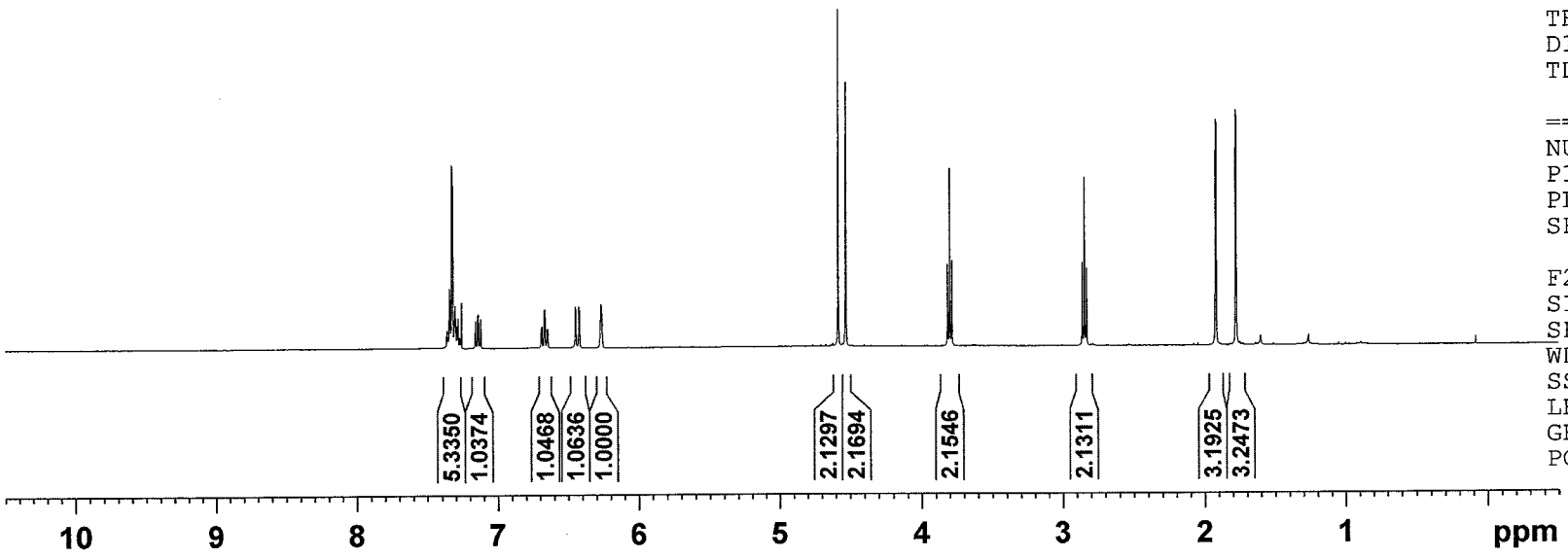
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





9



Current Data Parameters

NAME	LJE-261
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

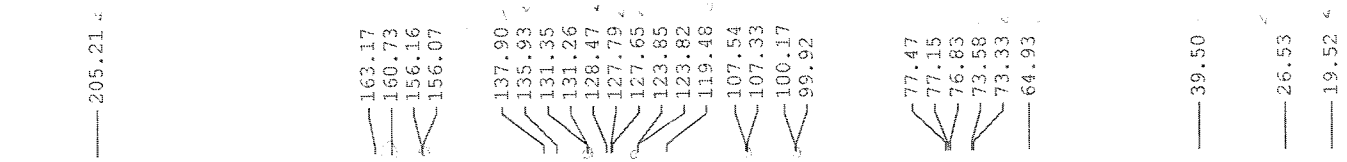
Date_	20121114
Time	20.15
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	2
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	76.56
DW	60.800 usec
DE	6.50 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1

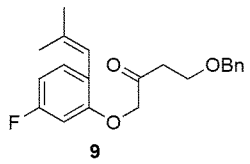
======== CHANNEL f1 ========

NUC1	1H
P1	15.00 usec
PLW1	12.00000000 W
SFO1	400.2124715 MHz

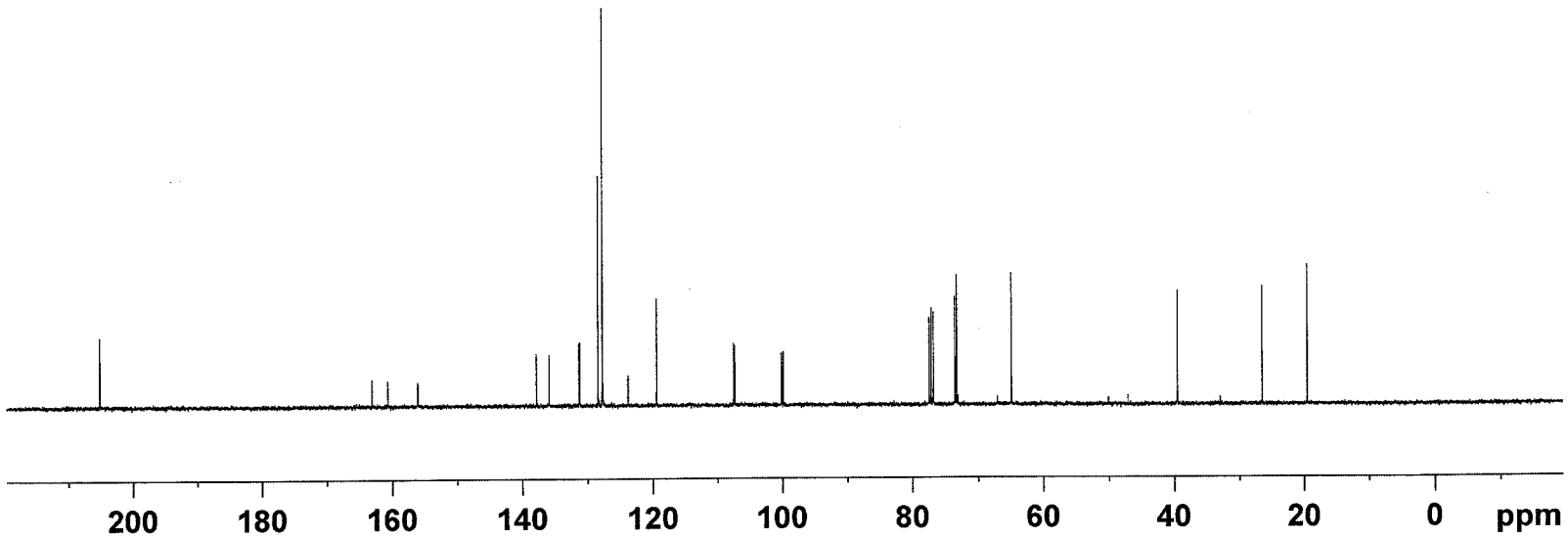
F2 - Processing parameters

SI	65536
SF	400.2100134 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





9



BRUKER

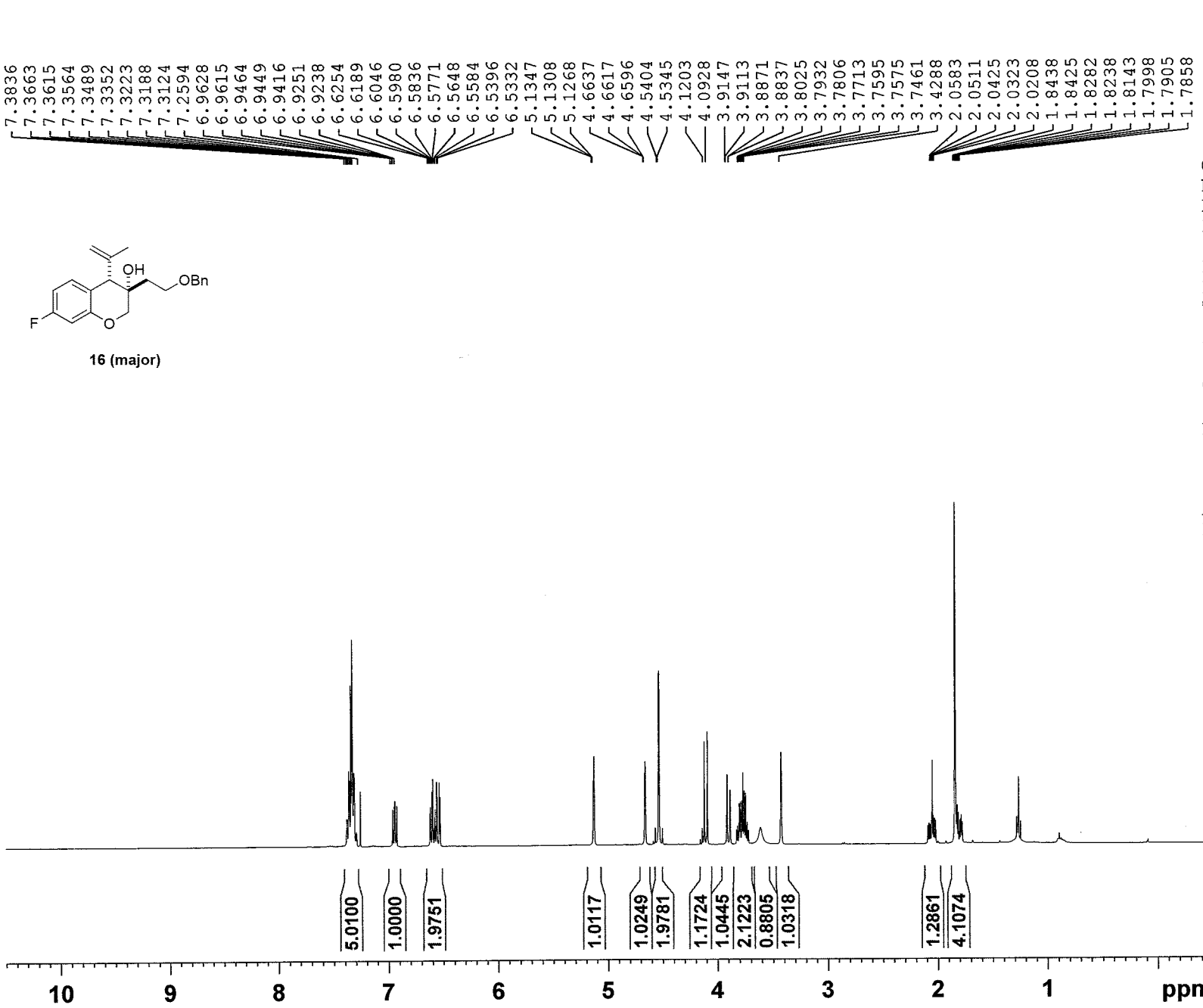
Current Data Parameters
NAME LJE-219
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120823
Time 20.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 57
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	LJE-265
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

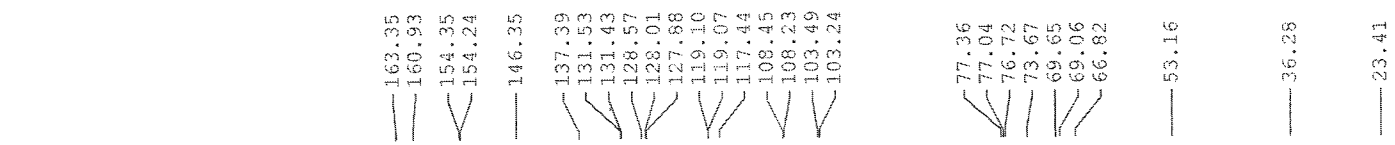
Date_	20121119
Time	13.44
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	58.64
DW	60.800 usec
DE	6.50 usec
TE	297.1 K
D1	1.00000000 sec
TD0	1

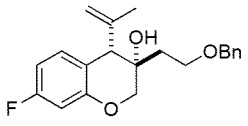
======== CHANNEL f1 ========

NUC1	1H
P1	15.00 usec
PLW1	12.00000000 W
SFO1	400.2124715 MHz

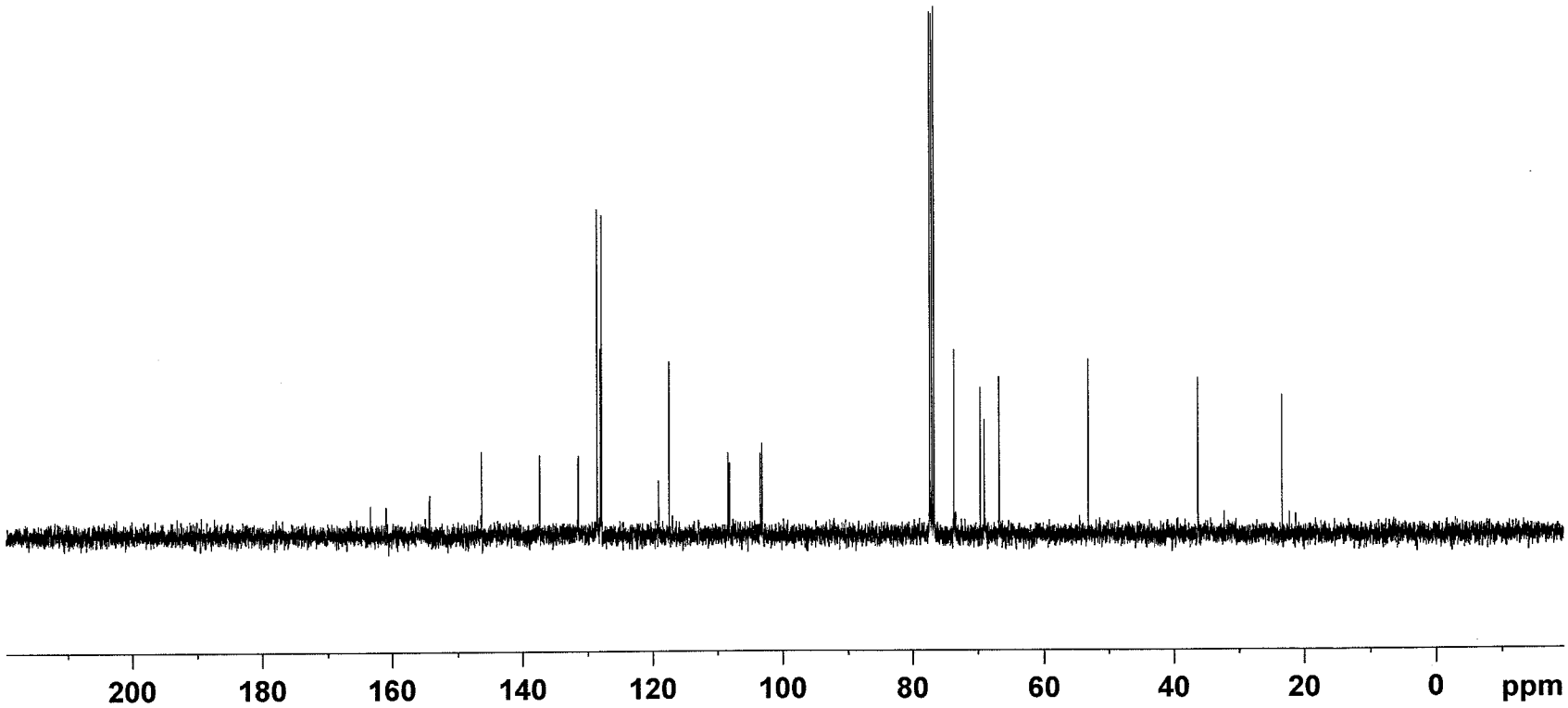
F2 - Processing parameters

SI	65536
SF	400.2100130 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

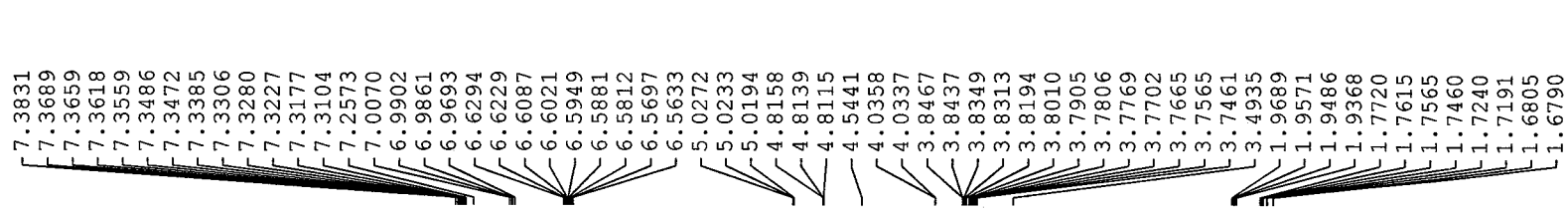


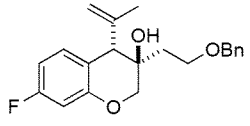


16 (major)

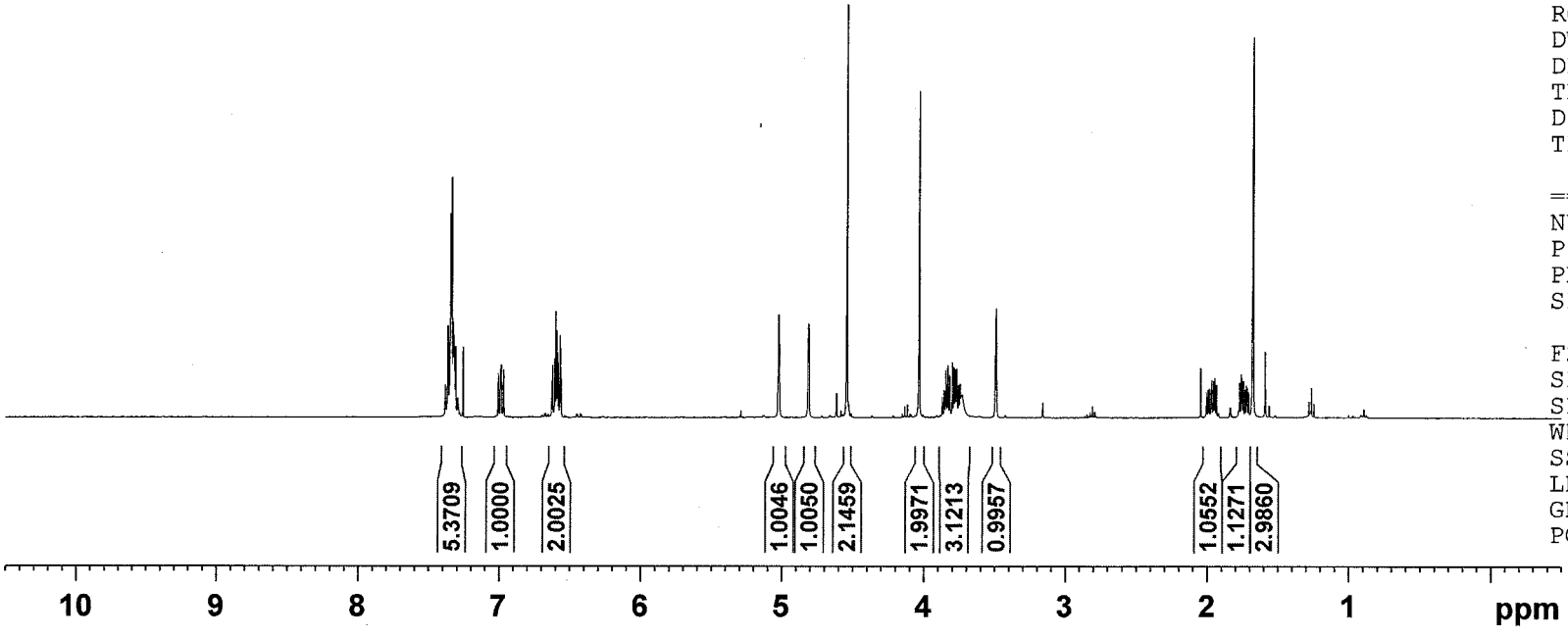



```
Current Data Parameters
NAME                  LJE-242
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121004
Time                  15.09
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      35
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                  190.62
DW                  20.800 usec
DE                   6.50 usec
TE                  299.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PLW1          52.00000000 W
SFO1         100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2          12.00000000 W
PLW12          0.33333001 W
PLW13          0.27000001 W
SFO2         400.2116008 MHz

F2 - Processing parameters
SI                    32768
SF           100.6328860 MHz
WDW                      EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```





16' (minor)

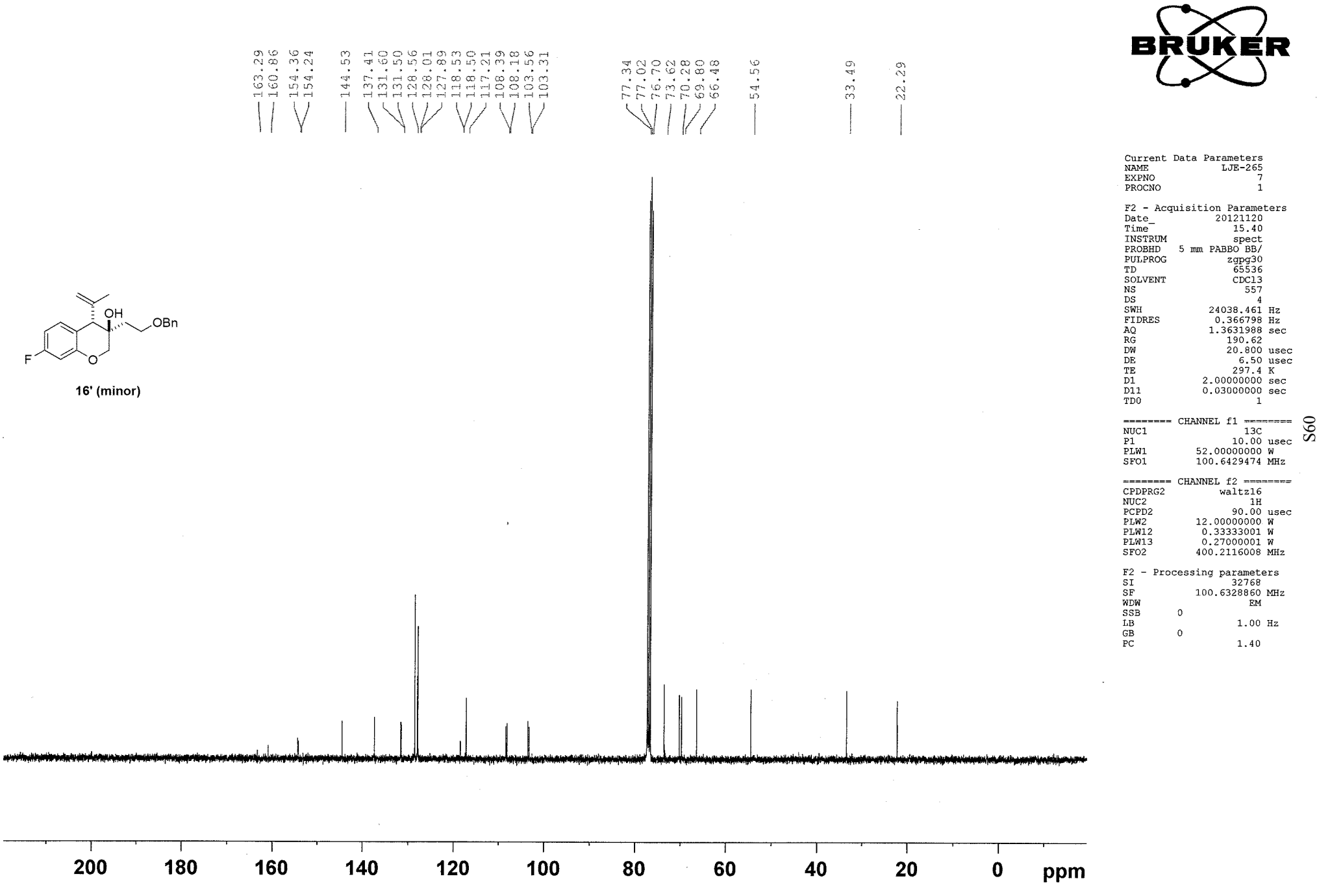


Current Data Parameters
NAME LJE-265
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121119
Time 13.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 69.69
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME LJE-265
EXPNO 7
PROCNO 1

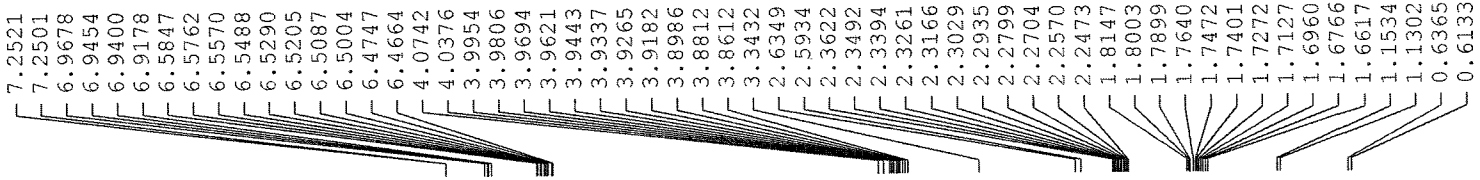
F2 - Acquisition Parameters
Date_ 20121120
Time 15.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 557
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

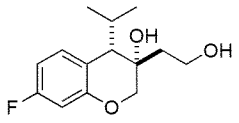
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

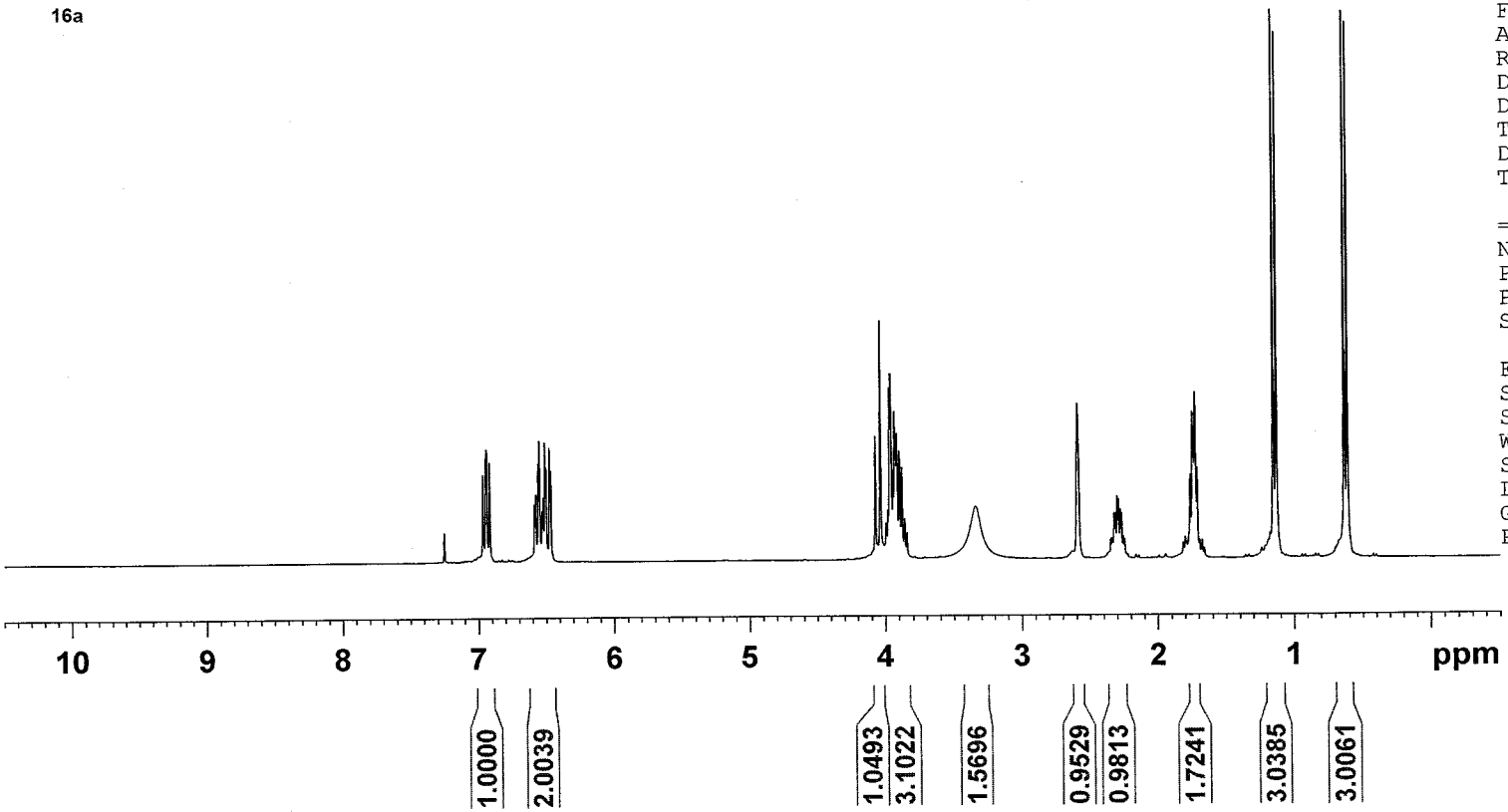
F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

LJE-163





16a



Current Data Parameters

NAME	LJE-163
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20120320
Time	20.52
INSTRUM	spect
PROBHD	5 mm DUL 1H-13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	90.5
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

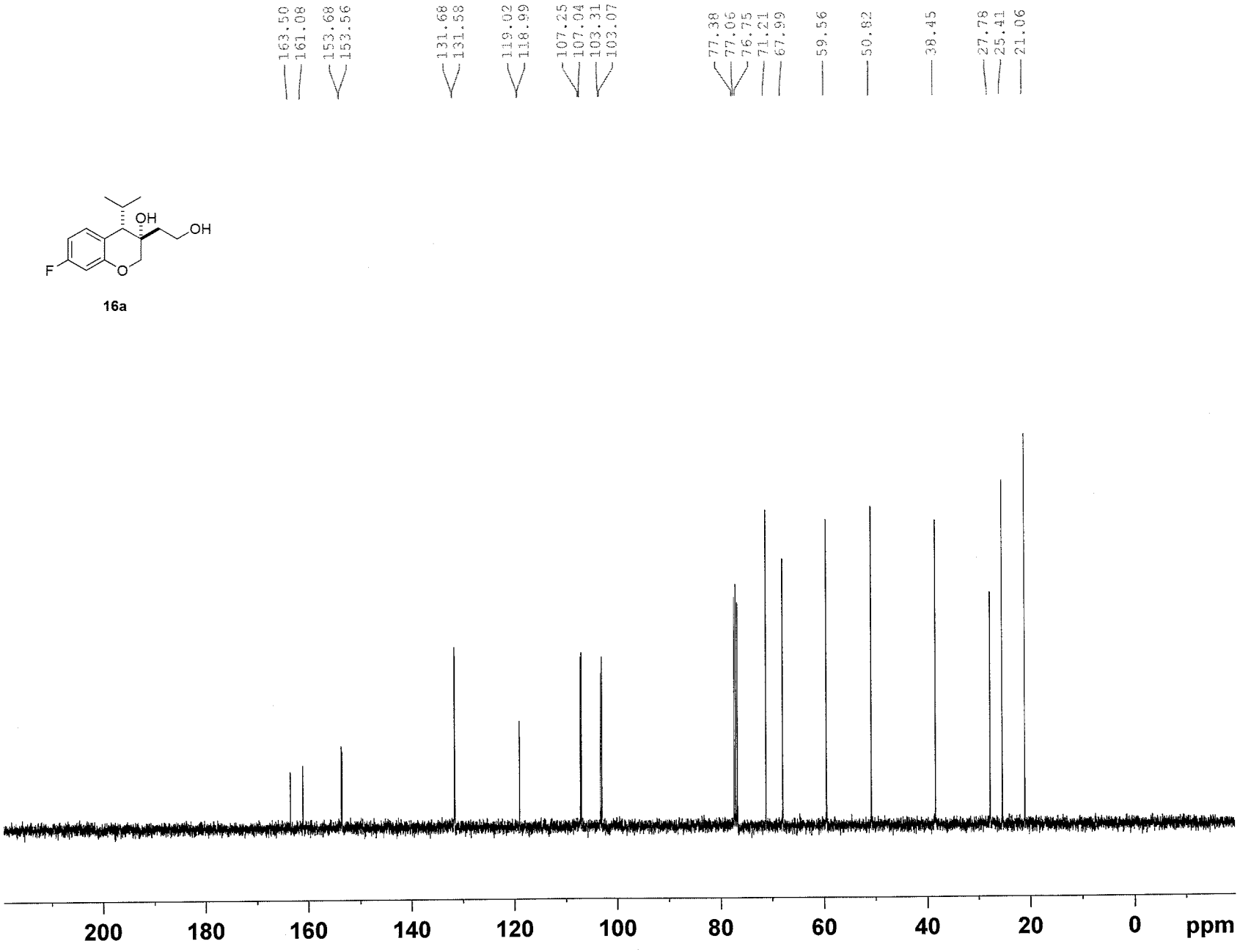
======== CHANNEL f1 ========

NUC1	1H
P1	10.50 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300147 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

16a



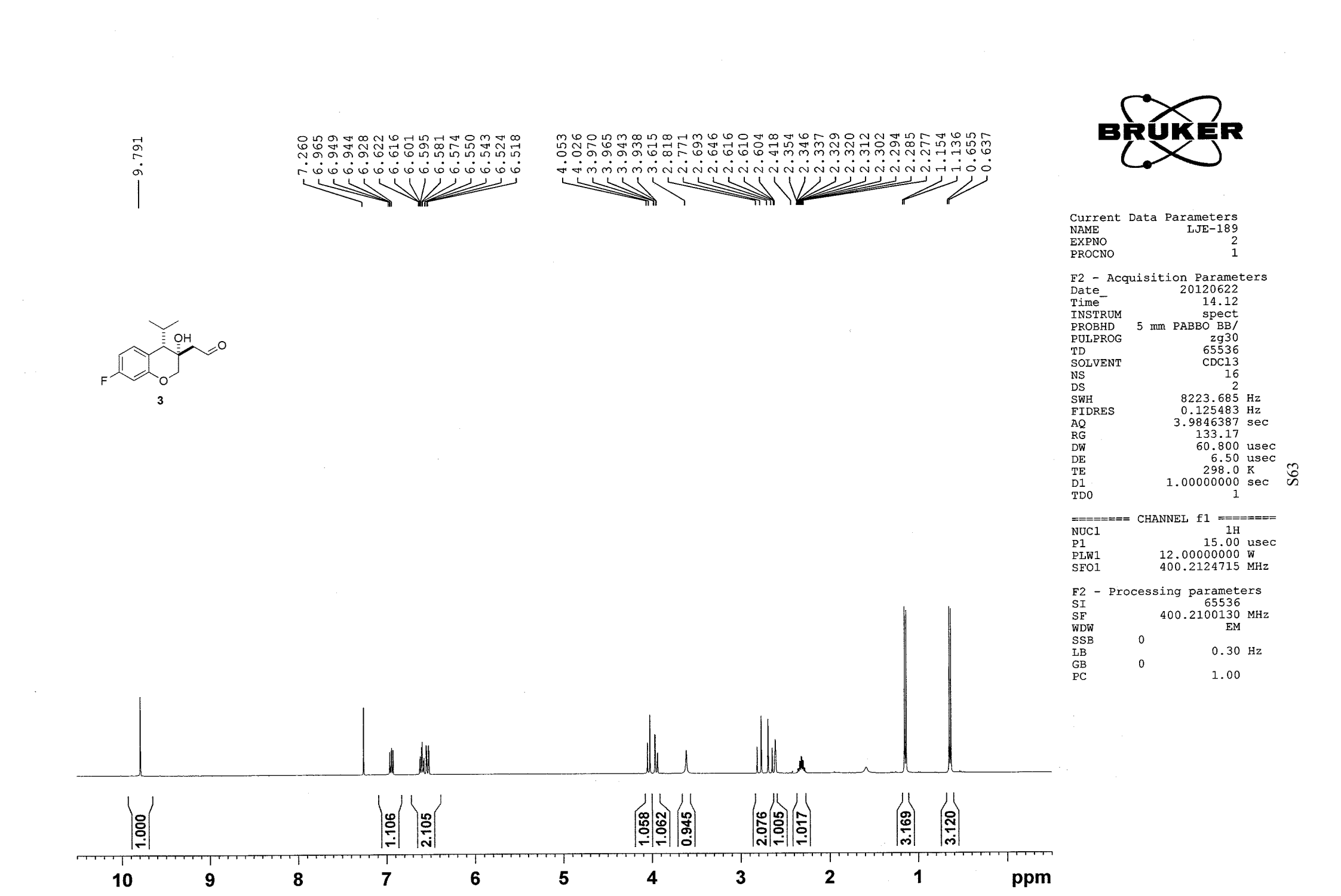
Current Data Parameters
NAME LJE-diol
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130416
Time 10.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 13
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



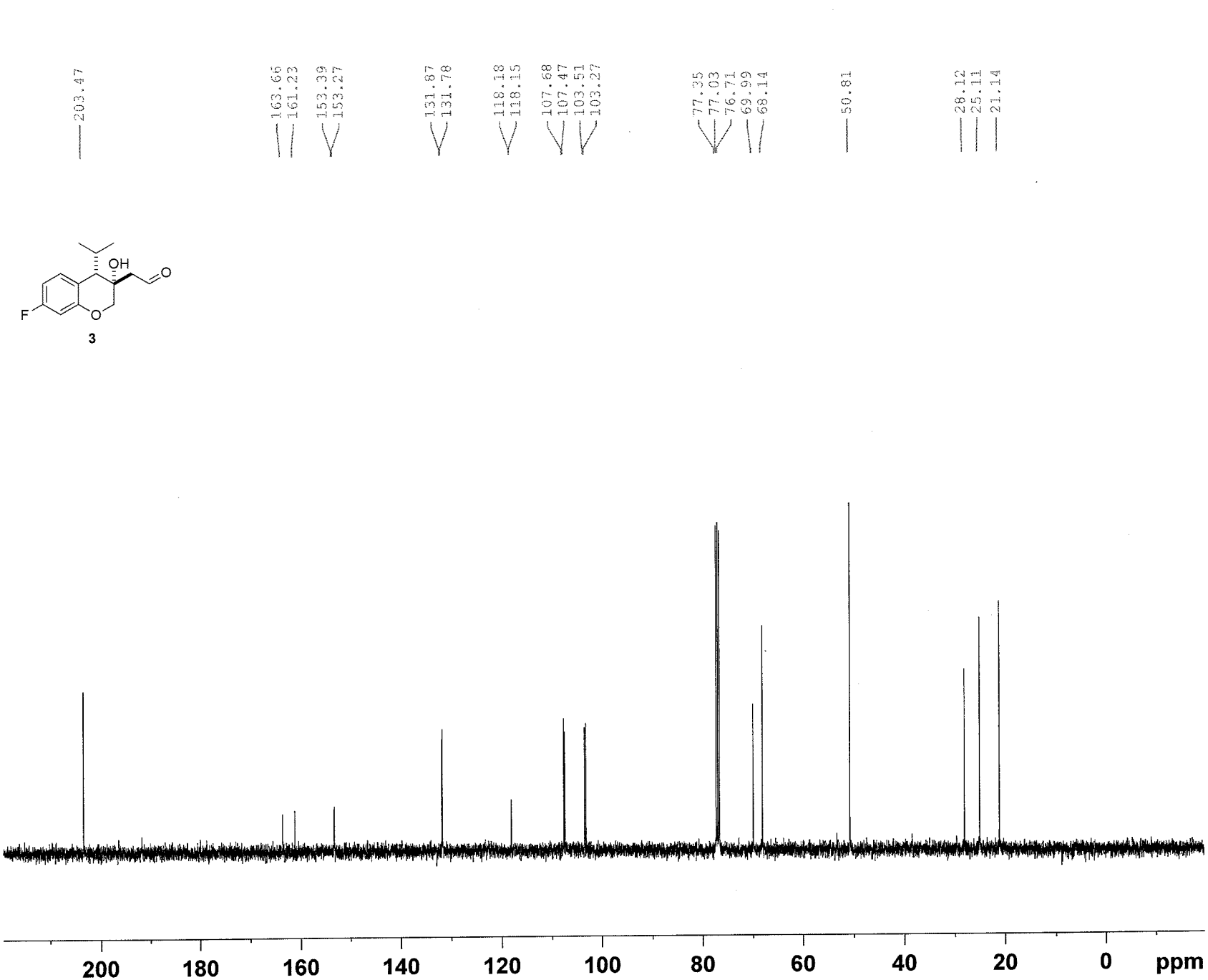
BRUKER

Current Data Parameters
NAME LJE-189
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120622
Time 14.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 133.17
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



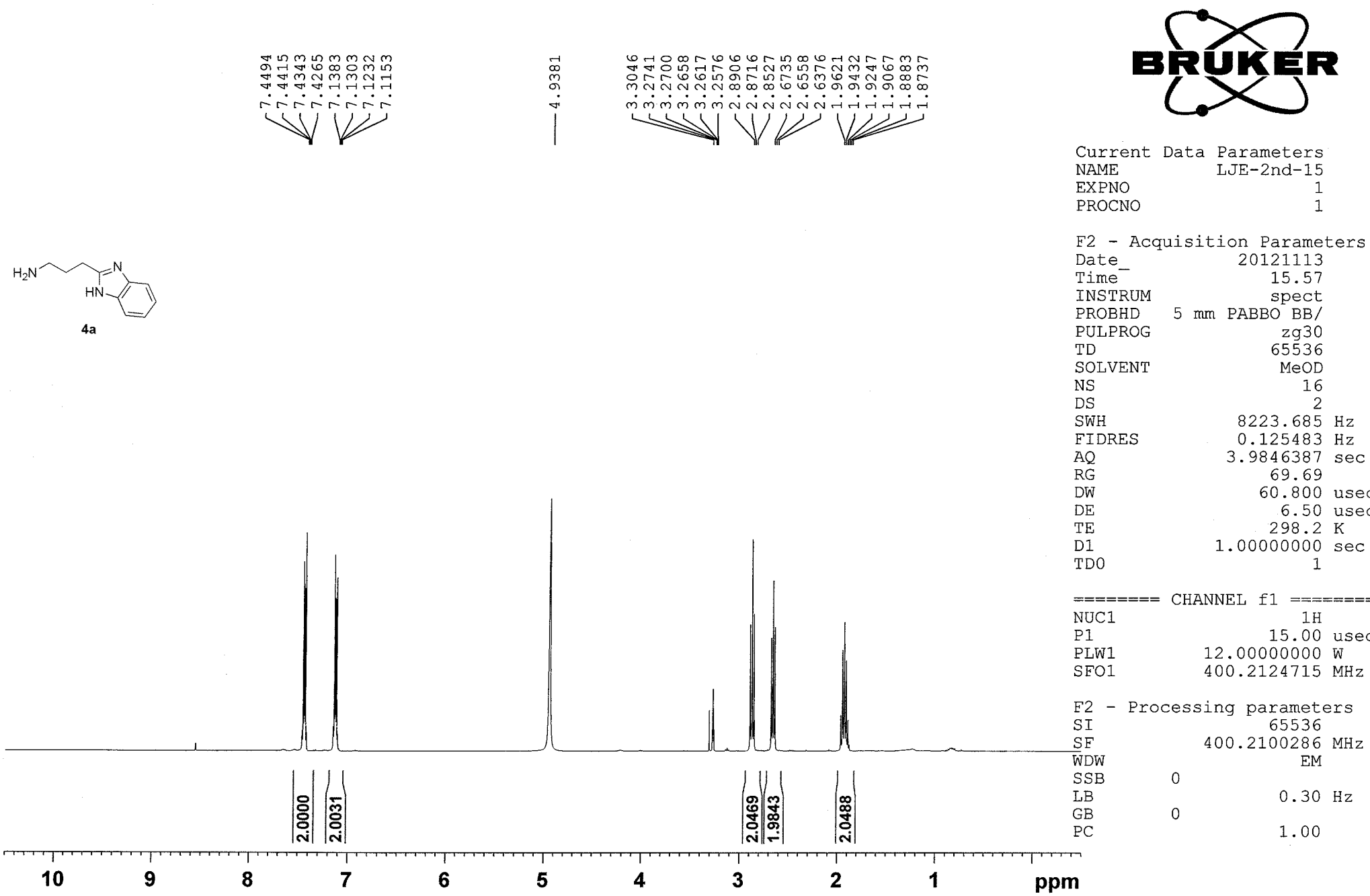
Current Data Parameters
NAME LJE-333
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130416
Time 21.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 39
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

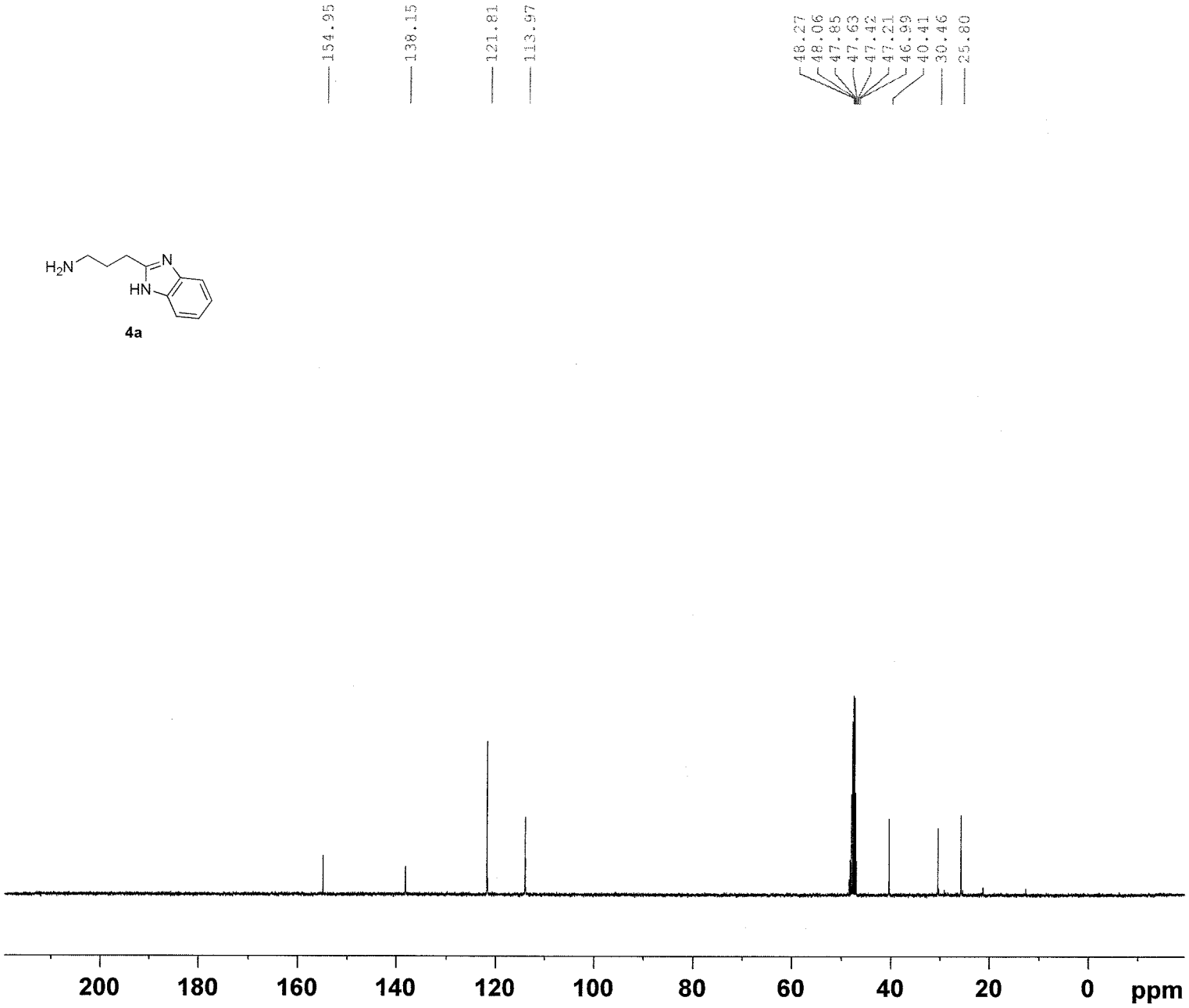
Current Data Parameters
NAME LJE-2nd-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121113
Time 15.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 69.69
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

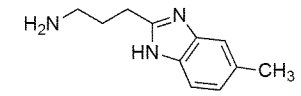
F2 - Processing parameters
SI 65536
SF 400.2100286 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

4a



BRUKER

```
Current Data Parameters
NAME          LJE-2nd-33
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120521
Time              14.35
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT            MeOD
NS                   56
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                190.62
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PLW1        52.00000000 W
SFO1        100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.33333001 W
PLW13        0.27000001 W
SFO2        400.2116008 MHz

F2 - Processing parameters
SI                32768
SF          100.6328860 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

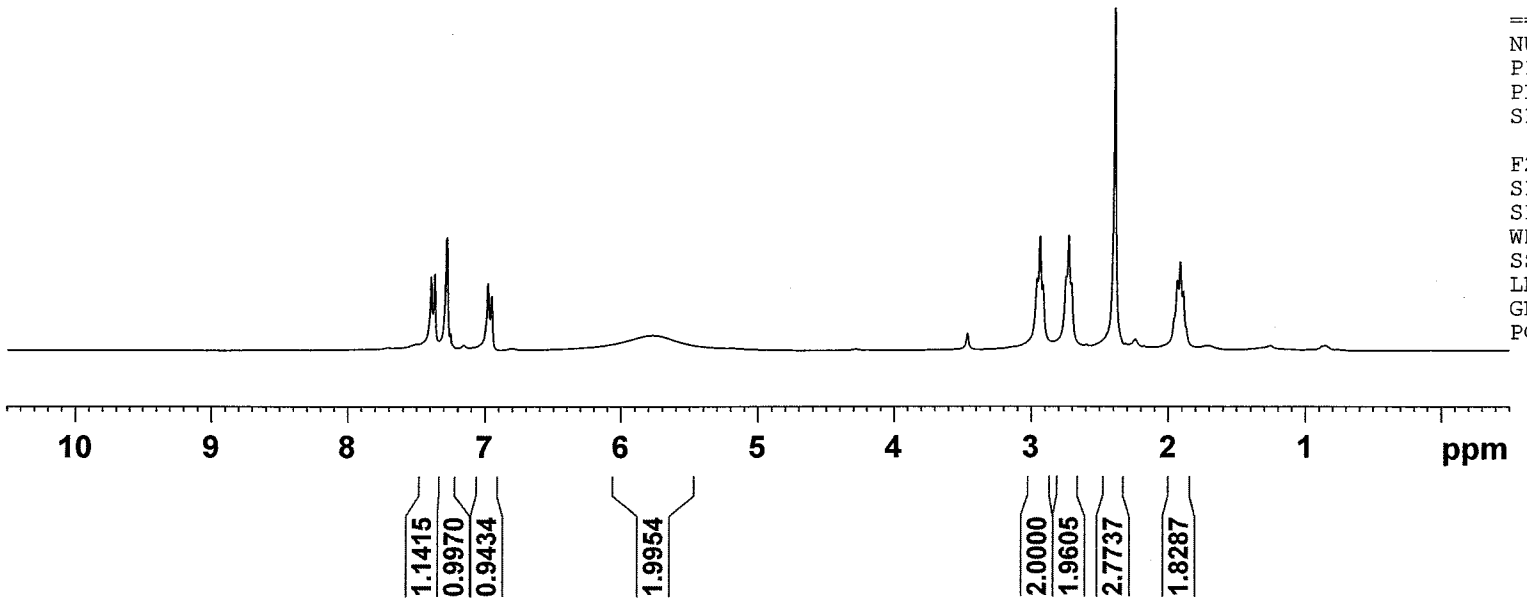


4b

7.3943
7.3673
7.2796
6.9805
6.9536

5.7715

2.9593
2.9370
2.9142
2.7462
2.7265
2.7062
2.3891
1.9331
1.9112
1.8891



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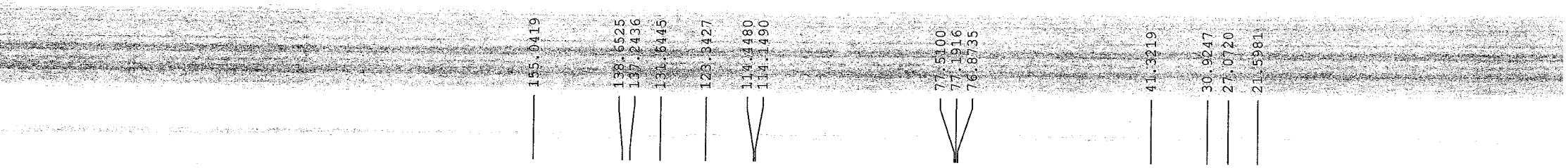
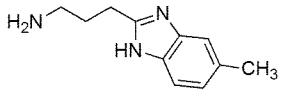
Current Data Parameters
NAME LJE-2nd-21
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120202
Time 10.59
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

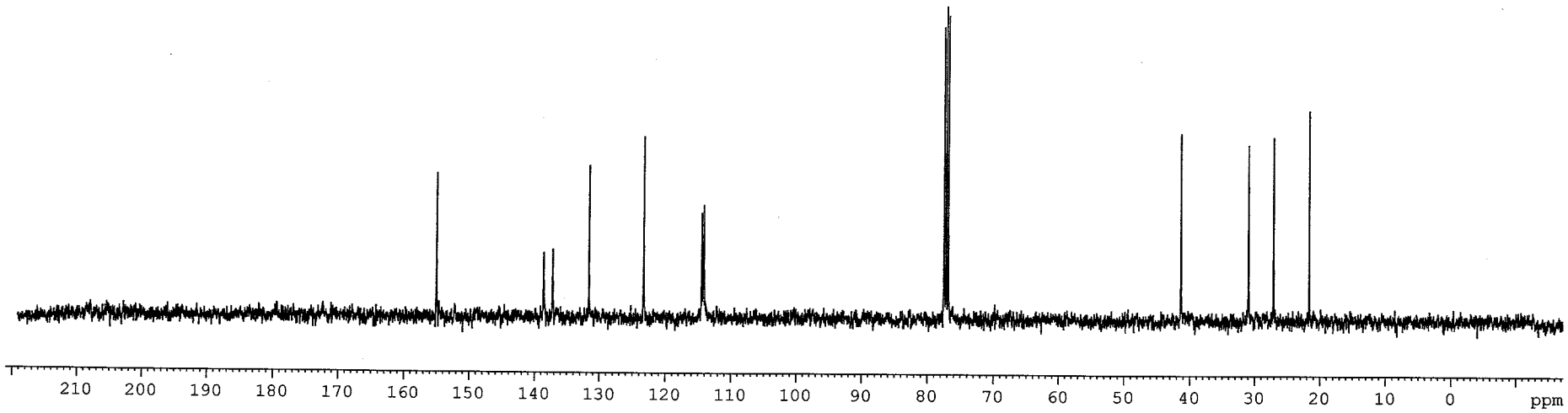
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

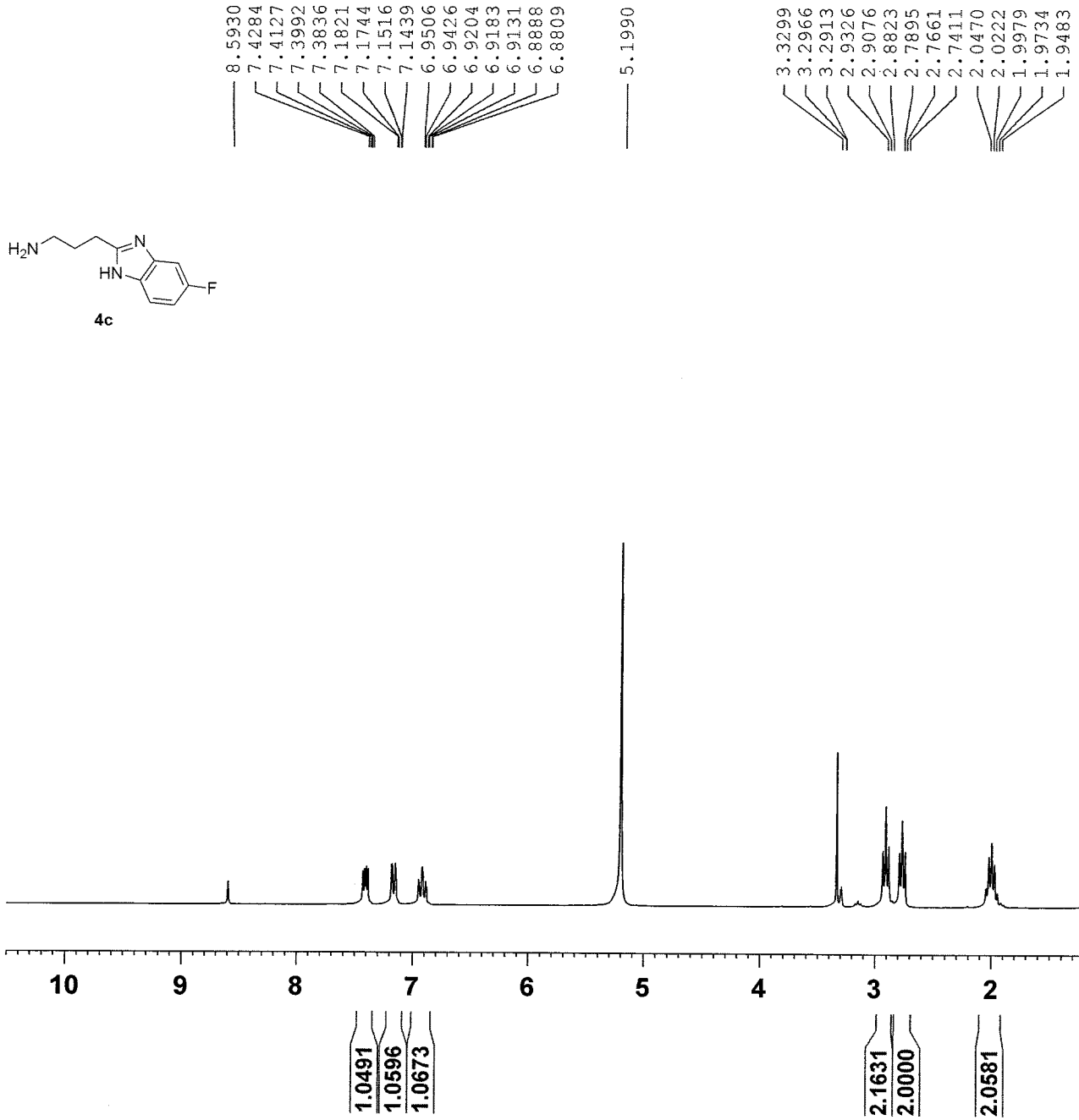

155.0419
138.6525
137.2436
134.6445
123.5427
114.4480
114.1490
77.5100
77.1916
76.8735
41.3219
30.9247
27.0720
21.5981


H2N
HN
N
CH3

4b


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

4c



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Current Data Parameters
NAME LJE-2nd-31
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

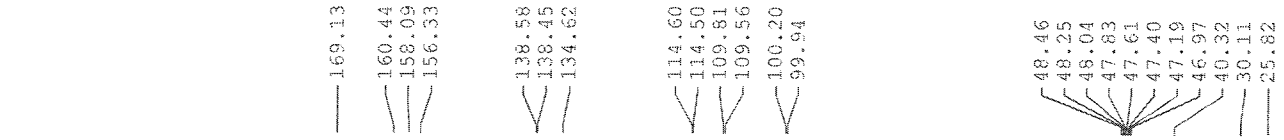
Date_	20120303
Time	16.35
INSTRUM	spect
PROBHD	5 mm DUL 1H-13
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	80.6
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

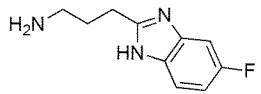
======== CHANNEL f1 ========

NUC1	1H
P1	10.50 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

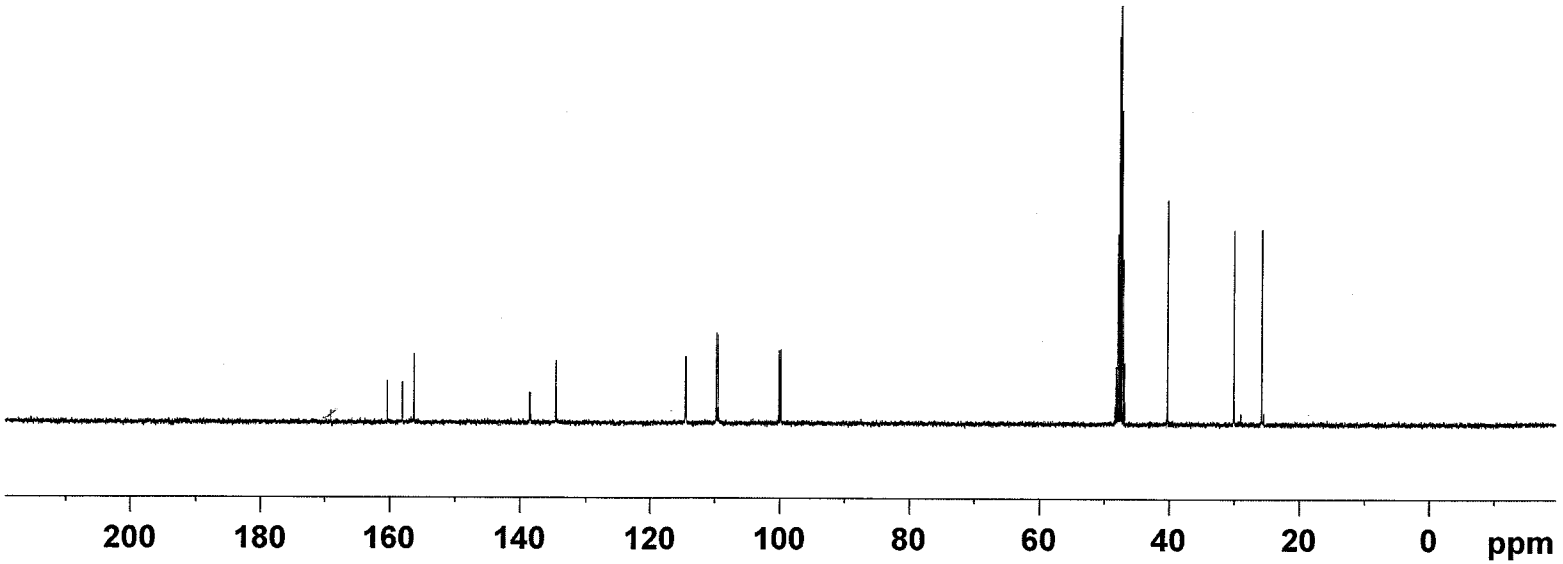
F2 - Processing parameters

SI	32768
SF	300.1300101 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





4c



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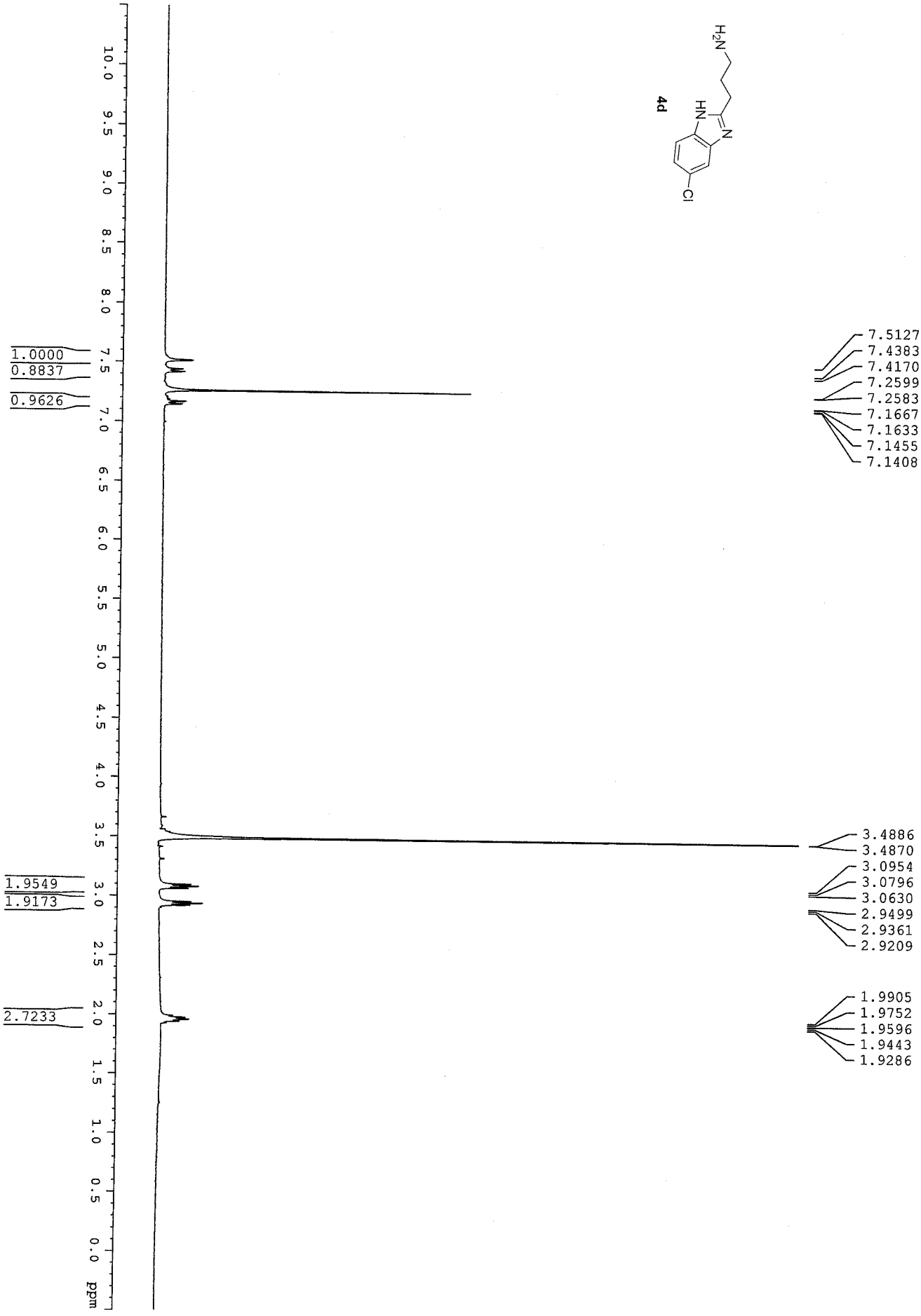
Current Data Parameters
NAME LJE-2nd-23
EXPNO 2
PROCNO 1

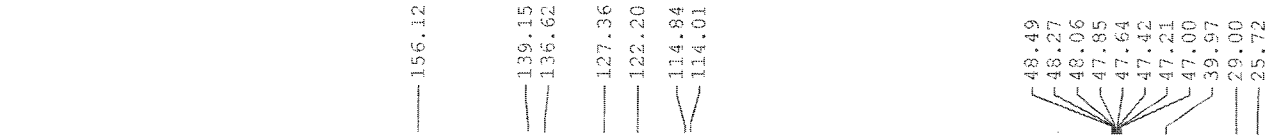
F2 - Acquisition Parameters
Date_ 20120518
Time 20.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 89
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

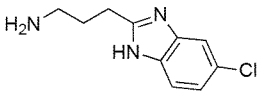
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

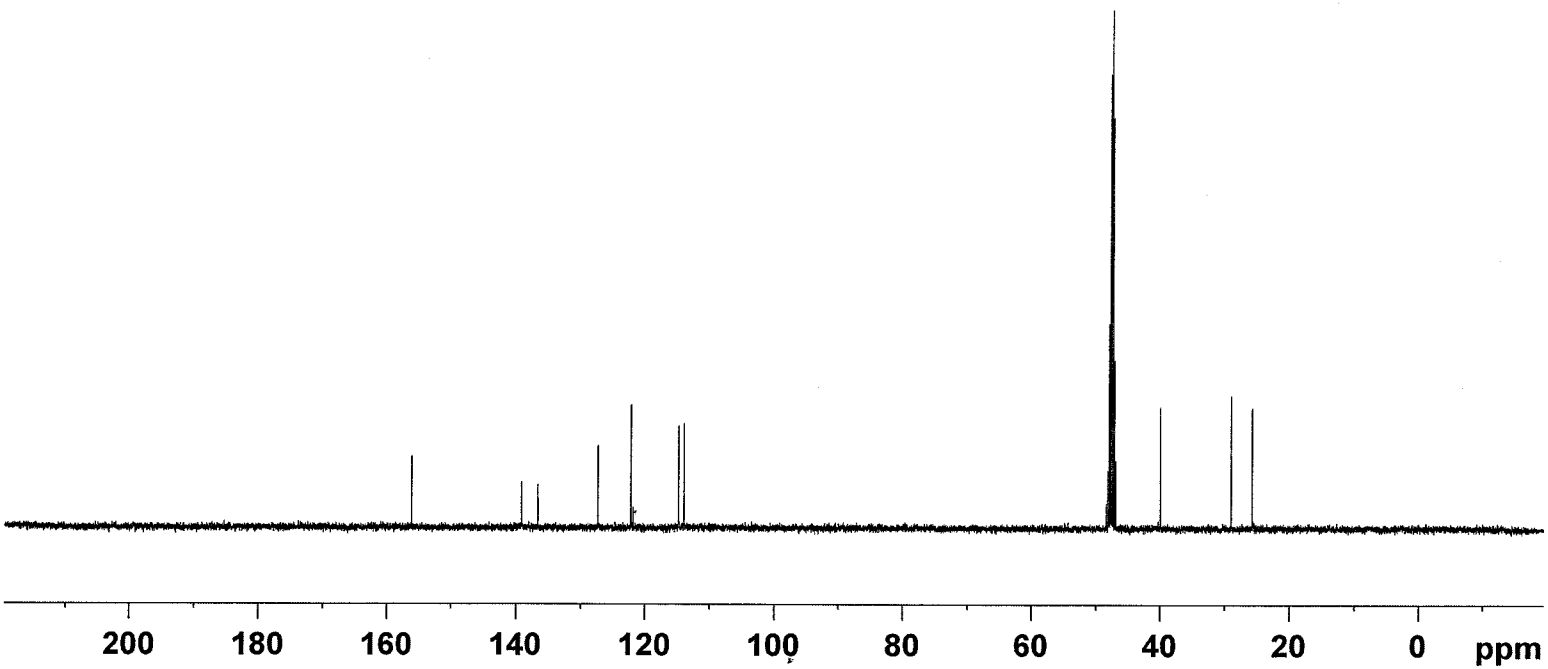
F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


4d
H2N
HN
N
Cl
7.5127
7.4383
7.4170
7.2599
7.2583
7.1667
7.1633
7.1455
7.1408
3.4886
3.4870
3.0954
3.0796
3.0630
2.9499
2.9361
2.9209
1.9905
1.9752
1.9596
1.9443
1.9286
1.0000
0.8837
0.9626
1.9549
1.9173
2.7233
ppm





4d



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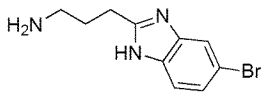
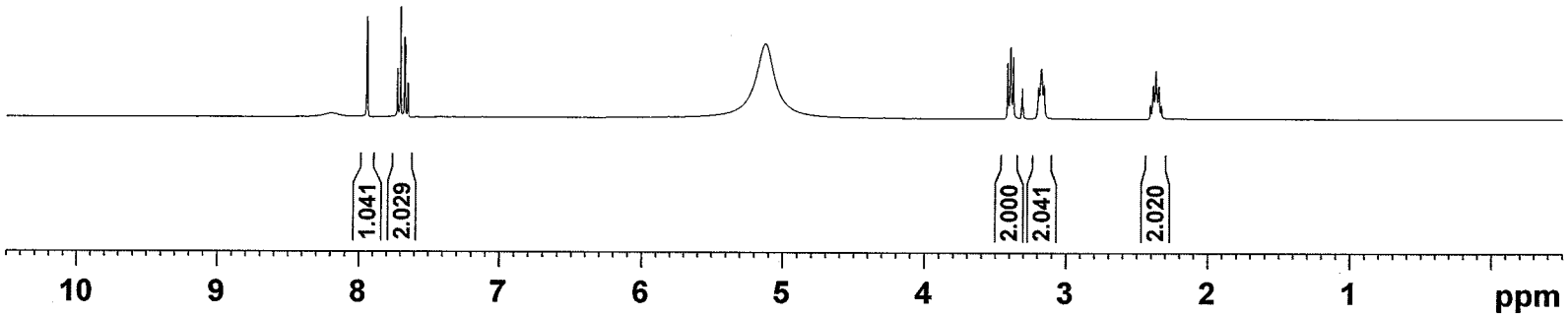
```
Current Data Parameters
NAME          LJE-2nd-44
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120518
Time               20.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                    47
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                190.62
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PLW1        52.00000000 W
SFO1       100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2        12.00000000 W
PLW12        0.33333001 W
PLW13        0.27000001 W
SFO2       400.2116008 MHz

F2 - Processing parameters
SI                 32768
SF         100.6328860 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4e

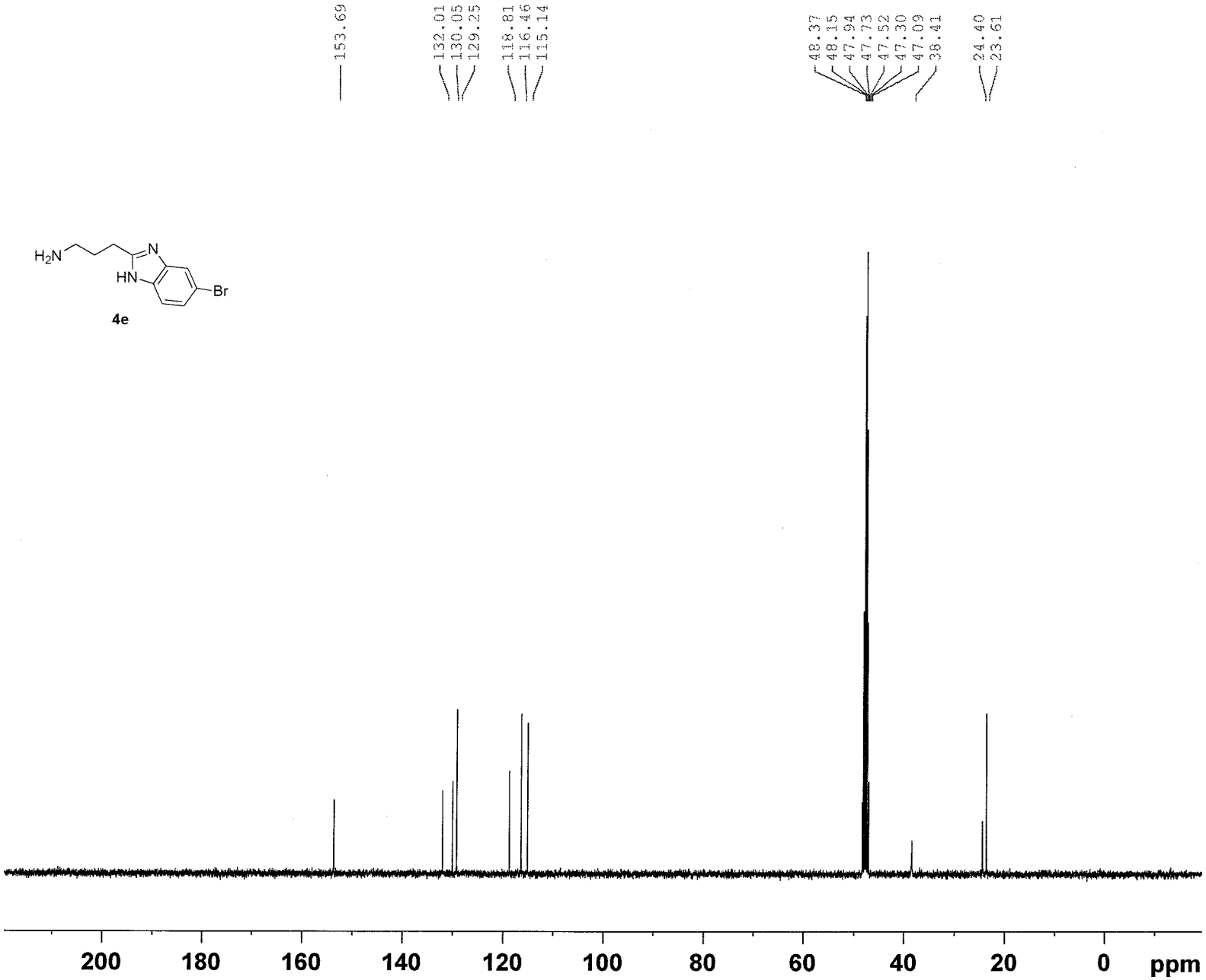


Current Data Parameters
NAME LJE-2nd-54
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120619
Time 14.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.2
DW 60.800 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



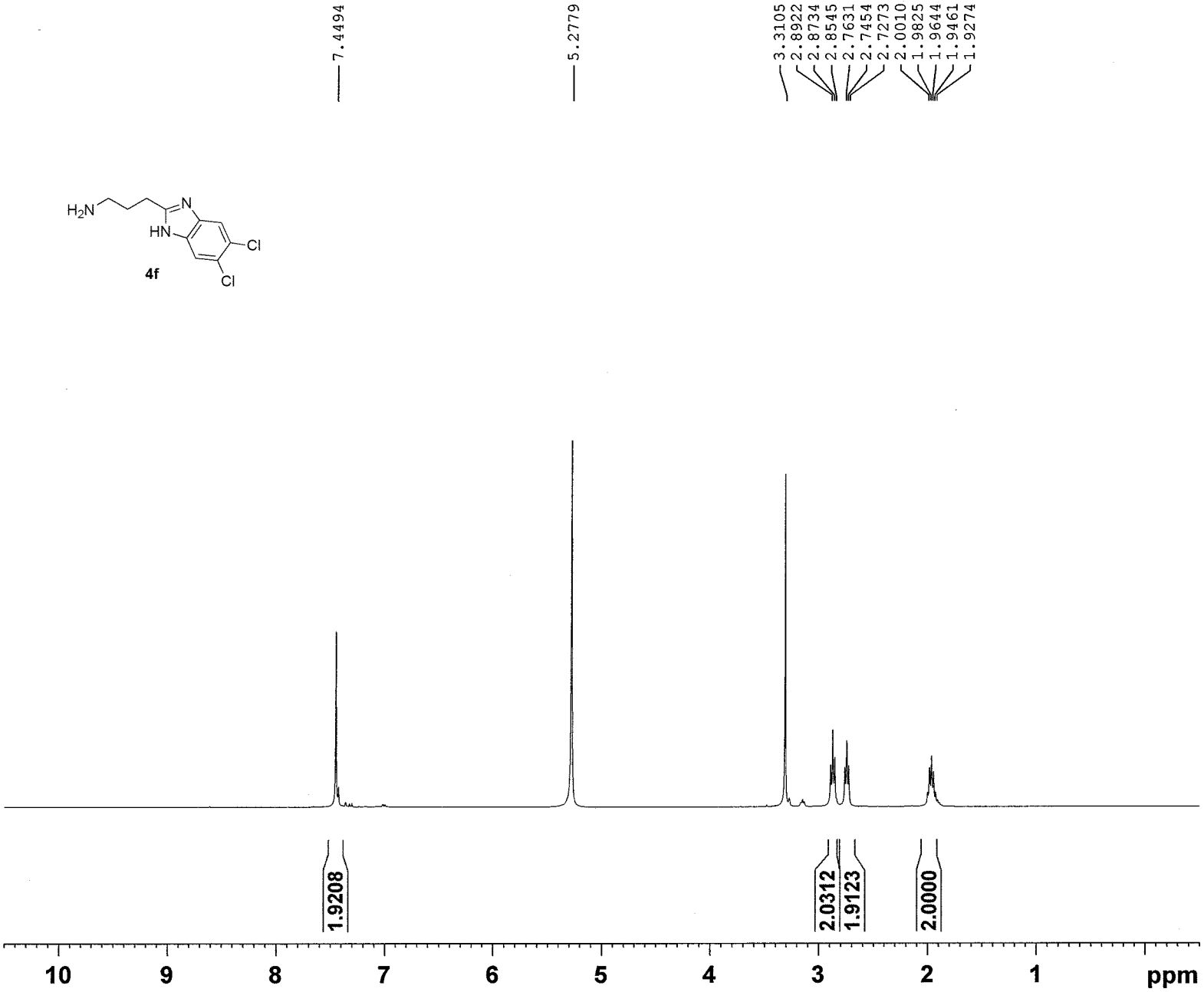
Current Data Parameters
NAME LJE-2nd-54
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120619
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 53
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

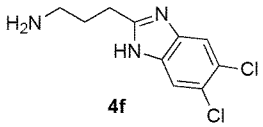


Current Data Parameters
NAME LJE-2nd-48
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120530
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.2
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1

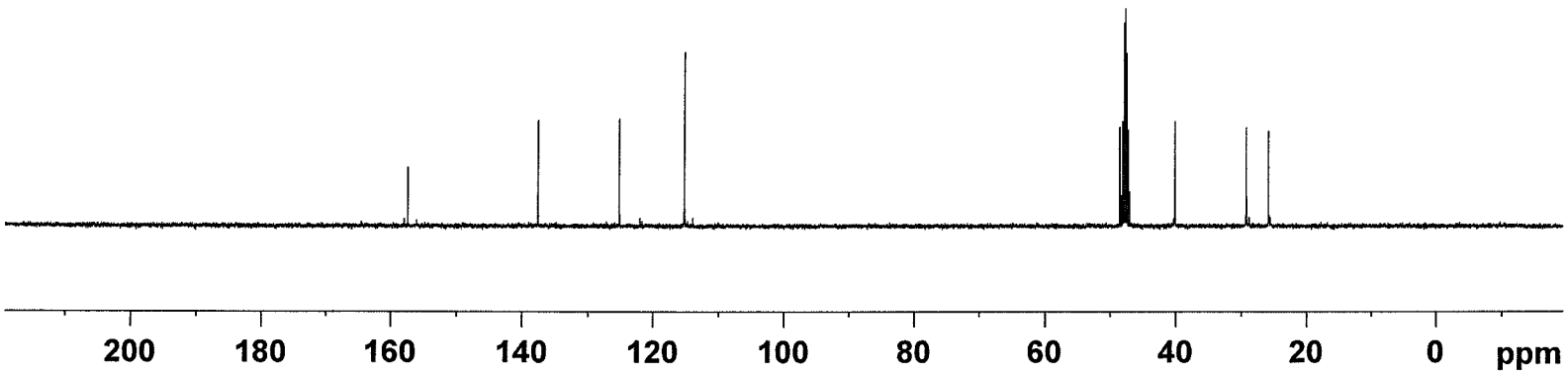
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100261 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4f

157.46
137.61
125.28
115.25
48.53
48.32
48.11
47.90
47.69
47.47
47.26
47.05
40.14
29.22
25.83



Current Data Parameters
NAME LJE-2nd-48
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20120530
Time	20.38
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	MeOD
NS	64
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	190.62
DW	20.800 usec
DE	6.50 usec
TE	299.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

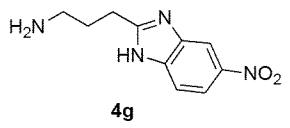
NUC1	13C
P1	10.00 usec
PLW1	52.00000000 W
SFO1	100.6429474 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PLW2	12.00000000 W
PLW12	0.33333001 W
PLW13	0.27000001 W
SFO2	400.2116008 MHz

F2 - Processing parameters

SI	32768
SF	100.6328860 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4g

8.6893
8.6831
8.4550
7.9904
7.9603
4.8960
3.4255
3.3990
3.3731
3.3083
3.3037
3.2986
3.2935
3.1844
3.1595
3.1341
2.3930
2.3656
2.3410
2.3161
2.2909

10 9 8 7 6 5 4 3 2 1 ppm

1.0000 0.9769 0.9810 2.0059 1.9571 1.9496

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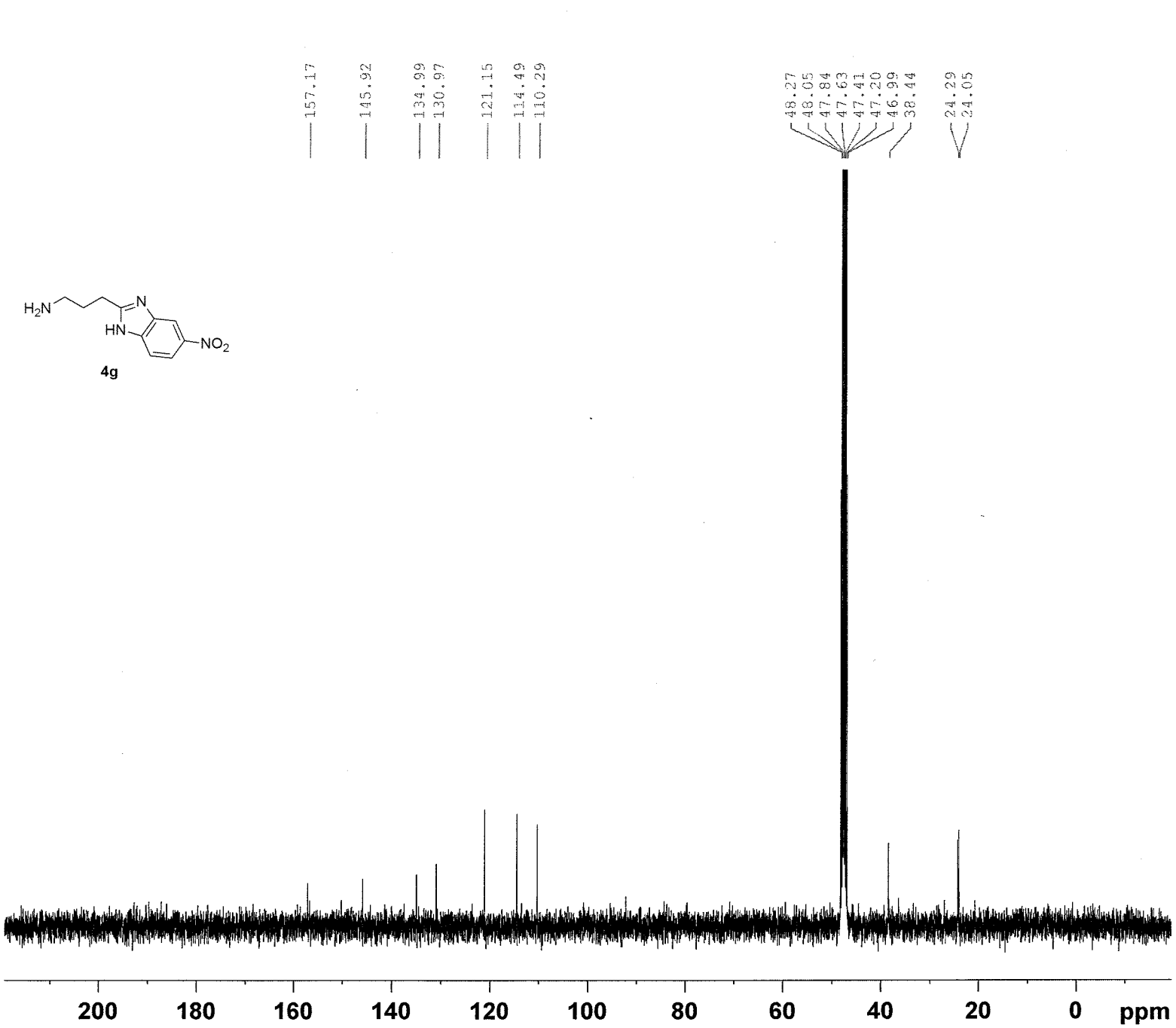
```
Current Data Parameters
NAME                  LJE-2nd-40
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20120411
Time                       20.44
INSTRUM                    spect
PROBHD   5 mm DUL 1H-13
PULPROG                     zg30
TD                         65536
SOLVENT                     MeOD
NS                            16
DS                             2
SWH                     6172.839 Hz
FIDRES                  0.094190 Hz
AQ                      5.3084660 sec
RG                         912.3
DW                        81.000 usec
DE                         6.00 usec
TE                        300.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        10.50 usec
PL1                       -1.00 dB
SFO1                300.1318534 MHz

F2 - Processing parameters
SI                         32768
SF                  300.1300067 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
```



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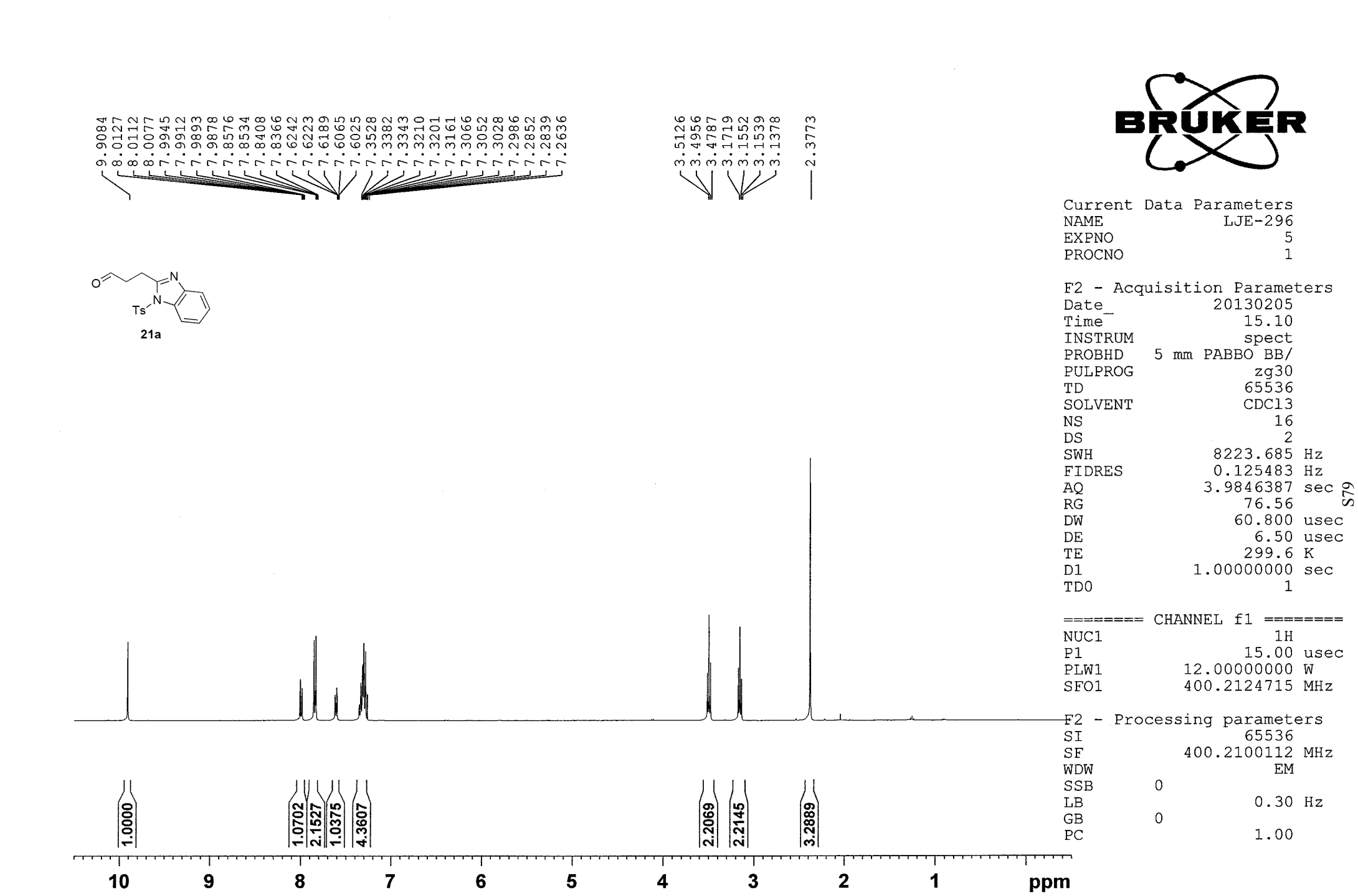
```
Current Data Parameters
NAME          LJE-2nd-37
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120520
Time               18.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             MeOD
NS                   213
DS                     4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG               190.62
DW              20.800 usec
DE               6.50 usec
TE              298.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PLW1        52.00000000 W
SFO1        100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.33333001 W
PLW13        0.27000001 W
SFO2        400.2116008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6328860 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



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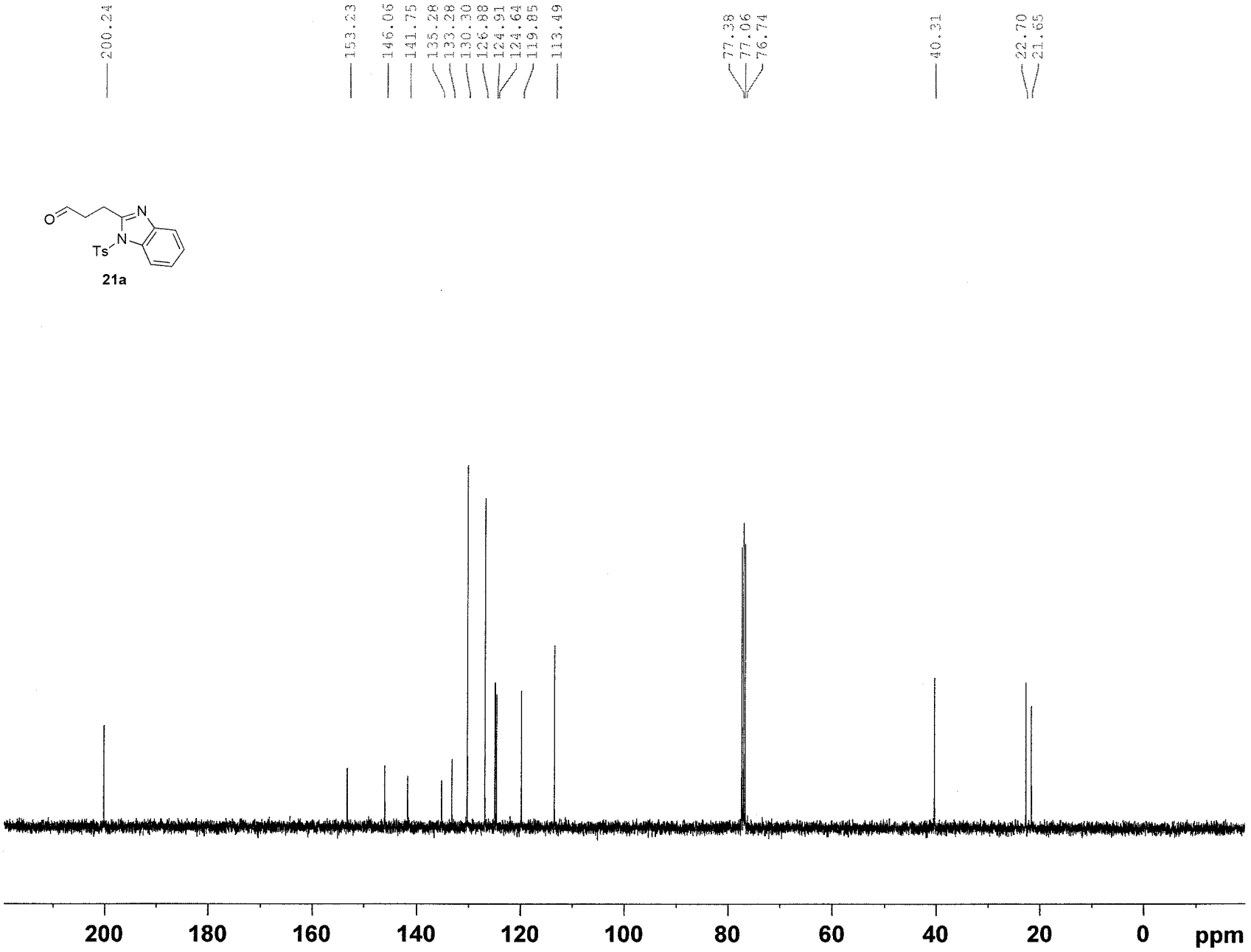
Current Data Parameters
NAME LJE-296
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130205
Time 15.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 76.56
DW 60.800 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



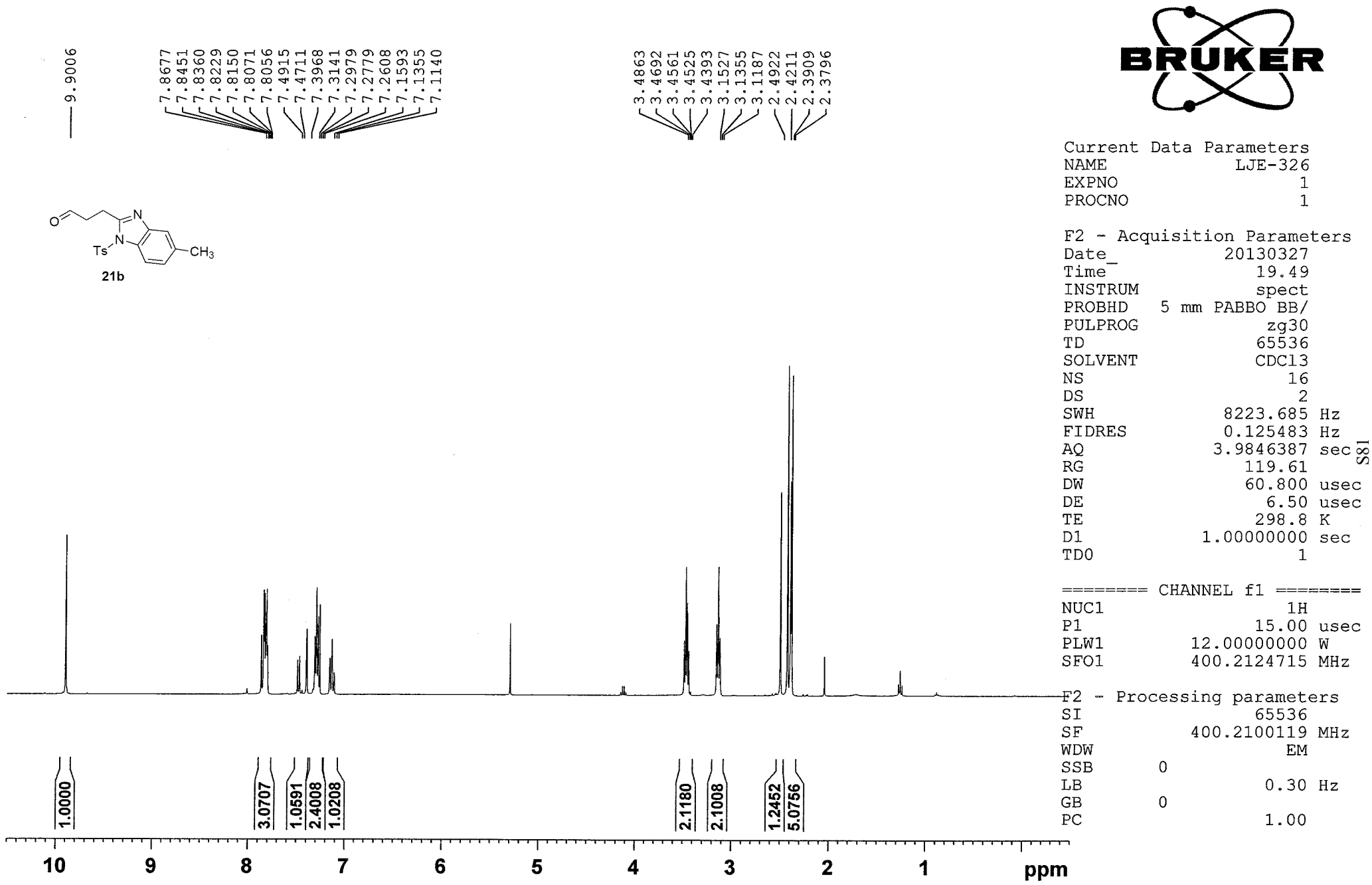
Current Data Parameters
NAME LJE-296
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130205
Time 15.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 34
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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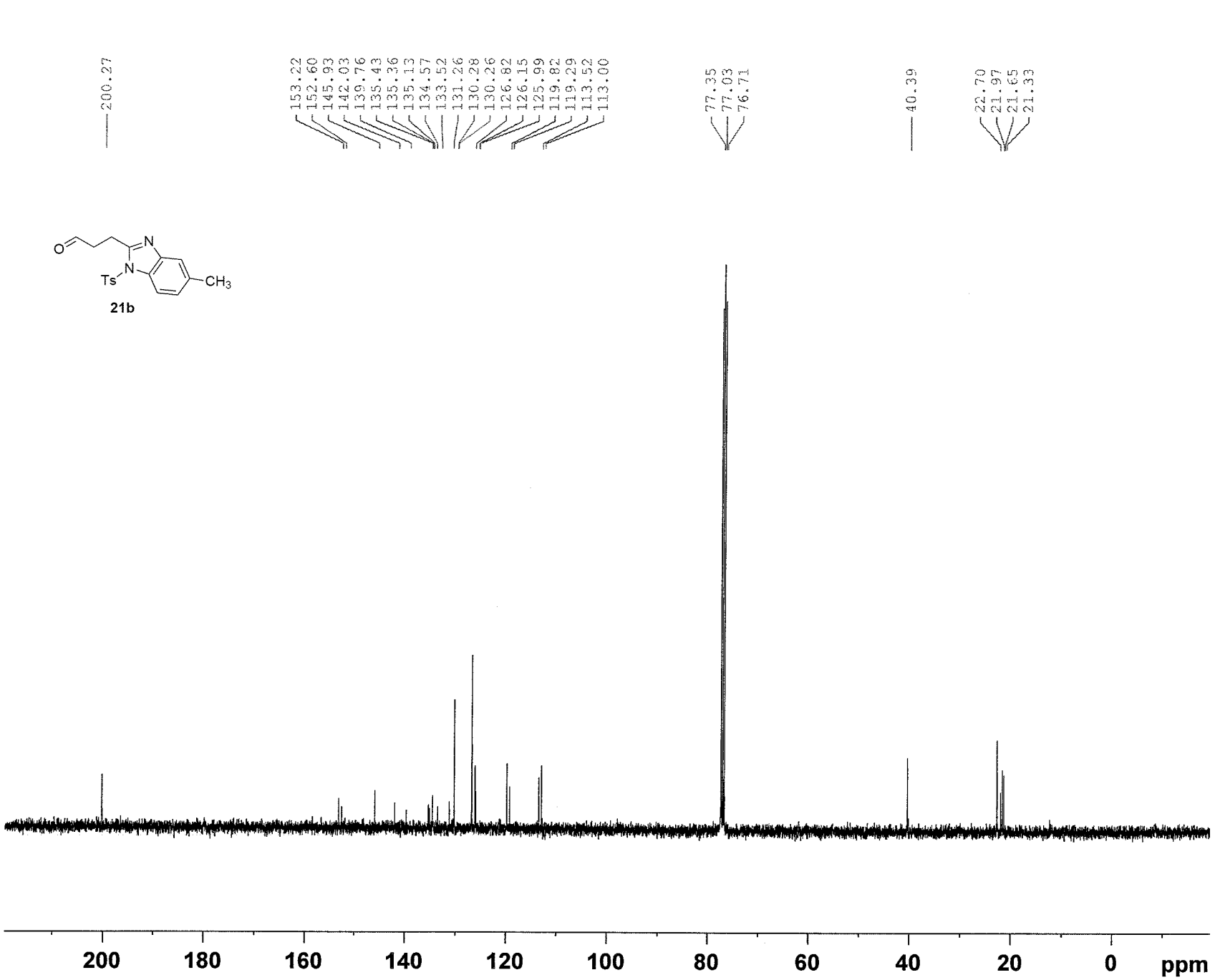
Current Data Parameters
NAME LJE-326
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130327
Time 19.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 119.61
DW 60.800 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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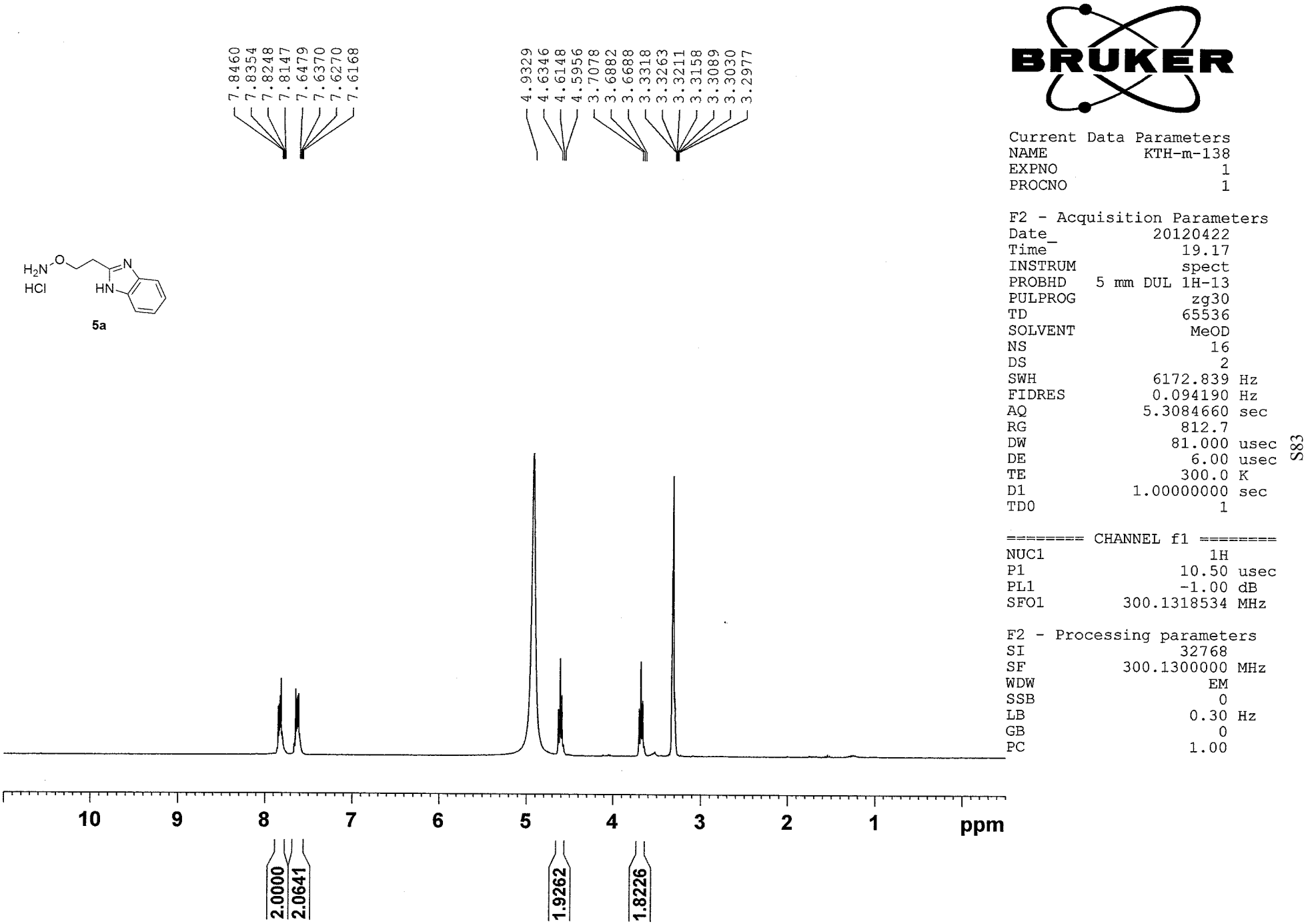
Current Data Parameters
NAME LJE-326
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130327
Time 19.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 73
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	KTH-m-138
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

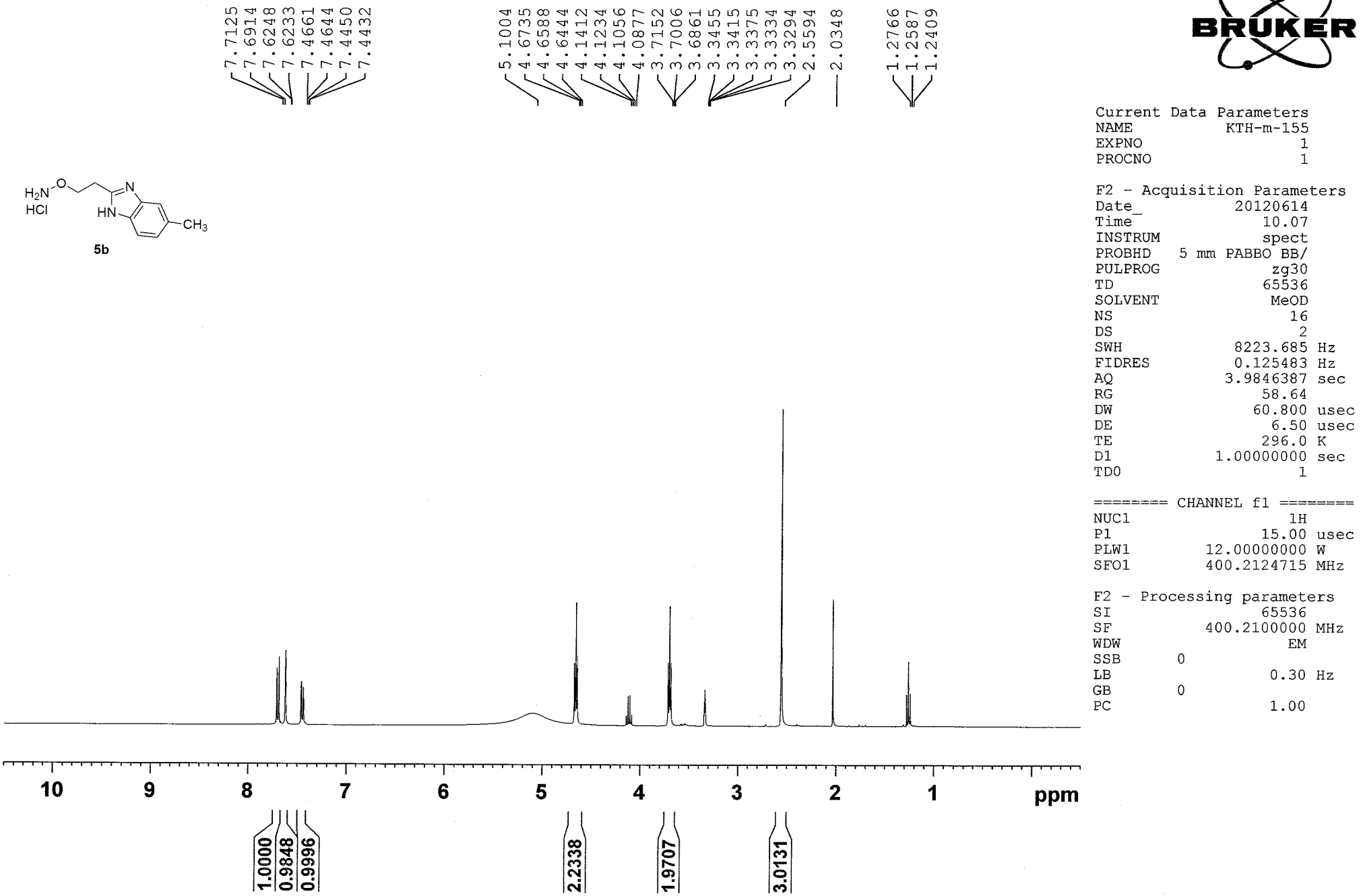
Date_	20120422
Time	19.17
INSTRUM	spect
PROBHD	5 mm DUL 1H-13
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	812.7
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	10.50 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



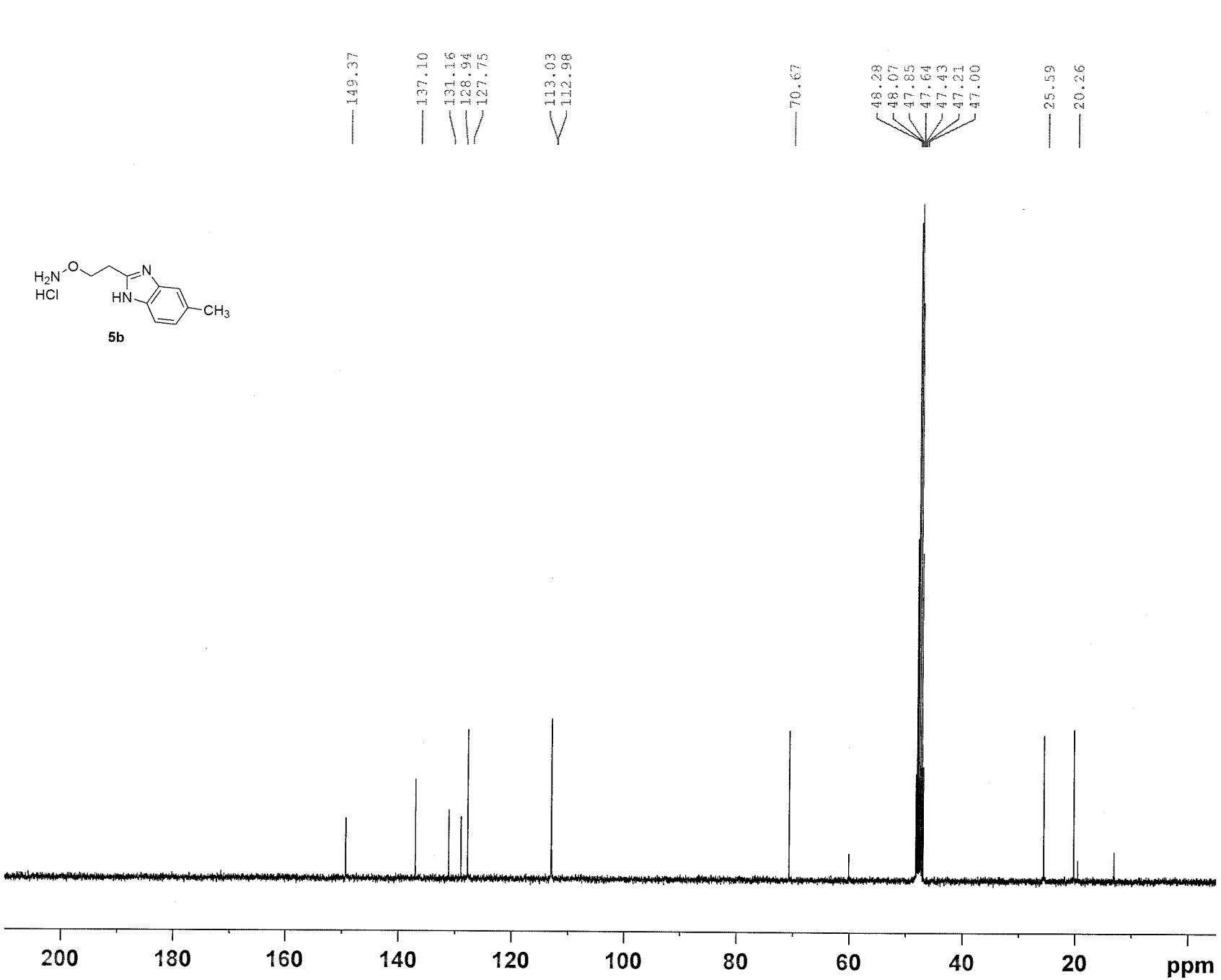
BRUKER

Current Data Parameters
NAME KTH-m-155
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120614
Time 10.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 58.64
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

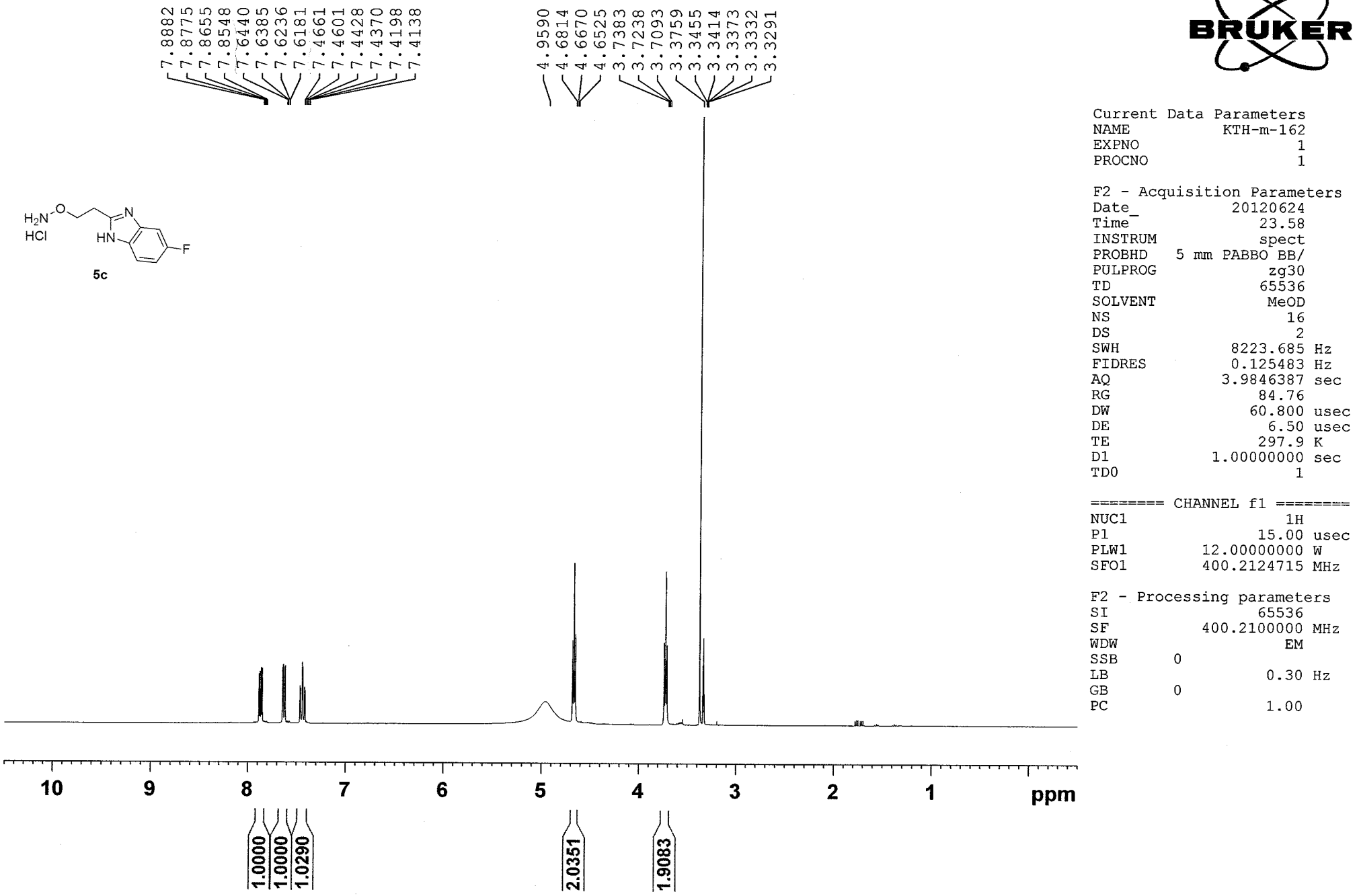
Current Data Parameters
NAME KTH-m-155
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120614
Time 10.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 127
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



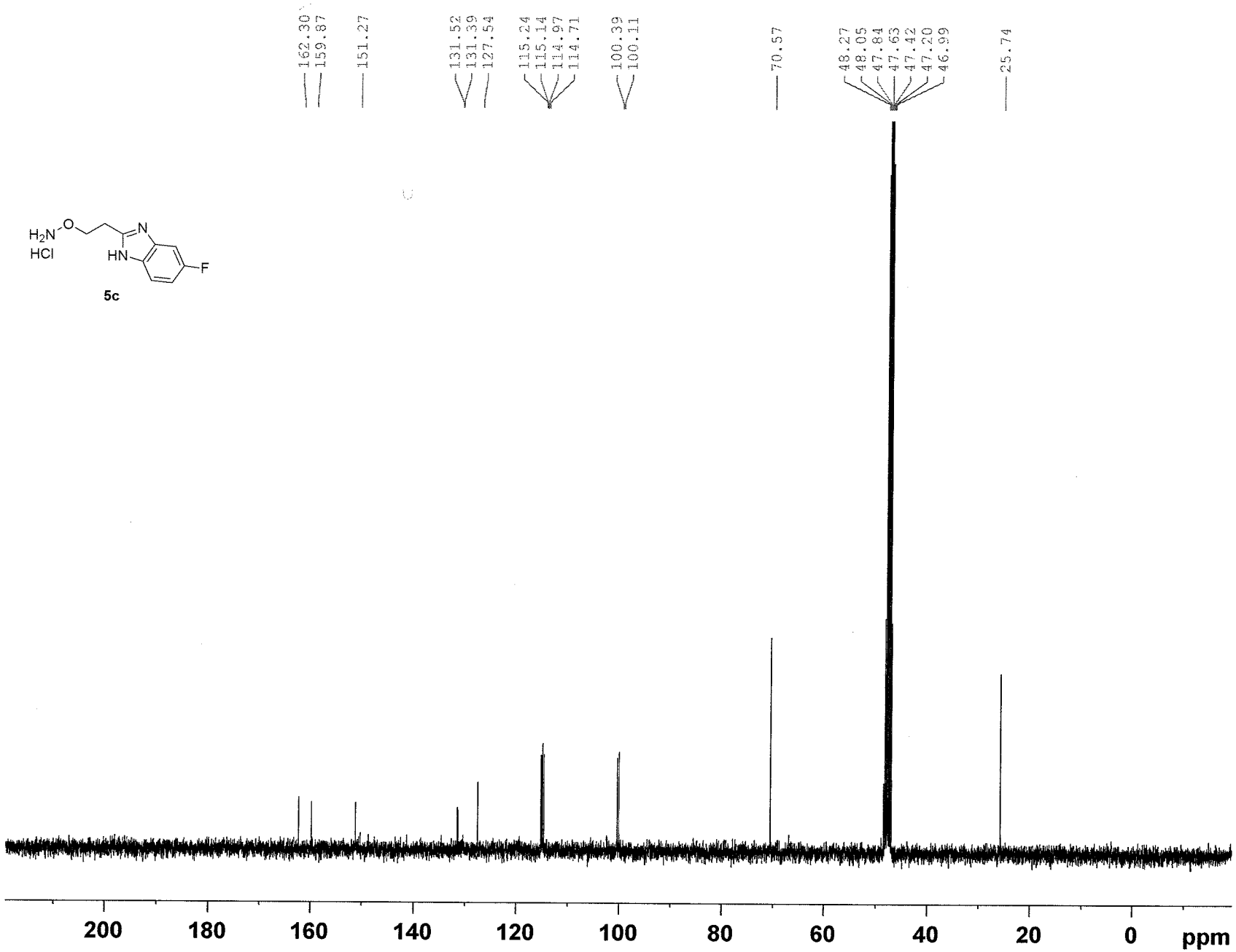
BRUKER

Current Data Parameters
NAME KTH-m-162
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120624
Time 23.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 84.76
DW 60.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

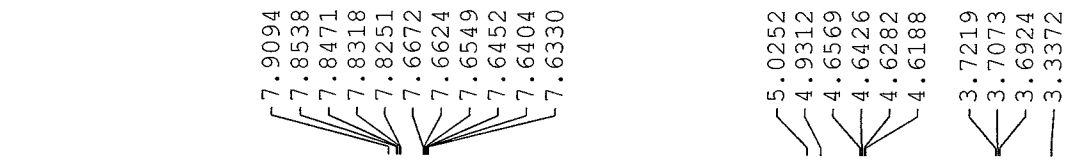
Current Data Parameters
NAME KTH-m-162
EXPNO 2
PROCNO 1

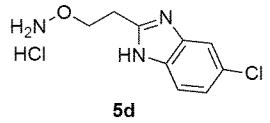
F2 - Acquisition Parameters
Date_ 20120625
Time 0.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 78
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

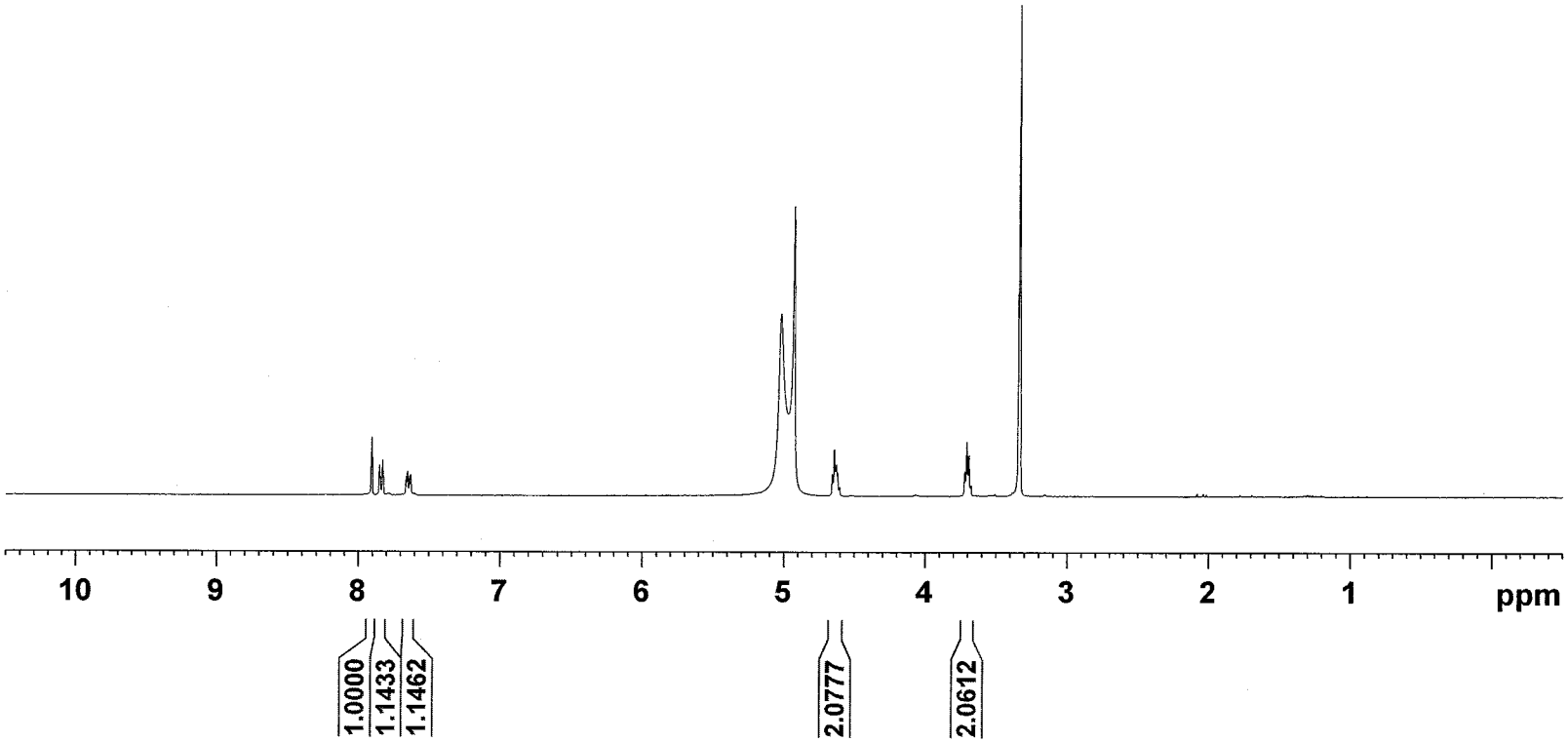
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





5d

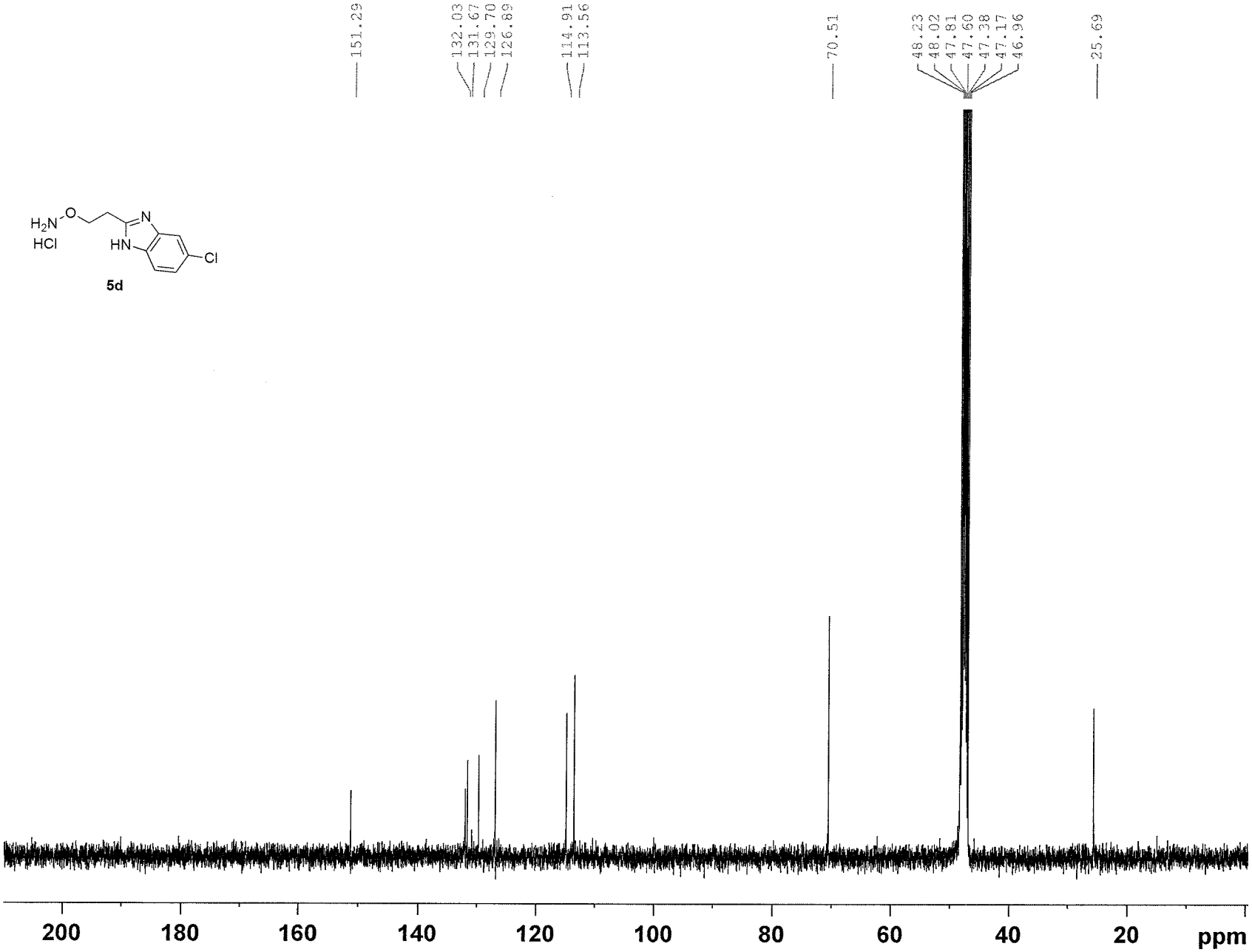


Current Data Parameters
NAME KTH-m-153
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120612
Time 21.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 171.62
DW 60.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



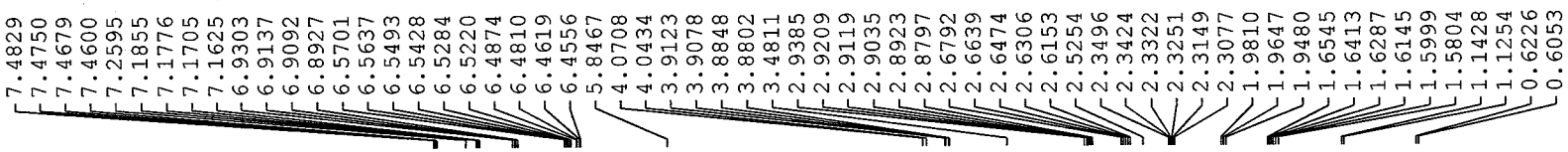
Current Data Parameters
NAME KTH-m-153
EXPNO 3
PROCNO 1

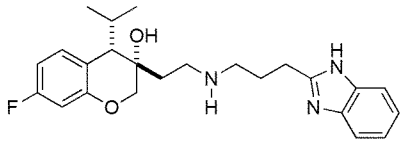
F2 - Acquisition Parameters
Date_ 20120613
Time 8.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 11477
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

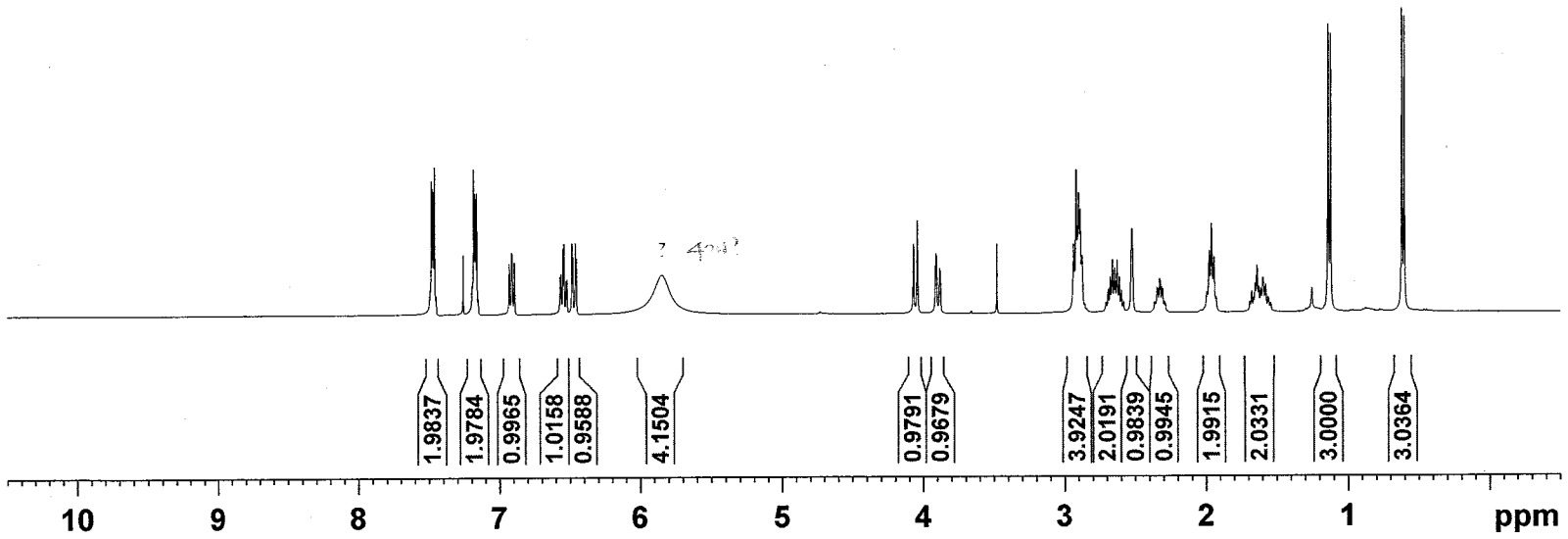
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2aa



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Current Data Parameters

NAME	LJE-182
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

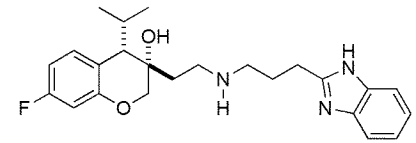
Date_	20120531
Time	20.35
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	33.2
DW	60.800 usec
DE	6.50 usec
TE	298.4 K
D1	1.00000000 sec
TD0	1

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

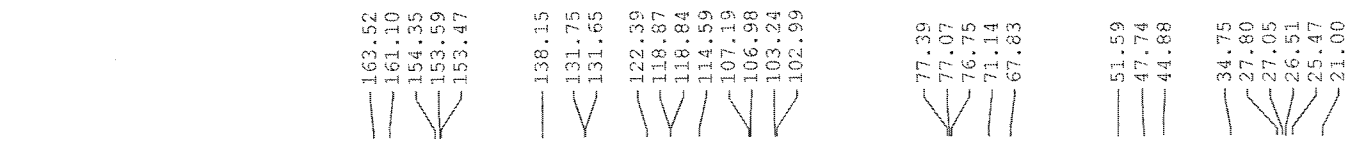
NUC1	1H
P1	15.00 usec
PLW1	12.00000000 W
SFO1	400.2124715 MHz

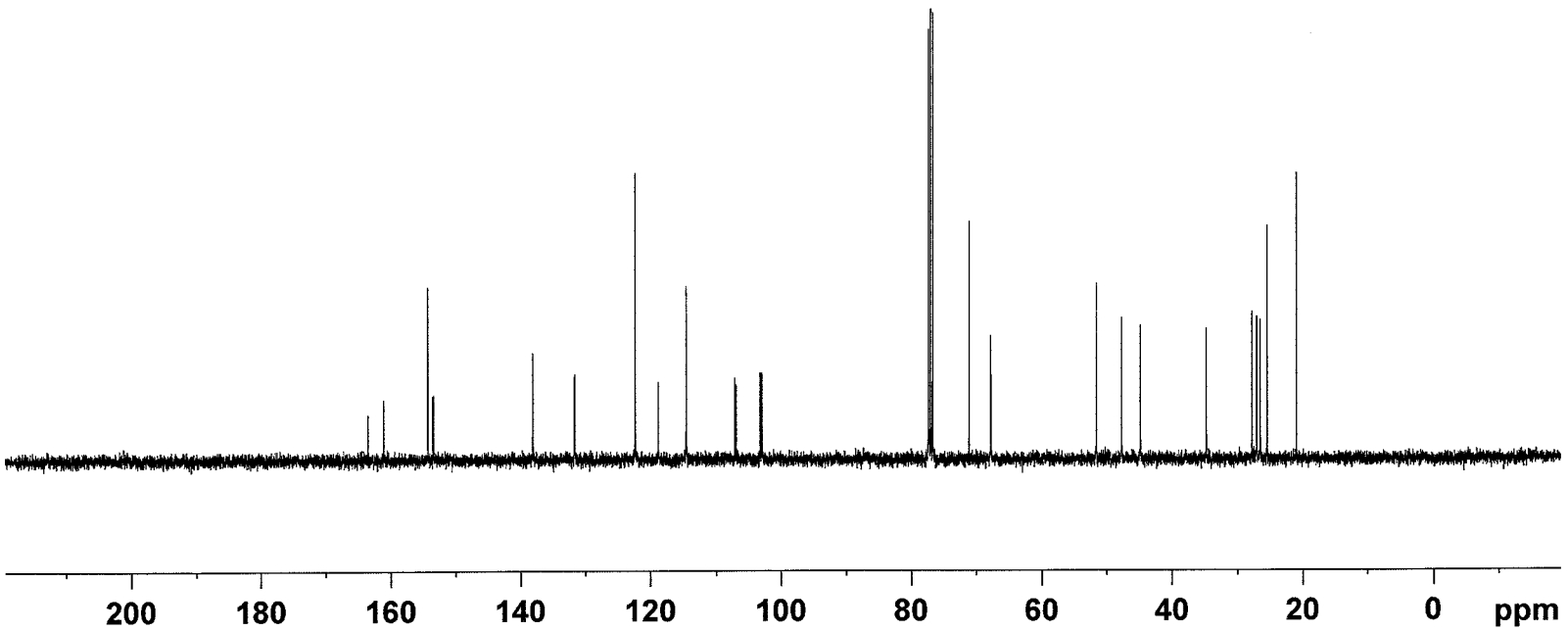
F2 - Processing parameters

SI	65536
SF	400.2100130 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

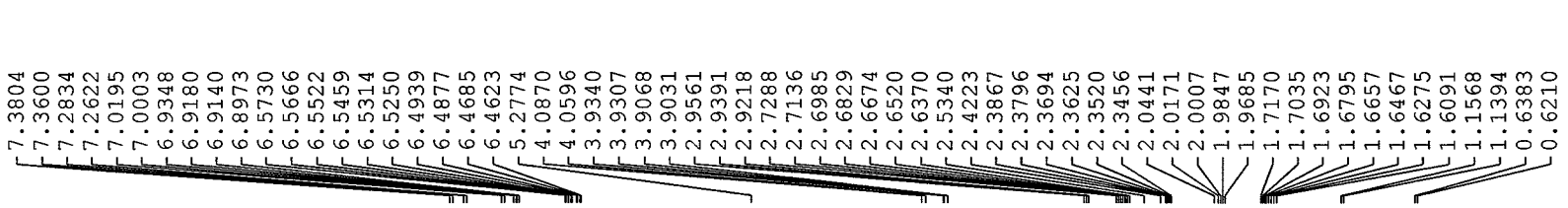


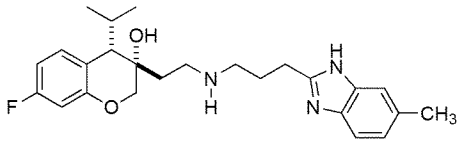
2aa



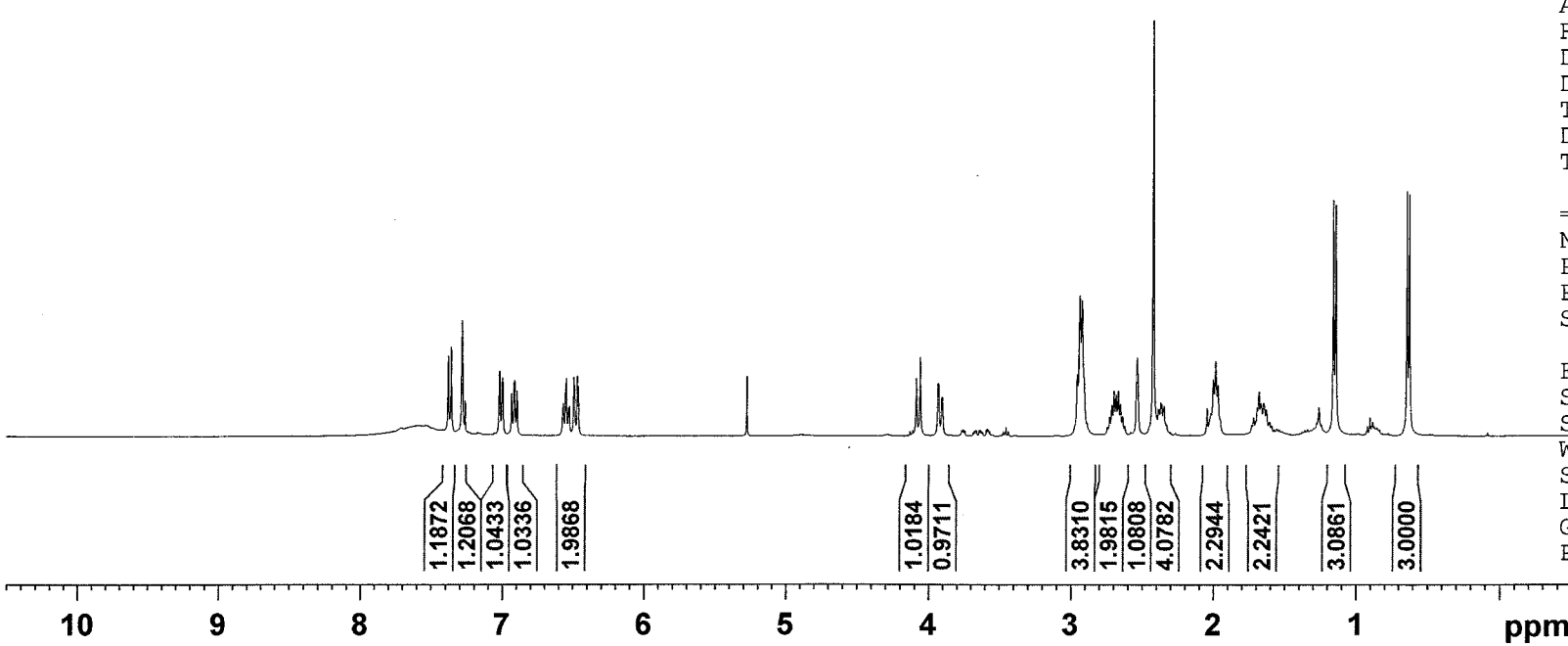



```
Current Data Parameters
NAME                  LJE-182
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20120531
Time                  20.42
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     100
DS                       4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                190.62
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                 10.00 usec
PLW1         52.00000000 W
SFO1         100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.33333001 W
PLW13         0.27000001 W
SFO2         400.2116008 MHz

F2 - Processing parameters
SI                   32768
SF           100.6328860 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



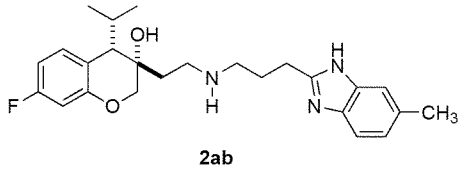


2ab

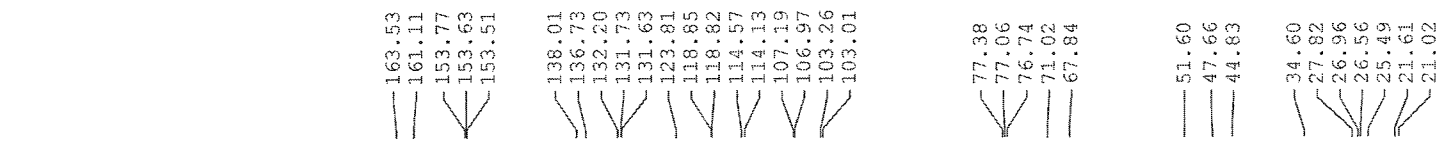


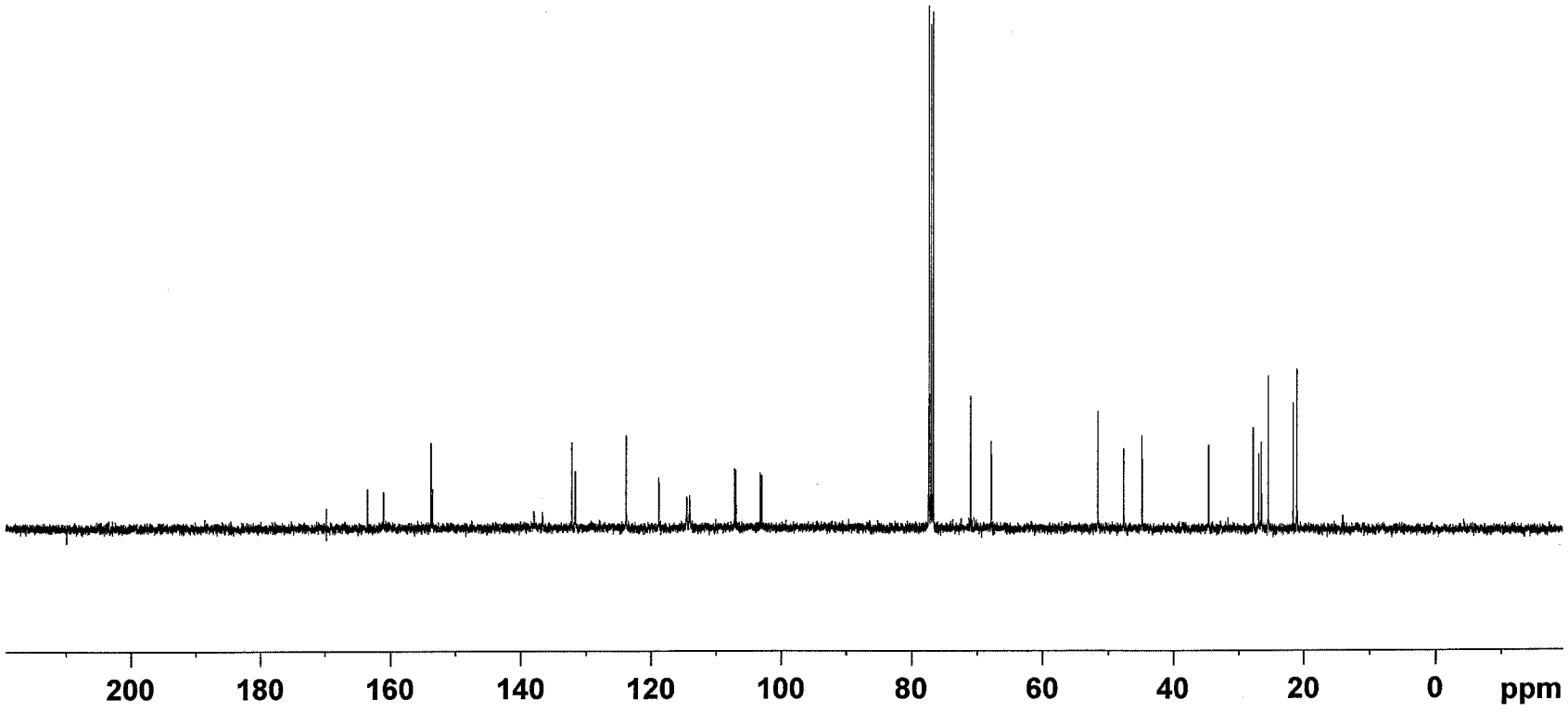
BRUKER

```
Current Data Parameters
NAME                  LJE-285
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130102
Time                  15.44
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        7
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     33.2
DW                   60.800 usec
DE                    6.50 usec
TE                   298.4 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1           12.00000000 W
SFO1           400.2124715 MHz

F2 - Processing parameters
SI                    65536
SF             400.2100118 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

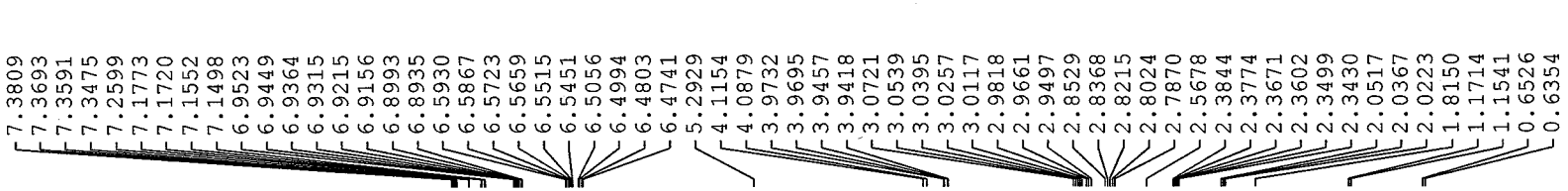


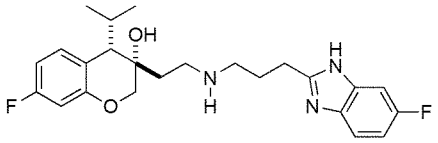
2ab



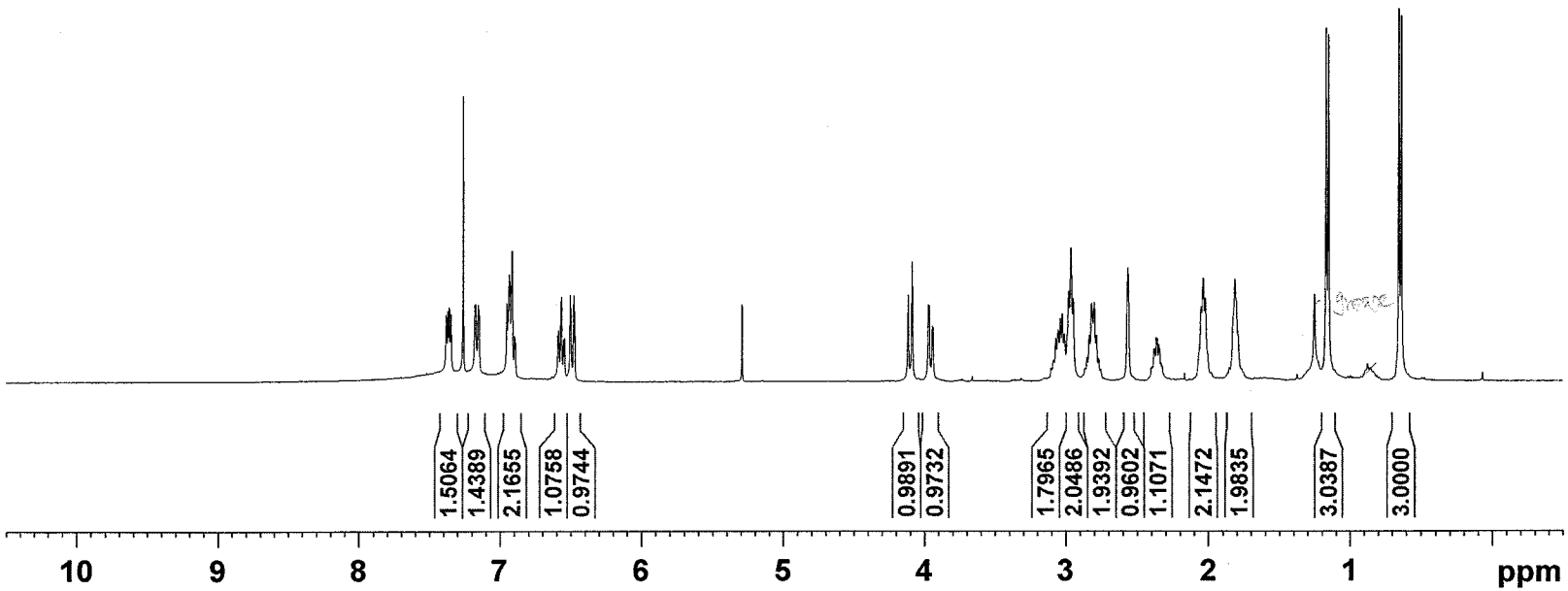



```
Current Data Parameters
NAME                  LJE-285
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130113
Time                  14.18
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      214
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                    190.62
DW                    20.800 usec
DE                     6.50 usec
TE                    295.6 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.00 usec
PLW1              52.00000000 W
SFO1             100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PLW2              12.00000000 W
PLW12              0.33333001 W
PLW13              0.27000001 W
SFO2             400.2116008 MHz

F2 - Processing parameters
SI                        32768
SF               100.6328860 MHz
WDW                          EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```





2ac



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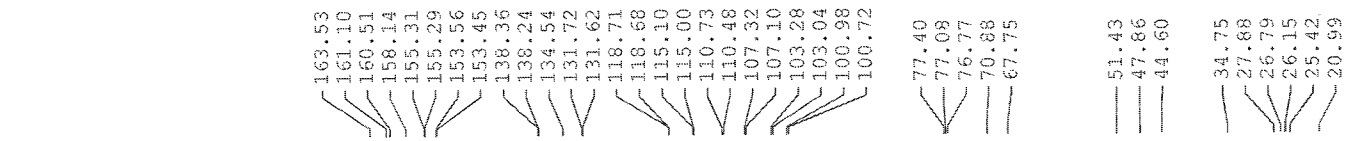
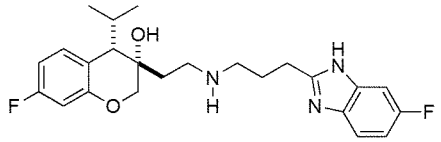
Current Data Parameters
NAME LJE-161
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120623
Time 17.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 119.61
DW 60.800 usec
DE 6.50 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1

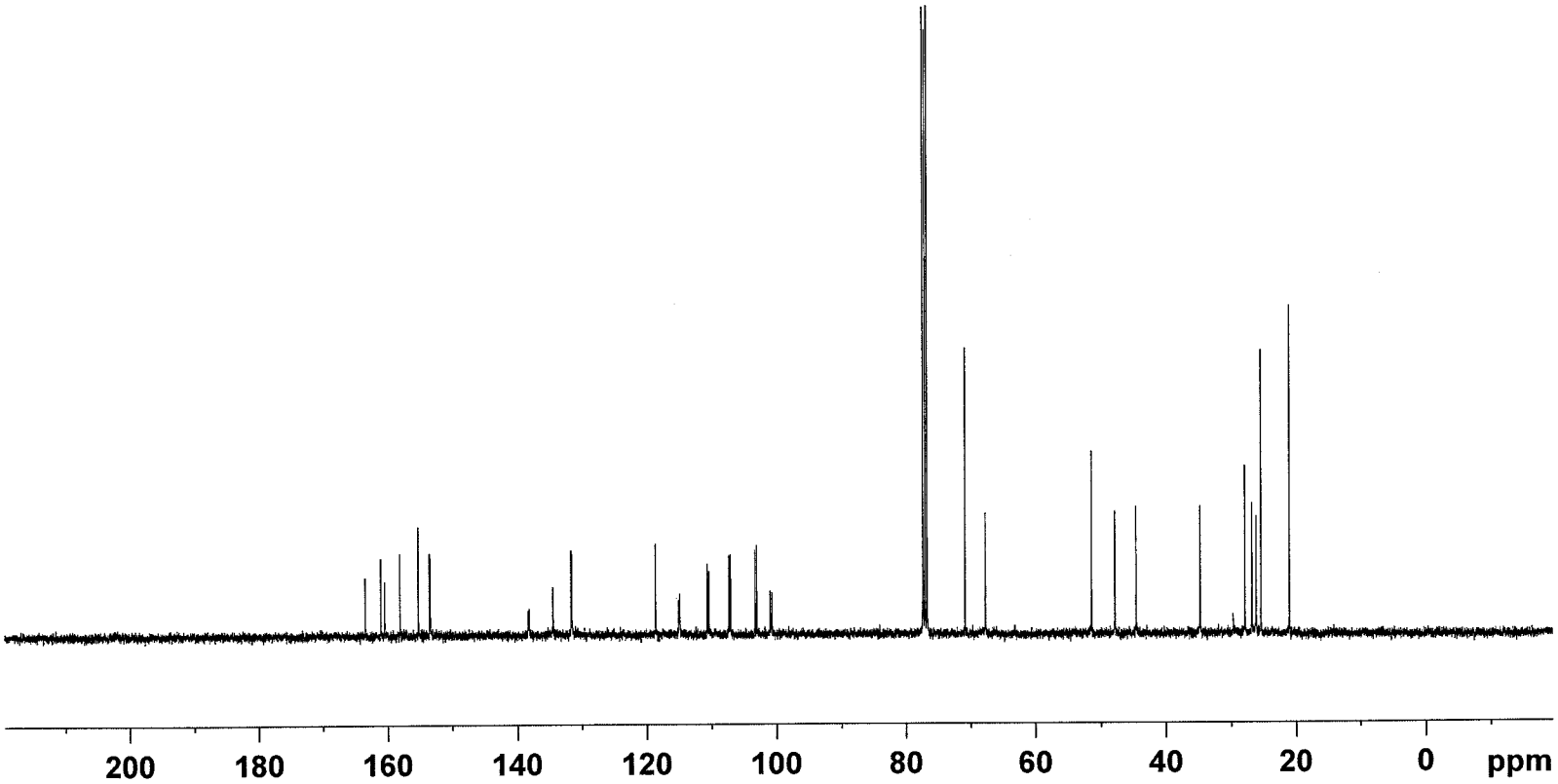
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





2ac



BRUKER

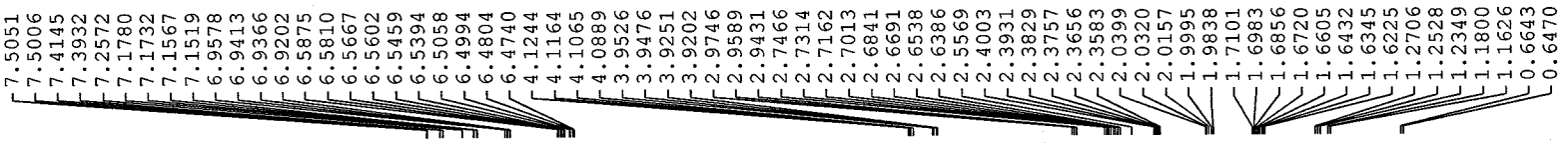
Current Data Parameters
NAME LJE-196
EXPNO 3
PROCNO 1

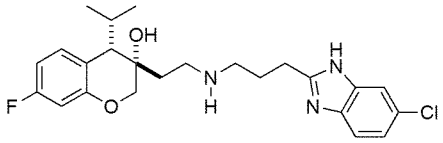
F2 - Acquisition Parameters
Date_ 20120709
Time 15.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 325
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

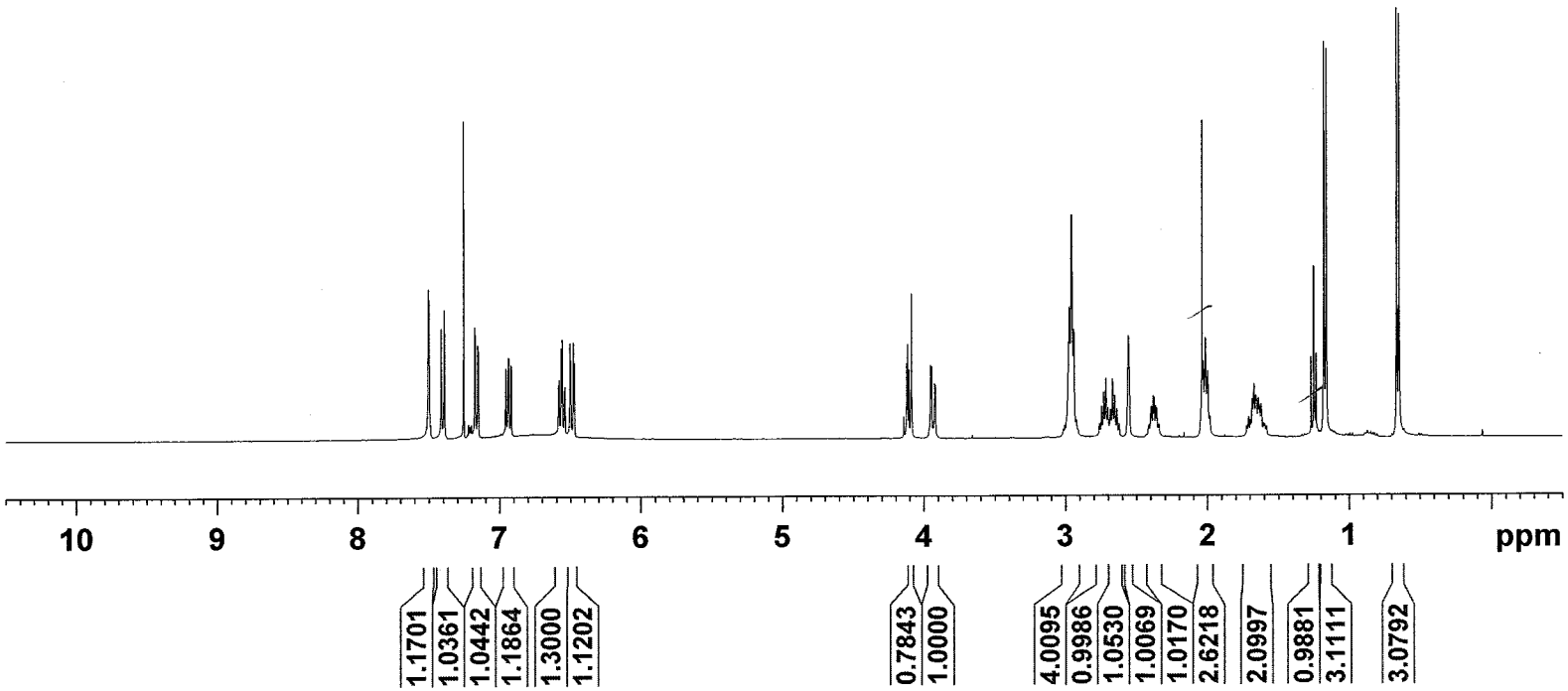
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2ad

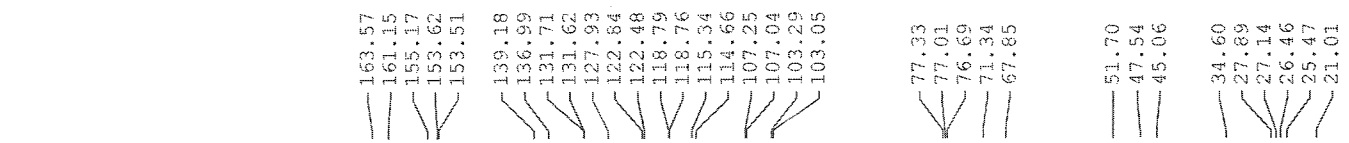


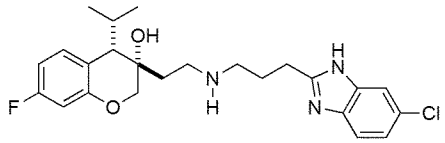
Current Data Parameters
NAME LJE-175
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120514
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.91
DW 60.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1

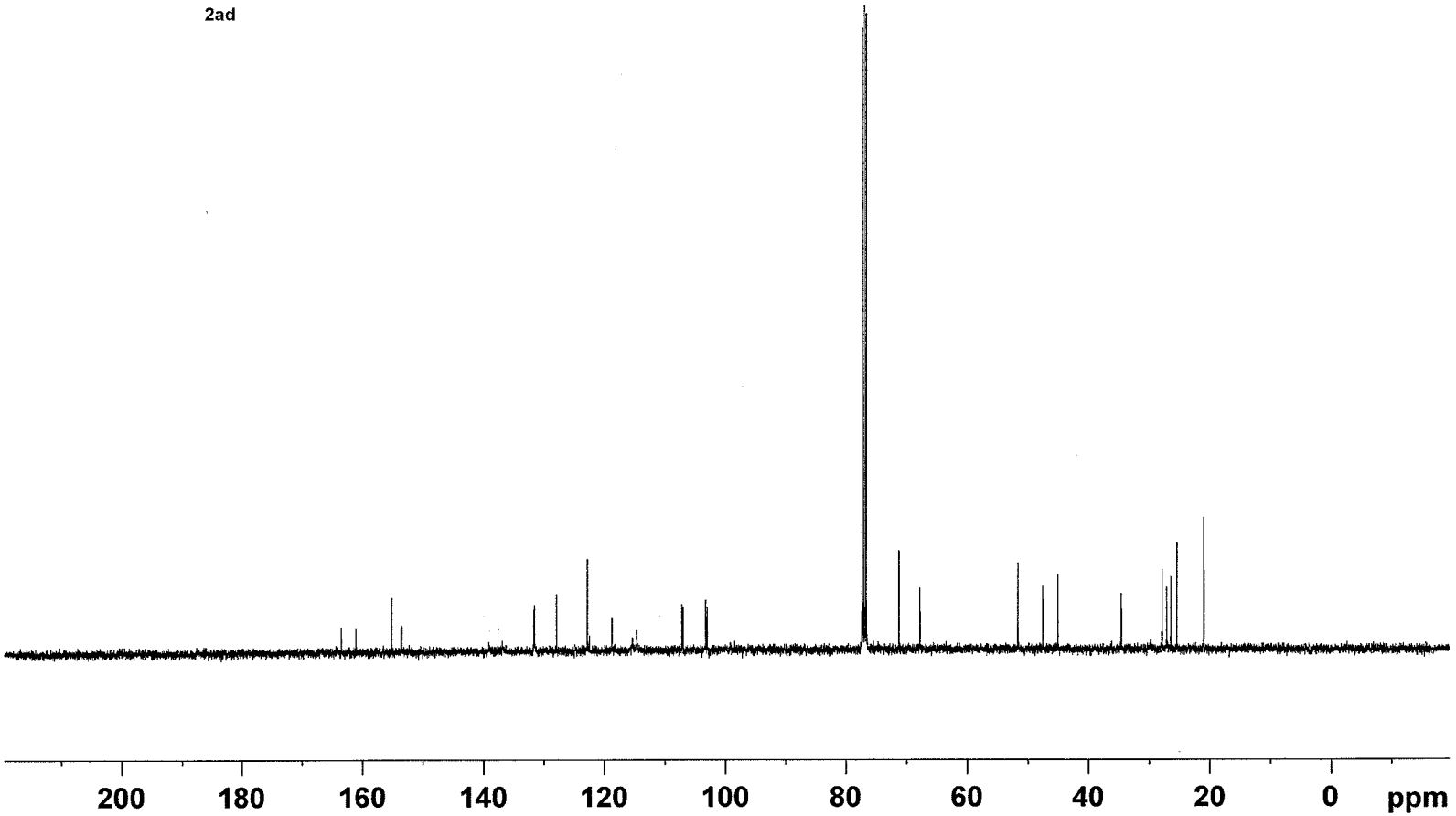
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





2ad



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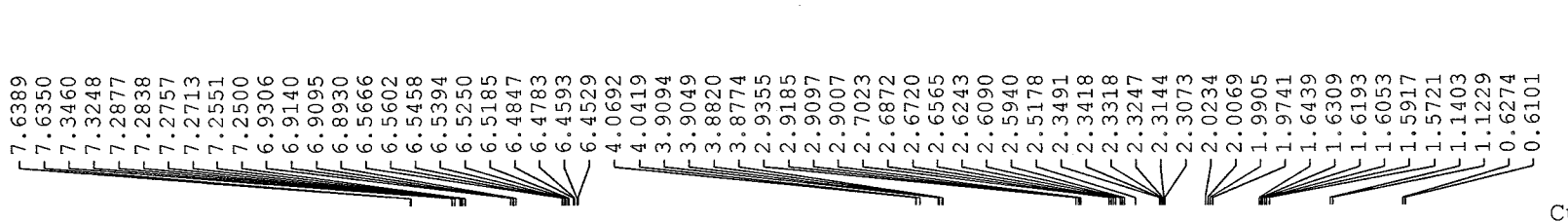
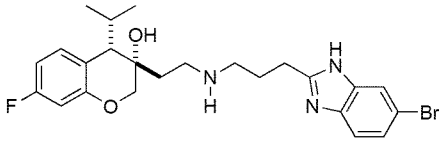
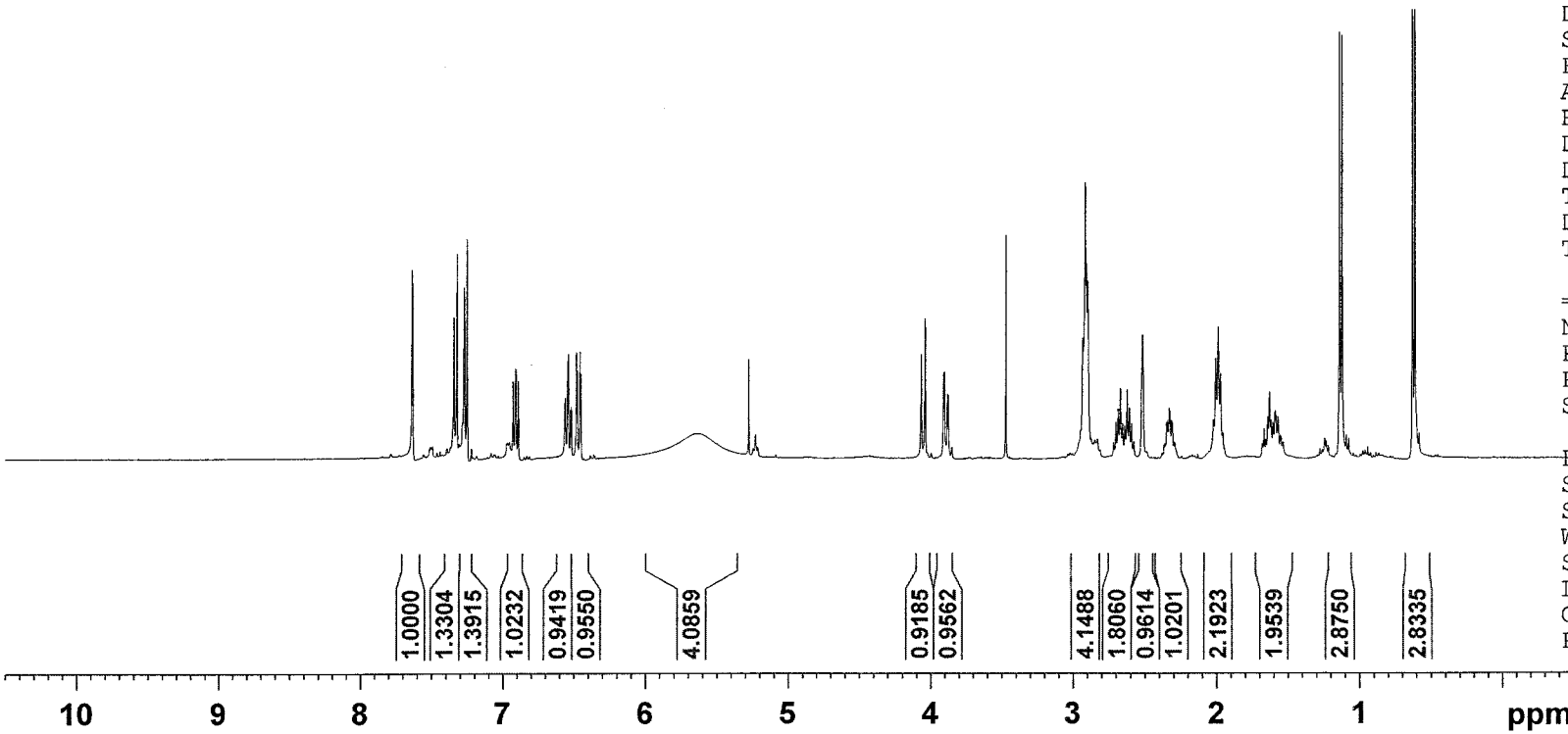
```
Current Data Parameters
NAME                  LJE-175
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_                20120623
Time                    16.31
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         495
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                     190.62
DW                     20.800 usec
DE                      6.50 usec
TE                     304.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PLW1              52.00000000 W
SFO1              100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PLW2              12.00000000 W
PLW12              0.33333001 W
PLW13              0.27000001 W
SFO2              400.2116008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6328860 MHz
WDW                         EM
SSB                0
LB                      1.00 Hz
GB                 0
PC                      1.40
```





2ae



Current Data Parameters

NAME	LJE-325
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

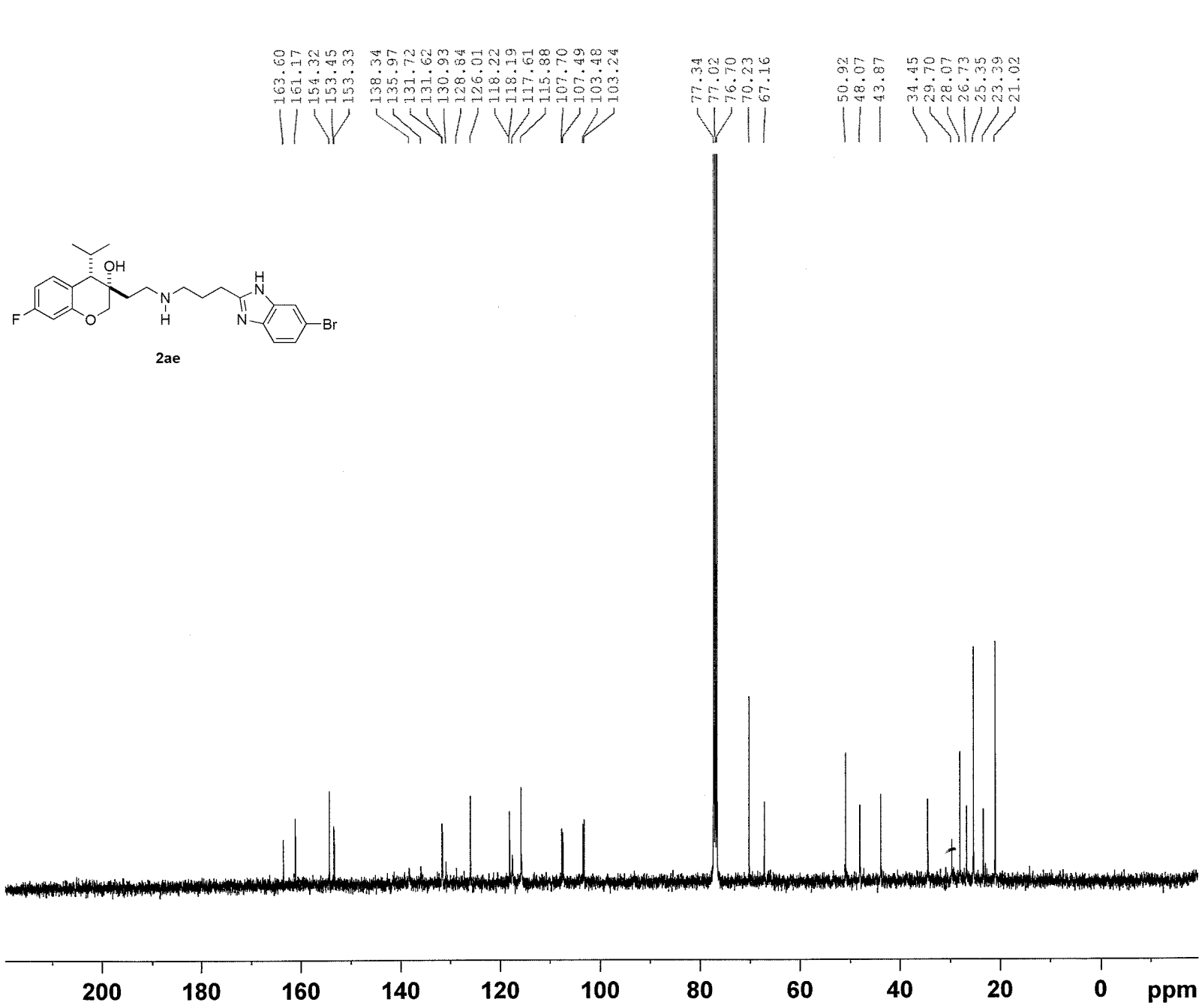
Date_	20130329
Time	15.24
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	69.69
DW	60.800 usec
DE	6.50 usec
TE	298.5 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	15.00 usec
PLW1	12.00000000 W
SFO1	400.2124715 MHz

F2 - Processing parameters

SI	65536
SF	400.2100148 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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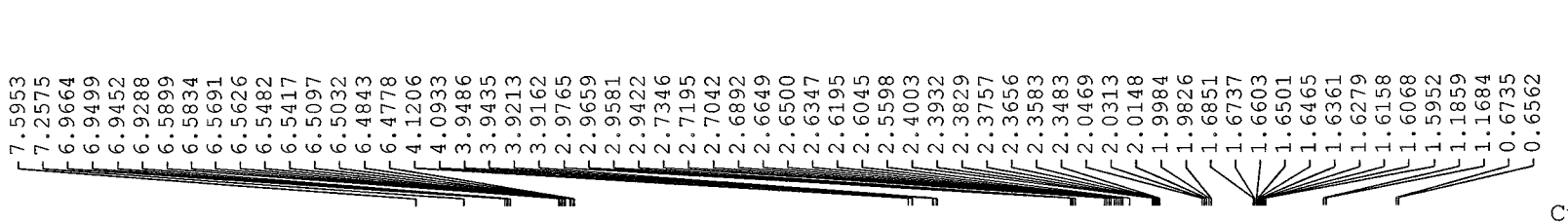
Current Data Parameters
NAME LJE-195
EXPNO 5
PROCNO 1

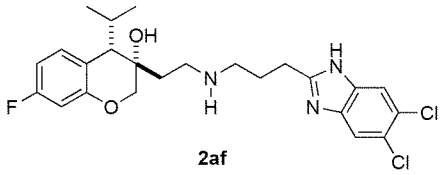
F2 - Acquisition Parameters
Date_ 20120715
Time 16.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2803
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

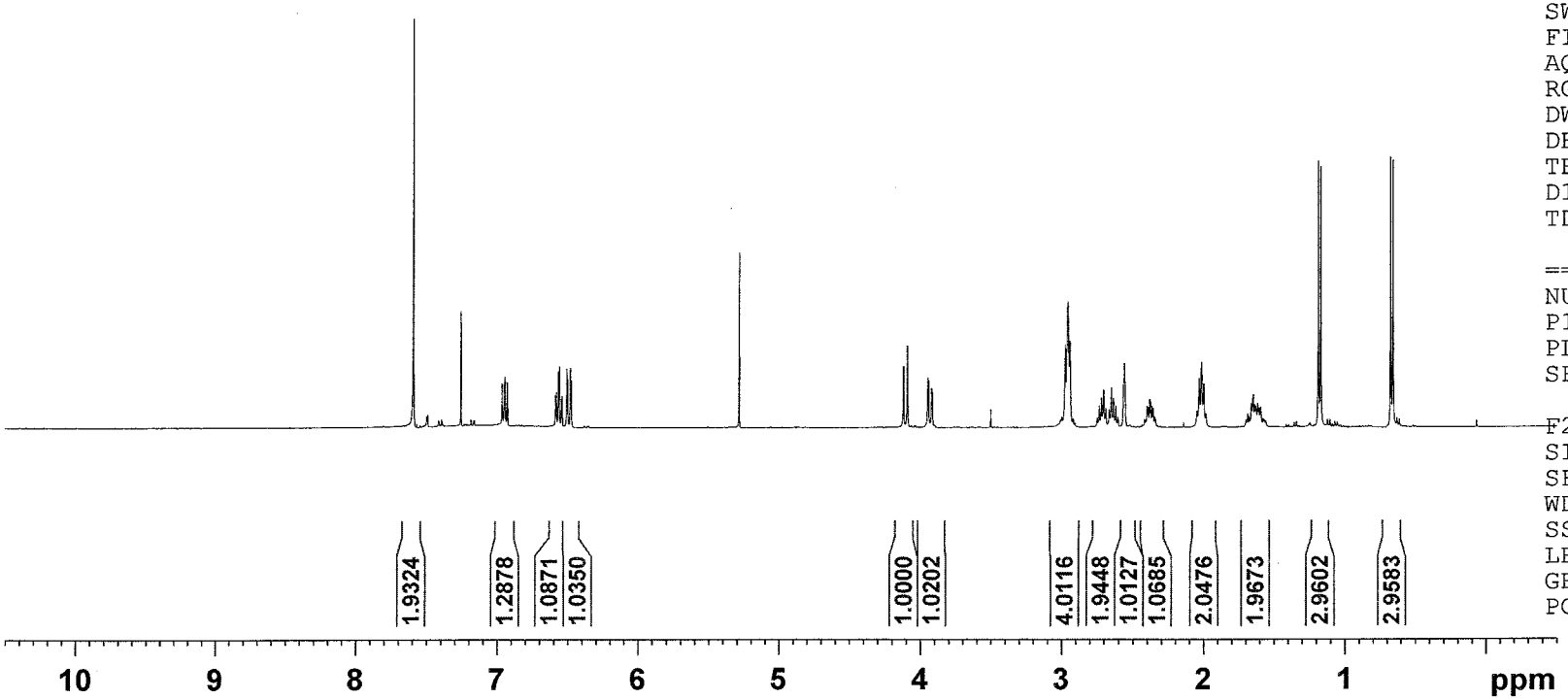
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2af



Current Data Parameters
NAME LJE-325
EXPNO 4
PROCNO 1

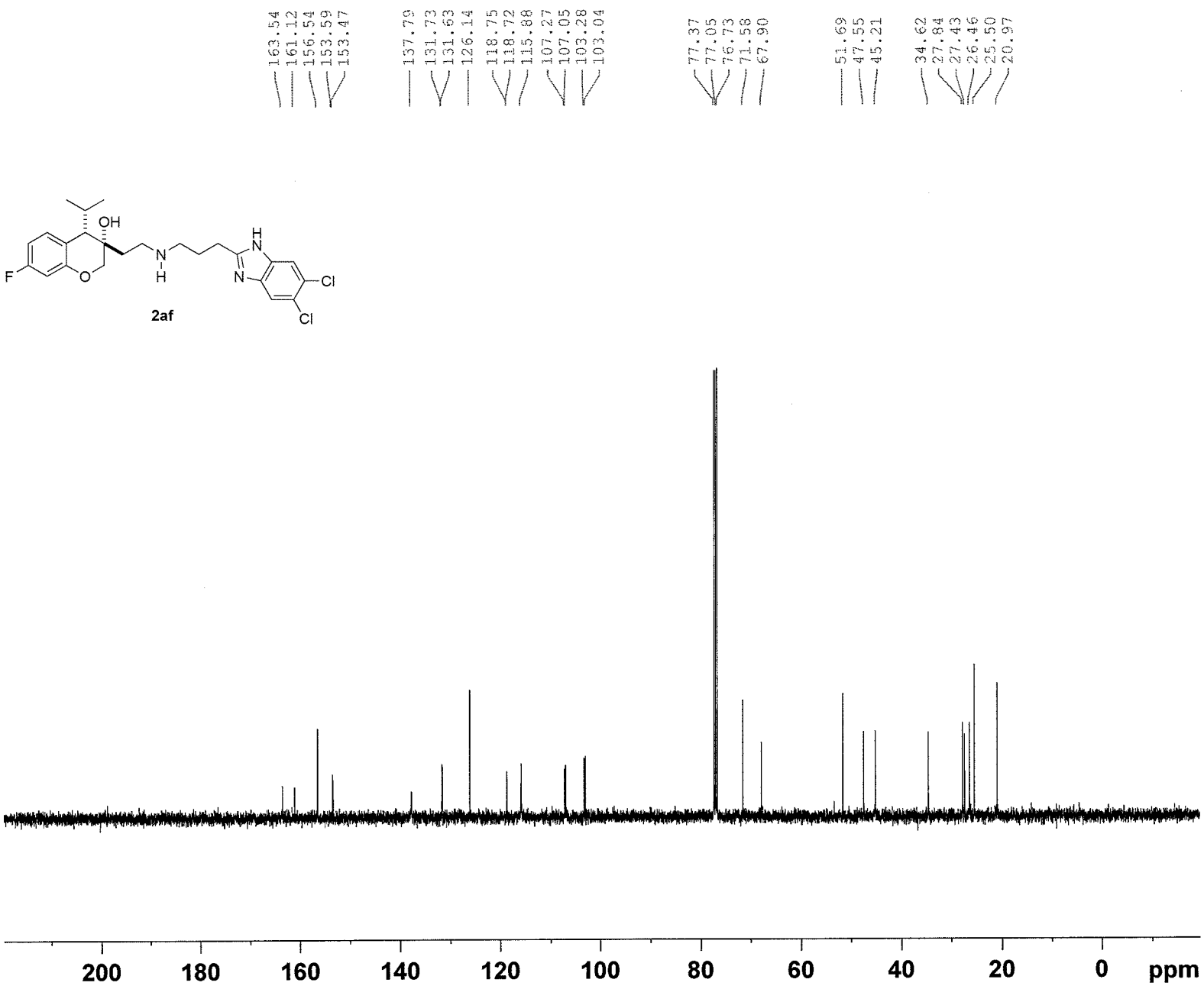
F2 - Acquisition Parameters
Date_ 20130329
Time_ 15.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 84.76
DW 60.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S100

2af



Current Data Parameters

NAME	LJE-183
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20120601
Time	17.14
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	75
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	190.62
DW	20.800 usec
DE	6.50 usec
TE	299.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

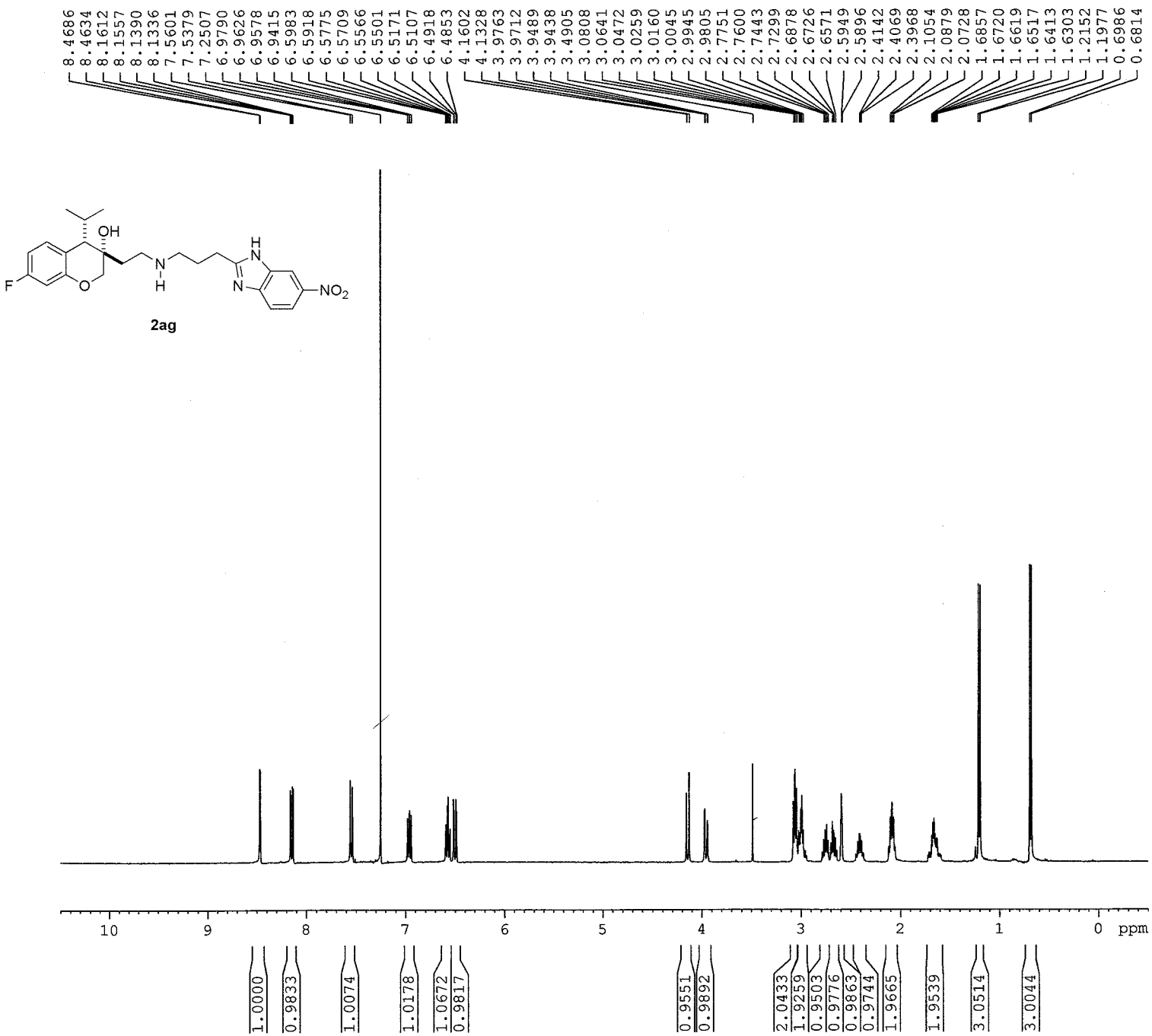
NUC1	13C
P1	10.00 usec
PLW1	52.00000000 W
SFO1	100.6429474 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PLW2	12.00000000 W
PLW12	0.33333001 W
PLW13	0.27000001 W
SFO2	400.2116008 MHz

F2 - Processing parameters

SI	32768
SF	100.6328860 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

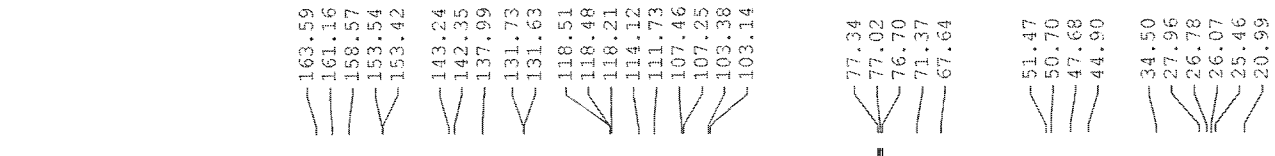


Current Data Parameters
NAME LJE-173
EXPNO 1
PROCNO 1

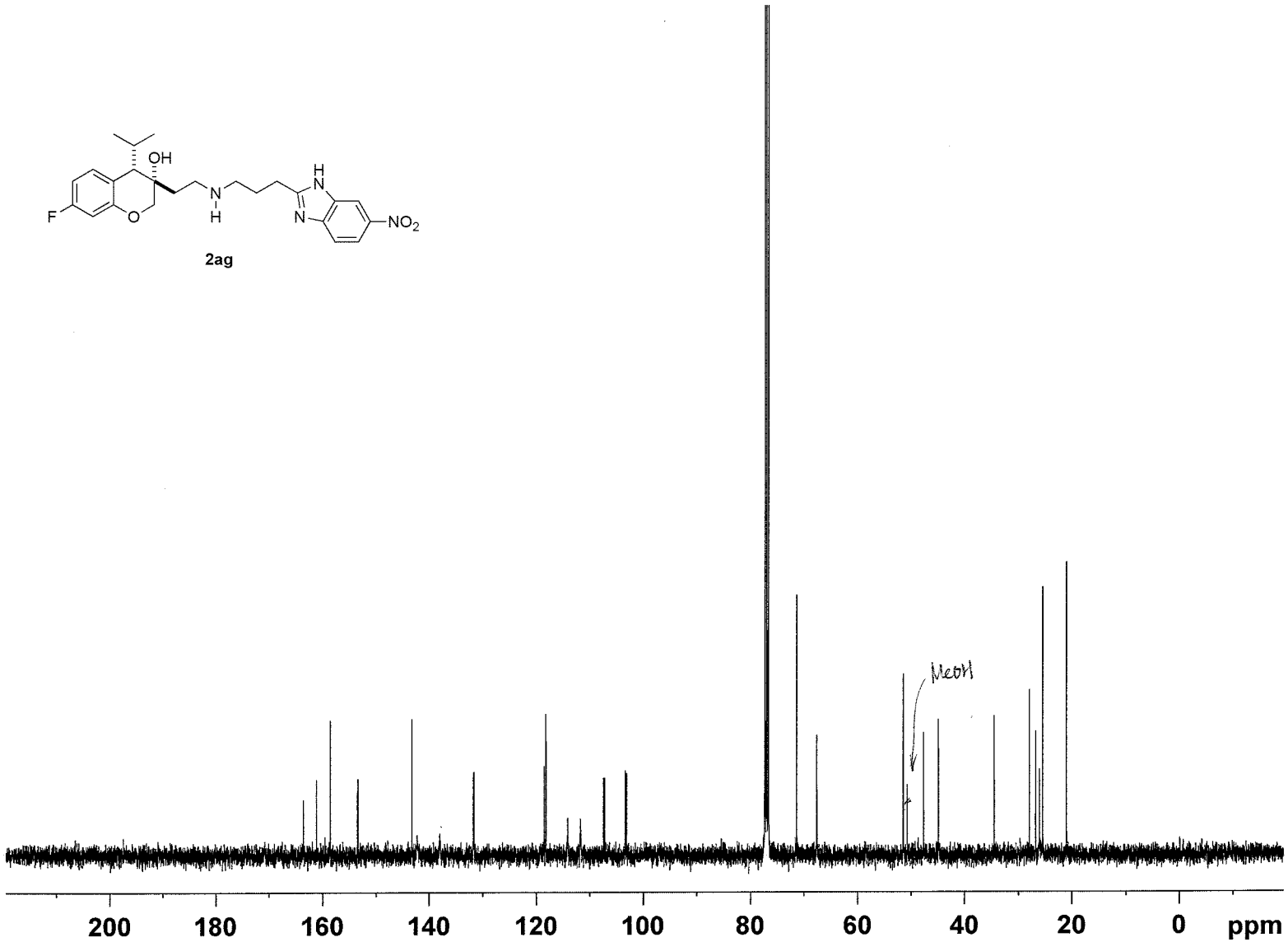
F2 - Acquisition Parameters
Date_ 20120420
Time 21.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 645.1
DW 60.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 4.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300136 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2ag



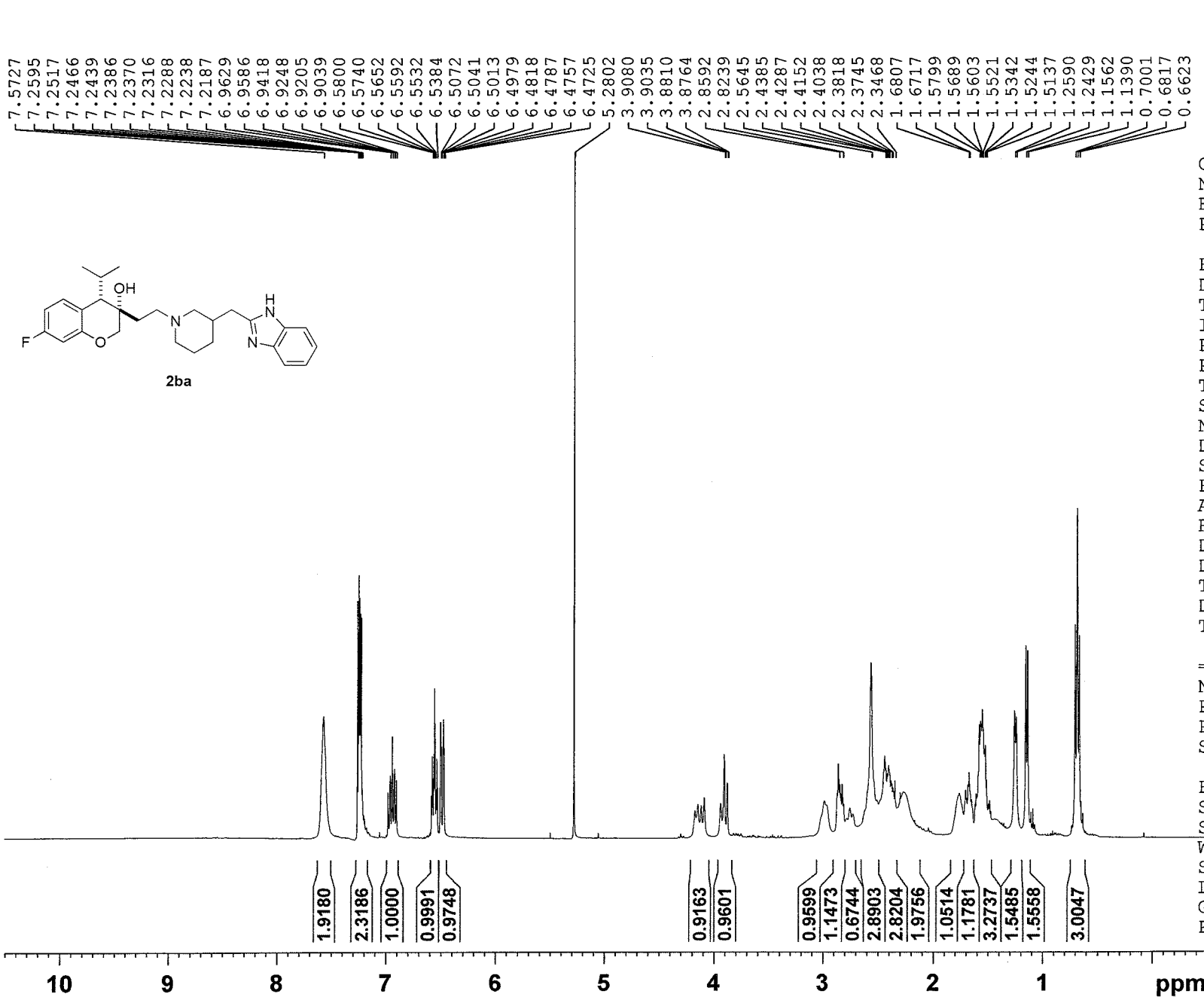

```
Current Data Parameters
NAME                  LJE-173
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20120521
Time                  14.45
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                    190.62
DW                  20.800 usec
DE                   6.50 usec
TE                   298.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PLW1             52.00000000 W
SFO1            100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 90.00 usec
PLW2             12.00000000 W
PLW12             0.33333001 W
PLW13             0.27000001 W
SFO2            400.2116008 MHz

F2 - Processing parameters
SI                        32768
SF              100.6328860 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```



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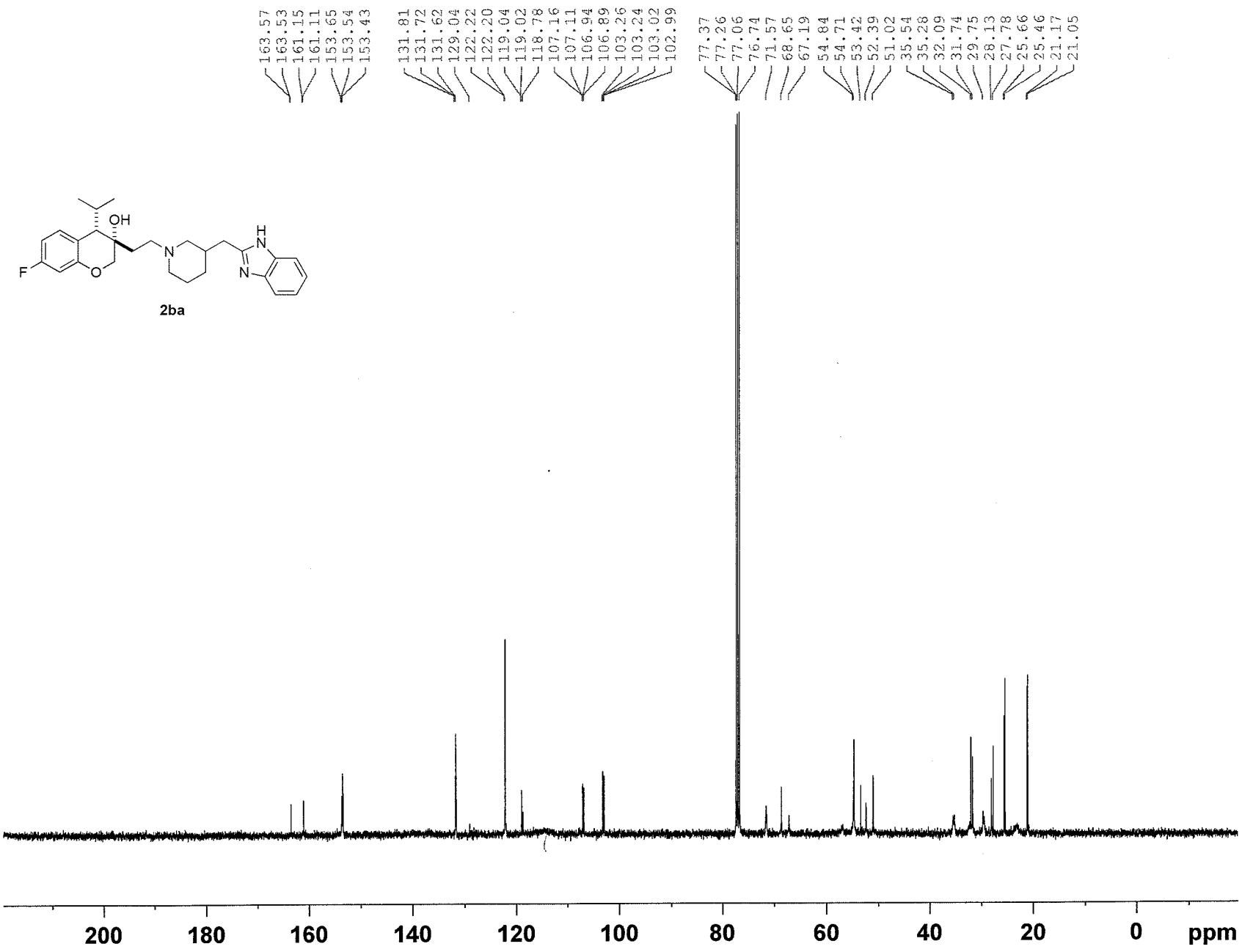
Current Data Parameters
NAME KTH-m-198
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121221
Time 15.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 58.64
DW 60.800 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100132 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



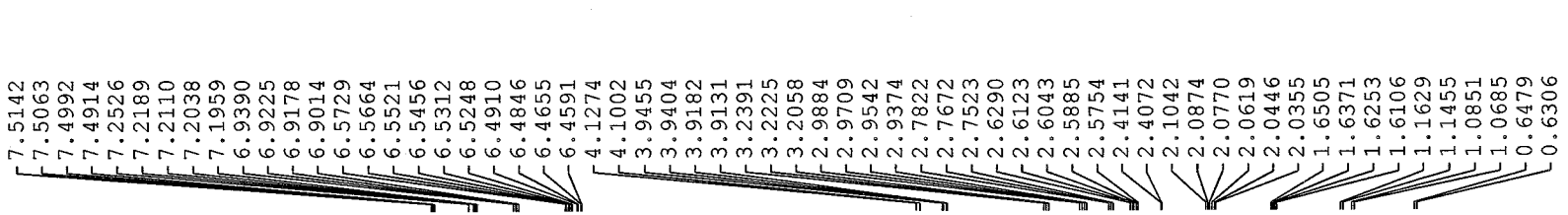
Current Data Parameters
NAME KTH-m-198
EXPNO 7
PROCNO 1

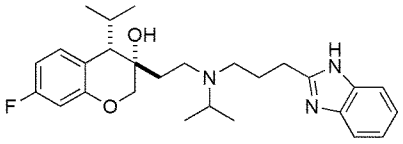
F2 - Acquisition Parameters
Date_ 20121221
Time 19.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 592
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

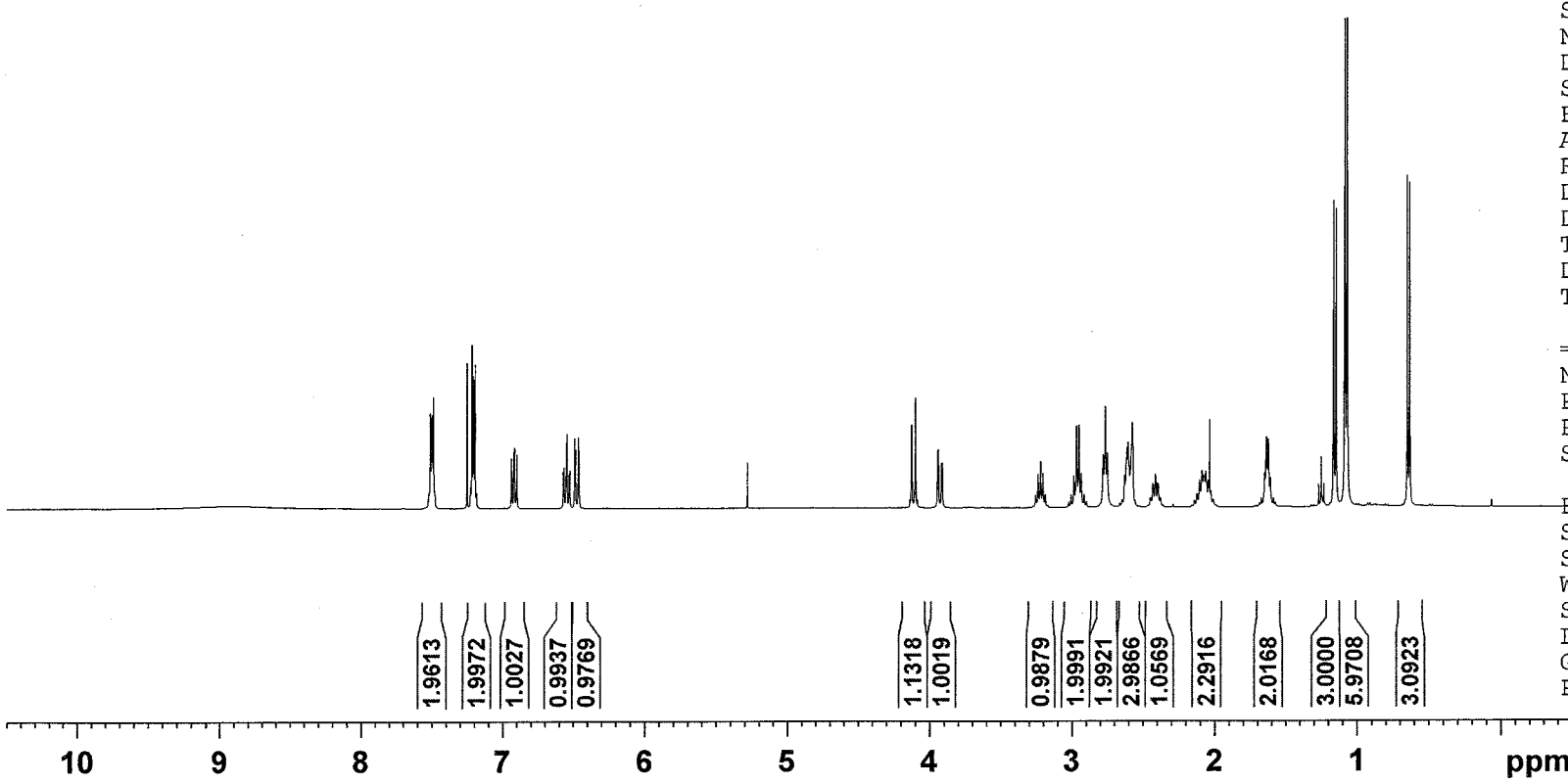
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2ca



Current Data Parameters

NAME	LJE-328
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

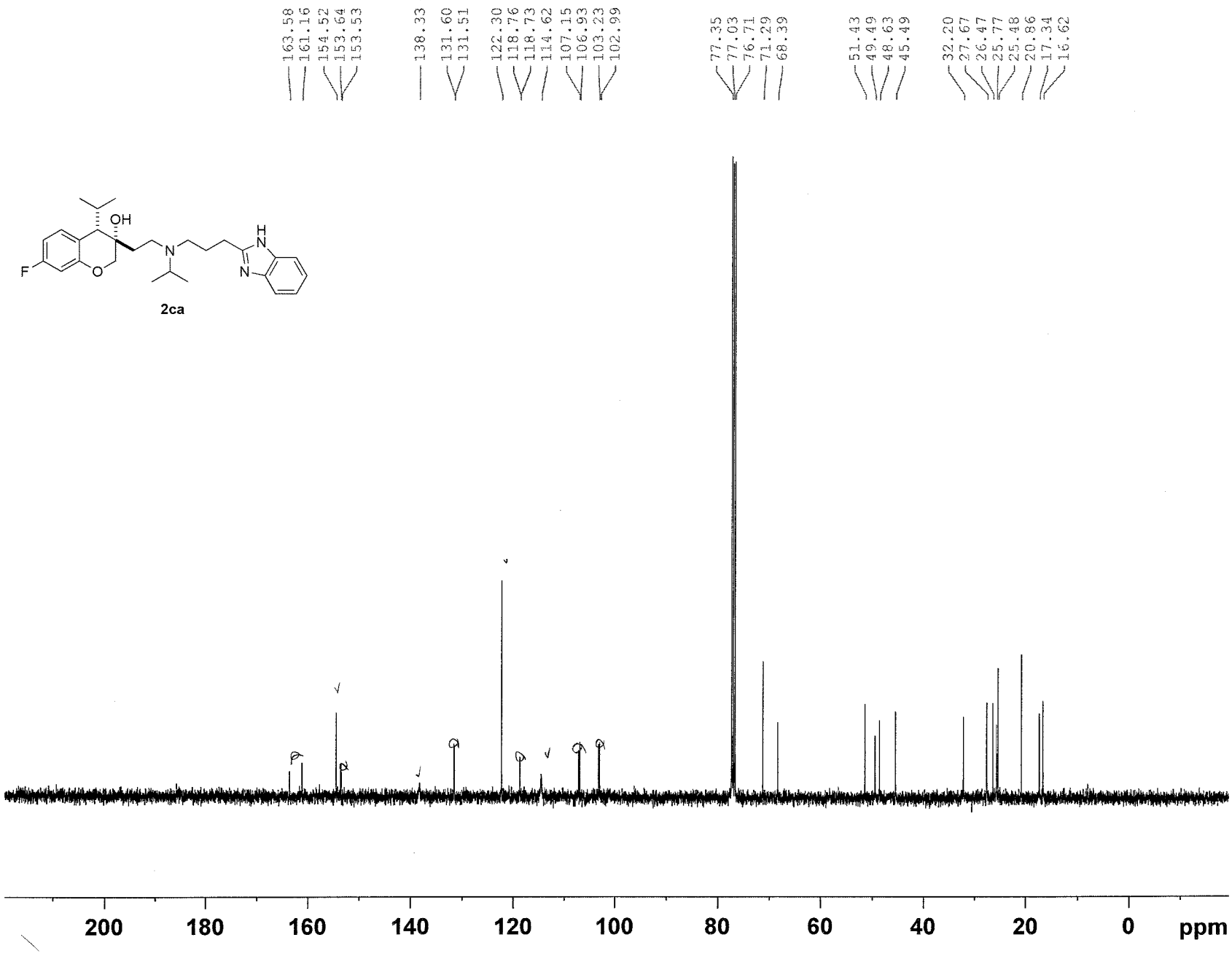
Date_	20130328
Time	19.03
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	84.76
DW	60.800 usec
DE	6.50 usec
TE	298.7 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	15.00 usec
PLW1	12.00000000 W
SFO1	400.2124715 MHz

F2 - Processing parameters

SI	65536
SF	400.2100157 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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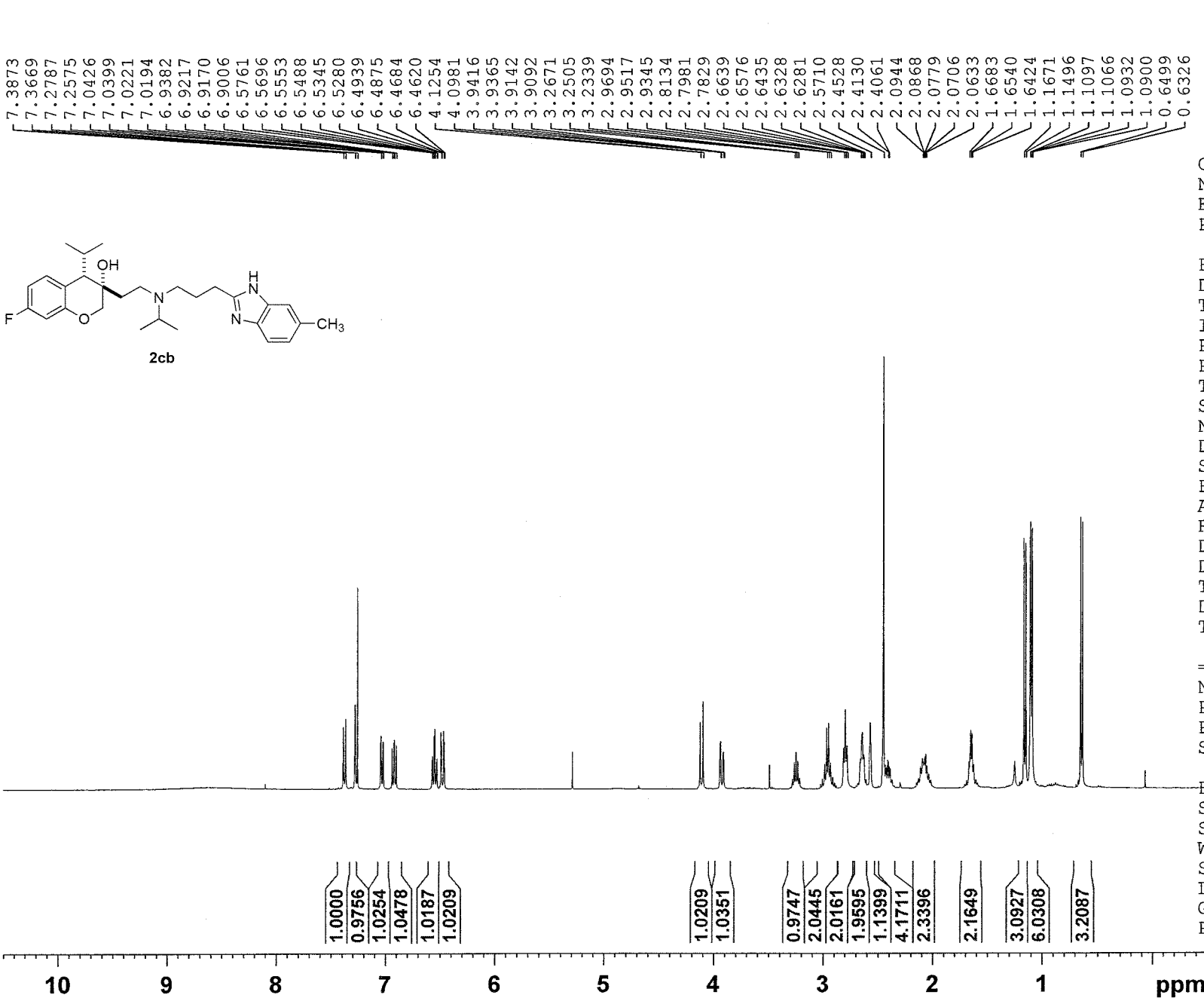
Current Data Parameters
NAME LJE-328
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130328
Time 19.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 133
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



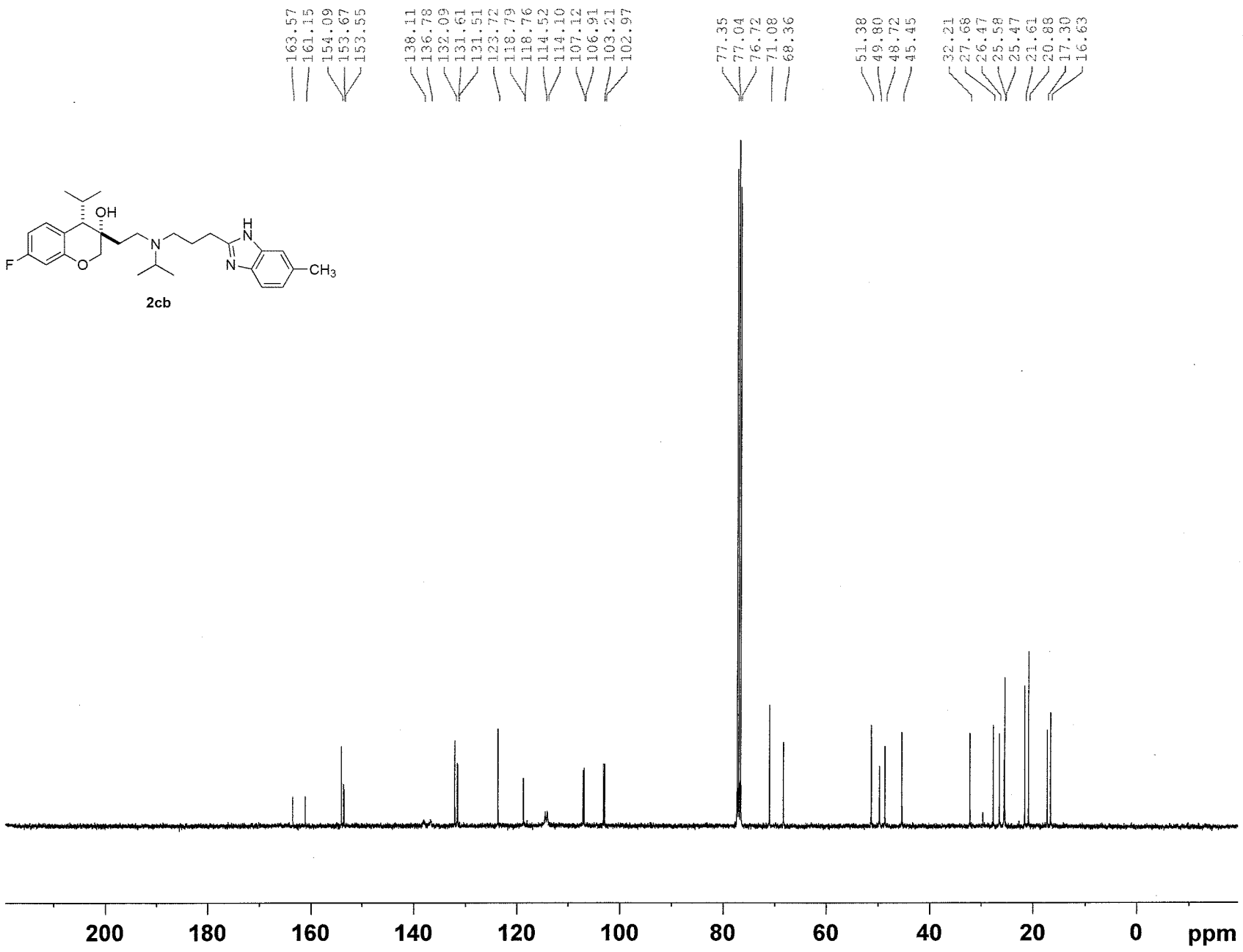
BRUKER

Current Data Parameters
NAME LJE-328
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130329
Time_ 15.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 103.95
DW 60.800 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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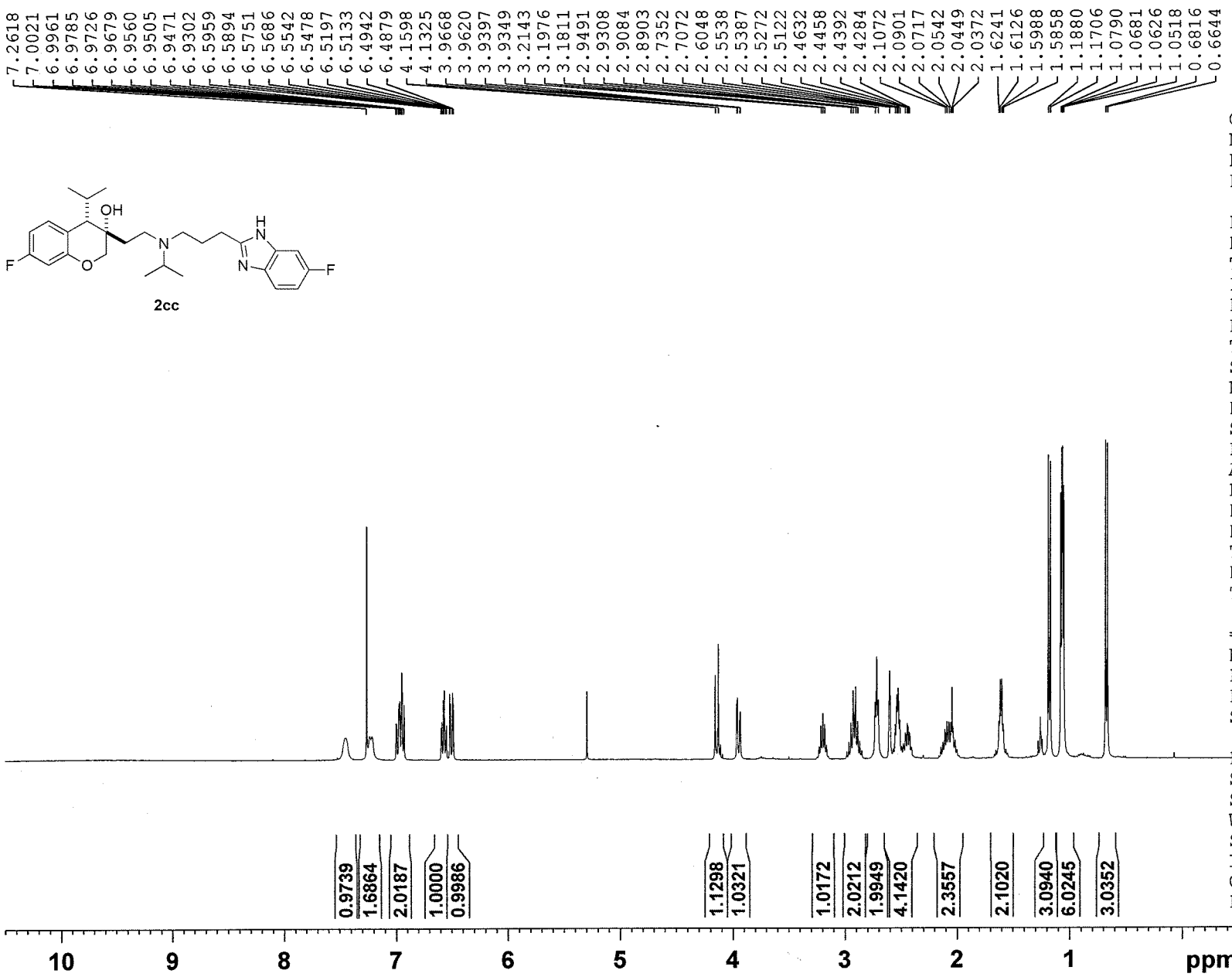
```
Current Data Parameters
NAME                  LJE-328
EXPNO                       7
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130401
Time                   16.56
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1308
DS                          4
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                     190.62
DW                     20.800 usec
DE                      6.50 usec
TE                     299.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PLW1              52.00000000 W
SFO1             100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.33333001 W
PLW13              0.27000001 W
SFO2             400.2116008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6328860 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```



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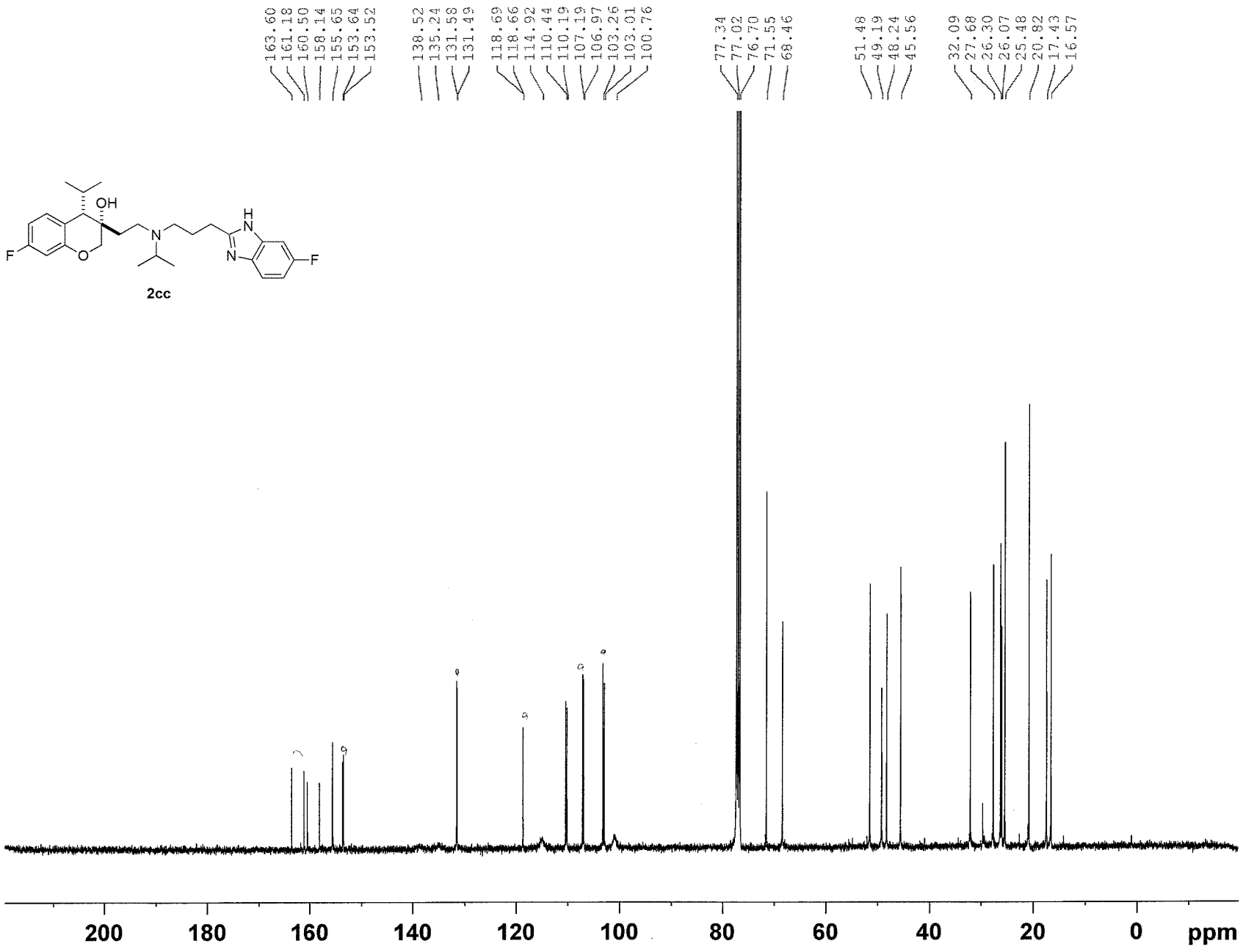
Current Data Parameters
NAME LJE-289
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130313
Time 19.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 133.17
DW 60.800 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

2cc



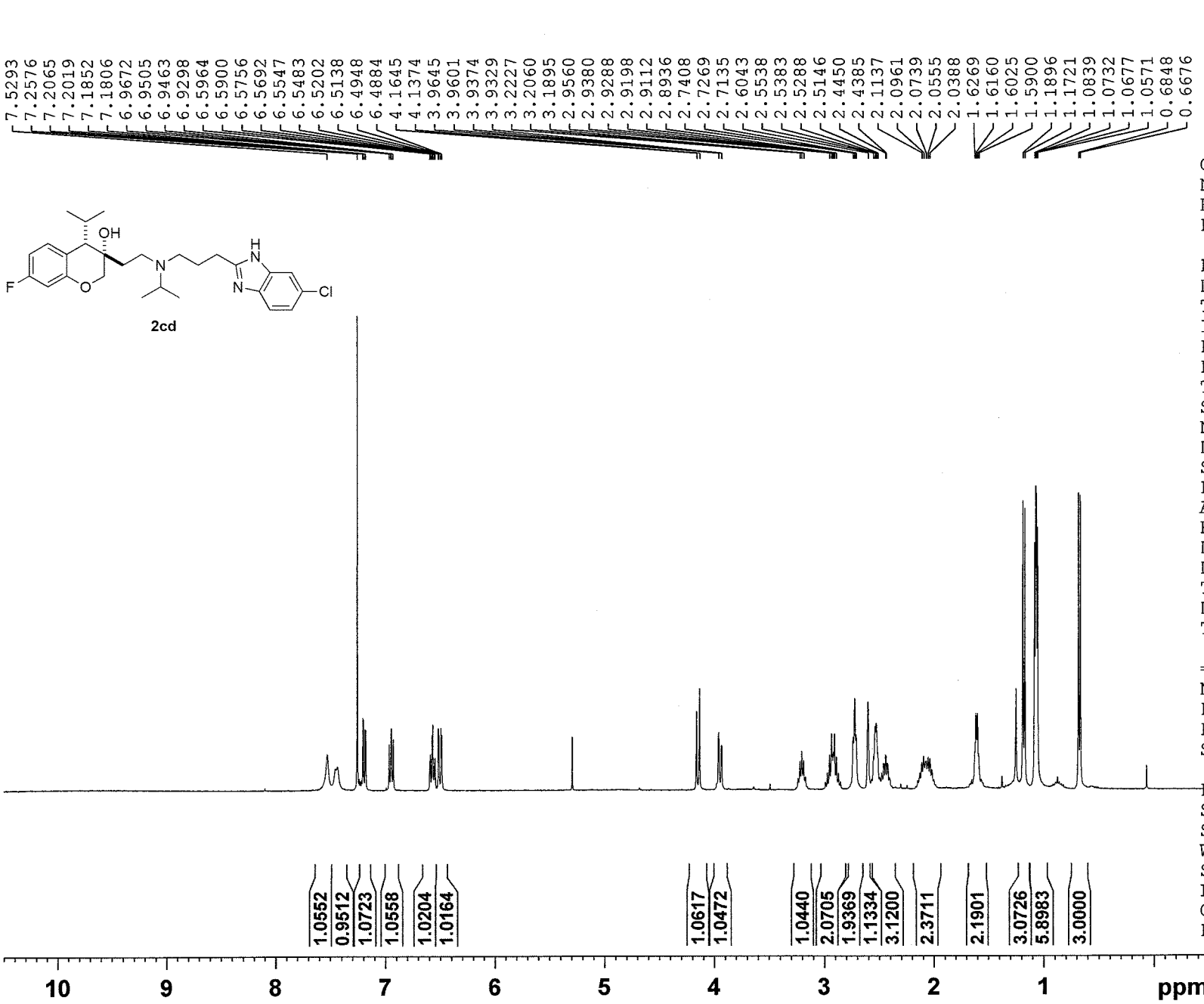

```
Current Data Parameters
NAME                  LJE-289
EXPNO                       8
PROCNO                      1

F2 - Acquisition Parameters
Date_                20130409
Time                    21.59
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                      11638
DS                          4
SWH                24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                        190.62
DW                        20.800 usec
DE                         6.50 usec
TE                       298.5 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.00 usec
PLW1               52.00000000 W
SFO1              100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 usec
PLW2               12.00000000 W
PLW12               0.33333001 W
PLW13               0.27000001 W
SFO2              400.2116008 MHz

F2 - Processing parameters
SI                         32768
SF                100.6328860 MHz
WDW                           EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

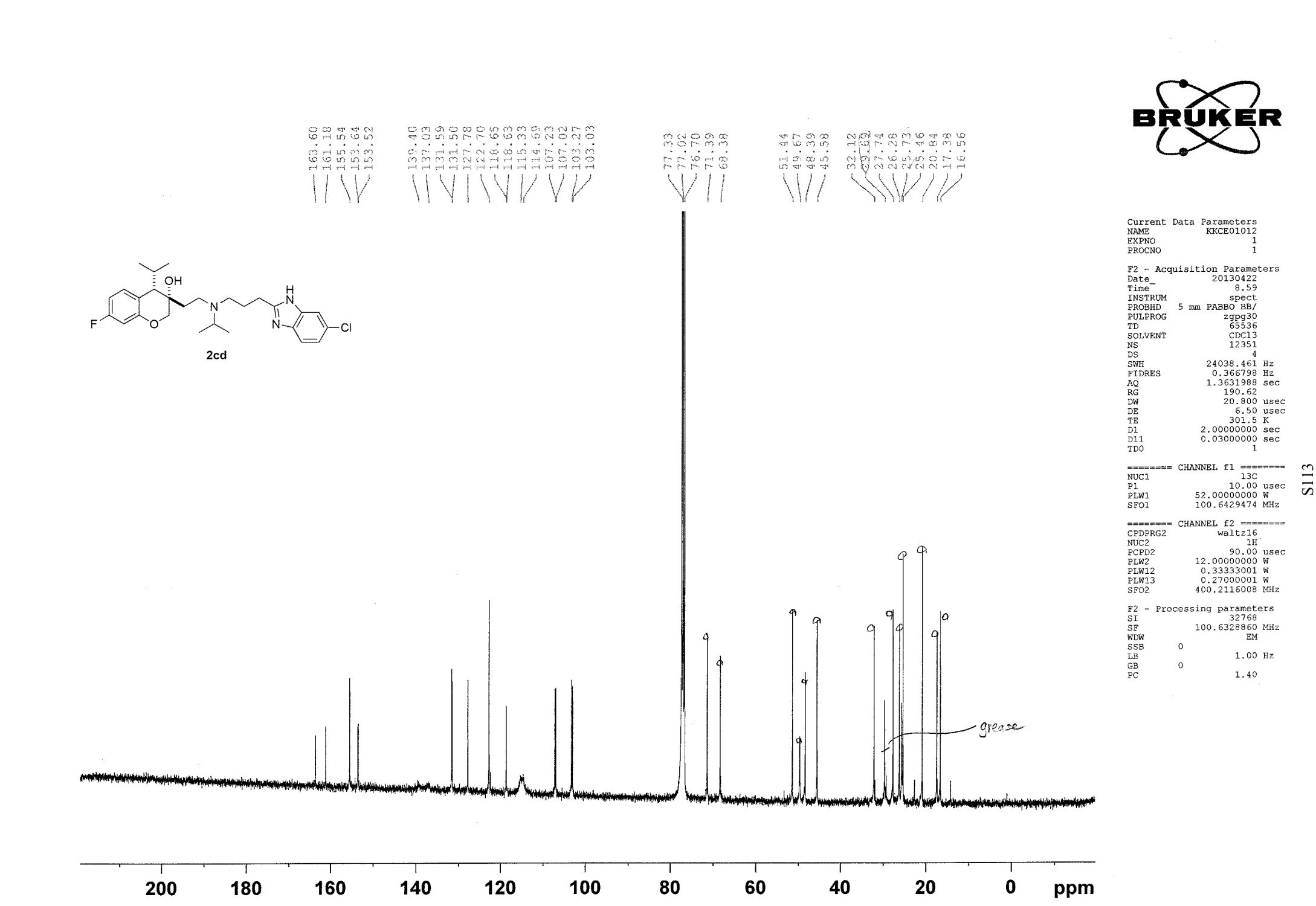


Current Data Parameters
NAME LJE-289
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130314
Time 15.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 171.62
DW 60.800 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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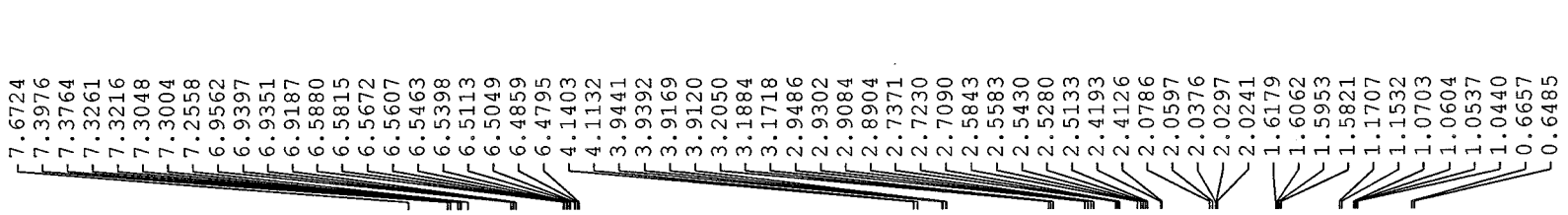
Current Data Parameters
NAME KKCE01012
EXPNO 1
PROCNO 1

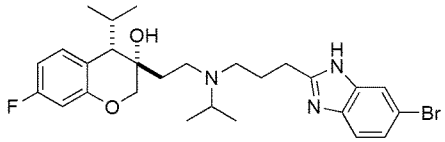
F2 - Acquisition Parameters
Date_ 20130422
Time 8.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12351
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 301.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

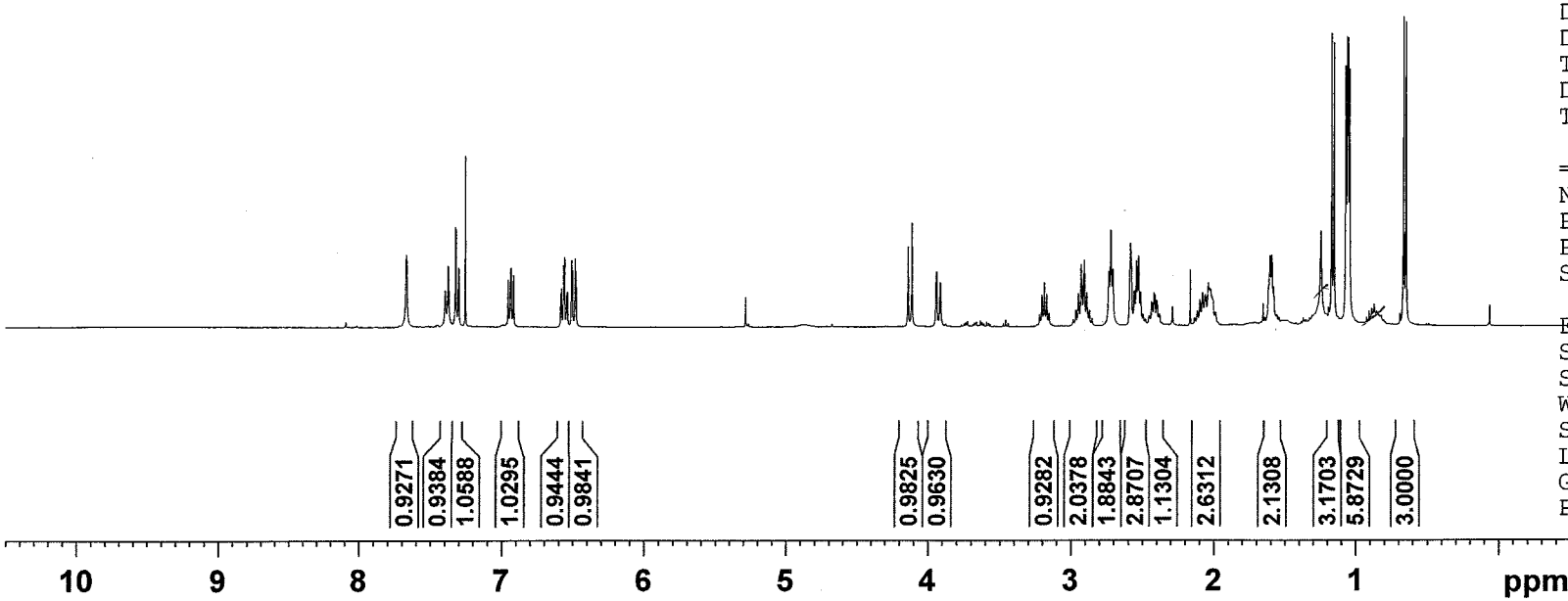
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2ce



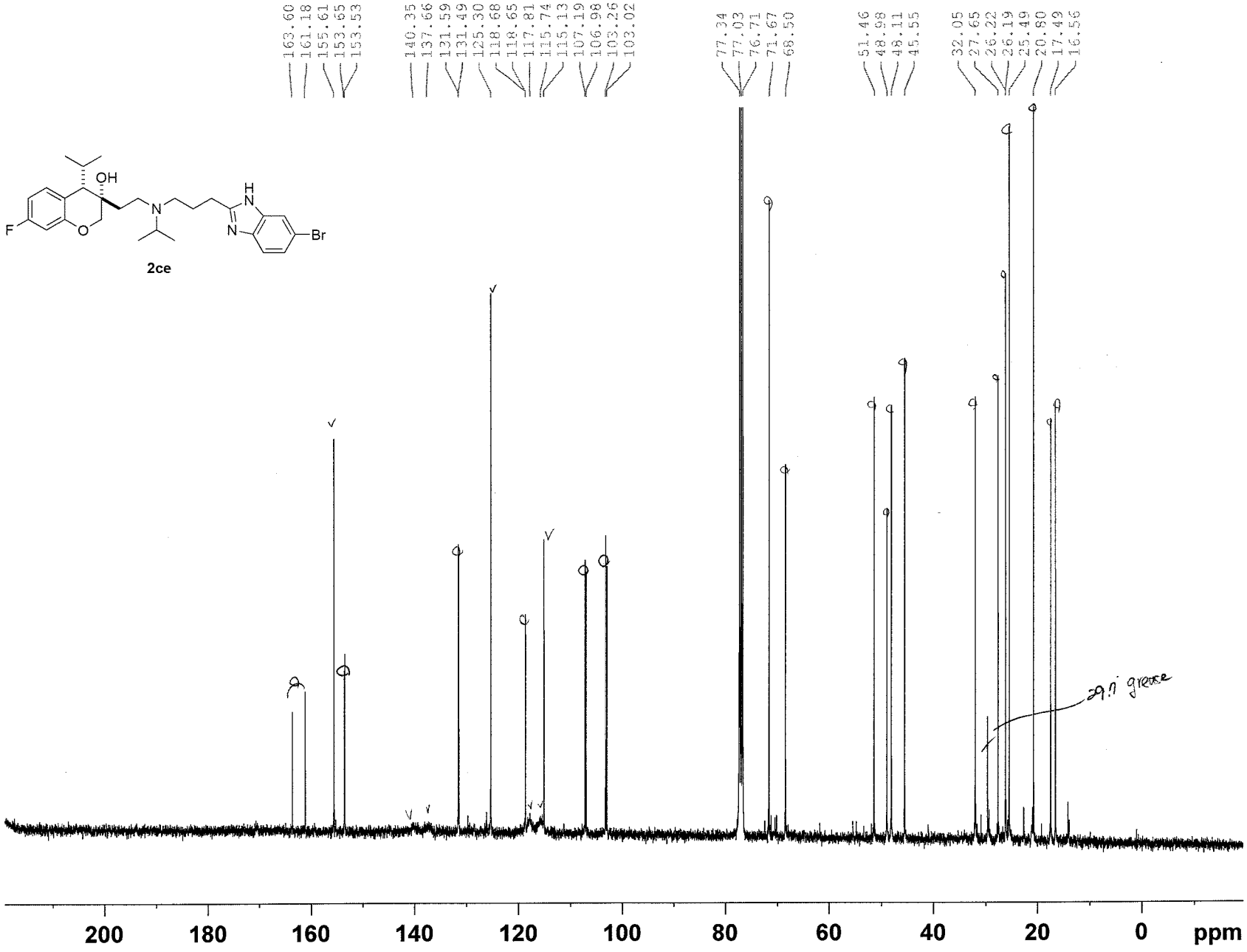
BRUKER

```
Current Data Parameters
NAME                  LJE-331
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130403
Time                 17.48
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                   84.76
DW                  60.800 usec
DE                   6.50 usec
TE                  298.3 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PLW1          12.00000000 W
SFO1          400.2124715 MHz

F2 - Processing parameters
SI                  65536
SF            400.2100145 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



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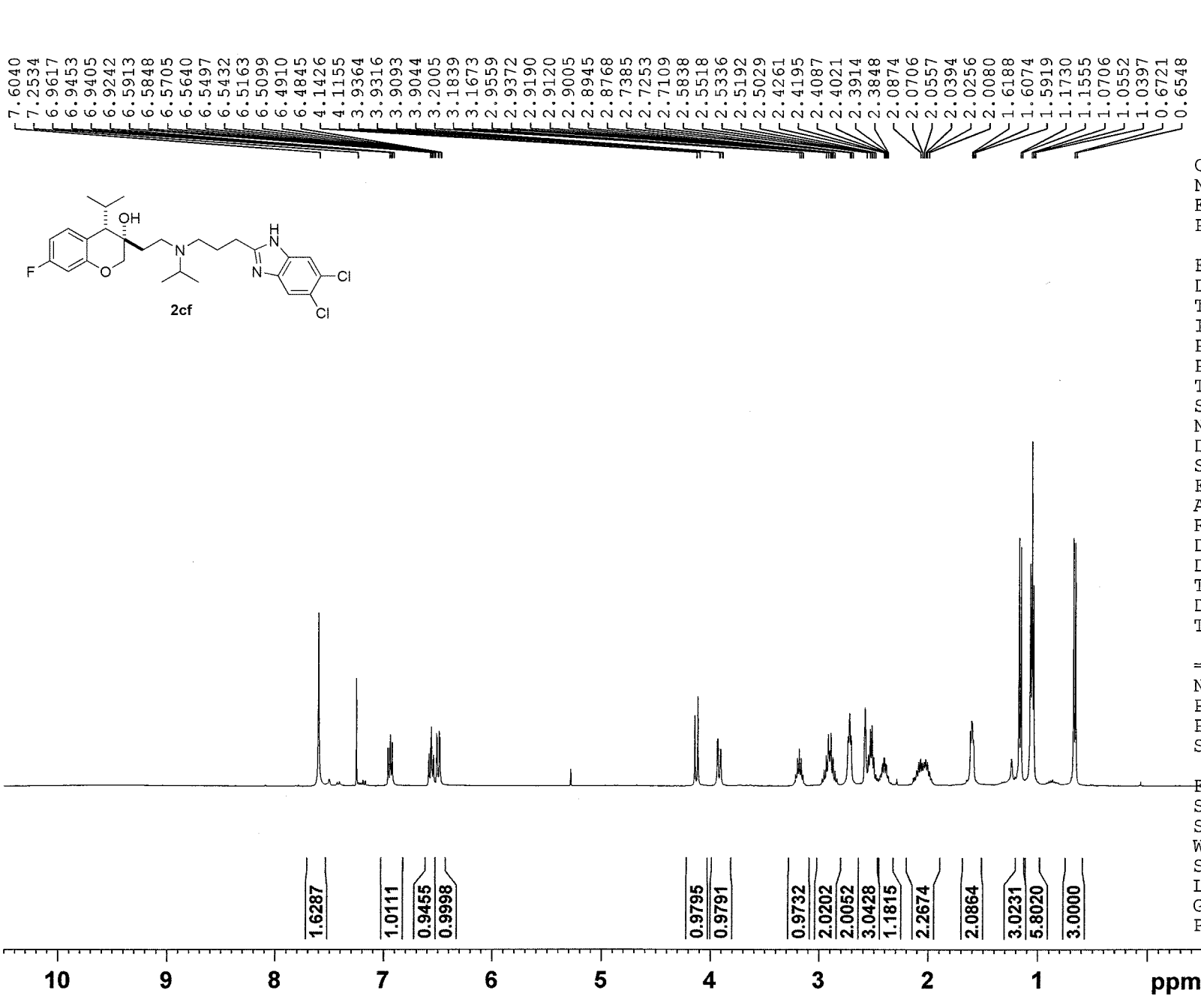
Current Data Parameters
NAME LJE-331
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130404
Time 21.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11344
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

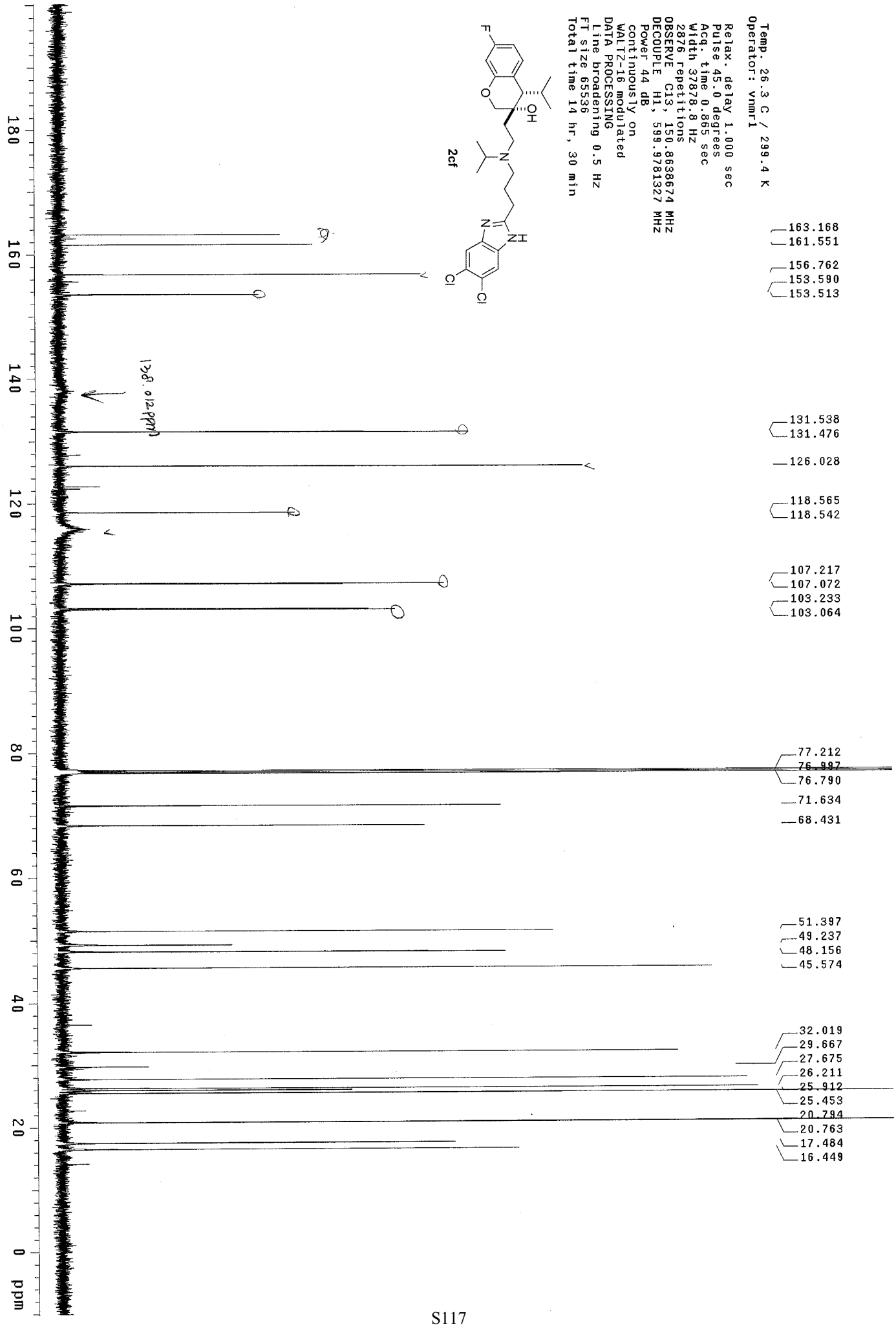


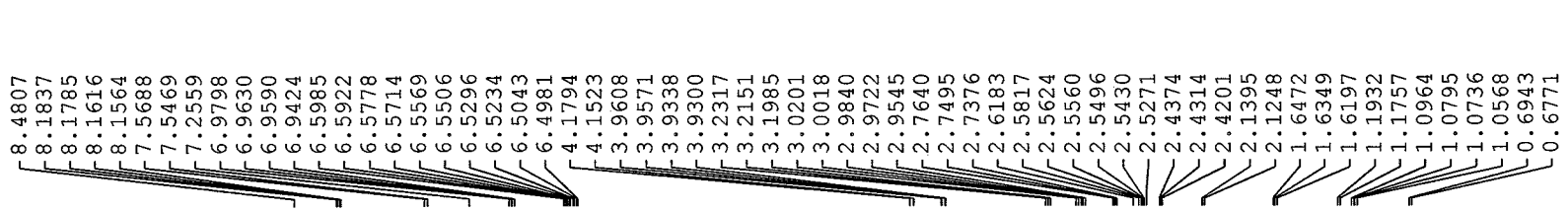
Current Data Parameters
NAME LJE-331
EXPNO 2
PROCNO 1

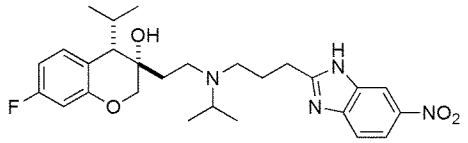
F2 - Acquisition Parameters
Date_ 20130403
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 84.76
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

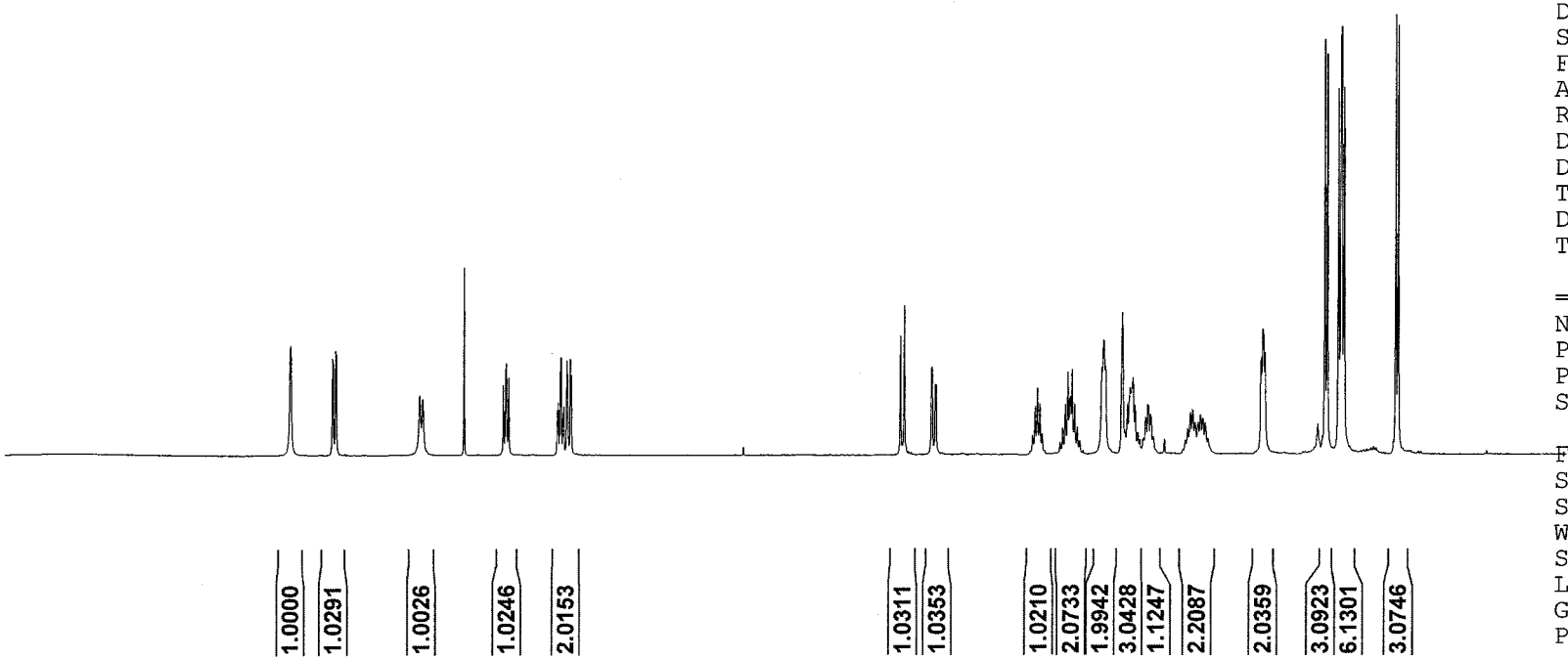
F2 - Processing parameters
SI 65536
SF 400.2100155 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

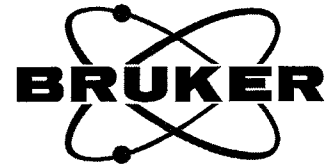

Temp. 26.3 C / 299.4 K
Operator: vnmr1
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 37878.8 Hz
2876 repetitions
OBSERVE C13, 150.8638674 MHz
DECOUPLE H1, 599.9781327 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 14 hr, 30 min
2cf
F
O
OH
N
N
H
Cl
Cl
138.012 ppm
163.168
161.551
156.762
153.590
153.513
131.538
131.476
126.028
118.565
118.542
107.217
107.072
103.233
103.064
77.212
76.997
76.790
71.634
68.431
51.397
49.237
48.156
45.574
32.019
29.667
27.675
26.211
25.912
25.453
20.794
20.763
17.484
16.449
180
160
140
120
100
80
60
40
20
0
ppm





2cg

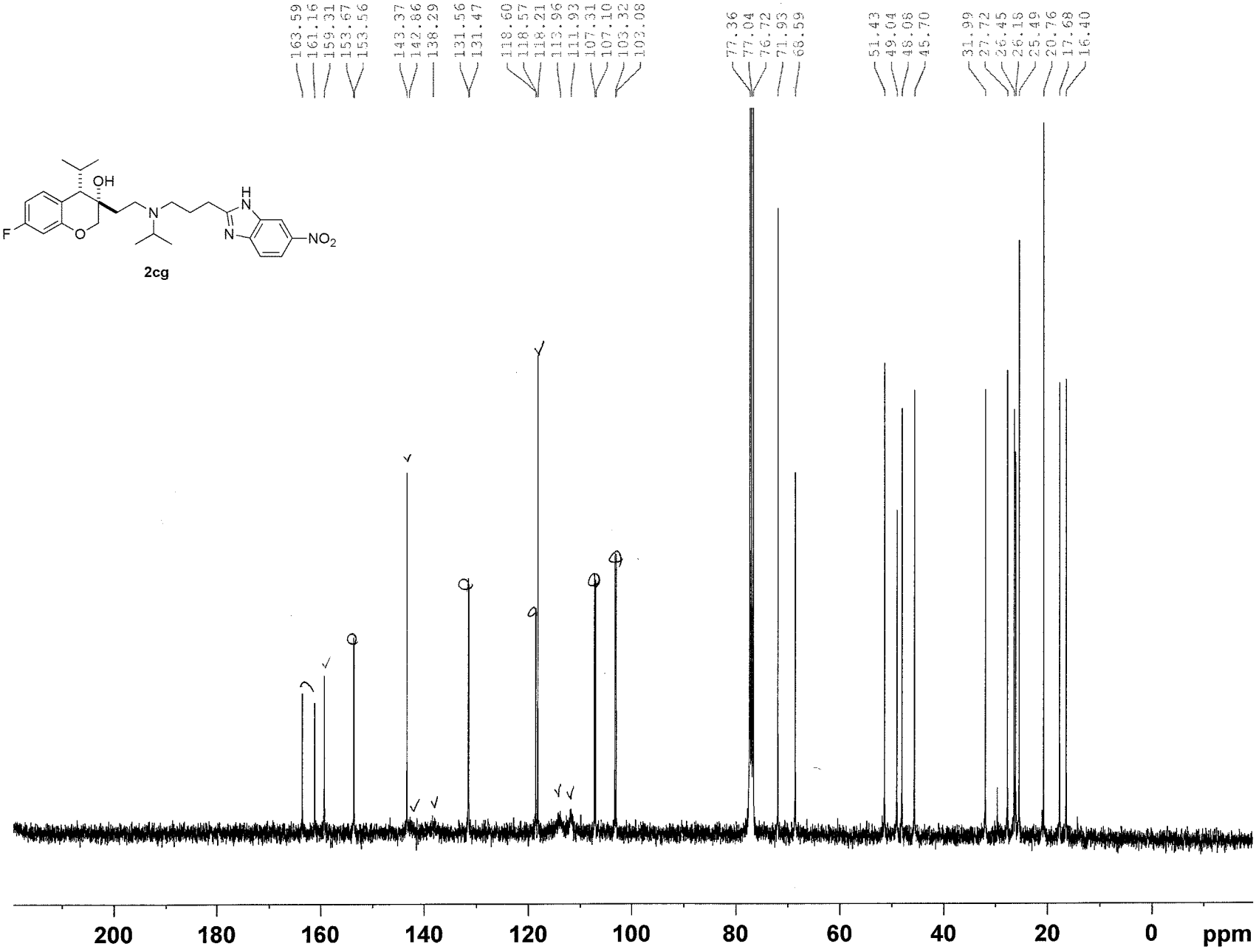





```
Current Data Parameters
NAME                  LJE-302
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130318
Time                  13.12
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                    91.91
DW                   60.800 usec
DE                    6.50 usec
TE                   296.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1          12.00000000 W
SFO1          400.2124715 MHz

F2 - Processing parameters
SI                    65536
SF            400.2100138 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

S118

2cg



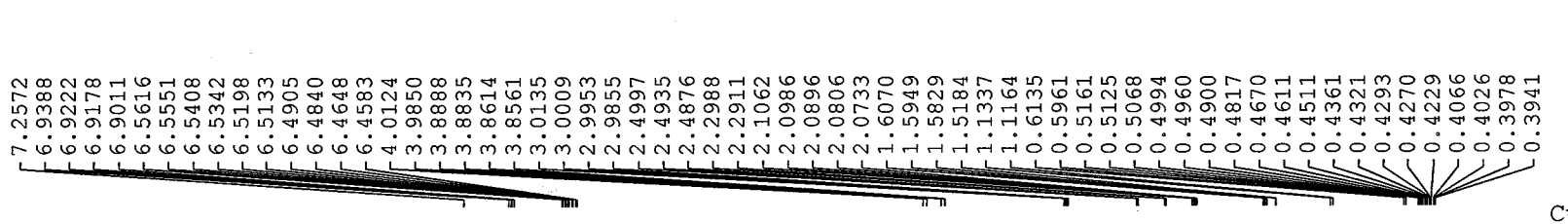
Current Data Parameters
NAME LJE-332
EXPNO 3
PROCNO 1

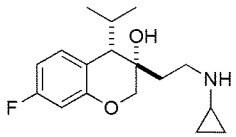
F2 - Acquisition Parameters
Date_ 20130405
Time 19.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

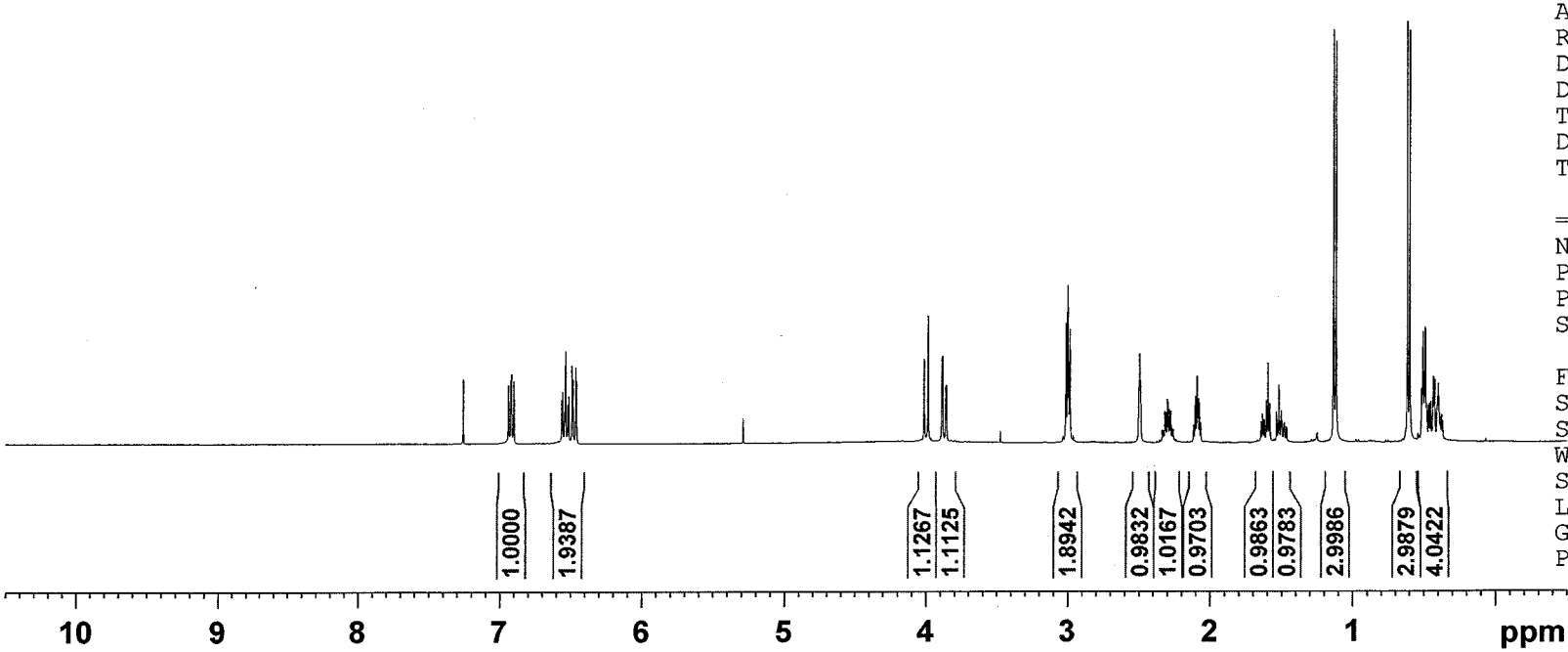
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



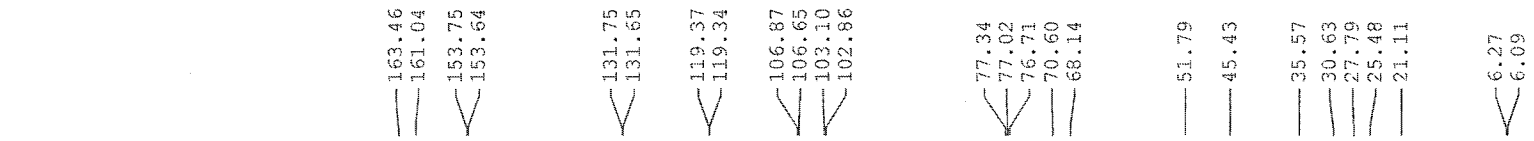


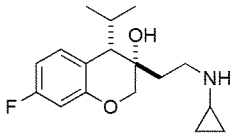
24a



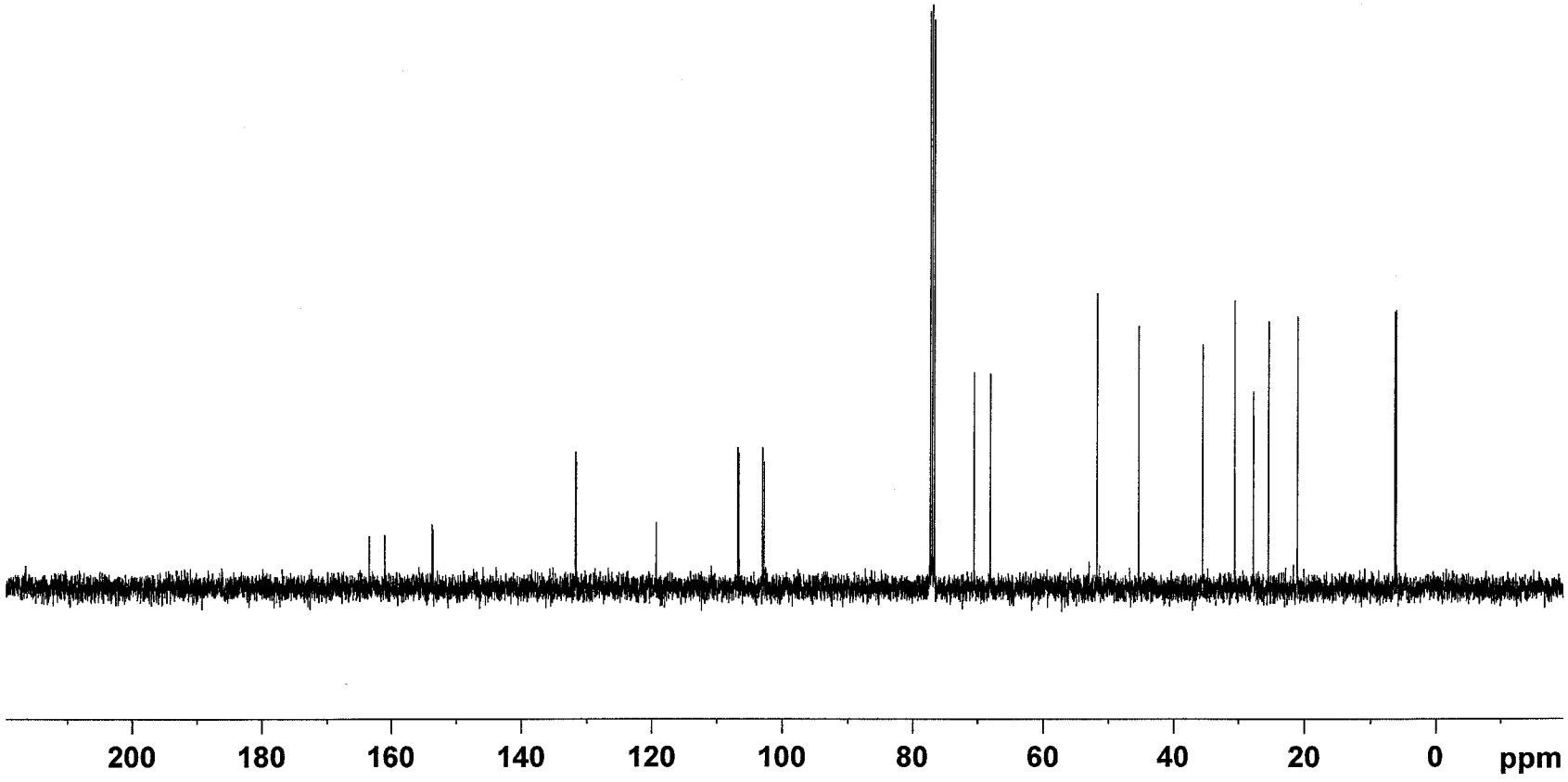
BRUKER

```
Current Data Parameters
NAME                  LJE-285
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121227
Time                  17.53
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                    69.69
DW                   60.800 usec
DE                    6.50 usec
TE                   299.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1          12.00000000 W
SFO1          400.2124715 MHz

F2 - Processing parameters
SI                    65536
SF            400.2100138 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

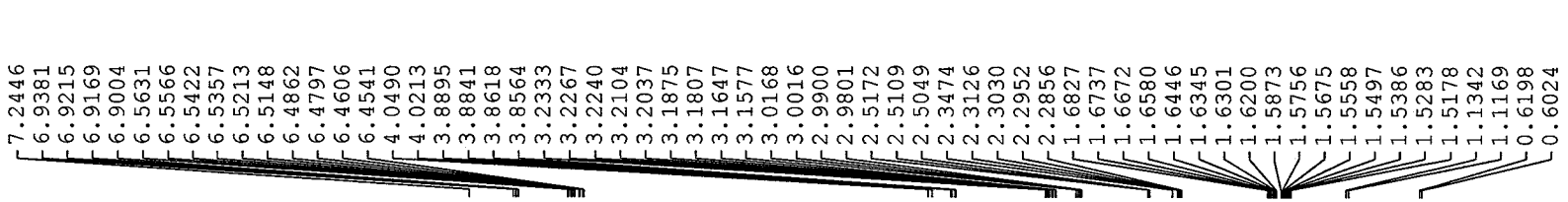


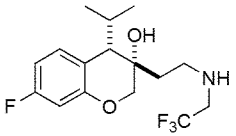


24a

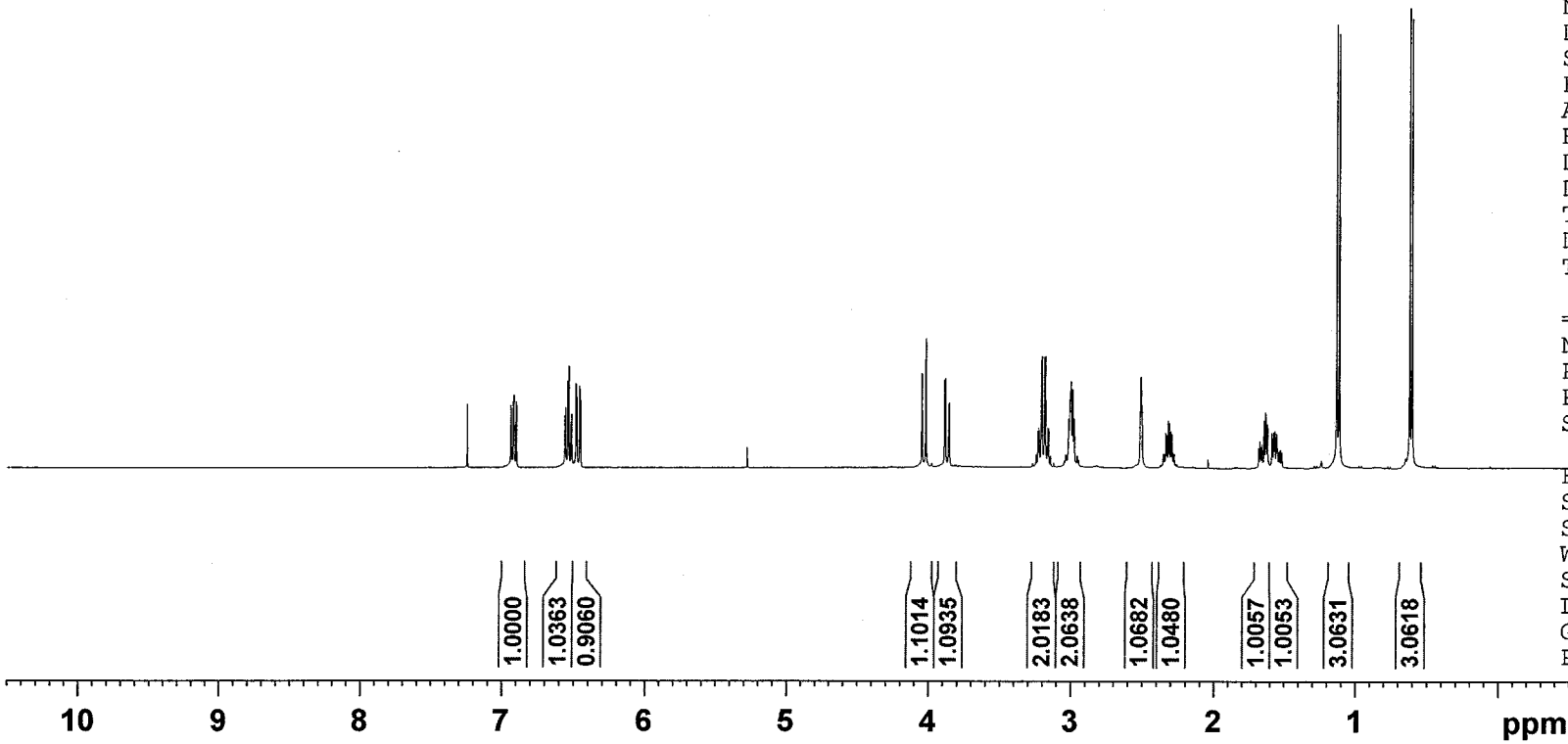



```
Current Data Parameters
NAME                  LJE-285
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_                20121227
Time                    17.56
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         31
DS                          4
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                    190.62
DW                   20.800 usec
DE                    6.50 usec
TE                   299.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.00 usec
PLW1               52.00000000 W
SFO1              100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PLW2               12.00000000 W
PLW12               0.33333001 W
PLW13               0.27000001 W
SFO2              400.2116008 MHz

F2 - Processing parameters
SI                         32768
SF                100.6328860 MHz
WDW                           EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```





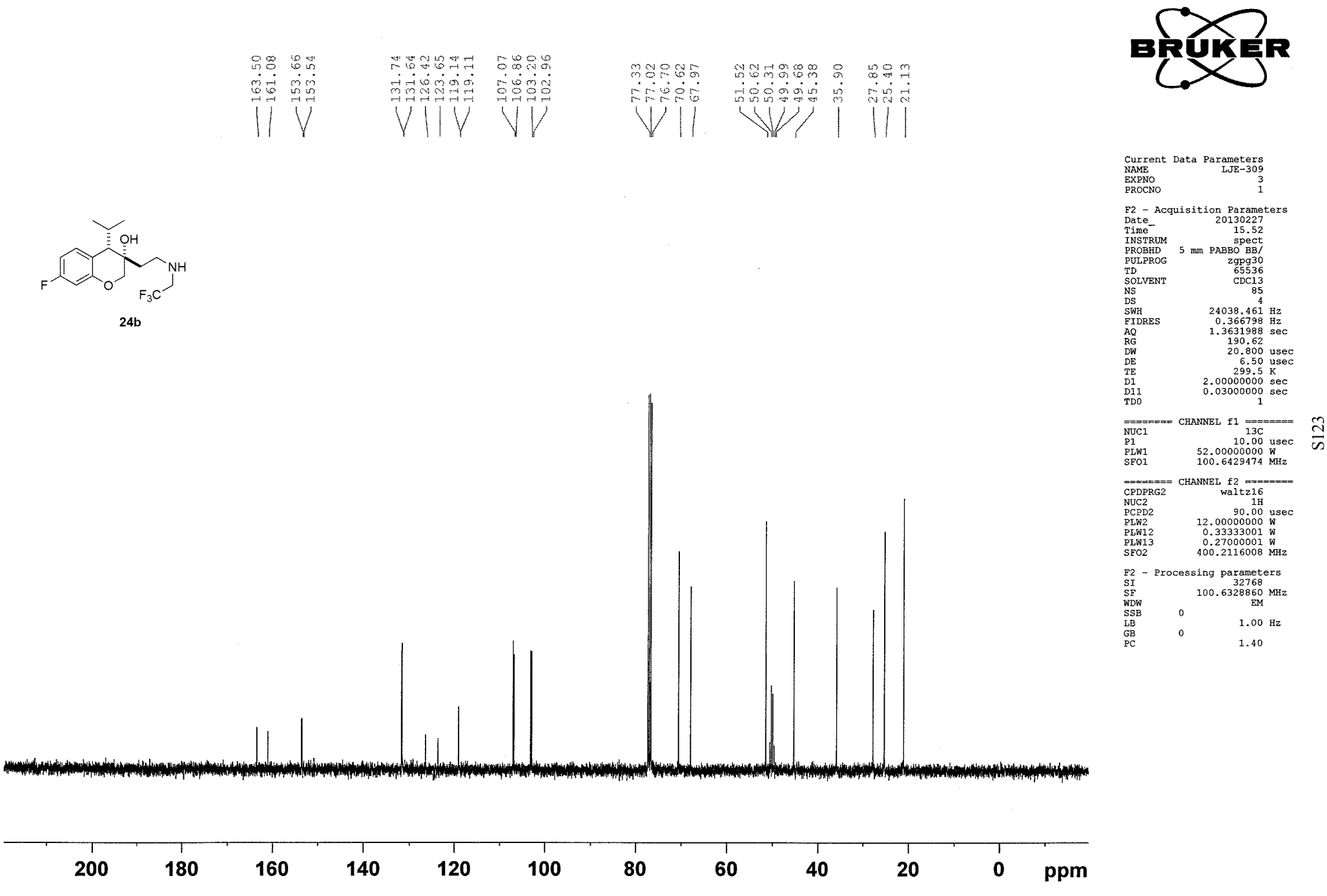
24b



BRUKER

```
Current Data Parameters
NAME                  LJE-309
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130227
Time                  15.45
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                    58.64
DW                  60.800 usec
DE                   6.50 usec
TE                  298.8 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1           12.00000000 W
SFO1           400.2124715 MHz

F2 - Processing parameters
SI                    65536
SF             400.2100190 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

S122



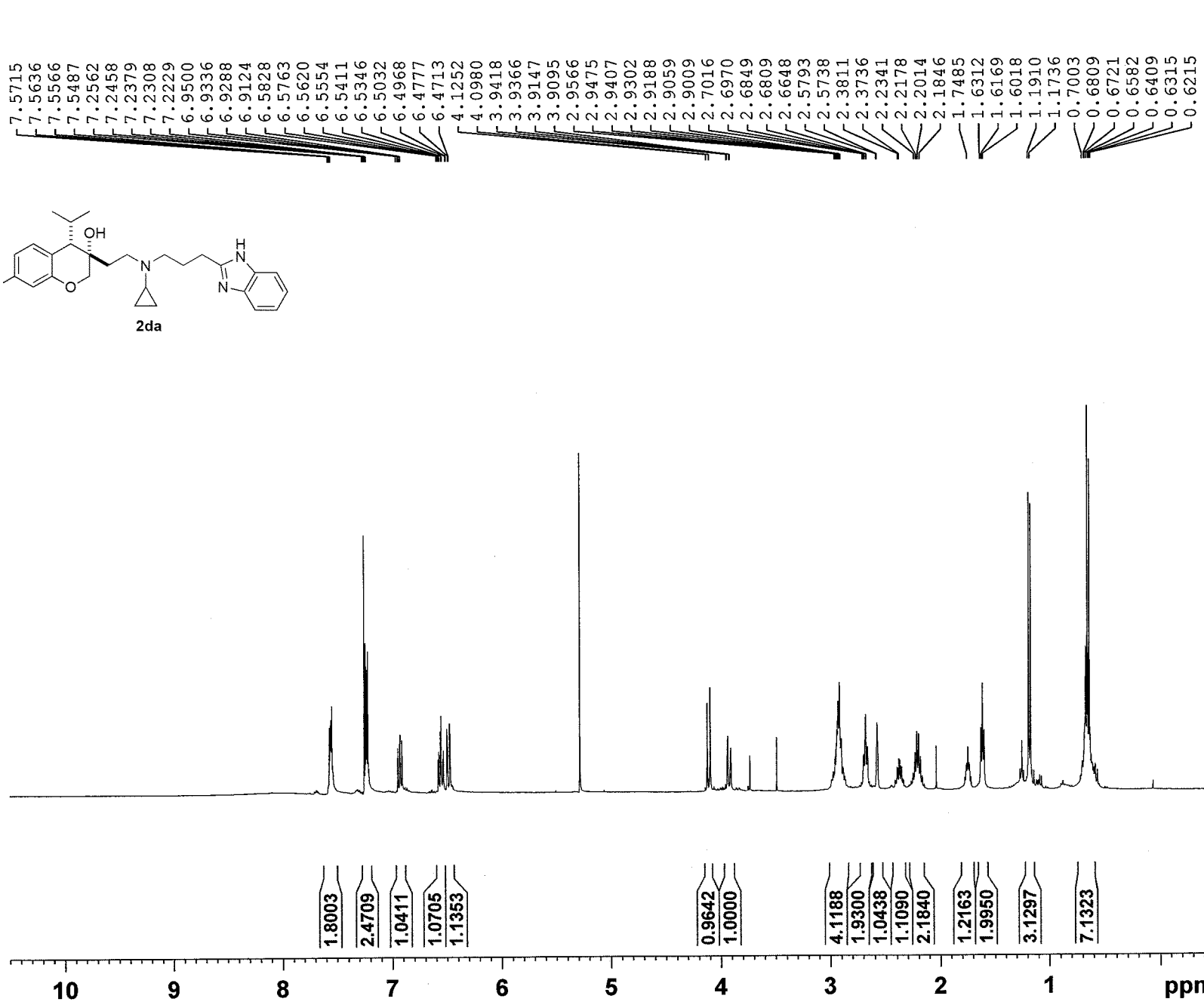
Current Data Parameters
NAME LJE-309
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130227
Time 15.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 85
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



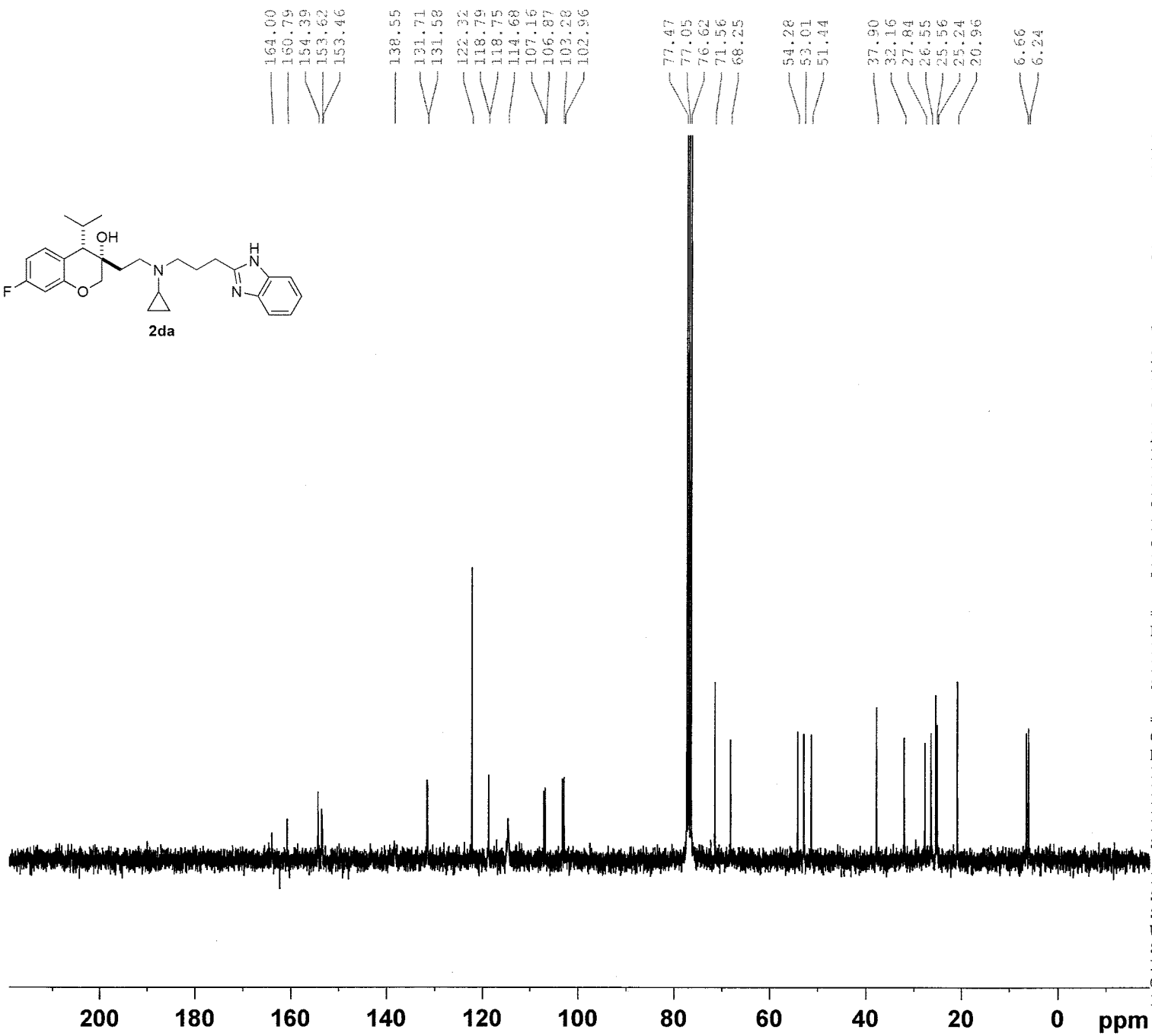
Current Data Parameters
NAME LJE-306
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130222
Time 17.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 103.95
DW 60.800 usec
DE 6.50 usec
TE 300.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

2da



BRUKER

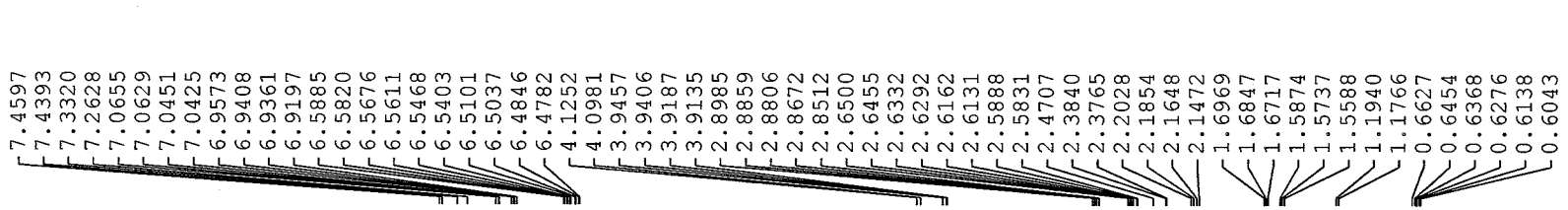
Current Data Parameters
NAME LJE-Final
EXPNO 8
PROCNO 1

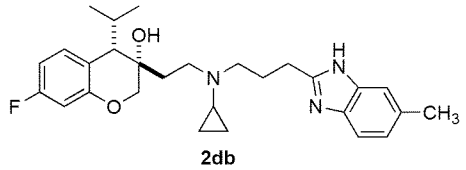
F2 - Acquisition Parameters
Date_ 20130328
Time 13.05
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 882
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

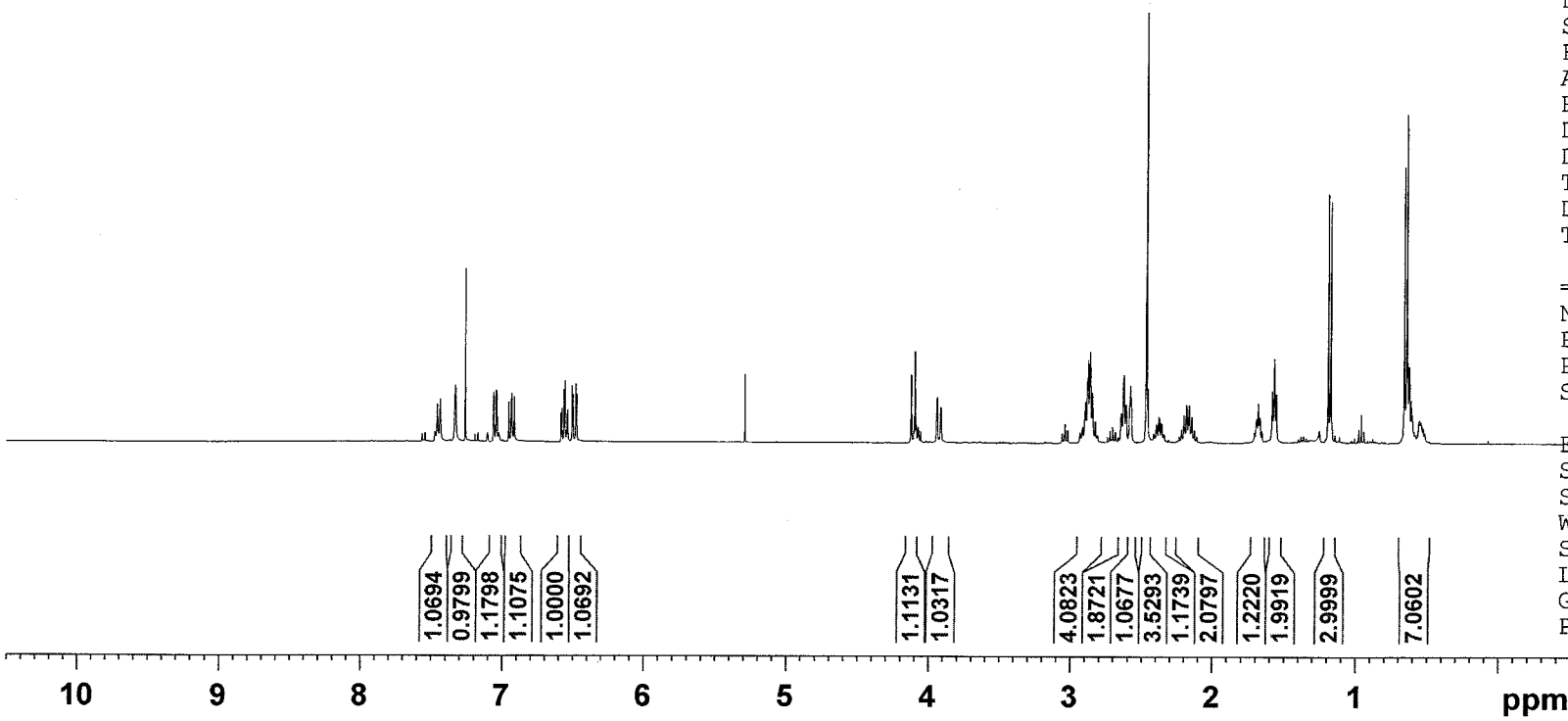
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 22.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2db

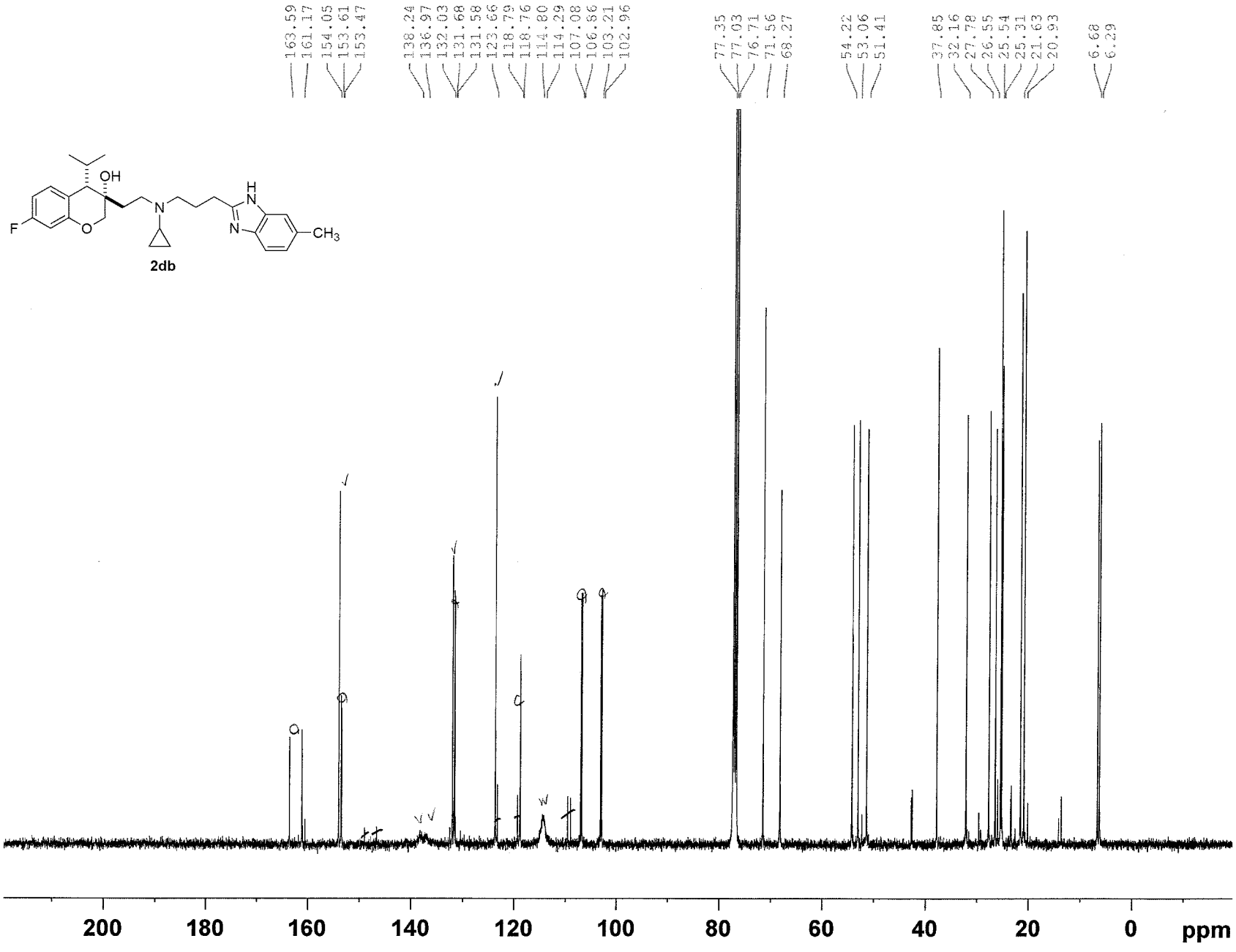


Current Data Parameters
NAME LJE-333
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130411
Time 17.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 103.95
DW 60.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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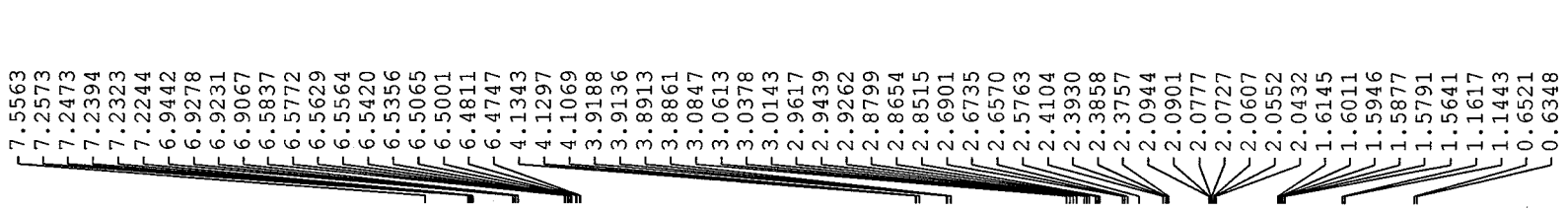
Current Data Parameters
NAME LJE-Final
EXPNO 5
PROCNO 1

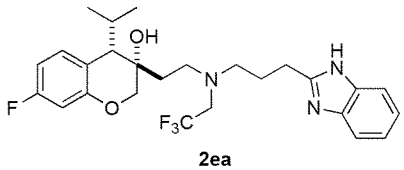
F2 - Acquisition Parameters
Date_ 20130417
Time 21.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11155
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

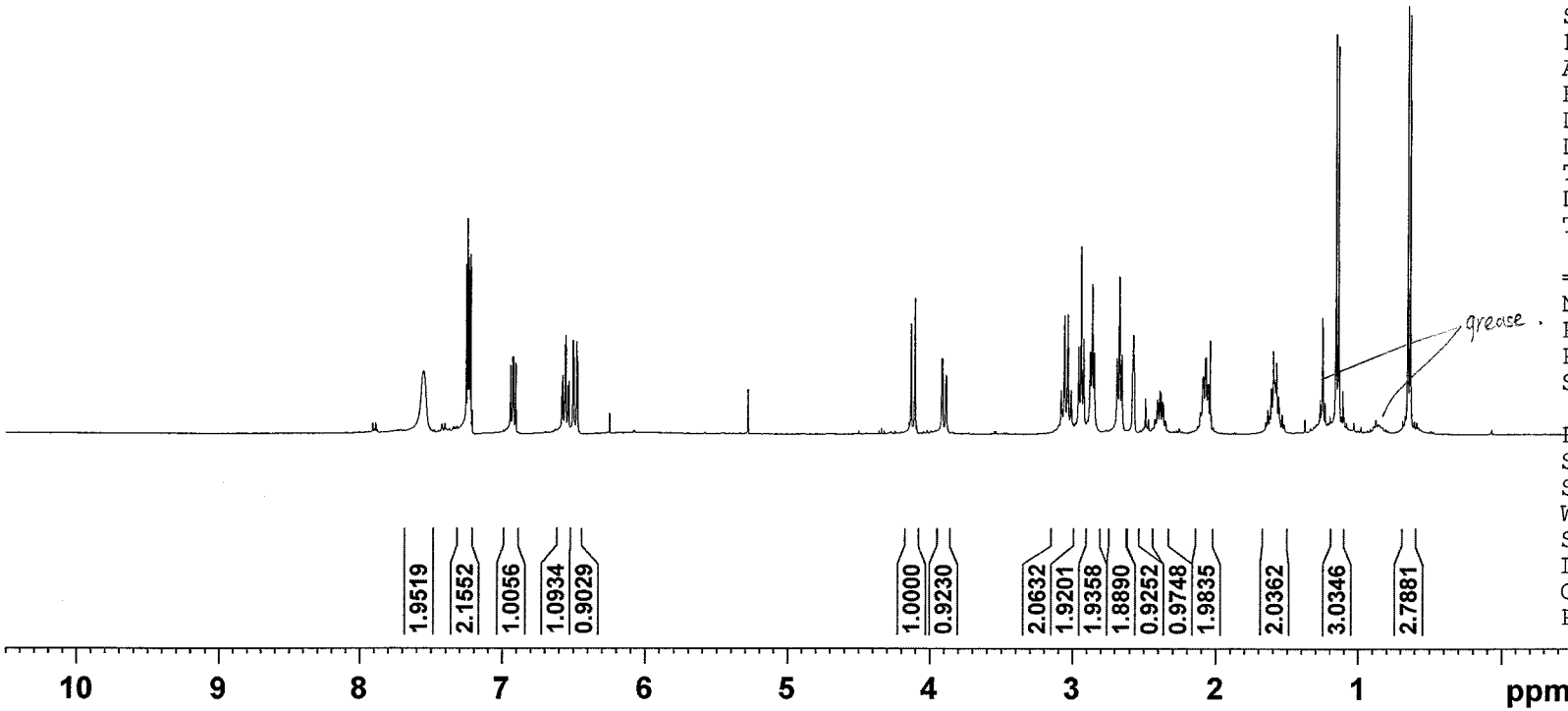
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2ea

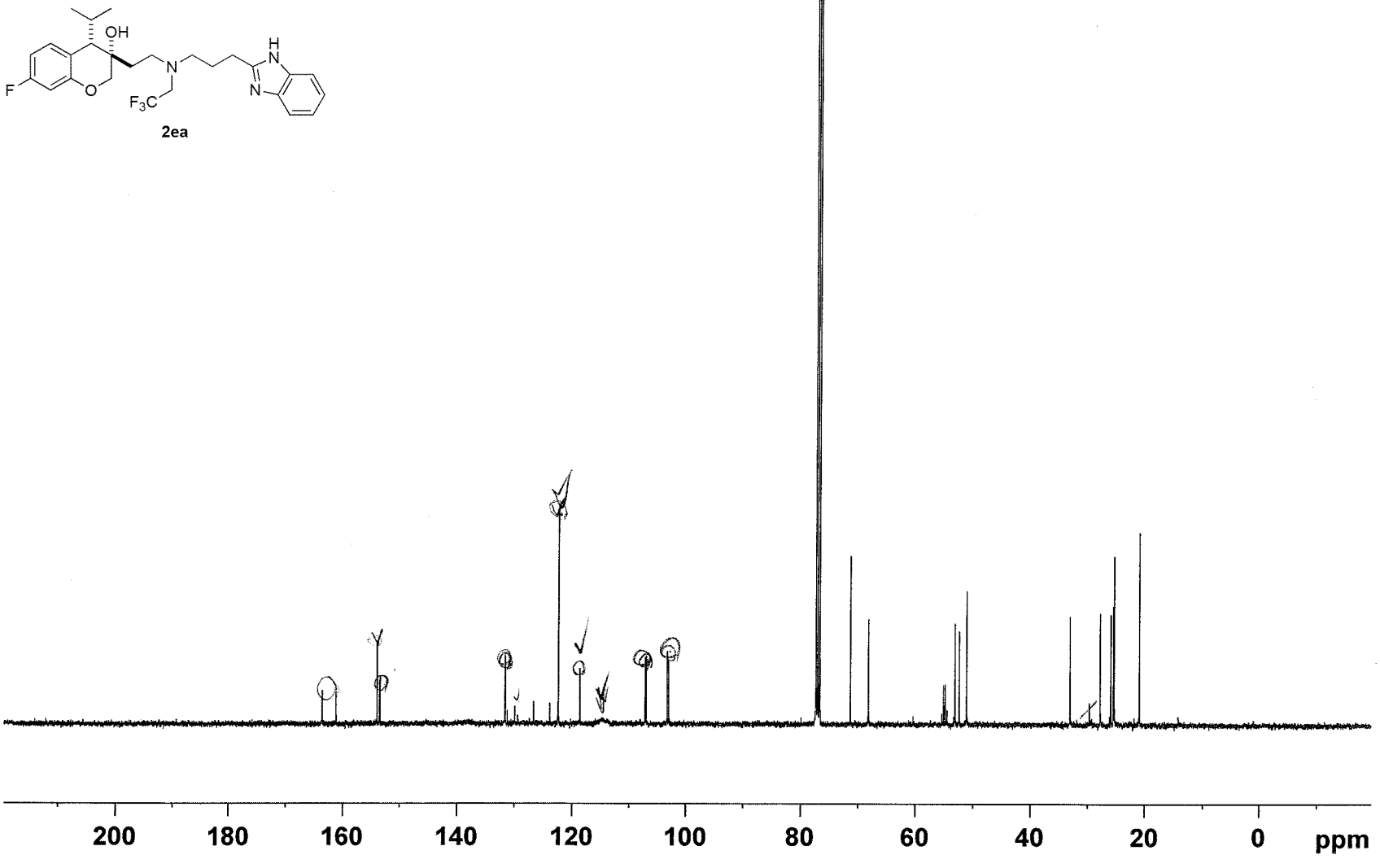



```
Current Data Parameters
NAME                  LJE-312
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130305
Time                  17.01
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                    69.69
DW                   60.800 usec
DE                    6.50 usec
TE                   299.7 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1           12.00000000 W
SFO1           400.2124715 MHz

F2 - Processing parameters
SI                    65536
SF             400.2100138 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



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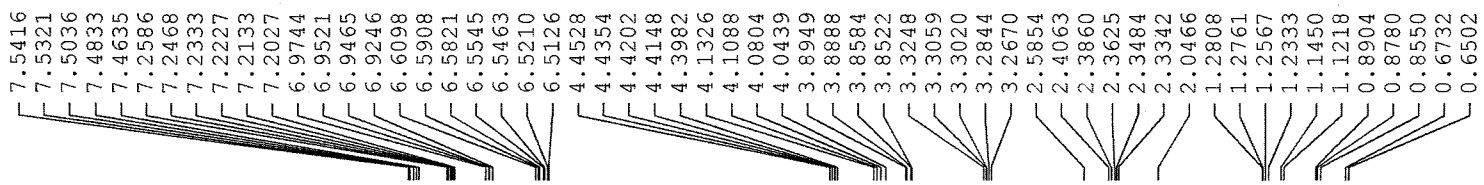
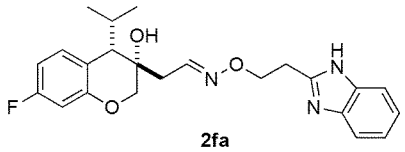
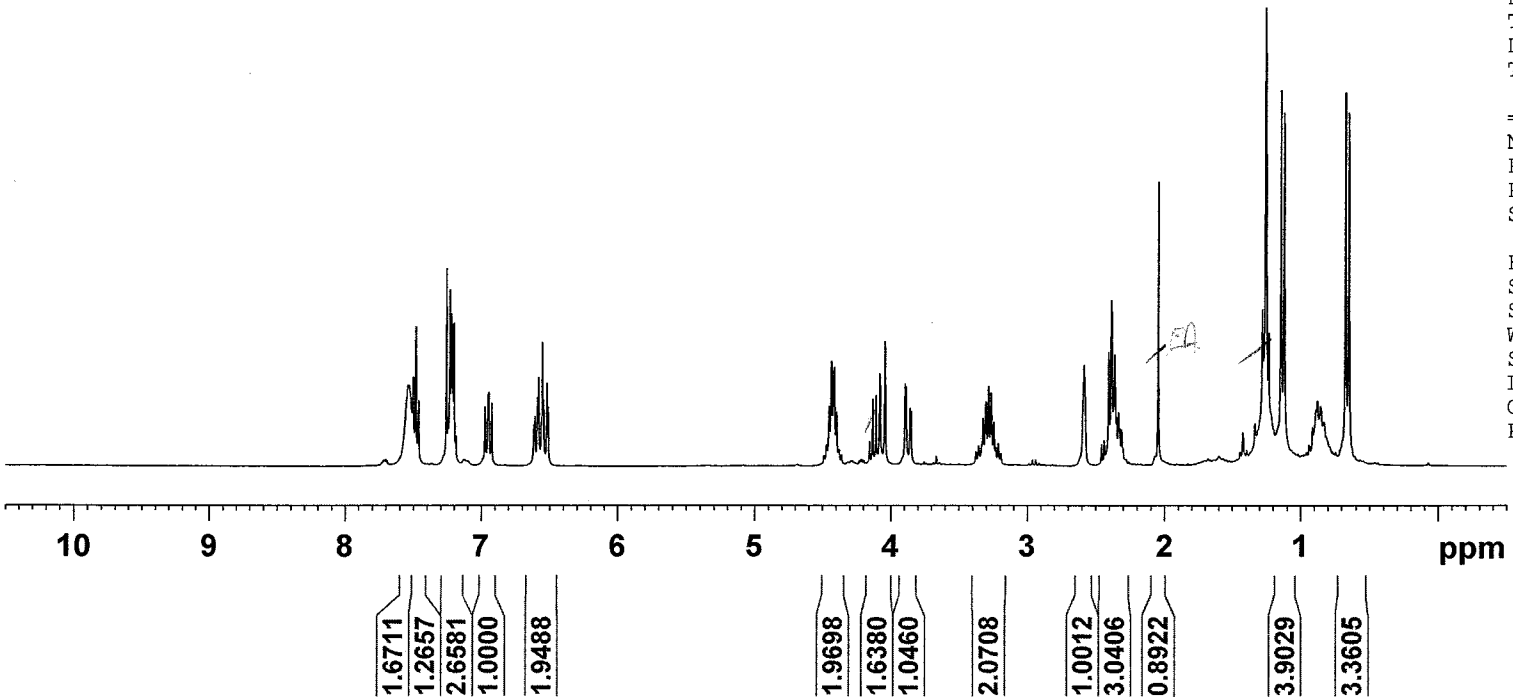
```
Current Data Parameters
NAME                  LJE-312
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_                20130327
Time                    20.49
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                       190.62
DW                      20.800 usec
DE                       6.50 usec
TE                      299.9 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PLW1               52.00000000 W
SFO1              100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PLW2               12.00000000 W
PLW12               0.33333001 W
PLW13               0.27000001 W
SFO2              400.2116008 MHz

F2 - Processing parameters
SI                         32768
SF                100.6328860 MHz
WDW                           EM
SSB                0
LB                      1.00 Hz
GB                 0
PC                      1.40
```





2fa

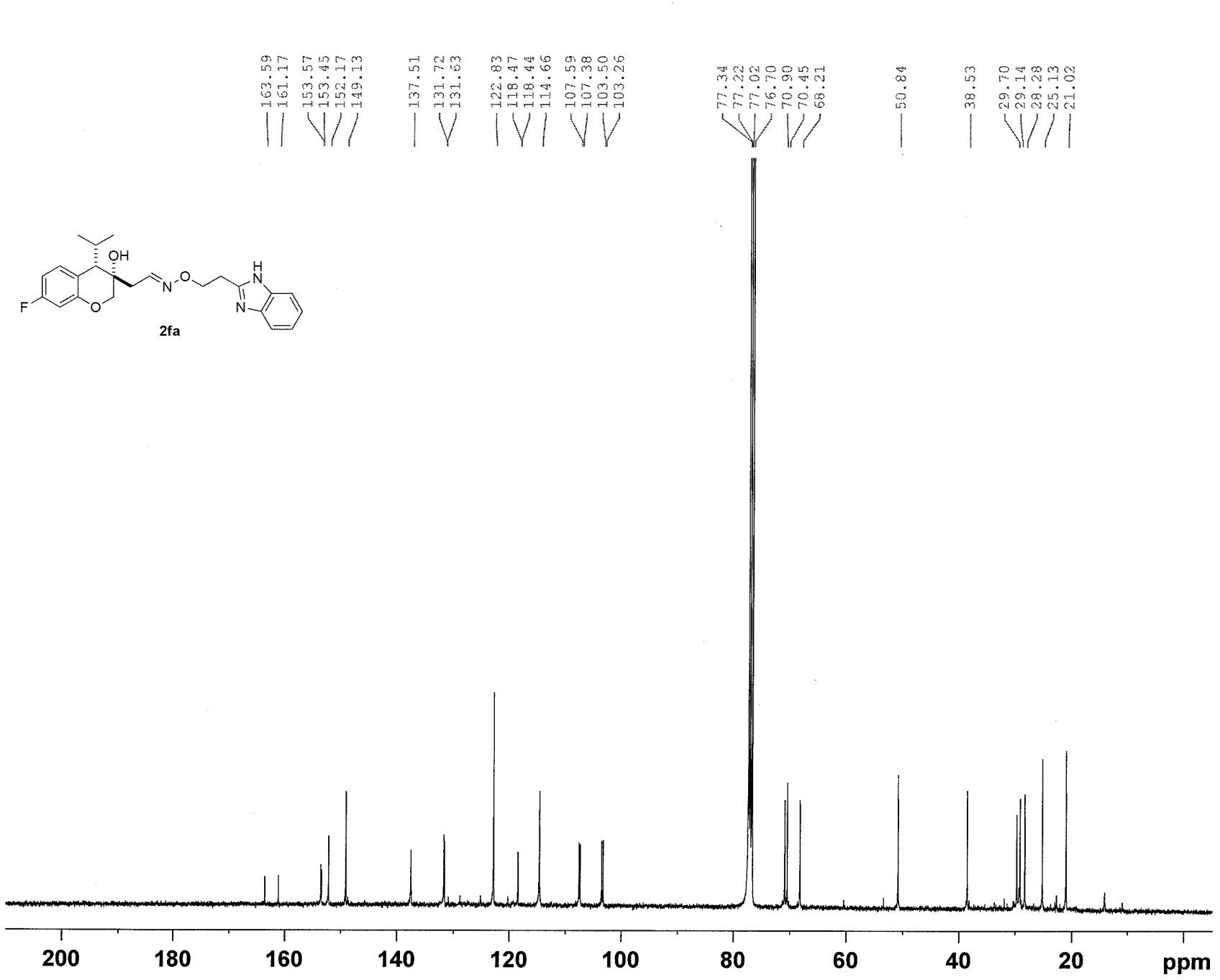


Current Data Parameters
NAME KTH-m-140
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120507
Time 14.04
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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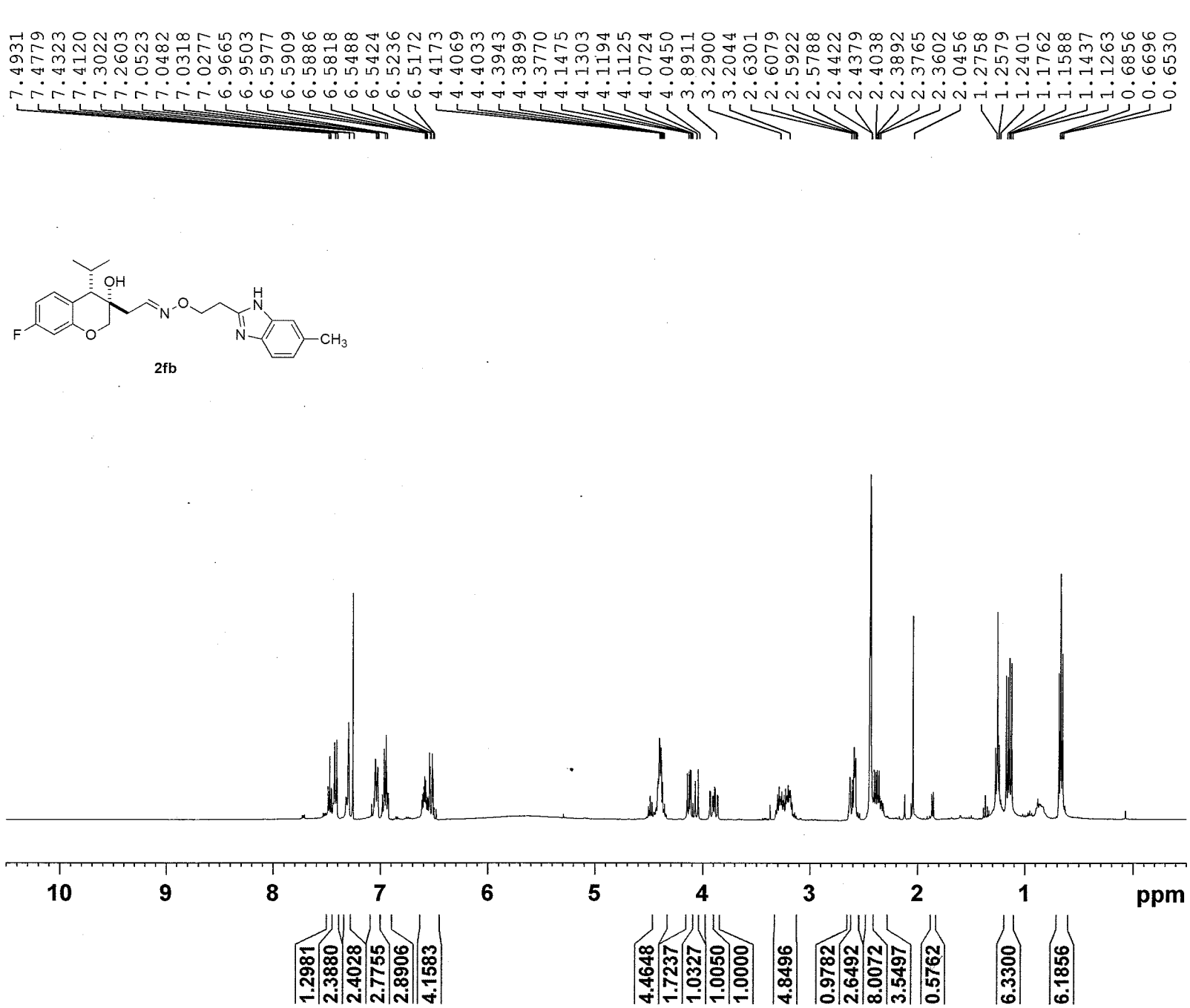
Current Data Parameters
NAME KTH-m-140
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120718
Time 9.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12288
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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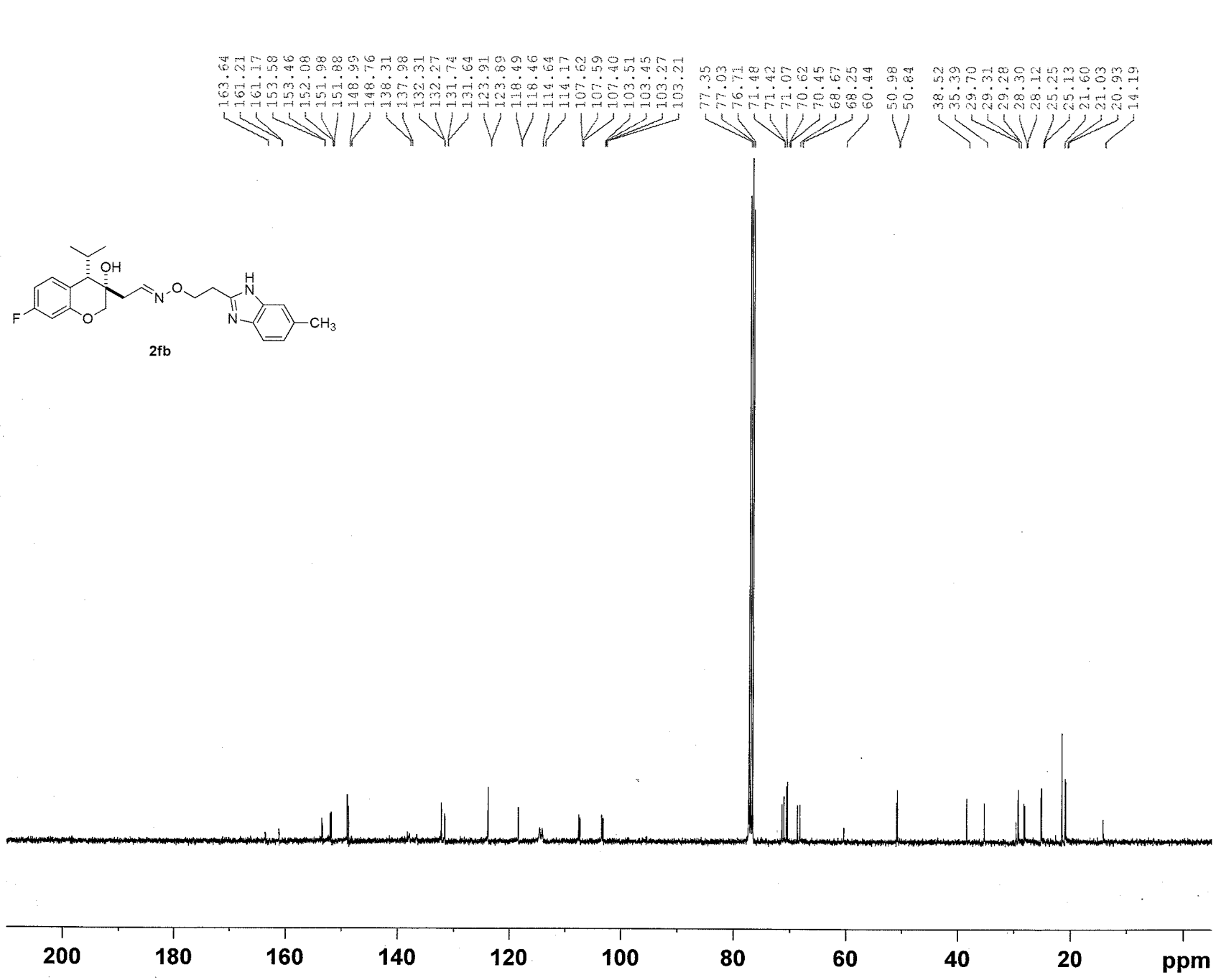
Current Data Parameters
NAME KTH-m-166
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120708
Time 21.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.91
DW 60.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



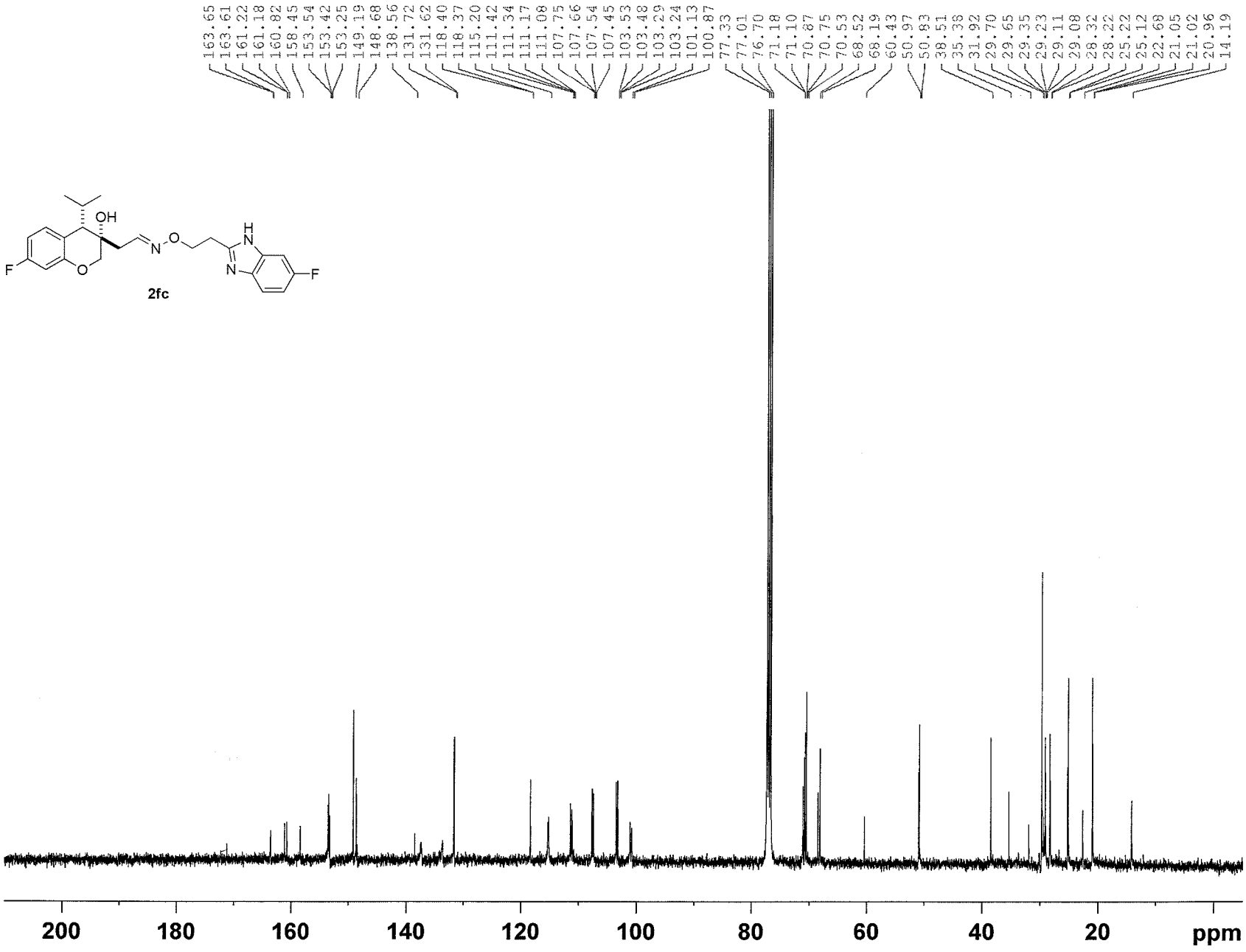
Current Data Parameters
NAME KTH-m-166
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120708
Time 22.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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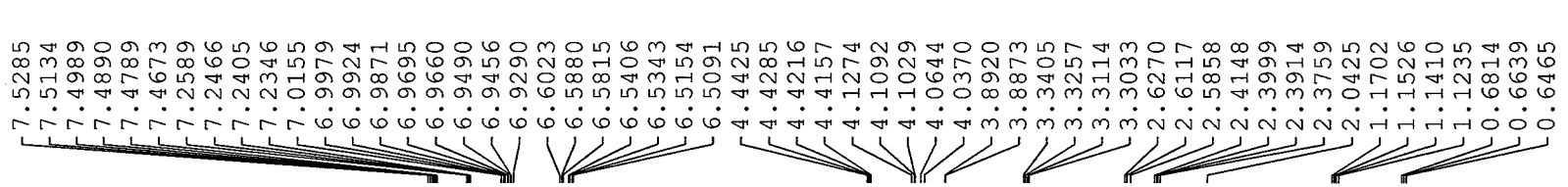
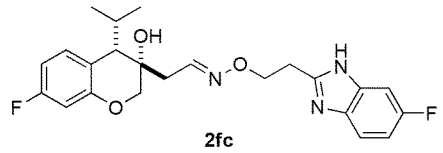
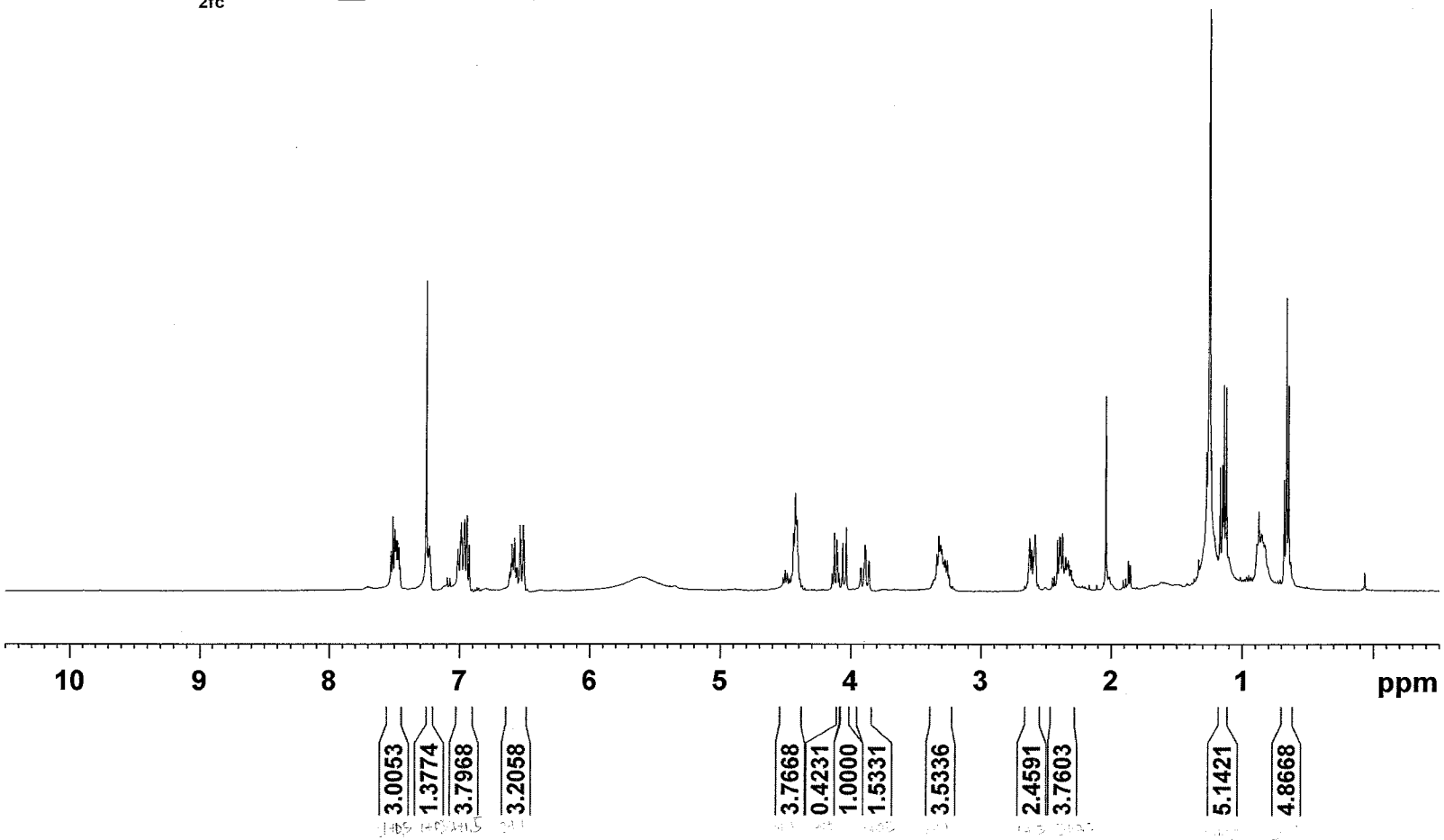
```
Current Data Parameters
NAME                  KTH-m-165
EXPNO                         5
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20120709
Time                      21.34
INSTRUM                   spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                       12170
DS                           4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631988 sec
RG                      190.62
DW                      20.800 usec
DE                       6.50 usec
TE                      298.9 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PLW1               52.00000000 W
SFO1              100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PLW2               12.00000000 W
PLW12               0.33333001 W
PLW13               0.27000001 W
SFO2              400.2116008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6328860 MHz
WDW                         EM
SSB                  0
LB                      1.00 Hz
GB                   0
PC                      1.40
```





2fc

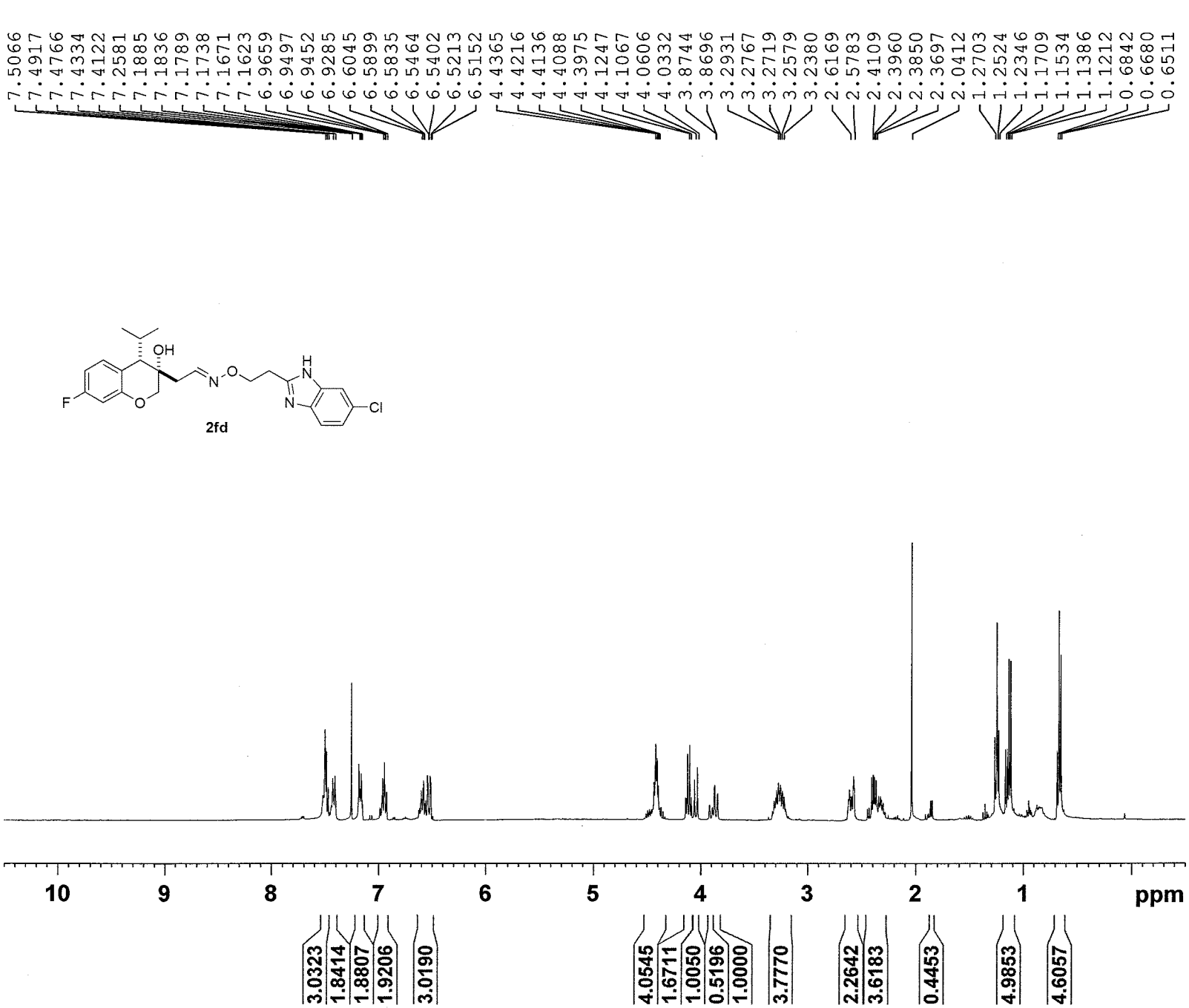


Current Data Parameters
NAME KTH-m-165
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120709
Time 21.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 119.61
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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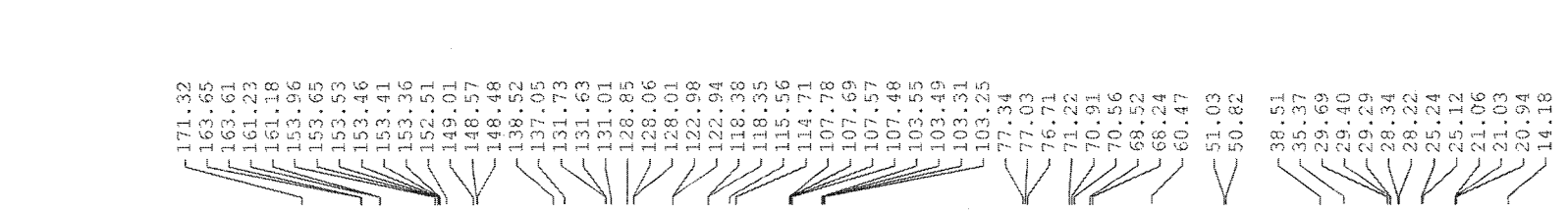
Current Data Parameters
NAME KTH-m-164
EXPNO 3
PROCNO 1

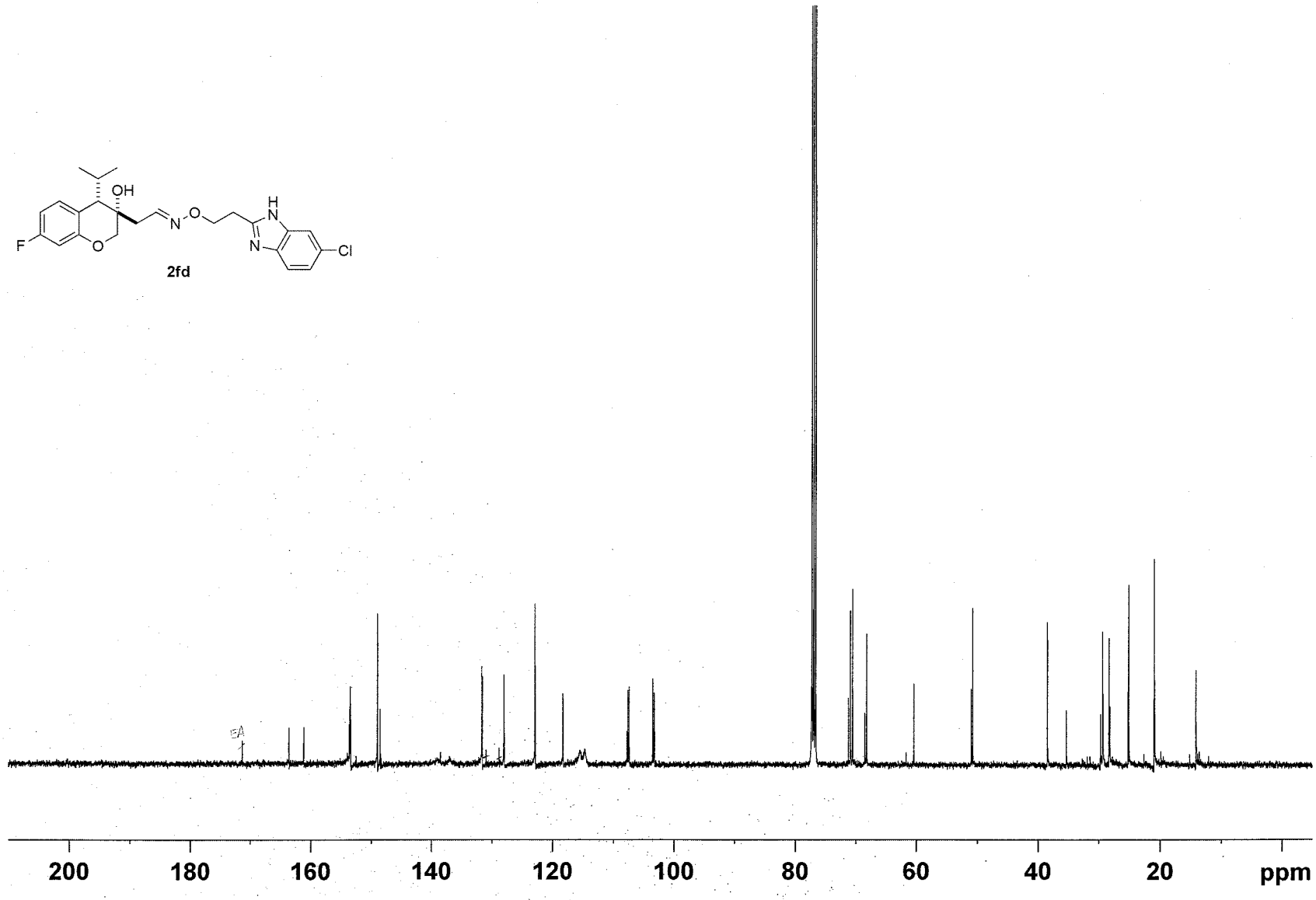
F2 - Acquisition Parameters
Date_ 20120708
Time 23.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.91
DW 60.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.2124715 MHz

F2 - Processing parameters
SI 65536
SF 400.2100138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2fd



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Current Data Parameters
NAME KTH-m-164
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120709
Time 4.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4848
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 190.62
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6429474 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.33333001 W
PLW13 0.27000001 W
SFO2 400.2116008 MHz

F2 - Processing parameters
SI 32768
SF 100.6328860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40