Supporting Information

Formal Total Synthesis of the Cytotoxic Marine Ascidian Alkaloid Haouamine A

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General Methods. All non-aqueous reactions were carried out under an inert atmosphere of argon or nitrogen in flame-dried glassware. Air and moisture sensitive liquid reagents were added via a dry syringe or cannula. Anhydrous tetrahydrofuran (THF), dichloromethane (CH_2Cl_2), diethyl ether (Et_2O) and toluene (PhMe) were obtained from a solvent dispensing system. All other solvents and reagents were used as obtained from commercial sources without further purification. Flash column chromatography was performed using EM Science silica gel 60 (230-400 mesh) or Aldrich basic alumina Brockmann I (~150 mesh). Analytical and preparative thin layer chromatography (TLC) were performed on EM Science silica gel 60 PF_{254} plates. ^1H and ^{13}C NMR spectral data were recorded on Bruker DPX-300, CPDX-300, AMX-360, or DRX-400 MHz spectrometers. Infrared spectral data were obtained using a Perkin-Elmer 1600 FTIR.

[2-(2-Hydroxyethyl)-6-methoxyphenyl]-(3-hydroxyphenyl)methanone (8). 3-Methoxyphenylmagnesium bromide (**7**, Aldrich, 1 M in THF, 18.7 mL) was added slowly to isocoumarin **6**[7] (3.01 g, 17.0 mmol) in CH_2Cl_2 (30 mL) at -78 ºC and the mixture was allowed to gradually warm to rt overnight. The mixture was quenched with saturated aqueous NH_4Cl (12 mL) and extracted with CH_2Cl_2 (3 x 12 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (50:50 EtOAc/hexanes) to afford ketoalcohol **8** as a colorless oil (4.77 g, 98%). IR (thin film) 3422, 1667, 1581, 1469, 1262 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.33 (bs, 1H, OH), 2.57 (t, *J* = 6.6 Hz, 2H), 3.55 (s, 3H), 3.63 (t, *J* = 6.6 Hz, 2H), 3.73 (s, 3H), 6.75 (d, *J* = 8.3 Hz, 1H), 6.85 (d, *J* = 7.5 Hz, 1H), 7.01 (dt, *J* = 7.0, 2.4 Hz, 1H), 7.16-7.20 (m, 2H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.35-7.36 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.2, 55.5, 55.8, 63.3, 109.1, 113.0,

120.3, 122.6, 122.8, 129.3, 129.6, 130.6, 137.8, 139.0, 156.9, 159.9, 198.3; HRMS *m/z* calcd for $C_{17}H_{19}O_4$ 287.1278 (MH^+), found 287.1281.

{2-[2-(*tert*-Butyldimethylsilanyloxy)ethyl]-6-methoxyphenyl}-(3-methoxyphenyl)-methanone (9). Imidazole (3.40 g, 50.0 mmol) and TBSCl (5.02 g, 33.32 mmol) were added to ketoalcohol **8** (4.77 g, 16.67 mmol) in CH_2Cl_2 (33 mL) and the mixture was stirred for 2 h at rt. The reaction mixture was diluted with H_2O (35 mL) and extracted with CH_2Cl_2 (3 x 35 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (15:85 EtOAc/hexanes) to yield ketone **9** as a white solid (6.67 g, 100%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.00 (s, 6H), 0.78 (s, 9H), 2.1 (t, *J* = 7.4 Hz, 2H), 3.64 (t, *J* = 7.4 Hz, 2H), 3.66 (s, 3H), 3.83 (s, 3H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.91 (d, *J* = 7.7 Hz, 1H), 7.09 (dt, *J* = 6.8, 1.6 Hz, 1H), 7.25 (s, 1H), 7.27-7.30 (m, 3H), 7.45 (t, *J* = 1.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ -5.4, 26.0, 36.8, 55.5, 55.7, 64.0, 108.9, 112.9, 120.2, 122.8, 123.1, 129.3, 129.6, 129.9, 137.4, 139.1, 156.7, 159.9, 197.6; HRMS *m/z* calcd for $C_{23}H_{32}O_4NaSi$ 423.1968 ($M+Na^+$), found 423.1953.

2-{3-Methoxy-2-[1-(3-methoxyphenyl)vinyl]phenyl}ethanol (10). The Tebbe-Petasis reagent[8] (0.5M in 50:50 THF/toluene, 1.6 mL) was added to a solution of ketone **9** (125 mg, 312 μmol) in toluene (1.5 mL). The mixture was heated in an oil bath maintained at 120 ºC for 15 h. After cooling the mixture to 40 ºC, saturated aqueous $NaHCO_3$ (1.5 mL) and MeOH (1.5 mL) were added to destroy the excess Tebbe-Petasis reagent. The mixture was stirred for an additional 1 h at 40 ºC and extracted with Et_2O (3 x 1 mL) after cooling to rt. The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (20:80 EtOAc/hexanes) to give olefin **10** as a pale

yellow oil (111 mg, 91%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.00 (s, 6H), 0.89 (s, 9H), 2.77-2.83 (m, 2H), 3.66-3.72 (m, 2H), 3.73 (s, 3H), 3.79 (s, 3H), 5.22 (d, *J* = 1.3 Hz, 1H), 6.06 (d, *J* = 1.3 Hz, 1H), 6.80-6.86 (m, 2H), 6.91-6.95 (m, 2H), 6.98 (dd, *J* = 7.7, 0.9 Hz, 1H), 7.22 (t, *J* = 8.2 Hz, 1H), 7.24 (t, *J* = 8.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ -5.2, 18.5, 25.8, 26.1, 37.0, 55.2, 55.9, 64.5, 108.9, 112.0, 112.8, 115.6, 118.8, 122.8, 128.2, 129.3, 130.6, 138.5, 141.6, 143.8, 157.3, 159.7.

***tert*-Butyl-(2-{3-methoxy-2-[1-(3-methoxyphenyl)vinyl]phenyl}ethoxy)-dimethylsilane (11).** TBAF (1 M in THF, 3.18 mL) was added to silylether olefin **10** (1.15 g, 2.89 mmol) in THF (29 mL) and the mixture was stirred at rt for 2 h. Saturated aqueous NH_4Cl (20 mL) was added and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were dried over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (20:80 EtOAc/hexanes) to afford hydroxy olefin **11** as a pale yellow oil (0.82 g, 100%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.68 (bs, 1H), 2.77-2.81 (m, 2H), 3.68-3.71 (m, 2H), 3.73 (s, 3H), 3.78 (s, 3H), 5.19 (d, *J* = 1.2 Hz, 1H), 6.03 (d, *J* = 1.2 Hz, 1H), 6.82 (ddd, *J* = 8.2, 2.5, 0.9 Hz, 1H), 6.85-6.93 (m, 3H), 6.95 (dd, *J* = 7.7, 0.8 Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.6, 55.3, 56.0, 63.5, 109.2, 112.0, 112.8, 115.9, 118.7, 122.2, 128.4, 129.4, 130.7, 138.1, 141.5, 143.6, 157.4, 159.7; HRMS *m/z* calcd for $C_{18}H_{21}O_3$ 285.1469 (MH^+), found 285.1485.

{3-Methoxy-2-[1-(3-methoxyphenyl)vinyl]phenyl}acetaldehyde (12). Dess-Martin periodinane[10] (0.82 g, 1.93 mmol) was added to hydroxy olefin **11** (0.50 g, 1.75 mmol) in CH_2Cl_2 (18 mL) at 0 ºC. The mixture was stirred at rt for 1 h. Aqueous NaOH (2 M, 30 mL) was added to the reaction mixture which was extracted with CH_2Cl_2 (3 x 35 mL). The residue was purified

by flash column chromatography on silica gel (14:86 EtOAc/hexanes) to yield aldehyde **12** as a white translucent gum (0.39 g, 79%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.55 (d, *J* = 2.0 Hz, 2H), 3.71 (s, 3H), 3.75 (s, 3H), 5.12 (d, *J* = 1.2 Hz, 1H), 5.98 (d, *J* = 1.1 Hz, 1H), 6.76-6.81 (m, 2H), 6.82-6.86 (m, 2H), 6.91 (dd, *J* = 8.3, 0.8 Hz, 1H), 7.14 (dt, *J* = 7.6, 1.1 Hz, 1H), 7.31 (dd, *J* = 8.2, 7.7 Hz, 1H), 9.54 (t, *J* = 2.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 48.6, 55.6, 56.4, 110.6, 112.4, 113.3, 117.0, 119.0, 123.2, 129.2, 129.8, 131.5, 132.7, 141.3, 143.7, 158.0, 160.1, 200.1.

Nitrone Cycloadditon Procedure. ***Method 1.*** Et_3N (0.44 mL, 3.14 mmol) was added to a solution of benzylhydroxylamine hydrochloride (0.47 g, 2.88 mmol) in toluene (1.0 mL) with Na_2SO_4 (0.10 g) and the mixture was stirred for 30 min at rt. Aldehyde **12** (0.74 g, 2.62 mmol) in toluene (2.6 mL) was added to the solution. The mixture was stirred for an additional 1 h and then heated at 115 ºC for 36 h. Concentration of the reaction mixture *in vacuo* gave a residue that was partitioned between brine (15 mL) and CH_2Cl_2 (10 mL) and extracted with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (10:90 EtOAc/hexanes) to yield **14** (640 mg, 63%) and 315 mg of an impure fraction containing **15**. The impure mixture containing the bridged cycloadduct **15** was heated in toluene (10 mL) at 115 ºC for 24 h to give additional linear product **14** (769 mg, 76% total yield).

Method 2. Et_3N (0.44 mL, 3.14 mmol) was added to a solution of benzylhydroxylamine hydrochloride (0.47 g, 2.88 mmol) in toluene (1.0 mL) along with Na_2SO_4 (0.10 g) and the mixture was stirred for 30 min at rt. Aldehyde **12** (0.74 g, 2.62 mmol) in toluene (2.6 mL) was added to the solution. The mixture was stirred for an additional 1 h and then filtered. The filtrate was heated in an oil bath maintained at 130 ºC for 45 h. The reaction mixture was concentrated *in vacuo*. The resulting residue was partitioned between brine (15 mL) and CH_2Cl_2

(10 mL) and extracted with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo*. The mixture was purified by flash column chromatography on silica gel (20:80 EtOAc/hexanes) to afford isoxazolidine **14** as a white solid (0.60 g, 73%).

1-Benzyl-4-methoxy-3a-(3-methoxyphenyl)-3,3a,8,8a-tetrahydro-1*H*-indeno[2,1-*c*]isoxazole (**14**). White solid, mp 109-111 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.62 (d, *J* = 17.1 Hz, 1H), 2.97 (dd, *J* = 17.0, 6.4 Hz, 1H), 3.39 (d, *J* = 6.3 Hz, 1H), 3.62 (s, 3H), 3.67 (s, 3H), 4.04 (s, 2H), 4.11 (d, *J* = 9.0 Hz, 1H), 4.57 (d, *J* = 9.0 Hz, 1H), 6.70-6.75 (m, 4H), 6.77 (d, *J* = 7.6 Hz, 1H), 7.13-7.32 (m, 6), 7.32 (dd, *J* = 7.8, 1.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.2, 55.4 (2), 61.3, 70.1, 76.8, 77.2, 77.7, 80.0, 82.5, 109.1, 111.3, 117.8, 119.0, 127.5, 128.5 (2), 129.3 (2), 129.8, 133.0, 144.3, 146.8, 156.5, 159.7; HRMS *m/z* calcd for $C_{24}H_{35}O_3Si$ 388.1895 (MH^+), found 388.1907. Suitable crystals for X-ray analysis were obtained by the slow evaporation of a 1:1 pentane/ Et_2O solution.

[2-Amino-7-methoxy-1-(3-methoxyphenyl)-indan-1-yl]methanol (16). 10% $Pd(OH)_2$/C (1.05 g) was added to a solution of isoxazolidine **14** (2.10 g, 5.17 mmol) in 1:1 CH_2Cl_2/methanol (50 mL). The mixture was stirred under one atmosphere of H_2 at rt for 12 h, filtered through a pad of Celite, and concentrated *in vacuo* to yield a translucent gum (1.45 g, 94%). The resulting amino alcohol was used without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.46 (bs, 2H), 2.65 (dd, *J* = 15.7, 5.3 Hz, 1H), 3.12 (dd, *J* = 15.7, 6.8 Hz, 1H), 3.63 (s, 3H), 3.71 (s, 3H), 3.80 (dd, *J* = 6.8, 5.4 Hz, 1H), 4.27 (d, *J* = 11.4 Hz, 1H), 4.37 (d, *J* = 11.5 Hz, 1H), 6.73-6.78 (m, 4H), 6.89 (d, *J* = 7.4 Hz, 1H), 7.16 (t, *J* = 8.2 Hz, 1H), 7.23 (t, *J* = 7.8 Hz, 1H).

1-(*tert*-Butyldimethylsilanyloxymethyl)-7-methoxy-1-(3-methoxyphenyl)-indan-2-ylamine (17). TBSCl (0.86 g, 5.69 mmol) and imidazole (0.40 g, 5.83 mmol) were added to amino alcohol **16** (1.45 g, 4.86 mmol) in DMF/CH_2Cl_2 (40:60, 50 mL), and the mixture was stirred for 12 h at rt. The reaction mixture was diluted with H_2O (50 mL) and extracted with Et_2O (3 x 50 mL). The combined organic layers were washed with H_2O (50 mL), dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (25:75 EtOAc/hexanes) to give amino silyl ether **17** as a colorless gum (1.99 g, 99%). ^{1}H NMR (400 MHz, $CDCl_3$) δ -0.31 (s, 3H), -0.11 (s, 3H), 0.74 (s, 9H), 1.78 (bs, 2H), 2.75 (dd, *J* = 15.4, 7.6 Hz, 1H), 3.14 (dd, *J* = 15.5, 7.9 Hz, 1H), 3.57 (s, 3H), 3.70 (t, *J* = 7.5 Hz, 1H), 3.71 (s, 3H), 4.19 (d, *J* = 9.8 Hz, 1H), 4.55 (d, *J* = 9.9 Hz, 1H), 6.67 (d, *J* = 8.3 Hz, 1H), 6.68-6.75 (m, 3H), 6.82 (d, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 7.9 Hz, 1H), 7.20 (t, *J* = 7.8 Hz, 1H)); ^{13}C NMR (75 MHz, $CDCl_3$) δ -5.9, -5.7, 18.1, 25.9, 42.2, 55.0, 55.3, 60.5, 109.0, 110.6, 113.0, 117.3, 119.0, 128.9 (2C), 132.1, 145.5, 147.7, 157.0, 159.5; HRMS *m/z* calcd for $C_{24}H_{36}NO_3Si$ 414.2464 (MH^+), found 414.2469.

2-(2-Bromo-5-methoxyphenyl)-*N*-[1-(tert-butyl-dimethylsilanyloxymethyl)-7-methoxy-1-(3-methoxyphenyl)-indan-2-yl]-acetamide (24). EDAC (75 mg, 0.383 mmol) was added to phenylacetic acid **23**[12] and amino silyl ether **17** in CH_2Cl_2 (3.2 mL) at -10 ºC. The mixture was allowed to gradually warm to rt overnight. H_2O (2.5 mL) was added to the reaction mixture and solution was extracted with CH_2Cl_2 (3 x 3 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (25:75 EtOAc/hexanes) to afford amide **24** as a white foam (201 mg, 99%). ^{1}H NMR (300 MHz, $CDCl_3$) δ -0.40 (s, 3H), -0.20 (s, 3H), 0.60 (s, 9H), 2.71 (dd, *J* = 15.6, 8.0 Hz, 1H), 3.28 (dd, *J* = 15.7, 8.9 Hz, 1H), 3.55 (s, 3H), 3.66 (s, 3H), 3.67 (s, 2H), 3.77

(s, 3H), 4.25 (AB_q, J = 24.7, 10.1 Hz, 2H), 4.93 (q, J = 8.9 Hz, 1H), 6.61-6.73 (m, 5H), 6.77 (d, J = 6.9 Hz, 1H), 6.92 (d, J = 3.0 Hz, 1H), 7.09 (t, J = 7.9 Hz, 1H), 7.18 (t, J = 7.8 Hz, 1H), 7.46 (d, J = 8.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 6.2, -5.6, 17.9, 25.8, 40.7, 44.8, 54.8, 55.2, 55.6, 59.8, 60.3, 65.2, 109.1, 111.3, 112.7, 115.3, 115.4, 117.1 (2C), 118.8, 129.0, 129.2, 130.8, 133.7, 136.1, 144.5, 145.8, 156.6, 154.4 (2C), 169.1; HRMS m/z calcd for $C_{33}H_{42}NO_5NaSiBr$ 662.1913 (MNa^+), found 662.1904.

2-(2-Bromo-5-methoxyphenyl)-*N*-[1-formyl-7-methoxy-1-(3-methoxyphenyl)-indan-2-yl]-acetamide (25). TBAF (1 M in THF, 117 μL) was added to silylether amide **24** (50 mg, 78 μmol) in THF (1.0 mL) and the mixture was stirred at rt for 30 min. Saturated aqueous NH_4Cl (1 mL) was added and the mixture was extracted with CH_2Cl_2 (3 x 1 mL). The combined organic layers were dried over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (50:50 EtOAc/hexanes) to yield the alcohol as a white gum (41 mg, 100%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.36 (bs, 1H), 2.74 (dd, J = 16.5, 1.9 Hz, 1H), 3.04 (dd, J = 16.4, 6.2 Hz, 1H), 3.62 (s, 2H), 3.67 (s, 3H), 3.74 (s, 3H), 3.75 (s, 3H), 4.16-4.22 (m, 2H), 4.83-4.88 (m, 1H), 6.45-6.46 (m, 1H), 6.57 (d, J = 7.8 Hz, 1H), 6.67 (dd, J = 8.8, 3.1 Hz, 1H), 6.69 (dd, J = 8.3, 2.6 Hz, 1H), 6.82 (d, J = 8.1 Hz, 1H), 6.86 (d, J = 3.1 Hz, 1H), 6.90 (d, J = 8.0 Hz, 1H), 7.11 (t, J = 8.0 Hz, 1H), 7.26-7.31 (m, 1H), 7.39 (d, J = 8.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 39.0, 44.6, 55.3, 55.6, 55.7, 62.3, 62.6, 66.3, 109.4, 111.7, 113.2, 115.1 (2C), 115.6, 117.1, 118.9, 119.2, 129.4, 129.5, 130.0, 133.6, 135.9, 144.3, 145.5, 156.8, 159.2, 159.7, 169.9.

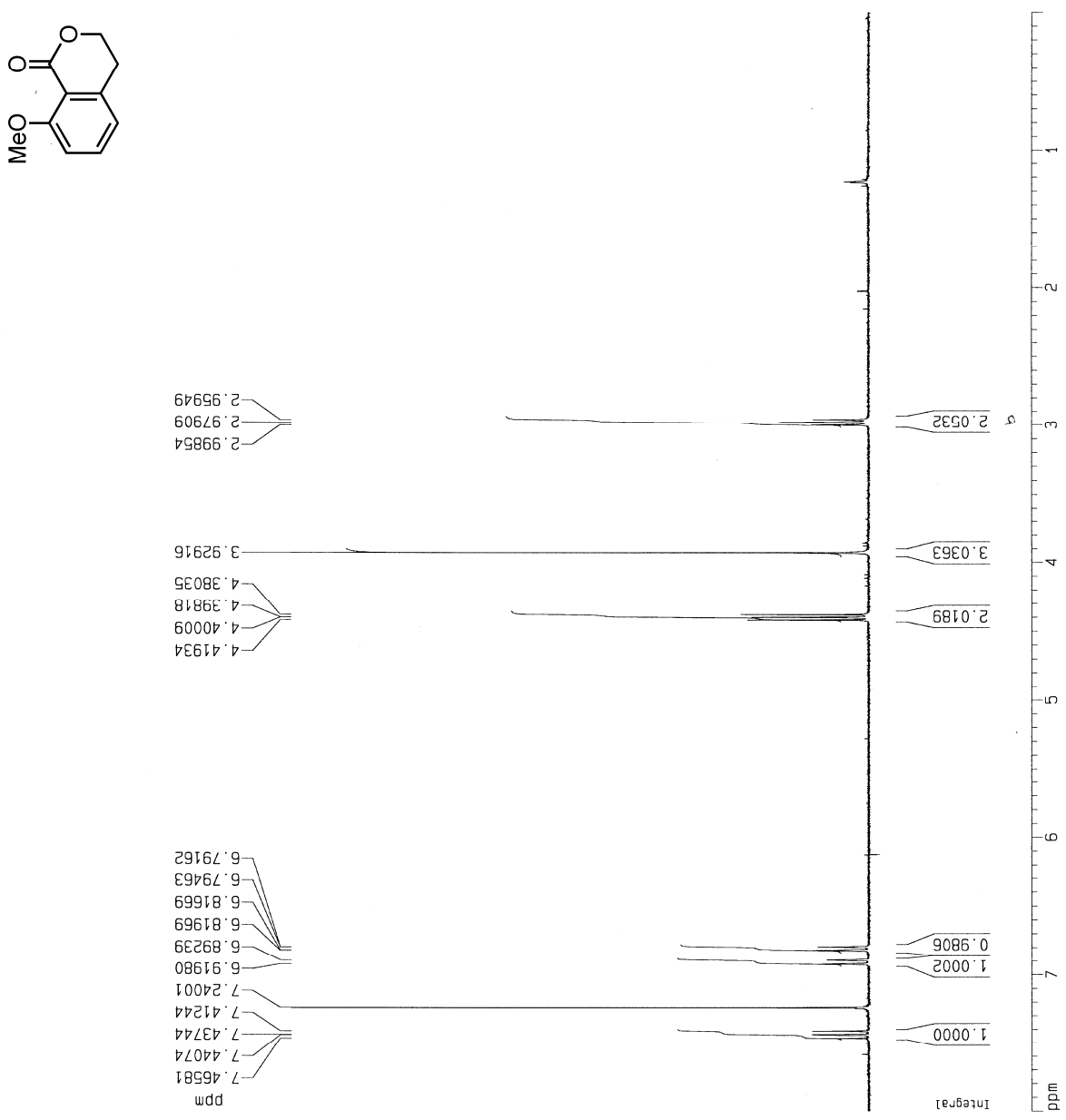
Water (78 μL, 78 μmol) and Dess-Martin periodinane[10] (50 mg, 117 μmol) were added to the above alcohol (41 mg, 78 μmol) in CH_2Cl_2 (2 mL), and the mixture was stirred at rt for 1 h.

Aqueous NaOH (1 M, 2 mL) was added to the reaction mixture which was then extracted with CH_2Cl_2 (3 x 2 mL). The residue was purified by flash column chromatography on silica gel (50:50 EtOAc/hexanes) to give aldehyde **25** as a white foam (40 mg, 98%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.79 (dd, *J* = 15.8, 7.6 Hz, 1H), 3.27 (dd, *J* = 15.8, 7.8 Hz, 1H), 3.59 (s, 5H), 3.70 (s, 3H), 3.76 (s, 3H), 4.78 (q, *J* = 7.7 Hz, 1H), 6.59 (s, 1H), 6.59 (d, *J* = 3.8 Hz, 1H), 6.71 (dd, *J* = 8.8, 3.1 Hz, 1H), 6.75-6.78 (m, 2H), 6.84 (d, *J* = 3.0 Hz, 1H), 6.87 (d, *J* = 7.5 Hz, 1H), 7.05 (d, *J* = 9.2 Hz, 1H), 7.18 (t, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.45 (d, *J* = 8.8 Hz, 1H), 9.69 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 38.2, 44.3, 55.3, 55.4, 55.7, 61.4, 69.9, 110.0, 112.7, 113.4, 115.4, 117.0, 118.1, 119.7, 126.1, 129.6, 131.0, 133.7, 135.7, 139.9, 144.7, 156.8, 159.3, 159.9, 169.8, 199.0; HRMS *m/z* calcd for $C_{27}H_{26}NO_5NaBr$ 546.0892 (MNa^+), found 546.0878.

3-(2-Bromo-5-methoxyphenyl)-5-methoxy-4a-(3-methoxyphenyl)-1,4a,9,9a-tetra-hydroindeno[2,1-b]pyridin-2-one (26). K_2CO_3 (16 mg, 1.114 mmol) was added to aldehyde **25** (62 mg, 0.118 mmol) in MeOH (10 mL) and the resulting mixture was heated at 60 ºC overnight. Concentration of the reaction mixture *in vacuo* gave a residue that was partitioned between brine (10 mL) and CH_2Cl_2 (10 mL). The aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (66:33:1 hexanes/EtOAc/conc. NH_4OH) to yield lactam **26** (59 mg, 98%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.11 (dd, *J* = 15.6, 6.6 Hz, 1H), 3.34 (dd, *J* = 15.8, 6.9 Hz, 1H), 3.60 (s, 3H), 3.73 (s, 3H), 3.75 (s, 3H), 4.17-4.22 (m, 1H), 6.26 (s, 1H), 6.69-6.80 (m, 5H), 6.89 (d, *J* = 3.0 Hz, 1H), 6.92 (t, *J* = 7.5 Hz, 1H), 7.18-7.24 (m, 1H), 7.29 (t, *J* = 7.7 Hz, 1H),7.42 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 40.9, 55.3, 55.4, 55.7, 60.6, 64.6, 110.1, 112.1, 113.2, 114, 6, 115.2, 117.3, 117.6, 119.3, 129.5, 130.0 (2C),

133.2, 134.7, 139.1, 141.1, 143.5, 145.0, 156.7, 158.9, 159.9, 163.7; HRMS *m/z* calcd for $C_{27}H_{42}NO_4NaBr$ 528.0786 (MNa^+), found 528.0783.

3-(2-Bromo-5-methoxyphenyl)-5-methoxy-4a-(3-methoxyphenyl)-2,4a,9,9a-tetra-hydro-1H-indeno[2,1-b]pyridine (3). $LiAlH_4$ (29.7 mg, 782 μmol) and $ZnCl_2$ (0.5 M in THF, 782 μL) were added to lactam **26** (33 mg, 65 μmol) in THF (5.0 mL) at 0 ºC and the mixture was allowed to gradually warm to rt over 1 h. After recooling the reaction mixture to 0 ºC, 10% H_2SO_4 (2 mL) was added slowly. The mixture was stirred for approximately 30 min until all of the solids had dissolved. The solution was basified with saturated aqueous $NaHCO_3$ and extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by preparative TLC (66:33:1 hexanes/EtOAc/conc. NH_4OH) to afford amine **3** as a white foam (24 mg, 75%): mp 58-61 ºC; IR (thin film) 2934, 1588, 1479, 1465, 1289, 1263, 1080, 1052, 1027 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.92 (dd, *J* = 16.1, 4.5 Hz, 1H), 3.15 (dd, *J* = 16.1, 7.0 Hz, 1H), 3.51 (d, *J* = 17.6 Hz, 1H), 3.61 (s, 3H), 3.62-3.67 (m, 1H), 3.74 (s, 3H), 3.75 (s, 3H), 3.79 (dd, *J* = 17.7, 2.0 Hz, 1H), 6.34 (s, 1H), 6.67 (dd, *J* = 8.7, 3.1 Hz, 1H), 6.71-6.75 (m, 4H), 6.80 (t, *J* = 2.1 Hz, 1H), 6.92 (d, *J* = 7.5 Hz, 1H), 7.16 (t, *J* = 7.9 Hz, 1H), 7.24 (t, *J* = 7.8 Hz, 1H), 7.41 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 36.3, 45.4, 55.4 (2C), 55.7, 66.7, 109.8, 111.5, 113.1, 113.3, 114.4, 116.2, 118.0, 119.7, 129.2, 129.3, 130.2, 132.6, 133.5 139.4, 143.8, 143.9, 147.6, 157.7, 159.0, 159.8; HRMS *m/z* calcd for $C_{27}H_{27}NO_3Br$ 492.1174 (MH^+), found 492.1194.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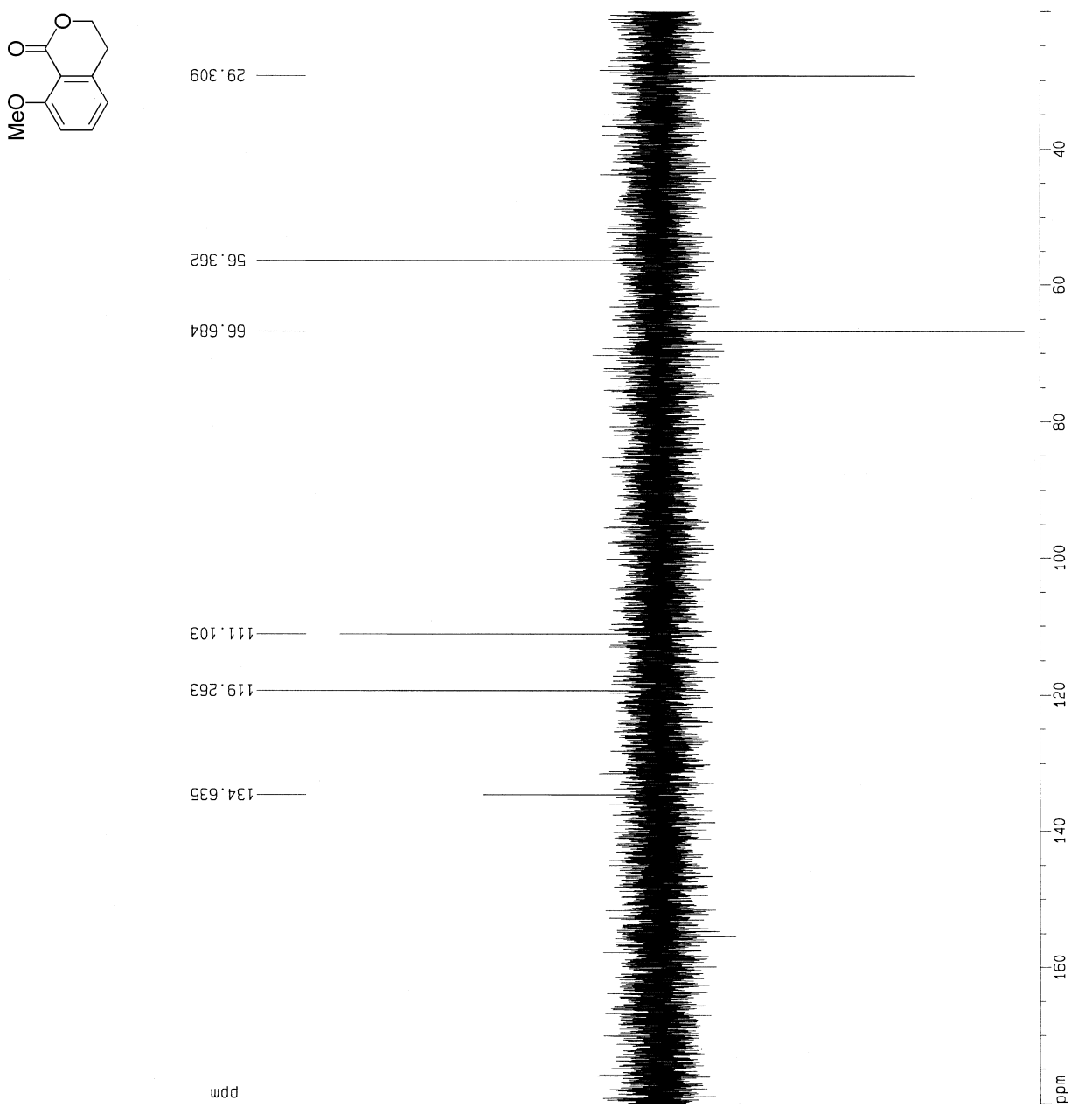
PULPROG zg30
TD 46294
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.133340 Hz
AQ 3.7498641 sec
RG 2048
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700159 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 2398.96 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40000 ppm/cm
HZCM 119.94801 Hz/cm
MeO
O
O
ppm
7.46581
7.44074
7.43744
7.41244
7.24001
6.91980
6.89239
6.81969
6.81669
6.79463
6.79162
4.41934
4.40009
4.39818
4.38035
3.92916
2.99854
2.97909
2.95949
Integral
0.9806
1.0002
1.0000
2.0189
3.0363
2.0532
ppm

Current Data Parameters
NAME jhj-04-29-03
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030429
Time 16.10
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AG 1.7433076 sec
RG 724.1
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023584 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 160.000 ppm
F1 13572.42 Hz
F2P 20.000 ppm
F2 1508.05 Hz
PPMCM 8.00000 ppm/cm
HZCM 603.21887 Hz/cm
MeO
29.309
56.362
66.678
76.803
77.227
77.650
111.107
119.262
134.627
142.619
ppm
40
60
80
100
120
140
160
ppm


MeO
O
O
29.309
56.362
66.684
111.103
119.263
134.635
ppm
ppm
Current Data Parameters
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EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030429
Time 16.30
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 93
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D2 0.00357143 sec
D12 0.00002000 sec
DELTA 0.00006688 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
P2 10.80 usec
PL1 -6.00 dB
SFO1 75.4058817 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.40 usec
P4 24.80 usec
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023584 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
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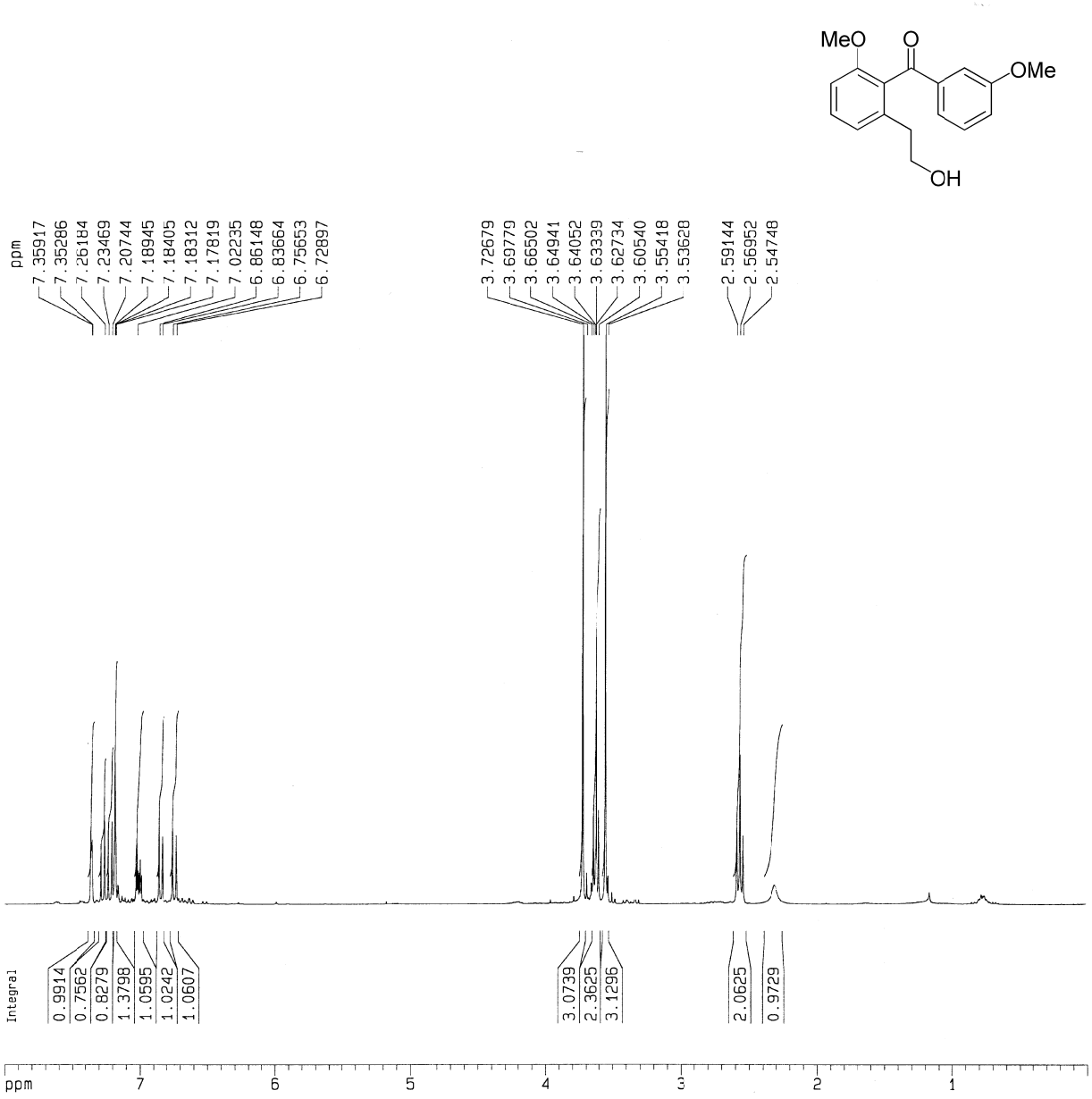
F1 13572.42 Hz
F2P 20.000 ppm
F2 1508.05 Hz
PPMCM 8.00000 ppm/cm
HZCM 603.21887 Hz/cm

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NAME        jhj-03-29-03
EXPNO                  1
PROCNO                 1

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Time               15.43
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PULPROG             zg30
TD                 46294
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           6172.839 Hz
FIDRES        0.133340 Hz
AQ            3.7498641 sec
RG                  71.8
DW               81.000 usec
DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec

============ CHANNEL f1 ========
NUC1                  1H
P1                11.70 usec
PL1                0.00 dB
SFO1        299.8718518 MHz

F2 - Processing parameters
SI                 32768
SF          299.8700347 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                20.00 cm
F1P               8.000 ppm
F1              2398.96 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          0.40000 ppm/cm
HZCM         119.94801 Hz/cm
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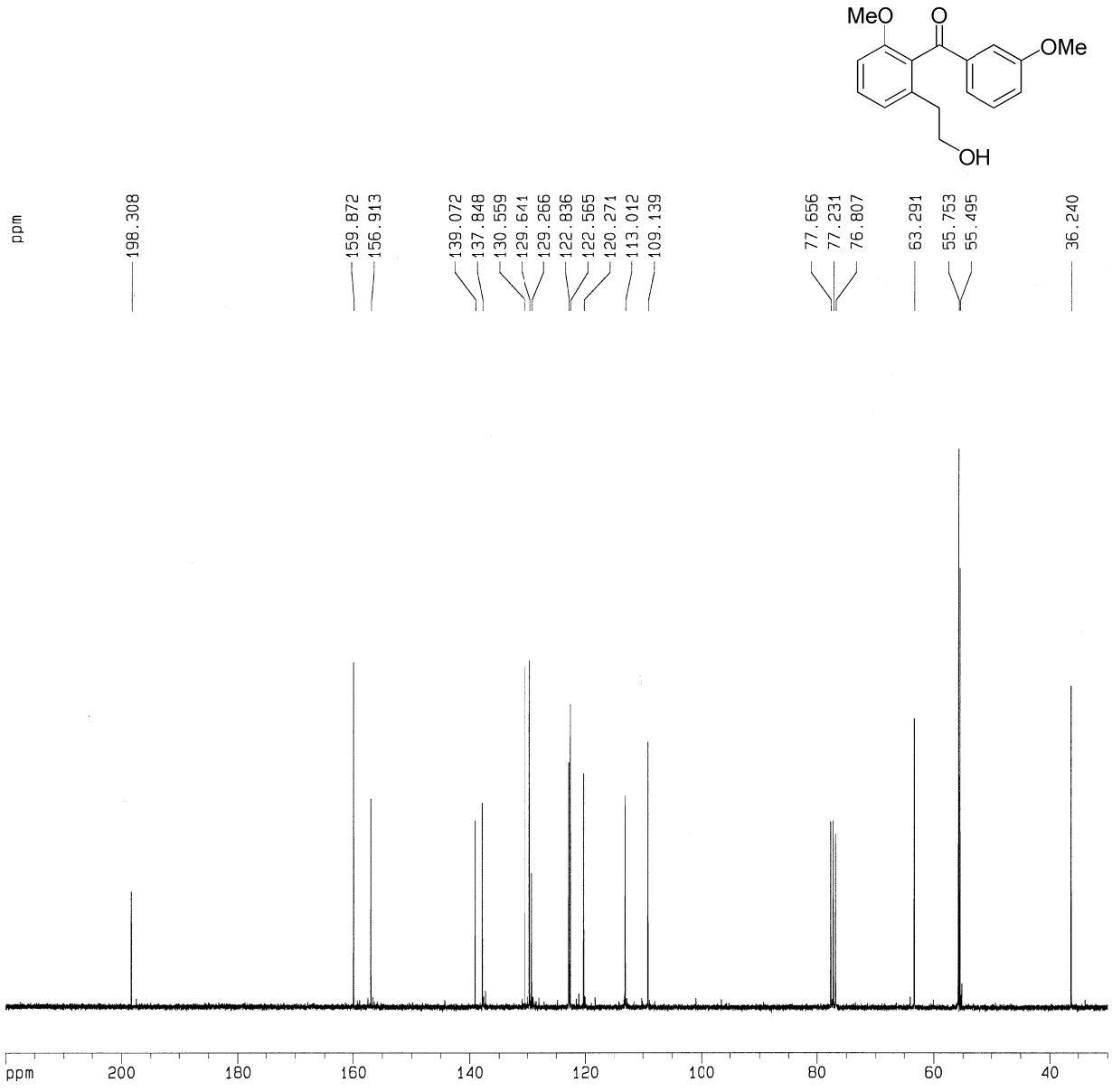

Current Data Parameters

NAME	jhj-03-29-03
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

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Time	15.50
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	900
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	1290.2
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

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

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

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

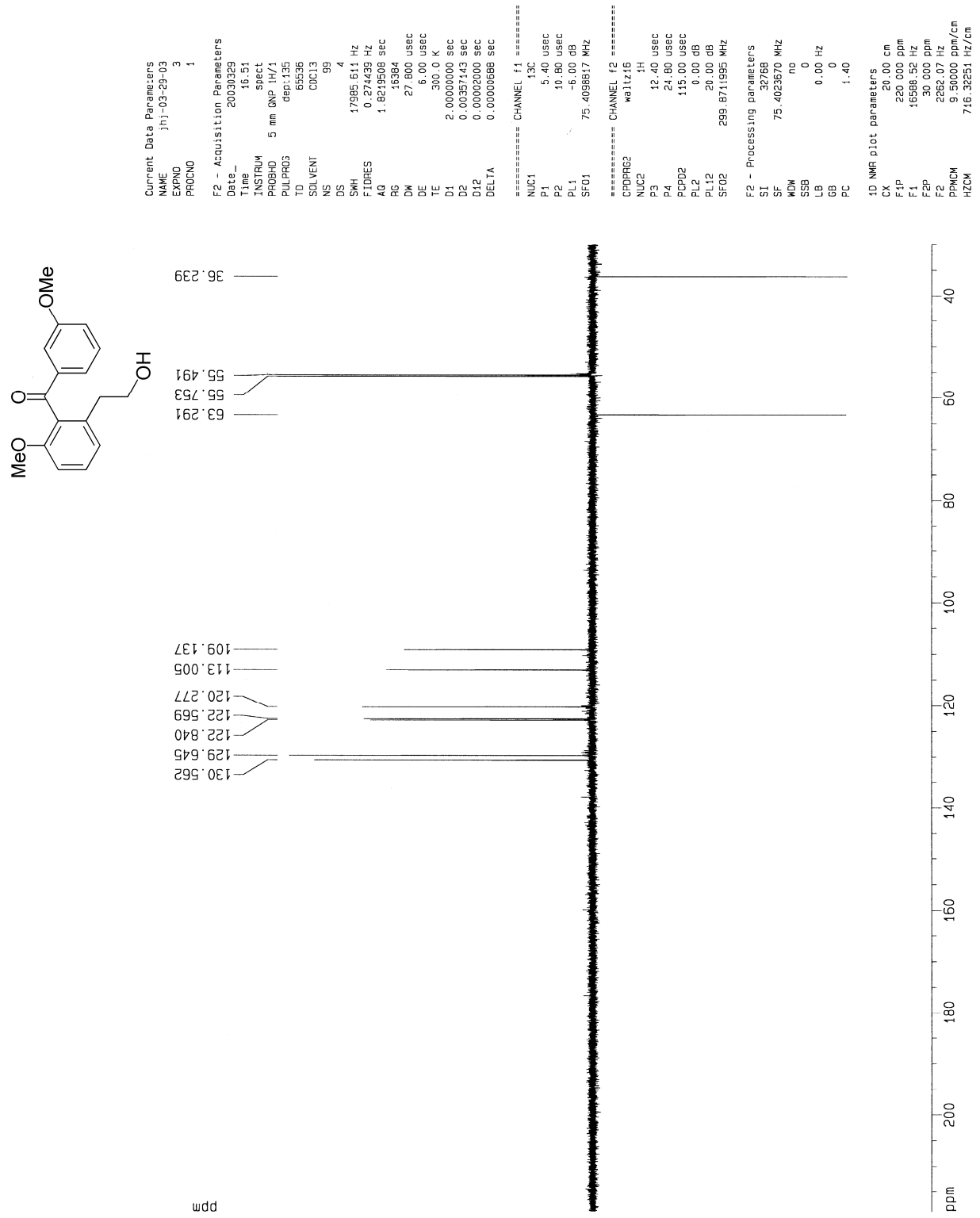
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

SI	32768
SF	75.4023670 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	16588.52 Hz
F2P	30.000 ppm
F2	2262.07 Hz
PPMCM	9.50000 ppm/cm
HZCM	716.32251 Hz/cm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NAME jhj-03-29-03
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
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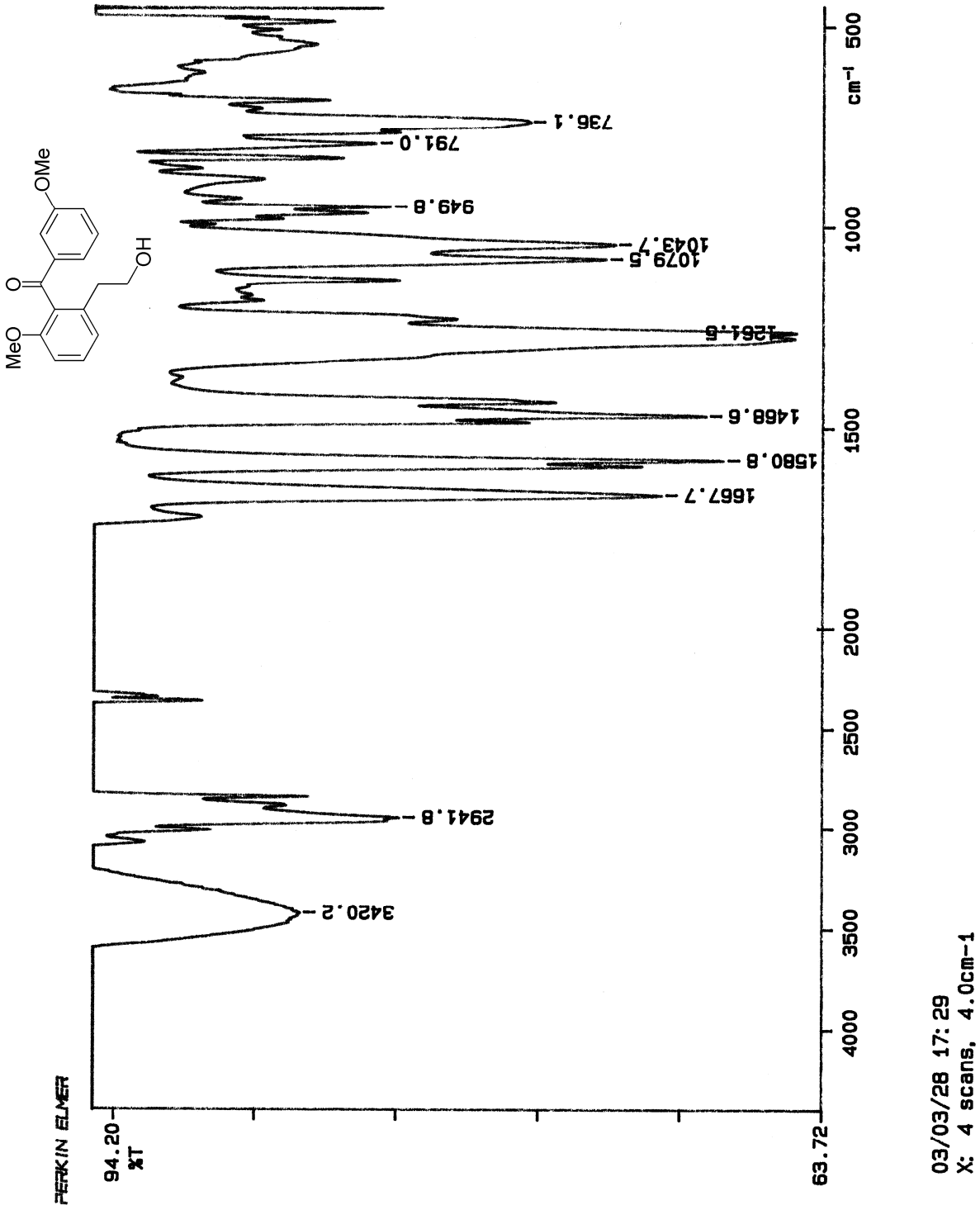
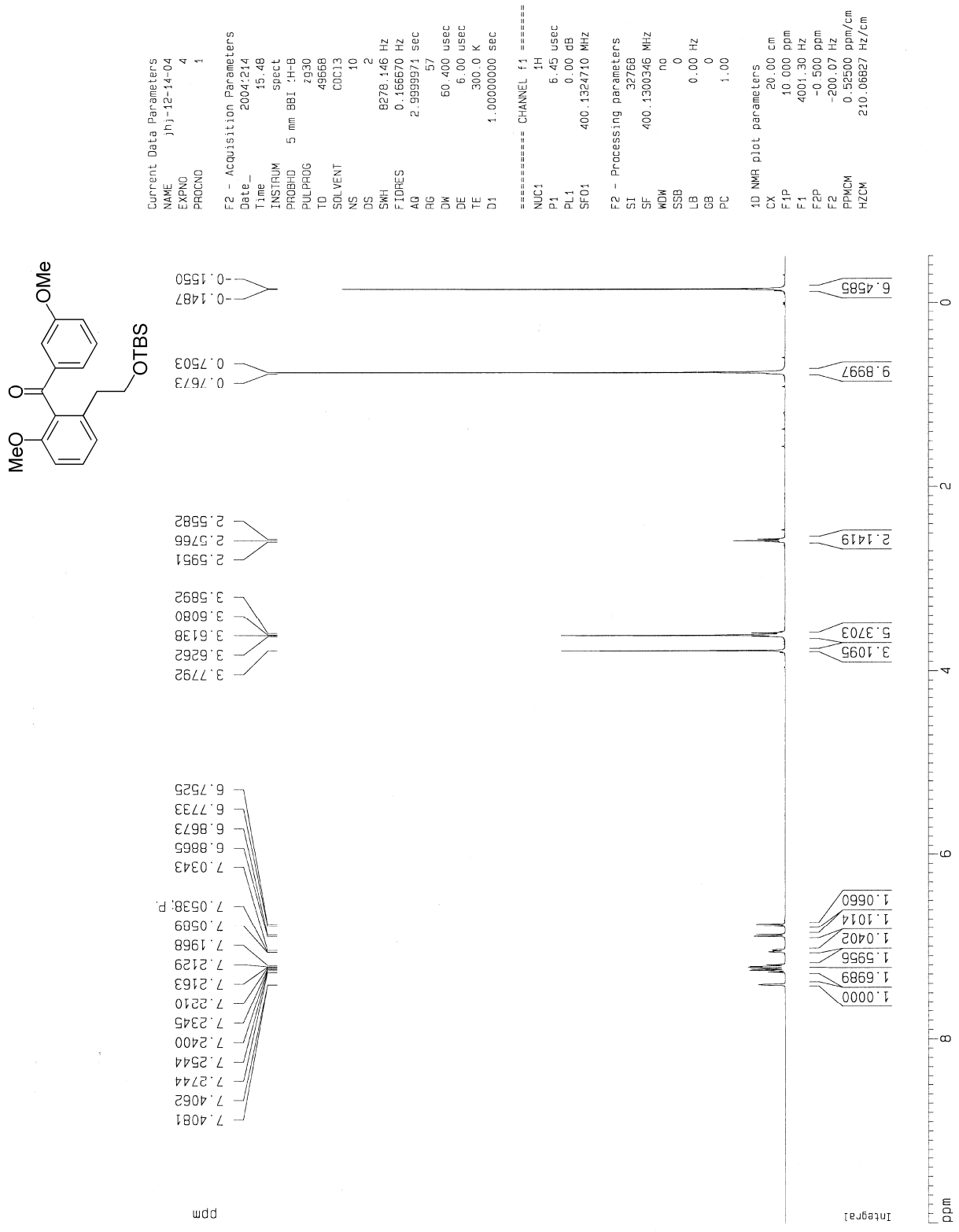
Time 16.51
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 99
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D2 0.00357143 sec
D12 0.00002000 sec
DELTA 0.00000688 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
P2 10.80 usec
PL1 -6.00 dB
SFO1 75.4098817 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.40 usec
P4 24.80 usec
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023670 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16588.52 Hz
F2P 30.000 ppm
F2 2262.07 Hz
PPMCM 9.50000 ppm/cm
HZCM 716.32251 Hz/cm
OMe
O
OH
MeO
36.239
55.491
55.753
63.291
109.137
113.005
120.277
122.569
122.840
129.645
130.562
ppm
200
180
160
140
120
100
80
60
40


PERKIN ELMER
94.20
%T
63.72
4000
3500
3000
2500
2000
1500
1000
500
cm⁻¹
3420.2
2941.8
1667.7
1580.8
1468.6
1261.6
1079.5
1043.7
949.8
791.0
736.1
OMe
MeO
O
OH
03/03/28 17:29
X: 4 scans, 4.0cm-1


OMe
O
OTBS
MeO
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EXPNO 4
PROCNO 1
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Date_ 2004:214
Time 15.48
INSTRUM spect
PROBHD 5 mm BBI '1H-B
PULPROG zg30
TD 49568
SOLVENT CDCl3
NS 10
DS 2
SWH 8278.146 Hz
FIDRES 0.166670 Hz
AQ 2.9999971 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300346 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06827 Hz/cm
-0.1550
-0.1487
0.7503
0.7473
2.5582
2.5766
2.5951
3.5892
3.6080
3.6138
3.6262
3.7792
7.4081
7.4062
7.2744
7.2544
7.2400
7.2345
7.2210
7.2163
7.2129
7.1968
7.0509
7.0538
7.0343
6.8665
6.8673
6.7733
6.7525
6.4585
9.8997
2.1419
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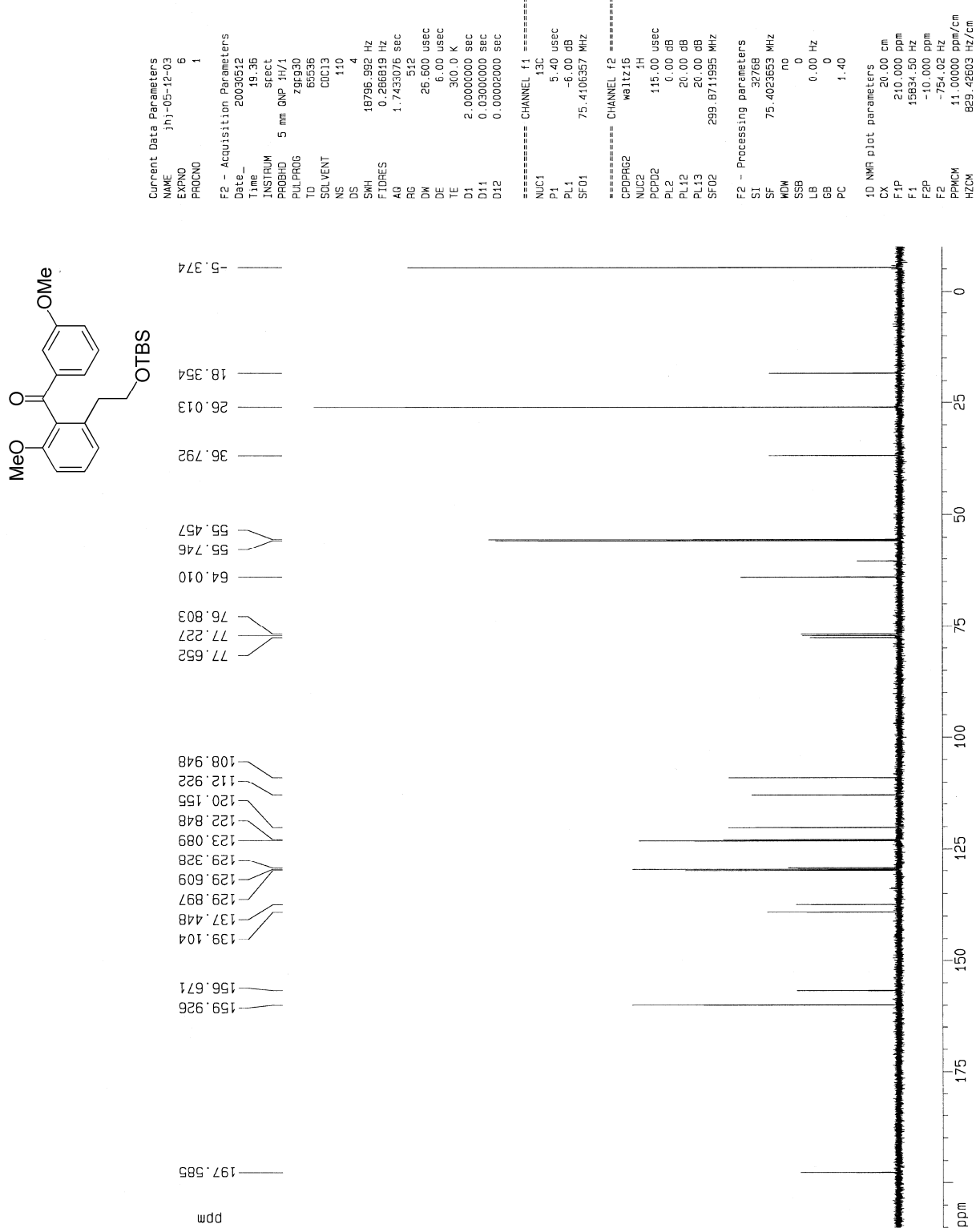
1.0000
1.6989
1.5956
1.0402
1.1014
1.0660
ppm
Integral
ppm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EXPNO 6
PROCNO 1
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Time 19.36
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 110
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023653 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 210.000 ppm
F1 15834.50 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 11.00000 ppm/cm
HZCM 829.42603 Hz/cm
-5.374
18.354
26.013
36.792
55.457
55.746
64.010
76.803
77.227
77.652
108.948
112.922
120.155
122.848
123.089
129.328
129.609
129.897
137.448
139.104
156.671
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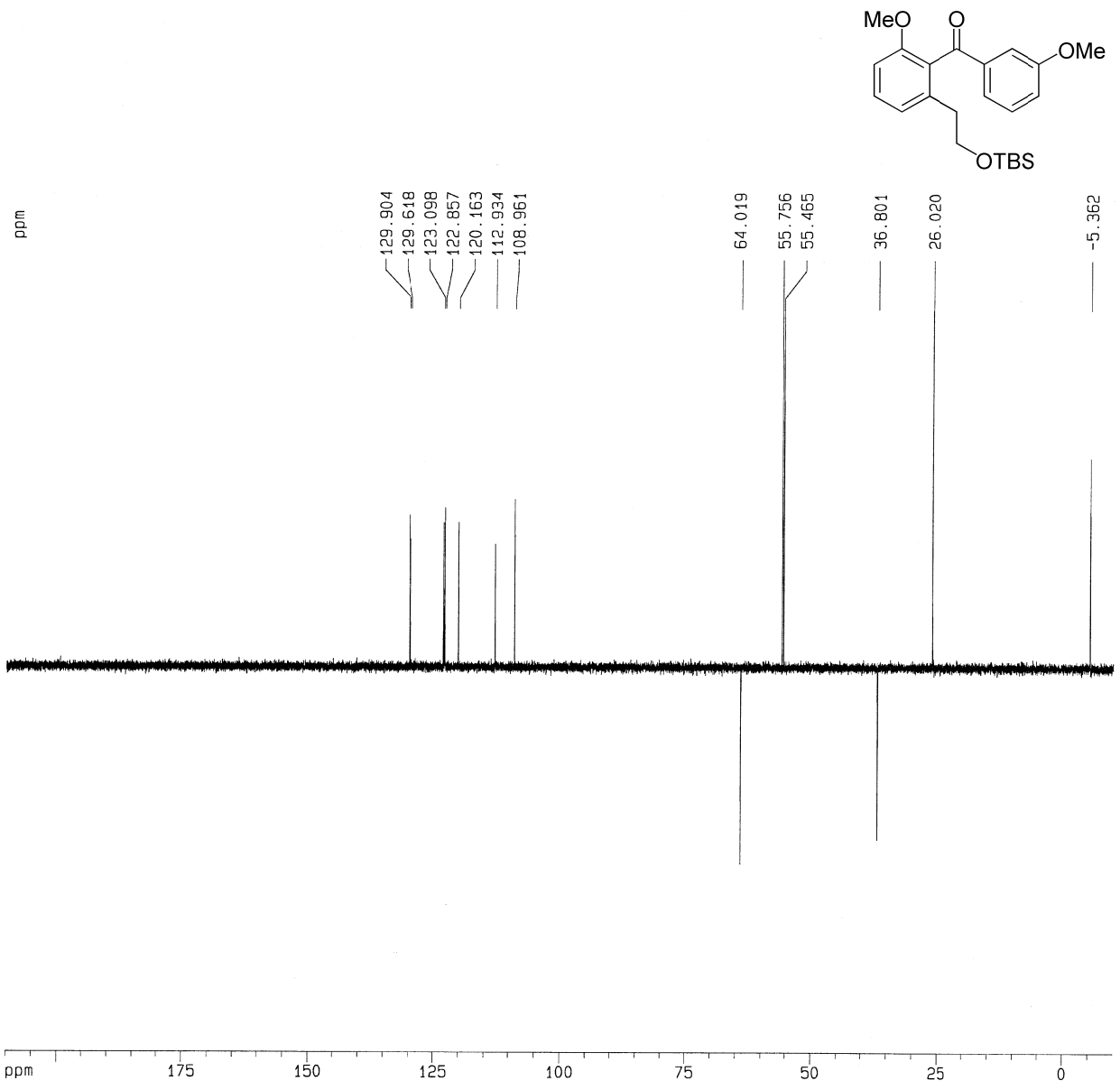
ppm
0
25
50
75
100
125
150
175
ppm
OMe
O
OTBS
MeO

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EXPNO                  7
PROCNO                 1

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Time               19.39
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PULPROG         dept135
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                16384
DW               27.800 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
D2            0.00357143 sec
D12           0.00002000 sec
DELTA         0.00000688 sec

============ CHANNEL f1 ========
NUC1                13C
P1                 5.40 usec
P2                10.80 usec
PL1               -6.00 dB
SFO1          75.4098817 MHz

============ CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
P3                12.40 usec
P4                24.80 usec
PCPD2            115.00 usec
PL2               0.00 dB
PL12             20.00 dB
SFO2         299.8711995 MHz

F2 - Processing parameters
SI                 32768
SF            75.4023645 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              210.000 ppm
F1             15834.50 Hz
F2P              -10.000 ppm
F2              -754.02 Hz
PPMCM          11.00000 ppm/cm
HZCM          829.42603 Hz/cm
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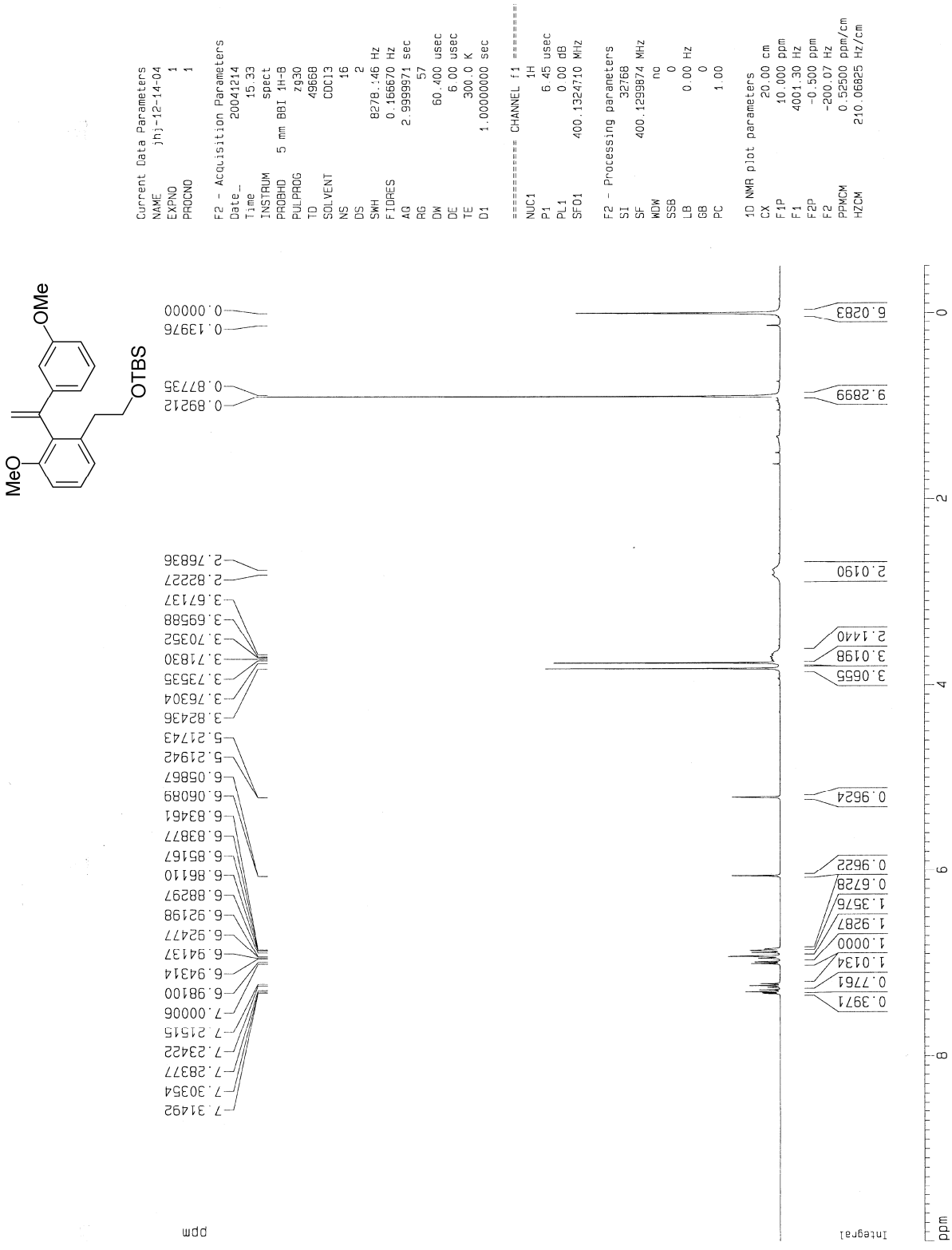
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OTBS
MeO
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EXPNO 1
PROCNO 1
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Date_ 20041214
Time 15.33
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 49668
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.166670 Hz
AQ 2.9999971 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1299874 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm
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0.87735
0.89212
2.76836
2.82227
3.67137
3.69588
3.70352
3.71830
3.73535
3.76304
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5.21942
6.05867
6.06089
6.83461
6.83877
6.85167
6.86110
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9.2899
2.0190
2.1440
3.0198
3.0655
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1.3576
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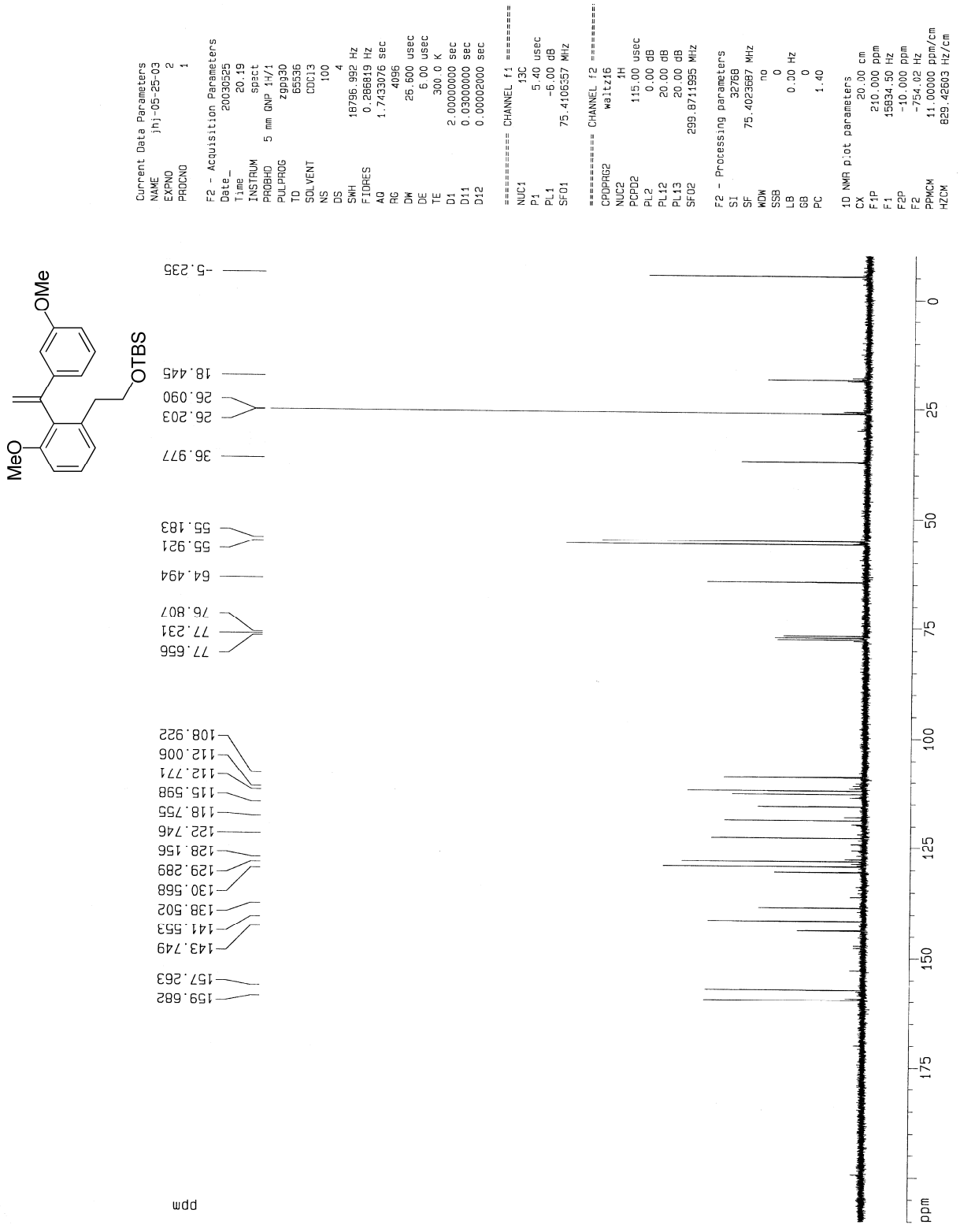
0.7761
0.3971
ppm
Integral
ppm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OTBS
MeO
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55.183
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77.231
77.656
108.922
112.006
112.771
115.598
118.755
122.746
128.156
129.289
130.568
138.502
141.553
143.749
157.263
159.682
ppm
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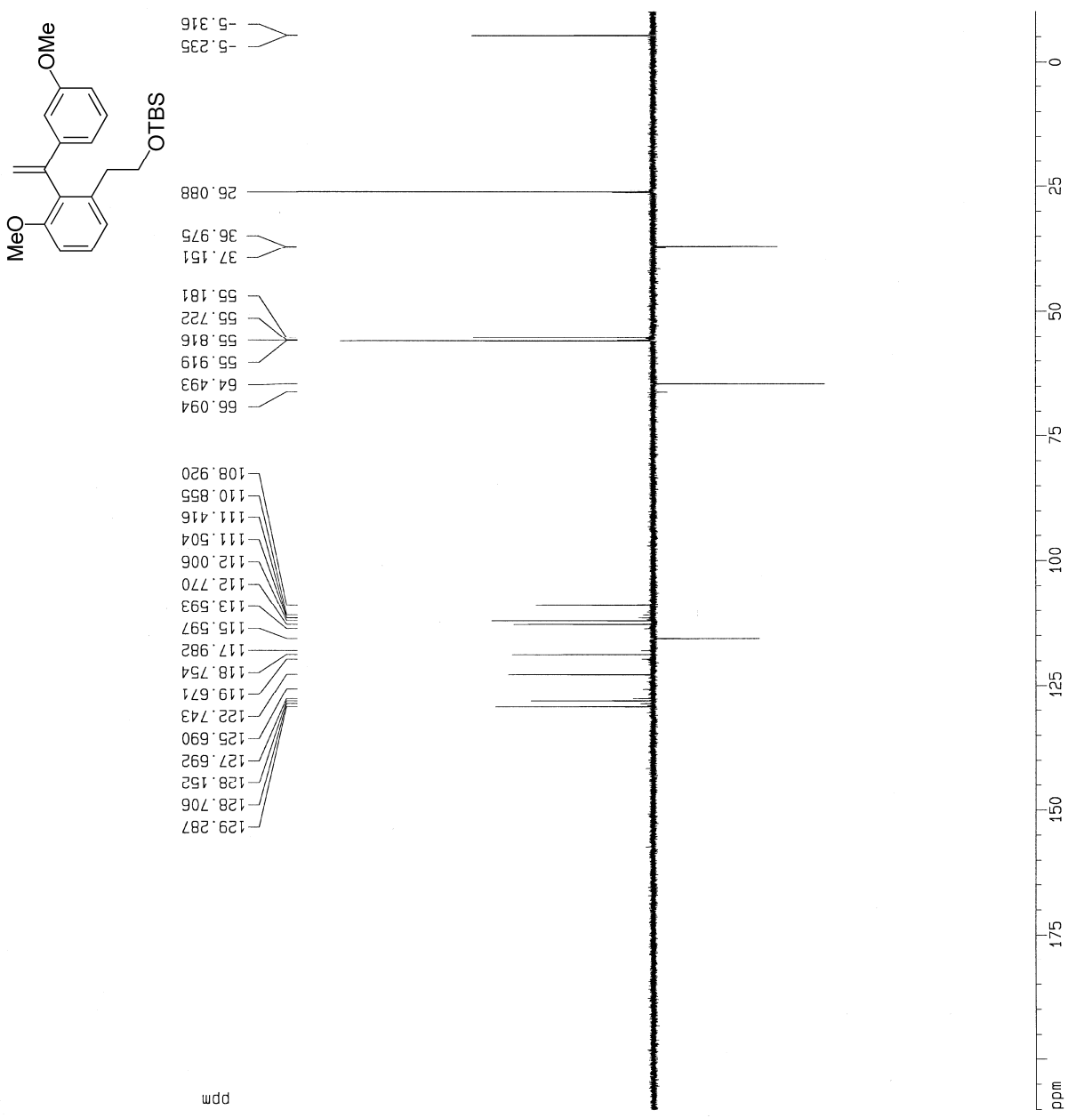
NAME jhj-05-25-03
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030525
Time 20.19
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 4096
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023687 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 210.000 ppm
F1 15834.50 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 11.00000 ppm/cm
HZCM 829.42603 Hz/cm
0
25
50
75
100
125
150
175
ppm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OTBS
MeO
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-5.235
26.088
36.975
37.151
55.181
55.722
55.816
55.919
64.493
66.094
108.920
110.855
111.416
111.504
112.006
112.770
113.593
115.597
117.982
118.754
119.671
122.743
125.690
127.692
128.152
128.706
129.287
ppm
Current Data Parameters
NAME jnj-05-25-03
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030525
Time 20.24
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 50
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D2 0.00357143 sec
D12 0.00002000 sec
DELTA 0.00000688 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
P2 10.80 usec
PL1 -6.00 dB
SFO1 75.4098817 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.40 usec
P4 24.80 usec
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023698 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 210.000 ppm
F1 15834.50 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 11.00000 ppm/cm
HZCM 829.42603 Hz/cm
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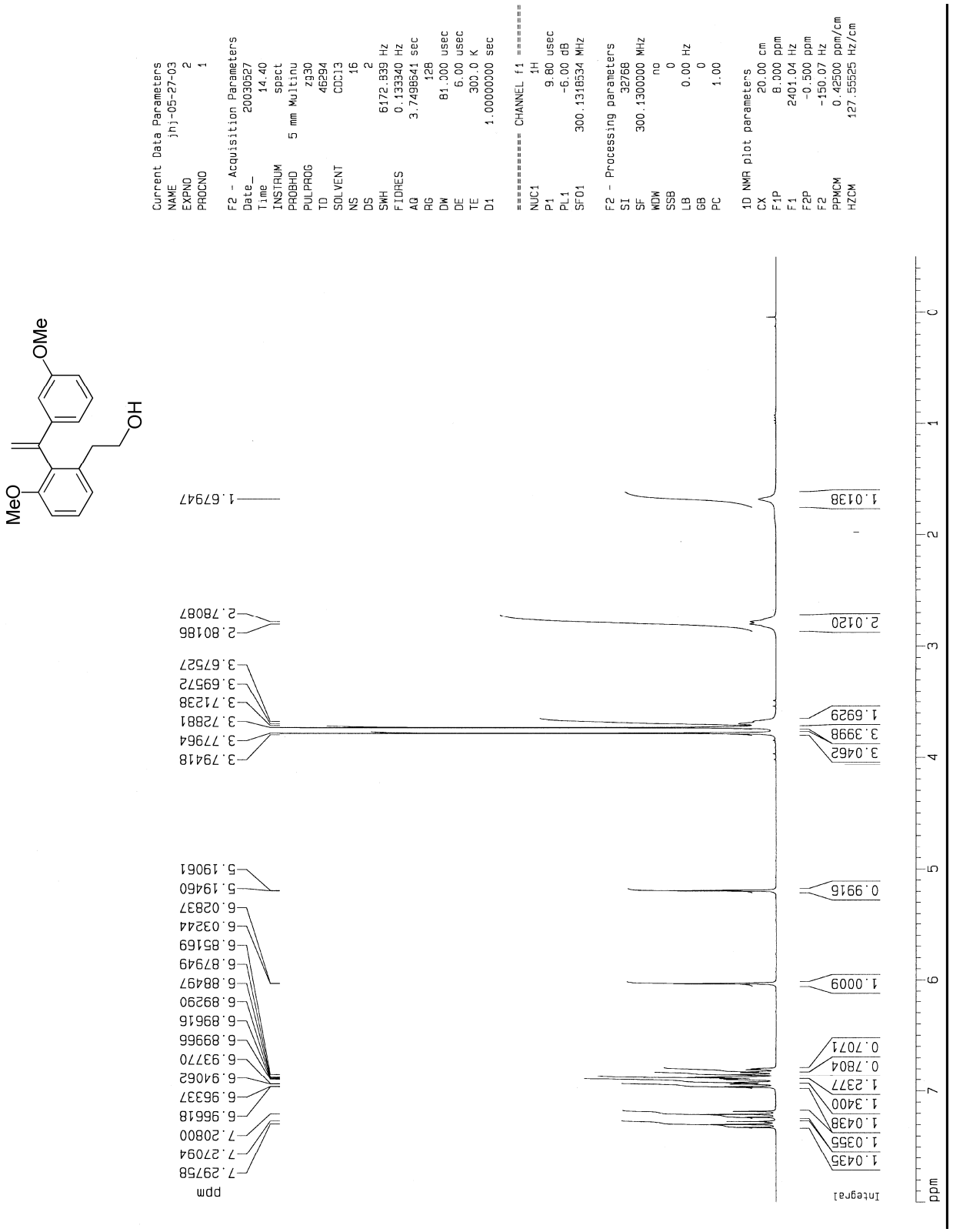
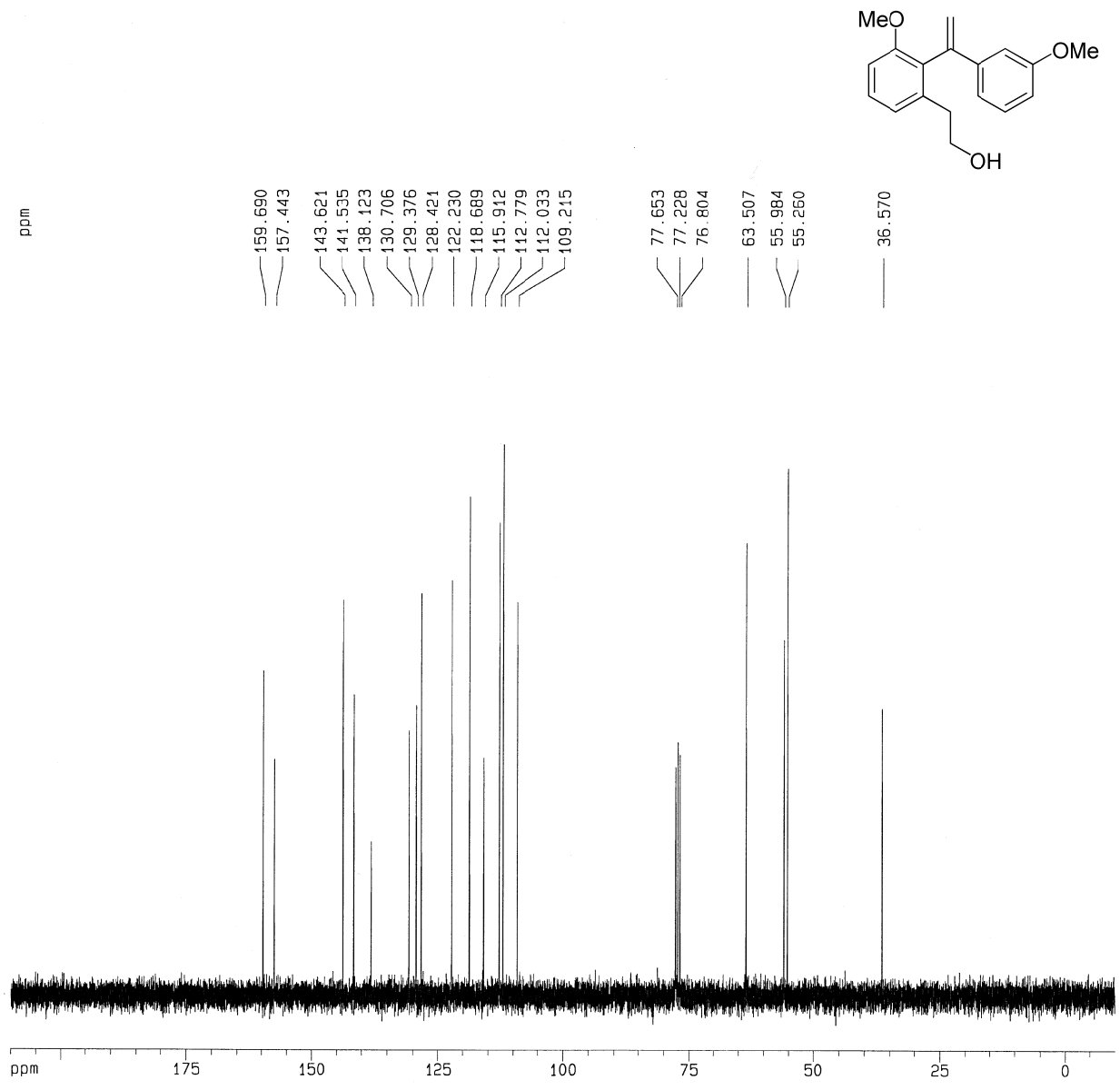
25
50
75
100
125
150
175
ppm


OMe
OH
MeO
Current Data Parameters
NAME jhj-05-27-03
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030527
Time 14.40
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 46294
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.133340 Hz
AQ 3.7498641 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 2401.04 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 127.55525 Hz/cm
1.67947
2.78087
2.80186
3.67527
3.69572
3.71238
3.72881
3.77964
3.79418
5.19061
5.19460
6.02837
6.03244
6.85169
6.87949
6.88497
6.89290
6.89616
6.89966
6.93770
6.94062
6.96337
6.96618
7.20800
7.27094
7.29758
ppm
1.0138
2.0120
1.6929
3.3998
3.0462
0.9916
1.0009
0.7071
0.7804
1.2377
1.3400
1.0438
1.0355
1.0435
Integral
ppm

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Current Data Parameters
NAME        jhj-05-27-03
EXPNO                  3
PROCNO                 1

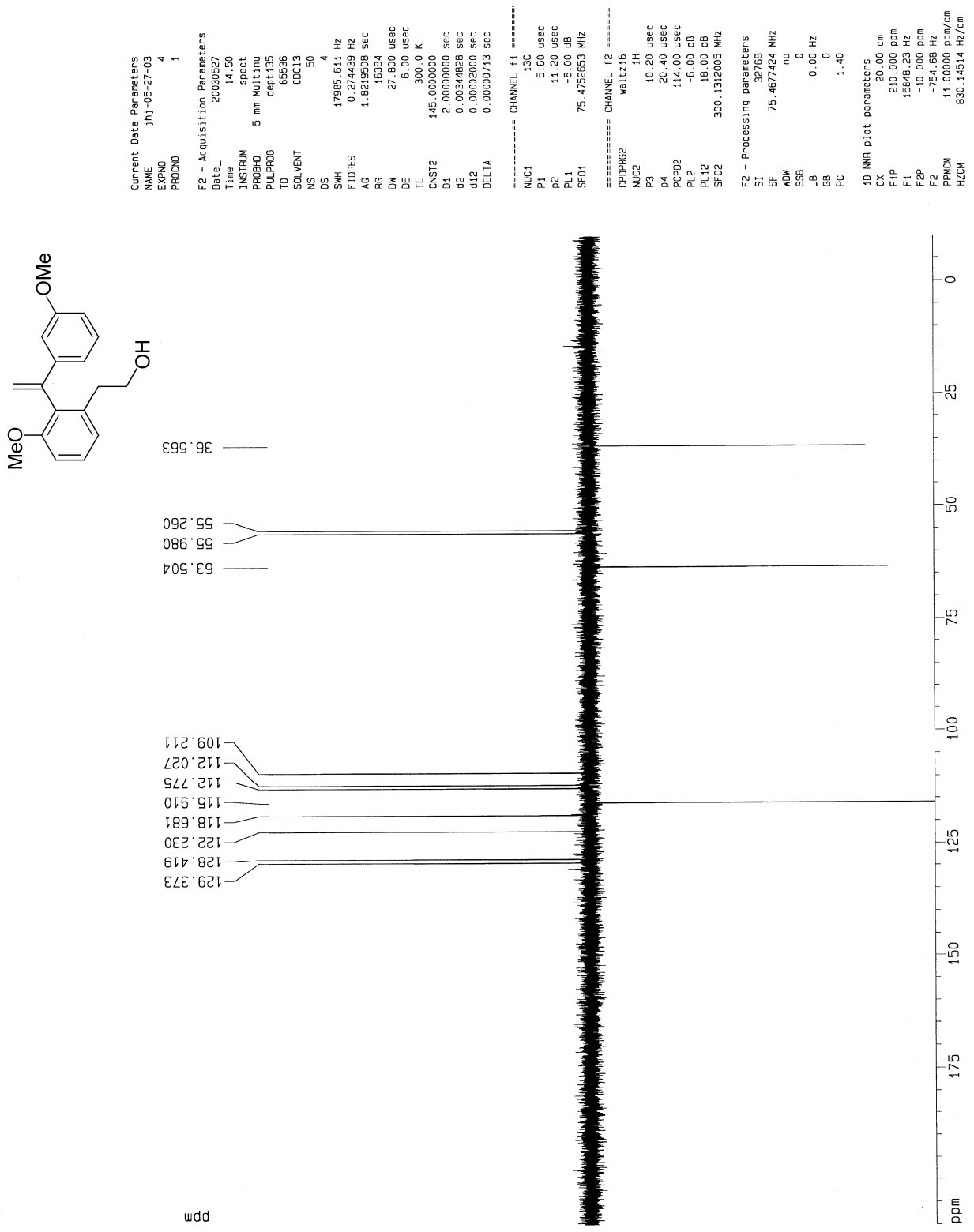
F2 - Acquisition Parameters
Date_           20030527
Time               14.46
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    2
SWH           18832.393 Hz
FIDRES         0.287360 Hz
AQ             1.7400308 sec
RG                13004
DW               26.550 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                 5.60 usec
PL1               -6.00 dB
SFO1         75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2           114.00 usec
PL2              -6.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677421 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               210.000 ppm
F1              15848.23 Hz
F2P               -10.000 ppm
F2              -754.68 Hz
PPMCM          11.00000 ppm/cm
HZCM          830.14514 Hz/cm
```



Current Data Parameters
NAME jhj-05-27-03
EXPNO 4
PROCNO 1

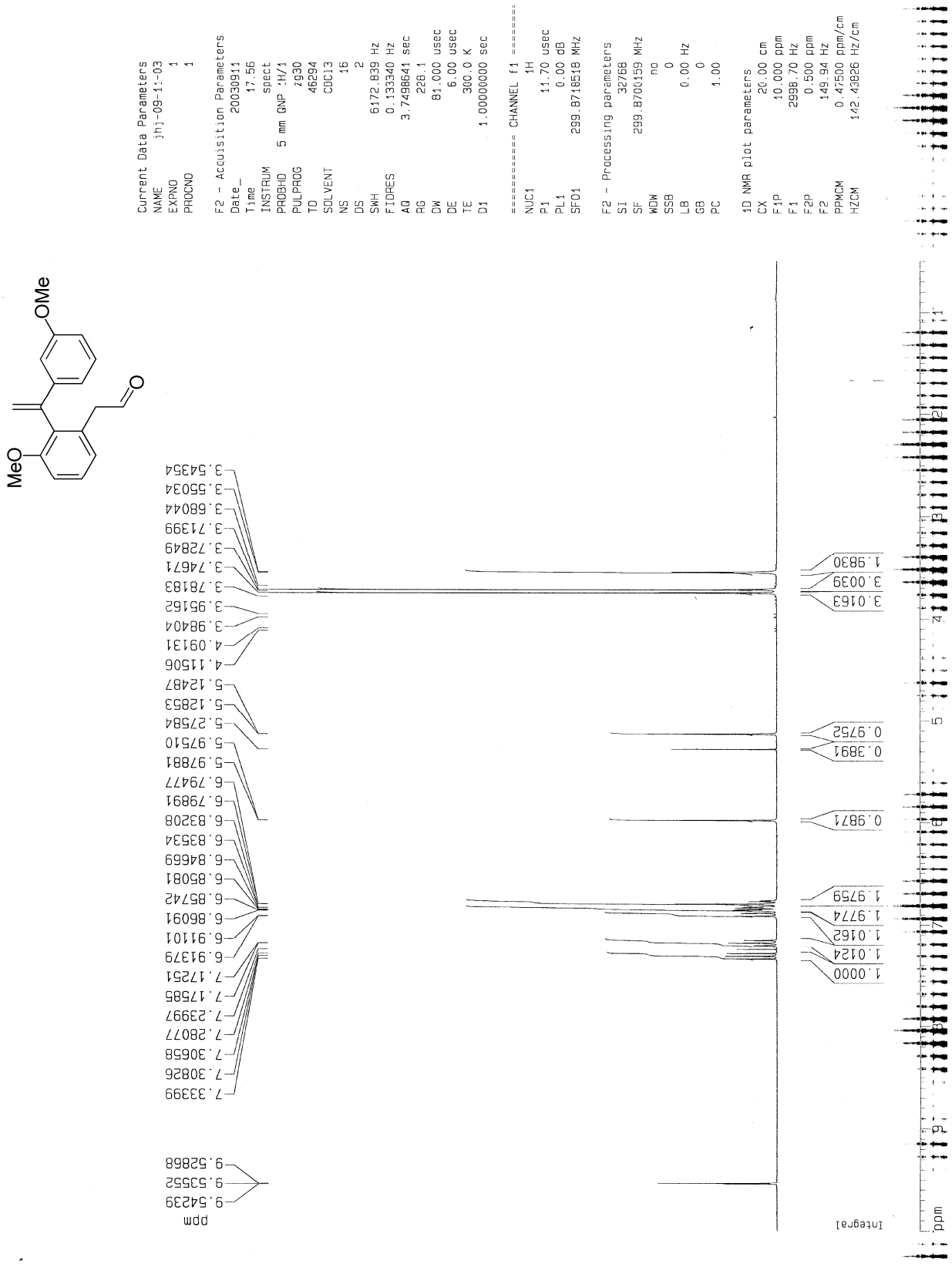
F2 - Acquisition Parameters
Date_ 20030527
Time 14.50
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 50
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000713 sec

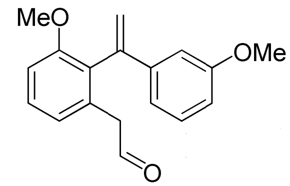
======== CHANNEL f1 ========
NUC1 13C
P1 5.60 usec
p2 11.20 usec
PL1 -6.00 dB
SFO1 75.4752653 MHz

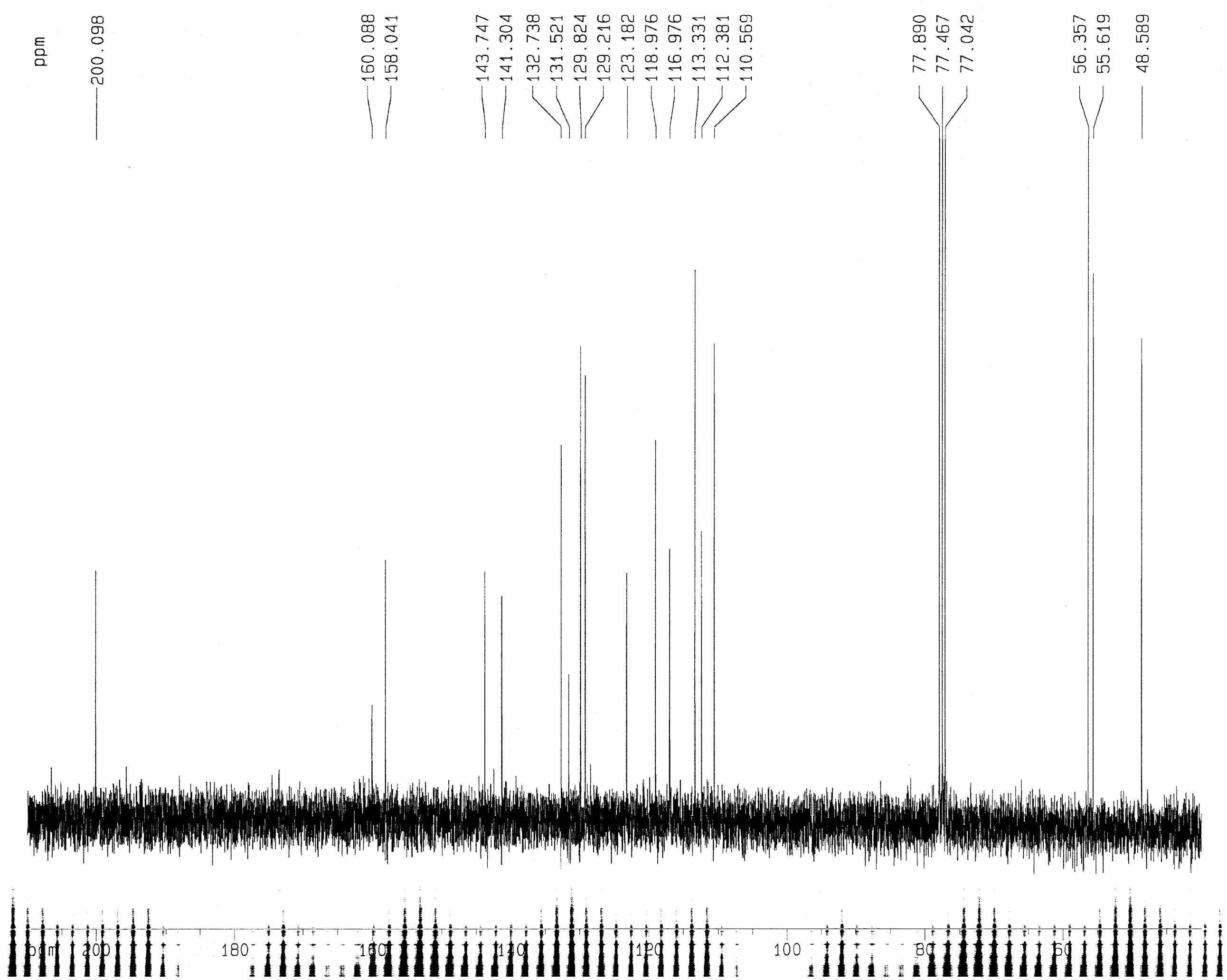
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 10.20 usec
p4 20.40 usec
PCPD2 114.00 usec
PL2 -6.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677424 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 210.000 ppm
F1 15848.23 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14514 Hz/cm


OMe
MeO
O
Current Data Parameters
NAME jhj-09-11-03
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030911
Time 17.56
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 46294
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.133340 Hz
AQ 3.7498641 sec
RG 228.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700159 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 2998.70 Hz
F2P 0.500 ppm
F2 149.94 Hz
PPMCM 0.47500 ppm/cm
HZCM 142.43826 Hz/cm
9.54239
9.53552
9.52868
7.33399
7.30826
7.30658
7.28077
7.23997
7.17585
7.17251
6.91379
6.91101
6.86091
6.85742
6.85081
6.84669
6.83534
6.83208
6.79891
6.79477
5.97881
5.97510
5.27584
5.12853
5.12487
4.11506
4.09131
3.98404
3.95162
3.78183
3.74671
3.72849
3.71399
3.68044
3.55034
3.54354
1.0000
1.0124
1.0162
1.9774
1.9759
0.9871
0.3891
0.9752
3.0163
3.0039
1.9830
ppm
Integral





Current Data Parameters
NAME jhj-09-11-03
EXPNO 2
PROCNO 1

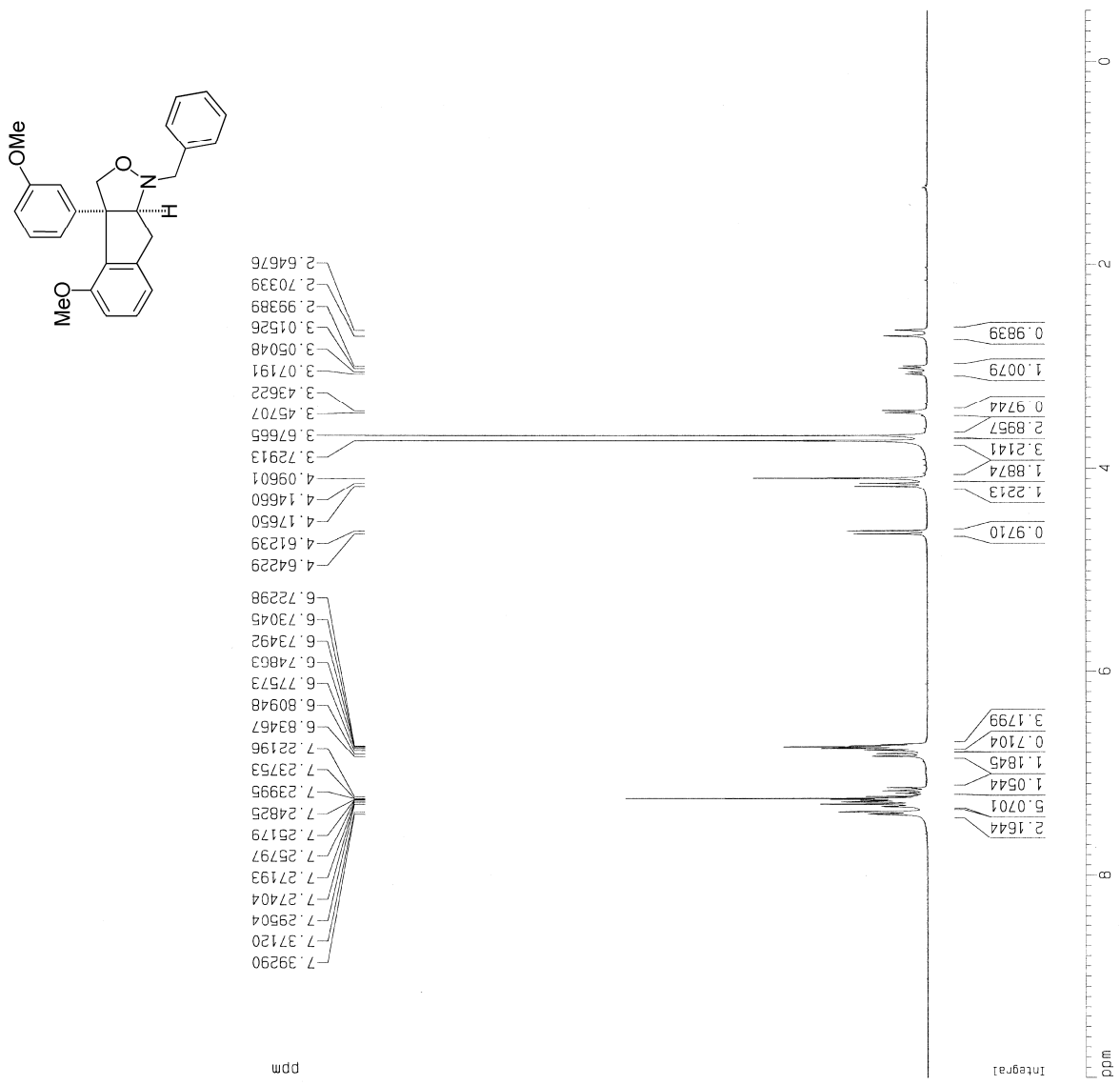
F2 - Acquisition Parameters
Date_ 20030911
Time 18.01
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

============ CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz

============ CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz

F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 210.000 ppm
F1 15834.49 Hz
F2P 40.000 ppm
F2 3015.09 Hz
PPMCM 8.50000 ppm/cm
HZCM 640.91992 Hz/cm


OMe
MeO
H
N
O
Current Data Parameters
NAME jhj-03-05-06
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060305
Time 20.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 724.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8716518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700159 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 2998.70 Hz
F2P -0.500 ppm
F2 -149.94 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.43176 Hz/cm
7.39290
7.37120
7.29504
7.27404
7.27193
7.25797
7.25179
7.24825
7.23995
7.23753
7.22196
6.83467
6.80948
6.77573
6.74603
6.73492
6.73045
6.72298
4.64229
4.61239
4.17650
4.14660
4.09601
3.72913
3.67665
3.45707
3.43622
3.07191
3.05048
3.01526
2.99389
2.70339
2.64676
0.9839
1.0079
0.9744
2.8957
3.2141
1.8874
1.2213
0.9710
3.1799
0.7104
1.1845
1.0544
5.0701
2.1644
Integral
ppm
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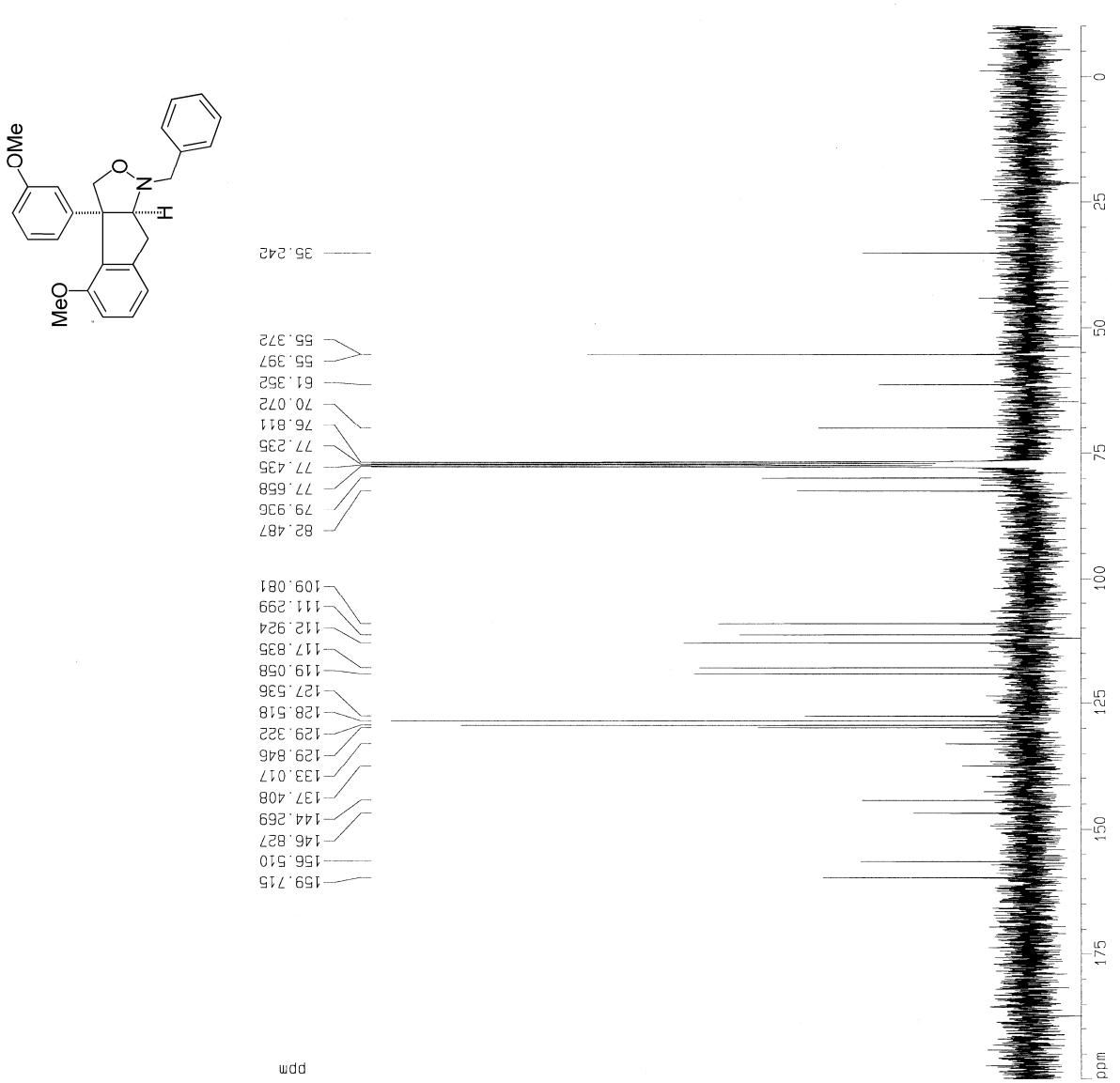
2
4
6
8


OMe
MeO
H
O
N
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77.235
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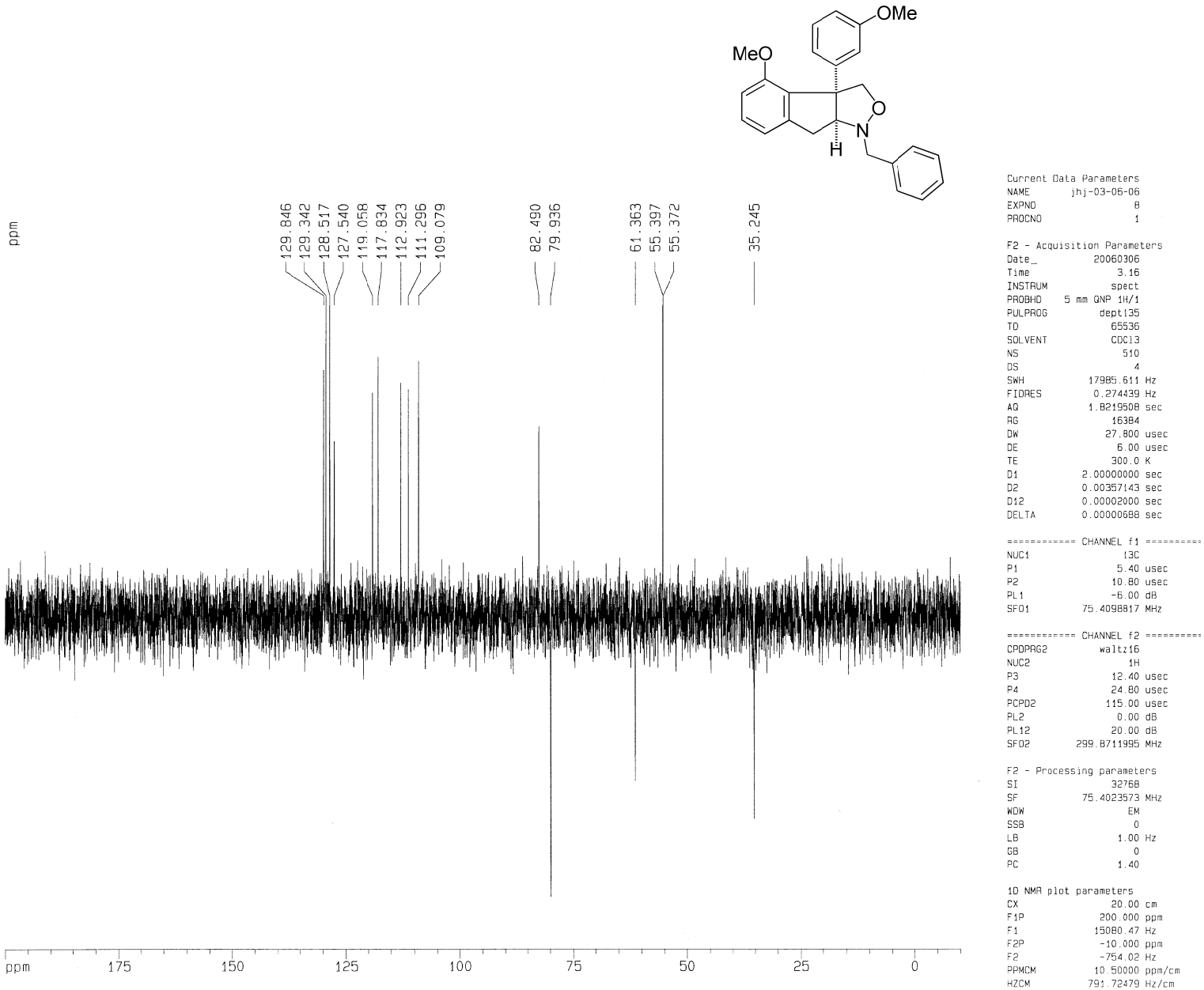
77.658
79.936
82.487
109.081
111.299
112.924
117.835
119.058
127.536
128.518
129.322
129.846
133.017
137.408
144.269
146.827
156.510
159.715
ppm
0
25
50
75
100
125
150
175
ppm
Current Data Parameters
NAME jhj-03-06-06
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060306
Time 1.10
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023572 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15080.47 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 10.50000 ppm/cm
HZCM 791.72479 Hz/cm



Current Data Parameters
NAME jhj-03-06-06
EXPNO 8
PROCNO 1

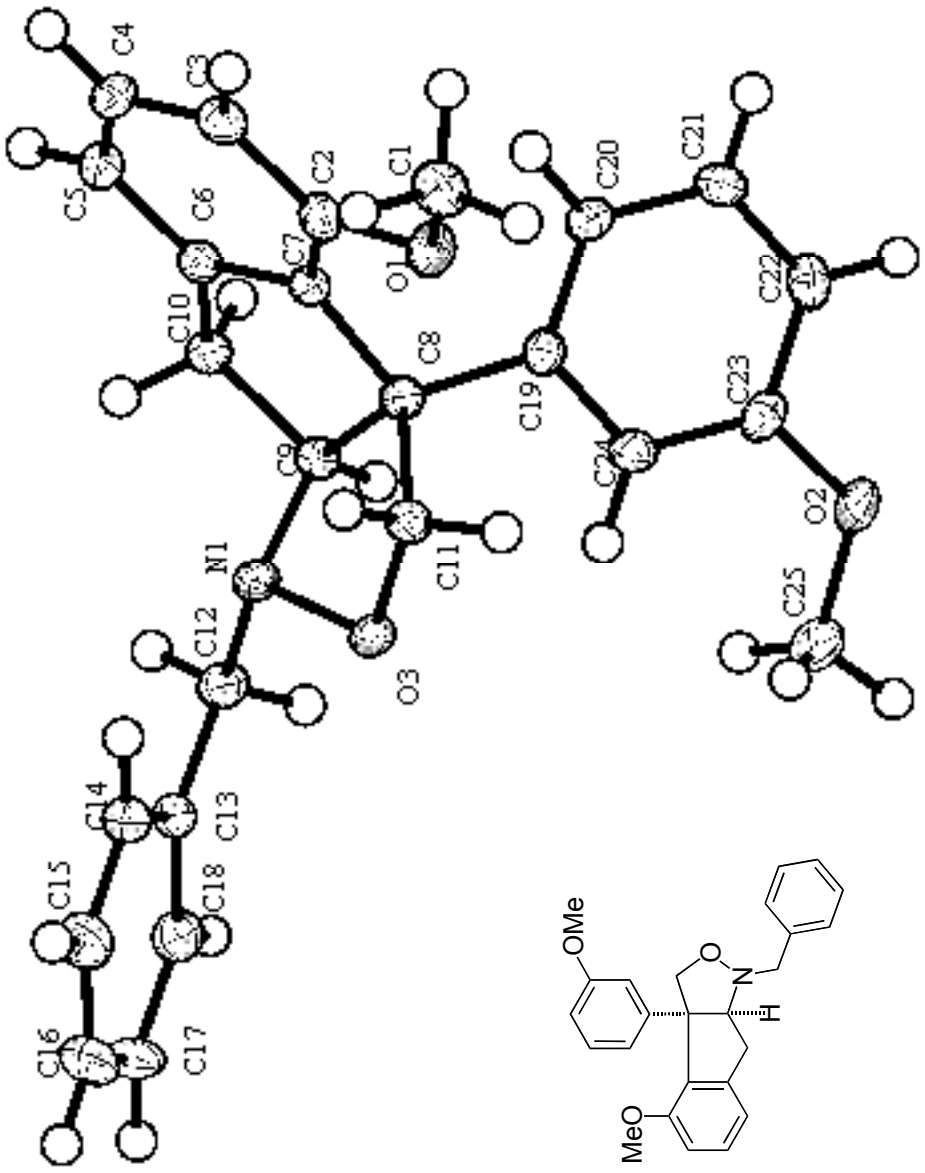
F2 - Acquisition Parameters
Date_ 20060306
Time 3.16
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 510
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D2 0.00357143 sec
D12 0.00002000 sec
DELTA 0.00000688 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 5.40 usec
P2 10.80 usec
PL1 -6.00 dB
SFO1 75.4098817 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
P3 12.40 usec
P4 24.80 usec
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 299.8711995 MHz

F2 - Processing parameters
SI 32768
SF 75.4023573 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15080.47 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 10.50000 ppm/cm
HZCM 791.72479 Hz/cm


C4
C3
C5
C6
C2
C1
C7
O1
C10
C8
C20
C21
C22
C19
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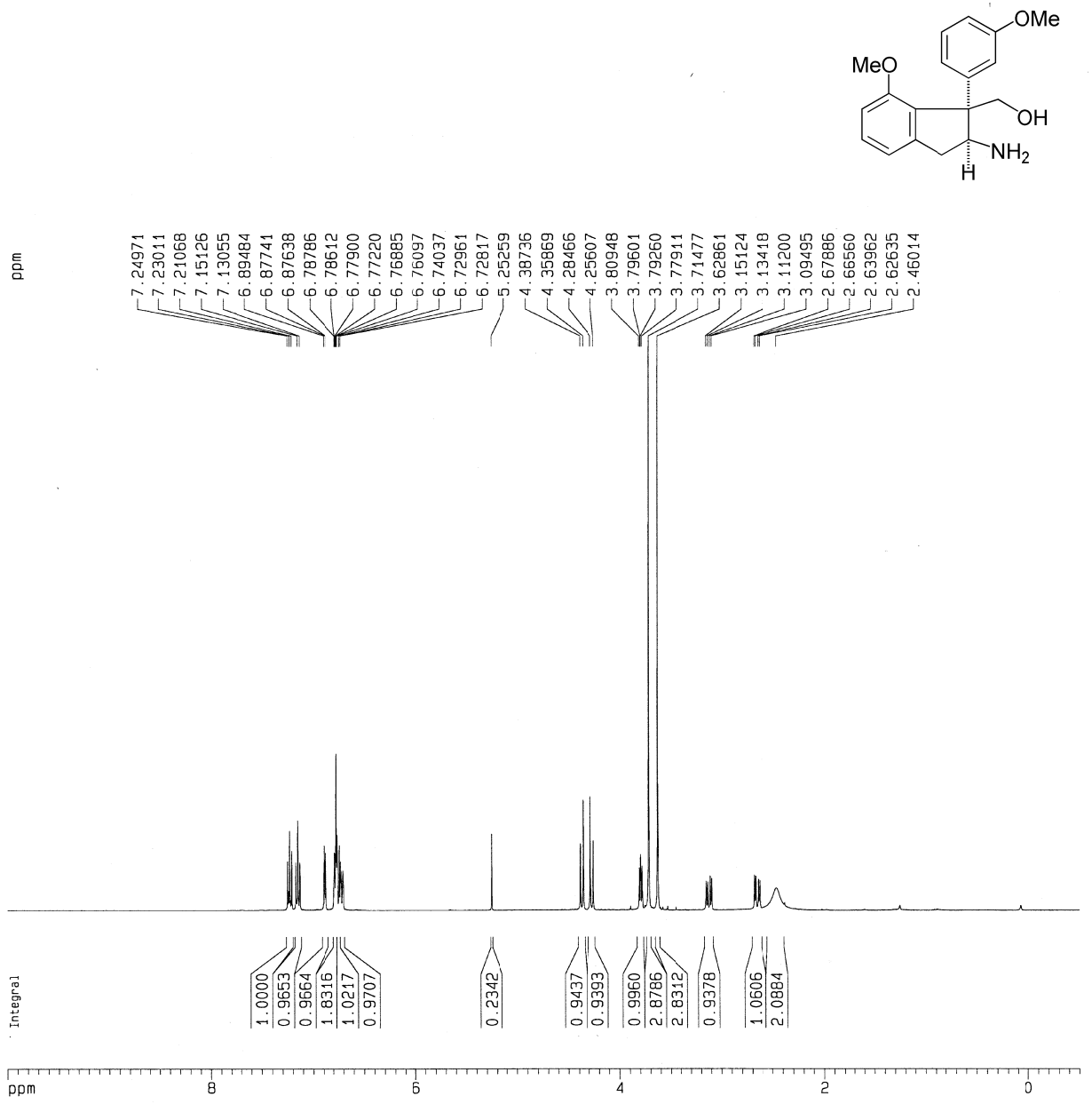
C9
C24
O2
N1
C11
C25
C12
O3
C14
C13
C15
C18
C16
C17
OMe
O
N
H
MeO



Current Data Parameters

NAME	jhj-10-14-4
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20041014
Time	13.13
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	33110
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.250019 Hz
AQ	1.9998940 sec
RG	90.5
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

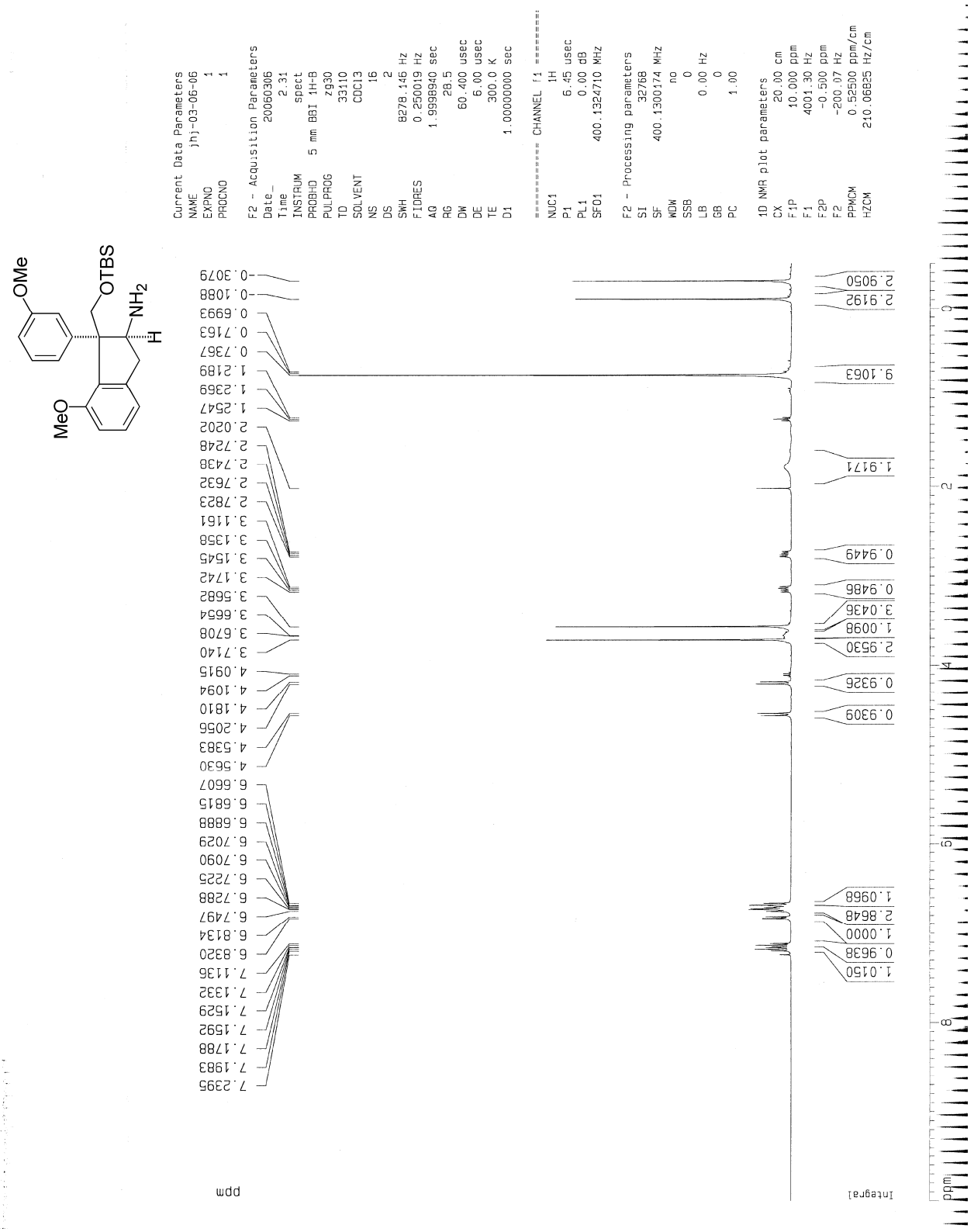
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

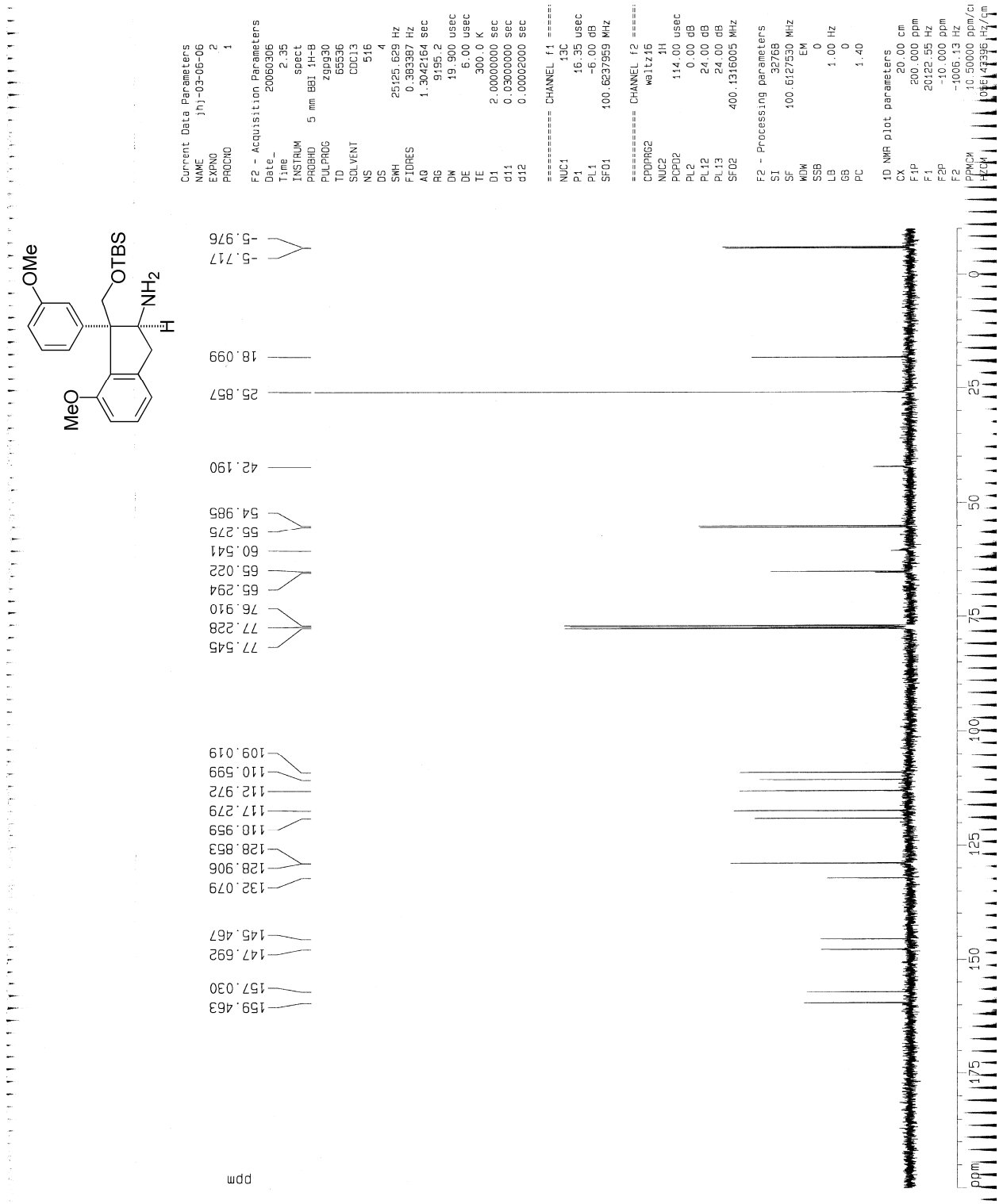
F2 - Processing parameters

SI	32768
SF	400.1300171 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm


OMe
OTBS
NH2
H
MeO
Current Data Parameters
NAME jhj-03-06-06
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060306
Time 2.31
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 33110
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.250019 Hz
AQ 1.9998940 sec
RG 28.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm
0.3079
0.1088
0.6993
0.7163
0.7367
1.2189
1.2369
1.2547
2.0202
2.7248
2.7438
2.7632
2.7823
3.1161
3.1358
3.1545
3.1742
3.5682
3.6654
3.6708
3.7140
4.0915
4.1094
4.1810
4.2056
4.5383
4.5630
6.6607
6.6815
6.6888
6.7029
6.7090
6.7225
6.7288
6.7497
6.8134
6.8320
7.1136
7.1332
7.1529
7.1592
7.1788
7.1983
7.2395
2.9050
2.9192
9.1063
1.9171
0.9449
0.9486
3.0436
1.0098
2.9530
0.9326
0.9309
1.0968
2.8648
1.0000
0.9638
1.0150
ppm
Integral


OMe
OTBS
NH2
H
MeO
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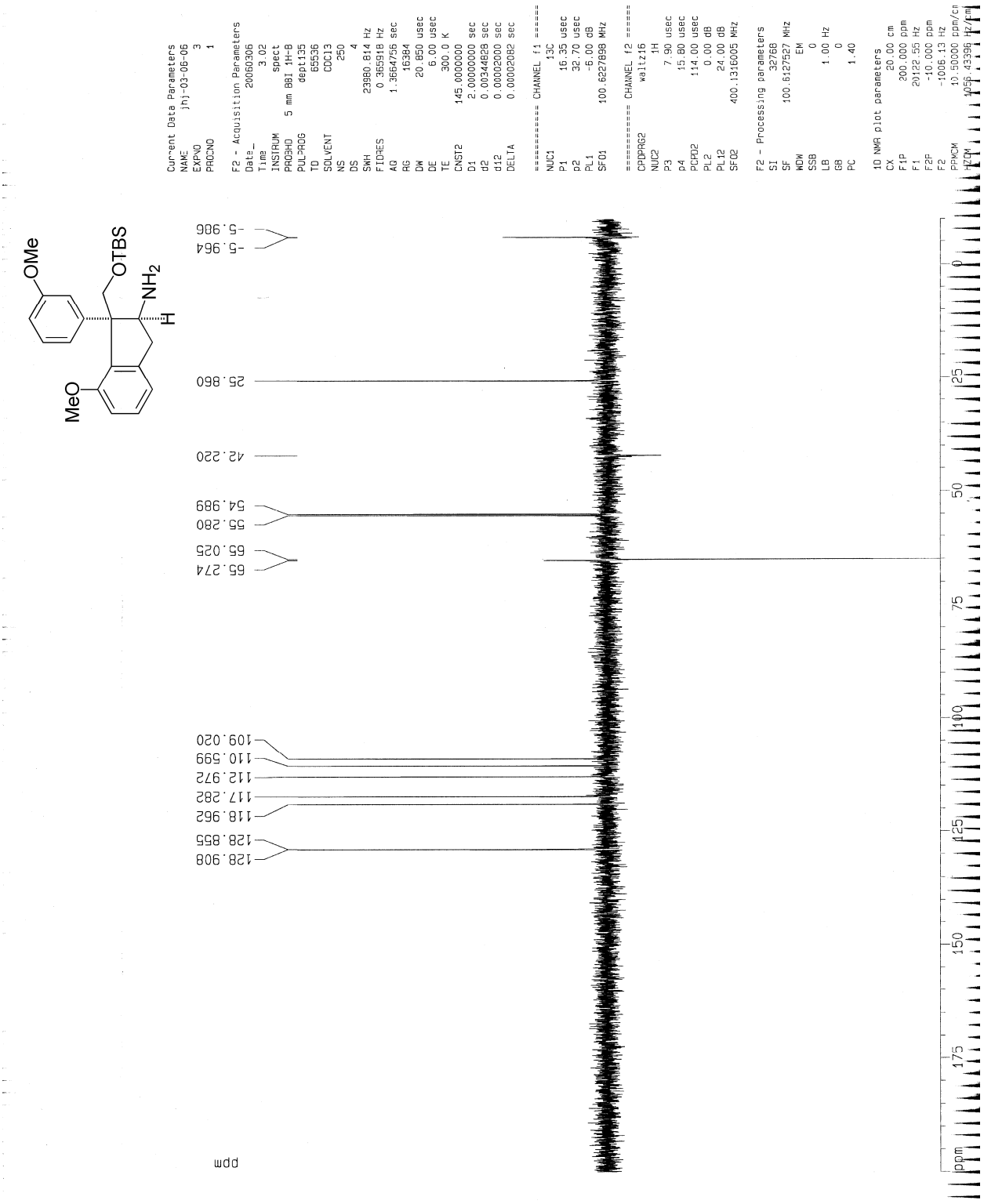
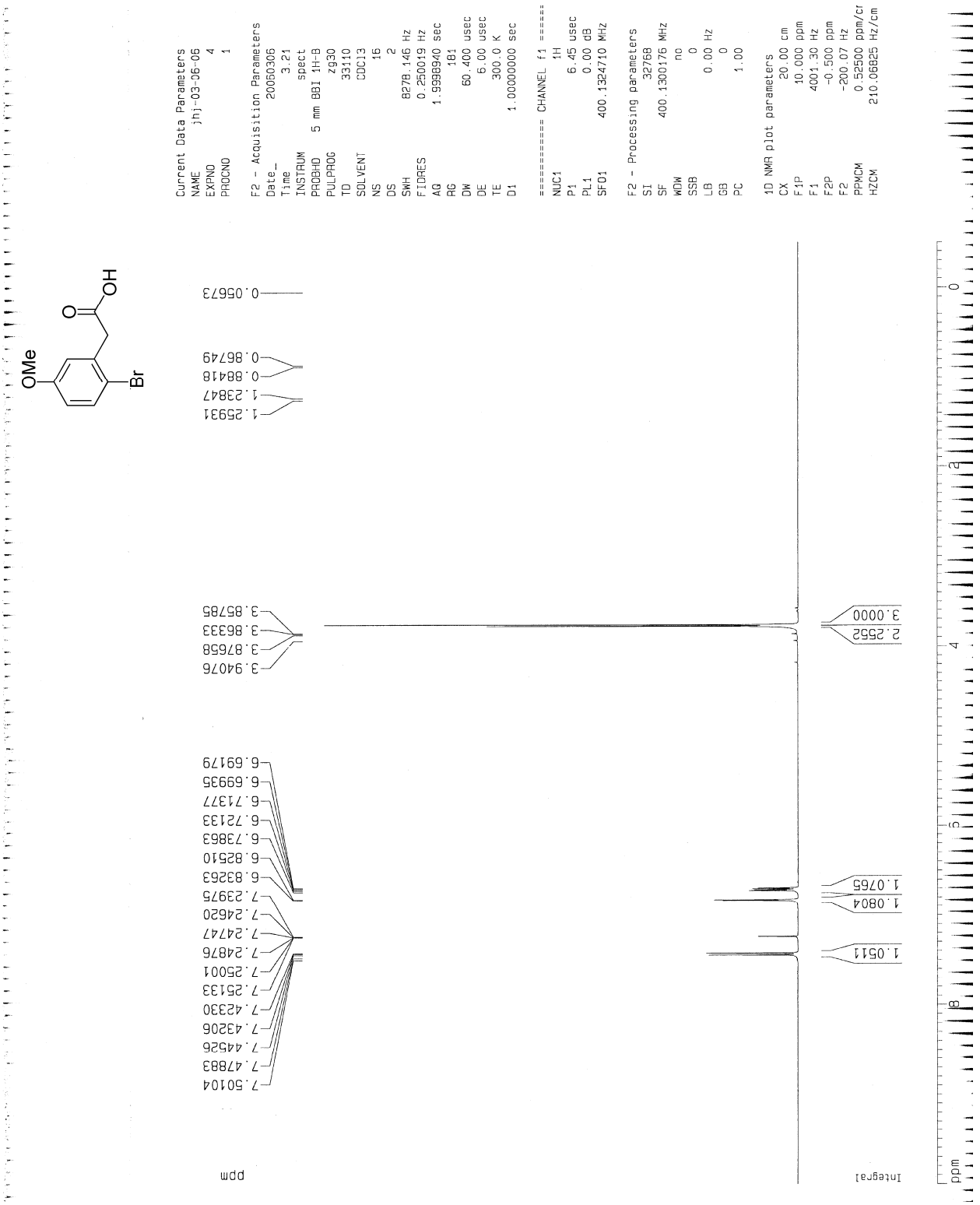
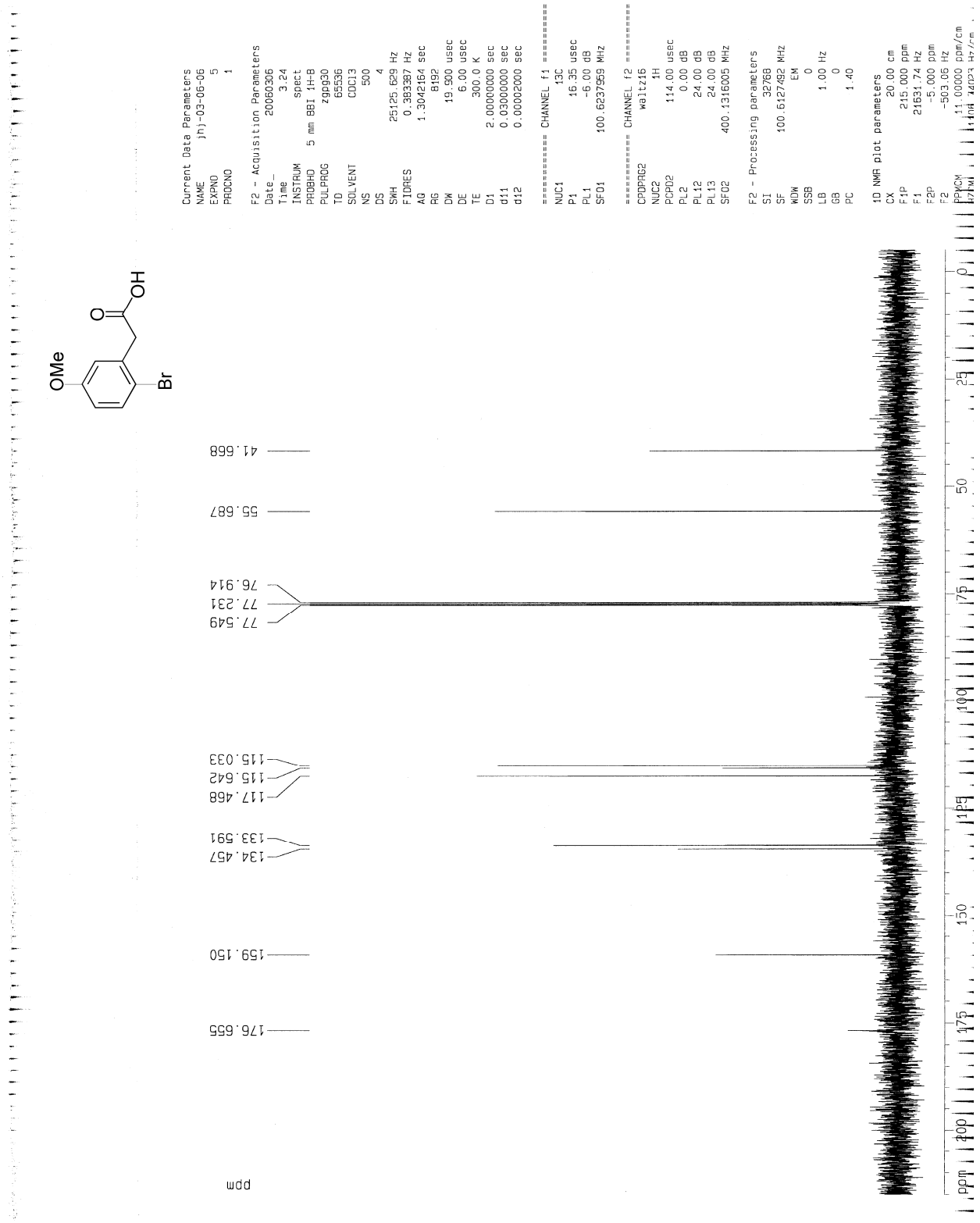
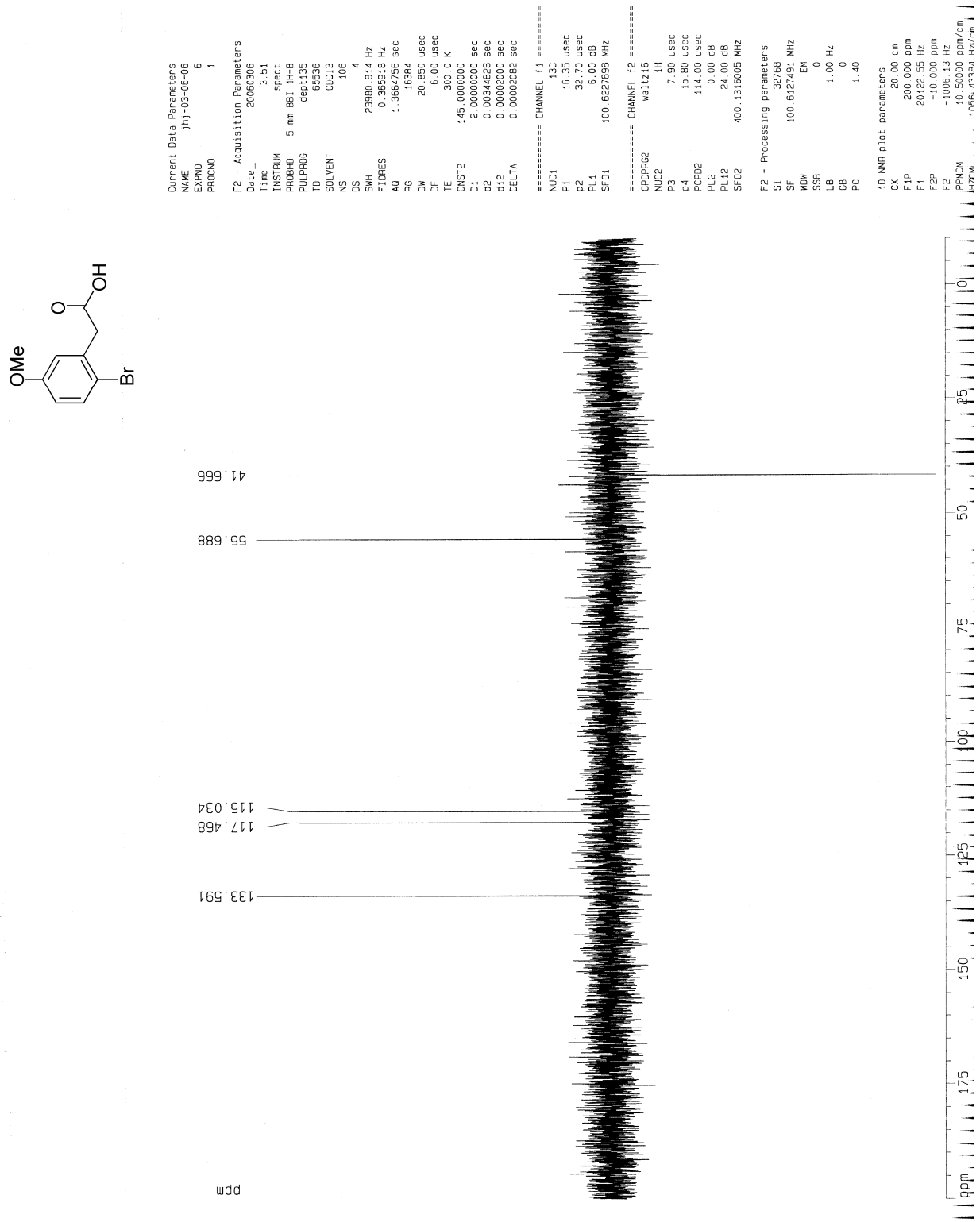
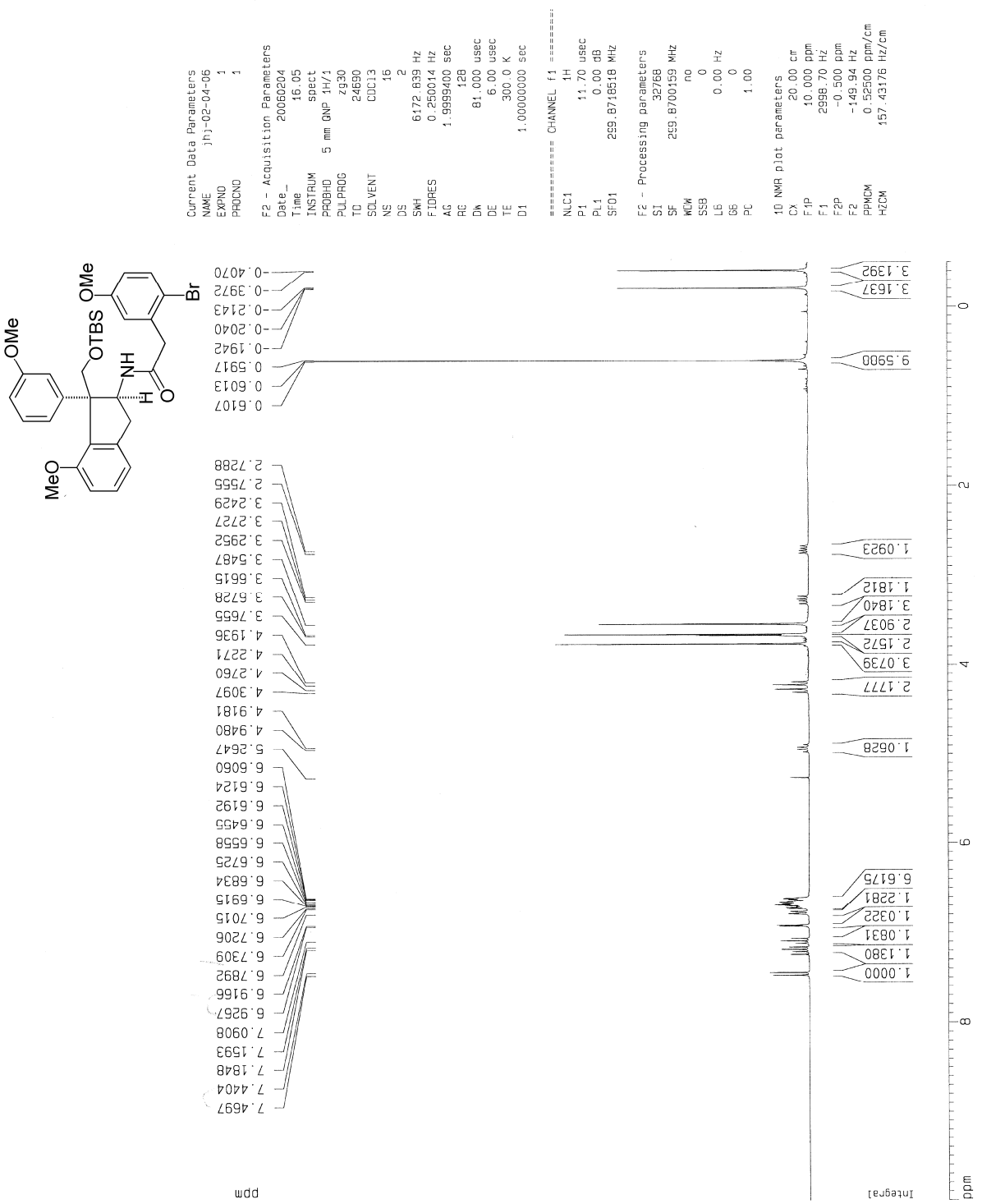
-5.717
18.099
25.857
42.190
54.985
55.275
60.541
65.022
65.294
76.910
77.228
77.545
109.019
110.599
112.972
117.279
118.959
128.853
128.906
132.079
145.467
147.692
157.030
159.463
ppm
Current Data Parameters
NAME jnj-03-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060306
Time 2.35
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 516
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 9195.2
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 16.35 usec
PL1 -6.00 dB
SF01 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 114.00 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127530 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 2114.33936 Hz/cm
ppm

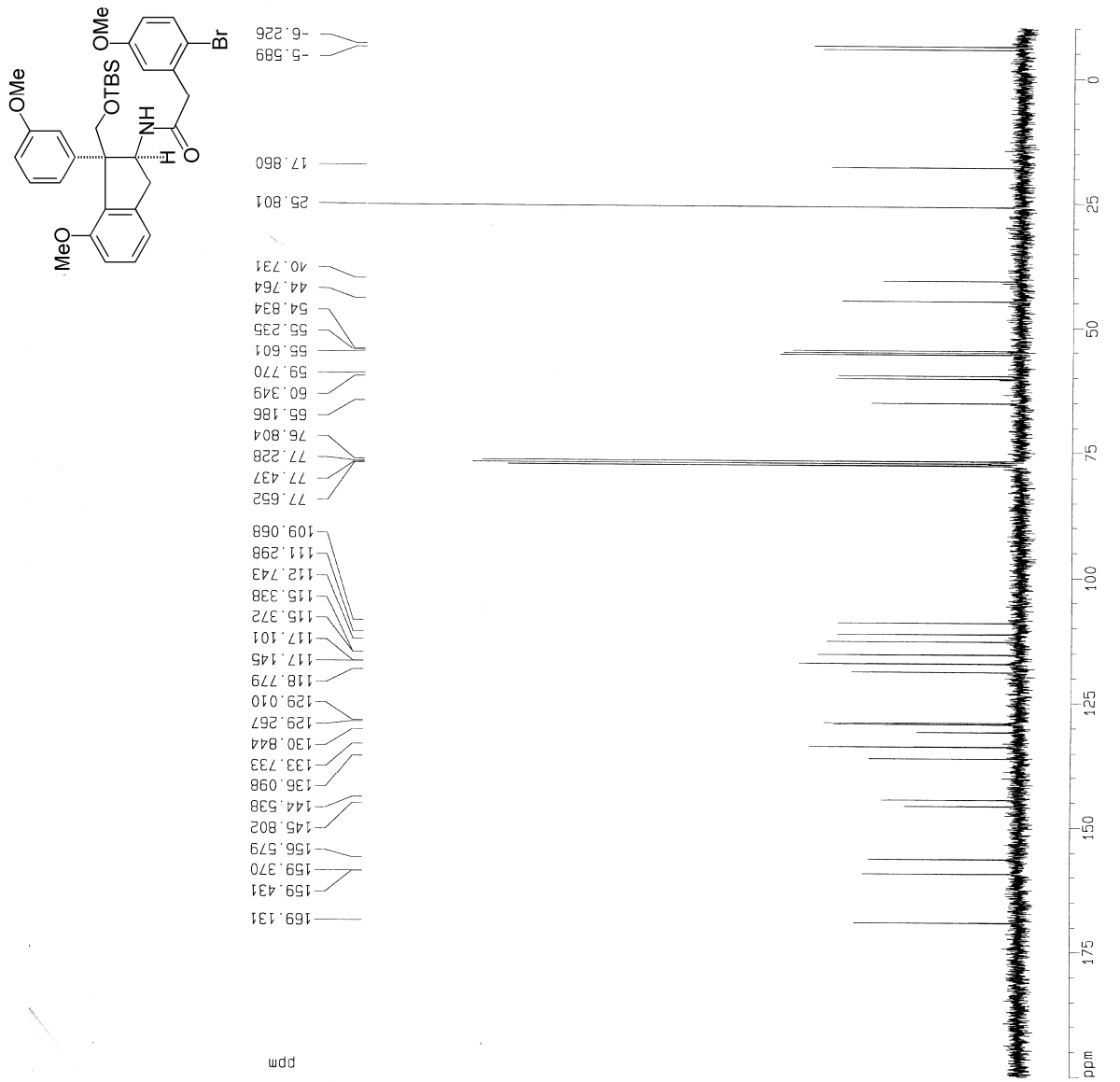

OMe
OTBS
NH2
H
MeO
-5.906
-5.964
25.860
42.220
54.989
55.280
65.025
65.274
109.020
110.599
112.972
117.282
118.962
128.855
128.908
ppm

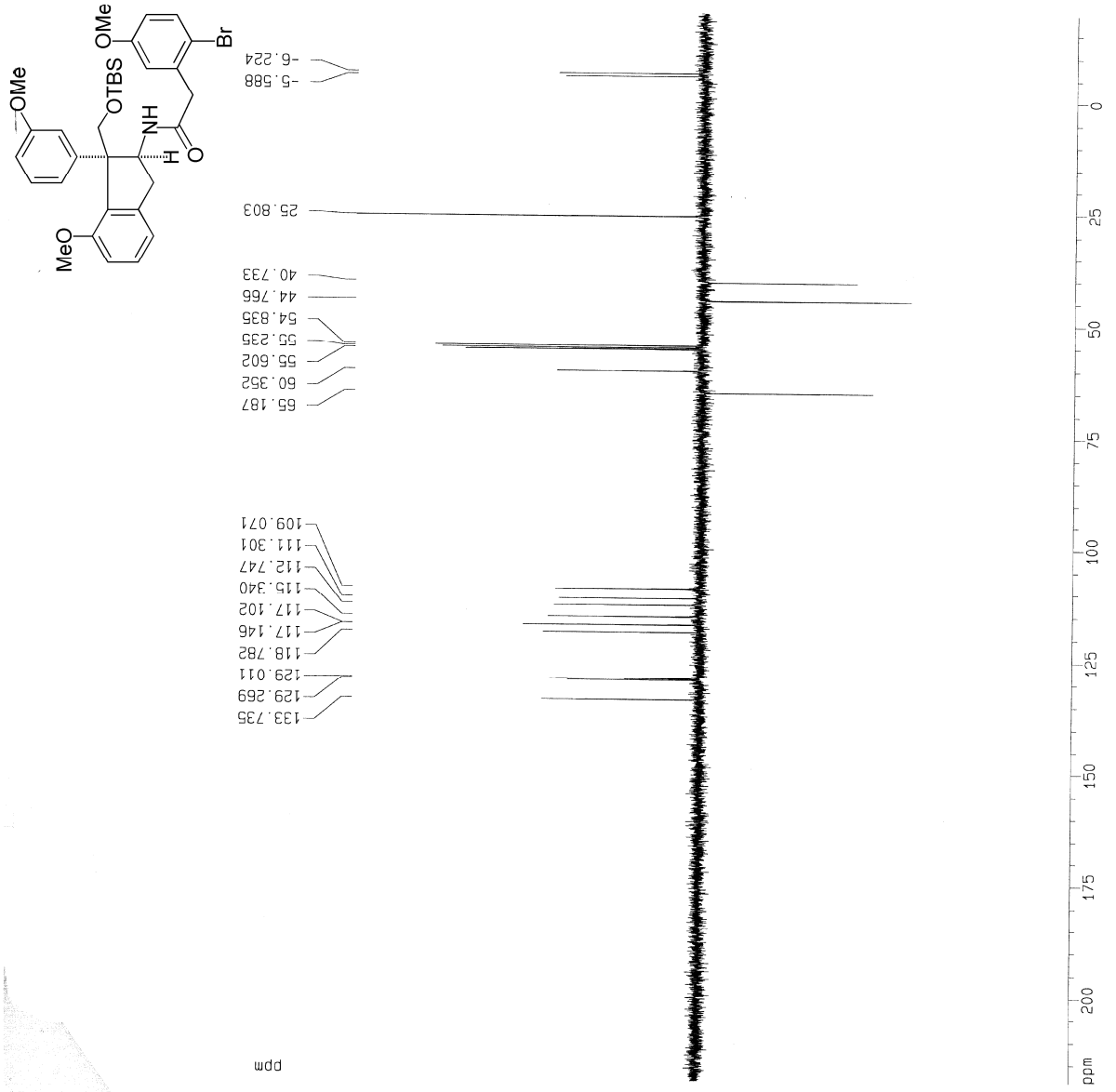

OMe
Br
O
OH
Current Data Parameters
NAME jhj-03-06-06
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060306
Time 3.21
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 33110
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.250019 Hz
AQ 1.9998940 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300176 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm
0.05673
0.86749
0.88418
1.23847
1.25931
3.85785
3.86333
3.87658
3.94076
6.69179
6.69935
6.71377
6.72133
6.73863
6.82510
6.83263
7.23975
7.24620
7.24747
7.24876
7.25001
7.25133
7.42330
7.43206
7.44526
7.47883
7.50104
3.0000
2.2552
1.0804
1.0765
1.0511
ppm
Integral

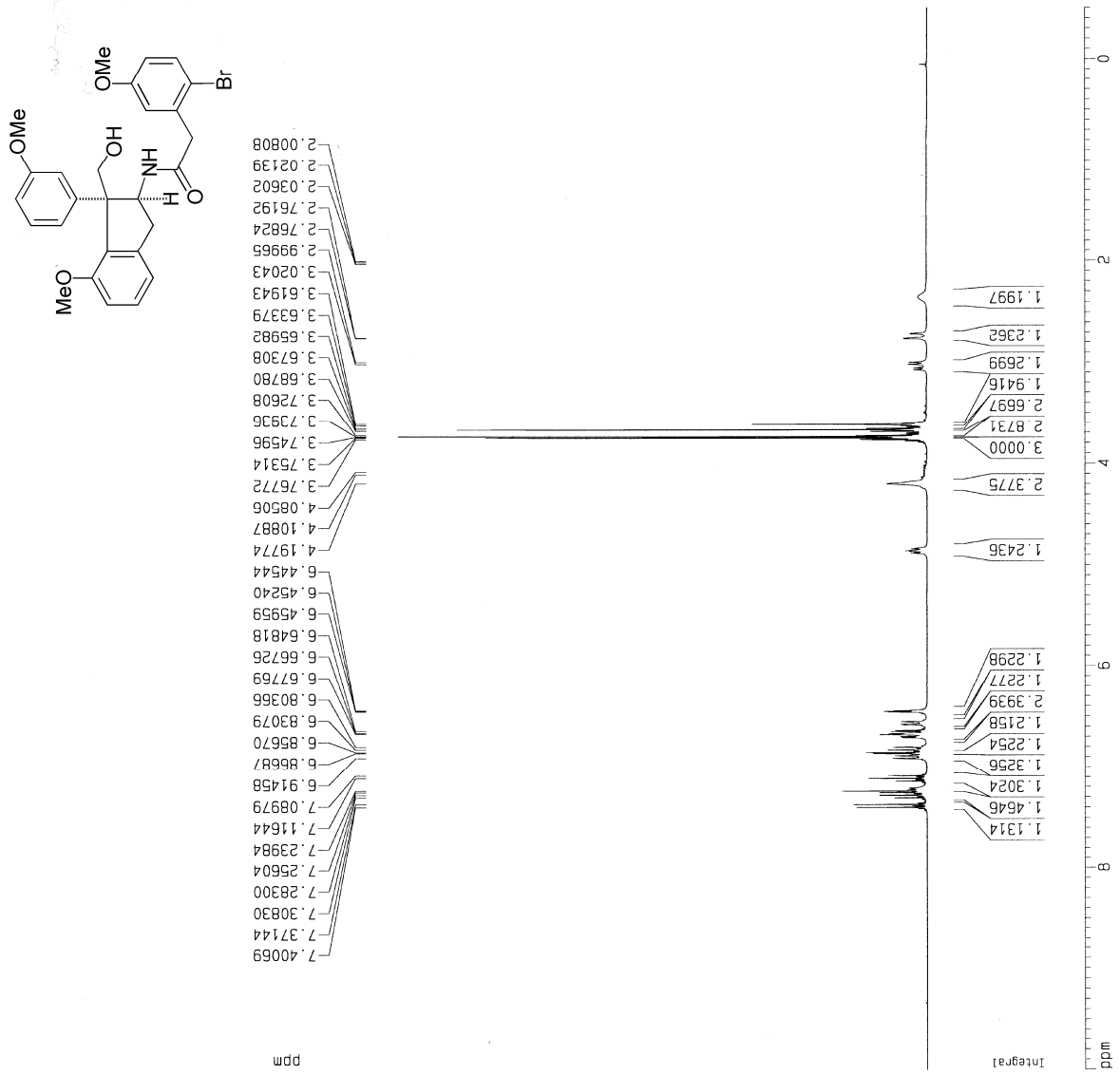

OMe
O
OH
Br
41.668
55.687
76.914
77.231
77.549
115.033
115.642
117.468
133.591
134.457
159.150
176.655
ppm

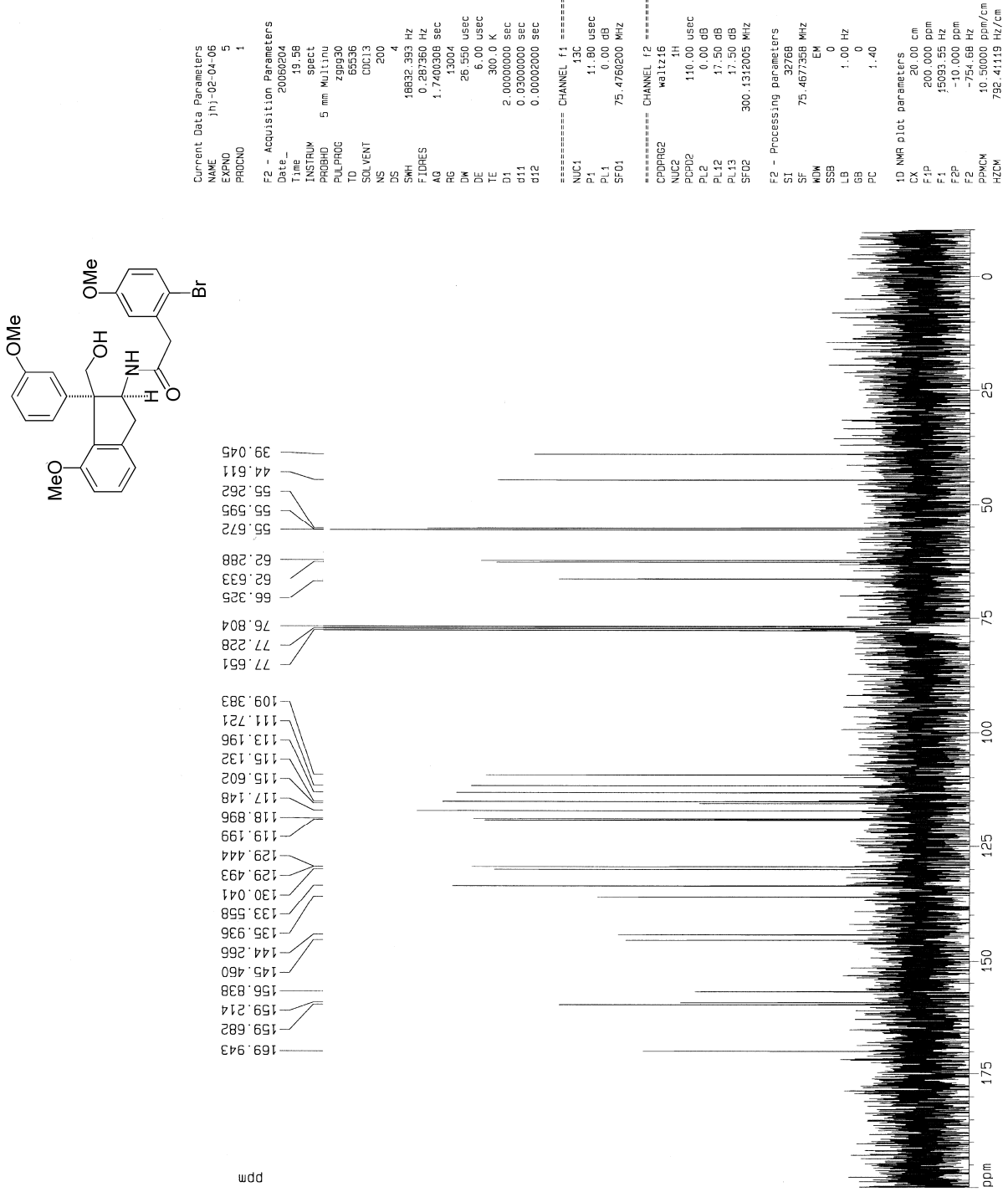

OMe
O
OH
Br
41.666
55.688
115.034
117.468
133.591
ppm

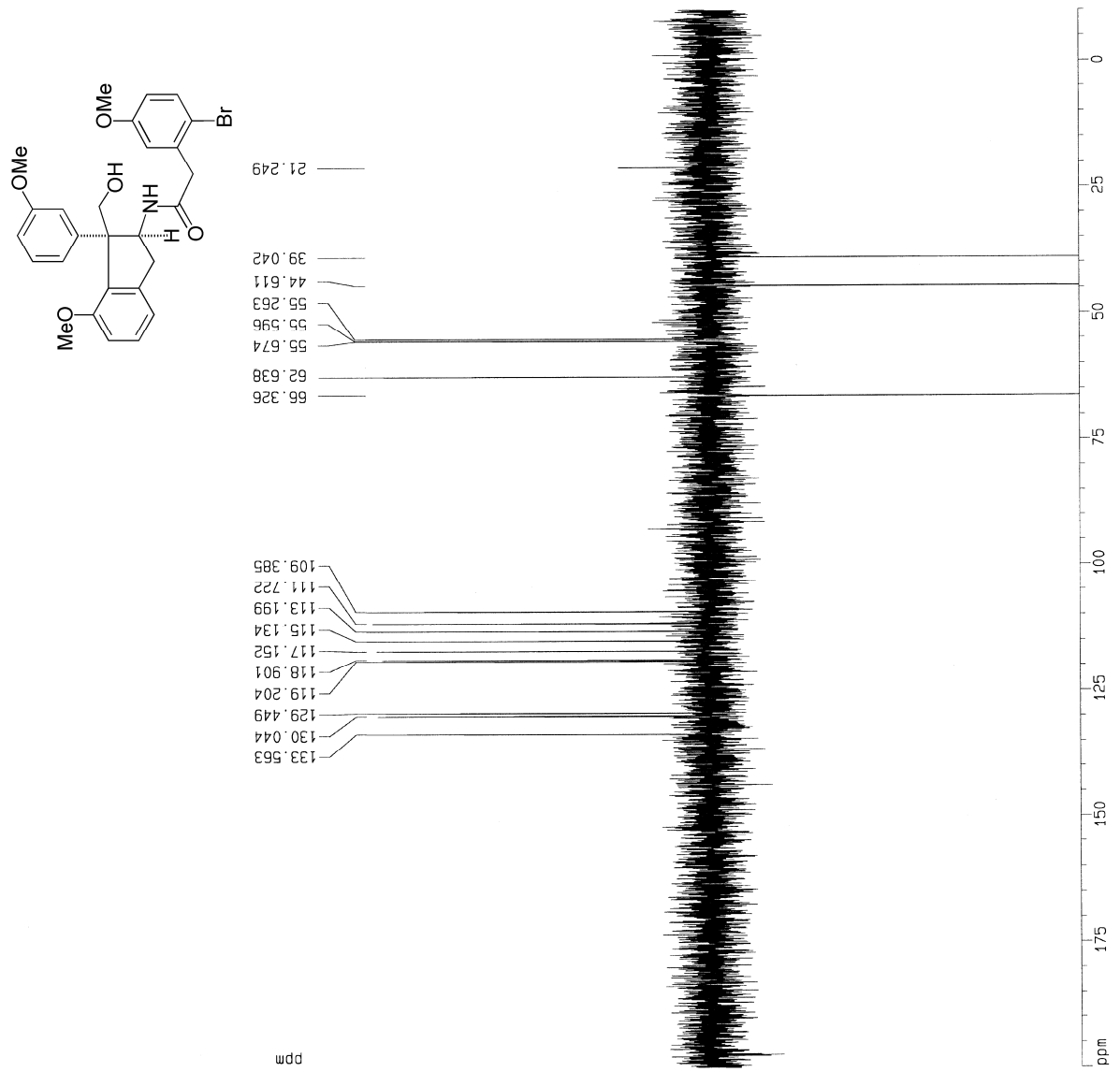

Current Data Parameters
NAME jhj-02-04-06
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060204
Time 16.05
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8716518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700159 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 2998.70 Hz
F2P -0.500 ppm
F2 -149.94 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.43176 Hz/cm
OMe
OTBS
OMe
Br
NH
H
O
MeO
0.4070
0.3972
0.2143
0.2040
0.1942
0.5917
0.6013
0.6107
2.7288
2.7555
3.2429
3.2727
3.2952
3.5487
3.6615
3.6728
3.7655
4.1936
4.2271
4.2760
4.3097
4.9181
4.9480
5.2647
6.6060
6.6124
6.6192
6.6455
6.6558
6.6725
6.6834
6.6915
6.7015
6.7206
6.7309
6.7892
6.9166
6.9257
7.0908
7.1593
7.1848
7.4404
7.4697
3.1392
3.1637
9.5900
1.0923
1.1812
3.1840
2.9037
2.1572
3.0739
2.1777
1.0628
6.6175
1.2281
1.0322
1.0831
1.1380
1.0000
ppm
Integral
0
2
4
6
8
ppm


OMe
OTBS
OMe
Br
NH
H
O
MeO
-6.226
-5.589
17.860
25.801
40.731
44.764
54.834
55.235
55.601
59.770
60.349
65.186
76.804
77.228
77.437
77.652
109.068
111.298
112.743
115.338
115.372
117.101
117.145
118.779
129.010
129.267
130.844
133.733
136.098
144.538
145.802
156.579
159.370
159.431
169.131
ppm
Current Data Parameters
NAME jnj-02-04-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060204
Time 16.08
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 150
DS 4
SWH 18796.392 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023607 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15080.47 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 10.50000 ppm/cm
HZCM 791.72479 Hz/cm
ppm
0
25
50
75
100
125
150
175


OMe
OTBS
OMe
Br
NH
H
O
MeO
-6.224
-5.588
25.803
40.733
44.766
54.835
55.235
55.602
60.352
65.187
109.071
111.301
112.747
115.340
117.102
117.146
118.782
129.011
129.269
133.735
ppm
Current Data Parameters
NAME jnj-02-04-06
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060204
Time 15.18
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 75
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D2 0.00357143 sec
D12 0.00002000 sec
DELTA 0.00000688 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
P2 10.80 usec
PL1 -5.00 dB
SFO1 75.4098817 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.40 usec
P4 24.80 usec
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023605 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 219.012 ppm
F1 16513.99 Hz
F2P -19.517 ppm
F2 -1471.62 Hz
PPMCM 11.00000 ppm/cm
ppm
200
175
150
125
100
75
50
25
0


OMe
Br
OMe
OH
NH
H
O
MeO
Current Data Parameters
NAME jhj-02-04-06
EXPNO 4
PROCNO 1
F2 - Accuisition Parameters
Date_ 20060204
Time 19.53
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 362
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 300.1316534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
2.00808
2.02139
2.03602
2.76192
2.76824
2.99965
3.02043
3.61943
3.63379
3.65982
3.67308
3.68780
3.72608
3.73936
3.74596
3.75314
3.76772
4.08506
4.10887
4.19774
6.44544
6.45240
6.45959
6.48418
6.66726
6.67769
6.80366
6.83079
6.85670
6.86687
6.91458
7.08979
7.11644
7.23984
7.25604
7.28300
7.30830
7.37144
7.40069
ppm
1.1997
1.2362
1.2699
1.9416
2.6697
2.8731
3.0000
2.3775
1.2436
1.2298
1.2277
2.3939
1.2158
1.2254
1.3256
1.3024
1.4646
1.1314
Integral
ppm


OMe
OMe
Br
OH
NH
H
O
MeO
Current Data Parameters
NAME jhj-02-04-06
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060204
Time 19.58
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 13004
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
========== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4760200 MHz
========== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677358 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41119 Hz/cm
39.045
44.611
55.262
55.595
55.672
62.288
62.633
66.325
76.804
77.228
77.651
109.383
111.721
113.196
115.132
115.602
117.148
118.896
119.199
129.444
129.493
130.041
133.558
135.936
144.266
145.460
156.838
159.214
159.682
169.943
ppm
0
25
50
75
100
125
150
175



Current Data Parameters
NAME jhj-02-04-06
EXPNO 6
PROCNO 1

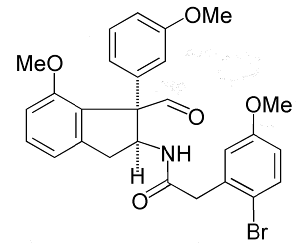
F2 - Acquisition Parameters
Date_ 20060204
Time 20.10
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 97
DS 4
SWH 17985.6:1 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
DELTA 0.00001502 sec

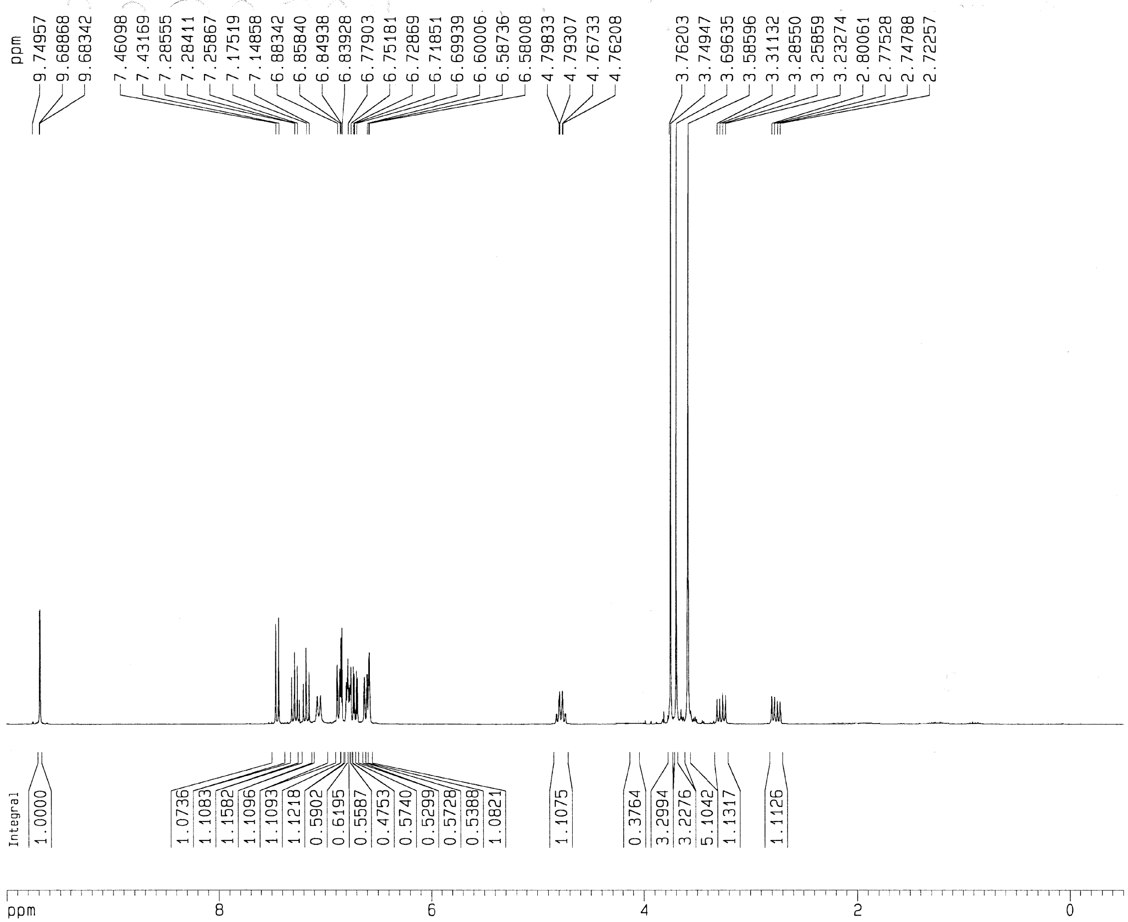
========== CHANNEL f1 ==========
NUC1 13C
P1 11.80 usec
P2 23.60 usec
PL1 0.00 dB
SFO1 75.4752653 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
P3 15.25 usec
P4 30.50 usec
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677357 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41119 Hz/cm



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Current Data Parameters
NAME          jhj-02-19-06
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20060218
Time                 17.11
INSTRUM              spect
PROBHD      5 mm Multinu
PULPROG               zg30
TD                   24690
SOLVENT              CDCl3
NS                      16
DS                       2
SWH            6172.839 Hz
FIDRES         0.250014 Hz
AQ            1.9999400 sec
RG                     128
DW              81.000 usec
DE                6.00 usec
TE                 300.0 K
D1           1.00000000 sec

============ CHANNEL f1 ========
NUC1                    1H
P1                9.60 usec
PL1               -6.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF           300.1300124 MHz
WDW                     no
SSB                      0
LB                 0.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                20.00 cm
F1P             10.000 ppm
F1               3001.30 Hz
F2P             -0.500 ppm
F2              -150.07 Hz
PPMCM        0.52500 ppm/cm
HZCM       157.56825 Hz/cm
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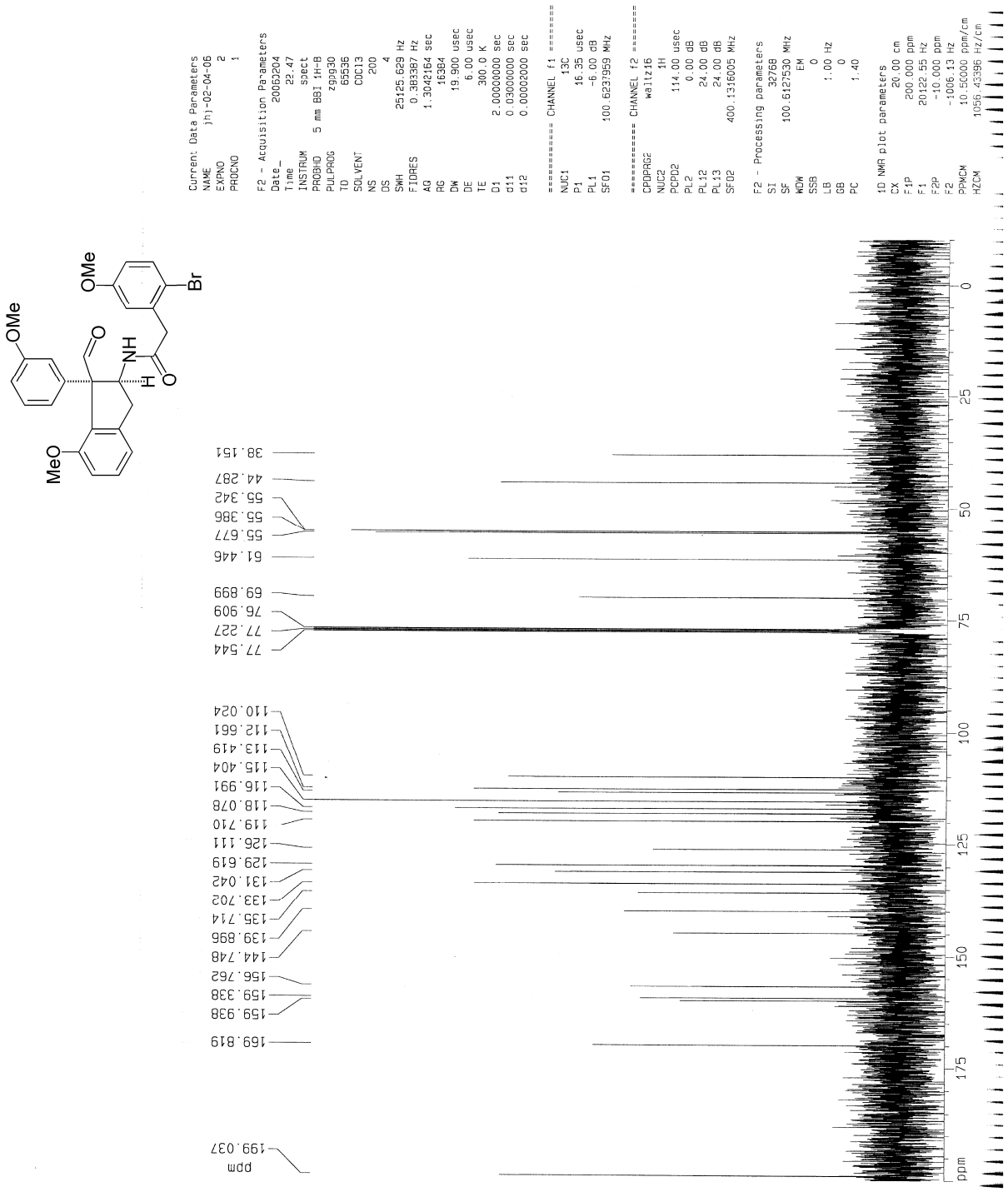
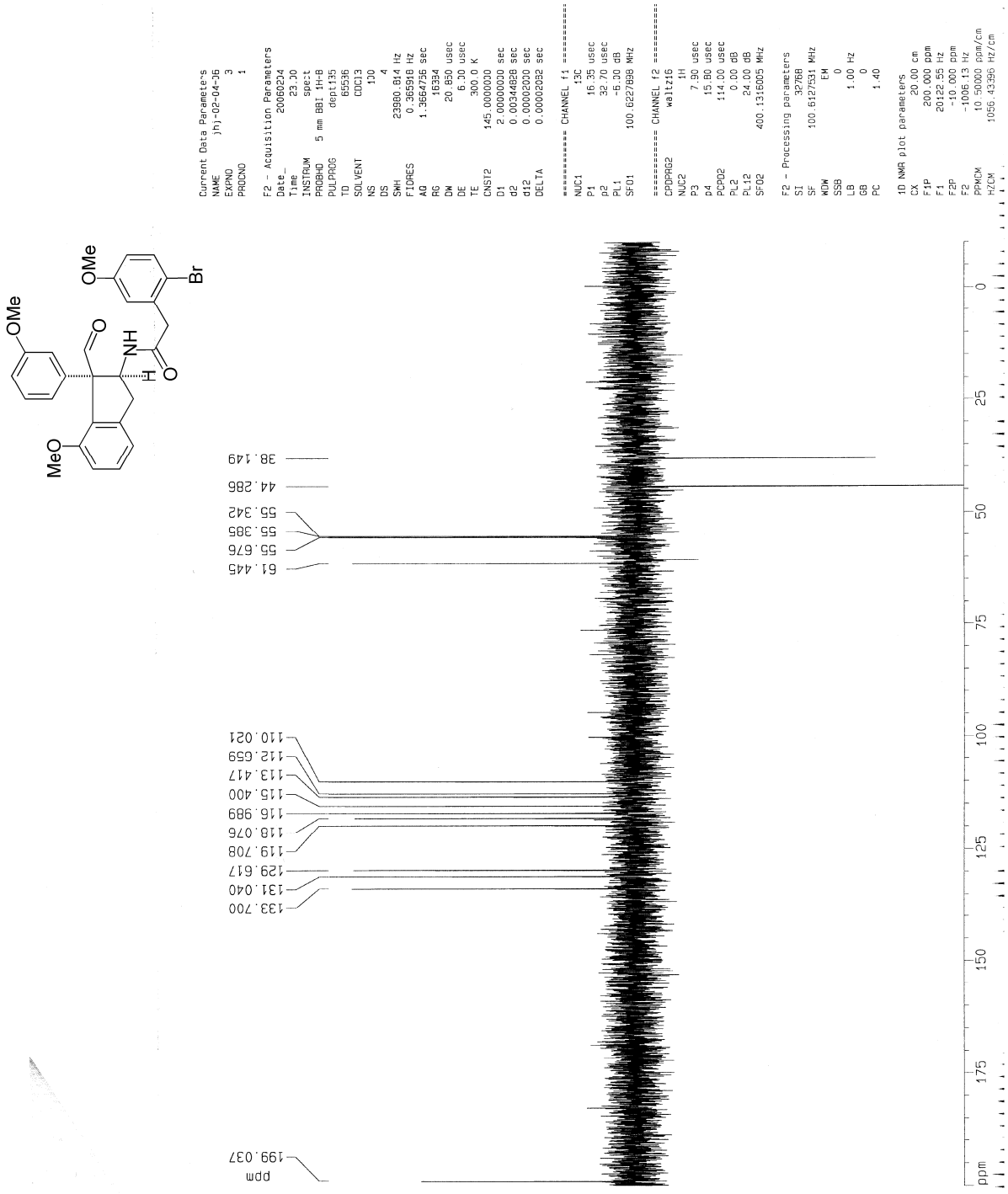
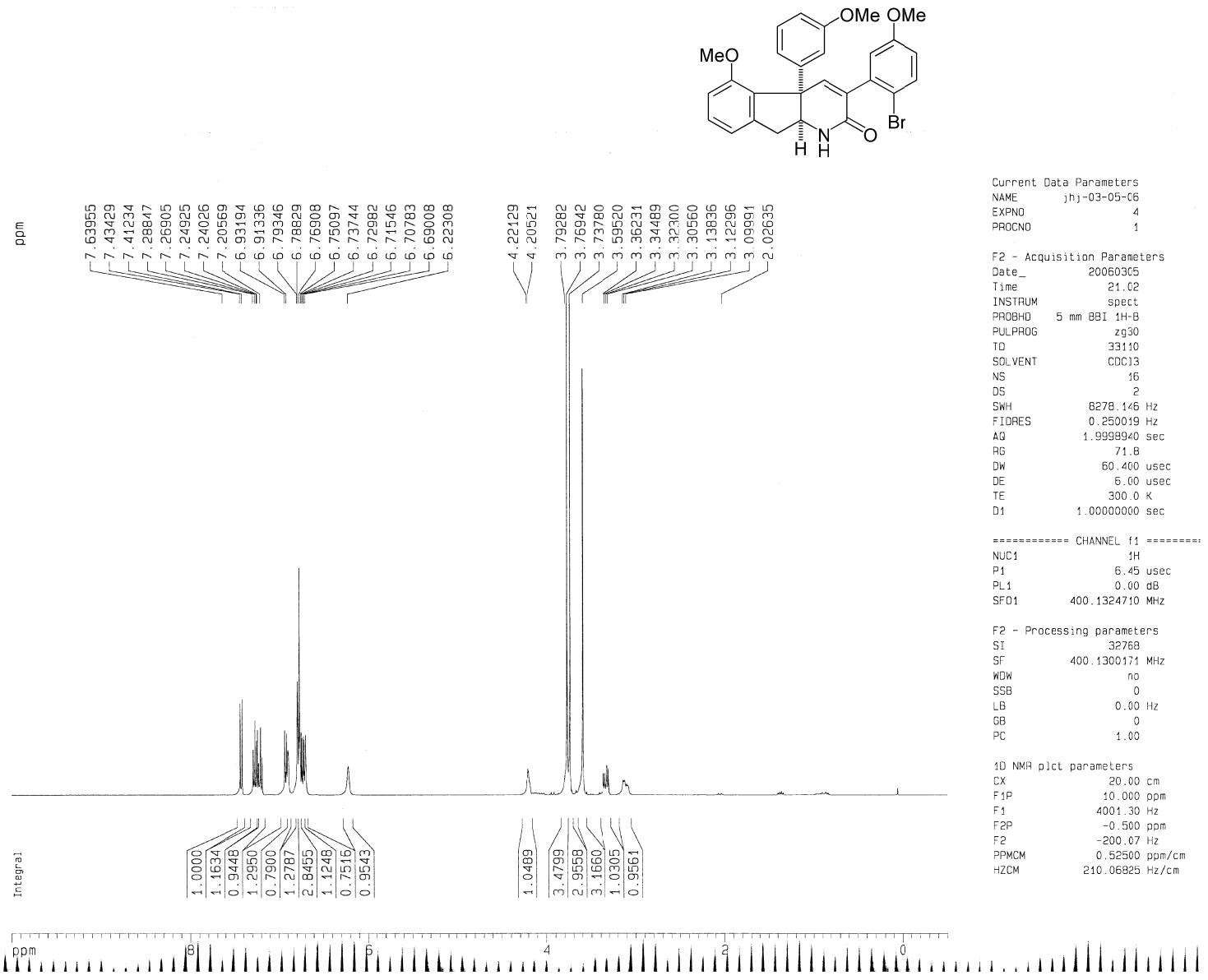

OMe
Br
O
NH
H
O
MeO
Current Data Parameters
NAME jhj-02-04-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060204
Time 22.47
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 16384
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 16.35 usec
PL1 -6.00 dB
SFO1 100.6237959 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 114.00 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127530 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43396 Hz/cm
38.151
44.287
55.342
55.386
55.677
61.446
69.899
76.909
77.227
77.544
110.024
112.661
113.419
115.404
116.991
118.078
119.710
126.111
129.619
131.042
133.702
135.714
139.896
144.748
156.762
159.338
159.938
169.819
199.037
ppm
0
25
50
75
100
125
150
175
ppm


OMe
Br
O
NH
H
O
OMe
MeO
38.149
44.286
55.342
55.385
55.676
61.445
110.021
112.659
113.417
115.400
116.989
118.076
119.708
129.617
131.040
133.700
199.037
ppm
Current Data Parameters
NAME jhj-02-04-J6
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060204
Time 23.30
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 23980.814 Hz
FIDRES 0.365916 Hz
AQ 1.3664736 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00002032 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 16.35 usec
p2 32.70 usec
PL1 -5.00 dB
SFO1 100.6227898 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
P3 7.90 usec
p4 15.80 usec
PCPD2 114.00 usec
PL2 0.00 dB
PL12 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43396 Hz/cm
ppm
0
25
50
75
100
125
150
175



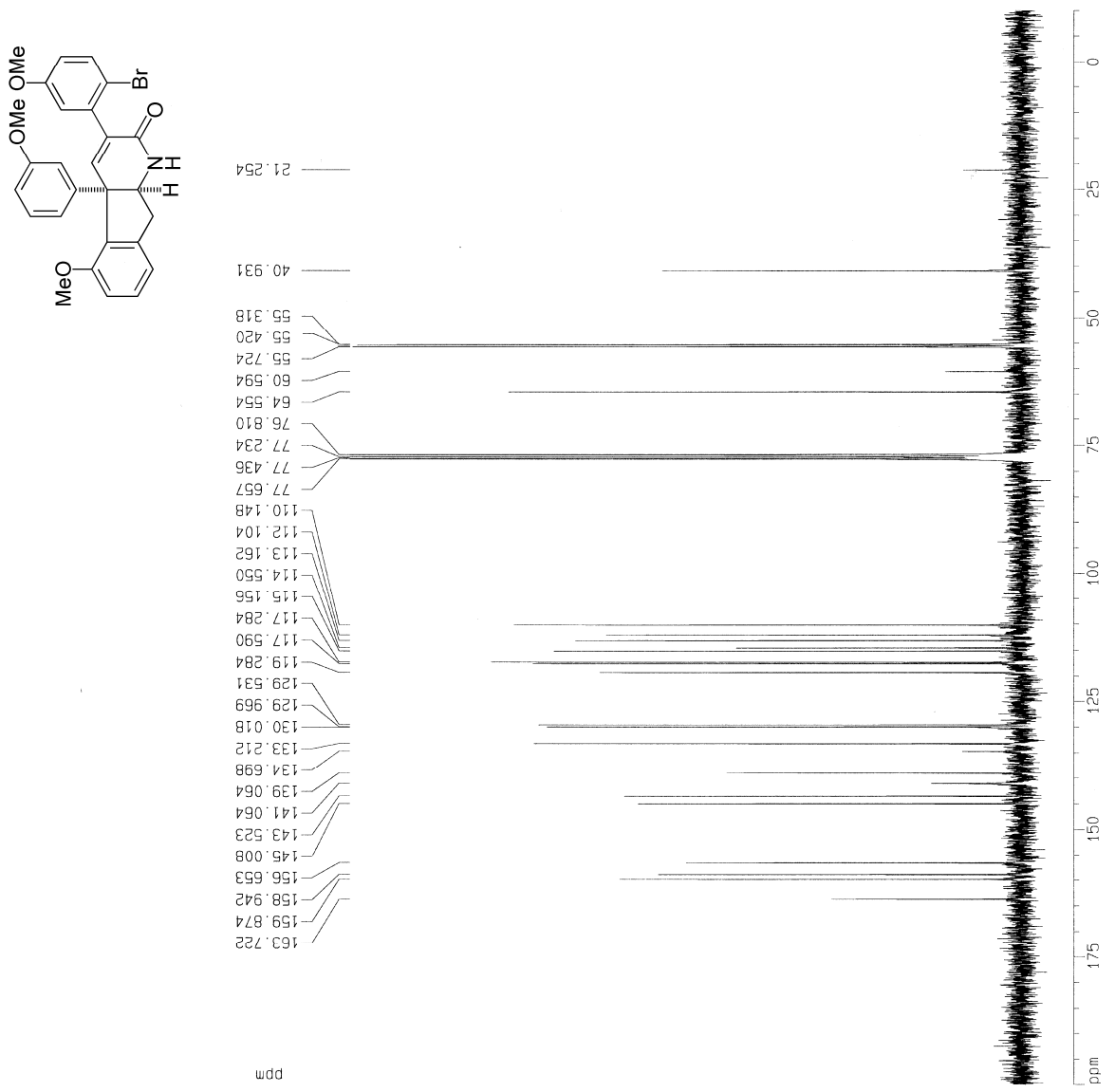

```
Current Data Parameters
NAME        jhj-03-05-06
EXPNO                  4
PROCNO                 1

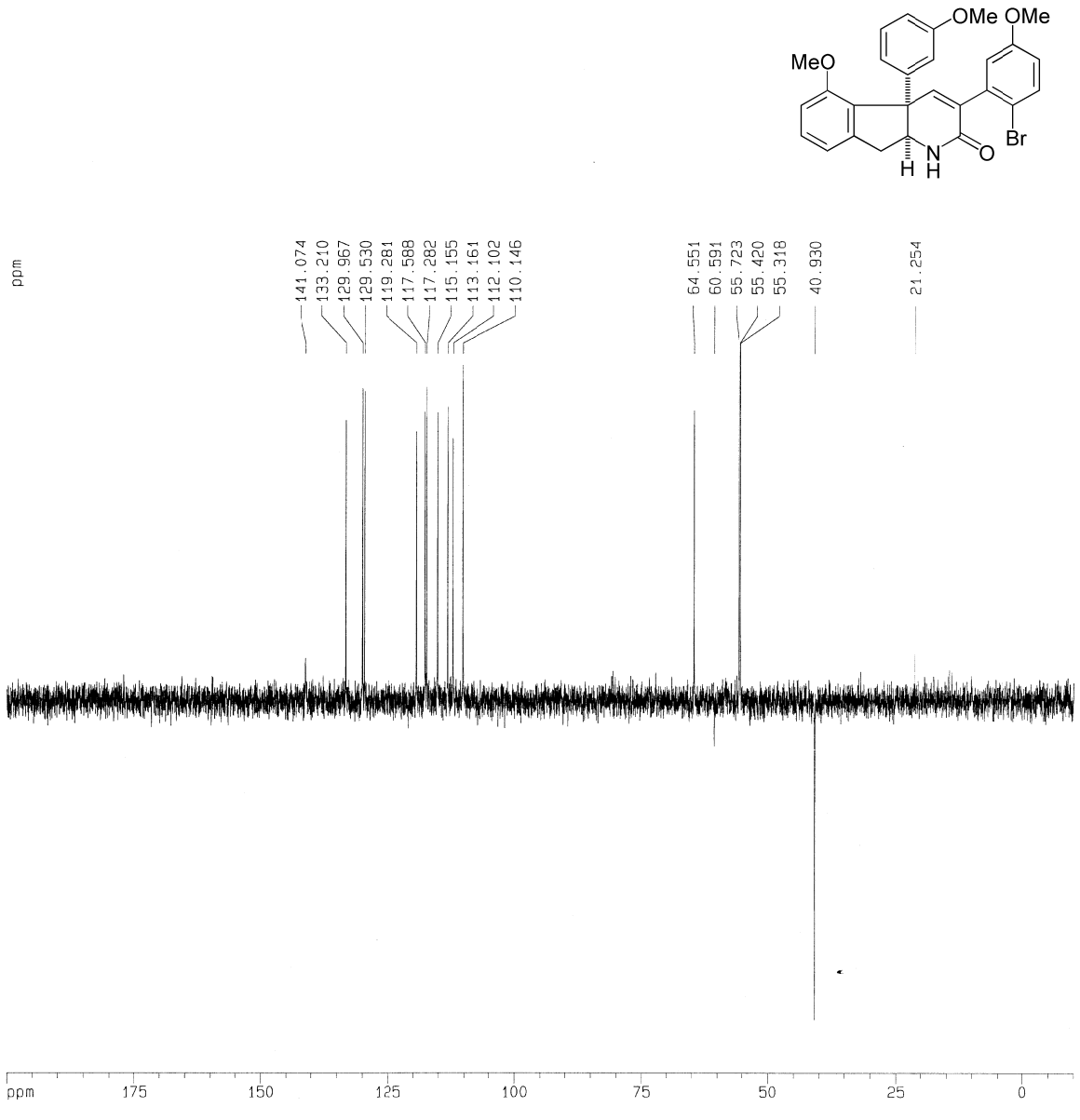
F2 - Acquisition Parameters
Date_           20060305
Time               21.02
INSTRUM            spect
PROBHD    5 mm BBI 1H-B
PULPROG             zg30
TD                 33110
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.250019 Hz
AQ              1.9998940 sec
RG                  71.8
DW                60.400 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec

============ CHANNEL f1 ========
NUC1                   1H
P1                  6.45 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300171 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -0.500 ppm
F2              -200.07 Hz
PPMCM            0.52500 ppm/cm
HZCM           210.06825 Hz/cm
```


OMe OMe
Br
O
N
H
H
MeO
21.254
40.931
55.318
55.420
55.724
60.594
64.554
76.810
77.234
77.436
77.657
110.148
112.104
113.162
114.550
115.156
117.284
117.590
119.284
129.531
129.969
130.018
133.212
134.698
139.064
141.064
143.523
145.008
156.653
158.942
159.874
163.722
ppm
Current Data Parameters
NAME jhj-03-06
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060305
Time 23.12
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1529
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023601 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15080.47 Hz
F2P -10.000 ppm
F2 -754.02 Hz
PPMCM 10.50000 ppm/cm
HZCM 791.72479 Hz/cm
ppm
0
25
50
75
100
125
150
175




```
Current Data Parameters
NAME        jhj-03-06-06
EXPNO                   6
PROCNO                  1

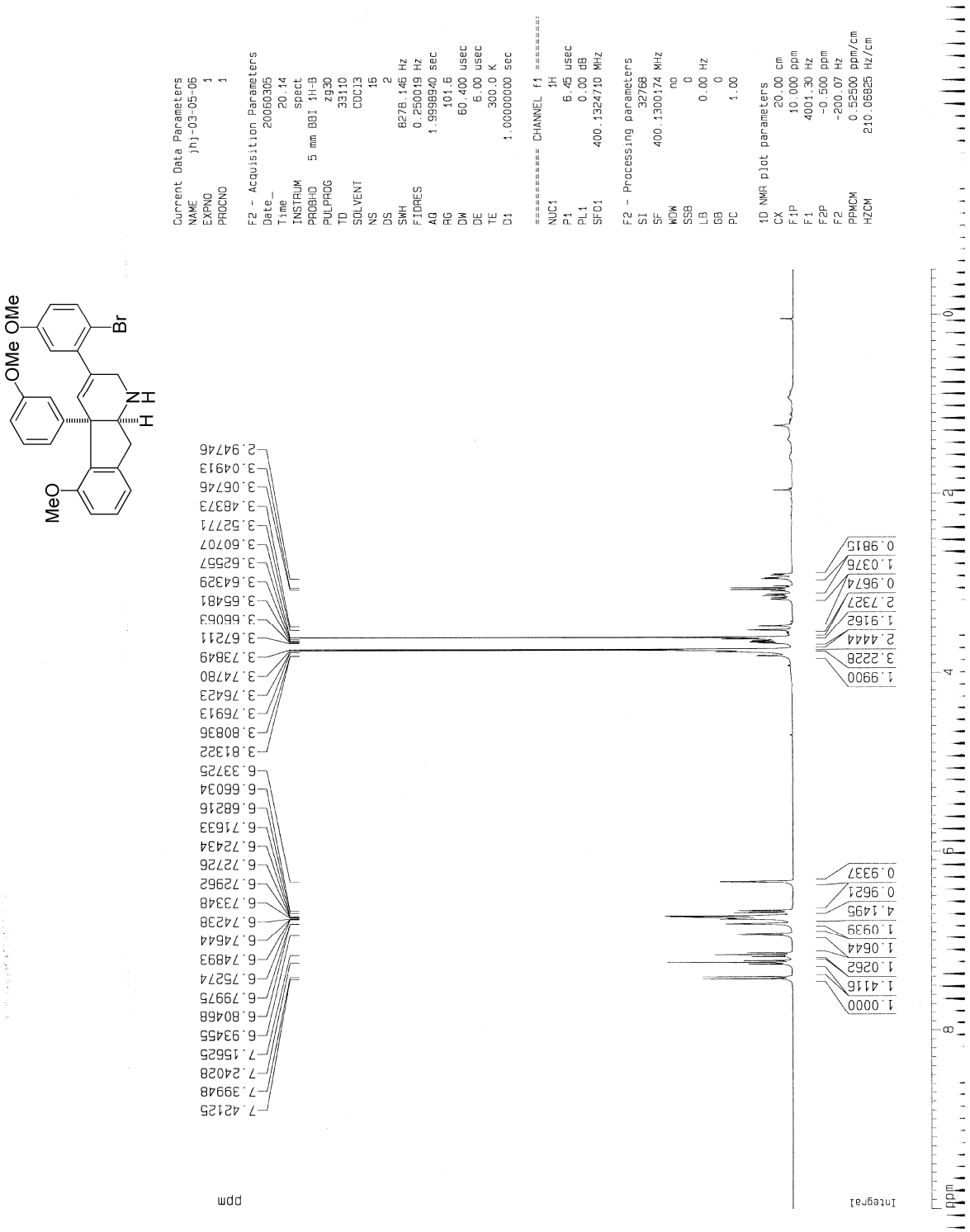
F2 - Acquisition Parameters
Date_            20060306
Time                 0.49
INSTRUM             spect
PROBHD    5 mm QNP 1H/1
PULPROG           dept135
TD                  65536
SOLVENT             CDCl3
NS                    301
DS                      4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  16384
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1            2.00000000 sec
D2            0.00357143 sec
D12           0.00002000 sec
DELTA         0.00000688 sec

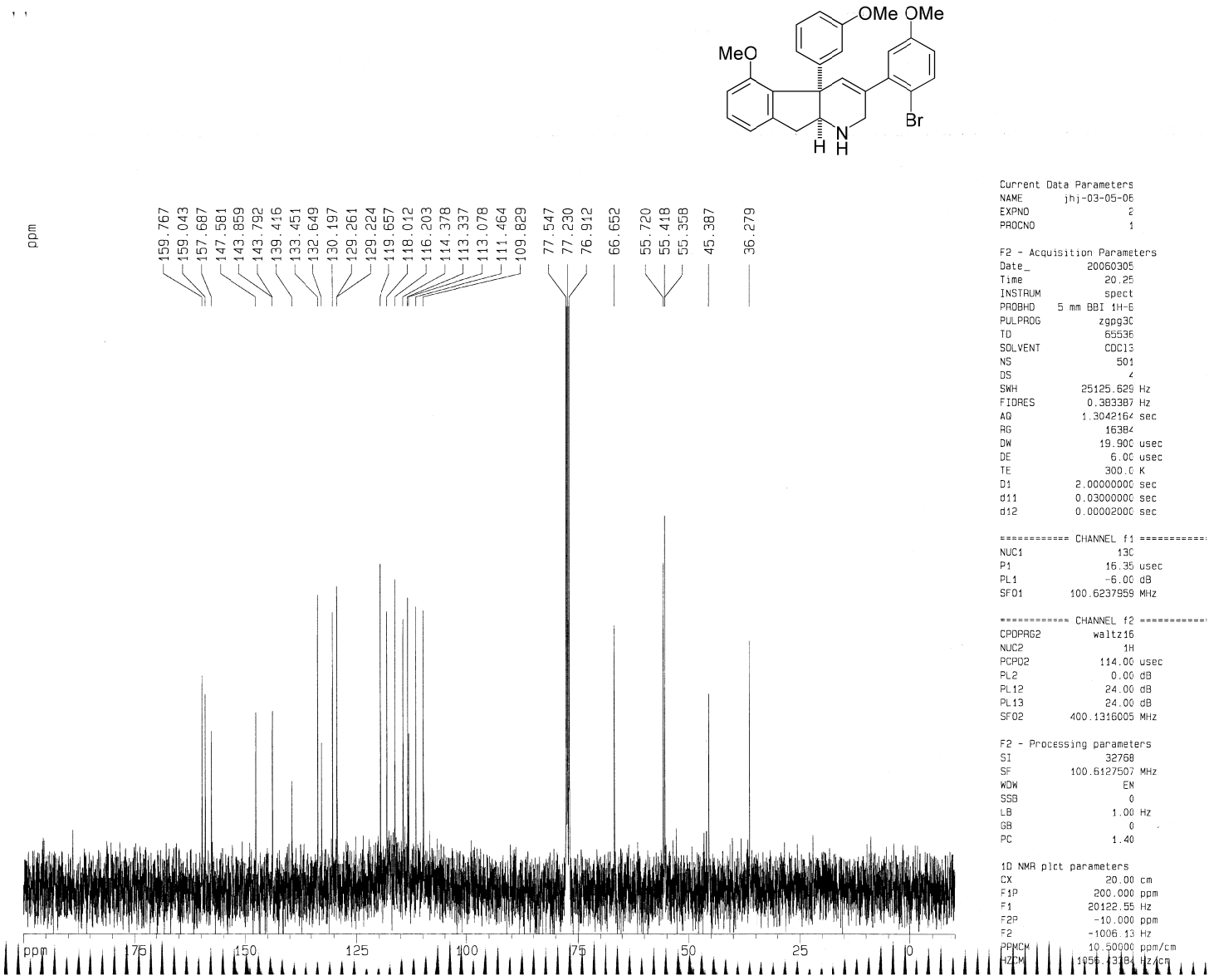
============ CHANNEL f1 ============
NUC1                  13C
P1                  5.40 usec
P2                 10.80 usec
PL1                -6.00 dB
SFO1          75.4098817 MHz

============ CHANNEL f2 ============
CPDPRG2           waltz16
NUC2                   1H
P3                 12.40 usec
P4                 24.80 usec
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               20.00 dB
SFO2         299.8711995 MHz

F2 - Processing parameters
SI                   32768
SF            75.4023602 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                200.000 ppm
F1               15080.47 Hz
F2P                -10.000 ppm
F2                 -754.02 Hz
PPMCM             10.50000 ppm/cm
HZCM             791.72479 Hz/cm
```


OMe OMe
Br
H
N
H
MeO
Current Data Parameters
NAME jhj-03-05-06
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060305
Time 20.14
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 33110
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.250019 Hz
AQ 1.9998940 sec
RG 101.6
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm
ppm
Integral




```
Current Data Parameters
NAME        jhj-03-05-06
EXPNO                  2
PROCNO                 1

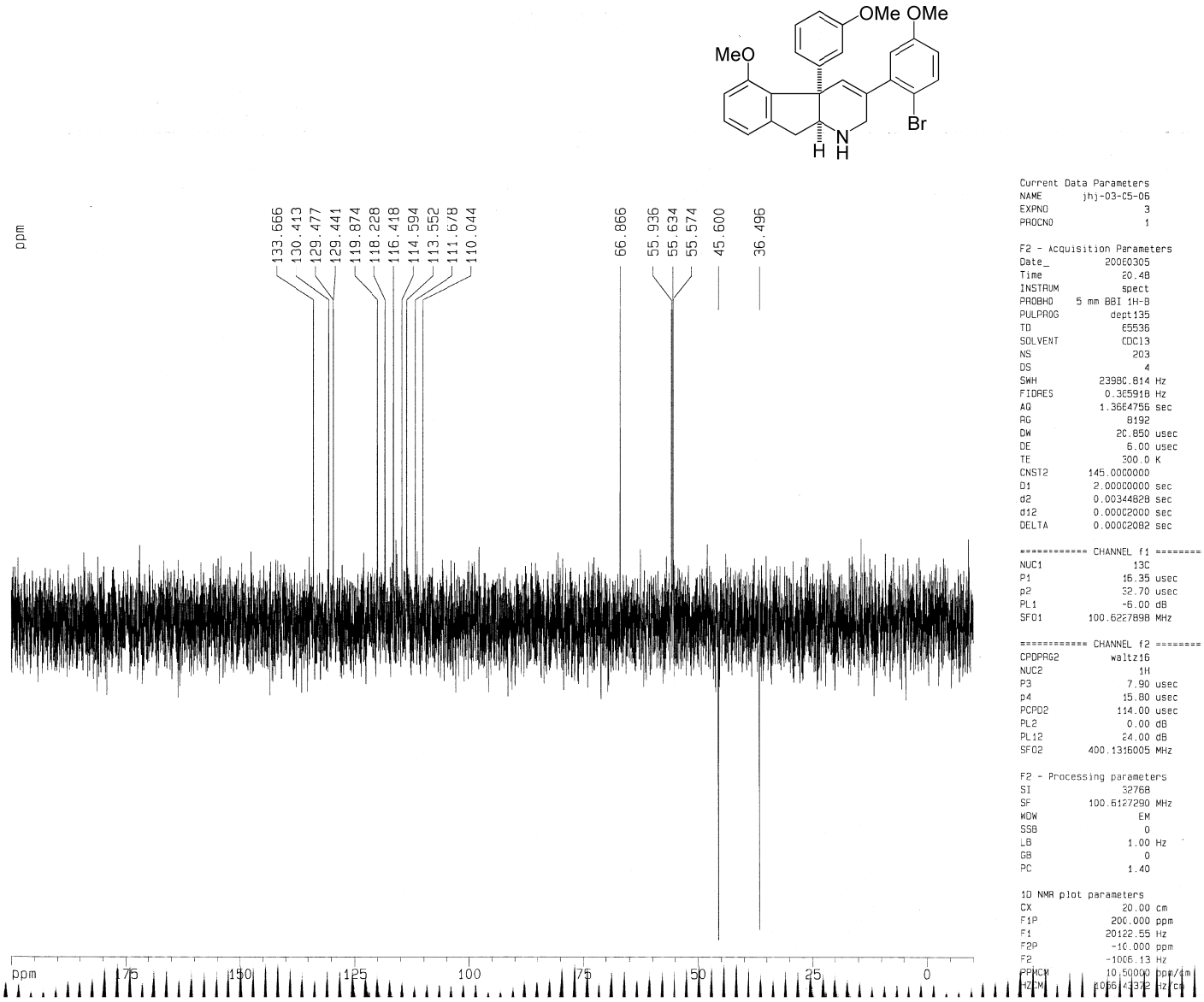
F2 - Acquisition Parameters
Date_           20060305
Time              20.25
INSTRUM           spect
PROBHD    5 mm BBI 1H-B
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  501
DS                    4
SWH          25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                16384
DW               19.900 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

========== CHANNEL f1 ==========
NUC1                13C
P1                16.35 usec
PL1               -6.00 dB
SFO1        100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2            114.00 usec
PL2                0.00 dB
PL12              24.00 dB
PL13              24.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127507 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM           10.50000 ppm/cm
HZCM          1056.43184 Hz/cm
```




```
Current Data Parameters
NAME        jhj-03-05-06
EXPNO               3
PROCNO              1

F2 - Acquisition Parameters
Date_         20060305
Time            20.48
INSTRUM         spect
PROBHD    5 mm BBI 1H-B
PULPROG         dept135
TD              65536
SOLVENT          CDCl3
NS                203
DS                  4
SWH         23980.814 Hz
FIDRES       0.365918 Hz
AQ           1.3664756 sec
RG               8192
DW             20.850 usec
DE              6.00 usec
TE             300.0 K
CNST2       145.0000000
D1           2.00000000 sec
d2           0.00344828 sec
d12          0.00002000 sec
DELTA        0.00002082 sec

============ CHANNEL f1 ========
NUC1              13C
P1              15.35 usec
p2              32.70 usec
PL1             -6.00 dB
SFO1       100.6227898 MHz

============ CHANNEL f2 ========
CPDPRG2         waltz16
NUC2              1H
P3              7.90 usec
p4             15.80 usec
PCPD2          114.00 usec
PL2             0.00 dB
PL12           24.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127290 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40

1D NMR plot parameters
CX             20.00 cm
F1P           200.000 ppm
F1           20122.55 Hz
F2P           -10.000 ppm
F2            -1006.13 Hz
PPMCM         10.50000 ppm/cm
HZCM        1056.42372 Hz/cm
```