SUPPORTING INFORMATION

A Mild Procedure for the Lewis Acid-Catalyzed Ring-Opening of Activated Cyclopropanes by Amine Nucleophiles

Olga Lifchits and André B. Charette*

Département de Chimie, Université de Montréal, P.O. Box 6128, Station Downtown, Québec, Canada H3C 3J7. E-Mail: andre.charette@umontreal.ca

General: All non-aqueous reactions were run under an inert atmosphere of argon with exclusion of moisture from reagents and glassware using standard techniques for manipulating air-sensitive compounds.[1] All glassware was stored in the oven or was flame-dried prior to use under an inert atmosphere of gas. Anhydrous solvents (benzene, diethyl ether, THF) were obtained by filtration through activated alumina columns. Flash column chromatography was performed using 230-400 mesh silica and the indicated solvent system according to standard technique.[2] Analytical thin-layer chromatography (TLC) was performed on precoated, glass-backed silica gel. Visualization of the developed chromatogram was performed by UV absorbance and/or aqueous potassium permanganate. Melting points were obtained on a melting point apparatus and are uncorrected. Nuclear magnetic resonance spectra were recorded on 300 MHz or 400 MHz spectrometers. Chemical shifts for ^{1}H NMR spectra are recorded in parts per million from tetramethylsilane with the solvent resonance as the internal standard (chloroform, δ = 7.27 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, m_c = centered multiplet and br = broad), coupling constant in Hz and integration. Chemical shifts for ^{13}C NMR spectra are recorded in parts per million from tetramethylsilane using the solvent resonance as the internal standard (chloroform, δ = 77.00 ppm). All ^{13}C NMR spectra were obtained with complete proton decoupling. When ambiguous, proton and carbon assignments were established using COSY, HMQC and DEPT 135 experiments. Where inseparable and/or interconvertible mixtures of diastereomers were obtained, the spectra are reported as observed; where chemical shifts are coincidental, they are reported with an integration of 1 H; where diastereoisomers display separate chemical shifts, integrations are reported as 1 H^{d1} and 1 H^{d2} for the first (d_1) and second (d_2) diastereoisomer, respectively. Infrared spectra were taken on an FTIR apparatus and are reported in reciprocal centimeters (cm^{-1}). High resolution mass spectra were performed by the Centre régional de spectroscopie de masse de l'Université de Montréal. Optical rotations were determined with a polarimeter at 589 nm. Data are reported as follows: $[\alpha]_{\lambda}^{temp}$, concentration (*c* in g/100 mL), and solvent. Analytical Supercritical Fluid Chromatography was performed on an instrument equipped with a diode array UV detector recording at 210 nm. Data are reported as follows: (column type, eluent, flow rate, pressure, column temperature: retention time (t_r)).

Reagents: Unless otherwise stated, commercial reagents were used without purification. Cyclopropanes **1a-1e**[3a] and *tert*-butyl 3-aminophenylcarbamate **2d**[3b] were prepared according to literature procedures.

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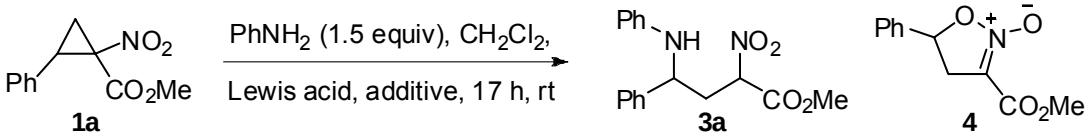
1 Shriver, D.F.; Drezdzon, M.A. *The manipulation of air-sensitive compounds*; 2nd Edition; Wiley: New York, 1986.

2 Still, W.C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

3 a) *Enantioenriched cyclopropanes **1a** and **1b**:* Moreau, B.; Charette, A.B. *J. Am. Chem. Soc.* **2005**, *127,* 18014. *Racemic cyclopropanes **1a-1e**:* Wurz, R. P.; Charette, A. B. *J. Org. Chem.* **2004**, *69*, 1262. b) Sauer, M.; Yeung, C.; Chong, J.H.; Patrick, B.O.; MacLachlan, M.J. *J. Org. Chem.* **2006**, *71*, 775.

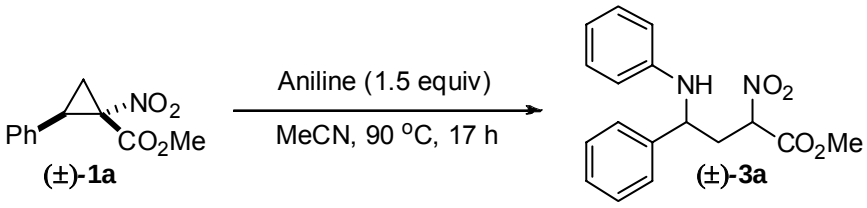
Table SI-1. Screening of the Lewis acid for ring-opening of **1a** with aniline



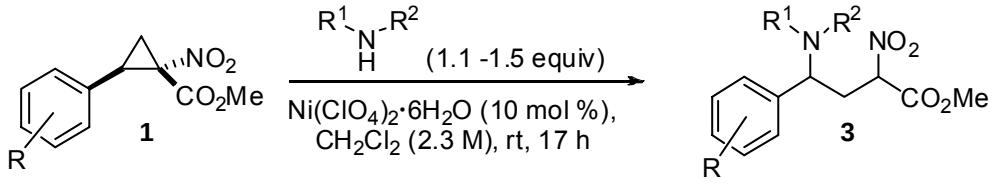
entry	Lewis acid	Additive	Ratio **1a: 3a: 4**[a]	ee of **4** (%)[b]
1	None	none	65:35:0	-
2	$Cu(OTf)_2$	none	45:48:7	-
3	$AlCl_3$	none	13:21:66	20
4	$BF_3{\cdot}OEt_2$	none	40:38:22	-
5	$SnCl_4$	none	0:0:100	10
6	$Ti(O^iPr)_4$	none	65:35:0	-
7	$ZnCl_2$	none	60:40:0	-
8	$Y(OTf)_3$	none	6:90:4	-
9	$Ni(ClO_4)_2{\cdot}6H_2O$	none	10:87:3	-
10	$Ni(ClO_4)_2{\cdot}6H_2O$	3Å MS	45:55:0	-

[a] Ratios were determined by ^{1}H NMR of the crude reaction mixture. [b] 1.0 equiv of the enantioenriched cyclopropane ***ent*-1a** (90% ee) used as the starting material

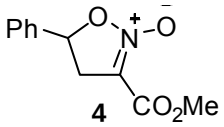
Experimental Procedures and Characterization Data



Nucleophilic ring-opening of methyl 1-nitro-2-phenylcyclopropanecarboxylate with aniline under thermal conditions. In a 2-mL microwave vial containing a magnetic stirbar, cyclopropane **(±)-1a** (100 mg, 0.45 mmol, 1 equiv) was dissolved in acetonitrile (1.5 mL), and aniline (58 µL, 0.68 mmol, 1.5 equiv) was added. The vial was sealed with a Teflon-lined cap and the reaction mixture stirred at 90 °C for 17 h. The crude reaction mixture was cooled to room temperature, evaporated under reduced pressure and purified by flash chromatography, eluting with 25% EtOAc in hexanes to afford pure **(±)-3a** as a yellow crystalline solid (122.0 mg, 0.39 mmol, 86%).

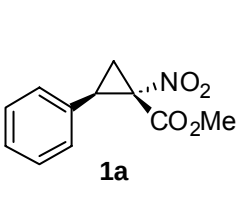


General procedure for the ring opening of methyl 1-nitro-2-arylcyclopropane carboxylates with amines under Lewis acid catalysis. In a 2-mL microwave vial containing a magnetic stirbar, cyclopropane **1** (0.23 mmol, 1 equiv) was mixed with the appropriate amine (0.34 mmol, 1.5 equiv), and dichloromethane (100 µL) was added, followed by $Ni(ClO_4)_2 \cdot 6H_2O$ (8.3 mg, 0.023 mmol, 0.1 equiv). The vial was sealed with a Teflon-lined cap to prevent solvent evaporation (a regular septum is sufficient on a larger scale), and the reaction mixture was stirred at room temperature for 17 h. The crude reaction mixture was evaporated under reduced pressure and purified by flash chromatography. In cases where the remaining excess of the aniline derivative was difficult to separate by flash chromatography, it was removed by the following aqueous workup prior to chromatographic purification: the organic layer was washed twice with 3 M HCl and the combined acidified aqueous layers were washed with diethyl ether twice. Combined organic layers were then neutralized with sat. aq. $NaHCO_3$, washed with sat. aq. NaCl and dried over Na_2SO_4.

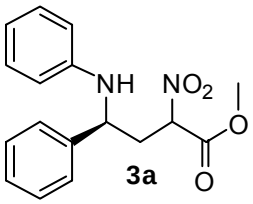


Methyl 5-phenyl-4,5-dihydroisoxazole-3-carboxylate 2-oxide (4). Beige solid; mp 89-91 °C; R_f = 0.35 (30% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.36 (m, 5H), 5.73 (dd, ^3J = 7.8 Hz, ^3J = 9.6 Hz, 1H), 3.87 (s, 3H), 3.81 (dd, ^2J = 16.9 Hz, ^3J = 9.6 Hz, 1H); 3.44 (dd, ^2J = 16.9 Hz, ^3J = 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.3, 137.6, 129.1, 129.0, 125.7, 107.7, 76.8, 52.6, 38.3; FTIR (neat) 2952, 1733, 1702, 1614, 1438, 1241, 1197, 977, 746, 700 cm^{-1}; HRMS Calcd for $C_{11}H_{11}NO_4Na$ $(M+Na)^+$: 244.0580. Found 244.0575.

SFC (Chiralcel AD-H, 5% MeOH, 2 mL/min, 200 bar, 25 °C): t_r 14.8 min (major enantiomer), t_r 19.2 min (minor enantiomer).



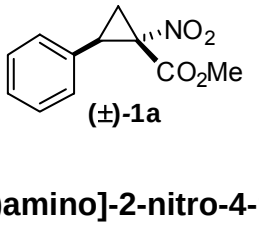
1a



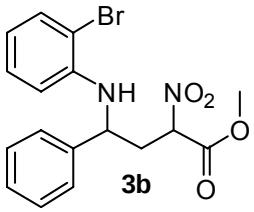
3a

Methyl (4*S*)-4-anilino-2-nitro-4-phenylbutanoate (3a). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **1a** (50.0 mg, 0.23 mmol, 1 equiv, 92% *ee*) and aniline (29.2 µL, 0.34 mmol, 1.5 equiv), and purified by the aqueous workup described above, followed by flash chromatography, eluting with 30% EtOAc in hexanes, to afford spectroscopically pure **3a** as a crystalline yellow solid (58.2 mg, 0.18 mmol, 82%, 55:45 dr, d_1 = 91.7% *ee,* d_2 = 89.7% ee). mp 64-67 °C; R_f = 0.44 (d_1), 0.50 (d_2) (30% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.42-7.25 (m, 5H), 7.17-7.10 (m, 2H), 6.72 (t, ^3J = 7.4 Hz, 1H), 6.60-6.56 (m, 2H), 5.47 (dd, ^3J = 5.1 Hz, ^3J = 8.8 Hz, 1H^{d1}), 5.15 (dd, ^3J = 5.1 Hz, ^3J = 8.6 Hz, 1H^{d2}), 4.57-4.50 (m, 1H), 4.10 (br. s, 1H), 3.84 (s, 3H^{d1}), 3.81 (s, 3H^{d2}), 2.94-2.80 (m, 1H), 2.67-2.55 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.2, 164.8, 146.2, 141.2, 140.8, 129.27, 129.25, 129.18, 129.0, 128.1, 127.9, 126.3, 126.0, 118.6, 118.4, 114.0, 113.7, 85.5, 85.3, 55.2, 54.6, 53.8, 53.7, 38.6, 37.9; FTIR (neat) 3399, 3028, 2957, 2247, 1750, 1601, 1559, 1504, 1372, 907, 729 cm^{-1}; HRMS Calcd for $C_{17}H_{19}N_2O_4$ $(M+H)^+$: 315.1339. Found 315.1337.

SFC (Chiralcel AD-H, 10% MeOH, 2.5 mL/min, 200 bar, 25 °C) t_r 5.2 min (minor enantiomer, minor diastereomer), t_r 6.3 min (minor enantiomer, major diastereomer), t_r 6.7 min (major enantiomer, minor diastereomer), t_r 11.7 min (major enantiomer, major diastereomer).

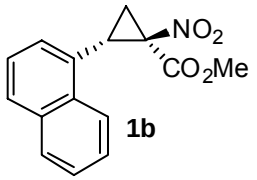


(±)-1a

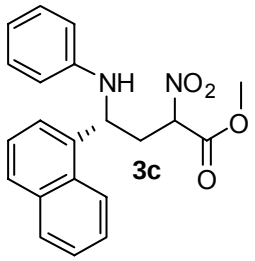


3b

Methyl 4-[(2-bromophenyl)amino]-2-nitro-4-phenylbutanoate (3b). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and 2-bromoaniline (58.3 mg, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 7% EtOAc in hexanes, to afford spectroscopically pure **3b** as a yellow oil (73.8 mg, 0.19 mmol, 83%, 50:50 dr). R_f = 0.76 (30% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.45-7.26 (m, 6H), 7.09-7.02 (m, 1H), 6.58 (m_c, 1H), 6.50 (ddd, ^3J = 8.3 Hz, ^3J = 13.0 Hz, ^4J = 1.4 Hz, 1H), 5.46 (dd, ^3J = 5.2 Hz, ^3J = 8.6 Hz, 1H^{d1}), 5.12 (dd, ^3J = 4.9 Hz, ^3J = 8.9 Hz, 1H^{d2}), 4.75 (m, 1H), 4.63-4.52 (m, 1H), 3.87 (s, 3H^{d1}), 3.82 (s, 3H^{d2}), 3.01-2.84 (m, 1H), 2.72-2.61 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.0, 164.7, 143.08, 143.03, 140.6, 140.1, 132.45, 132.43, 129.3, 129.1, 128.5, 128.4, 128.3, 128.0, 126.3, 125.9, 119.1, 118.9, 113.0, 112.7, 110.5, 110.3, 85.31, 85.29, 55.1, 54.7, 53.9, 53.8, 38.7, 38.0; FTIR (neat) 3393, 2954, 1750, 1595, 1558, 1453, 1268, 1019, 908, 741 cm^{-1}; HRMS Calcd for $C_{17}H_{18}N_2O_4$ $(M+H)^+$: 393.0444. Found 393.0449.



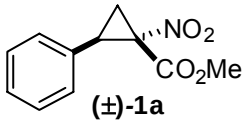
1b



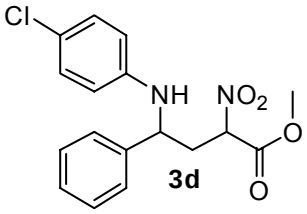
3c

Methyl (4*R*)-4-anilino-4-(1-naphthyl)-2-nitrobutanoate (3c). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **1b** (50.0 mg, 0.18 mmol, 1 equiv, 92% ee) and aniline (23.8 µL, 0.28 mmol, 1.5 equiv), and purified by the aqueous workup described above, followed by flash chromatography, eluting with a gradient of 15% to 30% EtOAc in hexanes, to afford spectroscopically pure **3c** as an off-white foam (49.3 mg, 0.13 mmol, 73%, 55:45 dr, d_1 = 91.5% ee, d_2 = 91.7% ee). R_f = 0.59 (d_1), 0.69 (d_2) (10% ethyl acetate in toluene); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 8.24 (d, ^3J = 8.3 Hz, 1H^{d1}), 8.19 (d, ^3J = 7.9 Hz, 1H^{d2}), 7.94 (d, ^3J = 8.2 Hz, 1H), 7.82 (d, ^3J = 6.8 Hz, 1H^{d1}), 7.80 (d, ^3J = 6.3 Hz, 1H^{d2}), 7.69-7.39 (m, 4H), 7.14-7.06 (m, 2H), 6.74-6.68 (m, 1H), 6.61-6.55 (m, 2H), 5.69 (dd, ^3J = 4.2 Hz, ^3J = 9.4 Hz, 1H^{d1}), 5.50-5.36 (m, 1H + 1H^{d2}), 4.29 (br. s, 1H), 3.87 (s, 3H^{d1}), 3.81 (s, 3H^{d2}), 3.16-3.06 (m, 1H^{d1}), 3.04-2.95 (m, 1H^{d2}), 2.83-2.73 (m, 1H^{d1}), 2.58-2.48 (m, 1H^{d2}); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.3, 165.0, 146.1, 146.0, 136.4, 136.2, 134.12, 134.06, 130.8, 130.6, 129.32, 129.30, 129.28, 129.25, 128.6, 128.4, 126.9, 126.0, 125.9, 125.6, 125.5, 122.7, 122.3, 122.1, 121.9, 118.6, 118.4, 113.8, 113.6, 85.5, 85.3, 53.81, 53.78, 50.8, 50.3, 37.7, 37.3; FTIR (neat) 3393, 3052, 2956, 1752, 1601, 1504, 1436, 1255, 778 cm^{-1}; HRMS Calcd for $C_{21}H_{21}N_2O_4$ (M+H)$^+$: 365.1496. Found 365.1494.

SFC (Chiralcel AD-H, 5% MeOH, 5 mL/min, 150 bar, 25 °C) t_r 5.3 min (minor enantiomer, minor diastereomer), t_r 6.3 min (major enantiomer, minor diastereomer), t_r 6.7 min (major enantiomer, major diastereomer), t_r 8.7 min (minor enantiomer, major diastereomer).

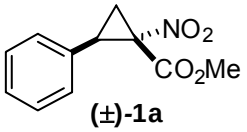


(±)-1a

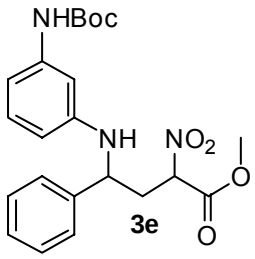


3d

Methyl 4-[(4-chlorophenyl)amino]-2-nitro-4-phenylbutanoate (3d). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and 4-chloroaniline (43.2, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 10% EtOAc in hexanes, to afford spectroscopically pure **3d** as a yellow oil (67.9 mg, 0.19 mmol, 86%, 50:50 dr). R_f = 0.52 (d_1), 0.59 (d_2) (10% ethyl acetate in toluene); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.42-7.26 (m, 5H), 7.07 (m_c, 2H), 6.50 (m_c, 2H), 5.45 (dd, ^3J = 5.1 Hz, ^3J = 8.7 Hz, 1H^{d1}), 5.14 (dd, ^3J = 5.0 Hz, ^3J = 8.8 Hz, 1H^{d2}), 4.47 (m_c, 1H), 4.12 (br. s, 1H), 3.83 (s, 3H^{d1}), 3.80 (s, 3H^{d2}), 2.92-2.79 (m, 1H), 2.66-2.54 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.1, 164.7, 144.8, 144.7, 140.7, 140.3, 129.2, 129.08, 129.07, 129.05, 128.3, 128.0, 126.2, 125.9, 123.1, 123.0, 115.1, 114.8, 85.4, 85.2, 55.3, 54.7, 53.80, 53.79, 38.4, 37.8; FTIR (neat) 3400, 3029, 1749, 1598, 1559, 1495, 1254, 908, 731 cm^{-1}; HRMS Calcd for $C_{17}H_{18}N_2O_4Cl$ (M+H)$^+$: 349.0950. Found 349.0948.

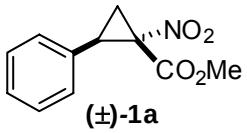


(±)-1a

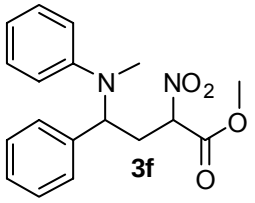


3e

Methyl 4-({3-[(*tert*-butoxycarbonyl)amino]phenyl}amino)-2-nitro-4-phenylbutanoate (3e). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and *tert*-butyl 3-aminophenylcarbamate **2d**[3b] (70.6 mg, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 20% EtOAc in hexanes, to afford spectroscopically pure **3e** as a crystalline yellow solid (64.2 mg, 0.15 mmol, 66%, 50:50 dr). mp 112-114 °C; R_f = 0.53 (30% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.39-7.23 (m, 5H), 7.00 (m_c, 1H), 6.87 (br. s, 1H^{d1}), 6.83 (br. s, 1H^{d2}), 6.60-6.53 (m, 1H), 6.40 (br. s, 1H), 6.22 (m_c, 1H), 5.44 (dd, ^3J = 5.1 Hz, ^3J = 9.0 Hz, 1H^{d1}), 5.12 (dd, ^3J = 5.3 Hz, ^3J = 8.8 Hz, 1H^{d2}), 4.51-4.47 (m, 1H), 4.12 (br. s, 1H), 3.83 (s, 3H^{d1}), 3.80 (s, 3H^{d2}), 2.89-2.77 (m, 1H), 2.63-2.48 (m, 1H), 1.50 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.2, 164.8, 152.6, 147.03, 147.02, 141.1, 140.7, 139.3, 129.76, 129.74, 129.1, 129.0, 128.1, 127.8, 126.3, 125.9, 108.6, 108.5, 108.1, 108.0, 104.3, 104.0, 85.4, 85.3, 80.3, 55.0, 54.3, 53.8, 53.7, 38.0, 37.8, 28.3; FTIR (neat) 3385, 2977, 1751, 1707, 1559, 1526, 1479, 1231, 908, 729.cm^{-1}; HRMS Calcd for $C_{22}H_{28}N_3O_6$ $(M+H)^+$: 430.1973. Found 430.1980.

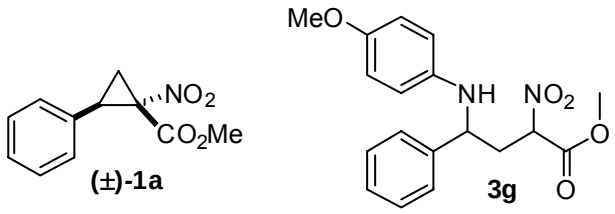


(±)-1a

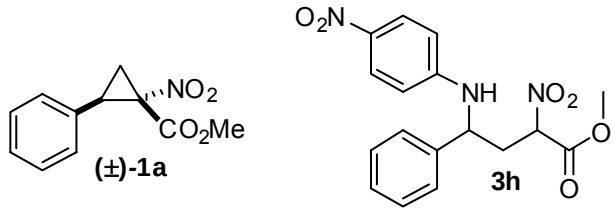


3f

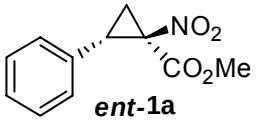
Methyl 4-[methyl(phenyl)amino]-2-nitro-4-phenylbutanoate (3f). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and N-methylaniline (36.7 µL, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 100% toluene, to afford spectroscopically pure **3f** as a yellow oil (59.2 mg, 0.18 mmol, 80%, 55:45 dr). R_f = 0.81 (10% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.39-7.16 (m, 7H), 6.88-6.81 (m, 3H), 5.45 (dd, ^3J = 4.2 Hz, ^3J = 9.5 Hz, 1H^{d1}), 5.29-5.20 (m, 1H^{d2} + 1H^{d1}), 5.10 (dd, ^3J = 4.6 Hz, ^3J = 11.5 Hz, 1H^{d2}), 3.84 (s, 3H^{d1}), 3.80 (s, 3H^{d2}), 3.11-2.91 (m, 2H), 2.62 (s, 3H^{d1}), 2.60 (s, 3H^{d2}); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.3, 164.9, 150.1, 150.0, 138.0, 137.8, 129.4, 129.3, 128.6, 128.5, 127.84, 127.77, 126.9, 126.8, 118.6, 118.4, 114.6, 114.3, 85.6, 85.4, 59.05, 59.03, 53.7, 53.6, 32.2, 32.0, 31.81, 31.78; FTIR (neat) 2955, 1750, 1596, 1558, 1372, 1266, 1108, 991, 750, 697 cm^{-1}; HRMS Calcd for $C_{18}H_{21}N_2O_4$ $(M+H)^+$: 329.1496. Found 329.1497.



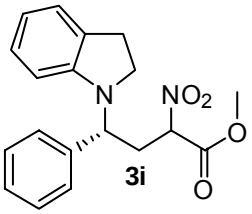
Methyl 4-[(4-methoxyphenyl)amino]-2-nitro-4-phenylbutanoate (3g). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and *p*-methoxyaniline (41.7 mg, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 30% EtOAc in hexanes, to afford spectroscopically pure **3g** as a dark yellow oil (55.0 mg, 0.16 mmol, 71%, 50:50 dr). R_f = 0.47 (d_1), 0.52 (d_2) (30% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.40-7.23 (m, 5H), 6.73-6.69 (m, 2H), 6.55-6.51 (m, 2H), 5.52 (dd, ^3J = 5.1 Hz, ^3J = 8.8 Hz, 1H^{d1}), 5.17 (dd, ^3J = 5.0 Hz, ^3J = 8.6 Hz, 1H^{d2}), 4.46-4.40 (m, 1H), 3.83 (s, 3H^{d1}), 3.81 (s, 3H^{d2}), 3.78 (br. s, 1H), 3.71 (s, 3H^{d1}), 3.70 (s, 3H^{d2}), 2.91-2.77 (m, 1H), 2.64-2.51 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.3, 164.9, 152.8, 152.7, 141.4, 141.0, 140.2, 140.1, 129.1, 129.0, 128.0, 127.8, 126.3, 126.0, 115.7, 115.3, 114.7, 85.6, 85.4, 56.1, 55.7, 55.59, 55.56, 53.7, 38.6, 38.0; FTIR (neat) 3372, 2956, 1750, 1708, 1557, 1439, 1357, 1237, 822, 735 cm^{-1}; HRMS Calcd for $C_{18}H_{21}N_2O_5$ $(M+H)^+$: 355.1445. Found 345.1443.



Methyl 2-nitro-4-[(4-nitrophenyl)amino]-4-phenylbutanoate (3h). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and *p*-nitroaniline (41.7 mg, 0.34 mmol, 1.5 equiv). To achieve full conversion, the reaction mixture was stirred for 48h before being evaporated under reduced pressure. The crude mixture was purified by flash chromatography, eluting with 5% EtOAc in toluene, to afford spectroscopically pure **3h** as a bright yellow viscous oil (74.9 mg, 0.21 mmol, 92%, 50:50 dr). R_f = 0.41 (5% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 8.49 (d, ^3J = 6.1 Hz, 1H^{d1}), 8.43 (d, ^3J = 7.8 Hz, 1H^{d2}), 8.20 (dd, ^3J = 8.7 Hz, ^4J = 1.5 Hz, 1H^{d1}), 8.18 (dd, ^3J = 8.6, ^4J = 1.5 Hz, 1H^{d1}), 7.45-7.31 (m, 5H + 2H^{d2}), 6.77-6.67 (m, 2H), 5.40 (dd, ^3J = 4.7 Hz, ^3J = 9.3 Hz, 1H^{d1}), 5.01 (dd, ^3J = 4.4 Hz, ^3J = 9.8 Hz, 1H^{d2}), 4.74-4.61 (m, 1H), 3.87 (s, 3H^{d1}), 3.81 (s, 3H^{d2}), 3.11-2.88 (m, 1H), 2.77-2.61 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.5, 164.3, 143.8, 143.6, 139.8, 139.1, 136.4, 136.3, 133.0, 132.8, 129.6, 129.4, 128.8, 128.4, 126.8, 126.4, 125.9, 116.8, 116.6, 114.8, 114.5, 85.0, 84.9, 54.4, 54.1, 54.0, 53.9, 38.7, 37.7; FTIR (neat) 3362, 2957, 1752, 1561, 1501, 1417, 1350, 1235, 1039, 910, 742 cm^{-1}; HRMS Calcd for $C_{17}H_{17}N_3O_6Na$ $(M+Na)^+$: 382.1010. Found 382.1006.



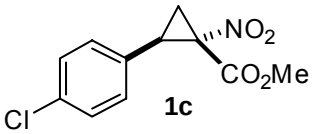
***ent*-1a**



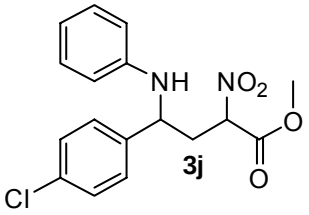
3i

Methyl (4*R*)-4-(2,3-dihydro-1*H*-indol-1-yl)-2-nitro-4-phenylbutanoate (3i). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane ***ent*-1a** (50.0 mg, 0.23 mmol, 1 equiv, 90% ee) and indoline (38 µL, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 20% EtOAc in hexanes, to afford spectroscopically pure **3i** as a beige solid (72.3 mg, 0.21 mmol, 94%, 55:45 dr, d_1 = 90.0% ee, d_2 = 89.6% ee). mp 82-85 °C, R_f = 0.76 (30% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.39-7.25 (m, 5H), 7.12-7.04 (m, 2H), 6.69-6.58 (m, 2H), 5.50 (dd, ^3J = 5.1 Hz, ^3J = 8.9 Hz, 1H^{d1}), 5.26 (dd, ^3J = 6.3 Hz, ^3J = 7.3 Hz, 1H^{d2}), 4.90 (dd, ^3J = 6.4 Hz, ^3J = 9.2 Hz, 1H^{d1}), 4.83 (dd, ^3J = 4.7 Hz, ^3J = 11.0 Hz, 1H^{d2}), 3.80 (s, 3H^{d1}), 3.79 (s, 3H^{d2}), 3.44-3.35 (m, 1H), 3.12-2.80 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.3, 164.9, 150.6, 150.5, 137.2, 137.1, 129.7, 129.5, 128.8, 128.6, 128.1, 128.0, 127.7, 127.5, 127.4, 127.3, 124.8, 124.7, 117.94, 117.91, 107.1, 107.0, 85.60, 85.58, 55.7, 55.0, 53.62, 53.58, 46.7, 46.2, 32.2, 31.8, 28.0; FTIR (neat) 3029, 2955, 2849, 1750, 1605, 1558, 1485, 1436, 1328, 1254, 1002, 873, 745 cm^{-1}; HRMS Calcd for $C_{19}H_{21}N_2O_4$ $(M+H)^+$: 341.1496. Found 341.1499.

SFC (Chiralcel OJ-H, 10% ^{i}PrOH, 2 mL/min, 150 bar, 25 °C) t_r 10.9 min (minor enantiomer, minor diastereomer), t_r 15.0 min (minor enantiomer, major diastereomer), t_r 18.6 min (major enantiomer, minor diastereomer), t_r 21.6 min (major enantiomer, major diastereomer).

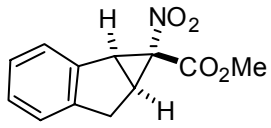


1c

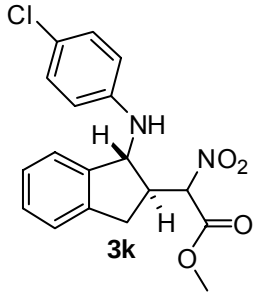


3j

Methyl 4-anilino-4-(4-chlorophenyl)-2-nitrobutanoate (3j). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **1c** (57.8 mg, 0.23 mmol, 1 equiv) and aniline (29.2 µL, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 100% toluene, to afford spectroscopically pure **3j** as a yellow oil (58.6 mg, 0.17 mmol, 74%, 50:50 dr). R_f = 0.43 (toluene); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.36-7.24 (m, 4H), 7.13 (m_c, 2H), 6.74 (t, ^3J = 7.3 Hz, 1H^{d1}), 6.73 (t, ^3J = 7.3 Hz, 1H^{d2}), 6.54 (m_c, 2H), 5.46 (dd, ^3J = 4.9 Hz, ^3J = 8.9 Hz, 1H^{d1}), 5.16 (dd, ^3J = 5.3 Hz, ^3J = 8.3 Hz, 1H^{d2}), 4.54-4.49 (m, 1H), 4.04 (br. s, 1H), 3.84 (s, 3H^{d1}), 3.82 (s, 3H^{d2}), 2.88-2.77 (m, 1H), 2.65-2.50 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.1, 164.7, 145.86, 145.84, 139.8, 139.5, 133.8, 133.6, 129.34, 129.32, 129.29, 129.19, 127.7, 127.4, 118.8, 118.7, 114.0, 113.8, 85.3, 85.2, 54.6, 54.1, 53.86, 53.84, 38.5, 38.0; FTIR (neat) 3394 (br), 2957, 1750, 1601, 1559, 1490, 1436, 1372, 1313, 1265, 1179, 1090, 826, 752, 692 cm^{-1}; HRMS Calcd for $C_{17}H_{18}N_2O_4Cl$ $(M+H)^+$: 349.0950. Found 349.0954.

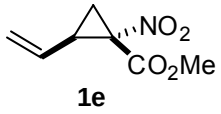


1d

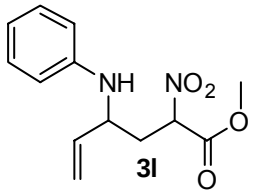


3k

Methyl {1-[(4-chlorophenyl)amino]-2,3-dihydro-1*H*-inden-2-yl}(nitro)acetate (3k). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **1d** (52.7 mg, 0.23 mmol, 1 equiv) and *p*-chloroaniline (43.2 mg, 0.34 mmol, 1.5 equiv), and purified by flash chromatography, eluting with 100% toluene, to afford the spectroscopically pure **3k** as an orange solid (60.3 mg, 0.18 mmol, 78%, 70:30 dr). mp 111-113 °C; R_f = 0.43 (toluene); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.31-7.15 (m, 6H), 6.71-6.64 (m, 2H), 5.47 (d, ^3J = 5.7 Hz, 1H^{d1}), 5.39 (d, ^3J = 8.7 Hz, 1H^{d2}), 5.26 (t, ^3J = 9.1 Hz, 1H^{d1}), 5.04 (t, ^3J = 9.1 Hz, 1H^{d2}), 3.91 (br. s, 1H^{d1}), 3.88 (br. s, 1H^{d2}), 3.81 (s, 3H^{d1}), 3.64 (s, 3H^{d2}), 3.40-3.30 (m, 1H), 3.25-3.10 (m, 1H), 2.93-2.84 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 164.4, 164.2, 145.7, 145.6, 142.5, 142.4, 139.4, 139.2, 129.41, 129.37, 128.54, 128.52, 127.4, 125.05, 125.02, 124.0, 123.8, 123.1, 123.0, 114.5, 114.4, 89.6, 88.1, 60.5, 59.6, 53.6, 43.4, 33.9, 32.7; FTIR (neat) 3392, 2955, 1751, 1598, 1501, 1459, 1293, 1179, 1004, 910, 817, 750 cm^{-1}; HRMS Calcd for $C_{18}H_{18}N_2O_4Cl$ $(M+H)^+$: 361.0950. Found 361.0945.

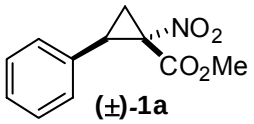


1e

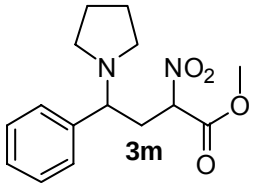


3l

Methyl 4-anilino-2-nitrohex-5-enoate (3l). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **1e** (39.0 mg, 0.23 mmol, 1 equiv) and aniline (29.2 µL, 0.34 mmol, 1.5 equiv). The crude reaction mixture was washed twice with 3 M HCl and the combined acidified aqueous layers were washed with dichloromethane twice. Combined organic layers were then neutralized with sat. aq. $NaHCO_3$, washed with sat. aq. NaCl and dried over Na_2SO_4. After evaporating the solvent under reduced pressure, the crude product was purified by flash chromatography, eluting with 30% EtOAc in hexanes, to afford spectroscopically pure **3l** as a yellow oil (40.0 mg, 0.15 mmol, 67%, 50:50 dr). R_f = 0.36 (15% ethyl acetate in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.18 (m_c, 2H), 6.76 (t, ^3J = 7.3 Hz, 1H), 6.62 (m_c, 2H), 5.83-5.70 (m, 1H), 5.47 (dd, ^3J = 4.9 Hz, ^3J = 8.8 Hz, 1H^{d1}), 5.33 (dd, ^3J = 5.4 Hz, ^3J = 8.3 Hz, 1H^{d2}), 5.31-5.18 (m, 2H), 4.02 (m_c, 1H), 3.84 (s, 3H^{d1}), 3.83 (s, 3H^{d2}), 3.56 (br. s, 1H), 2.74-2.59 (m, 1H), 2.49-2.31 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.2, 165.0, 146.4, 146.3, 137.4, 137.2, 129.34, 129.32, 118.7, 118.6, 117.8, 116.8, 114.1, 113.8, 85.2, 85.1, 53.7, 53.3, 52.8, 35.8, 35.4; FTIR (neat) 3384, 2957, 1749, 1601, 1557, 1498, 1360, 1310, 1217, 992, 751 cm^{-1}; HRMS Calcd for $C_{13}H_{17}N_2O_4$ $(M+H)^+$: 265.1183. Found 265.1174.

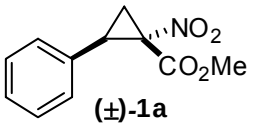


(±)-1a

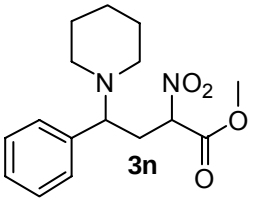


3m

Methyl 2-nitro-4-phenyl-4-pyrrolidin-1-ylbutanoate (3m). The title compound was prepared by the Lewis acid-catalyzed procedure described above using cyclopropane **(±)-1a** (50.0 mg, 0.23 mmol, 1 equiv) and pyrrolidine (39.5 µL, 0.48 mmol, 2.1 equiv), stirring the reaction mixture for 48 hours. It was purified by flash chromatography, eluting with 5% MeOH in dichloromethane, to afford spectroscopically pure **3m** as a yellow oil (60.5 mg, 0.21 mmol, 90%, 60:40 dr). R_f = 0.30 (5% MeOH in dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.40-7.22 (m, 5H), 5.22 (m_c, 1H^{d1}), 4.83 (dd, ^3J = 3.4 Hz, ^3J = 11.0 Hz, 1H^{d2}), 3.82 (s, 3H^{d1}), 3.76 (s, 3H^{d2}), 3.50 (m_c, 1H^{d1}), 3.19 (dd, ^3J = 4.6 Hz, ^3J = 10.0 Hz, 1H^{d2}), 3.06 (m_c, 1H^{d1}), 2.92 (m_c, 1H^{d2}), 2.68-2.35 (m, 5H), 1.79-1.66 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.2, 165.1, 139.7, 138.0, 128.7, 128.4, 128.3, 128.1, 127.9, 85.9, 85.6, 66.0, 63.9, 53.5, 53.4, 52.1, 50.2, 36.0, 35.0, 23.2, 23.0; FTIR (neat) 2959, 2795, 1754, 1561, 1454, 1436, 1262, 1210, 1135, 884, 704 cm^{-1}; HRMS Calcd for $C_{15}H_{21}N_2O_4$ $(M+H)^+$: 293.1496. Found 293.1498.

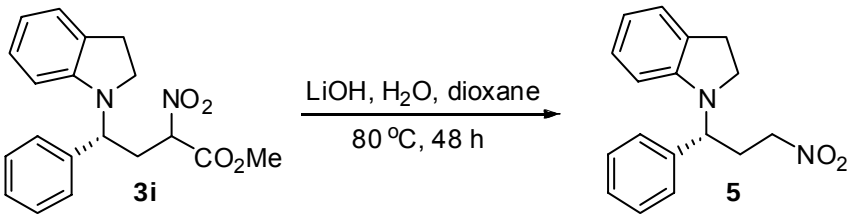


(±)-1a



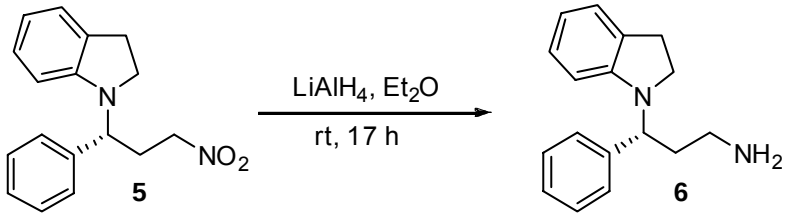
3n

Methyl 2-nitro-4-phenyl-4-pyrrolidin-1-ylbutanoate (3n). In a 2-mL microwave vial containing a magnetic stirbar, cyclopropane **(±)-1** (100 mg, 0.45 mmol, 1 equiv) was mixed with piperidine (67 µL, 0.68 mmol, 1.5 equiv), and dichloromethane (200 µL) was added, followed by $Ni(ClO_4)_2{\cdot}6H_2O$ (8.3 mg, 0.023 mmol, 0.1 equiv.) The vial was sealed with a Teflon-lined cap and the reaction mixture was stirred at room temperature for 48 h. The crude reaction was evaporated under reduced pressure and purified by flash chromatography, eluting with 5% MeOH in dichloromethane, affording spectroscopically pure **3n** as a beige crystalline solid (87.3 mg, 0.28 mmol, 63%, 50:50 dr). mp 68-71 °C; R_f = 0.20 (5% MeOH in dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 7.39-7.31 (m, 3H), 7.16 (m_c, 2H), 5.67 (m_c, 1H^{d1}); 5.22 (dd, ^3J = 5.6 Hz, ^3J = 8.3 Hz, 1H^{d2}), 3.87 (s, 3H^{d1}), 3.81 (s, 3H^{d2}), 3.58-3.52 (m, 1H), 3.15-3.04 (m, 1H^{d1}), 2.94 (m_c, 1H^{d2}), 2.56-2.38 (m, 4H^{d1} + 1H), 2.15 (m_c, 4H^{d2}), 1.50 (m_c, 4H), 1.33-1.27 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of 2 diastereomers) δ 165.9, 165.4, 136.4, 135.7, 128.5, 128.2, 128.0, 127.8, 127.7, 86.8, 86.1, 67.2, 63.5, 53.5, 50.8, 50.3, 32.5, 32.0, 26.3, 26.0, 24.4; FTIR (neat) 3029, 2933, 2806, 1751, 1558, 1436, 1371, 1160, 1100, 871, 702 cm^{-1}; HRMS Calcd for $C_{16}H_{23}N_2O_4$ $(M+H)^+$: 307.1652. Found 307.1654.



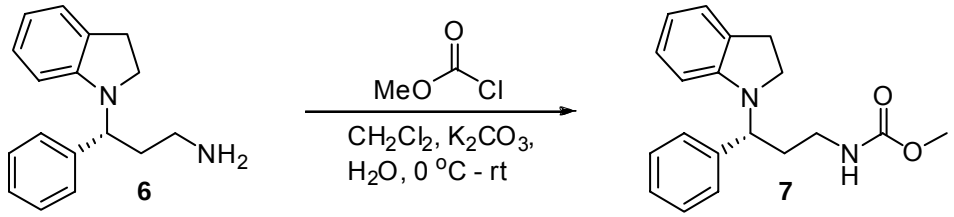
1-[(1*R*)-3-nitro-1-phenylpropyl]indoline (5). To a solution of **3i** (500 mg, 1.5 mmol, 1 equiv, 90% ee) in dioxane (10 mL) and water (5 mL) was added LiOH (53 mg, 2.2 mmol, 1.5 equiv), and the reaction mixture was stirred at 80 °C for 48 h, after which all of the starting material was consumed. The crude reaction mixture was cooled to room temperature, neutralized with 1 M HCl and partitioned with EtOAc. The aqueous phase was extracted with EtOAc three times, and the combined organic phases were washed with sat. aq. NaCl and dried over Na_2SO_4. After evaporating the solvent under reduced pressure, the crude product was purified by flash chromatography, eluting with 20% EtOAc in hexanes, affording spectroscopically pure **5** as a crystalline yellow solid (369.5 mg, 1.31 mmol, 89%, 90% ee). $[\alpha]_D^{20}$: + 150.8 (*c* 0.455, MeOH); mp 69-71 °C; R_f = 0.76 (30% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.26 (m, 5H), 7.06 (m_c, 2H), 6.64 (ddd, ^3J = 7.6 Hz, ^3J = 7.3 Hz, ^4J = 0.9 Hz, 1H), 6.56 (d, ^3J = 8.1 Hz, 1H), 4.82 (dd, ^3J = 5.9 Hz, ^3J = 9.6 Hz, 1H), 4.62-4.46 (m, 2H), 3.45-3.37 (m, 1H), 3.20-3.11 (m, 1H), 3.02-2.88 (m, 2H), 2.84-2.61 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.8, 138.0, 129.6, 128.7, 127.9, 127.6, 127.4, 124.7, 117.6, 106.8, 73.0, 56.0, 46.7, 28.9, 28.1; FTIR (neat) 3027, 2925, 2848, 1604, 1546, 1381, 1255, 1024, 919, 873, 744 cm^{-1}; HRMS Calcd for $C_{17}H_{19}N_2O_2$ $(M+H)^+$: 283.1441. Found 283.1434.

SFC (Chiralcel OD-H, 20% MeOH, 4 mL/min, 100 bar, 25 °C) t_r 5.8 min (minor enantiomer), t_r 15.5 min (major enantiomer).



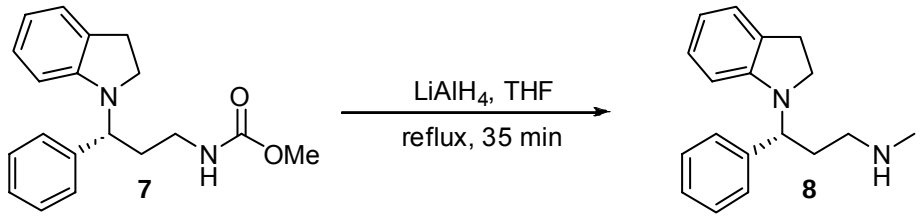
(3*R*)-3-(2,3-dihydro-1*H*-indol-1-yl)-3-phenylpropylamine (6). In a flame-dried 25 mL round bottom flask, **5** (200 mg, 0.71 mmol, 1 equiv) was dissolved in anhydrous diethyl ether (6 mL) under an atmosphere of argon. $LiAlH_4$ (107 mg, 2.8 mmol, 4 equiv) was added quickly in one portion at 0 °C. When the exotherm ceased, the reaction mixture was warmed up to room temperature and stirred overnight. The crude reaction mixture was quenched with several drops of H_2O at 0 °C, and several drops of 2M NaOH were added so that a thick white precipitate formed. This suspension was filtered through a pad of Celite washing with diethyl ether, yielding a clear yellowish solution, which was evaporated under reduced pressure to afford spectroscopically pure **6** (177 mg, 0.70 mmol, 99%, 90% ee) which was used without further purification. $[\alpha]_D^{20}$: + 155.4 (*c* 0.975, MeOH); R_f = 0.15 (20% MeOH in dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.25 (m, 5H), 7.07-7.02 (m, 2H), 6.61–6.53 (m, 2H), 4.79 (dd, ^3J = 7.1 Hz, ^3J = 7.9 Hz), 3.49 (m_c, 1H), 3.28 (m_c, 1H), 3.04-2.88 (m, 2H), 2.83 (t, ^3J = 7.2 Hz, 2H), 2.28-2.02 (m, 2H), 1.29 (br. s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.4, 140.2, 129.5, 128.3, 127.7, 127.2, 127.1, 124.5, 116.6, 106.3,

53.4, 46.8, 39.7, 35.0, 28.1; FTIR (neat) 3025, 2924, 2847, 1605, 1487, 1329, 1258, 1157, 837, 742, 631 cm^{-1}; HRMS Calcd for $C_{17}H_{21}N_2$ $(M+H)^+$: 253.1699. Found 253.1697.



Methyl (3*R*)-3-(2,3-dihydro-1*H*-indol-1-yl)-3-phenylpropylcarbamate (7). To a solution of **6** (165 mg, 0.65 mmol, 1 equiv) in dichloromethane (5 mL) was added methyl chloroformate (61 µL, 0.78 mmol, 1.2 equiv). The solution was cooled to 0 °C and K_2CO_3 (362 mg, 2.6 mmol, 4 equiv) and H_2O (5 mL) were added. The reaction was warmed to room temperature and stirred for 30 min. Water (5 mL) was added and the reaction mixture was extracted with dichloromethane three times, dried over Na_2SO_4 and evaporated under reduced pressure. Flash chromatography, eluting with 30% EtOAc in hexanes, afforded the spectroscopically pure **7** as a colourless oil (191.0 mg, 0.61 mmol, 94%, 90% ee). $[\alpha]_D^{20}$: + 117.0 (*c* 0.675, MeOH); R_f = 0.33 (30% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.27 (m, 5H), 7.10-7.06 (m, 2H), 6.63 (t, ^3J = 7.2 Hz, 1H), 6.56 (d, ^3J = 8.0 Hz, 1H), 4.97 (br. s, 1H), 4.75 (t, ^3J = 7.1 Hz, 1H), 3.70 (s, 3H), 3.53-3.47 (m, 1H), 3.40-3.23 (m, 2H), 3.04-2.89 (m, 2H), 2.26 (m_c, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.0, 150.2, 138.5, 128.5, 127.3, 126.6, 126.3, 126.2, 123.6, 115.9, 105.4, 55.4, 51.0, 45.6, 37.9, 30.3, 27.0; FTIR (neat) 3030, 3026, 2946, 1698, 1604, 1519, 1452, 1188, 1023, 918, 741 cm^{-1}; HRMS Calcd for $C_{19}H_{23}N_2O_2$ $(M+H)^+$: 311.1754. Found 311.1747.

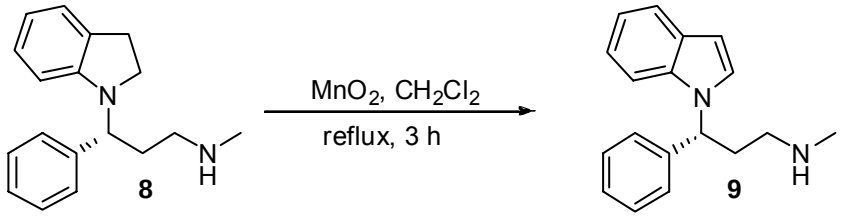
SFC (Chiralcel OD-H, 20% MeOH, 4 mL/min, 100 bar, 30 °C) t_r 5.9 min (minor enantiomer), t_r 7.1 min (major enantiomer).



(3*R*)-3-(2,3-dihydro-1*H*-indol-1-yl)-*N*-methyl-3-phenylpropan-1-amine (8). In a flame-dried 25 mL round bottom flask, **7** (57.5 mg, 0.18 mmol, 1 equiv) was dissolved in anhydrous tetrahydrofuran (3 mL) under an atmosphere of argon and cooled to 0 °C. $LiAlH_4$ (28.1 mg, 0.74 mmol, 4 equiv) was added quickly in one portion. When the exotherm ceased, the reaction was warmed up to room temperature, and then stirred at reflux for 35 min, after which all of the starting material was consumed. The crude reaction mixture was quenched with several drops of H_2O at 0 °C, and several drops of 2 M NaOH were added so that a thick white precipitate formed. This suspension was filtered through a pad of Celite washing with diethyl ether, yielding a clear yellowish solution, which was evaporated under reduced pressure to afford pure **8** (49.2 mg, 0.18 mmol, quant., 90% ee) which was used without further purification. If desired, the product can be further purified by flash chromatography, eluting with 20% MeOH in dichloromethane or by filtration through a silica plug eluting with MeOH. $[\alpha]_D^{20}$: + 149.7 (*c* 0.625, MeOH); R_f = 0.10 (methanol); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.22 (m, 5H), 7.07-7.02 (m, 2H), 6.61-6.53 (m, 2H),

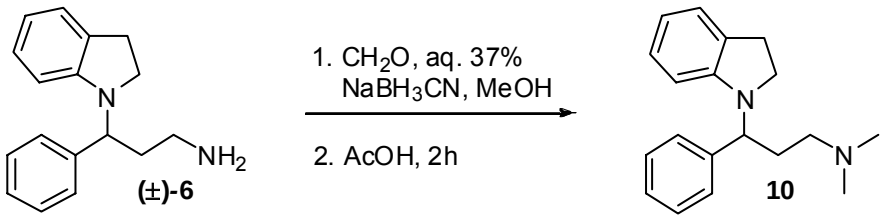
5.77 (t, ^{3}J = 7.3 Hz, 1H), 3.49 (m_c, 1H), 3.25 (m_c, 1H), 3.02-2.84 (m, 2H), 2.70 (m_c, 2H), 2.44 (s, 3H), 2.31-2.11 (m, 2H), 1.81 (br. s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.4, 140.1, 129.5, 128.3, 127.7, 127.2, 127.1, 124.4, 116.6, 106.4, 56.9, 49.5, 46.8, 36.5, 31.4, 28.1; FTIR (neat) 3026, 2930, 2793, 1605, 1472, 1328, 1263, 1157, 1024, 735 cm^{-1}; HRMS Calcd for $C_{18}H_{23}N_2$ $(M+H)^+$: 267.1856. Found 267.1854. Spectroscopic data are in full agreement with the reported values.[4]

SFC (Chiralcel OD-H, 20% [MeOH + 0.2% NEt_3], 2 mL/min, 100 bar, 40 °C) t_r 6.4 min (minor enantiomer), t_r 8.0 min (major enantiomer).



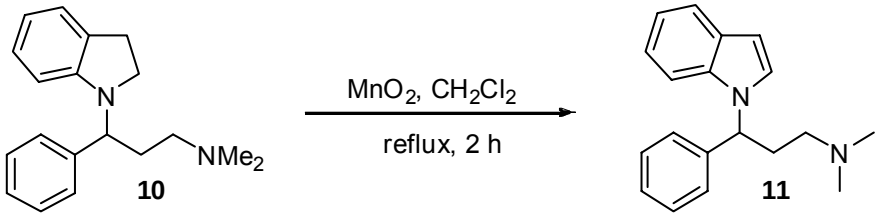
(3*R*)-3-(1*H*-indol-1-yl)-*N*-methyl-3-phenylpropan-1-amine (9). To a solution of **8** (49 mg, 0.18 mmol, 1 equiv) in dichloromethane (3 mL) was added MnO_2 (161 mg, 1.8 mmol, 10 equiv) and the reaction mixture was stirred at reflux for 3 h, after which all of the starting material was consumed. The crude reaction mixture was filtered through a pad of Celite, washing with dichloromethane, evaporated under reduced pressure and purified by flash chromatography eluting with 20% MeOH in dichloromethane, affording spectroscopically pure **9** as a yellowish oil (40.3 mg, 0.15 mmol, 83%, 90% ee). $[\alpha]_D^{20}$: + 81.2 (*c* 0.50, MeOH); Lit.[4]: $[\alpha]^{25}_D$ = + 79.2 (*c* 1.0, MeOH); R_f = 0.13 (20% MeOH in dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, ^{3}J = 7.6 Hz, 1H), 7.37 (d, ^{3}J = 3.2 Hz, 1H), 7.40-7.24 (m, 6H), 7.20 (ddd, ^{3}J = 7.1 Hz, ^{3}J = 7.0 Hz, ^{4}J = 1.1 Hz, 1H), 7.14 (ddd, ^{3}J = 7.9 Hz, ^{3}J = 7.0 Hz, ^{4}J = 1.1 Hz, 1H), 6.63 (d, ^{3}J = 3.2 Hz, 1H), 5.71 (dd, ^{3}J = 6.4 Hz, ^{3}J = 8.6 Hz, 1H), 2.63-2.45 (m, 4H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.3, 136.3, 128.6, 128.5, 127.5, 126.2, 124.9, 121.4, 120.8, 119.5, 109.9, 101.8, 57.2, 48.7, 36.4, 35.3; FTIR (neat) 3028, 2932, 1609, 1509, 1474, 1308, 1212, 1013, 738 cm^{-1}; HRMS Calcd for $C_{18}H_{21}N_2$ $(M+H)^+$: 265.1699. Found 265.1704. Spectroscopic data are in full agreement with the reported values.[4]

SFC (Chiralcel OD-H, 20% [MeOH + 0.2% NEt_3], 2 mL/min, 100 bar, 35 °C) t_r 7.1 min (minor enantiomer), t_r 12.9 min (major enantiomer).



***N*-[3-(2,3-dihydro-1*H*-indol-1-yl)-3-phenylpropyl]-*N*,*N*-dimethylamine (10).** To a solution of **(±)-6** (295 mg, 1.17 mmol, 1 equiv) in MeOH (6 mL) was added 37% aq. formaldehyde (483 mg, 5.95 mmol, 5 equiv) at room temperature, upon which a white suspension formed which dissolved under 1 min. $NaCNBH_3$ (120 mg, 1.90 mmol, 1.6 equiv) was added, and the reaction mixture was stirred for 2 h at room temperature. Several drops of glacial acetic acid were added (with gas

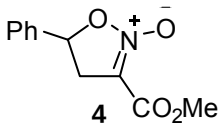
evolution) and the reaction mixture was stirred for additional 2 hours. The reaction mixture was basified to pH 9 with 2 M NaOH and extracted with dichloromethane three times. The combined organic layers were washed with sat. aq. NaCl, dried over Na_2SO_4 and evaporated under reduced pressure. Flash chromatography, eluting with 20% MeOH in dichloromethane, afforded spectroscopically pure **10** as a colourless oil (234.9 mg, 0.84 mmol, 72%). R_f = 0.43 (20% MeOH in dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.22 (m, 5H), 7.06-7.02 (m, 2H), 6.61-6.53 (m, 2H), 4.74 (m_c, 1H), 3.50 (m_c, 1H), 3.44 (m_c, 1H), 3.03-2.85 (m, 2H), 2.56-2.37 (m, 2H), 2.32 (s, 6H), 2.30-2.14 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.3, 139.9, 129.5, 128.3, 127.4, 127.22, 127.20, 124.5, 116.6, 106.4, 56.9, 56.8, 46.7, 45.3, 29.1, 28.1; FTIR (neat) 3026, 2943, 2854, 2764, 1605, 1487, 1388, 1304, 1024, 738, 629 cm^{-1}; HRMS Calcd for $C_{19}H_{25}N_2$ $(M+H)^+$: 281.2012. Found 281.2000.



3-(1*H*-indol-1-yl)-*N*,*N*-dimethyl-3-phenylpropan-1-amine (11). To a solution of **10** (234.9 mg, 0.84 mmol, 1 equiv) in dichloromethane (18 mL) was added MnO_2 (727 mg, 8.4 mmol, 10 equiv) and the reaction mixture was stirred at reflux for 2 hours. The reaction mixture was filtered through a pad of Celite washing with dichloromethane and evaporated under reduced pressure to afford spectroscopically pure **11** as a colourless oil (233.8 mg, 0.84 mmol, quant.). R_f = 0.33 (20% MeOH in dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, ^3J = 7.8 Hz, 1H), 7.43-7.13 (m, 9H), 6.65 (d, ^3J = 3.2 Hz, 1H), 5.72 (dd, ^3J = 6.3 Hz, ^3J = 8.7 Hz, 1H), 2.56-2.37 (m, 2H), 2.33-2.27 (m, 2H), 2.27 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.4, 136.3, 128.6, 128.5, 127.4, 126.3, 124.9, 121.4, 120.8, 119.4, 110.0, 101.8, 57.2, 56.2, 45.5, 33.3; FTIR (neat) 3030, 2944, 2860, 2768, 1510, 1407, 1213, 906, 727, 648 cm^{-1}; HRMS Calcd for $C_{19}H_{23}N_2$ $(M+H)^+$: 279.1856. Found 279.1862. Spectroscopical data are in full agreement with the literature values.[4]

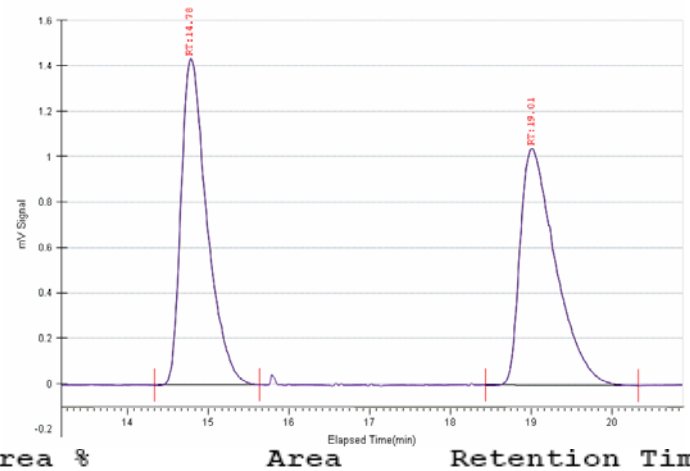
[4] Mahaney, P. E. *et al. Bioorg. Med. Chem.* **2006**, *14*, 8455.

SFC chromatograms of enantioenriched compounds



4

racemic



Area %	Area	Retention Time
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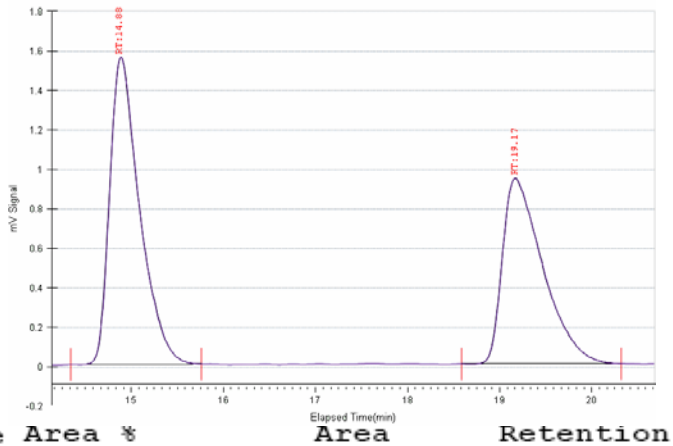
49.9446	31.5277	19.01 min

enantioenriched (Table SI-1, entry 5)



Area %	Area	Retention Time
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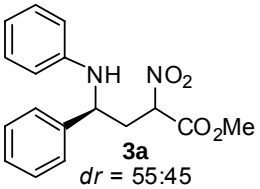
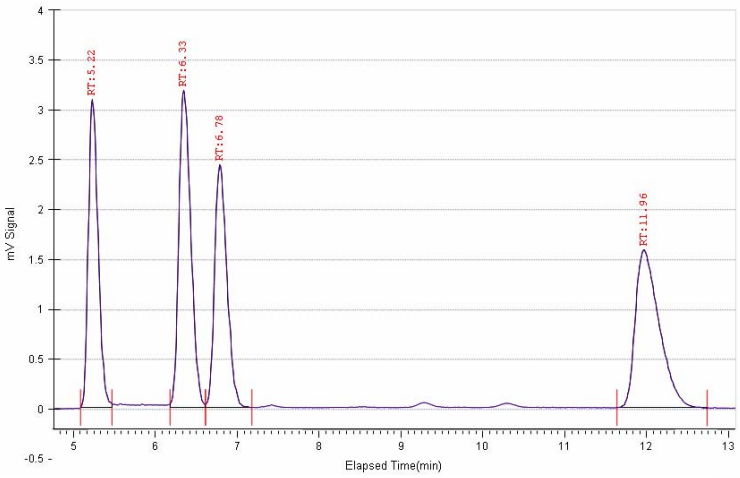


3a

dr = 55:45

racemic



Area %	Area	Retention Time
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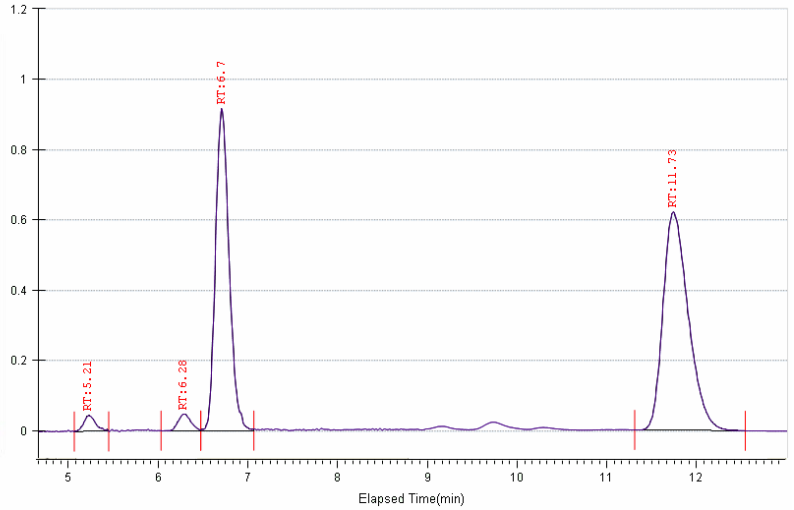
22.0758	24.7544	6.78 min
28.1145	31.5258	11.96 min

enantioenriched



Area %	Area	Retention Time
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42.4095	9.4036	6.7 min
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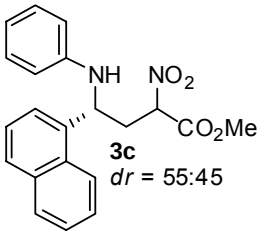
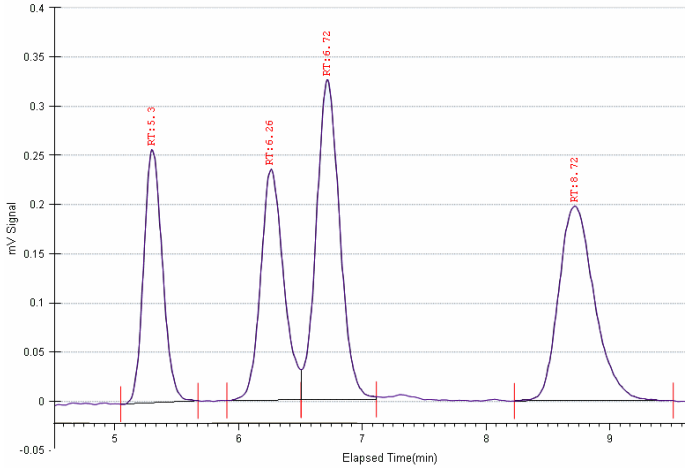


3c
dr = 55:45

racemic



Area %	Area	Retention Time
19.2457	2.6413	5.3 min
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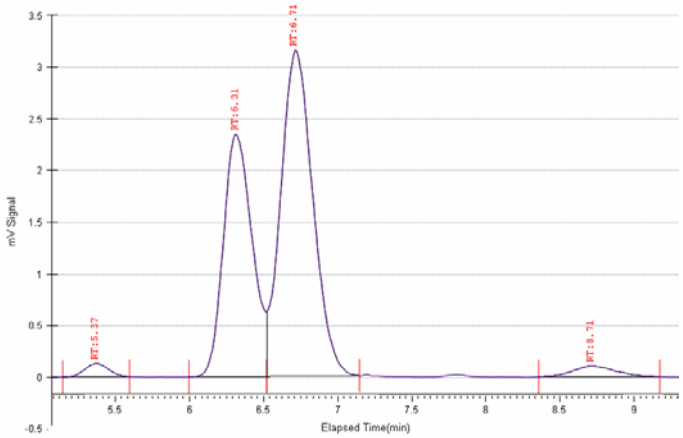
30.5376	4.1909	6.72 min
28.6838	3.9365	8.72 min

enantioenriched



Area %	Area	Retention Time
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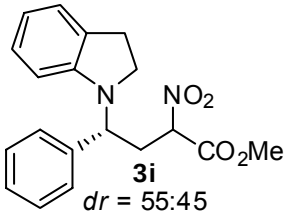
57.6323	46.1594	6.71 min
2.4818	1.9877	8.71 min



3i
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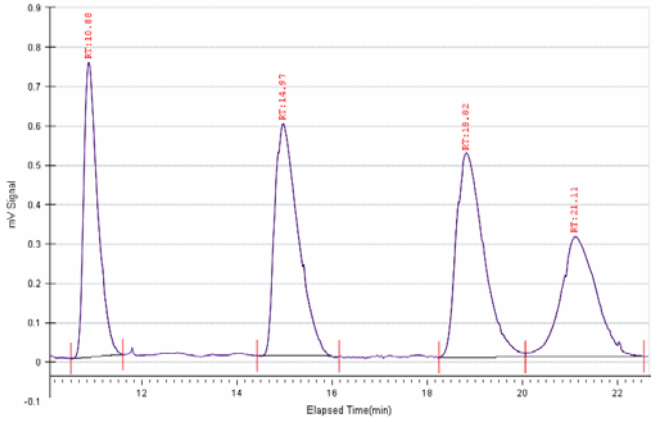
racemic



Area %	Area	Retention Time
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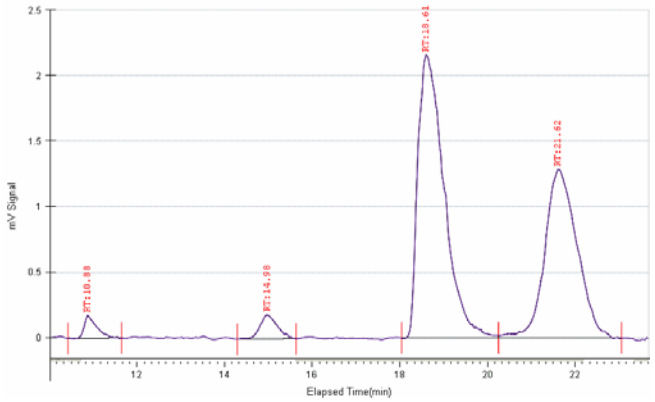
28.7382	20.5177	18.82 min
21.851	15.6006	21.11 min

enantioenriched



Area %	Area	Retention Time
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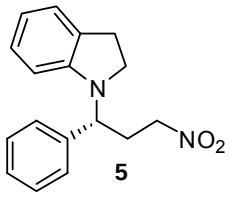
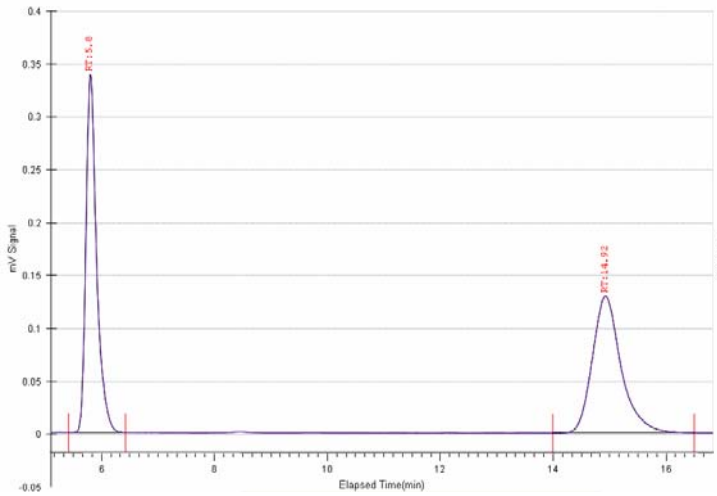
3.0666	4.9759	14.98 min
54.7373	88.8175	18.61 min
40.1359	65.1251	21.62 min

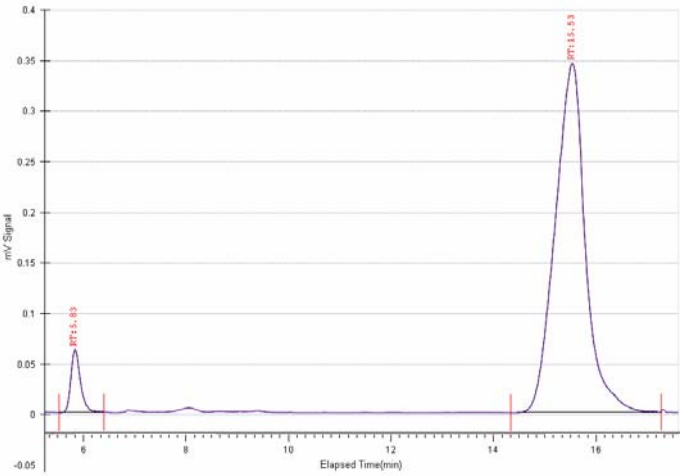


5

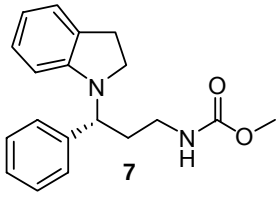
racemic



enantioenriched

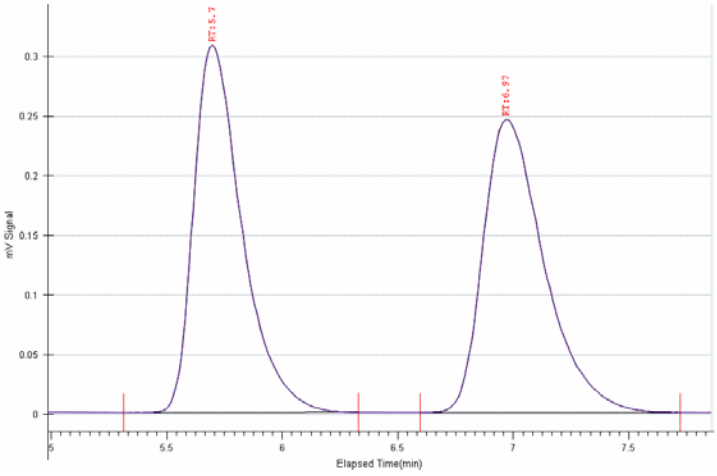


Area %	Area	Retention Time	Area %	Area	Retention Time
49.4258	4.3671	5.8 min	5.3035	0.7851	5.83 min
50.5742	4.4685	14.92 min	94.6965	14.0186	15.53 min

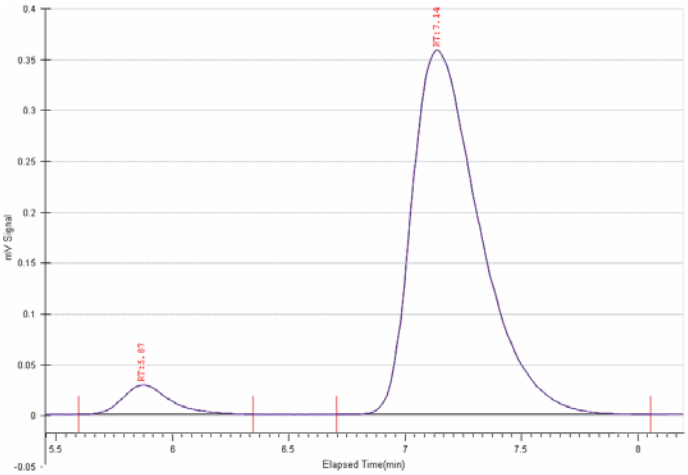


7

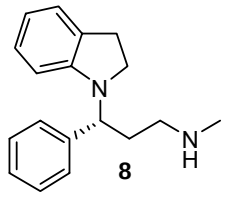
racemic



enantioenriched

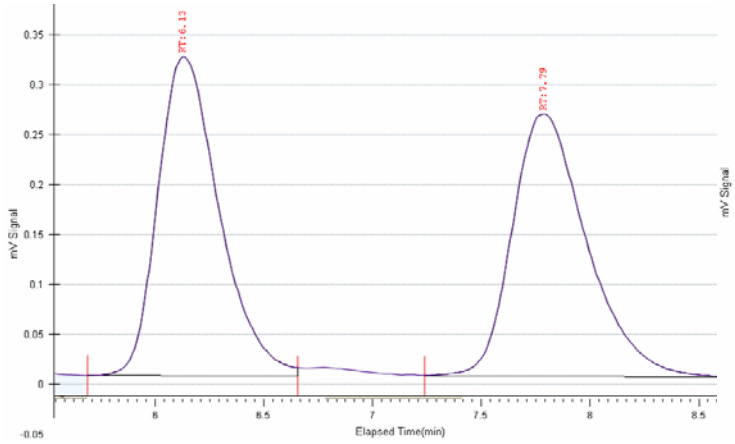


Area %	Area	Retention Time	Area %	Area	Retention Time
49.7832	4.401	5.7 min	5.1748	0.3898	5.87 min
50.2168	4.4393	6.97 min	94.8252	7.1435	7.14 min



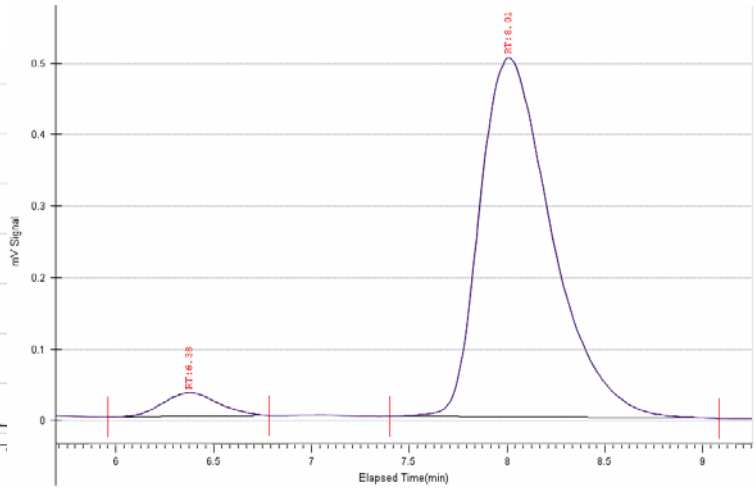
8

racemic

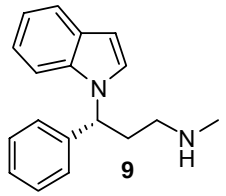


Area %	Area	Retention Time
49.8896	6.1562	6.13 min
50.1104	6.1835	7.79 min

enantioenriched

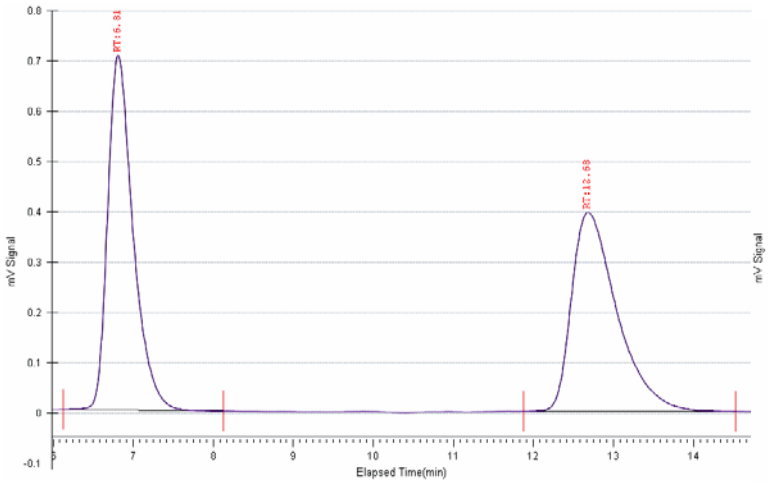


Area %	Area	Retention Time
4.7693	0.6377	6.38 min
95.2307	12.7331	8.01 min



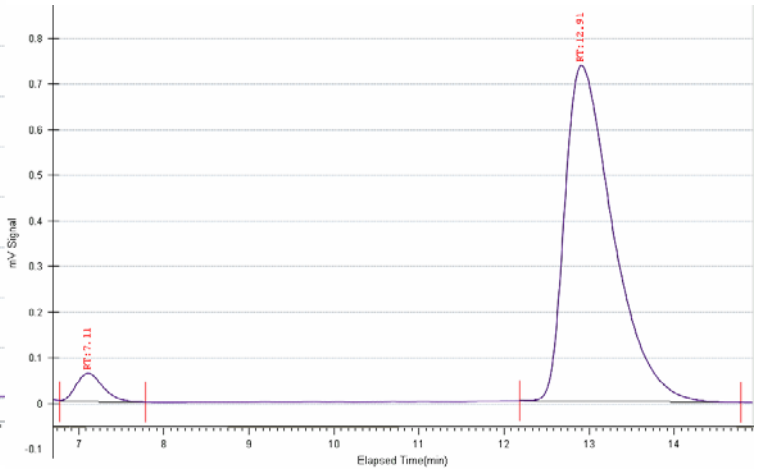
9

racemic



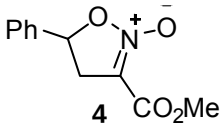
Area %	Area	Retention Time
49.9237	15.4094	6.81 min
50.0763	15.4565	12.68 min

enantioenriched

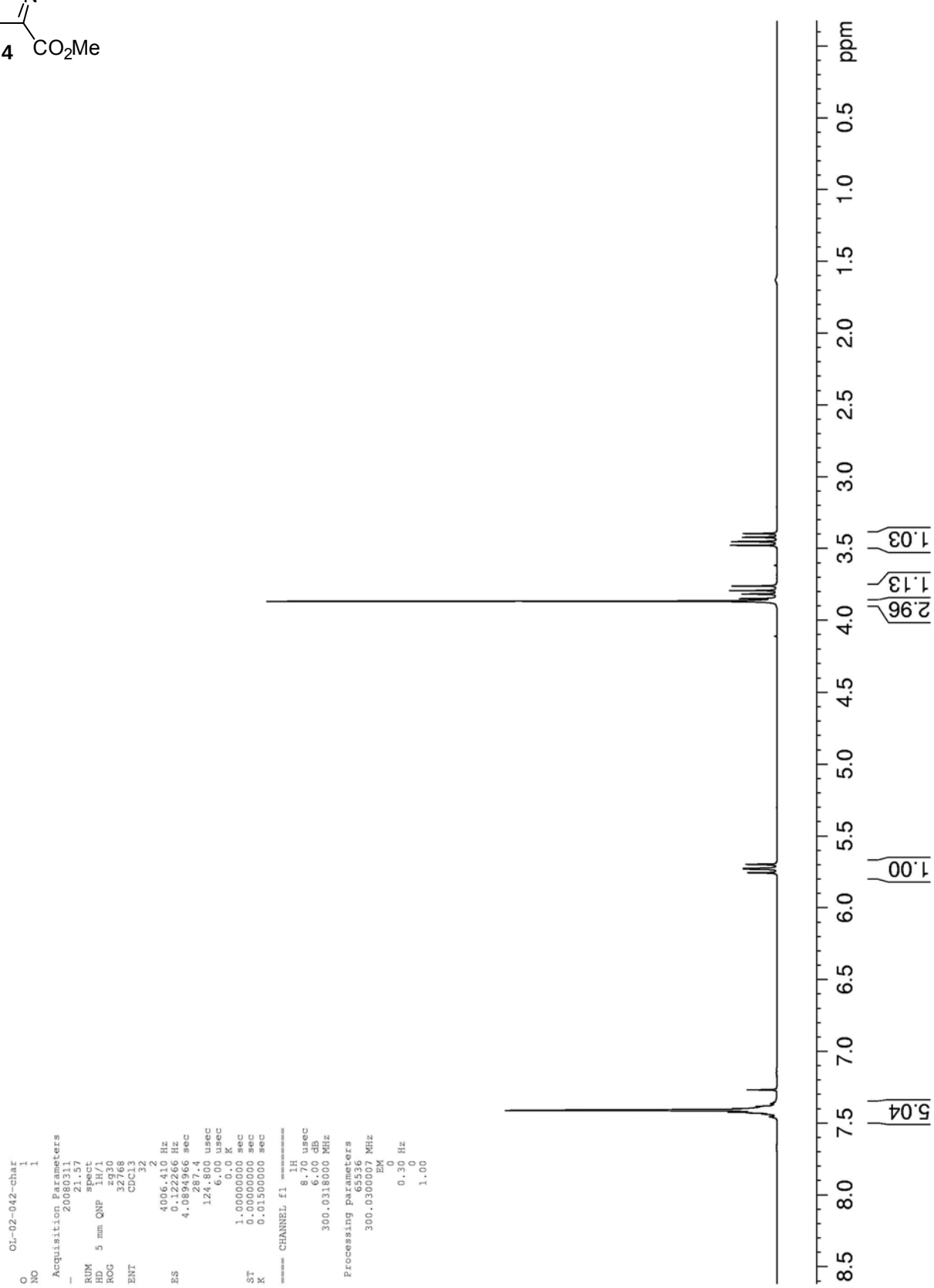


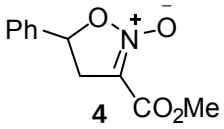
Area %	Area	Retention Time
4.4598	1.3402	7.11 min
95.5402	28.7101	12.91 min

^{1}H and ^{13}C NMR spectra of new compounds

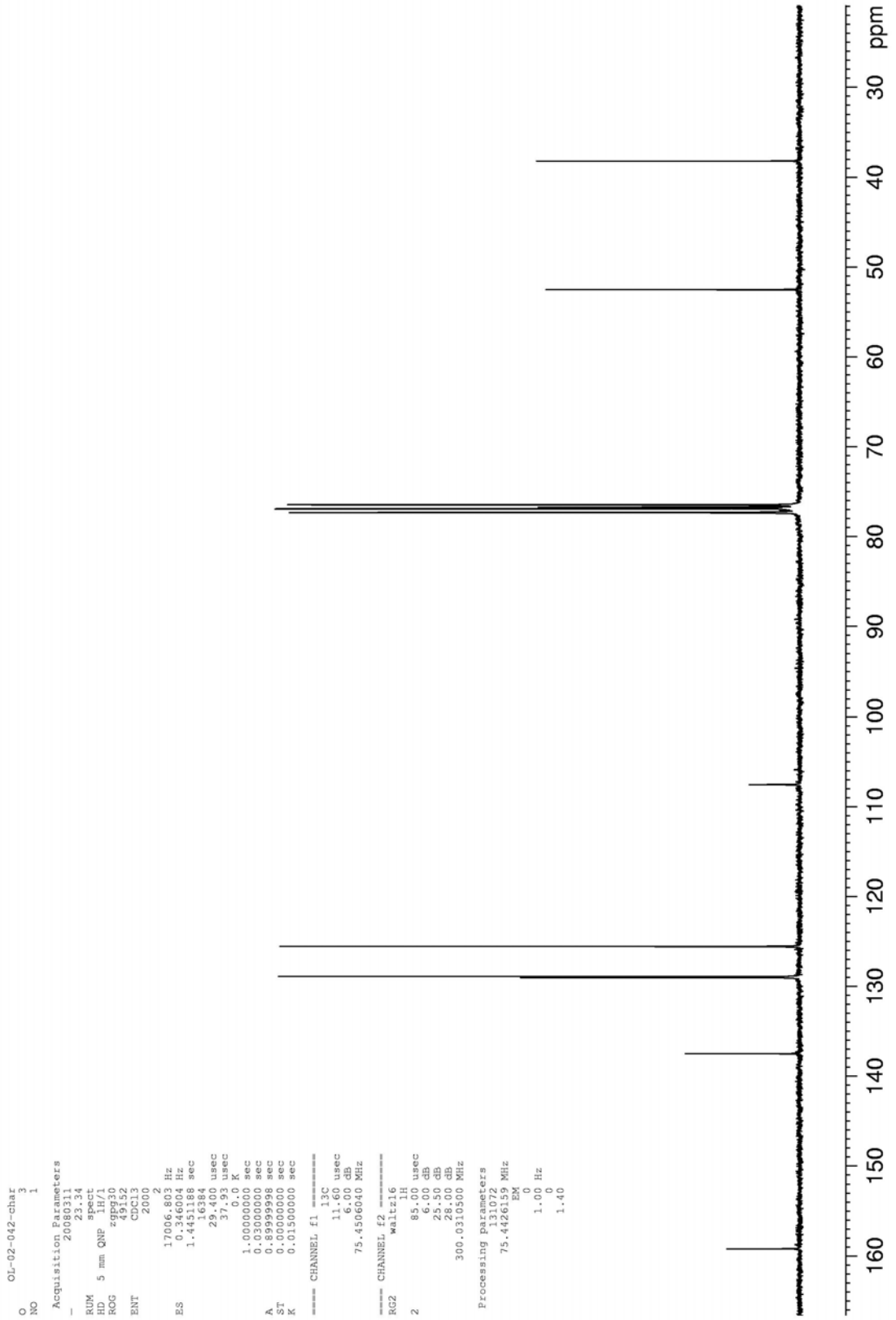


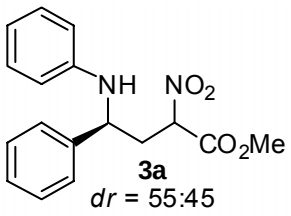
4



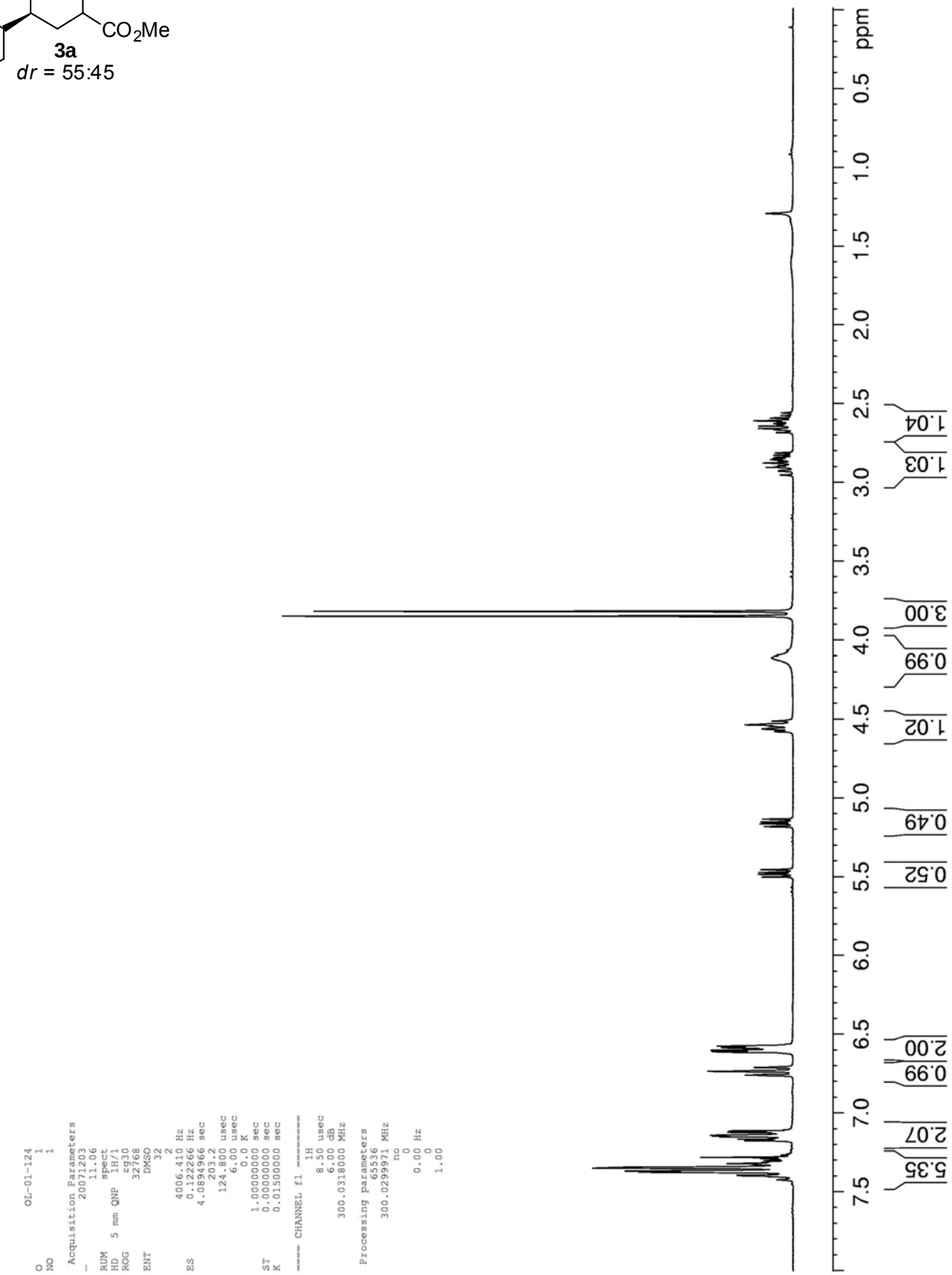

Ph
O
N
O
CO2Me

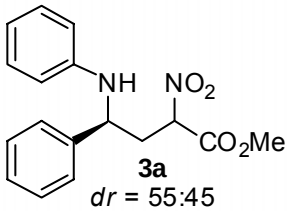
4


OL-02-042-char
O 3
NO 1
Acquisition Parameters
20080311
23.34
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
ENT CDC13
2000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec
CHANNEL f1
13C
11.60 usec
6.00 dB
75.4506040 MHz
CHANNEL f2
RG2 waltz16
1H
2 85.00 usec
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4426159 MHz
EM
0
1.00 Hz
0
1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


NH
NO2
CO2Me

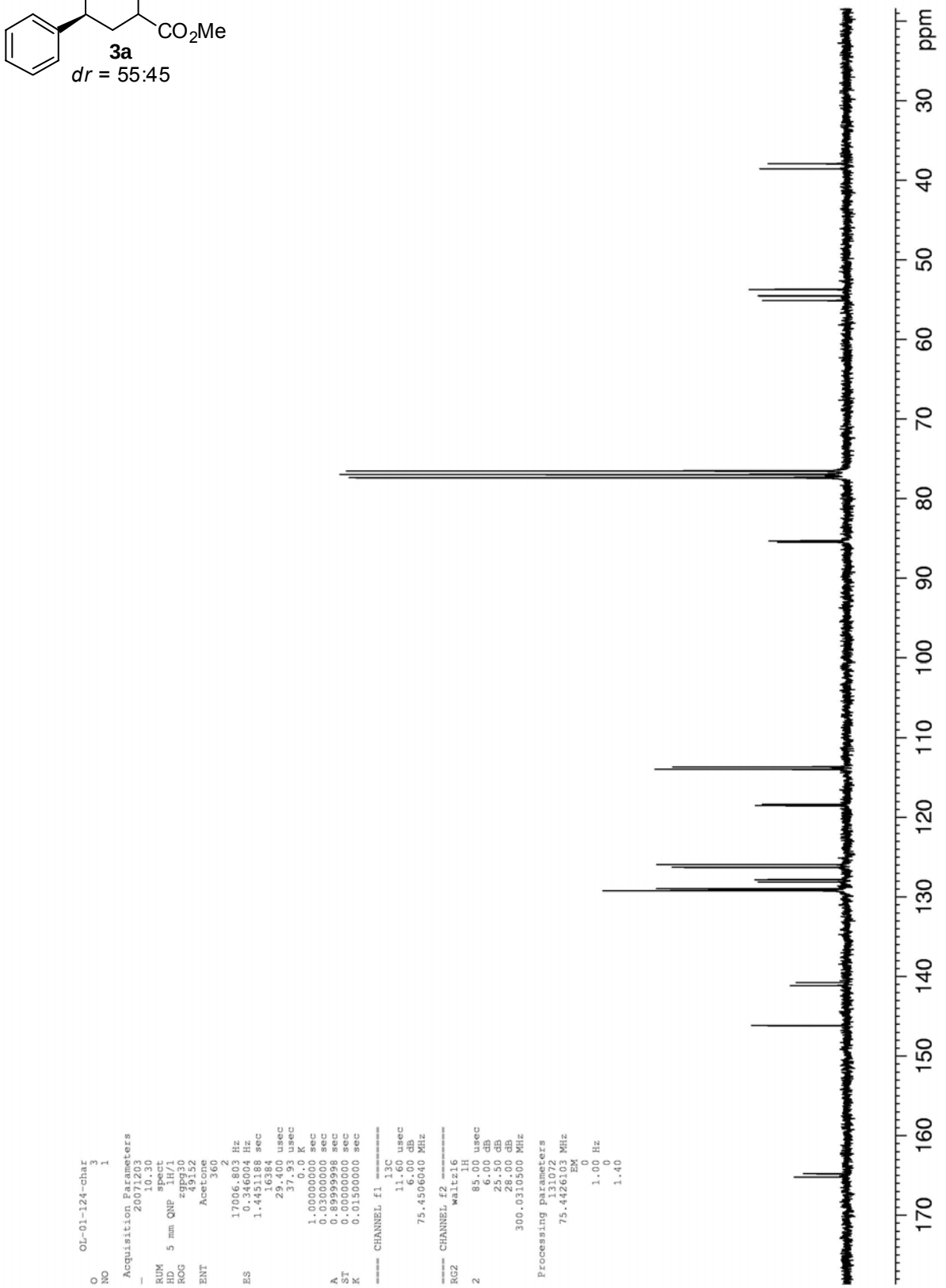
3a
dr = 55:45

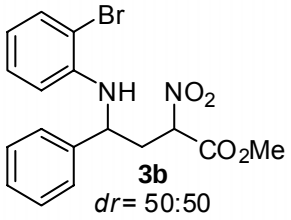

ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.35
2.07
0.99
2.00
0.52
0.49
1.02
0.99
3.00
1.03
1.04



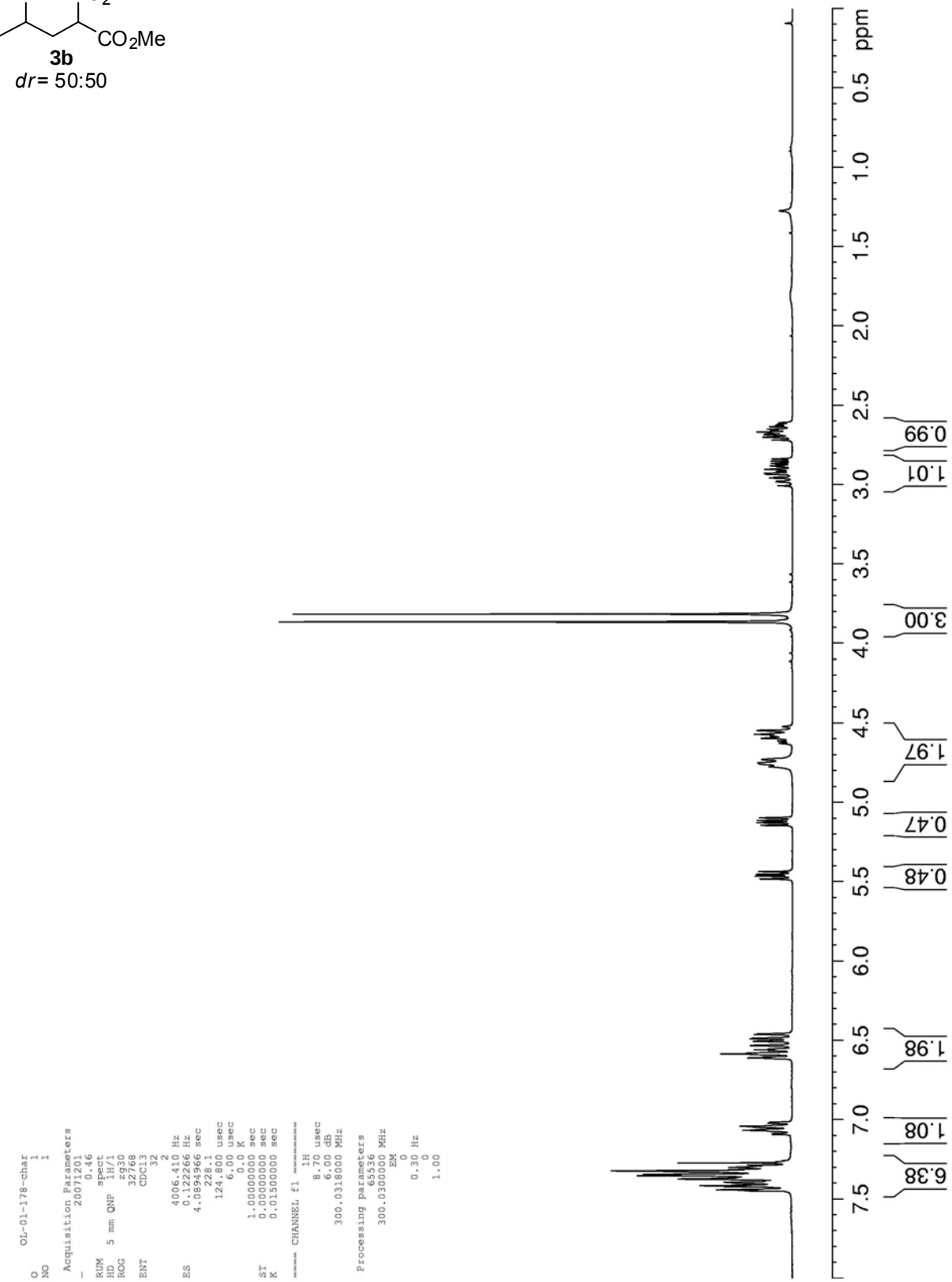
3a

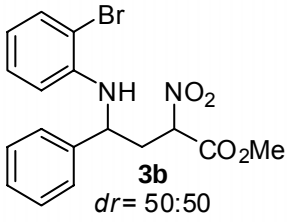
dr = 55:45




Br
NH
NO2
CO2Me

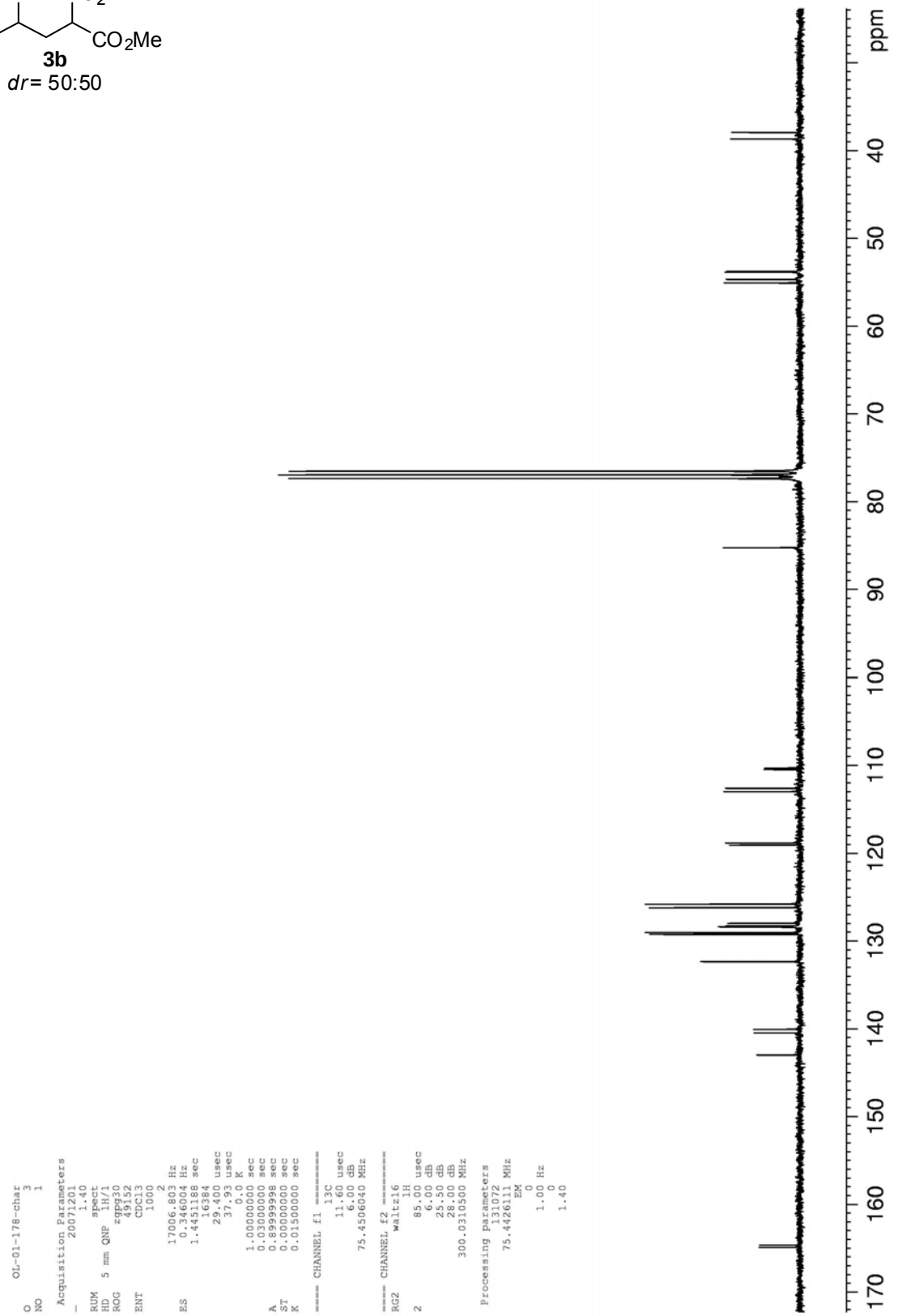
3b
dr = 50:50


ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
0.99
1.01
3.00
1.97
0.47
0.48
1.98
1.08
6.38



3b

dr = 50:50



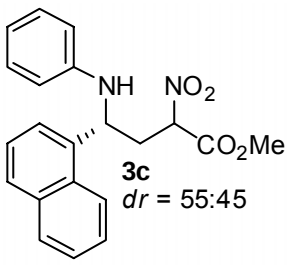
OL-01-178-char
O 3
NO 1

Acquisition Parameters
20071201
1.40
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
1000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec

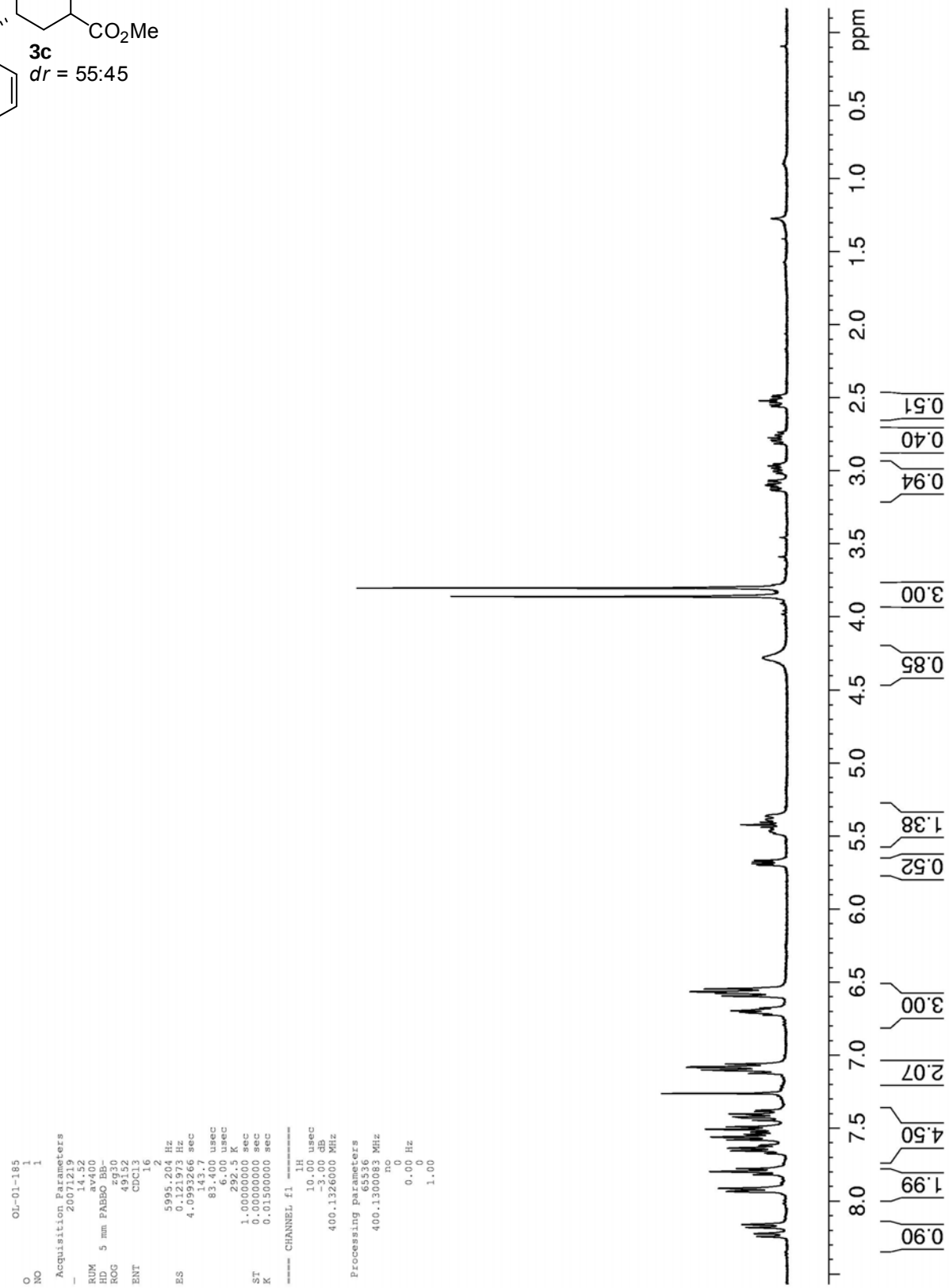
===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506040 MHz

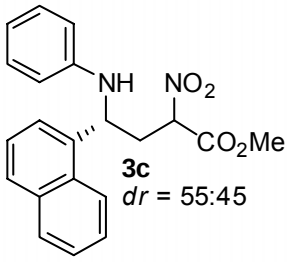
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 usec
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz

Processing parameters
131072
75.4426111 MHz
EM
0
1.00 Hz
0
1.40

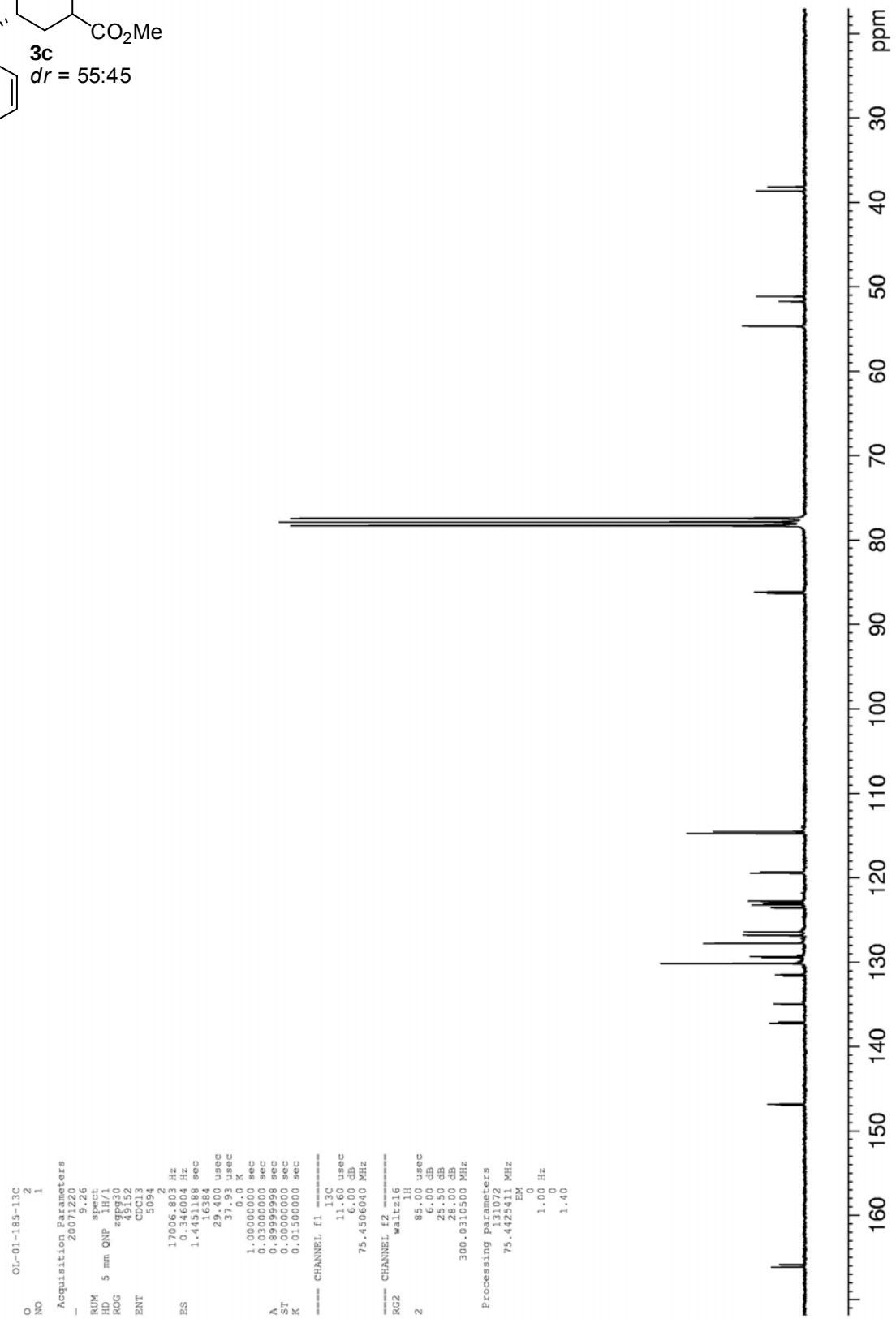


3c
dr = 55:45

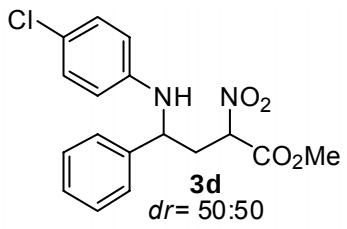




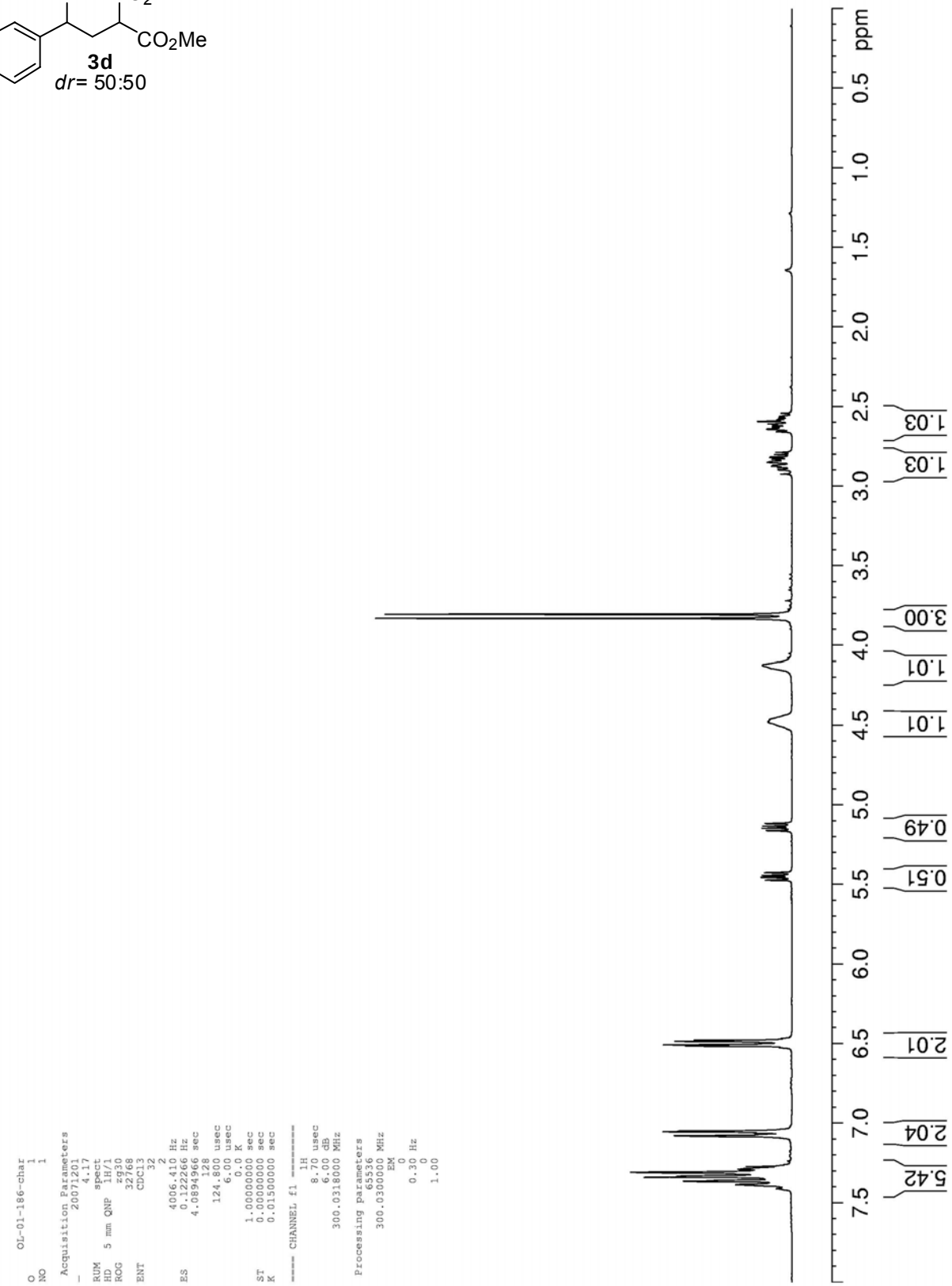
3c
dr = 55:45

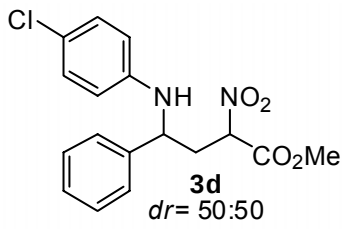



```
OL-01-185-13C
O                2
NO               1

Acquisition Parameters
_              20071220
                   9.26
                  spect
RUM
HD   5 mm QNP  1H/1
ROG            zgpg30
                 49152
ENT              CDCl3
                  5094
                     2
              17006.803 Hz
ES                0.346004 Hz
              1.44451188 sec
                   16384
                29.400 usec
                37.93 usec
                     0.0 K
             1.00000000 sec
A            0.03000000 sec
ST           0.89999998 sec
K            0.00000000 sec
             0.01500000 sec

===== CHANNEL f1 ========
                     13C
                11.60 usec
                 6.00 dB
              75.4506040 MHz

===== CHANNEL f2 ========
RG2              waltz16
                      1H
2                85.00 dB
                  6.00 dB
                 25.50 dB
                 28.00 dB
             300.0310500 MHz

Processing parameters
                131072
              75.4425411 MHz
                      EM
                       0
                  1.00 Hz
                       0
                    1.40
```

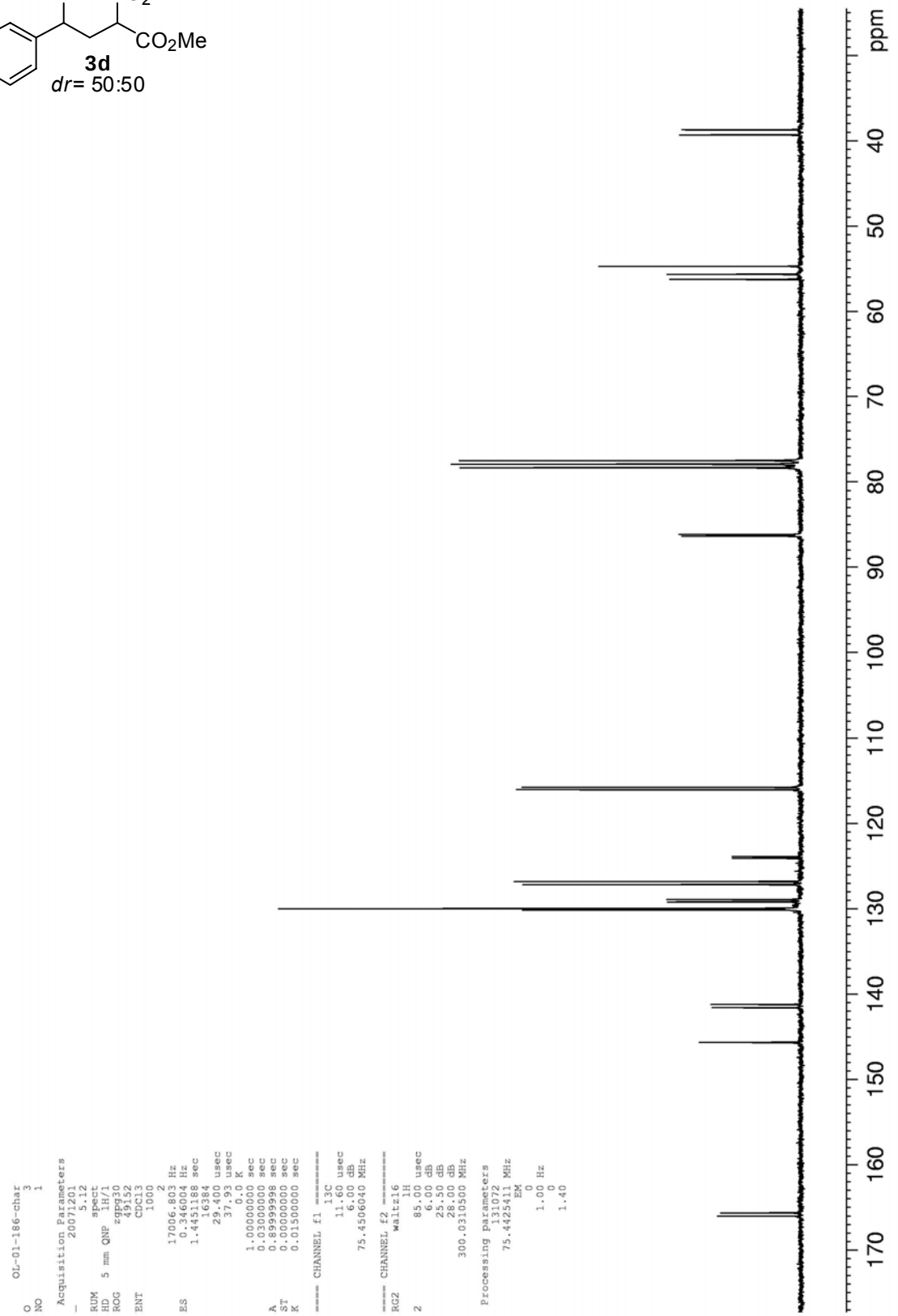

Cl
NH
NO2
CO2Me

3d
dr= 50:50

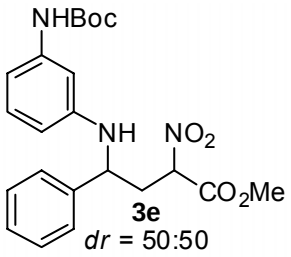

ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.42
2.04
2.01
0.51
0.49
1.01
1.01
3.00
1.03
1.03



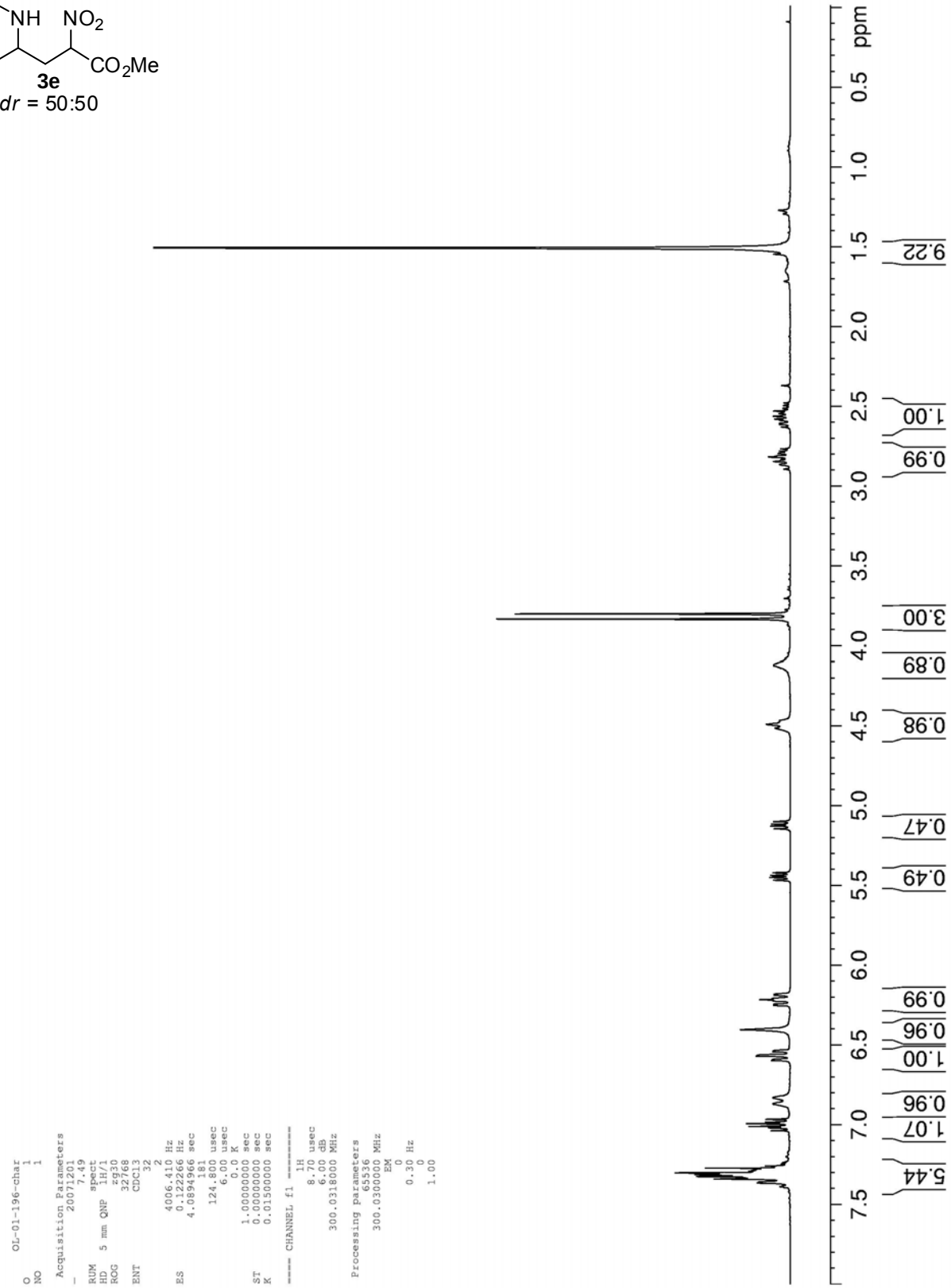
3d
dr= 50:50

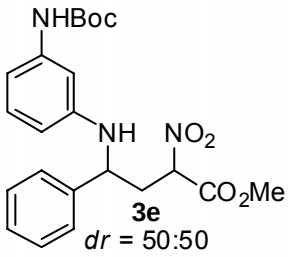


OL-01-186-char
O 3
NO 1
Acquisition Parameters
20071201
5.12
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
1000
2
ES 17006.803 Hz
0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec
===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506040 MHz
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 usec
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4425411 MHz
EM
0
1.00 Hz
0
1.40


NHBoc
NH
NO2
CO2Me

3e
dr = 50:50


ppm
9.22
1.00
0.99
3.00
0.89
0.98
0.47
0.49
1.00
0.96
0.99
1.07
0.96
5.44



3e
dr = 50:50

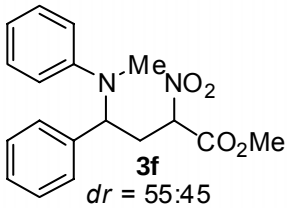
OL-01-196-char
O 3
NO 1

Acquisition Parameters
20071201
8.44
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
1000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec

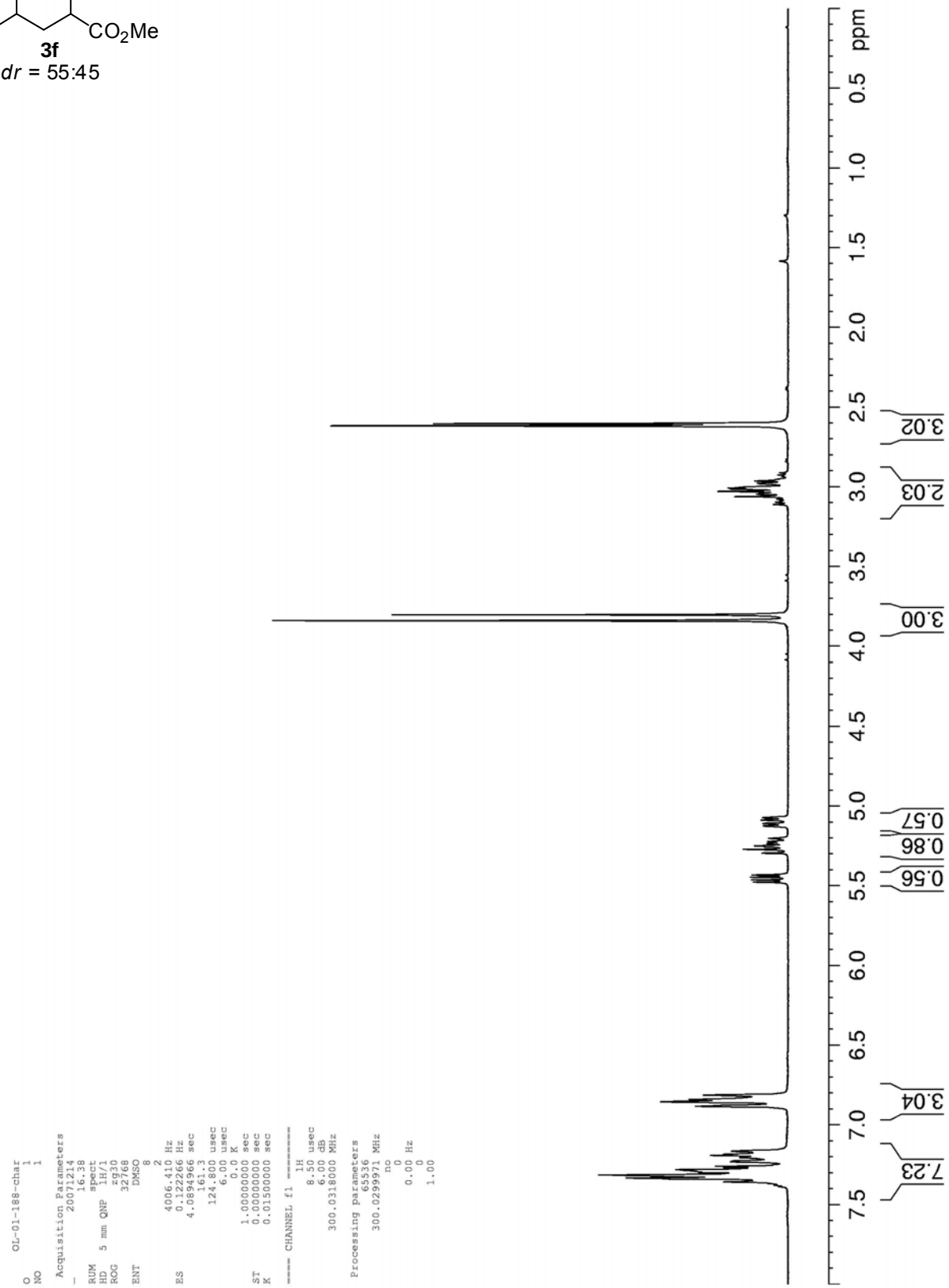
===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506640 MHz

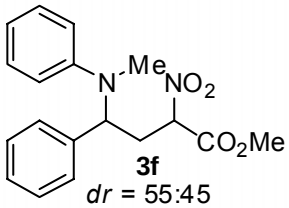
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 usec
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz

Processing parameters
131072
75.4425411 MHz
EM
0
1.00 Hz
0
1.40

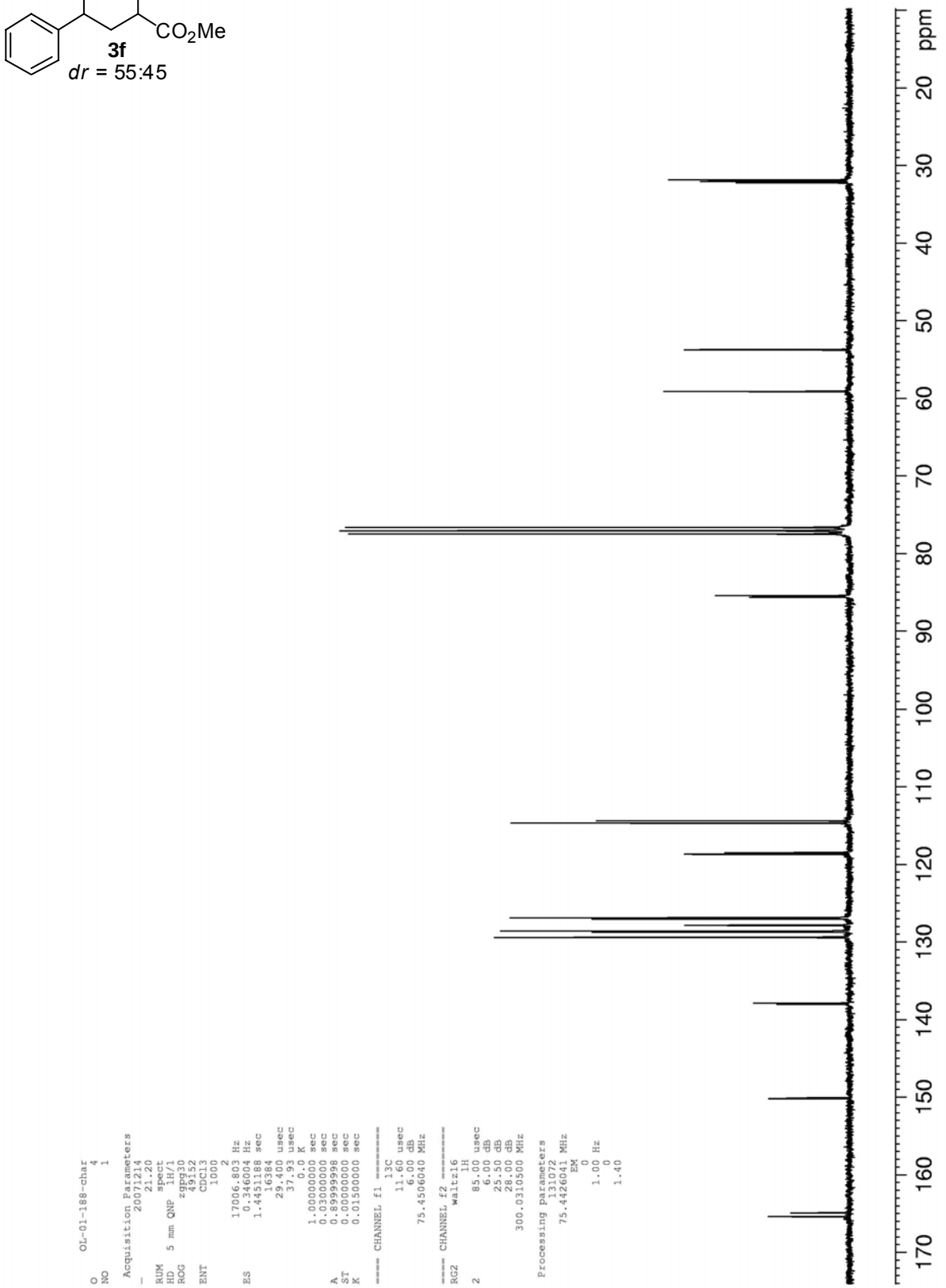


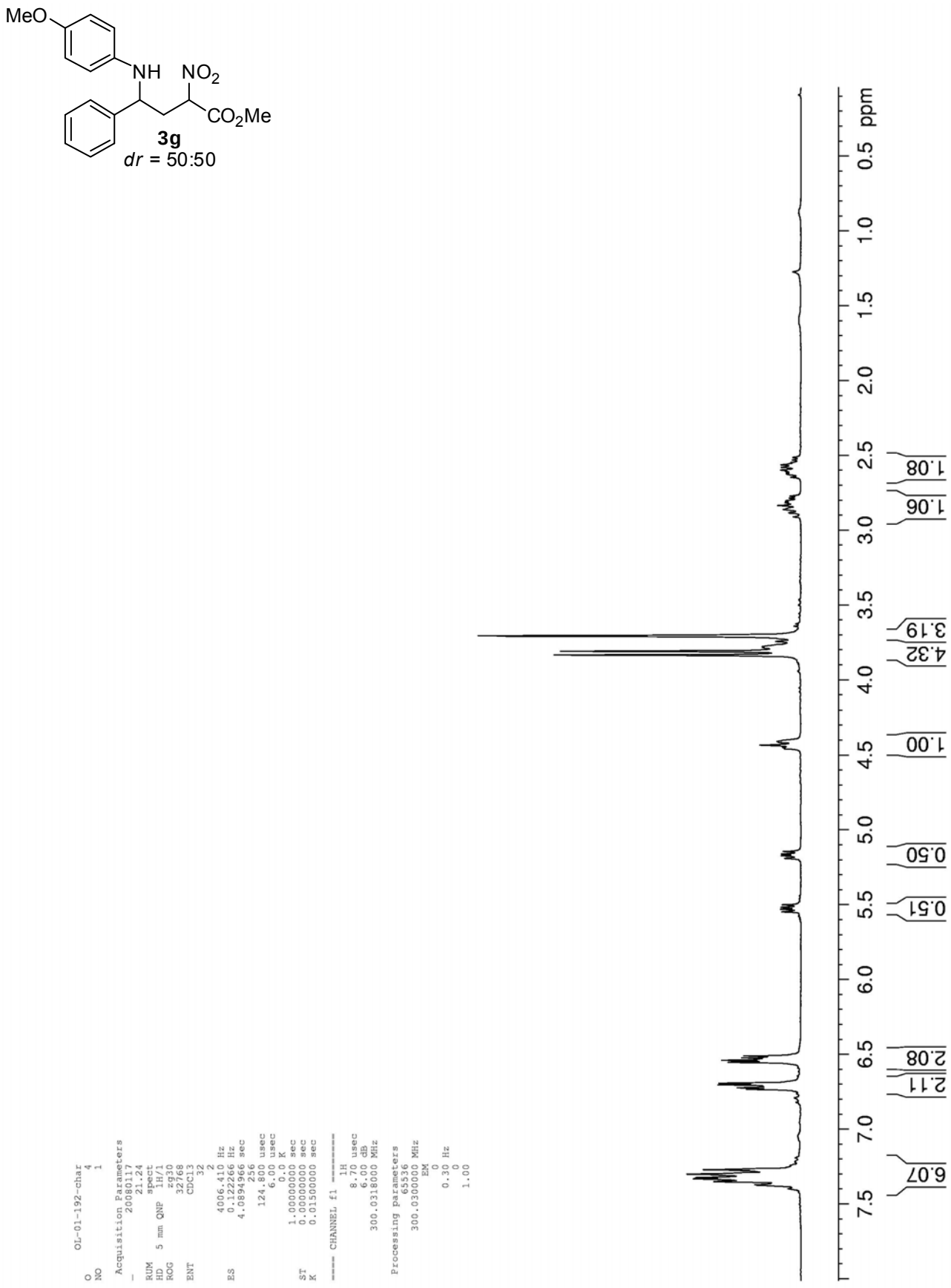
3f
dr = 55:45

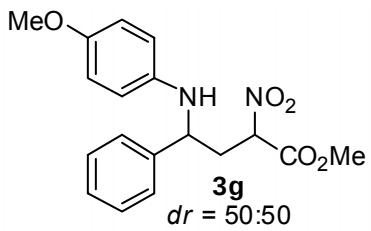



Me
N
NO2
CO2Me

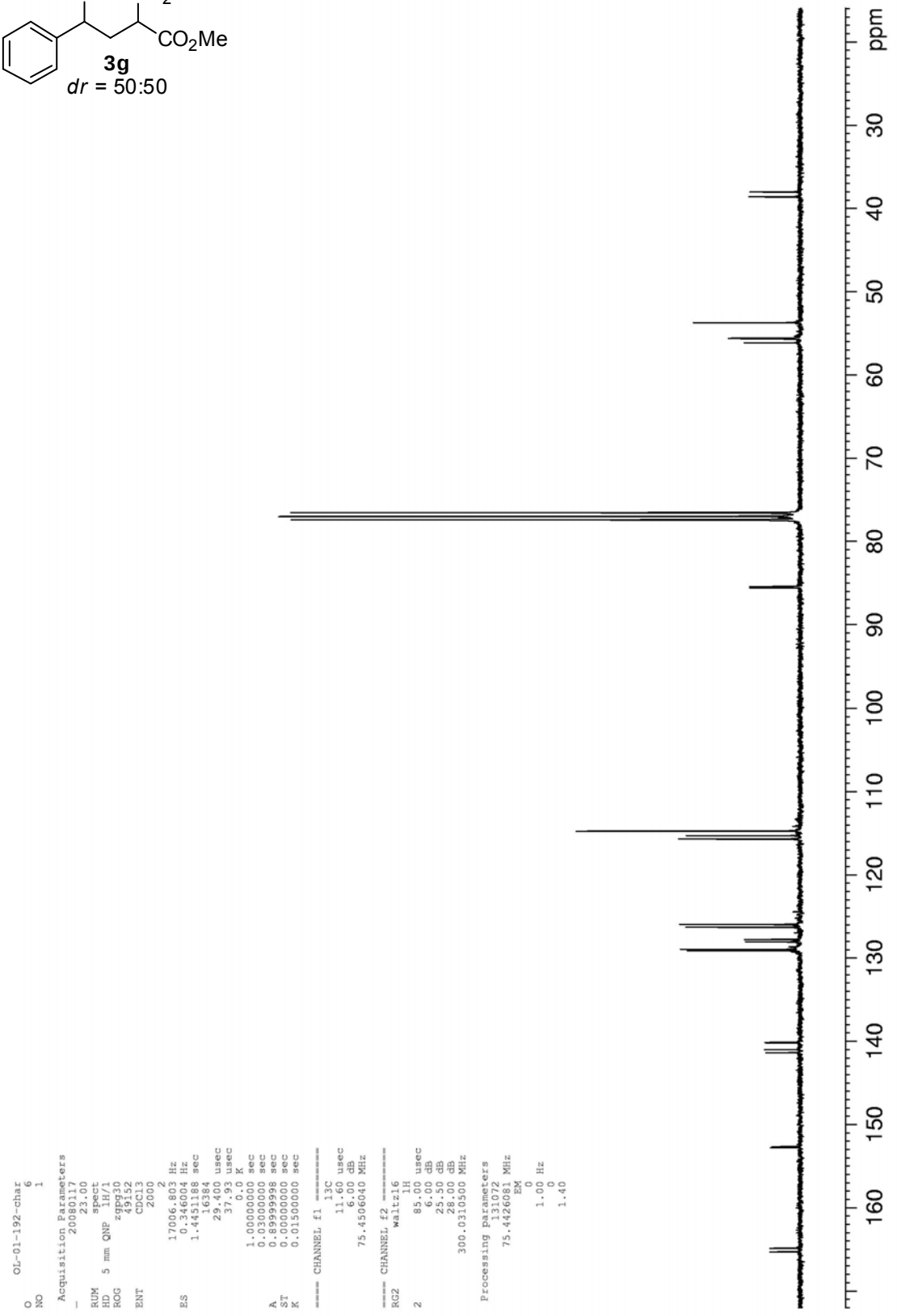
3f
dr = 55:45


OL-01-188-char
4
1
Acquisition Parameters
20071214
21.20
spect
1H/1
zgpg30
49152
CDCl3
1000
2
17006.803 Hz
0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
0.89999998 sec
0.00000000 sec
0.01500000 sec
CHANNEL f1
13C
11.60 usec
6.00 dB
75.4506040 MHz
CHANNEL f2
waltz16
1H
85.00 dB
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4426041 MHz
EM
0
1.00 Hz
0
1.40
ppm

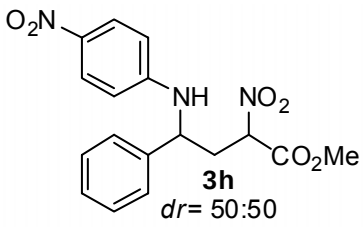

MeO
NH
NO2
CO2Me
3g
dr = 50:50
ppm
6.07
2.11
2.08
0.51
0.50
1.00
4.32
3.19
1.06
1.08



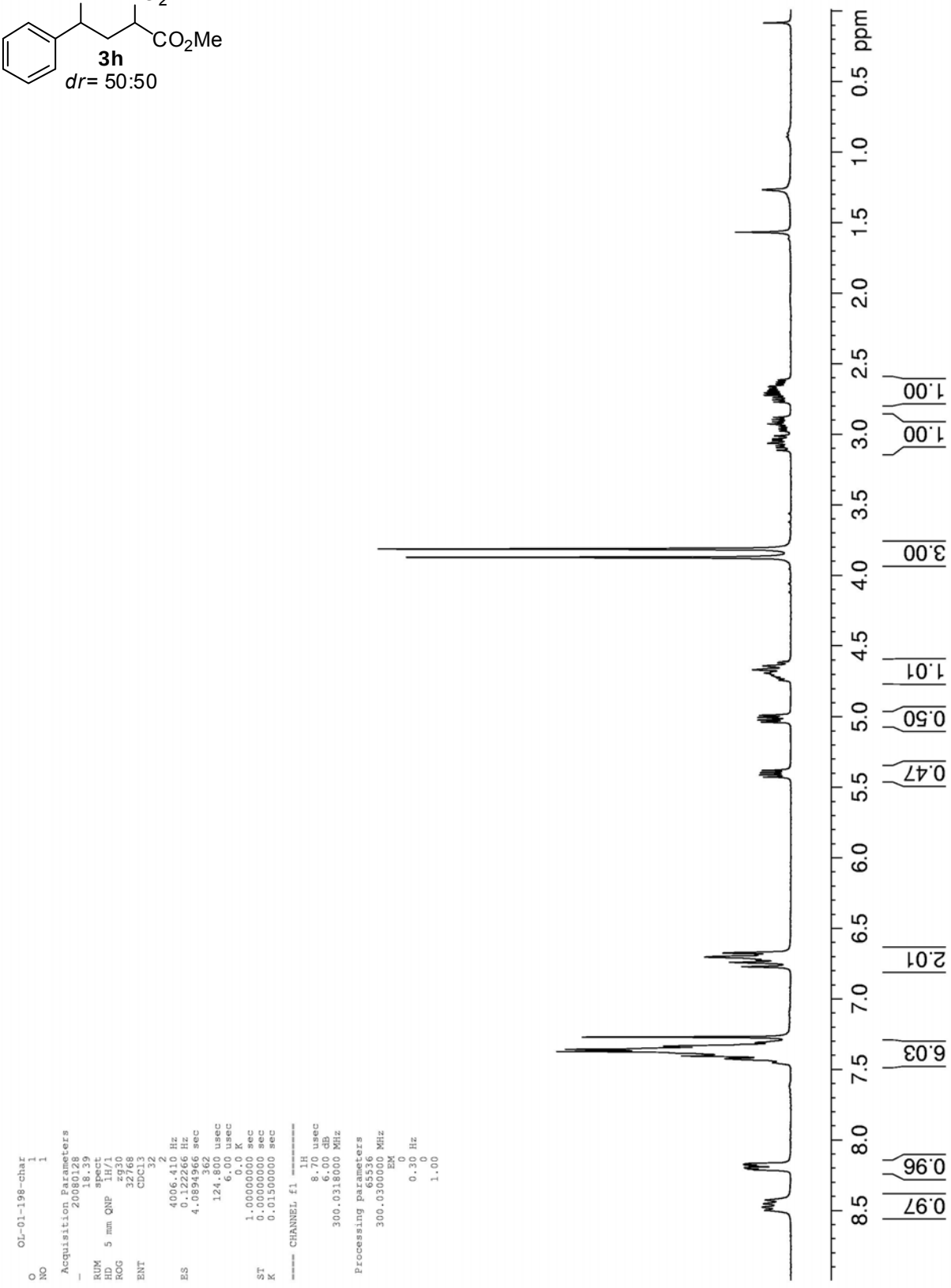
3g
dr = 50:50

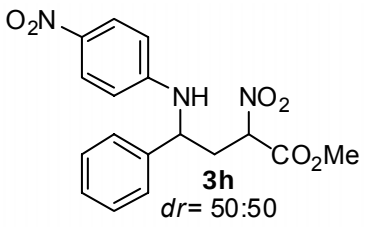



```
   OL-01-192-char
O                  6
NO                 1

  Acquisition Parameters
_              20080117
                  23.00
RUM               spect
HD   5 mm QNP   1H/1
ROG            zgpg30
                  49152
ENT               CDCl3
                   2000
                      2
                17006.803 Hz
ES              0.346004 Hz
                1.4451188 sec
                    16384
                   29.400 usec
                   37.93 usec
                      0.0 K
                1.00000000 sec
A               0.03000000 sec
ST              0.89999998 sec
K               0.00000000 sec
                0.01500000 sec

===== CHANNEL f1 ==========
                      13C
                   11.60 usec
                    6.00 dB
                75.4506040 MHz
===== CHANNEL f2 ==========
RG2             waltz16
                      1H
2                 85.00 usec
                   6.00 dB
                  25.50 dB
                  28.00 dB
               300.0310500 MHz

  Processing parameters
                 131072
              75.4426081 MHz
                      EM
                       0
                1.00 Hz
                       0
                1.40
```

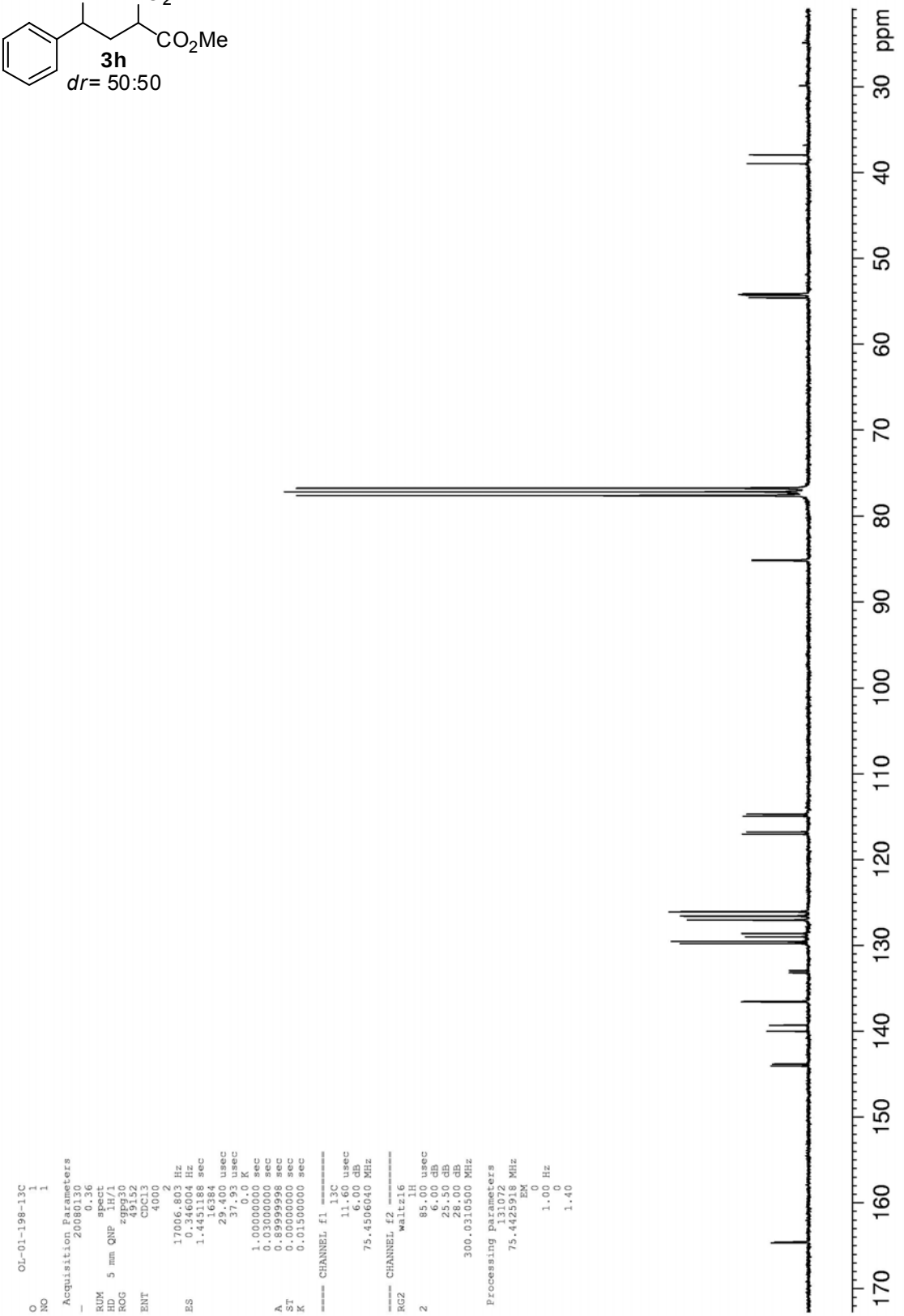

O2N
NH
NO2
CO2Me

3h
dr= 50:50

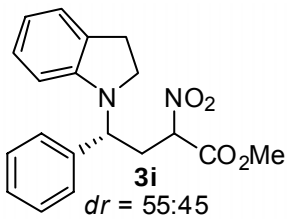

OL-01-198-char
Acquisition Parameters
CHANNEL f1
Processing parameters
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.97
0.96
6.03
2.01
0.47
0.50
1.01
3.00
1.00
1.00



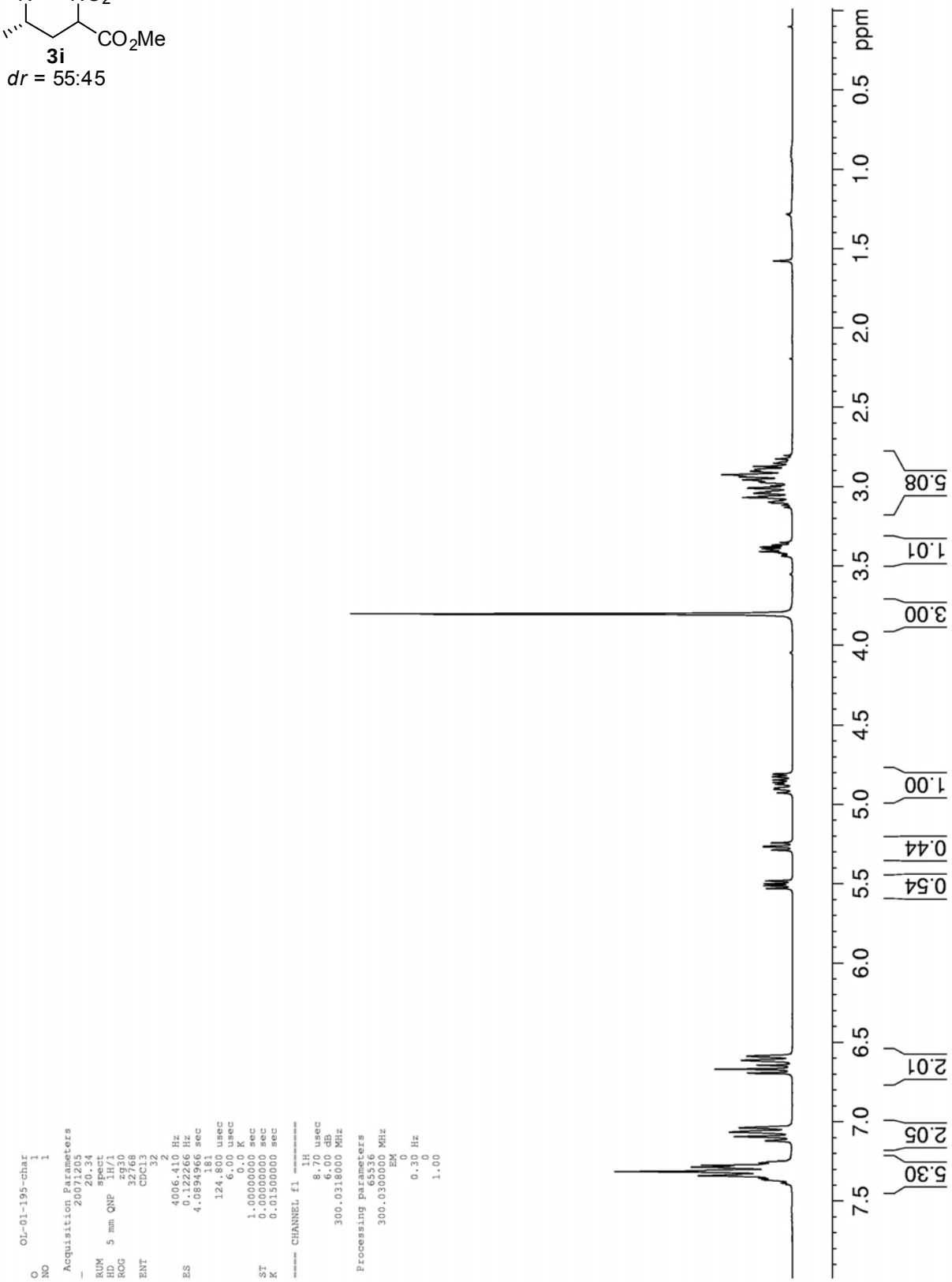
3h
dr= 50:50

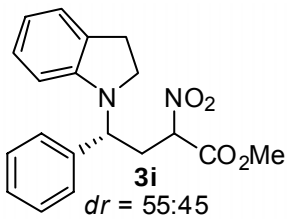



```
  OL-01-198-13C
O               1
NO              1

  Acquisition Parameters
_            20080130
                0.36
                spect
RUM
HD  5 mm QNP  1H/1
ROG             zgpg30
               49152
ENT             CDCl3
                4000
                   2
            17006.803 Hz
ES              0.346004 Hz
             1.44511188 sec
                16384
             29.400 usec
             37.93 usec
                 0.0 K
          1.00000000 sec
          0.03000000 sec
A         0.89999998 sec
ST        0.00000000 sec
K         0.01500000 sec

===== CHANNEL f1 ========
                  13C
             11.60 usec
                 6.00 dB
            75.4506040 MHz
===== CHANNEL f2 ========
RG2           waltz16
                   1H
2               85.00 dB
                 6.00 dB
                25.50 dB
                28.00 dB
           300.0310500 MHz

  Processing parameters
            131072
            75.442918 MHz
                EM
                   0
                1.00 Hz
                   0
                1.40
```

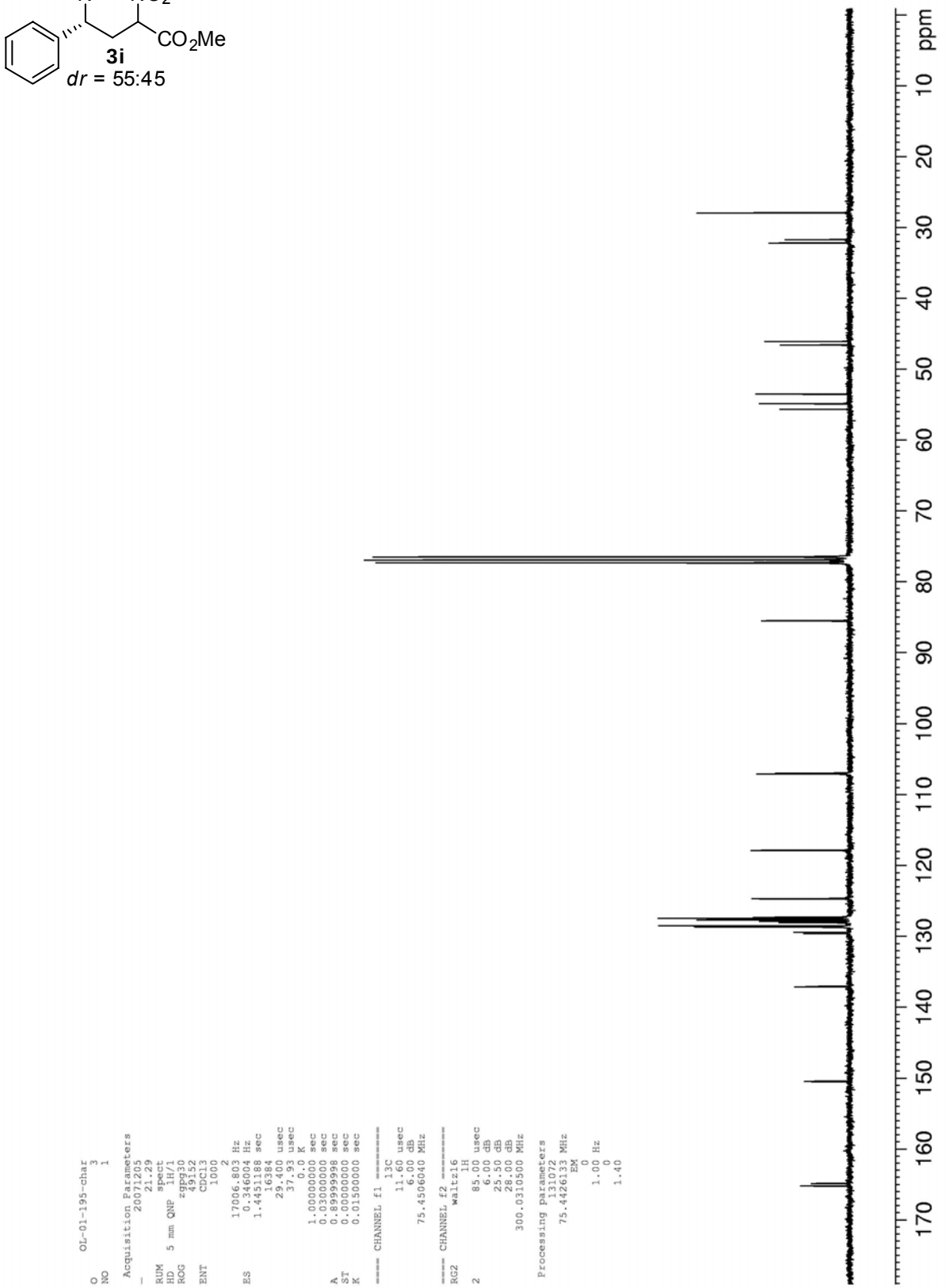

N
NO2
CO2Me

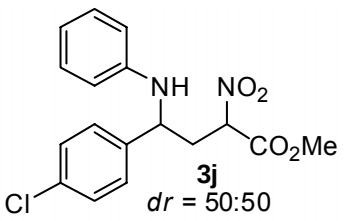
3i
dr = 55:45


ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.30
2.05
2.01
1.00
0.44
0.54
1.00
3.00
1.01
5.08


N
NO2
CO2Me

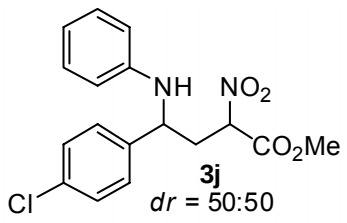
3i
dr = 55:45


OL-01-195-char
Acquisition Parameters
zgpg30
CDCl3
CHANNEL f1
CHANNEL f2
waltz16
Processing parameters
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

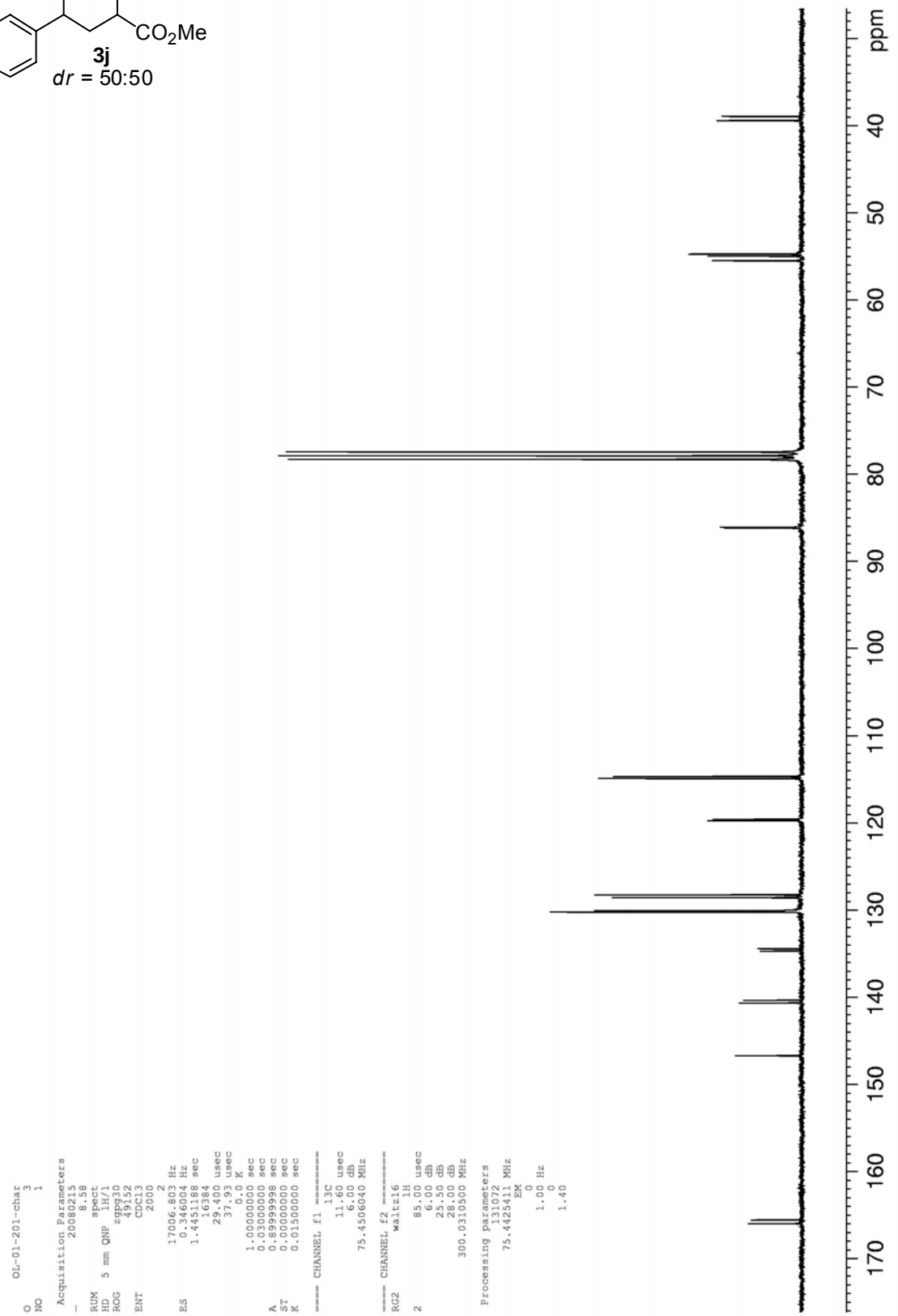

NH
NO2
CO2Me
Cl

3j
dr = 50:50

OL-01-201-char
Acquisition Parameters
20080215
7.21
spect
1H/1
zg30
32768
CDCl3
32
2
4006.410 Hz
0.122266 Hz
4.0894966 sec
228.1
124.800 usec
6.00 usec
0.0 K
1.00000000 sec
0.00000000 sec
0.01500000 sec
CHANNEL f1
1H
8.70 usec
6.00 dB
300.0318000 MHz
Processing parameters
65536
300.0300000 MHz
EM
0
0.30 Hz
0
1.00
4.29
2.06
1.04
2.03
0.51
0.48
1.00
0.95
3.00
1.01
1.02
ppm



3j
dr = 50:50



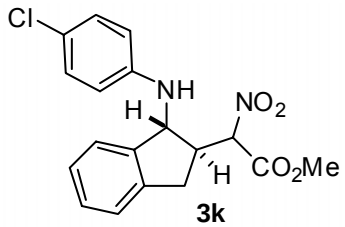
OL-01-201-char
O 3
NO 1

Acquisition Parameters
20080215
8.58
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
2000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec

===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506040 MHz

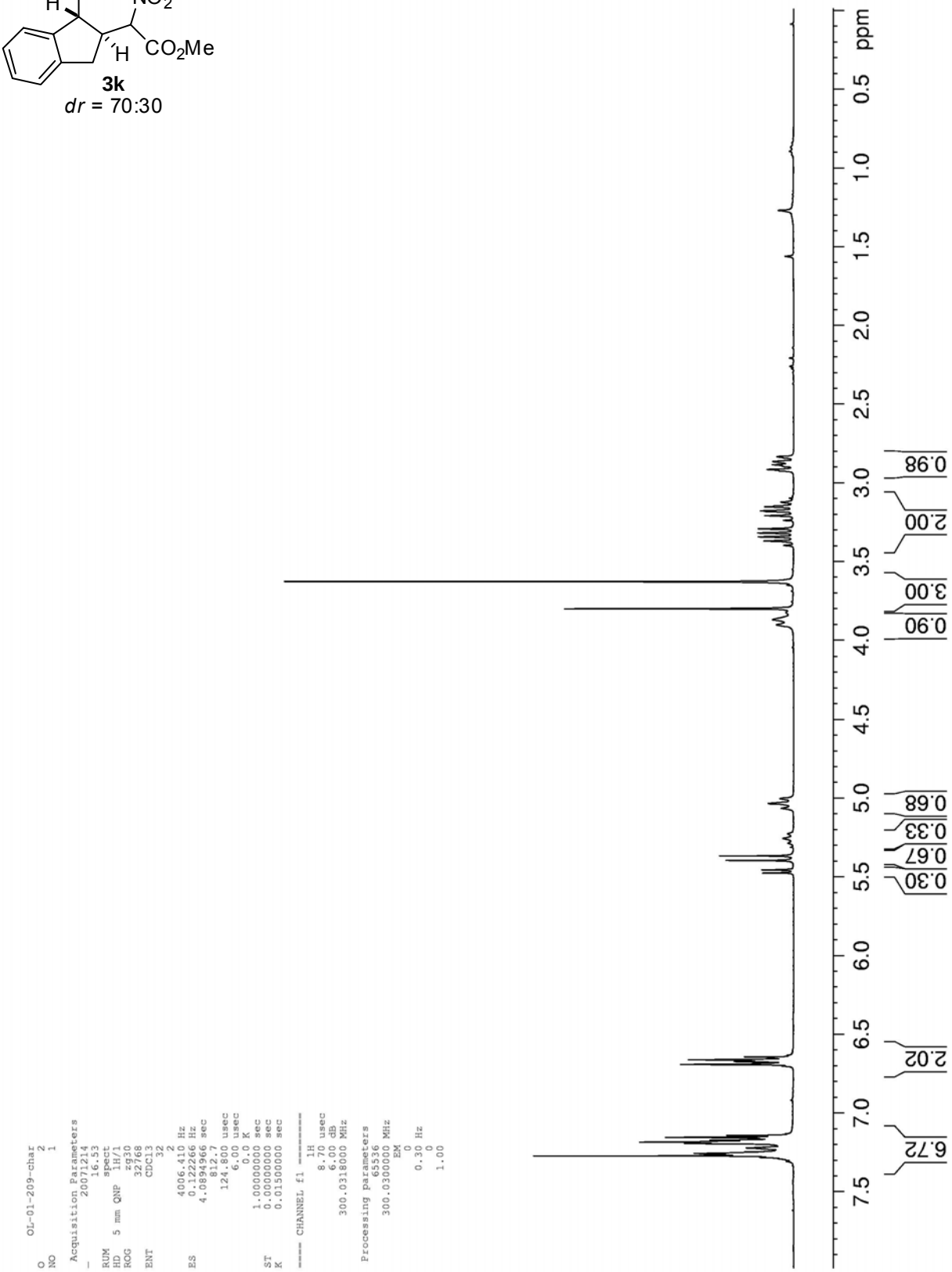
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 dB
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz

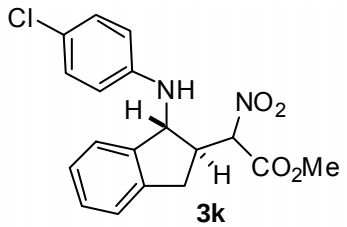
Processing parameters
131072
75.4425411 MHz
EM
0
1.00 Hz
0
1.40


Cl
NH
H
NO2
H
CO2Me

3k

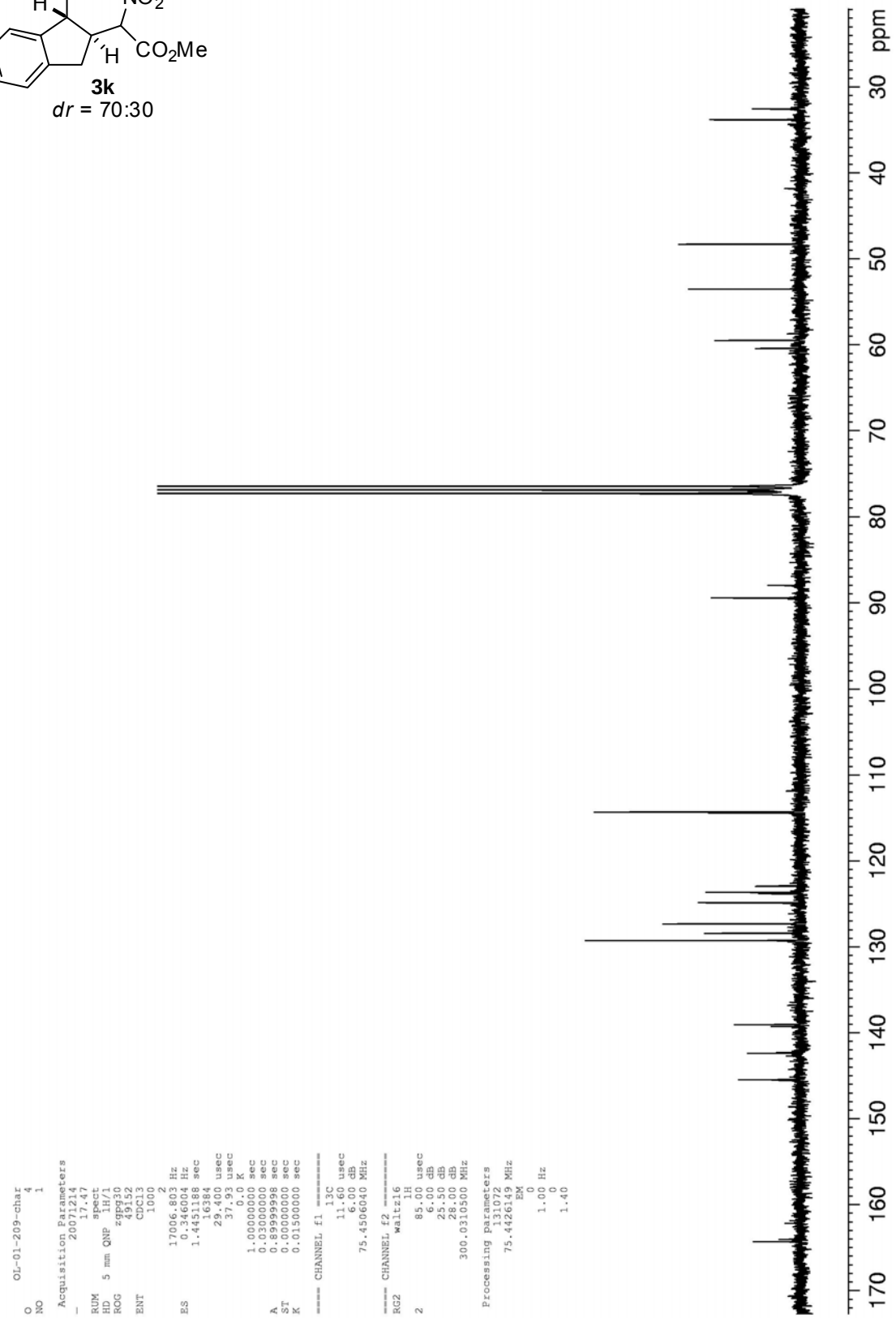
dr = 70:30


ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
6.72
2.02
0.30
0.67
0.33
0.68
0.90
3.00
2.00
0.98



3k

dr = 70:30



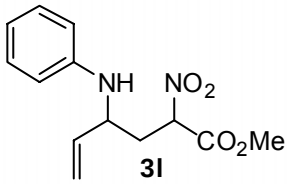
OL-01-209-char
O 4
NO 1

Acquisition Parameters
20071214
17.47
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
1000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec

===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506040 MHz

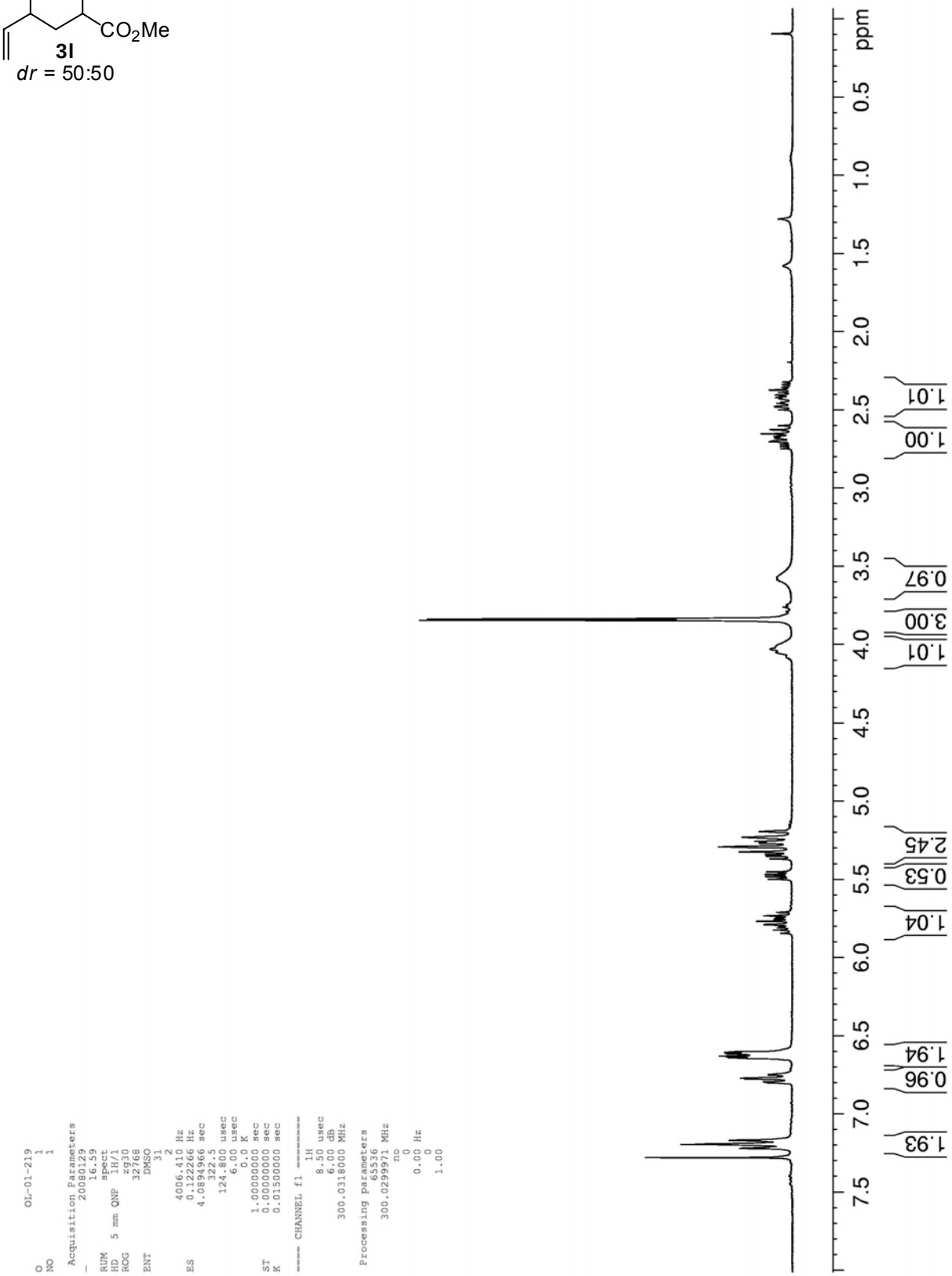
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 dB
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz

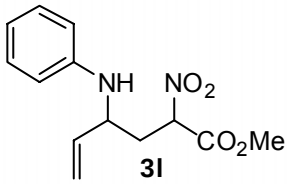
Processing parameters
131072
75.4426149 MHz
EM
0
1.00 Hz
0
1.40


NH
NO2
CO2Me

3l

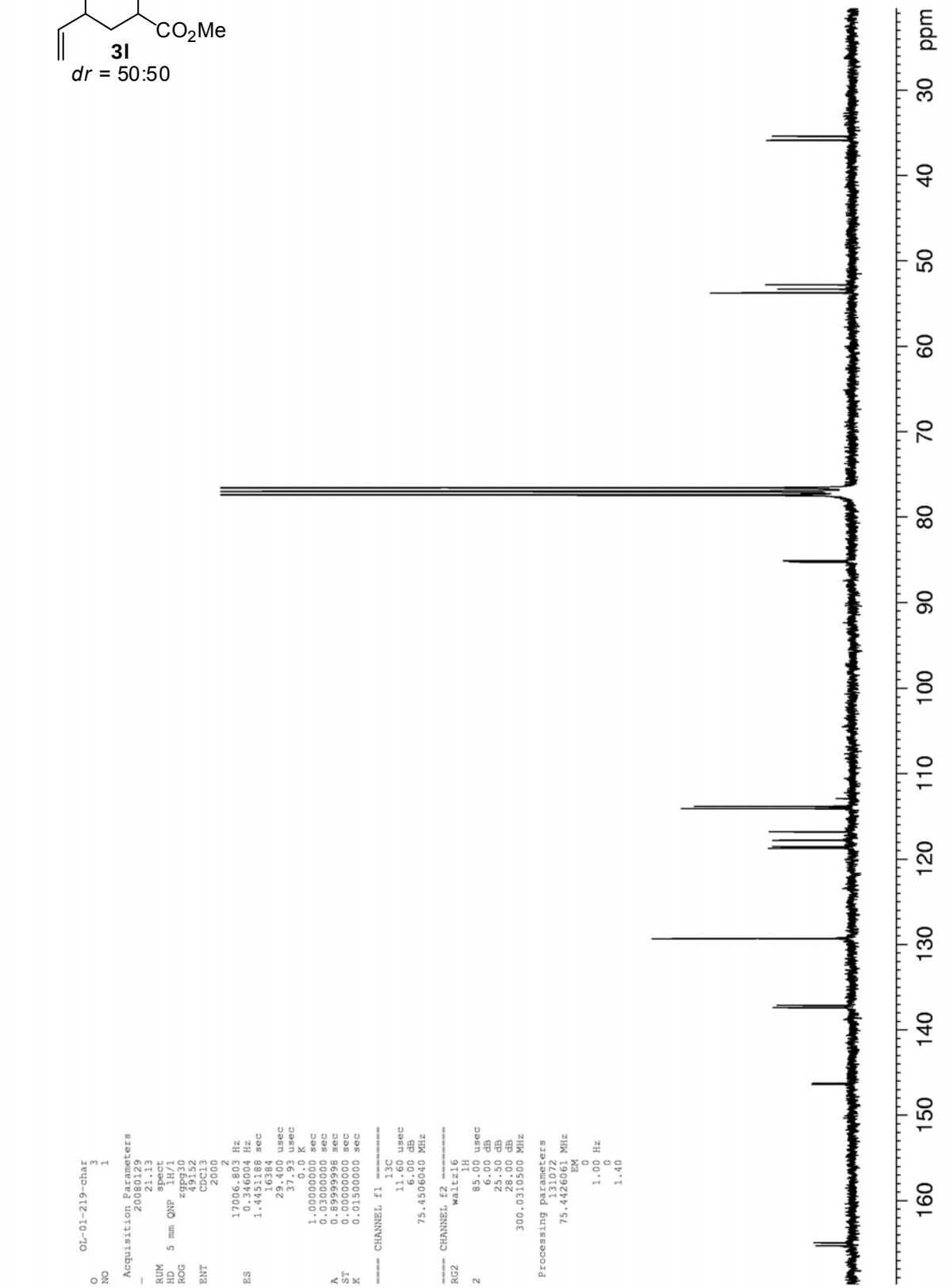
dr = 50:50

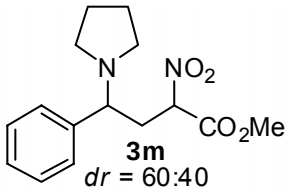

ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
1.93
0.96
1.94
1.04
0.53
2.45
1.01
3.00
0.97
1.00
1.01


NH
NO2
CO2Me

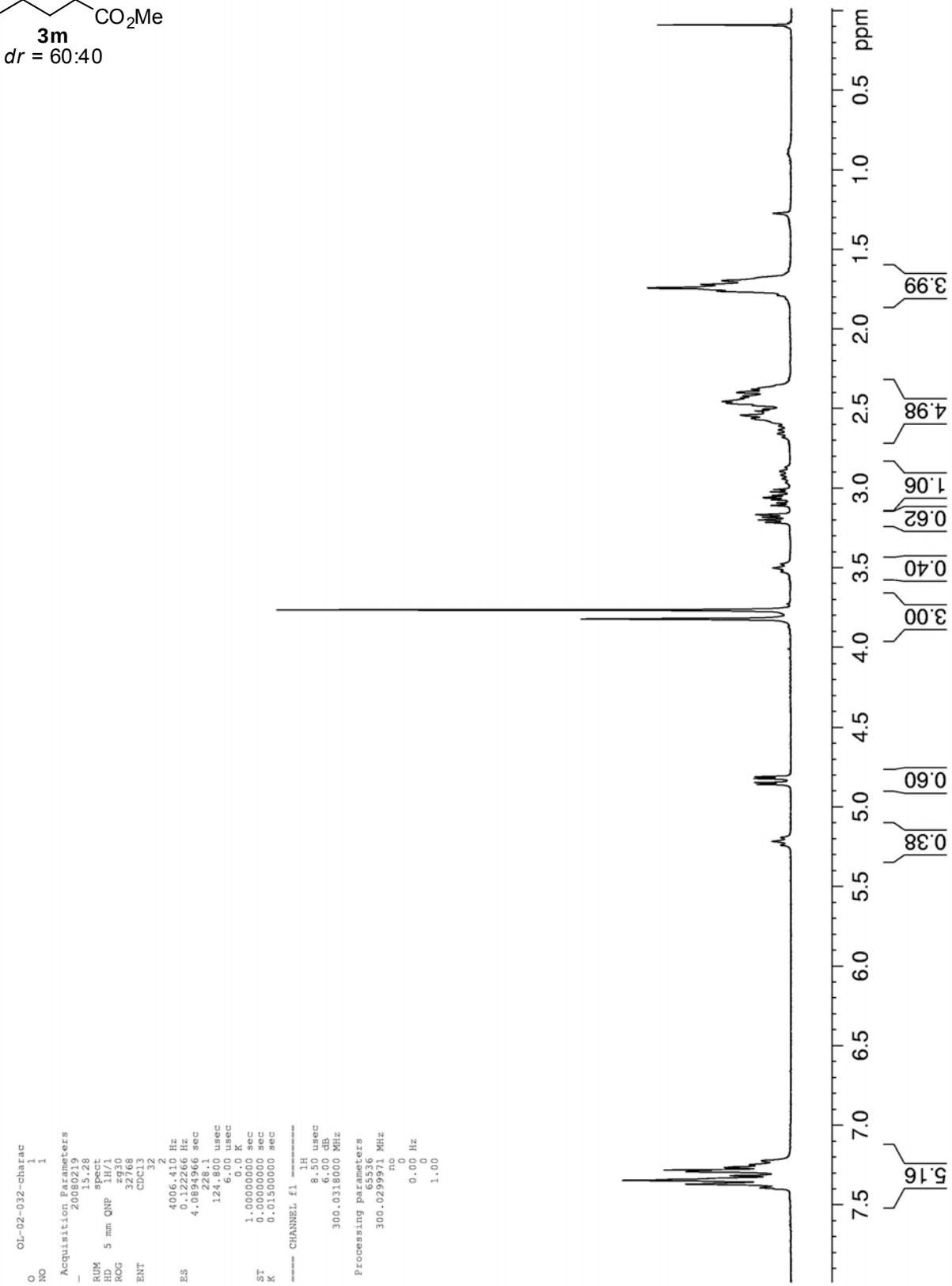
3l

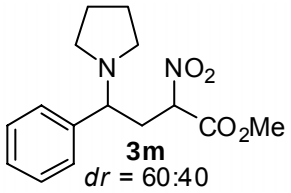
dr = 50:50


OL-01-219-char
O 3
NO 1
Acquisition Parameters
20080129
21.13
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
2000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec
===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506040 MHz
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 dB
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4426061 MHz
EM
0
1.00 Hz
0
1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

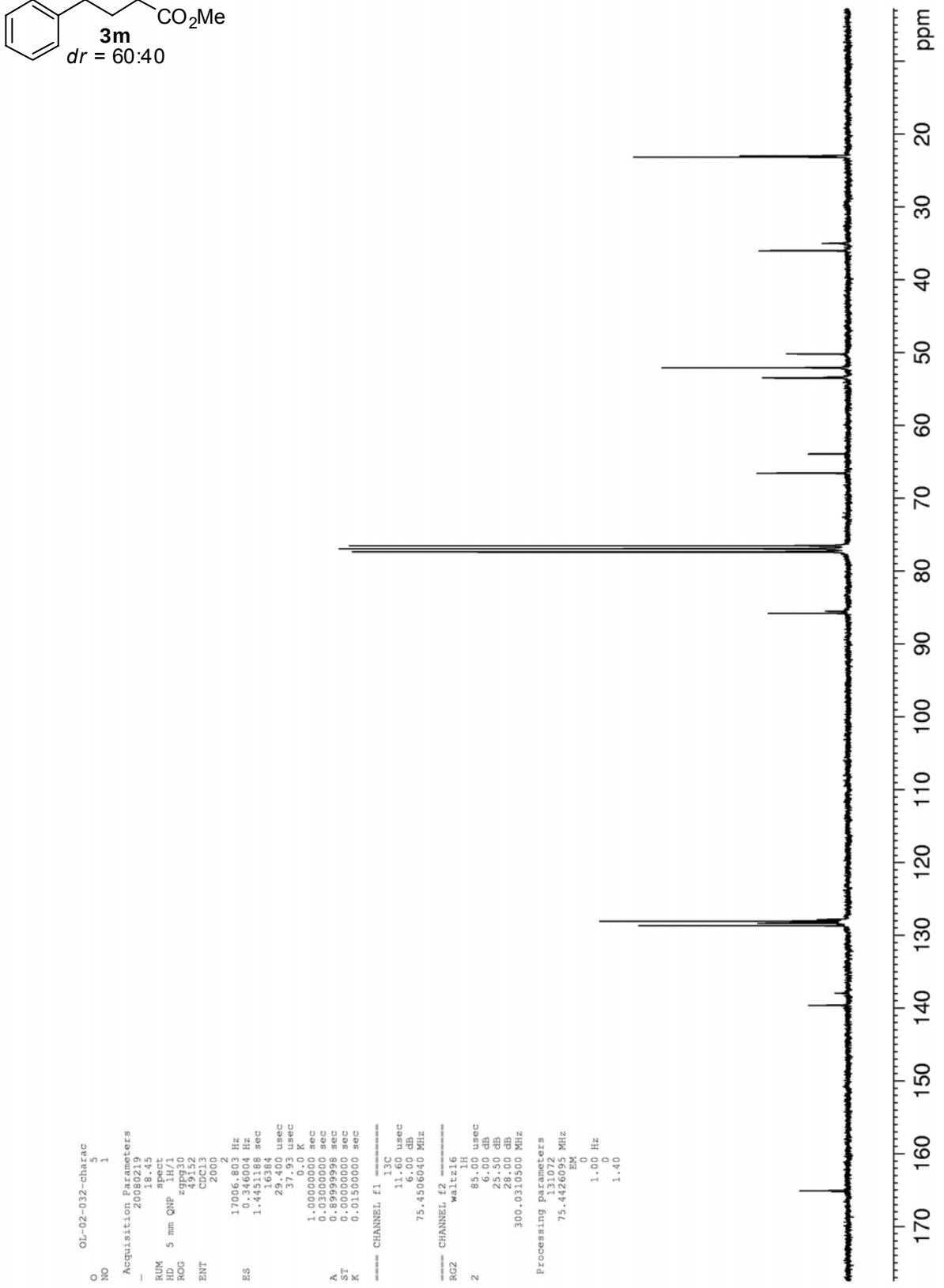

N
NO2
CO2Me

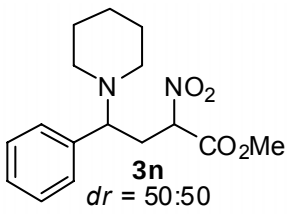
3m
dr = 60:40


ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
3.99
4.98
1.06
0.62
0.40
3.00
0.60
0.38
5.16

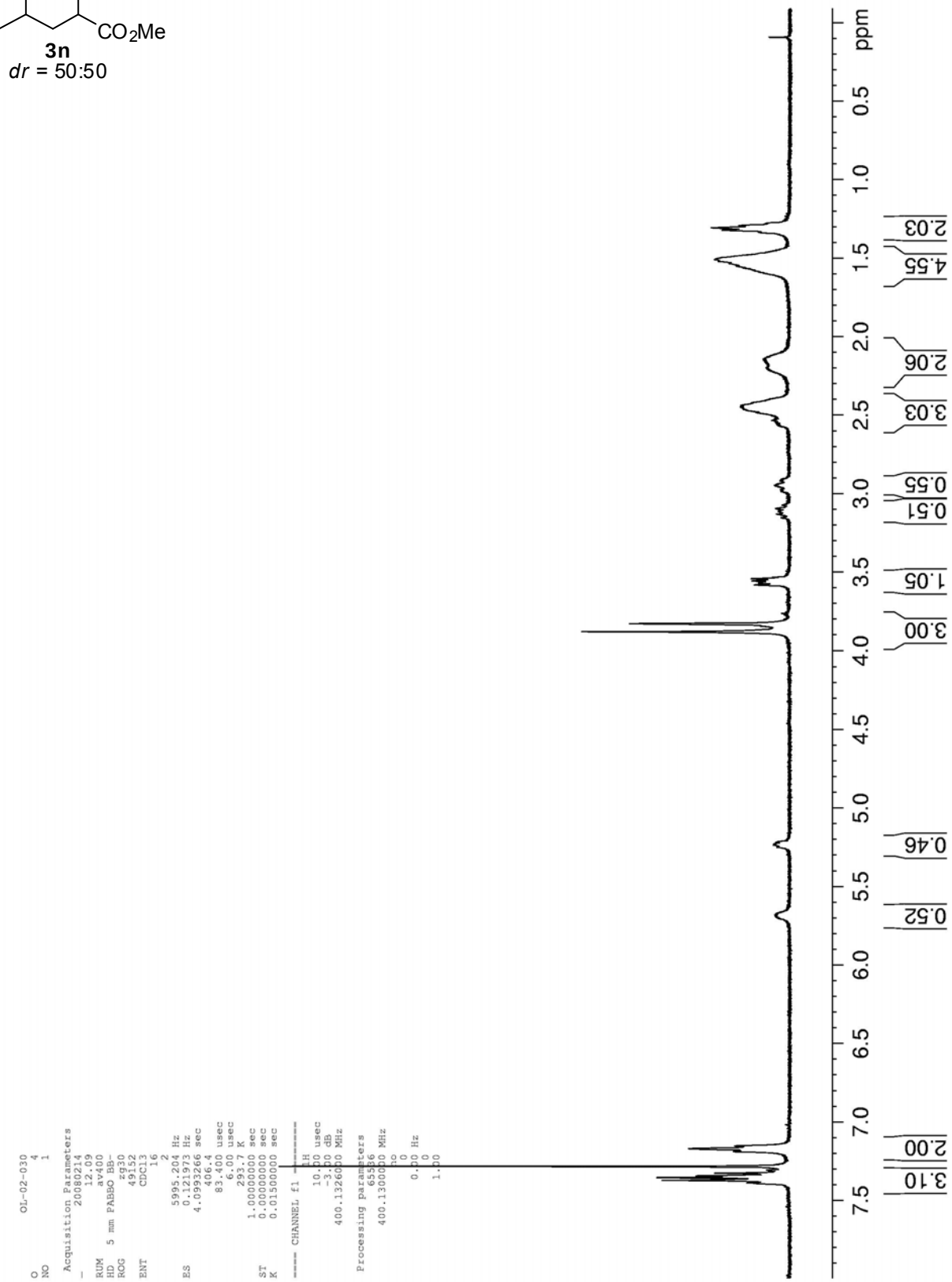

N
NO2
CO2Me

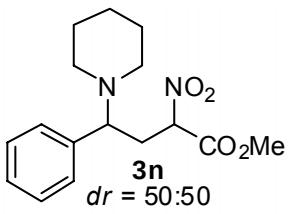
3m
dr = 60:40


OL-02-032-charac
5
1
Acquisition Parameters
20080219
18.45
spect
5 mm QNP 1H/1
zgpg30
49152
CDCl3
2000
2
17006.803 Hz
0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
0.89999998 sec
0.00000000 sec
0.01500000 sec
CHANNEL f1
13C
11.60 usec
6.00 dB
75.4506040 MHz
CHANNEL f2
waltz16
1H
85.00 dB
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4426095 MHz
EM
0
1.00 Hz
0
1.40
ppm

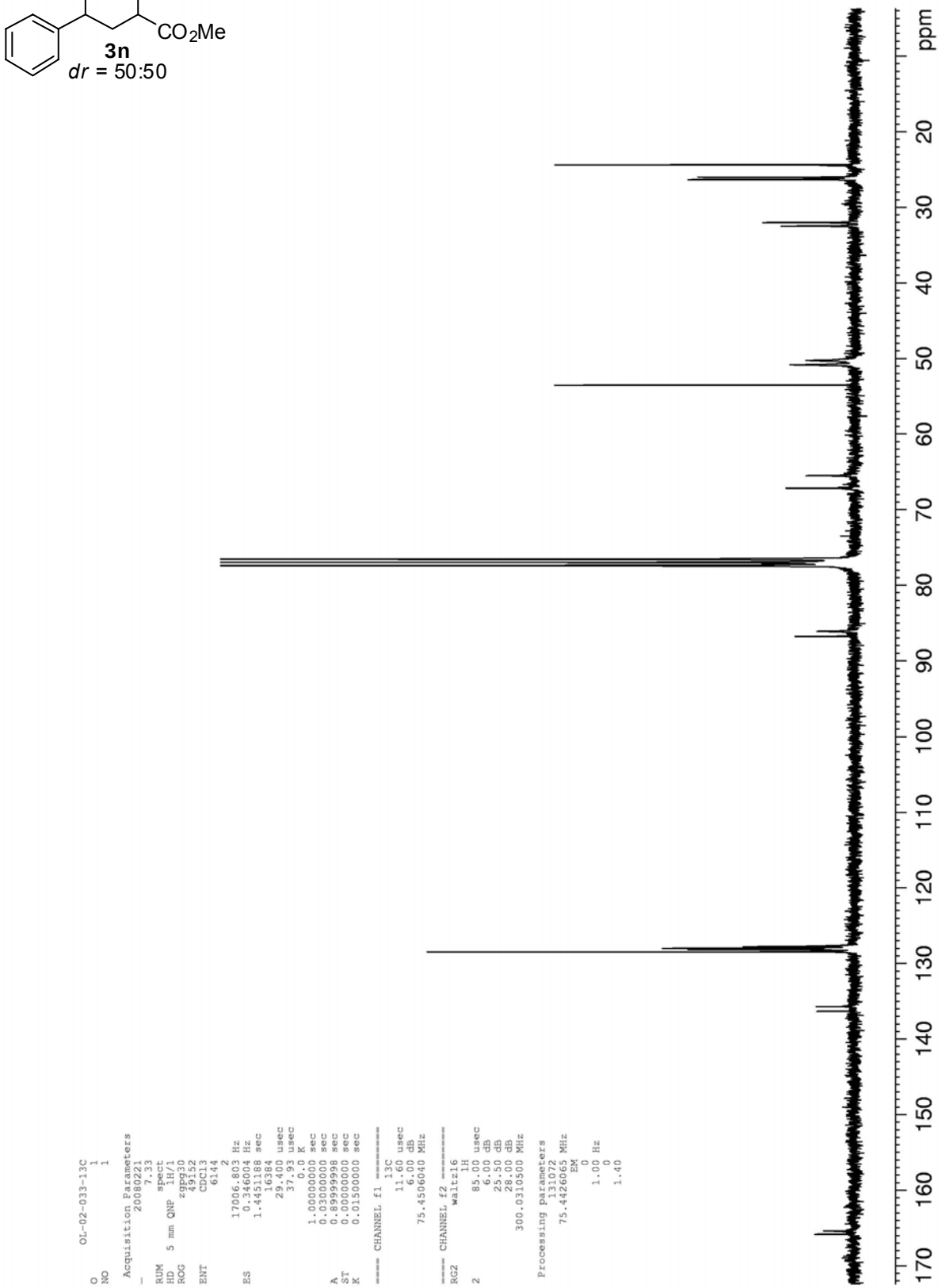

N
NO2
CO2Me

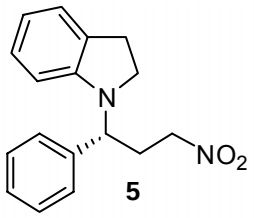
3n
dr = 50:50


ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
3.10
2.00
0.52
0.46
3.00
1.05
0.51
0.55
3.03
2.06
4.55
2.03

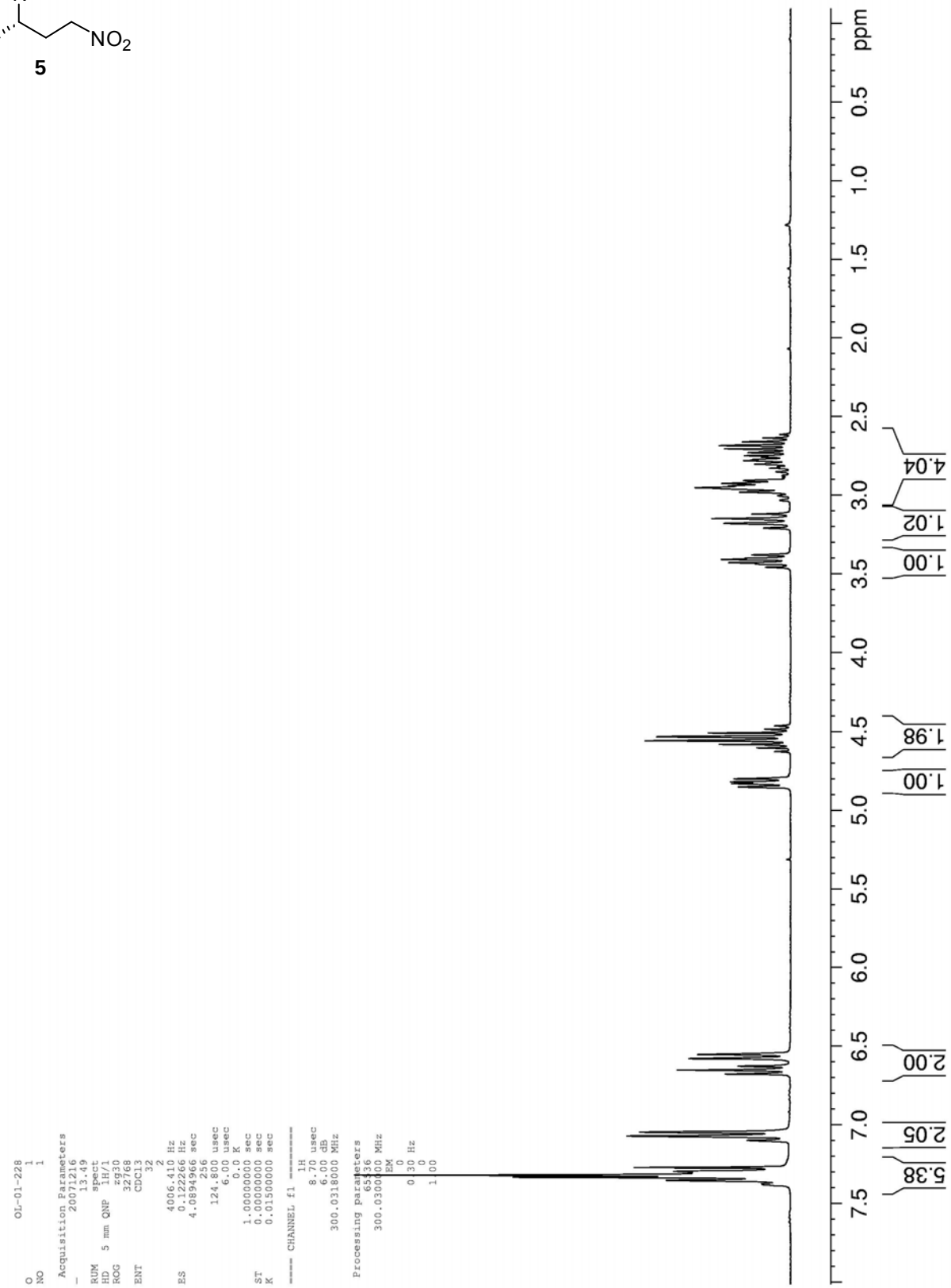

N
NO2
CO2Me

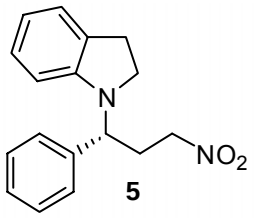
3n
dr = 50:50


ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

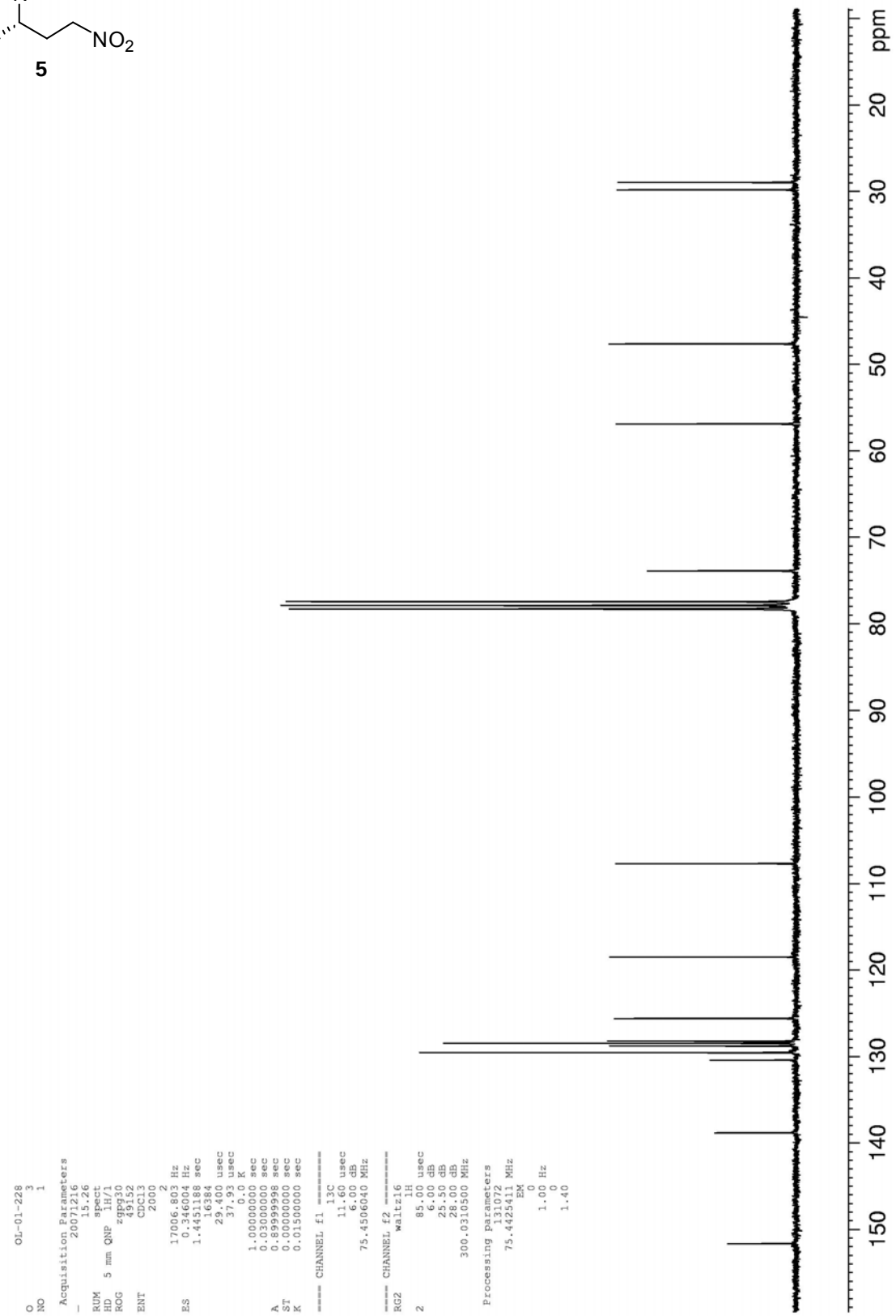

N
NO2

5

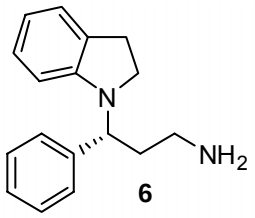

OL-01-228
1
NO
1
Acquisition Parameters
20071216
13.49
spect
RUM
HD 5 mm QNP 1H/1
ROG zg30
32768
ENT CDCl3
32
2
4006.410 Hz
0.122266 Hz
ES 4.0894966 sec
256
124.800 usec
6.00 usec
0.0 K
1.00000000 sec
ST 0.00000000 sec
K 0.01500000 sec
===== CHANNEL f1 =====
1H
8.70 usec
6.00 dB
300.0318000 MHz
Processing parameters
65536
300.0300000 MHz
EM
0.0
0.30 Hz
0
1.00
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.38
2.05
2.00
1.00
1.98
1.00
1.02
4.04



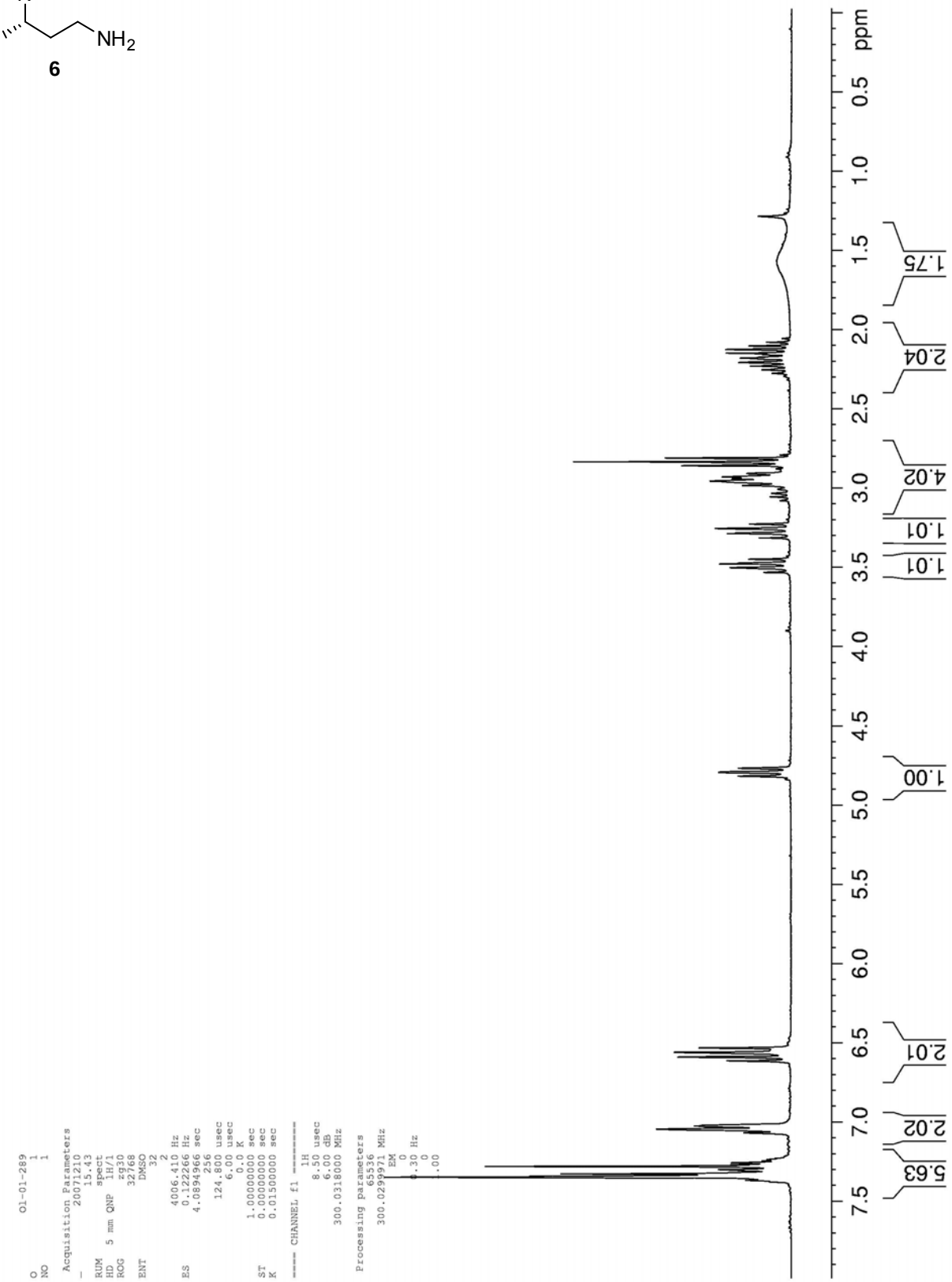
5

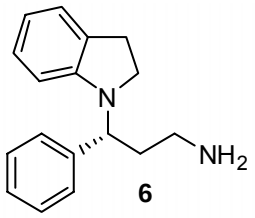



```
        OL-01-228
O             3
NO            1

  Acquisition Parameters
_        20071216
              15.26
RUM           spect
HD  5 mm QNP  1H/1
ROG           zgpg30
              49152
ENT           CDCl3
               2000
                  2
          17006.803 Hz
ES            0.346004 Hz
          1.44451188 sec
                16384
           29.400 usec
           37.93 usec
              0.0 K
          1.00000000 sec
          0.03000000 sec
A         0.89999998 sec
ST        0.00000000 sec
K         0.01500000 sec

===== CHANNEL f1 ========
                  13C
           11.60 usec
               6.00 dB
          75.4506040 MHz
===== CHANNEL f2 ========
RG2           waltz16
                   1H
2               85.00 dB
                 6.00 dB
                25.50 dB
                28.00 dB
         300.0310500 MHz

  Processing parameters
            131072
          75.4425411 MHz
                  EM
                   0
                1.00 Hz
                   0
                1.40
```

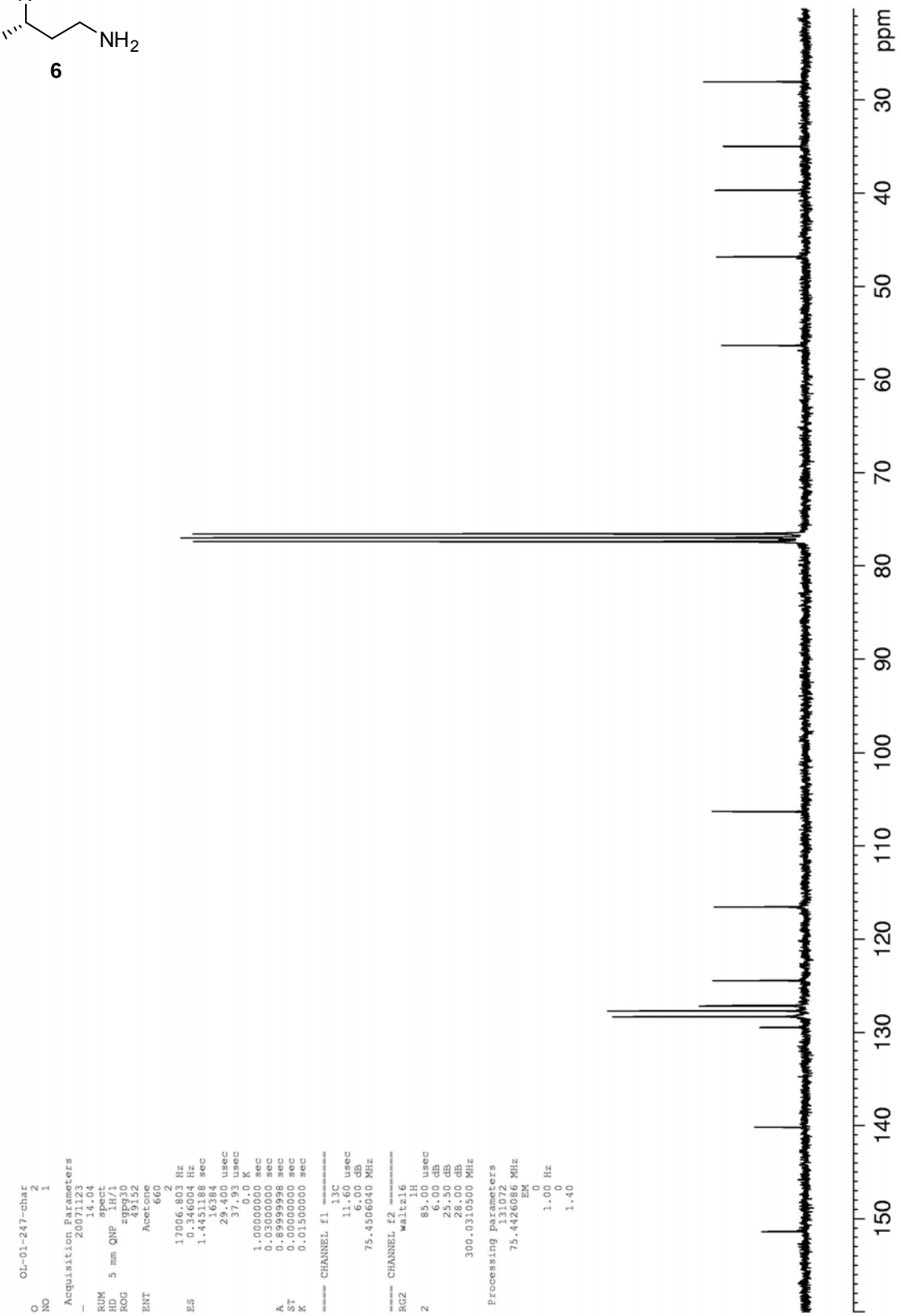

N
NH2

6

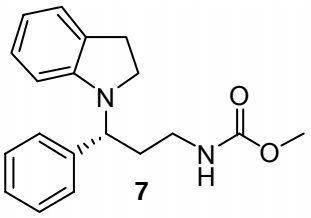

ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.63
2.02
2.01
1.00
1.01
1.01
4.02
2.04
1.75



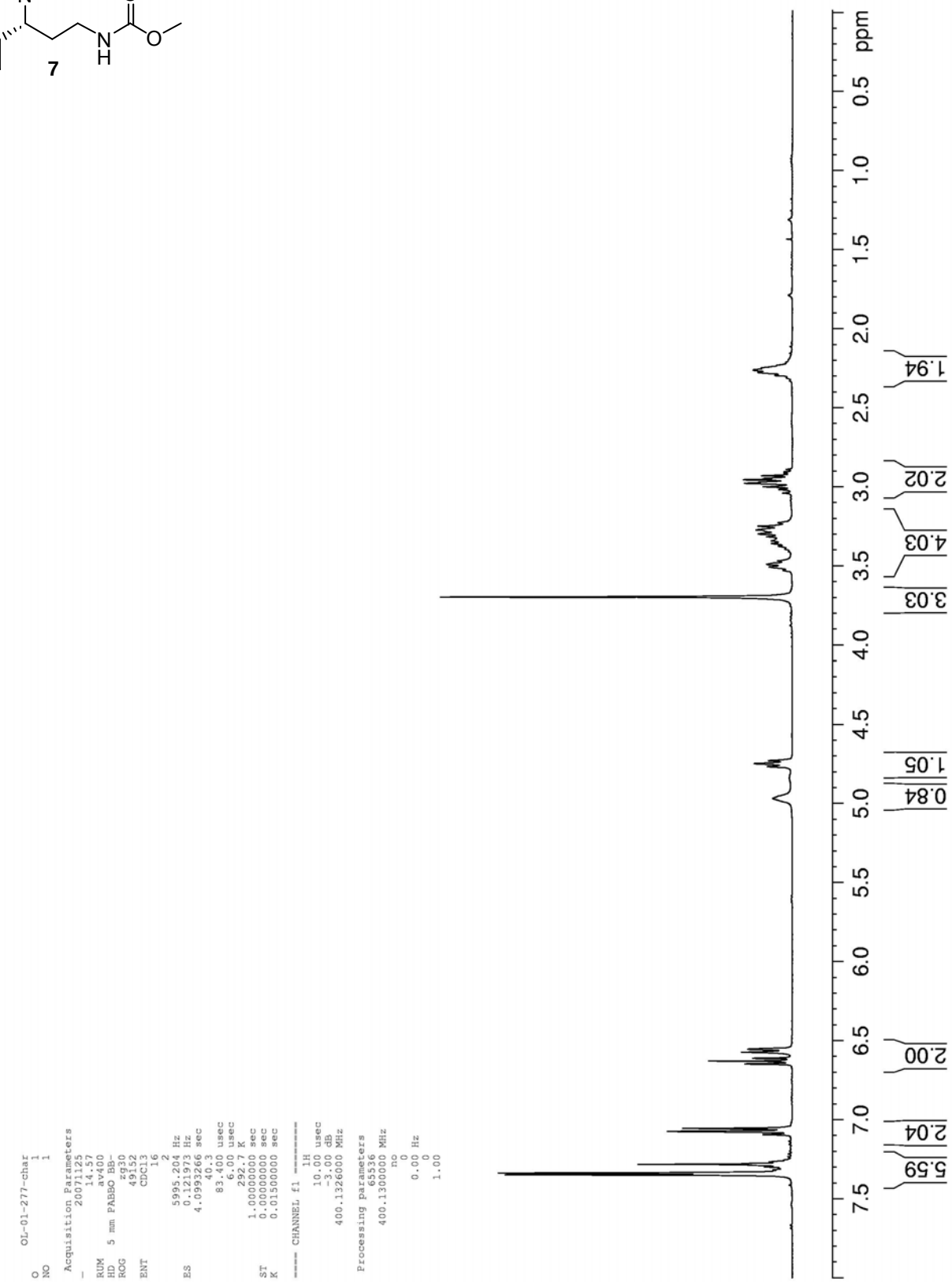
6

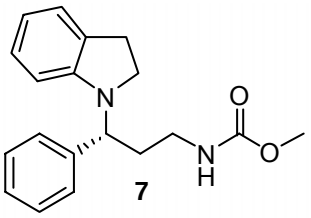



```
OL-01-247-char
O                2
NO               1

Acquisition Parameters
_            20071123
                14.04
                spect
RUM
HD  5 mm QNP  1H/1
ROG             zgpg30
                49152
ENT           Acetone
                  660
                    2
            17006.803 Hz
ES            0.346004 Hz
              1.4451188 sec
                16384
                29.400 usec
                37.93 usec
                  0.0 K
              1.00000000 sec
              0.03000000 sec
A             0.89999998 sec
ST            0.00000000 sec
K             0.01500000 sec

===== CHANNEL f1 ========
                  13C
                11.60 usec
                 6.00 dB
              75.4506040 MHz
===== CHANNEL f2 ========
RG2           waltz16
                  1H
2                85.00 usec
                 6.00 dB
                25.50 dB
                28.00 dB
             300.0310500 MHz

Processing parameters
              131072
              75.4426086 MHz
                  EM
                   0
                1.00 Hz
                   0
                1.40
```

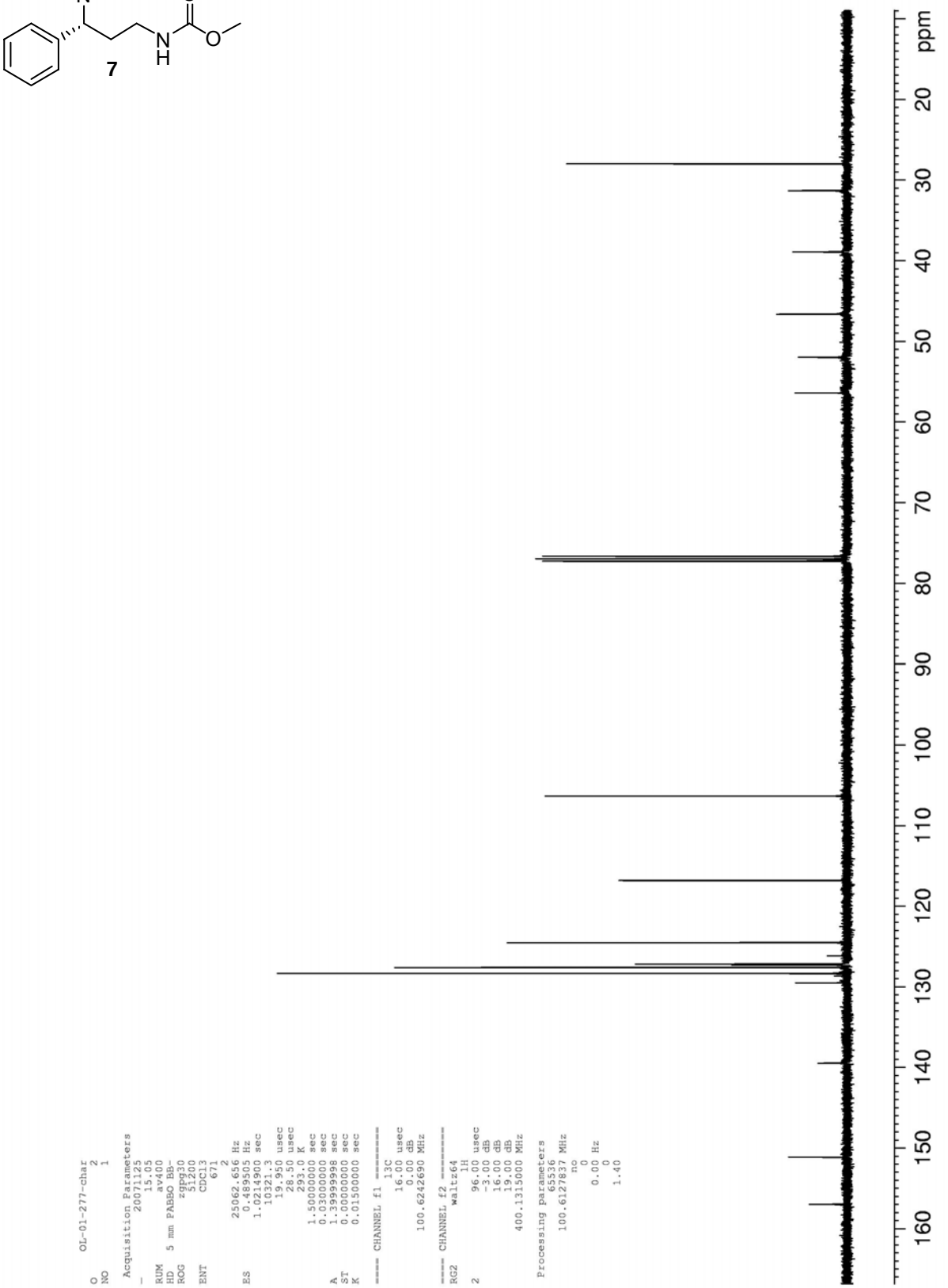

N
O
N
H
O

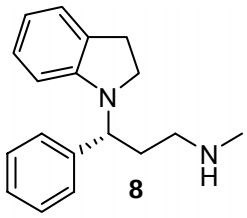
7


OL-01-277-char
Acquisition Parameters
5 mm PABBO BB-
CHANNEL f1
Processing parameters
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.59
2.04
2.00
0.84
1.05
3.03
4.03
2.02
1.94

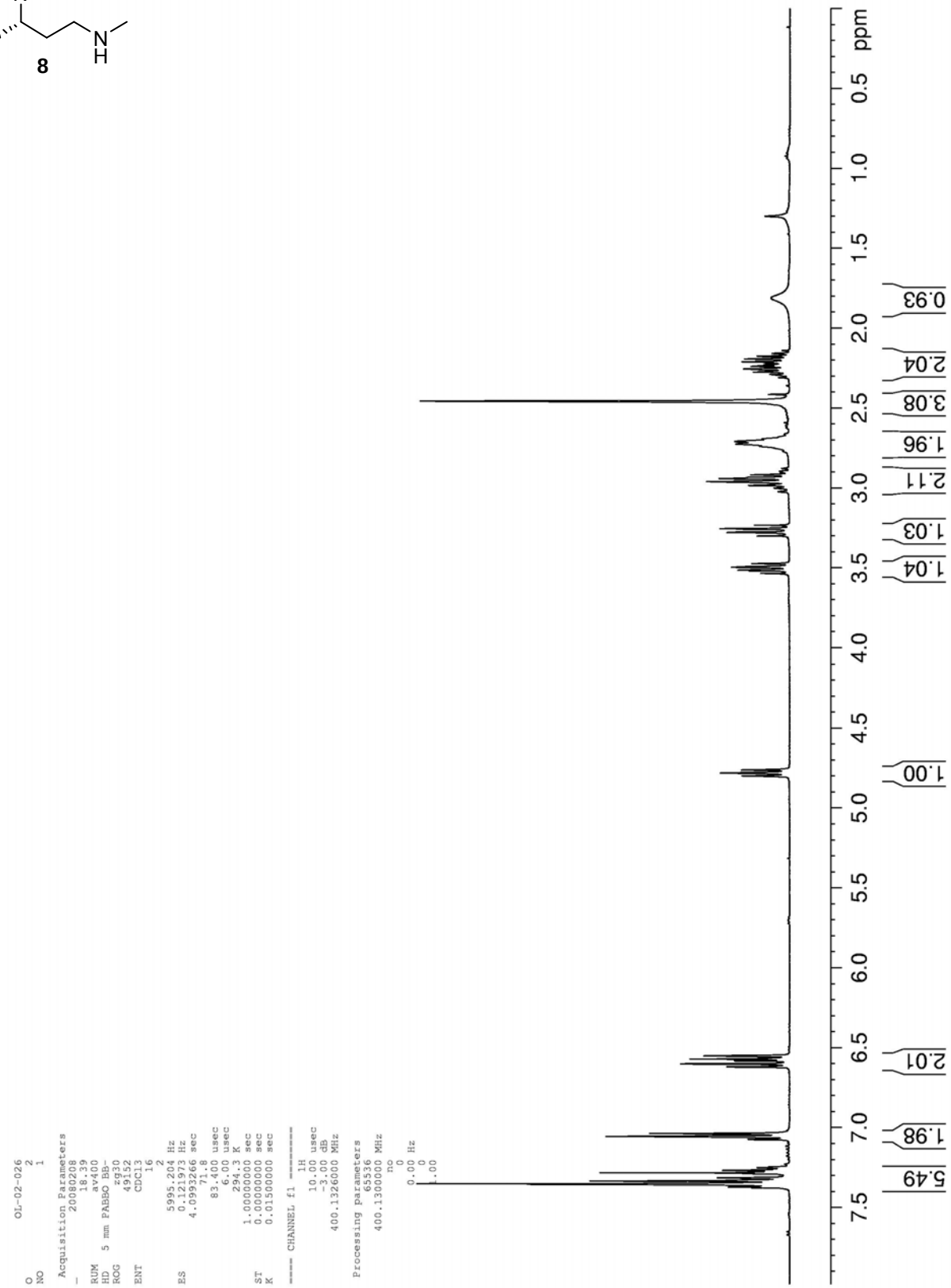

N
O
N
H
O

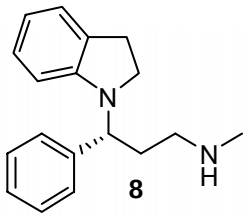
7


OL-01-277-char
2
1
Acquisition Parameters
20071125
15.05
av400
5 mm PABBO BB-
zgpg30
51200
CDCl3
671
2
25062.656 Hz
0.489505 Hz
1.0214900 sec
10321.3
19.950 usec
28.50 usec
293.0 K
1.50000000 sec
0.03000000 sec
1.39999998 sec
0.00000000 sec
0.01500000 sec
CHANNEL f1
13C
16.00 usec
0.00 dB
100.6242690 MHz
CHANNEL f2
waltz64
1H
96.00 usec
-3.00 dB
16.00 dB
19.00 dB
400.1315000 MHz
Processing parameters
65536
100.6127837 MHz
no
0
0.00 Hz
0
1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

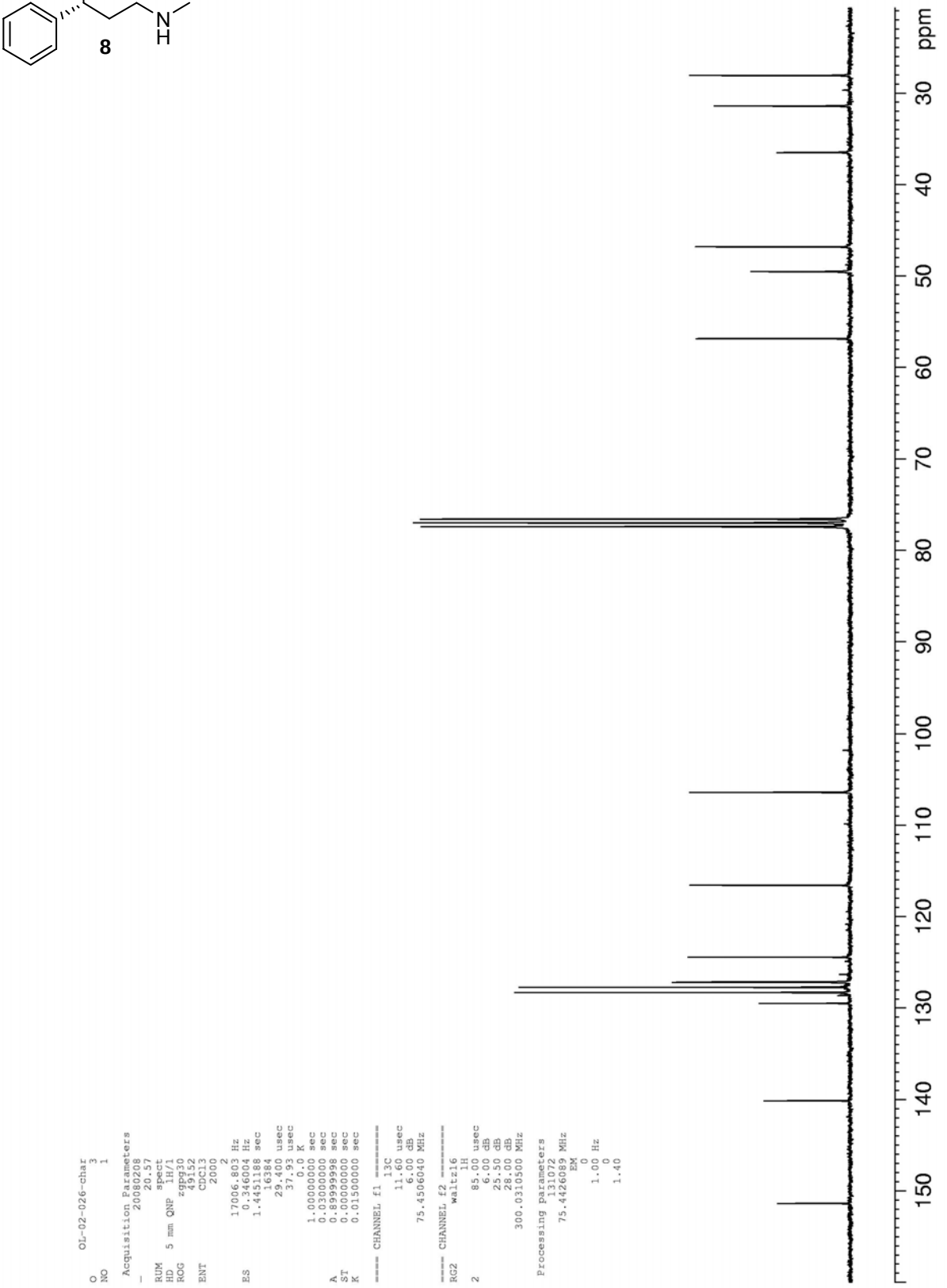


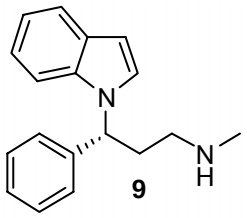
8


OL-02-026
2
NO 1
Acquisition Parameters
20080208
18.39
av400
RUM
HD 5 mm PABBO BB-
ROG zg30
49152
ENT CDCl3
16
2
5995.204 Hz
ES 0.121973 Hz
4.0993266 sec
71.8
83.400 usec
6.00 usec
294.3 K
1.00000000 sec
ST 0.00000000 sec
K 0.01500000 sec
===== CHANNEL f1 =========
1H
10.00 usec
3.00 dB
400.1326000 MHz
Processing parameters
65536
400.1300000 MHz
no
0
0.00 Hz
1.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.49
1.98
2.01
1.00
1.04
1.03
2.11
1.96
3.08
2.04
0.93

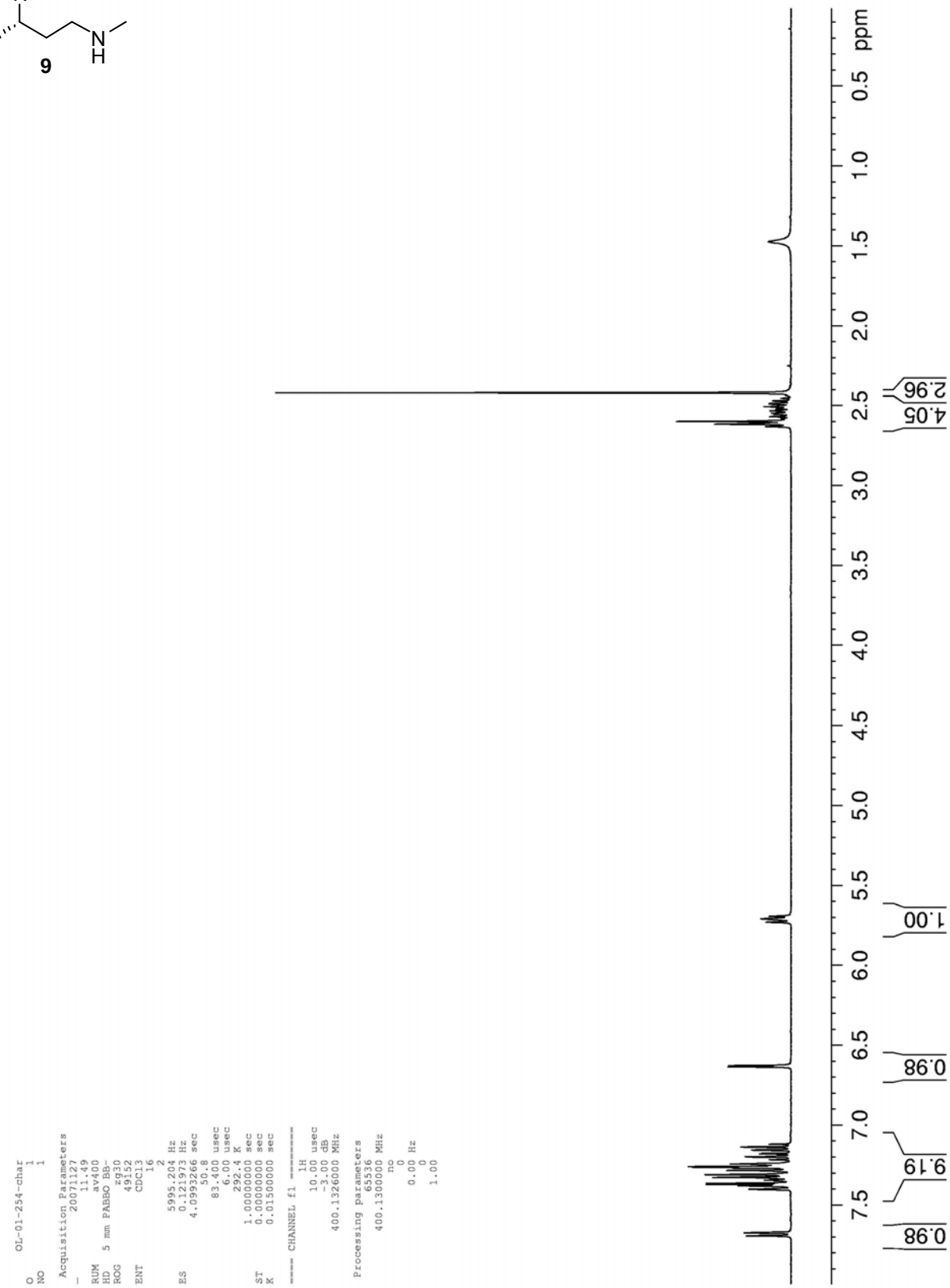


8

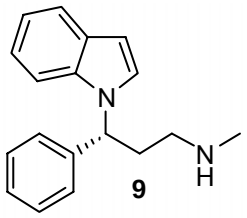

OL-02-026-char
O 3
NO 1
Acquisition Parameters
20080208
20.57
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
ENT CDCl3
2000
2
17006.803 Hz
ES 0.346004 Hz
1.44451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec
==== CHANNEL f1 ========
13C
11.60 usec
6.00 dB
75.4506040 MHz
==== CHANNEL f2 ========
RG2 waltz16
1H
2 85.00 usec
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4426089 MHz
EM
0
1.00 Hz
0
1.40
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm



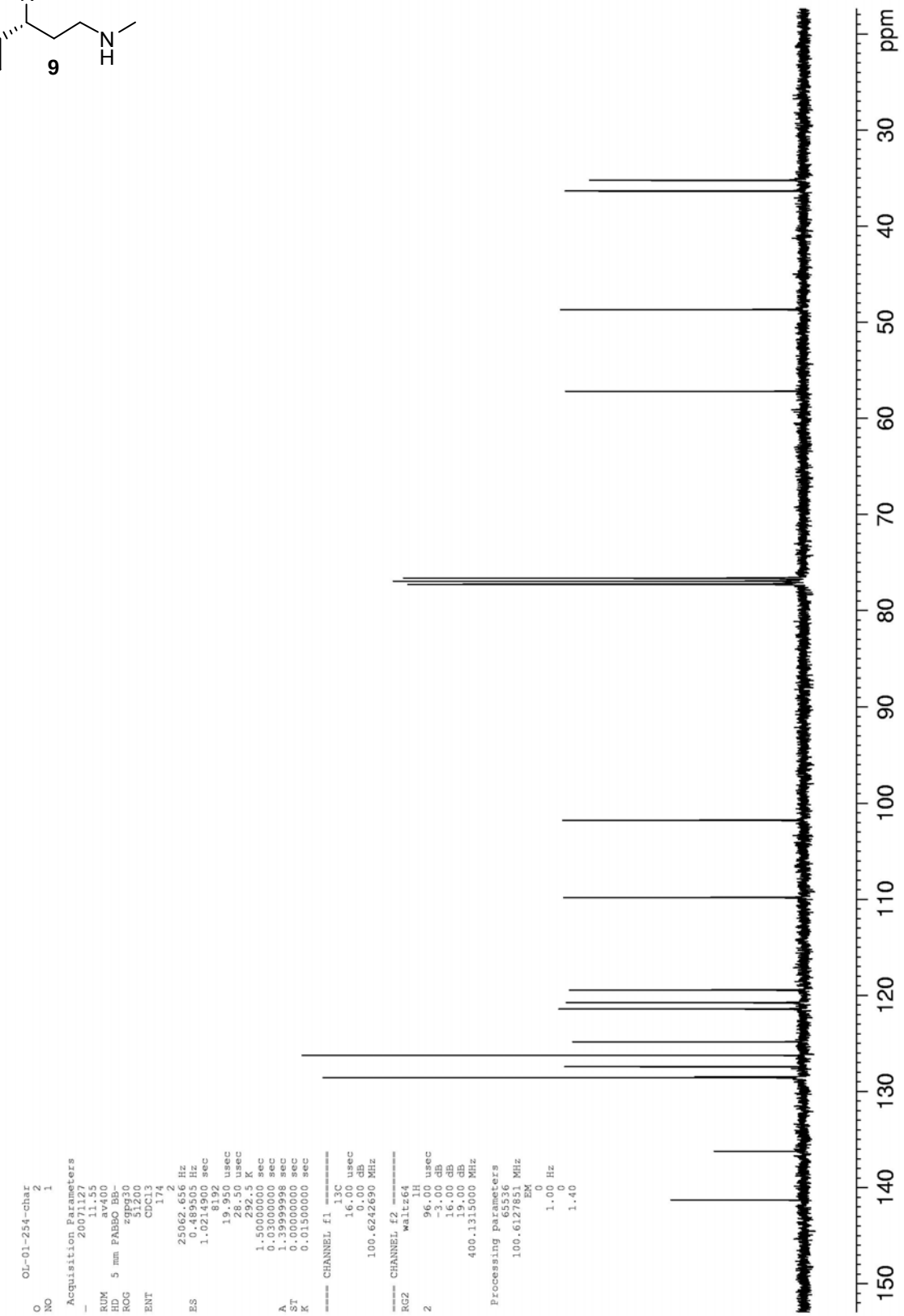
9

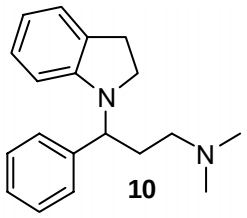



```
OL-01-254-char
O                1
NO               1

Acquisition Parameters
_              20071127
                  11.49
RUM               av400
HD  5 mm PABBO BB-
ROG                zg30
                  49152
ENT               CDCl3
                     16
                      2
                5995.204 Hz
ES              0.121973 Hz
                4.0993266 sec
                     50.8
                   83.400 usec
                    6.00 usec
                   292.4 K
                1.00000000 sec
ST              0.00000000 sec
K               0.01500000 sec

====== CHANNEL f1 ========
                      1H
                   10.00 usec
                    3.00 dB
                400.1326000 MHz

Processing parameters
                    65536
                400.1300000 MHz
                      no
                       0
                    0.00 Hz
                       0
                    1.00
```

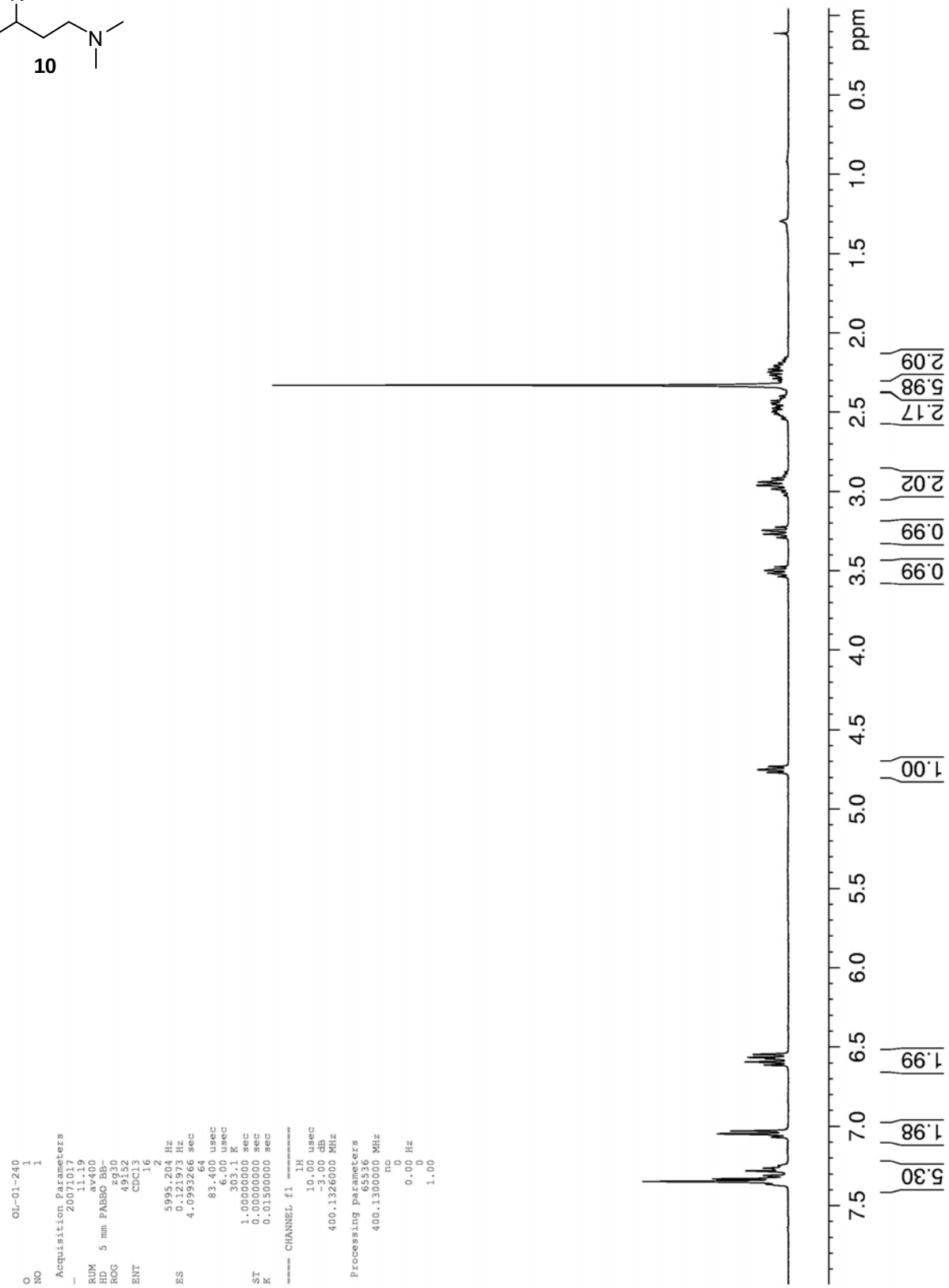


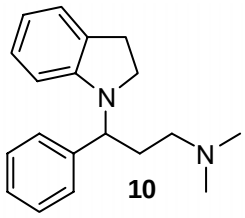
9


OL-01-254-char
2
1
Acquisition Parameters
20071127
11.55
av400
5 mm PABBO BB-
zgpg30
51200
CDCl3
174
2
25062.656 Hz
0.489505 Hz
1.0214900 sec
8192
19.950 usec
28.50 usec
292.5 K
1.50000000 sec
0.03000000 sec
1.39999998 sec
0.00000000 sec
0.01500000 sec
CHANNEL f1
13C
16.00 usec
0.00 dB
100.6242690 MHz
CHANNEL f2
waltz64
1H
96.00 usec
-3.00 dB
16.00 dB
19.00 dB
400.1315000 MHz
Processing parameters
65536
100.6127851 MHz
EM
0
1.00 Hz
0
1.40
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

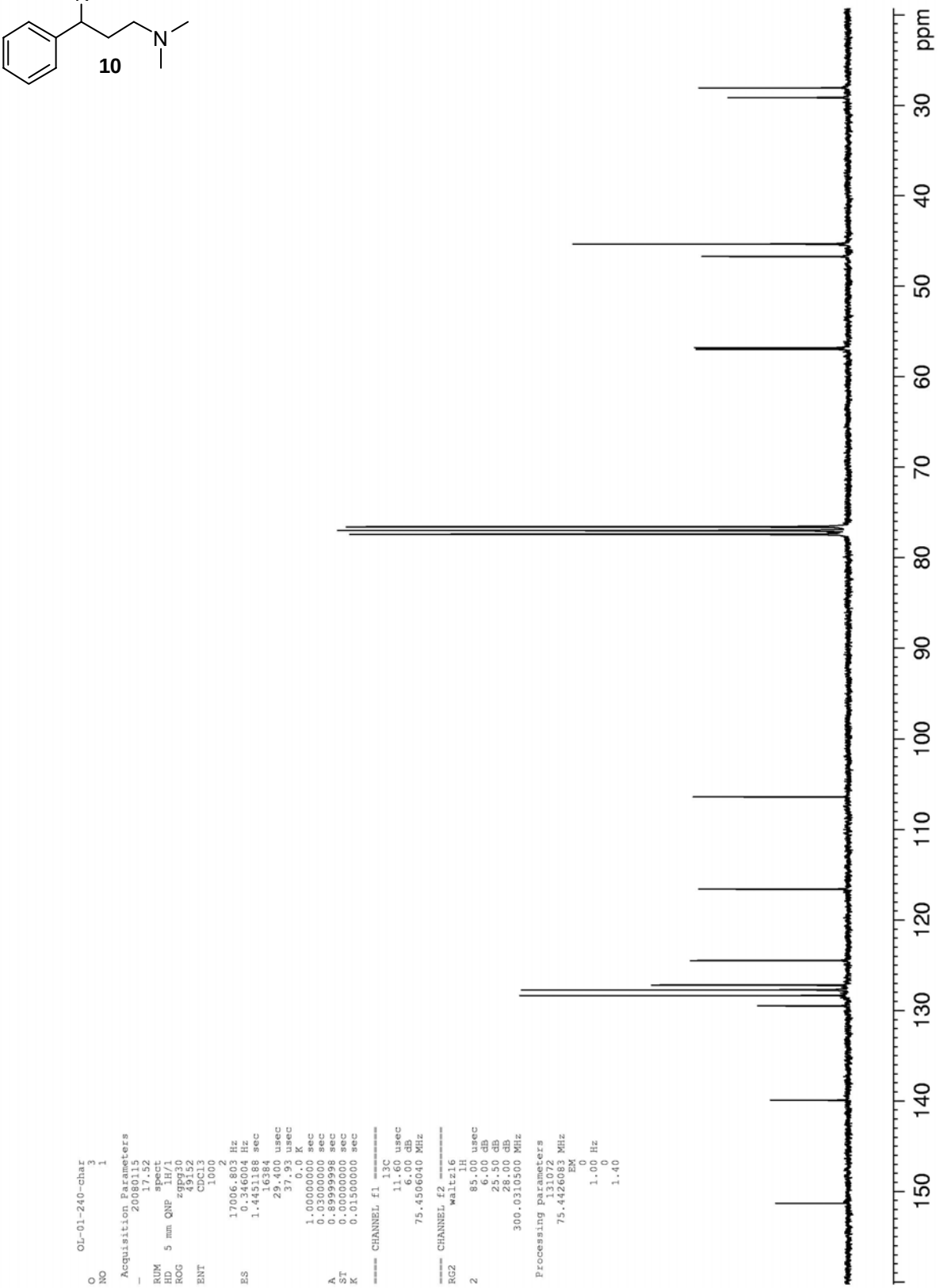


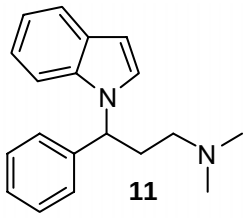
10


ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
5.30
1.98
1.99
1.00
0.99
0.99
2.02
2.17
5.98
2.09

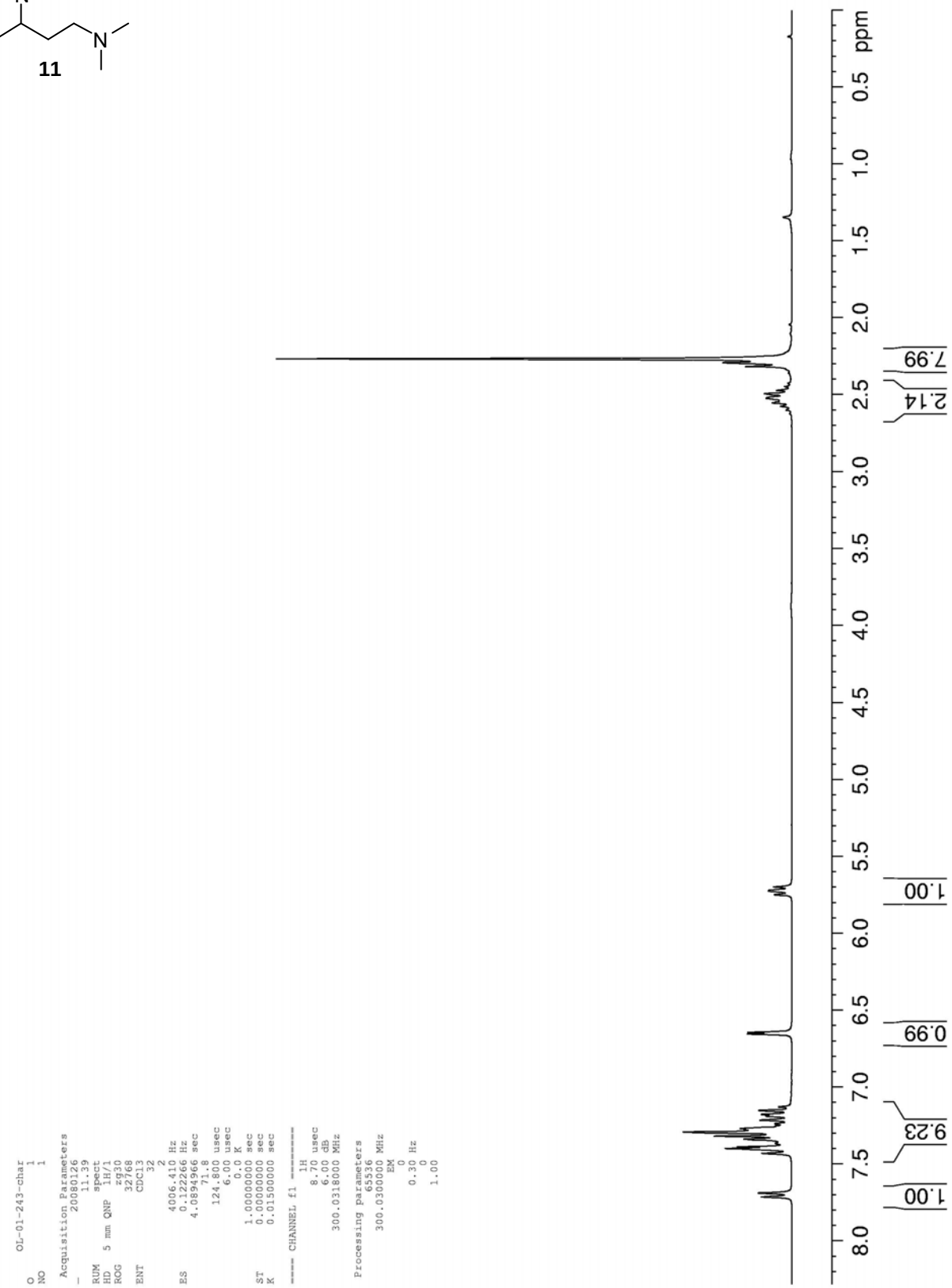


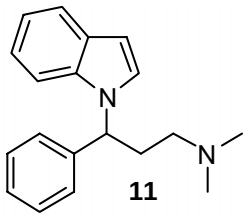
10


OL-01-240-char
O 3
NO 1
Acquisition Parameters
20080115
17.52
spect
RUM
HD 5 mm QNP 1H/1
ROG zgpg30
49152
CDCl3
1000
2
17006.803 Hz
ES 0.346004 Hz
1.4451188 sec
16384
29.400 usec
37.93 usec
0.0 K
1.00000000 sec
0.03000000 sec
A 0.89999998 sec
ST 0.00000000 sec
K 0.01500000 sec
===== CHANNEL f1 =====
13C
11.60 usec
6.00 dB
75.4506040 MHz
===== CHANNEL f2 =====
RG2 waltz16
1H
2 85.00 usec
6.00 dB
25.50 dB
28.00 dB
300.0310500 MHz
Processing parameters
131072
75.4426083 MHz
EM
0
1.00 Hz
0
1.40
150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

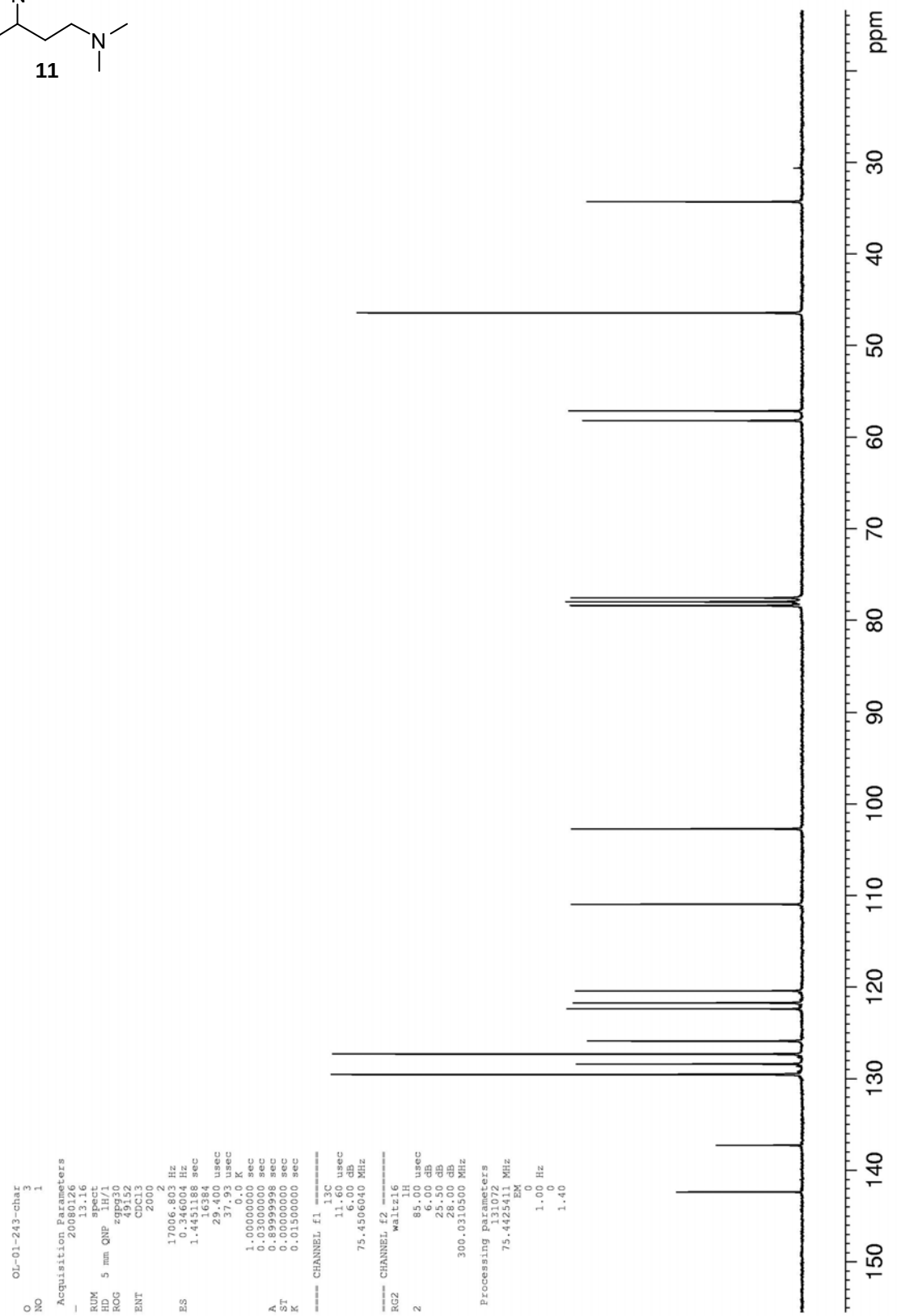


11


OL-01-243-char
1
1
Acquisition Parameters
20080126
11.39
spect
1H/1
zg30
32768
CDCl3
32
2
4006.410 Hz
0.122266 Hz
4.0894966 sec
71.8
124.800 usec
6.00 usec
0.0 K
1.00000000 sec
0.00000000 sec
0.01500000 sec
CHANNEL f1
1H
8.70 usec
6.00 dB
300.0318000 MHz
Processing parameters
65536
300.0300000 MHz
EM
0.0
0.30 Hz
0
1.00
ppm
1.00
9.23
0.99
1.00
2.14
7.99



11


OL-01-243-char
Acquisition Parameters
13.16
spect
5 mm QNP 1H/1
zgpg30
CDCl3
CHANNEL f1
13C
CHANNEL f2
waltz16
Processing parameters
EM
ppm